221-40323

GAS CHROMATOGRAPH

GC-14B

USER'S MANUAL

READ AND UNDERSTAND THIS MANUAL
BEFORE OPERATION. SAVE THIS MANUAL.

SHIMADZU CORPORATION
ANALYTICAL INSTRUMENTS DIVISION
KYOTO, JADAN

Information in this publication is subject to change without notice and does not represent a commitment on the part of the vendor.

Any errors or omissions which may have occurred in this publication despite the utmost care taken in its production will be corrected as soon as possible, but not necessarily immediately upon detection.

Note that Shimadzu does not have any obligation concerning the effects resulting from the application of the contents of this manual.

Shimadzu Corporation will continue to supply original Shimadzu maintenance parts associated with a given product for a period up to 10 years from the cessation of production of that product. Please be informed that after this period of time, Shimadzu Corporation cannot guarantee supply of such original maintenance parts. However, following discontinuation of a product, the period of availability of maintenance parts which have been produced on a subcontract basis is up to the discretion of the concerned Subcontracting company.

MS-DOS and MS-Windows are trademarks of the Microsoft Corporation.

Safety Precautions

This gas chromatograph is an analysis system designed to perform qualitative and quantitative analysis.

For safe operation, observe the following precautions. If not observed, the safety may be compromised.

1. Use the unit only for the purpose for which it is intended.
2. Follow the procedures, warnings and precautions described in the manual.
3. Do not disassemble or modify the unit.
4. For repairs, contact our sales office or service representative.

THREE CATEGORIES OF DANGER SYMBOLS ARE USED THROUGHOUT THE MANUAL.

WARNING	**Used for situations that may cause death or serious injury.**
CAUTION	**Used for situations that may cause slight injury or damage to the instrument.**
NOTE	**Emphasizes additional information or provides tips for easier operation.**

WARNING LABELS

WARNING

DO NOT TOUCH

High temperature injection ports, detectors and upper cover.

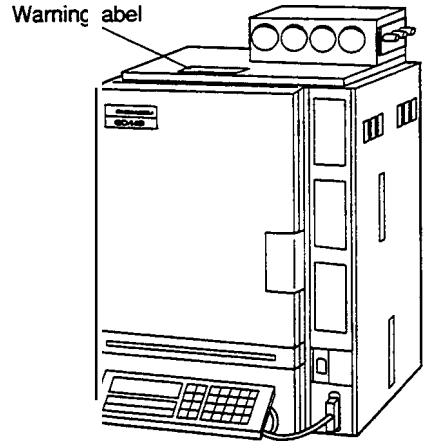


Fig. 1

WARNING

WARNING IN USING HYDROGEN

Shut off hydrogen and cap unused column fittings to prevent accumulation of hydrogen in oven **and** possible explosion.

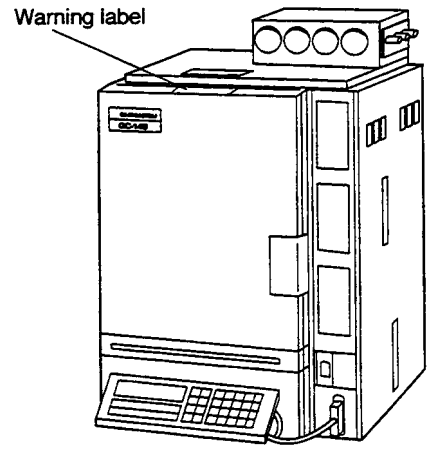


Fig. 2

WARNING

HOT AIR EXHAUST

Keep temperature sensitive materials away from opening.

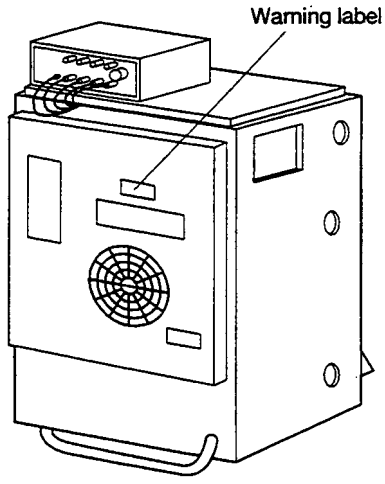


Fig. 3

WARNING

HIGH **VOLTAGE**

Disconnect power cable before removing cover. Refer servicing to qualified service personnel.

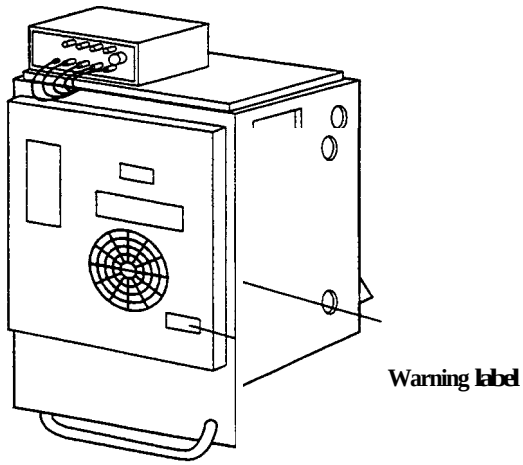


Fig. 4

WARNING

WARNINGS IN USING HYDROGEN

When hydrogen gas is in use, care should be exercised in order to prevent accident.

1. Connect gas lines correctly. Do not connect the hydrogen line to the air inlet, or hydrogen will leak excessively.
2. When the device is not in use, the main valve of the hydrogen gas cylinder or generator must be closed. Also, make sure that there is no gas leakage from the main valve of the supply.
3. The flow line for hydrogen gas should be checked for leakage whenever it is used.
4. To prevent buildup of explosive concentration in case the hydrogen gas leaks, the room in which the device is used should be well ventilated.
5. When analyses are completed, close the main valve of the hydrogen gas container immediately before performing other procedures.

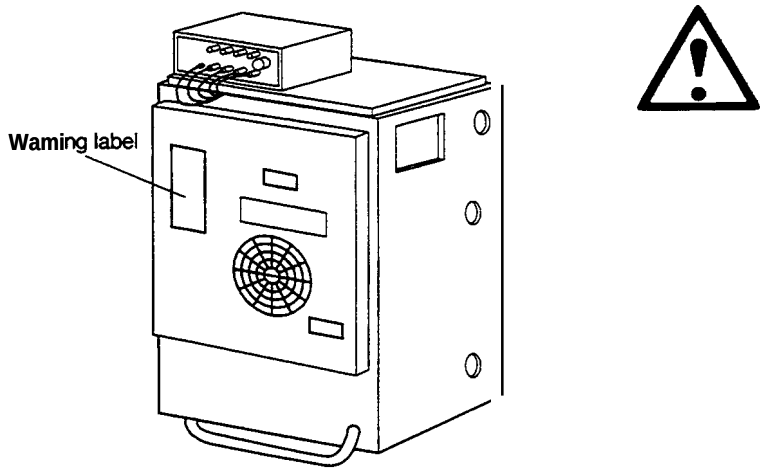


Fig. 5

EMERGENCY MEASURES

In an emergency such as trouble detected in the gas chromatograph, take the following countermeasures.

Before restarting operation, check the gas chromatograph. Contact our service personnel if necessary.

Emergency stop operation

1. Turn **OFF** the power switch for the gas chromatograph.
2. Turn **OFF** all power switches of the supplied equipment.
3. Close the main valve that supply the carrier gas, hydrogen, air, and make-up gas.
4. Shut down the power supply.

- When the power cable is fixed to the switchboard with the screws, turn **OFF** the switch on the board.
- When the power cable is connected with the plug, disconnect the power cable.

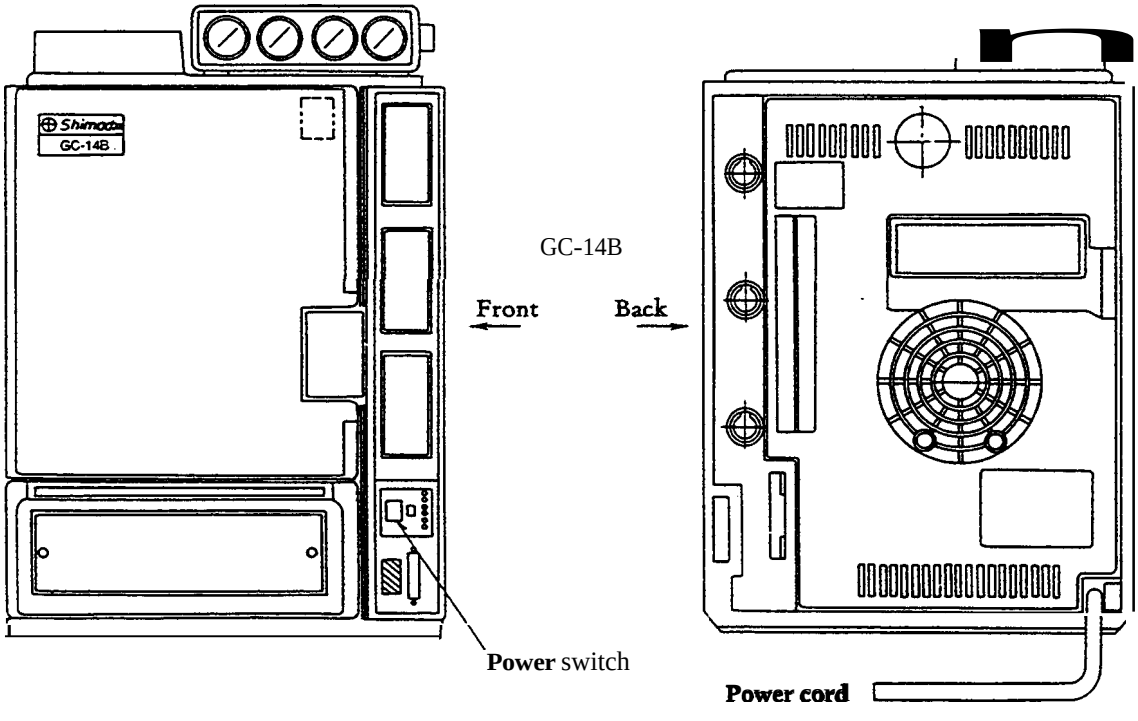


Fig. 6 Power switch and power cable for the gas chromatograph

Other Symbols

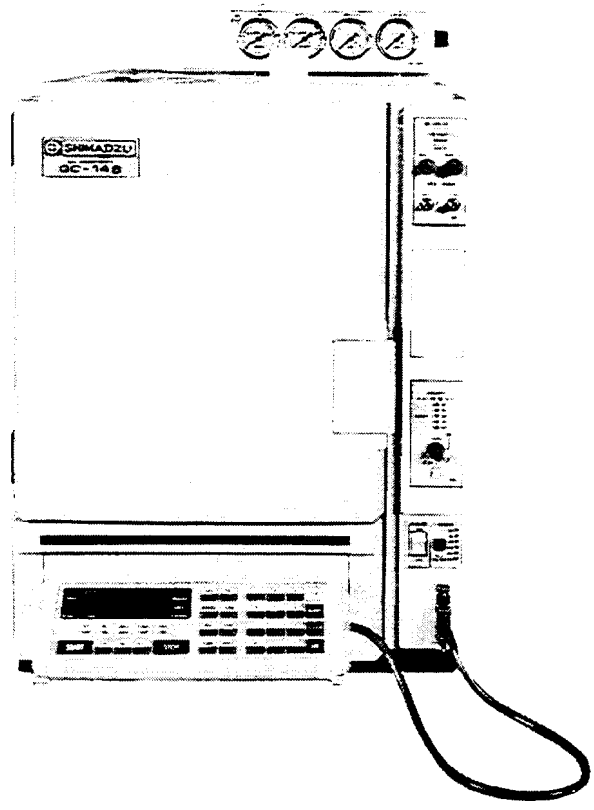
indicates protective conductor terminal.
Grounding circuit continuity is vital for safe operation of equipment. Never operate equipment with ground conductor disconnected.

indicates functional earth terminal.

Contents

12. Hydrogen Flame Ionization Detector (FID)

1. Introduction



GC-14BPFSC

Thank you very much for your purchase of Shimadzu's **GC-14B** series Gas Chromatograph.

The **GC-14B** series Gas Chromatograph, which is controlled by microcomputer, is a multipurpose gas chromatograph exhibiting high performance. This GC unit has particularly distinguishing features such as small installation area requirements and large system expansion capabilities.

1.1 Installation Environment

1.1.1 Operating Temperature, Specified Temperature, humidity

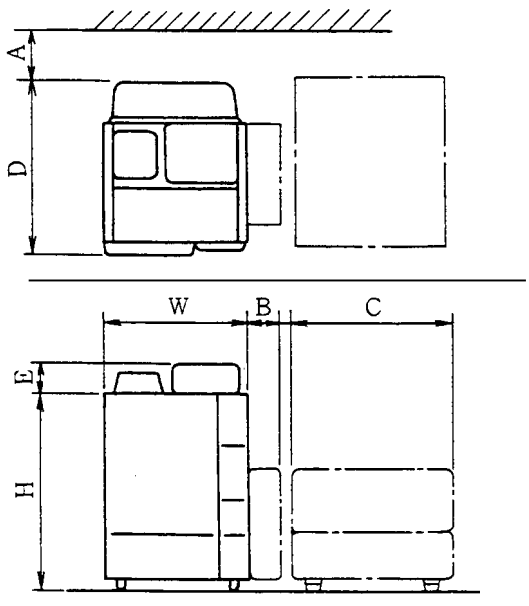
Although the unit operates in the range of ambient temperatures of 5°C ~ 40°C and relative humidity of 5% ~ 90%, use it within the range of 10°C ~ 30°C ambient and 50 ~ 60% relative humidity for longer service life and the optimum specifications. (Operating temperature range: 5°C ~ 40°C, specified temperature range: 10°C ~ 30°C)

Installation place

Warning	As high-temperature (400°C) air is exhausted from the rear of the unit, do not place combustible things around the unit.

The following installation area is required. Install the unit at place which is protected from vibration and direct sunlight.

Dimensions of installation place



External dimensions of the unit

H : Height 520mm
W : Width 400mm
D : Depth 475mm
E : Height of flow controller 70mm
A : Required space behind the unit
300mm or more
B : With option unit 76mm
C : For **C-R7A** Chromatopac 364mm

Weight of **GC-14B** main body: Approx. 40kg

Caution	The sample is vaporized and discharged from the split vent, purge vent, and detector vent, etc. When analyzing toxic substance, properly ventilate the place and provide a means of recovery if necessary.

1.1.2 Electro-magnetic wave and the noise of power source

This unit should not be used in the strong electro-magnetic field. And it should not be used with the power source which conteins a strong noise. Such a noise will disturb the unit to give correct data.

1.1.3 Power

115/220V/230V/240V AC (50Hz/60Hz) and 2.2kVA max. capacity are necessary for the power source of the unit (with no satellite oven installed.)

Column oven	1.3kVA
INJ block	150VA
DET block	300VA
TCD block	200VA

Total consumed power is calculated by totalling the power consumed by INJ, DET, column oven, and AUX blocks.

Range of source voltage	± 10%
Specified range of source voltage	± 5%

Power cable

Warning	High Voltage 1. When connecting the power cable directly to the terminal block on the distribution panel, be sure to turn **OFF** the switch on the distribution panel before installation. 2. Always supply the power from the power source that incorporates the leakage breaker. 3. Do not put any heavy articles on the power cable.

In the case of 115V power, the maximum capacity of power exceeds 15A. ***So*** the power cannot be supplied from a general outlet. Accordingly, supply power from a specially-wired terminal with large capacity. Exercise caution in wiring in accordance with the functions and color of the power cable, as described below.

Color of power cord leads of GC main body	Pole	Remarks
Black	**HOT**	Electric potential of 115/220V against ground.
White	**NEUTRAL**	No electric potential OV against ground.
Green	GROUND	Ground

In the case of 220V/230V/240V power, the plug of the power cable can be connected to a general outlet. The rated voltage is printed on the equipment.

1.2 Parts List

1. Main body of GC ..... 1 (Note 1)
2. Flow controller ..... 1 (Note 2)
3. Pack of accessories ..... 1 (Note 3)

Note 1. At shipment, detectors TCD and FID are incorporated in the main body, and ECD, and FPD are usually separately packaged.

Note 2. Quantity and type of detectors are different depending upon the ordered types and combination.

Note 3. Refer to the attached sheet for the contents of the accessories package.

STANDARD ACCESSORIES FOR GAS CHROMATOGRAPH

(P/N221-32372-91)

201-35584
Injection Rubber Septa

221-15858
Insert Ring

221-15561-91
Glass Column Joint Set

Blind Plug Set

081-16103 Tweezers

221-14122 Grounding Wire

Optional Accessory
201-35184
Silicon Rubber Gaskets

086-03011 Wrenches

086-03802 2mm
086-03804 3mm
Allen Wrenches

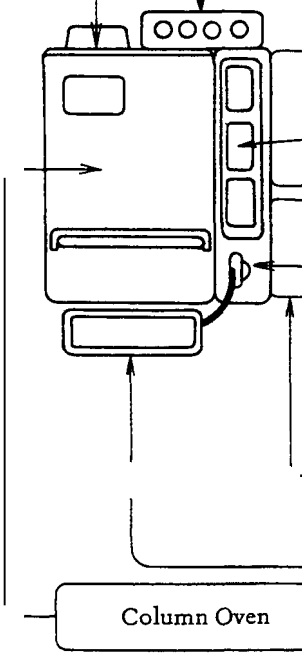
Optional Accessory
201-47614
Silicon Rubber "O" Rings

221-14093 Glass Insert for Injection Port

201-35183
Aluminum Gaskets

221-41123-91 Signal Cable

201-47616
Silica Wool



Injection Port System
A variety of injection port artangements are available depending upon the analytical requirements.

Flow Controller
Flow control configuration depends upon analytical requirements

Optional Controller Case
Allows simultaneous installation of upto **4** detectors

Operator Interface
Detachable keyboard and display unit for parameter setting, control and monitoring, etc., of the instrument.

2. Specifications

■ Column oven

Dimensions of column compartment: 230 (W) X 140 (D) X 360 (H) mm

Length of columns to be accommodated:	Stainless steel column	10m X 2
	Glass column	3m X 2
	Capillary column	100m X 1

Range of temperature setting:	Temperature;	-80~+399	1°C step
	Rate of temperature rise;	0~40°C/min	0.1°C step
	Constant temperature hold time;	0—655 min	0.1°C step
	Program stages	5 stages max.	

Range of temperature control (with power voltage of 100V)

Range of linear temperature increase:	30°C/min	150°C or less
	20°C/min	250°C or less
	10°C/min	330°C or less
	5°C/min	399°C or less

Lower-limit temperature: When INJ and DET temperature is 300°C, room temperature + 15°C

When INJ and DET temperature is 150°C, room temperature + 10°C

Additional cryogenic equipment is required for controlling at lower temperatures than those above.

Cooling speed: Approx. 9 min to reduce from 399°C to 100°C with room temperature of 25°C.

Approx. 14 min for reducing from 399°C to 50°C with room temperature of 25°C

■ Detector oven

Range of temperature setting: Room temperature ~ 399°C (in 1°C steps)

■ TCD oven

Range of temperature setting: Room temperature ~ 399°C (in 1°C steps)

■ Sample injection port unit

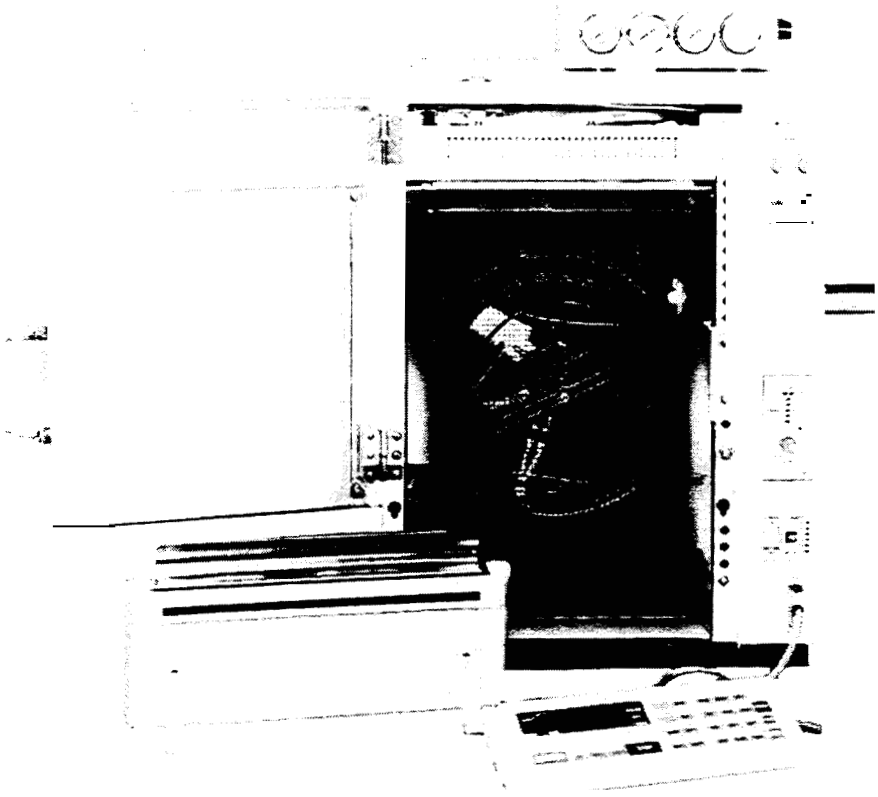
Range of temperature setting: Room temperature ~ 399°C

Sample injection port unit (Either one of the following units is provided)

Single injection port unit:	For packed glass column, combination type of glass insert and on-column injection, 1 flow line
Dual injection port unit:	For packed glass column, combination of glass insert and on-column injection, 2 flow lines
Injection port unit for for capillary columns:	Exclusive injection port for capillary analysis

- Overheat protection circuit 3 circuits
  1. 450°C-fixed independent protection circuit.
  2. Protection circuit for which the upper-limit temperature can be set via key operation.
  3. Overheat protection circuit by CPU abnormality detection
- Combination of Detectors
  1. Four detectors at maximum from among TCD, FID, ECD, FPD, and FTD can be simultaneously installed to the detector oven of the GC main body. Two TCD detectors cannot be set simultaneously.
  2. Only two types of detector controllers (except FTD) can be installed simultaneously to the control section of the GC. Another unit should be applied for installing three or more detector controllers, or FTD.

2. Installation



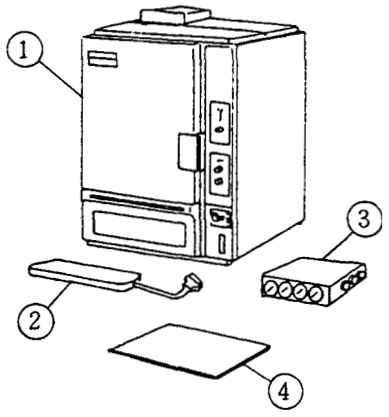
1. Installation

Note **GC-14B** weighs more than 40kg.

So, it should be carried by two people, and care should be given not to drop it or knock it during unpacking.

1.1 Inspection at Unpacking of GC Main Body

- Main body of GC-14B Gas Chromatograph



① Main body .....

② Keyboard unit 1

③ Flow controller

Single model 1

Dual model 2

④ Package of standard accessories

- Detectors

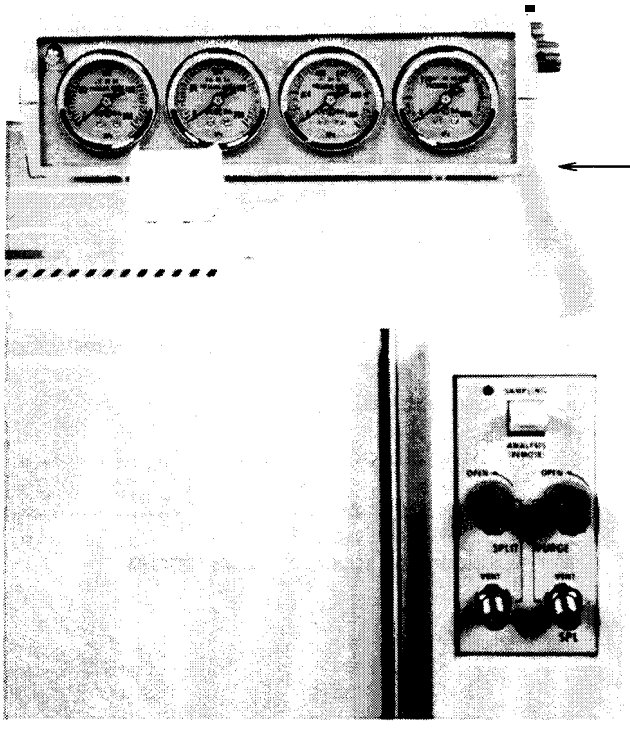
TCD ..... Incorporated in the **GC-14B.**

FID .....Incorporated in **GC-14B.** For dual flow lines, they may be packed separately for each flow line, and added on installation.

Other detectors .....They are packaged in separate boxes, and added on installation.

- Flow controller

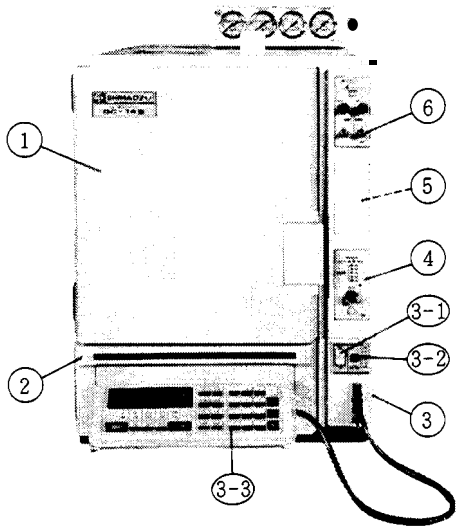
The flow controller is included in the package of the main body. Mount it at the place shown in the diagram below on installation.



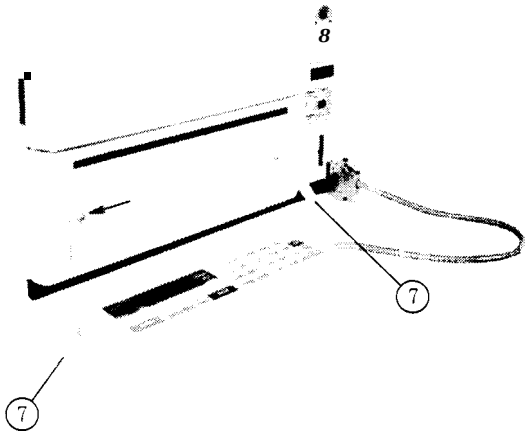
Flow controller
(Another **flow** controller is placed above for dual **flow** lines.)

1.2 Installation

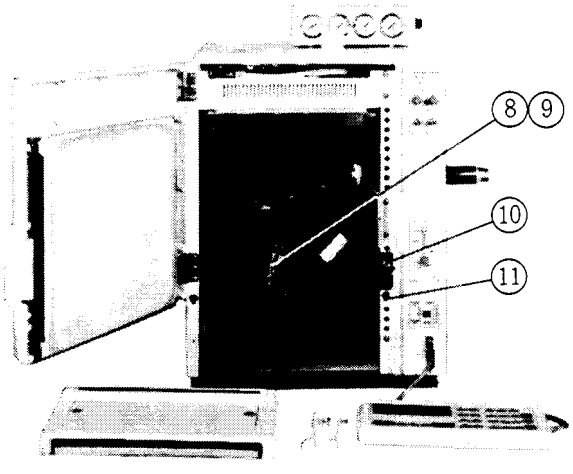
Operations	Remarks
1) Place it on the table.	① Column oven door ② Sub door ③ Operation panel (keyboard) of the main body ③-1 Power switch ③-2 Heater switch ③-3 Keyboard unit cable connector ④ Detector control section (normally TCD) ⑤ Detector control section (normally FID) ⑥ Capillary split/splitless injection system controller



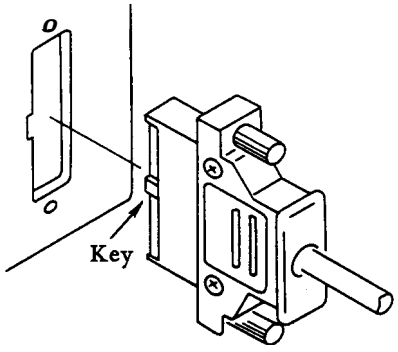
2) Installation of keyboard unit



3) Open the oven door and check that no foreign matter exists inside.



Insert the keyboard unit cable connector into the recess in the column oven as shown below. Observe the key on the connector. Tighten the two knurled screws firmly.



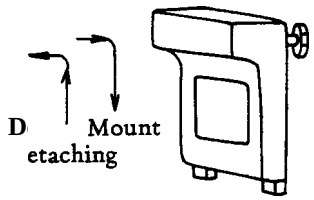
⑦ For fixing the keyboard unit to the sub door, mount the metal brackets provided by the screws indicated by the arrows.

⑧ Temperature control sensor

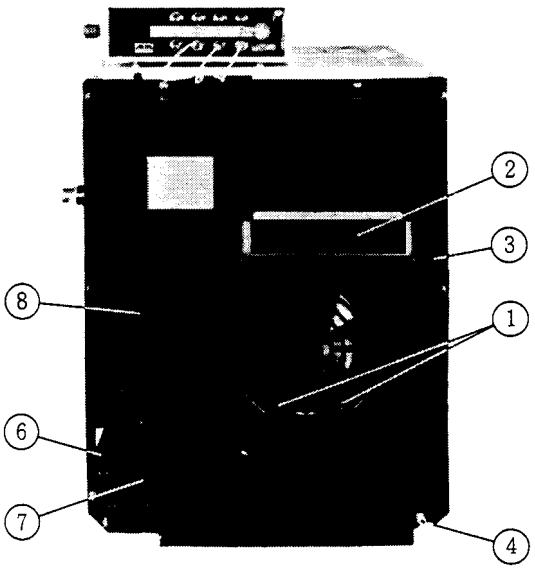
⑨ Overheat prevention thermocouple

⑩ Door switch (Heater is turned OFF if the door is opened.)

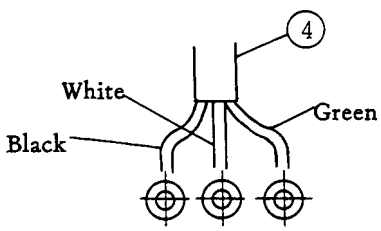
⑪ Sub door mounting hole



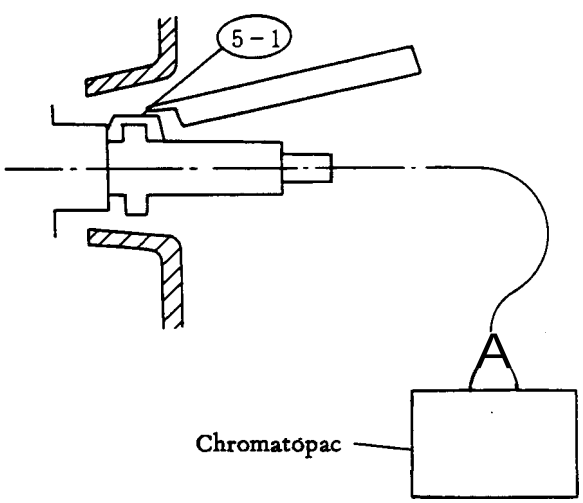
4) Inspect the rear of the oven



5) Connection of power in the case of 115V power.

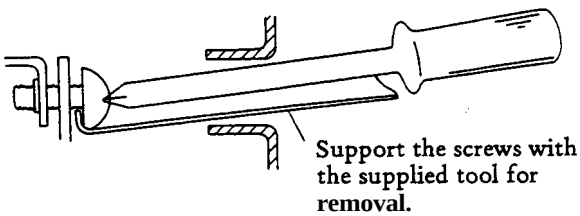


6) Insert of signal cable



① Fan motor clamping screws

Remove the fan motor clamping screws located at ① before operation. **As** these screws are only used for fixing the motor during transportation, they should be removed prior to operation to prevent vibration.



② Exhaust port during cooling

As hot **air** is exhausted from this port, do not place any dangerous materials near the back of the oven. (30cm or more clearance is required to prevent danger.)

③ Intake port during cooling

④ Power cord (Specifications are different depending upon source voltage.) Check that the power switch on the front panel is **OFF** before connection.

Connect as follows in the case of 115V power.

black ..... connected to **HOT** side of **AC** line

white ..... connected to NEUTRAL side of **AC** line

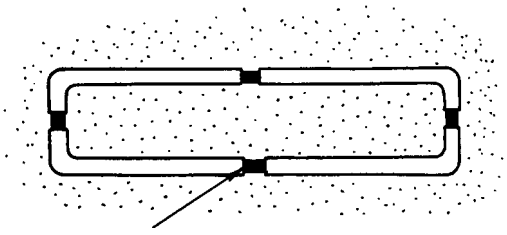
green ..... grounded

to a power source with capacity of 115V AC. In the case of 220V/230V/240V power, connect the plug of the power cord to the power source.

⑤ signal cable connect port

Insert **signal** cable to connection port at the rear of the control section of the detector to be used. For detaching the signal cable, first remove lock (5-1) by screwdriver or the supplied tool, then pull the connector out.

7) Lead wire feed through



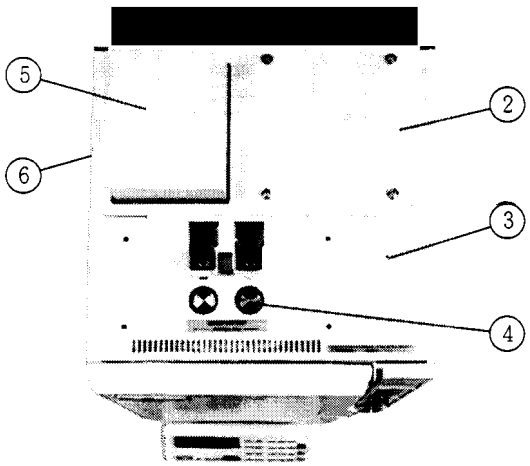
Cut the plastic joints **to open the feed through when necessary.**

⑥ Feed through for current loop and/or RS-232-C interface cords

⑦ Feed through for external control signals

AUX terminal board

8) Inspect the top



② Top cover

③ INJ/DET cover

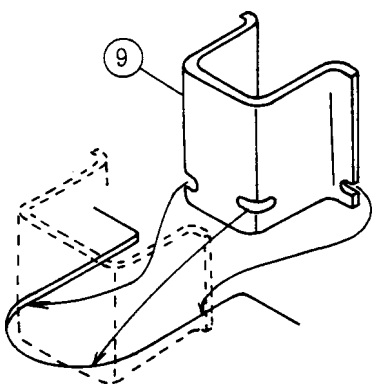
④ INJ unit

When an INJ unit is already installed in the main body, inspect it in that condition (or after inserting a glass insert). For changing the position or type of INJ unit, refer to paragraph **3.**

⑤ TCD oven

When the GC unit is not provided with a TCD, this surface is continuous with ② .

9) Set FID guard



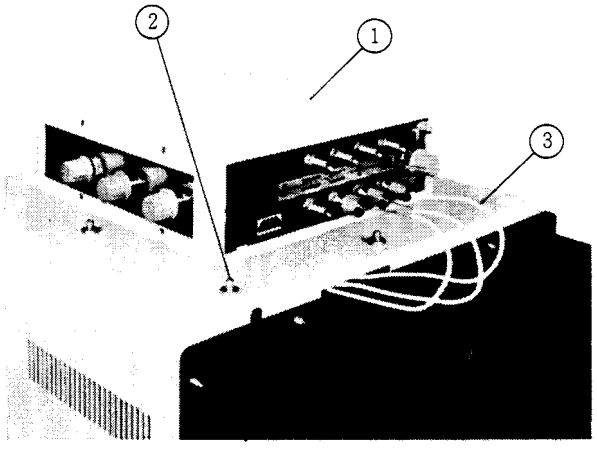
⑥ Left-side face, TCD vent

⑨ Set FID guard

FID guards are incorporated in the main body at the plant before shipment.

If adding **an** FID later, set the guard **as** in the method shown in the diagram on the left.

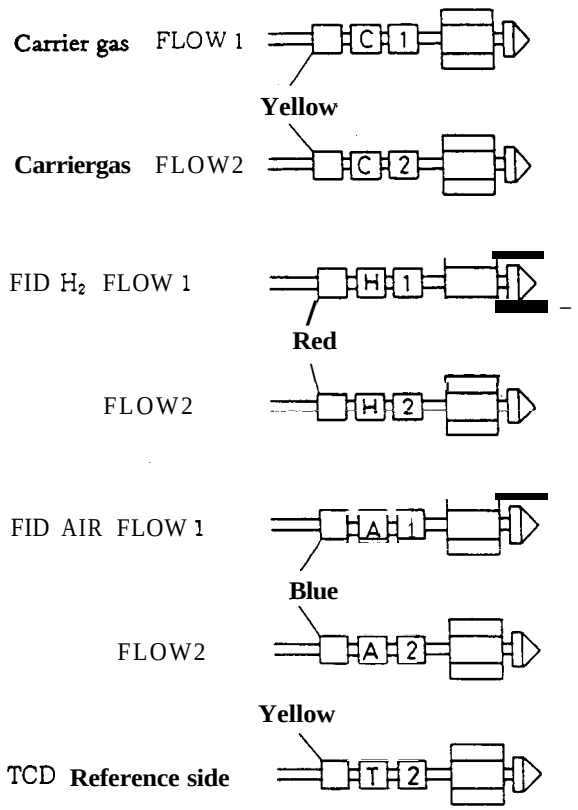
10) Connect flow controller



① Flow controller
② Positioning pin
③ Piping

Connect the pipes exiting from the notch in the back of top cover to the OUT-side joints on the back panel of the flow controller. The pipes are color-coded and labeled for identification. Be sure to connect each pipe to the correct joint.

Identification of pipes



Installation of column .....Refer to paragraph 2 of the "Installation of Column" section.

Details of gas supply and flow controller ..... Refer to the instruction manual for the flow controller.

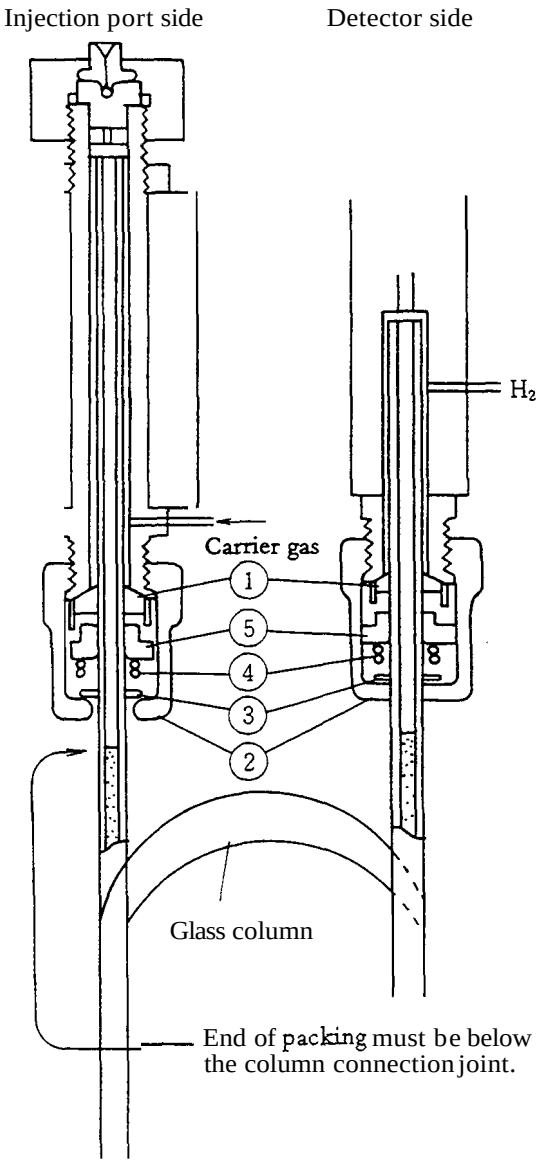
Addition of INJ unit ..... Refer to the paragraph with reference to addition, and change of position of INJ unit.

Addition of detector .....Refer to the instruction manual attached to each detector.

Note **Do not turn on the power if a** TCD **is installed without first establishing carrier gas flow.** TCD **filaments may** be **damaged otherwise.**

2. Setting of Column

2.1 Glass Column (On-column type)



1) Fix parts ① ~ ⑤ on the inlet andoutlet sides of the glass column as shown in the diagram on the left. Be careful about the orientation of back rings ⑤ . (The ends with the smaller outer diameter should contact the graphite ferrules.)

2) Lightly tighten nuts ② by hand, and press the column up until the end of the column bottoms in the column joint on the detector side.

3) With the column correctly positioned, tighten the joint at the injection port side with wrenches.

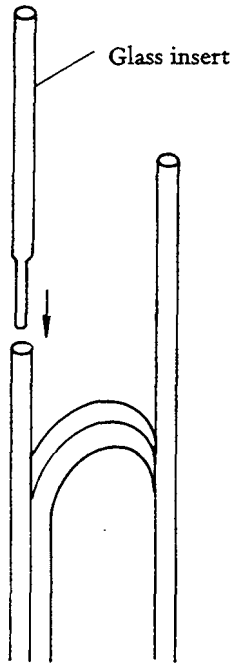
> **Caution** As excessive tightening may damage the column, tighten the joint gradually while checking leakage with soapy water applied to the joint until the optimum extent of tightening is found.

If this procedure is conducted while watching the carrier gas pressure gauge, when carrier gas is controlled by mass flow controller, the pressure gauge indication rises when the leakage stops.

4) Tighten the column joint on the detector side to a similar extent.

	Parts No.	Name of parts	
①	221-15563-91	Graphite ferrule (4)	
②		Cap nut	Glass column joint P/N 221-15561-91
③		Washer	
④		Spring	
⑤		Back ring	

2.2 Glass Column (Insert type)



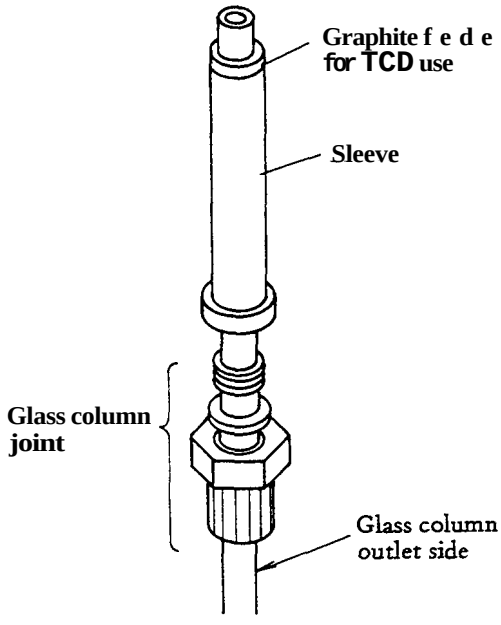
Inlet and outlet necks of the column are of different lengths. Insert the glass insert into the shorter neck, and set the assembly into the injection port.

Tightening method is the same as that for the on-column type described in the previous paragraph.

* Two types of glass inserts are available to fit the inner diameter of the glass column to be used. A glass insert of 3.2ϕ is provided in the set of standard accessories.

Glass insert, 3.2ϕ	P/N 221-14093	For columns of inner diameter 3 ~ 3.4ϕ
Glass insert, 2.6ϕ	P/N 221-14094	For columns of inner diameter 2.5 ~ 2.7ϕ

2.3 When Connecting Glass Column to TCD

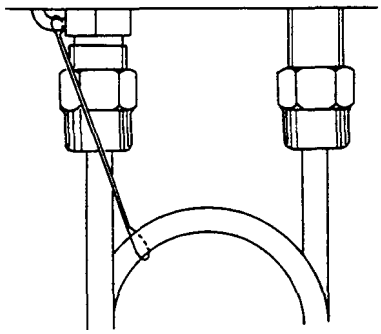


The TCD has no passage to purge the column outlet as in other detectors. With the FID, for instance, hydrogen gas enters at the column outlet to purge dead space.

Accordingly, for TCD, the graphite ferrule and back ring are special ones, and the seal occurs at the bottom of the column connection port to minimize dead space.

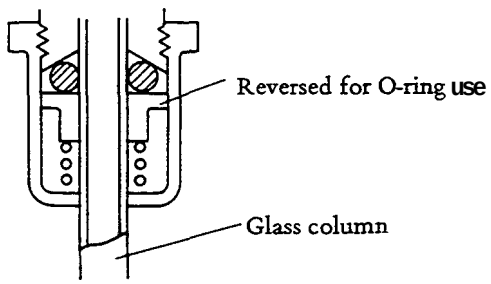
Graphite ferrule for TCD use	Set of 10 P/N 221-10076-91
Sleeve	P/N 221-10073

Warning support the column with wire, etc. to prevent it from popping out of the joints due to the pressure of the carrier gas. If the column slides down by 40mm or more, hydrogen gas will leak from the detector (FID), thus causing a dangerous condition.



Caution An O-ring can be used as the gasket of a glass column joint. In this case, exercise caution about the following points.

1. The back ring of the glass column joint should be set in the opposite direction of that for graphite ferrule use.



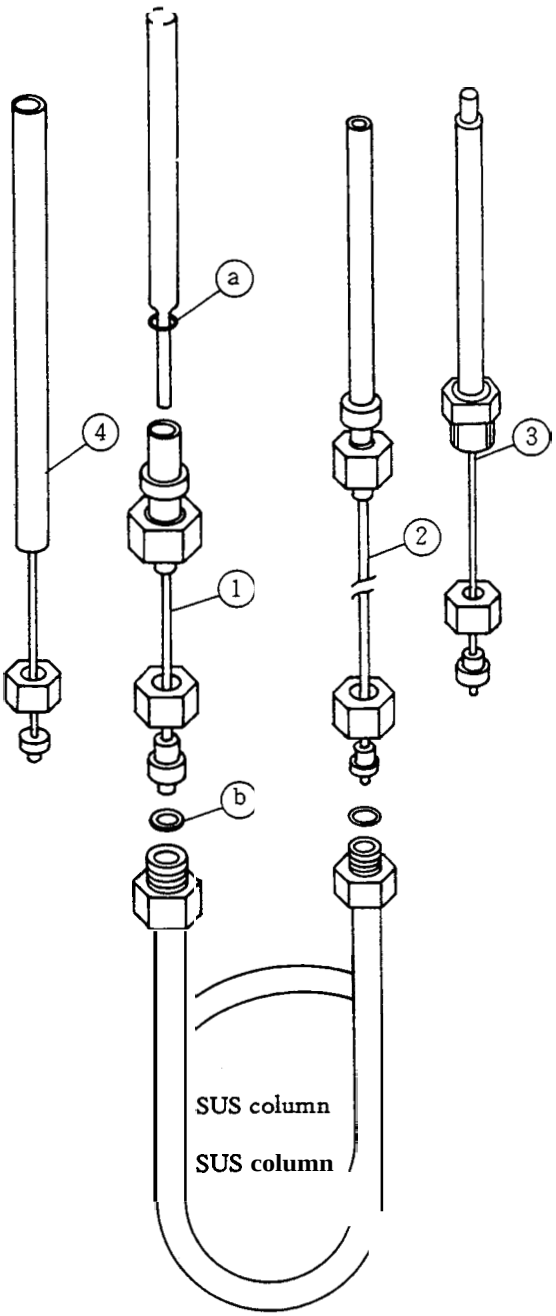
2. Although O-rings can be used easily, all O-rings have certain heat-resistance limits. Observe the limit for the type of O-ring used to prevent elution of compounds which will cause ghost peaks.
(Silicon rubber O-ring ..... 250°C max.)

2.4 Stainless Column

As no parts for connecting a stainless column are provided with the standard unit, purchase stainless steel column adapters separately.

Stainless steel column adapters can be used interchangeably for Shimadzu GC-7A, GC-9A,

1) Stainless steel column adapters for Shimadzu gas chromatographs (for SUS column)



) Stainless steel column adapter
P/N 221-14087-91 for INJ

) Stainless steel column adapter
P/N 221-08882-91 for DET

) Stainless steel column adapter
P/N 221-10079-91 for TCD

Caution When inserting a glass insert into a **SUS** column adapter, set an insert ring ⓐ (P/N 221-15858) as shown in the diagram. This prevents breakage of the glass which may otherwise occur due to the difference in heat expansion between the glass and metal.

Note For connecting a **SUS** column, tighten with column gasket ⓑ . (When aluminum gaskets are applied, overlap a few gaskets together.)

Column gasket (Aluminum)
Set of 100 P/N 201-35183

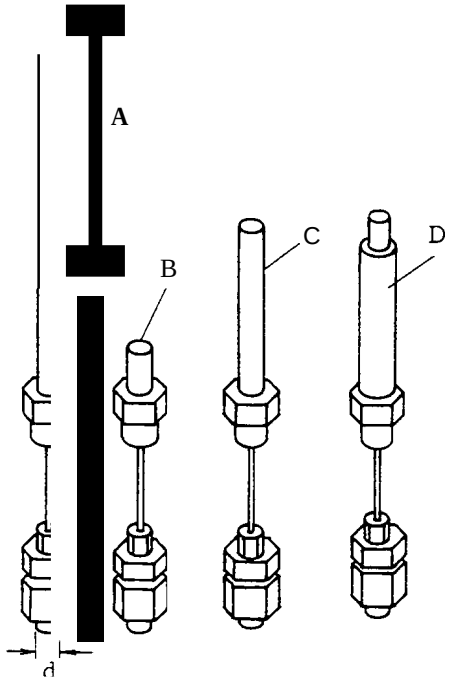
Column gasket (Silicon rubber)
Set of 50 P/N 201-35184

4) Some **SUS** column adapters on the INJ side use stainless steel pipes instead of glass inserts. In this case, join by glass column joint.

SUS column adapter (On-column)
P/N 221-17920-91

2) Adapters for use when fractional-inch-sized stainless steel pipe is used for the column

These adapters are for Shimadzu stainless steel column in 1) with swagelock type joints.



Note Fractional-inch-sized columns or materials for such columns are not supplied by Shimadzu except for the adapters.

A. SUS column adapter on INJ side (On-column type)

Column outer diameter (d)	P/N
For 1/8-inch column	221-22910-32
For 3/16-inch column	221-22910-33
For 1/4-inch column	221-22910-34

B. SUS column adapter on INJ side (Glass insert type)

Column outer diameter	P/N
For 1/8-inch column	221-22910-22
For 3/16-inch column	221-22910-23
For 1/4-inch column	221-22910-24

C. SUS column adapter on DET side

Column outer diameter	P/N
For 1/8-inch column	221-22910-72
For 3/16-inch column	221-22910-73
For 1/4-inch column	221-22910-74

D. SUS column adapter on **TCD** side

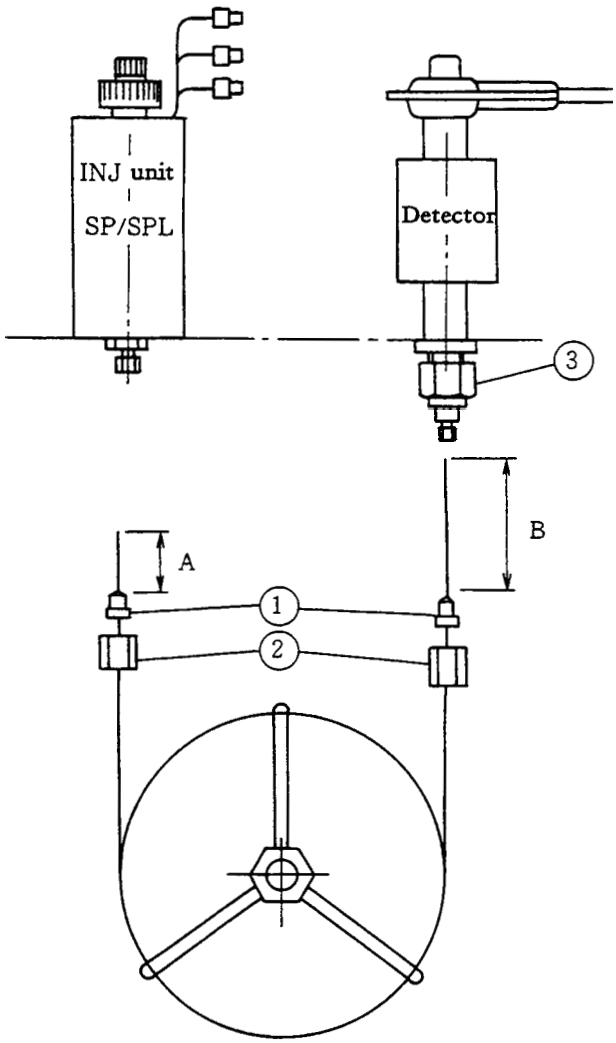
Column outer diameter	P/N
For 1/8-inch column	221-22910-62
For 3/16-inch column	221-22910-63
For 1/4-inch column	221-22910-64

2.5 Setting of Capillary Column

For capillary column analysis, use the split/splitless system (SPL-14). The GC-14B capillary model (GC-14B SC model) is provided with the SPL-14 as standard. For the standard GC-14B packed column model the SPL-14 is an optional accessory.

Refer to chapter 7 for details of the capillary system.

1. When setting a capillary column to the SPL-14

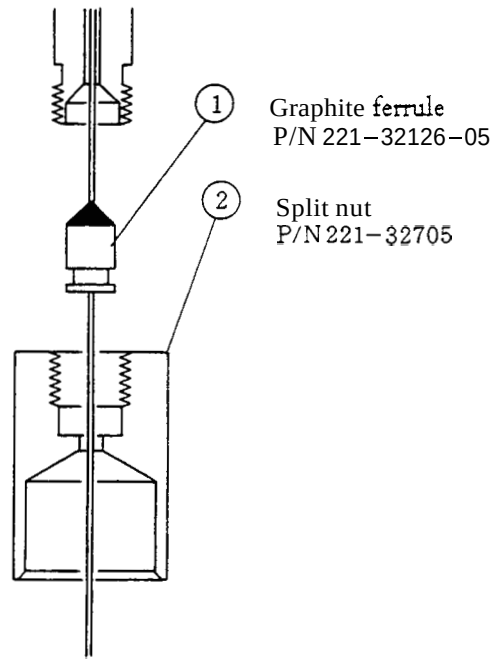


Setting of graphite ferrules for the capillary column

Slide a graphite ferrule (1) onto both ends of the capillary column, and tighten by nuts (2) . The nuts can be tightened sufficiently by hand.

> **Caution**
>
> Excessive tightening by wrenches will cause breakage of the neck of the joint.

Expanded diagram of capillary column joint

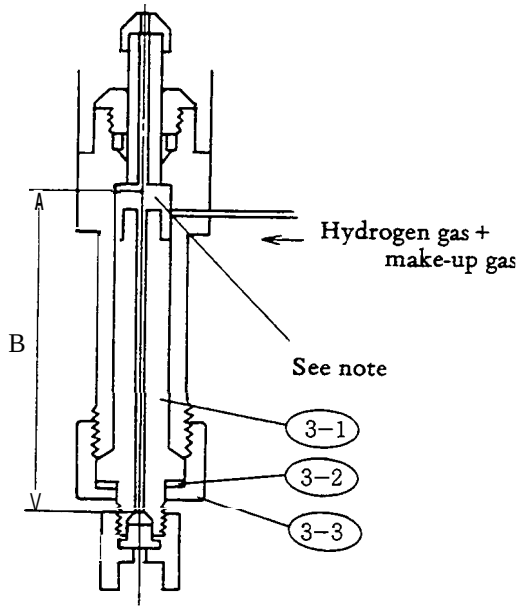


Length between graphite ferrule and column end

Injection port side	**A**	35mm
Detector side	B	75mm

When the detector is an FID, and the column is to be inserted upto the middle of the nozzle, the

Adapter on detector side



length becomes the dimension B above +20mm.

Adapter on detector side		P/N 221-33193-91
(3-1)	Adapter	P/N 221-33193-90
(3-2)	Washer WM	P/N 201-30051
(3-3)	Nut MF	P/N 201-30008

Note Install so that the end of capillary column will enter the nozzle from adapter (3-1) .

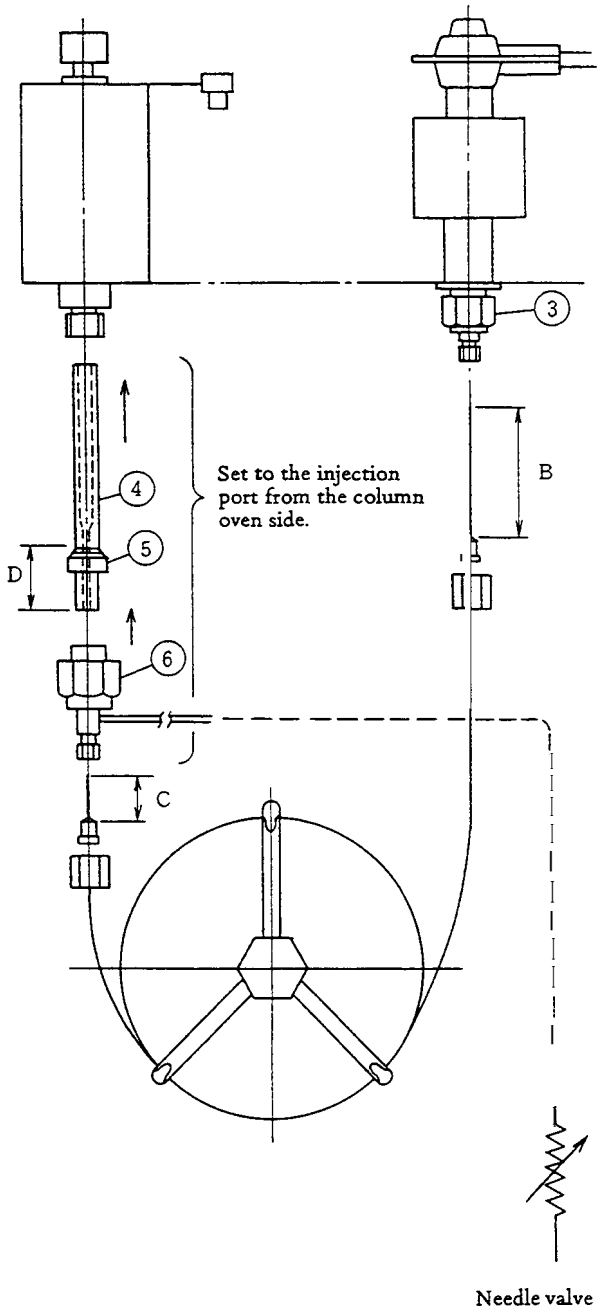
* When inserting into the nozzle, insert the column by dimension B + approx. 10mm. In this case, however, replace the 0.54 inner diameter quartz nozzle to one of 1.2ϕ to allow clearance for both the column and gas flow.

