

**DIFFERENTIAL REFRACTIVE DETECTOR
RID-10A
FOR SHIMADZU
HIGH-PERFORMANCE LIQUID CHROMATOGRAPH
USER'S MANUAL**

Read the instruction manual thoroughly before you use the product. Keep **this instruction manual** with care so that you can use it any time you need it.

SHIMADZU CORPORATION
CHROMATOGRAPHIC & SPECTROPHOTOMETRIC
INSTRUMENTS DIVISION
KYOTO, JAPAN

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Precautions for Safety Operation

The RID-10A is the differential refractive index detector for the high-performance liquid chromatograph.

In order to operate the unit safely, strictly observe the following points.


1. Do not use the unit for any purpose other than the above mentioned analysis.
2. Follow the procedures described in the instruction manual.
3. Observe the warnings and cautions.
4. Do not disassemble or modify the unit without approval from Shimadzu. Failing to do so may lead to a dangerous situation or damage of the unit.
5. For internal repair of the product, contact your Shimadzu Service Representative.
6. The pink-color pages and the meshed parts are for our service engineers, and are not intended for our clients. Do not attempt installation of the instruments as serious damage could result.

WARNING IN THE INSTRUCTION MANUAL

This instruction stipulates the content of warnings as follows:

- | | |
|----------------|---|
| WARNING | Applied in a case that could result in death or serious injury. |
| CAUTION | Applied in a case that could result in slight injury or physical damage. |
| NOTE | Applied for improvement of operating efficiency or help in understanding. |

Warning labels indicated on the equipment

For safety of operation, this unit is provided with the  mark at the portion where special cautions are required.

When operating the parts where this mark is indicated, exercise special caution after reading the **manual**.

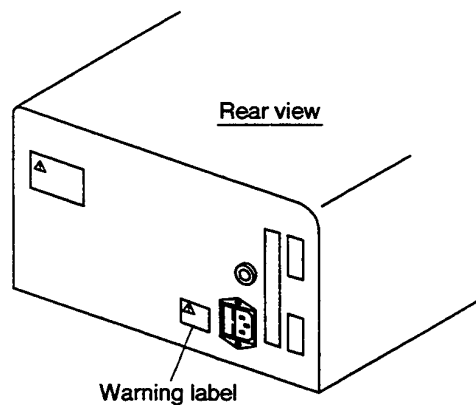
WARNING

Replacement of fuses

This unit uses the following fuse. Be sure to replace the fuse of same type and capacity.

Rated voltage : 100-240V

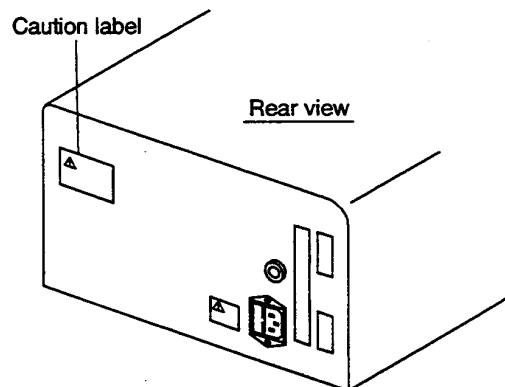
Part No. 072-01652-23 250V 5AT



CAUTION

Do not bend the optical cable for remote control with the radius of 35mm or less.

Bending it with the radius smaller than 35mm may damage the cable, which may cause malfunction of the unit.



Precautions on installation site and handling of the unit

Generally, a large amount of organic solvents is used in the high-performance liquid chromatograph. Sufficient care should be taken in installation site and handling of the unit. Please take care of the notices in the text, not to mention the precautionary requirements listed below:

1. **Ventilation**
Solvents used in the high-performance liquid chromatograph are inflammable and/or toxic. Be sure to ventilate the room well.
2. **Fire**
The lighting of fires is prohibited in the room where high-performance liquid chromatograph is installed. Do not install any devices which may emit sparks in the room. It is also necessary to provide a fire extinguisher preparing for the worst.
3. **Safety goggles**
Wear safety goggles in handling solvents.
4. **Other equipment**
A washstand or the like is necessary to be furnished nearby preparing for the cases such that solvent comes into the personnel's eyes or the personnel touches toxic solvent.
5. **Power source**
Working voltage range and power consumption of this system are as follows. Be sure to connect to the power source conforming to them.

Part no.	Power source	Capacity
228-32000-91	100V	150VA
228-32000-92	120V	150VA
228-32000-94	220~240V	150VA

6. **Grounding**
To prevent electric shock and to secure safe operation of the system, always make grounding.
7. **Repair and maintenance of the unit**
Normal maintenance of this unit can be performed without removing the cover. Do not remove the cover at the normal maintenance. Contact our sales office or agent of repair requires to remove the cover of the main body.

8. Solvent not usable

HFIPA (Hexafluoroisopropyl alcohol) affects the materials used in the flow path and deteriorate the strength of the material. In the worst case, pipe may explode and high-pressure solvent scatters. **As** it is very dangerous, never ~~use~~ HFIPA.

9. Others

The equipment should not be exposed to direct sunlight or strong air currents. It is also recommended to install in a room where temperature fluctuation is small.

Precautions on Static Electricity

Liquid chromatography using flammable organic solvents as mobile phase requires proper care against fire, explosion, etc. Particularly, among various possible accidents, those caused by static electricity are difficult to anticipate, and tend to occur only with unexpected conditions which often make countermeasures insufficient.

At a site where preparative liquid chromatography is practiced, a large amount of flammable substances may be used. Therefore, once an accident happens, it could lead to tremendous damage.

The mechanism of accident caused by static electrical discharge and preventive measures are described below. Take due care in safety measures in handling of equipment.

1. Mechanism of Static Electrical Discharge Accident (Example)

Accidents caused by static electricity take place through the following processes.

Occurrence of Static Electricity

When liquid is fed at high speed through a small-diameter tube like the pipe of a liquid chromatograph, static electrical charge occurs by friction between solid and liquid as shown in Fig. 1.

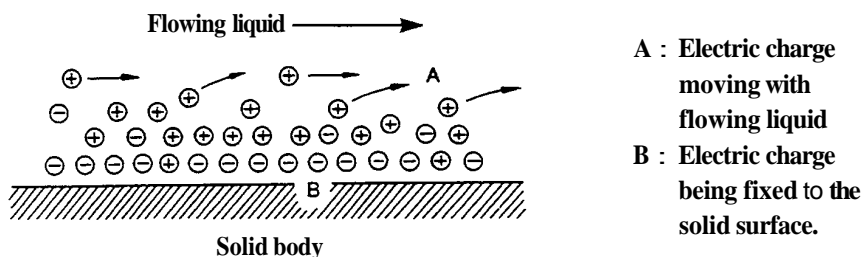
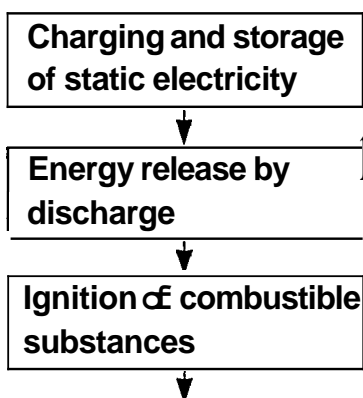


Fig. 1 Occurrence of Static Electricity by Friction between Solid and Liquid



When the charged liquid is collected in an insulated vessel, the static charge accumulates gradually, and the voltage can easily reach a few kilovolts.

If some other conductive object is brought near the vessel, electricity is discharged at a certain distance from the vessel releasing heat energy.

If flammable gas of sufficient concentration exists nearby, ignition is caused by this energy.

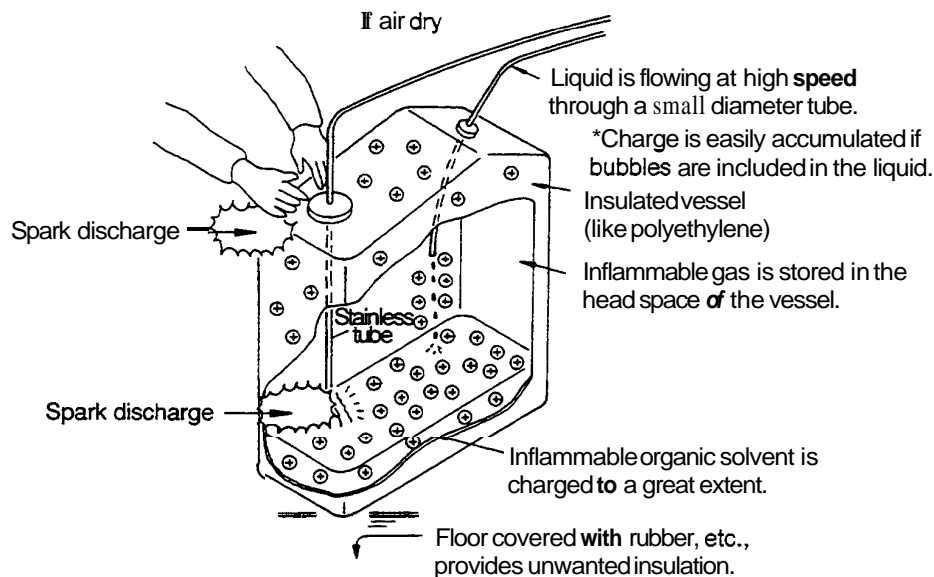


Fig. 2 Conditions which may cause Accidents

2. Preventive Measures against Accidents

The principal preventive measure is the prevention of “charging and storage of static electricity” among those items shown in “Mechanism of Static Electrical Discharge Accident.” The preventive measures are shown below. It is recommended to exercise two or more measures simultaneously.

✱ Particularly when a large quantity of flammable solvent is held in a large vessel, be sure to observe the preventive measures 1, 2, and 3.

Preventive measure 1.

Use metallic (conductive) waste liquid vessel which is well grounded. This releases the charge of the waste liquid and vessel to ground.

The following items are available.

- (1) Grounding wire with clip P/N 228-21353-91
- (2) Metallic 18 liter can P/N 038-00044
- (3) Metallic 4 liter can P/N 038-00043-01

- ✱ Be sure to ground the vessel properly. Disconnecting of grounding wire or poor grounding defeat the purpose of using a metallic vessel.
- ✱ There are some metallic cans which have no conductivity due to an oxidized coating or lacquer on their surface. Be sure to confirm the grounding of vessels by a tester before application.
- ✱ When a liquid with almost no conductivity (of 10^{-10} S/m or less) is discharged into the vessel, it is necessary to mix it with another liquid with some conductivity. (The other liquid can be placed in the vessel in advance.)

Preventive measure 2.

Minimize the clearance of both inlet and outlet of vessel to prevent flame from entering the vessel.

- (1) Cap with three holes for 18 liter and 4 liter cans (P/N 228-21354-91) is available.

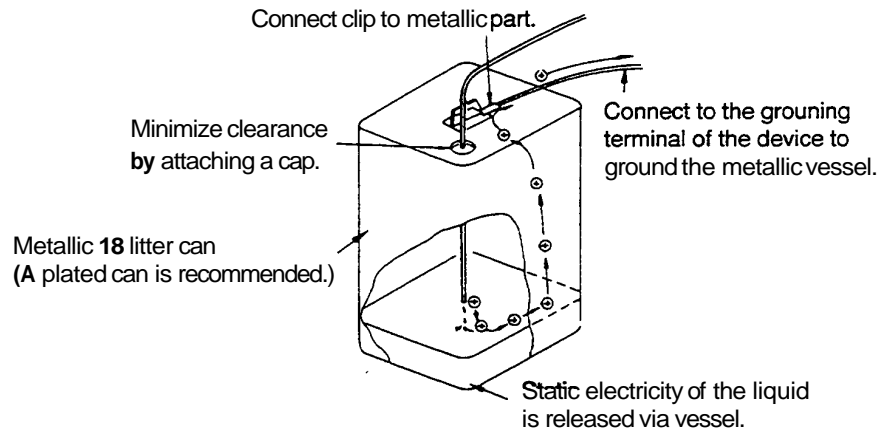


Fig. 3 Anti-Static Electricity Measures for Vessel

Preventive measure 3.

Do not approach the vessel with charged objects including the human body.

Charging prevention measures for human body

- a) Prevention of charging of shoes and clothes
- b) Grounding of human body
- c) Make working floor conductive

Suitable products to be used for those measures a), b), and c) are available on the market.

- ✱ When persons who use no charge prevention measures approach dangerous sections, they have to be grounded beforehand. (For example, they should contact grounded metal by hand.)

Preventive measure 4.

Use pipes with inner diameter of **2mm** or more for waste liquid line for large flow rates.

- ✱ Inclusion of bubbles in the **tube** may increase the amount of charging by ten times. Check that there is no inclusion of **air** via **tube** joints.

Preventive measure 5.

When it is impossible to use a conductive vessel, use caution in the following points.

- a) Set the vessel so that the pipe outlet will be placed below the liquid level in the vessel. Or, dip a grounded metal (ex. pipe connected to the main body of device) in the liquid.
- ✱ This method is not effective for liquid with small conductivity (10^{-10} S/m or less).
- b) Use **a** vessel of ~~the~~ smallest possible capacity to minimize the damage by fire if it should occur.
- c) Prevent the room from **being** dry. Humidity of 65% or more has charge prevention effects.

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Chapter 1 General

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RID-10A is the differential refractive index detector for the high performance liquid chromatograph developed as a module of the **LC-10A** series.

The **RID-10A** improves analysis productivity and convenience of use like a UV detector. Its shortened stabilization time, the fact that it covers various applications from high sensitivity analysis to sampling analysis preparation **and** more, **RID-10A** is much improved in convenience than conventional models.

Only handling instructions for **RID-10A** and related accessories are described in this instruction manual. For handling instructions of other components, please refer to the pertinent instruction manual.

- 1. Excellent stability**

Stabilization time after turning on the power has been shortened to reduce the waiting time for start of analysis. Excellent stability has been realized by employing the dual temperature control structure of the optical system and by improving the thermal design.
- 2. Various applications**

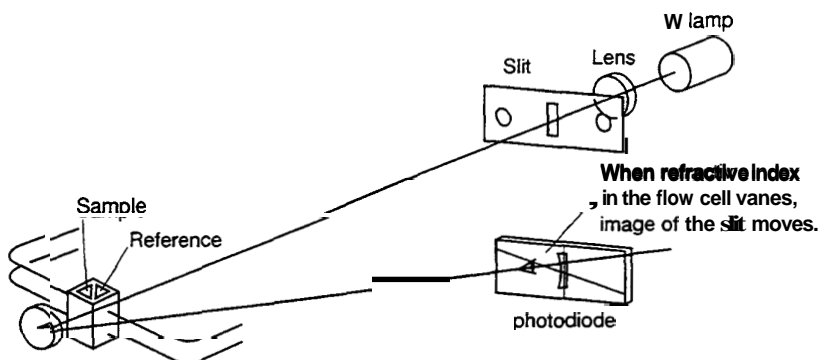
By adapting the original four-partitioned photodiode, this equipment now covers the wide dynamic range of various applications from **high** sensitive analysis to large scale preparation of high concentration samples.
- 3. Safety measure**

The standard model is equipped with a leak sensor which enables automatic performance of operations such as stopping the pump in the early stage of organic solvent leakage.
- 4. Corresponding to the LC-10A series**

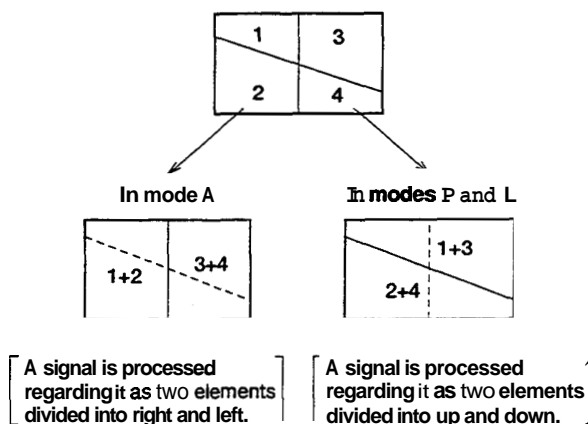
This equipment is designed for the LC-10A series to have excellent operability as a component of the total system, including control from the SCL-10A and data processing in the CLASS workstation, etc.

The optical system of this unit is shown in following illustration. The light radiated from the lamp passes through the lens slit and through the cell in the form of parallel rays, which is then reflected by the mirror and passed through the cell to form an image of the slit on the photodiode.

The flow cell consists of the sample side and the reference side. **When** the refractive index in the sample side cell varies, the image of the slit moves horizontally.



This unit has a photodiode partitioned in four as shown in the figure below. When selecting a piece of photodiode to use among these four, the unit is able to provide measurements for both analytical and preparative work.



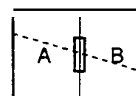
Signal processing in mode A

A signal is processed using the right and left portions of the photodiode as individual elements. Data processing is performed as a two-partitioned photodiode divided into right and left.

When the refractive index in the cell varies, the balance of the incident light intensity into right and left parts of the photodiode changes. The change in the right/left balance is converted into refractive index and then can be recorded.



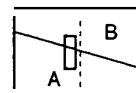
$$RI \propto \frac{A-B}{TOTAL}$$



Signal processing in mode P

Signal processing is performed using the upper and lower parts of the photodiode as individual elements. Since the boundary between the upper and lower parts is slanted against a horizontal line, when an image of the slit moves horizontally, the balance of the light intensity in the upper/lower parts of the diode changes. This change in balance is converted into refractive index. The change of the balance against the variation of the refractive index is one-twentieth that of mode A. In mode A, when the slit image goes beyond the center line, measurement becomes impossible because the balance value stops varying, while in mode P, measurement is possible under these conditions enabling measurement of a sample with high concentration.

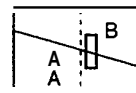
$$RI \propto \frac{A-B}{TOTAL}$$



Signal processing in mode L (An optional flow selection block is necessary)

Signal processing is performed as the same as in mode P, using each of the upper and lower parts of the diode as individual elements. However, since the reference side with less flow line resistance is used as the sample cell, the image moves in the reverse direction of that of mode P. Thus polarity is turned over before the change in balance is converted into a refractive index.

$$RI \propto \frac{B-A}{TOTAL}$$



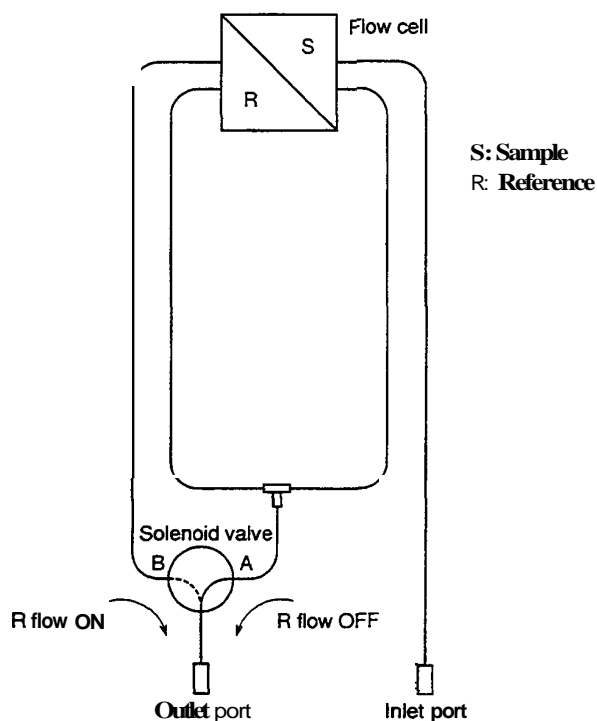
The diagram shows flow lines in the RID-10A.

At R flow **OFF**

When the R flow switch is OFF, the solenoid valve is open at side **A**. The solvent passing through the sample cell flows into the outlet port, not into the reference cell.

At R flow **ON**

When the R flow switch is ON, the solenoid valve is open at side **B**. The solvent passing through the sample cell flows into the outlet port after passing through the reference cell. This is used to fill the sample cell and the reference cell with solvent of equal refractive index before starting the measurement.



Tubing volume (At R flow OFF)

Inlet port to flow cell	63.5	μL
Flow cell volume	9	μL
Flow cell to cell outlet port	280.2	μL

Chapter 2 Parts List

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[Caution]

When connecting the flow line and performing maintenance, be sure to use the **parts** described on this page or “12.1, Consumable and Repair **Parts**”. Normal function of the system is not **guaranteed** when other **parts** are used.

