228-10832A

Solvent Delivery Module LC-6A for The Shimadzu High Performance Liquid Chromatograph Service Manual

SHIMADZU CORPORATION

ANALYTICAL INSTRUMENTS DIVISION

κύοτο, μάραν

Contents

1.	Introduction	1
2.	Specifications	2
3.	Construction and Function	3
3	3.1 Front panel	3
3	3.2 Rear panel	5
3	3.3 Left side panel	6
3	3.4 Flow line	7
3	3.5 Solvent delivery pump main unit	9
3	3.6 Electrical system	11
	3.6.1 Checking connector	11
	3.6.2 Relation between deformed cam rotating angle and pulse	14
	3.6.3 Usage of the REMOTE connector	15
	3.6.4 Circuit diagram	19
4.	Maintenance	22
4	.1 Inspection of clogging and stains and cleaning of LC flow line	22
4	.2 Replacement of plunger seal	23
4	.3 Replacement of line filter	25
4	.4 Zero point adjustment of pressure sensor	25
4	.5 Lubrication of the moving parts of the pump	25
4	.6 Inspection of check valve	26
5.	Troubleshooting	28
5	.1 General troubleshooting	28
	5.1.1 Large drift and noise are generated on the base line	28
	5.1.2 Even when samples are charged, no peak is produced	29
	5.1.3 Peak retention time changes, abnormally short or abnormally long	30
5	.2 Solvent delivery flow rate is unstable, and solvent delivery flow pulsation	
	is noticeable	31
	5.2.1 Checking method	31
	5.2.2 Cause and remedy	31
5	.3 No pump performs high-speed suction operation	34

Flow rate at no load does not fall within the specification with respect to		
the present value	35	
Abnormal noise is generated from one pump	36	
Sample symptoms for pressure base	36	
Checking circuit	38	
	Flow rate at no load does not fall within the specification with respect tothe present valueAbnormal noise is generated from one pumpSample symptoms for pressure baseChecking circuit	

1. Introduction

This Service Manual is intended for users operating this equipment, and describes the maintenance method for Type LC-6A solvent delivery module for Shimadzu high performance liquid chromatograph.

Explanation is given mainly of a general troubleshooting for malfunctions which are apt to take place during operation, maintenance and check points for routine work and detailed construction of this equipment.

Regarding electronic circuits such as printed circuit boards, this manual merely shows the circuitry explanation and circuit diagram, no explanation is given if elements such as IC's and transistors. When performing operation, use this manual together with the instruction manual. With due care taken for installation hints for equipment operation and operating precautions, you are required to carry out adequate and more effective maintenance of this equipment.

2. Specifications

1.	Pump	:	Single small plunger reciprocating pump of constant	
			discharging and quick suction type (100µl/stroke)	
2.	Mode of delivery	:	Constant flow or constant pressure delivery	
(1	Constant flow delivery			
	• Range of flow rate		$0.1 \sim 5 \mathrm{m}\ell/\mathrm{min.} (10 \sim 500 \mathrm{kgf/cm^2})$	
			$0.1 \sim 9.9 \mathrm{m}\ell/\mathrm{min.} (10 \sim 250 \mathrm{kgf/cm^2})$	
	• Accuracy of flow rate setting	:	Within $\pm 2\%$ or 20μ l/min., which is higher (0.1 $\sim 5m$ l	
			min.)	
	• Stability of flow rate	:	Within ±0.5% (within the $0.1 \simeq 5 \mathrm{m}\ell/\mathrm{min.}$ range)	
2	Constant pressure feeding			
	• Pressure setting range	:	$10 \simeq 500 \text{kgf/cm}^2$ (at 10kgf/cm^2 intervals)	
	• Accuracy of pressure setting	:	Within $\pm 20\%$ of the setting or $\pm 25 \text{kgf/cm}^2$, whichever is	
			smaller	
3.	Pressure limit setting			
	• High pressure limit	:	$0 \simeq 500 \text{kgf/cm}^2$ continuously variable	
	• Low pressure limit		OFF, $0 \simeq 300$ kgf/cm ² continuously variable	
4.	4. Pressure indicating range		$0 \simeq 500 \text{kgf/cm}^2$	
5.	Dampener	:	Flow-through type with interval capacity of 1300μ l	
6.	Materials in contact with solvent	:	SUS316, Hastelloy C, ruby, sapphire, Kel-F, Teflon	
			tempered with graphite	
7.	Suction filter	:	5µm mesh	
8.	Line filter	:	2μm mesh	
9.	Optimum operating temperature	:	$10 \sim 40^{\circ} C$	
10.	Size	:	260W × 160H × 400D mm	
11.	Weight	:	About 15kg	
12.	Power requirements (Power requi	reme	ents vary depending upon the line voltage.)	
	P/N 228-14000-91	AC	100V ± 10V 1.5A 50, 60Hz	
	P/N 228-14000-92	AC	$115V \pm 10V$ 1.5A 50, 60Hz	
	P/N 228-14000-93	AC	$220V \pm 20V$ 0.8A 50, 60Hz	
	P/N 228-14000-94	AC	$240V \pm 20V$ 0.8A 50, 60Hz	
	P/N 228-14000-95	AC	$230V \pm 20V$ 0.8A 50, 60Hz	

3. Construction and Function

3.1 Front panel



Fig. 3.1 Front Panel

No.	Name of Part	Function
1	Power indication lamp	Lights when the power switch is turned ON.
2	Pump operation indication lamp	Lights when pump switch is ON.
3	Constant flow mode indication lamp	Lights when pump feeds liquid in constant flow delivery mode.
4	Constant pressure mode indication lamp	Lights when the pump feeds solvents in the constant pressure solvent delivery mode.
5	Upper limit pressure indication lamp	Lights when the delivery pressure of the pump exceeds the upper limit pressure setting.
6	Lower limit pressure indication lamp	Lights when the delivery pressure of the pump drops below the lower limit pressure setting.
7	Pump monitor lamp	Lights when the pump plunger retreats. For replacement of the seal, stop the pump when the lamp is lit.
8	Pressure indicating meter	Indicates discharging pressure of the solvent delivery pump, with graduations of 20kgf/cm ² . Error tolerance is ±12.5kgf/cm ² of set value.

