## **RECYCLING PREPARATIVE HPLC** LC-92XX NEXT SERIES

# **USER'S MANUAL**



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# **USER'S MANUAL**



RECYCLING PREPARATIVE HPLC

# Preface

Thank you for introducing Recycling Liquid Chromatograph model LC-92XX NEXT. This manual contains instruction of the LC-92XX NEXT's operational manual, technical information and safety information.

Refer the manual thoroughly, for applying LC-92XX NEXT series to your research and investigation with safety operation.

Keep the manual near by the LC-92XX NEXT Liquid Chromatograph for referring any time.

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- •Manuals with missing pages or incorrect collating will be replaced.

# **Warranty Statement**

JAI warrants the Recycling Preparative HPLC LC-92XX NEXT series as follows, only when you operate it correctly following the instructions in this manual and without remodeling it.

Warranty period

LC-92XX NEXT Products are warranted for a period of one year from the date of installation.

#### Warranty detail

If the product is found to be defective due to a manufacturing fault during the warranty period, JAI will repair or replace the product or defective parts without charge.

#### Negative assurance

Even during the warranty period, the defects caused by the following are not covered by this warranty:

- •Improper use.
- Operations under the condition not authorized in this document or our company.
- Abnormal power supply or use under the conditions of irregular voltage or frequency.
- Defect is coming from other factors, not from the LC-92XX NEXT.
- Use under severe environmental conditions such as corrosive gas, high temperature and humidity, or continuous vibration.
- •Flowing a highly corrosive liquid.
- Force majeure such as earthquake, fire, or lightning.
- •Removal or transfer of the product after installation.
- Remodeling or Disassembly of the LC-92XX NEXT with not written way in this manual.
- ·Using an instrument that is resold without our approval.
- Defects of the consumables or the parts of which the warranty period is limited are not warranted.

Parts of consumption :

Pump seal, pump plunger, pump plunger guide, pump check valve, line filter, manual sample injector (rotor seal/stator face), selector valve (solvent selector, recycle flow 1, recycle flow 2, drain, C/D selector)

The items which are not included in this paragraph are out of the warranty.

Direct or indirect damages coming from the evident or implied warranties of this instrument are not compensated.

Direct or indirect damages coming from destroy of the instrument are not compensated.

# How to Use This Document

This document "LC-92XX NEXT SERIES USER'S MANUAL" consists of the following four volumes.

Choose the volume to use as needed.

Each volume has its own table of contents at the beginning.



# **To Operate in Safety**

LC-92XX NEXT series instrument is a recycling preparative liquid chromatograph to apply the separation analysis and separation collection of the samples by using the principle of liquid chromatography.

Please read and protect to precaution for safely operations of the LC-92XX NEXT series.

#### Cautions

- Do not use the LC-92XX NEXT series instrument other than a liquid chromatography.
- Please read and adhere the instruction in this manual.
- Take safety measures to prevent an accident.
- Please note that the LC-92XX NEXT products that are remodeled or disassembled without our prior notice are not warranted.
- In long term stop of the LC-92XX NEXT, disconnect Power cable. Seal column ends with correct plugs to prevent column drying. Fill tubing with noncorrosive solvent to prevent collision of tubing and flow line assemblies, but not flow the solvent into the column.
- •To avoid possible eye injury and skin trouble, wear eye protector and gloves.
- Prevent solvent leakage in the instrument.
- Do adequate ventilation in LC-92XX setting laboratory.

# Support

### Support on our Web site

Latest applications and soft ware revisions put on the site. News of our exhibition and Events also put on the site.

We are awaiting your question, conference and request information of our instrument.

Our Web site http://www.jai.co.jp/

# **Emergency Measure**

In emergency situation of the instrument or in disaster, do following emergency measure.

Restart should be carefully to check the instrument performance. If you have any worry, contact us or our representative.

#### **Emergency stop**

- •Turn POWER SWITCH off to stop the LC-92XX NEXT.
- •Turn off every POWER SWITCH of peripheral instruments.
- •Avoid solvent spill and leakage.
- •Cut off the power supply.



# Volume 1 Daily Operation Manual

Daily operations and information for new users

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# **Preface**

This manual includes basic operation of the liquid chromatograph model LC-92XX NEXT series to control.

For more details of information, installation, and maintenance of the instrument, please refer to other volumes, "General Information Manual", "Installation Manual", and "Maintenance Manual" on the tabs.

# **Cautions for Operation**

- If a failure or wrong operation occurs in the LC-92XX NEXT, an error message appears on the LCD display. At the beginning of the error message, "ERROR" is displayed followed by a number. Based on this number, refer to a section in "Volume 4 Maintenance Manual" to resolve the cause of failure.
- If you press the [Co/Dr] key for collecting fractions from a fraction nozzle during the [RECYCLE] operation, an error message appears and fractions cannot be collected. Stop the [RECYCLE] operation, and then press the [Co/Dr] key in [FRACTION] to collect fractions.
- Similarly, if you want to execute the [RECYCLE] operation during the [FRACTION] operation, stop [FRACTION] before executing [RECYCLE] by pressing the [Co/Dr] key in [FRACTION].
- •For the keys located on the sheet switches other than [PURGE] and [AUTO ZERO], you can press a key to execute its function, and press the key again to cancel the function.