This equipment consists of the following components. Upon unpacking, confirm that all parts listed below are included in your shipment.

- ① RID-IOA Main Body
- ② Standard Accessories

Part Name	Parts No.	Quantity
Signal cable	228-25089-92	1
Power supply cord (120V) (220~240V)	071-60814-01 071-60814-06	1
Optical cable	070-92025-51	1
Remote cable	228-28253-91	1
Syringe	046-00001	1
Syringe adapter	228-15672-91	1
Coupling 1.6C	228-16004-03	1
Male nut PEEK	228-18565	5
SUS tubing	228-22310-00	1m
	228-22305-00	50 cm
Teflon tubing	228-18495-03	2 m
Drain tubino kit	228-18495-03	1
Locking plate	228-18751	1
Instruction manual	228-30178	1
SC Coil ASSY (220~240V)	228-34050-91	1

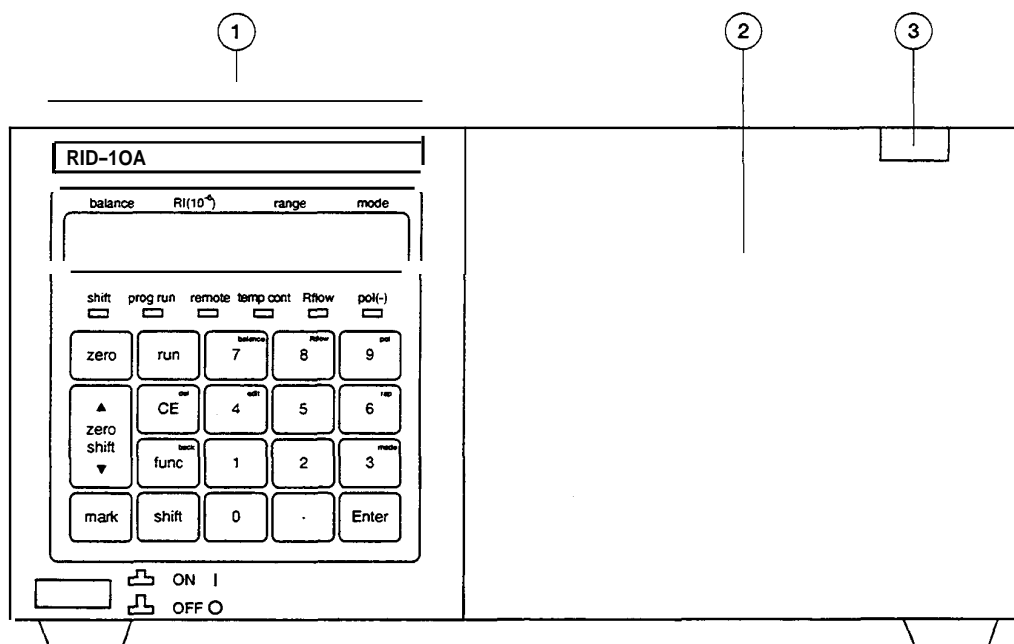
Chapter 3 Component Location and Function

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3. 1

Front View

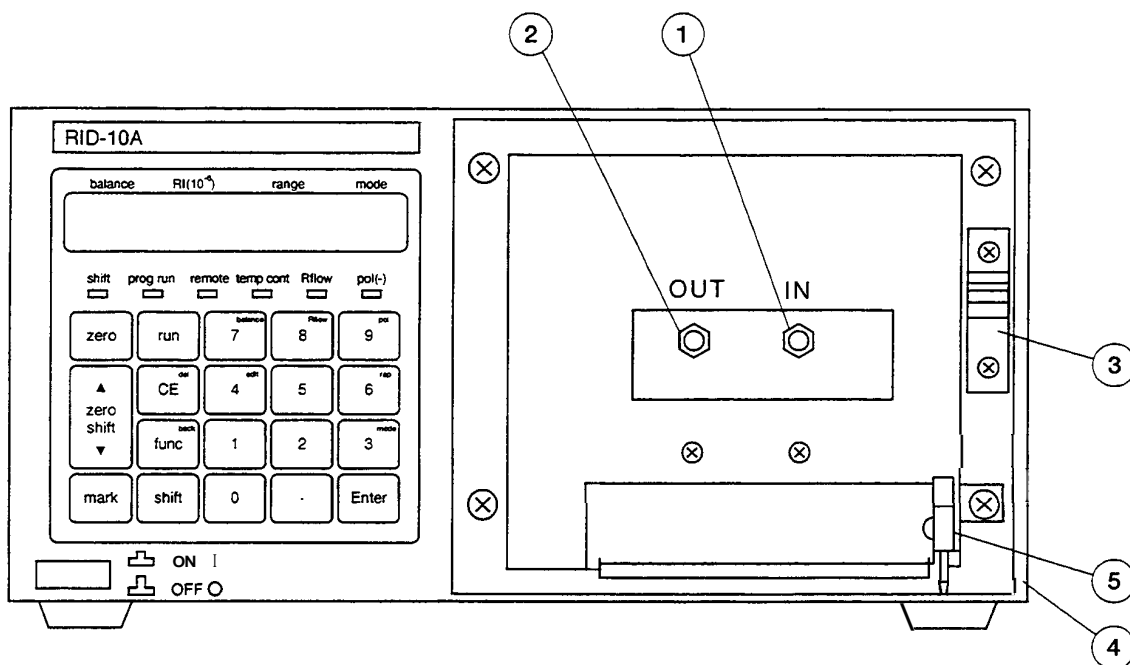


Component Location and Function

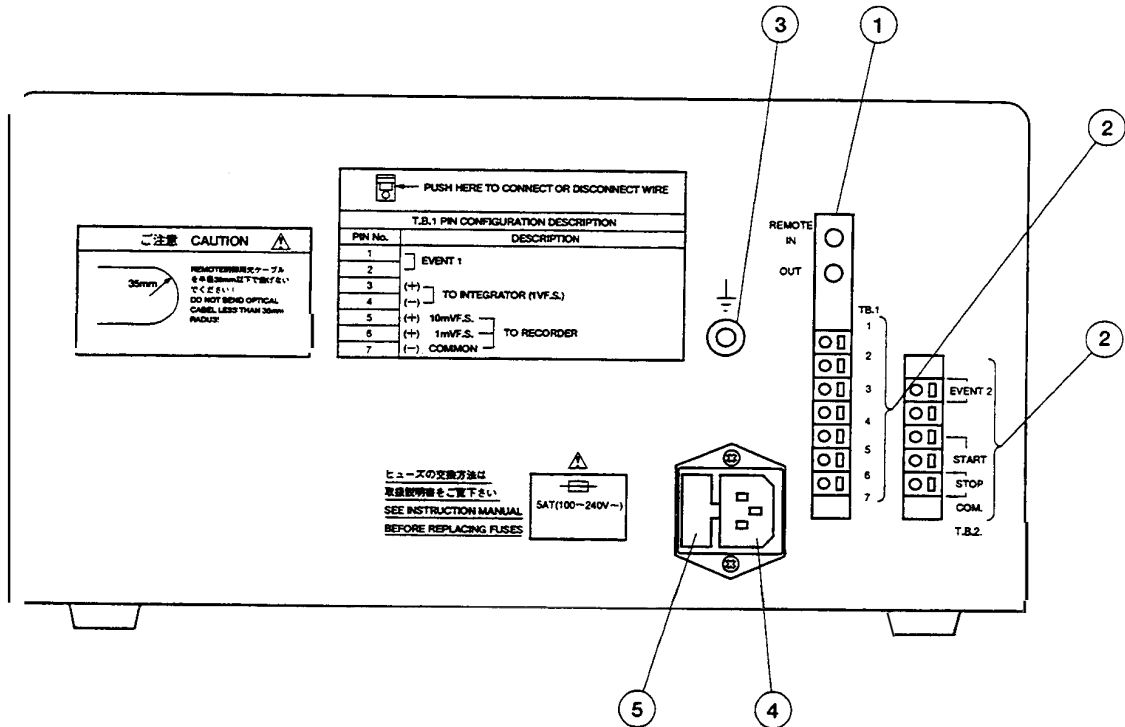
No.	Description	Function
①	Control panel	Performs parameter setting and displays set values.
②	Front cover	The front cover is opened for mounting and dismounting of flow cell and piping.
③	Front cover opening button	Press this button to open the front cover.

3. 2

Interior of Front Cover, Right Side



No.	Description	Function	Refer to page
①	Cell inlet port	Connects the tube from column.	4-8
②	Cell outlet port	Normally connects back pressure tube (0.3mm ID.x ID).	4-9
③	Tube holder	Secures the connected tube.	
④	Connection port for leak tube	Connects the leak tube to this port .	4-13
⑤	Solvent leak sensor	Detects solvent leaks and outputs an error signal.	10-4



No.	Description	Function	Refer to page
①	REMOTE connector	This is the connector for SCL-10A.	7-2
②	External input and output terminals	These terminals connect external equipment.	7-16
③	Grounding terminal	This terminal is used for grounding.	
④	Power supply cord connector	The power supply cord is connected here.	4-8
⑤	Fuse holder	Two fuses are provided in this holder.	8-7

Chapter 4 Installation

4

Installation

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Warning

To take full advantage of the RID-10A's performance capabilities and to ensure its operational stability over a long service life, verify that the selected installation site satisfies the following requirements.

1. Ventilation

Ventilate the room where the high-performance liquid chromatograph is located since the solvent used is flammable and/or toxic.

2. Fire

Never use fire in the same room where the high performance liquid chromatograph is installed. Also, avoid installation in the same room of other devices which may spark. **Always** keep a fire extinguisher nearby in case of accident.

3. Sink

Install a sink nearby for flushing eyes or skin which have been in contact with solvent.

4. Corrosive gas and dust

Avoid installation in a place exposed to corrosive gases or dust.

5. Electromagnetic noise

Avoid locations subject to intense magnetic or electromagnetic fields. Use an additional noise filter if power line noise interferes.

6. Space requirements

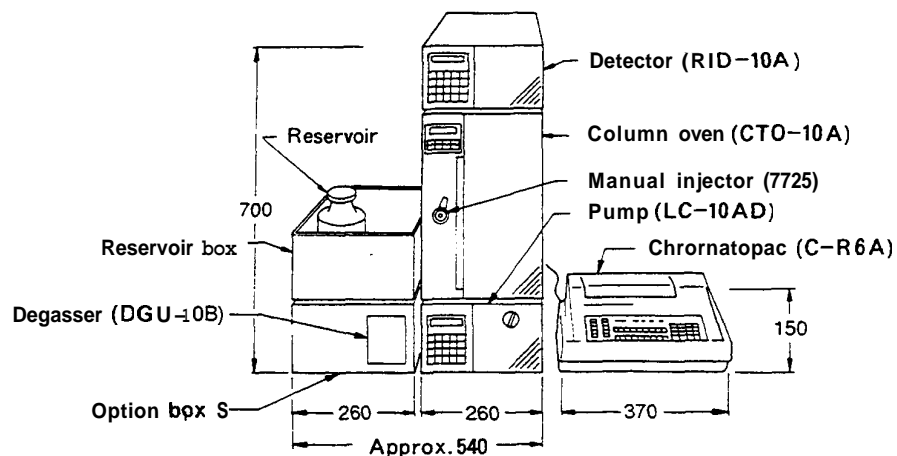
This system is designed to be used on a table or stand, preferably a solid and flat surface with a depth of 60cm or more. See "4.2, Example of System Layout" for typical configurations of systems and installation space.

7. Others

Select an installation site with the following parameters to maintain full performance of the system.

- (1) ~~Maintain~~ room temperature within 4~35°C, without extreme fluctuations.
- (2) Avoid direct output of a heater or a cooler.
- (3) Avoid exposure to direct sunlight.
- (4) Avoid locations subject to strong vibrations or prolonged weak vibrations.
- (5) Maintain relative humidity within 45–85%.

Example of liquid chromatograph system layout using this equipment and space requirements are shown below.

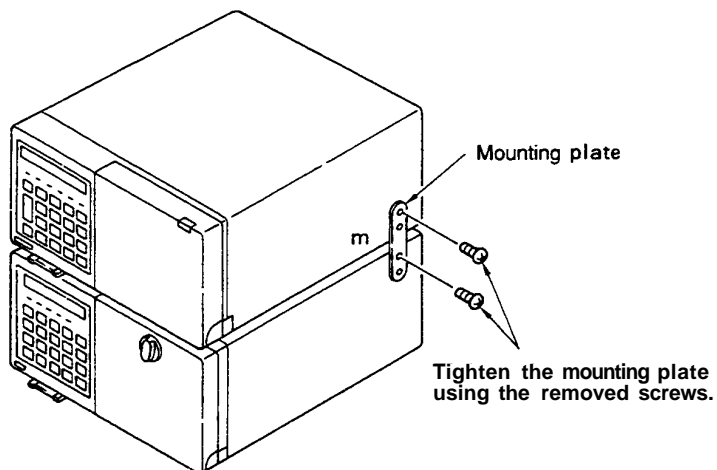


Caution

Each component of the LC-10A has a little clearance at the bottom. Be careful to keep fingers clear when installing the unit.

RID-10A may be stacked on the pump (LC-10AS/LC-10AD/LC-10AT/LC-10Ai) or the column oven (CTO-10A/10AC). The equipment can be stacked and locked for safety in case of an earthquake, etc. using the supplied locking plate.

- (1) Refer to the figure below and remove the screws fastening the equipment cover.
- (2) Attach the mounting plate using the removed screws.



Warning

Check the following points before connecting power supply.

- Supply voltage and capacity

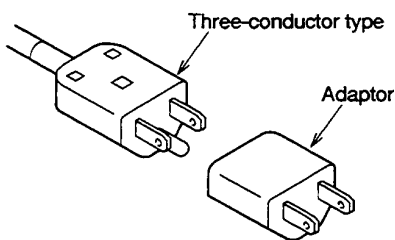
Supply voltage and capacity	Part Number
100V 150VA	228-32000-91
120V 150VA	228-32000-92
220 — 240V 150VA	228-32000-94

When the power supply is not stable or the capacity insufficient, satisfactory performance is not possible. Verify the total power supply for the system before preparing the power supply.

- Verify that the power switch of the main unit is **turned OFF**.

1. Connection to outlet

- (1) Connect the female connector of the power supply cord supplied with the unit to the power supply cord connector at the rear of the unit, and plug the male connector into a power supply outlet.
- (2) The supplied power cord is a three-conductor (3-prong) type. When connecting to a two-conductor (2-prong) type power supply outlet, use the provided power supply adapter.

**2. Grounding**

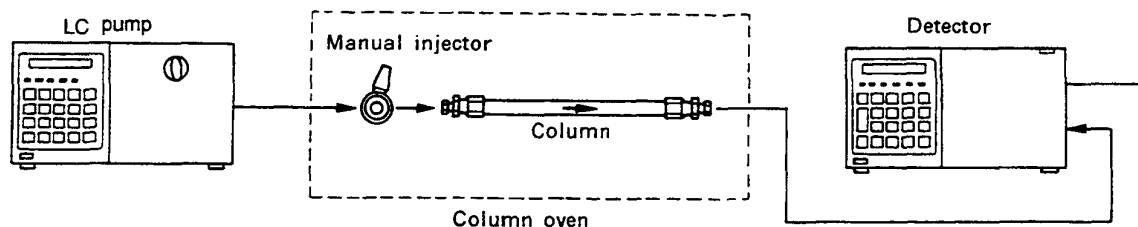
- (1) When the three-conductor type power supply outlet is used, the unit is grounded by the power supply cord.
- (2) When the two-conductor type power supply outlet is used, the unit is not grounded. In this case, ground from the grounding terminal on the rear panel of the unit.

Warning

To prevent electric shock and to secure safe operation of the system, always ground the unit.

This section describes proper connection in a typical flow line as shown in the figure below:

1. Flow line in the system



Refer to the column oven instruction manual for installation of the manual injector and column oven. When the column oven is not used, an injector holder and a column holder are optionally available for installation of the manual injector and the column.

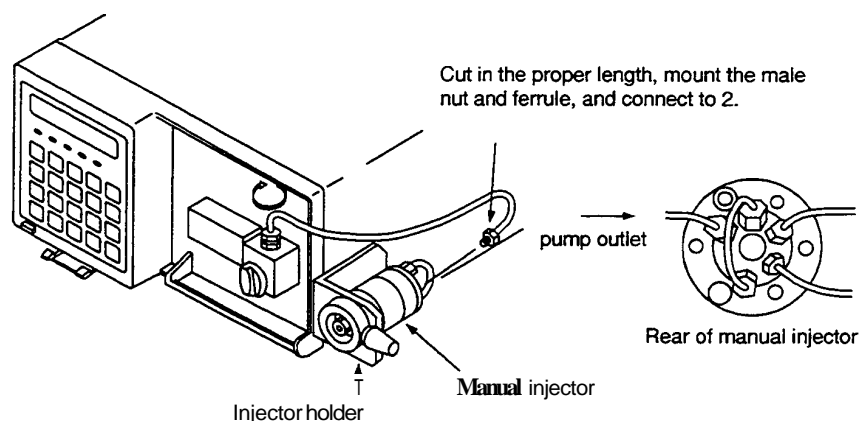
2. Connecting the solvent delivery pump and manual injector

Connection of the solvent delivery pump and the manual injector are described below.

- (1) Cut the **SUS** pipe (1.6 x 0.3 supplied as a liquid pump accessory) to the necessary length for the pump outlet and injector port 2.
- (2) **Install** the male nut and ferrule to both ends of the **SUS** pipe. Attach the male nut 1.6MN and ferrule 1.6F (accessories of the pump) to the pump outlet side, and the male nut and ferrule (accessories of the manual injector) to the manual injector.
- (3) Connect **an** end of the **SUS** pipe to the pump outlet and the other end to the injector.

Note

- Connect the drain tube to the manual injector ports 5 and 6. Adjust the tip of the drain tube to meet the needle port of the injector.
- For all connections of the manual injector, use the male nut and ferrule supplied as the manual injector accessory.



3. Connecting injector and column

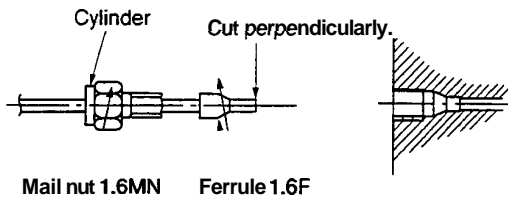
A typical connection of a manual injector and a column is shown below:

- (1) Cut the **SUS** pipe (1.6 x 0.3) supplied to the pump, to the necessary length for connecting the injector and the column.
- (2) Attach a male nut and a ferrule to the ends of this pipe.
- (3) Connect the ends of the **SUS** pipe to the injector and the column respectively.

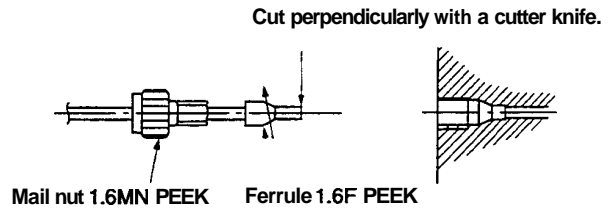
Note

- Attach the ferrule and the male nut supplied with the injector to the injector and those supplied to the pump to the column.
- Make the pipe length between the injector and the column **as** short as possible to prevent sample band broadening.
- Cut the pipe on the perpendicular and connect it securely to avoid dead volume.

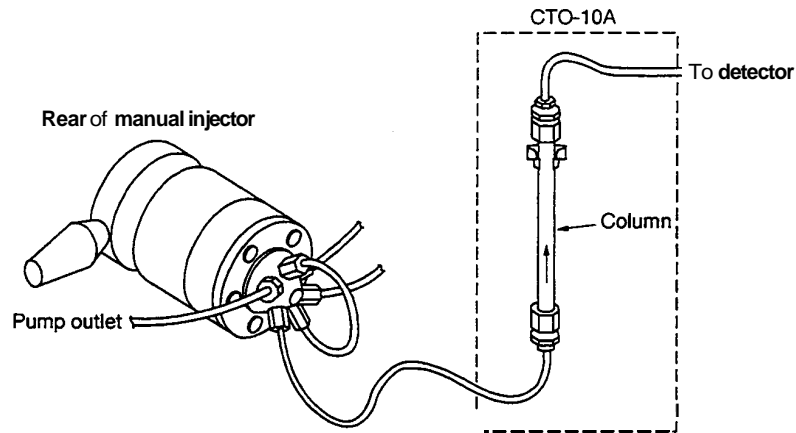
SUS pipe



PEEK pipe



Installation



4. Connecting detector and column

A typical connection between the RID-10A and a column is described in this section.