No.	Name of Part	Function
9	Zero adjustment screw for the pressure indicating meter	Turn the screw with a small regular screwdriver so that the pressure indicating meter points to zero when power switch is turned OFF.
10	Flow-pressure setting switch	Sets the flow rate for the constant flow solvent delivery mode and the pressure for the constant pressure solvent de- livery mode. Flow rate can be set at intervals of $0.1m^2/min$. and pressure at intervals of 10kgf/cm^2 .
11	Upper limit pressure setting switch	Set the upper limit of the discharging pressure of the pump.
12	Lower limit pressure setting switch	Set the lower limit of the discharging pressure of the pump. The switch must not be turned ON before actuating the pump. The pressure should be set after the pump is actuated and the system pressure has stabilized.
13	Solvent delivery mode selection switch	Release the switch for delivery mode. Depress it for constant pressure delivery mode. (Note) Before selecting the mode by this switch, be sure to turn OFF the pump switch.
14	Pump ON/OFF switch	 Depress this switch to turn the pump ON and OFF: the pump stops if the switch is depressed while the pump operates, and it starts operation if the switch is depressed when the pump is OFF. The pump LED turns ON and OFF accordingly. If the pressure limiter (upper limit) has been actuated, depress the switch to release the limiter.
15	Pressure sensor	Flow-through type sensor for detecting the delivery pressure of the pump.
16	Zero point adjustment trimmer for the pressure sensor	Turn the trimmer with a small screw driver so that the pressure indicating meter (8) points to zero when the power switch is turned ON, and the unit is not pumping.
17	Dampener	Eliminates fluctuations of the pump flow.
18	Drain valve	When the knob is turned counterclockwise (loosened), solvent fed from the pump flows through the drain tubing (21) and to the outlet (22). Therefore, if the outlet (22) is connected to an injector and a column, most of the liquid discharged from the pump flows to the drain tubing (21). When the knob is turned clockwise (tightened), liquid flows to outlet (22) alone.
19	Line filter	The filter is mounted in the liquid flow line.
20	Solvent delivery pump	
21	Drain tubing	Refer to (18) above.
22	Outlet	The tubing leading to the injector is usually connected here.



Fig. 3.2 Rear Panel

No.	Name of Part	Function	
1	Fan	Fan for cooling equipment interior (Caution) Cooling air is blown out from the equipment rear. Do not place obstacle in front of the air outlet.	
2	Grounding terminal	Be sure to use this terminal for grounding the unit when the power outlet is of two-wire type.	
3	Fuse	3A fuse for AC 100V or 115V 1.5A fuse for AC 220V	
4	Power cord connector	AC power input terminal for the equipment. Insert the supplied AC power connector into this connector.	
5	REMOTE connector	Connector for remote control by the System Controller SCL-6A (If not connected to SCL-6A, be sure to insert the shortcircuiting connector.) Please refer to Section 6.5 for other uses of this connector.	

3.3 Left side panel



Fig. 3.3 Left Side Panel of Equipment

No.	Name of Part	Function
1	Power switch	Throw the switch toward the front panel to turn power ON or toward the rear panel to turn power OFF.

3.4 Flow line



Fig. 3.4 Flow Line

No,	Name of Part	Part No.	Remarks	
1	Suction filter ASSY (including (3), (4) and (5))	228-12491-91		
2	Teflon tubing 2.0mm × 3.0mm	228-12373	0.9m	
3	Coil spring	228-12654	1	
4	Bushing 3.0mm with knurl	228-12492		
5	Ferrale 3.0 F-T	228-12493		
6	Check valve in assembly	228-12353-91		
7	Pump head LC-6A	228-12901		
8	Check valve out of assembly	228-09054-93		
9	Parts for tubing LC-6A ASSY	228-15510-91	Including (16) and (17) , each 2 pcs.	
10	Pressure sensor ASSY	228-14041-91		
11	Dampener ASSY	228-13622-92		
12	Drain valve ASSY	228-13942-93	Including (13). For internal structure, refer to Fig. 5.2	
13	Line filter ASSY	228-12642-93	-	
14	Lurelock ASSY	228-14083-91	Including (15).	
15	Teflon tubing 1.0mm × 2.0mm	016-37592	0.5m	
16	Male nut 1.6MN	228-16001	1	
17	Ferrule 1.6F	228-16000		

3.6 Electrical system

3.6.1 Checking connector

With the LC-6A, each circuit is checked, using connector JG (15-pin connector) on PC board PB-1 ass'y.

The connector inserted into connector JG is:

Connector PCN6-153-2.5E P/N 070-50441

Refer to 3.7 circuit diagram.

