SOLVENT DELIVERY MODULE LC-10AT SHIMADZU HIGH PERFORMANCE LIQUID CHROMATOGRAPH INSTRUCTION MANUAL

## SHIMADZU CORPORATION

CHROMATOGRAPHIC INSTRUMENTS DIVISION

KYOTO. JAPAN

		the units, to prevent them from toppling.
		In earthquake-prone areas, use the supplied tie plates when stacking
		tions of ambient temperature are extreme.
.7	Other Considerations	Avoid installation of the instrument in direct sunlight or where varia-
		to contact the field representative or service person.
		closed, do not open the cover. If you need to open the cover, be sure
		As maintenance may in general be carried out with the main cover
		that of the commercial power supply.
.9	Power Source	This instrument employs various voltages for each part, in addition to
		off.
		solvents splashed in the eyes or on the skin may be immediately rinsed
.5	Other Facilities Required	A sink should be located as close as possible to the unit so that toxic
<b>'</b> †	Protective Goggles	Wear protective goggles when using solvents.
		taken. See "Section 12.1 Precautions on Static Electricity".
3.	Static Discharge	Static discharge also may cause a fire. Appropriate measure should be
		Install a fire extinguisher at a conspicuous location nearby.
		sparks in the same room.
		chromatograph. Do not install other equipment that may generate
5.	Fire	Never use an open flame in the vicinity of a high performance liquid
		be well ventilated.
		inflammable and toxic, therefore the room in which they are used must
۱.	Ventilation	Solvents used in high performance liquid chromatography are often
		manual, in addition to those below.
		Be sure to pay due attention to the precautions noted throughout this
		appropriate installation site.
		volumes of organic solvent, requires proper care in handling and an
		High performance liquid chromatography, which often requires large

Precautions

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# Chapter 1 General

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The LC-10AT is a solvent delivery unit with a dual-plunger, tandem-flow (i.e., 2-stage) pump which has been developed for improving accuracy and sensitivity of analysis in high-performance liquid chromatography.

In addition to the solvent delivery unit(s), a high performance liquid chromatography system requires an automated or manual injector, column, injector holder, detector and so on, which may be separately ordered through your sales representative.

This instruction manual covers operations for the LC-10AT and relevant accessories. For use of the other modules and special accessories, please refer to the instruction manuals for each.

1

### Features

1.2

- 1. Stable Solvent Delivery<br/>with Little Flow PulsationThe LC-IOAT is a solvent delivery unit with a dual-planger, tandem<br/>-flow pump for use in high-performance liquid chromatography. It<br/>features stable solvent delivery with little flow pulsation.
- 2. A Variety of Functions for Gradient Elution
  Two gradient elution modes, low pressure and high pressure, are available. The high pressure gradient elution mode is highly accurate and allows a minimum time lag, while the low pressure gradient elution mode can handle solvent delivery of up to four liquids with a single pump. In addition, two types of gradient elution control are available depending upon the system setup. One is to use the SCL-IOA System controller, and the other allows control of gradient elution from the LC-1OAT alone.
- **3.** Long Life Plunger Seal The plunger seal is made of ultra-high-molecular-weight Polyethylene (UHMW-PE) which offers a low degree of wear.
- Seals and Plungers Protected Against Buffer Salts
   Buffer Salts
   Buffer Salts
   Buffer Salts
   Comparison of the plunger seal of the plunger seal by preventing the formation of salt deposits.
- 5. Easy Maintenance The LC-IOAT has a simple construction with a small number of components for easy maintenance. In addition, the replacement of plunger seals, plungers and check valves can be carried out from the front side of the LC-IOAT.

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# Chapter 2 Check Upon Delivery



2.1 Lists of Supplied Parts, etc. ..... 2-2







# Lists of Supplied Parts

The LC-10AT is composed of the following parts.

#### 1. LC-10AT Main Body

#### 2. Standard Parts and Accessories

Part Name	Part No.	Qty
100V power cord or 200V power cord	071-60814-01 071-608 14-06	Ι
Suction filter	228- 18740-91	Ι
Instruction manual (Japanese version) or Instruction manual (English version)	228-30055 228-30056	1
Accessory kit (See the following page.)	228-32133-91	1
Drain tube kit	228-28161-91	Ι

# Lists of Supplied Parts

#### 3. Accessory Kit

Category	Part Name	Part No.	Qty
Tools	Wrench, 8 x 10mm	086-03006	2
	Wrench, 13 x 17mm	086-03017	1
	Allen wrench, 3mm	086-03804	1
	Seal remover	228-25142	1
	File (for cutting SUS pipes)	670-18928-02	1
	Tie plate	228-18751	1
	Optical cable	070-92025-51	I
	Clip (for fixing tube)	046-00994-03	1
	Male nut, 1.6MN	228-16001	2
	Ferrules, 1.6F	228-16000	2
	Drain tube	228-25495-91	1
	SUS pipe, 1.6 x 0.3	670-10006-02	2m
Parts	Syringe needle (for disposable syringe)	228-18216-91	1
	Syringe, 20ml	046-00038-01	1
	Pipe clamp	670-11610-01	1
	Spiral wrap	018-26002	0.2m
	Lid	228-17644	1
	Bottle cap	228-18887	1
	Bushing 1.6MN PEEK	228-18565	2
	PTFE Tube 1 x 0.5	016-37502	2m
	Polyvinyl Tube	016-31401	0.12m
	Remote cable	228-28253-91	1
Consumables	Plunger seal	228-21975	1

The above are packed together as the accessory kit (P/N 228-32133-91).

#### 4. Drain Tube Kit

Category	Part Name	Part No.	Qty
	Drain tube for solvent leakage	228-25162-03	I
	Drain tube, elbow	228-28094	1
Parts	T joint	228-28162	1
	Straight joint	228-28163	1
	Pipe clamp	670-11610-01	1
	Instruction manual	228-10913	1

# Chapter 3 Construction and Functions

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# **Front Panel**

#### 1. Front Panel



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Fig. 3.1

No.	Part Name	Description
1	Operator's panel	For operation, see item 5.1.
2	Front cover	Covers the pump head and flow lines, ctc.
3	Drain valve knob	Turn this knob to open/close the flow line drain valve.
4	Front cover latch button	For opening the front cover.
5	Power switch	Push this button to turn on/off the instrument

3-2

## Front Panel

#### 2. Inside the Front Cover



Fig. 3.2

No.	Part Name	Description
1	Head holder	For mounting pump head. Includes a washing flow line for cleaning the plungers.
2	Pump head	The plunger reciprocates in the pump head to deliver solvent.
3	Drain pipe	Outlet of the washing flow line for the plungers
4	Check valve IN	Inlet check valve
5	Pump inlet	The reservoir suction filter (or selector valve outlet) is connected to this inlet.
6	Pump outlet	Usually for connecting the tubing to the injector.
Ø	Line filter	Traps dirt, etc. to prevent column clogging
8	Drain valve	The valve has a built-in pressure sensor, and is used to replace mobile phase or to release air from the flow line.
9	Drain port	Drain tube is connected to this port.
10	Check valve OUT	Outlet check valve
0	Тгау	If washing solution leaks from the washing seal, this tray catches the washing solution.



3. 2





Fig. 3.3

No.	Part Name	Description
1	External input/output terminal block	Used to make connection with external equipment.
2	Earth terminal	Used for grounding the instrument
3	Power cord connector	Used to connect a power cord.
4	Fuse holder	Two fuses are in the holder.
5	SOL.V connector	Used to connect a solvent selector valve unit FCV-I0AL/FCV-11AL.
6	REMOTE connector	Used to connect with SCL-IOA or an additional LC-IOAT. See Sec- tion 4.9 "Installation of the High Pressure Gradient Elution System."

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Fig. 3.4

1	Drain port of the pan in the front panel	For connecting the drain tube for solvent Icakage. See Section 4.5.5. "Connection of the Drain Tube for Solvent Leakage."
2	Mounting screws for the gradient mixer	A mixer or a manual injector can be mounted here.
3	Fan vent	For cooling the interior. Do not block this opening.

# Chapter 4 Installation

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For correct and safe use of the instrument, proper care about the installation site should be taken.

- 1. **Environmental Conditions** To assure long service life and good performance of the instrument, avoid installation in a place exposed to corrosive gases or dust.
  - Precautions on Ventila-Provide adequate ventilation when using flammable or toxic tion and Fire solvents as mobile phase.
    - Never use an open flame in the room particularly when flammable solvents are used.
- 3. **Electromagnetic Noise** Avoid installation in the vicinity of such equipment that generates a strong magnetic field. Use an additional noise filter if the power line has much noise.
- 4. **Installation Space** The LC-1OAT is designed to be used on a table or stand, preferably Requirements a solid and flat surface with a depth of 60cm or more. See Section 4.2 "Examples of System Configurations" for typical configurations of systems and installation spaces.
- <<Cautions>> In selecting the installation site, due care should be taken with regard to the following items in order to assure the optimum performance of the instrument.
  - Ambient temperature should be within 5 to 35°C, and without (1)extreme fluctuations.
  - Do not expose the instrument to the direct output from a (2)heater or a cooler.
  - Do not expose the instrument to direct sunlight. (3)
  - The installation site should be vibration-free. (4)
  - Relative humidity should be within 45% to 85%. (5)
  - LC-10AT is not designed in consideration of the safety regulation for medical equipments.
    - Therefore LC-IOAT can not be used in the patient environment." Patient environment: the area within a distance of 2.5m from
      - patients.

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5.

Use in the Patient

Environment

The following shows examples of HPLC system configurations incorporating the LC-1OAT.

1. Simple System An example of comparatively simple system configuration. It includes a column oven, detector, etc. that make a minimum set of modules required for stable isocratic analyses.



Fig. 4.1

2. Low Pressure Gradient Elution System 1

4. 2

An example configuration of a low pressure gradient elution system with manual sample injection. The system is highly economical and allows handling solvent delivery of up to four liquids for quanternary gradient elution.



Fig. 4.2

#### Examples of System Configurations

#### 3. Low Pressure Gradient Elution System 2

4. 2

A low pressure gradient elution system including an automatic injector controlled by the SCL-1OA System Controller. Centralized control of each module is available through the use of the system controller allowing great ease of operation. The system can also readily be used for automatic analyses.





4. High Pressure Gradient Elution System 1 An example configuration of a high pressure gradient elution system with manual sample injection. The LC-10AT high pressure gradient elution system allows highly accurate gradient analysis with a small time lag.





# Installation

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LC-10AT

#### **Examples of System Configurations**

#### 5. High Pressure Gradient Elution System 2

4. 2

A high pressure gradient elution system including an automatic injector controlled by the SCL-10A System Controller. In contrast with High Pressure Gradient Elution System 1, functions such as automatic sample injection, sample cooling, and selecting of mobile phase are available with this system, allowing flexible use for various types of analyses.



Fig. 4.5

## Mounting of Multiple Units

The LC-1OAT may be stacked one on another for use. The SPD-1OA detector, CTO-IOA column oven, SIL-10A automatic injector, etc. may also be stacked on an LC-IOAT. When stacking the units, it is possible to **fix** units together to prevent them from falling over during an earthquake or the like. The units should be fixed using the accessory tie plate.

- (1) Unscrew the screws that are used to fixed the unit cases.
- (2) Use the same screws to fix the tie plate.



Fig. 4.6

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LC-10AT

Before connecting the power source, confirm that the following conditions are met:

 Voltage and capacity of the power outlet Part number 228-31900-91, 92 90 - 130V~ 100VA 50/60Hz Part number 228-31900-93 200 - 250V~ 100VA 50/60Hz

The instrument will not exhibit satisfactory performance if operated on unstable line voltage or insufficient power capacity. In addition, the power capacity required from the overall system should be considered when preparing the power source.

• Make sure that the power switch on the main unit is turned off.

Connection to the power outlet
 Plug in the female end of the accessory power cord to the power connection on the back of the unit. Connect the male end to the power outlet.

<<Caution>>

- The LC-IOA employs a three-pronged power cord including a grounding wire. Be sure to connect the power cord to a three-pronged power outlet including a protective conductor terminal so as to ensure proper grounding.
- (2) For prevention of electric shock and to ensure stable operation, be sure to ground the instrument.

## Piping of the Unit

- 1. Preparation **c** Reservoir Prepare a reservoir of a capacity of 500ml or more.
- 2. Removing the Front Cover

Before connecting the flow lines of the unit, remove the front cover.

- (1) Press the latch button to open the cover.
- (2) The cover may be removed by pulling it while pressing the side of the cover as shown in the illustration below.



N/



- 3. Connection of the Suction Filter
- (1) Unscrew the transportation bushing dosing the pump inlet by hand.

Replace this bushing when the unit is not to be used for a long period of time in order to prevent the entrance of dust into the flow line.

- (2) Remove the 3D bushing from the suction filter tube.
- (3) Cut the tube to an appropriate length according to the distance between the reservoir and the pump inlet.
- (4) Put the filter in the reservoir as shown in the following illustration and run the tubing through the lid with four holes followed by the cap. Replace the 3D bushing back on the tube.
- (5) Connect the 3D bushing of the suction filter line to the pump inlet.
- (6) Fix the tube using the tubing clip as shown in the next illustration.





#### <<Cautions>>

- Keep the filter element clean to prevent clogging.
- For constantly stable analysis, the mobile phase in the reservoir should be degassed. See Section 4.6 "Connection of the Degasser."
- 4. Connection of the Drain (I) Tube (2)
- Remove the transportation cap mounted in the drain port. Screw the 1.6MN bushing (PEEK) of the accessory drain tubing into the drain port.
- (3) Prepare a waste container and put the other end of the drain tube in it.
- (4) Fix the drain tube using the tubing clip as shown in the next illustration.





#### **Connection of the Drain** Tube for Solvent Leakage

If liquid leaks in the unit, it accumulates in the pan on the front panel. Connect the drain tube for solvent leakage to lead the solvent to a waste container.

- (1)Fit the L-shaped drain tube to the drain port for solvent leakage at the right side of the unit.
- Connect the drain tube for solvent leakage and the L-shaped (2)drain tube using the straight joint.
- (3) Put the other end of the drain tube for solvent leakage in a waste container.

If the flow line of the drain tube for solvent leakage is located above the drain port for solvent leakage, the leaked solvent will not be discharged. Be sure to put the waste container lower than the unit, and direct the drain tube downward. Pour some water in the pan to check that waste will not back up but flow freely to the waste container,





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5.

<<Caution>>



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 Tubing for Solvent Leakage in the LC-10A System

Each component of the LC-IOA series has **a** drain port for solvent leakage at the front right side of the unit, which discharges liquid if leakage occurs in the unit. Tubing for solvent leakage should be conducted when installing the system.

- (1) Fit the accessory L-shaped drain tube to the drain port for solvent leakage on each component.
- (2) Connect the L-shaped drain tube fitted on the lowest component with a drain tube using a straight joint, and lead it to a waste container.
- (3) For the next higher component, use a T joint to connect on L-shaped drain tube and the tubing from upper component, and lead the other to the waste container. For the third and higher components, make interconnection with the other components using a T joint and a drain tube cut in an appropriate length. Be sure to incline the L-shaped drain tubes downward and fix them on the sides of the units using the accessory pipe clips, if necessary.

Place the waste container lower than the lowest unit.



Fig. 4.11

4-11

### Connection of the Degasser

Degassing of mobile phase is required for constantly stable analysis for the following reasons.

- (1) It prevents troubles that would occur from generation of bubbles.
- (2) It prevents unstable measurements that would occur due to variations in the concentration of dissolved gases.

Helium degassing and vacuum degassing through **a** resin membrane are popular in HPLC. Choose from these according to your needs. See Section 11.3 "Optional Units List."

As examples, the connection of DGU-2A and DGU-3A to LC-1OAT will be described in this chapter. The former is for helium degassing, and the latter is for vacuum degassing using a resin membrane.

