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-1**OA** 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 labels incleated on the equipment.

For safety of operation, this unit is provided with the $\int \mathbf{mark}$ at the portion where special cautions are required.

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

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 Image: Rear view Image: Rear view Image: Rear view Image: Rear view Image: 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, whith may cause malfunction of the unit.



Frecautions 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

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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.

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.

Capacity	Power source	Part NO.
VAOSI	100A	528-32000-91
AV021	120V	528-32000-92
VAOSI	520~540Λ	528-32000-94

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

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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.



- A : Electric charge moving with flowing liquid
- B : Electric charge being fixed to the solid surface.

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 10⁻¹⁰s/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 conductiveSuitable 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.

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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 $(10^{-10} \text{s/m or less})$.
- 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-1**OA** 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-1OA** 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-1 OA 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.





Principle of Measurement

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 **Gell** varies, the balance of the incident light intensity into right and left parts *G* 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.

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



Flow Schematic

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

At R flow OFF

When the R flow switch is OFT, the solenoid value 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	μĽ
Flow cell volume	9	μL
Flow cell to cell outlet port	280.2	μL



Chapter 2 Parts List



2.1Parts and Accessories2-2



[Caution]

Parts and Accessories

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**

Part Name	Parts No.	Quantity
Signal cable	228-25089-92	1
Power supply cord (120V)	071-60814-01	
(220~240V)	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
	228-22310-00	lm
Sus tubing	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

2 Standard Accessories

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Chapter 3 Component Location and Function

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Front View



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.



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 part	Normally connects back pressure tube (0.3mm ID.x Im).	4-9
3	Tube holder	Secures the connected tube.	
4	Connection port for leak tube	Connects the leak tube to this part.	4-13
5	Solvent leak sensor	Detects solvent leaks and outputs an error signal.	10-4



Rear View



No.	Description	Function	Refer to page
1	REMOTE connector	This is the connector for SCL-10A.	7-2
0	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
6	Fuse holder	Two fuses are provided in this holder.	8-7

Chapter 4 Installation

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Basic Installation Requirements

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 System Layout

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 bot-
	tom. Be careful to keep fingers clear when installing the unit.



Stacking Modules

RID-1OA may be stacked on the pump (LC-10AS/LC-10AD/ LC-10AT/LC-10Ai) or the column oven (CTO-10A/1OAC). 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

Warning	Cheo • S	ck the following points before connectin Supply voltage and capacity	ng power supply.
	–	Supply voltage and capacity	Part Number
		100V 150VA	228-32000-91
		120V 150VA	228-32000-92
		220 - 240V 150VA	228-32000-94
	Whe satis supp • V	en the power supply is not stable or the of factory performance is not possible. Ve oly for the system before preparing the p Verify that the power switch of the main u	capacity insufficient erify the total power ower supply. unit is turned OFF .
1. Connection to outlet	(1)	Connect the female connector of t supplied with the unit to the power su the rear of the unit, and plug the male supply outlet.	he power supply c upply cord connector connector into a po
	(2)	The supplied power cord is a three-co When connecting to a two-conductor supply outlet, use the provided power	onductor(3-prong)ty r (2-prong) type po supply adapter.
		Three-conduct	or type Adaptor
2. Grounding	(1)	When the three-conductor type powe the unit is grounded by the power sup	r supply outlet is us ply cord.
	(2)	When the two-conductor type power	supply outlet is us

4

(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.

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:

7

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.
- 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.

Note



3. Connecting injector and column

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

- (1) Cut the **SUS** pipe $(1.6 \times 0.3 \text{ supplied to the pump, to the necessary length for connecting the injector and the column.$
- (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.
- 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.

Note



4. Connecting detector and column

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

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

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

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. lighten the male PEEK nut firmly without using tools.

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.

Note

Note

Note



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) x length(cm)]	For extension [Inside diameter (mm) x length (cm)]	Remarks
3	0.3 X 100	1.0×100	
5	05 X 100	1.0×100	
20	0.8 X 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**.

EPC 104 propagative head	FRC-10A inlet pipe	FRC-10A outlet pipe	Max. operating flow rate
	Inside diameter $[mm] \times Length [cm]$	Inside diameter [mm] × Length [cm]	[mL/min]
Fraction correction head with valve	0.8 × 100	1.6× 100	20 (150')
Fraction correction head with valve	0.3 X 100	1.6×100	3
Fraction correction head without valve	e 0.3 x 100		

*) An optional flow selection **block** is necessary.

 Do not use the pipes listed in the table which are out of range.
 Do not use the pipes listed in the table which are out of range.

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.



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.

When the front cover is dosed.







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 Chrornatopac (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.






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 **Lrtype** 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

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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.	
		Front cover	
	2.	Precautions to prevent clogging of the flow cell.	
		Dusty or clogged flow cells are the most frequent causes	
		$\boldsymbol{\sigma}$ trouble in any detector. After analyzing a high concen-	
		tration 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.	