Connector Pin No.	Normal Voltage Value/Remarks				
1	+10V power source (+9.9 ~ +10.1V)				
2	-10V power source (-9.98 ~ - 10.02V) * Adjusted with trimmer R53: 5k Ω				
3	- 15V power source (- 14.3 ~ - 15.7V)				
4	0V * Voltage is measured with this pin as a reference.				
5	+15V power source (+14.3 ~ +15.7V)				
6	0 to $+5V (10mV/kg \cdot J/cm^2)$ Pressure sensor output signal is applied to this pin. When pressure is $500kgJ/cm^2$ as aforementioned, output voltage becomes $+5V$. With the LC-6A, two types of voltages are generated from the pressure sensor. The signal applied to this pin is quick in response (expressed as Vp in circuit diagram), which is used for compressivity correction, pressure recorder's recording, motor torque control, and pressure detecting signal when solvent is delivered at constant pressure. The other signal, further filtered to the signal in this pin (6), is slow in response. This signal (expressed as Vp' in circuit diagram) is used for pressure meter indication and pressure limit circuit.				
7	V/F converter output (expressed as (i) in circuit diagram) Voltage corresponding to the setting flow rate value (1/2 A2 output, expressed as (h)) is converted to frequency to drive the stepping motor by V/F converter consisting of inte- grator 1/2 A3, comparator A9 and reset pulse generator M11. The output voltage waveform of this pin is as illustrated with PUMP switch ON. $\int_{a}^{b} \int_{a}^{b} \int_{$				

Connector Pin No.	Normal Voltage Value/Remarks				
	$\Delta T = \frac{6 - 0.15 \text{ Fe}}{900 \text{ Fe}} \text{ sec}$ When Fe = 1ml/min., for example, ΔT is:				
	$\Delta T = \frac{5.85}{900} = 0.0065 \text{ sec} = 6.5 \text{ ms}$ However, when the pump is in the discharge stroke and in the suction stroke (quick re-				
	turn), $f \approx 2000 \text{ Hz} (\ \text{$\Delta T \approx 0.5 \text{ ms}$})$				
8	Voltage, corresponding to the setting flow rate is generated at this pin. This voltage is approximately: $-0.6 \times Fe(V)$ (Fe: Setting flow rate ml/min.) When Fe = 1ml/min., for example, the voltage is $-0.6V$. The flow rate is actually adjusted, using trimmer R9: 500Ω , so that the voltage on pin 8 differs from foregoing voltage slightly. When the pump is in the discharge stroke, a normal voltage will be: $-0.6 \times Fe(V) \pm 3\% \pm 5mV$ o With PUMP switch OFF, this voltage is approx. $+1V$. o When the pump is in quick return, the voltage on pin 8 is not $+1V$ but approx. $-6V$. The voltage waveform is as follows. $-0.6 \times Fe(V)$ Approx. $80ms$ at -1000 Jm^2 (Held at $-6V$ by $2/2 \text{ A2}$) Approx. $80ms$ at -1000 Jm^2 (Held at $-6V$ by $2/2 \text{ A2}$)				
9	This terminal is for voltage adjustment of pressure limit (upper/lower) circuit. Adjust trimmer R39: 100Ω so that this voltage is +2.55V.				
10	Voltage on this pin (Vg) is: $Vg = +0.1 \times 15^{(V)} \pm 0.3\% \pm 5 \text{ mV}$ $Dig. SW \times \text{ one digit}$ Dig. SW × ten digit				

Connector Pin No.	Normal Voltage Value/Remarks					
	(1) For CONST. FLOW setting, the flow rate setting voltage is:					
	$Vg \cong +1V/m\ell/min$					
	(2) For CONST. PRESS setting, the pressure setting voltage is:					
	$Vg \cong +0.01V/kgf/cm^2$					
	(3) However, when "PUMP" switch is OFF, the setting voltage is:					
	Vg ≅~-0.6V					
	Shows minus voltage					
11	+2V power source (+1.5 to +2.5V)					
12	+12 ~ +15V					
	(a) (b) $-12 \sim -15V$ (c) $-12 \sim -15V$					
	P. MONIT lamp on the front panel is lighting during this period.					
 a : When the plunger is retreated fully (start point of the discharge stroke) b : The plunger is in the discharge stroke, and in the position covering approx. 409 the entire stroke. 						
13	Voltage (for example, Va) on this pin is proportional to current flowing into the stepping motor. The LC-6A uses a 4-phase stepping motor, which is driven by the 2-phase exciting system. The exciting status is as illustrated below.					
	A phase When transistor Q., is ON					
	Ā phase 777					
	B phase " Q13 "					
	B phase 7/77 " Q14 "					
	In the LC-6A, this motor current is detected, using R_{62} : 0.1 Ω . The equipment is con- trolled so that motor current is always constant even when the flow rate changes. When pressure increases, the equipment allows 1/2 A5 amplifier setting signal to increase via R_{70} : 56.2 $k\Omega$ in proportion to the pressure so that motor current increases according to the pressure. The motor current is fixed as follows.					

Connector Pin No.	Normal Voltage Value/Remarks			
	Pressure Motor	current Va]	
	0kgf/cm ² Appro	x. 1.9A Approx. 190mV		
	200 "	2.7A 270mV	1	
	500 "	3.6A 360mV		
14	Spare terminal			
15	+5V power source (+4.8 to +5.2V)			

3.6.2 Relation between deformed cam rotating angle and pulse

(1) The relation between the plunger position and deformed cam angle is as follows.

Plunger Position	Deformed Cam Angle
Full depressed state	$\theta = 0^{\circ} (360^{\circ})$
Full protruded state	$\theta = 270^{\circ}$

 $0^{\circ} \le \theta \le 270^{\circ}$ Plunger is in the discharge state. $270^{\circ} \le \theta \le 360^{\circ}(0^{\circ})$ Plunger is in the suction state. (Quick return)

(2) Relation between the disk hole position and deformed cam angle



(3) Detecting home position

With PUMP switch ON, the motor rotates, and the home position (the state in which the plunger is fully retreated) is detected by the photocoupler near the pump disk. When the disk hole comes up to the photocoupler, collector voltage Q_6 is as illustrated below and flip flop M5 is set.



This home position is detected by the first turn of the disk. After that, the home position detection is ignored even when the disk home passes through the photocoupler. The home position thus detected once is maintained as it is except when "PUMP" switch is turned OFF (when flip flop M5 is once set), or pressure limiter is actuated. Once the home position is detected, the plunger position is then monitored, using counters M7, M8, M9, M10.