- (1) Cut the supplied SUS tubing (50cm long) to the necessary length for connecting the column outlet and the cell inlet port.

Note

Cut the tube on the perpendicular. When the tube is cut on a diagonal, dead volume is generated which deteriorates separation.

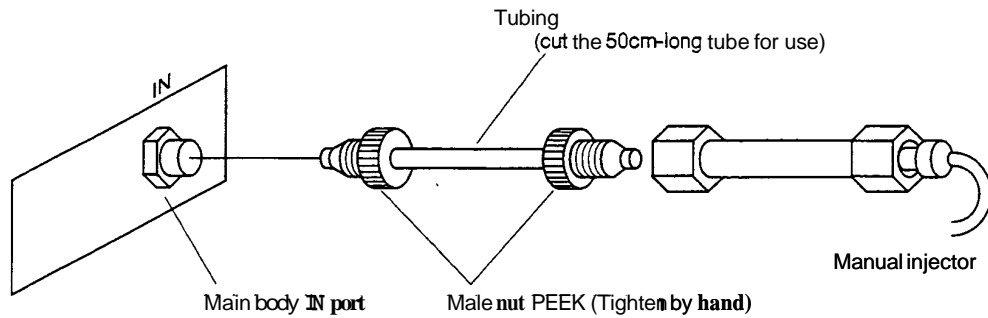
Note

When the flow selection block (option) is used, refer to section 5.3 Piping of Flow Selection Block (Option).

Connect the tubing between the column and the detector inlet with the supplied male PEEK nut as shown in the next figure. tighten the male PEEK nut firmly without using tools.

Note

Insert the tubing into the column joint and the male union securely until it comes to the end and then tighten the male PEEK nut. Similarly, insert the cell inlet pipe until it comes to the end and tighten the male PEEK nut so that dead volume is minimized.



5. Detector outlet side piping

- (1) Prepare a waste liquid bottle.

Connect the drain tube to the detector outlet. Select one from the drain tubes listed in the table below depending on the applied flow rate. Insert the outlet of the drain tube into the waste liquid bottle.

Max. flow rate [mL/min]	Drain tube [Inside diameter (mm) × length (cm)]	For extension [Inside diameter (mm) × length (cm)]	Remarks
3	0.3 × 100	1.0 × 100	
5	0.5 × 100	1.0 × 100	
20	0.8 × 100	1.6 × 100	
150	1.6 × 100	1.6 × 100	At mode L (option)

When using the FRC-10A fraction collector, connect the inlet tube of the FRC-10A to the detector outlet directly. Note that the **maximum** flow rate varies depending on the preparative head of the FRC.

FRC-10A preparative head	FRC-10A inlet pipe Inside diameter [mm] × Length [cm]	FRC-10A outlet pipe Inside diameter [mm] × Length [cm]	Max. operating flow rate [mL/min]
Fraction correction head with valve	0.8 × 100	1.6 × 100	20 (150')
Fraction correction head with valve	0.3 × 100	1.6 × 100	3
Fraction correction head without valve	0.3 × 100		

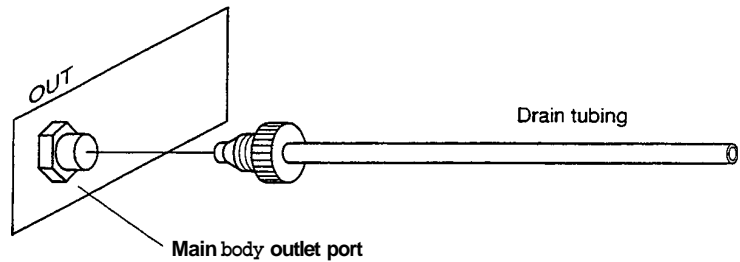
*) An optional flow selection block is necessary.

Caution

The solenoid valve and flow cells may be damaged by application of **back** pressure which exceeds the detector resistance pressure. Do not use the pipes listed in the table which are out of range.

Note

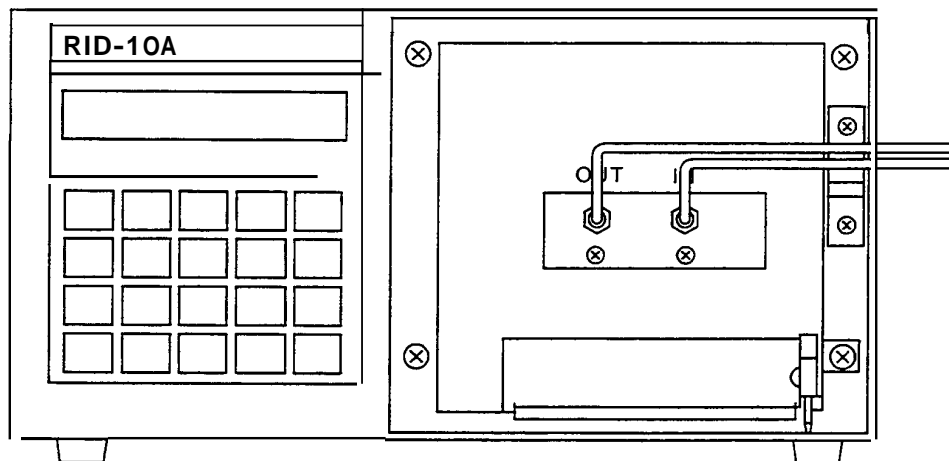
To prevent the detector **from** being damaged by back pressure, it is useful to connect the relief valve (option) to the detector outlet when the unit is used with large flow rate, or the piping is clogged.



Installation

6. Fixing the tubing

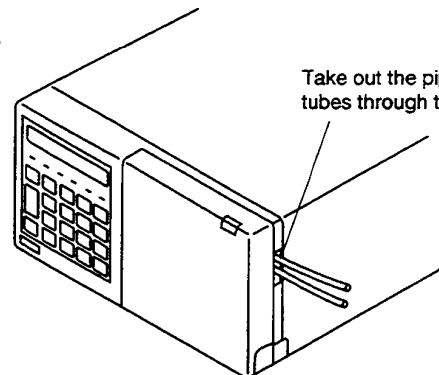
When piping for the detector inlet and outlet is completed, secure the two piping tubes in the tube holder as shown in the figure below.



Fit the tubes in the tube holder.



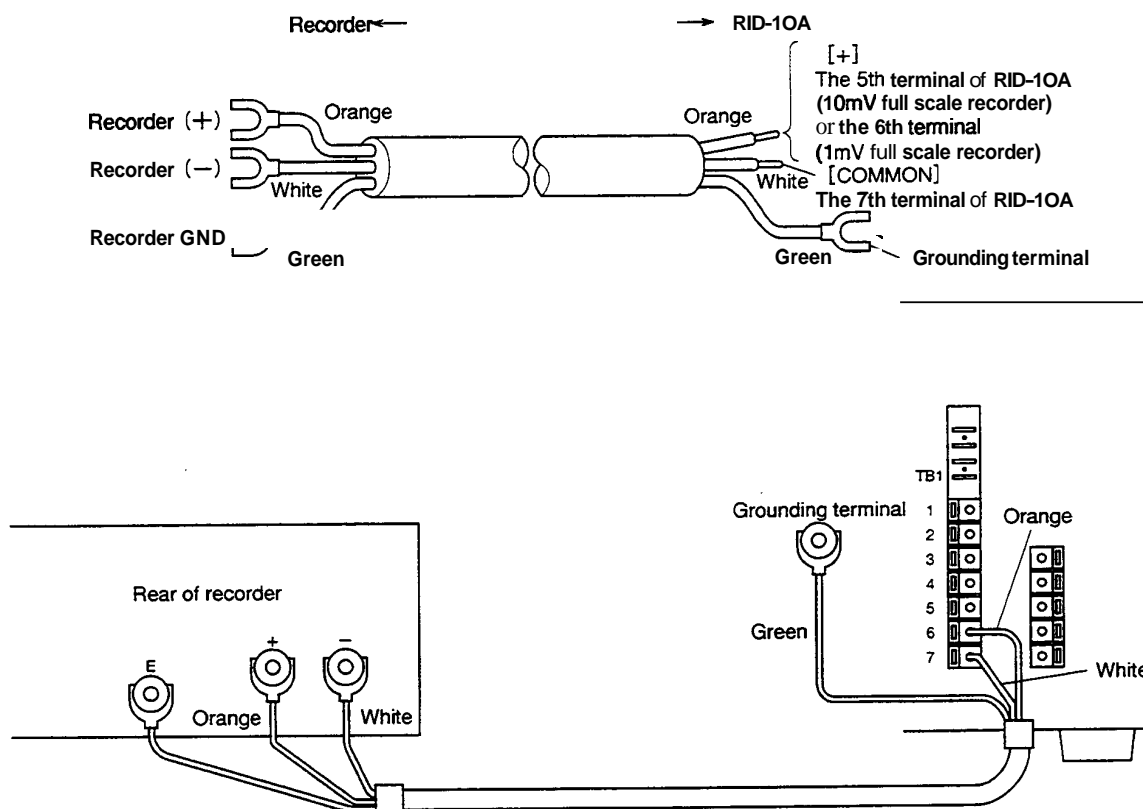
Take out the piping tubes through the cut.



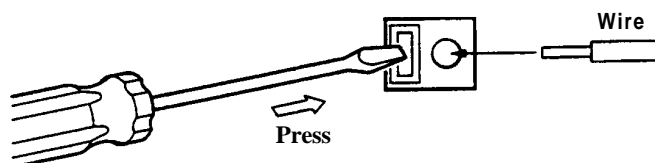
When the front cover is closed.

1. Connection with recorder

Connect the **RECORDER** terminal of the RID-10A and the recorder using the supplied signal cable.



When using a stranded wire, twist the end tightly, or tin it with solder. **Using** a small screwdriver or other tool, depress the rectangular button adjacent to the appropriate terminal hole. Insert the wire and release the button to **clamp** the wire in position, **as** shown in the **figure** below.



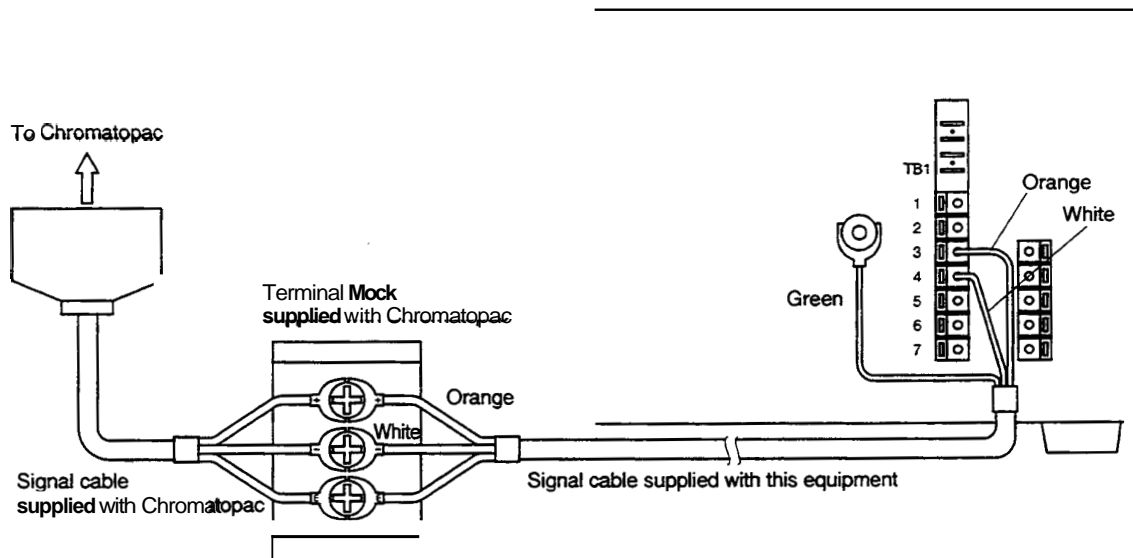
2. Connecting with Chromatopac (integrator)

Connect the Chromatopac to the **INTEGRATOR** terminals (TB1 No. 3 and No. 4) in the external input/output terminals at the rear of the RID-10A.

The connection procedure is similar to that in "1. Connecting with recorder." Connect the cable supplied with the Chromatopac to the Chromatopac side and connect each terminal of the signal cable to the terminal block supplied with the Chromatopac.



Installation

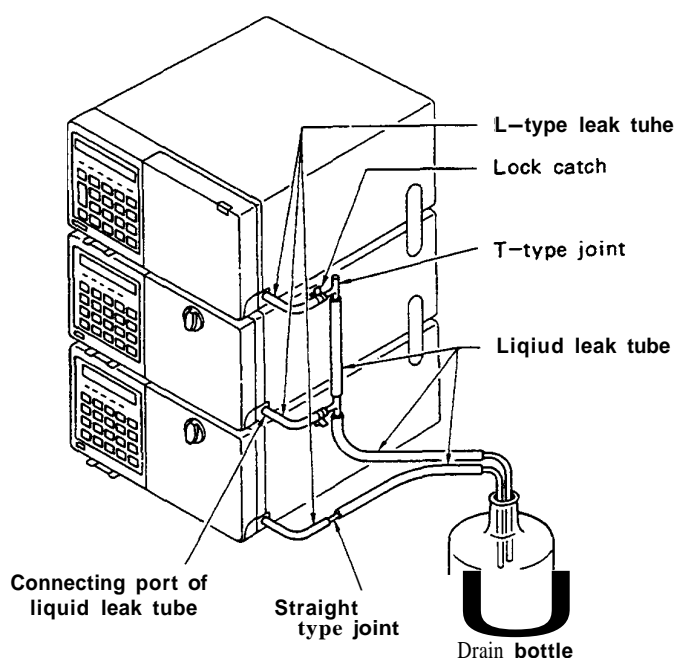


Each component in the LC-10A series is designed so that solvent leakage is discharged from the liquid leak tube connection port either at the right side or the lower front side of the equipment. Connect the liquid leak tube if necessary.

1. Connecting liquid leak tube in LC-10A system

Connect the supplied L-type leak tube to the connecting port in each component.

Connect the straight type joint to the liquid leak tube at the bottom of the equipment and insert it into the drain bottle.



Use a T-type joint to connect liquid leak tubes for multiple instruments.

Cut the supplied drain tube to an appropriate length.

Attach the L-type leak tubes horizontally or downward. Secure them to the side panels of the equipment with the supplied lock catches.

Position the waste liquid bottle lower than the bottom of the equipment.

Chapter 5 Operation

5

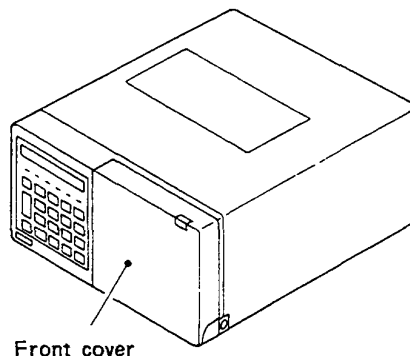
Operation

CONTENTS

5.1	Precautions for Operation	5-2
5.2	Fundamentals of Operation	5-3
5.3	Plumbing of the Flow Selection Block	5-13
5.4	Creating and Executing Time Programs	5-16
5.5	Additional Functions (AUX-FUNC).....	5-21

Caution

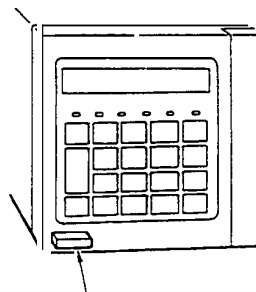
1. Be sure to close the front cover during measurement.
The baseline fluctuates when the front cover is opened or closed during high sensitivity analysis. Noise may be increased when the front door is kept open.



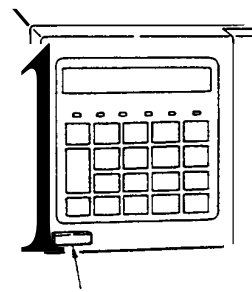
2. Precautions to prevent clogging of the flow cell.
Dusty or clogged flow cells are the most frequent causes of trouble in any detector. After analyzing a high concentration sample, thoroughly flush it from the flow cell, using a large amount of mobile phase.
Buffer solution crystallizes upon drying, and can clog the flow cell and tubing. Never leave buffer solution in the unit as mobile phase. Always flush the flow lines prior to shutdown of the instrument. Turn ON R flow several times during solvent delivery and replace the reference flow cell with water.

1. Turning power ON

- (1) Push the power switch on the front panel to turn the power ON and **OFF**.



Power switch OFF state



Power switch ON state

- (2) When the power is turned ON, the RID-10A operates as follows:

① Turning power ON



② All of the dots in the display unit and all of the indicator lamps light.



③ Control program version No. is displayed.

balance	RI (10 ⁻⁴)	range	mode
R I D - 1 0 A V ※ . ※			



④ The motor for optical balance adjustment is moved to the set position and the instrument **seeks** the home position.

balance	RI (10 ⁻⁴)	range	mode
S E E K I N G H O M E			



⑤ The position is adjusted so that the optical balance is at the optimum position.

balance	RI (10 ⁻⁴)	range	mode
1 0	B A L A N C E		A

- (3) After turning the power ON, a memory check is automatically performed and when no error is detected the following display appears, indicating **that** operation is possible. This is the initial State.

balance	RI (10 ⁻⁴)	range	mode
0	0	4	A

shift	prog run	remote	tamp cont	R flow	pol (-)
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

The balance and RI values vary depending on the types of instrument.

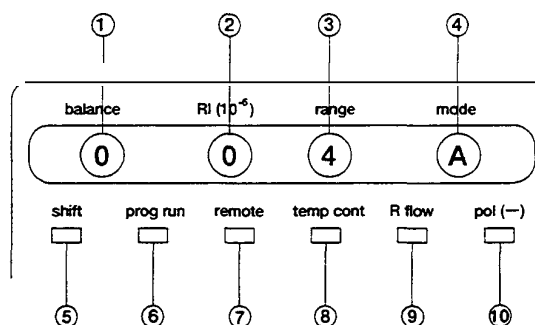
Note

If **NOT PROTECTED** message is displayed and an alarm sounds after turning the power ON, press **CE** key. When this message is displayed, the time program is initialized.

When any other error message is displayed, turn the power **OFF** and contact your Shimadzu Service Representative.

2. Display unit

The display unit consists of display screen and indicator lamps as shown below.



No.	Display or description	Function
①	balance	Displays position of the light on photodiode.
②	RI (10^{-6})	Displays refractive index (unit : $\times 10^{-6}$ RIU)
③		Displays full scale of refractive index output to the recorder terminal (unit : $\times 10^{-6}$ RIU)
④	mode	Displays measurement mode.
⑤	shift	Shift key indicator lamp.
⑥	prog run	Time program operation indicator lamp. Lights when time program is being executed.
⑦	remote	Remote mode indicator lamp. Blinks when controlled by SCL-10A.
⑧	temp cont	Lights when the power is being supplied to the temperature controlled heater for optical system unit.
⑨	R flow	Reference flow indicator lamp. Lights when the liquid is being supplied to the reference flow line side.
⑩	pol (—)	Polarity indicator lamp.

3. Keyboard

The nineteen keys on the front are used for operation and settings and classified into the following three types.

(1) STD-func keys

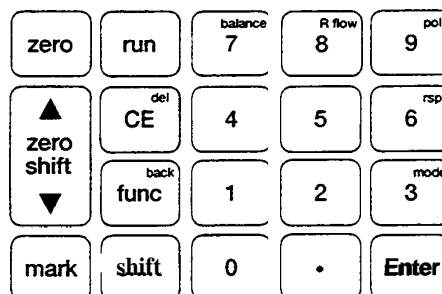
Press these keys to perform a specified operation immediately (**[Zero]** key, etc.)

(2) Shift-func keys

Press these keys after pressing a **[shift]** key to perform a specified operation (**[7 balance]** key, etc.).

(3) Edit keys

Use these keys to input parameters and edit a time program (ten-key, etc.).