Operation **G**



Fundamentals of Operation

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



(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.



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

5-3

Note

2. Display unit

If NOT **PROTECTED**) message is displayed and an alarm sounds after turning the power ON, press <u>CE</u> 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.

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



No.	Display ordescription	Function	
1	balance	Displays position of the light on photodiode.	
2	RI (10 ⁻⁶)	Displays refractive index (unit :×10 ⁻⁶ RIU)	
3		Displays full scale of refractive index output to the recorder terminal (unit :×10 ^{.6} RIU)	
4	mode	Displays measurement mode.	
6	shift	Shift key indicator lamp.	
6	prog run	Time program operation indicator lamp. Lights when time program is being executed.	
Ø	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 t

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 teams) 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 $4 \text{ zero shift } \bullet$.
▲zero shift▼ Zero shift key	Press this key to move the zero position on the recorder. It is moved upward by pressing \blacktriangle side and downward by pressing \blacktriangledown 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 value to replace the liquid inside the reference cell with the mobile phase.
(shift) + (pol) key	These keys switch the polarity of recorder output. $\boxed{pol(-)}$ LED is lit for (-) polarity.

5

shift + (mode] key	These keys select the measurement mode. The measurement mode changes from $\mathbf{A} \to \mathbf{P} \to \mathbf{L} \to \mathbf{A}$. \mathbf{A} : Analytical mode \mathbf{P} : Preparative mode \mathbf{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.
(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.
Snnt + 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 snow. 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 util the baseline stabilizes. When the balance value is more than 50, press shift and <u>Juance</u> 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		
	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. Setting the measurement mo	ode		
(Example)	When changing the measurement mode from A to P (1) Press shift and 3^{mod} .		

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

_	balance	Ri (10-4)	range	mode
	100	BALA	NCE	Р
_			\downarrow	
$\left(\right.$	0	0	4	Р

Note

When the above operation is repeated, the mode is changed form $\rightarrow A \rightarrow P \rightarrow L$. 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	
Δ	0.01~500×10° PIU	0.01 step for 0.01 to 1	
Ā		1 step for 1 to 500	
P·L	$1 \sim 5000 \times 10^{6} \text{RIU}$	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 \times 10⁻⁶ RIU or less, use mode A.

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

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

The procedure to set the recorder range is **as** follows:

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

(1) Press <u>func</u> once in the initial state to access the range parameter (blinking).



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

Note

Note

6. Setting measuring range (Example)

- 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:



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

(Example)

When that range = 1, ATTEN = 2 : Plot full scale = $1 \times 2^2/10 = 0.4$ [x 10⁻⁶RUFS]

(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 X 10 ⁻⁴ RIU/V	
2	1 X 103 RIU/V	Compatible with AUX-L of RID-6A
3	1×10^{-2} RIU/V	
4	2.5×10^{-4} RIUN	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 (X 10⁻⁶ RIU/FS) determined by AUX RANGE value and ATTEN value.

	AUXRANGE		(×10	⁻⁶ RIU)
ATTEN	1	2	3	4
0	0.1	1	10	0.25
1	0.2	2 1	. 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

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



Operation G

(Example)

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 <u>zero</u>, and the pen returns to almost 0 scale on the chart paper.
- (5) Press (zero shift $\triangle \bigtriangledown$) to move the baseline to a desired position and start analysis.
- (6) Press the zero key to return the baseline to the previous position.



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 (Letters in brack	e constant of analog CR filter ats are response of RID-6A.)	Minimum peak width at half-height
1	0.05	sec	02 sec
2	0.1	sec	0.4 sec
3	0.5	sec (FAST)	22 sec
4	1.0	sec	48 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





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

RESPONSE

(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 ≤ 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.





5-12



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

1. Usage of each mode

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

Mode Application Setting Analytical А 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. Ρ Preparative For measuring samples of high concentration. Samples ten times as concentrated as those for mode A can be measured. L For measuring samples of high Large-scale concentration at large flow rate. preparative Samples of the same concentration as those of the mode P can be delivered at 150mL/min.

Management	Refractive index measuring range	Flow rate range	Tubing
Measurement mode	(× 10 ⁻⁶ RIU)	(mL/min)	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).

- \downarrow **A** : Analytical mode
- \downarrow P : Preparative mode
- \downarrow L : Large-scale preparative mode

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

Ca	aut	tio	n
	_	_	

Note

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	$32 \times 16 \times 1000$
150	3.2×1.6×1000	32 × 16 × 1000

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 × 1 000	3.2 × 1.6 × 1000
50	(FRC-10A accessory)	(FRC-10A accessory)
450	20×16×1000	3.2 imes 16 $ imes$ 1000
150	3.2 × 1.6 × 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.





