SOLVENT DELIVERY MODULE FOR SHIMADZU HIGH PERFORMANCE LIQUID CHROMATOGRAPH LC-20AD INSTRUCTION MANUAL

Read the instruction manual thoroughly before you use the product. Keep this instruction manual for future reference.



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Introduction

Read this manual thoroughly before using the instrument.

Thank you for purchasing this instrument. This manual describes: the installation, operation, hardware validation, cautions for use, and details on the accessories and options. Read the manual thoroughly before using the instrument. Use the instrument in accordance with the manual's instructions. Keep this manual for future reference.

required.

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Original version is approved in English.

Warranty and After-Sales Service

Warranty

1. Validity

Please consult your Shimadzu representative for information about the extent of the warranty.

2. Term

The manufacturer will provide free replacement parts for, or repair free of charge, any instrument that fails during the warranty period, if the cause can be attributed to a defect in manufacturing.

3. Limitation of Liability

- In no event will Shimadzu be liable for any lost revenue, profit or data, or for special, indirect, consequential, incidental or punitive damages, however caused regardless of the theory of liability, arising out of or related to the use of or inability to use the product, even if Shimadzu has been advised of the possibility of such damage.
- In no event will Shimadzu's liability to you, whether in contract, tort (including negligence), or otherwise, exceed the amount you paid for the product.

4. Exceptions:

Failures caused by the following are excluded from the warranty, even if they occur during the warranty period.

- 1) Improper product handling
- 2) Repairs or modifications performed by parties other than Shimadzu or Shimadzu designated companies
- 3) Product use in combination with hardware or software other than that designated by Shimadzu
- 4) Computer viruses leading to device failures and damage to data and software, including the product's basic software
- 5) Power failures, including power outages and sudden voltage drops, leading to device failures and damage to data and software, including the product's basic software
- 6) Turning OFF the product without following the proper shutdown procedure leading to device failures and damage to data and software, including the product's basic software
- 7) Reasons unrelated to the product itself
- 8) Product use in harsh environments, such as those subject to high temperatures or humidity levels, corrosive gases, or strong vibrations
- Fires, earthquakes, or any other act of nature, contamination by radioactive or hazardous substances, or any other force majeure event, including wars, riots, and crimes
- 10) Product movement or transportation after installation

11) Consumable items

 Note: Recording media such as floppy disks and CD-ROMs are considered consumable items.
 If there is a document such as a warranty provided with the product, or there is a separate contract agreed upon that includes warranty conditions, the provisions of those documents shall apply.

 After-Sales Service If any problem occurs with this instrument, inspect it and take appropriate corrective action as described in the Section "6 Troubleshooting". If the problem persists, or symptoms not covered in the Troubleshooting section occur, contact your Shimadzu representative.

Replacement Parts Availability	Replacement parts for this instrument will be available for a period of seven (7) years after the discontinuation of the product. Thereafter, such parts may cease to be available. Note, however, that the availability of parts not manufactured by Shimadzu shall be determined by the relevant manufacturers.
Hardware Validation	Each LC component and the entire LC system should be checked periodically to ensure that they function normally, or the analysis data may not be reliable. To this end, it is necessary to carry out periodic hardware validation and keep records of the validation. There are two types of hardware validation - component validation and system validation. The purpose of component validation is to check that the individual components of the system function normally, while the system validation checks that the system as a whole (the several components in combination) functions normally. Before shipment from the factory, this instrument was rigorously inspected. The results are summarized in the Inspection Certificate accompanying the instrument. To inspect the instrument performance after installation, repeat the Hardware Validation as described in "7 Hardware Validation".
Hardware Validation Contract	This is a contract under which a qualified Shimadzu-approved engineer performs periodic component and system validation, and provides reports of the results. Details of the contract can be obtained from your Shimadzu representative.

Safety Instructions

- To ensure safe operation of the instrument, read these Safety Instructions carefully before use.
- Observe all of the WARNINGS and CAUTIONS described in this section. They are extremely important for safety.
- In this manual, warnings and cautions are indicated using the following conventions;

	Indicates a potentially hazardous situation which, if not avoided, could result in serious injury or possibly death.
	Indicates a potentially hazardous situation which, if not avoided, may result in minor to moderate injury or equipment damage.
NOTE	Emphasizes additional information that is provided to ensure the proper use of this product.

Application Precautions

This instrument is a solvent delivery module for use with a high performance liquid chromatography system.

Use this instrument ONLY for the intended purpose.

Using this instrument for any other purpose could cause accidents.

Installation Site Precautions

\land WARNING

- The solvents used in high performance liquid chromatograph are flammable and toxic. The room where the instrument is installed should be well ventilated; otherwise, solvent vapors could cause poisoning or ignite and cause a fire.
- High performance liquid chromatograph uses large amounts of flammable organic solvents. Use of open flame in the vicinity of this instrument must be strictly prohibited. Do not install the instrument in the same room with any other equipment that emits or could potentially emit sparks, since sparks could cause a fire.

Provide fire extinguishers for use in case of fire.

• Provide protective equipment near the instrument. If solvent gets into the eyes or on the skin, it must be flushed away immediately. Provide equipment, such as eye wash stations and safety showers, as close to the instrument as possible.



• The weight of this instrument is 10kg. During installation, consider the entire weight combined with other LC components.

The lab table on which this instrument is installed should be strong enough to support the total weight of the LC system. It should be level, stable and have depth of at least 600mm. Otherwise, the instrument could tip over or fall off the table.

• Keep at least 100 mm between the rear of the instrument and the wall.

This allows for sufficient air circulation and ventilation from the grille to provide cooling and prevent the instrument from overheating and impairing the performance.

· Avoid installation sites that are exposed to corrosive gases or excessive dust.

These adverse conditions may be detrimental to maintaining the instrument performance and may shorten its service life.

At least

600mm

Installation Precautions

• Take measures to prevent the instrument from falling in the event of an earthquake or other disaster.

Strong vibrations could cause the instrument to fall over, resulting in injury.

• The power supply voltages and power consumptions of this instrument are listed below. The power supply voltage of the instrument is indicated on the label on the back of the instrument. Connect the instrument to a power supply that complies with the capacity and use a power cord that complies with the capacity;

otherwise, fire or electric shock could result. Check that the power supply voltage is stable and that its current capacity is sufficient to operate all the components of the system. If not, the instrument will not operate at its rated performance.

Part No.	Power Supply Voltage (indicated on the instrument)	Power Consumption	Frequency	Rated Breaking Capacity [*]
S228-45000-31 S228-45000-41	AC100-120V (100-120V~)	150VA	50/60Hz	50A
S228-45000-32 S228-45000-42	AC100-120V (100-120V~)	150VA	50/60Hz	50A
S228-45000-38 S228-45000-48 S228-45000-58	AC220-240V (220-240V~)	150VA	50/60Hz	50A

* Connect the instrument to a power outlet that is equipped with a circuit breaker that shuts off the current at the described value or less.

Ground the instrument.

Grounding is necessary to prevent electric shock in the event of an accident or electrical discharge, and important for ensuring stable operation.

• To prevent electric shock and to maintain stability in operation of the product, be sure to ground the product.

The product will be grounded when the provided 3wired power plug is inserted into a 3-wired power socket equipped with a ground terminal.

• Do not place heavy objects on the power cord, and keep any hot items away.

The cord could be damaged, resulting in fire, electrical shock or malfunction. If the cord becomes damaged, contact your Shimadzu representative immediately.

• Do not modify the cord in any way. Do not bend it excessively or pull on it.

The cord could be damaged, resulting in fire, electrical shock or malfunction. If the cord becomes damaged, contact your Shimadzu representative immediately.



- When installing the instrument, be careful not to pinch your fingers between the system components, as this could result in injury.
- When opening the doors, be careful not to pinch your fingers as this could result in injury.



Operation Precautions

• Take thorough measures to prevent buildup of static electricity.

Static Electricity Precautions" P.XII Static electricity could result in fires or explosions.

- Always wear protective gloves and protective goggles when handling solvents and samples.
 If solvent gets into the eyes, blindness could result.
 Should solvent get into the eyes, flush immediately with large amounts of water and get medical attention.
- Always wear protective gloves when handling any toxic or biologically infectious samples.
- Never use a cracked reservoir bottle.
 If a helium degassing unit is used, pressure is exerted on the reservoir bottles and may cause cracks in the bottles.
 It could break the reservoir bottles and cause injury.
- Do not use flammable sprays (hair sprays, insecticide sprays, etc.) near the instrument. They could ignite and cause a fire.
- Be careful not to apply liquid to office equipment such as the PC as well as the instrument.







Precautions for Instrument Inspection, Maintenance, Adjustment and Care

• Unplug the instrument before inspection, maintenance, or parts replacement.

Otherwise, electrical shock or short-circuit accidents could occur.

• Never remove the main cover.

This may cause injury or malfunction of the instrument. The main cover does not need to be removed for routine maintenance, inspection and adjustment. Have your Shimadzu representative perform any repairs requiring removal of the main cover.

• Replace fuses only with fuses of the proper type and capacity.

Any other fuses could cause a fire.

- If the power cord plug gets dusty, remove the plug from the power outlet and wipe away the dust with a dry cloth. If dust is allowed to accumulate, fire could result.
- Replacement parts must be of the specifications given in "1.3 Component Parts" or "9.3 Maintenance Parts". Use of any other parts may result in instrument damage and malfunction.
- If any water gets onto the instrument, wipe it away immediately to prevent rust. Never use alcohol or thinner solvents for cleaning the instrument.

They could cause discoloration.

• Dispose of the waste liquid properly and in accordance with the instruction by your administrative department.



■ In an Emergency

If any problem is detected, such as pumping does not stop, something smells like burning, take the following action.

Also, when the instrument is used again, inspect the instrument and, if necessary, contact your Shimadzu representative to request servicing.

Emergency Shutdown Procedure

- 1) Turn OFF the power switch on the main unit.
- 2) Disconnect the power cable at the rear of the main unit.

During a Power Outage

Take the following measures in the event of a power outage.

- 1) Turn OFF the power switch on the main unit.
- 2) After confirming all related items in this section "Installation Precautions" and "Operation Precautions", use the standard startup procedure to start the instrument.

Static Electricity Precautions

Liquid chromatograph (LC) uses flammable organic solvent(s) as the mobile phase. LC systems are also often used where large amount of flammable substances are present. Thus, an accident can produce large scale damage. Operators must be constantly on guard against accidents involving fire or explosion.

The major cause of these accidents is static electricity. Devising preventative measures for static can be difficult, because the symptoms before an accident vary and can be hard to detect, since such accidents occur as a result of several simultaneous coincidences. Recommended methods for preventing static electricity accidents are provided below. Take thorough safety measures based on this information.

Typical Cause of Static Electricity Accidents

Static electricity accidents are generally caused by this sequence of events:



Preventing Static Electricity Accidents

The best way to prevent static electricity accidents is simply to prevent the occurrence and accumulation of electrostatic charges.

- It is important to take multiple preventive measures simultaneously.
- If large amounts of flammable solvents are collected in a large container, implement

preventative measures 1, 2, and 3 below.

Preventive Measure 1

Use a metal container for the waste liquid, and ground the container. This will ensure that the electrical charges of the container and liquid pass to the ground.

Accessories for this measure

(1)	Grounding wire with clip	Part No. S228-21353-91
(2)	18 Liter metal container	Part No. S038-00044

(3) 4 Liter metal container Part No. S038-00043-01

• Be sure to ground the metal waste container properly.

If the grounding wire is not properly attached or connected to the ground, static electricity can build up in the container.

- Some metal containers have surfaces that are laminated or oxidized, and therefore do not conduct electricity. After grounding the metal container, use a tester to verify that electricity is conducted to the ground.
- If the liquid to be drained into the waste container is virtually non-conductive (10⁻¹⁰ S/m or less), it will be necessary to add properly conductive, and therefore safe, liquid to the tank.

This conductive liquid may be added beforehand.

Preventive Measures for Static



Preventive Measure 2

Cover the spaces between the tubing and the sides of the inlet and outlet openings of the waste container with caps or other protective covering. This will prevent any sparks generated outside the container from getting inside.

Accessories for this measure Caps for 18 liter or 4 liter containers (with three 3mm diameter openings) Part No. S228-21354-91

Preventive Measure 3

Keep electrostatically charged objects, including the human body, away from the waste liquid container. To prevent electrostatic charging of the human body, take the following precautions:

- Wear anti-static clothing and shoes.
- Ground the human body with anti-static wrist straps. (For safety, the wrist strap should be connected to the ground using an intervening resistor of about 1MΩ.)
- Spread anti-static matting or the like on the floor, to make the floor conductive.

• Persons who have not taken anti-static precautions should touch some grounded metal object before coming near the waste liquid container, in order to drain static charges.

Preventive Measure 4

Use tubing with an inner diameter of at least 2mm for drain lines with high flow rates.

Periodically check the tubing connections for leaks.

Air bubbles in liquid can multiply the electrostatic charge by a factor of 20, 30 or more.

Preventive Measure 5

If it is not possible to use a conductive waste liquid container, take the following precautions:

• Ensure that the end of the inflow tubing is always submerged inside the container. Also, place some type of grounded metal object, such as a ground wire connected to the instrument, into the liquid.

The above precaution will be ineffective for low conductivity (less than 10⁻¹⁰ S/m) liquids.

- Use as small a container as possible to minimize damage in the event of fire.
- Keep the room at a proper humidity. Ambient humidity exceeding 65% will prevent static.

For Reference

Anti-static equipment (anti-static clothing, shoes and matting) and charge measurement equipment (potentiometer) are sold by specialty manufacturers.

Maintenance, Inspections, and Adjustment

In order to maintain the instrument's performance and obtain accurate measurement data, daily inspection and periodic inspection/calibration are necessary.

For daily maintenance, inspection, and replacement parts, see the "Maintenance" section of this Instruction Manual.

- Periodic inspection/calibration should be requested to your Shimadzu representative.
- Replacement cycles described for periodic replacement parts are rough estimate.
- Replacement may be required earlier than the described replacement cycles depending on usageenvironment and frequency.

Precautions for Mobile Phase Selection and Use

- Do not use resin parts for the high-pressure tubing while pumping at high pressures. Pumping at high pressure may cause resin tubing to be ruptured or disconnected, which could result in mobile phase leaks. Please note the maximum withstand pressure of each part when resin parts are used for the high-pressure tubing.
- If PEEK resin parts are used in the plumbing, do not use the following mobile phases. These mobile phases weaken the PEEK resin, which could result in cracked plumbing and mobile phase leaks.

Concentrated sulfuric acid, concentrated nitric acid, dichloroacetic acid, acetone, tetrahydrofuran (THF), dichloromethane, chloroform, dimethyl sulfoxide (DMSO), Hexafluoroisopropanol (HFIP) and other fluorinated organic solvents.

Note: Briefly using a weak solution of less than 0.5% acetone in water (e.g. in order to check gradient performance) will present no problems.

NOTE

- Use only HPLC grade or comparable mobile phase, and filter it with a filter of 0.45µm mesh or finer before use to remove particulates and foreign matter.
- Halogen ions can corrode the stainless steel material (SUS316L) used in the plumbing, so if such materials are used for the wetted parts of the equipment, avoid, as much as possible mobile phases that contain halogen ions - such as KCI, NaCI and NH₄CI or mobile phases that generate halogen ions in certain reactions. If such mobile phases must be used, clean all flow lines thoroughly with distilled water immediately after analysis.
- When SPD or a similar UV detector is used for high-sensitivity analysis, be sure to use HPLC grade mobile phases that have a low absorptivity of UV rays.
- Always degas the mobile phase, as air bubbles may tend to form during solvent mixing or during temperature or pressure changes. Air bubbles may cause pump malfunctions and detector signal noise.
- Understand the properties, including boiling points, firing point and viscosities, of the mobile phase.
 Image: "9.5 Mobile Phase Characteristics" P. 9-50.

Warning Labels

For safety operation, warning labels are affixed to where special attention is required. Should any of these labels peel off or be damaged, obtain replacements from Shimadzu Corporation.

S228-45000-32, -38 only



No.	Warning Label	Description
0	▲ WARNING WARNING	(Part No. S228-42603) See instruction manual before replacing fuses





No.	Warning Label	Description
0		(Part No.S037-72999-04) If the main cover needs to be removed, contact your Shimadzu representative to prevent electric shock.
0		 (Part No.S037-72999-02) Before replacing fuses, turn the power to the instrument OFF and unplug the instrument. Only use fuses of the correct type and rating for replacement. Failure to heed the above could result in fire, electric shock or short circuits.

Action for Environment (WEEE) To all users of Shimadzu equipment in the **European Union:**



Equipment marked with this symbol indicates that it was sold on or after 13th August 2005, which means it should not be disposed of with general household waste. Note that our equipment is for industrial/professional use only.

Contact Shimadzu service representative when the equipment has reached the end of its life. They will advise you regarding the equipment take-back.

WEEE Mark

With your co-operation we are aiming to reduce contamination from waste electronic and electrical equipment and preserve natural resource through re-use and recycling.

Do not hesitate to ask Shimadzu service representative, if you require further information.

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1

Configuration

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1.1 Overview

LC-20AD (hereinafter referred to as the instrument) is a dual-plunger parallel-flow solvent delivery module. It offers improved accuracy and sensitivity in high-performance liquid chromatograph. In addition to the instrument, a high performance liquid chromatography system requires an autosampler, column oven, detector and system controller at a minimum, which should be purchased separately. Consult your Shimadzu representative regarding the components required for your system.

1.2 Features

· Low Flow Pulsation and Pulsation Period Enable Precise Delivery

By reducing the discharge volume per plunger stroke to the microvolume level (10μ L), and applying highspeed drive, the flow pulsation and pulsation period have been reduced to levels significantly lower than those for other instruments. This feature enables the use of high-sensitivity detectors, including refractive index detectors and electrochemical detectors, which are sensitive to flow pulsation. It also enables gradient delivery with a degree of precision not usually possible, even with micro-level flow rates.

· Choice of Gradient Modes and Control Options

The LC-20AD can be used in both low- and high-pressure gradient modes. The high-pressure gradient mode uses two LC-20AD pumps and is highly accurate with minimal time lag. In low-pressure gradient mode, a single LC-20AD plus the gradient unit provides a low cost option to combine up to four solvents. The gradient program may be controlled by the LC-20AD itself or, for enhanced control capability, by the CBM-20A/20Alite system controller.

Automatic Plunger Rinsing

An optional automatic mechanism rinses the plunger and the back of the plunger seal, preventing premature seal failure due to crystallization of high-salt-concentration buffer solutions.

1.3 Component Parts

This instrument consists of the standard parts listed below. Check the parts against this list after unpacking. The standard parts provided depend on the power supply voltage. (See below.) After unpacking, verify that the correct types and quantity of parts have been provided.

The 2-digit numbers in the remark column in the table below indicate the power supply voltages for the part. -32, -42 is a 120 V power supply, and -38, -48, -58 a 220-240 V power supply.

Parts	Part No.	Q'ty	Remark
LC-20AD	-	1	
AC Power cord (for UL/CSA)	S071-60821-08	1	-32, -38 only
AC Power cord (for VDE)	S071-60825-51	1	-38, -48, -58 only
Suction filter	S228-45708-91	1	
Instruction manual	S228-30885	1	
Accessory pack (See next page for contents.)	S228-42650-91	1	
Signal cable	S228-35047-92	1	
Event cable	S228-28253-91	1	

Accessory Pack

Parts		Part No.	Q'ty	Remark
Tools	Wrench, 8 × 10	S086-03006	2	
	Allen wrench 4mm	S086-03805	1	
	Allen wrench A 3mm	S670-18928-04	1	
	Allen wrench B 3mm	S086-03804	1	
	Seal installer / remover	S228-18886	1	
	Box driver	S228-28767-91	1	
	File	S670-18928-02	1	For cutting SUS tubing
Parts	Optical cable	S070-92025-51	1	
	Tube clamp	S046-00994-03	1	For securing tubings
	Bottle cap	S228-18887	1	
	Lid	S228-17644	1	
	Pipe clip	S037-60177-05	1	For securing tubings
	Male nut, PEEK	S228-18565	2	
	Male nut, 1.6MN	S228-16001	2	
	Ferrule, 1.6F	S228-16000-10	2	
	Drain tubing	S228-25495-93	1	
	Tubing joint	S228-37146-01	4	
	Transparent tubing	S228-42203	1	1.0m
	SUS pipe 1.6mm O.D. × 0.3mm I.D.	S228-50579-91	1	2m
	Spiral tube wrap	S018-26020-02	1	0.2m
	Syringe	S046-00038-01	1	Volume: 20mL
	Syringe needle	S228-18216-91	1	
	Drain OUT, STD	S228-42205	1	
	Drain OUT, CTO	S228-42206	1	
	Silicone tubing	S228-25162-03	1	1m
	Straight tubing connector	S228-28163	1	
	L-type tubing connector	S035-61561-12	1	
	Drain adapter	S228-42204	1	
	Tubing holder	S228-42209	3	
	Stop joint D	S228-46054-91	1	
	Syringe tubing D	S228-46055-91	1	Volume: 2.5mL
1.4 Optional Parts

Optional units available for this instrument are listed below. For information about other optional units not listed below, contact your Shimadzu representative.

Option	Part No.	Features
Degassing unit (5 flow lines) DGU-20A₅	S228-45019-XX	Degasses mobile phase by passing through special tubing made of resin film, thereby reducing the pressure surrounding the tubing. Can separately degas up to 5 flow lines. Connected to pump unit.
Degassing unit (3 flow lines) DGU-20A ₃	S228-45018-XX	Same as above, but able to separately degas up to 3 flow lines.
Degassing unit DGU-10B	S228-45067-XX	Degasses up to four different mobile phases by means of helium. Can be switched ON/OFF at the instrument or with a system controller.
Low pressure gradient unit	S228-45040-91	 Can switch between up to 4 liquids of low-pressure gradient or mobile phases. Used for auto rinsing of column and flow lines. Installed inside of the instrument.
Reservoir switching valves FCV-11AL	S228-45048-XX	 Reservor switching valves used for switching between pairs of reservoir bottles (for up to 3 flow lines). Each valve switches between two liquids serving one pump (for example, switches between mobile phase and rinse solution). FCV-11AL can perfom switching for up to 3 pumps.
Reservoir switching valve FCV-11ALS	S228-45049-XX	 Switches between two liquids serving one pump (for example, switches between mobile phase and rinse solution). FCV-11ALS can perfom switching for a single pump.
Reservoir switching valve FCV-13AL	S228-45016-XX	 Auto mobile-phase switching valve with 7 ports and 6 positions. Can switch between up to 6 reservoir bottles. FCV-13AL can be controlled via LC workstation and are connected through an option box vp to the system controller.

1. Configuration

Option	Part No.	Features
Option box vp	S228-45060-XX	Can house up to two FCV-11AL, FCV-11ALS or DGU-10B. Can be placed under pump to save space.
Reservoir box	S228-45041-91	Holds up to seven 1-liter bottles.
Mixer 0.5-2.6mL HP	S228-45093-93	Gradient mixer with excellent gradient performance. Capacity can be varied in 3 stages. Can be used for high-pressure, low-pressure or semi-micro gradient analysis.
Mixer 100uL HP	S228-35830-93	Gradient mixer for high speed analysis allowing for gradient delay time. Can be installed inside the instrument using mixer installation kit (D/B) (P/N: S228-45935-91).
Automatic rinsing kit 20AD	S228-45567-91	For continuous automatic rinsing of the back side of the plunger seal, using a rinse solution. Used when system uses high-salt-concentration buffer solution.
Injector mounting plate	S228-35659-92	Plate for installing manual injectors on the instrument.
Column holder	S228-35655-94	For use if the column is installed on the instrument.
CBM-20Alite	S228-45011-XX	CBM-20Alite system controller can be installed inside of the instrument.

2

Parts Identification and Function

Contents

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2.2 Behind Front Cover

Parts marked with an asterisk are common to both pump heads.



 Left rinse-solution outlet Right rinse-solution outlet The outlet for the rinse flow line. The outlet for the rinse flow line. Connect rinse tubing to perform Connect rinse tubing to perform auto rinsing and manual rinsing. auto rinsing and manual rinsing. LC-20AD 00 G.E O func back edit CE \bigcirc (7) (8) (9) del O conc 4 5 6 3 \bigcirc \odot \odot enter Op er) \cap (::: 00 Left rinse-solution inlet Right rinse-solution inlet The inlet for the rinse flow line. The inlet for the rinse flow line. Connect rinse tubing to perform Connect rinse tubing to perform auto rinsing and manual rinsing. auto rinsing and manual rinsing.

2.3 Right Side and Base Panel



2.4 Back



LC-20AD | prominence LIQUID CHROMATOGRAPH

2.5 Names and Functions of Displays and Keypad

panel

This instrument is controlled through the keypad. The display allows verification of the instruments status.

NOTE

The display screen may become hot when in use.

2.5.1 Display Panel

The display panel consists of a display screen and LED indicators.

Names and functions of the display screen and the indications are given below.

olay																
Disp			purr O	ıp	prog. O	run	ren (note D	G	à.e. O		_				
			fun	c)	bac	:k)	ec	tit	(CE	7 (purr)			
			de)	7)		в	(9	(Ċ)			
pad			con	c)	4)		5	(6		rur)			
Keyl		,	C)	(1)		2	(3		purg				
		,	C)	0)		$\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{$	e	nter		VP				
			\subset)	$\left(\right)$	\bigcirc	(slee) p			
_			1)	(2)					(B)	4			
	_													-		
	1	2.	Q	10	Ø	ðп	11	MA	ĄΧ	•	14	3.	Ø			
		Ģ	0.	Ø	MF	^D Q		M:	ΓŅ		Ų	<u>)</u> .	Ø			\square
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	((\neg	/			
	(5			6		(5		(8	_/			(0	

No.	Display or Indicator	Function
1	flow/press	Displays set flow rate (in mL/min) in constant flow pumping mode, and set pressure (in units set with [PRS-UNIT] additional function) in constant pressure pumping mode.
2	pressure	Displays reading of pressure sensor (in units set with [PRS-UNIT] additional function).
3	p.max	Displays pressure upper limit (in units set with [PRS-UNIT] additional function).
4	p.min	Displays pressure lower limit (in units set with [PRS-UNIT] additional function).
5	pump (Pump indicator)	Illuminates when pump is running.
6	prog.run (Program indicator)	Illuminates when program is being executed.
7	remote (Remote mode indicator)	Illuminates when the instrument is controlled by system controller.
8	G.E. (Gradient mode indicator)	Illuminates when the instrument is running in gradient mode. Blinks when the analysis mode is set to Fast LC mode.
9	Status Indicator	Green : when power is ON. Red : when an error is generated. Orange : during sleep mode.

2.5.2 Keypad

The 24 keys on the keypad are used to operate the instrument and set parameters.



Кеу	Indicator	Function
	Display key	To show the operation keys.
pump	Pump key	To start and stop the pump.
run	Run key	To start and stop the time program. (If time program is not registered, this key is inoperative.)
purge	Purge key	To start and stop purging. Purging stops automatically 3 minutes after it begins. Purging can be stopped also by pressing the pump. The duration of purging can be changed using [P-TIMER] of auxiliary function.
VP	VP key	To move from the initial screen to VP mode.
sleep	Sleep key	To turn off the display screen. This key has no effect on operation.
del	Delete key	To delete individual lines of time program on the display screen.
edit	Edit key	To activate edit mode of time program (from the initial screen).
CE	Clear key	 To initialize the screen. To clear the values input up to that time in entering the values. To clear error messages and cancel alarms.
back	Back key	To scroll backward during time program editing.To scroll backward through auxiliary functions setting screens.
conc	Concentration key	To set liquid concentrations in gradient analysis.
func	Function key	 To scroll forward through basic functions. To scroll forward through auxiliary functions. To scroll during time program editing.
enter	Enter key	To validate input values for each item setting.
• - 9	Numeric keypad	To enter numeric values for each item setting.

3 Preparation

Contents

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3.2	Preparation before Operation	3-3
3.3	Checking Pressure Value	3-5

3.1 Turning Power ON/OFF

Press the power switch to turn the power ON. Press it again to turn the power OFF.