# Chapter 1 Operation Flow

Operation flow of LC-92XX NEXT series

## **Operation Panel Display**

This section describes the overview of the operation panel of the LC-92XX NEXT series.



## **Operation Flow**

Actual procedure is as follows.

Prepare the solvent, waste reservoir and sample.



Turn the power switch on.



Press the [CLEAN UP] key.



Set parameters.



Jurn on the pump.



Turn on the integrator or recorder.



Check the column stability.



Inject the sample.



Recycle separation.



Collect the fractions.



Shut down the LC.



# Chapter 2 General Operation

General operation flow of LC-92XX NEXT series

## Prepare the solvent, waste reservoir and sample.

#### Solvent

- Prepare sufficient volume solvent in a container.
- Place the solvent container in the LC-92XX solvent stage.
- Prepare the solvent purer than chemical grade reagent. If Chloroform (CHCL3) for the solvent is recommended, CHCL3 added ethanol for stabilizer is suitable.(Pure CHCL3 or CHCL3 added Amylene stabilizer is not recommended.)
- In case of using mix solvent, mix each solvent completely.
- In case of using mix solvent, solvent mixture should be degassed perfectly. For details of degassing, refer to "Appendix" P.2-48 in "General Information Manual".
- For the properties of typical solvent, refer to Appendix P.2-51 in "General Information Manual".
- Filter the sample to remove micro particles with a suitable membrane. For details of solvent's filtration, refer to Appendix P.2-53 in "General Information Manual".
- •Be careful to handle the solvent and waste solvent.
- Prevent solvent leakage in the instrument.
- Do adequate ventilation in LC-92XX setting laboratory.

#### Waste

- A waste reservoir volume should be more than the solvent container. Reservoir should be chemical proof against the solvent.
- Do not the reservoir to turn over and spill solvent.

#### Samples

• Sample should be filtered to remove particles before injection. For the recommended sample filtration, refer to Appendix P.2-54 in "General Information Manual".

## Turn the power switch on.

•Make sure that the receptacle is 100 VAC and enough to supply power for the LC-92XX. Turn power switch on.



• After 15 seconds from turning the power switch on, the computer in the LC-92XX boots up to indicate the initial stage.

## Press the [CLEAN UP] key.

• Press the [START] key of [CLEAN UP] at the lower right side of the operation panel.



•When the manual operation is required, a message appears on the LCD display with an intermittent beep.

- Follow the instructions displayed on the LCD display.
- After the completion of the [CLEAN UP] operation, turn the Column reject valve back to its original position ([CONNECT]).

\*The [CLEAN UP] operation needs 10 to 15 minutes.

\*While the pump is in operation, [CLEAN UP] cannot be executed. Execute the operation when the pump is stopped.

## Set parameters.

#### Pump flow rate setting



- •Press the [FLOW SET] key in [PUMP].
- The parameter to be changed blinks on the LCD display.
- ·Use the arrow keys to set the flow rate value to use.
- Press the [OK] key.
  - \*While the pump is in operation, the value cannot be changed. Perform the operation while the pump is stopped.

#### Pump pressure limit setting



- Press the [MAXIMUM SET] key in [PUMP].
- The parameter to be changed blinks on the LCD display.
- Use the arrow keys to set the maximum pressure (limit).
- Press the [OK] key.
- Similarly, press the [MINIMUM SET] key in the [PUMP] unit tab to enter the minimum pressure.
- \*The values can be changed while the pump is in operation. Set the values based on the Column pressure.
- \*The value "0" makes off pressure limit.
- \*Make sure the [MAXIMUM] value is more than the [MINIMUM] value.



- •When using the recorder, press the [RANGE] key and adjust the value by using the arrow key.
- •Normally use the RI detector with the [TEMP.C] key turned ON (LED is turned on).
- After the [CLEAN UP] operation, the [AUTO ZERO] operation is not yet executed. After the column equilibrium, press both the [AUTO ZERO] keys in the [UV DETECTOR] and [RI DETECTOR] unit tabs.

## Turn on the pump.



• Press the [ON/OFF] key in [PUMP].

## Turn on the integrator or recorder.

- •Turn on the INTEGRATOR or RECORDER and monitor the detector signal. Make sure to stabilize the base line of the detector signal.
- The INTEGRATOR or RECORDER is an optional product. Refer to each manual for correct operation.

## Check the column stability.

- In case an RI detector is attached, make sure that the base line of the RI detector signal is stable.
- •JAIGEL-H series (20φ x 600 mm), W series(20φ x 500 mm), GS series (20φ x 500 mm) the stability for each column, needs from 30 to 50 minutes. ODS and SIL column should be waited with monitoring of the detector signal.

\*In case of using the W and GS column, change of the solvent composition may need long time.

\*Before the sample injection do zero adjustment referring to P.1-8.