1.24 FID nozzle P/N 221-33265-02

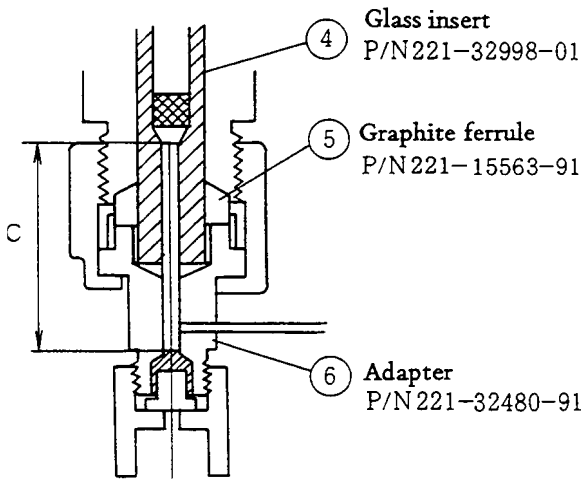
2.6 When setting a capillary column with an adapter for an injection port normally used for a packed column.

Capillary adapter CLH-14

P/N 221-32995-91



Insert the column into the glass insert (4) with graphite ferrule (5) and set the assembly into the injection port from the column oven side. Tighten the adapter joint (6) hand tight. See the diagram below for the assembly after tightening.



C = 60mm

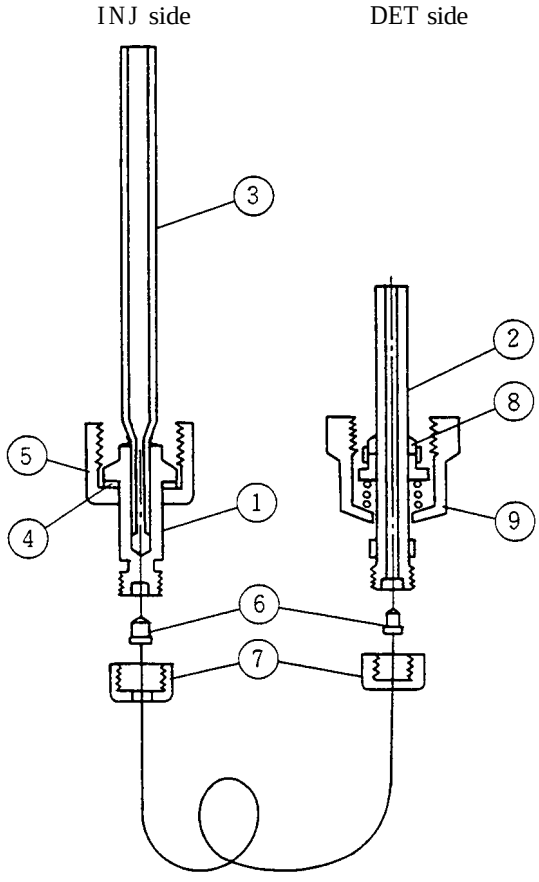
D = 11mm

The detector side of the SPL-14 uses the same joint (adapter) configuration.

2.7 When setting a wide-bore capillary column to an injection port meant for packed column use.

Some capillary columns have an inner diameter of 0.53mm (wide-bore column). Such columns do not aim at high resolution separations like those columns with small diameters (0.1mm~0.3mm). The value of such columns is the importance placed on the merits of the inactive stationary phase carrier, usually silica. Wide-bore columns can be set to the split/splitless system described in 1). However, as an easier (and less costly) method, a wide-bore column can be set to an injection port intended for packed column use as the column can be loaded with the whole sample quantity. The following adapters are used for this method.

Wide-bore column adapter (WBC attachment) P/N *221-29992-91*

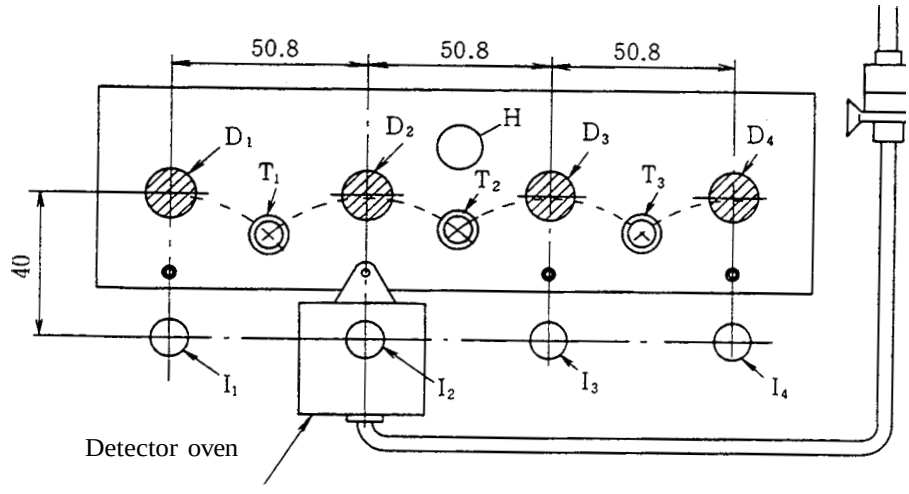


Wide-bore capillary column

	P/N	Name of parts	Q'ty
①	221-29676	WBC adapter, INJ	1
②	221-29757	WBC adapter, DET	1
③	221-38107	Glass insert	1
④	201-30051	Washer WM	1
⑤	201-30008	Nut MF	1
⑥	221-32126-08	Set of 10 graphite ferrules	1
⑦	221-32705	Nuts	2
⑧	221-15563-91	Set of 4 graphite ferrules	1
⑨	221-15561-91	Glass column joint	1

3. Addition or Change of Installation Place of Sample Injection Port Unit

Sample injection port and various detectors of the **GC-14B** are installed at the places shown below. Detector Location is fixed, however, the injection port is unitized for relocation as required.



Sample injection port unit

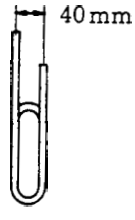
The diagram above is the top view of the detector section.

Setting holes	Uses
$D_1 \sim D_4$	Locations for detectors except TCD
T_1, T_2	Locations for column-to-TCD connection
T_3	Spare
H	Piping port to column oven
$I_1 \sim I_4$	Locations for injection port unit

3.1 Location of Injection Port Unit

o When a glass column is used.

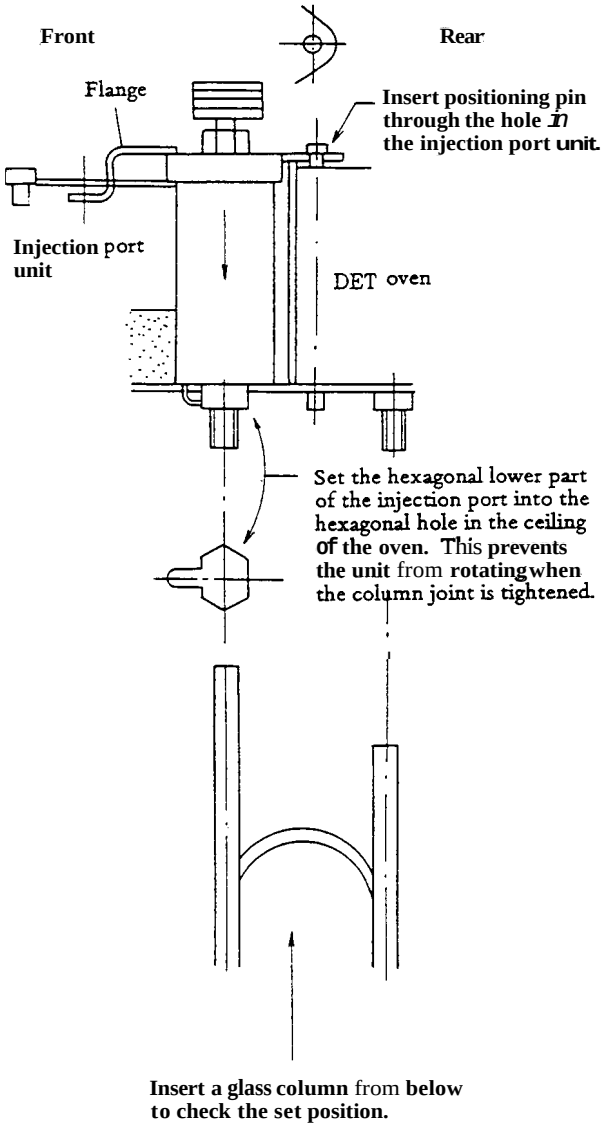
As shown here, the distance between glass column inlet and outlet should be 40mm on centers. For installing the column, fix the injection port unit just in front of the detector to be used. **As** the TCD detector uses T_1 and T_2 (see Section 11), T_1 can be used with injection port unit attached at I_1 or I_2, and T_2 can be used with the injector located at either I_1 or I_3.



○ When a stainless steel column or capillary column is used.

As the distance between centers of the column inlet and outlet is flexible to some extent, there is no restriction as to injection port location as with a glass column.

3.2 Installation of Injection Port Unit



Installation procedures

1) Remove the insulating material at the place where the injection port unit is to be installed.

(Any of I, ~ I_4 in the diagram shown before, as required.)

2) When setting the hexagonal part of the injection port unit into the hexagonal hole of the column oven, pass the locating pin through the positioning hole on the top flange of the unit.

3) When both items above are done, press the unit downward to assume the position shown in the diagram.

4) Fill any empty space around the unit with insulating material (ceramic wool). (Thickness of the insulating material should be approximately 20 ~ 40mm.)

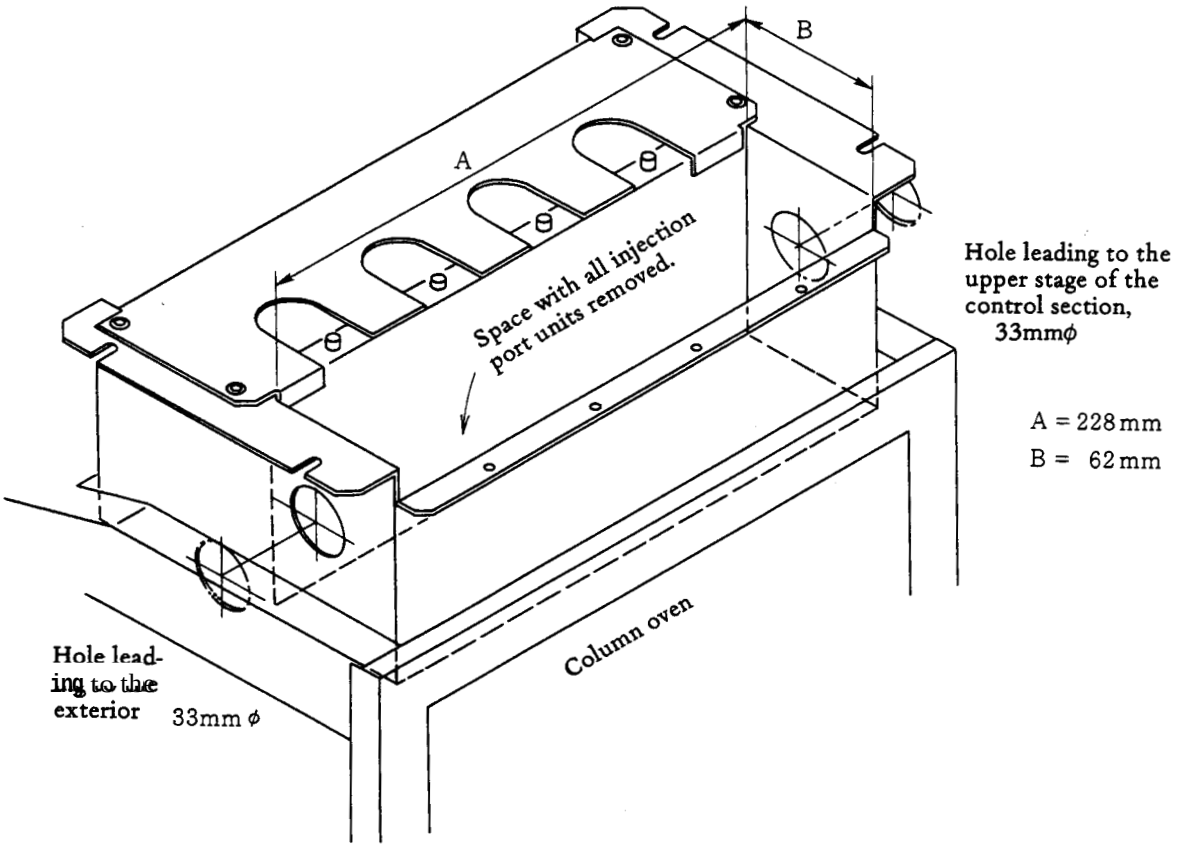
Note For those unused holes provided with no insulating material, apply aluminum foil to cover the holes before filling with insulating material.

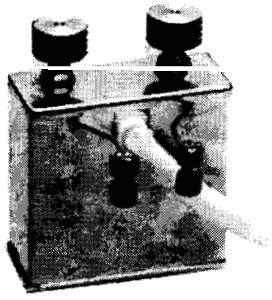
5) When using a glass column, after installing the unit, check that the glass column can be inserted from below smoothly (and falls freely when released). If the column binds, small adjustments should be made otherwise the column may fracture when the joints are tightened.

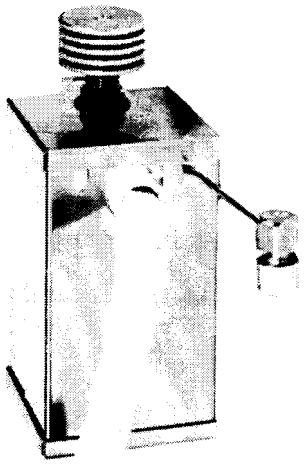
6) Replace the flange to hold the unit in place.

4. Types of Sample Evaporating Chamber Unit

This **GC** unit has the sample injection unit, which is installed to the top of column oven. With all sample injection port units removed, the space leading to the column oven as shown in the diagram can be obtained.



4.1 Injection Port Unit	Remarks (P/N)
	**Dual** injection port unit **Dual** injection port for packed column use For source voltage 115V (P/N 221-29280-92) 220V (P/N 221-29280-93) 230V (P/N 221-29280-38)



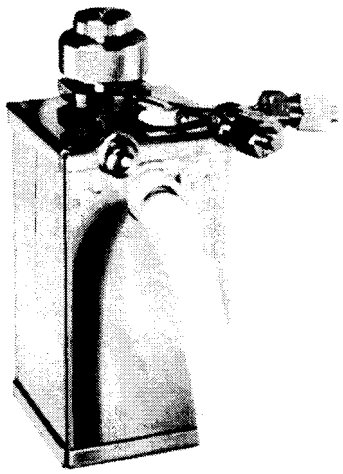
Single injection port unit

Single injection port for packed column use

For source voltage 115V (P/N 221-31222-92)

220V (P/N 221-31222-93)

230V (P/N 221-31222-38)



SP/SPL Injection port

Single split/splitless injection port for capillary column use

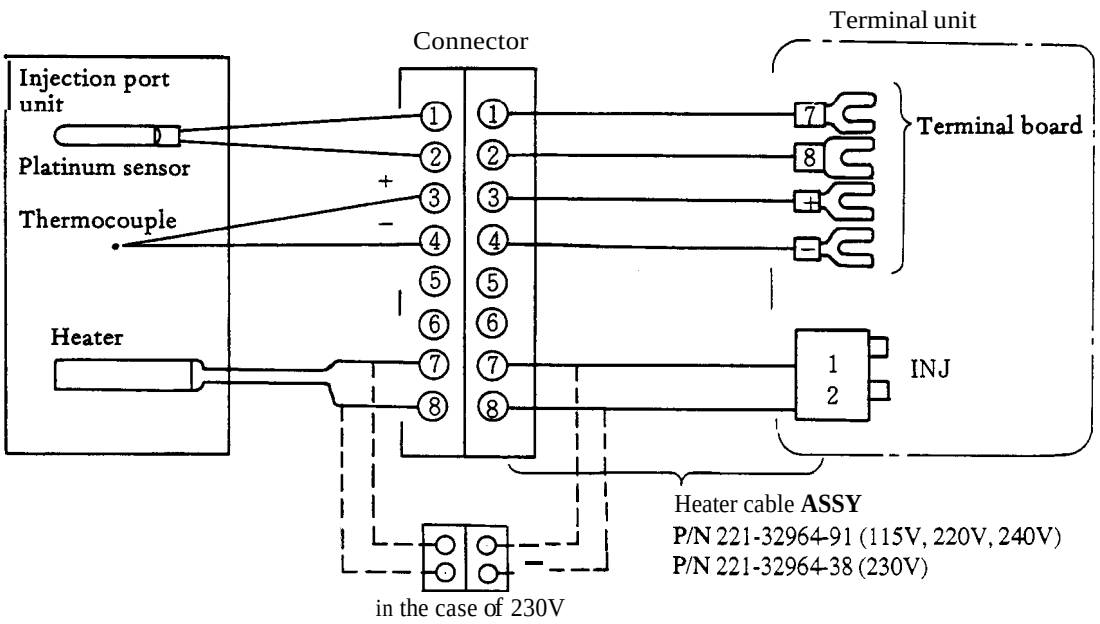
For source voltage 115V (P/N 221-32547-92)

220V (P/N 221-32547-93)

230V (P/N 221-32547-38)

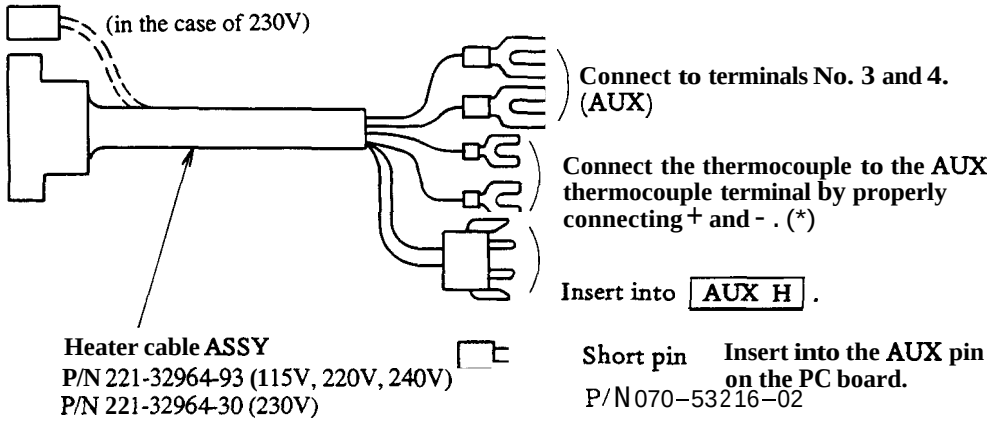
4.2 Wiring of Injection Port Units

Pin configuration for wiring of all injection port units is shown below.



When installing or removing injection port units be certain the power switch is OFF before plugging/unplugging connectors. If done with the power switch ON, the CPU sees a disconnection of the circuit, thus actuating the protective circuit and automatically turning **all** heaters **OFF** at that point.

Note When the AUX-2 temperature control function is not being used for any sections, injection port temperature can be controlled by this circuit. In this case, the following parts are necessary:



The most important thing for this operation is to correctly connect heater, heat sensor, and thermocouple to the PC board in accordance with the way shown. If they are not properly connected, temperature control does not function correctly causing excessive increase or decrease of temperature.

Note * As a standard, safety circuits with thermocouples are provided at **4** places in the GC-**14B** temperature control section. If the safety circuits are already used for all **4** places, the thermocouple of **an** additional unit cannot be connected.

In this case, apply either one of the following three methods.

1) Leave it without connection. In this case, this circuit monitors overheating with the heat sensor for temperature control.

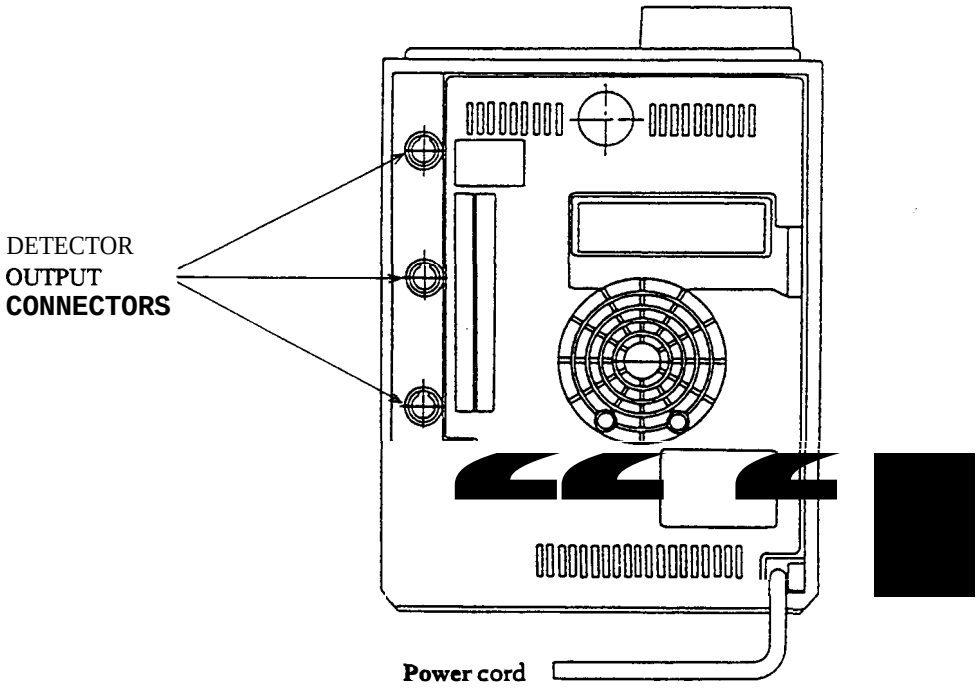
2) If any one of the safety circuits is not to be operated simultaneously with other circuits, remove the thermocouple of that circuit, and exchange it with the thermocouple of the added unit.

3) Add an external overheat protection device (option). In this case, temperature control functions for an additional four more zones.

5. All input and output connection

The connectors which are detachable for user are the outpuit connectors of detectors shown as the following figure.

The signal cable is connected to one of these connectors.

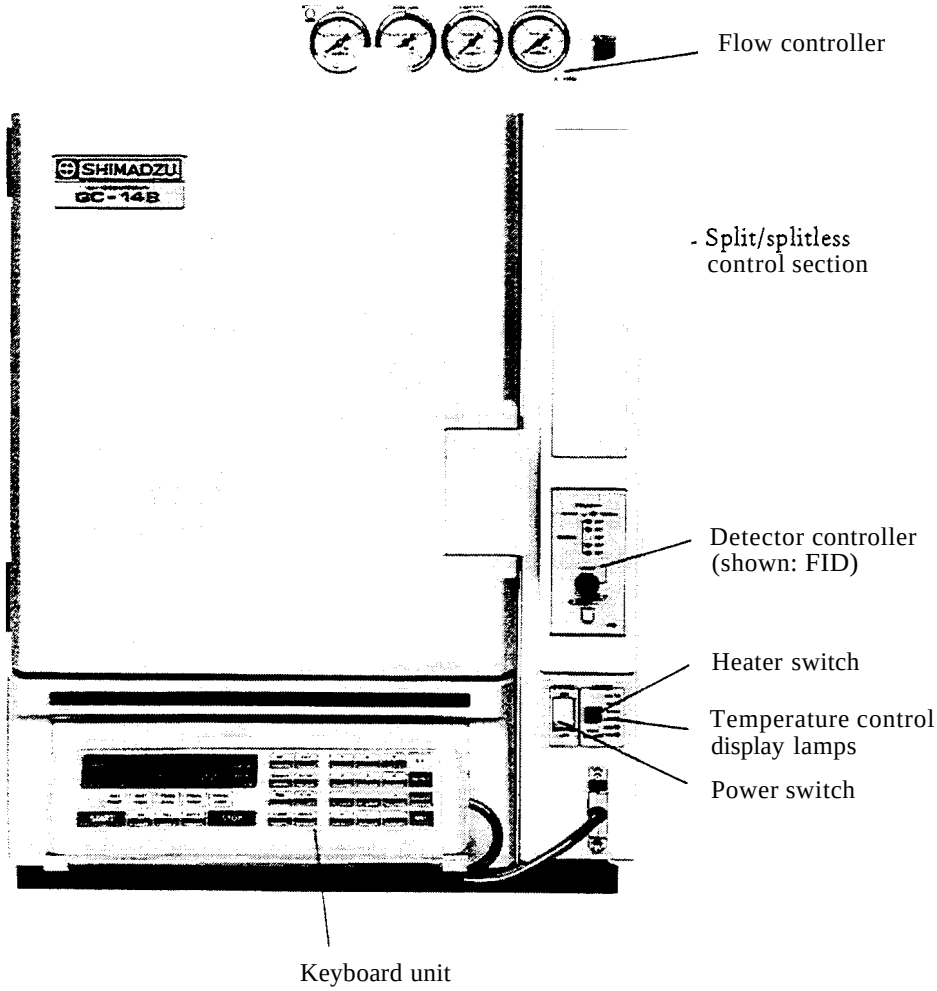


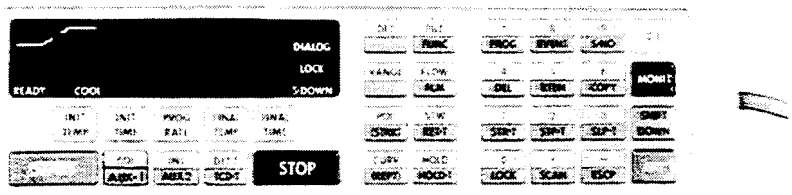
Note Don't touch the connector pins, or electrostatic discharge may damage the circuit.

3. Operation 1

This chapter describes operations necessary for power supply and checking the operating conditions of the instrument.

1. Explanation of Operation Panel





Warning For detectors such as the FID, **FPD,** and FTD, do not allow hydrogen to flow with no column connected. Otherwise, hydrogen will escape into the column oven creating an explosion hazard.

Caution Touching connectors or terminals with power on may damage instruments.

Caution Exercise caution in the following points when applying power with no column connected.

1. When a thermal conductivity detector (TCD) is provided, turn the pushbutton switch of the TCD control section OFF, or set TCD CURRENT to 2mA or less to prevent damage to the TCD filaments.
2. Check the inside of column oven. Particularly, check that no plastic objects (cushioning materials, etc.) are inside.

2. Setting Procedures for Operation Check

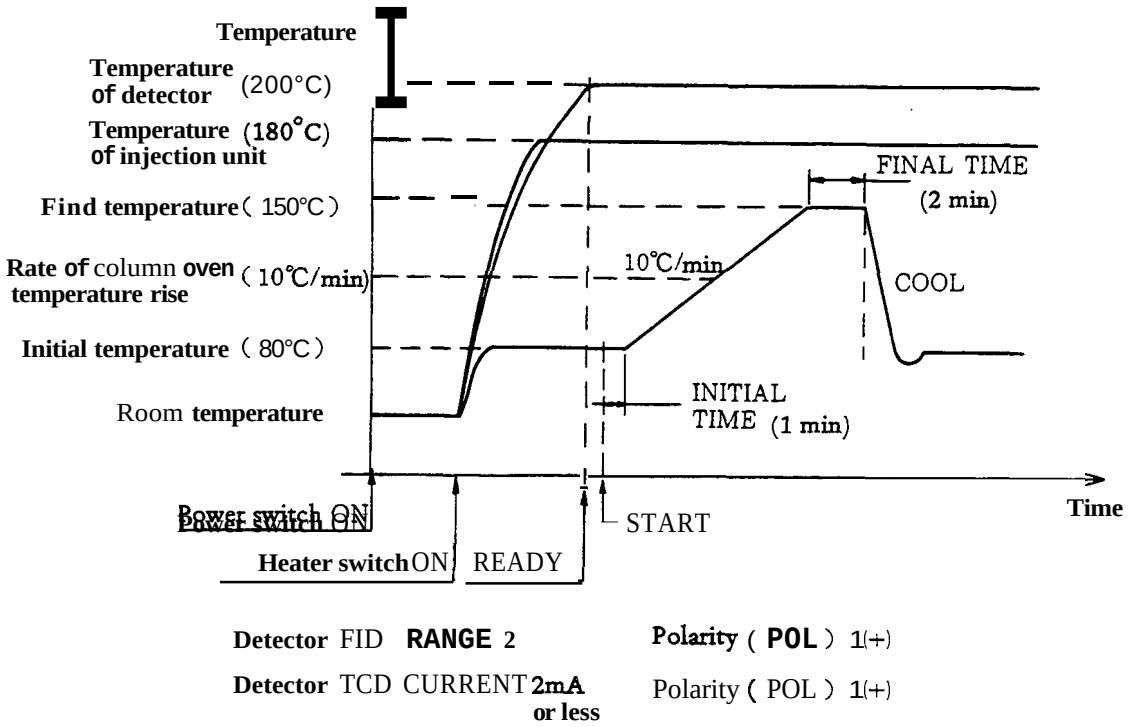


Fig. 32 Example of Condition Setting for Operation Check

Procedures for setting conditions above are described below.

Setting	Operation
1) **Turn** the power switch ON. **Note** Be sure to remove the clamp screws of the fan motor used for transportation.	○ **Fan** motor rotates. LED of **POLARITY** 1 or 2 of detector lights. Either LED of **RANGE** lights ON. Zero-point on-base **LED** may light. ○ CITP. XXX is displayed.
2) Set detector temperature. DET.T 2 0 0 ENT	o Display after pressing the ENT key. DETT 200
3) Set injection port temperature. INJ 1 8 0 ENT	INJT 180
4) Set the column oven initial temperature COL INIT TEMP 8 0 ENT	CITP 80
5) Set the initial temperature hold time COL INIT TIME 1 ENT	CITM 1.0
6) Set temperature rise rate COL PROG RATE 1 0 ENT	CPR1 10.0
7) Set final temperature COL FINAL TEMP 1 5 0 ENT	CFP1 150
8) Set final temperature hold time COL FINAL TIME 2 EMT	CFM1 2.0
in the **diagrams** above can be omitted when setting of column oven conditions is uninterrupted by other key operations.	

3

9) Set conditions of FID controller

DET — 1 — ENT

RANGE — 2 — ENT

POL — 1 — ENT

D1RG 2 **RANGE2** lamp of the FID controller lights.

DET 1 NON is displayed if the FID is not installed, or the controller switch is OFF.

D1PL 1 INJ1 (+) lamp of the controller lights.

10) Set TCD controller

DET — 4 — ENT

DET 4

CURR — 2 — ENT

D4CR 2

POL — 1 — ENT

D4PL 1 INJ1 (+) lamp of the TCD controller lights.

11) Monitor operation

in order to monitor the DET temperature or COL temperature, depress

MONIT DET.T or MONIT COL

respectively before exercising keying-in procedures for setting. ENT is not necessary for monitoring* (except special cases).

DETT. XX

CITP. XX

Even if power is turned ON, the GC is in stand-by with **no** heaters working **until** the **initial** START key is depressed.

DETT. 25 ← Room temperature is displayed.

CITP. 27 ← The temperature displayed may be somewhat higher than actual room temperature due to operation of the fan motor.

12) *(START/*

MONIT COL

13) Depress the heater switch.

If the heater is turned ON the temperature displayed **on** the monitor above increases. (The LED inside of the heater switch lights.)

Upon completion of these procedures, the temperature of each section starts rising towards the set value, and the READY lamp lights when the temperatures reach the set values. Program control starts by pushing [START] while the READY lamp is ON. After the FINAL TIME (2 min), the temperature is automatically reduced to return to the initial condition, and waits for the next key input for restarting. If the unit enters the cooling process (COOL) during the series of operations, hot air exhausts from the vent at the rear.

Display upon supplying power

CITP XX : This is the normal monitor display of column oven temperature. But the actual measurement value is not displayed as the [START] key has not yet been pressed after power is supplied. The actual measurement is displayed by depressing the [START] key.

ERROR CM : This is displayed when the contents of memory become unreliable due to changes while power is OFF, or changes of setting conditions just before turning power OFF. If this is displayed, turn power OFF, and ON again after approximately 10 seconds.

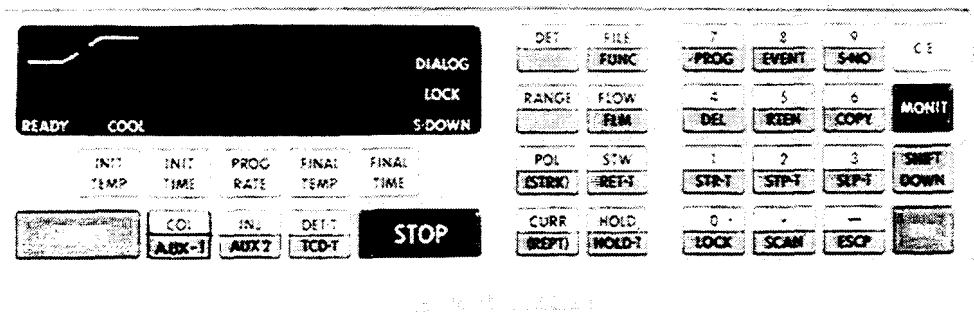
INITIAL When the contents of memory are greatly changed, the setting conditions in all files are initialized, and this display is shown. In this case, repeat of the conditions is necessary.

FAULT This is displayed if the system program has a fault. As the unit is unable to function properly in this condition, contact our serviceman.

4. Operation 2

In this chapter, the various **kinds** of keys used for setting are described along with their functions. For examples of operations which use these keys, please refer to chapter 5, Operation ***3.***

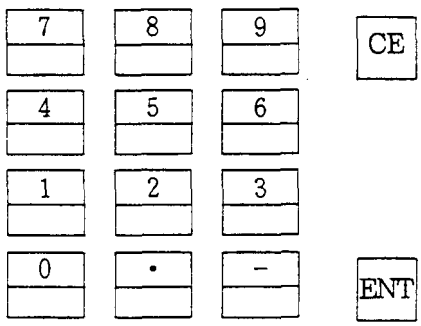
1. Classification *of* keys



4

1.1 Numeric keys

By pressing the numeric keys, the numerals selected are displayed. Actual input of the number then on display occurs with the subsequent pressing of the ENT key.



The range of figures that can be entered for display is up to **4** digits to the left of the decimal point and up to two digits to the right of the decimal point. If the integer portion of a figure entered exceeds four digits, the excess integer(s) becomes invalid. In the same way, if the decimal portion of a figure entered should exceed two digits to the right of the decimal point, the excess digit(s) becomes invalid.

Note

1. No zero-suppression is effected in entering numerals. (If numeral 0 is entered at the head of desired input for 1234, then actual input to the system is done as 0123, with the numeral 4 at the least significant digit being neglected.)
2. Be careful of the fact that pressing two or more keys at a time will result in an erroneous key input.

1.2 Command keys

The following 11 COMMAND keys are available for the outright execution of the selected command by pressing the relevant key. (There is no need for subsequent pressing of any of the numeral keys, following the pressing of such a COMMAND key.)

START STOP ENT CE ESCP HOLD
FLM SCAN LOCK SHIFT.D
STW

4

1.3 FUNCTION keys

Functions of the following keys become effective only when any of these keys has been operated, followed with input of an appropriate number (as described in para. 1.1) and by pressing the ENT key.

INIT TEMPI INIT TIME PROG RATE FINAL TIME FLVAL TEMP
COL INJ TCD-T AUX1 AUX2 DET-T
(STRK) (REPT) DET
RANGE POL CURR FILE FUNC
FLOW HOLD-T EVENT DEL COPY
RETN STR-T STP-T SLP-T

If, after pressing any of these keys, the ENT key is pressed without pressing a numeric key, the value then set for the system is indicated on the display

1.4 Keys available specifically for monitoring

The MONIT key is used when the actual measured values of the chromatograph are to be indicated on the display. The following keys cause display of measured values, as described below, when pressed just after the MONIT key:

INIT TEMP	Actual temperature of column oven
INIT TIME	Retention time counted from the moment when the START key was pressed
PROG RATE	Actual temperature in the column oven
FINAL TIME	Retention time counted from the moment when the START key was pressed
FINAL TEMP	Actual temperature in the column oven

COL	Actual temperature in the column oven
AUXl	Actual temperature in AUXl
INJ	Actual temperature in INJ (sample injection port)
TCD-T	Actual temperature in TCD (TCD oven)
AUX2	Actual temperature in AUX2 (auxiliary temperature control unit)
DET-T	Actual temperature in DET (detector unit)
S-NO	Sample number in AOC at that time
PROG	Step number to be executed next (time program)
STR-T	Time elapsed since Shift-down START
STP-T	Time elapsed since Shiftdown STOP
SLP-T	Time elapsed since Shift-down STOP
RET-T	Retention time counted from the moment when the START key was pressed
HOLD-T	Retention time counted from the moment when the START key was pressed

For each of the above keys, the relevant measurement value can be indicated on the DISPLAY as soon as the key is pressed following the MONIT key.

DET	Indicates the type of detector being used.
FLOW	Indicates measurement value for flow rate. (Obtainable, however, only when the optional **flow** measurement device is connected.)

For the two keys shown above, the relevant data is indicated on the display by operating keys as for example MONIT DET 1 ENT ;that is, by pressing the MONIT key first, followed with the pressing of either the DET or FLOW key, as desired, and of the desired numeral key, and the pressing of the ENT key to complete the necessary key operation.

1.5 Interactive function keys

A. Once PROG is pressed, the display and **all** the other keys are occupied for preparing the time program for the period till the pressing of the ESCP key.

By pressing the SHIFT. D, 7 / PROG keys, the DIALOG lamp lights and the display indicates TIME?, signalling that the time program editing routine is in effect. To escape this dialog routine for returning to the normal state, it is necessary to press the keys. SHIFT. D — / ESCP

B. By pressing the FUNC key, the whole period till the pressing of the ESCP key is dedicated to the necessary AUX function setting routine, with the content of the selected function being displayed.

If there is no need for changing the content (value) then on display, you can either call up the next function by the pressing of the ENT key, or you can escape from the AUX function routine by pressing the ESCP key. If the content then on display is to be changed, first enter the correct number and then press the ENT key. The display is then changed to the next function in the list along with its value (if any).

By pressing the SHIFT, D FILE/FUNC (desired number) ENT keys in that order, the DIALOG lamp lights and the content of the selected function is indicated on the display.

4

1.6 Setting functions not assigned to specific keys

Each of the following functions can be set by operaring the FUNC key. The functions available for setting and the corresponding code numbers are listed below.

Code-1	Function	
1	Selection of parameters required for AOC	*3
2	Request for listing of time program to Chromatopac	*1
3	Request for listing of AOC parameters to Chromatopac	*1
4	Request for listing of GC parameters to Chromatopac	*1
5	Setting upper limit temperature of detector and injection units	
6	Setting upper limit temperature in AUX1 oven	*4
7	Setting upper limit temperature in column oven	
8	Setting for link device (communications interface)	*1
13	Starting self-diagnosis function (When this function is executed, all the user settings are deleted and return to their default values.)	
14	Setting the value "K", the constant used for flow rate measurement	*2
15	Request for listing of temperature program to Chromatopac	*1

*1. Has meaning only if a Chromatopac is connected.

*2. Has meaning only if a flow rate measuring device is connected. On some occassion, however, this key may not be used depending on the type of particular flow rate measuring methods employed.

*3. Has meaning only if an automatic sample injector is connected. On some occassion, however, this key may not be used depending on the particular type of AOC used.

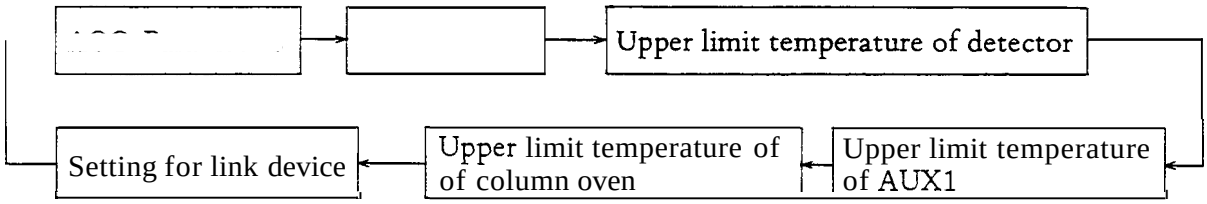
Explained below is an example of setting.

SHIFT. D | FILE / FUNC | 5 | ENT | 3 | 5 | 0 | ENT

In this case, 350°C is set as the upper limit for the detector temperature.

Note Once a desired function has been set by operating the FUNC key, all of the subsequent key operations are effected in the dialog system, with the DIALOG lamp being lit. It is necessary to press the ESCP key to escape from the dialog state effected with the FUNC kev.

When input has been made for 350°C, the upper limit value for the temperature in AUX1 oven, that has just been set, appears on the display. Then press the SHIFT. D | — / ESCP | ENT keys in that order to terminate the FUNC dialog.



It should be noted, however, that the function for requesting listing of parameters to the Chromatopac and the function of self-diagnosis are not included in this circular list and cannot be selected no matter how many times the ENT key is pressed.

If the function for setting parameters for the AOC is selected, each of the parameters involved is displayed in succession each time the ENT key is pressed.

AOC parameters available are the following 10 items. For details on them, please refer to the instruction manual for the AOC in use.

STRK	Syringe stroke
ISNO	Initial sample number
FSNO	Final sample number
REPT	Number of times of repetitious sampling
WASH	Selection of washing liquid
WSHT	Number of washing times
TWTE	Setting for waiting time
PROD	**Analysis** time
IJ1T	Number of times of injecting sample to the injection port No. 1
IJ2T	Number of times of injecting sample to the injection port No. 2

2. Explanation of Individual Keys

Key	Meaning	Example
START	(1) When pressed first after supplying power to the GC proper, it means starting of power supply to each of the temperature control zones. (Up till this moment, no power has been supplied to these temperature control zones.)	[START]
	(2) When pressed a second time and on after power is supplied to the GC, it means starting of the temperature program, time time program and automatic sample injector (AOC).	[START]
STOP	Executes stopping the operations started as per item (2) above. Relevant operation is executed from the beginning when the [START] key is pressed next time.	[STOP]
INIT TEMP	Sets initial temperature for column ovem or **AUXl.** If no program is involved, temperature is controlled at this setting. (isothermal analysis).	[COL/AUX1] [INIT TEMP] [1] [0] [0] [ENT] Column oven temperature is set at 100°C [SHIFT. D] [COL/AUX1] [INIT TEMP] [1] [0] [0] [ENT] Temperature for **AUXl** is set at 100°C.
INIT TIME	Sets intitial temperature hold time for column oven or **AUXI.** If the heating rate is set at 0°C/min, then the times set here becomes invalid.	[COL] [INIT TIME] [1] [0] [ENT] Initial time for column oven is set at 10 minutes. [SHIFT D.] [COL/AUX1] [INIT TIME] [1] [0] [ENT] Initial time for **AUXl** is set at **10** minutes.
PROG RATE	Sets heating rate (°C/min) for column oven or **AUXl** (Can be set in up to 5 stages.)	[COL] [PROG RATE] [5] [.] [2] [ENT] The 1st step heating rate is set at 5.2°C/min. Subsequent inputs with appropriate values allow setting for the 2nd and 3rd steps (and so on as required) for heating rate.
FINAL TEMP	Sets the temperature to be reached at the end of each rise stage of heating of the colum oven or **AUXl.**	[SHIFT.D] [COL/AUX1] [FINAL TEMP] [2] [1] [0] [ENT] 210° is set as the ultimate temperature to be attained in the 1st step of the heating of the **AUXl .** Subsequent inputs with appropriate values allows setting for the respective temperatures to be attained in the 2nd and 3rd steps of the program.

Key	Meaning	Example
FINAL TIME	Sets hold time for the final temperature of column overn or AUX1. (Final refers to the plateau, if any, after each temperature rise of the program.)	[COL] [FINAL TIME] [2] [0] [ENT] 20 minutes are set as the temperature hold time for the 1st step in the heating of the column oven. Subsequent inputs with appropriate values allows setting for the 2nd and 3rd steps (an so on as required) for the hold times in the different stages of the temperature program.
COL AUX1	(1) Designates whether the selected function from among INIT TEMP, INIT TIME, PROG. RATE, FINAL TIME or FINAL TEMP, mentioned above, is to be applied to the column overn or AUX1. Once selected, it is stored in the memory and is valid until AUX1 is selected. (2) In case of setting the temperature program, the step designation for PROG. RATE, FINAL TIME or FINAL TEMP is returned to the 1st step.	[COL/AUX1] * Any subsequent programming operations are for the column oven. [SHIFT.D] [COL/AUX1] * Subsequent programming operations are for the AUX1. *Pointers are all returned to the 1st step of the relevant program items.
INJ	Sets temperature for injection port.	[INJ] [2] [0] [5] [ENT]
TCD-T	Sets temperature for TCD.	[TCD-T] [3] [0] [0] [ENT]
DET	In setting parameters for a detector, (e.g. for setting POL, RANGE or CURR) designates the particular detector, which is to be selected for the setting, or on which some set value is to be read, by inputting the relevant detector number selected from among the detector Nos. 1 to 4. Once set, the setting is saved in the memory till the next setting is made.	[DET] [2] [ENT] Detector No.2 is selected, and the respective setting values for POL, RNG and CURR become effective with the detector No. 2 only.
RANGE	Sets Range for the selected detector.	[RANGE] [2] [ENT] 10^2 is selected as the range for the detector.
POL	Sets Polarity for the selected detector.	[POL] [1] [ENT] The signal output from the detector on the FLOW1 side will be positive.
CURR	Sets Current which is to be allowed to flow through the selected detector.	[CURR] [1] [0] [ENT] In case of TCD, 10mA is set as the current.

Key	Meaning	Example
FILE	Sets desired File No. from among 0 to 9. If set at a completely new number that has not been selected so far, or at a file that has once been erased by pressing the DEL key, a new file is opened with its contents being initialized. When a file No. for one that has already been used is set, that file is designated, and the GC is controlled according to the contents of that file. Accordingly, if the file No. is changed while the GC is at work, it is then controlled according to the contents of the file thus designated. Caution should be paid to this point when the GC is engaged in analysis.	[FILE] [1] [ENT]
STRK	Sets syringe stroke, where an AOC is attached. (Setting is possible only under PROG-controlled operation.)	
REPT	Where an AOC is attached, sets the number of times of injecting the same sample. (Setting is possible only under PROG-controlled operation.)	
FUNC	i) Sets conditions for an attached AOC. ii) Where a Chromatopac is connected via current loop, allows listing of the conditions set to the CRT or to the plotter of the chromatopac. iii) Setting max. temperature for each temp. control zone. iv) Self-diagnosis Setting is possible using any of the above-mentioned functions, through interactive operations with the display.	[SHIFT. D] [FUNC] [1] [ENT] Accesses the AOC parameter list with STRK as the first item displayed.
FLM	By adding a flow rate measurement unit, measurement of carrier gas flow rate is initiated by pressing this key.	It is necessary to set beforehand as to which flow rate is to be measured, by pressing the FLOW key followed by the relevant flow path number.
RET-T	A key dedicated to monitoring only, which allows display of the time elapsed from the pressing of START.	[MONIT] [SHIFT. D] [RET-T]

Key	Meaning	Example
HOLD-T	Used when it is desired to hold at the present temperature, while temperature program is running for either column overn or for AUX1. Can be held at the present temperature only for the period of time that has been set with this key.	SHIFT. D HOLD–T 3 ENT n this case, temperature is held for three ninutes.
STW	Enables the stop watch function. With the first push, display is cleared to start counting by 0.1 seconds, and the count is stopped when it is pressed the second time. When it is pressed the third time, the same state as in the first push is restored.	STW – STW
HOLD	Stops the time control. When it is pressed the first time, a HOLD message appears on the display. When it is pressed a second time, the HOLDing operation is released and the retention time is displayed. If the HOLD function is effected, the following functions are suspended: (while HOLD function is on) • AOC operation • PROG operation • Retention time • If in the HOLD TIME state in the temperature program mode, counting of the HOLD TIME is stopped. While heating is on in the program mode, however, no holding is done of any temperature reached during the heating.	HOLD – HOLD
PROG	In case of editing the time program, the interactive processes for the purpose are initiated. Thereafter, the dialog continues until the ESCP key is pressed.	SHIFT.D PROG ····· ESCP
EVENT	Can drive two internal relays (91, 92). Also, by adding a PRG PC board, externally arranged solenoid valves, or motors, can be driven, up to 10 elements in total.	SHIFT. D EVENT 9 1 ENT Relay No. 91 is turned on. If it is to be turned off, prefix with – sign before the desired numerals. (If this key operation is made at other times than in the course of executing the time program, the relay selected is put in motion instantaneously.)

Key	Meaning	Example
S-NO	i) Where an AOC is attached, the present sample number is indicated on the display. ii) Direct designation of the next sample	MONIT SHIFT.D S-NO
DEL	i) Deletion of contents of the selected file. ii) Deletes one step in the time program then in execution.	SHIFT.D DEL □ ENT All the contents of File No. **3** are deleted.
RETN	Allows setting only when PROG program is in execution. When a time program is executed, steps in the time program are repeated automatically with the selected File No. up to the particular step where this RETURN function is programmed beforehand. After having executed repetitively by the preset number of times, the fde is changed to the newly selected one for further execution.	SHIFT. D RETN 103 ENT 10³ └ No. of File to be executed next (selectable from among 0 to 9) └ Number of times to be executed repetitively with the present File No. (selectable from among **1** to 99.)
COPY	Used for copying the contents of the present file to **a** different file.	SHIFT.D COPY 4 ENT If the number of file then in use is 2, contents in said file are copied into File **4.**
STR T	Sets the wait time, the time duration till the starting of power supply to the heater in each of the temperature control zones after power supply to the GC. The time elapsed can be monitored by operating this key in combination with the MONIT key.	SHIFT.D STR-T 3 ENT START time is set at ***3*** minutes.
STP-T	Set the time before stopping power supply to the heater in each of the temperature control zones in the GC. When this time has elapsed, the GC enters the SLEEP state.	SHIFT.D STP-T □ ENT STOP time is set at 3 minutes.
SLP-T	Sets the time to be dormant after bringing the GC to the SLEEP state till power supply to the heater in each of the temperature control zones is to be resumed.	SHIFT.D SLP-T 5 ENT The GC will sleep for 5 minutes before power is resumed.
LOCK	Inhibits operation of all the keys, except for START and **STOP, SHIFT.** D and LOCK keys. Lock is released by pressing the LOCK key again.	SHIFT.D LOCK
SCAN	Used for cyclical display of actual measured temperatures in each of the temperature control zones. Each value is displayed for ca. *2* sec.	SHIFT.D SCAN

Key	Meaning	Example
ESCP	Used if it is desired to escape from the dialog state effected by pressing either the FUNC key or **PROG** key.	SHIFT. D ESCP
MONIT	Used when monitoring measured values.	MONIT COL Actual temperature measured in the column oven is displayed.
CE	Deletes the contents then indicated on the display. Used in case of an erroneous key operation. (In case an OVERheat alarm is given due to an overheating event in any of the temperature controlled zones, pressing this key will silence the alarm.)	
SHIFT. D	Used when selecting the function assigned to the lower part of each key.	
ENT	Used when entering numeric values or if it is desired to know the value that has already been entered.	

3. Control Parameters and Default Values

The following Table lists the reserved words of parameters available for control of the GC-14B, showing the parameters, maximum and minimum values available for input and their units. These can be used for setting conditions from an externally arranged computer, via RS-232C or current loop interface board (optional).

Parameter	Meaning	Max./Min. values	Unit	Default value
D1RG	DETECTOR 1 RANGE	0, 1, 2, 3	10^x	
D1PL	〃 POLARITY	2 (−), 1 (+)		
D1CR	〃 CURRENT		mA, nA	
D2RG	DETECTOR 2 RANGE			
D2PL	〃 POLARITY			
D2CR	〃 CURRENT		〃	
D3RG	DETECTOR 3 RANGE			
D3PL	〃 POLARITY			
D3CR	〃 CURRENT		〃	
D4RG	DETECTOR 4 RANGE			
D4PL	〃 POLARITY		〃	
D4CR	〃 CURRENT	0 ━ 200		
FLW1	FLOW CARRIER 1	0 — 999.8	mℓ/min	40.0
FLW2	〃 CARRIER 2	〃	〃	40.0
FLW3	〃 H_2 1		〃	30.0
FLW4	〃 H_2 2	〃	〃	30.0
FLW5	〃 AIR	〃	〃	200.0
INJT	INJECTION TEMP.	0 — 399	deg. ℃	200
DETT	**DETECTOR TEMP.**	0 — 399		200
AUXT	AUX. 2 TEMP.	0 — 399	〃	200
CITM	COLUMN INITIAL HOLD TIME	0 ~ 655	min	0.0
CITP	COLUMN INITIAL TEMP.	-99.0 ~ 399.0	deg. ℃	100.0
AITM	AUX1 INITIAL HOLD TIME	0— 655	min	0.0
AITP	AUX1 INITIAL TEMP	-99.0~ 399	deg. ℃	**100**
CPR1	COLUMN PROGRAM RATE1	0— 40.0	℃/min	0.0
CPR2	〃 2	〃	〃	〃
CPR3	〃 3	〃	〃	〃
CPR4	〃 4	〃	〃	〃
CPR5	〃 5	〃	〃	〃
CFP1	COLUMN FINAL TEMP1	-99.0 ~ 399.0	deg. ℃	200.0
CFP2	〃 2		〃	0.0

Parameter	Meaning	Max./Min. values	Unit	Default value
CFP3	〃 3	〃	〃	
CFP4	〃 4	〃	〃	〃
CFP5	5	〃	〃	
CFM1	COLUMN FINAL TIME 1	0 — 655	min	0.0
CFM2	〃 2	〃	〃	
CFM3	〃 3	〃	〃	〃
CFM4	4	〃	〃	〃
CFM5	〃 5	〃	〃	〃
APR1	AUX1 PROGRAM RATE1	0 — 250.0	℃/min	0.0
APR2	〃 2	〃	〃	
APR3	〃 3	〃	〃	
APR4	〃 4	〃	〃	〃
APR5	〃 5	〃	〃	〃
AFP1	AUX1 FINAL TEMP1	−99.0 — 399	deg.℃	200
AFP2	〃 2	〃	〃	0
AFP3	〃 3		〃	〃
AFP4	4	〃	〃	〃
AFP5	5	〃	〃	〃
AFM1	AUX1 FINAL TIME 1	0 — 655	min	0.0
AFM2	〃 2	〃	〃	
AFM3	〃 3	〃	〃	〃
AFM4	〃 4	〃	〃	〃
AFM5	〃 5	〃	〃	〃
TCDT	TCD TEMP	0 — 399	deg.℃	30
SNO	SAMPLE NUMBER	0 — 50		1
STRK	STROKE	10 — 80		
ISNO	INITIAL SAMPLE **NUMBER**	0 — 50		1
FSNO	FINAL SAMPLE NUMBER	0 — 50		49
REPT	REPEAT	1 — 10		1
WASH	WASHING LIQUID	0 (SAMPLE) 1 (SOLVENT)		1
WSHT	WASHING TIME	1 — 10		1
TWTE	TIME WAIT	1, 2, 3, ·····, 10		5
PROD	PERIOD	0 — 655	min	10
IJ1T	INJECTION1 TIME	0 ~ 10		1
IJ2T	INJECTION2 TIME	0 — 10		1
FLWK	FLOW K	0.01 — 999.8		500.0
DMAX	DETECTOR TEMP MAX	10 — 450	deg.℃	450

Parameter	Meaning	Max./Min. values	Unit	Default value
CMAE	COLUMN TEMP MAX	-99 ~ 400	〃	400
AMA)	AUXl TEMP MAX	-99 ~ 450	〃	〃
HLDA	HOLD AUXl	0 ~ 655	min	0.0
HLDC	HOLD COLUMN		〃	
SLPT	SLEEP TIME	0.1 — 3000	min	10
STRT	START TIME	0 — 3000	〃	〃
STPT	STOP TIME	0 — 3000		〃
LDVC	LINK DEVICE	0 - 55		0
STTS	STATUS (PRINT)			
FILE	FILE NUMBER	0 ~ 9		0
STAR	START			
STOP	STOP			
FLM	FLOW MEASURE			
AUXF	AUX. FUNCTION (PRINT)			
MON'I	MONITOR			
SSTR	SHIFT DOWN START			
SSTP	SHIFT DOWN STOP			
DET1	DETECTOR 1 (PRINT)			
RT	RETENTION **TIME (PRINT)**			
SCAN	SCAN			
COPY	FILE COPY	0 ~ 9		
KLOK	KEY LOCK	) (UNLOCK)		
		1 (LOCK)		
		2 (INVERSE)		
CKSM	CHECK SUM			
VALU	VALUE (PRINT)			
FNC$	FUNCTION (PRINT)			
DIGN	DIAGNOSTICS			
HOLD	HOLD	) (ON), 1(OFF)		
		3 (INVERSE)		
ESCP	ESCAPE			
EVNT	EVENT	0 - 255		
LPRG	LIST PROGRAM			
LAOC	LIST AOC **PARAMETERS**			
LPRM	LIST GC **PARAMETERS**			
AOC	40C (PRINT)			
PROG	PROGRAM			

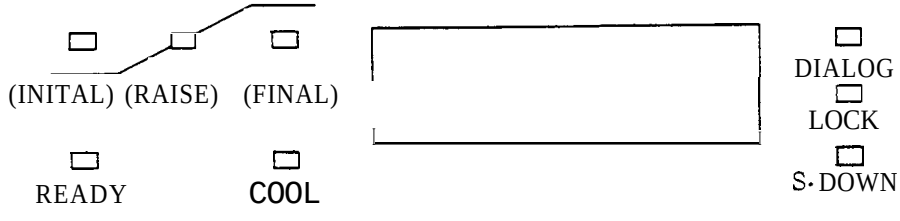
Param-eter	Meaning	Max./Min. values	Unit	Default value
TIME	TIME			
DELT	DELETE			
RETN	RETURN			
LTPR	LIST TIME PROGRAM			

Note HOLD TIME for COL/AUX1 is 655 minutes maximum including the total time for the HOLD TIME and the temperature program.

4. Meanings of Indication Lamps

READY	Lights when the temperature in the column oven is stabilized within ±1°C of the setpoint. The time program and/or temperature program and/or the AOC connected can be executed/operated. (START is possible.)
(FINAL)	Lights when the column oven temperature has risen to the next step and is held there in the temperature program mode. In case of a single-stage heating program, the temperature then equals the FINAL temperature.
(RAISE)	Lights when the column oven temperature is in the course of rising.
(INITIAL)	Lights when the column oven temperature, reading within ±1°C of the setpoint, is in the initial HOLD in the heating mode, or the time program or the AOC in use is at work.
COOL	Lights when the time of column oven FINAL temperature has elapsed in the program mode, and remains lit till it stabilizes at the initial temperature. If the INITIAL and the COOL lamps light simultaneously, it indicates that the heating program of the column oven has terminated and the AOC or the time program is now in the course of execution.
DIALOG	Lights while the interactive operations are going on after pressing the PROG key or FUNC key.
S. DOWN	When this lamp is on, it means that the key to be pressed next is in the SHIFT DOWN mode.
LOCK	When this lamp is lit, it indicates that all the key operations are locked.

Note In the transition period from FINAL, to COOL in the program for the column oven, a lamp other than COOL may light momentarily. This, however, can occur due to the time lag that can be caused with these indication lamps, because of the time-sharing software employed, and is not a symptom of failure.