Initial Screen



3 The memory is automatically tested, and after the memory check passes, the version number of the control program is displayed momentarily. The screen shown on the right appears and displays the ROM version [V^{*}.**] in this example.

When the power is first turned on, all the dots in

as shown on the right.

the display matrix and all the indicators illuminate,





NOTE

2

- The values displayed on the initial screen are the values last set.
- If an error is detected, an alarm sounds and an error message appears.

6.2 Error Message" P. 6-5



Pressure display [pressure]

3.2 Preparation before Operation

The pumps and internal plumbing of the pump are purged with nitrogen and dried prior to shipment. Prior to initial use (after installation) the pump must first be run for a short time using 2-propanol, in order to expel the air inside its flow lines. Then the instrument will be ready to receive the mobile phase to be used for analysis. The procedure is as follows.

- Pour approximately 100mL of 2-propanol into a beaker.
- 2 Place a suction filter into the beaker.



- 3 Connect one end of the SUS tubing (O.D. 1.6 × I.D. 0.3mm) provided to the pump outlet, and put the other end into the beaker.
- 4 Connect one end of the drain tubing to the drain tubing connection port, and put the other end into the waste container.
- Turn the power ON.
 The initial screen appears.
 "3.1 Turning Power ON/OFF" P. 3-2
- **6** Turn the drain valve knob 180° counterclockwise to open the drain valve.
- 7 Press purge. The pump runs, and the pump indicator illuminates.

NOTE

If the drain valve knob is turned more than 180°, any mobile phase that drains out may contain air bubbles. This is normal.









Fig. 3.4

3. Preparation

- 8 When purging is over, press **func** once, to change the flow rate. Set the flow rate to 5mL/min.
- 9 Turn the drain valve knob clockwise as far as possible, to close the drain valve.
- 10 Press pump. The pumps run, and the pump indicator illuminates.
- 11 After about 15 minutes, press **pump** again. The pump stops, and the pump indicator goes out. Preparation for operation is now completed.



Pump indicator

3.3 Checking Pressure Value

Before beginning operation, check the values for upper and lower pressure limits.





Fig. 3.5

2 To open the drain valve, turn the drain valve knob 180° counterclockwise.

NOTE

If the drain valve knob is turned more than 180°, any mobile phase that drains out may contain air bubbles. This is normal.



Fig. 3.6

3. Preparation

- **3** Press **CE** to return the initial screen.
- Check that the [pressure] value shown on the screen is in the range of -0.3 to 0.3MPa.

NOTE

If not, zero the value of the pressure sensor with [ZERO ADJ] function.

"Zero Adjustment of Pressure Sensor [ZERO ADJ]" P. 5-19

- Check that the pressure upper and lower limits are appropriate values.
 To change the values;
 "4.1 Setting Parameters" P. 4-2
- 6 Press purge

Pumping begins at a registered flow rate.

NOTE

Normally, pumping begins at the set flow rate as soon as **purge**) is pressed. But in the following situations, pumping is initially carried out at low speed (for a few seconds) in order to detect the pump home position, before rising at the set flow rate:

- **(purge**) is pressed for the first time after the power is turned on.
- (**purge**) is pressed for the first time after a pressure upper limit of 22MPa or more has been activated.
- 7 Observe the condition of the mobile phase that flows from the end of the drain tubing for about 10 seconds.

The mobile phase should flow continuously and be free of air bubbles.



Pressure upper limit



Pressure display [pressure] Pressure lower limit



Pump indicator

8 Press purge. The nump stops at

The pump stops, and the pump indicator goes out.

NOTE

• If the flow of mobile phase starts and stops repeatedly in synchronization with pumping:

There are probably air bubbles in the pump heads; press (purge) to expel air.

 If no mobile phase emerges: Attach the syringe needle to the disposable syringe (provided), insert the tip of the needle into the end of the drain tubing, and draw the mobile phase through the tubing.



Fig. 3.7

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4

Basic Operation

Contents

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4.1 Setting Parameters

Before operating this instrument, it is necessary to set the flow rate and the pressure value to protect flow line parts such as column.

The ranges, steps, initial values and their applicable modes of parameters are listed in the following table.

Mode	Parameter	Range	Steps	Initial Value
	flow	0 ~ 10.0000mL/min	0.0001 mL/min	0 mL/min
Constant Flow Delivery	p.max	1.0 ~ 44.0MPa (0 ~ 5.0000mL/min) 1.0 ~ 22.0MPa (5.0001 ~ 10.0000mL/min)	0.1 MPa	10 MPa
	p.min	0.0 ~ 44.0MPa (0 ~ 5.0000mL/min) 0.0 ~ 22.0MPa (5.0001 ~ 10.0000mL/min)	0.1 MPa	0 MPa
	press	1.0 ~ 40.0MPa	0.1 MPa	1.0 MPa
Constant Pressure Delivery	p.max	1.0 ~ 44.0MPa	0.1 MPa	10 MPa
	p.min	0.0 ~ 44.0MPa	0.1 MPa	0 MPa

1kgf/cm² = 0.098MPa = 0.98bar = 14.2psi

4.1.1 Prior to Key Operation

Press **H** to show the operation key.



4.1.2 Setting Flow Rate

The following is the basic procedure for setting a flow rate.

- Display the initial screen, either by turning the power on or by pressing CE several times.
- 2 Press (func). The cursor blinks in the display field, prompting input of a new value.

Enter the new value using the numeric keypad, and press (enter). The new value is set, and the

CE

screen returns to the initial screen.

* To cancel the new setting, press (

0.0	000ml	MAX	10.0
0.	0MPa	MIN	0.0
pump	prog.run	remote	G,E

Flow rate [flow] (with cursor prompting input)



- To change other parameters, press func. Pressing func accesses the items in the following sequence: [flow (press)] \rightarrow [P.max] \rightarrow [P.min]. When an item is accessed, the cursor blinks in the display field, prompting value input. Press func further to access the other auxiliary functions.
- **5** After setting a flow rate, press **CE** to return to the initial screen.



3

4.1.3 Setting Maximum Pressure Limit

The maximum pressure limit is the value of which the pressure in the flow line may not exceed. The purpose of maximum limit is to protect the column and other flow line components.

If pressure exceeds the maximum limit, pumping stops automatically, and an alarm sounds with the message as shown on the right.

ERF	R R	-MAX	
16.	ØMPa	MIN	Ø. Ø
pump	prog.run	remote	G.E

Setting Procedure

The procedure for setting the maximum pressure limit is as follows. For example, 15MPa will be set as the maximum pressure limit.

On the initial screen, press <u>func</u> twice. The cursor blinks in the [P.max] field, prompting input of a new value.

			[P.max]	
Ø. Ø Ø.	1000ml 0MPa	MAX MIN	∎Ø. Ø.	0 0
pump	prog.run	remote	(G.E
0	0	0		0

Press 1, 5 and enter. The new value appears (as shown on the right) on the initial screen.

0.0 0.	0000ml 0MPa	MAX MIN	15.0 0.0
pump	prog.run	remote	G.E.
0	0	0	0

4.1.4 Setting Minimum Pressure Limit

The purpose of the minimum pressure limit is to prevent pressure drop which occur in the following situation.

- When mobile phase in reservoirs runs out, air will be pumped through the flow lines.
- · When a leakage occurs in the flow lines.

If pressure drops to less than the minimum pressure limit and remains for more than 1 minute, pumping stops automatically and an alarm sounds with the message as shown on the right.

ERR Ø.	OR F ØMPa	[»] MIN MIN	2.0
pump	prog.run	remote	G.E
0	0	0	0

4

Setting Procedure

The procedure for setting the minimum pressure limit is as follows. For example, 2MPa will be set as the minimum pressure limit.

1 On the initial screen, press **func** three times. The cursor blinks in the [P.min] field, prompting input of a new value.

0.0	000ml	MAX	15.	0
0.	0MPa	MIN	0 0.	
pump	prog.run	remote		G.E.
O	O	O		O

Press 2, •, 0 and enter. The new value appears (as shown on the right) on the initial screen.

NOTE

When [0] is set as [P.min], pumping will not stop automatically and alarm will not sound when pressure drops.

Ø.(ØMPa	MIN	2.	Ø
pump	prog.run	remote		G.E.

4.2 Operation

There are two basic operating modes; one is in constant flow delivery mode and the other is in constant pressure delivery mode.

4.2.1 Operation in Constant Flow Delivery Mode Turn the drain valve knob clockwise as far as possible to close the drain valve. Press (CE to return to the initial screen. func) once, then, set the desired flow Press ([flow] rate. Example: To set 1mL/min, press (1 and 1.0000mlMAX 10.0 (enter). 0.0MPa MIN 0.0 G.E. pump prog.run remote 0 0 0 0 Press (pump) Δ The pumping starts, and the pump indicator 1.0000ml MAX 10.0 illuminates. 0.0MPa MIN 0.0 Observe the pressure value on the screen to 5 pump prog.run remote G.E. 0 \cap 0 ensure that the pump outlet pressure is rising. Pump indicator Pressure display To stop operation, press (**pump**) again. 6 The pumping stops, and the pump indicator goes out.

Close the drain valve if necessary. When it is left opened, mobile phase could flow out of the drain due to atmospheric pressure.

4.2.2 Operation in Constant Pressure Delivery Mode

- 1 Turn the drain valve knob clockwise as far as possible to close the drain valve.
- 2 Press **func** repeatedly until [CONTROL] appears on the screen.



Press enter).
Press func repeatedly until [MODE CHANG] appears.



Press enter.
 The mode changes from constant flow delivery mode to constant pressure delivery mode.
 Switching Solvent Delivery Mode [MODE CHANG]" P. 5-19



5 Press **CE** twice after mode is changed. The initial screen of constant pressure delivery mode appears.



Press **func**. The cursor blinks in the [press] display field, prompting value input.



6

Enter a value with the numeric keypad.
 Example: To enter 2.0MPa, press 2 and enter.

0.(8MPa	MIN	- <u>0</u> .	0
pump	prog.run	remote		G.E

- 8 Press **pump**. The pump indicator illuminates and pumping starts.
- **9** Observe that the pump outlet pressure rises and pressure display [pressure] stabilizes at about 2.0MPa.
- 2.0MPa MAX 10.0 2.ØMPa MIN 0.0 G.E. pump prog.run remote 0 0 0 • Pump indicator Pressure display [pressure]
- **10** To stop operation, press **pump** again. The pump indicator goes out and pumping stops.

4.3 High-Pressure Gradient Mode

When two pump units are used to form a high-pressure gradient system, two methods of control are possible:

- All pump units controlled by a system controller.
- Pump units connected together; one unit controls the other.

This section explains the second method. For details of the first method, see the system controller Instruction Manual.

4.3.1 Preparatory Settings

Press 2 to set the [SYS] parameter of the master pump unit (hereinafter referred to as pump A).
Setting System Control Parameter [SYS]" P.

Setting System Control Parameter [SYS]" P 5-21 Master pump unit (Pump A)



Press <u>1</u> to set the [SYS] parameter of the slave pump unit (hereinafter referred to as pump B).

Slave pump unit (Pump B)



Press 8 and 5 to set the [ADRS] parameter of both pump units.

 Image: Setting Link Address [ADRS]" P. 5-20

4 Set the maximum and minimum pressure limits of each pump unit.

The limits set for pump A will be valid for pump A only, and those set for pump B valid for pump B only.

From now on, operations will be controlled by pump A only.

"4.1.3 Setting Maximum Pressure Limit" P. 4-4
 "4.1.4 Setting Minimum Pressure Limit" P. 4-5



4.3.2 Setting Initial Conditions

• On the initial screen, press **func**.

2 Set the value of flow rate [flow], enter the flow rate (the total flow rate value of pump A and pump B).

 The mobile phase delivered by pump A is referred to as mobile phase A, and that of pump B as mobile phase B.

- **3** Press **conc**. The screen of setting concentration appears.
- 4 Enter the concentration of mobile phase B (unit:%).

The concentration of mobile phase A will be determined as follows:

Concentration of mobile phase A (%) = 100 (%) - Concentration of mobile phase B (%)

The concentration of mobile phase [B] can be set to any value between 0 and 100%. Minimum setting unit: 0.1%



Press **pump** on pump A. Pumping in pump A and pump B starts at the same time.

4.3.3 Operation in High-Pressure Gradient Mode

After setting initial conditions, press (pump) to start pumping.

Operation of Pump B

- The flow rate of pump B is controlled by pump A; all the other parameter settings of pump B remain effective for pump B.
- **(pump)** remains active and can be used to stop and start pump B at any time, during execution of a time program set on the side of pump A.
- (**purge**) is available to execute purging of pump B.



Do not press **run** on pump B.

If **run**) is pressed, the time program of pump B will start, which may cause incorrect operation.





B:

Π:

remote

0

0.0

0.0

G.E.

A: 100.0

0.0

prog.run

0

C:

gump

 \cap

4.4 Low-Pressure Gradient Mode

When an optional low-pressure gradient unit is connected, quaternary low-pressure gradient delivery can be controlled either by the instrument itself or by a system controller.

This section explains the operation of low-pressure gradient delivery controlled by the instrument itself. For operation controlled by a system controller, see the system controller instruction manual.

4.4.1 Preparatory Setting **0** to set the [FCV-TYPE] parameter. Press ("Selecting Flow Channel Valve Type [FCV 0.000 FCV-TYPE Ø TYPE]" P. 5-22 0:10AL 1:11AL remote G.E. pump prog.run 0 0 0 0 2 4) to set the [SYS] parameter. Press ("Setting System Control Parameter [SYS]" P. 0.000 SYS 4 5-21 Set System Param) twice to return to the initial screen. Press (CE G.E. pump prog.run remote \cap 0 0 Press (func) once. Flow rate [flow] 5.0000ml MAX 10.0 5 Set the flow rate. 0.0MPa MIN 0.0 Enter the total flow rate of mobile phases A, B, C and D. pump prog.run remote G.E. 0 0 0 In the example on the right, 5 mL/min is set for the • flow rate. Press (conc). 6 The screen of setting concentration appears.

Enter the concentrations.

Enter each concentration (%) for mobile phases B, C and D by using the numeric keypad. Press (enter) to move the cursor to the next mobile phase. In this case, since the settings are for initial operation of the gradient system (need to be completed for all flow lines), the same value of

Therefore, set 25% for each. The concentration of mobile phase A will be determined as follows:

mobile phases B, C and D should be entered.

A:	75.	Ø	B:	# 25.	Ø
0	Ø.	Ø	D:	Ø.	Ø
A:	50.	Ø	B:	25.	Ø
0:	25.	Ø	D:	Ø.	Ø
A:	25.	0	B:	25.	0
0:	25.	Ø	D:	# 25.	Ø

Mobile phase A (%) = 100 (%) - Mobile phase B (%) - Mobile phase C (%) - Mobile phase D (%)

The concentrations of mobile phases B, C and D can be set to any value between 0 and 100%. Minimum setting unit is 0.1%.

4.4.2 Initial Operation

- To open the drain valve, turn the drain valve knob 180° counterclockwise .
- Press (pump). Z Pumping starts, and the pump indicator illuminates.







Fig. 4.2

Wait for the flow lines to be filled with mobile phase.

Verify that the mobile phase is continuously flowing.

NOTE

There will be some air bubbles in the flow at first. Wait for a short time until air bubbles disappear.

Run the pump for about 6 minutes, to allow the Δ new mobile phase to completely replace the old mobile phase in the flow lines.

- Press pump.Pumping stops, and the pump indicator goes out.
- 6 Rotate the drain valve lever clockwise until it stops. Close the drain valve.

4.4.3 Operation in Low-Pressure Gradient Mode

When the required settings for flow rate and concentrations are completed, press (**pump**) to start pumping.

4.5 Plunger Rinsing When Buffer Solution is Used as Mobile Phase

When a buffer solution is used as the mobile phase, use distilled water to rinse the inner surfaces of the seals and the plunger surfaces.

The seals and plungers must be rinsed frequently when a buffer solution is used as the mobile phase. Buffer solutions crystallize upon evaporation, and crystals could damage the plungers and plunger seals, which may cause the shortening of their service life.

4.5.1 Operation Method for Automatic Rinsing Kit

Preparation

- Mount the automatic rinsing kit to the instrument using the method described in "9.1.10 Installation of Automatic Rinsing Kit (Optional)" P. 9-38.
- Pull the PTFE tubing connected to the automatic rinsing kit's outlet out of the rinse solution bottle.
 - Draw distilled water into the disposable syringe, and fill the rinse flow line with liquid.
 - * Attach a syringe needle to the tip of the disposable syringe before use.
- Return the PTFE tubing connected to the automatic rinsing kit's outlet to the original state.



Operation

3

4

- Set the flow rate to about 3mL/min.
- Press pump. Pumping starts, and the pump indicator illuminates.

Check whether liquid is coming out of the tip of the PTFE tubing connected to the automatic rinsing

If liquid is coming out, press (pump). The pump

indicator goes out and the pump stops.



Pump indicator



Fig. 4.4

kit's outlet.

- · As a rough guide, replace the rinse solution with distilled water once a day.
- If the volume of rinse solution increases during pumping, it is because of leakage from a plunger seal. Remove the rinsing kit, identify whether the leakage is from the left or right plunger seal, and replace the seal that is leaking in accordance with the procedure "8.2 Replacement of Plunger Seal" P. 8-5.
- If the volume of rinse solution decreases during pumping, it is because of leakage from a diaphragm. Confirm the leakage from the bottom of the head holder, and replace the diaphragm in accordance with the procedure "8.3 Cleaning and Inspection (Replacement) of Plungers and Diaphragms" P. 8-10.

4.5.2 Operation Method for Manual Rinsing

It is possible to manually rinse seals and plungers, without using the optional automatic rinsing kit. The following are examples of manual rinsing cycles.

- Phosphoric acid buffer solution (low concentration): Several times a day
- Boric acid buffer solution (low concentration): Frequently during the day
- Use an automatic rinsing kit for ammonium sulfate (high concentration).

Connection

- Cut the transparent tubing (both provided as accessories) to appropriate lengths.
- 2 Insert tubing joint into the transparent tubing cut off in step 1, as shown in the diagram on the right (Rinse tubing ①, ②).





Connect the rinse-solution outlet of the head holder on the left and right using piece ② of the rinse tubing.



Fig. 4.6

- 4 Connect piece ① of the rinse tubing to the left rinse-tubing inlet and connect the disposable syringe (accessory).
 - * Attach a syringe needle to the tip of the disposable syringe before use.
- 5 Connect piece ① of the rinse tubing to the right rinse-tubing inlet and insert the end of the tubing into a beaker.





Operation

- 1 Remove the disposable syringe from the Rinse tubing.
- 2 Draw rinse solution into the disposable syringe and reconnect it to the Rinse tubing.
- 3 Inject rinse solution into the rinse flow line using the disposable syringe.
 - * Manual rinsing is possible whether the pumps are running or not.

4.6 Replacing the Mobile Phase

The procedure for replacing the mobile phase varies depending on whether the new mobile phase is miscible with the old one, or whether a buffer solution is used for the mobile phase. Replace properly according to the appropriate procedure below. When the column is to be replaced also, remove the column first.

4.6.1 When New Mobile Phase is Miscible with the Old Mobile Phase

- Pour about 100mL of the new mobile phase into a 200mL beaker.
- 2 Remove the suction filter from the reservoir and put the filter into the beaker filled with the new mobile phase by step 1 above.
- 3 Shake the filter slightly to mix the mobile phases.



Fig. 4.8

- 4 Put new mobile phase into the reservoir. Then remove the suction filter from the beaker and put it into the reservoir.
- **5** Turn the drain valve knob 180° counterclockwise to open the drain valve.

NOTE

If the drain valve knob is turned more than 180°, any mobile phase that drains out may contain air bubbles. This is normal.



Fig. 4.9
Press (purge). 6

into the reservoir.

clockwise as far as it will go.

1

8

q

The old mobile phase will be completely expelled from the flow lines through the drain tubing.

Disconnect the tubing. Connecting the manual

injector outlet to the column, and put the tubing

Close the drain valve. Turn the drain valve knob

Drain valve knob Drain tubing





- Set the flow rate [flow] to about 1-3mL/min.
- Press (pump). Pumping starts, and the pump indicator illuminates.
- Press (pump) Pumping stops, and the pump indicator goes out.
- 2 Reconnect the tubing that was disconnected in step 7 to the manual injector outlet.
- **13** Press **pump** again to restart pumping. The mobile phase in the flow line downstream of the manual injector will be replaced.
 - * The flow rate should be set so that the pressure will be lower than the maximum column pressure.







4.6.2 When New Mobile Phase is not Miscible with the Old Mobile Phase

- Prepare an intermediate rinse solution (2-propanol or similar) that is miscible with both the old and new mobile phases.
- Replace the old mobile phase with the intermediate rinse solution.
 "4.6.1 When New Mobile Phase is Miscible with the Old Mobile Phase" P. 4-18, steps 1-11
- Replace the intermediate rinse solution with the new mobile phase.
 "4.6.1 When New Mobile Phase is Miscible with the Old Mobile Phase" P. 4-18, steps 1-11

4.6.3 When Buffer Solution is Used as Mobile Phase

If a buffer solution is used as the mobile phase, it deposits crystals in the flow line when it evaporates.

Distilled or de-ionized water should be used for purging. Use of organic solvents such as 2-propanol may cause crystals.

- Prepare distilled water or de-ionized water.
- Pump at least 200mL of the water prepared in step 1 through the flow line.
 1.4-18, steps 1-11
- Replace the water with the new mobile phase.
 Image: "4.6.1 When New Mobile Phase is Miscible with the Old Mobile Phase" P. 4-18, steps 1-11

4.6.4 Rinse When Buffer Solution is Used

When a buffer solution is used as the mobile phase, it deposits crystals in the flow line when it evaporates. Crystals on the plungers or plunger seals could damage them and shorten their service life, so when a buffer solution is used it is important to rinse the seal inner surfaces and plunger surfaces frequently with distilled water in order to protect them and prolong their service life.

"8.3 Cleaning and Inspection (Replacement) of Plungers and Diaphragms" P. 8-10

When the instrument is to remain inactive following operation with a buffer solution, be sure to replace the buffer solution in the flow lines with distilled or de-ionized water to prevent deposition of buffer solution crystals.

5

Application Operation

Contents

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5.1 Display Panel

5.1.1 Types of Screens

Turn the power ON, the initial screen appears.

By pushing the keys **func**, **VP** and **edit**, the screen can be switched from the initial screen to one of the three screens described below.

- · Basic setting screens and auxiliary function screens
- VP function screen
- Time program edit screen



Time program editing

Basic setting screen and auxiliary

function setting screens

5.1.2 Basic Setting Screen and Auxiliary Function Setting Screen

In this section, basic setting screens and auxiliary function setting screens are shown in the following flow diagrams.

On each screen, press (func) to show the next screen, and press (back) to return.

On auxiliary function group screens, press (enter) to enter each group.

Press **CE** to return the initial screen.

Initial screen 0.0000ml MAX 10.0 0.0MPa MIN 0.0 (func) (back) Flow rate Flow rate ■. 0000ml MAX 10.0 0.0MPa MIN 0.0 CE (func) (back) Maximum pressure limit P.4-4 0.0000m1 MAX 🔳0.0 0.0MPa MIN 0.0 CE (func) (back) Minimum pressure limit P.4-5 0.0000ml MAX 10.0 0.0MPa MIN 0.0 CE (func)









5.1.3 VP Function Screen

In this section VP function screens are shown in the following flow diagrams.

Press **VP** on initial screen to show each group screen.

Press **CE** to return to the initial screen.

Press func or back to switch the setting screen within the groups selected by (VP).

 $\label{eq:press} \fbox{CE} \mbox{ to return to the initial screen in the group.}$









5.2 Parameter in Auxiliary Functions

There are five groups for auxiliary functions:

Parameter Setting, File Operation, Control Setting, System Setting and Monitor Setting.

5.2.1 List of Auxiliary Functions

The auxiliary functions are listed in the tables below. [1] "5.1.2 Basic Setting Screen and Auxiliary Function Setting Screen" P. 5-3

Parameter Settings Group

Command	Operation	Description	Default value	Page
sv	Numeric keypad	To switch the solenoid valve to select mobile phase in optional low-pressure gradient unit or FCV-11AL.	1	P.5-15
EVENT	Numeric keypad	To control output relays.	0	P.5-16
COMP	Numeric keypad	To set a mobile phase compressibility for compensation (fine adjustment).	0.45	P.5-16
P-TIMER	Numeric keypad	To set a purge execution time.	3	P.5-17
P-FLOW	Numeric keypad	To set a purge flow rate.	8.0	P.5-17
P-PMAX	Numeric keypad	To set a maximum pressure limit at purging.	10.0	P.5-17

■ File Operation Group

Command	Operation	Description	Default value	Page
FILE NUM	Numeric keypad	To select a program file No.	0	P.5-18
FILE CPY	Numeric keypad	To copy a file. (including initial conditions and time program)	-	P.5-18
FILE DEL	enter	To delete a time program from a file.	-	P.5-18

About files:

The instrument can store up to 10 files of flow rate and other parameter values, and time programs in memory .

File No. 0 to 9 are assigned to each file. The auxiliary functions other than [SV] and [EVENT] are common functions.

Each value can be switched by [FILE NUM] in the File Operation Group.

* Operation in the table head shows the types of operation described below.

Display : Check the monitor.

(enter) : Press (enter) to activate the function.

Numeric keypad : Press - 9 to enter a value and press enter to determine the value.

Control Settings Group

Command	Operation	Description	Default value	Page
P-SET	Numeric keypad	To replace the plunger and plunger seal.	-	P.5-19
ZERO ADJ	enter	To perform zero adjustment of pressure screen.	-	P.5-19
MODE CHANG	enter	To change solvent delivery mode.	Constant flow solvent delivery mode	P.5-19

System Settings Group

Command	Operation	Description	Default value	Page
LOCAL	Numeric keypad	To select independent operation or control via system controller.	0	P.5-20
ADRS	Numeric keypad	To set address of the instrument for control via system controller.	3	P.5-20
KEY CLOSE	enter	To lock the keypad, preventing unexpected entries.	-	P.5-20
BRIGHT	Numeric keypad	To set brightness of display screen.	3	P.5-21
EXT-S	Numeric keypad	To close [EVENT1] output terminal at the start of time program, and [EVENT2] output terminal on detection of pump error.	0	P.5-21
SYS	Numeric keypad	To select a system control mode.	1	P.5-21
S-PROT	Numeric keypad	To reduce the flow rate without stopping the pumping when the pressure exceeds the p.max value.	0	P.5-22
FCV TYPE	Numeric keypad	To set a type of flow channel valve connected to the [SOL.V] connector on the back of the instrument.	0	P.5-22
PRS-UNIT	Numeric keypad	To set a unit of pressure.	0	P.5-22
RANGE	Numeric keypad	To set a full range for pressure signals for the recorder output.	10	P.5-22
CBM LINK	Numeric keypad	To set a link destination of system controller.	1	P.5-23
BEEP MODE	Numeric keypad	To set the operation of buzzer.	0	P.5-23

Monitor Settings Group

Command	Operation	Description	Default value	Page
MON TIME	Numeric keypad	To monitor the elapsed time in running a time program.	0	P.5-23
MON FLOW	Numeric keypad	To monitor the flow rate in constant pressure delivery mode.	0	P.5-24
MON ID	Numeric keypad	To monitor the pump ID.	0	P.5-24
MON SV	Numeric keypad	To monitor the flow line of solenoid valve unit.	0	P.5-25
MON REV	Numeric keypad	To monitor the pump revolution counter.	0	P.5-25
CONDITION	Display	To monitor the delivery conditions.	-	P.5-25
DEGAS PRS	Display	To monitor the vacuum pressure in degassing unit.	-	P.5-26

* Operation in the table head shows the types of operation described below.

Display : Check the monitor. enter

: $\ensuremath{\mathsf{Press}}$ (enter) to activate the function.

Numeric keypad : Press 9 to enter a value and press enter to determine the value. •)-

5.2.2 Showing the Auxiliary Function Screen

Press (func) four times to show parameter

settings group, the first group of the auxiliary

Pressing more than four times shows the other

* Press (**back**) to return the previous screen.

Select the desired group and press (enter).

groups of the auxiliary function.

1 Press CE. Initial screen appears.

2

3

function.