## Inject the sample.



- •Prepare filtered sample. (For the typical filtering method, refer to "Micro Particle Elimination in Sample Solution", Appendix P.2-54 in "Volume 2 General Information Manual").
- Suck the sample solution in a syringe to pull a plunger.
- •Wipe the drop out of the syringe needle with clean paper.
- Hold the syringe upwardly, push the plunger gently to purge air from the syringe. Push the plunger until the air is purged thoroughly.
- Insert the syringe into the manual injector thoroughly until the syringe needle touches the bottom.
- •Turn the handle of the [MANUAL INJECTOR] to the [LOAD] position.
- Push the plunger thoroughly.
- •Turn back the handle to the [INJECT] position and take the syringe.
- •Clean the needle port with clean solvent. (For details on how to clean the needle port, refer to "Needle Port Cleaning", Appendix P.2-54 in "Volume 2 General Information Manual").

## Separate recycles.



If the injector located at the [INJECT] position, the manual sample injector makes dead volume to diffuse the peaks on the separation of the recycling.

## Collect the fractions.



## Shut down the LC.



• After collecting fractions, press the [ON/OFF] key in [PUMP] to stop the pump.

• Press the [START] key of [CLEAN UP] at the lower right side of the operation panel.



- •When the manual operation is required, a message appears on the LCD display with an intermittent beep.
- Follow the instructions displayed on the LCD display.
- After the [CLEAN UP] operation, move the reject valve back to its original position to complete the [CLEAN UP] operation. If the LC-92XX NEXT will not be used for a long time, stay the valve position at [REJECT].
- \*If the column stays a long time in the stopped flow line, solvent may be lost by gravity to dry the column packing and to damage the column.
- Turn off the integrator, recorder or other attachment.
- •Turn off the LC-92XX NEXT.



# Volume 2 General Information Manual

General information of LC-92XX NEXT series (except optional accessories)

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# Chapter 1 Description of LC-92XX NEXT

LC-92XX NEXT compositions, operation flow-chart, and operation panel functions

## **Composition of LC-92XX NEXT Series**

The LC-92XX NEXT series is a high performance recycling preparative liquid chromatograph for separation and purification.

The main body, pump, sample injector, column\*, detector\*, and integrator\* are installed as standard equipment.

High Resolution Hydrophobic SEC Column, High Resolution GFC Column, High Resolution SEC Column, Optical Isomer separation Column, Various Polymer Column, Partition and Adsorption Column NEXT series can accommodate different separation method for both analytical and preparative scale.

NEXT series UV detector and RI detector can be fit into the body together.

\*Columns, Detectors, integrator, and recorder are optional accessories. Please select optional accessories according to your purpose.

For details on using accessories, refer to separate instruction manuals for correct operation.

## Name and Function of Each Part

#### <Front of the LC-92XX NEXT>



Name	Function
Operation Panel	To press the buttons to set conditions or execute the
	operation.
Manual Injector Valve	To inject the sample manually.
Column Reject Valve	To disconnect the flow line from the column.
Pump Drain Valve	To drain the solvent from the pump outlet.
Sinhan Bay	To collect fractions from the fraction nozzle or the
Siprion Box	siphon, or to collect the waste liquid.

#### $<\!$ Inside of the LC–92XX NEXT Front Door $\!>$



Name	Function
Dump Hood	Device using suction and pressure to force solvent
Ритр пеаа	into the flow line.
Solvent Table	A table to place a solvent reservoir.
LN / D = (= = (= = *1	Detector for absorption of the light after passing
	through the UV cell.
Power Switch	To turn on/off the power of the UV detector.
Wave Length Tuner <sup>*2</sup>	A tuner for wavelength range.
RI Detector <sup>*1</sup>	Detector for refractive index.
Ciphon Dov	To collect fractions from the fraction nozzle or the
	siphon, or to collect the waste liquid.
Front Door	To protect inner units from damages and leaking
	solvent.

\*1 UV detector and RI detector are sold separately.

\*2 Wave-length tuner is only available for variable wave length type UV detector.



#### <Inside of the LC-92XX NEXT Right Side Column Cover>

\*1 UV detector and RI detector are sold separately.



Name	Function
Power Switch	To turn on/off the LC-92XX.
Output Dort	To output recognized signals from detectors
Ouiput Poli	and valves.
Solvent Out <sup>*1</sup>	Solvent out being connected to the fraction
Solvent Out	collector. (Option)

\*1 The fraction collector is an optional product. Normally it is not used.


Name	Function
Siphon	Outlet of the eluent flowing from the detector.
Fraction Nozzle	Nozzle for collecting eluent fractions from the detector.
Pump Drain Out	Drain out pipe for solvent from pump drain valve.
Manual Sample Injector Drain Out	To drain surplus solvent out when to inject sample into manual injector or to clean needle port.

# **Specification**

If the explanation or specification is the same for both LC-9210 NEXT and LC-9225 NEXT, it is written as 92XX NEXT in this manual.

	LC-9210 NEXT	LC-9225 NEXT
Pump Model	FX-10	FX-25
Pump Type	Reciprocating	double plunger
Maximum Discharge Pressure	25 MPa	20 MPa*
Flow Rate	0.1 to 10.0 mL/min	0.1 to 25.0 mL/min
Operation Panel	8.4-inch LCD operation panel	
Manual Sample Injector	Rheodyne 7725i	
Sample Loop (Standard)	3 mL 10 mL	
Event Marker	Injection, recycle and collect	
Recycle System	Built-in	
Non-diffusion Function	Twin valve method	
Auto-Cleaning	Route washing by program	
Dimension of Mainframe	464(W) x 492(H) x 504(D)	
Weight	Aprox. 38 Kg	

\*In the LC-9225 NEXT, the maximum discharge pressure differs depending on the flow rate to be used.