1. Connection of the DGU-2A Use helium gas of high purity (99.995% or higher) for degassing.

- (helium degassing) (1)
  - ) Set a pressure regulator (optional) to the helium gas cylinder and connect the pressure regulator and the DGU-2A with a carrier gas pipe (optional).

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(2) For the reservoir, prepare a type LSI glass container. (A commonly used glass reagent container of a capacity of 500 or 1000ml).



Fig. 4.12

<<Caution>>

Never use a container with a crack or other flaw for a reservoir.

(3) Assemble the reservoir cap as shown in the illustration.



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Fig. 4.13

Category	Part Name	Description
1	Lid	Outer lid of the reservoir (B3)
2	Bottle cap	Inner lid of the reservoir
3	Teflon packing	Packing for the inner lid
4	Teflon tubing	3.0 x 2.0 x 230
* (5)	Teflon tubing	3.0 x 2.0, Connect to the pump inlet.
: 6	Filter element of the suction filter	Used to filter the mobile phase.
Ø	Helium gas bubbler frit	Helium gas bubbler frit
8	Teflon tubing	3 x 2 x 600 To be connected to an "OUT" (A, B, C, D) port of the DGU-2A.
9	Teflon tubing	3 x 2 x 600 To be connected to an "IN" (A, B, C, D) port of the DGU-2A.

= Parts (5) and (6) above are accessories supplied with the LC-10AT. Parts with other numbers are accessories supplied with the DCU-2A.

(4) Put the mobile phase in the reservoir. Also put the filter element assembled in (3) into the reservoir, then firmly close the cap.



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(5) Remove the caps set to DGU-2A ports to which you will make connection. Connect Teflon tubing led from the reservoir to OUT (A, B, C, or D) and IN (A, B, C, and D) ports of the DGU-2A, as shown in the illustration. Keep the unused ports covered with the caps.

Save the removed caps to use again to protect the degasser from entry of dust into flow lines when it is not to be used for a long time.



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LC-1**OAT** 

#### Connection of the Degasser

- 2. Ventilation of Helium Gas (1)
- Prepare a glass container. Fill it with a solvent that is miscible with the mobile phase.
- (2) Connect one end of the accessory Teflon tubing to the VENT port of the DGU-2A. Put the other end in the container and loosely cap the container with aluminum foil.
- (3) Place the container near a fume hood, ventilating fan or window so as to release the gas from the DGU-2A to outside the room.





**3.** Plumbing of the DGU-3A (1) Carry o

Carry out plumbing from the reservoir to the **DGU-3A** and from the **DGU-3A** to the LC-10AT as shown in the illustration below. First, remove the caps set to **DGU-3A** ports which you want to use. Keep unused ports covered with caps. Save the removed caps to use again to protect the degasser from entry of dust into flow lines when it is not to be used for a long **period.** 



Fig. 4.17

For gradient elution using the LC-IOAT, a special mixer (P/N 228-28000-91, optional) that is excellent in mixing solvents is available. Follow the instructions below for mounting the mixer and selecting mixer capacity.

1. Mounting Position

The mixer may be mounted in any of the following positions.

- (1) The right side panel of the LC-10AT
- (2) The inside left of the CTO-10A
- (3) The inside right of the CTO-10A

#### 2. Mixer Capacity

The following three capacities can be selected. Select the right mixer capacity according to the analysis requirements.

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STC:

- (1) **2.6**m*l*
- (2) 1.7 ml
- (**3**) 0.5m*l*
- 3. Selecting Mixer Capacity Mixer capacity can be changed by altering the plumbing. When it is shipped, it is set to 2.6ml. To change the setting, follow the instructions below:
  - (1) Remove the mixer cover.





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#### Mounting the Mixer

- (2) According to the desired capacity, change the plumbing as shown in the following illustration:
- (A) Plumbing for 0.5ml
- (B) Plumbing for 1.7ml
- (C) Plumbing for 2.6ml (Factory default)





(I)

- 4. Mounting the Mixer on the LC-10AT
- Move the pre-mixer section forward and fasten it by screws as shown in the following illustration:



Fig. 4.20

(2) Unscrew the screws on the right side of the pump, and fix the mixer with the screws as shown below:



Fig. 4.21

(3) Carry out piping between the mixer and injector referring to Section 4.9 "Installation of the High Pressure Gradient Elution System." E.,

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(4) Remount the mixer cover.



The pre-mixer joint sticks out of the cover.

Fig. 4.22

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#### 4-18

(1)

5. Mounting the Mixer in the CTO-10A

Mount the mixer on the inner wall of the CTO-IOA, using the accessory screws as shown in Fig. 4.24.

The mixer is **usually** mounted at the position indicated in (A). Note, however, that when mounting the 7725 manual injector with position sensor on the CTO-10A, the mixer should **be** mounted at the position shown in (B).



Fig. 4.23

(A) Mounting on the inside left wall









The following example of connections apply to the basic system shown in the illustration below. Connections should be made according to each system, referring to the examples.





For the method to mount the model 7725 manual injector and a column in the CTO-IOA column oven, refer to the instruction manual for the column oven. If not using the column oven, an injector holder and column holder (optional) are available for mounting the 7725 manual injector and column on the LC-10AT.

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Connection of the

As an example, the connection between the LC-10AT and model 7725 manual injector will be described.

- (1)Cut the accessory SUS pipe  $1.6 \times 0.3$  to the length required for piping from the pump outlet to the injector port 2.
- (2)Fit a 1.6MN bushing and 1.6F ferrule (that are supplied with the unit) at one end of the SUS pipe and connect it to the pump outlet. Also fit a bushing and ferrule (that are supplied with the manual injector) at the other end of the SUS pipe and connect that end to the model 7725 manual injector.
- The discharging pipes are connected to ports 5 and 6 of the 7725 manual injector. The other ends of the discharging pipes should be located at the same height as the injector.
- For pipe connections to No. 1 to 6 of the manual injector model 7725, use the bushings and ferrules that are supplied with the model 7725.

4

# Installation

#### 1. Manual Injector

<<Cautions>>

4-20



Fig. 4.26

**Connection between the** The following is an example of a typical connection between the manual injector model 7725 and column.

- (1) Cut the accessory SUS pipe  $1.6 \times 0.3$  to the length required for tubing from the injector to the column.
- (2) Fit bushings and ferrules at both ends of the SUS pipe.
- (3) Connect these ends of SUS pipe to the injector and column.

#### <<Cautions>>

2.

- In order to minimize sample broadening, make the tubing between the injector and column as short as possible.
- In order to eliminate dead volumes from the flow line, cut the pipe in such a manner that the cut face is perpendicular to the pipe axis.



Fig. 4.27
# Connection of the Injector, Column and Detector

## 3. Connection between Column and Detector

As an example, the connection between column and the SPD-10A detector will be described.

- (1) Cut the ETFE tube  $(1.6 \times 0.3)$  that is supplied with the SPD-10A to the length required to make connection from the column outlet to the cell inlet of the SPD-10A.
- (2) Fit bushings (I.6MN PEEK) at both ends of the ETFE tube.
- (3) Connect the ends of the ETFE tube to the column outlet and cell inlet (with a blue mark) of the detector as shown in the following illustration.

See cautions for "Section 4.8.2 Connection between the Injector and Column."





High pressure gradient elution systems using LC-IOAT are possible in two types of configuration characterized by the control method.

- (1) A high pressure gradient elution system of two or three solvents controlled from the SCL-IOA system controller.
- (2) A two-solvent high pressure gradient elution system controlled by the LC-IOAT. Installation for both types of configuration will be described in the following paragraphs.
- 1. Wiring for a System Controlled by the SCL-10A
- (1) Connect the REMOTE connector on the rear of the unit and the REMOTE connector on the rear of the SCL-IOA using the optical cable included in accessories for the unit. Channel 1 and 2 of the REMOTE connector are reserved for the SIL-IOA and FRC-10A. Channels 3 to 8 can be used for the pump(s).
- (2) The ADRS parameter of each LC-IOAT must be set to the channel number with which the unit is connected to the SCL-10A. (See Section 5.5 "Auxiliary Functions (AUX. FUNC).")



Fig. 4.29

4

## Installation of a High Pressure Gradient Elution System

- 2. Wiring for a System Controlled by the LC-10AT
- Connect two LC-10ATs by the REMOTE connectors on the (1) rear panels, using the accessory optical cable.



Fig. 4.30

- Set 85 to the ADRS parameter of each LC-IOAT. (2)(See Section 5.5 "Auxiliary Functions (AUX.FUNC).")
- Piping of the High **Pressure Gradient Elution System**

As an example, piping for a high pressure binary gradient elution system with two LC-10ATs, a degasser and a mixer will be described.





Carry out piping referring to the following connection method.

Prepare a reservoir. Degassing of mobile phases is necessary (1)for stable gradient elution of good reproducibility. Carry out piping up to the pump inlet referring to Section 4.6 "Connecting the Degasser."



4-24

- (2) Mount the mixer referring to Section 4.7 "Mounting the Mixer."
- (3) After mounting a mixer on the lower LC-10AT, make connection of the LC-10ATs and mixer inlets as shown in the following illustration.

First remove the plugs on inlets A and B of the pre-mixer section, and carry out piping from the outlets of each LC-10AT to inlet A and B using the accessory SUS pipe (1.6  $\times$  0.3).

For a ternary gradient system, remove the stop joint from inlet C and carry out piping from the inlet C to the pump outlet of the third LC-10AT.



Fig. 4.32

- (4) Connect the injector and the mixer outlet with the SUS pipe  $(1.6 \times 0.3.)$
- (5) Fix on the cover of the mixer referring to Section 4.7 "Mounting the Mixer."

This pump allows configuration of low pressure gradient elution systems that can handle up to four liquids, using the FCV-IOAL low pressure value module. The installation of the system is described in the following paragraphs.

The simplest low pressure gradient elution system consists of the units and parts listed below:

- (1) LC-10AT 1 ea.
- 2 Mixer 1 ea.
- (3) FCV-IOAL Low pressure value module 1 ea.
- (1) Make sure that the power switch of the module is turned Off.

E.

- (2) Using the cable included in the accessories for the FCV-IOAL, connect the FCV-IOAL and the SOL.V connector on the rear of the pump module.
- (3) Fasten the fixing screws on the connector of the module with a screw driver. The FCV-IOAL end of the cable has a flat cable connector; As such, fixing by screws is not done for the FCV-10AL connector.



Accessory cable FCV-10AL rear panel



In order to reduce dead volume in the flow line and allow a minimum time lag for gradient elution, install the FCV-10AL as close to the LC-IOAT as possible.

Carry out piping referring to the following connecting method.

 Prepare reservoir containers. Because degassing of the mobile phases is needed, carry out piping to the inlet joints of the FCV-1OAL referring to Section 4.6 "Connecting the Degasser."

- 1. Configuration œ pressure gradient elution system
- 2. Wiring to the FCV-10AL

<<Cautions>>

3. Piping of the Low Pressure Gradient Elution System

(2) Connect the outlet of the 5-way branching block and the pump inlet of the module with the accessory tube (40cm length).

The FCV-IOAL has four inlet ports (from A to D). If not every port is used, disconnect the pipe corresponding to the unused flow line from that inlet of the 5-way branching block. Then, be sure to fit the unused inlet with a plug (included in the accessories for the FCV-1OAL).



Fig. 4.34

(3) Remove the normal drain value line filter with the supplied wrench. Mount the low pressure line filter included with the FCV-1OAL.



- (4) Install the mixer, referring to the instruction in 4.7 "Mounting the mixer".
- (5) Connect the LC-IOAT and the inlet of the mixer as shown in the figure below.

Remove the plugs of inlets A and B of the pre-mixer, and connect the pump outlet of the LC-IOAT with inlet B by using the  $1.6 \times 0.3$  SUS pipe provided with the mixer.

Cover inlet A with the stop joint included with the mixer.





Fig. 4.36

- (6) Connect the injector and the mixer outlet with the  $1.6 \times 0.3$  SUS pipe.
- (7) Mount the cover, referring to the instruction in 4.7 "Mounting the mixer".

# **Chapter 5 Operation**

5

# Contents

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The operation of the unit is performed through the keys on the operator's panel. The status of operation may be checked on the display at any time. When using the SCL-IOA for control, see Section 7.2 "Control from the SCL-10A."

#### 1. Start-up

(1) Press the power switch located at the lower left of the front panel to turn on the unit. The unit is turned off by repressing the switch.



(2) When the power is switched on, a memory check is carried out automatically. If there is nothing irregular in the memory, the ROM version number is displayed for several seconds and then the display shown in the following illustration will appear to enable operation. (Values displayed vary according to setting.) This is the initial state.





If an error message is displayed after turning on the unit, see Section 9.2 "Error Messages" for appropriate measures.

<<Caution>>

# **Basic Operation**

2. Description of the Display The display has a screen and LED lamps. The function of each part is as follows:



Fig. 5.3

No.	Indication or Name	Function
1	flow/press	Indicates the set flow rate (ml/min) when the module is in the constant flow solvent delivery mode, or the set pressure value ( $\times 10^5$ Pa) in the constant pressure solvent delivery mode.
2	pressure	The pressure value measured by the pressure sensor $(x \ 10^5 \ Pa)$
3	p.max	Indicates the set upper limit pressure $(x 10^5 Pa)$
4	p.min	Indicates the set lower limit pressure $(x \ 10^5 \ Pa)$
\$	pump	Pump operation indicator lamp The lamp is lit when the pump is in operation.
6	program	Programmed operation indicator lamp The lamp is lit when <b>a</b> program is running.
Ø	remote	<b>REMOTE</b> mode indicator lamp Flashes when the pump is controlled by the SCL-10A.
8	c.flow	Constant flow mode indication lamp The lamp is lit when the module is in the constant flow solvent delivery mode.
9	c.press	Constant pressure mode indication lamp The lamp is lit when the module is in the constant pressure solvent delivery mode.

Note.  $10^5$  Pa = 1 bar = 1kgf/cm<sup>2</sup>

# 5. 1 Basic Operation

# 3. Description of Keys

The module has 20 keys on the front panel which are used for operation and setting. Functions of each key are as follows.

pump	run	7	8	9
purge	CE	4	5	6
del	back	1	2	3
edit	func	0	•	Enter

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**N**.,

<b>pump</b> Pump key	Key for start/stop of the pump. Press this key to start the pump. By pressing this key during operation, the pump stops. If it is pressed when purging, purging stops.
<b>purge</b> Purge key	Key for stadstop of purging. Press this key to start purging. Purging stops automatically after <b>a</b> period which can be set with the P.TIMER parameter. Purging can be <b>also</b> stopped manually by pressing this key during operation.
(del) Delete key	Deletes one line from the program during time program editing.
(edit)Edit key	Shifts to the time program edit mode.
run Run key	Key for stadstop of <b>a</b> time program. (If no time program has been set, this key has no effect.)
CE Clear entry key	<ul> <li>Sets the display screen to the initial state.</li> <li>If it is pressed during entry of numeric values, values entered so far are cleared.</li> <li>If it is pressed when an error message is on the display, the screen is cleared and the alarm is canceled.</li> </ul>
back Back key	<ul> <li>Press this key to select the previous parameter during the editing of a time program.</li> <li>If this key is pressed during the setting of the parameters for basic operation, entry position for setting of the parameter moves to the left, i.e., to the next left parameter.</li> <li>If it is pressed during AUX.FUNC setting, the setting display shows the previous parameter in the list.</li> </ul>

[func Function key	<ul> <li>If this key is pressed during the setting of a parameter for basic operation, the entry position for setting of a parameter moves to the right, i.e., to the next right parameter.</li> <li>Press this key to select the next parameter for entry during the editing of a time program.</li> <li>If it is pressed during AUX.FUNC setting, the setting display shows the next parameter in the list.</li> </ul>
[Enter) Enter key	Pressing this will register the value keyed in from the numeric keys.
0 - 9 Numeric keys	Used to key in numeric values for each settable parameter.