5. Functionof Files

Ten files in all are made available from File Nos. 0 to 9, for accommodating such functions as temperature setting for individual temperature controlled zones, temperature programs, setting conditions for individual detectors, flow conditions, and time programs. Memory area for each file is not fixed so that as much memory area as would be required can be occupied depending on the size of each file. Therefore, the length of a time program may be made fairly long if the number of files used is only a few. (Example: About 250 steps are available when the number of previously opened files is only one.)

4

When a new file is opened, all of its contents are initialized bringing the time program for that file to the blank state. Deletion of a file that has been opened is accomplished by operating the DEL key. It should be noted that no deletion can be executed of the presently used file.

If key operations are made as SHIFT. D 4/DEL ☐ ENT , File *3* is deleted.

If it is desired to copy the contents of the present file to another file, copying is accomplished by using the COPY key. If key operations are made as SHIFT. 6/COPY 4 ENT , contents of the present file are copied into File 4. In such a case, it is necessary that the file selected as the destination for the copying should have never been opened before (that is, no setting has ever been made in that particular file in the past.) Copying to a file that has been opened in the past should be made by first deleting the contents in that file then by executing copying, otherwise, the contents of the destination file may be corrupt.

(Ex.) Copy to an already used file (No.4) as follows:

SHIFT. D 4/DEL 4 ENT

SHIFT. D 6/COPY 4 ENT

Display of **"LIMIT"** appearing during operations to a file indicates that there is no reserve for expansion of the file. In such a case, unwanted files need to be deleted to secure more memory.

Note Only one file can be designated at a time. Accordingly, in case of a change in file number, the GC is controlled from the moment of the change according to the contents of the file that has newly been selected. When temperature control operation is going on in the GC, the setting temperature may change in some cases if another file is selected inadvertently if the contents of that file have not been prepared correctly. To avoid such trouble, new setting for several files or checking of their contents should be done before pressing the START key.

* When power is turned ON, the default file number selected is always 0.

6. Error Messages, etc.

Error Message displayed	Contents
OVER COL	Temperature of the column oven exceeds preset MAX. temperature.
OVER INJ	Injection port or detector temperature exceeds preset MAX. temperature.
OVER AUX	Temperature of the oven controlled by AUX1 exceeds present MAX. temperature.
ERROR CM	File contents changed during the period of power off.
ERROR CR	Trouble has occurred in the transmission of data between this unit and a Chromatopac data processing unit.
ERROR 1	Error occurred in data transmitted or received.
ERROR 2	Inability of measurement with externally arranged flow rate measuring device (optional item (optional item)
ERROR 3	Operation error; no function available which corresponds to the one selected for input
ERROR 4	Operation error; value input is beyond the allowable limit.
NON	No detector attached at the selected DET NO. Or switch on the detector control unit is in the off state.
CITP. XX	Temperature control has not been started yet. Appears at the time of power supply.
SET. LDVC	When performing program listing, a number has not been set for the link device.
NOT. LINK	No linkage has been established between the 1/0 devices used for data transmission.
LIMIT	Given when no extra space is available for expansion of a file, or if heating program has b been set for 6 or more stages.
INITAL	Automatic initialization occurred as a result of a radical change in the data saved in the memory due to some reason such as lightning or freezing temperatures.
FAULT	Given in case of partial destruction of the data in ROM. A service request is necessary is such a case.

5. Operation 3

In this chapter, details of operation are described.

1. Temperature Setting

Contents

1.1 Outline

This unit allows temperature control at *six* points in all, of which programmed heating is possible for the column oven and for the AUX1.

This chapter describes in detail how temperature settings are made and how such heating programs are prepared.

1.2 Temperature setting procedures

1. Column oven

Temperature setting range for column oven: – 99°C to 399°C

When setting column temperature at 300°C, for example:

	Key operation	Display	
1.	COL AUX.1	INJT 200	Previous display remains as is, which in this case, is the injector temperature.
2.	INIT TEMP	INJT 200	
3.	□	3	
4.	0	30	
5.	0	300	
6.	ENT	CITP 300	No setting can be made unless the ENT key is pressed

To set at a temperature below 0°C, operate keys as " □ 5 and 0 ", for example.

2. Injection port

Temperature setting range for injection port: 0 to 399°C

(Default value is set at 200°C.)

(1) Setting procedure

When setting injection port temperature at 350°C:

	Key operation	Display	
1.	INJ AUX.2	CITP 300	Previous display remains as is, which, in this case, is the present value of the column oven temperature.
2.	□	3	
3.	5	35	
4.	0	350	
5.	ENT	INJT 350	This sets the injection port temperature at 350°C.

3. Detector

Temperature setting range for detector: 0 to 399°C

(Default vdue is set at 30°C.)

Setting procedure:

When setting detector temperature at 100°C:

	Key operation	Display	
1.	DET–T / TCD–T	INJT 200	Previous display remains as is, which, in this case, is the injection port temperature.
2.	1	1	
3.	0	10	
4. 1	0	100	
5.	ENT	DETT 100	Now, detector temperature is set at **100°C.**

5

4. AUXl

In this GC, temperature control of up to two auxiliary heating zones can be made, in addition to that for the column oven, detector and the injection port. (e.g. for AUXl and AUX2) Furthermore, programmed heating is made available for AUXl, in the same manner as for the column oven.

Temperature setting range for AUX1: -99°C to 399°C

Default value: Set at 100°C

Temperature setting procedure:

Ex. When temperature of AUXl is to be set at 150°C:

	Key operation	Display	
1.	SHIFT.D	CITP 200	Previous display remains as is, which, in this case, is the initial temperature of the column oven. S.DOWN lamp is lit when SHIFT.D key is pressed.
2.	COL / AUX. 1	CITP 200	SHIFT.D lamp goes out.
3.	INIT TEMP	CITP 200	
4.	1	1	
5.	5	15	
6.	0	150	
7.	ENT	AITP 150	Now, temperature at **AUX1** (initial temperature) is set at **150°C.**

5. AUX2

A heating block connected to AUX2 is controlled at a constant temperature; no programming is possible.

AUX2 temperature setting range: 0 to 399°C

Default value: Set at **200°C**

Setting procedure:

Ex. When setting **AUX2** temperature at 150°C:

	Key operation	Display	
1.	SHIFT.D	INJT 200	Previous display remains as is, which, in this case, is the injection port temperature. S.DOWN lamp is lit when this key operation is done.
2.	INJ. AUX.2	INJT 200	S.DOWN lamp goesout.
3.	1	1	
4.	5	15	
5.	0	150	
6.	ENT	AUXT 150	**Now,** AUX2 temperature is set at 150°C.

6. TCD

TCD temperature setting range: 0 to 399°C

Default value: Set at **200°C**

(1) Setting procedure

Ex. When setting TCD temperature at 150°C:

	Key operation	Display	
1.	SHIFT.D	INJT 200	Previous display still remains as is, which, in this case, is the injection port temperature. S.DOWN lamp is lit when this key is pressed.
2.	DET-T TCD-T	INJT 200	S.DOWN lamp goes out.
3.	1	1	
4.	5	15	
5.	0	150	
6.	ENT	TCDT 150	**Now,** TCD temperature is set at 150°C.

1.3 How to look up preset temperatures

If you wish to look up the temperature set for a zone, you can get the value displayed by effecting the following key operations. (Listing of preset values is also available.)

1. Column

	Key operation	Display	
1.	COL / AUX. 1	INJT 200	Previous display remains as is, which, in this case, is the injection port temperature.
2.	INIT TEMP		
3.	ENT	CITP 300	Preset value for column oven temperature is displayed, which, in this case, has been set at 300°C.

2. Injection port

	Key operation	Display	
1.	INJ / AUX. 2	CITP 300	Previous display remains as is, which, in this case, is the injection port temperature.
2.	ENT	INJT 200	Preset value for injection port temperature is displayed, which, in this case, has been set at 200°C.

3. Detector

	Key operation	Display	
1.	DET–T / TCD–T	INJT 200	Previous display remains as is, which, in this case, in injection port temperature.
2.	ENT	DET–T	Preset value for detector temperature is displayed, which, in this case, has been set at 30°C.

4. AUX1

	Key operation	Display	
1.	SHIFT D	DET–T 30	Previous display remains as is, which, in this case, is detector temperature. S.DOWN lamp is lit when this key is pressed.
2.	COL / AUX. 1		S.DOWN lamp goes out.
3.	INIT TEMP		
4.	ENT	AITP 150	Preset value for AUX1 temperature is displayed, which, in this case, has been set at 150°C.

5

5. AUX2

	Key operation	Display	
1.	SHIFT.D	AITP 150	Previous display remains as is, which, in this case, is AUX1 temperature.
2.	INJ / AUX.2		
3.	ENT	AUXT 250	Preset value for AUX2 temperature is displayed, which, in this case, has been set at 250°C.

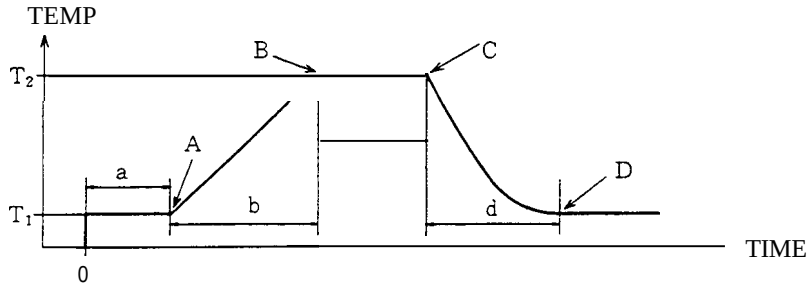
6. TCD

	Key operation	Display	
1.	SHIFT.D	AUXT 250	Previous display remains as is, which, in this case, in AUX2 temperature.
2.	DET-T / TCD-T		
3.	ENT	TCD-T 200	Preset value for TCD temperature is displayed, which, in this case, has been set at 200°C.

1.4 Heating program

Heating of the column oven can be executed by a desired multi-step heating program of up to five steps.

1.4.1 Basic single-step heating



Parameters necessary to prepare the single-step heating program of the heating pattern shown above are as follows:

		unit
INIT TEMP	setting temperature T_1	"C
INIT TEMP	setting time period a	min
PROG RATE	setting for $\frac{T_2 - T_1}{b}$	°C/min
FINAL TIME	setting time period c	min
FINAL TEMP	setting temperature T_2	°C

Key operations for inputting these parameters are done as follows:

[COL] [INIT TEMP] [6] [0] [ENT]

[INIT TIME] [1] [0] [ENT]

[PROG RATE] [2] [•] [5] [ENT]

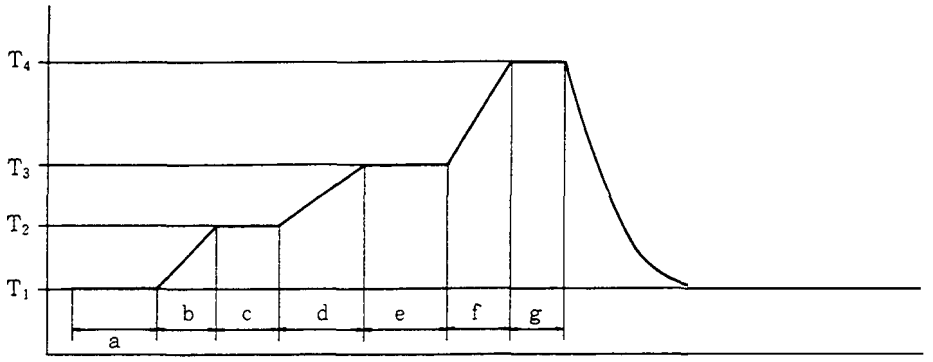
[FINAL TIME] [1] [5] [ENT]

[FINAL TEMP] [2] [0] [0] [ENT]

Through the above shown key operations, the necessary parametrs are set as shown below.

INIT TIME	10 minutes
INIT TEMP	60°C
PROG RATE	2.5°C/min
FINAL TIME	15 minutes
FINAL TEMP	200°C

1.4.2 Heating in multi-steps (case of 3-step heating)



In case of a multi-step heating program, necessary parameters are associated with respective keys as shown below.

5

INIT TIME ..... a

INIT TEMP ..... T_1

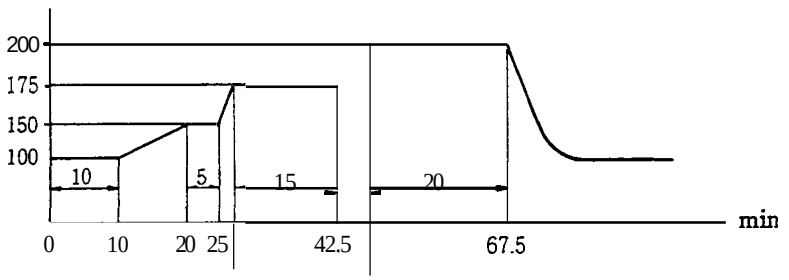
PROG RATE ..... $\frac{T_2-T_1}{b}$, $\frac{T_3-T_2}{d}$, $\frac{T_4-T_3}{f}$

FINAL TIME ..... c, e, g

FINAL TEMP ..... T_2, T_3, T_4

It is recommended that a list be prepared as shown below to show **all** the necessary parameters to facilitate subsequent setting.

	PROG RATE	TEMP	TIME	
Initial state	×	1 0 0	1 0	← Initial Temp, Time
1st step	5	1 5 0	5	} Final Temp, Time
2nd step	1 0	1 7 5	1 5	
3rd step	5	2 0 0	2 0	



Key operations for inputting parameters are done as follows:

COL	INIT TEMP	1 0 0 ENT
	INIT TIME	1 0 ENT
	PROG RATE	5 ENT 1 0 ENT 5 ENT
	FINAL TEMP	1 5 0 ENT
		1 7 5 ENT 2 0 0 ENT
	FINAL TIME	5 ENT 1 5 ENT 2 0 ENT

When preparing a multi-step heating program, one push of the COL key preceding inputs for the parameters effect those parameters set for the 1st step of the program. Thereafter, each round of parameter setting causes the steps to progress by one step without the need to push COL.

Unless there is change in function selected, you can go on pressing relevant numeral keys, followed with the pressing of the ENT key repetitively, for one step after another, to load a multi-step program.

In case of an erroneous loading of any of the parameters, a complete reloading from the beginning is necessary for an erroneous entry on Program Rate. If, however, an erroneous entry occurred for either Final Temp or Final Time, keep on pressing the ENT key, without pressing any numeral keys, until the value one step before the one to be corrected is indicated on the display. When it appears on the display, enter the right value via numeral keys and then press the ENT key to effect the correction.

Note Effective time for a heating program is 655 minutes in total.

Preparing Heating Program

1. Column oven

(1) Setting initial temperature

The initial temperature is the temperature that has been set in section 1.2 "Column Oven"

(2) Setting initial temperature HOLD time

Initial temperature HOLD time (Time period from START to the moment when the first heating rise is started.)

Allowable setting range 0 to 655 min

Default value 0.0 min

Setting procedure

Ex. When initial temperature HOLD time for column oven is to be set at 20 min:

	Key operation	Display	
1.	[COL/AUX. 1]	CITP 100	Previous display remains as is, which, in this case, is the initial temperature of the column oven.
2.	[INIT TIME]		
3	[2]	2	
	[0]	20	
4.	[ENT]	CITM 20.0	Initial temperature HOLD time for the column oven has been set at 20 minutes.

(3) Setting PROG RATE

PROG RATES are set for each of the heating rises to be programmed for each step of heating.

Allowable setting range: 0 to 40.0°C/min

Default value: at 0.0°C/min for each step

Setting procedure

Ex. Column heating rate in step 1. at 5°C/min

" step 2. at 20°C/min

" step 3. at 5°C/min

" step 4. at 10°C/min

" step 5. at 20°C/min

	Key operation	Display	
1	COL / AUX 1	CITM 20.0	Previous display remains as is, which, in this case is the initial temperature HOLD time for the column oven.
2	PROG / RATE		
3	□	5	
4	ENT	CPR1 5.0	PROG RATE 1 for the column oven is set at 5.0°C/min.
5	2	2	
	0	20	
6	ENT	CPR2 20.0	PROG RATE 2 for the column oven is set at 20.0°C/min.
7	5	5	
8	ENT	CPR3 5.0	PROG RATE 3 for the column oven is set at 5.0°C/min.
	1	1	
	0	10	
10	ENT	CPR4 10.0	PROG RATE 4 for the column oven is set at 10.0°C/min.
	2		
	0		
12	ENT	CPR5 20.0	PROG RATE 5 for the column oven is set at 20.0°C/min.

Note From then on, even if numerals are input followed with pressing of the ENT key, no changes occurs in the display, etc., as the limit of the steps for P.RATE has been reached.

(4) FINAL TEMP

This sets the final temperature for each step in the programmed heating. The final temperature set for a step means the starting temperature for the next step in the heating program. It does not matter if the final temperature set for a step is lower than that for the preceding step.

Allowable setting range: -99 to 399°C

Default value: at 200°C for FINAL TEMP 1, others are set at 0°C

Setting procedures:

Ex. When FINAL TEMP settings for the column oven are to be set as follows:

FINAL **TEMP** for step 1 ..... at 200°C
step 2 ..... at 300°C
step 3 ..... at 100°C
step 4 ..... at 250°C
step 5 ..... at 350°C

	Key operation	Display	
1	COL AUX. I	CPR5 20.0	Previous display remains as is, which in this case is the PROG RATE 5 for the column oven.
2	FINAL TIME		
3	2	2	
	0	20	
	0	200	
4	ENT	CFP 1 200	FINAL TEMP 1 for the column oven is set at 200°C.
5	3	3	
	0	30	
	0	300	
6	ENT	CFP2 300	FINAL TEMP 2 for the column oven is set at 300°C.
7	1	1	
	0	10	
	0	100	
8	ENT	CFP 3 100	FINAL TEMP 3 for the column oven is set at 100°C.
9	2	2	
	5	25	
	0	250	
10	ENT	CFP4 250	FINAL TEMP 4 for the column oven is set at 250°C.
11	3	3	
	5	35	
	0	350	
12	ENT	CFP5 350	FINAL TEMP 5 for the column oven is set at 350°C.

(5) FINAL TIME

Sets find temperature HOLD time for each step in the programmed heating.

Allowable setting range: 0 to 655 rnin

Default value: 0.0 min for all steps

Setting procedure:

Ex. When setting **FINAL TIMEs** for the steps as follows:

Step 1 ..... at 30 min

Step 2 ..... at 0 min

Step 3 ..... at 20 rnin

Step 4 ..... at 0 min

Step 5 ..... at 60 min

	Key op'on	Display	
1	COL AUX. 1	CFP5 350	Previous display remains as is, which in this case is the FINAL TEMP 5 for the column oven.
2	FINAL TIME		
4	3	3 1	
	0	30	
5	ENT	CFM1 30.0	FINAL TIME 1 for the column oven is set at 30.0 min.
6	0	0	
7	ENT	CFM2 0.0	FINAL TIME 2 for the column oven is set at 0.0 min.
8	2	2	
	0	20	
9	ENT	CFM3 20.0	FINAL TIME 3 for the column oven is set at 20.0 min.
10	a	0	
11	ENT	CFM4 0.0	FINAL TIME 4 for the column oven is set at 0.0 min.
12	6	6	
	0	60	
13	ENT	CFM5 60.0	FINAL TIME 5 for the column oven is set at 60.0 min.

2. AUX1

	Key operation	Display	
1	SHIFT.D	CITP 100	Previous display remains as is, which in this case is the INIT TEMP of the column oven.
2	COL AUX.1		
3	INIT TIME		
4	3	3	
	0	30	
5	ENT	AITM 30.0	INIT TIME for AUX1 is set at 30 min.

(3) Setting PROG RATE

Allowable setting range: 0 to 250.0°C/min

Default value: 0.0°C/min for all steps

Setting procedure:

Ex. When setting PROG RATE for the steps as follows:

Step 1 ..... at 10°C/min

Step 2 ..... at 20°C/min

Step 3 ..... at 5°C/min

Step 4 ..... at 2.5°C/min

Step 5 ..... at 10°C/min

	Key operation	Display	
1	[SHIFT.]	AITP 100	Previous display remains as is, which in this case is the INIT TEMP for AUX1.
2	COL AUX.1		
3	PROG RATE		
4	1	1	
	0	10	
5	ENT	APR1 10.0	PROG RATE 1 for AUX1 is set at 10.0°C/min.
6	2	2	
	0	20	
7	ENT	APR2 20.0	PROG RATE 2 for AUX1 is set at 20.0°C/min.
8	5	5	
9	ENT	APR3 5.0	PROG RATE 3 for AUX1 is set at 5.0°C/min.
10	2	2	
	.	2	
	5	2.5	
11	ENT	APR4 2.5	PROG RATE 4 for AUX1 is set at 2.5°C/min.
12	1	1	
	0	10	
	ENT	APR5 10.0	PROG RATE 5 for AUX1 is set at 10.0°C/min.

(4) Setting FINAL **TEMP**

Allowable setting range: -99 to 399°C

Default value: 0°C for **all** the steps except step 1 which is set at 200°C

Setting procedure:

Ex. When setting FINAL TEMPs for the steps as follows:

Step 1 ..... at 200°C

Step 2 ..... at 300°C

Step 3 ..... at 100°C

Step 4 ..... at 150°C

Step 5 ..... at 300°C

5

	Operation Key	Display	
1	SHIFT D	APR5 10.0	Previous display remains as is, which in this case is the PROG RATE 5 for AUX1.
2	FINAL TIME		
3	2	2	
		20	
	0	200	
4	ENT	AFP1 200	FINAL TEMP 1 for AUX1 is set at 200°C.
5	3	3	
	0	30	
	0	300	
6	ENT	AFP2 300	FINAL TEMP 3 for AUX1 is set at 300°C.
7	1	1	
	0	10	
	□	100	
8	ENT	AFP3 100	FINAL TEMP 3 for AUX1 is set at 100°C.
9	⊟	1	
	5	15	
	0	150	
10	ENT	AFP4 150	FINAL TEMP 4 for AUX1 is set at 150°C.
11	3	3	
	□	30	
	0	300	
	▼	AFP5 300	FINAL TEMP 5 for AUX1 is set at 300°C.

(5) **Setting** FINAL TIME

Allowable setting range: **0 to *655* min**

Default value: **0 min for all the steps**

Setting procedure:

Ex. When setting FINAL TIMEs **for the steps as follows:**

Step 1 ..... at 10 min

Step 2 ..... at 0 min

Step 3 ..... at 30 min

Step 4 ..... at 20 min

Step 5 ..... at 20 min

	Key operation	Display		
1	SHIFT.D	AFP 5	30	**Previous display remains as is,** which **in this** case **is the FINAL** TEMP **5.**
2	COL AUX. 1			
3	FINAL TIME			
4	1		1	
	0		10	
5	ENT	AFM1	10.0	**FINAL TIME 1 for AUX1 is set at** 10 min.
6	0		0	
7	ENT	AFM2	0.0	**FINAL TIME 2** for **AUX1 is set at 0 min.**
	3		3 1	
	0		30	
9	ENT	AFM3	30.0	**FINAL TIME 3 for AUX1 is set at 30 min.**
10	2		2	
	0		20	
11	ENT	AFM4	20.0	**FINAL TIME 4 for AUX1 is set at 20 min.**
12	2			
	0		20	
13	ENT	AFM5	20.0	**FINAL TIME 5 for AUX1 is set at 20 min.**

3. General precautions

[COL] and [AUX.1] keys

It has been described, in setting for a heating program, that either the [COL] key or [AUX 1] key should be pressed first to designate whether the setting is for the column oven or for AUXl (by pressing [SHIFT.D] and [COL / AUX.1] keys for AUX1. Refer to the relevant paragraphs on column oven temperature and AUXl temperature.)

Either of these two keys, however, remains effective, once pressed, till the other one is pressed. If setting is being done for the column oven, for example, you don't have to press the [COL] key each time you set for PROG RATE or FINAL TEMP, but you may press directly either the [PROG RATE] or [FINAL TEMP] key.

5

	Key operation	Display	
1.	[COL / AUX. 1]	AITP 100	Previous display remains as is, which in this case is the initial temperature of **AUXl.** This remains effective till next operation is performed as [SHIFT.D] [COL / AUX.1] and [ENT]. (Or, till some other operation is performed for AUX1.)
2.	[INIT TIME]	AITP 100	
3.	[1]	1	
4.	[0]	10	
5.	[ENT]	CITM 10.0	Setting for initial temperature HOLD time for the column oven.
6.	[PROG RATE]	CITM 10.0	
7.	[5]	5	
8.		CPRl 5.0	Setting **PROG RATE** for the column oven.
9.	[FINAL TEMP]	CPRl 5.0	
10.	[2]	2	
11.	[ ]	20	
12.	[0]	200	
13.	[ENT]	CFP1 200	Setting **FINAL** TEMP for the column oven.

Once either the column oven or AUX1 is selected, no change occurs in that status till the other one is selected.

	Key operation	Display	
1.	COL / AUX. 1	CITP. 100	Previous display remains as is, which in this case is the **initial** column oven temperature.
2.	ENT	CITP. 100	Column oven is selected by this operation.
3.	INIT TIME	CITP. 100	
4.	ENT	CITP 100	Observation of the setting of the initial temperature of the column oven.
5.	INIT TIME	CITP 100	
6.	ENT	CITM 5.0	Observation of the initial temperature HOLD time for the column oven.
7.	MONIT	CITM 5.0	
8.	SHIFT. D	CITM 5.0	S.DOWN lamp lights.
9.	COL / AUX. 1	AITP. 150	**Now,** the temperature of AUX.1 is monitored. But, this is not the operation for selecting **AUX.l** status.
10.	PROG RATE	AITP. 150	
11.	ENT	CPR 1 3.0	PROG **RATE** for the column oven is displayed.

Precautions for programming

In preparing a temperature program, for example, while programming for heating the column oven, you can proceed to set for INIT TEMP, INIT TIME, PROG RATE, FINAL TEMP and FINAL TIME in an optionally selected sequence.

	Key operation	Display	
1.	COL / AUX. 1	INITIAL	Previous display remains as is, which in *this* case is the initialized state.
2.	INIT TEMP	INITIAL	
3.	1 0 0		
4.	ENT	CITP 100	INIT TEMP .....100°C
5.	INIT TIME	CITP 100	
6.	1 0	1 10	

7.	[ENT]	CITM 10.0	INIT TEMP HOLD time ..... 10 min
8.	[PROG RATE]	CITM 10.0	
9.	[5]	5	
10.	[ENT]	CPR1 5.0	PROG RATE 1 ..... 5°C
11.	[ ] [ ]	1 10	
12.	[ENT]	CPR2 10.0	PROG RATE 2 ..... 10°C/min
13.	[FINAL TEMP]	CPR2 10.0	
14.	[1] [5] [0]	1 15 150	
15.	[ENT]	CFP1 150	FINAL *TEMP* 1 ..... 150°C
16.	[2] [0] [0]	2 20 200	
17.	[ENT]	CFP2 200	FINAL *TEMP* 2 ..... 200°C
18.		CFP2 200	
19.	[ ] [0]	1 10	
20.	[ENT]	CFM1 10.0	FINAL *TEMP* 1 ..... 10.0 min
21.	[0]	0	
22.	[ENT]	CFM2 0.0	FINAL *TEMP* 2 ..... 0.0 min
23.	[3] [0]	3 30	
24.	[ENT]	CFM3 30.0	FINAL *TEMP* 3 ..... *30* min
25.	[PROG RATE]	CFM3 30.0	
26.	[5]	5	
27.	[ENT]	CPR3 5.0	PROG RATE *3* ..... 5.0°C
28	[FINAL TEMP]	CPR3 5.0	
29	[1] [5] [0]	1 15 150	
30	[ENT]	CFP3 150	FINAL *TEMP 3* ..... 150.0°C

Even if other operations as for setting INJ temperature, for example, are done midway in the programming operation, the parameter set can be programmed for the next step succeedmg the one for which a setting has been made, if any one of the program parameters is keyed in. By pressing [FINAL TEMP] key, for example, the next F.TEMP step can be programmed. It should be noted, however, that any key operations prefixed by the [COL AUX.1] key, e.g., [COL AUX.1] [ENT], or [COL AUX.1] [PROG RATE] [1] [0] [ENT], etc., resets the step pointer to step No. 1 rendering the subsequent setting as starting with step 1.

If the "LIMIT" message should appear in the display in the course of setting, it indicates that setting has exceeded the limit of the five steps allowed. For example, if further key-in operation is performed by pressing [FINAL TEMP] [3] [0] [0] [ENT], while settings have already been made up to CFP5, a "LIMIT" message is displayed.

However, if you tried to set FINAL TEMP by pressing the [ENT] key only right after having set CFP5, for example, immediately before it, no such "LIMIT" message appears, as shown below.

	Key operation	Display	
1.	[FINAL TEMP]	CPR5 10.0	Previous display remains as is.
2.	El □ ⊟	3 30 300	
3.	[ENT]	CFP5 300	Setting for FINAL TEMP 5.
4.	[5] □	3 35 350	
5.	[ENT]	350	Display does not change, with nothing being set indicating that it is a setting intended for a step beyond the allowable 5 steps.

1.5 How to look up a preset program

If you wish to look up the contents of the preset program, proceed as follows.

Column oven heating program

(1) INIT TEMP (Initial Temperature)

	Key operation	Display	
1	COL AUX.1	INJT 200	Previous display remains as is, which in this case is the injection port temperature which has been set at 200°C.
2	INIT TEMP		
3	ENT	CITP 100	Preset initial temperature of the column ovenisdisplayed.

5

(2) INIT TIME (Initial Temperature HOLD Time)

	Key operation	Display	
1	COL AUX. 1	INJT 200	Previous display remains as is, which in this case is the injection port temperature which has been set at 200°C.
2	INIT TIME		
3	ENT	CITM 10.0	Preset INIT TIME (Initial temperature HOLD time) is displayed, which in this case has been set at 10 min.

(3) PROG RATE

	Key operation	Display	
1	COL AUX. 1	CITM 10.0	Previous display remains as is, which in this case is the INIT TIME.
2	PROG RATE		
3	ENT	CPR1 5.0	PROG RATE 1 is displayed, which in this case has been set at 5.0°C/min.
4	ENT	CPR2 10.0	PROG RATE 2 is displayed.
5	ENT	CPR3 5.0	PROG RATE 3 is displayed.
6	ENT	CPR4 2.5	PROG RATE 4 is displayed.
7	ENT	CPR5 0.0	PROG RATE 5 isdisplayed.

(4) FINAL TEMP

	Key operation	Display	
1	COL / AUX.1	CPR5 0.0	Previous display remains as is, which in this case is PROG **RATE** 5.
2	FINAL TEMP		
3	ENT	CFP1 200	FINAL TEMP 1 is displayed, which in this case has been set at 200°C.
4	ENT	CFP2 250	FINAL TEMP 2 is displayed.
5	ENT	CFP3 150	FINAL TEMP 3 is displayed.
6	ENT	CFP4 250	FINAL TEMP 4 is displayed.
7	ENT	CFP5 0	FINAL ***TEMP*** 5 is displayed.

(5) FINAL TIME

	Key operation	Display	
1	COL / AUX.1	CFP5 0	Previous display remains as is, which in this case is FINAL TEMP 5.
2	FINAL TIME		
3	ENT	CFM1 0.0	FINAL **TIME** 1 is displayed, which in this case, has been set at 0.0 min.
4	ENT	CFM2 10.0	FINAL TIME 2 is displayed.
5	ENT	CFM3 30.0	FINAL TIME 3 is displayed.
6	ENT	CFM4 0.0	FINAL TIME 4 is displayed.
7	ENT	CFM5 0.0	FINAL **TIME** 5 isdisplayed.

AUX.1 heating program

Contents of the heating program can be looked up in the same manner as described above. In this case, key operation is to be made as SHIFT.D COL / AUX 1, instead of the pressing of only the COL / AUX.1 key. Formats of displays given to indicate the initial temperature, etc. are as shown below.

AUX.1 INIT TEMP ..... AITP
INIT TIME ..... AITM
PROG RATE 1 ~5 ..... APR1 ~ 5
FINAL TEMP 1 — 5..... AFP1 ~ 5
FINAL TIME 1~ 5 ..... AFM1 ~ 5

Precation: [COL AUX.1] key

As was described in the section on how to prepare a heating program, key-in operations as [COL AUX 1] [ENT] , or as [COL AUX.1] [INIT TEMP] [ENT] , or [COL AUX.1] [PROG RATE] [ENT] , ... etc. cause respective displays to be returned to the first step in the heating program. Therefore, the following operations are also possible, fore xample:

	Key operation	Display	
1	[COL AUX. 1]		Display is **cleared.**
2	[INIT TEMP]		
3	[ENT]	CITP 100	**INIT** TEMP
4	[INIT TIME]		
5	[ENT]	CITM 10.0	**INIT TIME**
6	[PROG RATE]		
7	[ENT]	CPR 1 5.0	**PROG RATE 1**
8	[FINAL TEMP]		
9	[ENT]	CFP 1 150	**FINAL TEMP 1**
10	[FINAL TIME]		
11	[ENT]	CFM 1 0.0	**FINAL TIME** 3
12	[PROG RATE]		
13		CPR2 10.0	**PROG RATE 2**
14	[ENT]	CPR3 5.0	**PROG RATE 3**
15	[FINAL TEMP]		
16	[ENT]	CFP2 250	**FINAL *TEMP* 2**
17	[ENT]	CFP3 150	**FINAL TEMP 3**

By pressing the [COL AU .1] key here, display returns **again** to step 1.

20	[ENT]	CPR1 5.0	**PROG RATE 1**
21	[FINAL TEMP]		
22	[ENT]	CFP 1 150	**FINAL TEMP 1**
23	[ENT]	CFP2 250	**FINAL TEMP 2**

1.6 Partial change to a program

If partial change to the heating program is required, proceed as follows.

Ex. When column FINAL TEMP 3 is to be changed to 150°C, and FINAL TIME 2 is to be changed to 20 min.

	Key operation	Display	
1	COL / AUX 1	CITP 100	Previous display remains as is, which in this case, is the INIT TEMP of the column oven.
2	FINAL TEMP		
3	ENT	CFP 1 150	FINAL TEMP 1 is displayed.
4	ENT	CFP 2 250	FINAL TEMP 2 is displayed. Key in the desired value while the item just before the one to be changed is still displayed.
5	1	1	
6	5	15	
7	0	150	
8	ENT	CFP3 150	FINAL TEMP 3 is set at 150°C.
9	FINAL TIME		
10	ENT	CFM 1 0.0	Key in desired value in the same manner while the item just before the one to be changed is on display.
11	2	2	
12	0	20	
13	ENT	CFM 2 20.0	FINAL TEMP 2 is set at 20 min.

Note Once PROG RATE is set, the PROG RATE for the next step is automatically set at 0°C/min.

This is to prevent an unwanted heating rise to be executed if the value in the new program is smaller than that in the previous program in preparting the heating program. (No heating is performed for the step if the PROG RATE for the step has been set at 0°C/min.)

Accordingly, in case of a partial change attempted in the PROG RATE, it is necessary to set all the PROG RATEs again for the steps following the step for which the PROG RATE has been changed.

1.7 Setting the maximum temperature

For both the column ovem and the AUX.1, it is possible to set the upper limits for their temperatures. Temperature control will stop if the temperature in either of these parts should exceed the preset upper limit.

(1) Maximum value for column oven temperature

Maximum value for column temperature can be set within the range of –99°C to 400°C. (Default value: **400°C)** If the column oven exceeds the preset maximum value, the "OVER COL" warning message appears on the display, and the buzzer sounds continously and heating with the oven heater is stopped. To release this, either press the [CE] key when the column oven temperature has fallen sufficiently, or set the maximum value for the column oven temperature at a new value which is higher than the present column oven temperature, and then press the [CE] key.

Setting procedure (for setting at 350°C)

	Key operation	Display	
1	[SHIFT.D]	CITP 300	Previous display remains as is, which in this case is the column temperature that has been set at **300°C.** S.DOWN lamp is lit up.
2	[FILE/FUNC]		
3	[7]	7	S.DOWN lamp goes out.
4	[ENT]	CMAX 250	Displayed is the max. value then ***set*** for column oven temperature. DIALOG lamp is lit up.
5	□	3	
6	[5]	35	
7	□	350	
8	[ENT]	LDVC 11	**Now,** the upper limit for column oven temperature is set at 350°C, with display shifted one function forward in the list.
9	[SHIFT.D]		S.DOWN lamp is lit up.
10	[ESCP]		While the display remains **as** is, the DIALOG lamp goes out.

(2) Maximum value for AUX. 1 temperature

Maximum value for AUX. 1 temperature can be set within the range of –99°C to 450°C. (Default value: 400°C)

If AUX.1 temperature should exceed the preset maximum value, the "OVER AUX" warning appears on the display and the buzzer keeps on sounding. At the same time, heating with the heater is stopped. To release this condition, press the [CE] key when the temperature of AUX. 1 has fallen sufficiently, or set a new value, which is higher than the present max. AUX. 1 temperature, as the revised max. oven temperature, and press the [CE] key.

Ex. When setting the upper limit for AUX1 temperature at 350°C:

	Key operation	Display	
1	[SHIFT. D]	AITP 150	Last display remains as is, which in this case is the INIT TEMP set for AUX.1. S.DOWN lamp is lit up.
2	[FILE / FUNC]		S.DOWN lamp goes out.
3	[6]	6	
4	[ENT]	AMAX 200	Set value for max. AUX1 temperature is displayed, with the DIALOG lamp lit up. Skip to step 9 if no change is desired for the value.
5	[□]	3	
6	[5]	35	
7	[0]	350	
8	[ENT]	CMAX 400	350°C is set for the max. AUX1 temperature, with display shifted one function forward in the list.
9	[SHIFT]		S.DOWN lamp is lit up.
10	[ESCAPE]		Both DIALOG and S.DOWN lamps go out.

Operations 9 and 10 are intended to let you escape from the DIALOG mode, hence they are musts before performing other operations.

2. Detectors

Contents

5

2.1 Outline

Five different types of detectors, e.g. FID, TCD, FTD, FPD and ECD, are available for connection with this unit. Matching these different types of detectors with particular numbers selected from among DETECTOR 1 to 4 has been determined beforehand on the detector PC board, at the time of shipment or installation of the unit.

2.2 Selecting a particular detector

Selection must be made as to which one of the detectors provided for the unit is to be used for the analysis.

	Key operation	Display	
1	DET	CITP 100	The last display remains as is, which in this case is the column oven temperature, which has been set at 100°C.
	4	4	
3	ENT	DET 4	Now, detector 4 is selected. If a TCD is connected as DETECTOR 4, for example, the TCD is selected as the detector for use.

2.3 How to look up the selected detector

a. **To** look up the number of the detector presently selected

	Operation	Display	
1.	DET	INJT 200	The last display remains as is, which in this case is the injection port temperature, which has been set at 200°C.
2.	ENT	DET 4	The number assigned to the selected is displayed, which in this case is DETECTOR 4.

Note When performing this operation, if the display given already consists only of a number, that value is set for the DETECTOR No. To avoid this, such a display should be cleared beforehand by pressing the CE key.

	Operation	Display	
1.	DET	2	A numeral key, e.g. key *2* in this case, was pressed before this operation with *2* remaining on the display.
2.	ENT	DET 2	Now, DETECTOR *2* is set.

b. **To** identify the type of detector with the assigned detector number (from *among* **DETECTOR** Nos. **1 ~ 4)**

	Operation	Display	
1	MONIT	INJT 200	
2	DET	INJT 200	
3	2	2	Enter DETECTOR No. *2*.
4	ENT	FID	Indicates that DETECTOR *2* is FID.

2.4 RANGE, POLARITY and CURRENT

a. Setting procedure

After selecting a particular one of the detectors, RANGE, POLARITY and CURRENT are set for the detector (as applicable).

Ex. Setting POLARITY

	Key operation	Display	
1.	[POL]	DET 4	The last display remains as is, which in this case is selection of DETECTOR No. 4.
2.	[1]	1	
3.	[ENT]	D4PL 1	Now, "1" is set as the POLARITY of DETECTOR 4. (That is, signal is output as positive when sample is injected into the injection port at side 1.)

Note If only the numeric value entered remains in the last display appearing after these key-in operations, it indicates that no parameter settings have been made for that particular detector. In such a case, some other necessary parameters settings must first be made for the detector.

If you attempt to set kwith any value that is beyond the allowable setting range specified for the detector (in this regard, please refer to the section concerning these detectors), an "ERROR 4" message appears on the display, with no change occurring from the last setting value.

In case of a setting for RANGE, POLARITY or CURRENT, attempted for a DETECTOR No., to which no detector is connected, or in case of a setting attempted for such an item that is prohibited for that detector, (refer to the section on Detectors) a "NON" message will appear, telling that such a setting is not possible for that detector or the detector doesn't exist (the switch might be OFF).

3. Starting and Ending Temperature Control

Contents

3.1 Outline

Upon completion of settings for temperature, temperature control operation is started. At this time, the starting and the ending times can be set for the intended temperature control.

3.2 Starting and ending temperature control

By pressing the [START] key, controlled heating and holding operations are started at each of the temperature-controlled zones, toward the respective setpoints. For the column oven and the AUX.1, temperature control is executed at their respective INIT Temps. The READY lamp lights up when the column oven temperature has become settled within ±1°C of the set value.

3.3 Timer functions

Starting and ending the temperature control can be done automatically by utilizing the following timer functions.

(1) SHIFT DOWN Start

This operation is performed when it is desired to start the temperature control after a certain time delay, while no present temperature control is being done.

(a) First, the START TIME is set, which is the time period desired before starting the temperature control. Duration of this **START** TIME is set at the default value of 10 minutes at the time of turning on power. It can be set at a desired value within the range of 0.1 min to 3000.0 min. It should be noted that the START TIME appearing on the display is the value obtained by counting fractions over 5 as the unit and disregarding the rest in tenths

place. For example, if it is set at 0.1 min, display will appear as START TIME "0", but actually it is set exactly at 0.1 min (or 6 seconds).

(Ex. When setting START TIME at 10 hr)

	Key operation	Display	
1.	SHIFT. D	CPR 4 5.0	The last display remains as is, which in this case is column oven PROG RATE 4. S.DOWN lamp is lit.
2.	1 / STRT	CPR 4 5.0	S.DOWN lamp goes out.
3.	□	6	
4.		60	
5.	0	600	
6.	ENT	STRT 600	600 min (10 hr) is set for START TIME.

(b) Execution

The following key operations are to be performed to get the temperature control started after the expiration of the START TIME in actual operation.

	Key operation	Display	
1.	SHIFT. D	CITP 100	The last display remains as is, which in this case is he-column oven INIT TEMP, which has been set at 100°C. S.DOWN lamp is lit.
2.	START	SLEEP	Display changes to read as shown on the left, entering a WAIT condition, which last for the time set for the START TIME.
		* *	Temperature control starts when the time set for the START TIME has expired.

If the above key operations are performed while the temperature control is going on, illuminaation of READY lamp goes out momentarily with display changing to read "SLEEP". But, the temperature control is resumed immediately thereafter, with the display appearing as "**".

Note Execution of these key-in operations while the heating program is going on brings about a HOLD state, with the counting of retention time being suspended. To leave this state, either press the HOLD key, or press the START key again. Note that, upon completion of the execution of the program, display changes to "**".

(2) SHIFT DOWN Stop

While the temperature control is going on, it can be suspended temporarily after a certain period of time and then can be resumed for continued operation.

(a) Setting

First, the STOP TIME (the time period up to the moment when the temperature control is to be suspended) is to be set. The default value for the STOP TIME 10 min, and it can be set within the range from 0.1 to 3000 min. Setting can be made down to the tenths place, but in such a case, the value on the display will appear by counting fractions over 5 as the unit but disregarding the rest.

Ex. When setting STOP TIME at 5 hr

	Operation Key	Display	
1.	SHIFT. D	CITP 100	The last display remains as is, which in this case is the column oven INIT TEMP. S.DOWN lamp is lit up.
2.		CITP 100	S.DOWN lamp goes out.
3.	3	3	
4.	0	30	
5.	□	300	
6.	ENT	STPT 300	5 hr (300 min) is set for STOP TIME.

Next the SLEEP TIME (the time period during which the temperature control is to be suspended) is set, which lasts for 10 min as the default value.

SLEEP TIME can be set as desired within the range from 0.1 to 3000 min. It may be set down to the tenths place, but the value appearing on the display is rounded off by counting fractions over 5 as the unit but disregarding the rest.

Ex. When setting SLEEP TIME at 12 hr

	Key operation	Display	
1.	SHIFT. D	INJT 200	The last display remains as is, which in this case is the injection port temperature. S.DOWN lamp is lit up.
2.	3 / SLP.T	INJT 200	S.DOWN lamp goesout.
3.	7	7	
4.	2	72	
5.	0	720	
6.	ENT	SLPT 720	12 hr (720 min) is set for SLEEP TIME.

5

If you attempt to set a value beyond the allowable limit for the setting, an "ERROR 4" message appears with no change occurring in the previously registered value.

(c) Execution

The following key operations are to be performed to have the operations of stopping and starting of temperature control executed in actual operation.

	Key operation	Display	
1.	SHIFT. D	CITP 100	The last display remains as is, which in this case is the INIT TEMP of the column oven. S.DOWN lamp is lit up.
2.	STOP	WAIT	Message as shown on the left appears on the display, and temperature control is carried out for the period as has been set for STOP TIME.
		SLEEP SLEEP	After the expkation of the time set as the STOP TIME, display changes to read as shown on the left, and the temperature control stops. Remains in standby for the period of time as has been set for the SLEEP TIME.
		* *	Temperature control is resumed after the time set for the SLEEP TIME has expired.

No change occurs if the above operation is executed while temperature control is not going on.

If the START key is pressed to have the program executed while in the WAIT state (standby) while performing temperature control, execution of the program is started and it is kept on to the last even if the preset STOP TIME expires before the completion of the

program, i.e., the START key has priority override. If the set STOP TIME has not expired yet even when the program has come to the end, the GC remains in standby while performing temperature control, with READY lamp remaining lit. If the STOP TIME has expired already, the temperature control terminates at once.

If SHIFT DOWN STOP operation is made while the heating program is going on, the situation is a little different from that described above; counting of the STOP TIME starts after completing the execution of the program. Therefore, the temperature control comes to a stop when the STOP TIME has expired after the completion of execution of the program.

	Key operation	Display	
		INJT 200	**The last display remains as is, which in this case is the set value of the injection port temperature.**
2.	SHIFT.D	INJT 200	**S.DOWN lamp is lit up.**
3.	3 / SLP-T	SLPT. 27	**S.DOWN lamp goes out. Current count of** SLEEP **TIME is displayed in minutes, with the display advancing in increments of one minute.**

Temperature control starts when the preset SLEEP TIME has expired, but the display remains on without clearing even after the temperature control has started.

If this operation is performed during the WAIT time which spans from the SHIFT DOWN STOP operation to the completion of temperature control, then the current count of the STOP TIME at that moment is displayed. (Be careful, however, in that the display appears as "SLPT. ".) The temperature control stops when the preset STOP TIME has expired, then display is reset to "0" and counting of SLEEP TIME is started.

4. Operations Performed During Analysis

Contents

4.1 Outline

After all the settings necessary for analysis have been completed, including column oven temperature, heating program, setting for the detector, etc., actual analysis is started. Various operations are possible during analysis.

4.2 Starting analysis

(1) START **(1)**

By pressing the [START] key, temperature control is started.

	ope Key on	Display	
1.	[START]	* *	*Starting* **temperature control.**

By pressing the START key, the column oven (and also the AUX.1, if it is connected) is controllably heated to the temperature set as the INIT TEMP. Also, the injection port and the detector (and the TCD and/or AUX.2 as well, if such options are connected) are heated to and controlled at their preset temperatures.

The READY lamp is lit up when the column oven temperature has reached and settled within ±1°C of the set temperature. The temperature may fluctuate slightly before the column oven temperature has stabilized causing the READY lamp to be turned off once or twice, but this is stabilized soon afterwards.

(2) START (2)

By pressing the START key once again, the heating program is executed and the analysis is started.

	Key operation	Display	
1.	START	RT. 0.00	Retention time is displayed in "min".

○ Case of heating the column

If this operation is performed before the column has reached the preset INIT TEMP, the heating program and/or the time program are started in the absence of the normal temperature control.

If the START key is pressed, execution of the heating program is started even when the detector temperature has not reached the setpoint yet, To avoid such trouble, be sure to check the detector temperature to see if it has reached the setpoint before starting operation.

Progress of the execution of the heating program can be monitored with the lamps provided on the control panel; first, when the step is entered, where the column oven temperature is held at the set INIT TEMP, the READY lamp goes out and the INITIAL lamp is lit up instead. The RAISE lamp is lit when the heating step is entered, and the FINAL lamp is lit when that rise is completed, where it is to be held at the FINAL TEMP in the heating process (FINAL TIME). When entering the next heating cycle, the RAISE lamp is lit again, and the FINAL lamp is lit when arriving at the FINAL TEMP step.

These processes are repeated as many times as have been programmed. In case of a heating process, where the FINAL TEMP for the step is set at a lower temperature than the START TEMP for the next step, however, the RAISE lamp lights momentarily but it is

replaced with the lighting of the FINAL lamp soon thereafter, and temperature starts falling at that moment. (As described already in the section on the Heating Program, the FINAL TIME step sets in at that moment. Therefore, the FINAL TIME for the step should be provided sufficiently long enough to allow for the stabilization at that FINAL TEMP for the step.)

When the heating program has come to the end (if the PROG RATE for the next step has been set at 0.0°C/min, it is judged to be the end of the heating program.), the COOL lamp lights and the column oven temperature is lowered to the INIT TEMP set for the column oven. At the *same* time, counting of retention time terminates, and the READY lamp lights when the set INIT TEMP is reached.

Even when the FINAL TEMP for the last step is lower than the INIT TEMP set for the column oven, the COOL lamp lights upon completion of execution of the program. In this case, however, the column temperature continues rising even after the COOL lamp lights, to return to its INIT TEMP.

(3) Case of heating the AUX.1

Heating is carried out in the same manner as for the column oven, but no facility for monitoring status with indication lamps is provided.

4.3 Operations during measurement

(1) Monitoring of temperature

Current temperature at each of the temperature control points can be monitored during measurement.

	Key operation	Display	
1.	MONIT	RT. 5.36	The last display remains as is, which in this case is the retention time.
2.	COL / AUX.1	CITP. 122	Temperature of the column oven at that time is displayed prefixed by "CITP", but that does not show the INIT TEMP set for the column oven. It can be differentiated from the INIT TEMP setting with the flashing "period" sign appearing between CITP and the temperature value.

	Key operation	Display	
		CITP. 122	The last display remains as is, which in this case is the column oven temperature.
2.	INJ / AUX. 2	INJT. 199	Current temperature of injection port.

	Key operation	Display	
1.	MONIT	INJT. 199	The last display remains as is, which in this case is the injection port temperature.
2.	DET–T / TCD–T	DETT. 50	Current temperature of TCD.

	Key operation	Display	
1.	MONIT	DETT. 50	The last display remains as is, which in this case is detector temperature.
2.	SHIFT. D	DETT. 50	S.DOWN lamp is lit up.
3.	COL / AUX. 1	AITP. 100	Current temperature of AUX.1. S.DOWN lamp goes out.

	Key operation	Display	
1.	MONIT	AITP. 100	The last display remains as is, which in this case is the temperature of AUX.1.
2.	SHIFT. D	AITP. 100	S.DOWN lamp is lit up.
3.	INJ / AUX 2		S.DOWN lamp goes out. Current temperature of AUX.1 is displayed.

	Key operation	Display	
1.	MONI	AUXT. 150	The last display remains as is, which in this case is the temperature of AUX.2.
2.	SHIFT. D	AUXT. 150	S.DOWN lamp goes out.
3.	DET–T / TCD–T	TCDT. 200	S.DOWN lamp goes out. Current temperature of TCD is displayed.

By effecting these key operations, temperature at the selected measurement point, which undergoes constant change, is kept on display until the next key operation is made.

Note To differentiate the displays shown above from the setpoints for those different points of measurement, a flashing "period" appears between the displayed code word and the measured temperature value, when the current temperature at that point is being displayed.

The [SCAN] key is provided to display the temperatures measured at the *six* different points in sequence at intervals of about two seconds.

	Key operation	Display	
1.	[SHIFT]	CPR1 0.0	The last display remains as is, which in this case is the PROG RATE 1 set for the column oven. S.DOWN lamp is lit up.
2.	[SCAN]	CITP. 100	Indicates current column oven temperature. S.DOWN lamp goes out.
		AITP. 100	Current AUX.1 temperature
		INJT. 200	Current injector temperature
		DETT. 200	Current detector temperature
		AUXT. 150	Current AUX.2 temperature
		TCDT. 40	Current TCD temperature
		CITP. 100.1	Current column oven temperature

5

Note Current temperatures of those zones listed above appear on the display in the sequence shown above, though you cannot know which one comes at the moment you pressd the [SCAN] key. Display of these current temperatures at the different parts is kept on in succession until the next key-in operation is made. To terminate the display, either press the [CE] key, or effect other key-in operation.

These temperatures can be monitored irrespective of whether an **AUX.** or TCD is connected or not. If no such additional part is connected, display will appear as AITP, **–**105, **AUXT, –***5,* or as TCDT, **–***5,* for example.

(2) HOLD (HOLD.T)

(a) Holding temperature

This refers to the operation performed during the execution of **a** program, to suspend the heating for **a** while **during** which period, counting of retention time is continued.

	opKey.on	Display	
1.	COL / AUX. 1	CITP. 100	The last display remains as is, which in this case is the column oven temperature.
2.	SHIFT. D	CITP. 100	S.DOWN lamp is lit up.
3.	HOLD. T	CITP. 100	S.DOWN lamp goes out.
4.	1	1	
5.	□	10	
6.	ENT	HLDC 10.0	In this case, temperature is held at the temperature measured when the ENT key is pressed, for 10 min.

	opKey.on	Display	
1.	COL / AUX. 1	RT. 0.00	The last display remains as is, which in this case is the retention time.
2.	SHIFT. D	RT. 0.00	S.DOWN lamp is lit up.
3.	HOLD / HOLD. T	RT. 0.00	S.DOWN lamp goes out.
4.	ENT	HLDC 2.00	The last set value is displayed, and is set again at that value.

(b) Holding retention time

It is possible to temporarily hold the retention time while measurement is going on.
By pressing the [HOLD] key, counting of retention time is suspended temporarily.
By pressing the [HOLD] key again, counting is resumed.

	Key operation	Display	
1.	[HOLD / HOLD. T]	HOLD	**Counting of retention time stops.**

	Key operation	Display	
1.	* [HOLD / HOLD. T]	RT. 3.67	**Counting of retention time is resumed with that value.**

* **You may press the [START] key instead.**

If these key operations are performed while the column oven temperature is held at either its INIT TEMP or at its FINAL TEMP, the column oven temperature is also held at the constant level, along with the retention time, until the holding function is released.
When HOLD is released, execution of the program is resumed returning to the point where the HOLD was effected.

If the HOLD function is effected while the column temperature is rising, the column oven temperature will rise up to the FINAL TEMP and then be maintained at that level.
When HOLD is released, the temperature is returned to the temperature at the final stage of the heating program, and execution of the program is resumed. In other words, after HOLD is released, the column oven temperature is held for the duration of the FINAL TIME set for the said step in the heating program, and then moves on to the next heating step.

Even if in the HOLD state before execution of the program, a pressing of the [START] key enables you to leave the HOLD state without any exception.

If you are not certain whether the GC is in HOLD state or not, due to a change occurring in the display as a result of other key-in operation effected after entering the HOLD state, make judgement by monitoring the retention time by making the operation described in the next section; if execution of the program is going on, the retention time value displayed will increment.

(c) Holding the temperature of AUX. 1

As with the case of the column oven, it is also possible to temporarily suspend execution of program for the AUX. 1, so that the AUX. 1 temperature will be kept at that level for the duration of the set time period and execution of the program will be resumed after the expiration of the hold time period.

	Key operation	Display	
1.	SHIFT. D	AITP. 219	The last display remains as is, which in this case is the temperature of AUX.1 S. DOWN lamp is lit up.
2.	COL / AUX. 1	AITP. 219	S.DOWN lamp goes out.
3.	SHIFT. D	AITP. 220	S.DOWN lamp is lit up.
4.	HOLD / HOLD. T	AITP. 220	S.DOWN lamp goes out.
5.	1	1	
6.	0	10	
7.	ENT	HLDA 10.0	For the AUX.1, temperature is held for 10 min at the temperature measured at the moment when this step 7 is effected, and then execution of the program is resumed upon expiration of the HOLD time.

(3) Monitoring retention time

Count of the retention time is displayed simultaneous with the pressing of the START key. If you wish to display the current retention time count again after the display has disappeared due to the monitoring of temperature or otherwise, perform the following key-in operations:

	Key operation	Display	
1.	MONIT	CITP. 100	The last display remains as is, which in this case is the column oven temperature.
2.	SHIFT. D	CITP. 100	S.DOWN lamp is lit up.
3.	STW / RET. T	RT. 5.37	Count of retention time is displayed. S.DOWN lamp goes out.

If these key-in operations are performed with execution of the program not being started yet, the display appears as "RT. 0.00". If the retention time is in the HOLD state, the display remains still indicating the retention time at the moment when the HOLD was effected.

(4) Monitoring the time program

The number of the program step to be executed next can be monitored using this function. By effecting the following key-in operations, the step to be executed next is displayed identified by the step number assigned in the sequence of execution of the preset time program. (The number which appears when monitoring the time program can also be displayed via the printer or on the CRT display of the Chromatopac.)

	op Key on	Display	
1.	MONIT	CITP. 100	**The last display remains as is, which in this case is the column oven temperature.**
2.	SHIFT D	CITP. 100	**S.DOWN lamp is lit up.**
3.	7 / PROG	PROG. 5	**S.DOWN lamp goes out. Number of the program step to be executed next is displayed. In this case, steps up to No. 4 have already been executed, and step No. 5 is to be executed next.**

While being monitored, the display changes as the step with the number then appearing on the display is executed.

If these key operations are performed where no time program has been loaded, "PROG 0" appears on the display, indicating that there is no time program then at work.

Or, if these operations are performed while no temperature control is going on, the display appears as "PROG. XX".

(5) Direct operation using the EVENT key

Shown below is an example of outputting a desired EVENT using relevant keys at a desired time, without relying on any time program.

- EVENT 0 ····· Reversal
- EVENT -0 ····· All the EVENTs are turned OFF.
- EVENT 1 ~ 8 ····· Relevant triac output is turned ON.
- EVENT 91, 92 ····· Relevant relay output (mounted in the GC-14A) is turned ON.

	opKey.on	Display	
1.	SHIFT.D	CITP. 150	**The last display remains as is, which in this case is the column temperature. S.DOWN lamp is lit up.**
2.	8 EVENT	CITP. 150	**S.DOWN lamp goes out.**
3.	1	1	
4.	2	12	
5.	ENT	EVNT 12	**When step ☐ above is performed, output for EVENT 12 is turned ON.**

If an EVENT that has already been turned ON undergoes further ON operation, the same contents appears on the display, with no change occurring in the state of operation.