- Press **func** and select the desired parameter.
- **5** Press **func** or **back** to move to the other functions.
- 6 Press CE to show the group screen. Press CE to return to the initial screen.

G.E.

0

remote

0

pump

Ο

prog.run

0

5.2.3 Parameter Settings Group

This setting group is for pumping parameters.



Setting Solenoid Valve Position [SV]

Selects the position of the solenoid valve in using the low-pressure gradient unit or FCV-11AL reservoir switching valve (option). Enter the value corresponding to the selected mobile phase and press (enter).

Solenoid valve type	Set value	Selected mobile phase
	1	Solvent A
Low-pressure	2	Solvent B
gradient unit	3	Solvent C
	4	Solvent D
Solenoid	Set	Selected mobile phase
valve type	value	
	0	All channels are set to the A side.
Reservoir	1	Channel 1 is set to the B side.
(FCV-11AL)	2	Channel 2 is set to the B side.
(

The above settings can be combined in use.

3

Example: Set [SV] 123 to set all 1, 2 and 3 channels to B side.

Channel 3 is set to the B side.

NOTE

Before setting [SV], select a solenoid valve unit by [FCV TYPE].

"Selecting Flow Channel Valve Type [FCV TYPE]" P. 5-22



5

Setting EVENT Output Terminals [EVENT]

Sets the relays ON (close) / OFF (open) accessed by the EVENT output terminals on the back of the instrument. Enter the value and press (enter).

Set value	[EVENT1] output		[EVENT	2] output
0	Relay 1	OFF	Relay 2	OFF
1	Relay 1	ON	Relay 2	OFF
2	Relay 1	OFF	Relay 2	ON
12	Relay 1	ON	Relay 2	ON



Setting Fine Adjustment of Compressibility Compensation [COMP]

This instrument performs compressibility compensation to minimize pulsation due to mobile phase compressibility. The value set for this parameter is the compressibility of the mobile phase used. This value is utilized to fine-tune the compensation.

For low-pressure pumping, there is no particular need to alter this parameter, but when a high-compressibility mobile phase such as hexane or methanol is delivered at high pressure of 20MPa or more, alter this parameter to the compressibility of the mobile phase used.

The setting units are $(GPa)^{-1}$. Enter the value using numeric keypad, and press **enter**). The initial setting is 0.45.

Mobile phase	Compressibility (GPa) ⁻¹
Water	0.45
Acetonitrile	1.20
Methanol	1.25
Hexane	1.60



■ Setting Purge Time [P-TIMER]

Sets purge time.

Enter a time using the numeric keypad and press (enter).

Setting range is 1-20 (minutes) with 1 minutes step as setting unit.

0.000 P-FLOW**2**8.0

prog.run

0

Input

pump

0

0.1-10.0mL

remote

0

G.E.

0

Setting Purge Flow Rate [P-FLOW]

Sets the flow rate during purging.

It is available to both purging by **purge**) and autopurging by ststem controller.

Enter a flow rate using the numeric keypad and press (enter).

Setting range is 0.1-10.0 (mL/min) with 0.1mL/min step as setting unit.

Setting Maximum Pressure Limit during Purging [P-PMAX]

Sets the maximum pressure limit during auto-purging by system controller.

During purging by **purge**), the maximum pressure limit is 2.0MPa.

Enter a maximum pressure limit using the numeric keypad and press (enter).

[5.5 Control by CBM-20A or CBM-20Alite System Controller" P. 5-56

Set Range	Setting Steps
1.0 ~ 10.0 MPa	0.1 MPa
10 ~ 102 kgf/cm ²	1 kgf/cm ²
10 ~ 100 bar	1 bar
142 ~ 1451 psi	1 psi

 $1 \text{kgf/cm}^2 = 0.098 \text{MPa} = 0.98 \text{bar} = 14.2 \text{psi}$



5

5.2.4 File Operation Group

This is the group for file operation.

File Number [FILE NUM]

This function selects a file.

Up to 10 files of time program can be created and stored in memory.

Enter the desired file number using the numeric keypad [0-9] and press (enter).



FILE

to

prog.run

0

Select

G.E.

0

remote

0

0.000

Enter

pump O

Copying a File [FILE CPY]

The contents of the file currently selected by [FILE NUM] are copied to the selected file number. Enter the file number and press (enter).

Deleting a Time Program [FILE DEL]

The contents of the time program of the currently selected file are deleted. Press (enter).





5.2.5 Control Settings Group

This is the group for control.



Plunger Set [P-SET]

Manually moves the plungers to a safe position for seal replacement or removal of pump head.

Enter the desired value and press (enter).

Pump indicator illuminates and plunger moves.

Set value	Function
1	The left plunger is retracted. In this state, remove the left pump head.
2	The right plunger is retracted. In this state, remove the right pump head.
3	The plungers move to an intermediate position.



Zero Adjustment of Pressure Sensor [ZERO ADJ]

Clears the value of the pressure sensor to zero in atmospheric pressure.



Open the drain valve.

Pressure sensor is released from the pressure.

Press (enter).



Switching Solvent Delivery Mode [MODE CHANG]

Switches constant flow solvent delivery mode or constant pressure solvent delivery mode. Press (enter) to switch between two solvent delivery modes.



NOTE

Switch the delivery mode during pumping stopping. When the delivery mode is changed during pumping, the system may not run correctly.

5.2.6 System Settings Group

This is the group for system settings.



0.000 LOCAL

prog.run

0

Ø: Remote

pump

0

0 0

```
G.E.
```

G.E.

0

1:Local

0

remote

0

Selecting Local/Remote Mode [LOCAL]

Sets whether this instrument is operated by system controller or the instrument operates independently when system controller is connected.

Enter the desired value, and press (enter).

Set value	Mode	Function
0	Remote	Operate via system controller (initial setting)
1	Local	Operate independently (in local mode)

Setting Link Address [ADRS]

Sets the address (channel number) when this instrument is connected to system controller.

Enter the address number, and press (enter).

Address	Function
1-12	Channel numbers when this instrument is connected to system controller.
85	For a case of high-pressure gradient mode, in which two of this instruments are connected in use.

Disables Key Entry [KEY CLOSE]

Press **enter** to prohibit the key entry. After this, key operation is not available.

* To release this function, press (del) while pressing

CE).





Setting Brightness of Display Screen [BRIGHT]

Sets the brightness of display screen. Enter the set value and press **enter**. Value range is 1 to 4 and 4 is the brightest.

Ø.Ø	00	Ŀĸ	16HI	
Ent	er	1	- 4	
pump	pro	g.run	remote	G.E.

External Signal Functions for EVENT Output Terminals [EXT-S]

This feature is typically used to control external devices through the EVENT outputs (relays 1 and 2). Enter the desired value and press (enter).

Set value	Function
0	Relay contact points are controlled by the [EVENT] set value.
1	Relay 1 ([EVENT1] terminals) closes at the start of a time program.
2	Relay 2 ([EVENT2] terminals) closes on detection of an error.
3	Both functions of above 1 and 2 are enabled.



NOTE

The corresponding [EVENT] parameter will not be available when [EXT-S] function is activated.

Setting System Control Parameter [SYS]

Sets system control parameters. Enter the desired value and press (enter).

Set value	Function
1	When used with this instrument operated independently or by any other system.
2	When used as a master pump in high-pressure gradient mode of two units.
4	In low-pressure gradient mode.



Values other than above are invalid.



Setting System Protection [S-PROT]

Halves the flow rate repeatedly until pressure does not exceed [P.MAX].

Enter the desired value and press (enter).

Set value	Function
0	Cancels system protection.
1	Set system protection.

* To clear [ERROR P-MAX], press **CE**. It turns the alarm off, and the flow rate will be increased as before.

Selecting Flow Channel Valve Type [FCV TYPE]

Selects the type of flow control valve connected to the [SOL.V] connector on the back of the instrument. Enter the desired value and press (enter).

Set value	Valve unit
0	When low-pressure gradient unit is built-in
1	FCV-11AL

Selecting a Unit of Pressure [PRS-UNIT]

Sets the units by which the pressure is displayed. Enter a set value and press (enter).

Set value	Unit
0	MPa
1	kgf/cm ²
2	bar
3	psi

1kgf/cm² = 0.098MPa = 0.98bar = 14.2psi

Setting Pressure Recorder Range [RANGE]

Sets the full scale of the pressure recorder output. Enter the desired value and press (enter).

Full scale = (Set value) × 5.0MPaFS

Examples:

- When set value is 1, the full scale is 5.0MPaFS.
- When set value is 10, the full scale is 50.0MPaFS.









Switching Remote Connector internal/external [CBM LINK]

Sets the link destination of system controller. Enter the desired value and press (enter).

Set value	Function
0	Link with CBM-20Alite (option) inside of the instrument.
1	Link with external system controller by optical cable connected to [REMOTE] connector.

■ Setting Operation of Buzzer [BEEP MODE]

Sets the operation of buzzer. Enter a set value and press (enter).

Set value	Function
0	Alarm sound when error occurs and key entry sound are enabled. (default)
1	Only alarm sound when error occurs is enabled. Key entry sound is disabled.
2	All sounds are disabled.

5.2.7 Monitor Settings Group

This is the group for monitor setting.







Monitoring Program Elapsed Time [MON TIME]

Monitors the time elapsed from the start of the time program.

Enter a set value and press (enter).

Set value	Function
0	Clear this function
1	Set this function



If [1] is set, the initial screen will be as shown on the right while the program is running. However, if other monitor functions are set, this screen may not be displayed.

0.0000m	l T	1.00
0.0MPa	MIN	0.0
pump prog.run	remote	G.E.

Monitoring Flow Rate in Constant Pressure Solvent Delivery Mode [MON FLOW]

Monitors the flow rate in the constant pressure solvent delivery mode.

Enter a set value and press (enter).

Set value	Function
0	Clear this function
1	Set this function

If [1] is set, the initial screen in constant pressure solvent delivery mode will be as shown on the right. However, if other monitor functions are set, this screen may not be displayed.





Monitoring ID for Remote Control [MON ID]

Monitors the ID (A, B, C or D) connected to a system controller.

Enter a set value and press (enter).

Set value	Function
0	Clear this function
1	Set this function

If the ID is [A], the initial screen will be as shown on the right.





If the instrument is not connected to a system controller, the initial screen will be as shown on the right.



Monitoring Flow Line of Solenoid Valve Unit [MON SV]

Monitors the flow line of the solenoid valve unit. Enter the set value and press **enter**.

Set value	Function
0	Clear this function
1	Set this function

If [1] is set, the initial screen will be as shown on the right.

NOTE

This function is available also by low-pressure gradient mode. However, the screen sometimes shows the different flow line from the actual, because low-pressure gradient system switches the flow line at very high speed.

Monitoring Pump Revolution Counter [MON REV]

Monitors the total number of pump revolution counter. Enter a set value and press (enter).

Set value	Function
0	Clear this function
1	Set this function

Monitoring the pump revolution counter provides an indication of the timing to replace seals.

Approx. 4,500,000 times of revolution (approx. 1,500 hours by 1mL/min) by running at 10MPa ($102kgf/cm^2$) with 2-propanol is the aim to replace seals.

If [1] is set, the initial screen will be as shown on the right. However, if other monitor functions are set, this screen may not be displayed.

Display of Conditions [CONDITION]

Displays the current instrument conditions. In the normal state, [Press Stable] is displayed.





 Ø. ØØØ MON REV
 III

 Ø: Clear 1: Set
 III

 pump prog.run remote 0.E.
 G.E.

 0
 0
 0





5. Application Operation

If the pressure for the right pump decreases during discharge, [Press Dec Right] is displayed.

If the pressure for the left pump decreases during discharge, [Press Dec Left] is displayed.

If the pressure has been at least 10% higher than the

level shown 5 minutes ago, [Press Increase] is





 Ø. ØØØ
 CONDITION

 Press
 Increase

 pump
 prog.run
 remote
 G.E.

 0
 0
 0
 0

If the pressure has been at least 10% lower than the level shown 5 minutes ago, [Press Decrease] is displayed.

Ø. Pr	000 ess	CO De	ND cr:	IT Pa	IC ==	IN
pump	pro	g.run	ren	note		G.E.
0	(С		0		0

NOTE

displayed.

- [Press Increase] and [Press Decrease] are also displayed during normal operation, such as when the pumps start and stop.
- If [Press Dec Right] is displayed, it may indicate one of the following:
 - ① Air remains inside the right pump head.
 - $\ensuremath{\textcircled{O}}$ There is a leak in the right pump head's seal.
 - ③ The right pump head's inlet check valve or the left pump head's outlet check valve are not operating properly.
- If [Press Dec Right] is displayed, it may indicate that conditions ①, ②, or ③ above have occurred for the left pump head.

Monitoring Vacuum Pressure in Degassing Unit [DEGAS PRS]

Monitors the pressure in the DGU-20A degassing unit.

- When the pressure value is standard, [(OK)] is displayed.
- When the pressure value is not standard, [(NG)] is displayed.
- When the DGU-20A degassing unit is not connected, [(NOT CONNECTED)] is displayed.

Ø.	000 _ \$	DE)3	EGAS kPa	PRS (OK)	
pump	prog	ı.run	remote	ə G	.E.
0	. ()	0	(С

5.3 VP Functions

VP functions support the validation of the instrument by check functions or displaying the instrument information.

There are five groups for VP functions: Mobile Phase Monitor, Product Information, Maintenance Information, Validation Support, and Calibration Support.

5.3.1 List of VP Functions

The VP functions are listed in the tables below.

Mobile Phase Monitor Group

Command	Operation	Function	Page
MOBILE PHASE ^{*1}	Display/ Numeric keypad	To monitor the remaining volume and set volume of mobile phase	P.5-30
ALARM LEVEL	Numeric keypad	To set the alarm level of remaining mobile phase	P.5-30

*1 Mobile phase monitor functions are available only for one type of liquid in standard mode. However, 4 types of liquide (liquide A-D) can be available in low-pressure gradient mode.

Product Information Group

Command	Operation	Function	Page
SERIAL NUMBER	Display	To show the serial number of the instrument	P.5-31
S/W ID	Display	To show the version number of software	P.5-31

Maintenance Information Group

Command	Operation	Function	Page
TOTAL OP TIME	Display	To show the total cumulative operating time of the instrument	P.5-31
L SEAL DELIVERED	Display/ enter	To show the total volume of flow rate through left seal, and volume at a timing to replace a seal	P.5-32
R SEAL DELIVERED	Display/ enter	To show the total volume of flow rate through right seal, and volume at a timing to replace a seal	P.5-32
PART REPLACEMENT	Numeric keypad	To record the replaced parts number	P.5-32
DGU OP TIME	Display/ Numeric keypad	To show and reset the operating time of the DGU-20A degassing unit connected to this instrument	P.5-32
MAINTENANCE LOG	Display/ enter	To show maintenance log	P.5-33
ERROR LOG	Display/ enter	To show error log	P.5-34
OPERATION LOG	Display/ enter	To show operation log	P.5-34

* Operation in the table head shows the types of operation described below.

Display : Check the monitor.

enter : Press enter to activate the function.

Numeric keypad : Press - g to enter a value and press enter) to determine the value.

■ Validation Support Group

Command	Operation	Function	Page
DATE	Display/ Numeric keypad	To show/set the date	P.5-35
TIME	Display/ Numeric keypad	To show/set the time	P.5-35
MEMORY CHECK	enter	To run the memory check	P.5-36
PULSE CHECK	Numeric keypad/	To run the pulsation check	P.5-36
FLOW CHECK	enter	To run the flow rate check	P.5-37
GE TEST PROGRAM	Numeric keypad	To set the time program to check concentration accuracy by gradient mode	P.5-37
LEAKAGE TEST	enter	To run the leakage check	P.5-37
LEAK SENSOR TEST	enter	To run the leak sensor check	P.5-38

Calibration Support Group

Command	Operation	Function	Page
Input PASSWORD ^{*1}	Numeric keypad	To input password	P.5-39
FLOW COMP FACT	Numeric keypad	To enter the flow rate compensation factor (ALPHA)	P.5-39
PRESS COMP FACT	Numeric keypad	To enter pressure sensor sensitivity compensation factor	P.5-39
LEAK THR	Numeric keypad	To enter leak sensor operation level	P.5-40
SEAL REPLACEMENT	Numeric keypad	To set the volume at which seals are replaced	P.5-40
OP MODE	Numeric keypad	To select the operation mode	P.5-40
INITIALIZE PARAM	enter	To initialize parameters	P.5-40
CHANGE PASSWORD	Numeric keypad	To change password	P.5-41
CBM PARAMETER	enter	To show/set CBM-20A(lite) parameters	P.5-41

*1 If the password is not input correctly, the functions after [FLOW COMP FACT] in the Calibration Support Group cannot be accessed, even (func) is pressed.

* Operation in the table head shows the types of operation described below.

Display : Check the monitor.

(enter) : Press (enter) to activate the function.

5.3.2 Displaying the VP Functions



- 4 Follow the further instructions of the selected function.
- 5 To select a different VP Function Group, press VP repeatedly. To select the desired function, press func or

(back)

6 To return to the initial screen, press **CE**.

5.3.3 Mobile Phase Monitor Group

This is the group for mobile phase monitor function.



Showing/Setting Mobile Phase Volume [MOBILE PHASE]

Shows the current remaining volume (\mathbb{O}) and set volume (\mathbb{O}) of mobile phase.

Enter the mobile phase set volume in mL and press (enter).

Remaining volume is automatically calculated by pumping.

Only one type of mobile phase is shown in standard mode. However, 4 types of mobile phases (A-D) can be shown in low-pressure gradient mode.

MOB	ILE	PHASE	
	2	/ 🔢000	2 mL
pump O	prog.ru O	in remote O	G.E . O



Setting Alarm Level of Remaining Mobile Phase [ALARM LEVEL]

Shows the warning indication when the remaining volume of mobile phase is decreased less than the setting value (%). The pumping will continue.



Input the value, and press **enter**). When 0% is registered, this function will be deactivated.

Warning screen



5.3.4 Product Information Group

This group provides the information about the instrument.

PRO Pre	DUCT ss fu	INFO nc or	VP
pump	prog.run	remote	G.E.
0	0	0	0

Showing Serial Number [SERIAL NUMBER]

Shows the serial number of this instrument.



Showing S/W Version No. [S/W ID]

Shows the name of software (same as the model name) and version.

	S/W ID: LC-20AD	V ≭ . ≭≭	
--	--------------------	-----------------	--

5.3.5 Maintenance Information Group

This group provides the maintenance-related information.



■ Showing Total Operating Time [TOTAL OP TIME]

Shows the total operating time of this instrument.

1 (h)		TOTAL	OP	TIME 1(h)	
-------	--	-------	----	--------------	--

Showing Timing to Replace Left Plunger Seal [L SEAL DELIVERED]

Shows the volume of mobile phase delivered through the left plunger seal in use (①), and the volume at which the plunger seal should be replaced (@). (Unit:L) After the seal is replaced, press **enter** to reset the value to [0]. The date will be recorded in the maintenance log.

 The left plunger seal is [L], as viewed from the front of the instrument.



DELIVERED

remote

0

90

Ż

G.E.

0

■ Showing Timing to Replace Right Plunger Seal [R SEAL DELIVERED]

Shows the volume of mobile phase delivered through the right plunger seal in use (①), and the volume at which the plunger seal should be replaced (@). (Unit:L) After the seal is replaced, press **enter** to reset the value to [0]. The date will be recorded in the maintenance log.

* The right plunger seal is [R], as viewed from the front of the instrument.

Entering Replaced Part No. [PART REPLACEMENT]

Enters the replaced part No.

The part No. is recorded in the maintenance log.



SEAL

Ø

prog.run

Ó

/

R

pump

0

Showing Operating Time of DGU-20A Degassing Unit [DGU OP TIME]

Shows the operating time of the DGU-20A degassing unit connected to this instrument.

When the degassing unit is in operation, its status is shown on the lower right part of the display as [(ON)]. Similarly, [(OFF)] is shown when the degassing unit is stopped, and [(NC)] is shown when the degassing unit is not connected. Enter a value for the operating time to start counting the time from the entered value. The entered value is saved in the maintenance log. If 999999 (h) is set, [******] is displayed and the operating time will not be shown.



NOTE

- To use this function, the DGU-20A degassing unit must be connected to this instrument correctly with the power supply cable and vacuum pressure signal cable. If two or more pump units are used, both the power supply cable and the vacuum pressure signal cable must be connected to the same pump unit. For details on connection, refer to the DGU-20A instruction manual.
- To suspend the operation of the DGU-20A degassing unit, use the shutdown function of the CBM-20A/ 20Alite system controller or LC workstation. For details, refer to the CBM-20A/20Alite or LC workstation instruction manual.
- The recommended replacement cycle of the vacuum pump of the DGU-20A degassing unit is 3 year or 8,000 operation hours. Contact your Shimadzu service representative for replacement of the vacuum pump.

Showing Maintenance Log [MAINTENANCE LOG]

Shows the maintenance log, which contains the most recent parts replacement records (part No. and date) (up to 20).

Press **(enter)** repeatedly to show Log1 to Log20 in sequence, and return to the title screen.

In the example on the right, Log1 indicates that part No. 228-30000-91 was replaced on May 12, 2003.

If less than 20 logs are recorded, the screen displays the message as shown on the right.

 $\label{eq:press} \ensuremath{\mathsf{CE}}\xspace\ensuremath{)} \mbox{to return to the title screen.}$



Showing Error Log [ERROR LOG]

Shows the error log, which contains the most recent errors (up to 10) with their dates.

Press **enter** repeatedly to show Log1 to Log10 in sequence, and return to the title screen.

In the example on the right, Log1 indicates that error on maximum pressure limit occurred on May 12, 2003.

If less than 10 logs are recorded, the screen displays the message as shown on the right.

Press **CE** to return to the title screen.



Showing Operation Log [OPERATION LOG]

Shows the operation log, which contains the most recent password settings, parameter initialization, etc. (up to 10).

Press **enter** repeatedly to show Log1 to Log10 in sequence, and return to the title screen.

In the example on the right, Log1 indicates that password was changed on May 12, 2003.

If less than 10 logs are recorded, the screen displays the message as shown on the right.

 $\label{eq:press} \ensuremath{\mathsf{CE}}\xspace\ensuremath{)} \mbox{to return to the title screen.}$


5.3.6 Validation Support Group

This group checks whether the instrument is running correctly.

VAL	IDATI	ON	VP
Pre	SS fu	nc or	
pump	prog.run	remote	G.E

Entering Date [DATE]

Shows/enters the date.

However, the value returns to the initial value [00-00-00] after turning the power OFF.

When the instrument is controlled by a system controller,

the date is transmitted in connecting.

Example: Set January 2, 2003.



YY-MM-DD

03-01-02

Enter year, month and day in 2 digits.

2 Press enter).

Entering Time [TIME]

Shows/enters the time.

However, the value returns to the initial value [00:00:00] after turning the power OFF.

When the instrument is controlled by a system controller, the time is transmitted in connecting.

Example: Setting 5:30:55 p.m.



Entered

Date



1 Enter hour, minute and second in 2 digits. The display uses the 24-hour clock.

Ent	er	ed				
	Ī	1E	H: 7:	M	M : 2	33 55

2 Press enter.

Checking Memory [MEMORY CHECK]

Runs the memory check on ROM and RAM. Press **enter** to start. Results are shown when checking is completed.



Checking Pulsation [PULSE CHECK] Set the criterion of pressure fluctuation width (pulse). PULSE CHECK The unit is MPa. CRITERIA: 🛄. 20MPa Press (enter) to start. 2 remote G.E. pump prog.run 0 0 0 0 Pumping starts at 1mL/min and measuring starts in ten minutes. (enter) During measuring, set flow rate (or pressure value), actual pressure value, pressure fluctuation PULSE CHECK width and remaining time. to Start Enter Results are shown when measuring is complete. (enter) 3 Press (enter). The date and results are recorded. 1.000 wait... 1.99min left NOTE 6.5 1.000 0.07 Ø.99min left When measuring, ensure that parameters for solvent compressibility compensation are set correctly. Setting Fine Adjustment of Compressibility Compensation [COMP]" P. 5-16

PULSE

Ø. Ø7MPa

Enter to Memory

0K

Checking Flow Rate [FLOW CHECK]

- Press enter. Pumping starts at 1mL/min. Prepare for measuring.
- 2 When the preparation for measuring is done, press enter again to measure the required time for flowing liquid to fill 5mL of measuring flask.
- 3 Enter the measured time in seconds. Flow rate accuracy (%) and the check result are shown.

The criterion is within $\pm 2\%$.

NOTE

At the measuring, ensure that the parameter for solvent compressibility compensation are set correctly.

Setting Fine Adjustment of Compressibility Compensation [COMP]" P. 5-16



Setting Time Program to Check Concentration Accuracy of Gradient Mode [GE TEST PROGRAM]

Enter a file number 0-9 of time program in which the default gradient test program is to be copied. This file is automatically loaded for execution.

* The test program is the gradient time program described in P.7-22.



Checking Leakage [LEAKAGE TEST]

- Insert a plug into the pump outlet.
- **Press (enter)** during the pump stopped.
- 3 The pump will run, and stop when the pressure reaches about 30MPa. Then, pressure fluctuations will be monitored.
 - * If the pressure drops, there could be a leakage; inspect the entire flow line.



Checking Leak Sensor [LEAK SENSOR TEST]

Carries on the operation test for leak sensor.

- **1** Use a syringe filled with water to wet the thermosensor at the bottom of the leak sensor.
- 2 Wait about 10 seconds. Then, press (enter). If the sensor detects a leakage, [GOOD] will be shown. If not, [NO GOOD] will be shown.





Fig. 5.1

Press <u>CE</u> to clear the result. If the result is [NO GOOD], adjust the detection level with [LEAK THR] function in the Calibration Support Group.

[Setting Operation Level of Leak Sensor [LEAK THR]" P. 5-40

NOTE

After wetting and testing the leak sensor, wipe away the water on the leak tray completely.



5.3.7 Calibration Support Group

This group calibrates the instrument.



The instrument is adjusted before leaving the factory. Do not change values unnecessarily.

Inputting Password [Input PASSWORD]

Password should be registered by a system manager. Input five numbers and press (enter).

* Be sure to input five numbers. The default password is [00000].

If the password is input correctly, [FLOW COMP FACT] function (subsequent function) appears.

If the password is not input correctly, [FLOW COMP FACT] cannot be accessed.





Inp	ut	PAS	SMORD
PAS	SWO	RD	WRONG

Setting Flow Rate Compensation Parameter (ALPHA) [FLOW COMP FACT]

Enter the value and press **enter**. Reset if the flow rate is not accurate. To increase the flow rate, enter the surplus value (additional flow rate to the original flow rate). To decrease the flow rate, enter the value of which should be decreased from the original value. For example, to increase the parameter by 1.45% (because the current flow rate is 1.45% lower than the set rate) when the current setting value of the parameter is 5, enter 5 + 1.45 = 6.45. This will increase the flow rate by 1.45%.



Setting Pressure Sensor Sensitivity Compensation Factor [PRESS COMP FACT]

Enter the value and press (enter). Reset when the pressure sensor is replaced. Enter the value as a multiple of 100 for PRS-1 (in [1:]) and PRS-5 (in [5:])[XX.XX] shown in the data label of the pressure sensor.



Setting Operation Level of Leak Sensor [LEAK THR]

Sets the level at which the leak sensor is actuated. Enter the level and press **enter**. Use this function to reset the level when the sensor fails [LEAK SENSOR TEST].



■ Setting Timing to replace Plunger Seal [SEAL REPLACEMENT]

Sets the timing to replace the plunger seal. (Unit:L) Enter the set value by numeric keypad and press (enter). It is recommended to replace seals after pumping 90L (equivalent to 1,500 hours at 1mL/min).

SEAL	REPI 90 L	LACEM	ENT
pump	prog.run	remote	G.E.
0	0	0	0

0:20A 1:VP 2:10Ā

prog.run

0

remote

0

G.E.

Ο

OP MODE

pump

Ο

Selecting Operation Mode [OP MODE]

Select the operation mode according to the type of connected system controller.

Enter the number and press **(enter)**.