These counters are reset every 1200 pulses (one stroke of plunger). When the plunger is in the discharge stroke, the motor rotates for 900 pulses. When the plunger is in the suction stroke, the motor rotates for 300 pulses.

(4) Compressibility correction circuit

The LC-6A employs a compressibility correction circuit such that when the plunger returns to thereby suck solvent from the reservoir into the pump chamber, and re-starts discharge, the quick return speed is maintained as it is up to a certain angle of deformed cam according to the pressure, thus allowing the compression of solvent inside the pump chamber at high speed.



Quick return speed is maintained during this interval,

Actual compressibility correction is adjusted simply in the following manner.

- (1) Measure the flow rate whose back pressure is nearly 0kgf/cm^2 .
- 2 Apply a proper back pressure (column, etc.) 100 to 200kgf/cm², and measure the flow rate at that time. Turn trimmer R₈₂ to adjust the flow rate so that it is identical to the measurement (1) above.

3.6.3 Usage of the REMOTE connector

The REMORE connector at the rear panel of the equipment is used for connection to our System Controller SCL-6A. When it is not used with the SCL-6A, the pump can be

controlled externally as shown in Fig. 6.5, according to the purpose.



REMOTE Connector

Fig. 3.6 Wiring Diagram for REMOTE Connection

(1) A voltage of 10mV is applied across the No. 2 terminal (+) and No. 3 terminal (-, 0V) of the connector, for 1kgf/cm² of the pump delivery pressure from the pressure sensor. If the pump delivery pressure is to be recorded by a recorder with an appropriate full scale for, for example, divide the voltage across these terminals by the resistance of the recorder.

Resistance of the device connected to these terminals should not be lower than $10k\Omega$.

- (2) A pressure signal with voltage divided according to partial pressure in advance inside the equipment is applied across the No. 1 terminal (+) and No. 3 terminal. If the voltage across these terminals is connected to a recorder of 1mV full scale, the pressure can be recorded with a 500kgf/cm² full scale value.
- (3) The No. 4 terminal (+) and No. 15 terminal (-, 0V) of the connector are for an external flow rate input signal. The flow rate can be set externally by inputting a voltage between these terminals with the No. 4 and No. 5 terminals left open (Flow rate setting cannot be performed from the front panel, however, if the terminals are open.).

The flow rate is set at $1m\ell/min$. for 1V of voltage input. The range of input voltages is $0 \sim \pm 10V$ (not higher than 3mA in current input). Output impedance of the voltage input must not be higher than 10Ω .

Note that flow rate cannot be set externally if the constant pressure solvent delivery mode is selected at the front panel.

(4) A reed relay contact output can be connected between the No. 7 and No. 8 terminals. This relay contact is closed when either the upper or lower pressure limiter is actuated. If connected to these terminals, external equipment will automatically stop when the pressure limiters are triggered.

Rating of the relay contact is 5VA (DC 50V, 0.1A).

If external equipment with capacity higher than the contact capacity is to be driven by this relay contact output, connect a power relay to the external equipment and drive the equipment through the power relay.

- (5) To turn ON or OFF the pump externally, use the No. 9, No. 10 and No. 11 terminals: the pump is turned ON if the circuit between the No. 10 and No. 11 terminals is shorted or it can be turned OFF by shortcircuiting by contact relay between the No. 9 and No. 11 terminals momentarily (about 0.1 sec.).
- (6) The lead wire used for connecting the REMOTE connector to external equipment should be made as short as possible so as to minimize noise. Ground the 0V line at a point outside the equipment.

No	Part Name	Part No.	Remarks			
1	SW. paddle M-2022-2-W	064-14086-01	" POWER " switch			
2	Fan PE2B55	042-60085	Cooling fan for 100 or 115V			
L	Fan PE2B60	042-60086	" 200~240V			
3	Fuse F7161 3A 125V	072-01026	100, 115V			
	Fuse ULCS-61M 1.5A	072-01660-16	220~240V			
4	Transformer 6A 100V system	228-13799	Power transformer for 100V or 115V			
	Transformer 6A 200V system	228-13993	" 220~240V			
5	ZNR. ERZ-C10DK201	061-80703-12	Surge absorber for 100V or 115V			
	ZNR. ERZ-C10DK431	061-80703-17	" 220 ~ 240V			
6	PC board PB-1 ASSY	228-13485-91	PC board assembly			
7	*C. #PC4558C	075-31135	A1, A2, A4, A5, A6, A7, A8			
8	// #PC4082C	075-31139	A3			
9	// #PC55A	075-31104	A9			
10	SN74LS73N	075-20073-02	M12			
11	SN74LS05N	075-20005-02	M13			
12	" NE555V	075-23001	M11			
13	" TC 4011 P	075-33340-11	M4			
14	" TC4017BP	075-33340-17	M8,M9,M10			
15	" TC 4013 P	075-33340-13	M5,M6,M7			
16	Transistor 2 SA 1015-O	060-21015-01	Q4			
17		060-25815-02	Q1,Q3,Q5,Q6,Q7,Q15			
18	FET 2N4392	060-38801	Q2,Q8			
19	Diode 1 S 1588	060-01588	Parts on			
20	~ 3BZ61	060-15017	$D27 \sim D30, D32, D33$			
21	// 1BZ61	060-15011	D23~D26, D56			
22	// 2B4DM	060-13812	D21			
23	<i>∞</i> 1 SZ 45 A	060-14074-01	D22			
24	₩ 05 Z 9.1 L	060-14317-20	D5			
25	// 05 Z 15 U	060-14317-36	D31			
26	Reed relay PRA-4. DC5V	065-63056	K1			
27	Diode 05 Z 4.3 Y	060-14318-23	D15. D16 D38			
28	iС. µРС14315Н	075-31134	MI			
29	« MC7915C−Т	075-22026	M2			
30	" #PC14305H	075-31131	M3			
31	Transistor 2 SD633	060-27633	Q11~Q14			
32	" 2SC790	060-24790	Q16			
33	Diode RD51FB	060-14155				
34	Transistor 2SD588Q	060-27588-03	Parts mounted on radiator in			
35	<i>*</i> 2SD797~Y	060-27797-02	PB-1 ass'y			
36	SW. Digital 177601MN	064-92210	Flow rate setting switch, one digit			
37	Meter TRM45(100 μ A) # 1 - 4	080-48051-05	Pressure indication meter			
38	LED. TLG124A	061-78068-02	"POWER" indication lamp			
39	# GL=2PG1	061-78038-06	"PUMP" indication lamp			
40	" TLR-205	"U LIMIT" "L I IMIT" indication farmer				
41	" TLG205 061-78067-02 CECP PMONIT indication fame					
42	VR. RV24YN20SB 50K	053-12111-02	3-12111-02 Pressure limiter setting resistor			
43	SW, KSD1-2-0-LL-DC 064-56041-13 C.FLOW/C.PRESS select switch					
44	SW. SUN11 064-56058-04 PUMP ON/OFF switch					