The following key operations are to be performed when turning OFF an EVENT.

	opKey.on	Display	
1.	SHIFT.D	CITP. 100	**The last display remains as is, which in this case is the column oven temperature. S.DOWN lamp is lit up.**
2.	8 EVENT	CITP. 100	**S.DOWN lamp goes out.**
4.	2	- 2	
5.	ENT	EVNT - 2	**Output for EVENT 2 is turned OFF.**

Further OFF operation effected on an EVENT that has already been put in the OFF state causes no changes to take place in the state although it is displayed as such.

Selection of EVENT 0 causes all triac or relay outputs then in the ON state to be reversed to the OFF state, and those which are in the OFF state to be reversed to the ON state. EVENT-0 causes all the triac and relay outputs then in the ON state to be turned OFF.

Portion of an EVENT number in decimals is disregarded and only the integer portion thereof is accepted as the EVENT Number. If you try to set any value that cannot be accepted for EVENT number setting (any value smaller than −255, or that is greater than 255), an "ERROR 4" message appears on the display, with no changes occurring in the state.

Care should be paid to the case of acceptance of an unwanted EVENT number, which may result from the following key operations:

	Key operation	Display	
1.	SHIFT.D	CITP 40.0	The last display remains as is, which in this case is the column oven temperature. S.DOWN lamp is lit up.
2.	8 / EVENT	CITP 40.0	S.DOWN lamp goes out.
3.	ENT	EVNT 40.0	If the ENT key is pressed without setting a desired EVENT No., the value then appearing in the least four digits on the display is taken and set as the desired EVENT No. In this case, it is accepted as EVENT 40.

5

Unlike other kinds of operations, function for monitoring of current ON or OFF states is not provided with operations concerning EVENTs.

(6) Others

In addition to those mentioned above, the current sample number can be monitored in case of using an auto-sampler. (Refer to the section concerning the Auto-sampler.)

5. Time Program

Contents

5.1 Outline

A time program consists of a set of programmed parameters to be changed at such and such times elapsed after the program start. A variety of time programs can be edited for each of the files used for the unit.

5.2 Items available for programming

(a) RANGE, POL, CURR

Sensitivity, polarity of a detector, and elecmcal current allowed to flow therethrough can be changed as desired.

Ex. "DIRG 2" sets sensitivity to 10^2

(b) EVENT 91, EVENT 92

Those two relays are available for ON/OFF control of external devices as desired.

"EVNT-91" : EVENT 91 OFF

(c) Other EVENTs available as options

Eight different triac outputs are available by adding optional PC boards (PRG boards).

"EVNT 1" : EVENT 1ON

(d) RETN.

This allows repetitive execution of a program as desired.

Further, after the program has been executed for the desired number of times, the file number is changed for further execution.

For example, if set to "RETN 102", first execution is repeated 10 times with the current file number and then select file number 2 for execution of the program therein.

(e) INJ

Allows changes in temperature of the injection port.

(f) AUX2

Allows changes in temperature of the temperature controlled zone AUX.2.

(g) STRK

Allows changes in syringe stroke of the AOC.

"STRK 60" 60% of the full stroke

(h) REPT.

Changes the number of times of repeat sample injection to be performed by the AOC.

"REPT 5" Repetition for 5 times

(i) DET

This alone has no meaning.

Used for designating a particular one of the detectors prior to setting for RANGE, POL and/or CURR.

Ex. [DET] [□] [ENT] [RANGE] [2] [ENT]

(j) STOP

Execution of the program can be suspended by using this key.

5.3 Editing the time program (Time can be set in minutes down to the hundredths place.)

	Key operation	Display	
1	[SHIFT. D]	CITP 100	The last display remains as is, which in this case is the column oven temperature. S.DOWN lamp is lit up.
2	[7 PROG]	TIME ?	DIALOG lamp is lit up, with inquiry made first asking for the time when the step is to be executed. S.DOWN lamp goes out.
3	[1]	1	
4	[0]	10	Has been set at 10 min after ***START***.
5	[ENT]	FUNC VL ?	Inquires as to which function should be manipulated at that time.
6	[SHIFT. D]		
7	[8 EVENT]		
8	[1]	1	

9	[ENT]	TIME ?	**EVENT 1** is to be set to the ON state. Prompts for the time when the next step is to be executed.
10	[1]	1	
11	[5]	15	
12	[ENT]	FUNC VL ?	**Has** been set at 15 min after START.
13	[SHIFT.D]		
14	[8 EVENT]		
15	[-]	-	
16	[1]	-1	
17	[ENT]	TIME ?	Set EVENT 1 to the **OFF** state at 15 minutes after START. Prompts for the time when the next step is to be executed.
18	[1]	1	
19	[5]	15	Two or more actions may be performed in the same step, ie., at the same time.
20	[ENT]	FUNC VL ?	
21	[RANGE]		
22	[1]	1	
23	[ENT]	TIME ?	Range is to be set to RANGE **1.**
24	[5]		The actual execution may come slightly earlier or later.
25	[ENT]	FUNC VL ?	
26	[RANGE]		
27	[2]		**RANGE 2** is to be set.
28	[ENT]	TIME 10.0	Though the time of the next step is the same as one that has been previously set, you may proceed on editing the program under this condition.
29	[3]		
30	[0]		
31	[ENT]	FUNC VL ?	
32	[SHIFT.D]		
33	[5 RETN]		
34	[5]	5	
35	[0]	50	
36	[ENT]	TIME ?	This time program is repeated five times (5 STARTs executing the heating program as well five times, of course), then returns to file 0.
37	[SHIFT.D]		S.DOWN lamp is lit up.
38	[ESCP]		Both S.DOWN lamp and **DIALOG** lamp go out, and the **14B has** left the **DIALOG** state.

5

5.4 How to look up the time program loaded

First, call the file into which the time program in question is loaded. (FILE/FUNC 3 ENT)

Be careful of the fact that if the file is changed while temperature control is going on, all the temperatures under control shift from the temperatures set in the file then in use to those set in the new file to which the transfer has been made.

	Key operation	Display	
1	SHIFT.D	INJT 200	The last display remains as is, which in this case is the injection port temperature. SHIFT.D lamp is lit up.
2	7 PROG	TIME 5.00	Time of the first program step is displayed.
3	ENT	DIRG 2	Parameter related to that time.
4	ENT	TIME 10.00	Proceed in the same manner thereafter.
5	ENT	EVNT1	
6	ENT	TIME 15.00	
7	ENT	EVNT −1	
8	ENT	TIME 15.00	
9	ENT	DIRG 1	
10	ENT	TIME 30.00	
11	ENT	REPT 50	
12	ENT	TIME ?	Indicates that the end of the time program has been reached. If any addition is required, just go ahead and input further. Any desired value can be entered for the time, the program loaded is sorted automatically by the GC.
13	SHIFT.D		S.DOWN lamp is lit up.
14	ESCP		S.DOWN lamp and DIALOG lamp are extinguished.

5.5 Changes to the time program

If it is required to change part of the time program loaded, input while the display reads either "TIME ?" or "TIME XX" (XX represents any given values), if only for the addition of new parameters.

To delete part of the time program, proceed as follows.

	Key operation	Display	
1	SHIFT.D	CITP 100	Previous display remains as is. SHIFT.D lamp lights.
2	7 PROG	TIME 5.00	DIALOG lamp lights.
3	ENT	DIRG 2	Depress the ENT key repeatedly to step through the program.
	⋮		
4	ENT	TIME 15.00	When the time of the step to be deleted is displayed...
5	SHIFT.D		Press SHIFT.D followed by ...
6	4 DEL	TIME 30.00	DEL. The step will then be deleted and the time of the next step (if any) will be displayed.
7	SHIFT.D		If no further deletions are required, press SHIFT.D (the SHIFT.D lamp lights) ...
8	ESCP		and ESCP to terminate the dialog. (The DIALOG lamp and SHIFT.D lamp are extinguished.)

5

5.6 Time program memory requirements

If when programming the time for a step when the prompt "TIME ?" appears, and "TIME 0.00" is displayed upon input of some value, then there is insufficient memory available. Secure additional memory by deleting unwanted files.

5.7 Time program START and STOP

As in the case of the heating program, a time program is started up by pressing the **START** button. (Refer to the section on Heating Program.)

If execution of the time program is to be stopped midway, press the STOP key. You cannot continue execution of the heating program with only the execution of the time program being suspended.

6. Files

Contents

5

6.1 Outline

It is possible to generate up to 10 files for this unit. And it is possible to save time programs, GC parameters, heating programs and AOC parameters in each of these files. It is to be noted, however, that, as for the AOC parameters, except for the four items (IJ1T, IJ2T, STRK and REPT) they are used commonly for all the files.

Each of the fdes is made to have a variable length, that is, area of a file can only be secured in the memory by setting the file. Accordingly, a number of files can be generated if each of them is small in size (up to 10 fdes), but not so many files can be generated if each of them is large in size.

"File 0" is open at the time of turning on the power switch (e.g. when no setting has been made for file yet), therefore, all the programs loaded without selecting a file are saved in "File 0".

6.2 Calling a file

Prior to the editing of time program, *GC* parameters and heating program, the following key-in operations are to be made to set a file into which they are to be loaded. (If no setting is made, File 0 is set.)

Ex. When calling File **4:**

	Key operation	Display	
1.	FILE / FUNC		
2.	4	4	
3.	ENT	FILE 4	

6.3 How to look up the file No. then in use

	Key operation	Display	
1.	MONIT	CITP 100	The last display remains as is, which in this case is the colum oven temperature.
2.	FILE / FUNC	FILE 4	Number of the file then in use is displayed, which in this case is FILE **4.**

6.4 Copying the contents of a file

The contents of file then in use can be copied into another file as they are. If copying is to be made into a file that has already been opened, it is recommended that deletion of the file should be made first to clear it (referring to **Para.** 6-5) before copying to it.

	Key operation	Display	
1.	SHIFT.D	INJT 200	The last display remains as is. S.DOWN lamp is lit up.
2.	6 / COPY		S.DOWN lamp goes out.
3.	□	3	
4.	ENT	COPY 3	Contents of the current file used have been copied into FILE **3.**

6.5 Deletion of the contents of a file

It is possible to delete the contents of file(s) other than the one then in use.

Procedure for deletion

Ex. When contents in FILE **3** are to be deleted:

	op Key.on	Display	
1.	SHIFT D	TCDT 30	The last display remains as is, which in this case is the **TCD** temperature. S.DOWN lamp is lit up.
2.	4 DEL	TCDT 30	S.DOWN lamp goes out.
3.	3	3	
4.	ENT	DELT 3	**Now,** the contents of FILE **3** are deleted.

Enough space for a new file may not be secured if a large time program is extent or a number of large files have been prepared.

For example, if a **"LIMIT"** message appears on the display when attempting to copy a file or when setting for a new file is intended, it indicates that there is no more space for accommodating expansion of files. In such a case, delete unnecessary files to secure space for the new file.

Even if there is no more memory space to accommodate a new file, there may be some extra memory space that can accommodate some more time programs or heating programs.

Note Only one file can be designated at a time. Accordingly, in case of a change in file number, the GC will be controlled according to the contents of the newly designated file from then on. When the GC is under the temperature control, selection of a different file made inadvertently may cause change in the setting temperature, if the contents of the file newly selected are incorrect. When you are newly setting several files or wish to look up the contents of these files, these new settings and look-up of their contents should be made before pressing the **START** key after switching on the GC.

- In the state right after turning on the power switch, the file number selected is always "FILE 0"

7. Other Operations

Contents

	Key on	Display	
1.	SHIFT D	INJT 200	**The last display remains as is, which in this case is the injection port temperature. S.DOWN lamp is lit up.**
2.	FILE / FUNC	INJT 200	**S.DOWN lamp goes out.**
3.	1	1	
4.	□	13	**Self-diagnosis is provided as AUX function 13.**
5.	ENT	CITP. ××	**Display appears in the format shown on the left if no abnormality is found. If there is any sign of abnormality noted, the address of the faulty part is displayed.**

7.2 Listing

(1) Listing

It is possible to list on an external unit (Chromatopac, etc.) the AOC parameters, heating program, time program and GC parameters of the file designated.

(2) Operation for listing

The number assigned for the device selected for output of the list is set according to the instructions in Para. **7.3** on Link Device. ***Also*** the file number selected for listing is set accord-

	Key operation	Display	
1.	SHIFT.D	CITP 100	The last display remains as is.
	2	2	
4.	ENT	LIST. P	DIALOG lamp is lit up, indicating the DIALOG mode.
5.	1	1	Key in "1" when listing is desired.
6.	ENT	LIST. P	While listing is going on, the DIALOG lamp remains on. Upon completion of listing, the DIALOG lamp goes out to show escape from the DIALOG mode.

5

	opKey.on	Display	
1.	SHIFT.D	CITP 100	The last display remains as is. S.DOWN lamp is lit up.
2.	FILE FUNC	CITP 100	S.DOWN lamp goes out.
3.	3	3	Listing of AOC parameters is available as AUX function 3.
4.	ENT	LIST. A	DIALOG lamp is lit up.
5.	1	1	Key in "1" when listing of parameters is desired.
6.	ENT	LIST. A	Listing of parameters starts. Upon completion of listing, DIALOG lamp goes out.

2.3 Listing GC parameters

	opKey.on	Display	
1.	SHIFT.D	CITP 100	The last display remains as is. S.DOWN lamp is lit up.
2.	FILE FUNC	CITP 100	S.DOWN lamp goes out.
3.	4	4	Listing of GC parameters is available as AUX function 4.
4.	ENT	LIST GI	DIALOG lamp is lit up.
5.	1	1	Key in "1" when listing of parameters is desired.
6.	ENT	LIST GI	Listing of parameters starts. Upon completion of listing, DIALOG lamp goes out.

2.4 Listing of heating program

	Key on	Display	
1.	SHIFT.D	CITP 100	The last display remains as is. S.DOWN lamp is lit up.
2.	FILE / FUNC	CITP 100	S.DOWN lamp goes out.
3.	□	1	
4.	5	15	Listing of heating program is available as AUX function 15.
5.	ENT	LIST GP	DIALOG lamp is lit up.
6.	□	1	Key in "1" when listing of parameters is desired.
7.	ENT	LIST GP	Listing of parameters starts. Upon completion of listing, DIALOG lamp goes out.

5

(3) When no listing of parameters is required:

When no listing of parameters is required, key in "0" instead of "1". Then, no listing is performed and DIALOG lamp goes out, allowing escape from the DIALOG mode. You can also escape from the DIALOG mode by pressing the SHIFT.D and ESCP keys.

(4) Error display

If either of the following error messages appears on the display when you attempt to list, it indicates failure to perform some necessary setting. If either error message is given, the unit leaves the DIALOG mode.

"SET LDVC"

This error message appears when the listing device (printer, CRT, etc.) has not been designated. In such a case, set the appropriate link device code. (Refer to 7.3 Link Device.)

"NOT LINK"

This display appears when no link has been established between the GC and the external output device. In this case, make necessary steps needed for establishing a proper linkage with the output device (e.g., "OPEN TRS 1", etc., at the Chromatopac). See next.

7.3 Connection with an external data processing unit

By adding a proper current loop interface, the unit can be operated in combination with a Chromatopac. By making use of the BASIC capability of the Chromatopac, the GC functions can be enhanced substantially.

For details on the operating procedures for the Chromatopac, refer to chapter 10; "Other Curren Loops", or the instruction manual for the Chromatopac.

1. Setting of the **link** device code

(1) Link device

When the contents of time program, AOC parameters, GC parameters, etc. used for the unit are to be output to **an** external device, setting should be made beforehand for the code number of the selected external output device. External output device numbers available for setting are between 0 and 55, and the default value is 0.

(2) Setting procedure

	opKey.on	Display	
1.	SHIFT.D	CITP 100	The last display remains as is. S.DOWN lamp is lit up.
2.	FILE / FUNC	CITP 100	S.DOWN lamp goes out.
3.	8	8	Setting for link device is available as AUX function 8.
4.	"TI	LDVC 0	The link device then set is displayed. DIALOG lamp is lit up entering the DIALOG mode.
5.	1	1	Link device 11 is set.
6.	1	11	
7.	ENT	STRK 10	11 is set for link device and display advances to the next item in the parameter list.
8.	SHIFT.D	STRK 10	When setting is complete, operation must be made to escape from the DIALOG mode. S.DOWN lamp is lit up.
9.	ESCP	STRK 10	DIALOG lamp goes out. S.DOWN lamp goes out. Now, the GC has escaped from the DIALOG mode.

(3) In case of erroneous setting

In case of an erroneous setting, if you haven't pressed the [ENT] key yet, delete the display by pressing the [CE] key, and then enter the right number. If you have pressed the [ENT] key already, call up the LDVC display once again by pressing the [ENT] key by the required number of times, or escape temporarily from the DIALOG mode by effecting steps [8] and [ ], and start again from step [ ].

What you have to bear in mind in this regard is the need for escaping from DIALOG mode temporanly upon completion of necessary setting. Otherwise, you cannot make any other key-in operations.

(4) To look up the set value:

	Operation	Display	
1.	[SHIFT.D]	CITP 100	The last display remains as is. S.DOWN lamp is lit up.
2.	[FILE/FUNC]	CITP 100	S.DOWN lamp goes out.
3.	[8]	8	
4.	[ENT]	LDVC 11	The link device currently set is displayed. DIALOG lamp is lit up.
5.	[SHIFT.D]	LDVC 11	S.DOWN lamp is lit up. Take necessary steps for escaping from DIALOG mode.
6.	[ESCP]	LDVC 11	S.DOWN lamp goes out. DIALOG lamp goes out. Now, has escaped from DIALOG mode.

(5) Meaning of LDVC

LDVC stands for Link Device Code, used to specify the selected output device, e.g. either the CRT of the external device (Chromatopac) or a plotter, etc. Setting is done with two digits; the MSD is for setting the particular channel of the Chromatopac to which signals from the detector are connected. ("3" is set if connected with both channels 1 and 2.) The LSD is used for designating either CRT or the plotter of the Chromatopac. Designate 2 if listing is desired on the CRT, or set 1 if listing is desired via the plotter.

LDVC 22 means that signals from the detector are connected with channel 2 and listing is to be made on the CRT.

Appendix 1. Error Display

The following error displays appear in case of an erroneous operation by the user, or due to an abnormal occurrence in the unit, or other causes.

"OVER COL"

This error message appears on the display if column oven temperature exceeds the set maximum column oven temperature, and the buzzer keeps on sounding. At the same time, temperature control for the column oven stops.

If the setting of the maximum temperature is too low, while the column oven temperature is the right temperature as has been set, raise the maximum temperature setpoint. If column oven temperature is abnormally higher than the setpoint, which suggests a failure, turn off the power switch and investigate.

To escape from this abnormal condition, press the [CE] key after the column oven temperature has fallen sufficiently lower than the set maximum value. Then, the buzzer stops sounding and temperature control operation is resumed.

"OVER INJ"

If temperature of the injection port exceeds 450°C, or the temperature of the detector exceeds the set maximum value, this error message appears and the buzzer keeps on sounding. Since such a high temperature cannot be set for the injection port, this is a symptom of failure.

Care should be given also to the fact that the same error message appears if the temperature of the detector should exceed the set maximum temperature. If this error message is given, all temperature control is suspended.

"OVER AUX"

This error message appears if the temperature of AUX.1 exceeds the set maximum temperature, and the buzzer keeps on sounding.

"ERROR CM"

This error message appears if there was a change to the contents of the files while the power supply was off due to battery discharge or lightning, etc. The file contents are now presumed to be corrupt.

"ERROR CR"

This error message appears in case of trouble in information exchanged between the gas chromatograph and the Chromatopac. In such a case, switch off both the gas chromatograph and the Chromatopac temporarily and establish the link between them correctly.

"ERROR 1"

Error in data transmitted or received.

"ERROR 2"

This message tells that either no gas is flowing in flow rate measurement, or the flow rate is extremely low disabling measurement. (Option)

"ERROR 3"

General error display. This error message appears if there is no such function as has been selected.

"ERROR 4"

General error display. This error message appears if the value input is unacceptable.

" NON"

This indicates that the particular one of the detectors with the number selected is not connected, in monitoring detectors.

"NON "

This indicates that, in setting for a detector, there exists no such detector as selected when entering a number other than 1 to 4.

"CITP.XX", "INJT.XX", "PROG.XX"

These pseudo-error messages appear, when monitoring current temperature or current program number, etc., if temperature control operation has not yet been started. As the temperature control operation starts by pressing the START key, the right value is displayed in place of "XX".

"SET LDVC"

This appears, in listing of a program, etc., if no setting has been made for the link device code.

"NOT LINK"

This appears, in transferring/receiving to or from the output/input units (data processing unit), if no linkage has been established with such an external unit.

Other displays

"**"

Indicates that temperature control operation is started, or is now going on.

"LINK OK"

This appears when linkage is established with the external unit, with the buzzer sounding for about two seconds.

"LIMIT"

This appears if there is no more space available for expanding a file, or if it is attempted to create a heating program involving 6 or more steps.

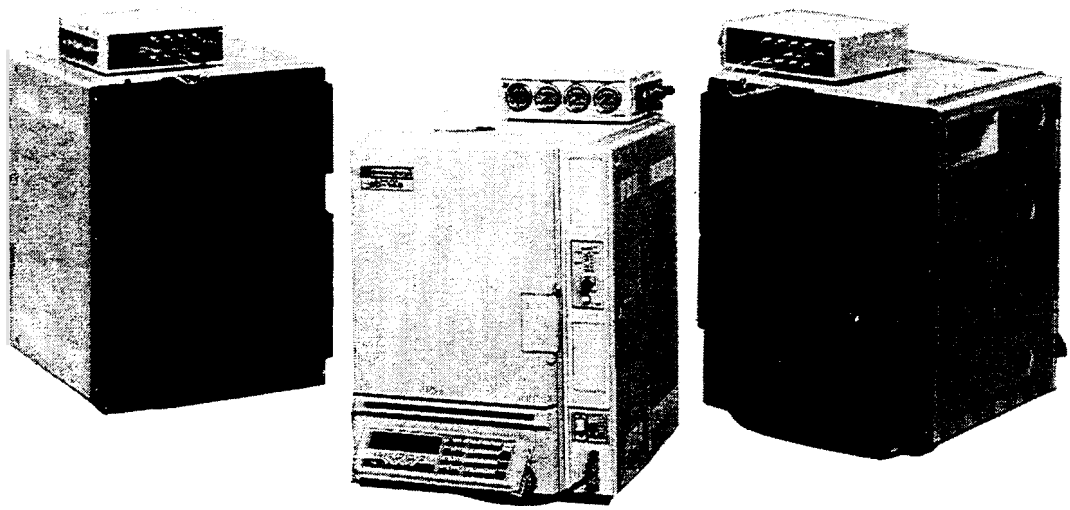
"INITIAL"

This message appears in case of a catastrophic change occurred in the contents of memory, with all the setting conditions being initialized. In such a case, set the conditions again from the beginning.

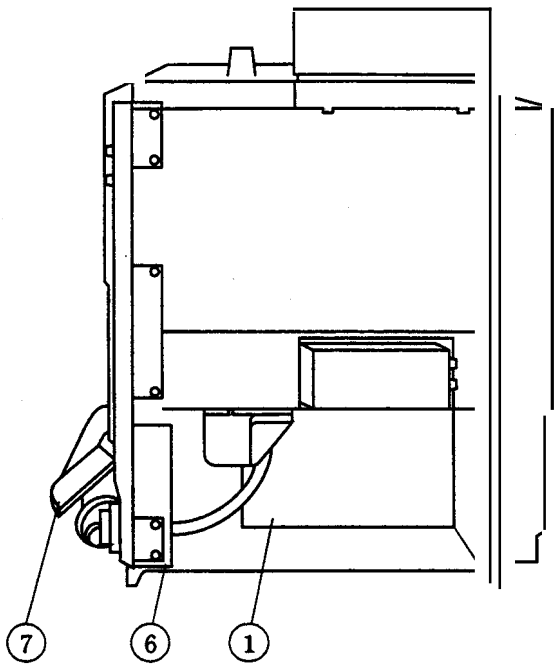
"FAULT"

This appears if part of the system program (ROM) has been destroyed. In such a case, contact our service staff, as it is impractical to use the unit with this condition.

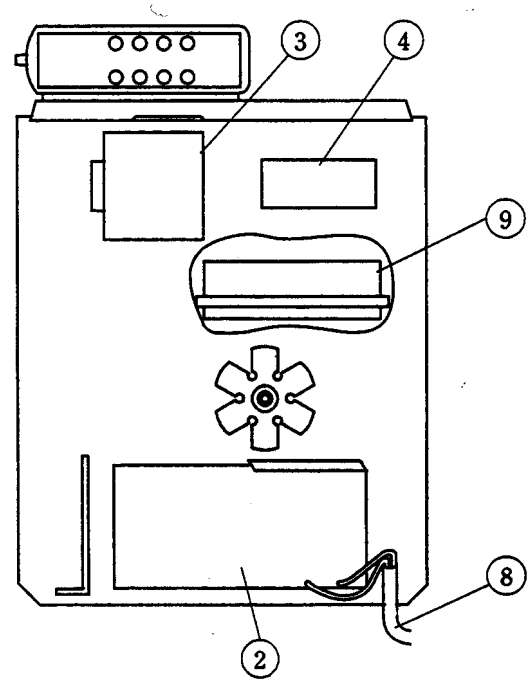
6. Construction and Maintenance



1. Construction



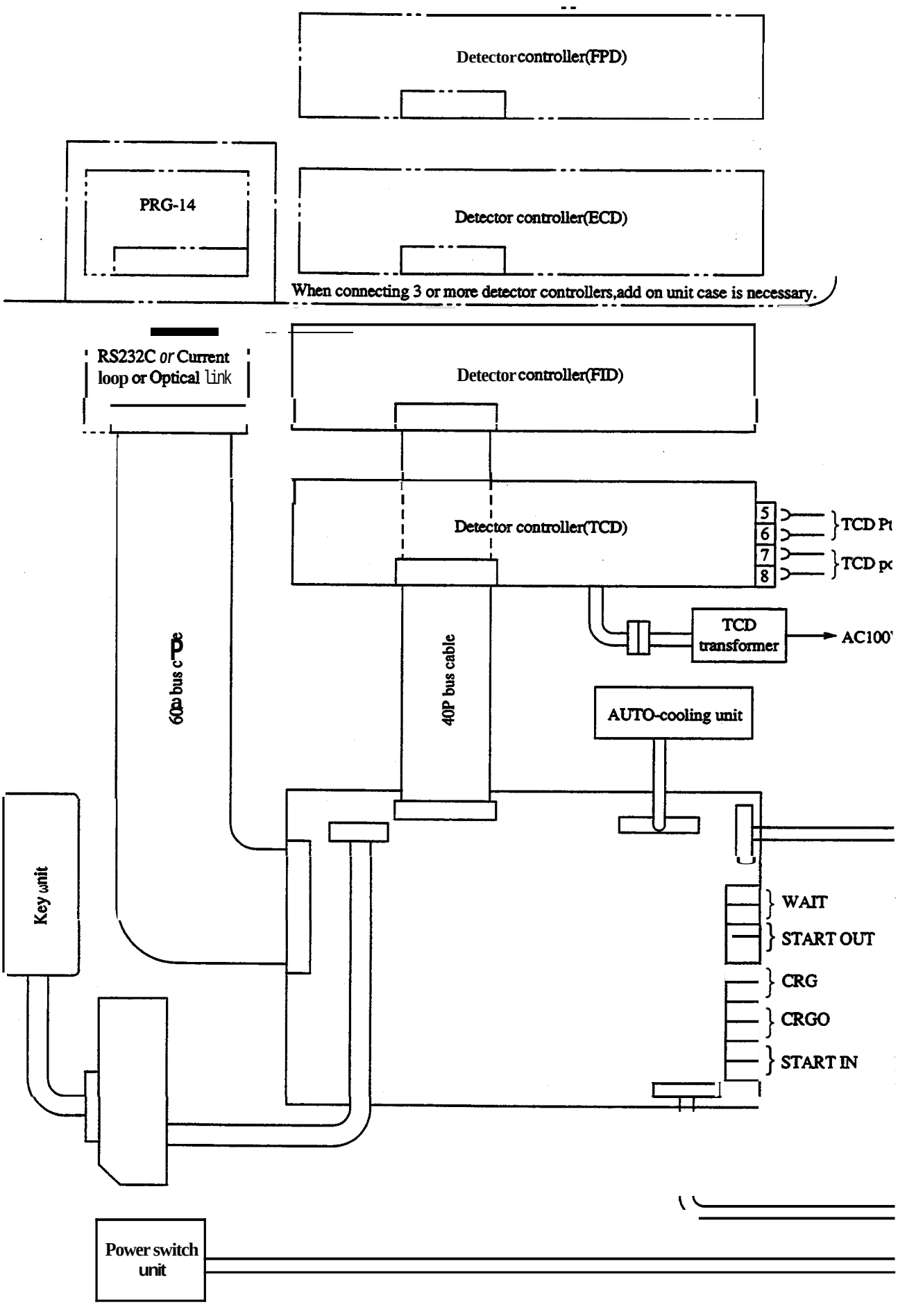
Side view



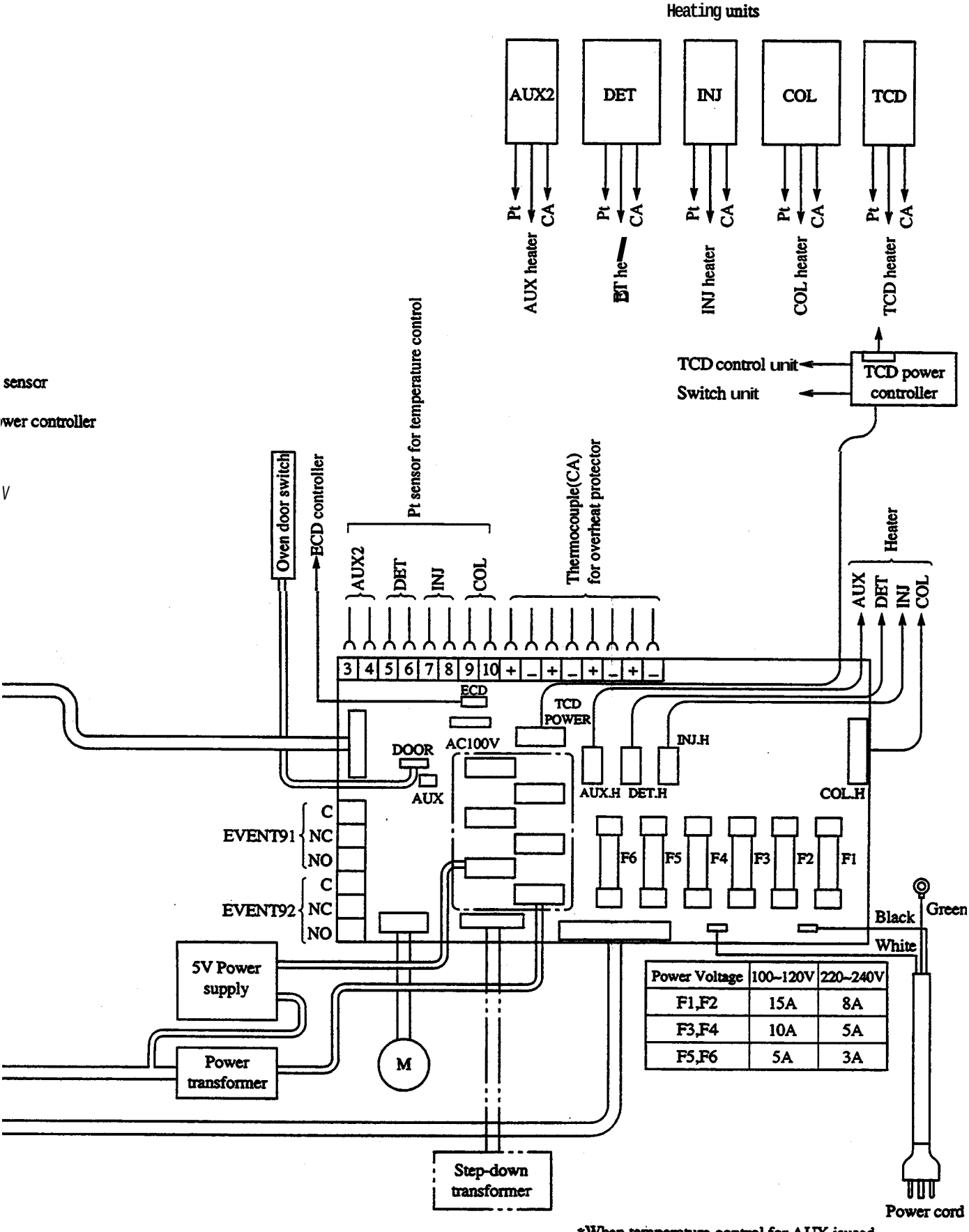
Rear view

6

① GC-Base unit	P/N 221-44388-91
② Terminalunit	P/N 221-44403-92 (115V power supply)
	221-44403-93 (220V, 230V, 240V, power supply)
③ 5V power supply	P/N 074-80642-15
④ TCD power controller	P/N 221-32896-96
⑥ PCB switch unit	P/N 221-44193-91
⑦ Key unit	P/N 221-31772-96
⑧ Power cord assembly	P/N 221-43318-93
⑨ Auto-cooling unit	P/N 221-32068-91


Detector controller(FPD)
PRG-14
Detector controller(ECD)
When connecting 3 or more detector controllers,add on unit case is necessary.
RS232C or Current loop or Optical link
Detector controller(FID)
Detector controller(TCD)
5
6
7
8
TCD Pt
TCD pc
TCD transformer
AC100
60P bus cable
40P bus cable
AUTO-cooling unit
Key unit
WAIT
START OUT
CRG
CRGO
START IN
Power switch unit

Block Dia

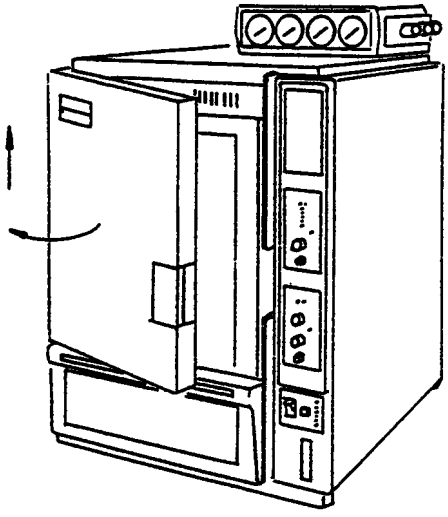


*When temperature control for AUX isused, set pin to AUX ,(P/N075-53216-02)

gram of Electrical systems

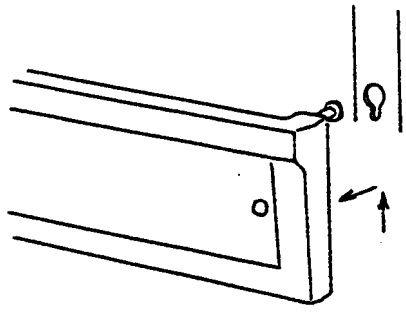
Removal and replacement of column oven door, rear panel and side panel

1. Removal of column oven door and subdoor



To dismantle the column oven door, open the door and raise the whole assembly to dismount the door.

Reverse the procedure when replacing the column oven door.



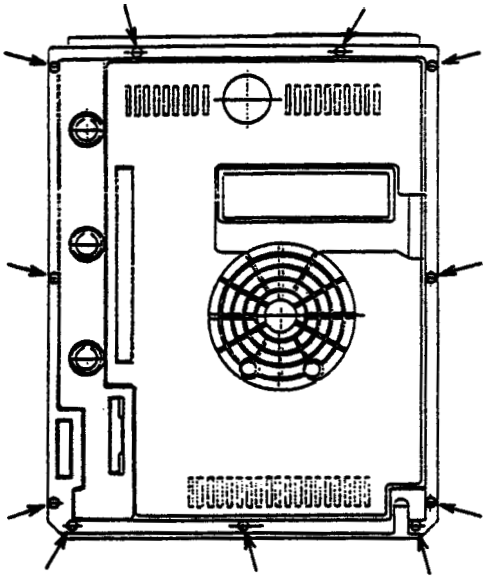
To detach the column oven subdoor, raise the subdoor slightly and pull it toward you.

Reverse the procedure to replace the sub-door.

Warning High Voltage

You may get an electric shock. Only the authorized service engineer is allowed to detach the rear panel and right side panel.

2. Removal of rear panel



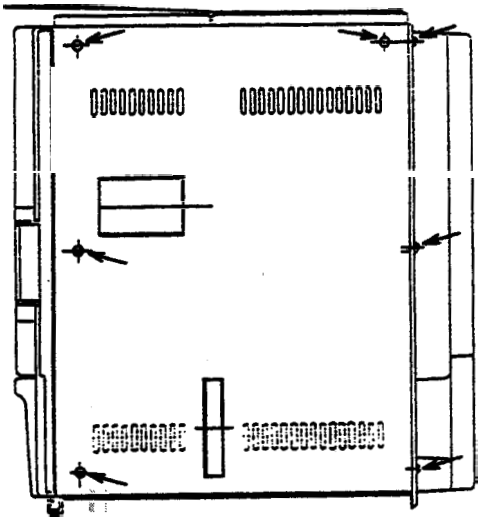
To detach the rear panel, first disconnect wiring for the detector control unit and for the optional boards.

By removing the 11 screws shown in the drawing, the rear panel can be detached easily.

Reverse the procedure to replace the rear panel.

To ensure safety, be sure to disconnect the power cord for GC-14B from the outlet without fail.

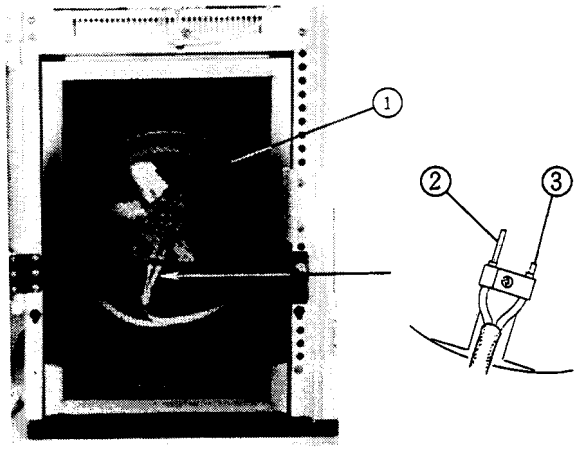
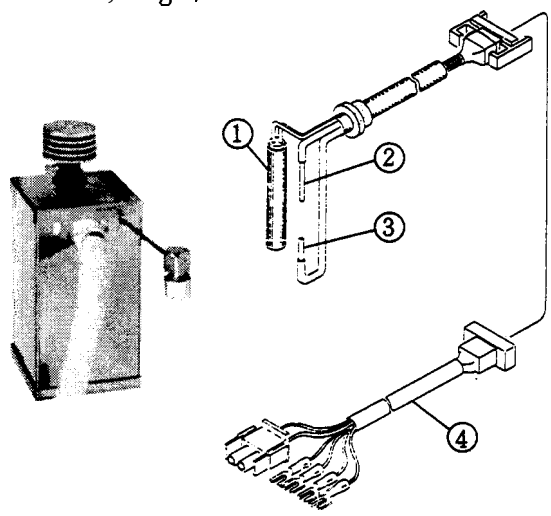
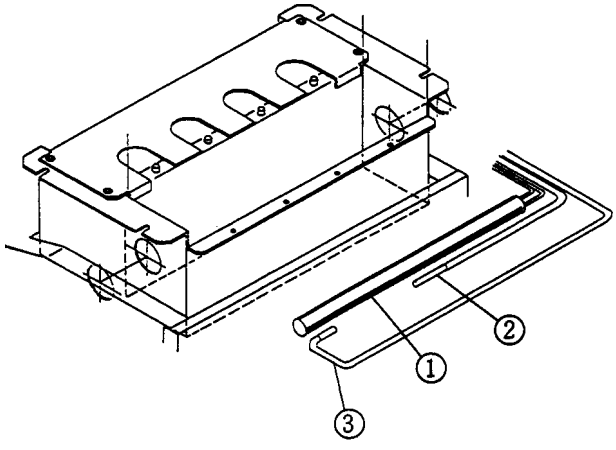
3. Removal of right side panel



To detach the right side panel, remove the four screws on the side and three screws on the rear face as shown in the *figure.* Then the panel can be removed easily. Be careful as catches protruded from the top panel.

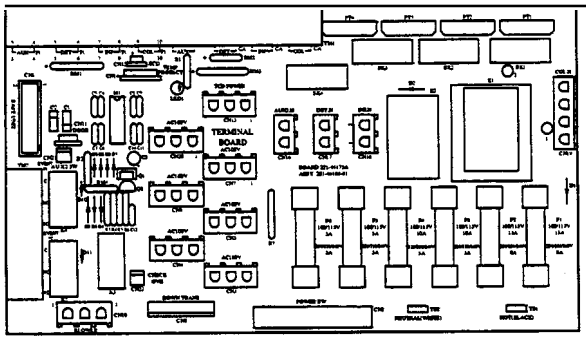
Reverse the procedure to replace the side panel.

Construction of basic components and spare parts required are as follows.

Construction	Spare parts
Column oven  	① Column oven heater assembly (1.3kW) Part No. for 115V power supply **221-28919-92** for 220V ~ 240V power supply 221-28919-93 Heater insulator **221-28919-05** Sleeve insulator **221-32897** ② Ptsensor 221-41059-92 ③ Thennocouple **221-32977-92**
INJ unit, single/dual  	① Single **INJ** heater (80W) for 115V power supply 221-32877-01 for 220V ~ 240V power supply 221-32877-03 ① **Dual** INJ heater (150W) for 115V power supply 221-29281-01 for 220V ~ 240V power supply 221-29281-03 ② Pt sensor 221-44685-91 ③ Thermocouple **221-32977-92** ④ Heater wiring cord 221-32964-91 (115V, 220V, 240V) -38 (230V) ④′ Heater wiring cord used for temperature Control with AUX2 oven 221-32964-93 (115V, 220V, 240V) -30 (230V)
DET oven  	① DET heater assembly (200W) for 115V power supply 221-32980-91 for 220V ~ 240V power supply 221-32980-93 ② Pt sensor 221-44685-92 ③ Thermocouple **221-32977-92**

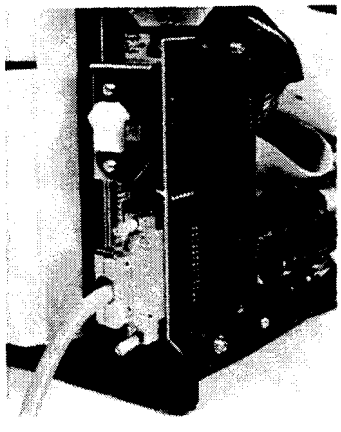
Electrical system and related parts (Refer to the Block Diagram of Electrical Systems)

Construction	Spare parts
Terminal unit and **peripheral parts**	See fuses information page 6-7



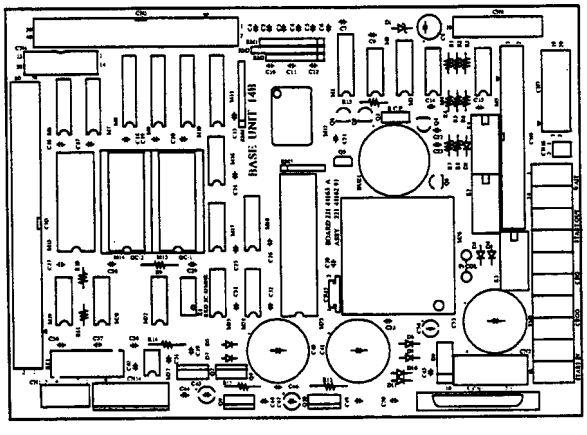
6

Switch unit



Door switch	221-29781-91
Power switch	064-14021
Lamp switch	064-54604-06

Base unit



Lithium battery	074-73307-01
Service life 5 years	

Fuses information

Terminal unit fuses

Fuse designation	Line Voltage	Fuse rating and type	Part Number
F1, F2	115V	AC250V 15A UL STD 198G ceramic body	072-01665-36
F1, F2	220V/230V/240V	AC250V 8A UL STD 198G ceramic body	072-01665-33
F3, F4	115V	AC250V 10A UL STD 198G ceramic body	072-01665-34
F3, F4	220V/230V/240V	AC250V 5A UL STD 198G glass body	072-01664-23
F5, F6	115V	AC250V 5A UL STD 198G glass body	072-01664-33
F5, F6	220V/230V/240V	AC250V 3A UL STD 198G	072-01664-30

3. Troubleshooting and Remedy

To ensure safety, be sure to disconnect the power plug of the **GC-14B** from the outlet before checking electrical part(s) inside the unit **main** body.

Troubleshooting procedures for failure occurring in the unit **main** body and countermeasures taken

Phenomenon	Cause	Check and countermeasure taken
1. Does not work at all.	a. AC100V is not supplied. b. Fuse burnt out. c. Faulty power switch.	a. Check with tester. b. Check with tester. c. Check with tester.
2. Heater switch cannot be turned on.	a. Faulty heater switch.	a. Check with tester.
3. Temperature of each part does not rise.	a. Break in heater. b. Break in heat-sensitive element. c. Overheat protector is activated due to overheat in thermo-couple. d. e. Faulty relay used in power supply unit. f. Faulty temperature control circuit.	a. Disconnect connector and check with tester. b. OVER (abnormal temperature) message appears and buzzer sounds. Disconnect terminals and check with tester. c. Check performance of protector with LED on terminal unit. d. Check output from triac with tester or on oscilloscope. e. Check with tester. f. Repair or replace.
4. Temperature in column oven does not rise. (Requirement in item 3 above is met.)	a. Faulty door switch.	a. Check with tester.
5. Temperature in AUX.2 does not rise. (Requirement in item 3 is met.)	a. AUX.2 oven short pin is open on terminal unit.	a. Check with tester.
6. Temperature at each part does not stabilize.	a. Faulty heat-sensitive element. b. Faulty triac. c. Faulty temperature control circuit.	a. Disconnect terminals and check with tester. b. Check on oscilloscope. c. Repair or replace.
7. There is a certain difference between the setting point and measurement value.	a. Faulty heat-sensitive element. b. Faulty base unit.	a. Disconnect terminals and check with tester. b. Adjust, repair or replace.

Phenomenon	Cause	Check and countermeasure taken
8. Unable to perform key inputs. Abnormal characters appear on display.	a. Faulty contact between key unit and main body.	a. Check to see if connector is positioned correctly. If not, set it correctly.
	b. Faulty switch unit.	b. Set connector correctly.
	c. Faulty 5V power supply.	c. Check with tester.
	d. Faulty base unit.	d. Repair or replace.

Resistance of the heat-sensitive element in normal conditions is 100 + 0.4 X temperature of the heat-sensitive element (°C)Ω.

4. Maintenance

Periodic maintenance and inspection of the GC-14B will ensure good analysis results. The items covered in this chapter are listed below.

1. Overtemperature protection system
2. Cleaning of cover
3. Glass insert
4. Septum
5. Graphite ferrule
6. Capillary column
7. Hydrogen flame ionization detector (FID)

4.1 Overtemperature protection system

Check the overtemperature protection system every six months.

- Checking procedure

(1) Disconnect the column.
(2) Set the maximum temperature of COL to 90°C. (FUNCTION 7)
(3) Set the column oven temperature to 100°C.
(4) Confirm that the overtemperature protection system operates properly, and the temperature falls.
(5) Check the temperature control zones respectively in the same way.

4.2 Cleaning of cover

Cleaning of cover should be done with a soft cloth slightly damped with water or a solution of water and a mild detergent. Carry out appropriate decontamination if hazardous material is spilt on or inside the equipment on user's responsibility. Before using any cleaning or decontamination method except those recommended by the manufacturer, user should check with the manufacturer that the proposed method will not damage the equipment.

4.3 Maintenance of glass insert

CAUTION

HIGH **TEMPERATURE**

Maintenance of the injection port should be performed only once the temperature has been lowered. Also, turning the injection port nut when hot can cause the nut to seize.

Note For details on handling the glass insert, refer to "Chapter 7: Split/splitless injection port".

When to perform maintenance and inspection

(a) Before starting a series of analysis

(b) If the reproducibility of retention time and peak areas low.

(c) If a ghost peak is detected

What *to* inspect

(a) Type of glass insert (if the wrong type of glass insert is used, results will be poor).

(b) Damage to the glass insert (can cause poor reproducibility)

(c) Silica wool inside the glass insert
(Improper packing can cause poor reproducibility.)

(d) Contamination on the glass insert wall or pieces of septum in the glass insert
(Contamination can cause poor reproducibility or ghost peaks.)

Cleaning the glass insert

Note When removing contamination in the glass insert, the graphite ferrule should also be removed. Adsorption of solvent to the graphite ferrule cause ghost peaks.

1. Removing pieces of septum from the glass insert

Push out the silica wool using a long, thin stick. Clean the insert before repacking with silica wool.

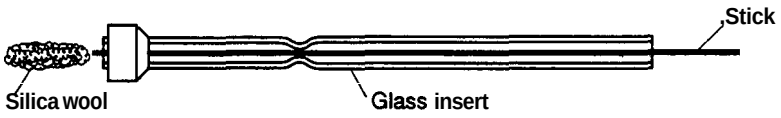


Fig. 6.4.1 Removing the silica wool

2. Removing contamination adhering to the glass in-sert. .

After removing the silica wool, clean the insert with a piece gauze soaked in solvent (acetone etc.).

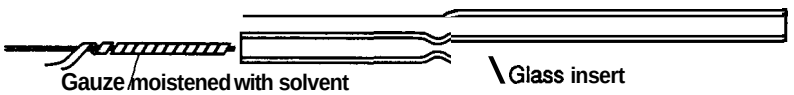


Fig. 6.4.2 Cleaning the glass insert wall

3. If contamination on the glass inner wall is pronounced

Soak the contaminated portion of the glass insert in solvent (acetone etc.) for several hours. Then, clean the insert with the gauze moistened with solvent.

Some methods may also require de-activation of the glass insert.

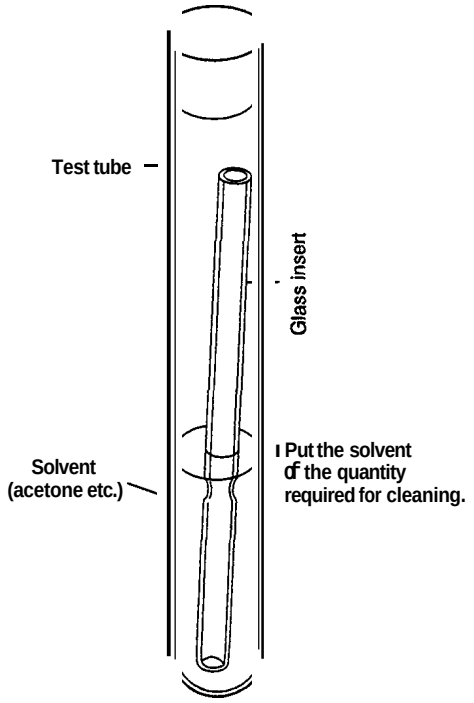


Fig. 6.4.3

4.4 Maintenance of septum

CAUTION

HIGH TEMPERATURE

Maintenance of the injection port should be performed only once the temperature has been lowered. Also, turning the injection port nut when hot can cause the nut to seize.

Note For details on handling the septum, refer to "Chapter 7: Split/splitless injection port".

■ When to perform maintenance and inspection

(a) Before starting a series of analysis
(b) When reproducibility of retention times and peak areas are poor.
(c) When a ghost peak is detected
(d) After approximately 100 injections.

■ What to inspect

- Gas leakage (This will be evidents as retention times shift.)
- Contaminated septum (This will be evident as ghost or extra peaks appear.)

■ Conditioning of silicon rubber septum

When high sensitivity analysis is performed, impurities from the silicon rubber septum may be detected as a ghost peak.

In such a case, perform conditioning of the silicon rubber septum according to the procedures below.

It is recommended to perform this conditioning as just before use as possible. If the silicon rubber septum is left unused for long hours after conditioning, impurities may adhere again.

(1) Soak the septum in hexane and leave it for 10 to 15 hours. The septum absorbs hexane and expands to double. Prepare a container that has cover with large taking port.

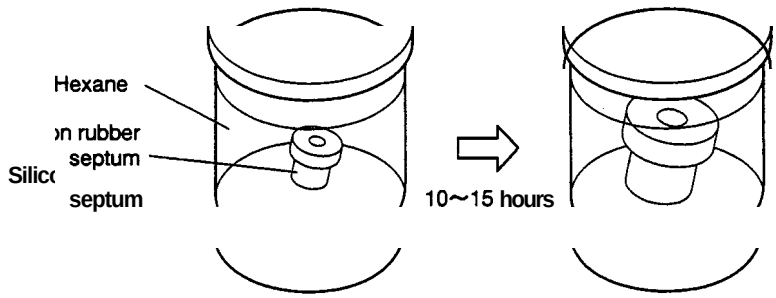


Fig. 6.4.4

(2) Put the septum to the clean container.

The septum expanded by absorbing the hexane is liable to break. Be careful when handling it.

(3) **Dry** the septum in the clean air spontaneously.

(4) After having dried, bake it in the column oven at 130-150°C for about two hours.

4.5 Maintenance of Graphite Ferrule

CAUTION

HIGH **TEMPERATURE**

Maintenance and inspection of the graphite ferrule should be carried out after the temperatures of the oven, injection port, and detector have been lowered. Also, turning the nut when hot can cause the nut to seize.

Areas where graphite ferrules are used

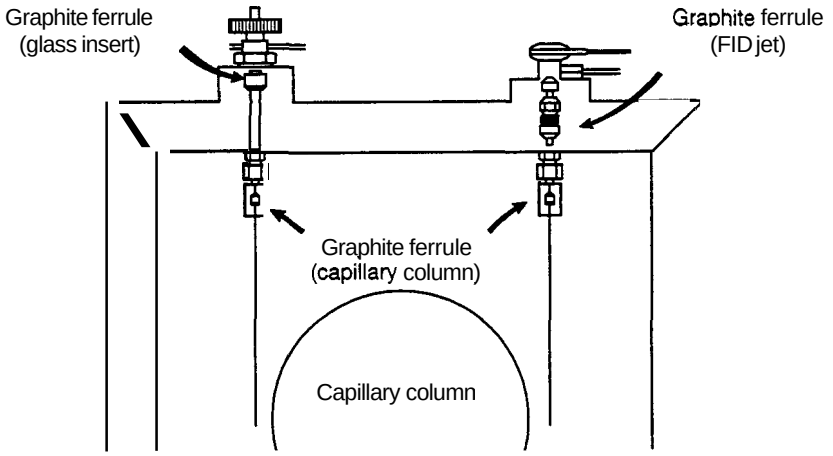


Fig. 6.4.5

When to perform maintenance and inspection

(a) When installing a new graphite ferrule

(b) When a ghost peat is detected in the Programmed analysis

(c) When a large baseline drift occurs during programmed analysis

What to inspect

- Gasleakage

  (Leakage of carrier gas caused by deterioration of the graphite will cause poor reproducibility.)

- Impurities in the graphite

  If impurities are present at the connection of the glass insert or column inlet, a ghost peak may appear. (In the typical split analysis, column graphite ferrule contamination is much less likely to cause ghost peaks because of the carrier gas flow path.)

  If impurities are present at the connection of the column outlet or FID jet, baseline drift may occur.

Conditioning of graphite ferrule

If impurities in the graphite ferrule cause problems, condition the graphite ferrule using any of the following methods. Perform conditioning just prior to analysis. After extended periods of non-use, conditioning may become necessary.

1. Conditioning with gas torch

   Place the graphite ferrule into blue flame of gas torch for approx. 30 seconds.

Caution	Use tweezers or a wire to hold the ferrule in the flame.

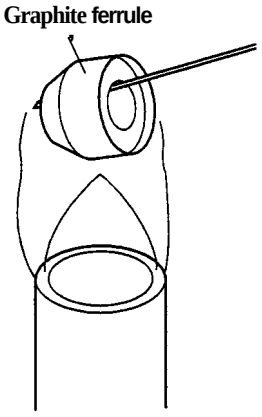


Fig. 6.4.6

2. Conditioning in the column oven

   Place the graphite ferrule in the column oven and condition at 399°C for 2 - 3 hours.

4.6 Maintenance of Capillary Column

CAUTION

HIGH TEMPERATURE

Maintenance and inspection of the graphite ferrule should be carried out after the temperatures of the oven, injection port, and detector have been lowered. Also, turning the nut when hot can cause the nut to seize.

Note For details of handling of the capillary column, consult the column instruction manual.

■ When to perform maintenance and inspection

(a) When using a new graphite ferrule or a graphite ferrule that has not been used for an extended period

(b) If ghost peaks are is detected

(c) If the baseline is unstable

■ What to inspect

(a) Baseline (If impurities have adhered to the liquid phase of the capillary column, the baseline becomes unstable or a ghost peak may be detected.)

(b) Visual inspection of capillary column

■ Conditioning the capillary column

1. Changing the carrier gas flow rate

When conditioning the column, it is not necessary to modify the flow rate of the carrier gas from that which is required for analysis. However, carrier gas can be conserved by decreasing the split ratio.

2. Setting the column oven temperature

Set the temperature of the column oven approximately 30°C higher than that is required for analysis. However, do not exceed the maximum allowable temperature of the column.

3. Setting temperature of the injection port and the detector

It is not necessary to change the temperature of the injection port. Set the temperature of the detector approximately. 30°C higher than that of the column oven.

4. Conditioning time

Perform conditioning for two or three hours, depending on the degree of contamination.

Note Some types of column liquid phase are easily damaged. Take note of the maximum temperature of the column and conditioning time. In addition, if the presence of oxygen in the carrier gas can seriously damage the liquid phase.

4.7 Maintenance of FID

CAUTION

HIGH TEMPERATURE, HIGHLY FLAMMABLE, SHOCK HAZARD

Before performing maintenance and inspection of the FID, be sure to first perform as follows.

1. Cur off the hydrogen gas supply to extinguish the flame.
2. Lower the detector temperature to below 100°C.
3. Turn **OFF** the power to the instrument.
4. Remove the capillary column from the FID.

If maintenance is performed when the **FID** is hot, a burning hazard exits. Damage to the detector may occur when disassembly is performed when hot. The threaded set screws and nuts may potentially seize.

Note For details on handling the FID, refer to Chapter 12 "Hydrogen flame ionization detector".

■ When to perform maintenance and inspection

Perform inspection and maintenance in the situations listed below.

(a) FID cannot be ignited.
(b) No peaks are detected
(c) Noise is severe.
(d) Peak shape is abnormal.