Set value	System controller
0	CBM-20A/20Alite
1	SCL-10Avp
2	SCL-10A

Initializing Parameters [INITIALIZE PARAM]

Initializes the parameters and deletes the time programs. Press **enter** to return to the default value and to delete the time programs.



Changing Passwords [CHANGE PASSWORD]



5

Press (enter) to return to the title screen.

Showing/Setting CBM Parameter [CBM PARAMETER]

Shows/sets parameters of CBM-20A/20Alite which

controls the instrument. Press **enter** to move to the CBM parameter setting screen.

To select the desired function, press **func** or **back** repeatedly.

To return to the title screen on the right, press (CE



NOTE

When the instrument is not connected with CBM-20A/ 20Alite, or is set to local mode, the CBM parameter setting screen will not appear, even (enter) is pressed.

List of CBM Parameters

SERIAL NUMBER	To show the serial No. of CBM
S/W ID	To show the program version No. of CBM
INTERFACE	To set the transmitting protocol to data processing unit
ETHERNET SPEED	To set the transmitting speed of ethernet *1
USE GATEWAY	To set usage of default gateway or DHCP server *1
IP ADDRESS	To set IP address of CBM *1
SUBNET MASK	To set subnet mask *1
DEFAULT GATEWAY	To set default gateway *1*2
TRS MODE	To select the communication destination when connecting to a LC workstation or a Chromatopac

*1 Available only to show, when not allowed to change on CBM-20A/20Alite side.

*2 Not available when [Default gateway] is not used.

NOTE

Each parameter is activated after CBM is restarted. Refer to CBM-20A/20Alite instruction manual for details of each parameter.

Showing Serial No. [SERIAL NUMBER]

Shows the serial No. of CBM which controls the instrument.



Showing S/W Version No. [S/W ID]

Shows the name of software and version No.of CBM which controls the instrument.



Setting Transmitting Protocol to Data Processing Unit [INTERFACE]

Sets the transmitting protocol from CBM which controls the instrument to data processing unit.

Enter the value by numeric keypad and press (enter).

Set Value	Transmitting Protocol
0	To connect with optical cable
1	To connect with serial transmission (RS-232C)
2	To connect with ethernet



Setting Transmitting Speed of Ethernet [ETHERNET SPEED]

Sets transmitting speed of CBM ethernet which controls the instrument.

Enter the value by numeric keypad and press (enter).

Set Value	Transmitting Speed
0	Auto Detect
1	10Mbps, Half Duplex
2	10Mbps, Full Duplex
3	100Mbps, Half Duplex
4	100Mbps, Full Duplex

ETH Inp	IERNET ut Ø,	SPEED 1-4	
pump	prog.run	remote	G.E.
0	0	0	0

Setting Usage of Default Gateway or DHCP Server [USE GATEWAY]

Sets usage of default gateway or DHCP server of CBM which controls the instrument. Enter the value and press (enter).

Set Value	Description
0	Not use of Default Gateway
1	Use of Default Gateway
2	Automatic Acquisition of IP address from DHCP server

Setting IP Address [IP ADDRESS]

Sets IP address of CBM which controls the instrument. Enter the value and press (enter).





Setting Subnet Mask [SUBNET MASK]

Sets subnet mask of CBM which controls the instrument. Enter the value by numeric keypad and press (enter).



Setting Default Gateway [DEFAULT GATEWAY]

Sets default gateway of CBM which controls the instrument.

Enter the value by numeric keypad and press (enter).



Setting Serial Transmission [TRS MODE]

Selects the communication destination when CBM is connected to a LC workstation or a Chromatopac. Enter the value by numeric keypad and press (enter).

Set Value	Remark
0	To connect in manual setting at CBM
2	To connect to CLASS-VP
3	To connect to LCsolution
11	To connect to C-R8A
12	To connect to C-R7A/C-R5A
13	To connect to C-R4A
14	To connect to C-R6A (without extended ROM board)
15	To connect to C-R6A (with extended ROM board)

TRS MODE $\blacksquare 3$ Input 0, 1-19pumpprog.run \circ \circ \circ \circ

NOTE

Value other than above are invalid.

5.4 Creating Time Program

Time programs can be used to execute commands (for flow rate, etc.) automatically at specified times during operation. Time programs are stored under file numbers (there are 10 files available). Before writing a program, check the file numbers already used. File numbers can be checked and specified using the [FILE NUM] auxiliary function in "File Number [FILE NUM]" P. 5-18.

5.4.1 Time Program Command

Command	Description	Setting Range	Remark	Page
FLOW	To set flow rate (Available only in constant flow solvent delivery mode)	0 -10.0000mL/min	Minimum setting unit: 0.0001mL/min	P.4-3
PRESS	To set pressure (Available only in constant pressure solvent delivery mode)	1.0 - 40.0 MPa 10 - 408 kgf/cm ² 10 - 400 bar 142- 5804 psi	Minimum setting units: 0.1 MPa 1 kgf/cm ² 1 bar 1 psi	P.4-7
SV	To set opening/closing of the solenoid valves of the low-pressure gradient units or FCV-11AL (S) (Optional) (only active when [SYS] ^{*2} = 1 or 2)	0, 1, 2, 3, 4 0 - 123 ^{*1}	Refer to *1	P.5-15
EVENT	To set ON/OFF for EVENT output (of relay contacts at back of the instrument)	0, 1, 2, 12	One of 4 values (For details, P.5-16.)	P.5-16
LOOP	To repeat program	0 - 255 Setting 0 will repeat the program 256 times.	Minimum setting units: 1	P.5-54
STOP	To stop program	N/A		P.5-53
GOTO	To switch to other programs (up to 10)	0 - 9		P.5-54
BCNC	To specify concentration of mobile phase B (only active when $[SYS]^{*2} = 2 \text{ or } 4$)	0 - 100% ^{*3}	Minimum setting units: 0.1%	P.4-9 P.4-11
CCNC	To specify concentration of mobile phase C (only active when $[SYS]^{*2} = 4$)	0 - 100% *3	Minimum setting units: 0.1%	P.4-11
DCNC	To specify concentration of mobile phase D (only active when $[SYS]^{*2} = 4$)	0 - 100% * ³	Minimum setting units: 0.1%	P.4-11

The commands for the time program are listed below.

*1:If Low-Pressure Gradient unit is set for the [FCV TYPE] parameter, the setting range is 1, 2, 3, 4. If FCV-11AL (S) is set, the range is 0-123, using combinations of 0, 1, 2 & 3.

"Selecting Flow Channel Valve Type [FCV TYPE]" P. 5-22

*2:For details of the [SYS] (system parameter) auxiliary function, [Setting System Control Parameter [SYS]" P. 5-21

*3:The total of the settings for [BCNC], [CCNC] and [DCNC] may not exceed 100%. That is: [BCNC] + [CCNC] + [DCNC] ≤ 100. The concentration of mobile phase A will be the value substructed the total value of the other concentrations from 100(%), for example: Concentration A = 100 - ([BCNC] + [CCNC] + [DCNC]).

5.4.5 Creating High-Pressure Gradient Programs" P. 5-51

5.4.6 Creating Low-Pressure Gradient Programs" P. 5-52

5.4.2 Description of Time Program Screen

To create a time program, access the edit screen as described below.

- Press <u>CE</u>. Initial screen appears.
- 2 Press edit.

The screen appears as shown on the right.

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

This example shows that 10 steps of time program have been set, and that 310 steps remain.



10.0

0.0000m1 MAX

3 Press enter.

Editing screen appears.

- ① Elapsed time from program start (minutes)
- ② Command
- ③ Set value

For the following programming procedure, refer to the "5.4.3 Time Program Creating Flow" P. 5-47.



5.4.3 Time Program Creating Flow

In this section, the creating procedure of time programs are shown in the following flow diagrams.



5.4.4 Creating Time Program

The following example gives the steps to create a time program to change the flow rate automatically as shown on the right. (initial flow rate : 1mL/min)

Step	TIME (min)	FUNC	VALUE (mL/min)
1	15.00	FLOW	2.0000
2	20.00	FLOW	5.0000
3	30.00	STOP	



Fig. 5.2

Press **CE**. Initial screen appears.

	¥* #	4.1
remote		G.E
	remote	remote

Press edit. The number of steps already created and remaining steps appear.

US	ED Ø	LEFT 320	
pump	prog.run	remote	G.E.

3 Press enter. The setting screen appears.

Time setting



Step	1	
4	Press 1 , 5 and enter .	Select of command
		15.00 ■LOW 0.0 10.0000 pump prog.run remote G.E. ○ ○
5	Press func repeatedly until [FLOW] appears.	Input the value of the command (Flow rate)
6	Press enter).	15.00 FLOW VALUE 0.0 - 10.0000 mL pump prog.run remote G.E. 0 0 0 0
7	Press 2 and enter .	Determination of step 1
		#5.00 FL0W2.0000 0.01 999.99min pump prog.run remote G.E. 0 0 0 0

Step 2

8 Press 2, 0 and enter to enter the time. Press enter to select [FLOW]. Press 5 and enter to enter the flow rate.

Determination of step 2



Step 3

Select of command



5

- **1** Press **func** repeatedly until [STOP] appears.
- Determination of step 3

0.0	1 - 9	99.99	min
pump	prog.run	remote	G.E.

- Press enter.
- 12 Press **CE** to complete creating program. 3-step time program has created.

U D		LE+ 	
	·:	01/	

13 Press **CE** to save the time program. Initial screen of step 1 appears.



NOTE

When setting several steps, they are ordered automatically in sequence.

5.4.5 Creating High-Pressure Gradient Programs

[BCNC] command of time programs can be used to specify particular concentration of mobile phase B (and thereby concentration of mobile phase A also) at particular times during operation. Concentration of mobile phase A is determined relative to concentration of mobile phase B as follows:

Concentration of mobile phase A (%) = 100 (%) - concentration of mobile phase B [BCNC] (%)

The value of [BCNC] command can be set to any concentration between 0 and 100%. Minimum setting unit is 0.1%.

- Example of Setting [BCNC]
- Enter the time program edit mode.
 1 5.4 Creating Time Program" P. 5-45
- **2** Enter the time.
- **?** Press **func** repeatedly until [BCNC] appears.
- Enter the concentration and press (enter).



As an example of creating a high-pressure gradient program, changing the concentration of mobile phase B is shown at right. (The initial value is 0.0%.)

Step	TIME (min)	FUNC	VALUE (%)
0	20.00	BCNC	100.0
2	30.00	BCNC	100.0
3	30.01	BCNC	0.0
4	40.00	STOP	





Fig. 5.3

5.4.6 Creating Low-Pressure Gradient Programs

[BCNC], [CCNC] and [DCNC] commands of a time program can be used to change the concentrations of mobile phases A, B, C and D at specified times.

The concentration of mobile phase A is determined as follows:

Mobile phase A% = 100 (%) - Mobile phase B% [BCNC] - Mobile phase C% [CCNC] - Mobile phase D% [DCNC]

The values of [BCNC], [CCNC] and [DCNC] can be set to any value between 0 and 100%. Minimum setting units: 0.1%.

Example of Setting [BCNC]

Enter the time program edit mode.
 1 5.4 Creating Time Program" P. 5-45

- 2 Enter the time.
- Press func repeatedly until [BCNC] appears.
 * To set [CCNC] or [DCNC], press func to show [CCNC] or [DCNC].
- Lenter the concentration and press enter.

Time Mobile phase Concentration BCNC 100. 20.00 17 999.99min 0.01 ••••• G.E. remote pump prog.run 0 0 0

5.4.7 Deleting Steps

Call up the steps and press (del).

An example to delete the step 1 of the program set in "5.4.4 Creating Time Program" P. 5-48 is shown as follows:

- 1 Show the step to be deleted. To display the step, follow the same procedure as creating the programs.
 - * To delete a subsequent step, press **enter**) repeatedly until the step appears.



Press <u>del</u>. Step 1 will be deleted, and the subsequent step appears.



5.4.8 Starting and Stopping a Time Program

Starting a Time Program

To start a time program, proceed as follows: Press **run** to start the time program. [prog.run] indicator illuminates.

Stopping a Time Program

There are two ways to stop the time program:

- Press **run** to stop the running program forcefully. The [prog.run] indicator goes out.
- · Insert [STOP] command of the program.
- "Setting Stop Command of Time Program [STOP]" P. 5-53



5.4.9 Commands Used Only For Time Program

This section describes the commands used only for the time program.

Setting Stop Command of Time Program [STOP]

Sets the time when the time program will stop. Enter the time in the last step of time program, and press **func** until [STOP] appears. Enter **enter**, [STOP] command will be set.

NOTE

In the following cases, do not set [STOP] command at the end of the program.

①Operating a time program endlessly.

⁽²⁾Coupling on program files by the [GOTO] command.

"Switching File [GOTO]" P. 5-54



Setting Loop Count of Time Program [LOOP]

[LOOP] function can register the loop count of time program.

30.	00 LO	0P	3
0.0	1 — 9	99.99	Min
pump	prog.run	remote	G.E.

Set as right table for example. Step ${\rm \textcircled{O}}$ and ${\rm \textcircled{O}}$ will be repeated three times in 30 minutes interval.

Step	TIME (min)	FUNC	VALUE (mL/min)
1	15.00	FLOW	2.0000
2	20.00	FLOW	5.0000
3	30.00	LOOP	3

NOTE

• After the time program is repeated as set for [LOOP], how the time program will automatically stop depends on whether [GOTO] command following [LOOP] has been entered or not as follows.

With [GOTO] command:

Until the time set for [GOTO] command is reached, time program maintain the setting values when [LOOP] is ended, and then the next time program starts.

Without [GOTO] command:

The time program will stop when [LOOP] has finished.

- · Steps set after [LOOP] command will deactivate except for [GOTO] command.
- Value of [LOOP] command can be set to 255. When [0] is set, time program will be repeated 256 times.

Switching File [GOTO]

[GOTO] command switches the specified file. When [GOTO] command switches the file, the initial screen of the file newly called up appears, and the instrument waits in standby. However, the program can set to start automatically after [GOTO] command is completed. Follow steps 1 to 6 to set [GOTO] command to start automatically.

Insert the event cable into the external input/ output terminal.

5.7.2 Connection of Event Cable" P. 5-59

Enter the program No. to be switched to.





Fig. 5.5

Add the following step to the original time program.
 ①Set a time earlier than [GOTO] command.
 ②Show [EVENT].

3Set [0].

- **3** Press **CE** to complete the time program.
- Change the file number into the one newly called up.
 "File Number [FILE NUM]" P. 5-18

5 Set the EVENT output terminal of the newly called up file specified in step 4 as [1]. Setting EVENT Output Terminals [EVENT]" P. 5-16

6 Set the file number back to the original file number.





5

5.5 Control by CBM-20A or CBM-20Alite System Controller

5.5.1 Preparation

To control the instrument by the CBM-20A or CBM-20Alite system controller, set the parameters as follows:

Command	Set Value	References
LOCAL	0 : Remote	Selecting Local/Remote Mode [LOCAL]" P. 5-20
ADRS	Link address	Setting Link Address [ADRS]" P. 5-20
OP MODE	0 : 20A	Selecting Operation Mode [OP MODE]" P. 5-40

5.5.2 Basic Parameters

A max. of four pump units can be connected to a CBM-20A/20Alite. CBM-20A/20Alite can set flow rate (pressure), maximum pressure limit, minimum pressure limit, solenoid valves, pumping ON/OFF, time program, etc. and control binary high-pressure gradient, ternary high-pressure gradient and quaternary low-pressure gradient. Also by combining the SIL-20A autosampler, auto purging function is available. Refer to the CBM-20A/20Alite instruction manual.

5.6 Control by SCL-10Avp or SCL-10A System Controller

5.6.1 Preparation

To control the instrument by the SCL-10Avp or SCL-10A system controller, set the parameters as follows:

Command	Set Value	References
LOCAL	0 : Remote	Selecting Local/Remote Mode [LOCAL]" P. 5-20
ADRS	Link address	Setting Link Address [ADRS]" P. 5-20
OP MODE	1 : VP * ¹ 2 : 10A ^{*2}	Selecting Operation Mode [OP MODE]" P. 5-40

*1 To connect to SCL-10Avp : the instrument is recognized as LC-10ADvp.

*2 To connect to SCL-10A : the instrument is recognized as LC-10AD.

5.6.2 Basic Parameters

A max.of three pump units can be connected to a SCL-10Avp or SCL-10A. SCL-10Avp/SCL-10A can set flow rate (pressure), maximum pressure limit, minimum pressure limit, solenoid valves, pumping ON/OFF, time program, etc. and control binary high-pressure gradient, ternary high-pressure gradient and quaternary low-pressure gradient. Refer to the SCL-10Avp/SCL-10A instruction manual.

5.6.3 Attention

When the instrument is connected to a SCL-10Avp or SCL-10A, the instrument will work in compatibility mode for LC-10ADvp or LC-10AD. In this case, the following applies.

1) Maximum limit of flow rate is 9.999mL/min and is set in steps of 0.001mL/min.

2) Maximum value of [P.max] is 43.2MPa.

5.7 Connection to External Input/Output Terminals

The external input/output terminals are connected to a event output device or other external devices with a provided event cable.

Details of the terminal and wiring are described as follows.

- Before connecting the cable, turn off the power and unplug the instrument.
- Use only the specified cable.
- Connect as specified.

Otherwise, fire, electric shock or malfunction may occur.

5.7.1 External Input/Output Terminals



Fig. 5.6

Signals	Description	Remark
EVENT1	[Event1/2] output terminals. To connect to relay and be turned ON/	
EVENT2	OFF according to a time program or the [EVENT] value of auxiliary function.	Contact rating: 30VDC/1A
PUMP ON	[PUMP ON] input terminal. To start pumping.	These signals are
PUMP OFF	[PUMP OFF] input terminal. To stop pumping.	implemented by shorting
START	[START] input terminal. To start the time program. When a start signal is received while program is running, time program is restarted from time [0].	the appropriate wire pair between the input command terminal and the
ERR IN	Error input terminal. To detect error.	Duration of shorting (tc)
СОМ	Common terminal for [PUMP ON], [PUMP OFF], [START], and [ERR IN].	should be as follows. 0.5 sec < tc < 10 sec.

5.7.2 Connection of Event Cable

Peel the cable about 10mm.It is not necessary for provided event cable.



Fig. 5.7

2 Insert the cable.

When the cable has the single core wire, just insert the cable.

When the cable has the stranded wires, strand the wires enough and insert with pressing the button of the terminal.

When removing the cable, remove the cable by pressing the button of the terminal.

NOTE

The instrument provided one event cable. When more than 2 cables are required, use the following cables.

- Cable with single wire : ϕ 0.4 to ϕ 1.2 (AWG26 to 16)
- Cable with stranded wire : 0.3 mm² to 1.25 mm² (AWG22 to 16), diameter of single wire thicker than ϕ 0.18.

The cable with stranded wire is suitable to prevent disconnection.

NOTE

If [EVENT1] or [EVENT2] signal is used, set [EVENT] and [EXT-S] parameters.

Setting EVENT Output Terminals [EVENT]" P. 5-16P. 5-16P. 5-17P. 5-18P. 5-16

Terminals [EXT-S]" P. 5-21

0.	000	ΕVI	ENT	1
Ø,	1,2	or	12	
pump	pro	g.run	remote	G.E.
0	(С	0	0



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6 Troubleshooting

Contents

6.1	Troubleshooting and Corrective Action	6-2
6.2	Error Message	6-5

6.1 Troubleshooting and Corrective Action

This section describes the probable causes of problems that can arise, and the corrective action to be taken to eliminate the causes. For more detailed procedures, refer to the indicated page.

If the problem cannot be resolved even after taking the indicated measures, or if there are problems not included in the following tables, contact your Shimadzu representative.

Symptom	Probable Cause	Corrective Action	Page
Power does not turn ON even after switching ON power.	Power plug is disconnected.	Connect plug correctly.	P.9-5
	Power cord internal wires are cut.	Replace with a new cord of the same type.	P.1-3
	Power supply does not meet specifications for this instrument.	 Use power supply that meets specifications for this instrument. 	P.9-4
	Fuse is blown.	Replace the fuse.	P.8-24
Key operation is not possible.	iii) was not pressed.	 Press <u>III</u>. Operation keys are shown to operate. 	P.2-8
No liquid is pumped. (The instrument does not run.)	(pump) was not pressed. (Pump indicator does not illuminate.)	Press pump. The pump indicator will illuminate.	P.4-6
		 Have the system controller send a signal to start pumping. 	*1
	Flow rate is set to [0].	Set a flow rate other than 0.	P.4-3
		Have the system controller set a flow rate.	*1
	Error message (P.MAX, P.MIN etc.) appears.	Press <u>CE</u> to clear the message and make corrective action for errors.	P.6-5
The instrument is running, but no liquid is pumped.	Air bubbles are generated inside pump head.	 Press (purge) to purge out the bubbles. Insert the disposable syringe into the drain tubing outlet and draw out the bubbles. 	P.3-7
	Air bubbles are produced through suction filter and pump inlet.	 Make sure that the filter bushing is tightly fitted. 	P.9-14
	Check valve is not working properly.	 Push in 2-propanol etc. from the inlet check valve.^{*2} 	

*1:Refer to the system controller instruction manual for details.

*2:How to push in solvent

Unplug the PTFE tubing connected to the inlet check valve. Attach the provided stop joint D to this unplugged PTFE tubing. Connect the provided syringe tubing D to the inlet check valve and push in 2-propanol etc.

*3:How to check clogging of filter

Take off the filter part of the suction filter and record a pressure waveform.

If the pressure waveform becomes ideal by taking off the filter, that suction filter is clogged.

Symptom	Probable Cause	Corrective Action	Page
Pumping is unstable, with large pump pulsation.	Air bubbles are generated inside pump head.	 Press (purge) to purge out the bubbles. Insert the disposable syringe into the drain tubing outlet and draw out the bubbles. 	P.3-7
	Previous mobile phase is still remaining inside pump head.	 Press (purge), and thoroughly purge the old mobile phase. 	
	Air bubbles are generated in filter tubing.	• Press (purge), and thoroughly purge the old mobile phase.	
		 Shake the suction filter to drive out the bubbles. 	
		 If the suction filter is clogged, clean it in an ultrasonic bath, Otherwise, replace it.^{*3} 	P.8-21
		Degas the mobile phase.	P.1-5
	Check valve is not working properly.	 Push in 2-propanol etc. from the inlet check valve.^{*2} 	
		Pump 2-propanol to clean check valve.	P.8-15
		 If cleaning check valve is not effective, clean the valve in an ultrasonic bath or replace it. 	P.8-16
	Liquid is leaking from gap between pump head and head holder, or from rinse flow lines.	Replace the plunger seal.	P.8-5
		 Replace the plunger seal. Otherwise, replace the plunger. 	P.8-10
	Liquid is leaking from flow line connection.	 Retighten the male nuts. Retighten the male nuts. Otherwise, replace the nuts and ferrules. 	P.9-7
	Flow line is (partially) clogged.	Clean the line filter in an ultrasonic bath or replace it.	P.8-19
		Identify the clogged parts and replace it.	
	Plunger seals wear out too quickly.	Replace plunger.	P.8-10
Pumping is unstable,	Check valve is not working	Pump 2-propanol to clean check valve.	P.8-15
with large pump pulsation at low flow rate of 50µl/min or less.	properly.*4	 If cleaning check valve is not effective, clean the valve in an ultrasonic bath or replace it. 	P.8-16

*4:NOTE (Pumping at low flow rate of 50µl/min or less)

Take care not to pump at high flow rate and high pressure (approx. 3ml/min (20MPa) or more) before pumping at low flow rale of 50µl/min or less.

(Pumping may be unstable because of the seal wearing.)

6

Symptom	Probable Cause	Corrective Action	Page
Flow rate is below set value.	Check valve does not work	Pump 2-propanol to clean check valve.	P.8-15
	properly.	 Pump 2-propanol to clean check valve. Otherwise, clean the valve in an ultrasonic bath or replace it. 	P.8-16
	Suction filter is clogged.* ³	Clean Suction filter in an ultrasonic bath. Otherwise, replace it.	P.8-21
Retention time cannot	Check valve does not work	Pump 2-propanol to clean check valve.	P.8-15
be reproduced.	properly.	 If cleaning check valve is not effective, replace the valve or clean it in an ultrasonic bath. 	P.8-16
In a high-pressure gradient system, the two pump units show	Zero points of the pressure sensors of two pump units are not the same.	Use ZERO ADJ auxiliary function to execute zero adjustment of the pressure sensors.	P.5-19
different pressures. (Difference of up to 2.0MPa or 5% is	Line filter of one of the pump units is clogged.	 Clean the line filter in an ultrasonic bath, or replace it. 	P.8-19
normal.)	Flow line(s) is/are clogged at the junction of the two pump unit flow lines.	 Identify the clogged lines and replace it. 	
Pressure does not rise.	Drain valve open.	Close drain valve.	P.2-2
	Liquid is leaking from flow line connections.	 Retighten the male nuts. Otherwise, replace the nuts and ferrules. 	P.9-9
Pressure rises too much. (Remove the column to check.)	Line filter is clogged.	 Clean the line filter in an ultrasonic bath or replace it. 	P.8-19
	Flow lines are clogged.	Identify the clogged parts and replace them.	
	Inner diameter of tubings is too small.	Use tubings with the specified inner diameter.	
The volume of the automatic rinsing kit's rinse solution increases.	Liquid is leaking from the gap between a pump head and a head holder, or from a rinse- solution outlet.	 Replace the plunger seal. If liquid is still leaking after replacing the plunger seal, replace the plunger. 	P.8-10
The volume of the automatic rinsing kit's rinse solution decreases.	Rinse solution is leaking from the back of the head holder.	Inspect (or replace) the diaphragm.	P.4-16

*3:How to check clogging of filter

Take off the filter part of the suction filter and record a pressure waveform.

If the pressure waveform improves by taking off the filter, that suction filter is clogged.

6.2 Error Message

The instrument has several diagnostic functions. Upon detection of a problem, an alarm sounds and an error message appears on the display panel.

The following list describes the error messages along with the causes and corrective actions.

NOTE

Each message is classified into the following three types. The type is indicated under the type column.

- Fatal:
 The instrument stops operation.

 Pressing
 CE will not clear the error message.
- Alarm: The instrument stops operation. Press **CE** to clear the error message.
- Warning: The instrument does not stop operation. Press \bigcirc CE to clear the error message.