4. Maintenance

Always check the following items and take necessary actions.

Section 4.1 describes maintenance requirements for the LC flow line system in general. Section 4.2 through 4.8 describe maintenance requirements for each part of the LC-6A main body.

- 4.1 Inspection of clogging and stains and cleaning of LC-flow line
 - (1) Inspection

Clogging or contamination of the flow line may be the reason for abnormally high pump pressure, large drift of the detector baseline, or variation of peak retention time or peak separation. Clean each portion of the flow line in the direction of flow.

- (2) Cleaning
 - (a) Cleaning operation of the flow line between suction filter and sample injector
 When the suction filter becomes clogged after long term operation, normal flow cannot
 be established due to the increased resistance of the flow line or generation of bubbles in
 the inlet tubing.

Contamination inside the pump head, check valve or line filter also prevents normal flow rate and causes drifting of the baseline.

Allow isopropyl alcohol to pass through the LC-6A for cleaning the line.

(b) Detector cell

Clean the cell according to the instruction manual of the detector.

(c) Column

If peak retention time or peak separation fluctuates in an analysis with the composition and flow rate of the solvent remaining unchanged or if drift occurs on the base line, the most probable cause is a contaminated column. For slight contamination, continuously pump fresh mobile phase, for a relatively long period of time, usually at a flow rate not higher than the analytical flow rate and below the maximum permissible pressure for the column. For heavy contamination, clean the column with a strong solvent suitable to the column packing materials. (For details, refer to the instruction manual of the column or contact our Analytical Application Laboratory.)

(d) Entire flow line

Buffer solutions will produce crystalline substances or residues upon dehydration or evaporation of the solution. If this occurs and the mobile phase is left in the solvent line for a long period of time, the tubing may become clogged, or the plunger and pump seal become damaged, causing leakage. After using such a mobile phase, therefore, wash the flow line thoroughly in distilled or deionized water. The operations described in Section 7.5 are recommended. If a buffer solution accidentally enters into the back of the pump head when replacing the damaged plunger seal, salt may deposit between the pump main body and the bearing (positioned right behind the pump head). If this occurs, it may be difficult to later disconnect the bearing. In such a case, slowly turn the screws used for setting the pump head alternately into the two screw holes provided at the angle of 45° above the bearing, and disconnect the bearing when it dislodges. Rinse away the salt from all parts with water.

4.2 Replacement of plunger seal

The service life of a plunger seal varies depending upon operating conditions (flow rate, pressure and mobile phase). If liquid leaks from the bottom of the pump head or from the small stainless steel drain pipe beneath the bearing behind the pump head, re-tighten the two bolts that set the pump head to the bearing. If the leak persists, replace the plunger seal. Prior to replacing the plunger seal, set the flow rate at $1m\ell/min$. or lower and actuate the pump. Then turn the pump switch OFF immediately after the pump monitor lamp lights. The plunger stops at its maximum position of withdrawal by the above operation, and therefore, the plunger seal can be replaced without breaking the plunger. Replace the seal in the following sequence.



1 Disconnect the tubing from the top and bottom check valves of the pump head.



(2) Loosen two bolts fixing the head, alternately little by little to remove them.



3 Pull the head away from the pump carefully.

Fig. 4.1

Fig. 4.2

Fig. 4.3





- (4) Take out the seal with tweezers or the like pointed tool, taking care so as not to damage the inside of the head.
- (5) Clean the inside of the head. Set the spacer as shown in Fig. 4.6 and set a new seal flat against the surface, using your thumb.

Fig. 4.5



(Caution) Set the spacer with the grooved side facing away from the pump body.

Fig. 4.6

Fig. 4.4



(7) Holding the head firmly with the thumb, tighten the bolts alternately little by little so that the head is set correctly and evenly. Firmly tighten the bolts at the end.



(8) Replace the tubing at the top and bottom check valves of the pump head.

Fig. 4.7

6 Mount the head correctly as

shown.

Fig. 4.9

4.3 Replacement of line filter

If the pressure gauge indicates a high value even when solvent is pumped without a column, a possible cause is a clogged line filter. In such a case, disassemble and clean the line filter, or replace it with a new one.

4.4 Zero point adjustment of pressure sensor

If the pressure gauge does not indicate zero when the power switch is OFF, turn the zero adjustment screw beneath the pressure gauge so that the gauge indicates zero. If the pressure gauge does not indicate zero when power switch is ON, turn the pressure sensor zero point adjustment trimmer ((16) in Fig. 3.1) slowly with a small screwdriver so that the gauge indicates zero.