**Basic Operation** 

4. Settable Parameter List

5. 1

Settable parameters, their ranges, minimum values, initial values, and flow modes for basic operation are as follows:

Settable parameter	Settable range	Settable step	Default or Initial value	Mode
flow	0 – 9.999m <i>l</i> /min	0.001 ml/min	0ml/min	Constant now solvent delivery
press	10 - 400 [x 10 <sup>5</sup> Pa]	l [x 10' Pa]	10 [x 10 <sup>5</sup> Pa]	Constant pressure solvent delivery
p.max	10 - 440 [x 10' Pa] <sup>Note</sup>	[× 10' Pa]	$100 \ [\times 10^5 \ Pa]$	
p.min.	0 – 440 [x 10' Pa] <sup>Note</sup>	1 [× 13' Pa]	0 [x 10' Pa]	

Note: The maximum pressure value is limited to 220 [x 10<sup>5</sup> Pa] if the flow is set to 5.001ml/min or higher.

- 5. Basic Setting Procedures The basic procedures used for entry of the values in the setting display are as follows:
  - (1) Set the unit to the initial state (that appears when power is turned on). If the unit is not in the initial state, press  $\overline{CE}$  to set it to the initial state. Then numerics corresponding to FLOW/PRESS in the display will flicker, indicating that the entry of numeric values to this parameter is enabled.

flow/press	pressure	p.max	p.min	
0.000	0	100	0	_1

- (2) Key in **a** value with the numeric keys then press [Enter] to register the value.
- (3) If you want to set another parameter, press <u>func</u> to move the flashing field to the right.



## **Basic Operation**

- (4) To cancel a value just keyed in, press (CE) before pressing (Enter).
- (5) To return the display to the initial state after setting, press  $\overline{(CE)}$ .

6. Setting of the Maximum Pressure Value
To protect columns and other components in the flow line, set an upper pressure limit. If the pressure value measured by the pressure sensor exceeds the upper pressure limit, the limiter will be activated to stop solvent delivery automatically.

When the limiter is activated, an alarm beeps and the following message will be displayed.

flow/press pressure p.max p.min ERROR **P - MAX** 

The setting procedures are as follows:

Example: To set p.max (upper pressure limit) to  $150 \times 10^5$  Pa]: (1) Press func to enable the entry of p.max (the entry field flashes).

(2) Press (1), (5), (0), and (enter). The following display appears, and the setting is complete.

flow/press	pressure	p.max	p.min
0.000	0		0

7. Setting the Lower Limit Pressure To prevent drawing air into the flow line when the mobile phase in a reservoir has been exhausted, or for safety measures in the case solvent is leaking from the flow line, a lower pressure limit should be set. The limiter will be activated if the pressure is still lower than the minimum pressure even one minute after starting solvent delivery. When the limiter is activated, the alarm beeps, the solvent delivery stops automatically and the following message will be displayed.

5

### <<Setting Example>>

<<Setting Example>>

# **Basic Operation**

flow/press	Pressure	p.max	p.min
ERROR	P - MIN		

The setting procedures are as follows:

Example: To set p.min (minimum pressure) to 20  $[x 10^5 Pa]$ :

(1) Press func to enable the entry of p.min (the entry field flashes).

flow/press	Pressure	p.max	p.min
0.000	0	150	

(2) Press (2), (0), and (Enter). The following display will appear, and the setting is complete.

flow/press	Pressure	p.max	p.min
0.000	0	150	20

<<Caution>>

A value of 0 disables the low pressure limiter function.

- An Operation Example of The following is an example of operation for constant flow solvent delivery:
   Delivery (1) Prepare a mobile phase and pour it into a reservoir followed
  - (1) Prepare a mobile phase and pour it into a reservoir followed by the suction filter.
  - (2) Carryout piping of flow line referring to Section 4.8 "Connection of the Injector, Column and Detector."
  - (3) Turn the drain valve knob counterclockwise by 180° to open the drain valve. Make sure that the drain tube is put into a waste container.



Fig. 5.4

- (4) Press the power switch.
- (5) Check that the pressure value indicated on the display is in the range from -3 to  $3 \ [\times 10^5 \text{ Pa}]$ . If not, carry out zero adjustment for the pressure sensor. (See Section 5.5 "AUX.FUNC Functions.")
- (6) Check that the upper pressure limiter and lower pressure limiter are appropriate. (See Section 5.1 "Basic Operation.")
- (7) Press purge , then the pump will operate with a flow rate of about 9.9ml/min.

In normal operation, solvent delivery at the preset flow rate starts immediately after the pump or purge key is pressed. In the two cases (**a.** and **b.**) below, however, solvent is delivered at a low rate for a few seconds until the home position of the pump is detected. After the home position is detected, the solvent delivery rate increases to the preset value.

- a. The <u>pump</u> or <u>(purge</u>) key is pressed for the first time after power-on.
- b. The (pump) or (purge) key is pressed for the first time after the high pressure limiter is activated with a limit value of 220 [x 10<sup>5</sup> Pa] or higher.
- (8) Observe the condition of solvent discharged from the drain tube. If the liquid is being discharged smoothly without bubbles, the unit is operating normally.

<<Note>>

# **Constant Flow Solvent Delivery**



Fig. 5.5

<<Caution>>

If solvent does not flow, use the disposable syringe and syringe needle to draw the mobile phase through to clear the flow path as shown in the illustration below.





- (9) Press purge or (pump) to stop the pump.(10) Set the desired flow rate.
- (10) Set the desired flow rate. Example: When you want to set 1ml/min, press 1 and Enter.



<<Caution>>

# **Constant Flow Solvent Delivery**

- (11) Turn the drain valve knob clockwise to the stop to close the drain valve.
- (12) Press (pump) to operate the pump.
- (13) Check that the pressure at the pump outlet increases and becomes stable.

If the drain valve is left open, mobile phase may siphon out from the drain port.

Keep the drain valve closed except for the time when draining or purging the system.

A.

R.

# An Example of Operation The following is an example of the operation for constant pressure solvent delivery. Solvent Delivery (1) Carry out the operating procedures from (1) to (9) of Section

- 5.2 "Constant Flow Solvent delivery."(2) Change the solvent delivery mode from the constant flow solvent delivery mode to the constant pressure solvent
  - solvent delivery mode to the constant pressure solvent delivery mode. First, press func until the following display appears on the screen.

flow/press	pressure	p.max	p.min
0.000	MODE	СНАМ	I G E

(3) Each time Enter is pressed, the solvent delivery mode changes. When the unit is in the constant flow solvent delivery mode, the c.flow lamp is lit, and in the constant pressure solvent delivery mode, the c.press lamp. Now set the unit so that the c.press lamp is lit.

pump	prog run	remote	cflow	cpress

(4) Press (CE) after shifting the mode to return to the initial setting display. Check that the flow/press entry field is ready for entry of values (flashing).

flow/pre:	ss pressure	p.max	p.min
	0	100	0

(5) Set a desired pressure with the numeric keys.
Example: When you want to set 20 [x 10<sup>5</sup> Pa], press 2,
0, and Enter.

flow/press	pressure	p.max	p.min
	0	100	0

- (6) Turn the drain valve knob clockwise to the stop to close the drain valve.
- (7) Press (pump) to operate the pump.
- (8) Check that the pressure increases and becomes constant at approx. 20 [ $\times 10^5$  Pa].

Using the time program function, parameters such as flow rate may be changed automatically at user-defined time intervals. The created time program may be stored as a file and it can be combined with other time program files for sequential running.

#### **1.** Command List The following con

The following commands can be used in a time program.

Command	Description	Settable range	Remarks
FLOW	Flow rate (effective only in the constant flow solvent delivery mode)	0 – 9.999ml/min	The minimum step is 0.001ml/min
PRESS	Pressure (effective only in the constant pressure solvent delivery mode)	10 - 400 [×10 <sup>5</sup> Pa]	The minimum step is I [x 10' Pal
sv	Open/close solenoid valve in the FCV-IOAL or FCV- 11AL(S) (optional).	0, 1, 2, 3, 123	See Note 1.
EVENT	Event output ON/OFF	0, 1, 2, 12	Select one of the four numeric values.
LOOP	Repeats a program from the first step.	from 0 to 255 Value 0 repeats a program 256 times.	The minimum stcp is 1.
STOP	Ends a program.	<u> </u>	
GOTO	Runs programs by linking files. (Up to 10 files)	0 - 9	
BCONC	Concentration of solvent B (effective only when $SYS = 2 \text{ or } 4$ )	0 - 100% Note 2	The minimum step is 0.1%.
CCONC	Concentration of solvent C (effective only when SYS=4)	0 - 100%	Same as above
DCONC	Concentration of solvent D (effective only when SYS=4)	0 – 100%	Same as above

Note 1. If the FCV-IOAL has been specified by setting the FCV TYPE parameter, the settable valve is 1, 2, 3, or 4. If the FCV-11AL(S) has been specified, the settable value is 0, 1, 2, 3 or combinations of those values.

Note 2. The concentration of solvent A is determined by subtracting BCONC, CCONC and DCONC from 100. ACONC = 100 - (BCONC + CCONC + DCONC)

5-12

1

## Time Program Creation and Execution

2. **Description** of the **Display** 

5.4

To create a time program, set the unit to the edit mode, then proceed with programming. The procedures are as follows:

(1) Press (edit) and the following display will appear.



- ① Number of steps already programmed
- ② Abbreviation of the word "used"
- ③ Number of free steps remaining
- (4) Abbreviation of the word "left"

The above example shows that 10 steps of the time program have already been written and that there are 310 remaining steps.

(2) Now press (Enter) and the following display will appear.



- ① Elapsed time (minutes and decimal fraction) from the start of program
- ② Command or function name
- ③ Set value or argument
- (3) Press <u>Enter</u>) once again; the first step of the program will appear.

flow/press	pressure	p.mox	p.min
5.000	FLO	N 5	5.00

For detailed creation of a program, see the following Section 5.4.3 "Creation of Time Program."

3. Creation of Time Program The sequence of creating a time program starting from the initial state is as follows. Arbitrary values are shown for example only.



E.

5.4

# **Time Program Creation and Execution**

5.4

An Example of Creating An example of creating a time program will be described taking the case of varying the flow rate. (Let's assume the initial flow rate is 1ml/min.)

TIME	FUNC	VALUE
15.0	FLOW	2.00
20.0	FLOW	5.00
30.0	STOP	



(1) Press (CE) to return to the initial display.

flow/press	pressure	p.max	p.min	
1.000	0	100	0	_1

(2) Press (edit)

fic	w/press	pressure		p.max	p.min
,	0	U	3	20	LΕ

(3) Press [Enter].

flow/press	pressure	p.max	p.min
ŤĬME	FUNC	V A	LUE

(4) Enter the time (minutes, tenths and hundredths) for the initial step. Press 1, 5 and Enter.



(5) Select an instruction. When an instruction other than FLOW is displayed, press func until FLOW is displayed and then press (Enter).

flow/press	pressure	p.max	p.min
15.00	FLOW	VÀ	Ļίίε

(6) Enter the flow rate. Press (2) and (Enter)

flow/press	pressure	p.max	p.min
12.160	FLOW	2.	00

(7) Like the procedure from (4) to (6) above, set the second step. Press 2, 0, Enter, Enter, 5 and Enter.

flow/press	pressure	p.max	p.min
20.00	FLOW	5.	00

(8) Enter a step for the STOP instruction. After entering a time value by the same procedure as (5), press | func | to display STOP, then press (Enter).

flow/press	pressure	p.max	p.min	
30.1000	STOP			_1

(9) Press (CE) to end the editing of the time program. A time program having three steps has now been completed in this example.



(10) By pressing <u>CE</u> once again, the initial display shown in
 (1) will return.

# Time Program Creation and Execution

5. Deleting a Step

Display the step you want to delete and press del. The following is an example of deleting the first step of the program. created in Section 5.4.4 "An Example of Creating a Time Program."

(1) As in the creation of a program, display the program step you want to delete.

 flow/press	pressure	p.max	p.min	
15.00	FLOW	2.	00	_

(2) Press (del).

flow/press	pressure	p.max	p.min
	FLOW	5.	0 0

Now the first step of the program is deleted and the second step is displayed. (This is now the first step of the program.)

6. LOOP Instruction

Use of the LOOP instruction permits repeating a program at a userdefined time interval for a specified number of times.

	TIME	FUNC	VALUE
ᠿ	15.00	FLOW	2.00
2	20.00	FLOW	5.00
	30.00	LOOP	3

With this setting the program will be executed four times; The initial execution plus 3 looped executions for a total time of 120 minutes as illustiated here:



<<Cautions>>

All steps set (timewise) after the LOOP command are ignored, with the exception of a GOTO command step.

## **Time Program Creation and Execution**

- After the LOOP is completed, the program stops automatically. If a GOTO instruction exists after LOOP instruction, the pump parameter conditions are maintained until the time of the GOTO instruction; at which time the program executes the GOTO instruction and stops. (See below)
- A number up to 255 may be set as VALUE for the LOOP instruction. Note, however, that setting 0 means LOOP 256 times. Delete the LOOP step to disable looping.
- When setting multiple steps, it is not necessary to enter steps in the correct time sequence. The unit sorts the steps automatically.
- Note that a STOP instruction at the last step is usually required except when you want to execute the time program endlessly, or link program files by the GOTO instruction.

To start or stop a time program that has been completed, follow the procedures below.

(1) Starting the Program
 Press run.
 The prog run lamp lights up and

the program starts.

(2) Stopping the Program

To stop the program, two methods are available. One is to forcibly stop it by pressing the STOP key, and the other is to stop by means of a STOP instruction step set in the program.

The case of a forced stop is described here.

Press (run).

The **prog run** lamp is turned off and the program stops.

tructionThe GOTO instruction stops the program and switches the activening viafile from the present file to another file whose number is specifiedgramin the GOTO instruction argument.

After the file is switched, the instrument parameters are set to the initial conditions specified in the new file.

The automatic start of the new file's time program (if extant) is possible by carrying out the following setting.

 Make connection between the external Input/Output terminals as illustiated here;

7.

Start/Stop

8. GOTO Instruction (File chaining via Time Program execution)



Fig. 5.7

- (2) Set the EVENT parameter to 0 in the initial conditions of the temporary file. Or carry it out at sometime in the time program *before* the GOTO instruction is executed.
- (3) Set the EVENT parameter to 1 in the initial conditions of the new file.

As soon as the new file is installed and activated (by the GOTO command), the relay closure (EVENT 1) will trigger the start of that file's time program.

Thus, each of the 10 files available can be installed and activated in any desired sequence ("chaining").



The unit has a number of auxiliary functions (AUX.FUNC). Use of these functions permits a variety of operations to be performed by this device. It is also possible to control the unit from an external device via some of these functions

## 1. AUX.FUNC List

5.5

The following table lists the AUX.FUNC functions. Details of each are on following pages

Туре	Name	Function	Factory default value
Ι	BCONC	Concentration of solvent B in the gradient elution mode.	0
1	CCONC	Concentration of solvent C in the gradient elution mode.	0
1	DCONC	Concentration of solvent D in the gradient elution mode.	0
1	FILE NUM	Selects tile.	0
1	FILE COPY	Copies a file.	
2	FILE DEL	Deletes a file.	<u> </u>
1	SV	Sets the condition of solenoid valve unit FCV-10AL or FCV-I IAL.	Ι
1	EVENT	Sets the state of relay contact point output.	0
1	EXT-S	Lets the EVENT l output work <b>as</b> a start signal for a time program. It also lets the EVENT 2 output work as <b>a</b> stop signal for external equipment in occurrence of an error.	0
Ι	S-PROT	Reduces flow rate without stopping the pump when the pressure exceeds the set value of p.max.	0
2			
1	MON FLOW	Monitors solvent delivery flow rate in the constant pressure solvent delivery mode.	0
1	MON TIME	Monitors elapsed time when running a time program.	0
1	MON REV	Monitors accumulated number of pump revolutions.	0
1	MON ID	Monitors pump's ID.	0
Ι	P-SET	Used when replacing the plunger and plunger seal.	<del></del>
1	COMP	Used for fine adjustment of compensation of solvent compressibility.	0.45
2	ZERO ADJ	Carries out zero adjustment for monitoring pressure.	
2	CLOSE KEY	Disables key entry.	_
1	RANGE	Sets the span for pressure signals in the recorder output.	10
1	SYS	Specifies isochratic or gradient system in use.	1
1	FCV TYPE	Sets the type of solvent selector valve connected to the SOL.V connector on the rear of the unit.	0
1	LOCAL	Selects whether control is made from the system controller (=1) or by the pump (=0).	0
1	P TIMER	Sets the period of purging	3
1	ADRS	Sets the address to which the unit is connected.	1

• BCONC is displayed only when SYS = 2 or 4.