5-14



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

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.

Note



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: ×10 ⁶ RIUFS,
RNGP	Designation of output range for recorder (mode P)	0, 1 - 5000	Recorder is short-circuited when
	Designation $\boldsymbol{\sigma}$ output range for recorder (mode L)		set to 0.
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 *ct* 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.

5-16

- **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
- 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:

- () 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 of shift and $(\underline{fun}, , , \underline{h})$ in this state returns the display to the previous setting.

- 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)**.

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.



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:

_	bala	ance	Ri (10**)	range	mode
$\left(\right.$	0	US	BED	32	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 **program** 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 program is dimmed, and the program stops.

4. Deleting a step (Example)

Note

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.



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

Туре	Command	Function	
4	TOTALEN	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 in 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)



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

CELL

(Lamp energy of each photodiode.)

balance	Ri (10 ⁻⁺)	range		mode	_
CELL	355	0	345	0	Ì

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



CELL TEMP		balance	Ri (10*)	range	mode	
(Cell temperature setting)		CELL	TEM	4 0	. 0	
	Sets the cell	temperati	ure at the m	neasurin	g unit.	
	When the fl	ow rate is	larger than	3 mL/n	un, turn this	OFF.
	When the f	low rate is	s less than	3 mL/n	nin, set the	temperature
	the room ter	nperature	+ 12℃			
ACT TEMP		baiance	Ri (10 ⁻⁴)	range	mode	<u> </u>
(Monitors actual cell temperature)		АСТ	TEMP	40.0)	
	Displays the	e actual cel	l temperat	ure in th	e optical un	it.
AUX RANGE		balance	Ri (10 ⁻⁴)	range	mode	
(Integrator output full scale)	(AUX	RANO	θE		_)
	1	1 1 X	10 ⁻⁴ RIU	/v		
	2	1×	10 ⁻³ RIU.	N	Compatible with	AUX-L of RID-
	3	1X	10 ⁻² RIU	⁄v		
	4	2.5>	< 10 ⁻⁴ RIU	/v	Compatible with	AUX-H of RID-
		1				
EVENT		balance	Ri (10 ⁻⁴)	range	mode	—
(Event output terminals)	(EVEI	ТИ	-	_)

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	alue EVENT1 EVEN	
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	RI (10*)	range	mode	
$\left(\right)$	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 pro-
	gram. 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)

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

rance

mode

Input the desired value, and press Enter

balance

RI (10*)

MONIT-TIME

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:



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)				
(Fromotion of key input)	This function locks keypad, preventing entry, and is invoked by pressing $\boxed{\text{Enter}}$ when the above display is shown. To restore keypad function, press $\boxed{\text{CE}}$ and $\boxed{\text{CE}}$ simultaneously.			
ADRS				
(Remote address)	balance Ri (10 ⁻¹) range mode			
	ADRS 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	balance RI (10 ⁻⁴) range mode			
(Setting of the local mode)	(LOCAL _)			
	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 (localmode).			
LAMP VOLT	Sets a lamp voltage. (Input range : 0 to 5.00)			
(Lamp voltage)	Normally, input the value such that value of TOTAL EN is within the range from 6,000 to 9,000.			
SPAN A	balance Ri (10⁻¹) range mode			
(Span factor in mode A)	(SPAN A 1.00)			
	Inputs the span value of the analytical mode.			
SPAN P				
(Span factor in mode P)	batance Ri (10") range mode			
	(<u>SPAN P 1.00</u>			
	Inputs the span value of the preparative mode.			

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RID-10A

SPAN L	balance	Ri (10 ⁻⁴)	range	mode
(Span factor in mode L)	SPAN	l 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.

Chapter 6 Initial Performance Test

6.1 System Performance Check	6.1	System Performance Check		6-2
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This chapter contains an explanation of the operation check for the simple system in the **figure** below.



Set the Chromatopac ATTEN to 4. (Plot full scale of the (7) Chromatopac becomes equivalent to 16x 10⁻⁶RIU.)

6-2

RID-10A

- (8) Press [ZERO], [S), [O) 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 ATTEN to 8 on the Chromatopac. (Chromatopac full scale becomes equivalent to about 256 x 10⁻⁶RIU)
- (12) Press ZERO, 2 , 0 and Enter on the Chromatopac.
- (13) Press (PLOT) and (Enter) on the Chromatopac to stop plotting.
- (14) Inject <u>sample to the injector</u>.
- (15) Press <u>START</u> 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 : Column :	Acetonitrile/Water = 75/25 Asahipak NH2P-50 4.6mm 0 x 25cm			
Flow rate :	1 mL/min		٦	i
Column temp :	40℃.			in 100mL
Sample :	Fructose Grucose	100 mg 100 mg		water, 10µL injection
	Sucrose	100 mg		L
	Maltose	100 mg		
	Raffmose	100 mg		

Initial Performance Test

(Example of chromatogram)



6

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



RID-10A



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 :

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 me a.file smpl/inj KEYLOK fre 9999.9 READY

3. Setting initial parameters

Press numeric key 0 in the main menu screen or move the cursor to the number 0 and press $\underbrace{\text{Enter}}$ to display the parameter screen for analysis file.