Error Message	Туре	Cause and Action
ROM FAILURE (ROM error)	Fatal	Cause: ROM error (electric failure) Action: Turn power OFF and contact your Shimadzu representative.
RAM FAILURE (RAM error)	Fatal	Cause: RAM error (electric failure) Action: Turn power OFF and contact your Shimadzu representative.
ERROR OVER HEAT (Overheating)	Fatal	Cause: Interior temperature has risen to an abnormal level.Action: Check to be sure that the internal fan can move, and that the rear exhaust vent is not blocked. If this message still appears, turn the power OFF and contact your Shimadzu representative.
OUT OF MAXSTEPS (Max. number of steps exceeded)	Alarm	Cause: More than 320 steps of time program have been set. Action: Delete unnecessary programs. []] 5.4 Creating Time Program" P. 5-45

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6. Troubleshooting

Error Message	Туре	Cause and Action
ERROR P-MAX (Max. pressure limit error)	Alarm	Cause: Pump discharge pressure has exceeded the set max. pressure limit, [P.MAX]. Pumping will automatically stop, unless [S-PROT] function has been activated, in which case pumping will continue at half flow rate. Setting System Protection [S-PROT]" P. 5-22 Action: Inspect the clogging in the flow line. If no clogging is found, set the appropriate [P.MAX] value.
ERROR P-MIN (Min. pressure limit error)	Alarm	 Cause: Pump discharge pressure has fallen below the set min. pressure limit, [P.MIN]. Pumping will automatically stop. Action: Inspect the leakage in the flow line. If no leakage is found, set the appropriate [P.MIN] value. Note that [P.MIN] function is not activated for first one minute of pumping. Image: "4.1.4 Setting Minimum Pressure Limit" P. 4-5
ERROR HOME POS (Home position error)	Alarm	 Cause: The motor home position cannot be detected, the motor does not run, or the motor is slipping. Action: Turn the power OFF and ON again, and press pump. If this message still appears, turn the power OFF and contact your Shimadzu representative.
OPEN DRAIN VALVE (Purge error)	Alarm	Cause: Pump discharge pressure has exceeded [P-PMAX] during purging. Action: Open the drain valve before pressing purge . If an error message appears during auto-purging by system controller, inspect the clogging in the flow line. If no clogging is found, set the appropriate [P-FLOW] and [P-PMAX] value. Setting Purge Flow Rate [P-FLOW]" P. 5-17 Setting Maximum Pressure Limit during Purging [P-PMAX]" P. 5-17 System Control by CBM-20A or CBM-20Alite System Controller" P. 5-56

Error Message	Туре	Cause and Action
ERROR LEAK (Leak error)	Alarm	Cause: Leak sensor has detected leakage. Action: Inspect the leakage in the flow line. Wipe away any liquid around the leak sensor.
ERROR EXTERNAL (External equipment error)	Alarm	Cause: An error signal was sent from the external device connected to the external input/output terminal [ERR IN].Action: Inspect the external device, and eliminate the cause of the error.
WARN : M-PHASE (Mobile phase alarm)	Warning	 Cause: The remaining volume of mobile phase is less than the alarm level. Action: Replace the mobile phase and reset the amount of mobile phase. Showing/Setting Mobile Phase Volume [MOBILE PHASE]" P. 5-30
WARN : DEGAS PRS (Degassing unit vacuum pressure error)	Warning	 Cause: Vacuum pressure of degassing unit (DGU-20A) connected to this instrument has been abnormal for a certain time. Action: Check the vacuum pressure of degassing unit. If the vacuum pressure is [NG], turn the power OFF once and turn it ON again. If the vacuum pressure is [OK], it is of no matter, for the degassing unit is returning to run automatically. Image "Monitoring Vacuum Pressure in Degassing Unit [DEGAS PRS]" P. 5-26
WARN : LOST PARAM (Lost parameters error)	Warning	Cause: Parameters may have been lost. Action: Parameter resetting is required. Contact your Shimadzu representative.

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7

Hardware Validation

This chapter provides instruction on hardware validation, which verifies the performance of individual components and the instrument as a whole.

Contents

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7.1 Overview of Hardware Validation

7.1.1 Hardware Validation

Hardware validation examines whether the LC system runs correctly and the instrument is suitable for the intended analysis. Validation is performed through LC system Installation, Operation and Performance Qualifications followed by periodic inspections. The performance of the LC system deteriorates with age, reflecting the wear of consumable parts. Hardware validation must therefore be performed periodically from the time of installation until the system is retired. Although validation aspects related to analysis, such as method validation and system suitability tests should also be performed, hardware validation is a prerequisite for these items.

7.1.2 Types of Hardware Validation

A High Performance Liquid Chromatograph consists of several LC components such as pump(s), autosampler, column oven, and detector(s). For this reason, hardware validation is divided into the inspection of individual components and system validation as a whole.



The operational protocol and criteria for this component and the HPLC system are described in this chapter to assist the user in conducting validation. Refer to each the instruction manual for each component for operational protocol of that specific component.
7.2 Implementation of Hardware Validation

7.2.1 Periodic Validation

Component and system validation must be performed at installation and every 6-12 months, as the performance of an LC instrument changes with age. It is also important to perform maintenance such as replacement of consumables in advance of hardware validation.

7.2.2 Daily Inspection

Daily inspection of the components and HPLC system examine the condition of maintenance parts to ensure a high level of analysis data reliability.

Items such as column deterioration and mobile phase adjustment are examined during system suitability tests.

7.2.3 Validation After Maintenance

After any maintenance, component performance must be re-validated. The type of validation depends on the actual work done.

If the maintenance inspection cannot be performed solely by the specific component validation, system validation is required.

NOTE

Maintenance information and results of hardware validation must be recorded and kept for future reference.

7.3 Validation Precautions

7.3.1 Environment

Instrument performance may be affected by abrupt changes in ambient temperature such as drafts from heating and air conditioning vents.

The equipment should be installed in a room with minimal (< 2°C) temperature fluctuation and away from sources of drafts and air currents.

7.3.2 Installation Site

The installation site is very important for ensuring correct validation. The site should satisfy the following conditions:

• Provide ample ventilation with no fire sources in vicinity When flammable or toxic solvents are used as the mobile phase, the room must be properly ventilated. When flammable solvents are used, open flame or other fire sources must be strictly prohibited.

- Avoid dust or corrosive gas
 Avoid installing the instrument in places subject to excessive dust or corrosive gas since service life
 and performance levels may be affected.
- Keep away from strong magnetic fields
 Do not install the instrument near equipment that generates strong magnetic fields. If the power supply line is subject to high electrical noise, use a commercially-available power surge protector.
- Provide adequate installation surface and space
 The weight of LC-20AD is 10kg. During installation, consider the entire weight combined with other LC components.

The lab table on which this instrument is installed should be strong enough to support the total weight of the LC system. It should be level, stable and have depth of at least 600mm.

If these precautions are not followed, the instrument could tip over or fall off the table.

When components are installed side by side, maintain a keep space of at least 30 mm between the components.

· Regulate room temperature and humidity

The room temperature should be between 4 and 35°C, with minimal temperature variations throughout the course of a day. Humidity should be kept within 20-85%.

• Position instrument properly in the room Install the instrument in a location that is free from vibration and away from sunlight, and heat/air conditioning drafts.

7.4 Equipment Required for Validation

The equipment and samples listed below are required for hardware validation. Prepare necessary equipment and samples depending on the system configuration of the instrument.

Testing Equipment

A list of testing equipment required for hardware validation is shown below. A certificate ensuring traceability or inspection results should accompany each item of testing equipment that is used.

Equipment	Description
Thermo recorder	For inspection of the temperature setting accuracy for the column oven and the autosampler's sample cooler. The thermo recorder must be certified as having an accuracy rating of ±1.0°C for the required temperature range (0°C to 50°C) at the time of inspection.
Resistance thermometer	For inspection of the temperature accuracy for the column oven. The resistance thermometer must have a testing accuracy of $\pm 0.5^{\circ}$ C for the required temperature range (0°C to 50°C) at the time of inspection.
Thermocouple	For inspection of the temperature accuracy for the column oven and autosampler's sample cooler. The thermocouple must have a testing accuracy of ±0.6°C for the required temperature range (0°C to 50°C) at the time of inspection.
DC voltage/current generator	For the hardware validation of the chromatopac. The DC voltage/current generator must be certified as having an accuracy rating of ±0.15% at the time of testing.
Stopwatch	For inspection of the flow rate accuracy for the solvent delivery module. The stopwatch must be certified at 5'30" \pm 0.3sec at the time of inspection.
Measuring flask	For inspection of the flow rate accuracy for the solvent delivery module. Obtain a 5mL-measuring flask.
Electronic balance	For inspection of the injection volume accuracy for the autosampler. The balance must be calibrated and able to perform measurement with a 0.001g precision at the time of inspection.

Standard Reagents for Validation

A list of standard reagents required for validation is shown below. The customer should prepare standard reagents to the stated specifications.

Standard sample	Part No.	Description
Caffeine set (5 concentrations)	S228-45725-42	For inspection of the absorbance linearity for the UV-VIS spectrophotometric and photodiode array detectors. For also inspection of system reproducibility for a system equipped with a UV-VIS spectrophotometric or photodiode array detector.
Caffeine (250mg/L)	S228-45725-06	For inspection of system reproducibility for a system equipped with a refractive index detector, inspection of autosampler carry-over, and inspection of the gradient concentration accuracy for gradient systems.
Naphthalene (60mg/L)	S228-32996-01	For inspection of system reproducibility for a system equipped with a spectrofluorometric detector.
Glycerol (0.872mg/L)	S228-32996-05	For inspection of the span for the refractive index detector.

■ Hardware Testing Supplies

A list of supplies required for hardware validation is shown below. Note that items such as autosampler vials or mobile phase solutions may be required in addition to the items listed.

Implement	Part No.	Description
		I.D. 0.13mm × 2m + I.D. 0.8mm × 2m
Resistor tube	S228-45726-91	For inspection of flow rate and gradient concentration accuracy for
		solvent delivery module, etc.
	S046 00001	For inspection of the absorbance linearity for the UV-VIS
Syringe	3040-00001	spectrophotometric and photodiode array detectors.
Gynnge		For also inspection of the span for the refractive index detector.
	3040-00030-01	This item is provided with detectors as a standard accessory.
Syringe adapter	S228-15672-91	Same as above.
Coupling 1.6C	S228-16004-13	For each kind of inspection and in plumbing the detector.
Male nut, PEEK	S228-18565	Same as above.
Plug	S228-16006	For inspection of the drift/noise for the refractive index detector.
Low-pressure	6000 20402	For inspection of the wavelength accuracy for the UV-VIS photodiode
Hg (Mercury) lamp set	5200-38423	array detector and the spectrofluorometric detector.
	6000 04470 04	For inspection of the wavelength accuracy for the UV-VIS photodiode
S228-34	5220-34170-91	array detector.
ng (mercury) lamp holder	S228-34478-91	For inspection of the wavelength accuracy for the spectrofluorometric
		detector.
DTEE block accombly	0000 04040 04	For inspection of the wavelength accuracy for the spectrofluorometric
	5228-34319-91	detector.
Column		Particle size: 5µm
Shim-pack VP-ODS	S228-34937-91	Column Dimension: I.D. 4.6mm × length 150mm
or	or	(An equivalent ODS column may also be used.)
LUNA C18 (2)	00F-4252-E0	For the system validation.

7.5 Validation: Pump

7.5.1 Check Terms

Check terms for the pump validation are listed below.

		-
	Check Term	Description
7.5.2	ROM, RAM Self Diagnosis	Checks whether the memory (ROM, RAM) functions correctly.
7.5.3	Firmware Version Check	Checks the version of firmware.
7.5.4	Display, LED Test	Checks the operation of display and LEDs.
7.5.5	Flow Rate's Control Parameter	Checks parameters to control the flow rate.
7.5.6	Pumping Stability Test	Checks that the pump's pressure fluctuation width does not exceed the rated value.
7.5.7	Pressure Limiter Test	Checks the operation of pressure limiter.
7.5.8	Leak Sensor Test	Checks the operation of leak sensor.
7.5.9	Flow Rate Accuracy Test	Checks the flow rate accuracy by using a measuring flask.
7.5.10	Solenoid Valve Test	Checks whether the valve switches correctly. (Only when the low-pressure gradient unit is connected.)

7.5.2 ROM, RAM Self Diagnosis

Objective

To check whether the memory (ROM, RAM) functions correctly.

- Check Procedure
- Turn the power switch ON.
- 2 Press VP four times to display [VALIDATION].
- **3** Press **func** three times to display [MEMORY CHECK].
- Press enter). Checking Memory [MEMORY CHECK]" P. 5-36

MEMO	RY	CHECK	
ROM	0K	/ RAM	OK
pump	prog.run	remote	G.E.
0	0	0	0

CHECK CRITERIA : [ROM OK / RAM OK] is displayed on the screen.

7.5.3 Firmware Version Check

Objective

To check the version of firmware.

Check Procedure

- Press **VP** two times on the initial screen. [PRODUCT INFO] appears.
- Press func two times to display the version number.

"Showing S/W Version No. [S/W ID]" P. 5-31

з/ W LC-	1U: 20AD	V*. **	
pump	prog.run	remote	G.E.

CHECK CRITERIA : Version number appears. The number is same as the administrated one.

7.5.4 Display, LED Test

Objective

To check the operation of display and LEDs.

Check Procedure

- Turn the power switch ON.
- 2 Check that all the dots on the screen and LEDs on the keypanel illuminate right after turning ON the power.



CHECK CRITERIA : All the dots and LEDs on the screen illuminate.

7.5.5 Flow Rate's Control Parameter

Objective

To check parameters to control the flow rate.

Check Procedure

- Press **VP** five times on the initial screen. [CALIBRATION] appears.
- 2 Press func once to display [Input PASSWORD].



FLOW COMP FACT

remote

0

prog.run

0

G.E.

0

5.00

pump

0

Enter the password as 5 digits and press enter.
 Imputting Password [Input PASSWORD]" P.
 5-39

[FLOW COMP FACT] appears.

4 Make a note of the displayed value of [FLOW COMP FACT] (ALPHA).

"Setting Flow Rate Compensation
 Parameter (ALPHA) [FLOW COMP FACT]"
 P. 5-39

- 5 Press func once. Make a note of the displayed two values (1: [PRS-1] and 5: [PRS-5]) of [PRESS COMP FACT].
 - Setting Pressure Sensor Sensitivity Compensation Factor [PRESS COMP FACT]" P. 5-39

PRESS COMP FACT 1:1456 5:8535 pump prog.run remote G.E. 0 0 0 0

CHECK CRITERIA : Parameters appear.

7.5.6 Pumping Stability Test

Objective

To check that the pressure fluctuation width does not exceed the rated value.

■ Items Required for Check

ltem	Description
Distilled water	Degas thoroughly using ultrasonic waves or an aspirator.
Resistor tube	

Check Procedure

- 1 Connect the resistor tube to the pump outlet instead of HPLC column.
- 2 Fill the reservoir with distilled water to replace the mobile phase in the flow line.
- **3** Press **func** repeatedly until [PARAMETER] appears and press **enter**).
- Press func or back repeatedly until [COMP] appears.
 Make a note of the current value.
- **5** Press $0 \cdot \bullet \cdot 4 \cdot 5 \cdot$ (enter) to set the value as [0.45].

Make note of this value.





- 6 Press **CE** two times to return to the initial screen.
- 7 Press **VP** four times to display [VALIDATION].

- 8 Press func four times to display [PULSE CHECK].
- 9 Press 0 · · 2 · enter to set [CRITERIA] as [0.20MPa].
- 10 Press enter.

The pump operates at 1mL/min and measurement starts one minute later. The set flow rate, the measured pressure, the pressure fluctuation, and the remaining time are displayed during measurement. After measurement, the measured value and the pass/fail status for the pressure fluctuation width are displayed.

Checking Pulsation [PULSE CHECK]" P. 5-36

The result and date having measured will be

stored at the instrument memory.



CHECK CRITERIA : The measurement result for pressure fluctuation width does not exceed 0.20MPa.

Resetting after Check

Press (enter).

- Press **func** repeatedly until [PARAMETER] appears, and press **enter**).
- 2 Press func or back repeatedly until [COMP] appears. Make a note the current value.
- Return the value to the one recorded in step 4.

7.5.7 Pressure Limiter Test

Objective

To check that the pressure limiter operates properly at the upper and lower limits.

Items Required for Check

Item	Description
Distilled water	Must be thoroughly degassed using ultrasonic waves or an aspirator.
Resistor tube	

Check Procedure

- 1 Connect the resistor tube to the pump outlet instead of HPLC column.
- Fill the reservoir with distilled water to replace the mobile phase in the flow line.
- 3 Turn the power switch ON. Initial screen appears.

Press (1

[1mL/min].

5

6

2. 0 0.	1000ml 0MPa	MAX MIN	10.0 2.0
pump	prog.run	remote	G.E.
0	0	0	0

Press **func** to activate setting of the flow rate.

Cursor (positioned to prompt input)



- - Press **func** two times to activate input of [P.MAX].

) and (enter) to set the flow rate to

7 Press 2, 0 and enter to set [20.0MPa] as [P.MAX].



11.

 \cap

Ø

G.E.

0

9.0

- Press (func) three times to activate input of 8 [P.MIN].
- 9 Press (0 and (enter) to set [0.0MPa] as [P.MIN].
 - Press (**pump**) to start pumping for 1 minute. * The pump indicator will illuminate while pumping.

follows:

* At this time, neither [ERROR P-MAX] nor [ERROR P-MIN] should appear on the screen. Observe the screen and verify that neither appears.





•

- While pumping, change [P.MAX] and [P.MIN] as 1.0000ml MAX 10.0MPa MIN [P.MAX] = actual pressure + 1.0MPa. remote pump prog.run
- [P. MIN] = actual pressure 1.0MPa. * For example, when the actual pressure value is [10.0MPa], set [11.0MPa] as [P.MAX] and [9.0MPa] as [P.MIN].
- Change the flow rate to [1.5mL/min], and observe the pressure.

When the actual pressure exceeds the [P.MAX] value set by step 11, [ERROR P-MAX] should appear.

- Press (CE) to clear [ERROR P-MAX].
- Reset the flow rate to [1mL/min], and press **pump**) again to restart pumping.

		I'I M 🔨	
11.	1MPa	MIN	9.0
pump	prog.run	remote	G.E

0



15 When the pressure has stabilized, change the flow rate to [0.5mL/min].

The actual pressure should fall below the [P.MIN] value set by step 11, and in several seconds, [ERROR P-MIN] should appear.

0.500	2ml MA	X 11.0
5.0M	Pa MI	N 9.0
pump pro	og.run rei	mote G.E.





5.	ØMPa	MIN	9.
pump	prog.run	remote	G
pump O	prog.run	remote O	

CHECK CRITERIA : [ERROR P-MAX] and [ERROR P-MIN] appear.

7.5.8 Leak Sensor Test

Objective

To check the operation of leak sensor.

Check Procedure

- Press **VP** four times on the initial screen. [VALIDATION] appears.
- Press back once to display [LEAK SENSOR TEST].

Checking Leak Sensor [LEAK SENSOR TEST]" P. 5-38

3 Use a syringe filled with water to wet the thermosensor at the bottom of the leak sensor.

LEAK Soak	SEN: and	30R Ent	TEST er
pump	prog.run	remote	G.E.
0	0	0	0



Fig. 7.1

In about 10 seconds, press **enter** to display the test result.

LEA SEN	IK SEN: ISOR GI	SOR DOD	TEST
			0.5
pump	prog.run	remote	G.E.

CHECK CRITERIA : [SENSOR GOOD] appears on the screen.

NOTE

After wetting and testing the leak sensor, wipe away the water on the tray completely.

7.5.9 Flow Rate Accuracy Test

Objective

To check the flow rate accuracy by using a measuring flask.

■ Items Required for Check

Item	Description
Distilled water	Degas thoroughly using ultrasonic waves or an aspirator.
Resistor tube	
Measuring flask (capacity 5mL), or measuring cylinder (capacity 10mL)	Flask/cylinder must have been validated.
Stopwatch	

Check Procedure

- 1 Connect the resistor tube to the pump outlet instead of HPLC column.
- 2 Fill the reservoir with distilled water to replace the mobile phase in the flow line.
- **3** Press **func** on the initial screen to activate setting of the flow rate.

Cursor (positioned to prompt input)



5

6

7

8

Press

0

Press (1 and (enter) to set the flow rate to Δ [1mL/min].

Press (func) repeatedly until [PARAMETER]

Press (func) repeatedly until [COMP] appears.

Then, make a note of the current [COMP] value.

Press (**pump**) to pump for at least 5 minutes.

* During pumping, Pump indicator illuminates.

(enter) to set [0.45] as [COMP].

4

5

and

appears and press (enter).

1.0 0.	000ml 0MPa	MAX MIN	10.0 0.0
pump	prog.run	remote	G.E
0	0	0	0

Make note of this value







Illuminates

- 9 Press (VP) four times on the initial screen. [VALIDATION] appears.
- Press (func) five times until [FLOW CHECK] 10 appears and press (enter).

Ent	w LHE er to	⊃R Start	
pump	prog.run	remote	G.E

Press (enter) again when it is ready to measure.

Ent	d 1⊴ ⊇fr	to	Meas	ure
pump	pro	g.run	remote	G.E.

12 Place the end of the tubing into the flask or cylinder. Then start measuring.

<For a measuring flask>

The inside of the flask must be completely dry.

- 1)Start the stopwatch as soon as the first drop of water emerges from the end of the tubing into the flask.
- 2)Stop the stopwatch when the water is pooled 5mL.





- < For a measuring cylinder>
- 1)Bring the end of the tubing into contact with the cylinder wall.
- 2) Determine a suitable water level inside the cylinder at which to start measurement. Start the stopwatch when the water reaches the determined start level.
- 3)Stop the stopwatch when the water is pooled 5mL.



NOTE

Make sure that the end of the tubing remains above the water in the measuring cylinder throughout the measurement.

13 Enter the time having measured with the numeric keypad and press <u>enter</u>). The flow rate accuracy (%) and the pass/fail status

are displayed.

CHECK CRITERIA : within ± 2.00%

Inpu	ut Ti	me(se	->
299.	3 +	0.07%	OK
pump	prog.run	remote	G.E

7. Hardware Validation

Resetting after Check

- Press **func** repeatedly until [PARAMETER] appears. Then, press **enter**).
- Press func or back repeatedly until [COMP] appears.
- Return the value to the recorded value by step 6.

7.5.10 Solenoid Valve Test

Objective

To check whether the valve switches correctly. Carried out only when the low-pressure gradient unit is connected.

Check Procedure

- Press **func** repeatedly until [SYSTEM] appears and press **enter**.
- Press func or back repeatedly until [SYS] appears.

Then, make a note of the current [SYS] value.

- 3 Press <u>1</u> · <u>enter</u> and set the pumping mode as isocratic.
- 4 Press **CE** two times to return to the initial screen.
- **5** Press **func** once to activate setting of the flow rate.
- 6 Press 1 · enter to set the flow rate to [1mL/min].

Set	Syst	em Pa	ram
pump	prog.run	remote	G.E.



7 Press func repeatedly until [PARAMETER] appears and press enter . [SV] appears.



- **9** Hold up all the suction tubings from the reservoir.
- 10 Press pump to start pumping. Mobile phase A's suction tubing sucks air bubbles approximately 10mm.
- 11 In the same way, change [SV] to [2], [3], and [4] respectively so that the suction tubings of mobile phases B, C, and D suck air bubbles.
- 12 Check that the settings for [SV] correspond to the valves that actually open by observing the movement of the air bubbles.

CHECK CRITERIA : Valve switches correctly.

7.6 Validation: Gradient Pump System (For Gradient System)

7.6.1 Gradient Concentration Accuracy Test for High-Pressure Gradient System

Objective

To check the gradient concentration accuracy for high-pressure gradient system.

■ Items Required for Check

Item		Description
High-pressu	ure gradient system	Pumps, gradient mixer
UV detector	r	SPD-10A, 10Avp, 20A series
Data processor		Chromatopac, LC workstation or similar
Resistor tube		
	Distilled water	
Mobile phase [*] 500mL for each	Caffeine solution (10mg/L)	 (How to prepare) <when for="" reagents="" shimadzu's="" standard="" using="" validation=""></when> Weigh 20mg of caffeine (250mg/L, Part No. S228-45725-06) and bring it to 500mL volume with water. <in by="" case="" customer="" of="" preparation=""></in> Weigh 20mg of anhydrous caffeine, transfer them to a 100mL measuring flask and bring it to 100mL volume with water. Then take 25mL of the solution, and add water to bring it to 500mL volume.

* Both mobile phases must be degassed using helium degassing unit ,or degassing unit.

Check Procedure

- 1 Connect the resistor tube between mixer outlet (1.7mL) and detector.
- 2 As mobile phase, prepare distilled water for mobile phase A, and caffeine solution for mobile phase B (500mL each).
- 3 Degas the mobile phases.
- 4 Open the drain valve and press **purge** of pump A and B to replace the flow lines of each pump.

5 Set the pumping condition as follows and press **pump** to start pumping until the baseline of detector stabilizes.

Total flow rate	= 3mL/min
Concentration of mobile phase B	= 50.0%

3.0 Ø.	0000ml 0MPa	MAX MIN	30.0 0.0
pump	prog.run	remote	G.E
•	0	0	•



Set the parameters of the control device as follows and pump until the baseline stabilizes.

6

LC-20AD

•	Pumping system	Pumping mode	B.GE	
		Flow rate	2.0mL/min	
		Concentration of mobile phase B	0.0%	
•	UV detector	Wavelength	272nm	
		RESPONSE	3 (0.5sec)	
		AUX RANGE	2 (1.0AU/V)	
•	Data processor	1,024 mAU full scale (for Chromatopac, ATTEN=10)		
		(With LCsolution, the intensity axis	s is set automatically.)	
		Chart speed 5mm/min (for Chroma	atopac, SPEED=5)	

- 7 Enter the time program given in the table below, which will produce the operation pattern shown on the right.
 - If a system controller or LC workstation is used, select time program from each menu, and create the program.
 - If control is local, create the time program on pump.



- * The [GE TEST PROGRAM] of VP function can copy the time program to a specified file automatically.
- Setting Time Program to Check Concentration Accuracy of Gradient Mode [GE TEST PROGRAM]" P. 5-37

Time Program

TIME	FUNC	VALUE
5.00	B.CONC	0.0
5.01	B.CONC	10.0
10.00	B.CONC	10.0
10.01	B.CONC	50.0
15.00	B.CONC	50.0
15.01	B.CONC	90.0
20.00	B.CONC	90.0
20.01	B.CONC	100.0
25.00	B.CONC	100.0
25.01	B.CONC	0.0
30.00	STOP	

For C-R8A/R7A/R4A

LINE	PROGRAM
10	LPRINT LEVEL(1);
20	WAIT 30
30	GOTO 10
40	END

For C-R3A/R6A

LINE	PROGRAM
10	PRINT LEVEL;
20	WAIT 30
30	GOTO 10
40	END

For C-R5A

LINE	PROGRAM
10	LPRINT LEVEL;
20	WAIT 30
30	GOTO 10
40	END

If a Chromatopac is used as the data processor, create the BASIC programs given on the right, which print the signal level at every 30 seconds (according to the model).

Zero the baseline, start plotting on the data processor and continue with the gradient time program.

If the Chromatopac is used as data processor, run the BASIC program.

9

8

10 Record the absorbance levels at the following concentrations, and use the data to calculate the actual concentrations.

NOTE

Measure a signal level when the baseline stabilizes.

<Calculation method>

Actual concentration of 10% =	(Absorbance level at set concentration of 10%) (Absorbance level at set concentration of 100%)	(Absorbance level at set concentration of 0%) (Absorbance level at set concentration of 0%)	- × 100
Actual concentration of 50% =	(Absorbance level at set concentration of 50%) (Absorbance level at set concentration of 100%)	(Absorbance level at set concentration of 0%) (Absorbance level at set concentration of 0%)	- × 100
Actual concentration of 90% =	(Absorbance level at set concentration of 90%) (Absorbance level at set concentration of 100%)	(Absorbance level at set concentration of 0%) (Absorbance level at set concentration of 0%)	- × 100

11 If the high-pressure gradient system uses 3 liquids, replace mobile phase of B (pump B) with C (pump C) and test as the same.

CHECK CRITERIA : Within ± 1.0% of set value

7.6.2 Gradient Concentration Accuracy Test for Low-Pressure Gradient System

Objective

To check the gradient concentration accuracy for low-pressure gradient system.

■ Items Required for Check

		Item	Description
Low-press	sure grad	lient system	Pump, low-pressure gradient unit, gradient mixer
UV detect	or		SPD-10A, 10Avp, 20A series
Data proc	essor		Chromatopac, LC workstation or similar
Resistor to	ube		
	А	Distilled water	
Mobile phase [*] 500mL for each	B,C,D	Caffeine solution (10mg/L)	 (How to prepare) <when for="" reagents="" shimadzu's="" standard="" using="" validation=""></when> Weigh 20mg of caffeine (250mg/L, Part No. S228-45725-06) and bring it to 500mL volume with water. <in by="" case="" customer="" of="" preparation=""></in> Weigh 20mg of anhydrous caffeine, transfer them to a 100mL measuring flask and bring it to100mL volume with water. Then take 25mL of the solution, and add water to bring it to 500mL volume.

* Both mobile phases must be degassed using helium degassing unit ,or degassing unit.

Check Procedure

- Connect the resistor tube between mixer outlet (1.7mL) and detector.
- 2 As mobile phase, prepare distilled water for mobile phase A, and caffeine solution for B, C, and D (500mL each).
- **3** Degas the mobile phases and replace the four flow lines with each mobile phase.

4 Set the conditions given below on the control device. Then open the drain valve and run the pumps for 5 minutes to clean the flow lines.