■ Points of inspection

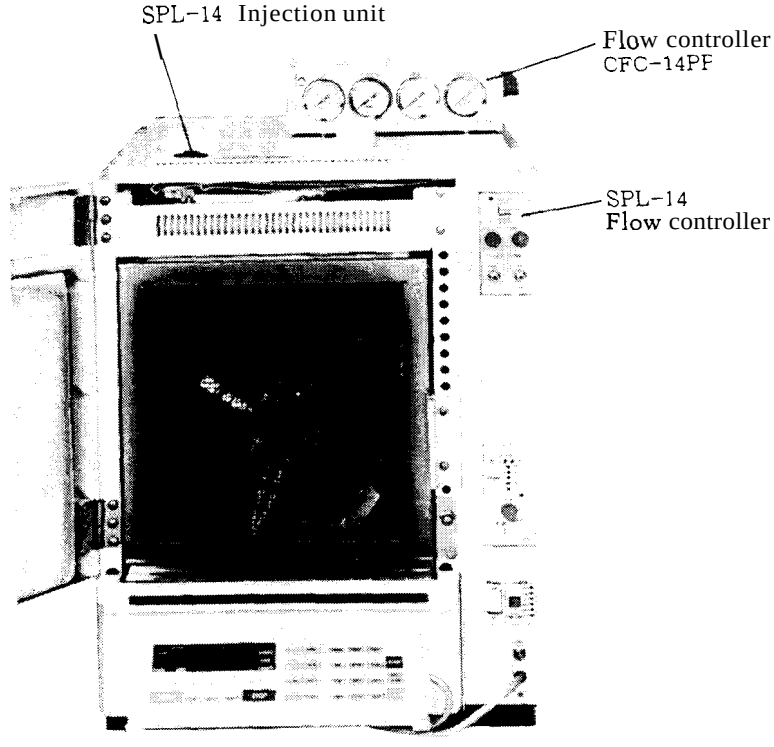
- Clogged jet. (Clogging of the jet may cause ignition failure or no detection of peaks.)
- Contaminated jet. (Contamination may cause noise.)
- Damaged jet. (Damage can cause abnormal peak shape.)

■ Cleaning the FID jet

If thip of the FID jet is clogged, remove the **FID** jet reffering chapter12 and insert a thin stick into the jet tip to clean it.

7. SPL-14 Split/Splitless Sample Injector

In this chapter, explanation is given on how samples are introduced for the capillary system. The model GC-14BSC is equipped with this **SPL-14** split/splitless sample injector as standard.



1. Split/Splitless Sample Injection

The capillary sample injection part of the unit is adaptable to either split or splitless type sample injection. In order to avoid ghost peaks that may result from impurities eluted from the rubber septum, which presents problems in trace analyses, the sample injection port is provided with a flow-line for purging the rubber septum, which is called the "septum purge flowline".

Solenoid valve SV1 is provided before the split **flow** control resistance (needle valve) to allow ON-OFF of the gas flow.

Also, a filter is provided for each of these flowlines to protect these parts against attack by organic substances.

Explained briefly are the principles of the split and splitless sample injection methods.

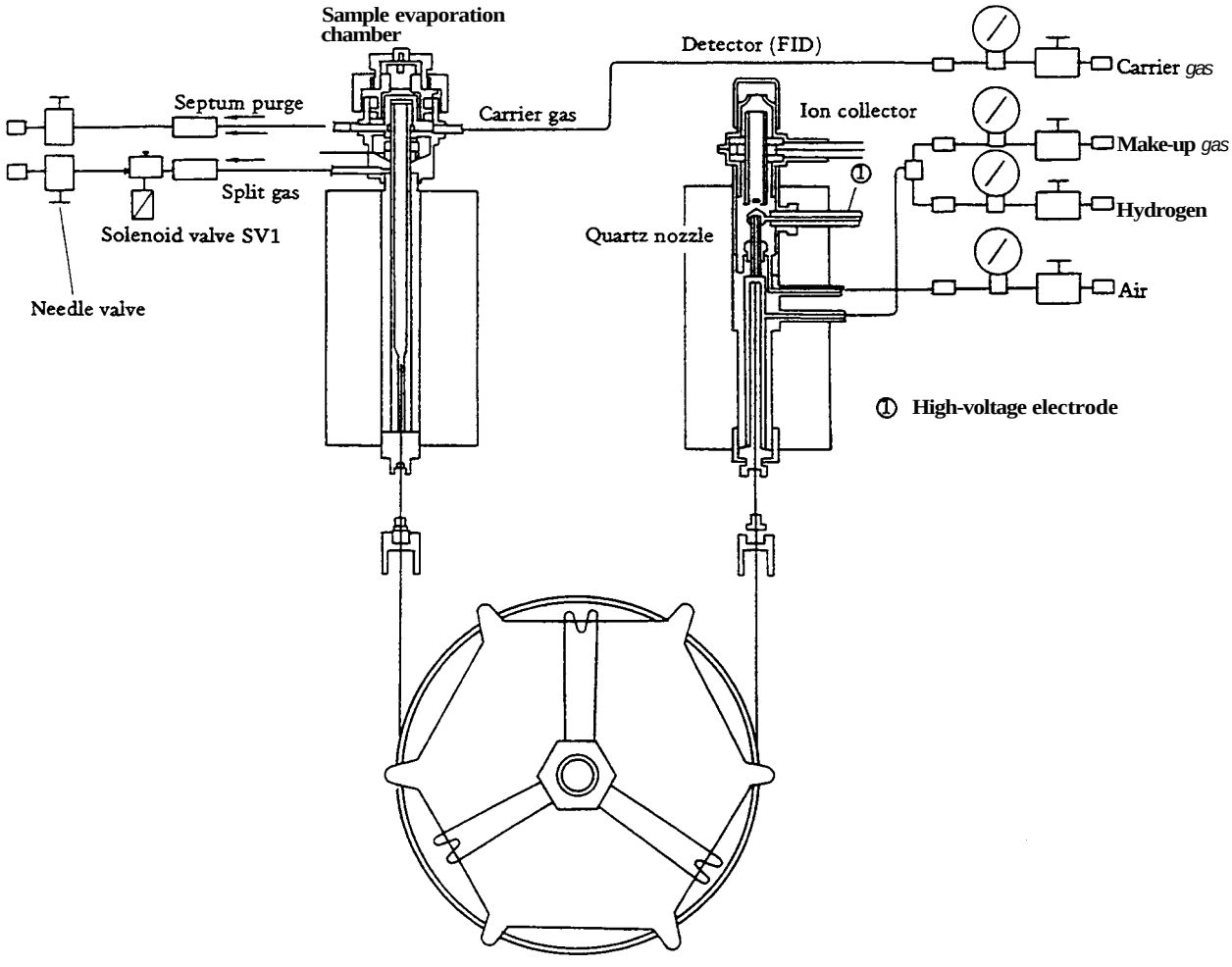


Fig. 7.1 Split/splitless sample injection method

1.1 Split sample injection method

(1) Three different kinds of gases can be used as carrier gas; of these helium gas is generally used as the carrier gas for its high resolving power and less time needed for analysis. Control of carrier gas flow rate is usually done in constant flow rate control with a packed column or wide-bore capillary column. With capillary columns, a constant-pressure control method is usually employed, because the flow rate of fluid flowing in the capillary column can be maintained constant when adjusting the split ratio, as to be explained below.

(2) The inlet side of the capillary column is inserted into the glass insert in the injection port, and carrier gas supplied is divided into two separate flowlines; the capillary column and the splitter, respectively. Assuming that the flow rates of carrier gas flowing in these two separate flowlines to be v_1 andv, [mℓ/min], then the split ratio can be expressed as $v_1 : v_1 + v_2$.

(3) Split ratio is selected usually in the range between 1/20 and 1/200. The reason for providing a splitter is that the absolute quantity of sample needs to be kept low to obtain the high separation performance that can be attained with the capillary column. Accordingly, when the split ratio is set at 1/50, 1/50 of the quantity of sample injected is introduced into the capillary colum and the remaining sample is discharged to the outside by way of the splitter. (Although accurate control may be necessary if concentration of the sample is extremely low or high, split ratio is normally set at around 1/50 or at 1/100.)

(4) To obtain separation on the capillary column in high efficiency, selection of proper flow rate of the carrier gas is essential. When helium is used as the carrier gas, around 40cm/sec is appropriate, for example, in terms of the linear velocity. The optimum flow rate of carrier gas is obtained on the H-U curve as to be explained later with reference to Fig. 7.2. Flow rate inside the capillary column is adjusted with the column inlet pressure. Two methods are available for measurement of flow rate; (1)direct measurement at the column outlet using a soap film flow meter, and (2) with the column connected with the detector, methane gas is injected and the carrier gas flow rate (linear velocity) is calculated from the retention time measured.

(5) The outlet side of the capillary column is connected with the detector, and a gas called make-up gas is fed to the connection part, at a rate of 40 to 60mℓ/min in normal cases.
The reasons for adding such a make-up gas are; (1)to obtain carrier gas flow rate necessary for maintaining the sensitivity of the detector (because of the low flow rate in the capillary column) and (2) to increase the gas flow rate for preventing expansion of peaks at the junction of the detection and the column.

(6) In the case of split sample injection, once the flow rate in the capillary column, split flow rate and the make-up gas flow rate have been decided, subsequent operations are carried out just in the same manner as those for ordinary analysis using packed columns. This method is most commonly used for analysis where concentrations of samples are relatively high. When concentration of sample is low, however, splitless sample injection, as to be described next, becomes necessary.

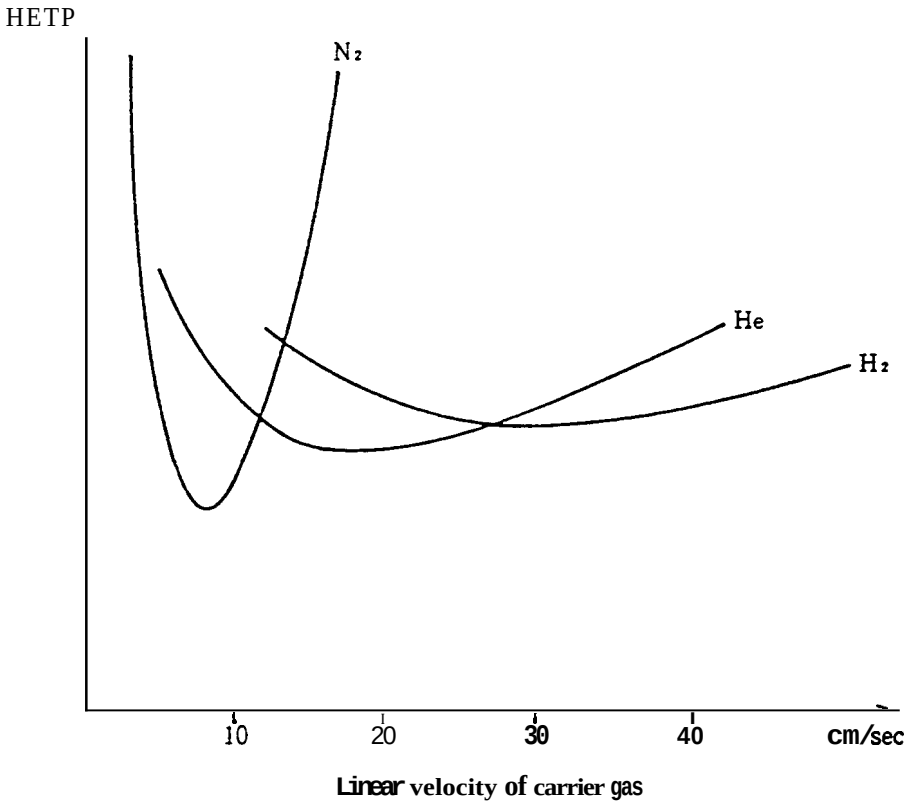


Fig. 7.2 Influence by the kinds of carrier gas (H-U curves)

1.2 Splitless sample injection method

This sample injection method is used when concentration of sample is low (at 10ppm or lower, for example), which cannot be detected accurately by the split sample injection method. This method (called the Grob method) can be used interchangeably with the split sample injection method. Samples applicable to this method are limited to samples that have been diluted with solvents.

To avoid ghost peaks (peaks resulting from impurities), a rubber septum purging flowline is provided. Solenoid valve SV1 is provided before the split resistor (needle valve) to allow ON-OFF of gas flow.

(1) Solenoid valve SV1 is installed in the split gas flowline, which closes immediately before sample injection.

Since the carrier gas flow rate is controlled by the pressure regulator, the pressure at the inlet part of the capillary column is maintained constant even when the solenoid valve opens or closes. As a result, flow rate of carrier gas fed through the capillary column is always maintained at a constant rate.

Flow rate of carrier gas allowed to flow in the injection port is low, measuring at around 1 to 2mℓ/min, when the solenoid valve is closed, but it is increased to 50 to 100mℓ/min (when the split ratio has been set at around 50).

(2) Sample injected into the glass insert with solenoid valve SV1 closed is fed to the capillary column vaporized. Since the inner volume of the injection port is approximately 0.4mℓ, most of the sample is to enter the capillary column in 0.5 to 1 minute, if carrier gas is allowed to flow at a rate of 1 to 2mℓ/min.

If analysis is conducted under this condition, however, heavy tailing may occur due to the solvent that has diffused and is staying within the injection port. To avoid this, solenoid valve SV1 is opened later to increase the flow rate of carrier gas flowing in the port, so as to expel such residues to the outside of the system through the split discharge outlet. Thus, trouble due to the tailing of the solvent peaks can be minimized with the Grob method.

(3) Initial temperature of the capillary column is kept low for the stage where sample is fed from the injection port to the capillary column. It is generally considered that the appropriate temperature for the stage will be the temperature lower by 10 to 40°C than the boiling point of the sample solvent. The reason for this is that, if the temperature is kept with this range, the solvent evaporated within the injection port will condense immediately after entering the capillary column to form a liquid film on the inner surface of the capillary, which in turn will act as a sort of fixed phase, so that even the components of relatively low boiling points can be trapped in the narrow zone to suppress expansion of peaks. (Needless to say that this effect cannot work with those components with boiling points lower than that of the solvent, which cannot be trapped.) Such an effect is called the "solvent effect", and is differentiated from the trapping attained by cooling in normal cases. Therefore, when analyzing components of relatively low boiling points on this unit, proper selection of the types of solvents used and setting optimum initial temperature of the column are especially important. In this unit, analysis is made on such components only as can be introduced into

the column from the injection port within a certain period of time. Therefore, in some cases, substances of high boiling points may not be fed to the column completely.

(4) Selection of the proper type of carrier gas, flow rate in the capillary column, split flow rate and the make-up gas flow rate, etc. is done in the same manner as for the split sample injection method. The only differences from those for the split method are that the initial temperature of the column needs to be changed depending on the boiling point of the solvent used for the sample, and that analysis needs to be executed under heating to meet the purpose.

1.3 Specifications

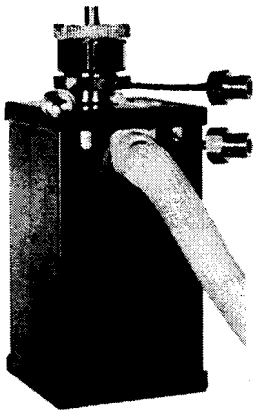
Method : Grobmethod

Range of temperature : Room temperature—399°C

2. Installation

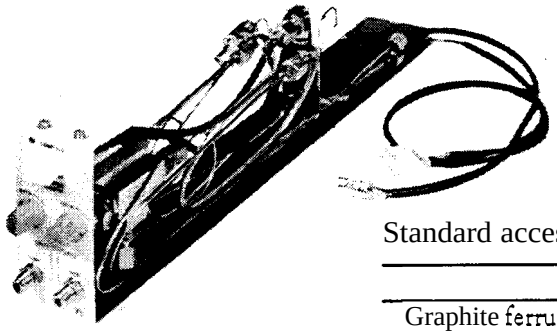
1. Details of components

The split/splitless sample injector (SPL-14) consists of the following components. In the case of a gas chromatograph main body of the capillary model, the components 1) and 2) are assembled in the main body before shipment.

1) 

SP/SPL INJ unit — Part No.

221-32547-91 (for 100V)
221-32547-92 (for 115V)
221-32547-93 (for 220V, 240V)
221-32547-38 (for 230V)

2) 

SP/SPL Flow controller unit **221-32420-91**

3) 

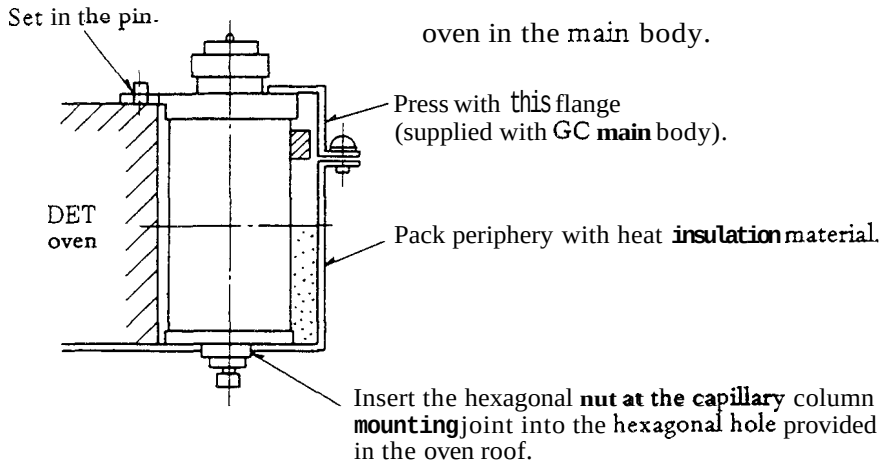
Standard accessory pack 221-32750-91

Description	P / N	Quantity
Graphite ferrule (for glass insert)	221-15563-91	1 (4pcs. per pack)
" G, 0.5 (for capillary column)	221-32126-05	1 (10pcs. per pack)
Column mounting nut (w/slit)	221-32705	2
Pipe MN2W-GN2W L700	221-33462-70	1
Branch-off pipe, for make-up gas	201-48386	1
Capillary adapter, for DET side	221-33193-91	1
Capillary column holder	221-31567-91	1
Aluminum packing	221-32543	1
Aluminum gasket (for column)	201-35183	1
Pipe insert, G6	201-36364-01	1
Spanner, 6 X8	086-03003	1
O-ring, silicone P12	036-11013	1
Glass insert, for split	221-37574-01	1
Glass insert, for splitless	221-32544	1
Graphite forming jig (MM5)	221-32510	1
" (Back ring)	221-12105	1
" (Nut)	221-32790	1
" (Forming shaft)	221-35549	1
Spanner SPL, for evaporation chamber	221-34123	1
FID nozzle for capillary	221-37304-03	1

2. Assembling in the main body

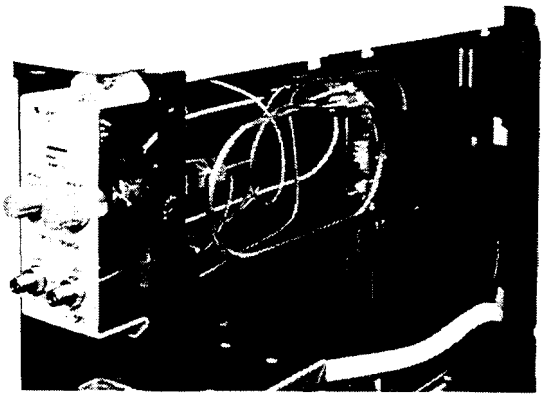
(1) Mounting SP/SPL INJ unit

Mount as shown on the left, at the left end of the INJ unit mounting part in front of the DET oven in the main body.

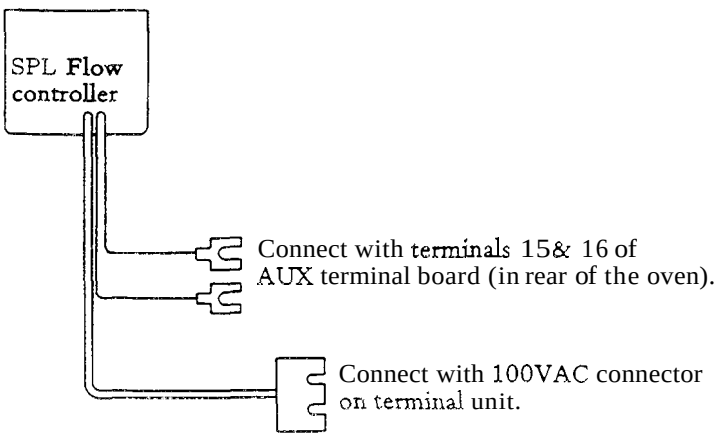


(2) Mounting the SPL **flow** controller

Remove the front panel for the control unit (refer to chapter 6. Construction, Removal of Right Side Panel.), and mount the flow controller in the uppermost position.

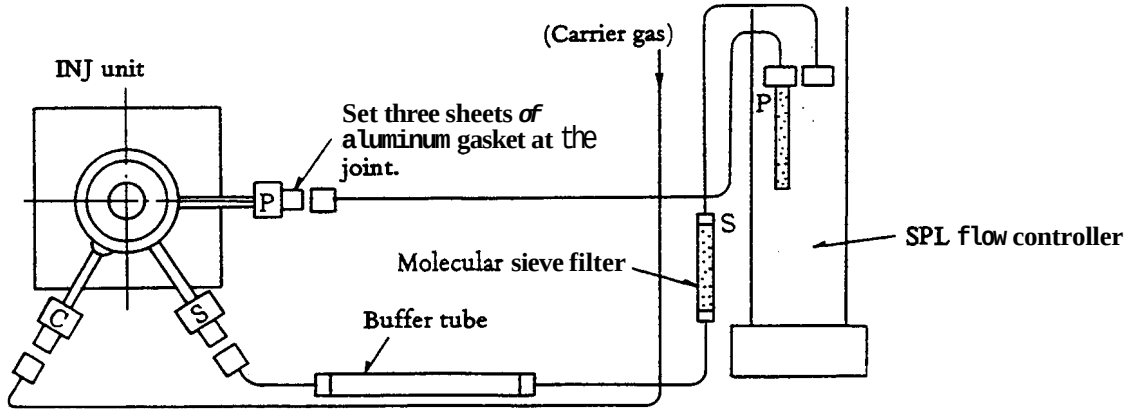


Wiring for SPL flow controller



(3) Connection of INJ unit with **SPL flow** controller

Molecular sieve filters in the **SPL flow** controller are connected with the split outlet and the septum purge outlet of the INJ unit. Connect a buffer tube to the split outlet.



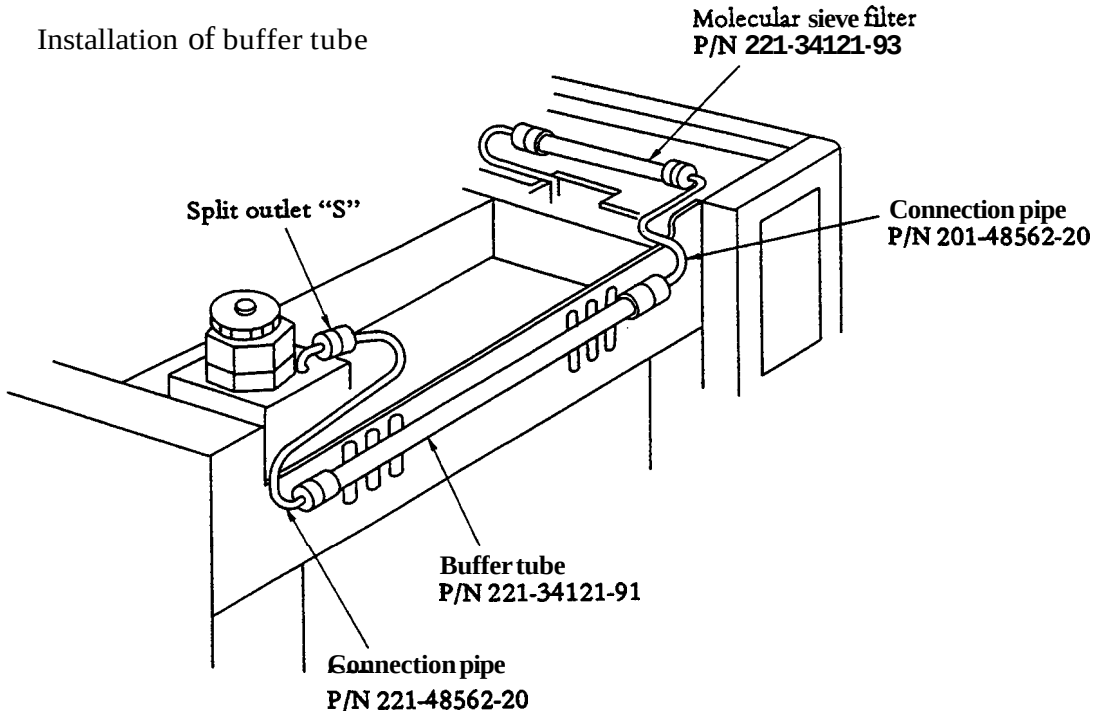
Marking on the joints

C. Carrier gas inlet – To be connected with carrier gas flow controller

S. Split outlet – To be connected with molecular sieve tube in split flowline.

P. Septum purge outlet – To be connected with molecular sieve tube in septum purge flowline.

Installation of buffer tube

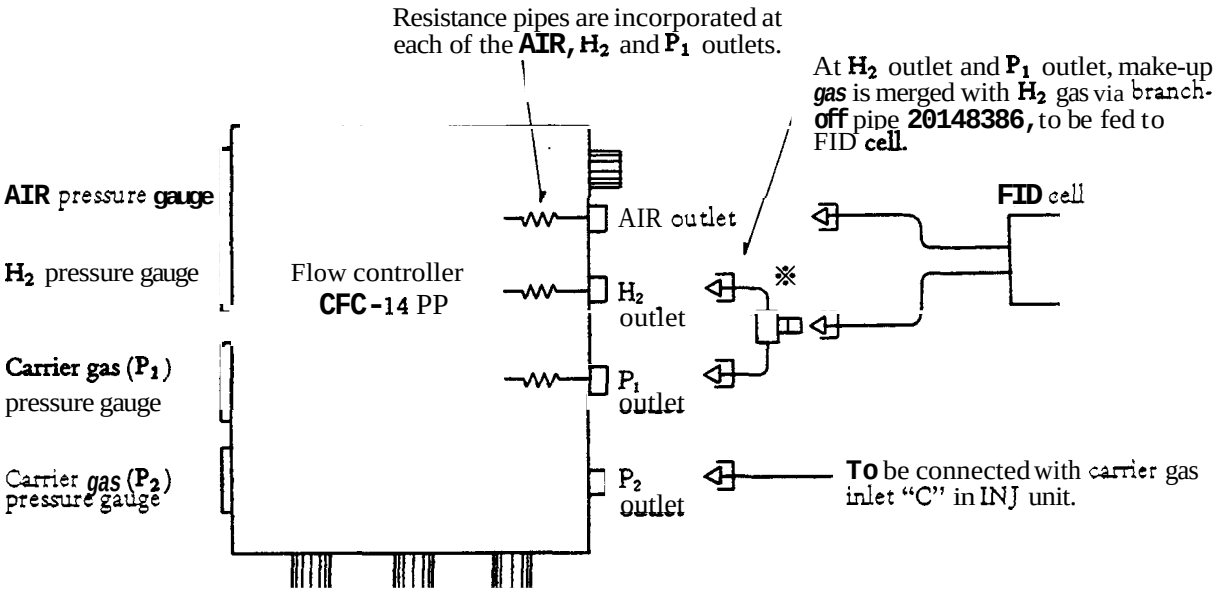


For installation of the buffer tube and the filter, use alminium gaskets for column included in the standard accessory pack of Chromatograph main body. This filter is for protection of solenoid valve to controll split ratio and needle valve. Remove the filter after injections of 50 to 100 times to check its contamination. To refresh the filter, put it in the column oven

of high temperature (approximately 300°C), flow carrier gas and drive out the contamination or refill molecular sieve (filler).

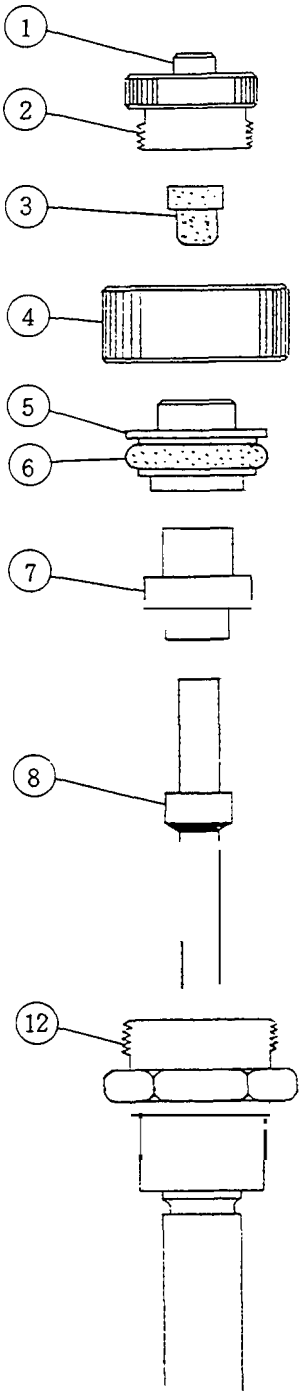
(4) Connecting carrier *gas* and make-up gas branch-off pipe

When used in the capillary split/splitless mode, the constant-pressure control method is employed, in which the column inlet pressure is controlled. Connections with the INJ unit and with the make-up gas flowline are done as shown below.



* Before setting this make-up *gas* branch-off pipe, check reading on the P_1 pressure gauge and the quantity of make-up gas fed to the P_1 outlet. (Flow rate of about 40mℓ/min can be obtained with N_2 *gas* when the pressure gauge indicates 50kPa.)

(5) Fitting the glass insert



Remove nut (4) and pull seal ring (5) upward. Then, O-ring (6) is exposed. Replace the O-ring if it is worn.

Using a pair of tweezers, extract ferrule holder (7) upward.

Insert glass insert (9) fitted with graphite ferrule (8) into the injection port main body (12) .

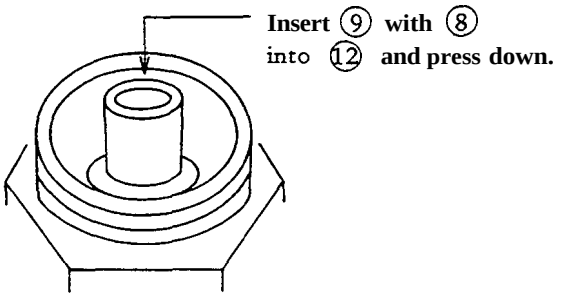
Refer to the section on Construction of Injection Port of SPL-14 to identify the numbers assigned for these parts.

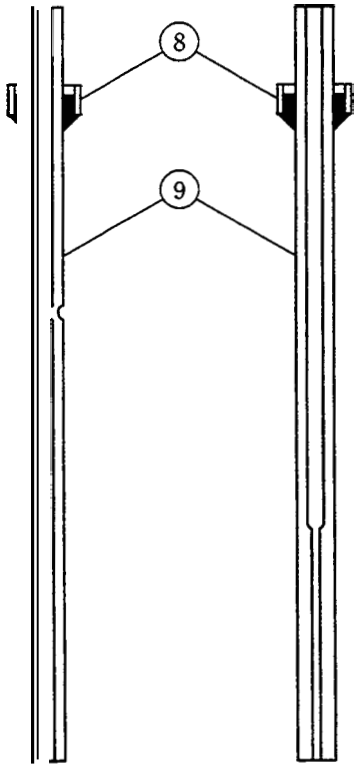
Reverse the above described steps to assemble, with good care taken to see that the ferrule holder (7) is positioned in the right direction (with the groove coming at the right position). The pipe used at the septum purge outlet should fit in this groove.

To replace rubber septum used at the injection port, remove nut (2) and replace septum (3) with a new one.

Press glass insert 9 securely so that its bottom edge reaches the bottom of the evaporation chamber.

If this is not enough, upper edge of the glass insert touches the injection rubber valve. This becomes the cause that normal septum purging flow is not obtained and there may be ghost peaks of septum.



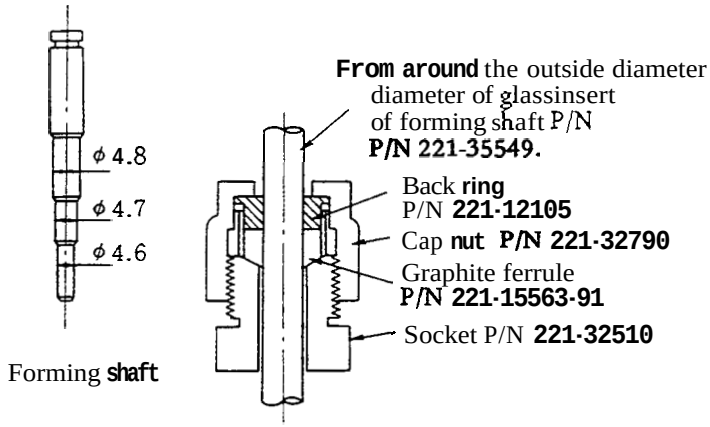


P/N221-37574-01
For split injection method

P/N221-32544
For splitless injection method

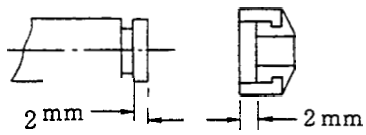
Fitting graphite ferrule (8)

Using the forming jig included in the accessory pack, reform the ferrule with the following sequence.



As shown in the above fiiure, put the graphite ferrule in the forming shaft and fasten its hexagonal nut with a spanner. Then, take out the graphite ferrule only and fix it in the glass insert.

The standard size of the graphite after forming is that the hollow from side rings is 2mm. The thickness of the head of the forming shaft is 2mm. Measure the hollow size by putting it in the hollow of the graphite.



When the hollow is smaller than 2mm, remove some graphite on the back ring side with tweezers to get the size. This is necessary for adjusting vertical positions of side holes of the carrier gas inlet of the injection port and side holes of the ferrule holder.

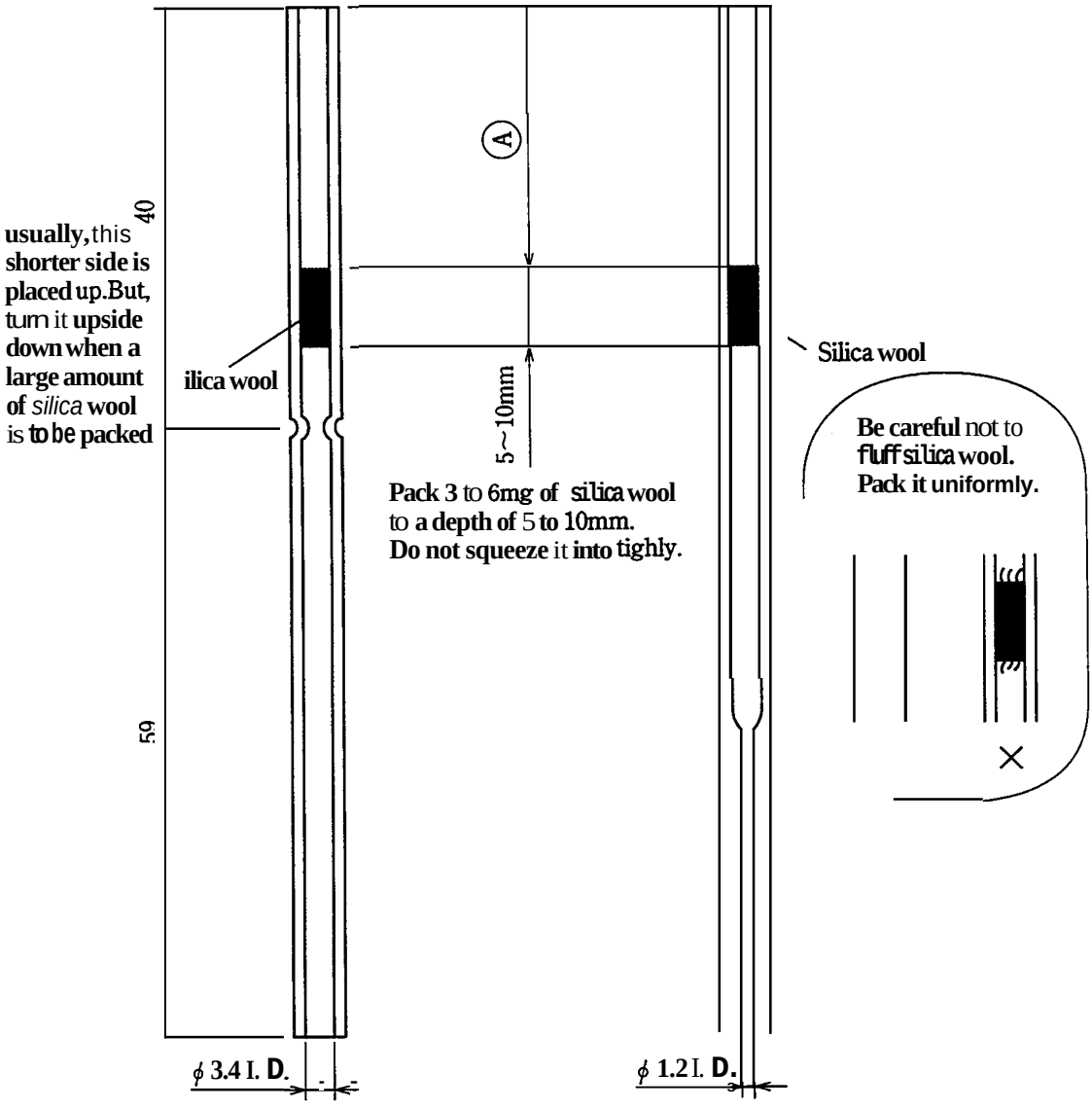
(6) Type of glass insert

The SPL-14 is provided with two different glass inserts; one for split analyses, and the other for splitless analyses.

For split analyses, glass insert having larger I.D. is used so that larger amount of sample can be injected. For splitless analyses, glass insert having smaller I.D. is used to increase the linear velocity.

Silica wool should be packed in the glass insert to prevent high boiling mist from entering the capillary column and to mix vaporized sample sufficiently. It is appropriate to pack silica wool at the position 1 or *2* mm apart from the needle tip at injection. If silica wool is too apart from the needle tip, satisfactory reproducibility may not be obtained.

Position of silica wool (length **(A)** in the figure) depends on the needle length of the syringe used and whether auto injector is used or not. Positions of silica wool in different cases are listed in the following table for reference.



Table

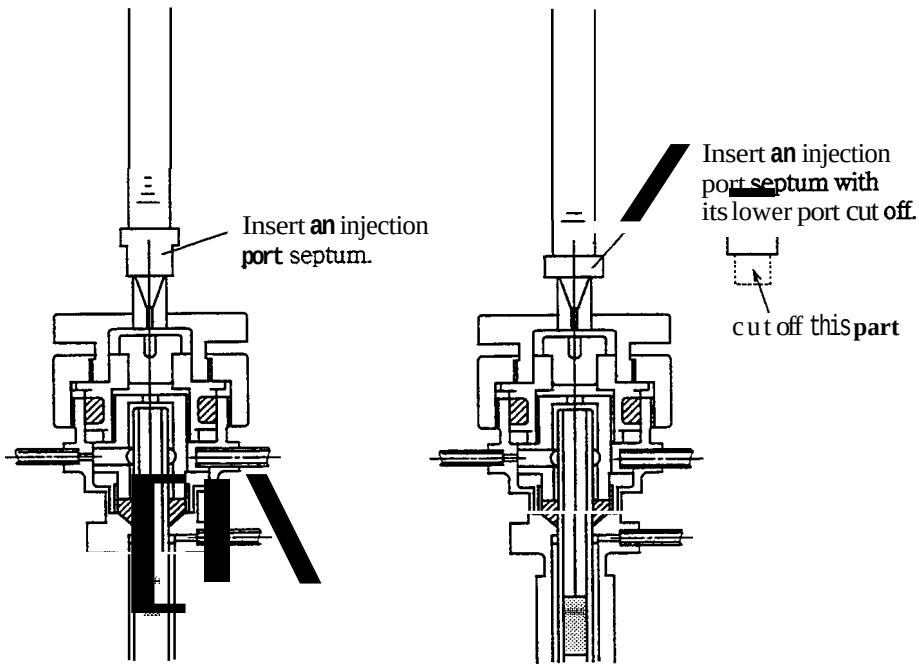
	Injection by AOC-14 (Needle length of syringe for AOC-14/17 is 43mm)	Injection by AOC-17 (Needle length of syringe for AOC-14/17 is 43mm.)	Manual injection using syringe for AOC-14/17 (needle length is 43mm.)	Manual injection using 50mm-long needle
Position of silica wool (dimension (A) in mm)	20mm	23mm	27mm	35mm

Note The above table indicates standard positions and weights of silica wool. For some particular samples, better result may be obtained by changing the amount of silica wool as follows.

1. For samples, such as agricultural chemicals, which have high adsorptivity, better result may be obtained by packing smaller amount of silica wool than listed.
2. For solvents, such as water, which have large latent heat of vaporization, better result may be obtained by packing larger amount of silica wool than listed.

Note Silica wool may be dislocated due to fluctuation of carrier gas flow rate during replacement of injection port septum or replacement of column. Before replacing injection port septum or column, return the inlet port pressure of the column to the atmospheric pressure. After replacement, return the inlet port pressure of the column gradually to the set pressure.

Note When injection is made manually in the INJ unit using AOC-14/17 (INJ unit in which silica wool position is adjusted for AOC-14/17), use a syringe for AOC-14/17.
To inject manually using a syringe for AOC-14/17 with silica wool at the same position as in AOC-14/17, insert an injection port septum into the bottom of the needle.

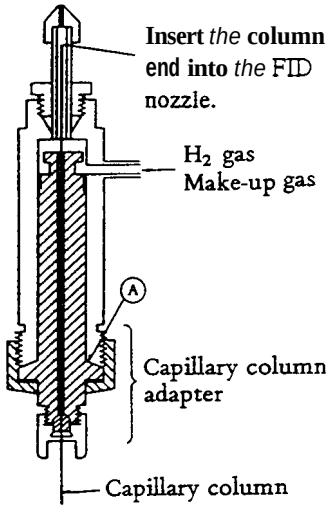


Manual injection when using AOC-14

Manual injection when using AOC-17

(7) Fitting capillary column adapter at the detector side

Fit capillary adapter (Part No. 221-33193-91) to the column connection side of the detector (FID).



The capillary column is attached in metal-to-metal contact (without using any gasket).

However, if airtightness is lost due to flaws on the contact surface produced after repeated fitting and removal, fit an aluminum gasket (optional part, Part No. 221-16053-91, available in a pack containing 50 sheets) at the position **(A),** to restore the airtightness.

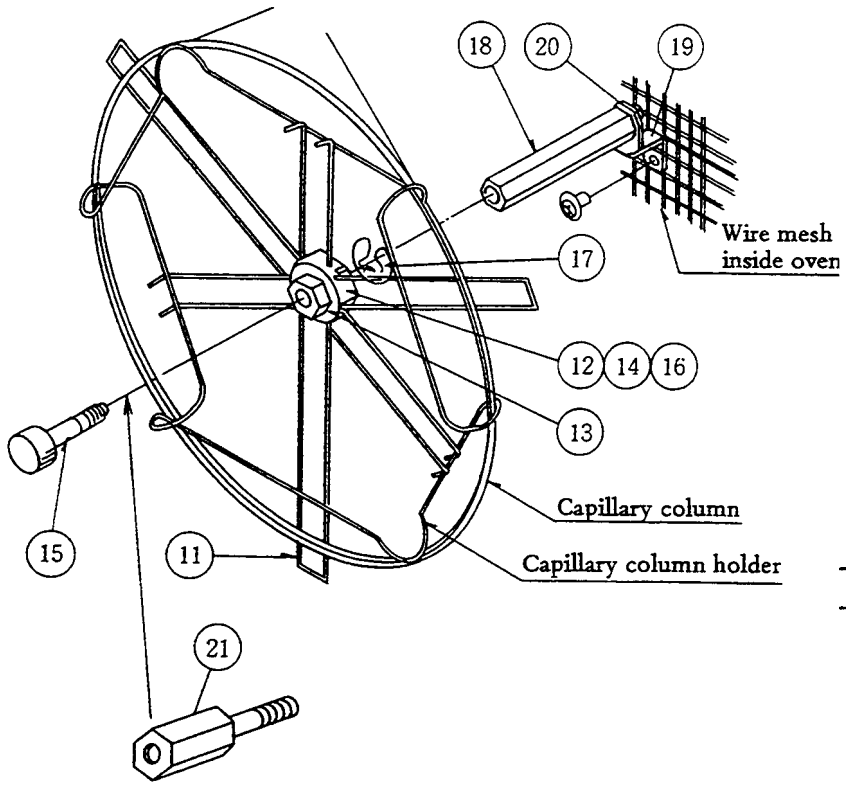
(7) Replace the FID nozzle with the nozzle for capillary (P/N 221-37304-03).

A nozzle for capillary is supplied in the **SPL**-14 accessory pack. Using this nozzle, the capillary column end can be inserted into the nozzle. Since the straight tube of the nozzle is a silica tube, small dislocation of the column does not affect performance only if the column end is in the nozzle. Differences in peak tailing and component adsorption caused by column installation can be prevented to improve operability.

If the nozzle for capillary is not used, lower the column a little (approx. 0.5mm) after the column tip hit the underside of the silica nozzle. Since peak tailing is often caused by this delicate installation, it is recommended to use the FID nozzle for capillary.

(8) Fitting the column holder

The column holder is included in the accessory pack, divided into two sub-assemblies, one consisting of the parts (11) to (17), and the other consisting of the parts (18) to (20). To install the column holder inside the gas chromatograph oven, insert the threaded portion of hook (20) into the wire mesh as shown in the figure and fasten it with the supplied screw.



When two capillary holders are fixed, fix the first holder with this spacer and fix another holder to the threaded portion of the spacer.

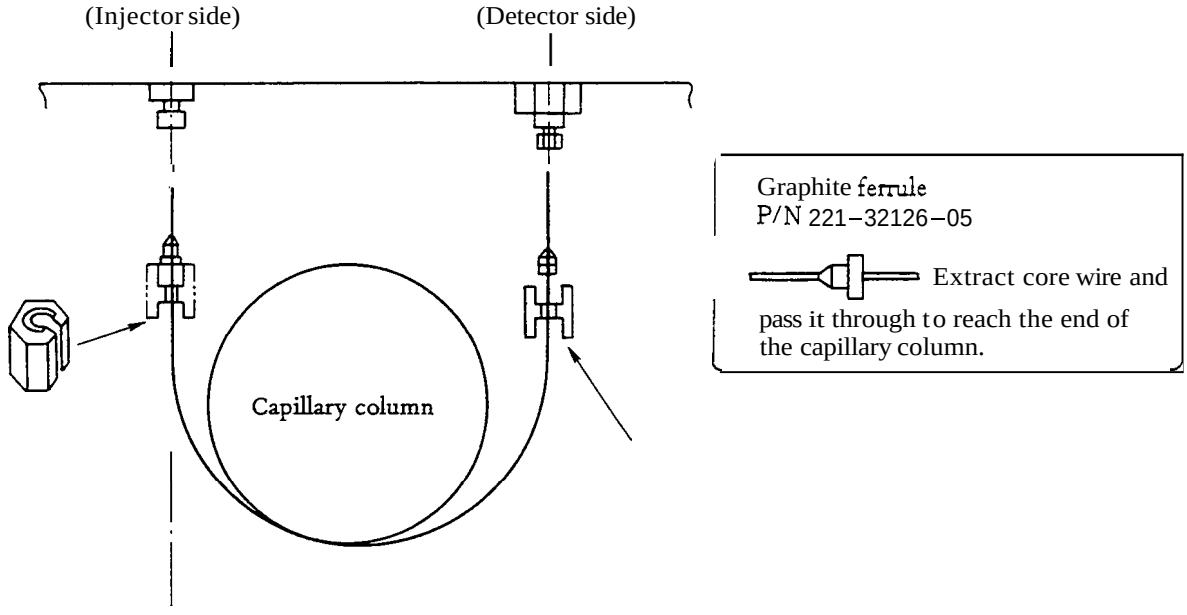
Column holder assembly
Part No. 221-31567-91

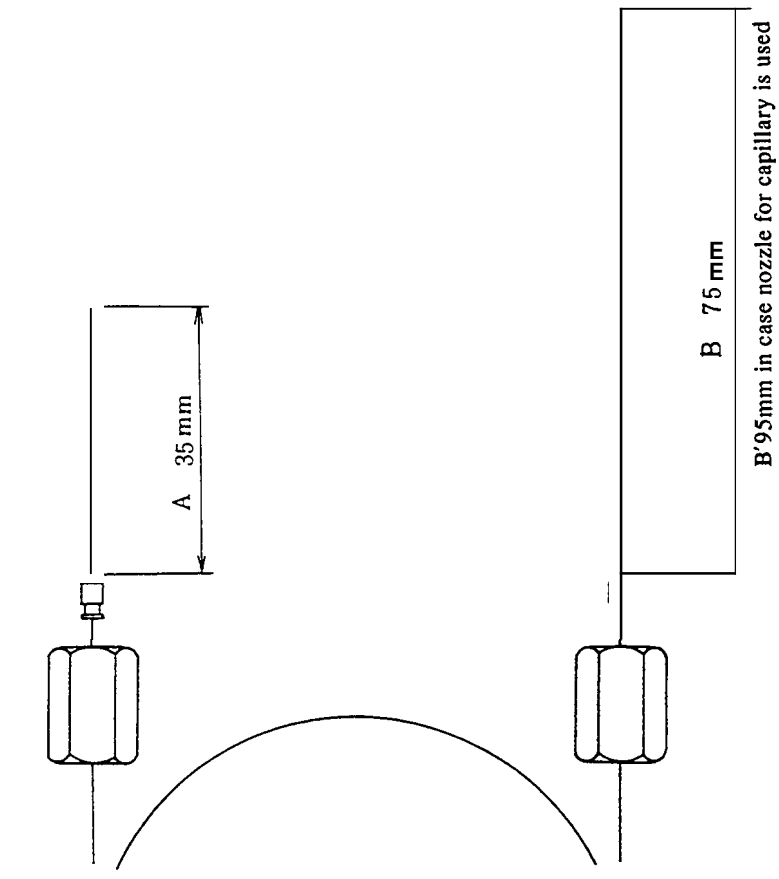
	Description	P/N
11	**Arm**	221-31568
12	**Guide, Arm**	221-31569
13	Fixing **screw**	221-31570
14	**Nut**	221-31571
15	**Screw**	221-31572
16	**Washer**	023-77050
17	**Stopper**	026-66204
18	**Post**	221-31573
19	**Mounting bracket**	221-32146
20	**Screw, M4X8**	020-46535
21	**Spacer**	221-34015

Set it at the central part in the oven, e.g. at around the center shaft of the fan motor, and fasten it securely.

The remaining parts (11) to (17) (in a sub-assembly) are fit to the post (18) with screw (15) after the column is set on the column holder. The arm (11), when assembled, is so constructed that the coil diameter held can be changed as desired in the range of 100mm and 220mm.

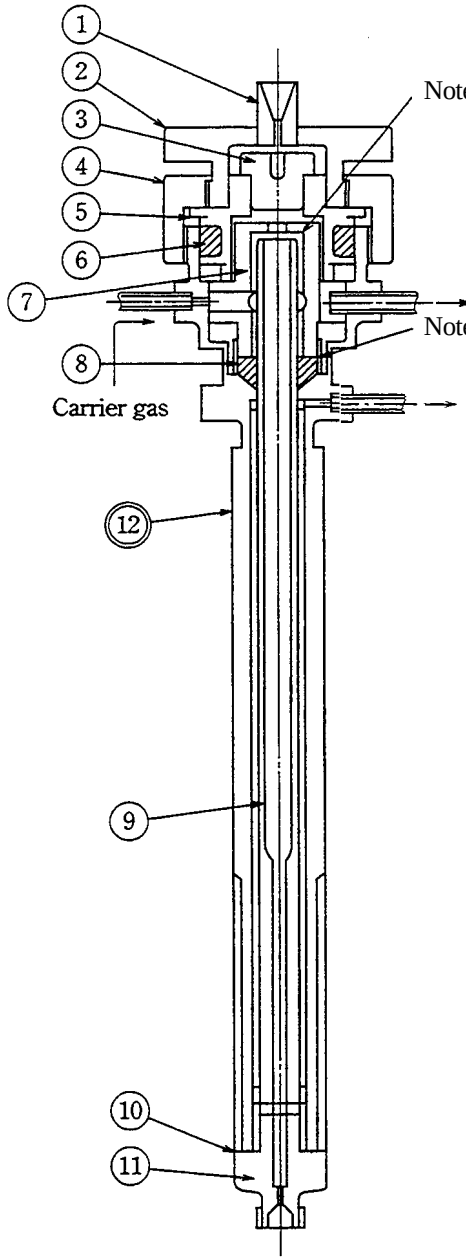
(9) Mounting column





3. Construction of SPL-14 injection port

Construction of the injection port is shown on the left. It is so constructed that the glass insert inside the evaporation chamber can be replaced through the injection side.



Note. This septum purge flowline may not be formed correctly if the glass insert is dislocated upward, resulting in ghost *peak* of septum. **Insert** the glass insert deeply into the vaporization chamber.

Septumpurgeout

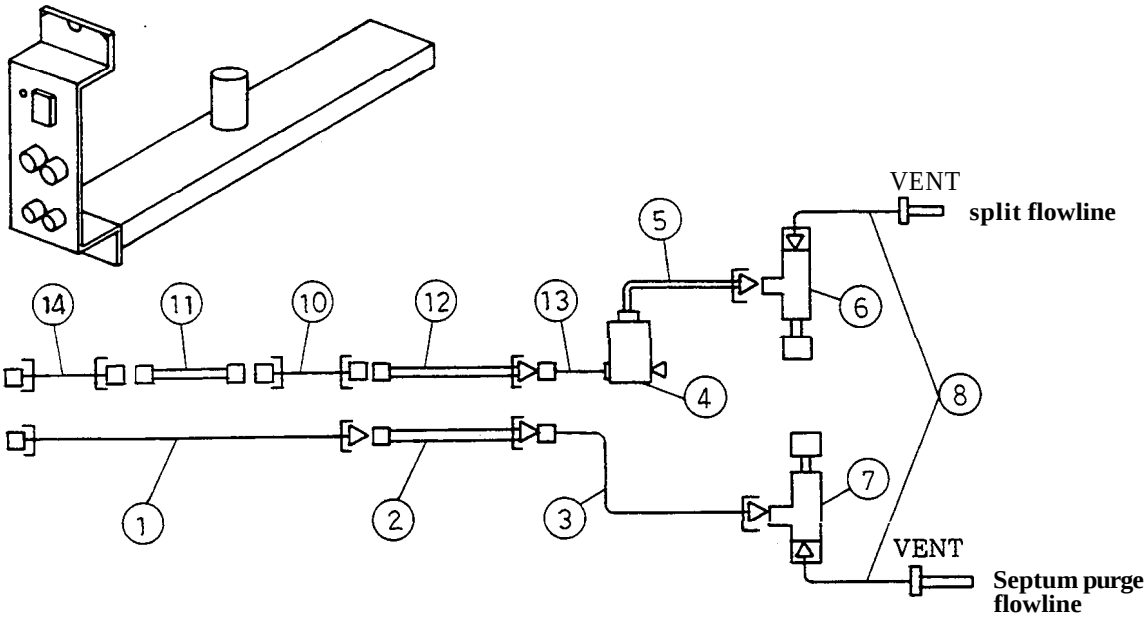
Note. Tighten the nut ④ securely not to allow clearance at this section. If the flowline is cut short at this section, septum purge will lost its effect.

split out

	Description	P / N
1	Needle guide	221-34579
2	Septum holder **A**	221-32538
3	Septum	201-47435
4	Nut	221-32539
5	Seal **ring**	221-32540
6	**O-ring**	036-11013
7	Ferrule holder	221-32541
8	Graphite ferrule	221-15563-91
9	Glass insert split	221-37574-01
	Glass insert splitless	221-32544
10	Gasket	221-32543
11	**Column** joint metal	221-32542
12	**Main** body of evaporation chamber	221-32535-91

Fig. 7.10 Injection port of SPL-14

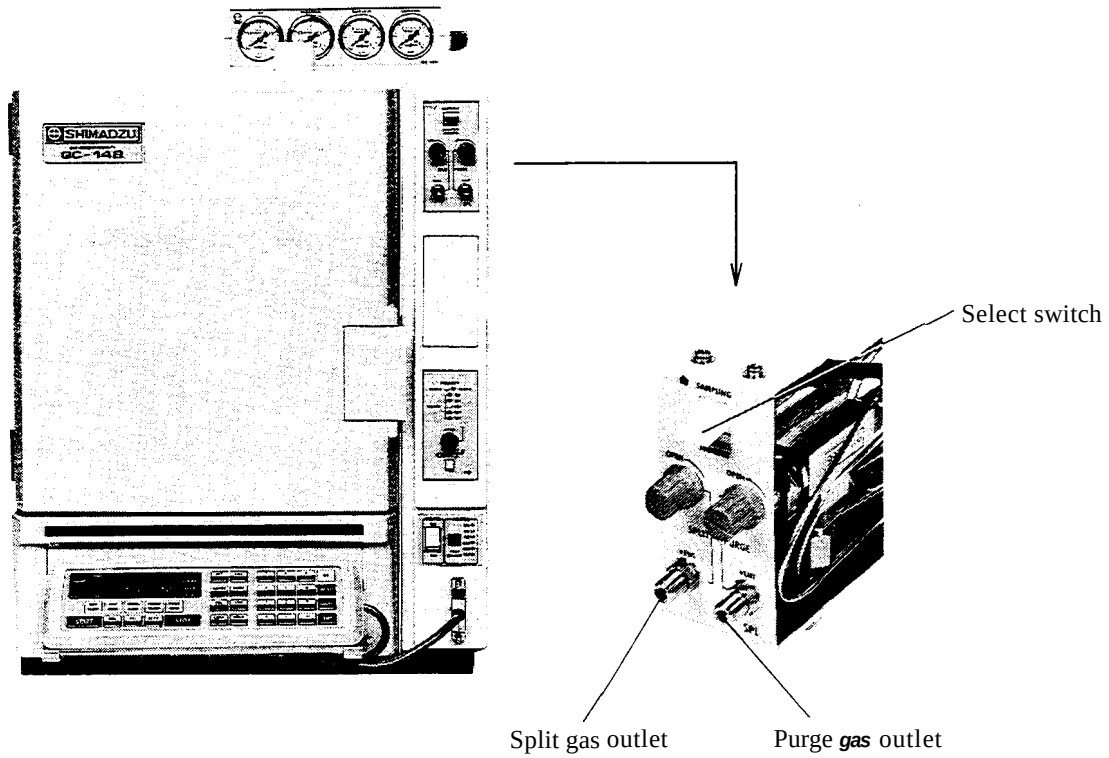
4. Construction *of* SPL flow controller



No.	Description	P I N
1	Pipe GF-MF	201-48560-50
2	Molecular sieve filter	201–36992–91
3	Pipe MM-MF	201-48557-30
4	Solenoid valve US-5M-37	040– 50271
5	Pipe M5-MF	221– 32428– 91
6	Needle vdve, split	221– 17482
7	Needle vdve, septum purge	221– 26995
8	Pipe GLMF	221– 31298– 91
10	Pipe GFGF	201-48562-20
11	Buffer tube	221– 34121–91
12	Molecular sieve filter	221–34121–93
13	Pipe M5-GF	221-32428-92
14	Pipe GF-GF	201-48562-25

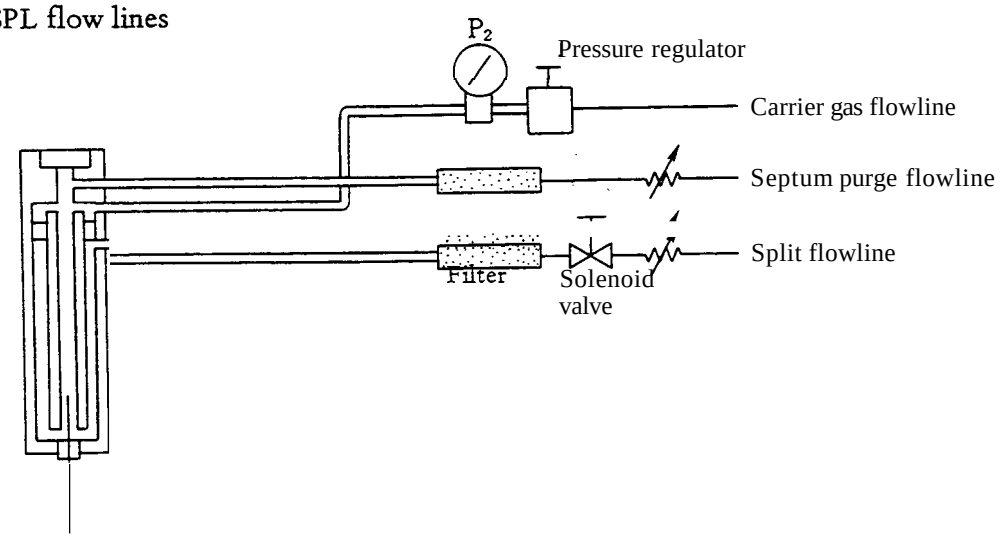
7

5. Operation of SPL-14



7

SP/SPL flow lines



1) Control parts of the SPL-14 are shown in the above figure.