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



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 <u>act</u> on the SCL keyboard to turn ON the <u>activate</u> lamp. When the <u>activate</u> lamp is lit, the parmeters set in this screen are sent to the detector.

(Example) If the <u>POL</u> parameter is set to -1, the **LED** for polarity on the detector is lit and the polarity is reversed.



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 <u>DET.A</u> and <u>DET.B</u> to switch between detectors.

(1) Press <u>func</u> to change the function display to the following display:

DAR		KO BARDIG

7-5

- 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
 (It) and (I), 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 (X10 ⁻⁶ RIU)	0, 0.01-500	(0.01-1.00) 0.01	100
			(1-500) 1	
	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 cr 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	I	2

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

PRUNT CLEAR COPY ZERO A ZERO B

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.

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

Performs zero adjustment of a detector A.



 $ZERO_A$ (f4)


6. Time program

Performs zero adjustment of a detector B.

DESP S MARK & MARK B AZBALL KEYLOK

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

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

Toggles between the detector A or B for parameter display.

Disables acceptance of any key input except for <u>KEYLOK</u> (lock release).

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.



Operation

- (1) 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.
- (2) Press (func) to change the function to the following display:

PRINT CLEAR COPHER TABLE TABLE

(3) Move the cursor to a desired device name in the following line using TABLE \leftarrow (f4) and TABLE \rightarrow (f4).

FUNCTION FOR (PUMP DET. B OTHER)

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

```
31 RESP 134 CELL. T 44 R. CLOS 46 RNG. A
32 MARK 135 ZERO 45 R. FLOW 147 RNG. P
33 POL 43 BALANC 1 I
```

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),	0,1	40
		30-60		
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	0, 0.01-500	0.01 (0.01-1)	100
	(X 10 ⁶ RIU)		1 (1-500)	
RNG. P	Recorder range in modes P and L	0,1-5000	1	100
	(X 10 ⁶ RIU)			

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

	GLENN.	0000	TABLE	TABLES
--	--------	------	-------	--------











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

Note) F. S. stands for full scale.

RID-10A

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



CautionA 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: $\phi 0.4 - \phi 1.0$ (AWG 26-18)
Stranded wire: $0.3 \text{mm}^2 \sim 0.75 \text{ mm}^2$ (AWG 22-20)
Element wire diameter : $\phi 0.18$ at the minimum

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

Mainten ance



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 mitric acid.

- 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 lies 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.





F-

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 verfied. 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 "RIU from that of pure water.

8.2.1 When span adjustment is performed using the Chromatopac

- (1) \mathbf{Turn} ON the power.
- (2) Set the parameters of the detector **as** follows:

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

 * When the RID-1OA is controlled from the system controller SCL-1OA: 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 x 10⁻⁶ RIU.

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

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 **CFF**.
- (6) When the baseline is stabilized, press (Shift) and (Balance) and perform optical balance adjustment.
- (7) Press <u>zero</u>, 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.

128×10" [RIU] $\times \frac{120}{150}$ mm = 102.4×10" [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 x 10⁻⁶ RIU, the span factor can be calculated by the following equation:

[SPAN-A] (new) = [SPAN-A] (old) x 100/R



(Example) When the measured refractive index is $102.4 \text{ R} \times 10^{-6}$ [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 in	hitial Conditions	
Pump Condi	tions : Isocratic] Detector A : RID-10A	rs
A.FLOW	1.000	mL/min	MODE 0 RANGE 100.00	- 0
B.FLOW	0.500	mL/min	POL 1 RESP 5	
C.FLOW	0.500	mL/min	CELL T AUXING	
APRES	100	Kgf/cm ²	Detector B : RF-10AXL	
B.PRES	0	Kgf/cm^2	EX.WAV 350 RANGE 1	
C.PRES	0	Kgf/cm^2	EM.WAV 450 RESP 3	
P.MAX	200	Kgf/cm^2	Martik	
P.MIN	0	Kgf/cm^2		
Other			-j	
sv		VEN.T 40		
EVENT	T 0	.MAX 85		
POWER	0			

Example 2 (CLASS-LCIO)

- Detec	tor RID-10A
Polarity	Response 1.5 ±
F Measure Mode	
O: Analytical	○ 1:1.0E-7 RIU/mV
	○ 3 1.0E - 5 RTU/mV
	4:2.5E-7 RIU/mV
Cell Temp.(C) 40	
Cell Temp.(C)	40 • •
ОКС	ancel <u>H</u> elp

	chromatogram on the analysis screen becomes more than
(Example)	When AUX RANGE setting is 2 set $\Delta TTEN = 7$
(Example)	(A) 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 µRIU .
	(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 $\mathbf{R} \times 10^{-6}$
	RIU, the span factor is calculated by the following equation:
	$[SPAN-A]) (new) = [SPAN-A] (old) \times 100/R$
(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
	(12) As for the adjustment of modes \mathbf{P} and \mathbf{L} shange the mode
	setting and repeat procedures from (4) to (12) Input the
	span value obtained in each mode into SPAN-P and
	SPAN-L.