• CCONC and DCONC are displayed only when SYS = 4.

The type column in the list shows the type of operating method.

- Type 1: Requires a number argument to execute; key in a value with the numeric keys then press  $\overline{(Enter)}$ .
- Type 2: Press [Enter] to directly execute the function.

Type 3: Monitor; The current value (condition) is displayed.

Operation

SV

Setting Procedures for

2.

# Auxiliary Functions (AUX.FUNC)

AUX.FUNC		flow/press	pressure	p.max	p.min
(Concentration setting)	0.000		BCONC		0. 0
	The concentration set for the gradier B, C or D with t	n (volumetrient nt elution mo he numeric k	c ratio) of de. Key in teys, then pr	solven the cond ress $En$	t B, C and/or b centration of sol ter). (Unit: %
FILE NUM	The unit permits	creation of	up to 10 t	files of	programs that
(File selection)	be stored in the	memory. A	file may b	be selec	cted (i.e., instal

through this parameter.

flow/press	pressure	p.max	p.min
0.000	FILE	MUN	И <u>1</u>

Key in the desired file number with the numeric keys, then press  $\overbrace{\text{Enter}}$  to install that file as the active file.

	flow/press	pressure	<b>p.max</b> p.min
(Copy of a file)	0.000	FILE	COPY 1

The program contents of the currently selected file (source file) are copied to **a** (destination) file of selected number. Key in the destination file number with the numeric keys, and press  $(\underline{Enter})$ .

FILE DEL	flow/press	nressure	n max n min	
(Deleting a program)	0.000	FILE	DEL	]

The time program of the currently selected file is deleted. Press  $\overline{(Enter)}$  . A new program can now be created in this file.

(Setting the state of	flow/press	pressure	p.max	p.min	
solenoid valves)	0.000	S V	\\ //	1	7

Any one of mobile phases can be selectively delivered by using the optionally available FCV-1OAL or FCV-11AL as a selector valve

# Auxiliary Functions (AUX.FUNC)

at the pump inlet. Key in the value corresponding to the desired mobile phase with a numeric key, then press Enter to switch the value.

Solenoid Valve type	Argument	Selected mobile phase
	1	Solvent A
FCV-I OAL	2	Solvent B
	3	Solvent C
	4	Solvent <b>D</b>

Solenoid valve type	Argument	Selected mobile phase	
	0	All channels arc set to the A side.	
FCV-11 <b>AL</b>	1	Channel I is set to the B side.	
	2	Channel 2 is set to the B side.	
	3	Channel 3 is set to the B side.	
In use, combine the arguments to select multiple channels.			
Example: By setting SV 123, channels 1, 2 and 3 are all set to the B side.			

Before setting SV, specify the kind of solenoid valve unit installed with the FCV TYPE parameter. See next.

FCV.TYPE	flow/proce	proceuro	n max	n min
(Specification of type of				
solenoid valve module)	0.000	FCV	IYPE	////

The optional solvent selector valve to be connected to the SOL.V connector on the rear of the unit is specified with this parameter. Key in the appropriate value with **a** numeric key, then press  $\boxed{\text{Enter}}$  to register the flow control value type.

Argument	Solenoid valve module
0	FCV-1OAL
1	FCV-11AL

EVENT		
(Setting	EVENT	relay
states)		

	flow/press	pressure	p.max	p.min
_	0.000	ΕVΕΝ	T //	// )   \ \

ON (contacts closed) and OFF (contact open) of the EVENT relay contacts (on the rear of the module) are set.

Key in the desired value with the numeric keys then press (Enter) to activate/deactivate the relay(s).

<<Note>>

Argument	EVENT 1 output	EVENT 2 output
0	Relay I OFF	Relay 2 OFF
1	Relay 1 ON	Relay 2 OFF
2	Relay I OFF	Relay 2 ON
12	Relay 1 ON	Relay 2 ON

# EXT-S (External signals) (Function setting for the EVENT relay terminals)

5.5

flow/press	pressure	p.max	p.min
0.000	ЕХТ —	s ``	

Sets control mode for the EVENT output (relays 1 and 2). key in the argument with a numeric key, then press (Enter).

Argument	Function		
0	Relays are controlled by the value set to EVENT. (Normal default condition)		
1	Relay 1 (EVENT 1) is used as a start output signal when the time program starts. (Event 2 operates normally)		
2	Relay 2 (EVENT 2) is used as an error output signal. (Event I operates normally)		
3	Combination of functions 1 and 2. (Normal EVENT 1/2 operations are disabled.)		

<<Note>>

The use of the EXT-S function disables normal operation of the EVENT parameter relevant to the corresponding EVENT terminal. Use care.

S-PROT (Setting the system protection function)



S-PROT controls solvent delivery in such **a** manner that when the P.MAX limiter is activated, flow rate is reduced by one half without stopping the pump, until the flow rate becomes lower than the P.MAX value. (Normally, exceeding P.MAX causes the pump to stop.)

Key in the argument with **a** numeric key, then press  $[\overline{Enter}]$ .

Argument	Function
0	Cancels the system protection.
1	Activates the system protection.

To cancel the P.MAX error alarm, press ICE).

5. 5 Auxilia	/ Functions (AUX.FUNC)
MODE CHANGE	flow/press pressure p.max p.min
(Selecting the solvent delivery mode)	0.000 MODE CHANGE
	Switches between the constant flow solvent delivery mode and t constant pressure solvent delivery mode. Press (Enter) to select the desired solvent delivery mode. T selected mode is indicated by lighting of either the C.PRESS
< <note>&gt;</note>	C.FLOW LED.

MON FLOW				
(Displaying flow rate in	flow/press	pressure	p.max	p.min
the constant pressure	0.000	MON	FLOV	<b>N</b> , 0
solvent delivery mode)				<u> </u>

Displays the approximate flow rate when in the constant pressure solvent delivery mode.

Key in the argument with a numeric key, then press (Enter).

Argument	Function	
0	Cancels the flow rate display function.	
1	Activates the flow rate display function.	

MON TIME (Monitoring the elapsed time of the time program)

flow/press	pressure	p.max	p.min
0.000	MON	ΤΙΜΕ	

Argument	Function
0	Cancels monitoring of the elapsed time of the time program.
1	Activates monitoring of the elapsed time of the time program.

# MON REV (Monitoring the accumulated number of pump revolutions)

flow/press		pressure	p.max p.min
	0.000	MON	REV

The accumulated number of pump revolutions is monitored. It counts up to 16,777,215, then resets to 0.

Key in the argument with a numeric key, then press (Enter).

## Auxiliary Functions (AUX.FUNC)

Argument	Function
0	Cancels monitoring the accumulated number of pump revolutions.
1	Activates monitoring the accumulated number of pump revolutions.

The accumulated number of pump revolutions serves **as** an indicator for seal replacement.

It is recommended to replace the seals every 2.5 million revs, i.e., approx. every 2000 hours (at 1ml/min,  $100 \times 10^5$  MPa using water).

# MON ID (ID indication for remote control)



If the unit is connected to the SCL-IOA, the pump connection address (A, B or C on the SCL-IOA) is indicated in the initial display. Key in the argument with a numeric key, then press Enter.

Argument	Function
0	Disables the 1D indication.
1	Enables the ID indication.

	flow/press	pressure	p.max	p.min
	0.000	PUMP	Α	

If the SCL-IOA is not connected, the following will be displayed.

flow/press	pressure	p.max	p.min
0.000	ΝΟΤ	LINK	ED



Moves the plungers and stops them in the home position for seal replacement.

Key in the argument with a numeric key, then press [Enter].

Argument	Function
1	The left and right plungers move until they reach the same position, and stop. Replace the plunger seals at this position.

COMP (Fine adjustment of compensation for fluid compressibility)

5.5

flow/press		pressure	p.max	p.min
	0.000	СОМР	0.	45

The LC-10AT employs compressibility compensation in order to reduce the fluctuation of the pressure due to solvent compressibility. Fine adjustment of compensation is possible by setting the compressibility factor for the solvent used.

For low pressure solvent delivery, changing of the parameter is not usually required, but for delivering solvents of greater compressibility such as hexane or methanol under a pressure of 200 [x 10<sup>5</sup> Pa] or higher, this parameter should be adjusted accordingly. Key in the compressibility factor  $(GPa)^{-1}$  as the set value with the numeric keys, then press [Enter]. The default value is 0.45.

Mobile phase	Compressibility (GPa) <sup>-1</sup>
Water	0.45
Acetone	1.24
Methanol	1.25
Hexane	1.6

Compressibility factors vary slightly with temperature; The values shown are for room temperature (~25°C). However, correction is usually not required for the usual range of temperatures encountered in HPLC.

(Zero adjustment for	flow/press	pressure	p.max p.min
pressure indication)	0.000	ZERO	ADJ

Output of the pressure sensor is reset to zero.

Press (Enter). The zero adjustment is carried out. Before using this function, open the drain valve and set the conditions so that no pressure is applied.

<<Note>>

ZERO AD.I

CLOSE KEY (Disabling key entry)	flow/press pressure p.max p.min 0.000 CLOSE KEY		
	Disables key entry. Press <u>Enter</u> ). The unit is set to the state in which key depression is ineffective. To cancel this key lockout effect, press <u>CE</u> and <u>func</u> simul- taneously.		
RANGE (Changes the output pressure signal FS range)	Sets the fullscale value for the output signal (through the "pressure" terminal on the rear of the unit). Key in the factor with the numeric		
	Full scale = (factor) x $50[\times 10^5 \text{ Pa}]$ . Full scale voltage is 1mV. Examples: When factor = 1, the full scale is $50[\times 10^5 \text{ Pa}]$ . When factor = 10, the full scale is $500[\times 10^7 \text{ Pa}]$ .		
< <note>&gt;</note>	The factor must be an integer value from 1 to 10; Fractional values are not accepted.		
SYS (Setting the system parameter)	flow/press pressure p.max p.min 0.000 SYS		

Sets a system parameter. Enter the set value with a numeric key, then press  $\overline{(Enter)}$ 

Argument	Function
1	Set this value when using the unit individually or when control is made externally.
2	Set this value when using the unit as a control pump in a two- module high pressure gradient elution system.
4	Set this value when using the pump in a low pressure gradient elution system.

<<Caution>>

Values other than the above should not be set.

5

LC-1**OAT** 

# LOCAL

5.5

(Setting the control mode)

flow/press pressure p.max p.min \\**I** // LOCAL 0.000 Ĩ١١

By setting this function when the unit is linked with SCL-IOA, the unit can be operated independently.

Key in the argument with a numeric key, then press [Enter].

Argument	Function	
0	Control is made by the SCL-IOA.	
1	The module is operated independently (local mode).	

P TIMER		
(Setting the	period of	purging)



Sets the period of purging (min).

Key in the required purge time with numeric keys, then press [Enter].

A value from 0.1 to 9.9 (min) can be selected.

ADRS			
(Setting the REMOTE address)	flow/press	pressure	p.max p.min
	0.000	A D R S	

If the unit is connected to the SCL-10A system controller or another LC-10AT, the connection address should be stated with this parameter.

Key in the address number with the numeric keys, then press [enter].

Address	Function
3 - 16	In the case when the unit is connected <b>to</b> the SCL-IOA, ADRS should be set to the <b>port</b> number of the SCL-IOA <b>to</b> which the unit is connected.
85	In the case when the unit is connected to another LC-IOAT, ADRS should be set to 85.

Values other than the above should not be set.

High pressure gradient elution is possible by controlling the 2 or 3 LC-10ATs in the system from the SCL-10A system controller, or by connecting two LC-10ATs with each other in such a manner that one of them (the "master") controls the other (the "slave"). The latter setup will be described here.

For the operating method for control by the system controller, see the System Controller Instruction Manual.

1. Preliminary Setting

3.

(I) Set system parameters for the master LC-10AT (called pump A) that controls the other (pump B) as follows:

$$SYS = 2$$
  
ADRS = 85

(2) Set system parameters for the slave LC-10AT (called pump B) that is to be controlled as follows:

$$SYS = 1$$
  
ADRS = 85

For the procedures for setting the system parameter, see Section 5.5 "Auxiliary Functions (AUX.FUNC)."

(3) Set the upper limit pressure (P.max) and lower limit pressure (P.min) for each pump. Values P.max and P.min set at each pump are effective.

From now on operation is carried out from the master pump A only.

It is advised to lock the keys of pump B to prevent misoperation.

- 2. Setting Initial Conditions (1) Set the total flow rate of the mobile phase (through the column, i.e., total flow rate of pump A and B) as the flow rate of pump A.
  - Referring to Section 5.5 "Auxiliary Functions (AUX.FUNC)," enter the concentration value for solvent B (unit: %) to parameter BCONC.

The concentration of solvent of pump A (ACONC) is as follows.

ACONC(%) = 100(%) - BCONC(%)

For the BCONC parameter, settable range is from 0 to 100%, and the minimum step is 0.1%.

- (3) By pressing (pump) on pump A, pumps A and B starts solvent delivery simultaneously.
- High Pressure GradientBy setting the concentration of solvent B in a time program, itDelivery Programis possible to change concentration of solvent A and B linearly (inverse percentage ratio) on a time basis.
### **High Pressure Gradient Elution Mode**

- 4. An Example of Program (1) Setting (2)
- **am** (1) Call the time program editor screen. (Press edit).)

(2) Enter a time.

- (3) Press <u>func</u> until BCONC is displayed on the setting screen, and then press <u>Enter</u>.
- (4) Enter concentration.

The following is an example of setting for changing the concentration through a time program.

(See Section 5.4 "Time Program Creation and Execution".) Initial value for BCNC: 0.0

TIME	FUNC	VALUE
10.00	BCONC	25.0
20.00	BCONC	100.0
30.00	BCONC	100.0
30.10	BCONC	0.0
40.00	STOP	

The program above results in a binary gradient elution in which the concentration of the solvent B can be graphically illustrated, as follows.



Note that the concentration delivered at any given time is based on a linear interpretation of proportionality between each defined point. (Nonlinear curves are available by control with the SCL-10A.)

- (1) Pump **A** controls flow rate of pump **B**, but all parameters of pump **B** except the flow rate (FLOW) are effective.
- (2) The pump key of each pump is always effective, so that pressing the pump key starts or stops delivery even if it is controlled by a time program of pump **A**.
- (3) When not delivering, pressing the (purge) key will allow for purging.

**Operation of Pump B** 

5.

<<Caution>>

Do not press the  $[\underline{RUN})$  key on pump B. If  $\underline{RUN}$  key is pressed, the time program (if any) in pump B will also **start.** This may result in faulty solvent delivery.

Operation

LC-1OAT

The module by itself allows running of **a** binary, ternery or quarternary low pressure gradient elution. (It is also possible to carry out the setting from the SCL-10A system controller.) The following is **a** description of setting **a** quarternary (A, B, C, and D) gradient elution using **a** single LC-1OAT equipped with **a** selector value and mixer, as described in Section 4.10.

### 1. Preliminary Setting

5.7

- (1) Enter 4 at the system parameter SYS of AUX.FUNC.
- (2) Enter total flow rate of the mobile phase (sum of flow rates of four liquids A, B, C and D) for the set value of the flow parameter.