(1) STD-func keys

zero Auto zero key

Press this key to perform zero adjustment. Returns the baseline to the zero position set by **▲ zero shift ▼**.

▲ zero shift ▼ Zero shift key

Press this key to move the zero position on the recorder. It is moved upward by pressing **▲** side and downward by pressing **▼** side.

mark Mark key

Press this key to add Mark to the data being recorded in the recorder by pressing this key. Mark is not valid in the integrator output.

run Run key

This key is a switch to *start* and stop a time program.

(2) shift-func keys

(+)key

These keys move to time program creation mode.

shift + **balance** key

These keys drive the zero glass and adjust its position to optimal.

shift + **R flow** key

These keys switch the solenoid valve to replace the liquid inside the reference cell with the mobile phase.

shift + **pol** key

These keys switch the polarity of recorder output. **pol(-)**
LED is lit for (-) polarity.

[shift] + **[mode]** key

These keys select the measurement mode.

The measurement mode changes from **A** → **P** → **L** → **A**.

A : Analytical mode

P : Preparative mode

L : Large-scale preparative mode

For the operation of each mode, refer to section 5.3.2, Changing the Measurement Mode.

[shift] + **[rsp]** key

These keys can be used to set time constant.

Values from 1 to 10 can be set. For the relation between each value and time constant, refer to section 5.2.9, Setting the Response.



Operation

(3) edit keys

[0] - **[9]** Numerical keys

Input numerical values.

[Enter] Enter key

Sets input values.

[CE] Clear key

Returns the display screen to the initial state.

Press this key to clear an input value when entering a numerical value. Press this key to clear the display and alarms when an error is displayed. Equipment failure errors cannot be cleared with this key.

[shift] + **[del]** Delete key

Deletes a line in the time program.

[func] Function key

Advances to the next item in the display screen.

AUX. **FUNC** setting screen is forwarded.

[shift] + **[back]** Back key

Returns to the previous item.

AUX. **FUNC** setting screen is scrolled backward.

4. Basic operation

Before starting analysis, flush the detector flow line with mobile phase. Supply the mobile phase at a flow rate of 1 mL/min, and then press **[shift]** and **[8 Rflow]**. The solenoid valve is switched and the **[R flow]** lamp is lit. Solvent flows **through** the sample and the reference sides of the detector cell replacing solvent in each. Supply solvent for approximately 20 minutes with Rflow ON. Then, turn Rflow ON/OFF several times to drive the bubbles out of the cell. Return to **Rflow OFF** state, and wait **until** the baseline stabilizes.

When the balance value is more than 50, press **[shift]** and **[7 balance]** to perform optical balance adjustment.

When the baseline is stabilized, **start** analysis.

Note

When the liquid inside the flow lines is not sufficiently replaced with the mobile phase, the baseline takes longer to become stabilized and the **drift** becomes large. To perform effective replacement, turn R flow ON/OFF several times at an interval of two minutes.

Caution

Switching flow lines during solvent delivery at large flow rate in mode L may damage the solenoid valve and the flow cell. Thus, the following message appears when performing R flow in mode L.

CHECK FLOW

7

Press **Enter** after the flow rate is changed to 1 mL/min.

To avoid trouble caused by bubbles, refer to the following.

- (1) When the pump sucks bubbles, degas the solvent with the ultrasonic cleaner. If dirt on the suction filter is the cause, clean the filter with the ultrasonic cleaner or replace it.
- (2) Bubbles may not be easily removed when using an aqueous solvent. Flush the flow lines with methanol or acetone.
- (3) When replacing the aqueous solvent with organic solvent or vice versa, bubbles may be generated successively. If bubbles can be observed, flush the flow lines with thoroughly degassed solvent.

5

Operation

5. Setting the measurement mode (Example)

When changing the measurement mode from A to P

- (1) Press **shift** and **2 mode**.

The mode is changed to P to perform optical balance adjustment.

balance	RI (10 ⁻⁴)	range	mode
1 0 0	BALANCE		P
↓			
0	0	4	P

Note

When the above operation is repeated, the mode is changed from $\boxed{A} \rightarrow \boxed{P} \rightarrow \boxed{L}$. Mode L is only applicable when an optional flow selection block is installed. When the block is not installed, do not perform measurement in mode L.

Refer to 5.3.1 for the mode setting when using the flow selection block.

Mode	Refractive index measurement range	Input step
A	$0.01 \sim 500 \times 10^{-6}$ RIU	0.01 step for 0.01 to 1 1 step for 1 to 500
P · L	$1 \sim 5000 \times 10^{-6}$ RIU	1 step for 1 to 5000

Select the mode corresponding to the refractive index of the sample to be measured. When the index is 500×10^{-6} RIU or less, use mode A.

Note

Note that the setting of mode P has wider measurement range while it has the larger baseline noise.

Note

The range value will default to 100 when the mode is changed as described below.

- ① When the mode is switched to mode P or L during measurement with the range of 0.01 to 1.00 in mode A.
- ② When the mode is switched to mode A during measurement with the range of 501 to 5000 in mode P or L.

6. Setting measuring range (Example)

The procedure to set the recorder range is as follows:

Changing the range from 4 to 16×10^{-6} RIUFS

- (1) Press **func** once in the initial state to access the range parameter (blinking).

balance	RI (10^{-6})	range	mode
0	0	4	A

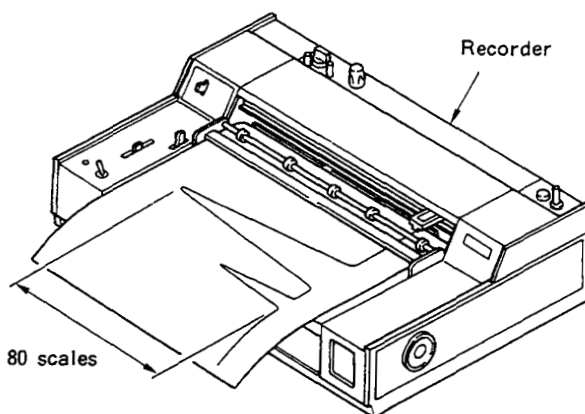
- (2) Input **1**, **6** and **Enter**

balance	RI (10^{-6})	range	mode
0	0	16	A

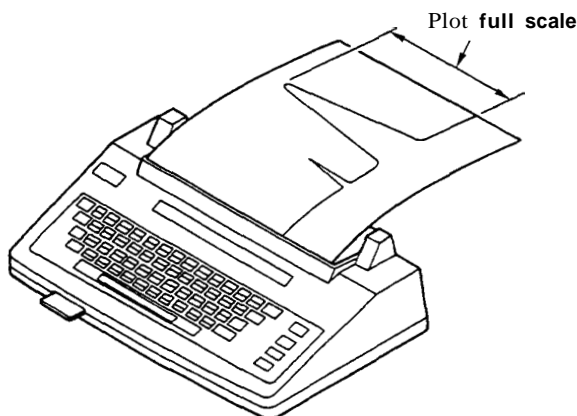
- (3) After a value is input, it returns to the initial state.

7. Changing the range

- (1) When a recorder is connected to the **RECORDER** terminal, set the range at a value about 1.2 times as much as the expected maximum peak value. Maximum peak will be about 80% of the full scale ensuring that peaks remain on-scale.



- (2) When a Chromatopac (integrator) is connected to the **RECORDER** terminal (10mV terminal), peaks having refractive index up to about 100 times as much as the setting range can be recorded. Set the measurement range at a value about 1/80 of the expected maximum peak refractive index. Normally, the recorder range is set at a value of about from 1 to 10 and the plot full scale is adjusted by changing **ATTEN** on the Chromatopac side. The relationship of the setting range and attenuation of Chromatopac (**ATTEN**) for the plot full scale when connected to the **RECORDER** terminal (10mV) is as follows:



$$\text{Plot full scale} = \text{Setting range} \times 2^{\text{ATTEN}} / 10 [\times 10^{-6} \text{RUFS}]$$

(Example)

When that range = 1, ATTEN = 2 :

Plot full scale = $1 \times 2^2/10 = 0.4$ [$\times 10^{-6}$ RIU/FS]

- (3) When a Chromatopac is used as a recorder, connect it to the INTEGRATOR terminal and set the range at ATTEN on the Chromatopac side. It is also necessary to make a rough range setting on the detector side since the dynamic range of the detector is extremely wide. This setting is made by setting a value in the parameter AUX RANGE. Relation between AUX RANGE value and INTEGRATOR terminal output are listed in the table below:

AUX RANGE value	INTEGRATOR terminal output	Remarks
1	1×10^{-4} RIU/V	
2	1×10^{-3} RIU/V	Compatible with AUX-L of RID-6A
3	1×10^{-2} RIU/V	
4	2.5×10^{-4} RIU/V	Compatible with AUX-H of RID-6A

Plot full scale for Chromatopac determined by AUX RANGE value and ATTEN value is as follows :

**Plot full scale refractive index ($\times 10^{-6}$ RIU/FS)
determined by AUX RANGE value and ATTEN value.**

	AUXRANGE ($\times 10^{-6}$ RIU)			
ATTEN	1	2	3	4
0	0.1	1	10	0.25
1	0.2	2	20	0.5
2	0.4	4	40	1
3	0.8	8	80	2
4	1.6	16	160	4
5	3.2	32	320	8
6	6.4	64	640	16
7	12.8	128	1280	32
8	25.6	256	2560	64
9	51.2	512	5120	128
10	102.4	1024	10240	256

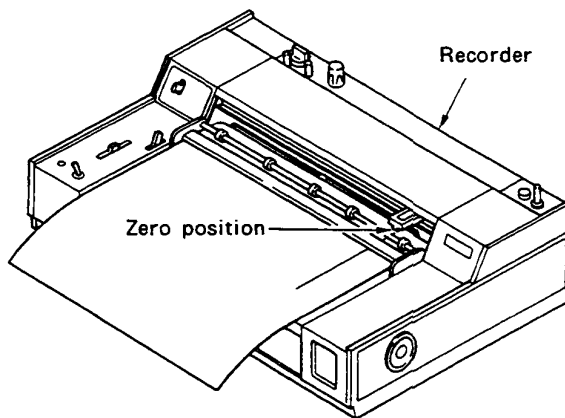
(Example)

When AUX RANGE = 2 and Chromatopac ATTEN = 7, the plot scale becomes 128×10^{-6} RIU.



8. Zero adjustment of recorder Before starting analysis, adjust the zero position of the recorder as follows:

- (1) Set the measuring range to 0 to short-circuit the recorder output. SHORT is displayed in the range display. (Refer to section 5.2.6, Setting the Range.)
- (2) Adjust the pen position to 0 scale on the chart paper using the pen position recorder adjusting knob.
- (3) Reset the measuring range at a value required for the analysis.
- (4) Press **[zero]**, and the pen returns to almost 0 scale on the chart paper.
- (5) Press **[zero shift $\Delta \nabla$]** to move the baseline to a desired position and start analysis.
- (6) Press the **[zerc]** key to return the baseline to the previous position.



Operation

9. Setting the RESPONSE

In this equipment, a digital noise filter is used to improve S/N ratio. Response improves when setting the filter response low, but the noise reduction effect becomes small. On the contrary, response is worsened when setting the response high, but the noise reduction effect becomes large. Ten digital filter response steps are available when setting the parameter to **RESPONSE**. **RESPONSE** values and corresponding time constants for analog filters are as shown in the table below:

RESPONSE value	Corresponding time constant of analog CR filter (Letters in brackets are response of RID-6A.)	Minimum peak width at half-height
1	0.05 sec	0.2 sec
2	0.1 sec	0.4 sec
3	0.5 sec (FAST)	2.2 sec
4	1.0 sec	4.8 sec
5	15 sec (STD)	7.2 sec
6	3.0 sec (SLOW)	13 sec
7	6.0 sec	26 sec
8	8.0 sec	36 sec
9	10.0 sec	45 sec
10	2.0 sec	9 sec

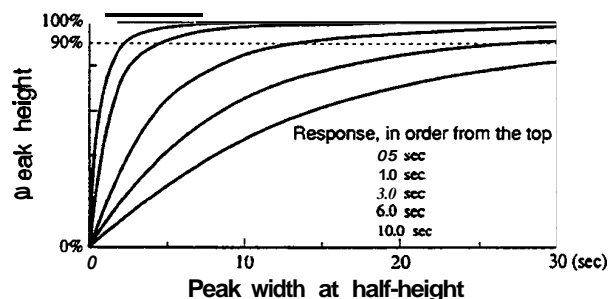
6^{res}

RESPONSE 5

- ② Input a value using numeral keys and press **Enter**.
- ③ Press **CE** to return to the initial display.

(Note) As response is increased, **data** processor response decreases, peak height decreases, and width at half-height increases. It is recommended that response be set at a value such that, for a given half-height width, the peak height drops no more than 10%. The relation between response time, **peak** half-height width, and peak height reduction is shown in the figure below.

To select a response using the figure, determine the width at half-height of the narrowest peak of interest. Using the graph, find the point of intersection between that width value and the **90%** height value. Set the response which corresponds to a value \leq the time constant **as** read from the graph. Note that response has no effect on peak **area**. **Peak area** does not change even when a low response value broadens the peak.



When the RID-10A is equipped with the flow selection block (option), measurement mode can be selected for high sensitivity analysis, preparative and large-scale preparative mode. Operation in the large scale preparative mode requires the optional block.

Note

Mounting of the flow selection block is performed by a service engineer. Refer to section 12.3.

1. Usage of each mode

Mode	Setting	Application
Analytical	A	For general analysis. The flow lines are the same as that of the RID-10A without the flow selection block. This mode is compatible with the RID-6A.
Preparative	P	For measuring samples of high concentration. Samples ten times as concentrated as those for mode A can be measured.
Large-scale preparative	L	For measuring samples of high concentration at large flow rate. Samples of the same concentration as those of the mode P can be delivered at 150mL/min.

Measurement mode	Refractive index measuring range ($\times 10^{-6}$ RIU)	Flow rate range (mL/min)	Tubing connection
Analytical (A)	— 500	20	Fig.1
Preparative (P)	—5000	20	Fig.1
Large-scale preparative (L)	—5000	150	Fig.2

2. Changing the measurement mode
(1) Selection of mode setting

The current mode is displayed on the LED display. Switch the measurement mode by pressing **shift** and **mode**.

↓ A : Analytical mode

↓ P : Preparative mode

↓ L : Large-scale preparative mode

Mode setting must be accompanied with corresponding tubing connection as shown in Figs.1 and 2.

Caution

Drain tube
Use the following types of **drain** tube for large-scale preparative mode. The longer and/or smaller I.D. tubing than the specified one causes **high back** pressure beyond the value in the specifications of the detector, which may lead to damage of the detector.

Max flow rate (mL/min)	Drain tubing O. D. x I. D. x Length [mm]	For extension O. D. x I. D. x Length [mm]
50	1.6 × 0.8 × 1000	32 × 16 × 1000
150	3.2 × 1.6 × 1000	32 × 16 × 1000

Note

When a fraction collector FRC-10A is used, use the following tubing.

Max flow rate (mL/min)	FRC-10A inlet tubing O. D. x I. D. x Length [mm]	FRC-10A outlet tubing O. D. x I. D. x Length [mm]
50	16 × 0.8 × 1000 (FRC-10A accessory)	3.2 × 1.6 × 1000 (FRC-10A accessory)
150	3.2 × 1.6 × 1000	3.2 × 16 × 1000 (FRC-10A accessory)

When using a fraction collector made by other manufacturer, check that the back pressure caused by connecting the fraction collector does not exceed the limit that the detector can resist. Pay attention to pressure change, as well as back pressure when switching the preparative valve.

- (2) Changing the tubing connection
Open the front panel and change the piping connection because large-scale preparative mode has different plumbing.

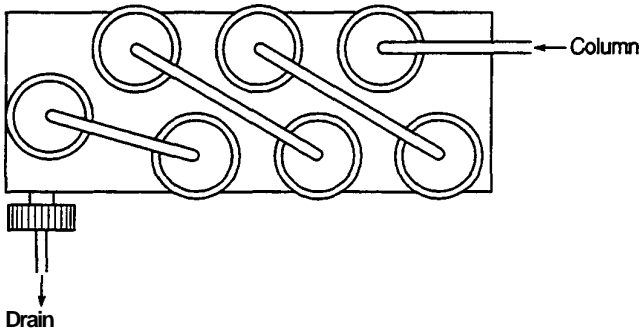


Fig. 1 Tubing connection in the analytical and preparative modes.

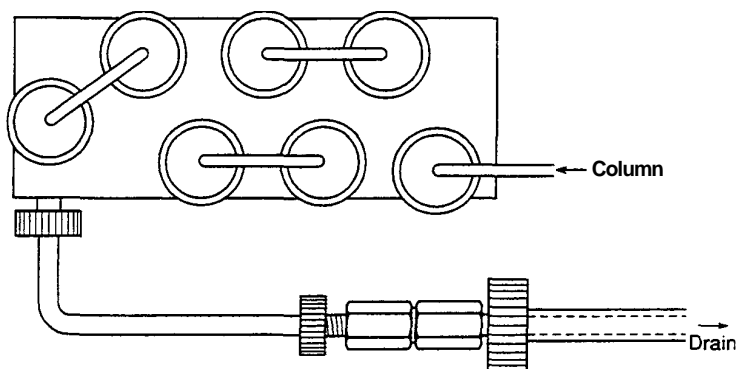


Fig. 2 Tubing connection in the large-scale preparative mode

Note

Do not set the instrument to mode L with analytical **and** preparative tubing connection. Similarly, do not set the instrument to mode A or P in large-scale preparative tubing connection.

5

**Op
tion**

1. Command list

Commands which can be used for a time program are listed below.

Command	Description	Setting range	Remarks
ZERO	Execution of zero adjustment	Not applicable	
MARK	Marking on output for recorder	Not applicable	
RNGA	Designation of output range for recorder (mode A)	0, 0.01 ~ 500	Unit: $\times 10^{-6}$ RIUFS, Recorder is short-circuited when set to 0.
RNGP	Designation of output range for recorder (mode P) Designation of output range for recorder (mode L)	0, 1 ~ 5000	
RESP	Designation of response	1 ~ 10	Refer to section 5.2. Fundamentals of Operation.
EVNT	EVENT output ON/OFF	0, 1, 2, 12	Refer to section 5.5, Additional Functions.
POL	Polarity setting	0, 1	Positive polarity Negative polarity
LOOP	Repeats all preceding steps in the program.	0 ~ 255	Value 0 repeats a program 256 times.
STOP	Ends a program.	Not applicable	

Note

Setting the RNGA at decimal number

RNGA enables increments of 0.01 unit steps. However, when set at a range more than 1.00, fraction of 5 and over is counted as unit and the rest is omitted.

(Example)

RNGA (setting value)	RNGE (execution value)
1.40	1.00
1.50	2.00

Note

Execution of RNGA and RNGP

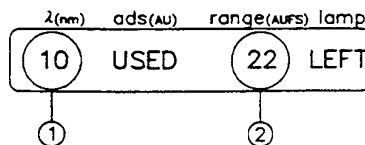
RNGP setting during a time program is ignored in mode A.

RNGA setting during a time program is ignored in mode P or L.

2. Explanation of display screen

The edit mode is used to create a time program.

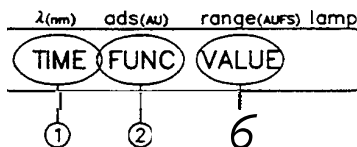
- (1) Press **shift** and **edit**. A screen similar to the one below will be displayed.



- ① Number of steps already set
 ② Number of remaining steps

The above example **shows** that the time program is set for 10 steps and there are **22** remaining steps.