(3)

Change display ATTEN so that the vertical axis of the

Maintenance



Warning	This unit uses two pieces of the following fuses. Be sure to
C	replace the fuses of the same type and capacity.
	Rated voltage: 100-240 VAC
	Eart 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 pus it in until it clicks.



8-7



Periodical Cleaning

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 C	omponent 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 S	ystem Validation	9-9
9.2.1	Test Procedure of Isocratic LC System	9-9



Component Validation

. Overview Each unit of the RID-10A system has been inspected stringently at the factory during the production process. The inspection procedures described m this manual have been produced by *compiling* and indicating the inspection activities that are bothactually 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.

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

rocedureValidation 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



3.

4. Validation procedure

Validation item

9-2

1.	Checking the lamp lit time	This test checks the lamp lit time		
	Content of Inspection	This test checks the famp in 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 intensit	V		
	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 of	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 then room temperature.		

9.1.1 Test Procedure of Stand-alone Unit

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
- (1) Set the parameters of the detector as follows.
 CELL TEMP: 40
 polarity: +
 mode: A (When performing mode A span adjustment)
- Set the detector so that the plot full scale of the data processor becomes 128 x 10⁻⁶RIU.
 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.

Test procedure

(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.

 128×10^{-6} [RIU] x 120 [mm]/150[mm] = 102.4×10^{-6} [RIU] 150 : Plot full scale of the C-R7A. (See the table below.)