Flow Rate (mL/min)	Maximum Discharge Pressure (MPa)
0.1 to 9.9	20
10.0 to 19.9	15
20.0 to 25.0	10

# **NEXT Series Features**

This section describes the functional characteristics of LC-92XX NEXT series.

# **Recycling preparative HPLC**

This is a liquid chromatography for separation and purification. "Recycling function" enables the same solvent and the sample to flow through columns more than one time to complete perfect separation as if using "simulated" infinite columns.

# Layout

The manual sample injector is located outside of the body, which reduces time for tasks such as seal exchange and improves the maintenance. The pump and detectors can be taken out from the front of the body more easily.

# Compact sized body

Body size made 1/3 smaller compared to our previous models with improved functions and performance.

# Easy to operate

A 8.4-inch LCD operation panel is installed. The keys on the operation panel are designed for easy intuitive operation.

# Auto-cleaning

Automated flow line cleaning function keeps flow channels clean and solvent changeover very easy.

# Non-diffusion function

During extended recycling period, the recycled sample in the loop can diffuse back into the solvent line with the danger of contaminating the solvent reservoir. The NEXT series incorporates a patent pending non-diffusion device that prevents the sample from backing into the solvent line. Thus, this keeps the lines of NEXT series constantly clean automatically.

# **Flow Channel Map**

The flow channel map of the LC-92XX NEXT series is as follows.

Valves and flow lines of LC-92XX NEXT



<Normal Flow Channel via Siphon>



<Normal Flow Channel via Fraction Nozzle>



<Recycle Flow Channel>



# **Operation Panel Display**

This section provides the overview of the operation panel of the LC-92XX NEXT series.



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# **Chapter 2 Description of Operation Panel**

Description of the LC-92XX NEXT series operation panel

# WAKE UP

WAKE UP is the state when the power is turned on.

It refers to the initial screen of the LCD display that starts when the power switch is turned on.

The initial screen changes to the normal screen after approximately 15 seconds from power on.



<Example: WAKE UP of LC-9210 NEXT>

\*The LC-9210 NEXT model is used as example.

\*While the initial screens 1 and 2 are displayed, the system performs the initialization,

verification of the previous settings, and preparation.

\*Key operations are not available on the initial screen 1 or 2.

# **Description of Arrow Keys and [OK] Key**

### <How to Operate Arrow Keys>



The arrow keys are mainly used to change various setting values. The up arrow key increments the numerical value at the blinking cursor, and the down arrow key decrements it. The right arrow key shifts the blinking cursor to the right, and the left arrow key shifts it to the left. After setting, press the [OK] key to update the setting value.

<How to Operate [OK] Key>



This key is used to update the setting values. After setting a numerical value, be sure to press the [OK] key.

\*When the manual [CLEAN UP] operation is required or when an error message is displayed, the LED blinks at the top of the key.

# **Description of Units**

# **PUMP** Unit



 In the [PUMP] unit, there are the keys for starting/stopping the pump, and displaying the setting screens of the flow rate, the maximum pressure limit, and the minimum pressure limit.

# <Starting/Stopping the Pump>



This is the key for starting/stopping the pump. Press **ON/OFF** to start the pump, and press **ON/OFF** again to stop it. At the top of the key, the LED is turned off when the pump is OFF (stopped) and turned on when the pump is ON (running).

<Setting Pump Flow Rate>



Press the [FLOW SET] key to display the setting screen in the LCD display. Use the arrow keys to set the numerical value at the blinking cursor. After setting, press the [OK] key to update the setting value.



The current pressure is displayed in the normal screen of the LCD display.

Press the [MAXIMUM SET] key to set the maximum pressure limit.

Press the [MINIMUM SET] key to set the minimum pressure limit.

<Setting Maximum Pressure Limit>

When it's over pressed, this function is to protect columns and detectors. Press the [MAXIMUM SET] key to display the setting screen in the LCD display. Use the arrow keys to set the numerical value at the blinking cursor. After setting, press the [OK] key to update the setting value. When the maximum pressure limiter is activated, the whole system stops and an error message appears.

- \*"(MINSETVAL: 00.0 MPa)" displayed in the setting screen indicates the setting value of the minimum pressure limit
- <Setting Minimum Pressure Limit>

When it's pressed too low, this is to protect columns and solvent from splitting. Press the [MINIMUM SET] key to display the setting screen in the LCD display. Use the arrow keys to set the numerical value at the blinking cursor. After setting, press the [OK] key to update the setting value. When the minimum pressure limiter is activated, the whole system stops and an error message appears.

\*"(MAXSETVAL: 00.0 MPa)" displayed in the setting screen indicates the setting value of the maximum pressure limit.

\*For details on how to operate the arrow keys, refer to P.2-17.

\*In case that the pressure limiter is activated to stop the system, refer to "Volume 4 Maintenance Manual" for trouble shooting.