- (2) Press **Enter** to display the following:



- ① Elapsed time from time program start (minute)
 ② Commandname
 ③ Set value

- (3) Press **Enter** again, to display the following:

The contents in the display are the same **as** those in (2)



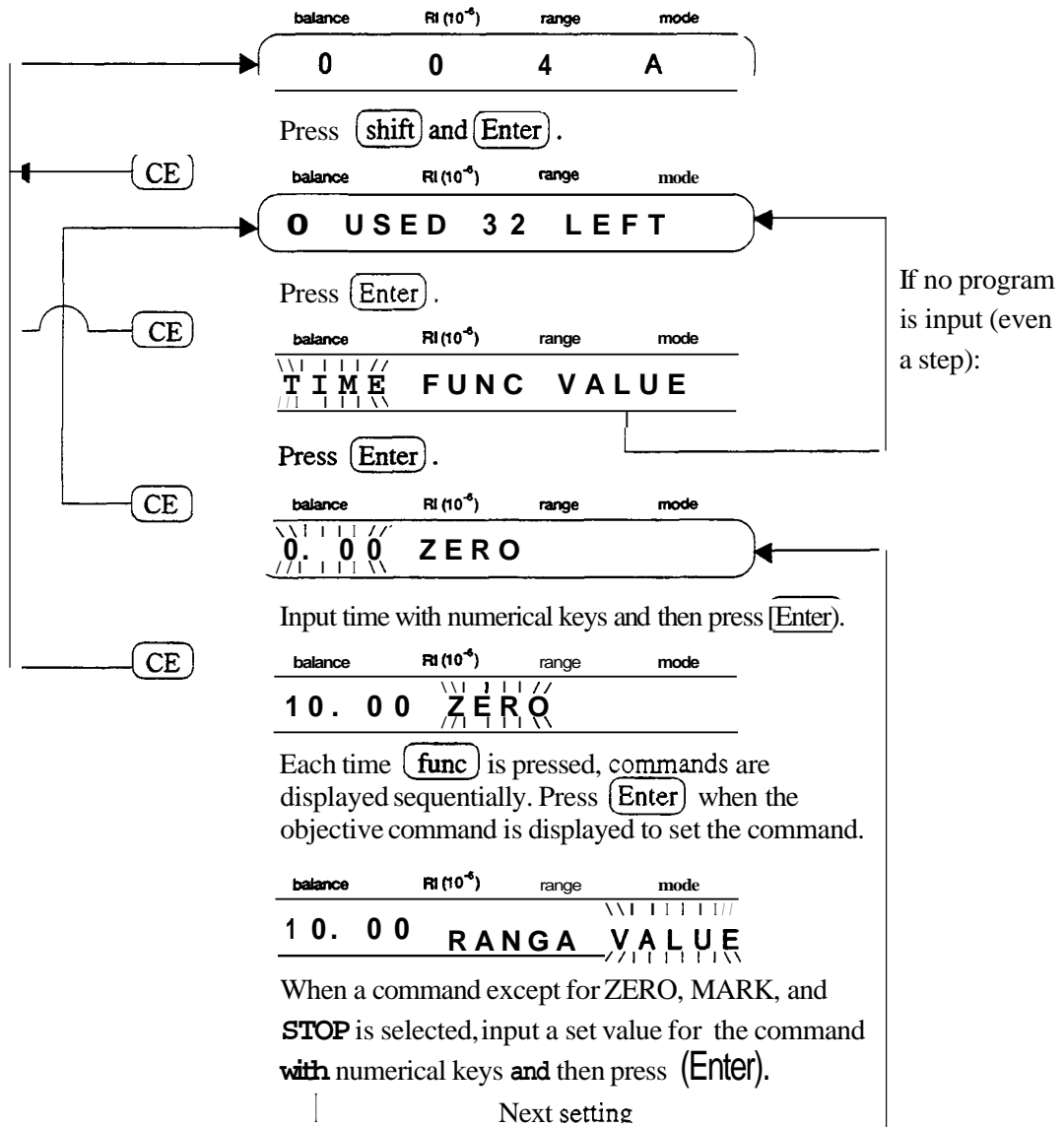
The above display shows that **AUTO ZERO** is activated one minute **after start** of the time program.

3. Flow of setting

Flow in setting a time program is shown below :



Operation



Operation of **shift** and **func** in this state returns the display to the previous setting.

Note

- It is unnecessary to set the time order of steps as they are automatically rearranged.
- Set a **STOP** command at the end of a program unless the program is to be operated continuously.
- When selecting a function, the previous function is displayed by pressing **shift** and **back**.

4. Deleting a step (Example)

Display the step to be deleted and press **shift** and **del**.
Deleting the first step in the program set in the previous section.

- (1) Display the step to be deleted.

balance	R1 (10 ⁻⁴)	range	mode
5.00		ZERO	

- (2) Press **shift** and **del**.

balance	R1 (10 ⁻⁴)	range	mode
20.00		RANGA	2 1

The first step in the program is deleted and the second step is displayed. When the second step is not set, the following is displayed:

balance	R1 (10 ⁻⁴)	range	mode
0		USED 3 2	LEFT

5. Start and stop

After setting a time program, start and stop the program according to the following:

- (1) Press **run**. The LED for **prog run** is lit, and the time program starts.
- (2) There are two methods to stop a time program: forced stop of a time program in process, and stop of a time program using the **STOP** command in the program. To stop the time program forcibly, Press **run** again. The LED for **prog run** is **dimmed**, and the program stops.



Operation

6. LOOP command

A program can be repeated any number of times using a LOOP command.

TIME	FUNC	VALUE
15.00	ZERO	
20.00	MARK	
30.00	LOOP	3

According to the above setting, steps (1) and (2) are repeated **3** times at an interval of **30** minutes.

Note

- When parameter is changed while executing a time program, the changed value is valid only until the end of the program. On completion of the time program, the parameter is reset to the value set before executing the time program.
- **Any** number up to 255 can be set to the **VALUE** for the LOOP command. When 0 is set for the value, LOOP is repeated 256 times.
- The program ~~that was~~ set after ~~the~~ **LOOP** command is not executed. The program stops at the end of the LOOP command.



Option

The following Additional Functions are equipped with the RID-10A.

Type	Command	Function
4	TOTAL EN	Indicates the total quantity of light irradiated on the photodiode.
4	CELL	Displays the quantity of light irradiated on each of the right and the left side of photodiode.
1	CELL TEMP	Sets the cell temperature.
4	ACT TEMP	Displays the actual temperature of the optical unit.
1	AUX RANGE	Sets output range to μ integrator.
1	EVENT	Controls output relays on the back of the RID-10A.
1	EXT-S	Sets control mode for the control of external equipment through the EVENT output relays.
1	MONIT TIME	Monitors when running a time program.
4	LAMP TIME	Displays the lamp lit time.
3	CLOSE KEY	Locks the keypad, preventing unwanted entries.
1	ADRS	Sets address of the RID-10A for control via system controller.
1	LOCAL	Selects independent operation or control via system controller.
1	LAMP VOLT	setting of the lamp voltage
1	SPAN A	Inputs the span value of the analysis mode.
1	SPAN P	Inputs the span value of the preparative mode
1	SPAN L	Inputs the span value of large-scale preparative mode (An optional flow selection block is necessary.)

“Type” in the above table indicates a kind of operating procedure.

Type 1 : Input a value using numerical key and then press **Enter** key.

Type 3 : Press **Enter** key to execute the function.

Type 4 : The status is displayed.

2. Setting procedures for AUX. FUNC

TOTAL EN

(Total lamp energy)

balance	RI (10 ⁻⁴)	range	mode
TOTAL EN		7 0 0 0	

Displays the total quantity of light irradiated into the photodiodes.
(unit : mV)

CELL

(Lamp energy of each photodiode.)

balance	RI (10 ⁻⁴)	range	mode
CELL	3 5 5 0	3 4 5 0	

Displays the quantity of light irradiated into each of the right and left photodiode. (unit : mV)

CELL TEMP

(Cell temperature setting)

balance	RI (10 ⁻⁴)	range	mode
(CELL TEMP 40.0)			

Sets the cell temperature at the measuring unit.

When the flow rate is larger than 3 mL/min, turn this **OFF**.

When the flow rate is less than 3 mL/min, set the temperature to the room temperature + 12°C

ACT TEMP

(Monitors actual cell temperature)

balance	RI (10 ⁻⁴)	range	mode
(ACT TEMP 40.0)			

Displays the actual cell temperature in the optical unit.

AUX RANGE

(Integrator output full scale)

balance	RI (10 ⁻⁴)	range	mode
(AUX RANGE —)			



Operation

1	1×10^{-4} RIU/V	
2	1×10^{-3} RIUN	Compatible with AUX-L of RID-6A
3	1×10^{-2} RIU/V	
4	2.5×10^{-4} RIU/V	Compatible with AUX-H of RID-6A

EVENT

(Event output terminals)

balance	RI (10 ⁻⁴)	range	mode
(EVENT —)			

This function **sets** the relays **accessed** by the EVENT output terminals on the back of the RID-10A. **OFF** is an open relay and ON is closed.

Input the desired value and press **Enter**.

Set value	EVENT1	EVENT2
0	OFF	OFF
1	ON	OFF
2	OFF	ON
12	ON	ON

EXT-S

(External starts, sets specific functions for the EVENT output terminals)

balance	R1 (10 ⁻⁴)	range	mode
EXT — S —			

This feature is typically used to control external equipment through the **EVENT** output (relay 1 and 2).

Input the desired value, then press **Enter**.

Set value	Control mode
0	Relay contact points are controlled by the EVENT set value.
1	Relay 1 (EVENT 1 terminals) closes at the start of a time program. Useful starting data processors or zeroing detectors.
2	Relay 2 (EVENT 2 terminals) closes on detection of the error of the RID-10A. Useful for communicating to external equipment
3	Both functions described above are enabled.

MONIT-TIME

(Monitors program elapsed time)

balance	R1 (10 ⁻⁴)	range	mode
MONIT-TIME —			

When **running** a time program, the time elapsed from the **start** of the program is displayed.

Input the desired value, and press **Enter**.

Set value	Function
0	Cancels display of elapsed time.
1	Sets display of elapsed time.

When setting this function, the time program display during operation is as follows:

balance	R1 (10 ⁻⁴)	range	mode
XXX	△△△	T	□□. □□
Elapsed time from the start of the program.			
balance	R1 (10 ⁻⁴)	range	mode
LAMP TIME 150			

LAMP TIME

(Monitors the lamp lit time)

Displays the integration time of the lamp lighting. (unit : hour)

The life of a lamp is 20,000 hours. When the lamp life is close to the end, contact your Shimadzu Service Representative.

CLOSE KEY

(Prohibition of key input)

balance	RI (10 ⁻⁴)	range	mode
C L O S E K E Y			

This function locks keypad, preventing entry, and is invoked by pressing **Enter** when the above display is shown. To restore keypad function, press **CE** and **a** simultaneously.

ADRS

(Remote address)

balance	RI (10 ⁻⁴)	range	mode
A D R S		3	

Sets a remote address setting (port No.) when this equipment is connected to the system controller (SCL-10A) for use. Input the address number, then press **Enter**.

For port No., see section 7.1, Connection with System Controller SCL-10A.

LOCAL

(Setting of the local mode)

balance	RI (10 ⁻⁴)	range	mode
L O C A L		—	

Selects independent control or control via an SCL-10A system controller. A set value 1 allows the RID-10A to be controlled from its own keypad even when connected to an SCL-10A.

Input the desired value, and then press **Enter**.

Set value	Function
0	Allows control via an SCL-10A.
1	Independent operation (local mode).

LAMP VOLT

(Lamp voltage)

Sets a lamp voltage. (Input range : 0 to 5.00)

Normally, input the value such that value of TOTAL EN is within the range from 6,000 to 9,000.

SPAN A

(Span factor in mode A)

balance	RI (10 ⁻⁴)	range	mode
(S P A N A		1. 0 0	

Inputs the span value of the analytical mode.

SPAN P

(Span factor in mode P)

balance	RI (10 ⁻⁴)	range	mode
(S P A N P		1. 0 0	

Inputs the span value of the preparative mode.



Operation

SPAN L

(Span factor in mode L)

balance	Rt (10 ⁻⁴)	range	mode
S P A N	L	1 .	0 0

Inputs the span value of large-scale preparative mode.

Large-scale preparative mode is applicable when the flow selection block option is installed.

**Operation**

Chapter 6 Initial Performance Test

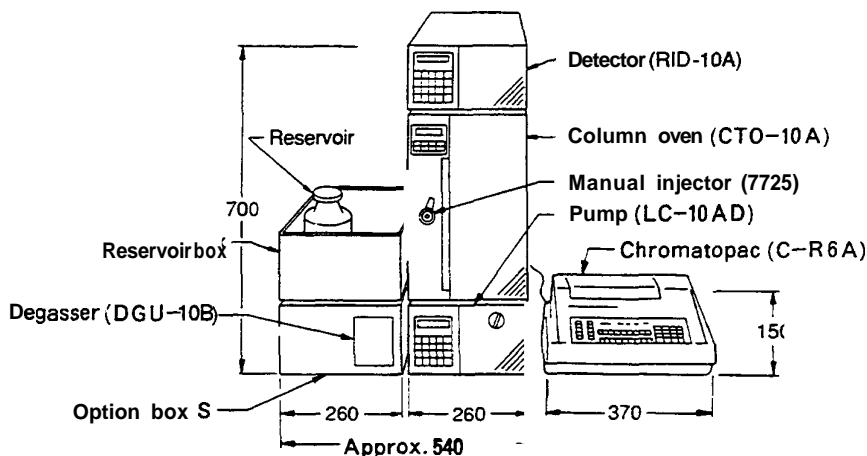
6

Initial Performance Test

CONTENTS

6.1	System Performance Check	6-2
-----	--------------------------------	-----

This chapter contains an explanation of the operation check for the simple system in the **figure** below.



1. Preparation

- (1) Prepare **required** reservoirs.
- (2) Prepare mobile phase. (Refer to section 4.)

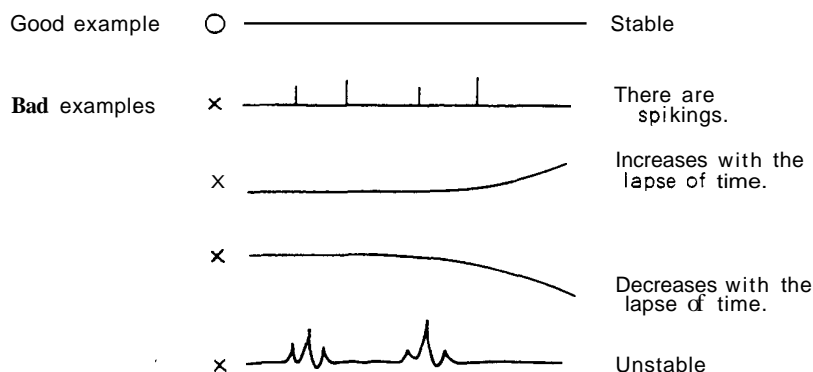
2. Connection

- (1) Connect the input cable of the Chromatopac to the **INTEGRATOR** output terminal of this equipment **using** the signal cable supplied to this equipment through the terminal block supplied to the Chromatopac.

3. Operation

- (1) Turn ON the system power.
- (2) **Open** the drain valve of the pump **and** purge the entire flow line to prime the pump. Suck out about 40mL using the syringe.
- (3) Close the **drain** valve and operate the pump at 1 mL/min.
- (4) Verify that pump pressure is stable **and** mobile phase is flowing **from** the outlet.
- (5) **Set** the temperature of CTO-10A to 40°C.
- (6) Set parameter AUX **RANGE** of RID-10A to 2. (AUX output range becomes 1×10^{-3} RIU/V.)
- (7) Set the Chromatopac ATTN to 4. (Plot full scale of the Chromatopac becomes equivalent to 16×10^{-6} RIU.)

- (8) Press **[ZERO]**, **[5]**, **[0]** and **[Enter]** on the Chromatopac to center the pen Position.
- (9) Press **[PLOT]** and **[Enter]** on the Chromatopac to plot the output.
- (10) Wait till the baseline becomes stable.



- (11) Set ATTN to 8 on the Chromatopac. (Chromatopac full scale becomes equivalent to about 256×10^{-6} RIU)
- (12) Press **[ZERO]**, **[2]**, **[0]** and **[Enter]** on the Chromatopac.
- (13) Press **[PLOT]** and **[Enter]** on the Chromatopac to stop plotting.
- (14) Inject sample to the injector.
- (15) Press **[START]** of the Chromatopac simultaneously with the sample injection.

4. Example of analysis result

An example of analysis under the following conditions is shown below.

Mobile phase : Acetonitrile/Water = 75/25

Column : Asahipak NH2P-50 4.6mm ϕ x 25cm

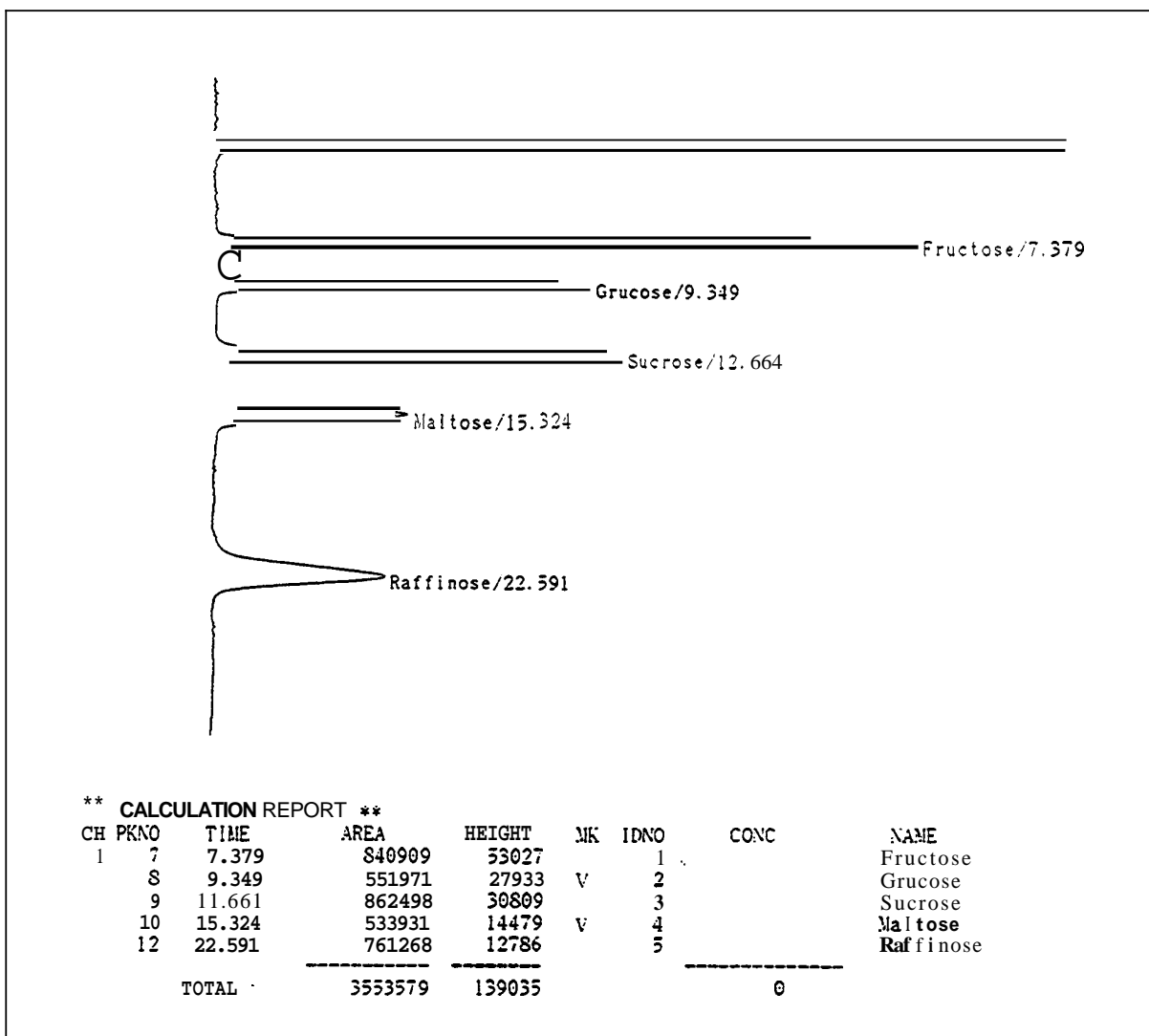
Flow rate : 1 mL/min

Column temp : 40°C.