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

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	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 I	MONITOR	screen]	
			ACT.TMP	40.0		
			TOTAL.E	7566		
			BALANCE	10		
			LAMP.TM	86	(Lamp lit time)	
				<u>-</u>		
		(2)	Record the value	e as the lan	np 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		t source intensity is sufficient.	
	Test procedure	(1)	Fill the cell with distilled water.		
		(2)	(2) Select the [6. MONITOR] on the main menu of the S		on the main menu of the SCL-10A
			to open the mo	nitor screen	l.
			(Example of	MONITOR	screen]
			DET.A		*******
			ACT.TMP	40.0	
			TOTAL.E	7566	(Lamp intensity)
			BALANCE	10	
			LAMP.TM	86	
		(3)	Record the ligh	nt intensity v	value.
		т	TOTAL EN is may		0
	Acceptance criteria	Whe flow	en the value does v cell. (Refer to th	not fall with ne section 8	thin reference value, first clean the 1.1 Cleaning the Flow Lines.) Then
	Acceptance criteria	Whe flow incre the s	en the value does cell. (Refer to the value of section 5.5 Additi	not fall with ne section 8 the LAMP	thin reference value, first clean the 3.1 Cleaning the Flow Lines.) Then VOLT to <i>6,000</i> or more. (Refer to ons.)
3.	Acceptance criteria Checking the temperature	Whe flow incre the s	en the value does v cell. (Refer to the the value of section 5.5 Addition 5.5	not fall with the section 8 the LAMP tonal Functi	thin reference value, first clean the 3.1 Cleaning the Flow Lines.) Then VOLT to <i>6,000</i> or more. (Refer to ons.)
3.	Acceptance criteria Checking the temperature Content of inspection	Whe flow incre the s e contro This prop	en the value does v cell. (Refer to the ease the value of section 5.5 Addition 5.5 addition	not fall with the section 8 the LAMP tonal Functional F	thin reference value, first clean the 3.1 Cleaning the Flow Lines.) Then VOLT to <i>6,000</i> or more. (Refer to ons.) temperature control is working
3.	Acceptance criteria Checking the temperature Content of inspection Test procedure	Whe flow incre the s e contro This prop (1)	en the value does v cell. (Refer to the ease the value of section 5.5 Additi ol system s test verifies we perly. Select the [3. SCL-10A to op	not fall with ne section 8 the LAMP fonal Functional Fu	thin reference value, first clean the Al Cleaning the Flow Lines.) Then VOLT to <i>6,000</i> or more. (Refer to ons.) temperature control is working TROL] on the main menu of the meter control screen of the detector.
3.	Acceptance criteria Checking the temperature Content of inspection Test procedure	Whe flow incre the s e contro This prop (1)	en the value does v cell. (Refer to the ease the value of section 5.5 Additi ol system s test verifies we berly. Select the [3. SCL-10A to op (Example of DET.A	not fall with ne section 8 the LAMP donal Function whether the DET CON pen the parameter DET CON	thin reference value, first clean the Al Cleaning the Flow Lines.) Then VOLT to <i>6,000</i> or more. (Refer to ons.) temperature control is working TROL] on the main menu of the meter control screen of the detector. TROL screen]
3.	Acceptance criteria Checking the temperature Content of inspection Test procedure	Whe flow incre the s e contro This prop (1)	en the value does v cell. (Refer to the ease the value of section 5.5 Additi ol system s test verifies we berly. Select the [3. SCL-10A to op (Example of DET.A RANGE	not fall with ne section 8 the LAMP fonal Function whether the DET CON pen the parameter DET CON DET CON 100.00	thin reference value, first clean the Al Cleaning the Flow Lines.) Then VOLT to <i>6,000</i> or more. (Refer to ons.) temperature control is working TROL] on the main menu of the meter control screen of the detector. TROL screen]
3.	Acceptance criteria Checking the temperature Content of inspection Test procedure	Whe flow incre the s e contro This prop (1)	en the value does y cell. (Refer to the ease the value of section 5.5 Additi ol system s test verifies we berly. Select the [3. SCL-10A to op (Example of DET.A RANGE RESP	not fall with the section 8 the LAMP tonal Function whether the DET CON the parameter DET CON the parameter DET CON 100.00 5	thin reference value, first clean the A.1 Cleaning the Flow Lines.) Then VOLT to 6,000 or more. (Refer to ons.) temperature control is working TROL] on the main menu of the meter control screen of the detector. TROL screen]
3.	Acceptance criteria Checking the temperature Content of inspection Test procedure	Whe flow incre the s e contro This prop (1)	en the value does v cell. (Refer to the ease the value of section 5.5 Additi ol system s test verifies we berly. Select the [3. SCL-10A to op (Example of DET.A RANGE RESP POL,	not fall with the section 8 the LAMP ional Function whether the DET CON the parate DET CON DET CON 100.00 5 1	thin reference value, first clean the Al Cleaning the Flow Lines.) Then VOLT to <i>6,000</i> or more. (Refer to ons.) temperature control is working TROL] on the main menu of the meter control screen of the detector. TROL screen]
3.	Acceptance criteria Checking the temperature Content of inspection Test procedure	Whe flow increation the s e contro This prop (1)	en the value does v cell. (Refer to the ease the value of section 5.5 Additi ol system s test verifies we berly. Select the [3. SCL-10A to op (Example of DET.A RANGE RESP POL, CELL.T	not fall with the section 8 the LAMP tional Function whether the DET CON the parate DET CON DET CON 100.00 5 1 40.0	thin reference value, first clean the A1 Cleaning the Flow Lines.) Then VOLT to <i>6,000</i> or more. (Refer to ons.) temperature control is working TROL] on the main menu of the meter control screen of the detector. TROL screen]
3.	Acceptance criteria Checking the temperature Content of inspection Test procedure	Whe flow incre the s e contro This prop (1)	en the value does v cell. (Refer to the ease the value of section 5.5 Additi ol system s test verifies we berly. Select the [3. SCL-10A to op (Example of DET.A RANGE RESP POL, CELL.T MODE,	a not fall with the section 8 the LAMP ional Functional Function whether the DET CON ben the parameter DET CON DET CON 100.00 5 1 40.0 0	thin reference value, first clean the Al Cleaning the Flow Lines.) Then VOLT to 6,000 or more. (Refer to ons.) temperature control is working TROL] on the main menu of the meter control screen of the detector. TROL screen]

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

Performance Verification

		(3)	Select the [6. MONITOR] on the main menu of the SCL-10A	
			to open the monitor screen.	
			(Example of DET A	MONITOR screen]
			ACT.TMP	40.0
			TOTAL.E	7566
			BALANCE	10
			LAMP.TM	86
		(4)	Record the value	ue of ACT.TMP as the actual temperature.
	Acceptance criteria	А	CT TEMP value	is within $+/-0.1$ °C of CELL TEMP .
		The	CELL TEMP valu	he must be 12°C higher than room temperature.
4.	Checking the span			
	Content of inspection	This	test checks whe	ether the refractive index value is accurately
		meas	sured and displa	yed by testing the standard sample whose
		index	x is already know	n.
		The	test requires the I	ollowing reagents and equipment.
		•	Distilled water	
		•	Standard samp	le (Glycerin solution)
		•	Refer to the sec	ction 8.2 Span Adjustment.
		•	Data processor	
		•	Data processor	
	Test procedure	(1)	Set the parame	ters of the detector as follows.
			LOCAL: 1 (*)
			CELL TEMP	2:40 (indicated as OFF)
			polarity: +	
			mode: A (W	hen performing mode A span adjustment)
			*This test mu	st be performed in LOCAL mode not in
			REMOTE mo	ode. After changing the operation mode, other
			parameters, s	such as CELL TEMP , polarity, and mode,
			should be set	directly to the detector.
		(2)	Set the detector becomes 128x	so that the plot full scale of the data processor 10 ⁻⁶ RIU.
			AUX RANGE	of the detector: 2 (or 4)
			ATTEN of the	Chromatopac: 7 (or 9)

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- (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.