Flow rate	= 9mL/min
Concentration of mobile phase B	= 25.0%
Concentration of mobile phase C	= 25.0%
Concentration of mobile phase D	= 25.0%

9.0000m]	LMAX	30.0
0.0MPa	MIN	0.0
pump prog.run	remote	G.E



5 Change the flow rate to 3.0mL/min, close the drain valve, and run the pumps until the baseline stabilizes.

3.0000ml	MAX	30.0
0.0MPa	MIN	0.0
pump prog.run	remote	G.E

6 Set the parameters of the control device as follows and pump until the baseline stabilizes.

 Pump system 	Pumping mode	LP.GE
	Flow rate	2.0mL/min
	Concentration of mobile phase B	0.0%
	Concentration of mobile phase C	0.0%
	Concentration of mobile phase D	0.0%
 UV detector 	Wavelength	272nm
	RESPONSE	3 (0.5sec)
	AUX RANGE	2 (1.0AU/V)
 Data processor 	1,024 mAU full scale (for chromatopac, ATTEN=10)	
	(With LCsolution, the intensity axis	is set automatically.)
	Chart speed 5mm/min (for chroma	topac, SPEED=5)

- 7 Enter the time program given in the table below, which will produce the operation pattern shown on the right.
 - If a system controller or LC workstation is used, select time program from each menu, and create the program.
 - If control is local, create the time program on pump.



- * The [GE TEST PROGRAM] of VP function can copy the time program to a specified file automatically.
- Setting Time Program to Check Concentration Accuracy of Gradient Mode [GE TEST PROGRAM]" P. 5-37

Time Program

TIME	FUNC	VALUE
5.00	B.CONC	0.0
5.01	B.CONC	10.0
10.00	B.CONC	10.0
10.01	B.CONC	50.0
15.00	B.CONC	50.0
15.01	B.CONC	90.0
20.00	B.CONC	90.0
20.01	B.CONC	100.0
25.00	B.CONC	100.0
25.01	B.CONC	0.0
30.00	STOP	

For C-R8A/R7A/R4A

LINE	PROGRAM
10	LPRINT LEVEL(1);
20	WAIT 30
30	GOTO 10
40	END

For C-R3A/R6A

LINE	PROGRAM
10	PRINT LEVEL;
20	WAIT 30
30	GOTO 10
40	END

For C-R5A

LINE	PROGRAM
10	LPRINT LEVEL;
20	WAIT 30
30	GOTO 10
40	END

If a Chromatopac is used as the data processor, create the BASIC programs given on the right, which print the signal level at every 30 seconds (according to the model).

Zero the baseline, start plotting on the data processor and continue with the gradient time program.

If the Chromatopac is used as data processor, run the BASIC program.

9

8

10 Record the absorbance levels at the following concentrations, and use the data to calculate the actual concentrations.

NOTE

Measure a signal level when the baseline stabilizes.

<Calculation method>

Actual concentration of 10% =	(Absorbance level at set concentration of 10%) (Absorbance level at set concentration of 100%)	(Absorbance level at set concentration of 0%) (Absorbance level at set concentration of 0%)	- × 100
Actual concentration of 50% =	(Absorbance level at set concentration of 50%) (Absorbance level at set concentration of 100%)	(Absorbance level at set concentration of 0%) (Absorbance level at set concentration of 0%)	- × 100
Actual concentration of 90% =	(Absorbance level at set concentration of 90%) (Absorbance level at set concentration of 100%)	(Absorbance level at set concentration of 0%) (Absorbance level at set concentration of 0%)	- × 100

11

Repeat the above check with a pair of mobile phases A and C, also A and D.

CHECK CRITERIA : Within ± 1.0% of set value

7.7 System Validation

- The LC system is comprised of many individual components. System validation is used to confirm the function of each component as well as the performance of the entire system.
- The standard system validation procedure described in this section is used to determine whether the LC system is functioning normally. This procedure constitutes the basis of the LC system capability inspection.
- System validation is performed at installation, and periodically thereafter. If a problem occurs during operation, system validation may be performed to determine whether the problem is in the LC system or in the analysis method.
- If the LC system passes the system validation, it can be assumed that the LC system is normal and that the problem lies in the particular analysis method or conditions being used.
- If the LC system does not pass the system validation, it may be assumed that there is an abnormality in the system, and component validation must be performed to identify the malfunctioning component(s).

7.7.1 Validation of Isocratic LC system

Objective

An analysis is performed and the retention time and peak area are obtained for each peak. The data is then examined to check for reproducibility. Reproducible data validates the system.

Generally, the system being validated consists of a minimum of the following components: pump, column oven, autosampler, detector, system controller and data processor.

Items Required for Validation

Item	Description
Mobile phase	Mixture of water and methanol (3/2, v/v) * Both the water (distilled) and the methanol should be HPLC grade.
Column	Shim-pack VP-ODS (Part No. S228-34937-91) , LUNA C18 (2) (Part No. 00F-4252-E0) or equivalent ODS column (Particle size 5µm, Column Dimension : I.D. 4.6mm × length 150mm)
Sample	20mg/L caffeine solution (included in Caffeine set (5 concentrations) Part No. S228-45725-42) <preparation> Weigh 20mg of anhydrous caffeine, transfer to a 100mL volumetric flask and dilute to volume with water. Transfer 1mL of the solution to a 10mL volumetric flask, and dilute to volume with water.</preparation>
Water	HPLC grade, or equivalent
2-propanol	HPLC grade, or equivalent

- Checking and Preparing the LC system
- 1 Check all the wiring connections in the LC system. Refer to individual component instruction manuals for details. If a Chromatopac is used, it should be connected to the detector with the signal cable connector provided with the Chromatopac, and the signal cable should then be connected to the integrator terminal of the detector.
 - If the system normally uses Chromatopac or LC workstation, the connections used for regular analysis will be satisfactory.
- 2 Check the LC system plumbing. Ensure that the tubing between (a) the autosampler outlet and the column inlet, (b) the column outlet and the detector inlet, has an I.D. of less than 0.3mm, and is shorter than 300mm. Keep the liquid volume that is not in the column as low as possible.
- 3 Clean the system flow lines using one of the procedures described below.

Before cleaning the flow lines, remove the column from the system, and connect the column inlet to the column outlet with a coupling 1.6C ("Fig. 7.6").

< For a new system >

Clean the flow lines first with 2-propanol, then with water. In each case, pass the liquid through the flow lines for 10 minutes, at a rate of 2mL/min.

< For a system in use that uses a mobile phase with a low dielectric constant, such as hexane>

The procedure is the same as that of a new system, given above.

< For a system that has been using a mixture of a water solution and an organic solvent as mobile phase, or water plus an organic solvent miscible with water (methanol, acetonitrile, etc.) >

Clean the flow lines with water. Pass water through the flow lines for 10 minutes, at a rate of 2mL/min.







- 4 When cleaning is finished, pour mobile phase (mixture of water and methanol (3/2, (v/v)) into the reservoir, and reconnect the column with the LC system ("Fig. 7.7"). Pour water into the reservoir as a rinse liquid, and purge the flow line.
- Validation Procedure
- Set the pumping flow rate to 1mL/min. See the pump's instruction manual for setting procedures.

Pump's display screen

1.0 0.	000ml 0MPa	MAX MIN	20.0 0.0
pump	prog.run	remote	G.E
0	0	0	0

2 Set the column oven temperature to 40°C. See the column oven's instruction manual for setting procedures.

3 Press **pump** on the pump keypad, and **oven** on the column oven keypad. Pumping and temperature regulation will start.

Verify that liquid flows through the detector outlet tubing, and that there are no leaks from any of the connections.

Set the detector parameters.

Validation" P. 7-31

See the detector's instruction manual for setting procedures.

Set the autosampler parameters.

Validation" P. 7-31

See the autosampler's instruction manual for setting procedures.

Set the data processor parameters.

Validation" P. 7-31

See the data processor's instruction manual for setting procedures.

Column oven's display screen



Detector's display screen



5

6

7 Monitor the baseline.

When the baseline has stabilized, press the detector (zero) key, then inject $10\mu L$ of mobile phase, and verify that no peaks are observed.

8 Inject 10µL of the test standard six times, and analyze the data obtained.

9 From the peak data obtained from the six analyses, derive the relative standard deviation (coefficient of variation (C.V.)) for: retention time and peak area ("Fig. 7.8").



Fig. 7.8

Parameter Settings for Isocratic System Validation

The parameters to be set for the various devices when validation analysis of an isocratic system is performed are given below.

• Pump	Flow rate	: 1mL/min
	P.Max	: 20.0MPa
Column oven	Oven temperature	: 40°C
Time program	5.00 STOP	
Autosampler	RINSE VOLUME	: 200µL
	RINSE SPEED	: 35µL/s
	SAMPLING SPEED	: 15µL/s
	RINSE MODE	: 0 (No needle rinsing)
Detector	Wavelength	: 272nm
	AUX RNG	: 2 (1AU/V)
	RESPONSE	: 3 (0.5s)
Data processor	WIDTH	: 5
	DRIFT	: 0
	T.DBL	: 1000
	ATTEN	: 10 (1,024mAUFS)
	SLOPE	: 1000
	MIN.AREA	: 100000
	STOP.TM	: 5

CHECK CRITERIA

The RSD (C.V.)'s obtained must satisfy the following criteria: Retention time RSD must not exceed 0.5%. Peak area RSD must not exceed 1.0%.

7.7.2 Validation of Gradient LC System

Objective

An analysis is performed and the retention time and peak area are obtained for each peak. The data is then examined to check for repeatability. Reproducible data validates the system.

Generally, the system being validated consists of a minimum of the following components: pump, column oven, autosampler, detector, system controller and data processor.

Items Required for Validation

Item	Description
Mobile phases	A: Distilled water B: Methanol A /B =60%/40% * Both the water (distilled) and the methanol should be HPLC grade.
Column	Shim-pack VP-ODS (Part No. S228-34937-91) , LUNA C18 (2) (Part No. 00F-4252-E0) or equivalent ODS column (Particle size 5µm, Column Dimension : I.D. 4.6mm × length 150mm)
Sample	20mg/L caffeine solution (included in Caffeine set (5 concentrations) Part No. S228-45725-42) <preparation> Weigh 20mg of anhydrous caffeine, transfer to a 100mL volumetric flask and dilute to volume with water. Transfer 1mL of the solution to a 10mL volumetric flask, and dilute to volume with water.</preparation>
Water	HPLC grade, or equivalent
2-propanol	HPLC grade, or equivalent

Checking and Preparing the LC System

- 1 Check all the wiring connections in the LC system. Refer to individual component instruction manuals for details. If a Chromatopac is used, it should be connected to the detector with the signal cable connector provided with the Chromatopac, and the signal cable should then be connected to the integrator terminal of the detector.
 - If the system normally uses Chromatopac or LC workstation, the connections used for regular analysis will be satisfactory.
- 2 Check the LC system plumbing. Ensure that the tubing between (a) the autosampler outlet and the column inlet, (b) the column outlet and the detector inlet, has an I.D. of less than 0.3mm, and is shorter than 300mm. Keep the liquid volume that is not in the column as low as possible.



Clean the system flow lines using one of the procedures described below.

Before cleaning the flow lines, remove the column from the system, and connect the column inlet to the column outlet with a coupling 1.6C ("Fig. 7.9").

< For a new system >

Clean the flow lines first with 2-propanol, then with water. In each case, pass the liquid through the flow lines for 10 minutes, at a rate of 2mL/min.

< For a system in use that uses a mobile phase with a low dielectric constant, such as hexane>

The procedure is the same as that of a new system, given above.

< For a system that has been using a mixture of a water solution and an organic solvent as mobile phase, or water plus an organic solvent miscible with water (methanol, acetonitrile, etc.) >

Clean the flow lines with water. Pass water through the flow lines for 10 minutes, at a rate of 2mL/min.

When cleaning is finished, pour mobile phase (A: water, B: methanol) into the reservoir, and reconnect the column with the LC system ("Fig. 7.10"). Pour water into the reservoir as a rinse liquid, and purge the flow line.

Validation Procedure

Set the pumping flow rate to 1mL/min, and set the concentration of mobile phase B parameter to 40%. See the pump's instruction manual for setting procedures.



Fig. 7.10

Pump's display screen



- 2 Set the column oven temperature to 40°C. See the column oven's instruction manual for setting procedures.
- Press pump on the pump panel, and oven on the column oven panel. Pumping and temperature regulation will start. Verify that liquid flows through the detector outlet tubing, and that there are no leaks from any of the connections.
- Set the detector parameters.
 Parameter Settings for Gradient System Validation" P. 7-35

See the detector's instruction manual for setting procedures.

5 Set the autosampler parameters. Parameter Settings for Gradient System Validation" P. 7-35

See the autosampler 's instruction manual for setting procedures.

Set the data processor parameters. Parameter Settings for Gradient System Validation" P. 7-35

See the data processor's instruction manual for setting procedures.

Monitor the baseline.

6

When the baseline has stabilized, press the detector (zero) key. Then inject 10µL of mobile phase and verify that no peaks are observed the second time.

- Inject 10μL of the test sample six times, and analyze the data obtained.
- **9** From the peak data obtained from the six analyses, derive the relative standard deviation (coefficient of variation (C.V.)) for: retention time and peak area ("Fig. 7.11").

Column oven's display screen



Detector's display screen



RSD(C	$(SD/\overline{X}) \times 100$
SD =	$\sqrt{\frac{\sum\limits_{i=1}^{n} (Xi-X)^2}{n-1}}$
$\overline{X} = (X$	1 + X2 •••• Xn - 1 + Xn)/n
n	: Number of analyses
X1••XI	n : Retention time (or areas) of each peak
X	: Average
SD	: Standard deviation
RSD	: Relative standard deviation
C.V.	: Coefficient of variation

Parameter Settings for Gradient System Validation

The parameters to be set for the various devices when validation analysis of a gradient system is performed are given below.

• Pump	Flow rate	: 1mL/min
	B.CONC	: 40%
	P.Max	: 20.0MPa
Column oven	Oven temperature	: 40°C
Time program	5.00 STOP	
Autosampler	RINSE VOLUME	: 200µL
	RINSE SPEED	: 35µL/s
	SAMPLING SPEED	: 15µL/s
	RINSE MODE	: 0 (No needle rinsing)
Detector	Wavelength	: 272nm
	AUX RNG	: 2 (1AU/V)
	RESPONSE	: 3 (0.5s)
Data processor	WIDTH	: 5
	DRIFT	: 0
	T.DBL	: 1000
	ATTEN	: 10 (1,024mAUFS)
	SLOPE	: 1000
	MIN.AREA	: 100000
	STOP.TM	: 5

CHECK CRITERIA

The RSD (C.V.)'s obtained must satisfy the following criteria: Retention time RSD must not exceed 0.5%. Peak area RSD must not exceed 1.0%.

7.8 If Validation Fails

Should the system fail to satisfy any of the system validation check criteria, or should a component fail to satisfy any of the component validation check criteria, proceed as follows.

- Check whether any consumable items have reached the end of their service life: The cause of failure to satisfy check criteria could be a consumable part that is no longer usable. Check consumable parts and replace them if necessary.
- Perform troubleshooting:

It is possible that some minor problem (such as air bubbles) has caused the system to fail the criteria. Perform troubleshooting to check for such problems, and take action to eliminate any problems found. For troubleshooting procedures for individual system components, see the applicable instruction manuals.

• If a cause cannot be determined, contact your Shimadzu representative: If you are unable to determine the cause of the failure, or if you are unclear about troubleshooting or corrective action procedures, contact your Shimadzu representative.

8 Maintenance

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8.1 Periodic Inspection and Maintenance

It is necessary to perform periodic inspections of this instrument to ensure its safe use. It is possible to have these periodic inspections performed by Shimadzu service representatives on a contractual basis.

For information regarding the maintenance inspection contract, contact your Shimadzu representative.

🕂 WARNING

• Unless the instructions here specified, turn off the power always and unplug the instrument prior to performing inspections and maintenance. Otherwise, fire, electric shock or malfunction may occur.

- When replacing parts, use only the parts listed in "1.3 Component Parts" and "9.3 Maintenance Parts". If any other parts are used, injury or malfunction may occur.
- Never remove the main cover. Otherwise, injury or malfunction may occur. Contact your Shimadzu representative to remove the main cover.

8.1.1 Prior to Inspection and Maintenance

- · Replace the mobile phase in the flow lines with water.
- Wipe away any dirt from the front panel and the main cover.
- Wipe away any dirt from the keypad with tissue paper or a soft cloth moistened with water.
8.1.2 List of Periodic Inspection and Maintenance

The replacement and maintenance periods listed in this table are presented only as guidelines. These are not guarantee periods. These will vary depending on usage conditions.

Inspection/Maintenance Item	1 year	2 years	3 years	Remark	Page
Replacement of Plunger seal	x			 Sealing efficiency decreases when seals are worn. Replace when the plunger is replaced. As a guideline, seals should be replaced after delivery of the respective volumes listed below when 2-propanol is pumped. (VP function, [L(R) SEAL DELIVERED], shows the total delivered volume.) Pumping Pressure Total Delivery 10 MPa (102 kgf/cm²) 90 L 30 MPa (306 kgf/cm²) 30 L *1 *1 With using optional automatic rinsing kit 	P.8-5
Cleaning and inspection (replacement) of Plunger	×				P.8-10
Inspection (replacement) of diaphragm		×		Replace when the plunger is replaced.	P.8-10
Inspection (replacement) and ultrasonic bath cleaning of outlet check valve	×				D 9 16
Inspection (replacement) and ultrasonic bath cleaning of inlet check valve	×				F.0-10
Inspection (replacement) of line filter		×		 Particulates in mobile phase clog the filter in prolonged use. 	P.8-19
Inspection (replacement) and cleaning of suction filter		×		Particulates in mobile phase clog the filter in prolonged use.	P.8-21
Replacement of Drain valve			×	Sealing efficiency decreases when the drain valves are worn.	P.8-22
Pump ASSY lubrication			×	Contact your Shimadzu representative.	-
Fuse replacement			×		P.8-24



8.1.3 Check after Inspection and Maintenance

After inspection and maintenance, check any leakage during pumping.

"6.2 Error Message" P. 6-5

8.2 Replacement of Plunger Seal

Plunger seals are mounted inside both (right and left) pump heads. The procedure given below is for replacement of the seal inside the right pump head (as viewed from the front of the instrument).

Necessary parts

Part	Туре	Part No.
Plunger seal	Consumable part	S228-35146

8.2.1 Before Removing Pump Head

Retract the plunger on the replacement side (i.e., on the right) to prevent it being bent.



Turn the power switch ON. Initial screen appears.

Initial screen

[P-SET] display

0.(0.	0000ml 0MPa	MAX MIN	10. 0.	10 12
pump	prog.run	remote		G.E
0	0	0		0

Press func repeatedly until [CONTROL] appears, and press enter. [P-SET] appears on the screen and the cursor blinks in the [P-SET] input field, prompting input.



Press 2 and enter.
 (When replacing the left plunger seal, press
 1 and enter.)
 The pump indicator illuminates for a short while, then goes out. The right plunger is retracted as far as possible.

"Plunger Set [P-SET]" P. 5-19

CE) two times.

Initial screen appears.





Δ

Press (

- 5 In order to prevent liquid from flowing out of the end of the tubing when plumbing is removed, take one of the following precautions:
 - Place the reservoir lower than the pump inlet.
 - Empty the reservoir and suction tubing of all mobile phase, and unscrew and remove the filter bushing from the pump inlet.

8.2.2 Removing Pump Head

- **1** Hold the outlet check valve using the provided wrench.
- Loosen the male nuts at both ends of SUS tubing B with the other wrench and remove SUS tubing B.

3 Loosen male nut PEEK on the inlet-side tubing and remove it from inlet check valve.













4 Using the provided Allen wrench, alternately loosen the two hex socket head bolts in the pump head and remove.

5 Remove the pump head.

Ease the pump head out gently, keeping it aligned with the plunger at all times. If it is pulled out forcefully, the plunger could be bent.



Fig. 8.5

8.2.3 Replacing Plunger Seal

Plunger seals are mounted inside the pump head. To remove the plunger seal, use the provided seal installer/remover.

Insert the seal installer/remover (flanged end) into the plunger seal.





Pull on the seal installer/remover. The plunger seal will be removed of the pump head.

After removing the seal, wipe the plunger head inner side and plunger seal outlet with clean gauze soaked in 2-propanol.

If seal material adheres to these surfaces, the sealing efficiency of the plunger cannot be maintained.



Fig. 8.7

- 3 Insert the seal installer/remover (straight end) into the new plunger seal.
- 4 Insert the new plunger seal vertically into the pump head. Then pull the installer/remover out of the seal.



Fig. 8.8

8.2.4 Installing Pump Head

1 Mount the pump head onto the head holder so that the arrow on the pump head is pointing upward.

Ease the pump head in gently, keeping it aligned with the plunger at all times. If it is pushed in forcefully, the plunger could be bent.





2 Place the two hex socket head bolts into the holes in the pump head, and screw them in alternately and uniformly with provided Allen wrench.

NOTE

Screw the bolts in alternately 90°. At the end, tighten the bolts securely with the Allen wrench.



Fig. 8.10

- 3 Screw the male nuts at the both ends of SUS tubing B into outlet check valve and the drain valve.
- 4 Screw the male nut PEEK on the inlet-side tubing into inlet check valve.
- 5 Return the reservoir or the suction filter bushing to its original position.



8.2.5 Check after Replacement

After replacing a plunger seal, check the following:

- Pumping (pressure value) is stable.
- There is no leakage from the gap between the pump head and the head holder.
- There is no leakage from the rinse flow line.

NOTE

When the phenomena above happen even after replacement of the plunger seals, the surfaces of the plunger may be scratched or nicked, in which case the plunger must be replaced.

10 "8.3 Cleaning and Inspection (Replacement) of Plungers and Diaphragms" P. 8-10

8.3 Cleaning and Inspection (Replacement) of Plungers and Diaphragms

Plungers and diaphragms are mounted inside both (left and right) pump chambers. The procedure given below is, for cleaning inspecting, and replacing of the plunger and diaphragm inside the right pump chamber (as viewed from the front of the instrument). When replacing a plunger, be sure to replace the plunger seal and the diaphragm.

Necessary parts

Part	Туре	Part No.
Plunger holder ASSY	Consumable part	S228-35281-95
Diaphragm	Consumable part	S228-32784-91

8.3.1 Removing Pump Head and Head Holder ASSY

- Remove the pump head.
 - "8.2.1 Before Removing Pump Head" P. 8-5,"8.2.2 Removing Pump Head" P. 8-6



- 2 Using the provided Allen wrench, alternately loosen the two hex socket head bolts in the pump head and remove.
- Remove the head holder.





Fig. 8.13

Ease the head holder out gently, keeping it aligned with the plunger at all times. If it is pulled out forcefully, the plunger could be bent. When the Head Holder Is Difficult to Remove

Reinsert the two hex socket head bolts into the head holder's M4 bolt holes and tighten them alternately. The force of the two hex socket head bolts pushing against the body causes the head holder to become detached.



8.3.2 Cleaning and Inspecting (Replacing) of Plungers

Using the provided box driver, rotate the plunger holder counterclockwise, and remove it.

When inserting the box driver into the plunger holder, be careful not to bend the plunger.





2 Wipe any seal material with clean gauze soaked in 2-propanol.

Seal material will interfere the sealing efficiency.



Fig. 8.16

8

Visually inspect the plunger for any scratches or nicks.

> If any scratches are found, replace the plunger holder Assy.

Plunger surfaces may be damaged by foreign matter (from the mobile phase) or buffer solution crystals inside the head holder.



Fig. 8.17

8.3.3 Inspection (Replacement) of Diaphragms

When replacing a plunger, be sure to replace the diaphragm.

- Remove the diaphragm.
- Inspect the diaphragm for scratches or deformation. If any scratches or deformation is found, replace the diaphragm.



Fig. 8.18

8.3.4 Re-Installing after Inspection



NOTE

Be sure to mount the diaphragm with the correct orientation, as shown on the right.





- 2 Holding the plunger by finger, insert it into the pump chamber.
- NOTE

Check that the diaphragm is centered properly.

3

Δ

Use the box driver to tighten the plunger holder.



Fig. 8.20



Fig. 8.21

Insert the head holder into the body.

Ease the head holder out gently, keeping it aligned with the plunger at all times. If it is pulled out forcefully, the plunger could be bent.

5 Insert the two hex socket head bolts in the head holder, and tighten them with the Allen wrench.

NOTE

Tighten the hex socket head bolts alternately, in 90° increments. At the end, tighten the bolts securely with the Allen wrench, holding the long part of the wrench like a handle.



Reinstall the pump head and the plumbing to the original state.

18.2.4 Installing Pump Head" P. 8-8



8.3.5 Check after Cleaning and Inspection (Replacement)

After cleaning and inspecting (replacing) a plunger and/or a diaphragm, use the following procedure to check the diaphragm.

- **1** Insert a syringe needle into the tip of the disposable syringe (accessory).
- 2 Attach the tubing joint and transparent tubing (cut it to about 5 cm long) (accessories) to the tip of the syringe needle, and insert it in the rinse solution outlet at the top of the head holder.
- **3** Block the rinse-solution inlet at the bottom of the head holder with your finger.





Push in the disposable syringe's plunger and release it at an appropriate position.
 If no air is leaked, the plunger will return to its original position.

If Air Is Leaked

Check the connection between the transparent tubing and the head holder and, if there are no irregularities, disassemble the components and check that the diaphragm is mounted properly.

8.4 Cleaning of Check Valves

The check valves are cleaned by pumping 2-propanol through the flow lines. The procedure is described below.



- 2 Put the suction filter into the reservoir filled with the 2-propanol.
- 3 Unscrew the male nuts from both ends of the column, and remove the column from the flow line.
- 4 Screw the male nuts from the column into the ends of a resistor tube (0.1mm I.D. × 2m length).
- 5 Turn the power switch ON. Initial screen will appear.



- 6 Press func once. The cursor blinks in the flow rate display field, prompting input of the flow rate.
- Flow rate setting screen Cursor blinks



- Press 2 and enter to set [2.0000] for the flow rate.
- Press pump.
 Pumping of 2-propanol will begin at a rate of 2mL/ min, and the pump indicator will illuminate.
 Pump for at least 1 hour.



8.5 Inspection (Replacement) and Ultrasonic Bath Cleaning of Check Valves

Necessary parts

Part	Туре	Part No.
Inlet check valve ASSY	Replacement part	S228-48249-91
Inlet check valve ASSY, 2 pieces	Replacement part	S228-48249-92
Outlet check valve ASSY	Replacement part	S228-45705-91

NOTE

Use one of the following measures to ensure that the mobile phase does not flow out of the end of the inletside tubing when the inlet-side tubing's male nut PEEK is removed.

- · Place the reservoir lower than the pump inlet.
- Empty the reservoir and tubing of all mobile phase, and unscrew and remove the filter bushing from the pump inlet.
- 1 Hold the outlet check valve using the provided wrench
- Loosen the male nuts at both ends of SUS tubing
 B using the other wrench and remove SUS tubing
 B.



g. 0.20

3 Loosen male nut PEEK on the inlet-side tubing and remove it from inlet check valve.





4 Use the provided wrench to unscrew and remove the inlet and outlet check valves.

Do not disassemble the check valves for any reason. If they are disassembled, their performance may be affected.



Fig. 8.27

- 5 Put the inlet and outlet check valves in a 2propanol bath, and clean in an ultrasonic bath for 5 minutes.
- 6 Screw the inlet and outlet check valves back into the pump head, and tighten with the wrench.
- 7 Return SUS tubing B, the inlet-side tubing, the suction filter, and the reservoir bottle to their original states.
- 8 Plug the instrument and turn the power switch ON. Initial screen appears.

8

8. Maintenance

9 To open the drain valve, turn the drain valve knob 180° counterclockwise.





Flow rate setting screen

Cursor blinks



Press 2 and enter to set [2.0000] for the [flow].

The cursor blinks in the [flow] display field,

prompting input of the flow rate.



12 Press pump.

10 Press func once.

The pump indicator illuminates, and liquid flows from the drain tubing.

If liquid flows continuously, the check valves are functioning normally.

If the liquid does not flow continuously from the drain tubing, the check valves must be replaced.



8.6 Inspection (Replacement) of Line Filter

Necessary parts

Part	Туре	Part No.
Stainless steel line filter	Consumable part	S228-35871-96

- Hold the line filter by the provided wrench.
- 2 Unscrew the male nuts on the end of the SUS tubing using another wrench, and remove the SUS tubing.