Sample :	Fructose	100 mg
	Glucose	100 mg
	Sucrose	100 mg
	Maltose	100 mg
	Raffinose	100 mg

in 100mL water, 10 μ L injection
--

(Example of chromatogram)



Initial Performance Test

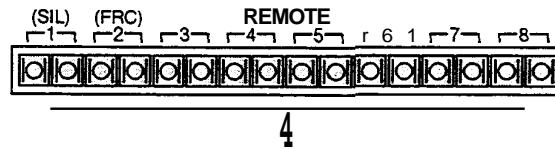
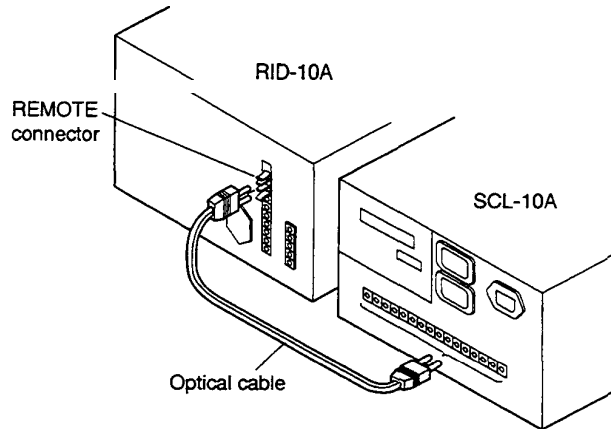
Chapter 7 Control from the External Equipment

CONTENTS

7.1	Connection with System Controller SCL-10A	7-2
7.2	Control from the SCL-10A	7-3
7.3	Connecting External Input and Output Terminals	7-10

1. Preparation

- (1) Using the supplied optical cable, connect the REMOTE connector at the rear panel of the RID-10A and the REMOTE connector of the SCL-10A

**Caution**

Do not bend the remote control optical cable at less **than** a 120° angle.

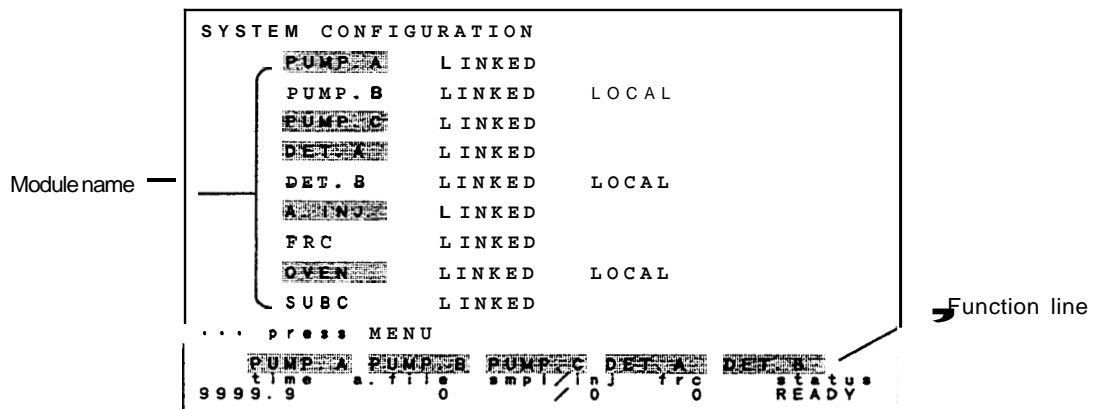
- (2) Set a value for ADRS in AUX. FUNC. Input the **port** No. of the SCL-10A to which the optical cable is connected to the parameter **ADRS** of this equipment.

Refer to the SCL-10A System Controller Instruction Manual for basic operation of the system controller.

1. Power ON and CONFIG screen

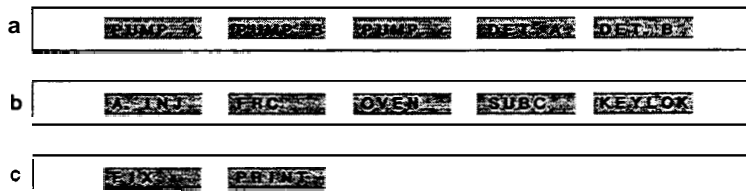
Turn ON the SCL power switch to display the CONFIG screen. When the detectors and the SCL are properly connected, **LINKED** is displayed at the right side of both **DET.A** and **DET.B** (when two detectors **are** connected).

Equipment connection for controlling the detectors by SCL is defined on this screen. Highlighting the name of the unit defines it as being present and the system controller will perform a link check upon power up. Unit definitions are displayed only once after the initial installation. These definitions are stored in the SCL and are required **only** once unless the system configuration is changed.



Define the detector connection according to the following procedure :

- (1) Display line a in the function line as in the following figure. When b is displayed in the function line, press **func** once to change the display.



- (2) Select the detector connected to the function key and the equipment name is highlighted on the screen.

Equipment of **LOCAL** display **does** not accept control by SCL even when its name is highlighted. Refer to section 5.5, Additional Functions to change the local mode to the remote mode.

2. Main menu screen

Press **(menu)** in the **CONFTG** screen to display the main menu screen. Press **(menu)** to access the main menu from any screen .

```

      MENU
ANALYSIS FILE PARAMETERS : 0
      TIME PROGRAM : 1
      PUMP CONTROL : 2
      DET CONTROL : 3
FRACTION COLLECTOR : 4
AUTO INJECTOR/ANAL SEQUENCE : 5
MONITOR : 6
SYSTEM : 7

time 9999.9 a. file 0 smpl / inj fr 0 KEY-LOCK status
READY

```

3. Setting initial parameters

Press numeric key 0 in the main menu screen or move the cursor to the number 0 and press **(Enter)** to display the parameter screen for analysis file.

This screen also displays parameters for equipment other than detectors. These parameters can be reset.

Control from the External Equipment

Solvent delivery unit

Solenoid valve

Contact output

Multiple terminal box

Column oven

Detector

Sub-controller

Help display

By pressing **(func)**, the setting range for the highlighted parameter can be displayed on the screen.

```

ANALYTICAL PARAMETERS
FILE 0
PUMP-----
( ISO. )
A. FLOW 0.000 ml/min
B. FLOW 0.000 ml/min
C. FLOW 0.000 ml/min
A. PRES 0 kg/cm²
B. PRES 0 kg/cm²
C. PRES 0 kg/cm²
P. MAX 100 kg/cm²
P. MIN 0 kg/cm²
SV 0
EVENT 0
POWER 1
OVEN. T 40 °C
T. MAX 85 °C
RV. A 0
RV. B 0
RV. C 1
RV. D 1
SVR. SI 1 µl/s
SVR. SO 1 µl/t
DEGAS 0
-----DET. A-----
RANGE 100.00
POL 1
CELL. T 40.0
-----DET. B-----
WAVE 195 nm
RANGE 2.5600 AUFS
WAVE2 200 nm
A. FLOW 0.9999 (ml/min) STEP 0.001
time 9999.9 a. file 0 smpl / inj fr 0 KEY-LOCK status
READY

```

When detectors are connected, the control parameters for the detectors are displayed in **DET.A** and **DET.B** as in the previous figure.

Other parameters necessary for the detector are displayed and set in the **DET CONTROL** screen.

4. DET CONTROL screen

Press **| 3 |** in the **main** menu screen to select the **DETECTOR CONTROL** screen.

The detector parameters, which cannot be input on the **ANALYTICAL PARAMETERS** screen, can be input on the **DETECTOR CONTROL** screen. These detector parameters control the operation of the detector from the **SCL** (described in section 5.2, Fundamentals of Operation).

Press **act** on the SCL keyboard to turn ON the **activate** lamp. When the **activate** lamp is lit, the parameters set in this screen are sent to the detector.

(Example)

If the **POL** parameter is set to -1, the **LED** for polarity on the detector is lit and the polarity is reversed.

```

DETECTOR CONTROL
FILE 0
-----DET. A-----
      (RID)
RANGE 100.00
RESP 5
POL -1
CELL. T 40 °C
MODE 0
AUX RNG 3

POL + (1) - (-1)
9999.9 a. t i l e s m p l / 0 f r c s t a t u s
READY
  
```

In the **DETECTOR CONTROL** screen above, where the **RID-10A** is connected as detector A, the type and parameters for one detector are displayed.

When no detector is connected, the following display appears in the center of the screen.

DETECTOR IS NOT CONNECTED

When two detectors are connected, use the function keys **DET.A** and **DET.B** to switch between detectors.

- Press **func** to change the function display to the following display:

DISP. MARK-A MARK-B A/B MARK KEYLOCK

- (2) Using function key **A/B** (**f4**), select the detector where parameters are to be set. This is only necessary when two detectors (e.g., A and B) are connected. When a detector B is selected and only one detector is connected, **DETECTOR IS NOT CONNECTED** is displayed.
- (3) Move the cursor to select the parameter with the cursor keys (**↑**) and (**↓**), input a desired value, and press **Enter** . Set and select the range and minimum parameter steps after accessing the help line with the **func** key.
- (4) To set the parameters for two detectors, refer to item (2) above. Parameters which can be set in the DET CONTROL screen are listed in the table below:

■ Parameter summary

Parameter	Description	Setting range	Minimum Unit	Default value
RANGE	Range for recorder in mode A ($\times 10^{-6}$ RIU)	0, 0.01-500	(0.01-1.00) 0.01 (1-500) 1	100
	Range for recorder in modes P and L ($\times 10^{-6}$ RIU)	0, 1-5000	1	100
RESP	Response	1-10	1	5
POL	Polarity	-1 or 1		1
CELL.T	Cell temperature setting	0 (=OFF), 30-60	0.1	40
MODE	Measurement mode	0-2	1	0
AUXRNG	AUX range	1-4	1	2

- (5) The following operations are possible in the detector control screen using the function keys.

PRINT **CLEAR** **COPY** **ZERO_A** **ZERO_B**

PRINT (**f1**)

Outputs detector parameters being displayed to the printer of the Chromatopac (requires the current loop interface).



Resets the parameters (including parameters for other devices such as solvent delivery unit, etc.) in the file No. being displayed to the initial values.

COPY (**f3**)

Copies parameter values **from** the currently displayed file to another file.

ZERO_A (**f4**)

Performs zero adjustment of a detector **A**.

ZERO_B (f5)

Performs zero adjustment of a detector B.



MARK_A (f2)

Sends marker signal to the recorder connected to the detector A.

MARK_B (f3)

Sends marker signal to the recorder connected to the detector B.

A/B (f4)

Toggles between the detector A or B for parameter display.

KEYLOK (f5)

Disables acceptance of any key input except for **KEYLOK** (lock release).

6. Time program

After setting the initial parameters in the **DET CONTROL** screen, press **menu** to return to the main menu to select **TIME PROGRAM**. In the time program screen, similar program to the time program is set on the SCL side (refer to section 5.4, Creating and Editing Time Programs). In the time program set in this screen, commands for the detector as well as other instruments are included, e.g., solvent delivery unit, etc.

Program input line

TIME PROGRAM				300 STEPS LEFT	
FILE	0	(B. GE)			
#	TIME	FUNC	VALUE		
0	30.00	B. CONC	100		
1	50.55	RANGE A	4		
2	6.000.0	STOP			
3					
4					
5					
6					
7					
8					
9					
FUNCTION FOR (PUMP) DET. A DET. B OTHER					
31	RESP	34 CELL. T	44 R. CLOS	46 RNG. A	
32	MARK	35 ZERO	45 R. FLOW	47 RNG. P	
33	POL	43 BALANC			
A. FLOW 0 - 9.999 (ml/min) STEP 0.001					
9999.9 a. fill 0 smp / 0 n 1 r 0 status					
READY					

Command table

■ Operation

- When no program is set and the cursor flashes in the **TIME** column in step 0 (see example above), input the desired time for the start of a command and press **ENTER**. The cursor then moves to the item FUNC.
- Press **func** to change the function to the following display:



- (3) Move the cursor to a desired device name in the following line using **TABLE←** (**f4**) and **TABLE→** (**f4**).

FUNCTION FOR (PUMP **DETA** DET. B OTHER)

Commands corresponding to the selected module are displayed in the command table. An example for the command table is shown below:

```

3 1 RESP | 3 4 CELL. T | 4 4 R. CLOS | 4 6 RNG. A
3 2 MARK | 3 5 ZERO | 4 5 R. FLOW | 4 7 RNG. P
3 3 POL | 4 3 BALANC |

```

■ Command summary for time program

Parameter	Description	Settingrange	Minimum Unit	Defaultvalue
RESP	Response	1-10	1	5
MARK	Marks on the recorder output.	--	--	--
POL	Polarity	-1 or 1	--	1
CELL. T	Cell temperature setting	0 (=OFF), 30-60	0.1	40
ZERO	Performs zero adjustment.	--	--	--
BARANC	Performs optical balance adjustment.	--	--	--
R. CLOS	Closes reference flow lines.	--	--	--
R. FLOW	Purges reference flow lines.	--	--	--
RNG. A	Recorder range in mode A ($\times 10^{-6}$ RIU)	0, 0.01-500	0.01 (0.01-1) 1 (1-500)	100
RNG. P	Recorder range in modes P and L ($\times 10^{-6}$ RIU)	0, 1-5000	1	100

The following operations are possible in the time program screen using the function keys.

PRINT **RESET** **COPY** **BASE** **TABLE**



PRINT (**f1**)

Outputs the time program on display to the printer connected to the Chromatopac.

CLEAR (**f2**)

Erases all of the time program in the file being displayed.

COPY (**f3**)

Copies the file being displayed to a different **file**.

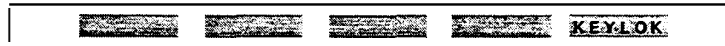
TABLE← (**f4**)

Moves the cursor at the device name in the command table to the left.

TABLE→ (**f5**)

Moves the cursor at the device name in the command table to the right.

Press **func** to change the function to the following display:

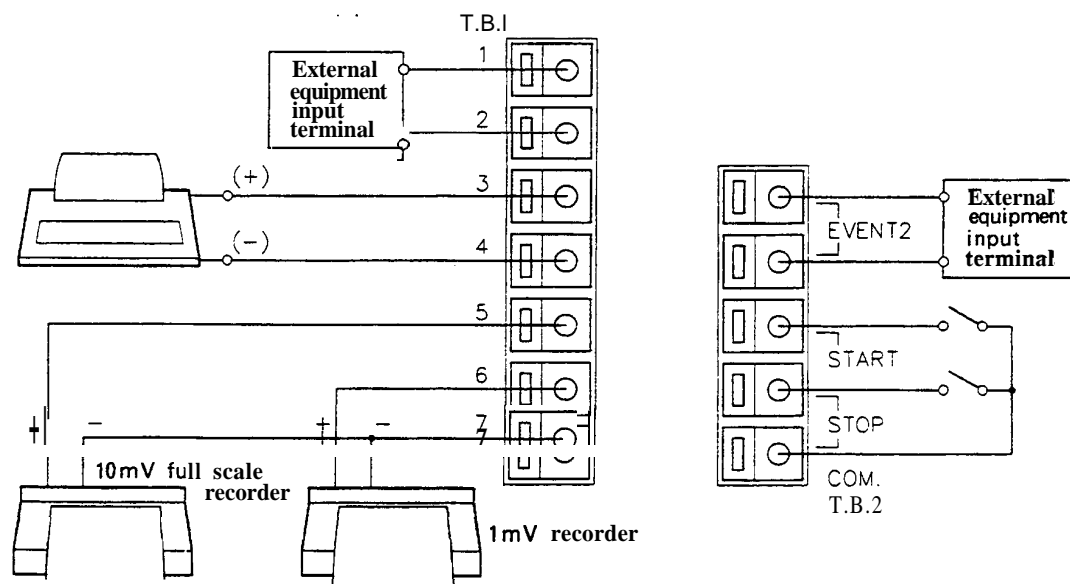


KEYLOK (**f5**)

Disables acceptance of any key input except **KEYLOK** (lock release).



The two external input and output terminals are T.B. 1 and T.B. 2.



T. B. 1

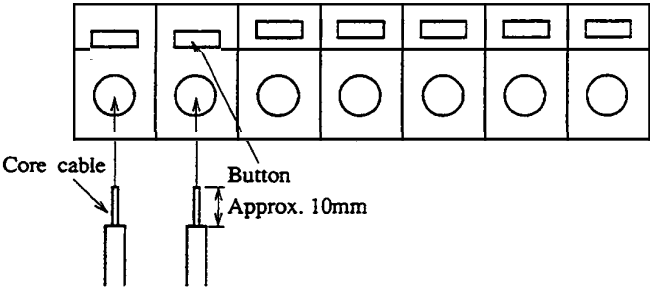
PIN.No.	Signal name	Explanation
1, 2	EVENT1 output terminal	Outputs relay contact. It is turned ON/OFF according to the program or the set value of EVENT parameter in AUX. FUNC.
3 (+) 4 (-)	INTEGRATOR output terminal	Signal output terminal connected to Chromatopac. (1V F.S.)
5 (+10mV F.S.) 6 (+1mV F.S.) 7 (-COMMON)	RECORDER output terminal	Signal output terminal connected to recorder.

Note) F. S. stands for full scale.

T. B. 2

Signal name	Explanation
EVENT2 Event output terminal 2	Relay contact It is turned ON/OFF according to the program or the set value of EVENT parameter in AUX.FUNC.
START input terminal	Time program is started by shorting this terminal with COM terminal.
STOP input terminal	Time program is stopped by shorting this terminal with COM terminal.
COM.	COM terminal for START and STOP terminals.

- (1) Strip the sheath of the connecting cable for approximately 10mm in **length** at the end. (It is unnecessary for supplied remote cable.)
- (2) When the core wire is solid, insert it into the hole of a terminal while pressing the button above the hole using a small screwdriver. When the wire is stranded, twist the end well and insert it into the hole while pressing the button above the hole using a small screwdriver. To disconnect the cable, press the button and remove the cable from the terminal.



Caution

A remote cable is supplied with the RID-10A. To protect the cable wire **from** breaking, use stranded cables. When connecting two or more circuits to the terminal, use the following wires with core diameter **as** indicated below:
Solid wire : $\phi 0.4\text{--}\phi 1.0$ (AWG 26-18)
Stranded wire : $0.3\text{mm}^2\text{--}0.75\text{mm}^2$ (AWG 22-20)
Element wire diameter : $\phi 0.18$ at the minimum



Control from the External Equipment

Chapter 8 Maintenance

CONTENTS

8.1	Cleaning the Flow Lines	8-2
8.2	Span Adjustment	8-3
8.3	Replacement of Fuse	8-7
8.4	Periodical Cleaning	8-8

Dirty cells and piping may cause phenomena such as an unstable baseline or a large baseline noise due to pulsation of the solvent delivery pump. In this case, clean the flow lines with the following procedures.

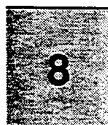
Item to be prepared	Qty
Syringe	1
Adapter	1
Water	100mL
Acetone	50mL
0.1N nitric acid	50mL

Mixing nitric acid with organic solvent may form an explosive substance. Be extremely careful when handling nitric acid.

- (1) Deliver acetone from the inlet port using the syringe and adapter. During supply, turn the Mow on^(*) to deliver solvent to the reference side.

(*) The state in which Rflow lamp is lit when **Shift** and **Rflow** is pressed.

- (2) Use distilled water to flush the flow lines as in step 1.
- (3) Flush the flow lines with 0.1N nitric acid.
- (4) Flush out the nitric acid with distilled water.
- (5) Replace with mobile phase. When the mobile phase used for analysis is not miscible with water, replace the flow lines with acetone and replace it with the mobile phase.



Span adjustment should be performed over each mode of A, P and L. When a Chromatopac is used, refer to 8.2.1, and when an LC workstation is used, refer to 8.2.2.

Inject the standard solution whose refractive index is known, and adjust the detector output so that the signal intensity can be verified. Following is the description for mode A span adjustment.

Items to be prepared	Qty
Chromatopac or LC workstation	
Syringe	
Adapter	
Water (HPLC grade)	100mL
Refractiveindex standard sample*	100mL

* Refractive index standard sample:

glycerin solution 0.872 g/L.

Dissolve 43.6 g of glycerin (USP grade or equivalent) in the 1L of water, and dilute this solution 50 times for the refractive index standard solution. (Reference document ASTM E1303-89) This sample shows difference of 100×10^{-6} RIU from that of pure water.

8.2.1 When span adjustment is performed using the Chromatopac

- (1) Turn ON the power.
- (2) Set the parameters of the detector as follows:

LOCAL	1 (*)
CELL TEMP	40
polarity	+
mode	A (When performing mode A span adjustment)

* When the RID-10A is controlled from the system controller SCL-10A: This adjustment is performed by the detector independently. First, input **LOCAL** = 1 to separate control (link) from the SCL. After that, input each set value using the numerical keys of the detector main unit

- (3) Set the detector so that the plot full scale of the Chromatopac becomes 128×10^{-6} RIU.