\*The maximum value cannot be less than the minimum value, and they cannot exceed 50.1 MPa.

# **Detector Unit**



• In the [UV DETECTOR] and [RI DETECTOR] units, there are the keys for adjusting the current UV/RI output to O mV and setting the recorder signal range.

<Output Value Display Screen>

FLOW	09.90 ml⁄min	
PRESS	1.0 MPa	
UV	0210	UV output
RI	0035	RI output

In the LCD display, the current UV/RI output is displayed in mV unit.

UV Unit	
1	UV DETECTOR
AUTO ZERO RANGE	SET

• In the UV unit, there are the [AUTO ZERO] key for adjusting the UV output to 0 mV, the [RANGE] key for setting the recorder signal range, and the [SET] key for setting the detector response speed.

<Zero Adjustment Key>



• Press the [AUTO ZERO] key to adjust the current UV output to 0mV. However, when the UV transmittance is too low, the AUTO ZERO function may not work.

<Recorder Signal Range Adjustment>



This function is used for output signal attenuation of pen recorder. Press the [RANGE] key to display the setting screen in the LCD display.
Press the arrows key to increment the signal range (the peak will be shown smaller), or to decrement it (the peak will be shown larger).

\*In case of monitoring the chromatogram with integrator, recorder signal range buttons are without the need for setup.

\*In case a peak-sense (sense threshold and peak height) fraction collector (optional product) is used, the range need to be adjusted.

### <Brightness Check>



 This function is used to perform the brightness check of the lamp installed on the UV detector. Press the [SET] key and select [MENU] to display the output values of [SAMPLE] and [REFERENCE].

\*If the [REFERENCE] value is too low, the lamp should be replaced.

<Setting Detector Response Speed>



This function is used to set the detector response speed. Press the [SET] key and select [PARAMETER] to display the response setting screen. You can select the response speed from the three levels, [SLOW], [MEDIUM], and [FAST]. They mean the time constants of 1.5 sec, 0.5 sec, and 0.05 sec respectively. To change the setting, press the response or respectively.

# **RI Unit**



 In the RI unit, there are the [AUTO ZERO] key for adjusting the RI output to 0 mV, the [RANGE] key for setting the recorder signal range, the [PURGE] key for replacing the solvent in the reference cell, and the keys for selecting the polarity and whether or not to control the temperature.

<Zero Adjustment Key>



- Press the [AUTO ZERO] key to adjust the current RI output to 0mV. When the optical balance is lost too much, the AUTO ZERO function may not work.
- \*If the zero adjustment function does not work, refer to "Volume 4 Maintenance Manual".

<Recorder Signal Range Adjustment>

 Adjust the recorder signal range in the same way as described in "<Recorder Signal Range Adjustment>" of the UV unit. For details, refer to the page 2-22.

### <Starting PURGE>



• This key starts/stops replacing the solvent in the reference cell. Press to change the flow channel to the reference cell for one minute. At the top of the key, the LED is turned off when the fraction is flowing through the sample cell, and turned on when it is flowing through the reference cell.

\*During the [CLEAN UP] operation, the solvent in the reference cell is also replaced automatically. This PURGE function may not be used in general operation.

### <Starting TEMP.C>



• This function is used to select whether or not to use the temperature adjustment function of the RI detector. When the LED is turned on, the temperature controlling heater is turned on to maintain the inside temperature as 35°C for stable operation. Normally this function should be turned on. When you use a low boiling solvent, you can press the [TEMP C] key to turn it off.

### <Selecting SIGNAL POLARITY>



 There are cases that current RI output is shown in negative, when the solvent with high index of refraction is used for mobile phase. This key can be used to reverse the negative signal to positive. When the LED is turned on, the signal is output as it is. Press the [POLARITY] key to turn the LED off and reverse the polarity from negative to positive.

### **FRACTION Unit**



 In the [FRACTION] unit, there are the keys for selecting the method of collecting fractions (siphon or fraction nozzle) and setting the dead volume.

### <Setting DEAD VOLUME>



•You can use this key to select the detector that detects the peak for the basis of collecting fractions. Press the or arrow key to select a detector. The time difference is calculated as dead volume from the selected detector to a fraction nozzle. If you select [ELSE], you can set an arbitrary value when pipe lines are extended or other detectors are installed. To set an arbitrary value, press and select [ELSE] to display the setting screen. Use the arrow keys to set the numerical value at the blinking cursor.

\*For details on how to operate the arrow keys, refer to P.2-17.

- \*The default value is preset in case our optional products (UV-254 NEXT,
- UV-370 NEXT, or UV-600 NEXT for UV detector, and RI-700 NEXT for RI detector) are installed.

\*[ELSE] can be used when other detectors or non-standard-equipped fraction collectors are installed.

### <Siphon/Fraction Nozzle Selection Key>



• You can use this key to collect the fractions of eluent from the detector by using the siphon or the fraction nozzle. The LED is turned off for the eluent from the siphon, and turned on for the eluent from the fraction nozzle. To collect fractions with the fraction nozzle, press <sup>err</sup> when the LED is off. Then the LED blinks, which indicates that the fraction is passing through the dead volume specified by [DEAD VOLUME SET]. After passing through the dead volume, the eluent will flow from the fraction nozzle. If you press <sup>err</sup> again, the LED blinks in the same way, and the eluent passes through the dead volume and then flows from the siphon.