Set the flow rate to 5ml/min here as an example.

(3) Next, set concentration parameters. As an initial operation is being done here, setting is made so that each flow rate is equal to fill **all** the flow lines with the liquids. Set each of BCONC, CCONC and DCONC to 25%. (See Section 5.5 "(AUX.FUNC)".

Concentration of liquid A (ACONC) is as follows.

ACONC(%) = 100(%) - BCONC(%) - CCONC(%) - DCONC(%)

For parameters BCONC, CCONC, and DCONC, the setting range is from 0 to 100%, and the minimum step is 0.1%.

Key input is automatically rejected if an attempt is made to enter **a** concentration to BCONC, CCONC or DCONC which causes the total conc. to exceed 100%.

- (1) Open the drain valve.
  - (2) Press (pump) to start the pump.
  - (3) Draw the mobile phase from the pump outlet to fill the flow line with the mobile phase.
  - (4) Make sure that no bubbles are in the flow line.
  - (5) Purge the flow line with the mobile phase for about 10 minutes.
  - (6) Press (pump) to stop the pump, and close the drain valve.
- **3.** Low Pressure Gradient (1) Set the (total) flow rate and the concentration(s) to the prescribed values, then press pump to start solvent delivery.
- 4. Low Pressure Gradient Delivery Program

**Initial Operation** 

By setting concentration of liquids B, C, and D at certain times in **a** time program, it is possible to vary the concentrations of liquids **A**, B, C, and D in linear proportions on **a** time basis.

5-32

<<Note>>

2.

# **Plunger Washing Method**

- 1. Connections
- (1) Connect the accessory Teflon tubes with the two Teflon tubes at the bottom of the head holder by using the accessory vinyl tubes.
- (2) Place the open end of one of the Teflon tubes into a waste container.



Fig. 5.0



Fig. 5.9

## **Plunger Washing Method**

2. Operation

<<Caution>>

- (1) Fill the disposable syringe with washing solution and insert the needle end into the rinsing tube.
- (2) Force the washing solution through the washing flow line. It is possible to conduct the washing irrespective of whether the pump is running or not.
- (3) Typical periods for manual washing are as follows. Phosphoric acid buffer solution (low concentration)
  : A few times/day Boric acid buffer solution (low concentration)
  - : Several times/day

Use of a buffer solution as the mobile phase will produce crystalline salts upon evaporation of the solution, and which can damage the plunger and plunger seal and shorten their service life. When using a buffer solution, accordingly, it is recommended to wash the flow line frequently. Continuous washing can be done using an inexpensive peristaltic pump or the like to pump water through the wash line at a few ml/min.

As a mobile phase may be or may not be miscible with another, or **as** the use of buffer solutions requires extra care in handling, change of **a** mobile phase should be conducted correctly according **to** the following description. If you also change the column to a different type, remove the first column and carry out the procedure before installing the next column.

- 1. Change between Miscible Mobile Phases
- (1) Put approx. 100ml of the fresh mobile phase into a container of 200ml capacity.
- (2) Take out the suction filter from the first reservoir and swirl it around in the 100ml of fresh mobile phase to remove **as** much of the old mobile phase from the accessable surfaces of the filter and its tube.
- (3) Put the suction filter into a newly prepared reservoir filled with fresh mobile phase, open the drain valve, and press purge to purge the previous mobile phase completely out of the flow line.



Fig. 5.10



### Change of Mobile Phase

(4) Break open the connection at the outlet of the manual injector and pipe the outlet to a waste container. Set the flow rate to 2-3ml/min then close the drain valve. Switch the injector between the load and inject positions and hold each position long enough to allow the sample loop and inner passages to be flushed. Thus, the mobile phase in the manual injector will be replaced.





(5) Reconnect the outlet of the manual injector, then replace the mobile phase in components in the flow line that follow the manual injector.

Flow rate should be so adjusted that the delivery pressure does not exceed the permissible pressure rating of the column.



Fig. 5.12

5

# Change of Mobile Phase

- 2. Change between Immiscible Mobile Phases
- (1) Prepare an intermediate washing solution (such **as** isopropyl alcohol) that is miscible with both previous and next mobile phases.
- (2) Replace the old mobile phase with the intermediate washing solution. See procedures (1) to (5) of Section 5.9.1 "Change between Miscible Mobile Phases."
- (3) Replace the intermediate washing solution with the fresh mobile phase. See procedures (1) to (5) of Section 5.9.1 "Change between Miscible Mobile Phases."

3. Replacement & Buffered Use of a buffered mobile phase may result in crystalline substances being deposited upon dehydration or evaporation of the solution. Accordingly, replace the buffer solution as follows. Note that if an organic solvent such as isopropyl alcohol is delivered, strongly bound salt deposits may form, so care should be taken.

- (1) Prepare distilled or deionized water.
- (2) Feed 200ml or more of the water to wash out the buffer solution. See procedures (1) to (5) of Section 5.9.1 "Change between Miscible Mobile Phases."
- (3) Then replace the water with the fresh mobile phase. See procedures (1) to (5) of Section 5.9.1 Change between Miscible Mobile Phases."
- Use of Washing Flow Line
   Use of a buffered mobile phase may result in the deposit of crystalline substances upon dehydration or evaporation of the solution: These salts can damage the plungers and plunger seals and shorten their service life. When using a buffer, it is advisable to frequently or continuously wash the back side of the plunger seals and plunger surfaces by pumping water through the built-in wash line. For the washing method, see Section 5.8 "Plunger Washing Method."

<Caution>> If you are not going to use the unit for some time (hours +) after feeding a buffer solution, replace the contents of the flow line with distilled or deionized water to prevent salt formation and deposition.

# Chapter 6 Performance Checking

	Contents	
6.1	Operation Checking for Simple Isochratic System	6-2
6.2	Checking Concentrations in High Pressure Gradient Elution	6-5



## **Operation Checking for Simple Isochratic System**

Operation checking for a simple isochratic system, as shown in the following illustration, is described here.





- Prepare as many reservoirs as required. (1)Prepare an appropriate mobile phase and sample. (2)
- 2. Connection Connect the input terminal of the Chromatopac and AUX terminal of SPD-10A by using the signal cable and terminal block included in the accessories for the Chromatopac and the signal cable included in the accessories for the SPD-IOA.
- 3. Operation

Preparation

6. 1

- (1)Switch power on for the whole system.
- (2)Purge the flow line (from the suction filter to the pump drain).
- Close the drain valve and operate the pump at 1ml/min. (3)
- Check that the pressure is stable and liquid is flowing from (4) the outlet of the SPD-IOA.
- Set the temperature of the CTO-IOA to 40°C. (5)
- On the SPD-IOA, set the wavelength to 254nm, response to (6) 4, and AUX RNG to 3. (The AUX output range becomes 2AU/V.)

6

1.

# **Operation Checking for Simple Isochratic System**

- (7) On the Chromatopac, set ATTEN to 4.
- (8) On the Chromatopac, press ZERO, 5, 0 and ENTER to set the pen position at the center.
- (9) On the Chromatopac, press <u>PLO</u> and <u>ENTER</u> to carry out plotting.
- (10) Wait until the baseline becomes stable.





- (11) On the Chromatopac, set ATTEN to 8.
  (The Chromatopac's full scale becomes equivalent to 0.5AU.)
  (12) On the Chromatopac, press ZERO, 2, 0, and ENTER].
  (13) On the Chromatopac, Dress PI OT, and FNTF R, to stop
- (13) On the Chromatopac, Dress <u>PLOT</u> and <u>FNTFR</u> to stop plotting.
- (14) Inject the sample. (see next page)
- (15) At the same time, press START of the Chromatopac.

## **Operation Checking for Simple Isochratic System**

4. Example of Analysis (conditions)

The following is an example of the performance check under the conditions below:

Mobile pha	se :	H <sub>2</sub> O/Acetonitrile	= 15/85		
Column	:	Shim-pack CLC	Shim-pack CLC-ODS 6.0mmø x 15cm		
Flow rate	:	1ml/min			
	:	Wavelength 254	nm, respor	nse 4	
Sample:	:	Naphthalene	10mg –	1	
		Biphenyl	5mg	in 100m <b>l</b>	
		Acenaphthene	30mg	Methanol	
		Anthracene	1mg	Inject 5µl	
		Pyrene	1Omg		

### (results)



The concentration of each solvent in high pressure gradient elution can be tested without a column or sample in the following method.

1.	Conditions	Solvent	А	:	0.3% acetone in water
			В	:	Distilled water
		Flow rate		:	1 m <i>l/</i> min
		Load press	sure	:	10 – 30 [×10 <sup>5</sup> Pa]
		Measureme	ent wavelength	:	254nm

#### 2. **Specification** At liquid A concentration ratios of 10, 50 and 90%, the concentration error should be within $\pm 1\%$ .

- 3. Precautions for Measurement
- (1)Be sure to degas the mobile phase. (2) Open the drain valve and wash the flow line for 5 minutes, with the flow rate set to 9ml/min and BCONC parameter to 50%.
- (3) Then set the flow rate to 3ml/min. Close the drain valve and wash the flow line for 20 minutes.
- (4) Last, set the flow rate to 2ml/min and the BCONC parameter to 100%. and check that the baseline has become stable, then start measurement.
- 4. **Measurement Example** Initial parameter setting Flow rate

: 1ml/min BCONC : 100



Time pro	gram	
TIME	FUNC	VALUE
0.10	BCONC	90.0
10.00	BCONC	90.0
10.10	BCONC	50.0
20.00	BCONC	50.0
20.10	BCONC	10.0
30.00	BCONC	10.0
30.10	BCONC	0.0
40.00	BCONC	0.0
40.10	BCONC	100.0

Calculation

$$\frac{A10}{A100} = 10\% \text{ actual concentration}$$
$$\frac{A50}{A100} = 50\% \text{ actual concentration}$$
$$\frac{A90}{A100} = 90\% \text{ actual concentration}$$

It is possible to read values of AIO, A50, A90 and A100 directly from the recorder chart, however, this method yields serious errors. Accordingly, it is recommended to run the following BASIC program on the Chromatopac, to print out the LEVEL.

BASIC	PROGRA	М
10	PRINT	LEVEL;
20	WAIT	60
30	GOTO	10

# Checking Concentration in Low Pressure Gradient Elution

1.	Conditions	Solvent A Solvent B, C, and D Flow rate Load pressure Measurement wave length	<ul> <li>0.3% acetone water solution</li> <li>Distilled water</li> <li>1ml/min</li> <li>10 - 30 [× 10<sup>5</sup> Pa]</li> <li>254nm</li> </ul>
2.	Specification	At Solvent A mixing ratios ±2% maximum	10, 50, and 90%, concentration error
3.	Precautions for Measurement	<ol> <li>Be sure to degas the</li> <li>Open the drain value a setting the flow rate to DCONC parameter to the unused inlets with with either solution u</li> <li>Then set the flow rate wash the flow line for</li> <li>Last, set the flow rate to 100%, CCONC and that the base line has be</li> </ol>	mobile phase. and wash the flow line for 5 minutes, 5 5ml/min and BCONC, CCONC, and 25%. In this case, be sure to connect the reservoir and conduct replacement sed. to 2ml/min. Close the drain valve and or 20 minutes. 1 ml/min and the BCONC parameter DCONC parameters to 0% and check become stable, then start measurement.

### 4. Measurement Example Low pressure gradient Elution using Solvent A and B



Initial para	meter setting
Flow rate:	1ml/min
B.CONC	: 100.0
C.CONC	: 0.0
D.CONC	: 0.0

Time	program		
	TIME	FUNC	VALUE
	0.10	BCONC	90.0
	10.00	BCONC	90.0
	10.10	BCONC	50.0
	20.00	BCONC	50.0
	20.10	BCONC	10.0
	30.00	BCONC	10.0
	30.10	BCONC	0.0
	40.00	BCONC	0.0
	40.10	BCONC	100.0

Calculation

A10 A100	= 10%	actual	concentration
A50 A100	= 50%	actual	concentration
A90 A100	= 90%	actual	concentration

It is possible to read values of A10, A50, A90 and A100 directly from the recorder chart, however, this method yields serious errors. Accordingly, **it is** recommended to run the following BASIC program on the Chromatopac, to print out the LEVEL.

BASIC	PROGRAM
10	PRINT LEVEL;
20	WAIT 60
30	GOTO 10

6

# Chapter 7 Control from External Equipment

7.1	Connections of External Equipment to the Input/Output Terminals	7-2
7.2	Control from the SCL-IOA	7-4



# Connections of External Equipment to the Input/Output Terminals

### 1. The Input/Output Terminals



Fig. 7.1

Signal name	Description	Remarks
EVENT 1	Reed relay contact point output. The contact points close and open according to EVENT parameter values set in a program or AUX.FUNC setting.	The rated values of the reed
EVENT 2	Reed relay contact point output. The contact points close and open according to EVENT parameter values set in a program or AUX.FUNC setting.	30VDC and 0.1A.
ON (PUMP)	The pump in the unit can be started by the external contact point signal through this terminal.	Control is made by short-cir- cuiting these signal terminals
OFF (PUMP)	The pump in the unit can be stopped by the external contact point signal through this terminal.	and "COM" terminal. The closure time (tc) should be as follows:
PRG START	The time program for the unit can be started by the external contact point signal through this terminal.	0.5sec < tc < 10sec, i.e., 500ms or more
COMMON	Common terminals for ON, OFF, START, and STOP signals.	
PRESSURE MONITOR	Outputs pressure values. The output scale may be set in ten steps within an output range of $(0 - 50 [\times 10^5 \text{ Pa}])$ to $(0 - 500 [\times 10^5 \text{ Pa}])$ . See Section 5.5 "Auxiliary Functions (Aux. FUNC)"	Full scale voltage = $1 \text{ mV}$

2. Wiring

- (1) Strip the insulation from the end of the connecting wire to leave approx. 10mm of exposed conductor. This treatment is not necessary for the accessory remote cable.
- (2) If the core wire is solid, just insert it into the hole of a terminal.

If it is stranded, twist the end well, then insert it into the hole while pressing the button on the right side using a small screwdriver or the like. To disconnect the cable, press the button and pull the cable.



Fig. 7.2

<<Caution>>

The accessory remote cable can be used for the wiring. If additional wire is required, carry out wiring with the following wires.

Solid wire:	Q0.4- ø1.0 (AWG26 - 18)
Stranded wire:	0.3mm <sup>2</sup> - 0.75mm <sup>2</sup> (AWG22 - 20)
	Element wire diameter: Ø0.18 at the minimum

To protect the wire from breaking, stranded wire is recommended. Twisted-pair cable composed of stranded wire with the exposed ends well tinned with solder is most highly recommended.

For the basic operation of the SCL-10A system controller, see the SCL-10A System Controller Instruction Manual.

1. Switching Power on When the SCL power switch is turned on, power is supplied and and the CONFIG Display a few seconds later the CONFIG display will appear.

If the solvent delivery unit and the SCL is connected correctly, LINKED is shown at the right of each unit name PUMP.A , PUMP.B and PUMP.C .

(In the case that three units are connected to the SCL.)

In this display, connection of units should be defined to control the solvent delivery unit from the SCL. If this definition is not carried out for  $\mathbf{a}$  unit, link is not checked for the unit before an analysis even if it is connected to the SCL.

This definition is required only once after installation. The SCL stores the information in itself, so reassignment is not required unless you change the system configuration.