AUXRANGE of the detector	ATTEN of the Chromatopac
2	7
4	9

- (4) Flow distilled water from the inlet port using the syringe and the adapter.
Make Rflow on^(*) to purge the reference side flow lines with water.
(*) **Shift** and **Rflow** is pressed, and **Rflow** lamp is lit.
- (5) After the sample cell/reference cell inside is replaced with distilled water, make Rflow **OFF**.
- (6) When the baseline is stabilized, press **Shift** and **Balance** and perform optical balance adjustment.
- (7) Press **zero**, and record the baseline level when the cell is filled with distilled water.
- (8) Inject the standard solution from the inlet port. The baseline will move up ward by 80% of the full scale. Record the baseline level in case of standard solution.
- (9) Read the difference between the base line levels of the distilled water and of the standard solution, and convert the value into RI unit.

(Example)

When the base line moves 120 mm on the chart paper with the Chromatopac C-R7A, the difference value can be converted into RI unit with the of following formula.

$$128 \times 10^{-6} [\text{RIU}] \times \frac{120}{150} \text{ mm} = 102.4 \times 10^{-6} [\text{RIU}]$$

150 : Plot scale of the C-R7A (see following table)

- * When the Chromatopac is used, record the value by pressing the **PLOT** key. The plot scale is as follows:

Chromatopac model	Full scale on chart paper
C-R7A	150mm
C-R4A	160mm
C-R5A/R6A	135mm

- (10) As a result of measurement, when the refractive index of the standard sample is $R \times 10^{-6}$ RIU, the span factor can be calculated by the following equation:

$$[\text{SPAN}-A] (\text{new}) = [\text{SPAN}-A] (\text{old}) \times 100/R$$



(Example)

When the measured refractive index is 102.4×10^{-6} [RIU] and the old span factor is 0.90, the new span factor is:

$$0.90 \times 100/102.4 = 0.88 \leftarrow \text{New SPAN-A}$$

- (11) Input the new span factor obtained by the above formula into **SPAN-A**.
- (12) Inject distilled water from the inlet port and finish the span adjustment.
- (13) For span adjustment of mode P/mode **L**, change the mode setting in (2) above to **P** or **L**, and follow the procedures from (4) above. Input the span value obtained at each mode into **SPAN-P** and **SPAN-L** each.

8.2.2 When span adjustment is performed using the LC workstation

- (1) **Turn ON** the power of the RID-10A and LC workstation.
- (2) Set the parameters of the RID-10A as follows:

■ Example 1 (CLASS-VP)

Instrument Initial Conditions

Pump Conditions : Isocratic

A.FLOW 1.000 mL/min

B.FLOW 0.500 mL/min

C.FLOW 0.500 mL/min

A.PRES 100 Kgf/cm²

B.PRES 0 Kgf/cm²

C.PRES 0 Kgf/cm²

P.MAX 200 Kgf/cm²

P.MIN 0 Kgf/cm²

Other

SV 1 OVEN.T 40

EVENT 0 T.MAX 85

POWER 0

Detector A : RID-10A

MODE 0 RANGE 100.00

POL 1 RESP 5

CELL.T AUX.RNG

Detector B : RF-10AXL

EX.WAV 350 RANGE 1

EM.WAV 450 RESP 3

More

RID-10A parameters

■ Example 2 (CLASS-LCIO)

Detector RID-10A

Polarity ☒ + ☐ - Response 1.5

Measure Mode

☒ 0: Analytical

☐ 1: Preparative

☐ 2: Large

AUX RANGE

☐ 1: 1.0E-7 RIU/mV

☒ 2: 1.0E-6 RIU/mV

☐ 3: 1.0E-5 RIU/mV

☐ 4: 2.5E-7 RIU/mV

Cell Temp.(C) 40

Cell Temp.(C) 40

OK Cancel Help

- (3) Change display ATTEN so that the vertical axis of the chromatogram on the analysis screen becomes more than 100 μ RIU.

(Example)

When AUX RANGE setting is 2, set ATTEN = 7.

- (4) Supply distilled water from the inlet port using the syringe and adapter. Then, replace the reference flow line with distilled water,
- (5) After both sample cell and reference cell are filled with distilled water, close R. flow.
- (6) Press **Test/zero** and **balance**, and wait until the baseline is stabilized.
- (7) Adjust the zero position, and record the baseline when the inside of the cell is filled with distilled water.
- (8) Inject the standard sample from the inlet port. The baseline will move toward + direction about 100 μ RIU.
- (9) When the baseline is stabilized, move the cursor to read the difference between the levels of the distilled water and the standard sample.
- (10) When the refractive index of the standard sample is $R \times 10^{-6}$ RIU, the span factor is calculated by the following equation:

$$[\text{SPAN}-A](\text{new}) = [\text{SPAN}-A](\text{old}) \times 100/R$$

(Example)

When the measured refractive index is 102.4×10^{-6} [RIU] and the old span factor is 0.90, the new span factor is as follows.

$$0.90 \times 100/102.4 = 0.88 \leftarrow \text{new SPAN}-A$$

- (11) Input the SPAN-A value in local mode. Press **func** several times and set LOCAL to 1. Press the **func** key several times and input the value obtained into SPAN-A. After setting a the new value, set LOCAL to 0 again to return to the remote mode.
- (12) Inject distilled water from the inlet port and complete the span adjustment.
- (13) As for the adjustment of modes P and L, change the mode setting, and repeat procedures from (4) to (12). Input the span value obtained in each mode into SPAN-P and SPAN-L.



Warning

This unit uses two pieces of the following fuses. Be sure to replace the fuses of the same type and capacity.

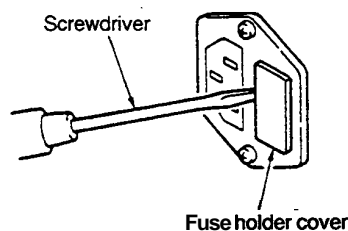
Rated voltage: 100-240 VAC

Part No. 072-01652-23

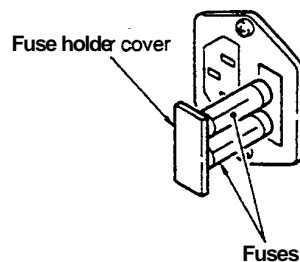
250V, 5AT

Replace the blown fuse according to the following procedure:

- (1) **Turn** the power switch **OFF**.
- (2) Disconnect the power cord from the power supply connector.
- (3) Catch the fuse holder cover with a regular screwdriver and take it out to this side.



- (4) Replace the fuse and push it in until it clicks.



If the cover or the panel of the unit is **stained**, wipe out dirt or dust with soft cloth or a piece of paper.

If necessary, use a non-ionic detergent.



Chapter 9 Performance Verification

CONTENTS

9.1	Component Validation	9-2
9.1.1	Test Procedure of Stand-alone Unit	9-3
9.1.2	Test Procedure Controlled from the SCL-10A	9-5
9.2	System Validation	9-9
9.2.1	Test Procedure of Isocratic LC System	9-9

1. Overview

Each unit of the RID-10A system has been inspected stringently at the factory during the production process. The inspection procedures described in this manual have been produced by compiling and indicating the inspection activities that are both actually necessary and can be practically executed by the user. That is, this manual is intended to serve as a reference book for inspection procedures that do not require specialized expensive equipment that is used at the factory during manufacture. When inspection results obtained by the procedure described in this manual meet the acceptance criteria, the RID-10A can be used without problem.

2. Frequency for executing hardware validation

Roughly speaking, perform hardware validation every 6 to 12 month. Adjust the frequency of the inspection based on the actual operating conditions of the LC system. When the LC unit is operated continuously, day and night, the intervals between inspections will necessarily be shorter. As mentioned above, when the equipment is judged to be improper, hardware validation must be executed immediately to correct any possible malfunctions.

3. Validation item

With the RID-10A, validation is performed on the following items. Statement is made in reference to the regulation in the ASTM (American Society for Testing and Materials) E1303-89.

- (1) Checking the lamp lit time
- (2) Checking the lamp intensity
- (3) Checking the temperature control system
- (4) Checking the span

4. Validation procedure

Validation procedure differs depending on the system configuration with which the RID-10A is used. This chapter describes the following methods of validation.

Control of the RID-10A	Data processing unit	Page
Stand alone	Chromatopac	9-3
SCL-10A	Chromatopac	9-6

9.1.1 Test Procedure of Stand-alone Unit

1. Checking the lamp lit time

Content of inspection

This test checks the lamp lit time.

Test procedure

- (1) Press the **func** key several times to display LAMP TIME.
- (2) Record the value as the lamp lit time.

Acceptance criteria

LAMP TIME is less than 20,000.

The lamp life is approximately 20,000 hours. This is equivalent to approximately 7 years of use 8 hours a day. It is recommended that the lamp be replaced before the expected end of life to avoid the inconvenience of instrument failure. To replace the lamp, contact your Shimadzu representative or service engineer.

2. Checking the lamp intensity

Content of inspection

This test checks whether the light source intensity is sufficient.

Test procedure

- (1) Fill the cell with distilled water.
- (2) Press the **func** key several times to display TOTAL EN.
- (3) Record the light intensity value.

Acceptance criteria

TOTAL EN is more than 6.000.

When the value does not fall within reference value, first clean the flow cell. (Refer to the section 8.1 Cleaning the Flow Lines.) Then increase the value of the LAMP VOLT to 6000 or more. (Refer to the section 5.5 Additional Functions.)

3. Checking the temperature control system

Content of inspection

This test verifies whether the temperature control is working properly.

Test procedure

- (1) Press the **func** key several times to display CELL TEMP.
- (2) Record the value as the set temperature.
- (3) Press the **func** key again to display ACT TEMP.
- (2) Record the value as the actual temperature.

Acceptance criteria

ACT TEMP value is within ± 0.1 °C of CELL TEMP.

The CELL TEMP value must be 12(C higher than room temperature.



4. Checking the span

Content of inspection

This test checks whether the refractive index value is accurately measured and displayed by testing the standard sample whose index is already **known**.

The test requires the following reagents and equipment.

- Distilled water
- Standard sample (Glycerin solution)
Refer to the section 8.2 Span Adjustment.
- Syringe
- Data processor

Test procedure

- (1) Set the parameters of the detector **as** follows.
CELL **TEMP: 40**
polarity: **+**
mode: A (When performing mode A span adjustment)
- (2) Set the detector so that the plot full scale of the data processor becomes 128×10^{-6} RIU.
AUX RANGE of the detector: 2 (or **4**)
ATTEN of the Chromatopac: 7 (or **9**)
- (3) Use the syringe and adapter to deliver distilled water to the inlet port. During delivery, press the **shift** and **R flow** keys to flow the water to the reference flow lines.
- (4) After the inside of the sample cell/reference cell is replaced with distilled water, turn **Rflow OFF** by pressing the **shift** and **R flow** keys again.
- (5) When the baseline is stabilized, press the **shift** and **balance** keys to perform optical balance adjustment.
- (6) Press the **zero** key and record the baseline level when the cell is filled with distilled water.
- (7) Inject the standard solution from the inlet port. The baseline moves up ward by 80% of the full scale.
Record the baseline level when the cell is filled with standard solution. When the Chromatopac is used, press the **PLOT** key to record the baseline.
- (8) Read the difference of the baseline level between the distilled water and the standard solution, and convert the value into RI unit.



(Example)

When the baseline moves 120 mm on the chart paper with the Chromatopac C-R7A, the value can be converted into the RI unit with the following formula.

$$128 \times 10^{-6} [\text{RIU}] \times 120 [\text{mm}] / 150 [\text{mm}] = 102.4 \times 10^{-6} [\text{RIU}]$$

150 : Plot full scale of the C-R7A. (See the table below.)

Chromatopac model	Full scale on chart paper
C-R7A	150 mm
C-R4A	160 mm

Acceptance criteria
 $100 \pm 5 \times 10^{-6} [\text{RIU}]$

When the value does not fall within reference value, perform span adjustment according to the section 8.2 Span Adjustment.

9.1.2 Test Procedure Controlled from the SCL-10A

1. Checking the lamp lit time

Content of inspection

This test checks the lamp lit time.

Test procedure

- (1) Select the [6. MONITOR] on the main menu of the SCL-10A to open the monitor screen.

(Example of MONITOR screen)

```

-----DET.A-----
ACT.TMP      40.0
TOTAL.E      7566
BALANCE      10
LAMP.TM      86      (Lamp lit time)
-----
```

- (2) Record the value as the lamp lit time.

Acceptance criteria

LAMP TIME is less than 20,000.

The lamp life is approximately 20,000 hours. This is equivalent to approximately 7 years of use 8 hours a day. It is recommended that the lamp be replaced before the expected end of life to avoid the inconvenience of instrument failure. To replace the lamp, contact your Shimadzu representative or service engineer.



2. Checking the lamp intensity

Content of inspection This test checks whether the light source intensity is sufficient.

Test procedure

- (1) Fill the cell with distilled water.
- (2) Select the [6. MONITOR] on the main menu of the SCL-10A to open the monitor screen.

(Example of MONITOR screen)

```
----DET.A-----  
ACT.TMP      40.0  
TOTAL.E      7566   (Lamp intensity)  
BALANCE      10  
LAMP.TM      86  
.....
```

- (3) Record the light intensity value.

Acceptance criteria TOTAL EN is more than 6,000.

When the value **does** not fall within reference value, first clean the flow cell. (Refer to the section 8.1 Cleaning the Flow Lines.) Then increase the value of the LAMP VOLT to **6,000** or more. (Refer to the section 5.5 Additional Functions.)

3. Checking the temperature control system

Content of inspection This test verifies whether the temperature control is working properly.

Test procedure

- (1) Select the [3. DET CONTROL] on the main menu of the SCL-10A to open the parameter control screen of the detector.

(Example of DET CONTROL screen)

```
----DET.A-----  
RANGE        100.00  
RESP         5  
POL,         1  
CELL.T       40.0  
MODE,        0  
AUXRNG       2  
-----
```

- (2) Record the value of CELL.T as the set temperature.



- (3) Select the [6. MONITOR] on the main menu of the SCL-10A to open the monitor screen.

(Example of MONITOR screen)

```
----DET A-----
ACT.TMP      40.0
TOTAL.E      7566
BALANCE      10
LAMP.TM      86
-----
```

- (4) Record the value of ACT.TMP as the actual temperature.

Acceptance criteria	ACT TEMP value is within $\pm 0.1\text{ }^{\circ}\text{C}$ of CELL TEMP.
---------------------	--

The CELL TEMP value must be $12\text{ }^{\circ}\text{C}$ higher than room temperature.

4. Checking the span
Content of inspection

This test checks whether the refractive index value is accurately measured and displayed by testing the standard sample whose index is already known.

The test requires the following reagents and equipment.

- Distilled water
- Standard sample (Glycerin solution)
Refer to the section 8.2 Span Adjustment.
- Syringe
- Data processor

Test procedure

- (1) Set the parameters of the detector as follows.
- LOCAL: 1 (*)
 - CELL TEMP: 40 (indicated as OFF)
 - polarity: +
 - mode: A (When performing mode A span adjustment)
- *This test must be performed in LOCAL mode not in REMOTE mode. After changing the operation mode, other parameters, such as CELL TEMP, polarity, and mode, should be set directly to the detector.
- (2) Set the detector so that the plot full scale of the data processor becomes $128 \times 10^6 \text{ RIU}$.
- AUX RANGE of the detector: 2 (or 4)
 - ATTEN of the Chromatopac: 7 (or 9)



- (3) Use the syringe and adapter to deliver distilled water to the inlet port. During delivery, press the **shift** and **R flow** keys to flow the water to the reference flow lines.
- (4) After the inside of the sample cell/reference cell is replaced with distilled water, turn **Rflow OFF** by pressing the **shift** and **R flow** keys again.
- (5) When the baseline is stabilized, press the **shift** and **balance** keys to perform optical balance adjustment.
- (6) Press the **zero** key and record the baseline level when the cell is filled with distilled water.
- (7) Inject the standard solution from the inlet port. The baseline moves to the **+** direction by 80% of the full scale. Record the baseline level when the cell is filled with standard solution. When the Chromatopac is used, press the **PLOT** key to record the baseline.
- (8) Read the difference of the baseline level between the distilled water and the standard solution, and convert the value into RI unit.

(Example)

When the baseline moves 120 mm on the chart paper with the Chromatopac C-R7A, the value can be converted into the RI unit with the following formula.

$$128 \times 10^{-6} [\text{RIU}] \times 120 [\text{mm}] / 150 [\text{mm}] = 102.4 \times 10^{-6} [\text{RIU}]$$

150 : Plot full scale of the C-R7A. (See the table below.)

Chromatopac model	Full scale on chart paper
C-R7A	150 mm
C-R4A	160 mm

Acceptance criteria
 $100 + / - 5 \times 10^{-6} [\text{RIU}]$

When the value does not fall within reference value, perform span adjustment according to the section 8.2 Span Adjustment.



1. Outline

For the holistic validation of HPLC system, chromatographic analysis is performed under the analytical conditions specified by the manufacturer. The system status can be judged based on the obtained results. This is because the failure of the LC system may depend on the analytical conditions. Holistic validation procedure described in this manual is the standard for checking the status of the LC system, serving as the basis of inspection.

In routine operation, the operators must perform a system suitability test under the predetermined analytical conditions. If any problem occurs under such conditions, perform holistic validation first described in this manual. If the result of holistic validation meets the acceptance criteria, the LC system itself is working properly and the cause of the problem is relate to the analytical method itself. **On** the other hand, if the results do not meet the acceptance criteria for holistic validation, it implies that there is a problem in the LC system itself and modular validation should be performed for diagnosis of each LC module.

2. Procedures for inspection

Details of standard operating procedure for holistic validation are described in 9.2.1.

The repeatability (relative standard deviation) of retention time, peak area, and peak height is measured to check whether the values meet the acceptance criteria.

9.2.1 Test Procedure of Isocratic LC System**1. Purpose**

The purpose of this test is to confirm that the chromatographic data can be obtained with good repeatability for the LC system to be inspected.

An HPLC System for this inspection consists of the pump, detector, column oven, auto injector, system controller, and data processor.

2. Preparation for Inspection

- (1) Prepare the following parts and reagents.
 - (a) Isocratic LC system
 - (b) Mobile phase (acetonitrile/water = 4/1 (v/v))
 - * Use acetonitrile and water of HPLC grade.
 - (c) Column (Shim-pack HRC-ODS or equivalent, 4.6mm ID x 150mm)

- (d) Sample

* Anthracene in acetonitrile

[Method for sample preparation]

Place 200mg of anthracene in the volumetric flask of 100mL capacity and add acetonitrile of HPLC grade to it.

- (e) Water (HPLC grade or equivalent)

- (f) Isopropyl alcohol

- (2) Check the connection of the units. Refer to the instruction manuals for details of the connection of each unit.
- (3) Before installing the column, observe tubing connection of the LC system. Use tubing with 0.3mm I.D. or less from the outlet of auto injector to the column inlet and from the column outlet to the detector inlet. Length of the tubing should be 30cm or less to minimize the extra-column band broadening.
- (4) Flush the flow line with appropriate solvents dependent on the operation status of the system. General guideline is shown below. **To** flush the flow line with solvents, connect the inlet and outlet tubing of the column directly using a proper union. Column should be connected after flushing.

• **Newly installed system**

Flush the flow line with isopropyl alcohol, then with water at a flow rate of 2 mL/min for ten minutes.

• **When low polarity solvent (such as hexane) is used as the mobile phase in the system**

Flush the flow line with isopropyl alcohol, then with water at a flow rate of 2 mL/min for ten minutes.

• **When mixture of water and organic solvent, or organic solvent miscible with water (methanol, acetonitrile, etc.), or buffer solution is used as the mobile phase in the system**

Flush the flow line with water at a flow rate of 2 mL/min for ten minutes.

- (5) Set the mobile phase (b) and flush the flow line, then connect the column (c) to the LC system.

3. Procedure

Outline of procedure is described as below:

- (1) Set the **flow** rate of the pump to 1mL/min and column oven temperature to 40 °C. **Start** pump **flow** and temperature control. Then, confirm that the solvent comes out **from** the outlet tubing of the detector and no leak of solvent **is** observed at the connection.