When the power switch for the gas chromatograph is turned on and the SAMPLING-ANALYSIS select switch is set at the SAMPLING side, power is supplied to the solenoid valves and the indication lamp is lit **up.** At this time, the split flowline is blocked.

When analysis is made in the split method, this select switch is to be set at the ANALYSIS side at all times.

When analyzing in the splitless method, sample is injected with the select switch set at the SAMPLING side, and then it is shifted to the ANALYSIS side when a certain period of time has elapsed.

Procedures to be taken after fitting the column are explained in the following paragraphs.

2) Pressure should rise gradually by turning the control knob on the side clockwise, while observing the pressure of carrier gas (P_2) on the pressure gauge. Set to optimum pressure according to the Table below, to match the inside diameter and length of the capillary column used. (The figures shown in the Table are for the case where helium is used as the carrier gas.)

I.D. of capillary column \ Length	25m	50m
0.20 mm	150kPa	300kPa
0.25 mm	130kPa	250kPa
0.30 mm	100kPa	200kPa
0.50 mm	50kPa	100kPa

When the pressure is set properly, the linear velocity of carrier gas inside the capillary column should become the appropriate flow. As those figures shown above are given as a guideline, make further fine adjustment as necessary.

3) Shift the select switch to ANALYSIS. (The indication lamp should go out. Connecting a soap film type flow meter to the VENT port, and adjust with the control knob so that flow rate in the septum purge flowline and that in the split flowline will become 5 to 10mℓ/min and to 40 to 80mℓ/min, respectively.

4) Setting temperature in the injection port

Setting of temperature in the injection port greatly affects the result of measurements. A too low injection port temperature tends to prolong the analysis time, or will result poor recovery rate of high boiling temperature substances. Conversely, a too high injection port temperature is likely to cause distillation effects at the syringe tip, to degrade the reproducibility. So, it is necessary to select optimum temperature for the injection port according to the boiling point of the substance aimed at and the kind of solvent used for the sample. Generally, it seems to be appropriate to operate with the temperature set in the range from 200 to 300°C.

5) Setting column initial temperature

Setting of column initial temperature is especially important in the splitless sample injection method. To make best use of the solvent effect, the initial column temperature is set 10 to 40°C lower than the boiling point of the sample solvent. In the case of a high boiling point substance, which does not need the solvent effect, the column initial temperature is selected at temperature 150 to 200°C lower than the boiling point of the target component(s).

6) Determination of wait time

Another important factor on which reproducibility of analysis is dependent is the wait time, the time period from injecting sample till the opening of the solenoid valve. After injecting sample with the select switch being set at the SAMPLING side, wait for a predetermined period of time before switching over to the ANALYSIS side. If this wait time is too short, there wil be large loss in the high-boiling point substances, whereas too long a wait time may cause expansion of peaks of low-boiling point substances. By observing the changes in peak areas with time, it is known that the high boiling points substances enter the capillary column requiring fairly long period of time.

To cope with this problem, it is necessary to take appropriate measures, for example, by extending the wait time, or by increasing the carrier gas flow rate, or by raising the temperature in the injection port.

Normally, wait time is set between 0.5 and 2 minutes.

In order to obtain good reproducibdity, it is essential to maintain the wait time at a constant value for each repeat analysis.

7) Determination of make-up gas flow rate

When detection is made with the FID after leaving the capillary column, inert gas is added to obtain optimum sensitivity. As in the case of carrier gas, helium can be used as the make-up gas. Alternately, nitrogen can also be used for this purpose, separately from its use as the carrier gas. Nitrogen allows slightly better sensitivity than that obtainable with helium gas.

When other types of detectors than FID are used, it is also necessary to add make-up gas in oder to prevent expansion of peaks that may otherwise occur at the connections between the detector and the capillary column. Kind of the gas used and flow rate should be determined properly depending on the actual detectors used for analysis.

(Example: ECD ... N_2, FTD ... He, FPD ... N_2 or He)

8) sample injection rate

When the splitless sample injection method is used, flow rate of the carrier gas at the time of sample injection is fairly low, measuring about 1 to 2mℓ/min. Therefore, if a liquid sample is injected in a large volume, it will evaporate and increase its volume, to flow backward in the carrier gas flowline, causing it to deposit on the wall of the flowline. In the splitless injection method, the normal quantity with which sample can be injected at normal speed is about 1μℓ. If it is necessary to inject sample in higher quantities, some considerations are necessary, for example, by pushing the plunger slowly and by increasing the flow rate of the carrier gas.

9) Selection of solvent

When analyzing high boiling point substances, it is advantageous to use solvents of relatively high boiling points, like decane (C_{10}) or tetradecane (C_{14}). When such kind of solvent is used, there is little distillation effect occurring at the tip of the syringe, even if it is injected slowly, enabling determination with good reproducibility. Furthermore, initial temperature of the column can be made higher, thus shortening the analysis time.

In case of analysis involving a sample containing many components ranging from low to high boiling points, it is inevitable to use solvent of the lower boiling point than that of the target component(s). In the case of such analysis, it is often advantageous in terms of reliable reproducibility and determination, to introduce low sample volume in an as quickly as possible injection operation.

It is generally said that paraffins or acetone, etc. should preferably be selected as solvent for assured long life of the liquid phase in the column, avoiding use of aromatics or polar solvents for the purpose.

10) Routine operations (in the case of splitless injection method)

Once optimum analytical conditions have been decided (as to the flow rate, temperature condition, injection rate and wait time), routine operations for analysis are to proceed as follows.

(1) Confirm that the column oven temperature is stabilized at the set initial temperature.

(2) Set the select switch at SAMPLING.

(3) Inject sample via a microsyringe.

(4) Start up heating* and the data processing unit.

(5) **Wait for a predetermined period of time. (Wait till the sample shifts to the capillary column.)

(6) **Set the select switch at **ANALYSIS.**

* It is necessary to set the time period during which the column to be held at the set initial temperature (INITIAL TIME) longer than the WAIT TIME.

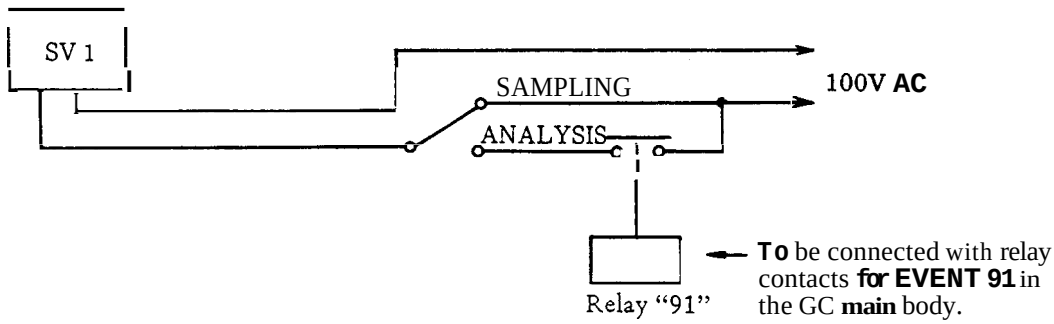
** For the SPL-14, these operations can be automated by using a time program. (Refer to 12 to follow.)

11) Use as a splitter

Changing the glass insert to the one intended for splitting (of 3.4mm I.D. with its one end reduced to 1.2mm I.D.), inject sample with the sampling switch set at the ANALYSIS position. Split ratio can be changed by turning the control knob for the needle valve in the SPLIT flow-line. Measure the split ratio by connecting a soap film type flow meter to the VENT port.

12) Case of operating solenoid valves for the SPL-14 using a time program:

The solenoid valves used in the SPL-14 are connected as shown in the circuit diagram shown below.



Accordingly, when this solenoid valve is to be operated via the time program functions provided for the *GC* main body, the solenoid valve SV1 can be turned ON and OFF via relay for EVENT 91, with the select switch set at ANALYSIS side. Even in this case, the indication lamp for monitoring the status of the stop valve is working effectively. That is, it indicates that the unit is then in SAMPLING state when the indication lamp is on, and indicates ANALYSIS state if the lamp is turned off. Key operations needed for standard time program are as follows:

① [SHIFT•D] [PROG] [ENT]

② [2] [ENT]

③ [SHIFT•D] [EVENT] [−] [9] [1] [ENT]

④ [SHIFT•D] [ESCP]

With the above key operations, ANALYSIS state is entered automatically in two minutes after starting injection and pressing the START button.

Needless to say, it is necessary to make key operation as shown below immediately before injecting sample to recover the SAMPLING state. (This can also be time programmed.)

SHIFT • D EVENT 9 1 ENT

13) **Handling** precautions

(1) When the glass insert has been replaced with a new one, there may a possibility of peaks resulting from surface soiling or otherwise in the next round of analysis. To avoid this, a blank test must always be conducted prior to the starting of actual analysis.

(2) Residue of sample deposited on the glass insert, if any, should be washed out with the proper solvent without fail.

(3) In some cases of splitless injection, column oven temperature may be lowered at the time of injection, and then raised abruptly at the time of analysis. If applied in such an operation, the column and the related parts made of PEG material may be damaged as they are susceptible to thermal shocks.

(4) If initial temperature of the column oven is too low, there may be a possibility of the liquid phase in the column being dissolved in the sample solvent.

(5) In case of a sample including components of wide boiling point range, the injection technique will have serious influence on the result of measurement.

Generally speaking, the injection method called "cold needle method" is adequate for the purpose. (Let air be drawn through the needle of the syringe. After sticking the needle tip into the septum, press the plunger promptly to inject sample. Extract the needle tip after 5 seconds or so after injecting the sample.)

5. Maintenance

Routine maintenance

(1) In order to protect the needle valves inside the flow controller, molecular sieve filter tubes are provided. Condition those filter tubes once a month.

(2) Clean the glass insert with proper solvent at regular intervals.

(3) In case of a high-sensitivity analysis, condition the graphite ferrule before use without fail.

(4) Some solvents may damage the liquid phase in the column.

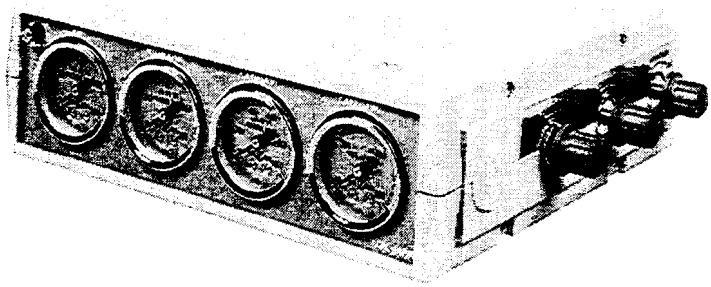
(5) There may be a case, where the liquid phase at the tip of the column may flow out as a result of repetitious splitless sample injection, degrading peak shapes. In such a case, cut off part of the column by 50cm or so from its tip and reconnect it in place.

Troubleshooting

Symptom	Countermeasure
Peaks of low boiling point substances expand.	• Lower initial temperature of column. • Change to solvent with higher boiling point. • Exchange column.
Peaks of high boiling point substances appear small.	• Extend wait time. • Raise temperature in injection port. • Increase flow rate of carrier gas. • Insert capillary column farther into the injection port.
Tailing of solvent peak is large.	• Increase split flow rate. • Increase septum purge flow rate. • Decrease injection rate.
Heavy ghost peaks appear.	• Condition graphite ferrule. *Increase septum purge flow rate. • Clean glass insert. • Clean carrier gas flowline. • Insert molecular sieve in the carrier gas flowline.

8. Flow Controller

The flow controllers of the **GC-14B** series are separate units. Description will be made on typical flow controller units.



1. Introduction

Carrier gas, hydrogen gas for the flame ionization detector, air for supporting combustion, and others are required for the operation of the gas chromatograph. The accuracy of the gas supply unit has a direct effect on the accuracy of analysis by the gas chromatograph and the flow controller is an important component of the chromatograph system.

The flow controller which is being outlined is a unit developed for gas chromatography permitting setting up an optimum arrangement for analysis and easy extension of the flow control system.

1.1 Gases used for gas chromatography

Detector	Type of Gas	Purity
Thermal conductivity detector (TCD) Gas with thermal conductivity considerably different from that of sample is generally used as the detector perceives the difference in thermal conductivity between carrier gas and sample.	Carrier gas o Helium (most optimum) o Hydrogen (requiring careful handling) o Nitrogen (usable but low in sensitivity)	99.99% min.
Hydrogen flame ionization detector (FID) Hydrogen and air are required in addition to carrier gas for mixing carrier gas with hydrogen and burning the mixture by supplying combustion support air.	Carrier gas o Nitrogen (most optimum) o Helium (usable) Combustion gas o Hydrogen o Air	99.99% As high a purity as possible. Supply compressed air from an oil-less air compressor after removing moisture.
Flame photometric detector (FPD)	Same as FID	
Flame thermionic detector (FTD)	Carrier gas o Heilum (most optimum) o Nitrogen (usable) Others same as FID	Use air from cylinder.
Electron capture detector (ECD)	Nitrogen gas	99.999% min.

1.2 Cautions about high pressure gas cylinders

High pressure gas cylinders are used as gas supply sources. Handle them carefully according to the cautions and guidance given by the high pressure gas cylinder supplier for preventing an accident.

(1) Place cylinders in an outdoor place of good ventilation protected from the direct rays of the sun and lead gases in by piping. This is compulsory in some countries when liquified gases are used.

(2) The temperatures of gas cylinders should be always kept below 40°C. Open flames must be kept away; at least 2m from gas cylinders.

(3) When high pressure gases are used, maintain good ventilation and check for gas leakage with soapy water, etc., before starting up the system. No smoking or use of open flames is permitted within 5m from the apparatus when flammable gases (for instance, acetylene, hydrogen and propane) or combustion supporting gases (for instance, oxygen and nitrous oxide) are used. Provide effective fire extinguishers also.

(4) Secure cylinders with a rope or the like so that they can not fall down. Liquefied gas cylinders (acetylene, propane, liquefied carbonic acid, nitrous oxide and others) must not be thrown down on their side.

(5) Use an oil-free reducing valve for the oxygen gas cylinder. Make sure that the interiors of the tubes and others coming into contact with oxygen are free from oil.

(6) Close the shutoff valve of the cylinder immediately after ceasing the use of the gas.

(7) Check the pressure gauges for function, at least every three months.

(8) We are sticking aluminum plates stating cautions about hydrogen gas handling to our products delivered to the users. If the plate is required urgently, contact us. We will send it free.

Pay attention to your national or local regulations on high pressure gases. Total amount of gases to be stored or maximum pressure of gases may be limited.

Exercise care in the following points for preventing a hazard when hydrogen gas is used.

Warning Warning in using hydrogen

(1) Close the shutoff valve of the hydrogen gas cylinder and ascertain that the gas does not leak from the valve when the system is not used.

(2) Check the hydrogen flow line from the hydrogen gas cylinder to the final outlet port through the apparatus for leakage every time hydrogen gas is used.

(3) Ventilate the room and don't use open flames unnecessarily in the room where the system is installed to prevent hydrogen gas explosion by an accident.

(4) Close the shut off valve of the hydrogen gas cylinder immediately up on completion of operating the apparatus, and switch off the power supply of the nearby apparatus before following other steps to be done at the end of operation.

(5) Install the protective blind caps to the connection port of the column and detector when the FID is not used.

2. Components of Flow Controllers

The flow controller is accommodated in a separate case from the gas chromatograph proper. The controller is available in the following types according to the internal flow line arrangement.

a. Model CFC-14PM

This type is equipped with one pressure regulator and one mass flow controller for the carrier gas flow line, two pressure regulators for the FID flow line, and one pressure gauge for each flow line.

b. Model CFC-14P

This type is equipped with two pressure regulators for the carrier gas flow lines, two pressure regulators for the FID flow lines, and one pressure gauge for each flow line.

c. Other types

Other types are outlined in Para. ***2.3.***

(cf) $1kPa = 1.02 \times 10^{-2} kgf/cm^2$

$1kgf/cm^2 = 98.1kPa$

2.1 Flow controller_model CFC-14PM, P/N 221-41806-91

The flow controller regulates the flow rate of carrier gas (mass flow control) and is most suitable for the packed column single flow line.

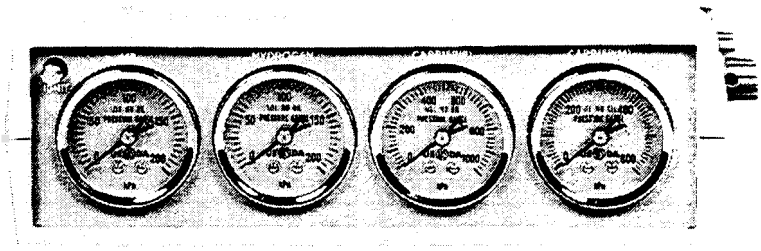


Fig. 84A CFC-14PM

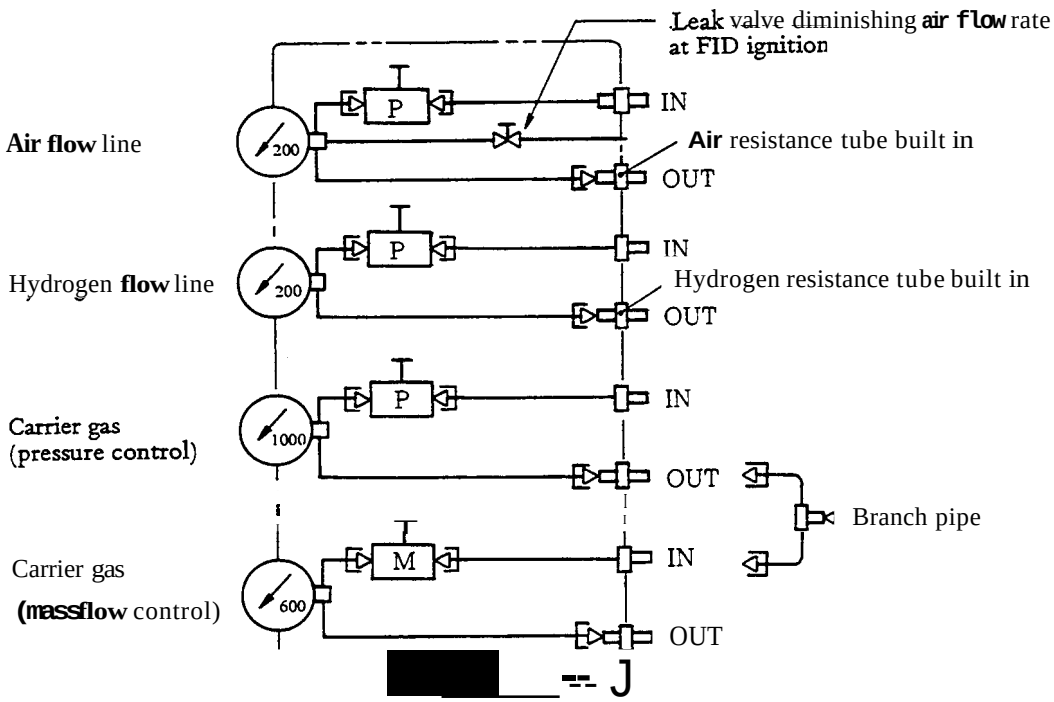
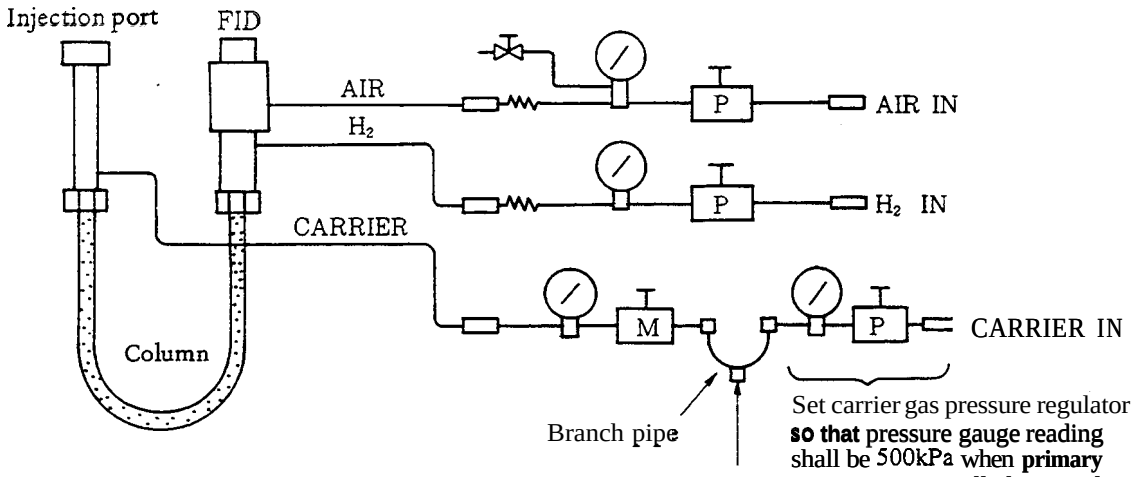


Fig. 84B Flow line diagram

Application Example 1 – For Packed Column



- Carrier gas regulated to constant pressure is acquired by branch pipe.
- Carrier gas is supplied from branch pipe to injection port evaporation chamber for regulating column inlet pressure of carrier gas to given level.

Fig. 85 Application example for a packed column

When the carrier gas flow line is regulated to a constant flow rate (mass flow control), the flow rate of carrier gas entering the detector is maintained constant and change in the sensitivity of the detector is prevented though the column resistance is changed with the rise of column temperature in temperature programmed analysis.

Carrier gas may be regulated in pressure (by the pressure regulator) when temperature programmed analysis is not made, as the column temperature is constant and the flow rate of carrier gas does not change during analysis. Pressure regulation is rather preferable when a part changing in inner volume such as a gas sample valve is connected to the upstream side of the column. Change in pressure due to valve switching is prevented and the fluctuation of the baseline by pressure change is minimized.

Application Example 2 – For Packed Column and TCD

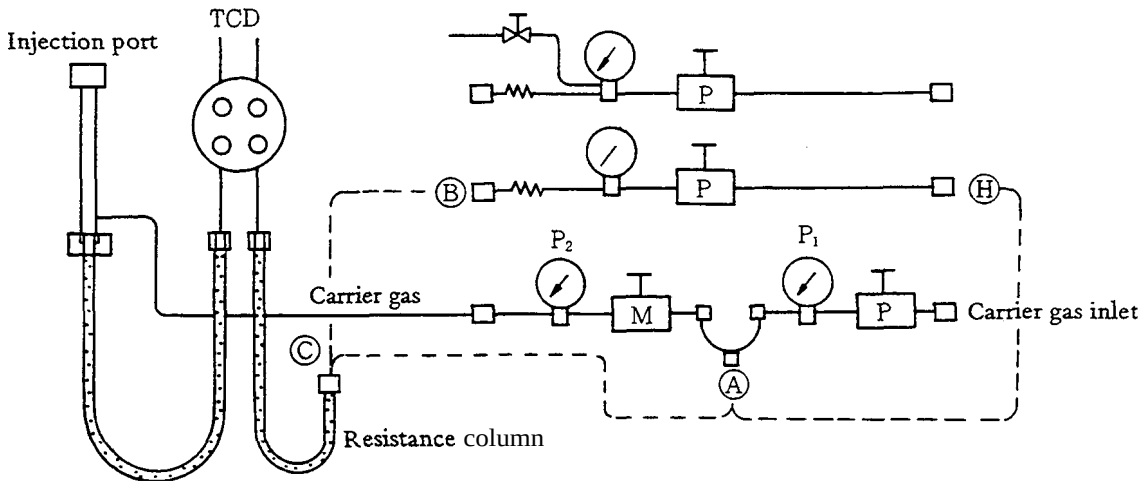


Fig. 86 Application example to packed column and TCD

The reference flow line of the TCD is arranged in one of the following methods.

1. The reference flow line is set up by connecting the resistance column to the port A of the branch pipe connected to the down-stream side of the primary pressure regulator. In this case, set pressure so that the reading of P_1 is higher by at least 50kPa than that of the column inlet port P_2.
2. The port A of the branch pipe is connected to the port H and the reference flow line is set up by connect the port B to the port C by utilizing the FID hydrogen pressure regulator.
3. The GC-14A provided with the TCD is equipped with a resistance tube (P/N 221-33431-91) as a substitute for the resistance column. The resistance tube provides a flow rate of 40mℓ/min (helium gas) at an inlet pressure of 500kPa.

Application Example 3 – For Dual Flow Lines and Dual Columns

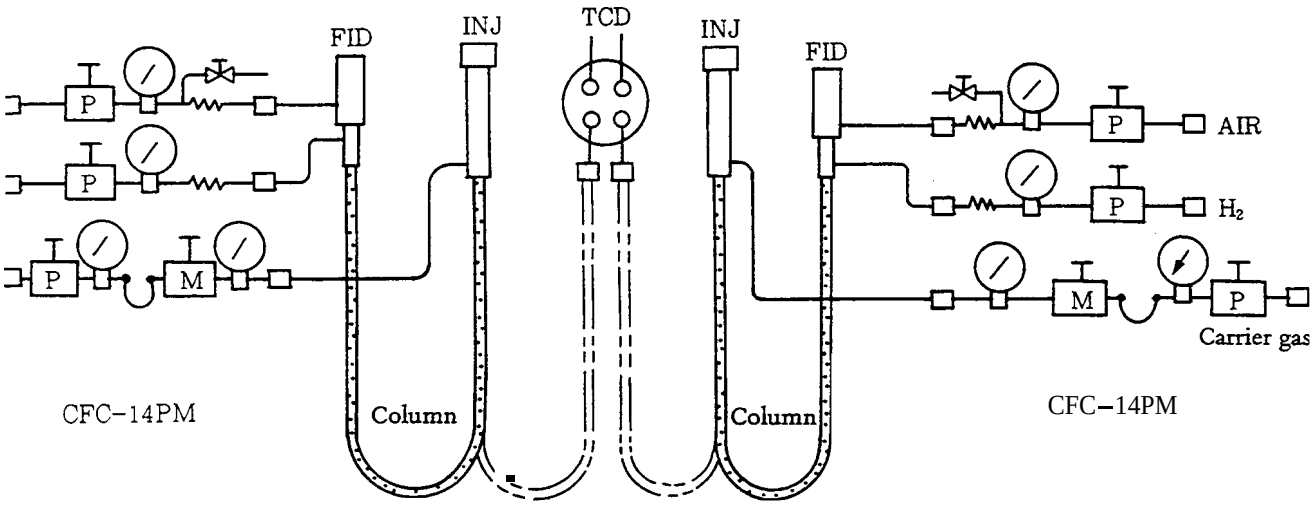


Fig. 87 Application for dual columns

In dual flow line and dual column arrangement, two model CFC-14PM flow controllers are used in parallel. Whereby a complete dual flow line and dual column system is set up.

8

2.2 Flow controller model CFC-14PP, P/N221-41805-91

This flow controller regulates the flow pressure of carrier gas constant (pressure control) and is most suitable for capillary column analysis.

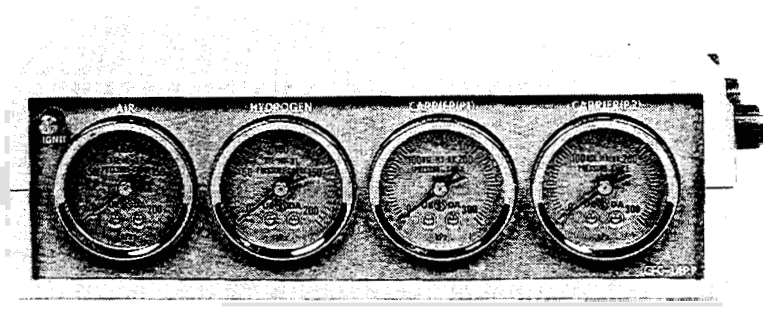


Fig. 88A CFC-14PP

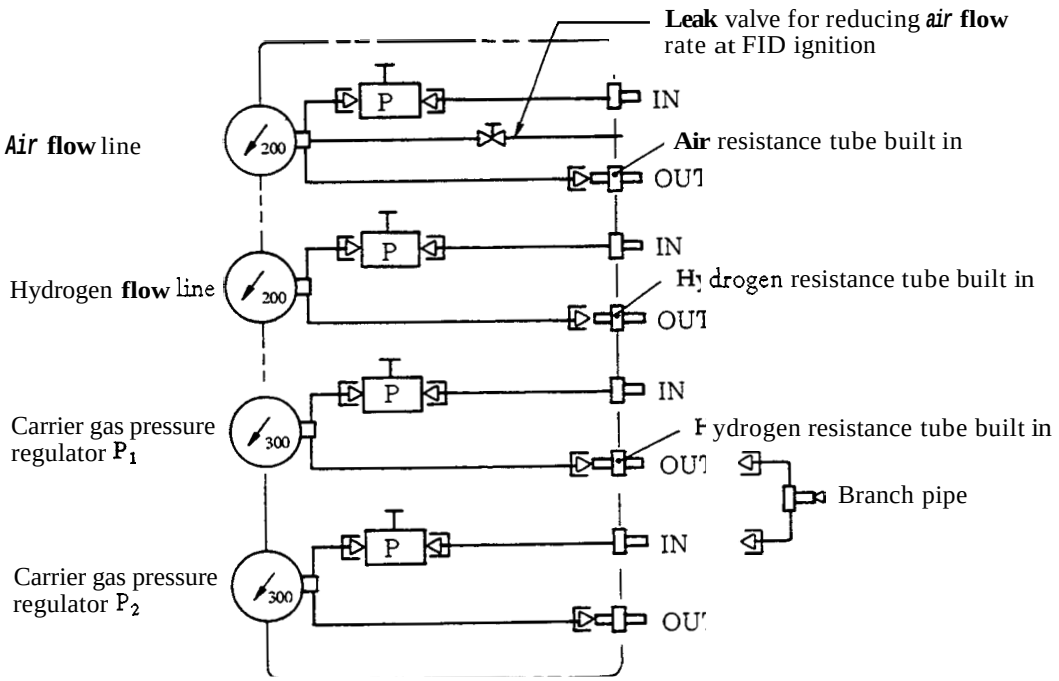


Fig. 88B Example of flow lines

Note The outlet port of the carrier gas pressure regulator P_1 is equipped with a resistance tube (indentical with that equipped in the hydrogen flow line). If gas is discharged from the outlet port via the resistance tube, a flow rate of 40ml/min is acquired for nitrogen gas at a pressure gauge reading 50kPa.

Application Example 1 – For Capillary Column

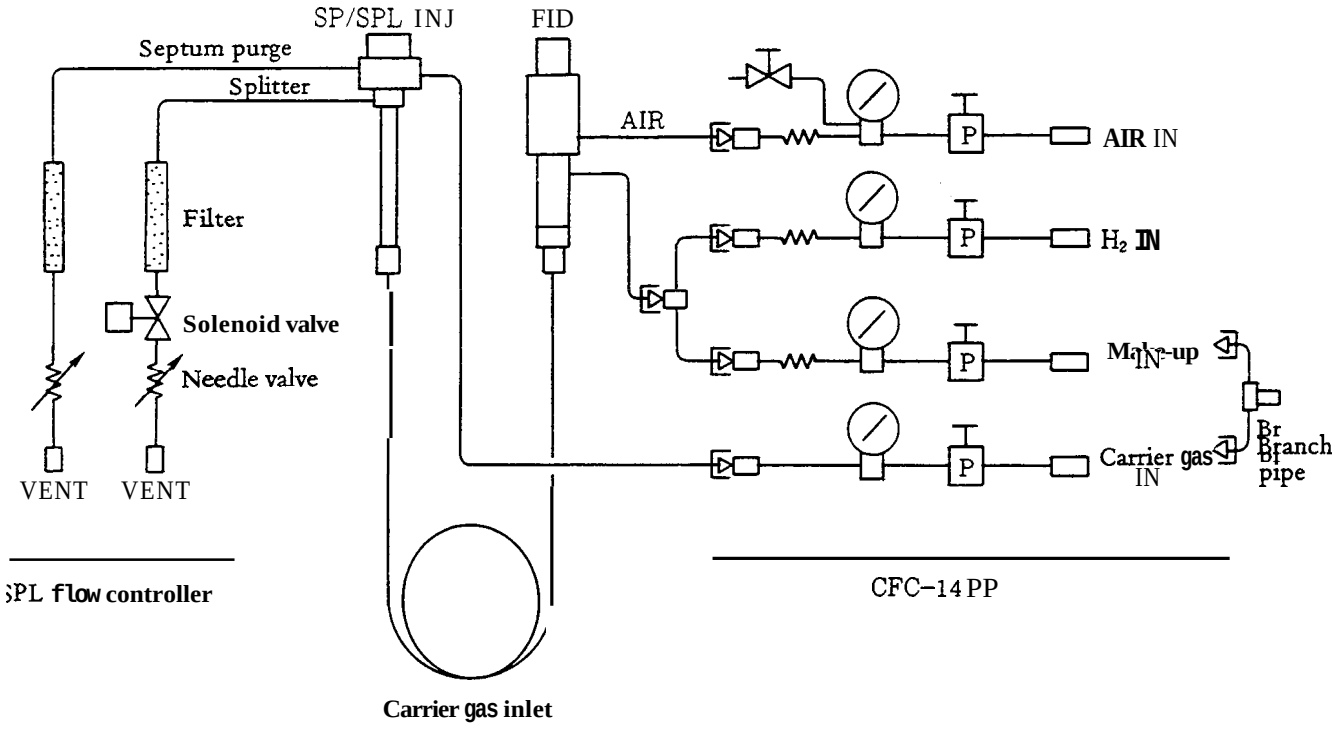


Fig. 89 Application example for capillary column

The model CFC-14PP carrier gas flow controller is equipped with a pressure regulator for carrier gas and one for make-up gas. The inlet ports are provided separately. When the same gas is used for carrier gas and make-up gas, divide the gas supply to the make-up gas inlet port and the carrier gas inlet port with the supplied branch pipe.

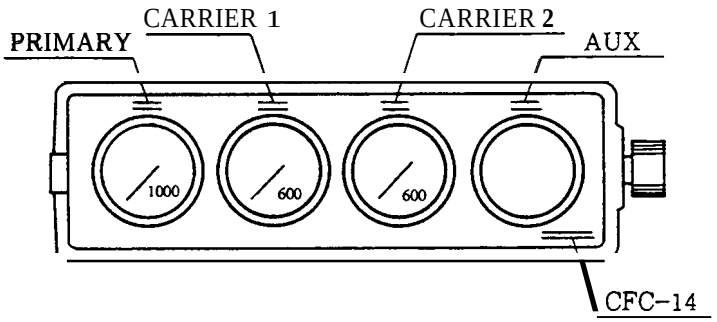
When the FID is used, the make-up gas is mixed with hydrogen of the FID cell at the outlet port of the flow controller or the outlet port of the capillary column. The effect, however, is the same. Make-up gas may be used for eliminating the dead space in the joint, and the mixing place may be changed according to the construction of the joint.

2.3 Other flow controllers

Other flow controllers of different combibations of pressure regulators and mass flow controller from those outlined in Para. 2.1 and 2.2 are available. Those controllers are options as they can be substituted for the controllers outlined in Para. 2.1 and 2.2. (The FPD flow controller model FpFC-14, however, is a standard unit for the chromatograph equipped with the FPD.)

(1) Carrier Gas Flow Controller Model CFC-14, P/N 221-32233-91

The flow controller incorporates a primary pressure regulator and two mass flow controllers. It is also provided with a space for installing one pressure regulator and one pressure gauge as standby instruments.



Flow line diagram

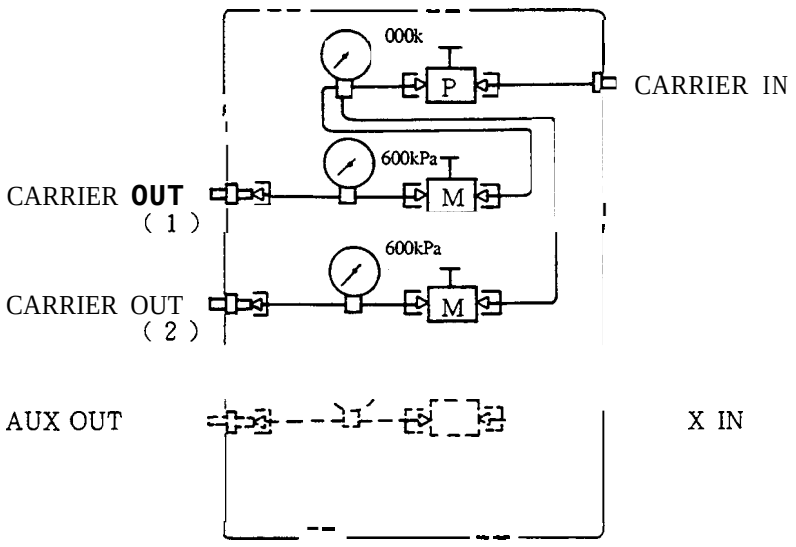
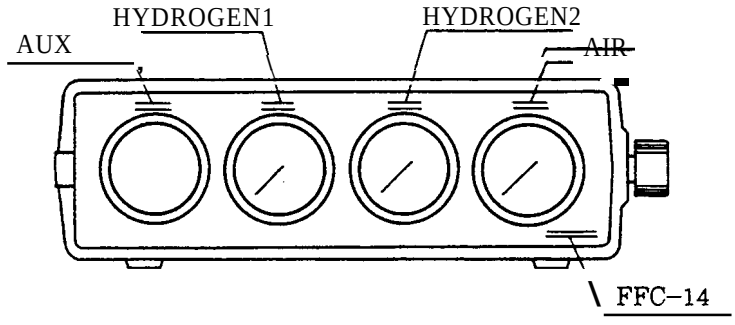


Fig. 810 FCF-14 .

(2) FID Flow Controller Model FFC-14, P/N221-32234-91

The controller regulates hydrogen and air for the FID.



Flow line diagram

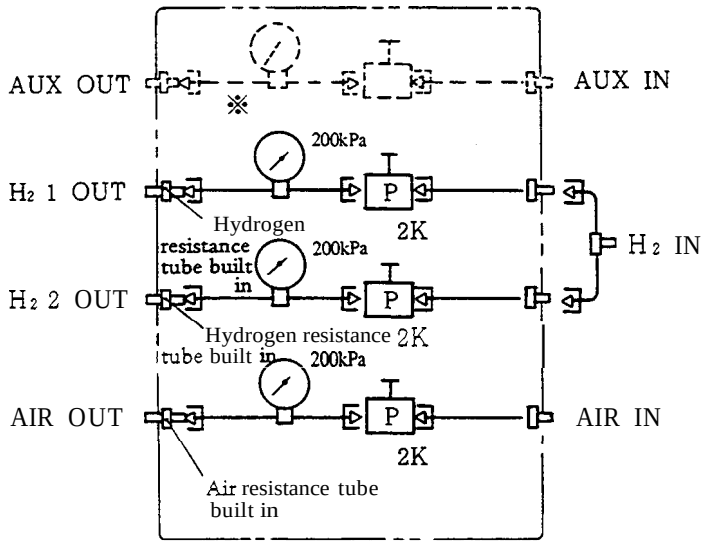
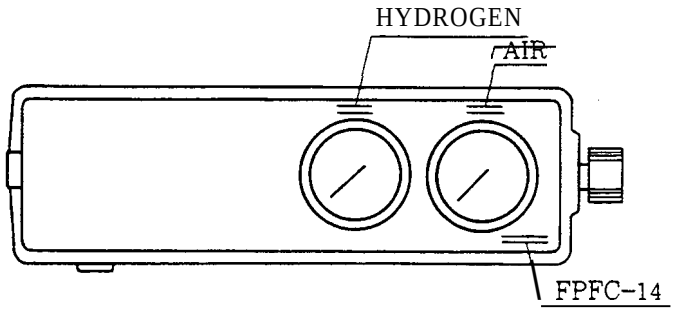


Fig. 811 FFC-14

8

(3) Flow Controller Model FpFC-14, P/N 221-32235-91

The controller is designed to regulate hydrogen and air of the FPD.



Flow line diagram

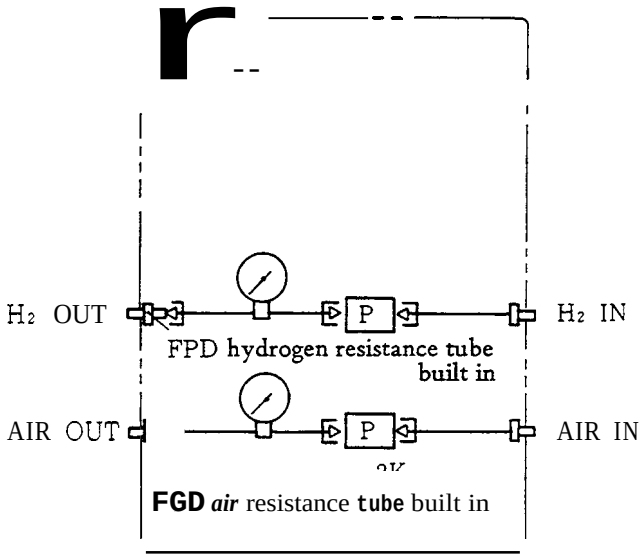
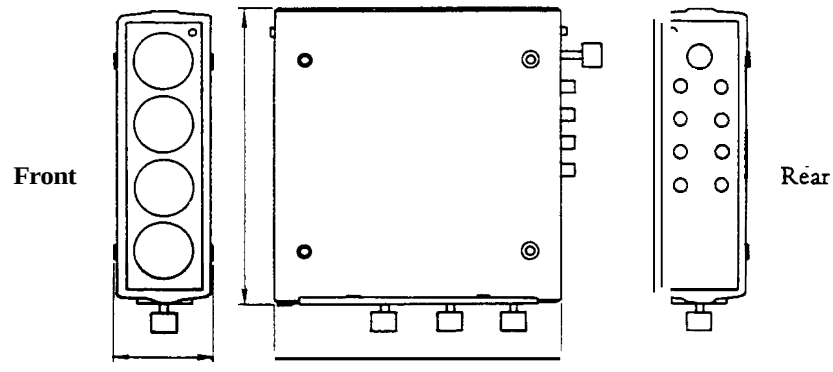


Fig. 812 FpFC-14

3. Specifications

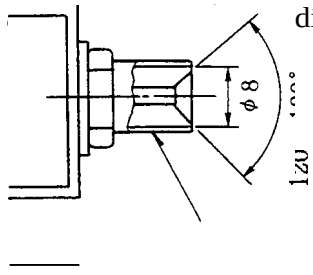
Overall dimensions



Weight: 2.6kg when four pressure regulators are built in.

Fig. 813A

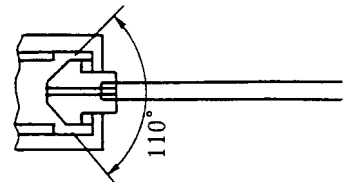
Gas inlet dimensions



M10P1

(Flow controller)

Fig. 813B



(Connection pipe)

Fig. 813C

Pressure regulators

Designation	Secondary pressure regulating range	Maximum inlet press	Accuracy	*Max.* flow rate
2K pressure regulator P/N 221-18150-91	10 ~ 200kPa	300kPa	**Less than 1% inlet pressure fluctuation**	1ℓ/min
6K pressure regulator P/N 221-18150-92	10 ~ 600kPa	800kPa	〃	〃

Mass flow controller	Inlet press	Accuracy	Max. flow rate
P/N 221- 32238 -91	500kPa	Less than 1% against 80% fluctuation of secondary resistance	200mℓ/min

4. Construction and Part List

Case

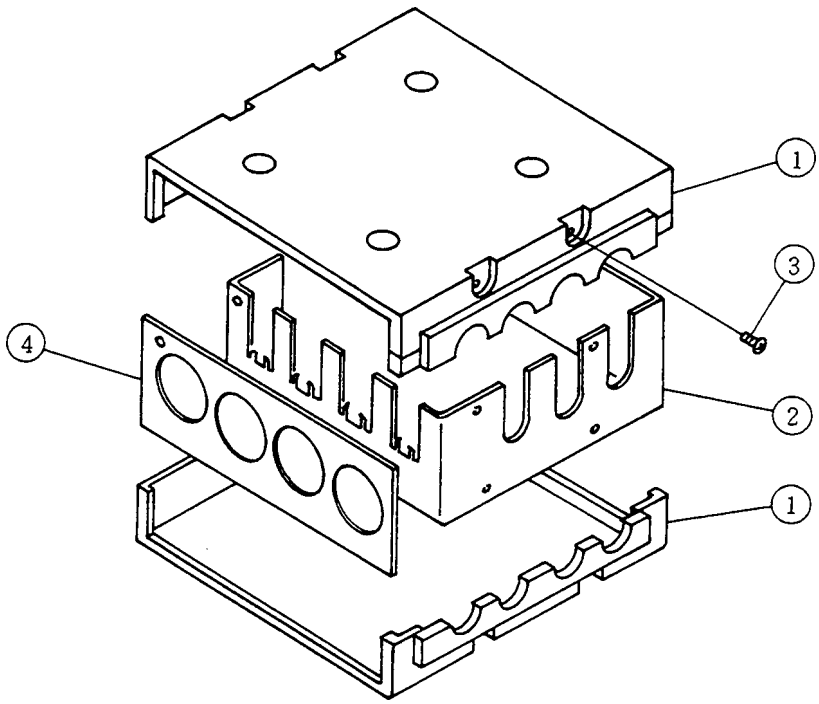


Fig. 814

8

①	221-31746	Case
②	221-31747	Chassis
③	020-37043	Screw M 3
④	221-31748-09	Panel , CFC-14PM
	〃 - 〃 -08	〃 , CFC-14 PP
	〃 - 〃 -04	〃 , CFC-14
	〃 - 〃 -05	〃 , FFC-14
	〃 - 〃 -06	〃 , FpFC -14
	〃 - 〃 -01	〃 , Plain

Pressure regulator and mass flow controller flow line **parts**

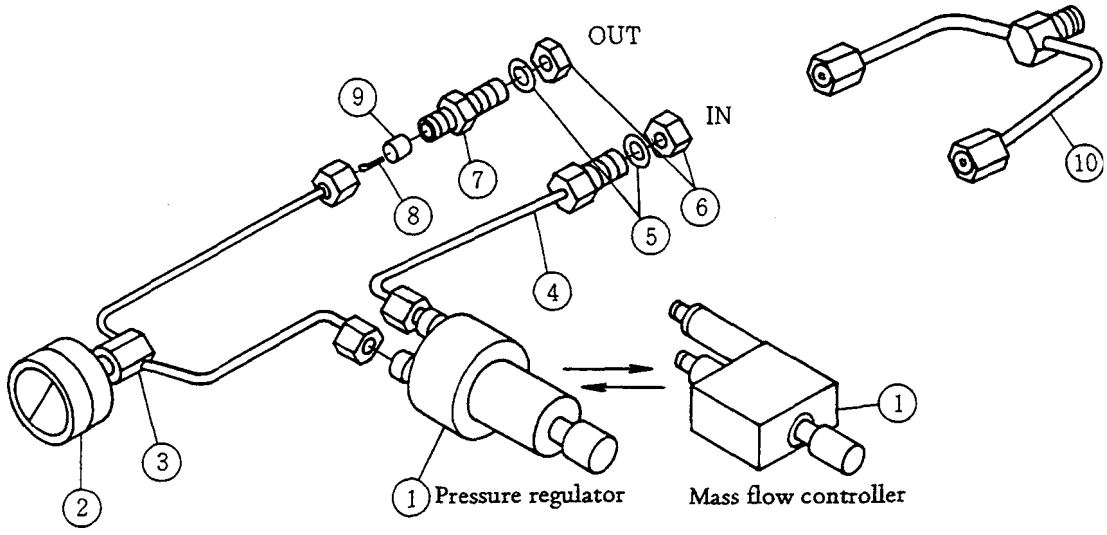


Fig. 815

Pressure regulator flow line

Ref. No.	Part Number	Part Name
1	221-18150-92	6K pressure regulator (for carrier gas)
	221-18150-91	2K 〃 (for FID hydrogen and air)
2	670-18558-14	1000kPa pressure gauge
	670-18558-13	600kPa pressure gauge
	670-18558-12	300kPa pressure gauge
	670-18558-11	200kPa pressure gauge
3	221-31739-93	Pipe PG-MF-MF
4	221-09864-18	Pipe MF-MML-17
5	201-30211-02	Washer
6	201-46833	Nut
7	221-31745	Resistance tube socket
8	221-19502-08	Resistance tube (for hydrogen)
9	201-35584 (20 pc/package)	Rubber septum (utilizing injection port septa)
10	201-48386	Branch pipe

	Part Number	Part Name
①	221-32238-91	Mass flow controller with filter

The resistance tube (9) and septum (9) are used only for the hydrogen flow line and make-up flow line.

FID and FPD **air** flow line **parts** of pressure regulator flow lines

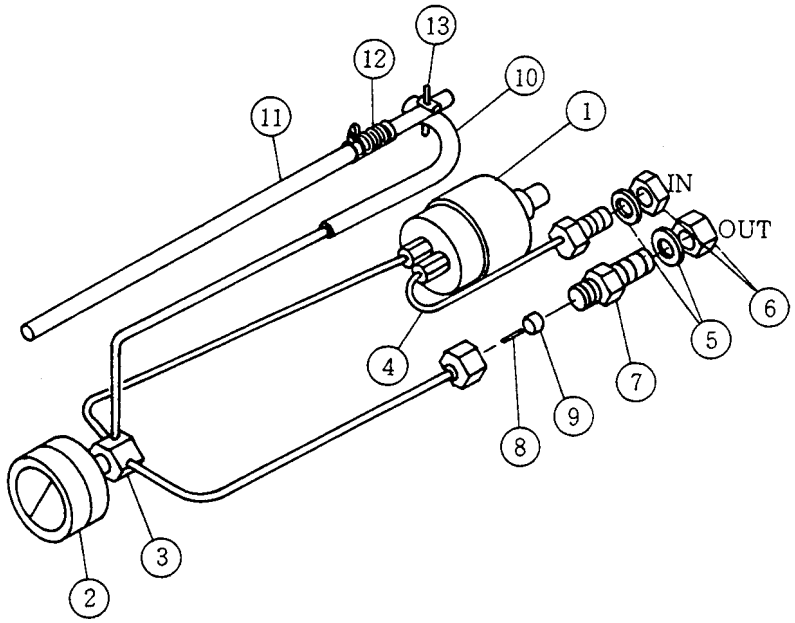


Fig. 816

Ref. No	**Part** Number	Part Name
1	221—18150—91	Pressure regulator (2K)
2	670—18558—11	**Pressure gauge 200kPa**
3	221—31738—92	Pipe PG-MF-MF-ϕ2
4	221—09864—18	Pipe MF-MML-17
5	201—30211—02	Washer
6	201—46833	Nut
7	221—31745	Resistance tube socket
8	221—19503—08	Resistance tube (for FID **air)**
	221—19502—06	Resistance tube (for FPD **air)**
9	201—35584(20pc/package	Septum (utilizing injection port septum)
10	016—31350—07	Silicon rubber tube 1 X 3 X 250mm
11	221—32848	Shaft
12	201—30248	**Spring**
13	221—32849	**Pin**

9. Capillary Column Assembly (Option)

1. Capillary Column Holder CLH-14 (P/N 221-32995-91)

1.1 Description

The CLH-14 capillary column holder allows the modification of packed column chromatographs (GC-14B, GC-15A/16A) to split/splitless capillary column configurations. The holder will accept narrow-bore, middle-bore, semiwide-bore, and wide-bore fused silica capillary columns with inner diameters of 0.1mm, 0.22mm, 0.33mm, and 0.53mm, respectively. The maximum outer diameter of the capillary column which can be accommodated by this holder is 0.8mm.

1.2 Specifications

(1)	Maximum column coil diameter:	22cm
(2)	Maximum column length:	5Om
(3)	Maximum column outer diameter:	0.8mm
(4)	Column type:	Fused silica capillary column with cage
(5)	Applicable GC:	Shimadzu Gas Chromatograph models GC-14B, GC-15A, GC-16A, GC-9A, GC-12A, and GC-7A

1.3 Structure

This holder includes splitter, split ratio adjustor, and column connection components. All components are shown and listed in Fig. 1 and Table 1, respectively.

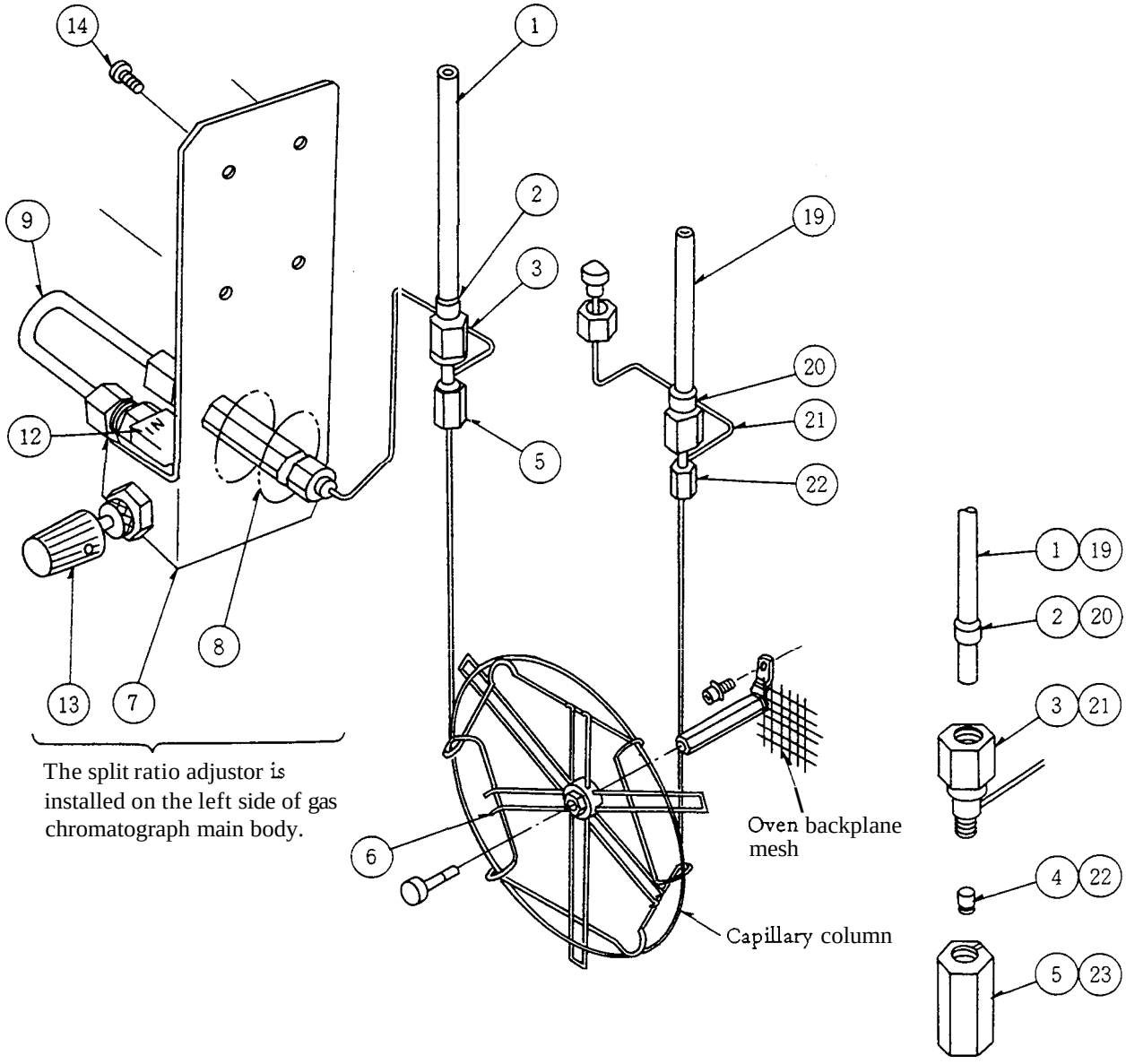


Fig. 1 Construction of CLH-14 (No. 1)

When the split ratio adjustor is installed on the left of the main body

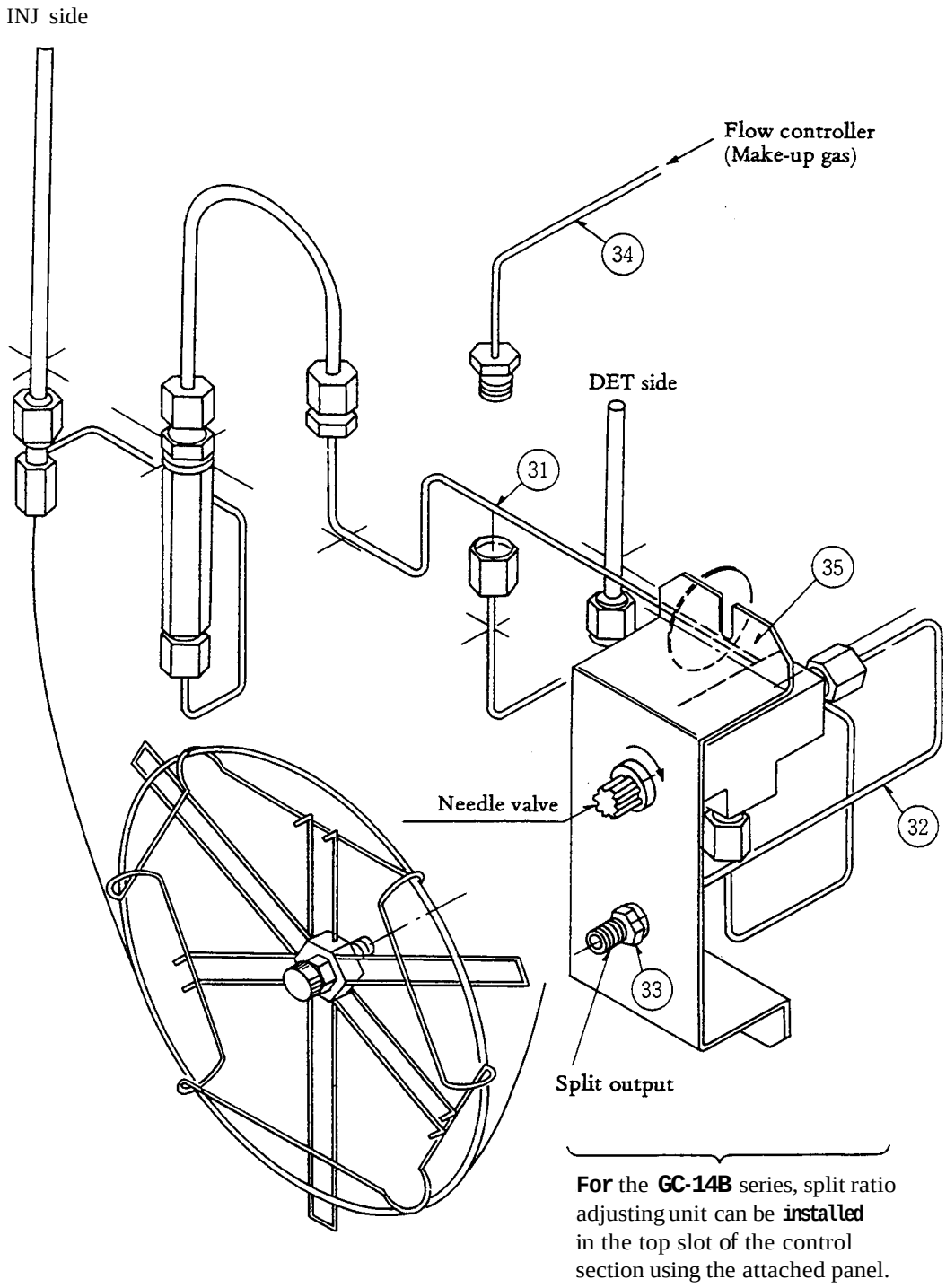


Fig. 2 Construction of CLH-14 (No. 2)

When the split ratio adjustor is installed in the top slot of the control section (**GC-14B** series only)

Table 1 Parts List for CLH-14 (221-32995-91)

	Ref. No.	Name of part	Part No.	Q'ty.	Remarks
Splitter	1	Glass insert INJ 0.8	221-32998-01	1	
	2	Graphite ferrule 5mm	221-15563-91	1	Note 1
	3	Splitter joint	221-32480-91	1	Note 2
	4	Graphite ferrule **0.5**	221-32126-05	1	Note *3*
	5	Capillary joint nut (Slotted)	221-32705	1	Note **4**
	6	Column holder	221-31567-91	1	
Split ratio adjustor	7	Needle valve holder plate	221-33001	1	
	8	Buffer	221-32997	1	
	9	MS-5A filter	221-32996-91	1	
	10	Nut	201-46833	1	
	11	Washer	201-30211-02	1	
	12	Needle valve	221-17482	1	
	13	Knob	037-02401-01	1	
	14	SUS bolt M4 X 16	020-46511	**4**	
	15	Graphite ferrule 4mm	221-23160	1	
	16	Side ring	221-21922	1	
	17	Back ring	221-23161	1	
	18	Cap nut M4	201-30008-04	1	
Detector connector	19	Glass insert **DET**	221-33000	1	
	20	Graphite ferrule 5mm	221-15563-91	1	Note 1
	21	Splitter joint	221-32480-91	1	Note 2
	22	Graphite ferrule 0.5	221-32126-05	1	Note *3*
	23	Capillary joint nut (Slotted)	221-32705	1	Note 4
Built-in components for GC-14B	31	Pipe **MF-MM** 250	201-48557-25	1	
	32	Pipe GL-MF	221-31298-91	1	
	33	Nut	201-48464	1	
	34	Pipe **MF-MM** 700	201-48557-70	1	
	35	Panel CLH-14	221-33464	1	

Note 1 **As** standard, one set of 5mm graphite ferrule (2) and 20 containing four pieces is provided.