Define connection of the solvent delivery unit as shown below.

unit **name** 

	PUMP. B	LINKED	LOCAL			
	PUMP. C	LINKED				
	DET.A	LINKED				
	DET.8	LINKED	LOCAL			
	A. I N J	LINKED				
	FRC	LINKED				
	OVEN		LOCAL			
	SUBC	LINKED				
	press MEN	U				
Pt	UMP. A PUM	P.B. PUMP. Is smpl/	C DET.A	DET.8	- Function	line
9999	ime a.fi ),9	le smpl/ O	inj fro	READY		

- (1) Call up display "a" in the following illustration at the function line.
- If "b" is displayed at the function line, press <u>func</u> once, then display of the line changes.
- A PUMP. A PUMP. B <sub>Pum</sub>p. C <sub>d</sub>et. A det. B
- b ALINU FRC OVEN SUBC KEYLOK
- (2) With the function keys, select the solvent delivery unit connected to the SCL. The selected unit will be highlighted on the screen.

The highlighted units on the screen are recognized by the SCL-1OA as connected units.

7

<<Note>>

If a unit is marked with **LOCAL**, control from the **SCL** is not accepted even if the name of the unit is highlighted. Set its local mode to 0, according to Section 5.5 (AUX.FUNC).

2. The Main Menu Screen From the CONFIG screen, press [menu) to enter into the main menu screen.

It is possible to return to the main menu display whenever you press (menu) from operation in any other display.



### 3. System Screen

Solvent selector valve unit FCV 10AL or FCV-11AL can be connected either to the pump unit or to the sub-controller. In any case the valve unit can be controlled by SCL-10A.

If the valve unit is connected to the pump unit or the sub-controller, state the connection in System Screen.

Select SYSTEM from the Main Menu to call the System Screen. In the System Screen the connection of optional units is stated. All information is stored in memory.

The CONFIG screen can be called from this screen.

SYSYTEM RELAY1 RELAY3 A.C.N P.L P.L P.E REC P.F RACT AUTTO.C PALAES PDE RACT AUTTO.C H ALAES PDE SV	start start online on on on on chi enable kgf/cm2 enable SUBC	stop stop offline offl off off off off off ch2 disable PUMP.A	error error error MPa PUMP.B	event1 event2 event3
CONFIG time 9999.9	a.f11e 0	s m p 1 / i	n J fro O O	KEYLOK <b>status</b> READY

Control from the SCL-10A

- (1) Select SV with cursor keys ( $\bigtriangleup$  ,  $\bigtriangledown$  ).
- (2) Select with cursor key  $([\triangleright], [\triangleleft])$  the unit name to which the valve unit is connected.
- 4. Setting the Initial Parameters
- Press (0) or move the flashing position to 0 using the arrow keys in the main menu screen, then press (Enter).
   The analysis file parameter control screen will be displayed. In addition to those for the solvent delivery units, parameters for any module may be displayed and updated from the analysis file parameter control screen.



• The <u>PUMP</u> column in the figure shows parameters for the solvent delivery units.

The solvent delivery mode in use is indicated inside the parentheses.

If an FCV-IOAL or FCV-11AL is connected to the pump unit, the parameter SV for the sub-controller works as the parameter SV of the pump unit.

(1)  $\operatorname{Press}(\operatorname{func})$  to display the following functions at the function line.

ISO, B.GE T.GE LP.GE KEYLOK

(2) Select a solvent delivery mode for the solvent delivery units using the corresponding function key.

 $\underline{ISO}((\underline{f1}))$  : Flow rates for up to three solvent delivery modules may be controlled independently.

B.GE (f2) : Conducts high pressure binary gradient elution using two solvent delivery units (A, B). Flow rate of solvent delivery unit

C is controllable independently.



Operation

## Control from the SCL-10A



: Conducts high pressure ternary gradient elution using three solvent delivery units. LP.GE ( (f4 )) : Low pressure quarternary gradient elution

is carried out using solvent delivery unit A. Flow rates of B and C are controllable independently.

The number and contents of the parameters displayed on the screen will vary according to the solvent delivery mode.

- Move the cursor with the arrow keys ( [ ](3) $\triangleright$  $\Delta$ and  $( \nabla )$  ) to the parameter you want to change. Key in a new value and press (Enter).
- If the activate lamp is lit, set parameters are sent to each unit at once for immediate execution.
- If the function display field is switched to the help information by pressing (func), the settable range and minimum step for the parameter at which the cursor is will be displayed.

**Examples:** 

A. FLOW 0 \_ 9,99 (m 1 / m i n ) STEP 0.01

EVENT 0. 1. 2. 3. 12. 13. 23. 123

Parameters displayed on the screen are as follows (for the solvent delivery module only):

Parameter	Description
A.FLOW	Flow rate for pump A (ml/min)
B.FLOW	Flow rate for pump B (ml/min)
C.FLOW	Flow rate for pump C (ml/min)
T.FLOW	Total flow rate (ml/min) in gradient elution mode
A.PRES	Pressure of pump <b>A</b> ( $\times 10^{5}$ Pa)
B.PRES	Pressure of pump B ( $\times 10^5$ Pa)
C.PRES	Pressure of pump C ( $\times 10^5$ Pa)
B.CONC	Concentration of solvent B (%)
C.CONC	Concentration of solvent C (%)
D.CONC	Concentration of solvent D (%)
B.CURV	Gradient curve of solvent B
C.CURV	Gradient curve of solvent C
D.CURV	Gradient curve of solvent D
P.MAX	Upper pressure limit (×10 <sup>5</sup> Pa)
P.MIN	Lower pressure limit ( $\times 10^5$ Pa)

7-7

<<Note>>

### Control from the SCL-10A

### 5. Time Program

When the setting of the initial parameters in the parameter control screen is complete, press <u>menu</u> to return to the main menu screen and select TIME PROGRAM .

From the time program screen, a time program, as described in Section 5.4 "Time Program Creation and Execution," may be set through the SCL.

In addition, program steps set in this time program editing screen can include parameters for other modules such as detectors.



Operation

(1) If a time program does not exist, the cursor is in the <u>TIME</u> field at step No. 0.
First at, using the numeric keys, key in the desired time for the parameter you want to change. After keying in values, press <u>Enter</u>, then the cursor moves to the <u>FUNC</u> field.

PRINT CLEAR COPY TABLE- TABLE-

- (2) Press (func) to select the function display that follows:
- (3) Use the TABLE← (f4) or TABLE→ (f5) key to move the highlighted box and select the module name in the FUNCTION FOR column.

FUNCTION FOR (PUMP DET. A DET. B OTHER)

(4) The contents of the parameter table vary with the module name which is displayed in the FUNCTION FOR column. The following is the parameter table for PUMP

 
 5
 B. CONC
 8
 B. CURV
 11
 A. PRES

 6
 C. CONC
 9
 C. CURV
 12
 B. PRES

 7
 D. CONC
 10
 O. CURV
 13
 C. PRES
 FLOW FLOW FLOW As for the parameters for another modules such as detectors, refer to the instruction manual of the SCL-IOA. If you enter a number shown in the parameter table using (5)the numeric keys, the **func** field will show the number. By pressing (Enter), the number disappears and the corresponding parameter will appear. The parameter table is displayed for ease of reference for parameter numbers. If a parameter has a number and it is not displayed on the screen, the entry of the value is possible anyway. If the selected parameter requires an argument (the entry of (6) values), the cursor will move to the **VALUE** field. Key in an appropriate value and press [Enter]. If you select the help information by pressing [func], the settable range and the minimum step of the argument required for the selected parameter will be displayed. If the parameter requires no argument, i.e., is a command, the cursor will move to the **TIME** field of the next step. Repeat the procedures (2) to (6) above and complete the time (7) program. The time you enter in the TIME field must be equal to or later <<Notes on Entry of than that in the previous step. program steps>> If you enter an earlier value, the alarm beeps when you press Enter and the entry is not accepted. (This is differnt than the time program editor of the LC-1OAT; See the SCL-1OA manual for insfructions on how to insert steps into a time program, etc.) The following operation may be carried out from the time (8) program screen using the function keys. By pressing [func], the function field switches and displays each function. PRINT CLEAR COPY TABLE← TABLE→

The time program on the screen is output to the printer connected to the Chromatopac.

All the steps of the time program corresponding to the file number on the screen will be deleted.

Control from External Equipment

7



### Control from the SCL-10A



TABLE  $\leftarrow$  ( [f4])

TABLE $\rightarrow$  ( (f5))

The time program corresponding to the file number on the screen will be copied to a file of another number.

Moves the box in the module name field of the parameter table to the left.

Moves the box in the module name field of the parameter table to the right.

KEYLOK

All key entry is disabled except KEYLOK (releases the key lock).

### 6. Pump Control Screen

(

f5)

KEYLOK

The Gradient profile or flow rate profile in a time program can be displayed graphically on the pump control screen so that it is possible to visualize and check the time program during its creation. To display the pump control screen, select **PUMPCONTROL** from the main menu.



- (1) The horizontal axis of this graph corresponds to time of the time program. The full scale for the horizontal axis is determined automatically so that the STOP instruction is located adjacent to the right end of the axis (if a STOP instruction is not included, the time of the last step).
- (2) The vertical axis corresponds to pump flow rates, concentration, or pressures. For flow rates and pressures, the maximum flow rate and pressure of each solvent delivery unit are shown. In the case of concentration, the full scal value is 100%.

Plot of change in solvent delivery parameters

Operation

### Control from the SCL-10A

- (3) The solvent delivery mode field at the right shows the currently selected mode in highlight. Below it, in addition, the functional parameter names for the solvent delivery modules in that mode are shown.
- (1) Press (func) to switch the function display as follows:

				a sama di ili ana sin
150.	8. G E	T.GE	LP.GE	PARAM

- (2) Select the desired solvent delivery mode with the function key.
- The selected solvent delivery mode is highlighted in the solvent delivery field at the upper right on the screen. Below it, parameter names corresponding to the mode are shown.
- (3) By pressing function key PARAM ( (f5)), the highlighted box moves down one line in the parameter name field. Move it to the desired parameter name for setting.
- Below the **TIME** field, the cursor flashes.
- It is the same **as** (func) of the time program.
- (4) Enter the desired time with the numeric keys and press Enter).
- The entered time is displayed, and the cursor moves to the position below the VALUE field.
- It is the same as TIME in the time program.
- (5) Enter a parameter value with the numeric keys and press (Enter).
- The entered value is displayed and the cursor will move back to the **[IIME]** field.
- It is the same as the parameter value in the VALUE field of the time program.
- If you set the solvent delivery mode to the high pressure gradient elution and selected **CONC** in (3) for parameter name, proceed to (8).
- (6) By the procedures from (3) to (5) the entry of 1 step in time programming is completed.Changes in flow rate (pressure) of the solvent delivery unit or concentration, which is caused by the entry of a new step, are displayed graphically on the screen.
- (7) Complete the time program by repeating the entry procedures from step (3).
- (8) By entering a parameter value for **CONC** with the numeric keys, the cursor moves further down by one step and flashes.

### Control from the SCL-10A

Now enter the value for the shape of the gradient curve that starts from the current time.

- This is the value for the CURV parameter in time programming.
- If the value for the gradient curve is 0 (linear), just press Enter).
- For the value and shape of the gradient curve, refer to the instruction manual for the SCL-1OA.
- When the entry is complete, the screen returns to (3).



- The entry of steps does not need to be carried out according to the order of time. Each new step is inserted in the time program in the correct order by an automatic sort function. If you change solvent delivery mode from the pump control
- screen, the solvent delivery mode on the parameter control screen will also be changed.

If a parameter for some other solvent delivery mode is entered while programming, it will be disregarded in the actual analysis.

It is impossible to correct **a** wrong entry from this screen. In such a case, press (menu] to return to the main menu and correct the wrong entry with the editing function from the TIME PROGRAM screen.

Switching the Screen Mode

<<Notes on Entry>>

The graphics display has two screen modes:

OVERLAY : Information about all parameters listed at the right of the screen are displayed on the screen at once.

SINGL The plot line of one parameter (selected with the PARAM key) is displayed.

Each time function key **G.MODE** ( **f1** ) is pressed, the screen mode switches.

The selected screen mode is highlighted at the right of the top line on the screen.

ISO

B.GE

( f2 )

# Control from the SCL-10A

The following operation is available using the function keys from the pump control screen.

> ISO. B.GE T.GE LP, GE PARAM

f1 ( ) How rates for up to three solvent delivery units may be controlled independently.

> Conducts high pressure binary gradient elution using two solvent delivery units (A, B). How rate for solvent delivery unit C is controlled independently.

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T.GE ( <b>f3</b> )	Conducts high pressure ternary gradient elution using three solvent delivery units.
LP.GE ( f4 )	Low pressure quartemary gradient elution is carried out using solvent delivery A. Flow rates for unit B and C is controlled independently.
	G. MODE ATTACK CARTER AND KEYLOK
PARM ( <b>f</b> 5 )	Selects parameter name.
G.MODE ( f1 )	Switches the screen display mode.
KEYLOK ( f5 )	All key entry is disabled except KEYLOK (releases the key

lock).

# Chapter 8 Maintenance

8.1	Replacement of Plunger Seals	8-2
8.2	Replacement of Plungers	8-5
8.3	Replacement of Washing Seal	8-7
8.4	Cleaning and Replacement of Check Valves	8-9
8.5	Replacement of Line Filter	8-10
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8.7	Cleaning	8-12

Contents

It is recommended that the plunger seal be replaced approx. every 2000 hours of solvent delivery. (Water delivery at 1ml/min, 100  $\times$  10<sup>5</sup> Pa.) See MON REV in Section 5.5 "Auxiliary Functions (AUX.FUNC)". When the plunger seal has been worn, the pressure becomes unstable, or solvent leaks from the gap between pump head and head holder or from the washing solution outlet. For replacement of the plunger seal follow the procedures below:

- (1) Assign 1 to the P.SET parameter. As for the setting method of the P.SET parameter, see Section 5.5 (AUX.FUNC).
- (2) The PUMP lamp will light up. Wait until this lamp goes off. Now the left and right plungers are set at the same position.
- (3) Remove the SUS pipe and teflon tube which are provided at the upper right and lower left areas of the pump head, respectively.



When removing the suction filter, keep the top end of the suction filter tube at a position higher than the level of the solution in the reservoir, or empty the tube.

(4) Loosen the three screws which fix the pump head gradually and alternately with the accessory Allen wrench. Then pull out the pump head.





<<Caution>>

8-2

LC-10AT

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- (5) Two seals are used in the pump head. Remove the seal which is causing the liquid leakage by using the accessory seal remover. Insert the rimmed end of the seal remover into the seal to be removed, and pull it out.
- (6) Two white spacers made of Teflon are provided at the end of the sealing area in the pump head. Remove these spacers by using tweezers, etc.. Be careful not to damage (scratch) the pump head.
- (7) Wet the new seal and the two spacers with alcohol.
- (8) Mount the large spacer to the original position in the pump head.
- (9) Fit the new seal onto the non-rimmed end of the seal remover. Then, fit the small spacer to the spring of the seal. (Being wetted with alcohol, both the seal and the spacers stick together easily when they contact.)
- (10) Insert the parts assembled in step (9) above into the pump head. Be careful that the assembly is not cocked. Insert it squarely into the head.







### **Replacement of Plunger Seals**

- (11) Mount the pump head **as** before and tighten the three screws alternately and uniformly. Finally, fit the short end of the hex wrench to each screw, grab the long handle and securely tighten each screw. Pay attention to the up/down orientation of the pump head. The left side has the check valve, while right side does not.
- (12) Reconnect the tubing at the top and bottom of the pump head as before.

<<Caution>> It may take some time after replacing the plunger seal until solvent delivery become stable. Accordingly, carry out running for about an hour using the solvent of usual with a flow rate setting of 1 to 5ml/min and pressure of 30 to  $100 \times 10^5$  Pa before start of analysis.

### <<Note>> If only one seal leaks after both seals have been in service for some time, it is advisable to replace both since the undamaged seal will probably fail next, and possibly soon.

If leakage persists after replacement of the plunger seal or the service life of the new plunger seal is very short, the plunger surface may be damaged. Defects may be caused by foreign matter contained in the pump head or crystalline substances of the buffer solution. In that case, the plungers need to be replaced.