# **RECYCLE Unit**



 This is the control unit of the recycle valve. You can use the key to start/stop the [RECYCLE] operation.

### <Starting RECYCLE>

•Press on to start the [RECYCLE] operation, and press again to stop the operation. •••••• At the top of the key, the LED is turned off when the [RECYCLE] operation is stopped (normal flow channel), and turned on when the [RECYCLE] operation is running.

# **CLEAN UP**

Press the [CLEAN UP] key at the lower right of the operation panel to execute the flow line cleaning automatically.



# LCD display during CLEAN UP operation



In the LCD display, there are the two sections; one indicates the place where the [CLEAN UP] operation is being executed, and the other the operation progress.

<STATUS Display>

The upper section of the LCD display shows the place where the [CLEAN UP] operation is being executed. For information on what the display indication means, refer to the following table:

Display indication	Where to clean
1.PUMP PURGE	Solvent filter to Pump head
2. BUFFER CLEANING	Buffer volume
3.SAMPLE LOOP CLEANING	Manual injector sample loop
4.VALVE CLEANING	Recycling valve
5.RI PURGE	RI reference cell
6.FINISH CLEANUP PROGRAM	

# <CLEAN UP Progress>

SAMPLE	LOOP	CLEANING
	50%	%
START		END

The lower section of the LCD display shows the progress of the [CLEAN UP] operation. 100% means the completion of the operation.

# <Emergency Stop>

If you press the [CLEAN UP] key during the [CLEAN UP] operation, the LED is turned off, and the operation is stopped forcibly. Please press the key only in emergency situation. After the emergency stop, the display returns to the normal display.

# <How to Operate>

When you press the [CLEAN UP] key, the operation procedures are displayed in the LCD display. Please follow the instruction. When a manual operation is required or when you need to verify settings, a message appears in the LCD display with an intermittent beep. The [CLEAN UP] operation ends in 10 to 15 minutes. After the completion, turn the column selector valve back to its original position ([COLUMN]) to use it.

<How to Use Solvent Suction Attachment>

In the LC-92XX NEXT, you must inhale the solvent by using a solvent suction attachment (standard accessory) at the preparation for the [CLEAN UP] operation.



Solvent Suction Attachment

### <How to Use>

CLEAN UP preparation screen





- When you press the [CLEAN UP] key, the operation procedures 1 - 5 are displayed in the LCD display. When you check and implement the items 1 to 4, then the item 5 instructs you to inhale the solvent.
- Set the solvent suction attachment to the pump drain out inside of the siphon box.
- 3. Set the luer-lock syringe to the solvent suction attachment.
- Pull the syringe to inhale the solvent (at least 10 mL of solvent must be inhaled).
- 5. After inhaling the solvent, remove the solvent suction attachment (pull out it while pushing the metal part located at the top of the pump drain out), and then press the [OK] key to start the [CLEAN UP] operation.

# 2-3

# Chapter 3 Recycling Separation

Description of recycling separation, setting of recycling condition parameter

# **Recycling Separation**

The recycling system is a standard specification of the LC-92XX NEXT series.

The recycling system is the feature that has the column eluent flow into column inlet again.

If the separation is insufficient in normal HPLC separation, the change is required in column (separation mode, length, particle size of packing) and/or solvent condition (solvent polarity, gradient curve, salt concentration).

Recycling system achieves hi-separation without change of such column condition or solvent condition as the HPLC case.

Insufficient separation area is introduced into the same column once more, and sufficient separation achieves.

# Relation between Peak Separation and the number of Recycling

In stead of using multiple columns, equivalent separation executes with the Recycling separation. As shown below, separation is multiplied according the numbers of the recycling.

MA .....

Recycling separation: COLUMN: JAIGEL-1H -F+JAIGEL-2H-F FROW RATE: 3.5 ml/min SAMPLE: Polystyrene oligomer (Mw. 495, Mw/Mn 1.20) Separation efficiency with n times Recycling is shown following formula.



To take a separation such as the figure, Rn+1 $\ge$ 1.5 is need. \*Peak width "W" with n times of recycling spreads to W(n+1)= {(n+1) W<sup>2</sup>+nW<sub>E</sub><sup>2</sup>}<sup>1/2</sup>.

# Note of good Recycling Separation

Recycling separation may have the following troubles. Before the real Recycling, pretest is recommended for the chromatogram acquisition without Recycling.

When the Recycling area is too wide or the Recycle times are too much, re-mixture trouble may occur. The top of recycled area will mix with the tail of the recycled area. Recycling area and times should be suitable value.



<Overlap Trouble>

If the Recycling area is set too fast retention time, 2nd cycle peak get past the 1st peak (symptom 1) or 2nd cycle peak overlaps with the later peak (symptom 2). Pretest is recommended for the chromatogram acquisition without Recycling to avoid overlap troubles.