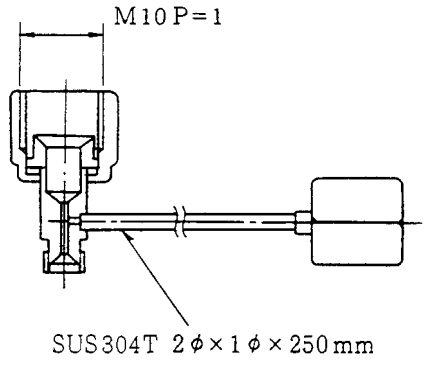
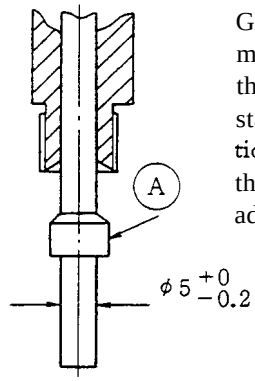
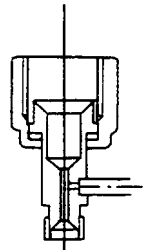
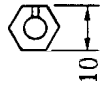
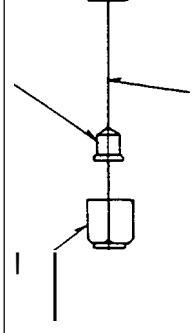
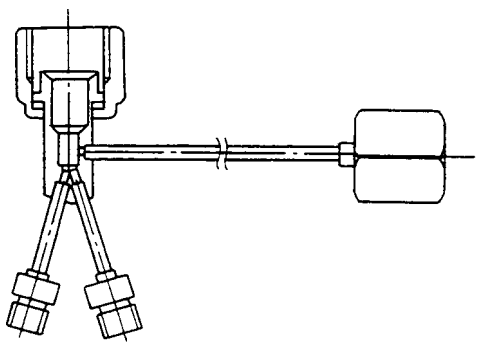
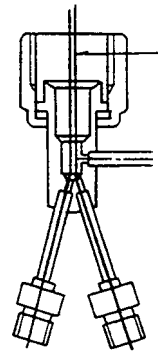
Note 2 **As** standard, **two** splitter joints (3) and 21 are provided.

Note **3** **As** standard, one set of 0.5mm graphite ferrules (4) and (22) containing 10 pieces is provided.

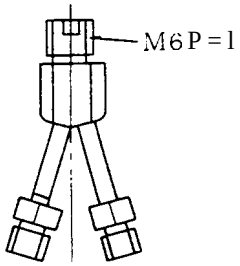
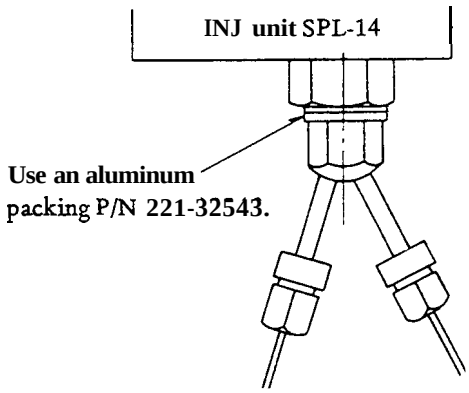
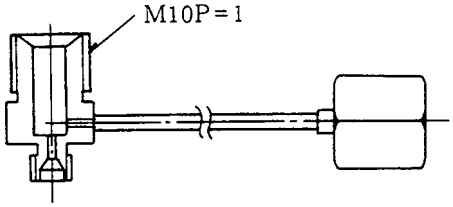
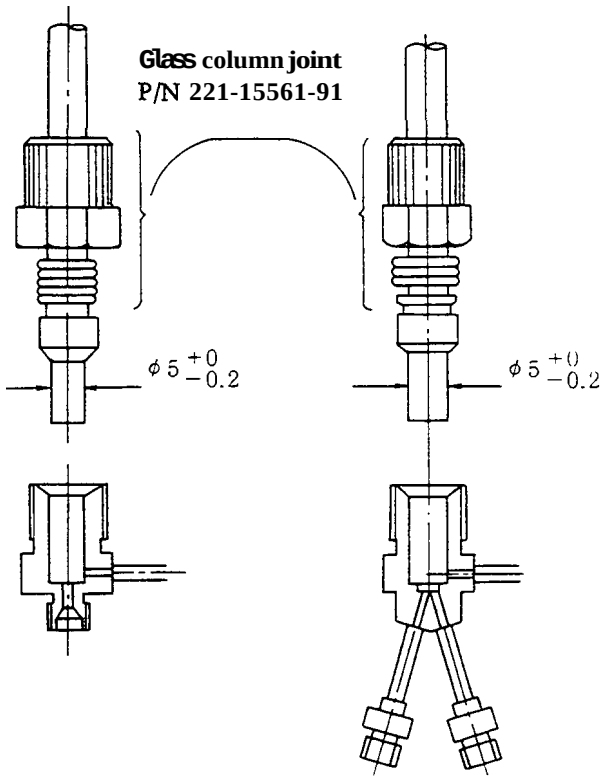
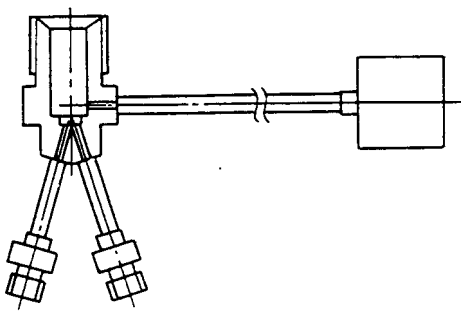
Note **4** **As** standard, **two** capillary joint nuts (5) and (23) (Slotted) are provided.

2. Components for Capillary Column Flowlines

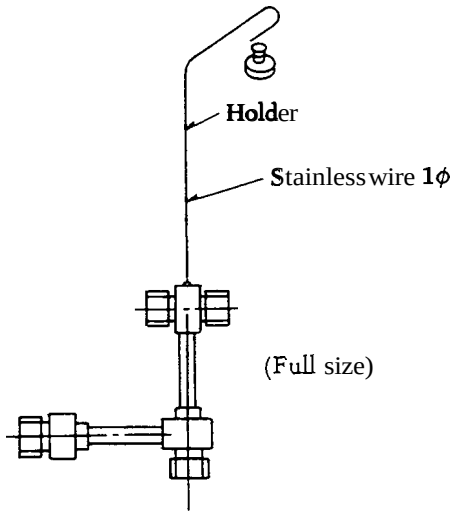
The following flowline components are used for constructing the appropriate flowline for capillary analysis.

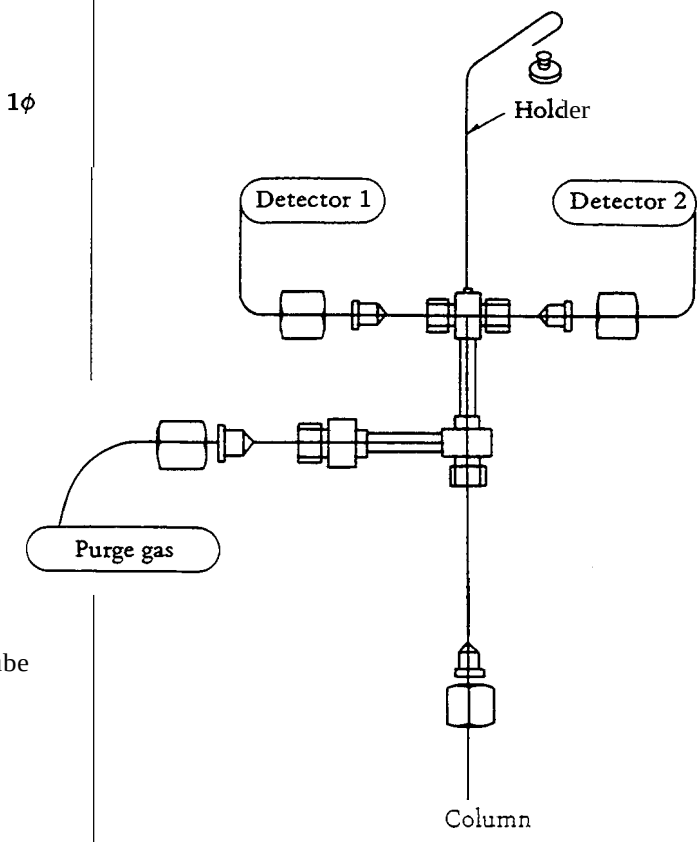
Name of part (P/N)	Application example
(1) Capillary adaptor P/N 221-32480-91 	 Glass insert (with outer diameter of 5mm) is inserted from the column oven side of the standard packed column injection port which is connected to the capillary column via this adaptor.
(2) Graphite ferrule for the capillary column (One set provided containing 10 pieces) G-0.5 P/N 221-32126-05 G-0.8 P/N 221-32126-08	 Graphite ferrule A to be attached to the glass insert should be P/N 221-15563-91 (one set provided containing four pieces).
(3) Capillary column nut Slotted nut (Large)  P/N221-32705 Non-slotted  P/N221-16325-01	
(4) Dual column adaptor P/N 221-32485-91 	**Two** capillary columns can be installed. See the installation example below. 

9

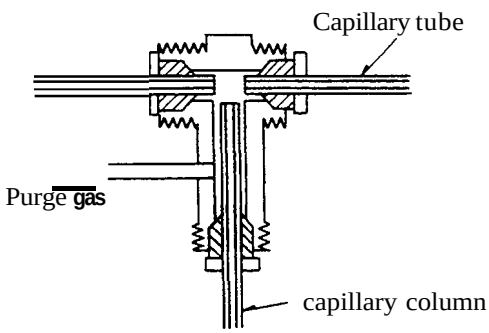
Name of part (P/N)	Application example
(5) **Dual column adaptor for SPL-14** **P/N 221-33487-91** 	**If the column joint of INJ unit P/N 221-32547-91 of the SPL-14 is replaced with this adaptor, *two* capillary columns can be installed as in (4).** 
(6) **Adaptor joint C-MM-MF** **P/N 221-32489-91** 	**This adaptor allows connection to a glass tube of 5mm outer diameter.** 
(7) **Adaptor joint C·C-MM-MF** **P/N 221-32491-91** 	

Name of part (P/N)	Application example
(8) Branch joint, detector P/N 221-32515-91	This adaptor branches the outlet of the capillary column to provide flow to two detectors. A make-up flowline is provided at the branch.

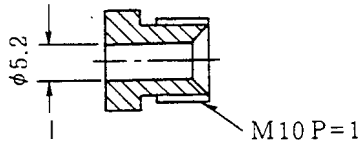
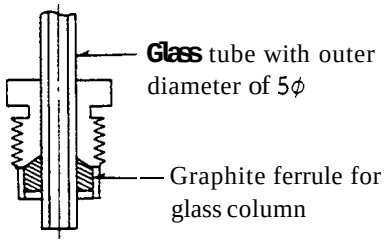
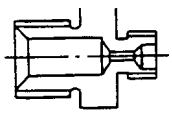
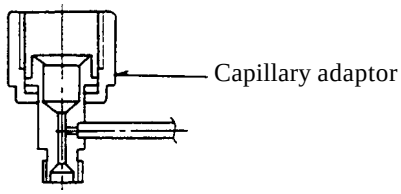
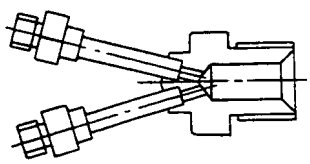
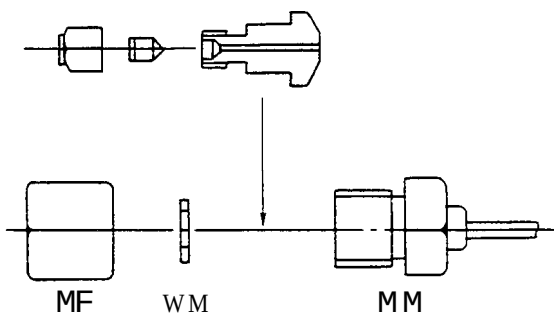
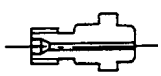
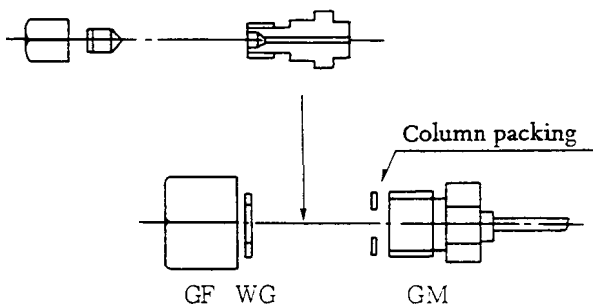




Cross section of branch



9

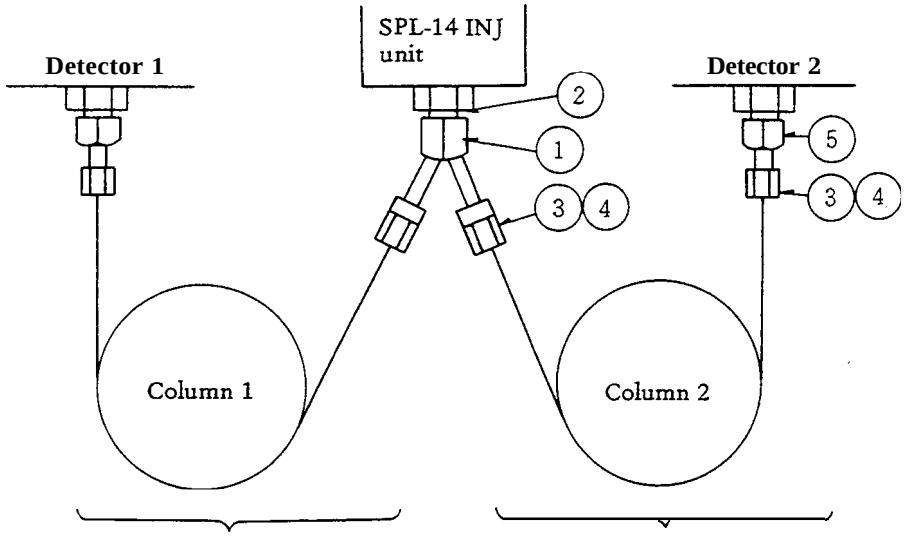
Name of part (P/N)	Application example
(9) Nut MM P/N 221-32510 	
(10) Adaptorjoint C-MM P/N 221-32490-01 	
(11) Adaptor joint C-C-MM P/N 221-32491-92 	These adaptor joints *(6)* and **(7)** without purge gas pipes are used to connect glass tube with outer diameter of 5φ and capillary column.
(12) Adaptor nipple C-MN P/N 221-32509 	This is the connection joint between pipingjoint and capillary tube. 
(13) Adaptor nipple CGN P/N 221-32508 	

9

3. Capillary Column Flowline Parts

3.1 Parallel branch column flowline parts

With SPL-14 capillary system

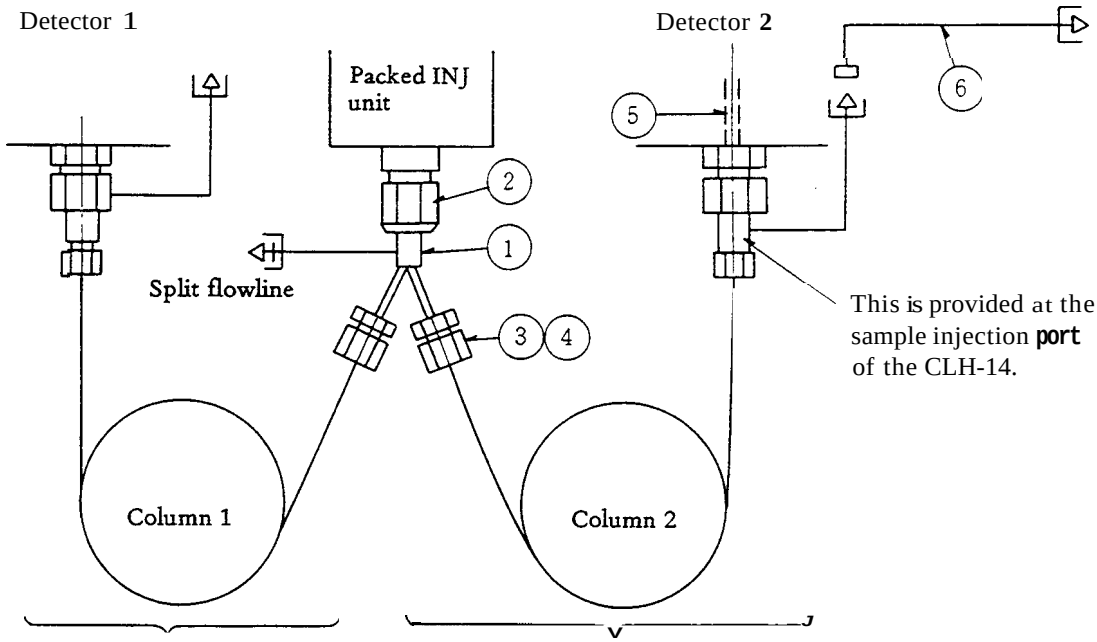


Dual column parts set (for SPL-14) P/N 221-33293-91

Ref. No.	P/N	Name of part	Q'ty.
①	221-33487-91	Dual capillary adaptor	1
②	221-32543	Aluminum gasket	1
③	221-16325-01	Nuts	4
④	221-32126-05	Graphite ferrule G0.5	1 (10 pcs.)
⑤	221-33193-91	DET-side adaptor	1
⑥	201-48386	Branch pipe make-up	1

Note **For organizing this flowline with a single flowline GC-14B, flow controller CFC-14PP (P/N 221-41805-91) or CFC-14PM (P/N 221-41806-91) is required for detector 2.**

With CLH-14 **capillary** system



Those parts shown in the following table are used for flowline 2.

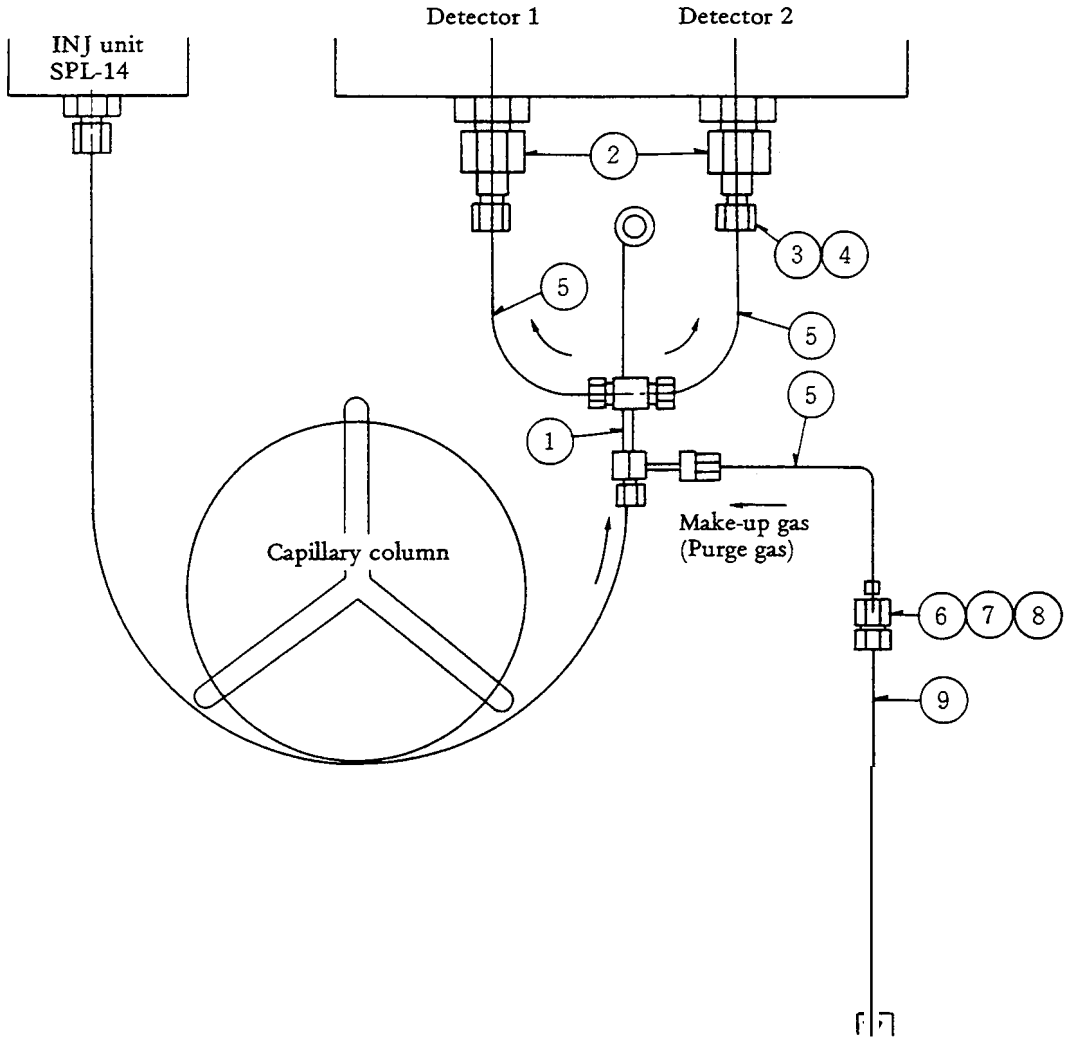
Dual column parts set (for CLH-14) P/N 221-33494-91

Ref. No.	P/N	Name of part	Q'ty.
①	221-32485-91	Dual capillary adaptor	1
②	221-15563-91	Graphite ferrules (for column)	1 (4pcs.)
③	221-16325-01	Nuts	4
④	221-32126-05	Graphite ferrules G 0.5	1 (10 pcs.)
⑤	221-33000	Glass insert for DET	1
⑥	201-48557-70	Pipe MF-MM 700	1

Note For organizing this flowline with a GC-14B with single flowline, flow controller CFC-14PP or CFC-14PM is required for detector 2.

3.2 Dual detector flowline

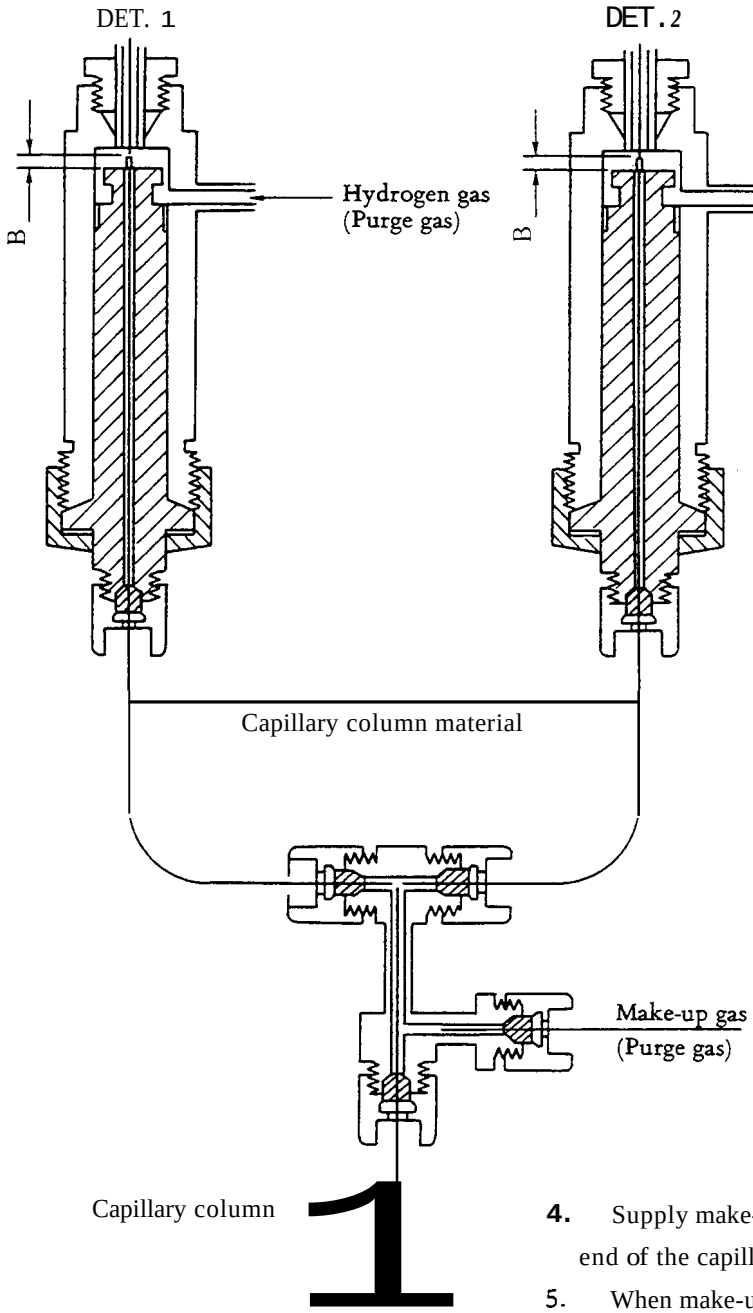
For capillary column



Detector-side branch parts set P/N 221-33495-91

Ref. No.	P/N	Name of part	Q'ty.	
①	221-32515-91	**Branch joint, detector**	1	
②	221-33193-91	**DET-side adaptors**	**2**	
③	221-16325-01	**Nuts**	**6**	
④	221-32126-05	**Graphite ferrules**	**1 (10 pcs.)**	
⑤	221-25964-02	**FQ tube**	2 m	**Cut to the appropriate length for application.**
⑥	221-32508	**Adaptor C-GN**	1	
⑦	201-30006	**Cap nut GF**	1	
⑧	201-30050	**Washer WG**	1	
⑨	201-48563-70	**Pipe GM-MF**	1	

Purge gas (make-up gas) flowline



1. Set the capillary column so that its end B can project from the adaptor by approx. 2mm.
2. Use hydrogen gas for purging the dead space for detectors using hydrogen.
3. Use purge gas flowline for ECD.

4. Supply make-up gas to the branch and purge the joint at the end of the capillary column.
5. When make-up gas in the branch is insufficient to obtain the optimum sensitivity of the detector, add make-up gas into the hydrogen-gas flowline. (Refer to page 7 - 7 in Chapter 7 of the Instruction Manual.)

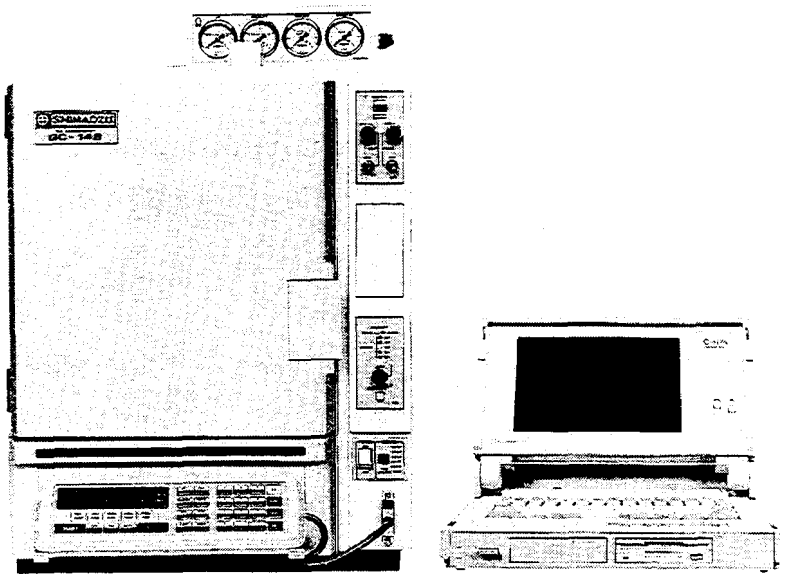
10. Outline of Optional Units

1. Current Loop

Outline

The current loop (Option P/N 221-24200-93) or optical link inerface (221-42171-91) (In this case, PC56N or PC57N is needed in chromatopac.) is designed to connect the Chromatopac (C-R2A (X), C-R3A, C-R4A, C-R5A, C-R6A or C-R7A) to the GC-14B. Whereby, the GC-14B parameter list setting, set value check and updating, measurement display, and others can be made by the Chromatopac, Other functions can be added by utilizing the BASIC functions of the Chromatopac.

The detail of GC-14B key operation is outlined in Chap. 5 "Operation", Para. 3.7.4 "List".



Operation

1. Port opening

The port should be opened in the following steps before transmitting through the current loop. Key in from the Chromatopac as stated below.

OPEN ␣ TRS ␣ 1 ↵
└ Port No.

The port No. is determined by the dip switch of the current loop PCB (printed circuit board) on the Chromatopac side. The port No. is generally 1. Another port No. may be set when two or more current loops are used.

When the port is opened, the GC-14B displays "LINK OK" and sounds the buzzer. (If the port has been opened previously, the GC-14B does not display or sound the buzzer.)

2. Link device code (LDVC)

The **link** device code comprises **two** figures having the following meanings.

LDVC = 1 1

1's position

- 1 : List is printed out by printer.
- 2 : List is displayed on CRT.

10's position

- 1 : Connected to channel **1** of the Chromatopac (start **signal**)
- 2 : Connected to channel **2** of the Chromatopac (When channel *2* is available.)
- 3 : Connected to both channels ***of*** the Chromatopac

For instance, LDVC = 11 means that the list is output by the printer and the channel 1 of the Chromatopac is started up by pressing the START key of the **GC-14B.**

2.1 Link device code setting

Example: Setting LDVC to **11**

(1) Setting from **GC-14B**

SHIFT · D	FILE / FUNC	8	ENT	Displaying LDVC
1	1	ENT		Setting **11**
SHIFT · D	- / ESCP			Ending communication with GC-14A

(2) Setting from Chromatopac

LDVC #1= 11

Port No.

On the C-R7A, port opening and link device code setting can be made on the basic setting menu. The details are given in the instruction manual of the C-R7A.

3. Check and updating of GC-14B settings and display of measurements by Chromatopac

3.1 Check of settings

(Example) Check of column initial temperature

PRINT ␣ CITP # 1 ↵

— PortNo.

Control parameter of column initial temperature

The column initial temperature setting in the current file of the GC-14A is printed out.

3.2 Updating of setting

(Example) Updating the injection port temperature setting to 200°C

INJT #1 = 200 ↵

Port No.

Control parameter of injection port temperature

The injection port temperature parameter in the current file of the GC-14A is set to 200°C

3.3 Display of measurements

(Example) Display of actual measured temperatures

PRINT ␣ DETT # 1 M ↵

Standing for monitor

Port No.

Control parameter of detector temperature

The measured detector temperature is printed out.

Refer to Chapter 4 "Operation," Sub-para. **2.3** "Control parameters."

4. Port closing

A transmission error is caused if the power supply of either the GC-14B or the Chromatopac is switched off with the current loop port opened. Close the port in the following manipulation from the Chromatopac before switching off the power supply for preventing the error.

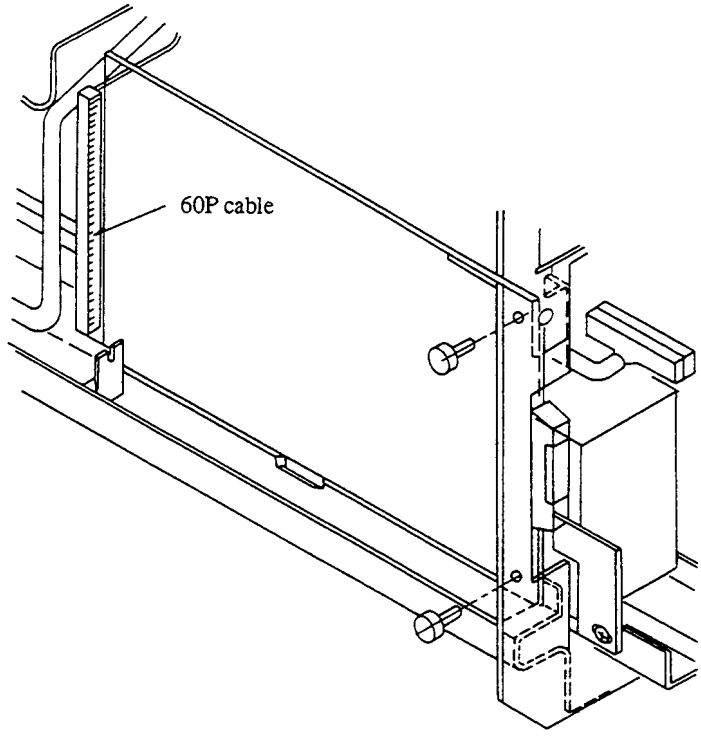
CLOSE ␣ TRS ␣ 1

Port No.

5. Installation of current loop PCB

Remove the current loop PCB mounting plate on the gas chromatograph side and fasten the PCB with two screws to the right side of the GC-14B proper as illustrated.

Connect the PCB to the GC-14B proper with the 60P cable inside the GC-14B.



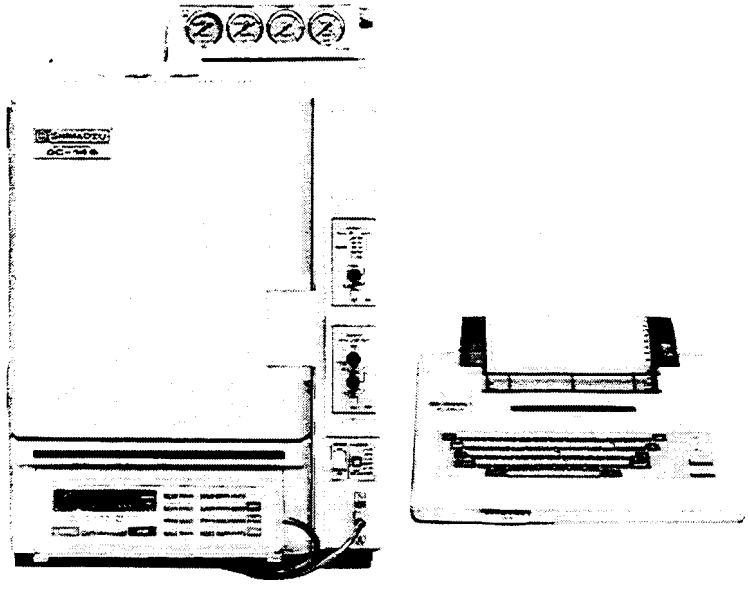
2. COS-GC14A

Outline

The COS (card operation system) model GC-14B is a system comprised of the model C-R5A Chromatopac with a current loop.

All the conditions are automatically set by just inserting the IC card. The GC parameters of the GC-14B and the analysis parameters of the C-R5A are automatically set. Manual setting is not necessary and an error in manual setting is eliminated.

The system is suitable for carrying out several types of routine analysis.



COS-GC14B system

Components required for setting up COS-GC-14B

(1)	GC-14B series gas chromatograph		
(2)	C-R5A Chromatopac		P/N 221-02135-91
(3)	Current loop for GC-14B		P/N 221-24200-93
(4)	IC card (with COS-GC program loaded)	10-card pack	P/N 221-32522-91
		1-card pack	P/N 221-32522-01

3. Control of GC-14B by Personal Computer Outline

The GC-14B series can be connected to a personal computer provided with an RS-232C interface (option P/N 221-27633-91). Whereby, it is possible to check and update the settings of the GC-14B and display measurements by the personal computer (as with the current loop outlined previously).

1. Specifications

Baud rate	110, 300, 600, 1200, and 2400 selectable
Start bit	1 fixed
Stop bit	1 or 2 selectable
Bit length	8 fixed
Parity	On-off selectable
Data type	Character **(ASCII)**

10

2. Setting of dip switches SW1 and SW2

SW1 Baud Rate Setting

	110	300	600	1200	2400
1	–	⊙	⊙	⊙	⊙
2	⊙	–	–	–	–
3	–	⊙	–	⊙	–
4	⊙	–	⊙	–	⊙
5	–	–	–	–	–
6	–	–	–	–	–

○ ····· ON
– ····· OFF

SW2 Baud Rate, Stop Bit and Parity Setting

	ON	OFF
1	1200 **or** 2400 baud	110,300 **or** 600 baud
2	**Parity** off	**Parity** on
3	Stop bit 1	Stop bit 2

3. Installation of RS-232C PCB

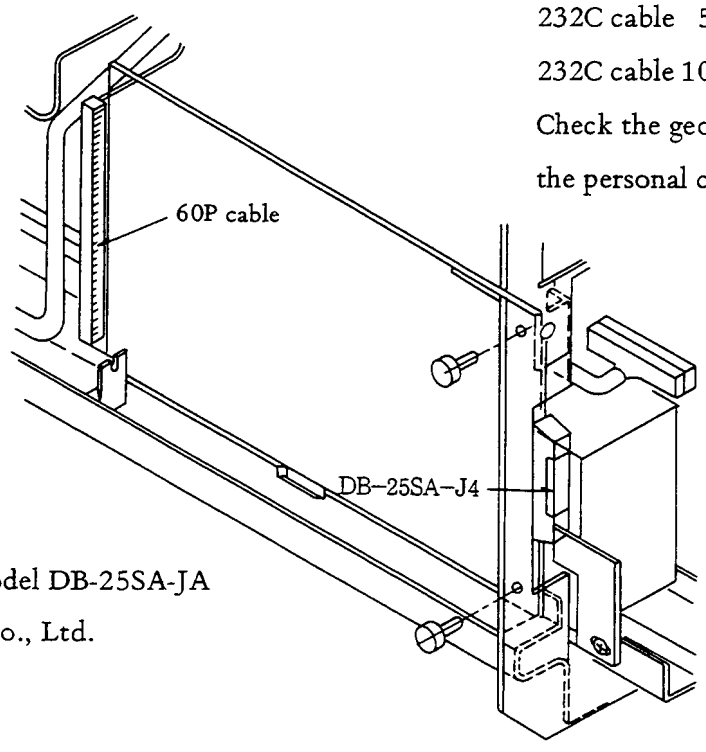
Remove the mounting plate of the RS-232C PCB and install the PCB with two screws as Illustrated. Connect the PCB to the GC-14B proper with the 60P cable (supplied with the GC-14B proper).

The 232C cable connector on the PCB is a D-sub connector 25P socket type*. Purchase a cable matching the connector. The cable is available also from us as stated below.

232C cable 5m P/N 221-33460-05

232C cable 10m P/N 221-33460-10

Check the geometry of the connector on the personal computer.



*Connector Model DB-25SA-JA
from Hirose Co., Ltd.

10

4. Operation

Refer to the instruction manual of the personal computer for the details of operating procedure! for port opening, baud rate setting, and others.

There are the following three control modes.

Parameter setting mode to GC

Parameter reading mode from GC

Measurement reading mode from GC

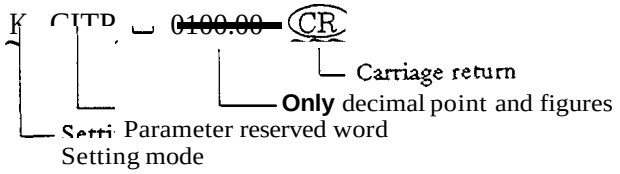
Prefix K, S, or M to the transmission command (shown in the next page) for specifying the control mode.

K: Setting mode

S: Setting request mode

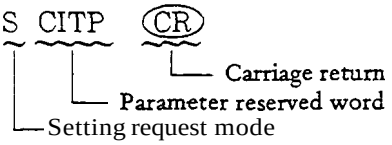
M: Measurement request mode

(Example) Setting column initial temperature to 100°C



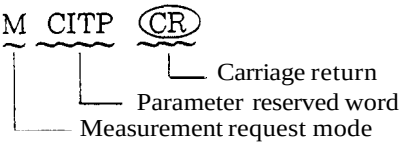
On receipt of the above message, the GC-14B answers ACK (acknowledged) or NACK (not acknowledged) in a character string. After ascertaining the message from the GC-14B, proceed to the next step.

(Example) Displaying column initial temperature setting



The GC-14A sends the data in 8 bytes and the computer receives and prints out the 8 bytes of data (INPUT$(8, #1)).

(Example) Displaying measured column temperature



The GC-14B sends data in 8 bytes. The computer receives the 8 bytes (INPUT$ (8,#1)) and prints them out.

10

5. RS-232C GC Parameter Reserved Words

CITP	STTS	CFM 4	FLW 5
AITM	DMAX	CFM 5	SNO
CITM	CMAX	APR 1	STRK
INJT	AMAX	APR 2	ISNO
DETT	RT	APR 3	FSNO
AUXT	CKSM	APR 4	REPT
AITM	STAR	APR 5	WASH
D1RG	STOP	AFP 1	WSHT
D1PL	CPR 1	AFP 2	TWTE
D1CR	CPR 2	AFP 3	PROD
D2RG	CPR 3	AFP 4	IJ1T
D2PL	CPR 4	AFP 5	IJ2T
D2CR	CPR 5	AFM 1	SLPT
D3RG	CFP 1	AFM 2	STRT
D3PL	CFP 2	AFM 3	SSTR
D3CR	CFP 3	AFM 4	SSTP
D4RG	CFP 4	AFM 5	
D4PL	CFP 5	FLW 1	
D4CR	CFM 1	FLW 2	
FILE	CFM 2	FLW 3	
TCDT	CFM 3	FLW 4	
EVNT			

Refer to Para. 4 "Operation," Sub-para. 2.3 "Control parameters" for the meanings of the reserved words.

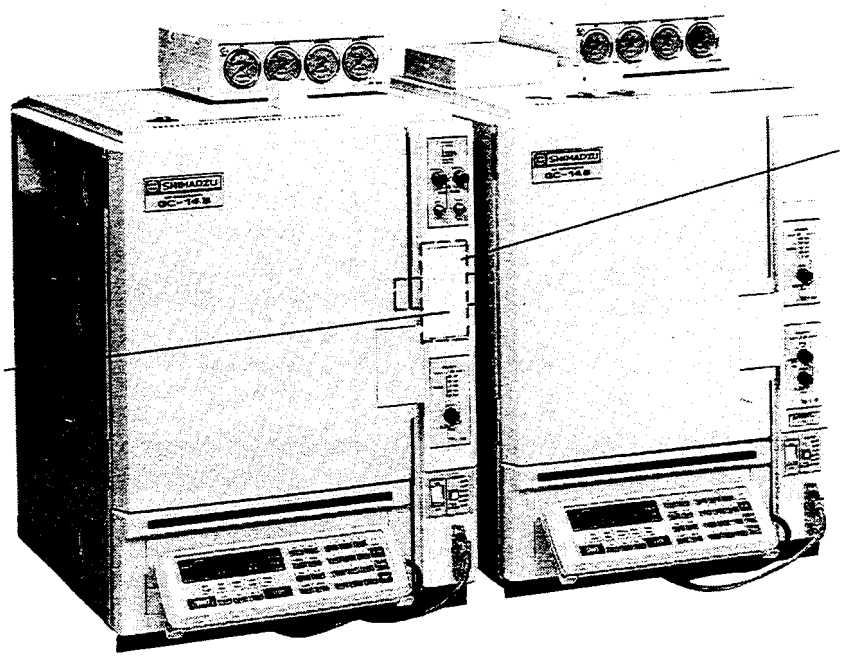
The words marked by "*", however, are not shown in the description of the control parameters.

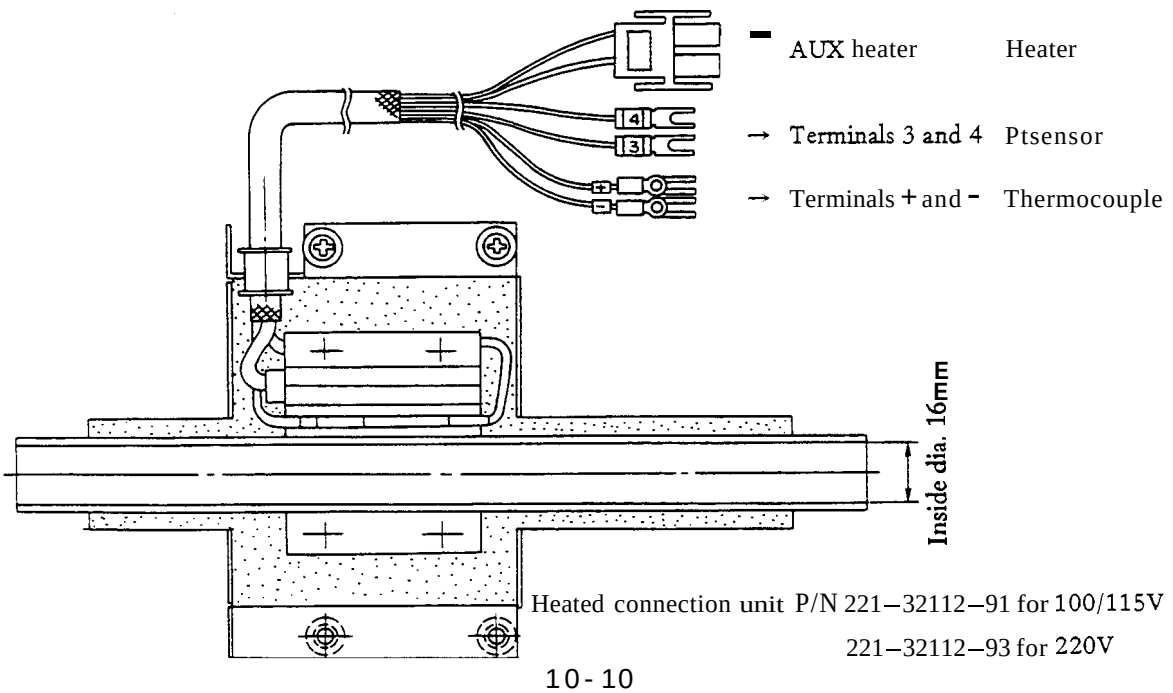
6. Example of a BASIC program for displaying measured GC-14B column, injection and detector temperatures through the RS-232C port of a PC-9800 (NEC).

```
 10  CLS
 20  LOCATE 10, 11 : PRINT " COL. TEMP "
 30  LOCATE 10, 12 : PRINT " INJ. TEMP "
 40  LOCATE 10, 13 : PRINT " DET. TEMP "
 50  OPEN "COM : N81NN " AS #1
 60  FOR 1=1 TO 3 : READ A$
 70  PRINT #1, A$ : C$ = INPUT$(8, #1)
 80  LOCATE 50, 10+I : PRINT C$ :
 90  NEXT
100  RESTORE 120
110  GOTO 60
120  DATA " MCITP ", " MINJT ", " MDETT "
```

4. Heated Connection Unit

This unit connects the column ovens of serveral gas chromatographs with heated connection lines as illustrated. The unit is connected to the second space provided for connecting the detector control unit. **As** a result, the detector control unit can not be connected to the second space when the unit is connected. The temperature of the connection line is regulated by the AUX-2 temperature control function. If the AUX-2 function is being used for another purpose, the temperature of the heating connection unit can not be controlled.





Heated connection unit P/N 221–32112–91 for 100/115V
221–32112–93 for 220V

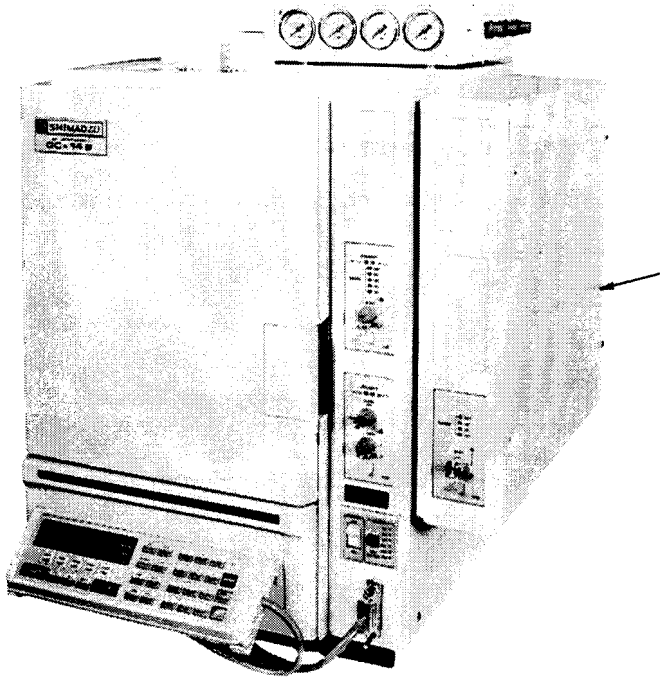
5. Add-on Unit Case

The add-on unit case is hung with a hook on the right side of the **GC-14B** as illustrated.

To add a detector control unit and **an** interface (PRG-14, AOC, etc.), the following extension bus cables are required.

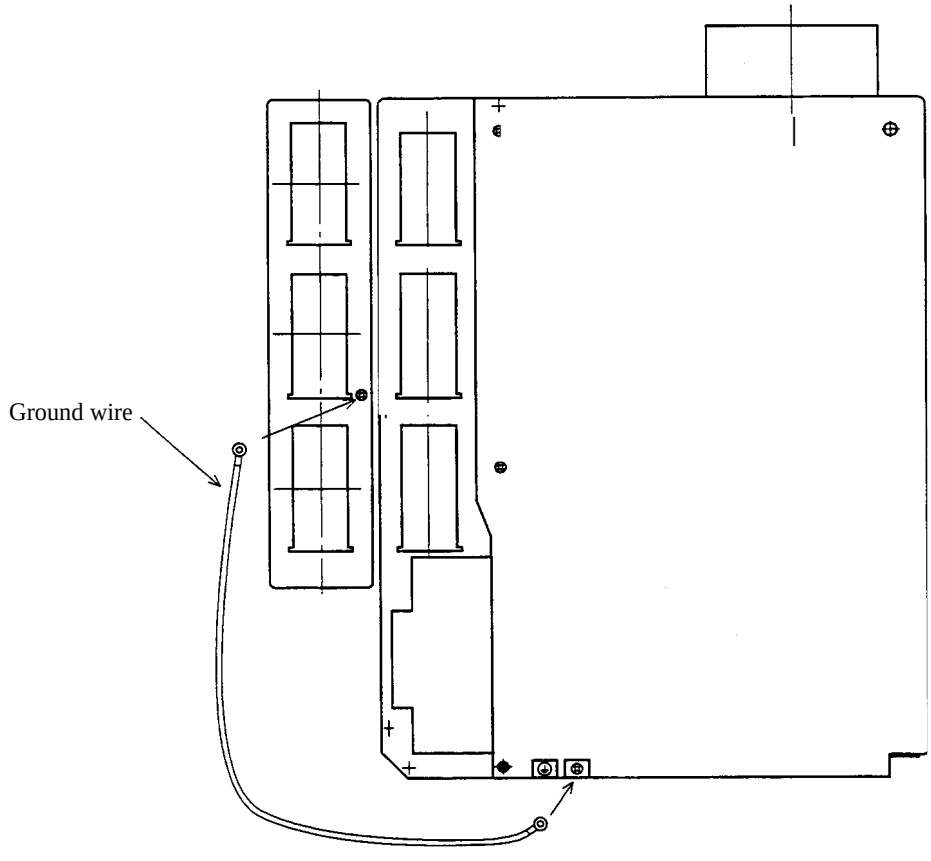
60P extension cable P/N 221-32374-91 (For adding interface)

40P extension cable P/N 221-32373-91 (For **adding** detector)



Add-on unit case, control unit
P/N 221-32364-91

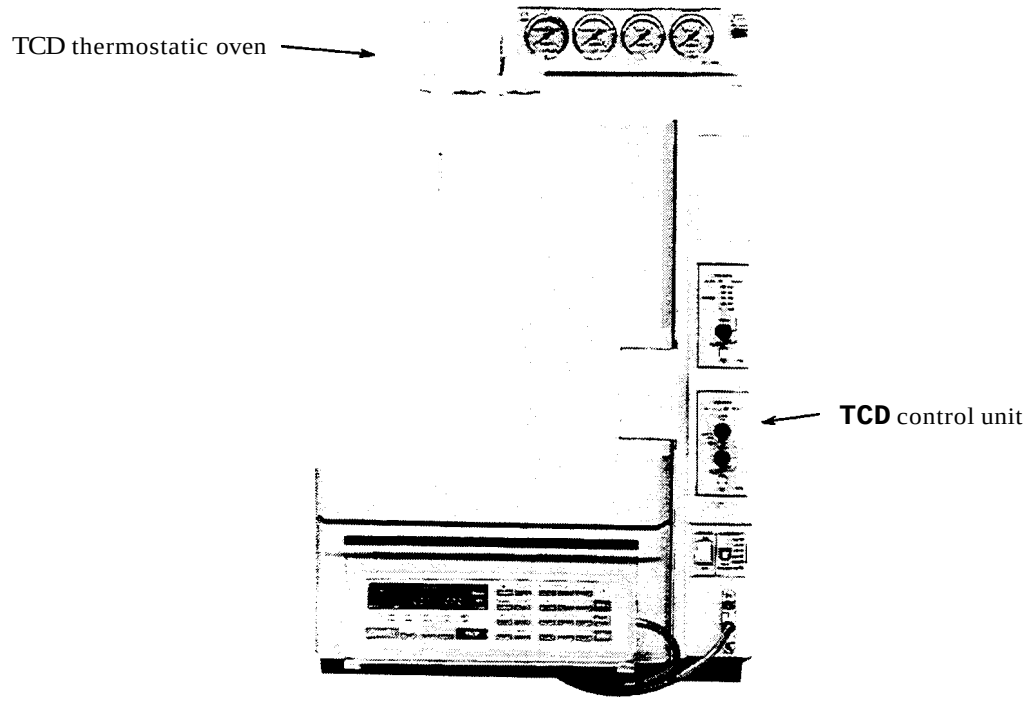
FIX the ground wire of add-on unit case to the rear panel of **GC-14B** as following figure.



11. Thermal Conductivity Detector (TCD)

This chapter mainly concerns the structure and operation of the TCD.

This detector is built into the main body and adjusted in the factory before shipment.