- For replacement of the plungers, follow the procedures below:
- (1) Remove the pump head in the same manner as procedures(1) to (4) of Section 8.1 "Replacement of Plunger Seals."
- (2) Remove the seal holder assembly by pulling it out.



- (3) Release and remove the two screws on the left and right sides of the head holder with the accessory Allen. wrench. Then, release the two screws at the center alternately and gradually, and remove the head holder and the springs gently so that the head holder does not become cocked and jamb.
- (4) Remove the plunger by turning it counterclockwise with the accessory wrench (13  $\times$  17) as shown in Fig. 8.6.
- (5) Mount the new plunger and fix it with the wrench.

### **Replacement of Plunger**





- (6) Thoroughly wipe the plunger surface with a piece of soft cloth or tissue paper.
- (7) Replace the head holder and spring to the original positions and tighten the <u>two screws at the center alternately and</u> <u>uniformly until secure.</u>

Then securely tighten the two screws on the left and right.

- (8) Clean the engaging section between the seal holder and head holder with a piece of soft cloth or tissue paper. Mount the seal holder to the original position.
- (9) Mount the pump head as before and tighten the three screws alternately and uniformly. Fit the short end of the hex wrench to each screw and securely tighten them.
- (10) Reconnect the tubing at the top and bottom of the pump head as before.

When the washing seal gets worn and washing solution leaks, the washing solution drips into the tray and is lead to the panel drain. **If** leakage of the solution is noticed, replace the washing seal.



- Remove the pump head in the same manner as procedures
   to (4) of Section 8.1 "Replacement of Plunger Seals."
- (2) Remove the seal holder by pulling it out.



Fig. 8.8
#### **Replacement of Washing Seal**

- (3) Remove the washing seal from the seal holder in the same manner **as** the replacement of the plunger seal, using the seal remover.
- (4) Slip **a** new seal over the non-rimmed end of the seal remover and push it vertically into the seal holder.





- (5) Thoroughly wipe the engaging section between the seal holder and head holder with a soft cloth or a piece of tissue paper, then remount the seal holder gently without cocking it.
- (6) Mount the pump head and tighten the three screws gradually **and** alternately with an equal torque.
- (7) Reconnect tubing at the top and the bottom of the pump head as it was before.

A defective check valve may result in poor reproducibility of retention time or unstable pressure in solvent delivery. In such **a** case clean or replace check valves in the following procedure.

- 1. Cleaning the Check<br/>ValvesReplace the mobile phase with 2-propanol. Connect a resistance<br/>tube (0.1 x 2m) in place of the column, then feed the solution at<br/>a flow rate of 2ml/min for one hour or more.
- 2. Replacement of check valve
- (1) Remove the **SUS** pipe and teflon tube at the top and the bottom of the pump head.





- (2) Remove the check valves with the accessory wrench.
- (3) Mount the new check valves.
- (4) Reconnect the tubing at the top and the bottom of the pump head as before.

<<Cautions>>

- Do not disassemble the check valves. If it is done, we cannot guarantee the functions of the check valve.
- Be careful not to set the IN check valve upside down, otherwise the parts inside the valve may fall out.

### **Replacement of Line Filter**

If a back pressure exists with the tubing at the pump outlet disconnected, a possible cause is a clogged line filter. To replace the line filter follow the procedures below:

- (1) Disconnect the tubing of the pump outlet.
- (2) Unscrew the line filter with the accessory wrench.
- (3) Take out the gasket that is in the drain valve main body.
- (4) Wipe off dirt in the connecting port for the line filter with a soft cloth or a piece of tissue paper.

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- (5) Mount a new line filter and a gasket.
- (6) Connect the outlet tubing as before.



Fig. 8.11

### Replacement of Fuse

When the fuse is broken, replace it as follows. Be sure to use the same type and the same rating of fuses. Type and rating of fuses;

250V 2AT (5 x 20mm) (for 90-130V~) 250V 1AT (5 x 20mm) (for 200-250V~)

- (1) Turn the power switch off.
- (2) Remove the power cord from the power cord connector.
- (3) With a slotted-head screwdriver, catch the cover on the fuse holder and slide it out.



Fig. 8.12

(4) After replacing the fuse, push the fuse holder cover back in until it clicks.



Fig. 8.13

## Cleaning

If the cover or the front panel of the unit is dirtied, wipe away the dirt or dust with a soft cloth or a piece of tissue paper. If necessory, use a synthetic detergent.

## **Chapter 9 Troubleshooting**

9.1	Symptoms and Countermeasures for Troubles	9-2

9.2 Error Messages .....

9-3

**9-**1

Pump operation troubles result from various causes all of which cannot be covered in this manual. This section describes the troubleshooting measures on the basis of the symptoms. As for the countermeasures, detailed information is given in each relevant section.

Symptom	Cause		Remedy		
	1.	Bubbles in the pump chamber.	<ol> <li>Flush liquid through the drain to purge bubbles.</li> <li>Suck up bubbles through the drain tubing with a syringe</li> </ol>		
	2.	Old solvent remains in the pump chamber.	1. Increase flow rate and flush solvent through the drain to purge the old mobile phase completely.		
Solvent delivery is unstable. Fluctuation in pr- essure is large.	3.	Bubbles in the suction filter enter the pump.	<ol> <li>Increase the flow rate to purge the old solvent through the drain tubing.</li> <li>Shake the suction filter to release bubbles.</li> <li>If the suction filter is clogging, carry out ultrasonic cleaning. If the ultrasonic cleaning is not effective replace the suction filter.</li> <li>Degas the mobile phase.</li> </ol>		
	4.	Malfunction of the check valves.	1. Clean or replace the check valves.		
	5.	Leak from the plunger seal.	<ol> <li>Replace the plunger seal.</li> <li>Replace the plunger.</li> </ol>		
	6.	Leak in the flow line.	<ol> <li>I. 'lighten the joints in the flow line.</li> <li>2. Replacement of defective parts.</li> </ol>		
	7.	Flow line clogged	<ol> <li>Clean or replace the line filter.</li> <li>Check the flow line.</li> </ol>		
The pump opera-	I.	Bubbles in the pump chamber.	<ol> <li>Flush liquid through the drain to purge bubbles.</li> <li>Suck up bubbles through the drain tubing with a syringe</li> </ol>		
not delivered.	2.	Air enters from the joint between the suction filter and inlet pipe.	1. Firmly connect the joint.		
Measured flow	Ι.	Malfunction of the check valves.	1. Clean or replace the check valves.		
ate is lower than .he set flow rate.	2.	Clogged suction filter.	1. Clean or replace the suction filter.		
Solvent is not de- livered. (Pump is not op- erating.)	1.	An error message is displayed. (P.MAX, P.MIN, etc.)	I. Press (CE) . Remove the cause of the error.		
Pressure does not	Ι.	The drain valve is open.	1. Close the drain valve.		
ise.	2.	Leakage in flow line.	<ol> <li>Tighten joints.</li> <li>Replacement of defective parts.</li> </ol>		
Pressure rises ex- cessively.	1.	Line filter clogged	<ol> <li>Clean line filter.</li> <li>Replace line filter.</li> </ol>		
Confirm it by	2.	Flow line clogged	1. Identify the clogged component and replace it.		
umn.)	3.	The inside diameter of tubing is excessively small.	1. Replace the tubing with another having an appropriate inside diameter.		

This unit has multiple self-diagnostic testing functions, which beep an alarm and show an error message if they detect any abnormalities. Except for the case (1) and (2), pressing  $\overline{CE}$  will stop the alarm beep and return the display to the initial screen.

#### 1. ROM Error

ROM FAILURE

This error message will be displayed when there is something irregular in the **ROM**.

If this error message is displayed, pressing (CE) will not release the system from the error.

Turn off the power source for the unit, and contact our office or agent.

2. RAM Error

#### RAM FAILURE

This error message will be displayed when there is something irregular in the **RAM.** If this error message is displayed, pressing CE will not release the system from the error.

Turn off the power source for the module, and contact our office or agent.

3. Lost RAM File Error

#### NOT PROTECTED

This error message will be displayed when the contents of the **RAM** have been lost or destroyed due to power source abnormalities. By pressing  $\overline{CE}$ , the module may become operable. However, if this error message is displayed every time the power switch is turned on, the problem may be caused by dead or low power backup batteries. Contact our office or agent for information on exchanging the backup batteries.

#### 4. Overstepping Error

#### OUT OF MAXSTEPS

This error message is displayed when the total number of steps used in the time programs exceeds 320.

If this error message is displayed, the time program can not be prepared any further. Continue entering program data after deleting unnecessary files. 9

#### Error Messages

#### 5. Maximum Pressure Error

#### ERROR P- MAX

This error message will be displayed when the upper limit pressure, p-max, has been exceeded by the actual delivery pressure. In this case solvent delivery stops. (However, if S.PROT function is activated, the unit only reduces the flow rate by one half without stopping the pump.) When this error message is displayed, check the flow line for clogging, and if there is no problem, set the **P-MAX** value to an appropriate value.

#### 6. Minimum Pressure Error

ERROR P- MIN

This error message will be displayed if the actual delivery pressure is less than lower limit pressure P.MIN after 1 min. of operation. In this case the limiter stop the solvent delivery. If this error occurs, check the flow line for leakage, and if there is no problem, set the p.min value to an appropriate value. This error trapping will not function in the first one minute after the start of the solvent delivery in order to allow for pressure equilibration when just beginning to pump.

#### 7. Home Position Error

**Purge Error** 

#### ERROR HOME POS

This error message is displayed when the home position of the motor can not be detected. It is also displayed when the motor slips.

In this case, press the **ICE**) key to cancel the error, then press pump to operate. If the same error appears again, turn off the power for the unit, then turn it on again after 10 or 15 seconds and try it again. If this doesh't work, contact our office or agent.

#### OPENDRAINVALVE

This error message is displayed if the actual delivery pressure of the pump exceeds  $5\text{kgf/cm}^2$  during purge operations started with the (**purge**) key. The **CE** key will release the system from the error. Start the purge operation again after opening the drain valve. If the error message is displayed even with the drain valve open, execute the zero adjustment of the pressure sensor (see ZERO ADJ in 5.5 "AUX.FUNC").

8.

## **Chapter 10 Specifications**

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## LC-10AT Unit Specifications

1.	Pump type	A 2-stage (tandem-flow) double-plunger reciprocating pump.
2. Modes of solvent delivery 2.1 Constant flow delivery • Flow rate range:Constant flow or con $0.001 - 5ml/min (10)$ $5.001 - 9.99ml/min (10)$ $5.001 - 9.99ml/min (10)$ $0.001 - 5ml/min (10)$ $0.01 - 5ml/min (10)$		Constant flow or constant pressure solvent delivery $0.001 - 5ml/min (10 - 400 [\times 10^5 Pa])$ $5.001 - 9.99ml/min (10 - 200 [\times 10^5 Pa])$ Within +2% or ±2µl/min of set value, whichever is higher (0.1 - 5ml/min, 10 - 400 [x 10' Pa], with water and room tempera- ture at 20°C ) Within ±0.3% (0.1 - 5ml/min, 10 - 400 [x 10 <sup>5</sup> Pa] with water and $10^{-5} Pa$
2.	2 Constant pressure deli	very
	<ul> <li>Pressure setting range:</li> <li>Pressure setting accuracy:</li> </ul>	10 - 400 $[x 10^5 Pa]$ (in 1 $[x 10^{\circ} Pa]$ steps) Within ±10% or ±10 $[x 10^5 Pa]$ of set value, whichever is larger
3.	Pressure limits:	User-settable High/Low pressure limits
4.	Materials in contact with solvent:	SUS316, UHMW-PE, Elgiloy <sup>®</sup> , Kalrez <sup>®</sup> , PEEK ETFE, PCTFE, PTFE
5.	Suction filter:	10µm mesh
6.	Line filter:	2pm mesh, liquid volume approx. 200µl
7.	Time program:	Flow rate, pressure, EVENT, LOOP (program repeat), 10 files, total 320 steps
8.	Pressure indication accuracy:	Within +2% or $\pm 5$ [x 10 <sup>5</sup> Pa], whichever is larger
9.	Plunger cleaning flow line:	Built-ing Cleaning of plunger seals is possible with the connection of syringe or pump.
10.	Size:	260W x 140H x 420D (except protruding components)
11.	Weight:	11kg
12.	Operating temperature range:	5 - 35°C
13.	Power requirements:	(Power requirements vary depending upon the line voltage.) P/N 228-31900-91, 92: 90–130V~ 50/60Hz 100VA P/N 228-31900-93. : 200–250V~ 50/60Hz 100VA

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- 1. LC-10AT Control High Pressure Gradient Elution Specifications
  - 1) Number of solvents: 2
  - 2) Gradient profile: Stepwise and linear, multiple processes are possible in a single program.
  - 3) Number of program files: 10 files with a total of 320 program steps
  - 4) Program mable time: 0.01 min 999.99 min, 0.01 min steps
  - 5) Settable Concentration range: 0 - 100%, 0.1% steps
  - 6) Concentration setting  $\pm 1\%$  (0 100%, 0.3 3ml/min, 10 400 [x 10<sup>5</sup> Pa], with aqueous accuracy: acetone/water)
- 2. SCL-10A Control High Pressure Gradient Elution Specifications
  - 1) Number of solvents: 2 or 3
  - 2) Gradient profiles: Stepwise, linear, and exponential functions, multiple processes are possible in **a** single program.
  - 3) Number of program files: 20 files, with a total of 400 program steps
  - 4) Programmable time: 0.01 min 9999.9 min, 0.01 min steps
  - 5) Settable Concentration range: 0 - 100%, 0.1% steps
  - 6) Concentration setting accuracy: ±1% (0 100%, 0.3 3ml/min, 10 400 [x 10' Pa], with aqueous acetone/water)



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L- LC-10AT Control - Low Pressure Gradient Elution Specifications

1)	Number of solvents mixed:	up to 4
2)	Gradient profiles:	Stepwise and Linear, multiple processes are possible in a single program.
3)	Number of program files:	a total of 10 files and 320 steps
4)	Programmable time:	0.01 - 999.99 min, 0.01 min steps
5)	Settable Mixing ratio range:	0 - 100%, 0.1% steps
6)	Concentration accuracy	± 2%
7)	Flow rate range	0.1 - 2ml/min (In the case of a flow rate of more than 2 ml/min, the life time of the valves of the low pressure gradient unit become short due to high speed cycling.)
2.	SCL-10A Control - Low I	Pressure Gradient Elution Specifications
1)	Number of solvents mixed:	up to 4

2) Gradient profiles: Stepwise, linear and exponential functions; multiple processes are prossible in a single program.

a total of 20 files and 400 steps

- Number of program files:
- **4)** Programmable time: 0.01 9999.9 min
- 5) Mixing ratio setting range: 0 - 100%, 0.1% steps
- 6) Concentration accuracy  $\pm 2\%$
- 7) Flow rate range
   0.1 2ml/min
   (In the case of a flow rate of more than 2 ml/min, the life time of the valves of the low pressure gradient unit become short due to high speed cycling.)

## Chapter 11 Spare Parts and

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	Part name	Part number	Remarks
Ι	Plunger seal (yellow)	228-21975	Seal for pump head

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Spare Parts and Optional Units



## List of Other Necessary Parts

#### 1. Mechanical Parts

	Part name	Part number	Remarks
1	Plunger assy	228-32654-91	
2	Head holder	228-31903	
3	Seal PE (white)	228-28499	Seal for washing solution
4	Pump head	228-31905	
5	Check valve OUT	228-32531-92	
6	Check valve IN	228-32166-91	
7	Pressure sensor assy	228-32252-92	Including line filter and drain valve
8	Filter assy standard	228-12642-93	
	for Low Pressure Gradient	(228-32467-91 <b>)</b>	Low volume type -> 228- 32693 - 9.
9	Gasket	228-12564	For line filter replacement
10	Suction filter assy	228-18740-91	
11	Photosensor assy	228-28288-91	For detection <b>of</b> the home position <b>of</b> pump
12	Drain valve shaft 10AS ASSY	228-28312-92	
13	Spacer (large)	228-32325-01	For plunger seel
14	Spacer (small)	228-32401	> roi piungei seai

	Part name	Part number	Remarks
1	PB-I assy ROM included	228-24785-95	ROM included
2	PB-I assy ROM not included	228-23685-93	ROM not included (Photocoupler included)
3	ROM LC-IOAT	228-24783-95	
4	Fuse IAT 5 x 20	072-01652-16	For 200V
5	Fuse 2AT 5 $\times$ 20	072-01652-19	For 100V

The following is a list of the optional units that may be used in combination with this unit. For details on the available optional units or for information on those not listed here, contact our office or agency.