### <Contamination Trouble>

The LC-92XX NEXT series standard specification has the backflow prevention system. The backflow prevention system works to reduce contamination during the recycle operation for good recycle separation. The backflow prevention system has a buffer-volume. The buffer-volume is cleaned during CLEAN UP operation. When the CLEAN UP is neglected or high-viscosity sample/low soluble sample is recycled, shock peak may appear at change over of the Recycling valve. When the shock peak appears, do CLEAN UP operation again. If the symptom cannot be remedied, cleaning-maintenance is required. Please refer to Volume 4 "Maintenance Manual".

Recycle Chromatogram with contaminations.



Recycle Chromatogram after CLEAN-UP



# For good Recycling efficiency

During Recycling separation, recycled sample flows unique flow line that the normal HPLC is not used, rear flow of the Detector, the Pump, and the Injector flow line.

Select suitable instrument and turn over the knob of the MANUAL INJECTOR to LOAD.

<Select Suitable Instrument>

In the LC-92XX NEXT series, the LC-9210 NEXT and LC-9225 NEXT are selected according to the Column size.

Double plunger reciprocal style pump is applied to the LC-92XX NEXT series. For the reduction of the diffusion, select suitable LC according Column size.

Sample diffusion in the plunger room can be ignored when the Column inside volume is enough larger than the plunger volume.



Plunger It moves frontward and backward and solvent is pressurized. The volume differs depending on the specification.

# Structure of the Plunger

# Table of Suitable Column Size $\phi$ for Recycling Separation

	LC	
Separation mode	LC-9210 NEXT	LC-9225 NEXT
SEC (Size exclusion	10% < column id < $20%$	10φ≤ column id ≤ 50φ
chromatograph)	$10\phi \le column$ id $\le 30\phi$	
Partition/Adsorption/Other mode	$10\phi \le column \ id \le 20\phi$	10φ≤ column id ≤ 30φ

		LC
Туре	LC-9210 NEXT	LC-9225 NEXT
Plunger volume	32 µL x 2	77µL x 2

<Turn Over the Knob of the MANUAL INJECTOR to LOAD> The LC-9210 NEXT has a 3 mL-Sample loop, and the LC-9225 NEXT has a 10 mL-Sample loop as a standard.

When multiple times Recycling is done, turn over the knob to LOAD to eliminate the sample loop from the flow line because the recycled sample diffuse in the sample loop.

Wait the manual injector knob at the [INJECT] position while injecting the sample, cleaning the needle port, and filling the sample loop with the new solvent. After that, turn the manual injector knob to the [LOAD] position.

When the pump flow rate of the LC-9210 NEXT is set at 3.0 mL/min or more (10 mL/min or more for the pump flow rate of the LC-9130 NEXT), wait for one minute after the injection, and then turn the manual injector knob to the [LOAD] position to eliminate the sample loop from the flow line.



# 2-4

# Chapter 4 Condition Setting

How to set conditions of LC-92XX NEXT series

# **Setting Operation**

# **Pump setting**

Set the pump flow rate and the pump pressure limit in the [PUMP] unit.



# <Setting Pump Flow Rate>

- Press the [FLOW SET] key.
- •The parameter to be changed blinks on the LCD display.
- •Set the flow rate for the column.

# <Setting Pump Pressure Limits>

- Press the [MAXIMUM SET] key].
- •The parameter to be changed blinks on the LCD display.
- Set the value at which the pressure maximum limiter works.

The pressure maximum limiter stops the pump when the pump pressure rises over the limit, so as to prevent a column and detector cell damage from the over pressure coming from injection of dust and precipitated insoluble in a column. An error message appears when the limiter works.

- •Press the [MINIMUM SET] key.
- The parameter to be changed blinks on the LCD display.
- •Set the value at which the pressure minimum limiter works.

The pressure minimum limiter stops the pump when the pump pressure drops below the limit, so as to prevent a column damage or solvent spill when the solvent loses or leakage of system occurs. An error message appears when the limiter works.

# <Limit of Setting Value>

The setting values of flow rate and pressure limits is limited in accordance with the LC model and the set flow rate.

Refer to the following table. When the setting value is over the limitation, an error message appears.

Flow Rate	LC-9210 NEXT
$\leq$ 10.0 mL/min	≤ 25.0 MPa

Flow Rate	LC-9225 NEXT
$\leq$ 9.9 mL/min	$\leq$ 20.0 MPa
≤ 19.9 mL/min	≤ 15.0 MPa
$\leq$ 25.0 mL/min	≤ 10.0 MPa

The flow rate can be set in the ranges of 0.1 to 10.0 mL/min and 0.1 to 30.0 mL/min for LC-9210 NEXT and LC-9225 NEXT, respectively. To stop a pressure limiter, set [0 MPa].

### Caution

When the Pump is running, the flow rate cannot be changed. Stop the Pump and reset the flow rate. When the pump flow rate is changed, change the [MAXIMUM] pressure limit to "monitoring pressure + 2.0 MPa" or more, and the [MINIMUM] pressure limit to "monitoring pressure - 2.0 MPa" or less.

# **Setting UV detector**

In the [UV] unit, the UV-370 NEXT, UV-254 NEXT, or UV-600 NEXT function can be set.



<Recorder Signal Range Adjustment>

This function is used for output signal attenuation of pen recorder. Press the [RANGE] key to display the setting screen in the LCD display. Press the arrows key to increment the signal range (the peak will be shown smaller), or to decrement it (the peak will be shown larger).