- **3** Using the wrench, unscrew and remove the line filter.
- 4 Wipe any dirt from the line filter hole with a cotton swab.



- 5 Screw the line filter back into the line filter opening, and tighten it with the open-ended wrench.
- 6 Screw the male nut on the SUS tubing back into the line filter, and tighten the nut with the openended wrench.





8. Maintenance

7 Turn the power switch ON. Initial screen appears.

- In a no load state, pump water at a rate of 1mL/ min.
 - If the pressure displayed does not exceed 0.3MPa, the line filter is functioning normally.
 - If the pressure displayed exceeds 0.3MPa, the line filter must be replaced.

v. Ø. 3	MPa	MIN	10.0 0.0
pump	prog.run	remote	G.E.

Filter is normal if this value is 0.3MPa or under.

NOTE

Void capacity

The void capacity of the line filter is about 70μ L, whereas that of the PEEK/PAT frit is about 10μ L. [Ref. PEEK frit part number: S228-48607-91 PAT frit part number: S228-32744 (Discontinued part)]

· Rinsing method to cope with ghost peaks

When using some mobile phases such as trifluoroacetic acid, ghost peaks may appear stemming from the line filter. In that case, rinse the line filter by the following procedure.

① Deliver 17% phosphoric acid-aqueous solution through the line filter at 1mL/min for 30 minutes.
 (Back pressure: 1-5MPa)

⁽²⁾ Deliver purified water through the line filter at 9mL/min for 30 minutes. (Back pressure: 1-5MPa)

Note for ion chromatography and mass analysis

When doing ion chromatography or mass analysis, this stainless steel line filter cannot be used. Use the PEEK frit (Part number: S228-48607-91) and the housing (Part number: S228-46358) instead.

- · How to replace the PEEK/PAT frit with the stainless steel line filter
 - Loosen and remove the housing of the PEEK/PAT frit.
 - Note: When the housing is removed, the frit might remain in the outlet of the solvent delivery module. In that case, take the frit out with a pair of tweezers or attach the provided syringe to the drain tubing and push out the frit by pressurizes the syringe.
 - $\ensuremath{\mathbb{Q}}$ Tighten the line filter with the provided wrench.

The filter element and the housing of the stainless steel line filter are made into one piece.

8.7 **Inspection (Replacement) and Ultrasonic Bath Cleaning of Suction Filter**

Necessary parts

Part	Туре	Part No.
Suction filter	Consumable part	S228-45707-91

- Pull the suction filter out of the suction tubing.
- 2 Clean the suction filter in a bath of 2-propanol, in an ultrasonic cleaning device for 5 minutes.



Fig. 8.32

- 3 Insert the suction tubing into the suction filter.
- Turn the power switch ON. Δ The initial screen appears on the display.
- 5 Pump water at 1mL/min for 10 minutes.
- 6 Check that air bubbles do not accumulate inside the solvent tubing. If they do, the tubing must be replaced.

1.0	000ml	MAX	10.0
0.	0MPa	MIN	0.0
pump	prog.run	remote	G.E

8.8 Replacement of Drain Valve ASSY

Necessary parts

Part	Туре	Part No.
Drain valve ASSY	Replacement part	S228-45574-95

1 Unscrew the drain valve ASSY by turning counterclockwise. And pull it in straight line.

NOTE

Confirm that there are no alien substance in the hole.



Fig. 8.33

2 Wet the seal of the new drain valve ASSY by using the 2-propanol, and insert it into the hole of the housing in a straight line.

NOTE

If the drain valve assy is inserted slantwise and forcefully, the seal can deform. As a result, the sealing of the seal cannot be maintained.

Screw the new drain valve ASSY as far as it will go to close.





4 Screw the knob to the drain valve ASSY and insert the cap into the knob, while the kob position is vertical.



Fig. 8.36

8.9 Replacement of Fuse

- Before replacing fuses, turn off the power and unplug the instrument.
- For replacement, only use fuses of the correct type and rating.
- Failure to heed the above could result in fire, electric shock or short circuits.

The correct rating of the fuses is: Necessary parts

Part	Туре	Part No.
250V 5AT (5 × 20)	Replacement part	S072-02004-23



Pull out the fuse holder by using, for example, a flat-head screwdriver.





2 Place new fuses into the fuse holder.



Fig. 8.38

3 Push the fuse holder in and fix by using a flat-head screwdriver.

8.10 Wiping of Leak Tray

The protective plate is attached on the leak tray in order to protect the leak sensor and to guide the leakage to sensor and drain outlet.

Wipe away the leakage on the leak tray completely, as below.

NOTE

When a buffer solution is used as the mobile phase, it may deposit crystals on the leak tray when it evaporates. Wipe the dirt on the leak tray with cloth soaked in water, as below.

Rotate the protective plate, to bring the leak tray and the leak sensor into sight.



Fig. 8.39

2 Wipe away the leakage around the leak sensor and on the leak tray completely.

NOTE

- Don't twist and pull the leak sensor.
- Wipe the dirt on the leak tray with cloth soaked in water.



Re-rotate the protective plate.



Make sure to re-rotate the protective plate after the wiping.
 Leak sensor may not detect leakage without the protective plate.

8

8.11 Exterior Cleaning

If the instrument cover or front panel becomes dirty, wipe it clean with a soft dry cloth or tissue paper. For persistent stains, clean the exterior using the following procedure.

- 1 Dip a piece of cloth in a dilute neutral detergent and twist firmly to remove excess liquid. Use this cloth to scrub the soiled area of the exterior surface of the instrument.
- 2 Dip a piece of cloth into water and twist firmly to remove excess liquid. Use this cloth to wipe away all the remaining detergent. Use a dry cloth to remove all moisture from the exterior surface of the instrument.

NOTE

Do not allow spilled water to remain on the instrument surface, and do not use alcohol or thinner-type solvents to clean the surfaces. These can cause rusting and discoloration.

9

Technical Information

Contents

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9.1 Installation

9.1.1 Installation Site

Suitable Sites and Preparation

To ensure safe operation, install the instrument in a suitable location that satisfies the following conditions.

🕂 WARNING

Ample ventilation

The solvents used with the HPLC system are often flammable and toxic. Therefore, the room where the instrument is installed must be well-ventilated.

- No fire sources used near the instrument
 The solvents used with the HPLC are often flammable. Therefore, the use of open flame where the instrument is installed must be strictly prohibited. Also, do not install in the same room with equipment that emits or could potentially emit sparks.
- Fire extinguishers permanently available Have fire extinguishers permanently available in case of fire.
- Protective equipment provided near the instrument
 If solvent gets into the eyes or onto the skin, it must be flushed away immediately.
 Provide equipment, such as eye wash stations and safety showers, as close to the instrument as possible.

- Avoid dust or corrosive gas
 To ensure a long service life of the instrument and preserve its performance levels, avoid installing it in places subject to large amounts of dust or corrosive gas.
- Keep away from equipment generating strong magnetic fields
 To ensure proper operation, do not install the instrument in places subject to strong magnetic fields.
 If the power supply line is subject to high electrical noise, install a surge protector.
- Install the instrument in the location that satisfies the following conditions to preserve the performance:
 - room temperature is between 4 and 35°C, with minimal temperature variation through a day.
 - air currents from heating or air conditioning equipment are not directed on the instrument.
 - sunlight does not shine directly on the instrument.
 - there is no vibration.
 - humidity stays within 20 85%.
 - place without condensation

Required Installation Space

• The weight of this instrument is 10kg. During installation, consider the entire weight combined with other LC components.

The lab table on which this instrument is installed should be strong enough to support the total weight of the LC system. It should be level, stable and have depth of at least 600mm.

If these precautions are not followed, the instrument could tip over or fall off the table.

• Keep at least 100mm between the rear of the instrument and the wall. This allows for sufficient air circulation to provide cooling and prevent the instrument from overheating and impairing the performance.

Typical system configurations and required installation spaces are shown in the figures below.



Fig. 9.1

9.1.2 Installation

Remove the Shipping Screws

In order to prevent shock during transportation, the instrument is fixed with the shipping screws. Remove these screws prior to installation.

NOTE

When the instrument is used without removing the shipping screws, system makes noise because of vibration.

Loose and remove the shipping screws (with washer).

"2.3 Right Side and Base Panel" P. 2-5

Installation

The instrument is designed for stacking with other Shimadzu HPLC components.

"9.4 Introduction to HPLC System" P. 9-47

When the LC-20A series components are stacked on each other, the clearance between the components is only 5 mm.

Use caution to avoid pinching your fingers between the components.





Stacking Brackets

The use of commercially available stacking brackets is recommended. These brackets limit the possibility of the instrument falling off the lab table during an earthquake or the like. Various grades of stacking brackets are available.

Fasten the instrument firmly in place by attaching stacking brackets to both the right and left sides. For more details, contact your Shimadzu representative.

An example of stacking bracket placement is shown in "Fig. 9.3".



Fig. 9.3

9.1.3 Power Supply Connection

Part No.	Power Supply Voltage (indicated on the instrument)	Consumption	Frequency	Rated Breaking Capacity [*]
S228-45000-31 S228-45000-41	AC100-120V(100-120V~)	150VA		
S228-45000-32 S228-45000-42	AC100-120V(100-120V~)		50/60Hz	50A
S228-45000-38 S228-45000-48 S228-45000-58	AC220-240V(220-240V~)			

The following table shows the electrical voltage, power consumption, and frequency.

* Connect the instrument to a power outlet that is equipped with a circuit breaker that shuts off the current at the described value or less.

Verify that the power outlet to be used for connection has sufficient capacity. If capacity is insufficient, a power outage or voltage drop can occur, affecting not only this instrument, but other instruments connected to the same power supply.

Connection to Power Outlet

Handle the power cord with care, and observe the following precautions to avoid cord damage, fire, electric shock or instrument malfunction.

- Do not place heavy objects on the cord.
- Keep hot items away from the cord.
- Do not modify the cord.
- Do not bend the cord excessively or pull on it.
- To unplug the instrument, pull the plug itself, NOT the cord.

If the cord is damaged, replace it immediately.





Before plugging in the instrument, make sure that the power switch is OFF.

- 1 Insert the connector side of the power cord into the power cord connector at the back of the instrument.
- 2 Insert the plug side of the power cord into the power supply outlet.



Fig. 9.5

■ Grounding

To prevent electric shock and to maintain stability in operation of the product, be sure to ground the product.

The product will be grounded when the provided 3-wired power plug is inserted into a 3-wired power socket equipped with a ground terminal.

9.1.4 Prior to Plumbing

Many different types of tubings and connectors are used to plumb the instrument at installation. It is necessary to cut tubings and mount connectors prior to the plumbing. In this section, instructions and precautions for these preparations are described.

Types of Tubings and Connectors

The tubing and connectors used for the plumbing are made of stainless steel (SUS) or resin as follows.

Stainless steel (SUS)

- Stainless steel tubing 1.6 O.D. × 0.3 I.D.
- Male nuts, 1.6 MN
- Ferrules 1.6F

- Resin
- FEP tubing, PTFE tubing, ETFE tubing, PEEK tubing, PE tubing, etc.
- Male nut PEEK
- PEEK ferrules
- PTFE ferrules

When resin tubing is used, pumping with the pressure of 20MPa or higher may cause tubing come off at the connection. Be sure to use this instrument with the pressure of 20MPa or less.

Cutting Tubings

Cut provided tubing to the proper lengths for installation.

Cutting SUS Tubings

Position the provided file (for cutting SUS tubing) diagonally against the tubing, and cut up around the tubing.

NOTE

Cut up the tubing so that the cut surface is at a right angle.





- 2 Holding the tubing at equal distances from the cutting up line, bend it up and down and from side to side to cut off.
- **3** File the cut surface to make smooth and straight.



Fig. 9.7

- Make the cut surface at right angle. Otherwise, dead volume will be created and may cause chromatographic peak broadening.
- Make sure that the inner diameter of the tubing is not deformed. Otherwise, the tubing may be clogged.

Cutting Resin Tubings

Cut off the resin tubing at a right angle using a cutter.



Fig. 9.8

■ Connecting Tubings

Mount a male nut and a ferrule to the tubing.

▲ CAUTION

Install stainless steel male nuts and ferrules on SUS tubing, and resin nuts and ferrules on resin tubing. If resin male nuts are mounted on SUS tubing, the connection can be loosened easily and leakage may occur.



The ferrule will be secured on the tubing.

- Insert the tubing completely into the opening, until it butts against the end of the opening.
 Otherwise, dead volume will be created and may cause chromatographic peak broadening.
- Do not overtighten the male nut. Otherwise, the threads will be damaged.









NOTE

Use the following as rough guide for the degree of tightening to tighten a male nut with a spanner when connecting the PEEK tubing with an SUS ferrule and a male nut.

Use the following as rough guide for the degree of tightening required to tighten the male nut with a spanner.

- 6-mm male nut: Tighten securely by hand and tighten another 120 (approx.) with a spanner.
- 8-mm male nut: Tighten securely by hand and tighten another 90 (approx.) with a spanner.

After connecting the PEEK tubing, pull the tubing to check that it does not come out.

9. Technical Information

NOTE

 For an SUS male nut: Use the open-end wrench (provided) to tighten and loosen the nut.

If the nut is to be connected to a union or other part that is not secured, use a second wrench to secure the union.

Loosen and move the male nut slightly to verify

that the ferrule is secured on the tubing.

 For a resin male nut: Tighten and loosen the nut by hand.





Moves Does not move

Fig. 9.12

Protective Plugs

Inlets and outlets of the instrument are fitted with protective plugs (bushings, stop plugs, caps and similar items) to keep out dirt and dust during shipment.

When the inlet and outlet are not connected to other thing, keep the protective plugs on. Otherwise, dirt and dust may cause clogging of the instrument.

Keep the plugs, and replace them when the instrument will be left not in use for a long time.



• For stop plugs:

Use the wrench provided to unscrew and screw in the plugs.

For resin plugs:

Remove and replace the plugs by hand.

9.1.5 Plumbing

A CAUTION

- Before plumbing, turn OFF the power supply to all the system components and unplug them.
- For plumbing, use the appropriate parts listed in "1.3 Component Parts".
- Connect only the tubing described in the instructions.

Otherwise, injury or equipment failure may cause.

The necessary plumbing is as follows:

- Plumbing of tubing for leakage Plumbing to evacuate If leaks occur in any of the instrument in a stack,

this tubing directs it down to the lowest device in the stack, and from there to a waste container.

NOTE

For details on plumbing when the optional degassing unit, low-pressure gradient unit and/or reservoir switching valve is/are used, see the instruction manual(s) for these options.



Reservoir and Waste Container Preparation

Before connecting the plumbing, prepare (a) reservoirs and (b) waste container (to receive the mobile phase after analysis and to collect leakage).

Do not use cracked or damaged bottles.

The reservoir should be made of glass and have a capacity of at least 500mL.

The waste container must be positioned lower than the instrument (for example, on the floor). If it is positioned higher than the instrument, liquid will not drain, and will leak from the connections.





Front Cover Removal

The front cover must be removed in order to perform the plumbing.



Press on the cover at the position indicated in the figure, and release to open the front cover.



Fig. 9.15



Hold the front cover upward and remove.

NOTE

The front cover can be removed at 120 degree of open angle.




Suction Filter (Accessory) Installation

NOTE

Prevent any dirt from adhering to the filter portion of the filter in installing it. Dirt on the filter could cause clogging and flow rate fluctuations.

NOTE

In order to ensure stable analysis at all times, the mobile phase should be degassed. For details of the degassing units, 1.4 Optional Parts" P. 1-5

- **1** Remove the ferrule, bushing and coil spring from the filter tubing.
- 2 Cut the tubing to a length appropriate for connecting the reservoir bottle and the pump suction port.
- **3** Pour mobile phase into the reservoir.





4 Put the filter into the reservoir, pass the tubing through the hole in the lid and through the bolt cap, place the lid into the bottle mouth, and screw the bottle cap on.

NOTE

The filter should touch the bottom of the bottle.

5 Replace the ferrule, bushing and coil spring on the tubing.



Fig. 9.18

9. Technical Information

6 Remove the shipping bushing from the pump inlet.

NOTE

Replace it if the instrument is out of use for a long time, to prevent foreign matter from entering the inlet.Retain the shipping bushing after removal.



Fig. 9.19



Fig. 9.20

Attach the ferrule and bushing on the end of the tubing into the pump inlet.

NOTE

When arranging the tubing upwards, fix it with tubing clamp.

Drain Tubing (Accessory) Installation

Remove the shipping cap from the drain tubing connection port.

NOTE

Keep the shipping cap after removal and attach it again if the instrument will not be used for a long time to prevent foreign matter from entering the port.



Fig. 9.21

- 2 Screw the male nut of the drain tubing into the drain tubing connection port.
- **3** Put the other end of the drain tubing into the waste container.

NOTE

To ensure a smooth flow of liquid, put the tubing into the bottle with its end pointing downward.



Fig. 9.22

Connecting Leakage Drain Tubing

This instrument is designed so that if leaks occur internally (except the column oven), the leaked liquid flows down to the lowest level of the instrument and is drained into the waste container.

The procedure for connecting leakage drain tubing is given below.

(Except for the waste container, all parts shown in the figure on the right are standard accessories.)

NOTE

- For connecting, cut a silicone tubing into the length in which both of the cut parts will not sag.
- Set the silicone tubing so that its edge does not touch the liquid surface in the waste container.



Fig. 9.23

9. Technical Information

Bottom of Instrument

- Insert the drain OUT, STD into leakage drain outlet from the front of the instrument.
- Turn the drain OUT, STD counterclockwise 45° to 2 secure.



Fig. 9.24

Connect one end of the silicone tubing adapter to the drain OUT, STD with a straight tubing

Straight tubing connector Silicone tubing

Fig. 9.25

- Cut the silicone tubing at the edge of the table, 4 and connect an L-Joint. Let the L-Joint head downward as in the right figure and connect the other cut part of the silicone tubing.
- Insert the other end of the silicone tubing into the 5 waste container.



Fig. 9.26

NOTE

3

connector.

To ensure a smooth flow of liquid, insert the silicone tubing into the container with its tip pointing downward.

Second Instrument from Bottom

NOTE

Leaks from the column oven are drained separately (See column oven Instruction Manual.).

If any components are installed on top of the column oven, carry out the same procedure as in "Installation on top of the column oven" on next page.

Also, when the bottom unit has no leakage hole ("Fig. 9.28"), carry out the same procedure as in "Installation on top of the column oven" on next page.

1 Insert the drain adapter into the position shown in the illustration, and slide it on the instrument of bottom.





- 2 The drain adapter connects the drain outlet to the leakage hole of the bottom unit.
- **3** Pour some water onto a spot near the drain outlet of the top unit, and verify that the water flows to the waste container.





Fig. 9.28

Installation on Top of the Column Oven

NOTE

When the bottom unit has no leakage hole ("Fig. 9.28"), carry out the same procedure described bellow.

1 Insert the drain OUT, CTO into leakage drain outlet from the front of the instrument.

2 Turn the drain OUT, CTO counterclockwise 45° to secure.





3 Connect one end of the silicone tubing adapter to the drain OUT, CTO with a straight tubing connector.



Insert the other end of the silicone tubing into the waste container.
Fig. 9.13"

NOTE

- To ensure a smooth flow of liquid, insert the silicone tubing into the container with its tip pointing downward.
- Set the silicone tubing so that its edge does not touch the liquid surface in the waste container. When the tip of tubing touches into the liquid of waste container, the drainage becomes difficult to flow.

LC-20AD

Front Cover Installation

- **1** After performing the plumbing, install the front cover using the reverse of the removal procedure.
- 2 Close the front door.



9.1.6 Installation of Manual Injector and Column

The procedures in this section covers installation of the manual injector and column installation. For similar column oven installation procedures, see the column oven instruction manual.

Installation of Manual Injector

Use the manual injectors listed below.

Option name	Part No.	Features	
Manual injector	0000 00040 04	Manual injector for general purpose analysis.	
Туре 7725	5228-32210-91	Standard sample loop: 20µL	
Manual injector		Same as type 7725, but with a position sensing switch.	
	S228-32210-93	Can send signals synchronized with injection of samples to system	
Type 77251		controller or Chromatopac.	
Semi-micro manual injector Type 8125	S228-23200-91	Manual injector for semi micro volume range.	
		Standard sample loop: 5µL.	
		Includes position sensing switch.	
		Can send signals synchronized with injection of samples to system	
		controller or Chromatopac.	
Non-metallic manual injector		Has liquid-contacting parts made of non-metallic materials. Maximum	
Туре 9725	5228-32650-91	use temperature: 60°C	
Non motallia manual inicator		Same as type 9725, but with a position sensing switch.	
	S228-32650-93	Can send signals synchronized with injection of samples to system	
		controller or Chromatopac.	

9. Technical Information

Attach the injector installation plate (option) onto the instrument with accessory screws.





2 Attach the manual injector to the installation plate with the screws attached to the injector. (For more detailed instructions, see the instruction manual for the used manual injector.)





- 3 Insert the vial holder provided with the installation plate into the vial holder opening in the plate.
- 4 Insert the vial provided with the installation plate into the vial holder.



Fig. 9.34

- Installation of Column Holder
- 1 Attach the column holder to the pump with the provided screws.
- Orient the column with its outlet at the top, and attach it to the column holder.
 The orientation of the column should be as shown in the figure on the right.
 Verify that the column is securely clamped.



Fig. 9.35

9.1.7 Flow Line Plumbing

Manual Injector Plumbing

NOTE

For connecting ports 1 to 6 of the manual injector, use the male nuts (with long bushing) and ferrules, provided as manual injector standard accessories.

- 1 Screw the sample loop male nuts (with long bushing) into ports 1 and 4 of the manual injector.
- Install a male nut (with long bushing) and ferrule to one end of each of the two waste liquid tubing sections. Then attach the tubing and ferrules into ports 5 and 6 of the manual injector. Tighten the nuts.

Back of manual injector



Fig. 9.36

9. Technical Information

- **3** Unscrew and remove the vial cap.
- 4 Route the other ends of the waste liquid tubing through the tubing opening and into the vial.



NOTE

To prevent liquid from flowing out due to the siphon effect, position the ends of the waste liquid tubing level with the needle port.





Fig. 9.38

Plumbing between Pump Unit and Manual Injector

- Cut the 1.6 O.D. × 0.3 I.D. SUS tubing (standard accessory of the pump) long enough to connect the pump outlet and port 2 of the manual injector.
- 2 Attach male nut and ferrule to both ends of the SUS tubing.
 - Pump outlet end: 1.6MN male nut and 1.6F ferrule provided as pump standard accessories.
 - Manual injector end: Male nut (long bushing) and ferrule (provided as manual injector standard accessories).
- 3 Insert the ends of the SUS tubing into the pump outlet and port 2 of the manual injector, and tighten the male nuts.



- Plumbing between Manual Injector and Column
- 1 Cut the 1.6 O.D. × 0.3 I.D. SUS tubing (standard accessory of the pump) to a length appropriate for connecting port 3 of the manual injector and the column inlet.

NOTE

In order to minimize peak broadening, cut the plumbing between the manual injector and the column as short as possible.

- Place a male nut and ferrule on both ends of the SUS tubing.
 - Manual injector end: Male nut (long bushing) and ferrule (provided as standard accessories of the manual injector)
 - Column inlet end: Male nut and ferrule (provided as standard accessories of the pump)
- **3** Unscrew and remove the stop plug from the column inlet.





4 Insert the ends of the SUS tubing into port 3 of the manual injector and the column inlet, and tighten the male nuts.



Plumbing between Column and Detector

The figure below shows the flow line from the column outlet to the detector and the waste container. Use the accessories provided with the detector to connect this tubing.

(For more detailed plumbing procedure, see the relevant sections of the detector instruction manual.)



Fig. 9.42

9.1.8 Installation and Plumbing of Mixer (Option)

A special SUS mixer with excellent solvent mixing performance is available for use with a gradient elution system.

Procedures for installing and plumbing the mixer are given below.

Preparation of Mixer

The mixer may be plumbed to accommodate three volumes; 0.5mL, 1.7mL and 2.6mL.

Select the appropriate capacity for the application. (The standard plumbing, installed at delivery, is for 2.6mL capacity.)



Remove the mixer cover.



Fig. 9.43

2 Configure the mixer tubing using the following illustrations.

NOTE

Attach caps to ports which are not used, to prevent dirt or dust entering through them.



3 The mixer assembly consists of two sections: a mixer section and a pre-mixer section.

The premixer section must be moved on the

assembly base plate before installing the mixer on

the instrument. Move and refasten the pre-mixer



Fig. 9.45



Fig. 9.46

Mounting the Mixer on the Instrument

The mixer can be installed in any of the following three positions:

· On the right side of the instrument

as shown in the figure.

· Left inside of column oven

Δ

Right inside of column oven

Installing the mixer on this instrument is described here.

For installing of the mixer on a column oven, see the column oven instruction manual.

- **1** Unscrew the cover installation screws attached on the right side of this instrument.
- 2 Install the mixer on this instrument using the unscrewed screws.



Fig. 9.47

- Mixer Plumbing (in Low-Pressure Gradient System)
- 1 Install the mixer on this instrument and remove the premixer caps from inlets A and B.





2 Screw the stop plug provided with the mixer into inlet A of the premixer.



3 Cut the two 1.6 O.D. × 0.3 I.D. SUS tubing sections provided with the mixer long enough to connect for the following:

- Pump outlet and premixer inlet
- · Mixer outlet and injector
- 4 Attach the 1.6MN male nuts and 1.6F ferrules provided with the mixer to both ends of each SUS tubing.
- 5 Attach the ends of one tubing section and ferrule into the pump outlet and premixer inlet B, and screw in the male nuts.



Fig. 9.50

6 Attach the ends of the other tubing and its ferrule into the mixer outlet and port 2 of the manual injector, and screw in the male nuts.

If an autosampler is used, see its instruction manual for procedures.



Fig. 9.51

- Mixer Plumbing (in High-Pressure Gradient System)
- Remove the premixer caps from inlets A and B.
- 2 Cut the 1.6 O.D. × 0.3 I.D. SUS tubing provided with the mixer to sections long enough for plumbing.
- 3 Attach the 1.6MN male nuts and 1.6F ferrules provided with the mixer to both ends of each SUS tubing section.
- 4 Connect the pump outlet to the premixer inlets A and B with the SUS tubing.



NOTE

For 3 mobile phases, also unscrew and remove the stop plug from inlet C, and connect inlet C to the pump 3 outlet with more SUS tubing.



5 Attach the ends of the third tubing section and ferrule into the mixer outlet and port 2 of the manual injector, and screw in the male nuts.

If an autosampler is used, see its instruction manual for procedures.



Fig. 9.54

Mixer Cover Installation

Secure the mixer cover with the screws.



Fig. 9.55

9.1.9 Wiring

🕂 WARNING

- Before performing wiring, turn OFF all components and unplug the power cables.
- Do not use any other than specified cables for wiring.
- Do not perform any other than the indicated wiring operations.
- Failure to observe the above cautions could result in fire, electric shock or instrument malfunction.

Connectors

- [PUMP PRESS] connector For connection to Chromatopac (or other integrator) or recorder. Outputs voltage proportional to pressure.
- External input/output terminals For connection to external equipment. For connection instructions, see
 "5.7 Connection to External Input/Output Terminals" P. 5-58.
- [DGU PRESS] connector. For connection to degassing unit to enter the pressure signal of vacuum pump inside of degassing unit.

Use the connectors above needed for the system. Connection instructions are provided on the following pages.



Fig. 9.56

Connecting the Optical Cable

The optical cable provided with this instrument is a two-way assembly for both transmission and reception of signals, and is connected to the [REMOTE] connector.

Instructions and precautions for connecting the optical cable are provided below.



Before connection, remove the cap from the connection channel to be used.

The caps on the [REMOTE] connectors prevent dirt or dust from getting into the connector.

If a [REMOTE] connector is not used, leave the cap on it to prevent dirt or dust from interfering with communication.

When a cap is removed, keep it in a safe place for future use.