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Optional unit identification	Part number	Description
DGU-2A	228-21996-91	A helium degasser for four solvents. Up to four solvents may be degassed.
DGU-3A	228-24200-91	A degasser for three solvent. It permits continuous degassing by pressure reduction through a resin membrane for up to 3 flow lines.
DGU-4A	228-24201-91	A degasser for two solvent. It permits continuous degassing by pressure reduction through a resin membrane for up to 2 flow lines.
DGU-10B	228-32235-91	A full automatic helium degasser for four solvent. Controlled by the LC- 10AT (or the SCL-IOA through option box L).
FCV-10AL	228-24861-91	<ul> <li>A solenoid valve unit for low pressure gradient elution analysis.</li> <li>Also usable as a four-solvent selector solenoid valve unit.</li> <li>Controlled by the LC-IOAT (or by the SCL-IOA through option box L).</li> </ul>
FCV-11AL	228-24813-91	<ul> <li>A solenoid valve unit for selecting solvents (for 3 flow lines).</li> <li>Three units of three way solenoid valves are built in, permitting selection of solvents for three flow line.</li> <li>Controlled by the LC-IOAT (or by the SCL-IOA through option box L).</li> </ul>
FCV-11AL(S)	228-24813-92	<ul> <li>A solenoid valve unit for selecting solvents (for 1 flow line).</li> <li>A three way solenoid valve is built in, permitting selection of two solvents.</li> <li>Controlled by the LC-IOAT (or by the SCL-IOA through option box L).</li> </ul>
FCV-13AL	228-24914-91	<ul> <li>A valve unit for selecting solvents.</li> <li>Having 6 setting positions, this valve unit permits switching 6 solvents for one flow line.</li> <li>Controlled by the SCL-IOA through option box L.</li> </ul>
A set of 3 suction fil- ters	228-18907-91	• A set of three suction filters. This set is for the FCV-13AL module. (The suction filters are not included with the FCV-13AL.)
Option Box L	228-25025-91(100V) 228-25025-92(200V)	<ul> <li>This unit has 3 functions.</li> <li>1. For control of the FCV-11AL, FCV-11AL(S), and FCV-I3AL. However, setting is made through the SCL-1OA.</li> <li>2. Up to 6 of the following units can be contained: FCV-11AL, FCV-11AL(S), FCV-13AL, DGU-1A, DGU-2A, and DGU-IOB.</li> <li>3. Supplies AC power for other modules included in a system. 100V x 8, or 200V x 6.</li> </ul>
Option Box S	228-25306-91	Up to 2 of the following units can be contained: FCV-IIAL, FCV-IIAL(S), FCV-13AL, DGU-IA, DGU-2A, DGU-IOB, etc. This unit can be placed under the LC-IOAT <b>so</b> it requires very little space.
Reservoir Box	228-25038-91	This box provides housing space for the reservoirs, and permits installation of a manual injector.
Mixer	228-28000-91	A static mixer for the high and low pressure gradient elution analysis.

Manual Injector Model 7725	228-32210-91	A manual injector for general analyses. The standard sample loop of $20\mu l$ is included.
Manual Injector Model 7725 i	228-32210-93	Manual Injector 7725 with the position sensing switch. A signal synchronized with sample injection is sent to the system controller and Chromatopac.
Manual Injector Model 8125 for semi-micro liquid chromatography	228-23200-91	A manual injector for semi-micro liquid chromatography. The standard sample loop of $5\mu l$ is included. The unit has <b>a</b> built-in position sensing switch.
Tubing <b>Parts</b> Kit for semimicro liquid chro- matography	228-23198-91	This unit consists of a <b>SUS</b> pipe with an inside diameter of 0.2mm and a joint for the manual injector.
Injector Holder	228-25468-91	This unit is used for mounting the manual injector on the right side of the LC-IOAT.
Column Holder	228-15418-93	This unit is used when fixing a column on the right side of the LC-IOAT.
Air Trap	228-23675-91	This is attached to the tube for the suction filter and used to let the air out of the flow line.

## **Chapter 12 Reference Material**

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## **Chapter 12 Reference Material**

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2.1	Precautions on Static Electricity	12-2
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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.

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

1. Mechanism of Static Electrical Discharge Accident (Example)

Occurrence	e of	Static	
Electricity			
			_

Charging and storage

Energy release by

Ignition of combustible

discharge

substances

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. 12.1.



A : Electric charge moving with flowing liquid

> Electric charge being fixed to the solid surface.

## Fig. 12.1 Occurrence of Static Electricity by Friction between Solid and Liquid

When the charged liquid is collected in an insulated vessel, the

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.



LC-10AT



Fig. 12.2 Conditions for 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 "Occurrence mechanism of static electrical discharge accidents." The preventive measures are shown below. It is recommended to exercise two or more measures simultaneously.

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

# **Preventive measure 1.** Use metallic (conductive) waste liquid vessel which is well grounded. This releases the charge of the waste liquid and vessel to ground.

The following items are available.

- (1) Grounding wire with clip P/N 228-21353-91
- (2) Metallic 18 liter can P/N 038-00044
- (3) Metallic 4 liter can P/N 038-00043-01
- Be sure to ground the vessel properly. Disconnecting of grounding wire or poor grounding defeat the purpose of using a metallic vessel.
- There are some metallic cans which have no conductivity due to an oxidized coating or lacquer on their surface. Be sure to confirm the grounding of vessels by a tester before application.
- \* When a liquid with almost no conductivity (of  $10^{-10}$ s/m or less) is discharged into the vessel, it is necessary to mix it

with another liquid with some conductivity. (The other liquid can be placed in the vessel in advance.)

#### Preventive measure 2.

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

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





Preventive measure 3.

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

Charging prevention measures for human body

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

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

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

**Preventive measure 4.** Use pipes with inner diameter of 2mm or more for waste liquid line for large flow rates.

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

12-4

Preventive measure 5.

12. 1

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

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

### **Mobile Phase Characteristics**

									(7) Watar		
			(3)		Dailina				water	( <b>0</b> )	(0)
(1	) *7~ 5°D ~45°	(2)	(5)	(4)	Doint	Viscosity	(5)	(6)	Solubility <i>o</i> rwin 20℃	(8) Dialactria	() D'
Solve	ent <b>**7&lt;.5cP,&lt;45</b> °	Source	Cutoff	(4) R.I. 25	$(\mathcal{C})$	(cP,25℃)	(3) p'	(0) e <sub>a</sub>	Solvent	Constant e <sup>20</sup>	0.25 e
1.	FC-78 (*)	(Particular	210 nm	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	2.6	< -2	25		1.9	(function in
2	L										proportion to intensity)
2.	Isooctane (*)(2,2,4-	IC	107	1 200	00	0.47	0.1	0.01	0.01.1	1.04	0.1
3	n Hentane (*)		197	1.369	99	0.47	0.1	0.01	0.011	1.04	0.1
з. 4	n Heyene (*)		195	1.365	90 60	0.40	0.2	0.01	0.010	1.92	0.5
4. 5	n-Pentane (**)		190	1.372	36	0.30	0.1	0.01	0.010	1.80	0.5
5. 6	Cyclobeyane		200	1.333	20 81	0.22	0.0	0.00	0.010	2.02	0.5
0. 7	Cyclonexale Cyclonextens (*)		200	1.423	40	0.90	-0.2	0.04	0.012	2.02	0.5
7. Q	L-Chlorobutane (*)		200	1.404	49 78	0.42	-0.2	0.05	0.014	7.4	2.8
0. 0	Carbon disulfide		380	1.400	16	0.42	0.3	0.20	0.005	7.4 2.64	2.0
9. 10	2 Chloropropaga (**)		220	1.024	40 26	0.34	1.2	0.15	0.005	2.04	1.7
10.	2-Chioropropane(**)		250	1.373	30 דד	0.50	1.2	0.29	0.008	9.62	2.7
12	n Dutul other	LC	203	1.457	142	0.90	2.1	0.16	0.008	2.24	2.5
12.	Tristhylemine		220	1.397	142	0.04	2.1	0.23	0.19	2.0	2.4
13. 14	Promosthana (*)			1.396	09 20	0.50	1.9	0.54		2.4	2. <del>4</del> 4.2
14.	i Propul other (*)		220	1.421	38 69	0.56	2.0	0.55	0.62	9.4 2.0	4.5
15. 16	Toluene	IC	220	1.303	110	0.58	2.4	0.20	0.02	3.9 2.4	2.0
10.	n Vulana	LC	203	1.494	120	0.55	2.4	0.29	0.040	2.4	2.9
17.	p-Aylelle Chlorobonzono		290	1.495	120	0.00	2.5	0.20		2.5	3.0 4.1
10.	Promohonzono			1.521	152	1.04	2.7	0.30		5.0	4.1
19. 20	Indehenzene			1.557	150	1.04	2.1	0.52		5.4	4.1
20. 21	Dhopyl other			1 590	250	2.2	2.0	0.55		27	27
21.	Dhamatala			1.500	230	5.5	5.4 2.2			3.7	5.7
22.	Fiber (**)		010	1.505	1/0	1.14	3.3	0.20	1.2	4.2	4.9
23.	Ethyl ether (**)		218	1.350	35	0.24	2.8	0.38	1.3	4.3	4.0
24. 25	Benzene	LC	280	1.498	80	0.60	2.7	0.32	0.058	2.3	3.0
25.	Fiber I in the			1 5 1 0	70	0.57	22			7.0	4.0
26.	Ethyl lodide		205	1.510	105	0.57	2.2	05	20	/.8	4.2
27.	n-Octanol		205	1.427	195	1.3	3.4 2.1	0.5	3.9	10.5	5.8
28. 20	Fluorobenzene			1.46	200	0.55	3.1			5.4	4.0
29. 20	Denzyletner Mathalana alalani da (**)		000	1.538	288	4.5	4.1	0.40	0.17	0.0	5.6
30. 21	Meunylene chioride (**)	LC	233	1.421	40	0.41	3.1	0.42	0.17	8.9	5.0
31. 22				1.514	154	0.9	3.8	0.61	0.0	4.3	4.6
32. 22	1-Pentanoi	LO	220	1.405	130	3.5	3.7	0.61	9.2	14./	1.3
33. 24	1,2-Dichloroethane	LC	228	1.442	83	0.78	3.5	0.44	0.16	10.4	6.3
34. 25	t-Butanol		210	1.385	82	3.6	4.1	0.7		12.5	0.2
35. 26	n-Butanol		210	1.397	811	2.6	3.9	0.7	20.1	17.5	8.3
36.	n-Propanol		240	1.385	9/	1.9	4.0	0.82	miscible	20.3	
37.	Tetrahydrofuran (*)	LC	212	1.405	66	0.46	4.0	0.57	miscible	7.6	
38.	Propylamine (*)			1.385	48	0.35	4.2	0 -0	miscible	5.3	
39.	Ethylacetate (*)	LC	256	1.370	77	0.43	4.4	0.58	8.8	6.0	5.8
40.	1-Propanol	LC	205	1.384	82	1.9	3.9	0.82	miscible	20.3	<b>.</b> .
41.	Chlorotorm (*)	LC	245	1.443	61	0.53	4.1	0.40	0.072	4.8	5.6
42.	Acetophenone		226	1.532	202	1.64	4.8	0.5	<u>aa (</u>	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

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12-6

### **Mobile Phase Characteristics**

										(7)		
				(2)		יוי ת				Water		(0)
(1	`	*7 < 6 - D > 45°	(2)	(3)	(4)	Boiling	Viceosity	(5)	$(\boldsymbol{\epsilon})$	Solubility	(8) Dialaatria	(9) I''
Solv	) ent	*7<.5cP,>45 **7<.5cP,<45°	(2) Source	u v Cutoff	(4) <b>R.I.</b> <sub>25</sub> ·	ronn (°C)	(cP,25℃)	(3) p'	(0) e <sup>°</sup> a	Solvent	Constant e 20	0.25 e
45.	Nitr	obenzene			1.550	211	1.8	4.4			34.8	13.2
46.	Benz	zonitrile			1.536	191	1.2	4.8			25.2	10.9
47.	Dio	xane	LC	215	1.420	101	1.2	4.8		miscible	2.2	
48.	Tetr	amethyl urea	LC	265	1.449	175		6.0	0.56		23.0	10.7
49.	Quii	noline			1.625	237	3.4	5.0			9.0	7.4
50.	Pyri	dine			1.507	115	0.88	5.3		miscible	12.4	
51.	Nitro	oethane		380	1.390	114	0.64	5.2		0.9		
52.	Ace	tone (*)	LC	330	1.356	56	0.30	5.1	0.71	miscible		
53.	Benz	zyl alcohol			1.538	205	5.5	5.7			13.1	8.8
54.	Tetra	amethyl guanidine						6.1	0.6			
55.	Met	hoxyethanol	LC	210	1.400	125	1.60	5.5		miscible	19.9	
56.	Tris	(cyanoethoxy)							0.56			
	1	propane	GC					6.6				
57.	Prop	ylene carbonate	LC					6.1				
58.	Etha	nol	LC	210	1.359	78	1.08	4.3		miscible	24.6	
59.	Oxy	dipropionitrile	GC					6.8				
60.	Anil	line			1.584	184	3.77	6.3			6.9	8.1
61.	Ace	tic acid			1.370	118	1.1	6.0		miscible	6.2	
62.	Ace	tonitrile (*)	LC	190	1.341	82	0.34	5.8		miscible	37.5	
63.	N, N	l-dimethylaceta-	LC	268	1.436	166	0.78	6.5	0.88		37.8	
	1	mide										
64.	Dim	ethylformamide	LC	268	1.428	153	0.80	6.4			36.7	
65.	Dim	ethylsulfoxide	LC	268	1.477	189	2.00	7.2	0.62	miscible	4.7	
66.	N-m	ethyl-2-pyrolidone	LC	285	1.468	202	1.67	6.7			32	
67.	Hex	amethyl phosphoric										
	ä	acid triamide			1.457	233	3	7.4	Q.65		30	
68.	Met	hanol (*)	LC	205	1.326	65	0.54	5.1		miscible	32.7	
69.	Nitr	omethane		380	1.380	101	0.61	6.0		2.1		
70.	m-C	resol			1.540	202	14	7.4			11.8	10.0
71.	N-m	ethylformamide			1.447	182	1.65	6.0		miscible	182	
72.	Ethy	lene glycol			1.431	182	16.5	6.9		miscible	37.7	
73.	For	namide			1.447	210	3.3	9.6		miscible	111	
74.	Wat	er	LC		1.333	100	0.89	10.2			80	

#### **Mobile Phase Characteristics**

- (\*) indicates solvents most suitable for LC, having convenient boiling points (>45°C) and low viscosity (≤0.5cp).
   (\*\*) indicates solvents with very low viscosity and boiling point.
- "LC" indicates that the solvents are commercially available specifically for LC from the 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 solvents are used as a stationary phase in the liquid-to-liquid LC.)

- (3) The wavelength shorter than which the solvent becomes opaque.
- (4) Refractive index at  $25^{\circ}$ 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 liquidto-solid adsorption
- (8) Value at 20°C
- (9) Function where P' is proportional to solvent strength and dielectric constant in ion pair chromatography.