4.5 Lubrication of the moving parts of the pump

To ensure long service life of the equipment, periodically lubricate the moving parts of the pump. Lubrication intervals are $3 \sim 4$ months in general. Remove the case cover of the equipment, grease two points shown in Fig. 4.10 while operating the pump. Be sure to use the lubricant supplied as standard accessory. Do not use other lubricants.

Lubrication point 1 Apply 4 or 5 drops of lubricant.

Lubrication point 2 Apply 4 or 5 drops of lubricant on the contact surfaces between cam and bearing.



Fig. 4.10

4.6 Inspection of check valve



Fig. 4.11 Check Valve

Fig. 4.11 illustrates the construction of inlet and outlet check valves.

The check valves can be easily disassembled, cleaned and reassembled by users in the following manner.

- 1. Remove check valves from the pump head.
- 2. To disassemble the check valves, pinch gasket B at point a of the outlet check valve, using narrow tweezers and take it out of the cartridge.
- 3. Then, take out the housing, tapping the cartridge. (Take care so that housing, seat A and ball do not come apart when taken out.)
- 4. Observe the contact surface (see Fig. 4.12) of the ball seat through a microscope of $80 \times$ magnification to check that the surface is not broken or stained.

If broken, replace with a new ball seat.

If stained, wipe off the stain.

Thoroughly clean the ball and housing with ethyl alcohol.



Fig. 4.12 Ball Seat

5. After inspecting the ball seat, reassemble the check valves, ensuring proper

orientation of the ball seat; the ball must be in contact with the surface of the ball seat. In assembling the check valves, also note that the orientation of the housing in outlet check value is reversed from that of the inlet check value. Assemble with care, referring to the illustration in Fig. 4.11.

6. Gasket A, seat A and gasket B, which are made of resin, serve to prevent liquid leakage between the parts of the check valves. Therefore, these parts must be correctly positioned in their proper states.

If seat A stretches excessively, the ball seat inclines or the ball does not come in contact with or off the ball seat smoothly. If gasket B expands radially, a spanner cannot be inserted into the cartridge. In these cases, replace seat A and gasket B.

- 7. Finally, mount the check valves on the pump head. Do not tighten with excessive torque; otherwise, gasket B may be damaged.
 - Note) Check that valve repair parts kit (P/N 228-11200-91) contains ball seats, gasket A, gasket B and seats A, 2 pcs. for each.

5. Troubleshooting

- 5.1 General troubleshooting
 - It is presumed that the malfunctions described below are caused by the following:
 - 1. Breakdown of the solvent delivery unit (LC-6A)
 - 2. Breakdown of equipment other than the solvent delivery unit
 - 5.1.1 Large drift and noise are generated on the base line.

Check faulty points and take proper corrective measures in accordance with the following flowchart.



5.1.2 Even when samples are charged, no peak is produced. Follow the flowchart below for checking and remedy.



5.1.3 Peak retention time changes, abnormally short or abnormally long.



- 5.2 Solvent delivery flow rate is unstable, and solvent delivery flow pulsation is noticeable.
 - 5.2.1 Checking method
 - (1) Measuring flow rate

A standard flow rate measurement is made under two conditions: no-load conditions and loaded conditions.

For flow rate measurement at no-load, with a \$\$\phi1.6 \times 0.3 \times 2000mm pipe connected to the pump outlet, measure actual flow rate at 1ml/min. setting flow rate. For this measurement, use a 5ml mespipet to calculate the flow rate from the time required for delivering 2ml methanol. A normal flow rate is 1ml/min. ±20µl. Even for the setting flow rate other than 1ml/min., the flow rate can be measured in the same manner. At this time, the flow rate is normal if it falls within the range of specification described in the instruction manual.

- (2) The flow rate measurement under loaded conditions is made with a \$\phi1.6 \times \$\phi0.1 \times 4m\$ pipe connected to the pump outlet. Measure actual flow rate at 5ml/min. setting flow rate in the same manner as in (1) above.
- (2) Recording pump discharge pressure

For pressure recording method, refer to para. 3.6 (1) and (2).

Pressure records are effective for checking flow rate stability. Ensure that irregularities, fluctuations, etc. of the output waveform are not present through one hour or more recording.

A normal pressure pulsation width is 5kgf/cm^2 or less at $1 \text{m}\ell/\text{min}$. setting flow rate when methanol is delivered with $\phi 1.6 \times \phi 1.4 \times 4 \text{m}$ pipe connected to the pipe outlet.

5.2.2 Cause and remedy

(1) Solvent leak at flow line units and remedy

When volatile solvent is used for mobile phase, it is hard to find solvent leak, so that mobile phase should then be replaced with distilled water (or deionized water). Raise the pump discharge pressure up to the pressure near the maximum withstand pressure of each flow line to check for solvent leakage and repair if faulty.

Solvent leakage at joint

Retighten the joint. If the leakage still persists, replace ferrule or wind PTFE tapes around the ferrule to prevent leak.

Solvent leakage at dampener for high pressure

No repair is possible. Replace the dampener with a new one. (Solvent flow has no

directional property.)

Solvent leakage at drain valve

The drain value is structured as shown in Fig. 5.2. If the knob (2) is kept turned fully clockwise, solvent does not flow into the drain tube side, but flows into the filter side only. If solvent should flow from the drain tube side under this condition, check and clean soft packing (1) and soft packing contact face on body (5) in the following sequence.