2 Insert the optical cable plug into the [REMOTE] connector until it clicks into place.

- Make sure there is no dirt or dust on the plug. Dirt or dust on the plug will get inside the [REMOTE] connector.
- Be careful not to insert the plug across two different channels.

Failure to follow these precautions above could result in malfunction or communication problems.



Fig. 9.57



Fig. 9.58

- Do not bend the optical cable less than 35 mm in radius.
- When inserting and removing the plug, grip the plug itself, not the cable.

• Do not bend the cable where it joins the plug. Failure to follow these above precautions could result in damage to the plug or a broken wire in the cable.





Connection to System Controller

Referring to "Connecting the Optical Cable", connect the pump and system controller [REMOTE] connector with the optical cable.

NOTE

Channels between 3 and 8 of the system controller [REMOTE] connector are typically used for this purpose.

2 Plug in the pump, and turn the power switch on.



Fig. 9.60







0

3 Check the [LOCAL] and [CBM LINK] parameter and set the [ADRS] parameter.

"5.2.6 System Settings Group" P. 5-20

- [LOCAL] : Check the value is [0] (initial value). (Remote mode)
- [CBM LINK]: Check the value is [1] (initial value). (External connection)
- [ADRS] : Enter the system controller's connector channel number.

Connection to System Controller (in case of the installed CBM-20Alite)

The optional CBM-20Alite can control a maximum of 5 LC components (including this instrument). Since it is installed in this instrument, the system requires a smaller space. Refer to the CBM-20Alite instruction manual.

\Lambda WARNING

- Only Shimadzu service representative is responsible for installation of CBM-20Alite. There is a risk of electric shock or short.
- By the installation of the instrument, remove the power plug of the instrument from the power outlet. There is a risk of electric shock or short.

Check the [LOCAL] parameter is [0:Remote], and set the [CBM LINK] parameter to [0:Int] (Internal Connection).

Selecting Local/Remote Mode [LOCAL]" P. 5-20

Switching Remote Connector internal/ external [CBM LINK]" P. 5-23





NOTE

It is not necessary to connect the optical cable, because the instrument controlled by the internal-connected CBM-20Alite.

In case of internal connection, the link address [ADRS] is the fixed value [5].

Setting Link Address [ADRS]" P. 5-20



Connection of Twin Pump Units

The wiring procedure for connecting twin pump units in a high-pressure gradient system without using a system controller, is given below.

Connect the pump [REMOTE] connectors with the optical cable.

NOTE

When three pump units are used in a high-pressure gradient system (for pumping 3 mobile phases), the pump units must be connected to a system controller.



Fig. 9.61

- 2 Plug the pumps in and turn the power switches ON.
- Check the [LOCAL] and [CBM LINK] parameter, and set the [ADRS] parameter.
 [Source 15.2.6 System Settings Group" P. 5-20
 - [LOCAL] : Check that the value is [0] (Initial value). (Remote mode)
 - [CBM LINK]: Check the value is [1] (initial value). (External connection)
 - [ADRS] : Enter [85].



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¹

Connection of [DGU/SOL.V] Connector

[DGU/SOL.V] connector attaches the reservoir selector valve or degassing unit to the pump.

"1.4 Optional Parts" P. 1-5

For detailed connection instructions, see the instruction manuals for the options. The procedure given here is for connection of the reservoir select valve (FCV-11AL).

Make sure that the power switch of the instrument is OFF before connecting or disconnecting the special cable.

Otherwise, the instrument could be damaged.

- **1** Using the accessory cable for reservoir select valve, connect the reservoir select valve to this instrument's [SOL. V] connector.
- 2 Tighten the [SOL. V] connector screws with a Phillips screwdriver. The connector on the reservoir select valve is a

flat cable connector and does not use screws.

3 Plug in the instrument, and turn the power switches on.

- Set the pump [FCV TYPE] (selecting solenoid valve unit) of auxiliary function.
 If necessary, also set its [SV] (opening/closing solenoid valve unit).
 - "5.2 Parameter in Auxiliary Functions" P. 5-12
 - [FCV TYPE] : Enter [1].
 - [SV] : Set the parameter depending on a kind of selected mobile phase.







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Connection of [PUMP PRESS] Connector

[PUMP PRESS] connector connects the instrument to a Chromatopac (or other integrator) or recorder. It outputs voltage proportional to pressure. The procedure given here is for a pump connected to a Chromatopac.





- Connect the provided signal cable to the [PUMP PRESS] connector. Then connect the instrument to the Chromatopac as shown in the figure above.
- **?** Plug the pump in and turn the power switch ON.
- 3 Set the pump [RANGE] parameter. [Setting Pressure Recorder Range [RANGE]" P. 5-22

0.0 Ran	00 RA GE*5.	NGE ØMPa/	FS FS
pump	prog.run	remote	G.E
0	0	0	0

Connection of [DGU PRESS] Connector

[DGU PRESS] connector is to enter the pressure signal of vacuum pump in degassing unit. Refer to the instruction manual of degassing unit.

9.1.10 Installation of Automatic Rinsing Kit (Optional)

The automatic rinsing kit is an optional product for automatically and continually rinsing the plungers and the plunger seals attached to the back of the pump heads. The installation method is described below.

Be sure to connect the plumbing in the way described here. Incorrect plumbing may shorten the life expectancy of the diaphragms attached to the main unit.

For a Single Pump Unit



Fig. 9.64

Insert tubing joint (accessory) into the rinsesolution outlet above the head holder.





- 2 Cut the rinse tubing assembly (accessory) in the way shown on the right with a cutter and insert it into the rinse-solution inlet below the head holder.
- 3 Cut the transparent tubing provided with the automatic rinsing kit to the required length in the way described in the automatic rinsing kit's instruction manual, and attach it by inserting it in the way shown on the right.









- 4 Prepare the small bottle provided with automatic rinsing kit filled with distilled water about four-fifths and close the cap.
- **5** Insert the two pieces of tubing into the top of the small bottle in the way shown on the right, and set the bottle in the instrument.



Do not pull the rinse tubing connected to the pump heads or any of the tubing of the automatic rinsing kit with excessive force. The tubing may come loose if it is pulled with excessive force.



■ For Twin Pump Units

Cut the transparent tubing (1,500mm) provided with the automatic rinsing kit to an appropriate length and connect it in the way shown below.



Fig. 9.69

9.2 Specifications

9.2.1 LC-20AD Pump Unit

Item		Specification		
Pump Type		Micro-volume double plunger pump (approx. 10µL/stroke)		
Pumping Methods		Constant flow delivery and constant pressure delivery		
	Flow Rate Setting Range	0.0001-5mL/min (1.0-40MPa) 5.0001-10mL/min (1.0-20MPa)		
Constant Flow Flow Rate Pumping Accuracy Flow Rate Precision		± 1% or ± 2μL/min, whichever is the greater. (0.01-2mL/min) ± 2% (2-5mL/min) (when water is pumped at 1-40MPa, with water and room temperature constant between 20-30 °C.)		
		Less than 0.06%RSD or 0.02minSD whichever is the greater.		
Constant Pressure	Pressure Setting Range	1.0-40MPa (0.1MPa steps)		
Pumping Pressure Accuracy		± 10% or 1.0MPa, whichever is the greater.		
Pressure limits function		Upper and lower limits		
Liquid-contacting part materials		SUS316L, PEEK, PTFE, ruby, sapphire, HastelloyC		
Suction Filter		10µm		
Line Filter		5μm mesh, capacity 70μL		
Time Program		Commands for flow rate, pressure, [EVENT] functions, [LOOP] (for program repetition), 10 files, total 320 steps		
Pressure Display Accuracy		\pm 2% or \pm 0.5MPa, whichever is the greater.		
Plunger Rinsing Line		Connecting the optional automatic rinsing kit enables automatic rinsing.		
Dimensions		W 260 × H 140 × D 420mm, excluding protruding parts		
Mass		10kg		
Operating Temperature Range		4-35 °C		

Item	Specification				
	Part No.	Power Supply Voltage (indicated on the instrument)	Power Consumption	Frequency	Rated Breaking Capacity [*]
Power Supply	S228-45000-31 S228-45000-41	AC100-120V (100-120V~)			
	S228-45000-32 S228-45000-42	AC100-120V (100-120V~)	150VA	50/60Hz	50A
	S228-45000-38 S228-45000-48 S228-45000-58	AC220-240V (220-240V~)			
	* Connect the instrument to a power outlet that is equipped with a circuit breaker that shuts off the current at the described value or less.				
Installation Environment (IEC)	Installation Category II Pollution Degree 2 Altitude 2000 m or lower Install inside the room.				

9.2.2 High-Pressure Gradient System

■ High-Pressure Gradient System controlled by System Controller (CBM-20A)

Item	Specification
Number of solvents mixed	2 or 3
Gradient profile	Step, linear and exponential functions (only when LC workstation controls) possible at multiple levels
Maximum program steps	Max. 400 steps per program. 20 programs files
Program duration	0.01-9999.9 minutes (in 0.01 minutes steps)
Mix ratio setting range	0-100% (in 0.1% steps)
Concentration accuracy	± 0.5% (at 0.2-2mL/min) ± 1.0% (at 2-5mL/min) (for binary gradient with water/caffeine solution, at 1.0-40MPa)
Flow rates possible	0.0001-10mL/min

■ High-Pressure Gradient System controlled by Pump Unit (LC-20AD)

Item	Specification
Number of solvents mixed	2
Gradient profile	Step and linear possible at multiple levels
Maximum program steps	Max. 320 steps per program. 10 program files.
Program duration	0.01-999.99 minutes (in 0.01 minutes steps)
Mix ratio setting range	0-100% (in 0.1% steps)
Concentration accuracy	± 0.5% (at 0.2-2mL/min) ± 1.0% (at 2-5mL/min) (for binary gradient with water/caffeine solution, at 1.0-40MPa)
Flow rates possible	0.0001-10mL/min

9.2.3 Low-Pressure Gradient System

■ Low-Pressure Gradient System controlled by System Controller (CBM-20A)

Item	Specification
Number of solvents mixed	Max. 4
Gradient profile	Step, linear and exponential functions (only when LC workstation controls) possible at multiple levels
Maximum program steps	Max. 400 steps per program. 20 program files.
Program duration	0.01-9999.9 minutes (in 0.01 minutes steps)
Mix ratio setting range	0-100% (in 0.1% steps)
Concentration accuracy	± 1% (for binary gradient with water/caffeine solution, at 0.1-2mL/min, 1-20MPa)
Flow rates possible	0.0001-2mL/min (rate over 2mL/min possible, but will shorten life span of low-pressure gradient unit's solvent valves)

■ Low-Pressure Gradient System controlled by Pump Unit (LC-20AD)

Item	Specification
Number of solvent mixed	Max. 4
Gradient profile	Step and linear possible at multiple levels
Maximum program steps	Max. 320 steps per program. 10 program files
Program duration	0.01-999.99 minutes (in 0.01 minutes steps)
Mix ration setting range	0-100% (in 0.1% steps)
Concentration accuracy	± 1% (for binary gradient with water/caffeine solution, at 0.1-2mL/min, 1-20MPa)
Flow rate possible	0.0001-2mL/min (rate over 2mL/min possible, but will shorten life span of low-pressure gradient unit's solvent valves)

9.3 Maintenance Parts

9.3.1 Consumable Parts

Part	Part No.	Remark
Plunger seal	S228-35146	For pump head
Stainless steel line filter	S228-35871-96	Lines filter's frit
Suction filter	S228-45707-91	Filter body only (cleaned)

9.3.2 Replacement Parts

Mechanical Parts

Part	Part No.	Remark
Plunger holder ASSY	S228-35281-95	
Plunger ASSY	S228-35601-93	
Holder ASSY	S228-35602-91	
Inlet check valve ASSY	S228-48249-91	
Inlet check valve ASSY, 2 pieces	S228-48249-92	
Outlet check valve ASSY	S228-45705-91	
Pump head	S228-34541	
Head holder	S228-38022	
Diaphragm, 2 pieces	S228-32784-91	
Suction filter ASSY	S228-45708-91	
Bush	S228-39084	For suction filter
Ferrule 3.0F-T	S228-12493	For suction filter
FEP tubing 3 × 1.5	S670-10321-05	For suction filter
Drain tubing ASSY	S228-25495-93	
Tubing joint	S228-37146-01	
Transparent tubing (1 m)	S228-42203	
ETFE tubing 1.6 × 0.8	S228-18495-01	For use between the inlet block and the check valve
Male nut, 1.6MN PEEK	S228-35403	For plumbing between the inlet block and the check valve
Inlet block	S228-47518	Pump inlet
Pressure sensor ASSY, 20AD	S228-45552-98	
Drain valve ASSY	S228-45574-95	

9. Technical Information

Part	Part No.	Remark	
Right panel ASSY, 20AD	S228-46513-94	Front cover	
Thrust	S228-34469		
SUS tubing A	S228-45590-91	For left head	
SUS tubing B	S228-45590-92	For right head	
Panel 20AD	S228-42208	For mounting the inlet block	

Electrical Parts

Part	Part No.	Remark	
Fuse 5AT, 250V	S072-02004-23		
Switch ASSY	S228-42287-91		
Power supply LEA75F-24	S074-80426-61		
PCB LC-20AD ASSY	S228-55130-45	Without EEPROM	
PCB sensor ASSY	S228-35842-92		
Left panel 20AD ASSY	S228-43200-41	Display panel and keypad assembly	
Leak sensor ASSY	S228-39247-95		
Fan for SPD60 LC-10A	S228-25246-91		

9.3.3 Maintenance Kit

A set of consumable parts and plumbing parts is provided as Maintenance Kit ASSY.

■ Maintenance Kit for LC-20AD (Part No.S228-45593-91)

Part	Part No.	Q'ty	Remark
Plunger seal	S228-35146	2	
Stainless steel line filter	S228-35871-96	1	
Suction filter ASSY	S228-45708-91	1	
Plunger holder ASSY	S228-35281-95	1	
Ferrule, 1.6F	S228-16000-10	4	
Male nut, 1.6MN	S228-16001	4	
Male nut, PEEK	S228-18565	4	
SUS pipe 1.6mm O.D. × 0.3mm I.D.	S228-50579-91	1	2m

9.4 Introduction to HPLC System

The Prominence LC (LC-20A) series components are for use with Shimadzu high performance liquid chromatography (HPLC) systems, which are designed to provide high accuracy and high sensitivity analyses. Example system configurations are provided below, along with descriptions of the operations of the various components.

9.4.1 Example of a Simple (Isocratic) System

Each component of the system is controlled locally. This is a simple system composed of the minimum number of components for stable analysis.



- •Mobile phase is drawn out of the reservoir bottle and pumped through the tubing by the pump.
- The degassing unit removes dissolved air from the mobile phase, preventing air bubbles and consequent rise, drift or other baseline irregularities caused by dissolved air.
- The pump sends the mobile phase through the manual injector, column and detector, in that order, and finally into the waste container.
- Samples are injected into the system by the manual injector, with a syringe.
- In the column, the components are separated by means of the mutual interactions of the mobile phase and the column packing (stationary phase).
- The detector detects the components eluted from the column, and sends the signal data to a Chromatopac or PC.
- Mobile phase from the detector drains into the waste container.

9.4.2 Example of Autosampler System (1)

Centralized control of all the components by a CBM-20Alite system controller enhances ease operation and is well suited for automated analyses. The CBM-20Alite can control a maximum of 5 LC components. Since it is installed in the pump unit or autosampler, the system requires a smaller space.

Solvent Flow
Reservoir bottle
Degassing Unit
Low-pressure gradient unit*
Low-pressure
Gradient unit*
Mixer
Mixer
Autosampler
Column

Detector

Waste container

8

(9





- Mobile phase is drawn out of the reservoir bottles and pumped through the tubing by the pump.
- The degassing unit removes dissolved air from the mobile phase, preventing air bubbles and consequent rise, drift or other baseline irregularities caused by dissolved air.
- The low-pressure gradient unit mixes up to 4 mobile phases that have been degassed by the degassing unit. (*This item is necessary for a low-pressure gradient system.)
- The pump sends the mobile phase through the autosampler, column and detector, in that order, and finally into the waste container.
- The mixer enhances the mixing efficiency of the mobile phases. This item is required for low or highpressure gradient system.
- The autosampler automatically injects the sample into the flow lines. By adding a rack changer, it is possible to automatically change the autosampler racks.
- In the column, the components are separated by means of the mutual interactions of the mobile phase and the column packing (stationary phase).
- The detector detects the components separated in the column, and sends the signal data to a Chromatopac or PC.

Mobile phase from the detector drains into the waste container.
9.4.3 Example of Autosampler System (2)

The CBM-20A system controller can control a maximum of 8 LC components (12 LC components as an option).

Use the same type of pumps for high-pressure gradient system.

■ Solvent Flow ■ Function of Components





- Mobile phase is drawn out of the reservoir bottles and pumped through the tubing by the pump.
- The degassing unit removes dissolved air from the mobile phase, preventing air bubbles and consequent rise, drift or other baseline irregularities caused by dissolved air.
- The pump sends the mobile phase through the autosampler, column and detector, in that order, and finally into the waste container.
- **4** The mixer enhances mixing efficiency of the mobile phases.
- The autosampler automatically injects the sample into the flow lines. By adding a rack changer, it is possible to automatically change the autosampler racks.
- In the column, the components are separated by means of the mutual interactions of the mobile phase and the column packing (stationary phase).
- The detector detects the components eluted from the column, and sends the signal data to a Chromatopac or PC.
- 3 Mobile phase from the detector drains into the waste container.
- The CBM-20A system controller can control a maximum of 8 LC components (12 LC components as an option) including a maximum of 4 pump units.

9.5 Mobile Phase Characteristics

	(1) Solvent (*)η≤ 0.5cp, B.P. > 45°C (**)η≤ 0.5cp, B.P. < 45°C	(2) Source	(3) UV Cutoff	(4) R.I.25°	Boiling Point (°C)	Viscosity (cP,25°C)	(5) p'	(6) e°a	(7) Water Solubility % ^W in ^{20°C} Solvent	(8) Dielectric Constant e ²⁰	(9) p'+ 0.25e
1	FC-78 (*) FC-75 (Fluorescent solvent) FC-43	(LC specific)	210nm 210 (opaque under 210)	1.267 1.276 1.291	50 102 174	0.4 0.8 2.6	< -2 < -2 < -2	25 25 25		1.88 1.86 1.9	p' and Dielect. const. (Function proportional to strength)
2	Isooctane(*)(2,2,4- tri methylpentane)	LC	197	1.389	99	0.47	0.1	0.01	0.011	1.94	0.1
3	n-Heptane(*)	LC	195	1.385	98	0.40	0.2	0.01	0.010	1.92	0.5
4	n-Hexane(*)	LC	190	1.372	69	0.30	0.1	0.01	0.010	1.88	0.5
5	n-Pentane(**)	LC	195	1.355	36	0.22	0.0	0.00	0.010	1.84	0.5
6	Cyclohexane	LC	200	1.423	81	0.90	-0.2	0.04	0.012	2.02	0.5
7	Cyclopentane(*)	LC	200	1.404	49	0.42	-0.2	0.05	0.014	1.97	0.6
8	I-Chlorobutane(*)	LC	220	1.400	78	0.42	1.0	0.26		7.4	2.8
9	Carbon disulfide	LC	380	1.624	46	0.34	0.3	0.15	0.005	2.64	1.7
10	2-Chloropropane(**)	LC	230	1.375	36	0.30	1.2	0.29		9.82	3.7
11	Carbon tetrachloride	LC	265	1.457	77	0.90	1.6	0.18	0.008	2.24	2.3
12	n-Butyl ether		220	1.397	142	0.64	2.1	0.25	0.19	2.8	2.4
13	Triethylamine			1.398	89	0.36	1.9	0.54		2.4	2.4
14	Bromoethane(*)			1.421	38	0.38	2.0	0.35		9.4	4.3
15	i-Propyl ether(*)		220	1.365	68	0.38	2.4	0.28	0.62	3.9	3.2
16	Toluene	LC	285	1.494	110	0.55	2.4	0.29	0.046	2.4	2.9
17	p-Xylene		290	1.493	138	0.60	2.5	0.26		2.3	3.0
18	Chlorobenzene			1.521	132	0.75	2.7	0.30		5.6	4.1
19	Bromobenzene			1.557	156	1.04	2.7	0.32		5.4	4.1
20	lodobenzene						2.8	0.35			
21	Phenyl ether			1.580	258	3.3	3.4			3.7	3.7
22	Phenetole			1.505	170	1.14	3.3			4.2	4.9
23	Ethyl ether(**)	LC	218	1.350	35	0.24	2.8	0.38	1.3	4.3	4.0
24	Benzene	LC	280	1.498	80	0.60	2.7	0.32	0.058	2.3	3.6
25	Tricresy phosphate										
26	Ethyl iodide			1.510	72	0.57	2.2			7.8	4.2
27	n-Octanol		205	1.427	195	7.3	3.4	0.5	3.9	10.3	5.8
28	Fluorobenzene			1.46	85	0.55	3.1			5.4	4.6
29	Benzylether			1.538	288	4.5	4.1				
30	Methylene chloride(**)	LC	233	1.421	40	0.41	3.1	0.42	0.17	8.9	5.6
31	Anisole			1.514	154	0.9	3.8			4.3	4.6
32	i-Pentanol			1.405	130	3.5	3.7	0.61	9.2	14.7	7.3
33	1,2-Dichloroethane	LC	228	1.442	83	0.78	3.5	0.44	0.16	10.4	6.3

	(1) Solvent (*)η≤ 0.5cp, B.P. > 45°C (**)η≤ 0.5cp, B.P. < 45°C	(2) Source	(3) UV Cutoff	(4) R.I.25°	Boiling Point (°C)	Viscosity (cP,25°C)	(5) p'	(6) e°a	(7) Water Solubility % ^W in ^{20°C} Solvent	(8) Dielectric Constant e ²⁰	(9) p'+ 0.25e
34	t-Butanol			1.385	82	3.6	4.1	0.7	miscible	12.5	
35	n-Butanol	LC	210	1.397	118	2.6	3.9	0.7	20.1	17.5	8.3
36	n-Propanol	LC	240	1.385	97	1.9	4.0	0.82	miscible	20.3	
37	Tetrahydrofuran(*)	LC	212	1.405	66	0.46	4.0	0.57	miscible	7.6	
38	Propylamine(*)			1.385	48	0.35	4.2		miscible	5.3	
39	Ethylacetate(*)	LC	256	1.370	77	0.43	4.4	0.58	8.8	6.0	5.8
40	i-Propanol	LC	205	1.384	82	1.9	3.9	0.82	miscible	20.3	
41	Chloroform(*)	LC	245	1.443	61	0.53	4.1	0.40	0.072	4.8	5.6
42	Acetophenone			1.532	202	1.64	4.8			17.4	8.7
43	Methylethyl	LC	329	1.376	80	0.38	4.7	0.51	23.4	18.3	9.1
44	Cyclohexanone			1.450	156	2.0	4.7			18.3	9.1
45	Nitrobenzene			1.550	211	1.8	4.4			34.8	13.2
46	Benzonitrile			1.536	191	1.2	4.8			25.2	10.9
47	Dioxane	LC	215	1.420	101	1.2	4.8		miscible	2.2	
48	Tetramethyl urea	LC	265	1.449	175		6.0	0.56		23.0	10.7
49	Quinoline			1.625	237	3.4	5.0			9.0	7.4
50	Pyridine			1.507	115	0.88	5.3		miscible	12.4	
51	Nitroethane		380	1.390	114	0.64	5.2		0.9		
52	Acetone(*) Benzyl alcohol	LC	330	1.356 1.538	56 205	0.30 5.5	5.1 5.7	0.71	miscible	13.1	8.8
53	Tetramethyl guanidine						6.1	0.6			
54	Methoxyethanol	LC	210	1.400	125	1.60	5.5		miscible	19.9	
55	Tris(cyanoethoxy) propane	GC					6.6	0.56			
56	Propylene carbonate	LC					6.1				
57	Ethanol	LC	210	1.359	78	10.8	4.3		miscible	24.6	
58	Oxydipropionitrile	GC					6.8				
59	Aniline			1.584	184	3.77	6.3			6.9	8.1
60	Acetic acid			1.370	118	1.1	6.0		miscible	6.2	
61	Acetonitrile(*)	LC	190	1.341	82	0.34	5.8		miscible	37.5	
62	N,N-dimethylaceta-mide	LC	268	1.436	166	0.78	6.5	0.88		37.8	
63	Dimethylformamide	LC	268	1.428	153	0.80	6.4			36.7	
64	Dimethylsulfoxide	LC	268	1.477	189	2.00	7.2	0.62	miscible	4.7	
65	N-methyl-2-pyrolidone	LC	285	1.468	202	1.67	6.7			32	
66	Hexamethyl phosphoric acid triamide			1.457	233	3	7.4	0.65		30	
67	Methanol(*)	LC	205	1.326	65	0.54	5.1		miscible	32.7	
68	Nitromethane		380	1.380	101	0.61	6.0		2.1		
69	m-Cresol			1.540	202	14	7.4			11.8	10.0
70	N-methylformamide			1.447	182	1.65	6.0		miscible	182	

	(1) Solvent (*)η≤ 0.5cp, B.P. > 45°C (**)η≤ 0.5cp, B.P. < 45°C	(2) Source	(3) UV Cutoff	(4) R.I.25°	Boiling Point (°C)	Viscosity (cP,25°C)	(5) p'	(6) e°a	(7) Water Solubility % ^W in ^{20°C} Solvent	(8) Dielectric Constant e ²⁰	(9) p'+ 0.25e
71	Ethylene glycol			1.431	182	16.5	6.9		miscible	37.7	
72	Formamide			1.447	210	3.3	9.6		miscible	111	
73	Water	LC		1.333	100	0.89	10.2			80	

(1) An asterisk (*) indicates solvents most suitable for LC, with low boiling points (>45 °C) and low viscosity (<0.5cp).

Double asterisks (**) indicates solvents with a very low viscosity and boiling point.

(2) "LC" indicates that a grade of solvent specifically for LC is commercially available from companies like the following:

Burdick & Jackson, Baker Chemical, Mallinckrodt Chemical, Fischer Scientific, Waters Associate, Manufacturing Chemists. Inc.

"GC" indicates that a 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 stationary phase in liquid-to-liquid LC.)

- (3) The wavelength below which the solvent becomes opaque.
- (4) Refractive index at 25°C.
- (5) Polarity parameter of solvent.
- (6) Solvent's strength parameter in relation to liquid-to-solid adsorption in alumina.
- (7) Water solubility (%W) at 20°C of solvent used in liquid-to -solid adsorption.
- (8) Value at 20°C.
- (9) Function consisting of p' (proportional to solvent strength) plus the dielectric constant, in ion chromatography.
- Source: A.M.Krstulovic and P.R.Brown, *Reversed-Phase High-Performance Liquid Chromatography,* Wiley Interscience, 1982.

9.5.1 Maximum Pressure Limit Setting for Gradient Elution Method

The Shimadzu solvent delivery modules allow the setting of the maximum pressure limit (P.Max) at which pumping will automatically stop for the protection of the system or the column if the pressure rises abnormally due to clogging or other problems.

With the gradient elution method, if mobile phases of different viscosities, such as water and methanol, are mixed together, the pumping pressure will vary according to the mobile phase composition.

This section explains the maximum pressure limit (P.Max) setting, taking examples of water (buffer solution) / acetonitrile and water (buffer solution) / methanol mobile phases, which are typically used in reversed phase chromatography.



Relation between Pressure and Organic Solvent Concentration

The graph above shows two pressure curves relative to the pressure at 100% water, as the acetonitrile and methanol concentrations change. From this graph, the maximum pressure is given at around 20% acetonitrile and around 50% methanol, which is 1.1 times and 1.6 times higher than that of 100% water, respectively. Consequently, for the gradient elution method, it is necessary to set the maximum pressure limit (P.Max) based on the maximum pumping pressure during gradient elution at the specific mobile phase composition. Conversely, when developing analytical conditions near the maximum pressure that the system or the column can withstand, determine the flow rate of the mobile phase, considering the maximum pumping pressure during gradient elution.

NOTE

- The graph above shows pressure changes at 40 °C. The relative pressure values vary depending on the temperature.
- The maximum withstand pressure varies depending on the system and the column being used.

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