Set the parameters of the RID detector.

AUX RANGE : 2 (1 × 10³ RIU/V)

RESPONSE : 5

- (1) Set the parameters **of** the data processor.

WIDTH : 5 SLOPE : 100

DRIFT : 0 MIN.AREA : 100,000

T.DBL : 0 STOP.TM : 7

ATTEN : 4 SPEED : 5

- (2) Monitor the baseline. When a stable baseline **is** obtained, press the zero point adjustment key of the detector. Then, inject 10uL **of** mobile phase and confirm no peak **is** observed.
- (3) Inject 10 uL of test sample solution. Repeat measurement at least five times.
- (4) Obtain the chromatographic data of retention time, peak **area**, and peak height **from** five successive analyses.
- (5) Calculate the average (X) of the data and %RSD **using** the equations **as** shown below.

$$SD = \sqrt{\frac{\sum_{i=1}^5 (X_i - XA)^2}{4}}$$

$$XA = (X1 + X2 + + X4 + X5) / 5$$

$$\%RSD = (SD / XA) * 100$$

X1X5 : Data

XA : Average

SD : Standard deviation

%RSD : Relative standard deviation



Performance Verification

9.2 System Validation

4. Parameter Setting for Inspection

Parameters to be set for the equipment at measurement are described below.

[LC parameters]

- | | |
|--|-------------------|
| * Initial condition | * LC Time program |
| FLOW : 1 | 7.00 STOP |
| OVEN.T : 40 | |
| P.MAX : 200 | |
| | |
| * Auto injector [SIL-10A/10Ai] | |
| SAMPLING SYRINGE SPEED : 2 | |
| EXCESS VOLUME : 50 | |
| (Use default value for others.) | |
| | |
| * Detector [RID-10A] | |
| AUX RANGE : $2 (1 \times 10^{-3} \text{ RIU/V})$ | |
| RESPONSE : 5 | |

5. Acceptance Criteria

The acceptance criteria for the %RSD are shown below.

- | | |
|----------------|----------------|
| Retention time | : 0.5% or less |
| Peak area | : 1.0% or less |
| Peak height | : 1.0% or less |









Chapter 10 Troubleshooting

CONTENTS	10.1 Symptoms, Causes and Remedies	10-2
	10.2 List of Error Messages	10-4



When **an** abnormality occurs in the equipment, follow the procedures below for **checking** the unit.

Symptom	Possible cause	Remedy
OVER is displayed on the display for the refractive index.	Recorder terminals are saturated in the negative end. No signal will be output	Press (zero) .
Recorder baseline does not change.	(1) Range is set to 0 . (2) Lamp does not light. (3) OVER condition exist. See OVER symptom.	(1) Set appropriate range. (2) Light the lamp. (3) Press (zero) .
Using Chromatopac, the obtained values for peak area or height are different from those obtained from the RID-6A.	AUX RANGE setting is inappropriate.	Refer to Chapter 5.2.7 Changing the range.
LED of INT ALARM remains lit. * The LED is lit when the TOTAL EN value is larger than 9000, or less than 5000.	(1) The solution in the reference cell is different from that in the sample cell. (2) It are stagnant in the flow cell.	* Purge the solution in the reference cell. * Perform optical balance adjustment by pressing (shift) and (balance) . * Adjust lamp voltage.
OVER is displayed on the display for BALANCE .	(1) Optical balance adjustment is requested. (2) Bubbles are stagnant in the flow cell.	* Purge the solution in the reference cell. * Perform optical balance adjustment by pressing (shift) and (balance) .

Symptom of malfunctions	Possible cause	Confirmation of cause	Countermeasures
spiking  Sawtooth baseline  Continuous Spiking occurring at every stroke of the pump  No equilibration of baseline  Drift  Noise is excessive  Swell	Bubbles flowing through the cell Bubbles trapped in the cell Cell window is stained. Impurities in the mobile phase	With the flow cell tubing connected, remove the cell, and check it under flow conditions, then check for bubbles and lens or cell fouling. The drift stops after stopping liquid supply.	(1) Degas the mobile phase. (2) Use methanol to rinse the cell interior. Remove the cell inlet tubing connector and force methanol from the outlet side with a syringe, then remove bubbles. (4) Check the pump,
	Light source lamp degradation	small.	

Should **any** trouble persist, contact our nearest branch office, sales office or agent.



This unit has several self-diagnostic functions. *So* when abnormality is detected, one of the following error messages is displays along with the output of alarm sound.

1. System error (Failure of the main unit)

(1) ROM error

balance	RI (10 ⁻⁴)	range	mode
ROM		FAILURE	

It is displayed when a ROM error is detected.

If this error is displayed, turn the power of the unit off and contact your Shimadzu Service Representative.

(2) RAM error

balance	RI (10 ⁻⁴)	range	mode
RAM		FAILURE	

It is displayed when a RAM error is detected.

If this error is displayed, turn the power of the unit off and contact your Shimadzu Service Representative.

(3) Home position error

balance	RI (10 ⁻⁴)	range	mode
ERR ZG		HOME POS	

It is displayed when the home position sensor of the motor for driving the xerography does not properly work.

If this error is displayed, *turn* the power of the unit off and contact your Shimadzu Service Representative.

(4) Leakage error

balance	RI (10 ⁻⁴)	range	mode
ERR LEAK		6 5 5 3 5	

It is displayed when the leakage occurs.

Remove the cause of leakage, and wipe off the liquid on the leak sensor. When the displayed value comes to 50000 or less, press the **CE** key to stop the alarm and use the leak sensor again.

(5) Overheat error

balance	RI (10 ⁻⁴)	range	mode
ERR OVER		HEAT	

It is displayed when the temperature of the measuring point rises to 63°C and over. If this error is displayed, *turn* off the power of the unit and contact your Shimadzu Service Representative.

2. Set value loss error

balance	RI (10 ⁻⁴)	range	mode
NOT		PROTECTED	

It is displayed if the parameters and time program set previously are lost at powering up. Press **CE** key and the unit becomes operational. Set parameters again.

Chapter 11 Specifications

CONTENTS

11.1	RID-10A Specifications	11-2
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11

Specifications

RID-10A Specifications

Measuring method	Deflection
Refractive index range (RIU)	1-1.75
Measurement range (RIU)	0.01-500 $\times 10^{-6}$ (Analysis mode) 1-5000 $\times 10^{-6}$ (Preparative and Large-scale Preparative modes)
Linearity (RIU) ※ 1	5 $\times 10^{-4}$ (Analysis mode) 5 $\times 10^{-3}$ (Preparative and Large-scale Preparative modes)
Maximum operating flow rate (mL/min.)	20 (with option installed : 150)
Baseline noise (RIU) ※ 1	0.25 $\times 10^{-8}$ or less (filled with water, time constant 1.5 sec., room temperature 25°C, analysis (A) mode)
Drift (RIU/h) ※ 1	1 $\times 10^{-7}$ and below (filled with water, time constant 1.5 sec., room temperature 25°C, analysis (A) mode)
Withstand pressure (MPa)	Detector : 0.4 (4kgf/cm ²) Cell : 1.9 (20kgf/cm ²)
Detector internal capacity (μL)	IN - cell inlet : 63.5 Cell : 9 Cell outlet - OUT : 280.2
Flow line switchover	Solenoid valve (pinch valve type) Maximum Operating pressure : 0.4MPa (4kgf/cm ²)
Materials in contact with solvent	SUS316, PTFE, quartz, fluoro-rubber, liquid crystal polymer
Cell temperature setting	30 - 60°C (by 0.1°C step), OFF
Zero adjustment	Optical balance (optical zero) Auto-zero, baseline shifting Fine-zero)
Polarity switching	Possible
Time program	Independent control of detector and control from system controller are both possible. Available parameters : Auto-zero, range, marker, response, event, polarity, loop
Output (recorder output) (integrator output)	1mV RECORDER terminal, 10mV RECORDER terminal Selectable in 4 steps of 1 $\times 10^{-4}$, 1 $\times 10^{-3}$, 1 $\times 10^{-2}$, 2.5 $\times 10^{-4}$ RIU/Volt
Safety measure	Bimetallic thermal breaker Solvent leak sensor
Weight	12kg
Size W x H x D (mm)	260 x 140 x 420
Operating temperature range (°C)	4 - 35
Power requirements	RID-10A is classified as follows according to supply voltage P/N228-32000-91 100V \pm 10V 150VA 50, 60Hz P/N228-32000-92 120V \pm 10V 150VA 50, 60Hz P/N228-32000-94 220V \pm 20V 150VA 50, 60Hz

※ 1 The specifications are determined according to the ASTM method below.

ASTM E1303-89

Practice for Refractive Index Detectors Used in LC.

Erratum in the instruction manual

There is an erratum in the description of time constant setting for noise specification in section 11-2(specification).

Erratum	Correction
Time constant : 1.5sec	Time constant : 3 sec

Chapter 12 Replacement Parts

CONTENTS

12.1	Consumables and Repair Parts	12-2
12.2	Optional Parts	12-4

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Replacement Parts

1. Consumables

W lamp	Life: 20,000 hours (equals to more than 7 years by 8 hours operation per day), compatible with RID-6A	228-32445-91
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2. Repair parts

Description	Remarks	Part Number
	Parts for electric system	
Panel, RID-10A		228-23009-98
Panel Switch, RID		228-23673-02
Power Switch		228-33968-91
PCB, PCB-CPU90		228-23527-91
ROM, RID-10A (CPU90)		228-33806-91
PCB, PB-1		228-31627-91
PCB, PB-2		228-31837-91
PCB, PB-3		228-32578-91
Leak Sensor		228-33743-91
Battery		228-25249-91
Heater Assy (for inner block)		228-33738-91
Heater Assy (for outer block)		228-33738-92
Fuse		072-01652-23
	Parts for optical system	
Light Source Block		228-32444-91
Slit Holder		228-32443-91
Photodiode		228-33748-91
H Zero-glass		228-33730-91
V Zero-glass		228-33732-91
Mirror holder with mirror		228-32442-91
Mirror		228-15706
Zero-glass motor		228-34210-91
Microswitch (for motor)		228-33736-91

Description	Remarks	Part Number
	Parts for flow lines	
Solenoid valve		228-341 14-91
Tube (for solenoid valve IN)		228-33816-01
Tube (for solenoid valve OUT)		228-33816-02
Flow cell		228-32004
Gasket (for flow cell)		228-32057
Port, IN side		228-16447
Port , OUT side		228-16447-01
Nut , (to mount port)		228-16034
Washer, (to mount port)		228-16033
Flow path, S side (with heat exchanger)		228-32017-91
Flow path, R side (with heat exchanger)		228-32018-91



Optional Parts

Description	Remarks	Part Number
Flow selection block	including a block, tubing and flow diagram 1) required when 20mL/min or larger flow volume is applied. (MAX 150 mL/min) 2) required for parallel flow configuration.	228-34102-91
Relief valve	for protection of cell and/or solenoid valve in case over pressure at high flow rate is applied, for example in preparative work.	228-33615-91

Chapter 13 Reference

13.1 Solvent Characteristics	13-2
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(1) Solvent	'7<.5cP, >45' '7<.5cp, <45'	(2) Source	(3) W Cutoff	(4) R.I. _D	Boiling Point (°C)	Viscosity (cP, 25°C)	(5) p'	(6) e° _s	(7) Water Solubility %* in 20°C Solvent	(8) Dielectric Constant e ²⁰	(9) p'+ 0.25 e
1.	FC-78 ^(*)	(Particular 210nm		1.267	50	0.4	<-2	-.25		1.88	P' and Dielect.
	FC-75 (Fluorescent	to LC)	210(opaque	1.276	102	0.8	<-2	-.25		1.86	const.
	FC-43 solvent)		210 or under)	1.291	174	26	c-2	-.25		1.9	(function in proportion to intensity)
2.	Isooctane ^(*) (2,2,4- tri methylpentane)	LC	197	1.389	99	0.47	0.1	0.01	0.011	1.94	0.1
3.	n-Heptane ^(*)	LC	195	1.385	98	0.40	0.2	0.01	0.010	1.92	0.5
4.	n-Hexane ^(*)	LC	190	1.372	69	0.30	0.1	0.01	0.010	1.88	0.5
5.	n-Pentane ^(*)	LC	195	1.355	36	0.22	0.0	0.00	0.010	1.84	0.5
6.	Cyclohexane	LC	200	1.423	81	0.90	-0.2	0.04	0.012	2.02	0.5
7.	Cyclopentane ^(*)	LC	200	1.404	49	0.42	-0.2	0.05	0.014	1.97	0.6
8.	1-Chlorobutane ^(*)	LC	220	1.400	78	0.42	1.0	0.26		7.4	2.8
9.	Carbon disulfide	LC	380	1.624	46	0.34	0.3	0.15	0.005	2.64	1.7
10.	2-Chloropropane ^(*)	LC	230	1.375	36	0.30	1.2	0.29		9.82	3.7
11.	Carbon tetrachloride	LC	265	1.457	77	0.90	1.6	0.18	0.008	2.24	2.3
12.	n-Butyl ether		220	1.397	142	0.64	2.1	0.25	0.19	2.8	2.4
13.	Triethylamine			1.398	89	0.36	1.9	0.54		2.4	2.4
14.	Bromoethane ^(*)			1.421	38	0.38	2.0	0.35		9.4	4.3
15.	i-Propyl ether ^(*)		220	1.365	68	0.38	2.4	0.28	0.62	3.9	3.2
16.	Toluene	LC	285	1.494	110	0.55	2.4	0.29	0.046	2.4	2.9
17.	p-Xylene		290	1.493	138	0.60	2.5	0.26		2.3	3.0
18.	Chlorobenzene			1.521	132	0.75	2.7	0.30		5.6	4.1
19.	Bromobenzene			1.557	156	1.04	2.7	0.32		5.4	4.1
20.	Iodobenzene						2.8	0.35			
21.	Phenyl ether			1.580	258	3.3	3.4			3.7	3.7
22.	Phenetole			1.505	170	1.14	3.3			4.2	4.9
23.	Ethyl ether ^(*)	LC	218	1.350	35	0.24	2.8	0.38	1.3	4.3	4.0
24.	Benzene	LC	280	1.498	80	0.60	2.7	0.32	0.058	2.3	3.6
25.	Tricresyl phosphate										
26.	Ethyl iodide			1.510	72	0.57	2.2			7.8	4.2
27.	n-Octanol		205	1.427	195	7.3	3.4	0.5	3.9	10.3	5.8
28.	Fluorobenzene			1.46	85	0.55	3.1			5.4	4.6
29.	Benzylether			1.538	288	4.5	4.1				
30.	Methylene chloride ^(*)	LC	233	1.421	40	0.41	3.1	0.42	0.17	8.9	5.6
31.	Anisole			1.514	154	0.9	3.8			4.3	4.6
32.	i-Pentanol			1.405	130	3.5	3.7	0.61	9.2	14.7	7.3
33.	1,2-Dichloroethane	LC	228	1.442	83	0.78	3.5	0.44	0.16	10.4	6.3
34.	t-Butanol			1.385	82	3.6	4.1	0.7	miscible	12.5	
35.	n-Butanol	LC	210	1.397	118	2.6	3.9	0.7	20.1	17.5	8.3
36.	n-Propanol	LC	240	1.385	97	1.9	4.0	0.82	miscible	20.3	
37.	Tetrahydrofuran ^(*)	LC	212	1.405	66	0.45	4.0	0.57	miscible	7.6	
38.	Propylamine ^(*)			1.385	48	0.35	4.2		miscible	5.3	
39.	Ethylacetate ^(*)	LC	256	1.370	77	0.43	4.4	0.58	8.8	6.0	5.8
40.	i-Propanol	LC	205	1.384	82	1.9	3.9	0.82	miscible	20.3	
41.	Chloroform ^(*)	LC	245	1.443	61	0.53	4.1	0.40	0.072	4.8	5.6

(1) Solvent	'7<.5cP, >45' ''7<.5cP, <45'	(2) Source	(3) W Cutoff	(4) R.I.2s'	Boiling Point (°C)	Viscosity (cP, 25 °C)	(5) p'	(6) ε°	(7) Water Solubility %* in 20°C Solvent	(8) Dielectric Constant ε ₂₀	(9) p'+ 0.25 e
42.	Acetophenone			1.532	202	1.64	4.8			17.4	8.7
43.	Methylethyl ketone"	LC	329	1.376	80	0.38	4.7	0.51	23.4	18.3	9.1
44.	Cyclohexanone			1.450	156	2.0	4.7			18.3	9.1
45.	Nitrobenzene			1.550	211	1.8	4.4			34.8	13.2
46.	Benzonitrile			1.536	191	1.2	4.8			25.2	10.9
47.	Dioxane	LC	215	1.420	101	1.2	4.8		miscible	2.2	
48.	Tetramethyl urea	LC	265	1.449	175		6.0	0.56		23.0	10.7
49.	Quinoline			1.625	237	3.4	5.0			9.0	7.4
50.	Pyridine			1.507	115	0.88	5.3		miscible	12.4	
51.	Nitroethane		380	1.390	114	0.64	5.2		0.9		
52.	Acetone'''	LC	330	1.356	56	0.30	5.1	0.71	miscible		
53.	Benzyl alcohol			1.538	205	5.5	5.7			13.1	8.8
54.	Tetramethyl guanidine						6.1	0.6			
55.	Methoxyethanol	LC	210	1.400	125	1.60	5.5		miscible	19.9	
56.	Tris (cyanoethoxy) propane	GC					6.6	0.56			
57.	Propylene carbonate	LC					6.1				
58.	Ethanol	LC	210	1.359	78	1.08	4.3		miscible	24.6	
59.	Oxydipropionitrile	GC					6.8				
60.	Aniline			1.584	184	3.77	6.3			6.9	8.1
61.	Acetic acid			1.370	118	1.1	6.0		miscible	6.2	
62.	Acetonitrile ^(*)	LC	190	1.341	82	0.34	5.8		miscible	37.5	
63.	N, N-dimethylacetamide	LC	268	1.436	166	0.78	6.5	0.88		37.8	
64.	Dimethylformamide	LC	268	1.428	153	0.80	6.4			36.7	
65.	Dimethylsulfoxide	LC	268	1.477	189	2.00	7.2	0.62	miscible	4.7	
66.	N-methyl-2-pyrrolidone	LC	285	1.468	202	16.7	6.7			32	
67.	Hexamethyl phosphoric acid triamide										
68.	Methanol"	LC	205	1.326	65	0.54	5.1		miscible	32.7	
69.	Nitromethane		380	1.380	101	0.61	6.0		2.1		
70.	m-Cresol			1.540	202	14	7.4			11.8	10.0
71.	N-methylformamide			1.447	182	1.65	6.0		miscible	182	
72.	Ethylene glycol			1.431	182	16.5	6.9		miscible	37.7	
73.	Formamide			1.447	210	3.3	9.6		miscible	111	
74.	Water	LC		1.333	100	0.89	10.2			80	



-
- (1) (*) indicates solvents most suitable for LC, having convenient boiling points ($> 45^{\circ}\text{C}$ cp) and low viscosity (20.5 cp).
(**) indicates solvents with very low viscosity and boiling point.
 - (2) "LC" indicates that the solvents are commercially available specifically for LC from following companies: Burdick & Jackson, Baker Chemical, Mallinkrodt Chemical, Fischer Scientific, Waters Associates and Manufacturing Chemists, Inc.
(Note: In Japan, they are also commercially available from the following companies: Wako Pharmaceutical Co., Ltd., Nakarai Pharmaceutical Co., Ltd. and Kanto Chemical Co., Ltd.)
"GC" indicates that the solvent is used as a stationary phase for ~~gas~~ chromatography, and can be purchased from companies selling **GC** columns and stationary phases. (These solvent are used **as** a stationary phase in the liquid-to-liquid LC.)
 - (3) The wavelength shorter than which the solvent become opaque.
 - (4) Refractive index at 25°C
 - (5) Polarity parameter of solvent
 - (6) Solvent strength parameter of liquid-to -solid adsorption on alumina
 - (7) Water solubility (W%) at 20°C for solvent used in liquid-to -solid adsorption
 - (8) Value at 20°C
 - (9) Function where P' is proportional to solvent strength and dielectric constant in ion pair chromatography.