- i) Loosen the knob counterclockwise approx. six turns to pull it a little forcedly and pull out the knob and shaft (3) fixed to the knob.
- ii) Use a toothstick or the like to take out soft packing (1) and drain packing B (4) remaining in interior body. Both packings (1) and (4) above will come out in step i) above, according to solvent use and force when the knob is pulled out.
- iii) Check the body contact surface. Remove dust or stains, if any.
 For reassembly, reverse the disassembly procedure described in i) above with care so that (4) may not be detached from (3) with (4) inserted into the hole drilled at the tip of (3). If leakage still persists, replace the drain valve with a new one.



No.	Parts Name	Parts No.	No.	Parts Name	Parts No.
1	Soft packing	228-07553	6	Filter F6 ass'y	228-12642-93
2	Knob K31, bronze	037-01049-02	7	Packing	228-12564
3	Shaft, drain	228-13946	8	Setscrew, WP M4 × 4	021-04632
4	Packing B, drain	228-12868-01	9	O-ring, Teflon P4	036-11402
5	Body drain	228-13947			

Fig. 5.1 Drain Valve Ass'y

Solvent leakage from pressure sensor flow line

No solvent leak can be corrected. Replace with a new one.

Solvent leak from plunger seal

When any leakage from the plunger seal is found, solvent leaks from the following two points.

• Pump head lower part and check valve (inlet) rear

• Pipe drain 6A ((42) in Fig. 3.6)

For these leaks, take corrective steps in the following manner.

- (a) Retighten pump head.
- (b) Replace the seal since it is worn out. (See para. 4.2.)
- (c) When no leakage can be stopped yet, check or replace plunger and bearing.
 CAUTION: When replacing plunger, ensure that the plunger is not damaged. If it is seriously damaged (the part where it contacts the seal), replace the plunger.

Disassembling pump head unit (See Fig. 3.6.)

- (a) Remove head (See para. 4.2.).
- (b) Remove bearing 6AR.
- (c) Remove screw (41) to take out plunger.

(2) Increasing flow resistance and countermeasures

It is presumed that one of the causes in which solvent delivery flow rate is not stable lies in changes and increase in flow resistance of suction filter, line filter, dampener, tubing, column, etc. A simple checking method is a diagnosis using the pressure waveform. At this time, use fully degassed mobile phase.

(a) Suction filter

Suction filter becomes clogged after long use, and depending on the conditions when mobile phase containing lots of dust and impurities is used. For checking method, remove metallic filter unit to record the output waveform. If the output waveform is better with the filter detached, it follows that the suction filter is in the clogged state.

As corrective steps, perform ultrasonic cleaning (use isopropyl alcohol), or replace with a nre one. The suction filter is equipped to prevent deterioration of the pump plunger seal, check valve, column, etc.: be sure to mount this filter before use except when inspection is carried out.

(b) Line filter (See Fig. 3.5.2.)

The line filter functions to prevent stains on the injector column and entry of dust therein, including seal chips generated in the pump.

The inspection method is the same as with the suction filter: remove the line filter to record the pressure waveform, and judge clogging conditions.

The remedy includes backwash with the exit side connected reversely and ultrasonic cleaning with isopropyl alcohol for approx. 30 minutes after disassembly. When no washing effect is produced, replace the line filter with a new one. After checkout and correction, mount the line filter securely to protect the column.

(c) Dampener, tubing, etc.

If these parts are used correctly, no flow resistance changes. When contaminants occur such that they get choked inside the pipe as can be seen in salt separating taking place when buffer mobile phase is used, it is also presumed that flow resistance may vary. To check this, as is the case with the suction filter and line filter, remove dampener, and tubings to find out the parts in which resistance is changing depending on output waveform changing conditions.

To remedy dampeners, one of the methods is to attempt to let solvent flow with the inlet and outlet reversed since the solvent flowing direction is not fixed.

(d) Changes in column resistance

Record and store flow resistance (flow rate, pressure, temperature, mobile phase name) at the earlier stages in which the column is used to thereby judge the column resistance changes.

- (3) Disassembling and washing check valve
 - Refer to para. 4.6.
- 5.3 No pump performs high-speed suction operation.



5.4 Flow rate at no load does not fall within the specification with respect to the present value.



- 35 -

5.5 Abnormal noise is generated from one pump (noise is too large).



5.6 Sample symptoms for pressure base

(1) Diagnosis by output waveform

In reference to para. 3.6-(1) and (2), allow the recorder to record the output waveform of column entry pressure, and check pump operating conditions, judging from the waveform symptom.

Note: The following should be noted during recording:

When the ambient temperature of equipment changes and it is exposed to the wind, the viscosity of mobile phase fluctuates, and since the column resistance value changes, the output waveform swells and drifts. Under this condition, a sure troubleshooting cannot be achieved: ambient conditions should be kept constant.

Output Waveform	Cause	Remedy			
	 Air dissolved in mobile phase. Mismatching or check valve OUT ball and seat. Mismatching of check valve IN ball 	∘ Degass mobile phase. ∘ Replace ball. ∘ Replace ball.			
Many spikes appear at the	and ball seat. • Solvent leak from plunger seal. • Bubbles sucked from plunger seal.	 Replace plunger. Retighten pump head mounting bolts equally. Replace plunger seal. 			
lower side,	• Air sucked from main unit con- nections of suction filter tube. • Pump head temperature rise.	 Retighten bushing. Lower room temperature (Air-condition- ing is required in summer). 			
	∘Bubbles stay in pump head.	• Let lots of mobile phase flow from DRAIN port.			
Moderate spikes appear at the lower side.	 Bubbles grow and flow away due to stained pump head interior or in- compatible previous residual mobile phase. Air dissolved in mobile phase. 	 Let isopropyl alcohol in air (50ml at 9ml/min.). Under pressure conditions (about 200 kgf/cm²), let isopropyl alcohol. Degass mobile phase. 			
Output waveform is irre-	 Check valve OUT ball and ball seat stained. Check valve OUT ball seat cracked or chipped. 	 Clean ball and ball seat. Replace ball seat. (Ball may sometimes be replaced.) 			
gular vertically.	•Check valve OUT loosely tightened.	• Re-tighten.			
Pressure suddenly lowers (occurs repeatedly).	∘Line filter clogged. ∘Column resistance changes.	 Backwash line filter (use isopropyl al- cohol). Replace. Replace column. 			
Pressure output waveform swells.	 Slight solvent leak from plunger seal. Solvent leak from part of flow line. Changes in column resistance due to changes in room temperature. 	 Replace plunger seal. Prevent leakage. Control changes in room temperature. 			