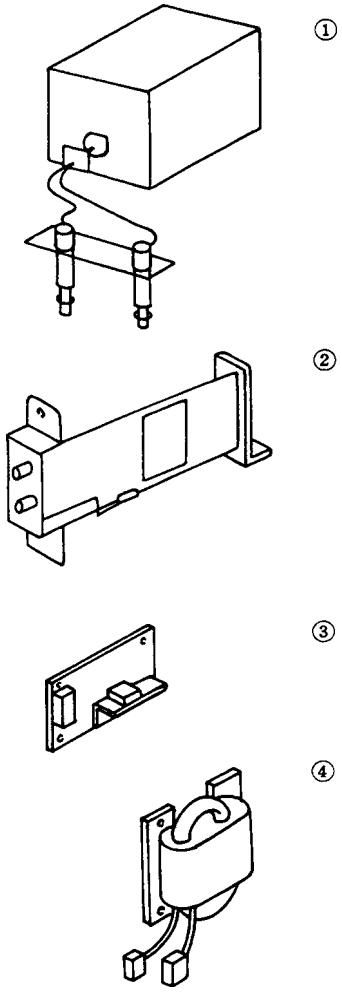
1. General Description

1.1 Principle of operation

When the metallic filaments heated with current flowing are present in a thermally conductive carrier gas such as helium, the sample components with lower thermal conductivity than the carrier gas raise the filament temperature when they pass through the carrier gas.

The chromatogram is obtained by measuring a change in the filament resistance caused by the temperature rise.

1.2 Major system components



The TCD for the **GC-14B** consists of the units shown at left.

①	TCD thermostatic oven (100～120V)	221-32743-91
	TCD thermostatic oven (220～240V)	221-32745-93
②	**TCD** control unit	221-23821-91 (115V, 220V, 240V) 221-23821-96 (230V)
③	Triac unit for thermal control	221-32896-96
④	Power transformer	221-32253-91 (115V, 220V, 240V) 221-32253-30 (230V)

Standard attachments

	P/N	Q'ty
SUS column adaptor INJ side	221−14087−91	2
SUS column adaptor **DET** side	221−10079−91	2
Graphite ferrule (X 10)	221−10076−91	1
Column **joint** parts	221−10078−91	2
Reference **flow** h e piping	201−48560−70	1

1.3 Specifications

Detector: Tungsten rhenium filaments (Approx. 10052 each at ambient temperature). 4-element filament semi-diffusion differential flow line type.

Maximum temperature: 399°C

Power source: Constant current control type

Current: 0 ~ 2-mA (In steps of 1mA)

Polarity selection: (In He carrier gas, a positive peak appears when the sample is injected to column 1 at POLARITY, INJ1(+).)

Zero adjustment: COARSE and FINE

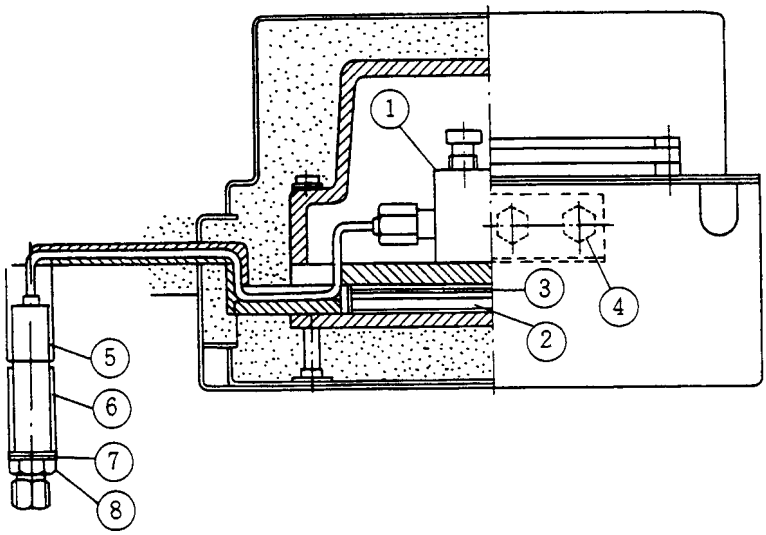
Built-in filament protection circuit

Sensitivity: Approx. 6000mV·mℓ/mg (Differs depending on the sample and operating conditions)

Note These specifications are applicable to the unit built into the main body and adjusted in the factory.

2. Structure

TCD Thermostatic Oven

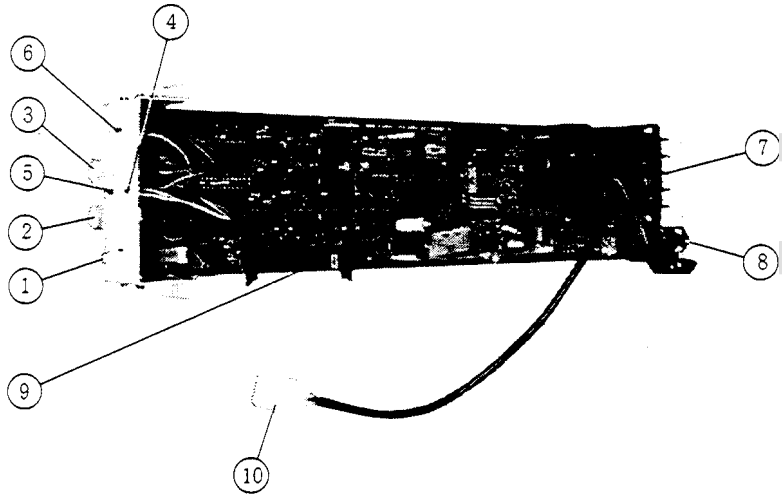


TCD cell ① is cased in the thermally controlled aluminium block. The inlet side of the TCD is connected from the lead pipe and column joint ⑤ to the column oven via the detector thermostatic oven.

The outlet side is connected to the the pipe ④ on the left side of GC main body.

①	TCD cell	201-40034	4-element filament provided
②	TCD heater 100V 220v	221-32974-91 221-32974-93	
③	Pt sensor	221-06625-93	
④	TCD vent pipe	221-13964-91	
⑤	Column joint FLOW 1 side FLOW 2 side	221-32577-91 221-32577-92	
⑥	Spacer	221-23552	
⑦	Washer	201-30211-02	
⑧	Nut	201-46833	

TCD Control Unit



① ON-OFF switch:' TCD ON-OFF switch

② Zero adjustment: **COARSE**

③ Zero adjustment: FINE

④ On-base pilot lamp: Lights when the zero point lies within approx. ±10mV of zero.

⑤ Overcurrent pilot lamp: Lights when the overcurrent protection circuit is actuated.

⑥ Polarity pilot lamp: **A** chromatogram at FLOW **1** side appears positive at INJ1(+).

⑦ Connecting terminals: **1~ 4:** TCD **signal** input

5, 6: Pt sensor for thermal control

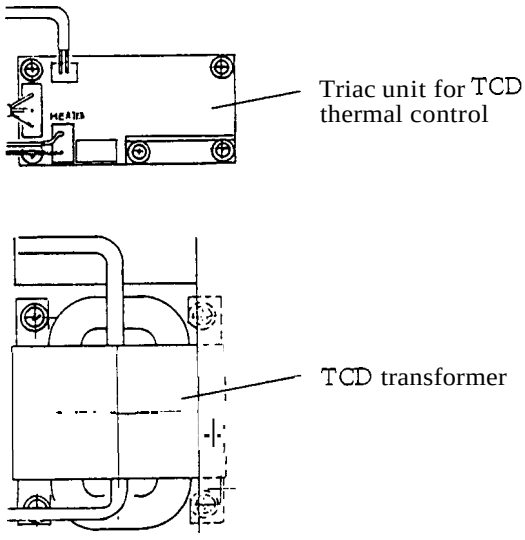
7, 8: Triac drive **signal**

⑧ Output connector

⑨ **Main** body connector **(40P** bus line)

⑩ TCD power connector

Triac Unit for Thermal Control, Power Transformer



① Triac **Assy** for thermal control: P/N 221-32896-96

TCD thermostatic oven heater power control parts

② TCD power transformer: P/N 221-32253-30 (for 230V)

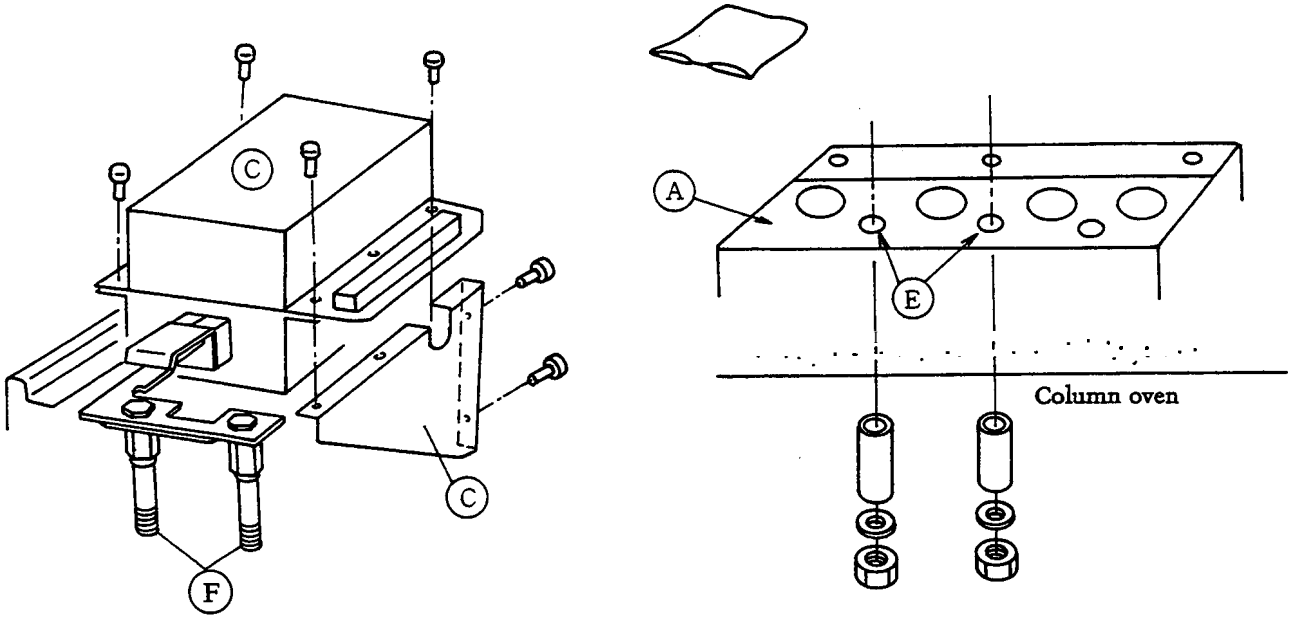
: P/N 221-32253-91 (for other power)

TCD control unit power transformer

3. Mounting the TCD in the Main Body

The TCD is built into the main body before fatctory shipment. The mounting procedures shown here will serve for maintenance.

3.1 Mounting the TCD thermostatic oven



The TCD thermostatic oven is provided at the rear of the detector thermostatic oven at the upper part of the column oven.

This detector delivers the chromatograph signal by sensing even a slight temperature difference. In mounting, the detector should be insulated with due care.

Mounting procedures

① Remove the detector thermostatic oven cover and insulating material, and expose the aluminum heating block. See (A) in the figure.

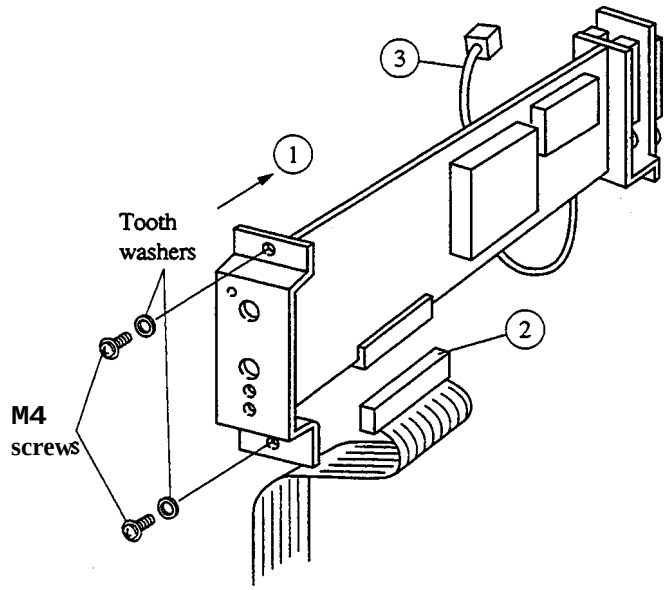
② Locate TCD thermostatic oven (C) (with the column connecting pipe supplied) at the right-hand side of the detector thermostatic oven at the upper stage of the oven case. (To make this operation easier, remove the mounting base (D) from the oven and attach it to the rear panel.)

③ Fit the column joints (F) into the detector block holes (E), and fix it with the sleeves, washers and nuts from the column oven side.

④ Bring the piping close to the detector block, and clamp the block with the pipe clamp. Then cover the block with aluminum foil (commercially available cooking foil), and charge with the insulating materials.

11

3.2 Mounting TCD control unit



The TCD control unit is contained in the control unit casing at the right of the oven. Normally it is mounted in the 1st position.

Procedures

① Remove the control unit front cover, side panel and rear cover. Insert the control unit form your side and fix it at the upper and lower parts of the front panel with M4 screws and tooth Washers.

② Connect 40P bus line.

Caution	If the power of the unit is turned ON without connecting the 40P bus line, control is disabled. An overcurrent may flow in the TCD cell.

③ Connect the lead coming from the power transformer.

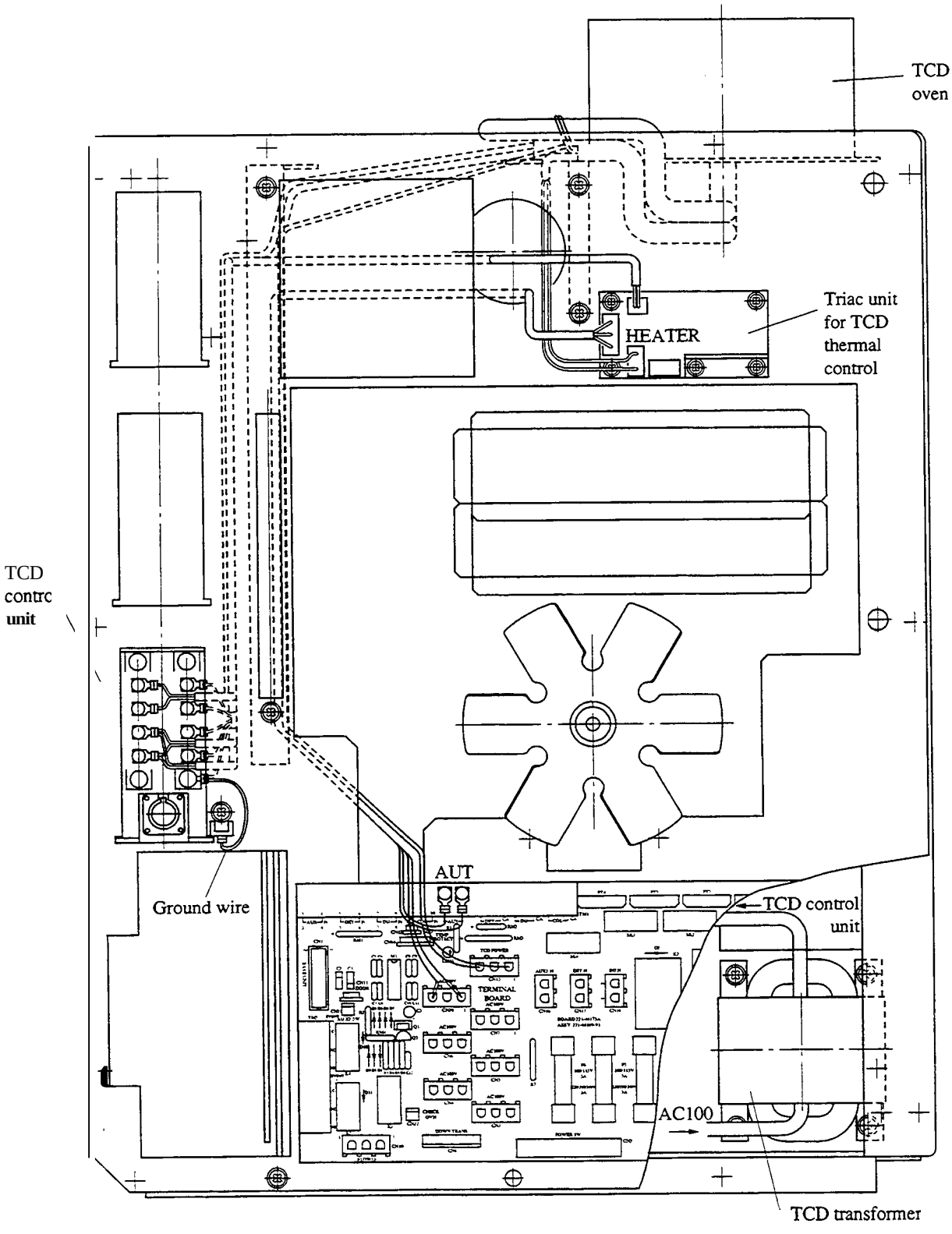
④ Attach the terminals to the rear terminal strip.

*If the unit is mounted in the 2nd position, high sensitivity recording may be interfered with by electromagnetic noise of the pulse motor at the column oven side. (Approx. 5μV of spike noise appears in the record at TCD current of 200mA).

⑤ Mount the ground wire of the control unit to the rear panel as the figure of 11-7.

3.3 Mounting TCD power transformer and triac assy for thermal control

The triac unit for TCD thermal control is mounted on the rear panel and TCD transformer on the mounting base inside the rear panel.



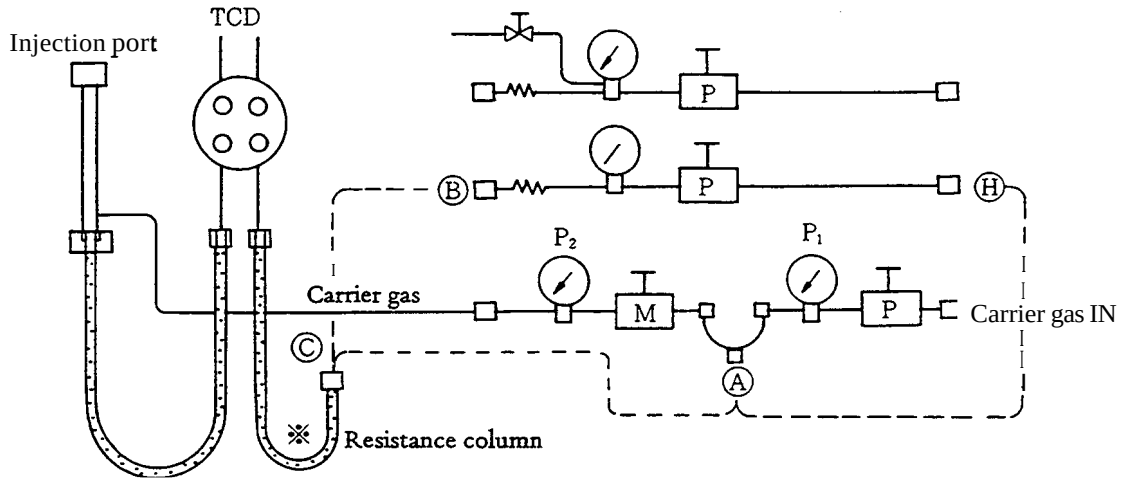
Layout of unit

4. Carrier Gas Flow Line

As the TCD is a density detector, the carrier gas flowing into the TCD should be maintained constant. Temperature programmed analysis is done with the mass flow controller line.

Schematic drawing for flow line

In Single Flow Line

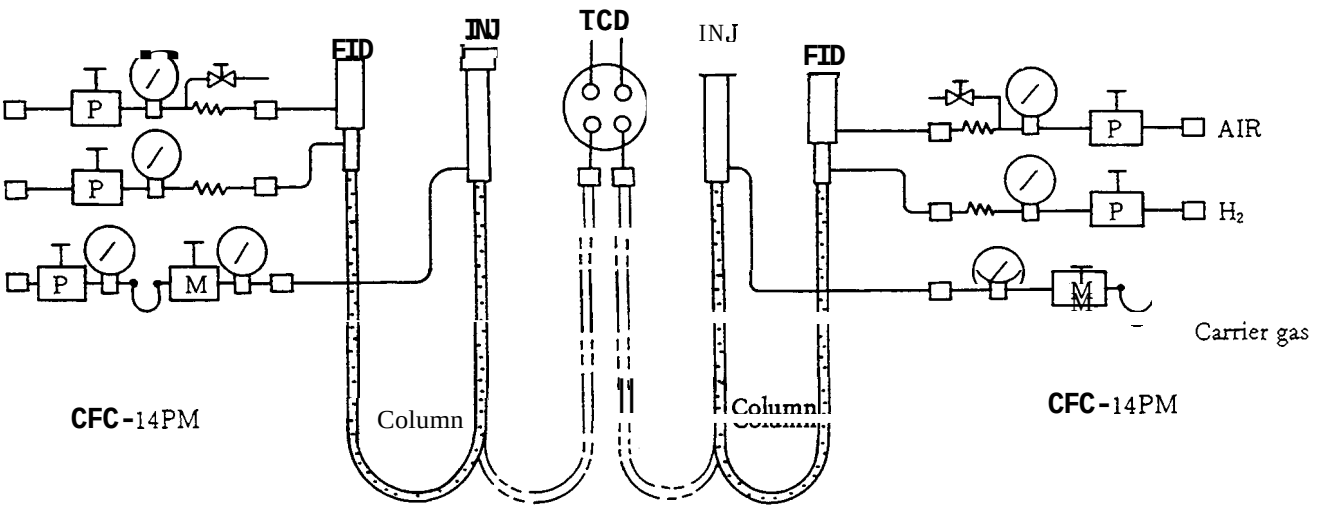


Follow either of the methods 1 or 2 to establish the TCD reference flow line.

1. Establish the reference flow line by connecting the short-circuit pipe Ⓐ behind the pressure regulator to the resistance column*. At this instance, set P_1 at least 50kPa higher than the column inlet pressure P_2.
2. Connect the short-circuit Ⓐ to Ⓗ and make the reference flow line from Ⓑ utilizing FID hydrogen pressure governor.

*A resistance tube charged with packing is available as an alternative resistance column. P/N 221-33431-91

In Dual Flow Line or Dual Column



In dual flow line or dual column, two units of the flow controller CFC-14M are used to establish the complete dual flow line or dual column system.

5. Operation

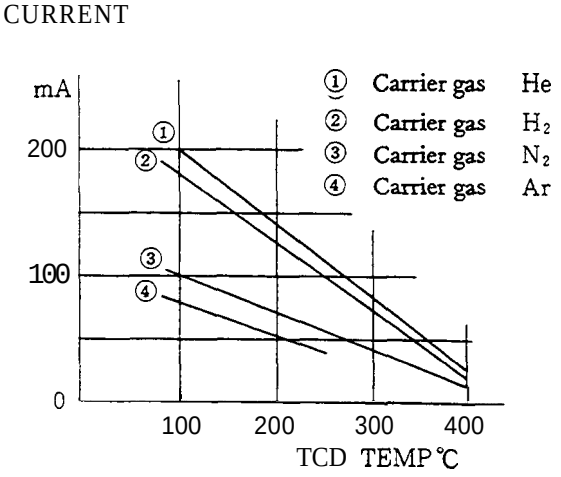
5.1 Temperature setting

To set TCD thermostatic oven temperature, hit the [S. DOWN] [TCD] keys first, enter the desired temperature, then hit the [ENT] key. As the maximum operating temperature is fixed at 399°C, the temperature cannot be set beyond this range. Be sure to set the temperature about 20 ~ 50°C higher than the column oven temperature (final temperature in temperature elevation).

As shown in the schematic drawing, the TCD analytical unit controls the sample injection port temperature and column-to-detector temperature separately. To set the sample injection port temperature, hit the [INJ] key first, enter the desired temperature, then hit the [ENT] key. To set the column-to-detector temperature, hit the [DET. T] key first, enter the desired temperature, then hit the [ENT] key.

5.2 Current setting

Current flowing in the TCD filaments is set from the keyboard. As the TCD is normally fixed at DET No. 4, operate the keys in the sequence of [DET] [4] [ENT] [CURR] [1] [0] [0] [ENT] . (At this instance, the current setting is 100mA.)



Current can be set from 0mA to 100mA in steps of 1mA. The upper limit of current supplied to the TCD filament differs, as shown in the figure at left, depending on the carrier gas type an TCD thermostatic oven temperature setting.

Sensitivity characteristics:

The detector sensitivity changes in proportion the cube of the current supply (CURRENT). If the sensitivity is insufficient, increase the current value* in the range shown in the figure,

Note that an unnecessarily large current flowing into the detector may increase the filament temperature and shorten the filament life, with resultant noise or drift.

*If the current setting is changed during analysis, the detector will be unstable for a while.

5.3 Polarity selection

Enter [1] to record the signal in the positive direction when the sample is injected into injection port 1, and enter [□] to record it in the reverse direction in the following sequences:

[DET] [4] [ENT] [POL] [1] [ENT]

[DET] [4] [ENT] [POL] [2] [ENT]

5.4 Zero adjustment

Zero the baseline by turning the two knobs indicated as ZERO on the TCD control panel.

5.5 Detector ON/OFF

When the TCD is not used, turn this switch OFF so that current does not flow into the TCD filaments.

The overcurrent pilot lamp may light when TCD detector ON/OFF switch is turned ON after the GC is energized. At this instance, be sure to hit the [CE] key, and enter the current value once again from the keyboard.

5.6 TCD filament protection circuit

When a large amount of air is included in the TCD when replacing the septum or gas cylinder, the control circuit is actuated to prevent burning the filaments. In this instance, OVER TCD is displayed and the CURRENT OVER lamp on the control unit lights. Should this occur, check the detector for proper carrier gas flow with a soap film flow meter connected to TCD VENT, and reenter the current value in the sequence of [CE] [DET] [4] [CURR] [1] [0] [0] [ENT]
If the CURRENT OVER lamp does not goes off after the key operation as above, enter a lower current setting.

Note When the TCD CURRENT OVER lamp lights:
AS current is set at 0mA automatically, be sure to reset the current value. The [CE] key turns off the TCD OVER lamp but does not contribute to current resetting.

- To set current value:
  Be sure to set the current flowing into the detector (CURRENT) within the specified range.
- To stop carrier gas:
  Be sure to set current (CURRENT) at 0mA when carrier gas is stopped temporarily for replacement of the column or septum, etc.
- To change the carrier gas type:
  Replace the gas sufficiently before supplying current (CURRENT).

6. TCD Troubleshooting

Trouble	Possible cause	Check and correction
1. Current does not flow.	a. TCD control swicth OFF.	a. Turn the switch ON.
	b. CURRENT OVER lamp lit.	b. Eliminate the cause and reset the current value.
	c. TCD filament burnt out.	c. Detach TCD lead from the terminal and check it with a tester.
	d. TCD control unit faulty.	d. Repair or replace.
2. Peak does not appear.	a. Carrier gas does not flow.	a. Check TCD VENT.
	b. Gasleakage.	b. Run the gas leak test.
	c. Meter deflected beyond the full scale.	c. Turn the zero adjustment.
	d. Sensitivity too low.	d. Check the current value and sample quantity.
	e. Sample adsorbed.	e. Conduct a repeat analysis. Check the analytical conditions.
	f. Data processor faulty.	f. Short-circuit the input signal and check for faulty operation.
3. Baseline unstable.	a. Supply voltage fluctuates.	a. Check with a tester and oscilloscope.
	b. Data processor faulty.	b. Short-circuit the input signal and check for faulty operation.
	c. Contact failure in the signal cable.	c. Vibrate the signal cable.
	d. Gas leakage.	d. Run the gas leak test.
	e. Detector contaminated.	e. Clean with solvent.
	f. Piping contaminated.	f. Replace the piping.
	g. Column contaminated.	g. Replace or age the column.
	h. Evaporator contaminated.	h. Clean with solvent.
	i. Purity of carrier gas.	i. Replace or attach the filter.
	j. Mass flow controller (or pressure regulator) faulty.	j. Replace the mass flow controller (or pressure regulator).
	k. Affected by the air-conditioner.	k. Stop the air conditioner.
	l. Flow control unit purged insufficiently.	l. Repeat purge.
	m. TCD cell defective.	m. Replace.
	n. TCD control unit defective.	n. Repair or replace.
4. Noise appears.	a. Power source noise.	a. Change the power source or attach a power filter.
	b. Data processor faulty.	b. Short-circuit the input signal and check for faulty operation.
	c. Sensitivity too high.	c. Check the current value and sample quantity.
	d. Zero-adjusting port defective.	d. Replace.
	e. Strong electro-magnetic wave.	e. Install GC-14B at another place or remove the noise source.

Trouble	Possible cause	Check and correction
	f. TCD filament contaminated. g. The unit vibrates. h. Data processor interfered with by electromagnetic waves. i. TCD cell defective.	f. Purge or replace TCD cell. g. Check the fan motor cushion. h. Keep away from a transceiver or other oscillators. i. Replace.
5. Peak becomes broad.	a. Carrier gas flow low. b. Column temperature low. c. Dead volume present. d. Column deteriorated. e. Erroneous selection of the column (type or length). f. Evaporator or detector temperature low.	a. Increase the flow. b. Increase the temperature. c. Check the column joint. d. Replace or age the column. e. Replace the column. f. Increase the temperature.
6. Peak becomes sharp.	a. Carrier gas flow high. b. Column temperature high. c. Column deteriorated. d. Erroneous selection of the column (type or length).	a. Reduce the flow. b. Reduce the temperature. c. Replace or age the column. d. Replace the column.
7. Peak tailing.	a. Glass insert damaged. b. Excessive injection amount. c. Erroneous selection of the column. d. Evaporator contaminated.	a. Replace. b. Check the current value and injection amount. c. Replace the column. d. Clean with solvent.
8. Abnormal peak appears.	a. TCD cell defective. b. The previous peak appears. c. Sample decomposed (changed). d. Glass insert contaminated. e. Injection rubber plug pierced with hole.	a. Replace TCD cell. b. Age the column c. Check the analytical conditions and sample storage method. d. Clean the insert and evaporator. e. Replace.

12. Hydrogen Flame Ionization Detector (FID)

This chapter mainly concerns the structure and operation of the FID.

This detector is built into the main body at the factory or at the installation site.

1. General Description

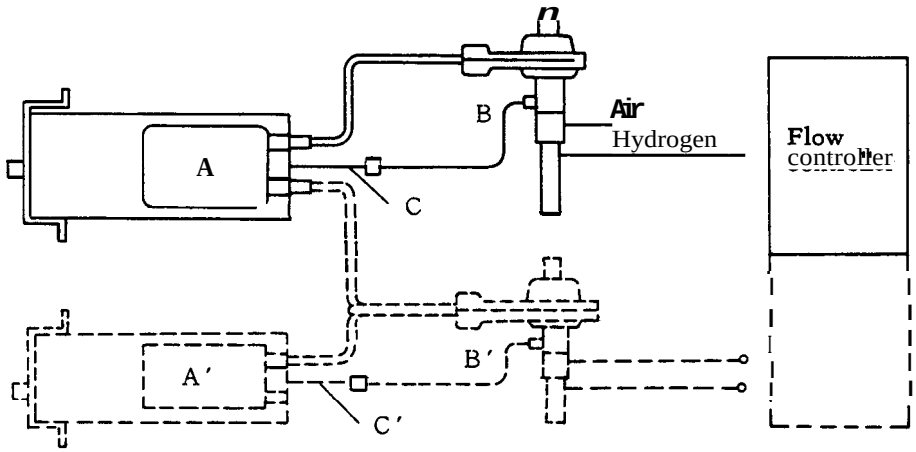
1.1 Principle of operation

When organic compounds are burnt in the hydrogen flame, ions are produced in the flame.

The electrode with DC voltage applied traps these ions whose current is measured with the electrometer.

1.2 Major system components

The FID for the GC-14B consists of the following units.



A: FID control unit (Electrometer) P/N 221-23820-96 (for 230V)
P/N 221-23820-92 (for 115V, 220V, 240V)

B: FID cell **P/N** 221-32361-91 (including mounting parts)

C: HV cord **P/N** 221-23710-94

Note FID control unit A' is required to control FID cell signals B and B' independently. To measure the difference in the signals B and B' of the FID cell, connect B and B' signal cables to FID control unit **A.**

Standard accessaries

Ignitor	P/N 670-12584	1
Battery **SUM-2**	P/N 074-70010	1
Sus column adaptor DET		1 **(2** peaces for Dual)

12

1.3 Specifications

FID cell

- Sample injection port evaporator: Glass insert type/on-column type
- FID (Hydrogen flame ionization detector)

Collector electrode:	Cylindrical type
Electrode applied voltage:	±200V
Minimum detecting amount:	3×10^{-12} g/sec (sample: diphenyl)
Dynamic range:	10^7
Maximum operating temperature:	400°C
Ignitor:	Filament externally attached, common to both flow lines
Nozzle:	Quartz

Electrometer

Range:	10^0, 10^1, 10^2, 10^3
Polarity selection:	INJ1 (+), INJ2(+)
Coarse zero adjustment:	k6.4 $\times 10^{-10}$ A
ON BASE lamp:	±10mV
Maximum sensitivity:	1×10^{-12} A/mV (full scale)
Linearity:	10^5 min
Noise:	1% or less at maximum sensitivity
Background compensation:	NORM position ±8 $\times 10^{-10}$ A
	EXT position ±80% of attenuation 1024 irrelevant of the range

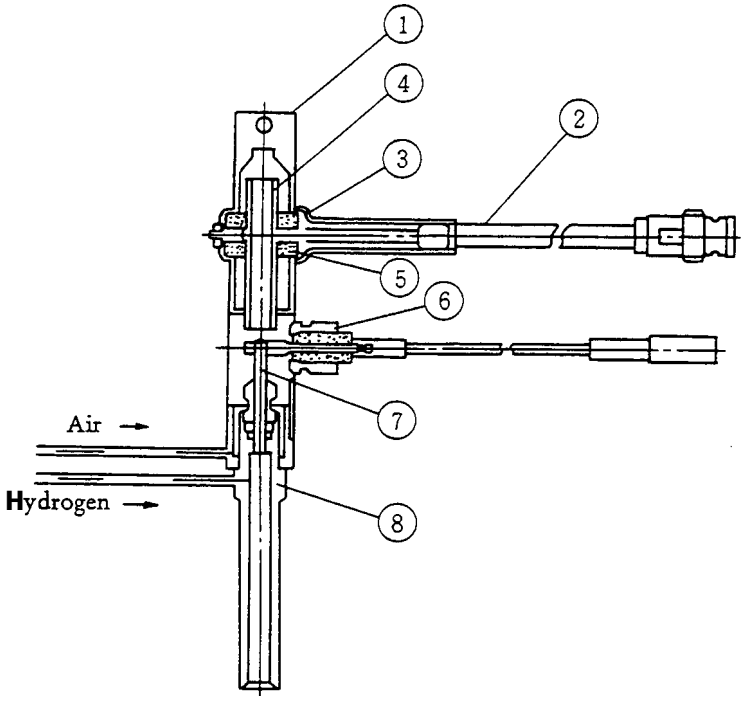
- Setting with key operation: Range, polarity selection

  Setting with electrometer: Coarse zero adjustment, ON/OFF switch
- FID ON/OFF switch: OFF mode does not allow key entries and maintains the setting before the switch was turned OFF.

12

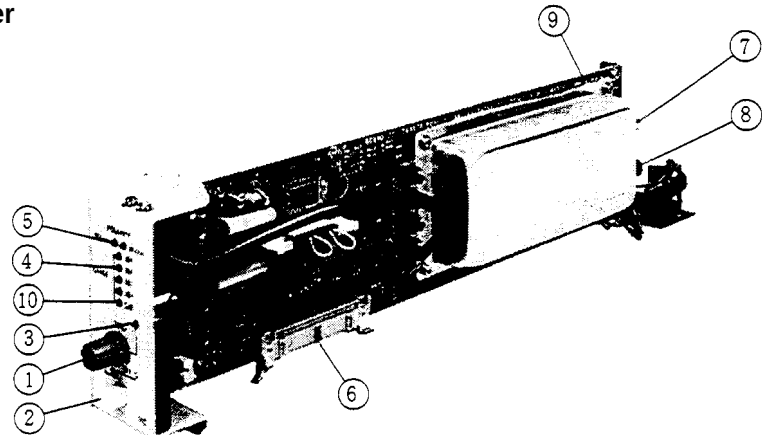
2. Structure

2.1 FID cell



No.	Designation	P/N
①	Cap	221-22830
②	signal cable	221-21912-92
③	Electrode (including ②, ④ and ⑤)	221-21906-94
④	Collector	221-21911
⑤	Insulator	221-21910
⑥	HV power supply	221-21925-92
⑦	Nozzle	221-21920-91
⑧	Nozzle base	221-21917-94

2.2 Electrometer



① Zero adjuster: $\pm 6.4 \times 10^{-10}$ A

② ON-OFF switch: OFF does not allow key entry.

③ On base pilot lamp: Lights within ±10mV

④ Range pilot lamp: 10^0, 10^1, 10^2, 10^3

12

⑤ Polarity pilot lamp: INJ(+) ..... Positive peak appears when the sample is injected to 1.

INJ(-) ..... Positive peak appears when the sample is injected to 2.

⑥ **40P** bus cord connector: Control signal

⑦ Signal inlet: Connected to FID cell collector

⑧ HV output: Connected to FID voltage electrode via FID and HV cord

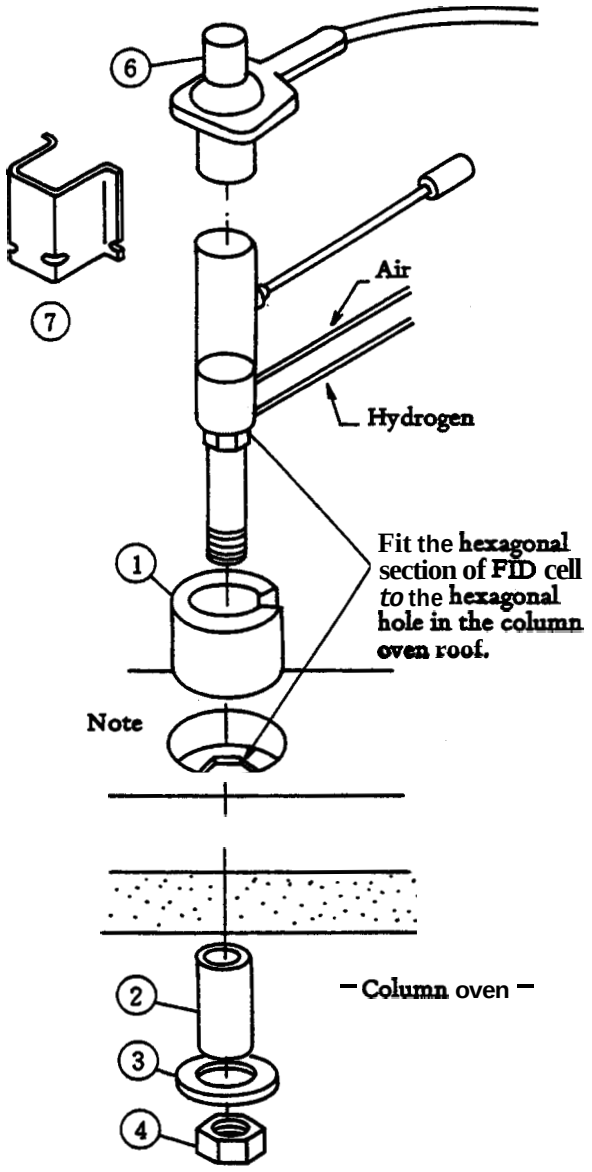
⑨ Backing mode selector switch: ↑ NORM ↓ EXT

⑩ SAT LAMP: Light more than 1V output

	Parts Designation	Parts No.	Remarks
VR	7276M 100K	054-22850	For zero adjustment
SW2	KSD1-2-0-LL-DC	064-50641-13	For ON-OFF SW
LED	LED GL3PR2	061-78037-07	For pilot lamp (red)
	LED 3HY2	061-78039-02	For pilot lamp (yellow)
SW1	SW slide	064-16035-02	For backing mode selection
K	Reed relay	065-63426-02	For range selection

3. Mounting FID in Main Body

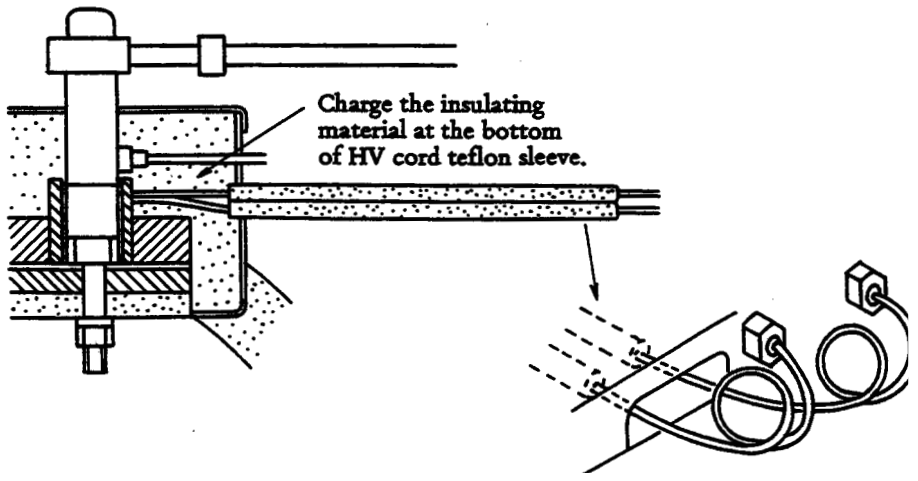
FID cell mounting



Mounting procedures

1) Remove the detector thermostatic oven cover and **insulating material,** fit **insulation** Sleeve (1), then insert FID **cell from** the top.
2) Fix the **cell by attaching sleeve** (2), washer (3) and nut (4) from the column **oven side.**
3) Let the hydrogen and **air** pipes **through** the **notch** of insulation sleeve (1) **and lead** them out **of** the oven **rear.**
4) Detach **HV electrode from** the detector heating block and charge the block with the **insulating** material.
5) **Charge the insulating material.** At this instance, blow out **any** dust if FID **electrode mounting parts** *are* **seen as** *dusty.*
6) Attach the electrode.

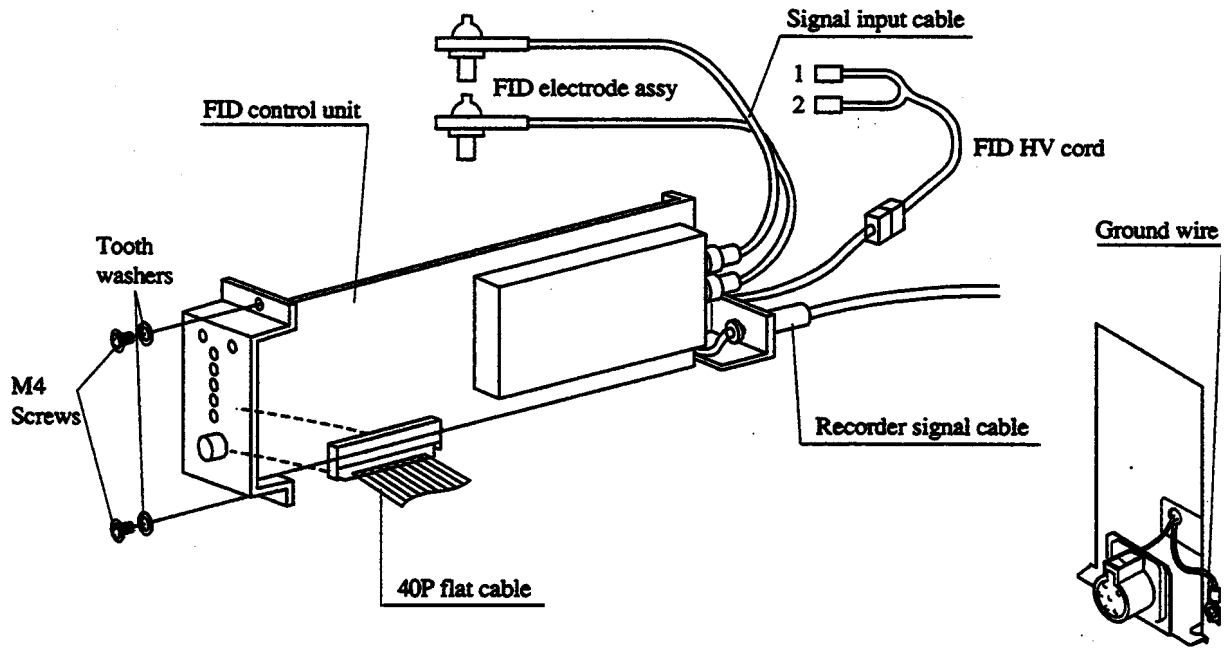
Note **If the FID is mounted in the second hole from the left side of the detector block and interfaced with the TCD, replace insulation sleeve (1) with P/N 221-32978 for TCD. (Standard attachment of TCD oven)**



Mounting parts

①	Insulation sleeve	P/N 221-22542	④	Nut	P/N 22146833
②	Sleeve	P/N 221-23552	⑥	Cell cap	P/N 221-22830
③	Washer	P/N 201-30211-02	⑦	Protector	P/N 221-32362

Mounting FID control unit



Mounting procedures

1) Remove the front cover, side panel and rear cover from the electric control unit at right of the oven. Insert FID control unit from your side, and fix it at the upper and lower parts of the front panel with M4 screws and tooth washers.
2) Attach the ground wire of FID control unit (green line with the terminal at the end) to the rear panel.
3) Insert 40P flat cable.
4) Connect FID signal input cable (one cable when FID is used singly) with HV cord. HV cord ends are branched into 1 and 2. When FID signal input cable is attached to 1, a peak appears in the positive direction with the polarity selection of POL(1).

12

4. Operation

4.1 FID control unit

(1) Range (RANGE) setting

Select the electrometer sensitivity from the keyboard.

Magnify the peak in steps of 10 times. The sensitivity becomes lower in the sequence of $1 \rightarrow 10 \rightarrow 10^2 \rightarrow 10^3$. Enter numericals of the exponent part from the key board

For instance, to set the range at 10 when FID is connected to DET No. 1, enter the keys in the sequence of [DET] [1] [ENT] [RANGE] [1] [ENT] . To set the range at 1, key in 0. Once DETl is set, FID remains at DETl until DET No. is changed. If [RANGE] [3] [ENT] are keyed to change the range or polarity without operating [DET] [1] [ENT] . FID range is switched to 10^3. The setting is displayed on the detector control panel. To display it on the keyboard panel, hit [RANGE] key

Sat lamp lights when the FID signal output is +1V or higher. Output signal at this time is around +1V. In this case, quantitation is not available. If quantitation is required, take appropriate measures such as diluting the sample or changing the split ratio.

(2) Polarity setting

Select the polarity from the keyboard. The signal from the column 1 appears positive at the recorder terminal output with 1 entered, while the signal from column 2 appears positive with 2 entered.

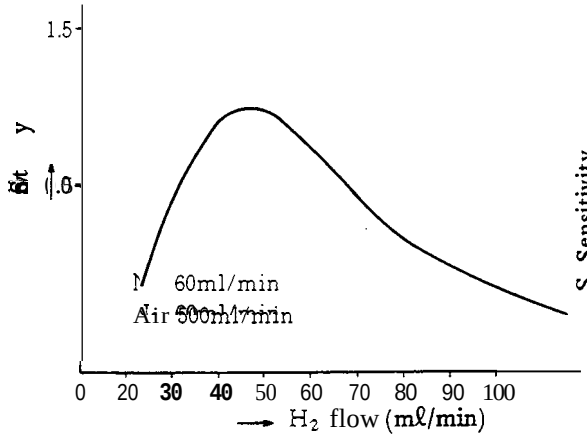
For instance, with [DET] [1] [ENT] (not required when DET1 was already selected) [POL] [1] [ENT] keyed, the signal from the column 1 appears positive at the recorder output. The setting is displayed by the lamp on FID control panel. INJ1(+) lamp being lit means that the signal from column 1 is positive. To display the setting on the keyboard panel, enter [POL] [ENTER].

(3) Backing mode selection

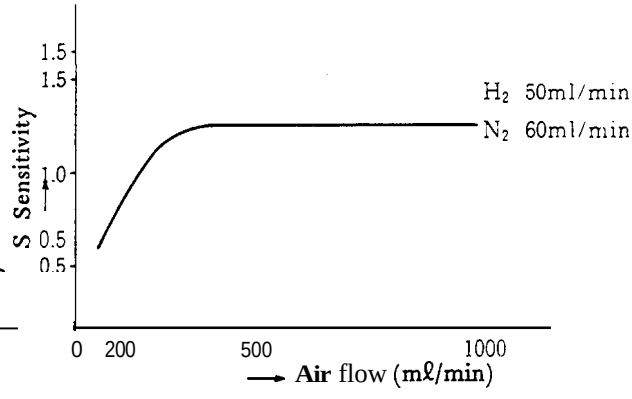
The backing mode selector switch is mounted on the PC board of the FID control unit. If FID background value is so large that zero adjustment is impossible simply by turning the zero adjusting volume, set the backing mode selector switch at "EXT" to examine the degree of unbalance. At this instance, the output signal becomes too large due to insufficient aging of the column or flow line contamination.

4.2 Hydrogen and air flow setting

The detector sensitivity differs depending on the mixing ratio of carrier gas (nitrogen),hydrogen and **air.** The optimum mixing ratio is required to maximize the sensitivity.



Mixing ratio of nitrogen and hydrogen, and sensitivity



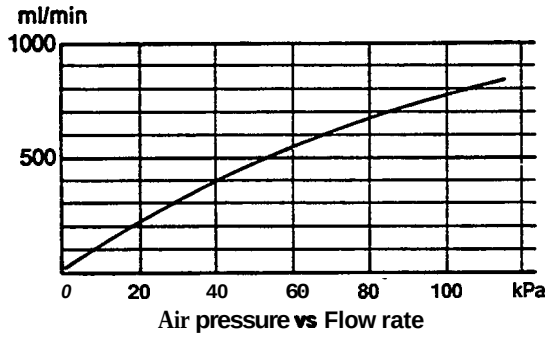
Air flow and sensitivity

In the 3mm-inner dia. column, set the flows as follows.

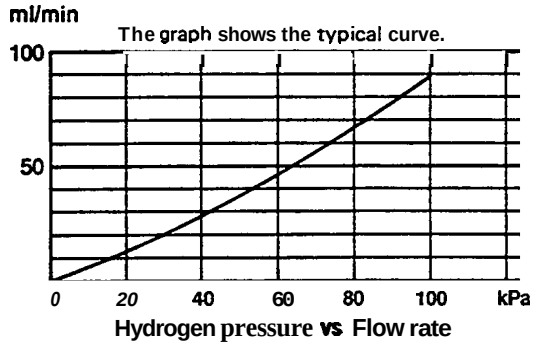
Carrier gas (nitrogen)	60mℓ/min (40～60mℓ/min)
Hydrogen*	50mℓ/min (30～60mℓ/min)
Air	500mℓ/min (400～600mℓ/min)

*Do not increase the hydrogen pressure beyond 100kPa.

The hydrogen and ***air*** flows are controlled with pressure regulators. The pressure ～ flow calibration curve shown in the figure below is useful in setting the flows from the pressure readings.



Air pressure vs Flow rate

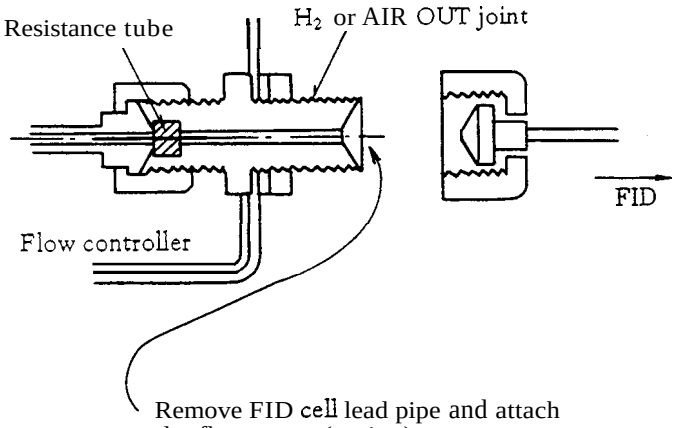


Hydrogen pressure vs Flow rate

12

4.3 Flow measurement

FID hydrogen and air flows are measured at the flow controller H_2 OUT or AIR OUT joint.

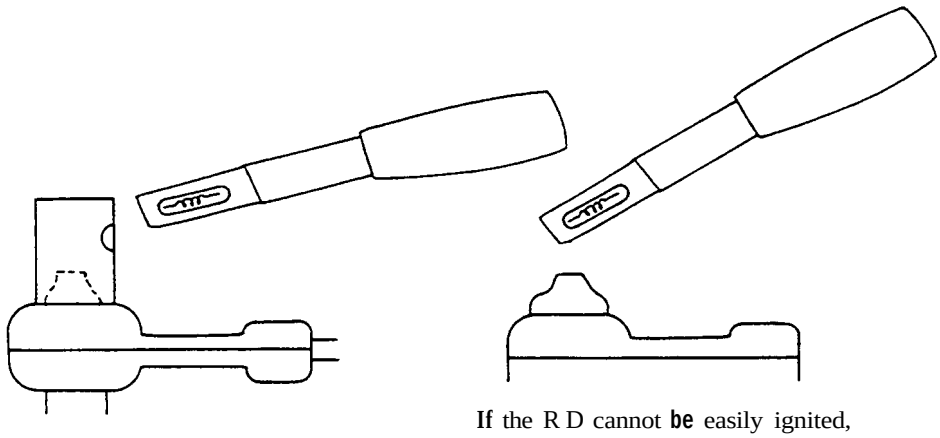


4.4 FID ignition

The FID does not ignite at the mixing ratio of hydrogen and air preset for analysis. To ignite, reduce the air flow. After ignition, return the flow to the specified value.

When the lighter is brought near the FID outlet cap as shown in the figure, push the button.

Note The lighter generates noises when igniting.
During ignition, do not bring the lighter into contact with the FID outlet cap or collector outlet. Keep distance of about 5mm. Also, do not use the lighter during analysis.



If the R D cannot be easily ignited, remove the cap and ignite.

12

4.5 Reduction of air flow

With IGNIT button of the flow controller depressed, the leak valve opens to reduce FID auxiliary combustion *air* flow, thereby making the FID ready for ignition. At this instance, ignite FID with the lighter as mentioned above.

After ignition, release IGNIT button so that the air is fed into FID cell at the flow preset with the pressure regulator.

4.6 Confirmation of ignition

A slight "pop" is heard upon ignition. The FID is ignited if moisture (vapors produced by hydrogen combustion) are deposited on a mirror or lustrous metallic surface that is brought close to the FID cell exhaust hole.

4.7 Nozzle cleaning

If FID is not ignited smoothly or a peak is not detected, the FID nozzle should be cleaned.

First turn off the power of the system and remove the FID collector. Insert a thin stick into the nozzle and clean inside.

<Option>

Wire for cleaning FID nozzle (P/N 221-24165-91) is convenient.

5. FID Troubleshooting

Trouble	Possible cause	Check and correction
1. FID does not ignite. Flame goes off.	a. Carrier gas, H and Air flows improper. b. Detector temperature low. c. Nozzle clogging. d. Air leak valve defective. e. H_2 flow line clogging.	a. Check the flows with the flow meter. b. Increase the temperature. c. Clean or replace. d. Check for proper operation. e. Replace.
2. Peak does not appear.	a. Flame goes off. b. HV not applied to the nozzle end. c. Gas leakage. d. Meter deflected beyond the full scale of -. e. Sensitivity too low. f. Sample adsorbed. g. Data processor faulty.	a. Re-ignite. b. Check with the tester. c. Run the gas leak test. d. Check by turning the zero adjusting volume. e. Check RANGE and sample quantity. f. Conduct repeated analysis. Check the analytical conditions. g. Short-circuit the input signal and check for proper operation.
3. Baseline unstable.	a. Supply voltage fluctuates. b. Contact failure in the signal cable. c. Gasleakage. d. Detector contaminated. e. Piping contaminated. f. Column contaminated. g. Sample evaporator contaminated. h. Purity of carrier gas. i. Mass flow controller (pressure regulator) defective. j. FID control unit defective.	a. Check with a tester or oscilloscope. b. Vibrate the signal cable. c. Run the gas leak test. d. Dismantle and clean. e. Replace. f. Replace or age the column. g. Clean with solvent. h. Replace or attach the filter. i. Replace. j. Repair or replace.
4. Noise appears.	a. Power noise. b. Zero adjusting volume defective. c. Nozzle contaminated. d. FID control unit defective. e. Data processor faulty. f. Contact failure in FID signal cable. g. Air supply silica gel deteriorated. h. Strong electro-magnetic wave	a. Change the power source or attach a power filter. c. Dismantle and clean. d. Repair or replace. e. Short-circuit the input signal and check for proper operation. f. Dismantle and clean. g. Replace. h. Install GC-14B at another place or remove the noise source.
5. Peak becomes broad.	a. Carrier gas flow low. b. Column temperature low. c. Dead volume present.	a. Increase the flow. b. Increase the temperature. c. Check the column joint.

Trouble	Possible cause	Check and correction
	d. Column deteriorated. e. Erroneous selection of the column (type or length). f. Sample evaporator or detector temperature low.	d. Replace or age the column. e. Replace the column. f. Increase the temperature.
6. Peak becomes sharp.	a. Carrier gas flow high. b. Column temperature high. c. Column deteriorated. d. Erroneous selection of the column (type or length).	a. Reduce the **flow.** b. Reduce the temperature. c. Replace or age the column. d. Replace the column.
7. Peak tailing.	a. Glass insert damaged. b. Excessive injection amount. c. Erroneous selection of the column. d. Sample evaporator contaminated.	a. Replace. b. Check RANGE and injection amount. c. Replace the column. d. Clean with solvent.
8. Abnormal peak appears.	a. Ghost appearing from the injection septum. b. Sample decompsed (or changed). c. The previous peak appears. d. **Glass** insert contaminated. e. Injection septum pierced with a hole.	a. Age the septum. b. Check the analytical conditions and sample preparation method. c. Age the column. d. Clean the insert and sample evaporator. e. Replace.

SHIMADZU CORPORATION

SHIMADZU CORPORATION. International Marketing Division
3. Kanda-Nishikicho 1-chome. Chiyoda-ku. Tokyo 101. Japan
Phone:81(3)3219-5641 Fax 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO Overseas Telex No.:0232-3291 (SHMDT J)

SHIMADZU SCIENTIFIC INSTRUMENTS. INC.
7102. Riverwood Drive. Columbia. Maryland 21046. U.S.A.
Phone:1(410)381-1227 Fax. 1(410)381-1222 Toll Free: 1(800)477-1227

SHIMADZU EUROPA GmbH
Albert-Hahn-Strasse 6-10. D-47269 Duisburg 29. F.R. Germany
Phone 49(203)7687-0 Fax.49(203)766625

SHIMADZU (ASIA PACIFIC) PTE LTD.
16 Science Park Drive = 01-01 Singapore Science Park Singapore 0511
Republic of Singapore
Phone 65-778 6280 Fax.65-779 2935

SHIMADZU OCEANIA PTY. LTD.
Unit 5 7 Stubbs Street Auburn NSW 2144 Australia
Phone 61(2)748-3233 Fax.61(2)748-3172

1995. I2 3500-12500-155TD *(A-742)*