 128×10^{-6} [RIU] x 120 [mm]/150[mm] = 102.4×10^{-6} [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	$100 + 7 - 5 \times 10^{\circ}$ [RIU]	

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



Outline 1. 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 Purpose 1. 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,

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.

column oven, auto injector, system controller, and data processor.

(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	:	$2(1 \times 10^{-3} \text{ RJU/V})$
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.

$$SD = \sqrt{\frac{5}{\sum (Xi-XA)^2}} \frac{1}{4}$$

 $XA = (X1 + X2 + \dots + X4 + X5) / 5$ %RSD = (SD / XA) * 100 X1X5 : Data XA : Average SD : Standard deviation %RSD : Relative standard deviation

4.	Parameter Setting for	Parameters to be set for the equipment at measurement are				
	Inspection	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 : $2(1 \times 10^{-3} \text{ RJU/V})$				
		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				

1

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





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. S	system error (Failure of the	main unit)	balance	RI (10⁵)	range	mode	
(1)	ROM error		ROM	F /	A I L U F	RE	
		It is displaye If this error your Shimae	ed when a is display dzu Servic	ROM erro ed, turn th e Represer	r is detect e power o ntative.	ed. f the unit off	and conta
(2)	RAM error	(balance RAM	RI (10 ⁻⁺) F /	^{range}	^{mode})
		It is displaye If this error your Shimae	ed when a is display dzu Servic	RAM erro ed, turn the e Represer	r is detect e power o ntative.	ed. f the unit off	and contact
(0)	11		balance	RI (10 ⁻⁴)	range	mode	
(3)	Home position error		ERR	ZG	НОМЕ	POS)
(4)	Leakage error	If this error your Shimad	is display dzu Servic balance ERR	ed, <i>turn</i> the re Represer RI (10 ⁻¹) LEAK	e power of ntative. range 6 5	f the unit off	and conta
		It is displaye Remove the sensor. When CE key to	ed when th cause of n the disp o stop the	e leakage leakage, a layed value alarm and	occurs. nd wipe o e comes to use the lea	ff the liquid 50000 or les lk sensor aga	on the lea ss, press the in.
(5)	Overheat error		balance	Ri (10 ⁻⁴)	range	mode	
(•)	overnear en el		ERR	OVER	HEA	Т	
		It is displaye 63°C and ov unit and con	ed when the rer. If this tact your s	ne temperat error is di Shimadzu S	ture of the splayed, <i>t</i> Service Re	measuring p <i>urn</i> off the p presentative	point rises t power of th
2 5	et value loss error		balance	RI (10 ⁻⁴)	range	mode	
2. 3	er value 1033 61101		ΝΟΤ	PF	ΟΤΕΟ	TED	
		It is display	ed if the	parameters	and time	program se	t previousl

are lost at powering up. Press (CE) key and the unit becomes operational. Set parameters again.

Troubleshooting

Chapter 11 Specifications

CONTENTS

11.1 RID-10A Specifications 11-2



RID-10A Specifications

Measuring method	Deflection			
Refractive index range (RIU)	1-1.75			
Measurement range (RIU)	0.01-500 x 10 ⁻⁶ (Analysis mode)			
	1-5000 x 10 ⁻⁶ (Preparative and Large-scale Preparative modes)			
Linearity (RIU) X 1	5 x 10 ⁻⁴ (Analysis mode)			
	5×10^{-3} (Preparative and Large-scale Preparative modes)			
Maximum operating flow rate (mL/min.)	20 (with option installed : 150)			
Baseline noise (RIU) 💥 1	0.25 x 10 ⁻⁸ or less (filled with water, time constant 1.5 sec., room temperature			
	25°C, analysis (A) mode)			
Drift (RIU/h) 💥 1	1 x 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 $= 1.9 (20 \text{kgf/cm}^2)$			
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)	1mV RECORDER terminal, 10mV RECORDER terminal			
(integrator output)	Selectable in 4 steps of 1×10^{-4} , 1×10^{-3} , 1×10^{-2} , 2.5 x 10^{-4} RIU/Volt			
Safety measure	Bimetalic thermal breaker			
	Solvent leak sensor			
Weight	12kg			
Size W x H \times D (mm)	260 × 140 × 420			
Operating temperature range (°C)	4-35			
Power reguirements	RID-10A is classified as follows according to supply voltage			
	P/N228-32000-91 100V ± 10V 150VA 50, 60Hz			
	P/N228-32000-92 120V±10V 150VA 50,60Hz			
	P/N228-32000-94 220V±20V 150VA 50,60Hz			

* 1 The specifications are determined according to the ASTM method below. ASTM EL303-89

Practice for Refractive Index Detectors Used in LC.