Table 1 Symptoms Developed on Output Waveform of Column Entry Pressure

5.7 Checking circuit

Should the circuit fail, first of all, refer to para. 3.6.1 to check voltage.

Description here is given of checking methods in para. 2 and 3.





ī

(3) Trimmer adjusting method

When parts are replaced due to parts breakdown, trimmer re-adjustment is required. Description is given here of each trimmer adjustment method.

Used for flow rate control. Measure solvent delivery flow rate actually, and adjust this trimmer for the actually measured flow rate value.							
Used for 1/2 A3 offset adjustment. Connect $100k\Omega$ resistor across TP-1 and TP-2 check termi- nals, and adjust this trimmer so that voltage on check connector pin No. (7) is $0 \pm 1 \text{mV}$ with "PUMP" switch ON (Flow rate is set at 0m /min.).							
 Used for frequency adjustment. When this trimmer is used for adjustment, 1) Set the flow rate to 5ml/min. 2) With "PUMP" switch ON, measure voltage on check connector JG pin No. (8). When this voltage does not fall within - 3000V ± 10mV, use trimmer R₉ so that the above voltage falls within - 3000V ± 10mV. (However, always record the voltage in advance so that the original value is obtained again after R₁₆ adjustment.) 3) Adjust this trimmer until the frequency of check connector JG pin No. (7) becomes 841 to 842Hz. 							
For -10V power source. Adjust R53 so that check connector JG pin No. (2) becomes -9.99 to -10.01V.							
For pressure limiter. Adjust R ₃₈ so that check connector JG pin No. (9) reaches +2.55V ± 0.05V.							
For compressibility correction adjustment. See para. 3.6.2(4).							
For adjustment of plunger "play" magnitude. For this adjustment, use distilled water for mobile phase, connect resistance tube $1.6\phi \times 0.1\phi$ \times 2m for back pressure, set the flow rate at $0.2m$ /min., record pressure at 50 kgf/cm ² full scale, and adjust this trimmer so that the waveform as illustrated below is obtained. (This pattern is not accepted.) 4 Quick return Adjust R ₁₂₂ so that this is reduced as far as possible. (Magnitude)							
_							

Table 2	Trimmer	Adjusting	Method
		–ajasung	itio uto u



SHIMADZU CORPORATION

TOKYO OFFICE

DOVO DEFICE P.O. BOX No.209, 40th Floor Shinjuku Mitsui Bidg., No. 1-1, Nishi-Shinjuku 2-Ohome, Shinjuku-ku, Tokyo 163, Japan Cable Add.:SHIMADZU TOKYO Overseas Telex No.:D232-3291 (SHMDT J) VOTO DEFICE

KYOTO OFFICE 1, Nishimokyo-Kuwabaraoho, Nakagyo-ku, Kyoto 604, Japan. Cable Add. SHIMADZU KYOTO Overseas Telex No.:05422-166 (SHMDS J)

3500-11600-400TD-E (811)



)

 \rightarrow

)



IND.	Parts Name	Parts No.	NO.	Parts Name	Parts No.	NO.	Parts Name	Parts No.	
1	Pulse motor KP8M2G-0001X	228-13956	21	Spacer pump head B	228-12284	41	Set screw, plunger	228-12906-01	
	" PH296-01S	228-14621	22	Seal UR304-125GFHST	228-11999	42	Pipe drain 6A	228-15851	
2	Gear head 8H6FB	132-53026-02	23	Bearing LC-6AR	228-14167-11	43	Drain tank	228-14594	
ļ	" 4GK6K	132-53651	24	Plunger ass'y	228-12904-93	51	Cartridge, single F6	228-15637-01	
3	Key-accessory	dependent	25	Thrust LC	228-13381	52	Gasket A	228-09028	
4	ROTEX TYPE 14	228-14038	26	Cross-head LC-6A	228-12907	53	Housing	228-09027-01	
5	Disk	228-12917	27	Bearing RTLB1602J	228-14036	54	Ball	018-98002	
6	B ody 5701	228012887	28	Bearing ARF0608LD	031-40233	55	Seat A	228-79574	
7	Photocoupler 6A ass'y	228-14307-91	29	Stopper	228-12911	56	Ball seat	018-98002	
8	Clamp E-20	037-60196	30	Circlip E type SUS4	026-66204	57	Gasket B	228-79029	
9	Parallel pin SR 1.2×8	026-11012	31	Holder, cam follower	228-12909	58	Pump head LC-6A	228-12901	
10	Bearing 6005 ZZ	030-03005	32	S srping pin 1.2×12	026-03515	59	Cartridge	228-12354	Fig
11	S spring pin 1.2×5	026-03511	33	Yoke return spring	228-12910				9.
12	Key fixing	228-08289-04	34	Snap ring E-25	026-66212				
13	Cam follower KR16LLX	031-07241	35	Bearing ARF0406LD	031-40231				
14	Cam LC-6A 7609	228-13359-01	36	Spring A	228-08720-04				
15	Oil mat	228-14274	37	Flange return spring	228-13382				

. 3.5 Solvent delivery pump main unit

.