Erratum in the instruction manual

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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





1. Consumables

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

2. Repair parts

Description	Remarks	Part Number
l	Parts for electric system	
Panel, RID-1 OA		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
Part, OUT side		228-16447-01
Nut, (to mount part)		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	228-34102-91
	1) required when 20mL/min or larger flow	
	volume is applied. (MAX 150mL/min)	
	2) required for parallel flow configuration.	
Relief valve	for protection of cell and/or solenoid valve in	228-33615-91
	case over pressure at high flow rate is	
	applied, for example in preparative work.	


Chapter 13 Reference

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Solvent Characteristics

									(7) Water		
(1)	"7- 50D - 15"	(2)	(3)	(4)	Boiling	Viccosity	(5)	(6)	Solubility	(8) Dialactria	(9)
Solvent	"7<.5cp, <45"	Source	vv Cutoff	(4) R.I.25*	rom (℃)	$(cP, 25^{\circ}C)$	(3) p'	(0) e°1	Solvent	Constant e^{20}	р+ 0.25 е
1.	FC-78 ^(*)	(Particula	210 n m	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	-02	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.	Tricthylamine			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		23	3.0
18.	Chlorobenzene			1.521	132	0.75	2.7	0.30		5.6	4.1
19.	Bromobenzene			1557	156	1.04	2.7	0.32		5.4	4.1
20.	lodobenzene						2.8	0.35			
21.	Phenyl ether			1580	258	3.3	3.4			3.7	3.7
22.	Phenetole		21 0	1.505	170	1.14	33			42	4.9
23.	Ethyl ether	LC	218	1.350	35	0.24	2.8	0.38	1.3	4.3	4.0
24.	Benzene Tritter tul alternaliste	LC	280	1.498	80	0.60	27	0.32	0.058	23	3.6
25.	Etherlie did									= 0	10
26.			205	1.510	72	0.57	2.2		•	7.8	4.2
27.	n-Octanoi		205	1.427	195	7.3	3.4	0.5	3.9	10.3	5.8
28. 20	Pluorobenzene			1.40	285	055	3.1			5.4	4.6
29. 30	Mathylana chlorida''''	10	222	1.538	288	4.5	4.1	0.42	0.17	20	5.6
30.	Anisole	LC	255	1.421	40	0.41	3.1 2.9	0.42	0.17	8.9	3.0 1.6
32	i Pentanol			1.014	134	0.9	3.8 27	0.61	02	4.5	4.0
32.	1 2-Dichloroethane	ιc	228	1.405	150 82	3.3 0.78	3.1 2.5	0.01	9.2	14.7	7.5 63
34	t-Butanol		220	1.442	83 82	36	33 41	0.44	miscible	10.4	0.3
35.	n-Butanol	LC	210	1.305	118	2.6	4.1 3 Q	0.7	20.1	125	83
36.	n-Pronanol	LC	210 240	1.327	97	19	4.0	0.82	miscible	203	0.5
37	Tetrahydrofuran ^(*)	LC	210	1.405	66	0.46	40	0.57	miscible	76	
38	Propylamine ^(*)	20	212	1 385	48	0.35	42	051	miscible	53	
39.	Ethylacetate ^(*)	LC	256	1370	-0 77	043	44	0.58	88	60 60	58
40.	i-Propanol	LC	205	1.384	82	19	39	0.82	miscible	20.3	2.0
41	Chloroform"	LC	245	1.443	61	053	41	040	0072	48	56
					01	0.00		5.10	5.672	1.0	5.0

13-2

									(7) Water		
	1		(3)		Boiling				Solubility	(8)	(9)
(1) Solvent	7<.5cP, >45 7<.5cp, <45	(2) Source	W Cutoff	(4) R.I.25	Point (°C)	(cP, 25°C)	(5) p'	(6) e°ı	Solvent	Dielectric Constant e ²⁰	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.	This (cyanoethoxy)							0.56			
	propane	GC					6.6				
57.	Propylene carbonate	LC					6.1				
58.	Etharol	LC	210	1.359	78	1.08	43		miscible	24.6	
59.	Oxydipropionitrile	GC					6.8				
60.	Aniline			1584	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-pyrolidone	LC	285	1.468	202	16.7	6.7			32	
67.	Hexamethyl phosphoric										
	acid triamide										
68.	Methanol"	LC	205	1.326	65	054	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	

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(1) (*) indicates solvents most suitable for LC, having convenient boiling points (> 45 "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.