\*In case of monitoring the chromatogram with integrator, recorder signal range buttons are without the need for setup.

\*In case a peak-sense (sense threshold and peak height) fraction collector (optional product) is used, the range need to be adjusted.


- •Remove the locking pin of wave-length selector inside the front door.
- •Turn the wave-length selector to set the wave-length.
- •Tighten the locking pin.
- ·Close to the front door.
- Press the [AUTO ZERO] key in [UV DETECTOR].



\*Setting method of UV-370 NEXT is above mentioned. Model UV-254 NEXT detector uses only one wave-length and not selected wave length. \*Select the wave-length at where using solvent has low wavelength absorption. 0 adjustment may not do at the wave-length where the solvent has high absorption. Please refer P.2-51, for the typical solvent UV cut-off wavelength.

\*Setting cut filter (Only UV-600 NEXT)

Wave-length	Cut filter
199 nm ~ 399 nm	Push
400 nm ~ 600 nm	Pull

#### **Setting RI detector**

In the [RI] unit, the RI-700 NEXT function can be set.



<Recorder Signal Range Adjustment>

This function is used for output signal attenuation of pen recorder. Press the [RANGE] key to display the setting screen in the LCD display.
 Press the arrows key to increment the signal range (the peak will be shown smaller), or to decrement it (the peak will be shown larger).

\*In case of monitoring the chromatogram with integrator, recorder signal range buttons are without the need for setup.

\*In case a peak-sense (sense threshold and peak height) fraction collector (optional product) is used, the range need to be adjusted.

<Setting SIGNAL POLARITY>

- •When the LED at the top of [POLARITY] is turned on, the output has the same polarity as the original signal. Press the [POLARITY] key to turn the LED off and reverse the polarity of the output signal.
- \*Reverse polarity is profitable when high refractive index solvent like toluene is used for eluent.

\*Please refer P.2-51, for the typical solvent refractive index.

#### <Setting TEMP C>

- •When the LED is turned on, the temperature controlling heater of the detector is on to maintain the temperature at 35°C. Press the [TEMP C] key to turn the LED off, which indicates that the temperature is not controlled.
- \*When the LED is turned on, more stable RI signal is obtained. Normally, the LED should be turned on.
- \*Using low boiling point solvent, turn the LED off to suppress bubbling of the solvent. \*Please refer P.2-51, for the typical solvent boiling point.

#### **Setting fraction**

In the [FRACTION] unit, the dead volume can be set.



<Setting DEAD VOLUME>

- •Press the [DEAD VOL SET] key.
- Selecting [UV], set the preset dead volume between UV-370 NEXT, UV-254 NEXT, or UV-600 NEXT, RI-700 NEXT, and Nozzle of Fraction Collector.
- Selecting [RI], set the preset dead volume between RI-700 and Nozzle of Fraction Collector.
- •When the external detector is used, or when the fraction collector is removed, select [ELSE] to set the dead volume.
- The specified dead volume is used to automatically calculate the dead volume flow time when the valve to be used is switched between the fraction nozzle and the siphon.
- \*The default value is preset in case our optional products (UV-254 NEXT, UV-370 NEXT, or UV-600 NEXT for UV detector, and RI-700 NEXT for RI detector) are installed.
- \*If [0.0] value is selected, the dead volume (delay time) is neglected. Immediately after pressing the [Co/Dr] key, the valve to be used is switched between the fraction nozzle and the siphon.
- \*When the [RECYCLE] operation is switched to ON/OFF, the dead volume (delay time) is neglected. Immediately after pressing the [RECYCLE] key, the valve is switched.

# 2-5

# Chapter 5 Appendix

Degassing, property of typical organic solvent, elimination of micro particles

#### Degassing

Solvent to use an eluent in the LC-92XX should be degassed. Pressure fluctuation of the peak retention time or the detector noise may occur with the babble in undegassed solvent. To result best chromatograph without noise and fluctuation, the solvent to use an eluent should be degassed.

<Degassing with ON-LINE DEGASSER>

- The ON-LINE DEGASSER should be connected between the solvent container and LC-92XX NEXT at the solvent supplying connector.
- •Read the ON-LINE DEGASSER instruction and according to the instruction, connect the ON-LINE DEGASSER to the LC-92XX NEXT.
- According to the instruction, turn the power switch on to make sure that the AC voltage is suited to the DEGASSER.

Be care that the DEGASSER is chemical proof against the solvent according to the instruction.

Strong organic solvent is sometime highly corrosive to the DEGASSER. In case the solvent which is corrosive to the DEGASSER, do degassing with an Ultra sonic cleaner and or vacuum pump described below.

In water solvent operation, the degassing may not be sufficient by the ON-LINE DEGASSER due to the flow rate and laboratory temperature environment.

Degassing with an Ultra sonic cleaner and or vacuum pump is also recommended.

The above method is a typical connecting method.

The connecting method may differ depending on the type of degasser or pipe line handling because these factors may cause a different filtering resistance.

Make sure to apply the suitable connection without much flow resistance according to the instruction.

<Degassing with Ultra Sonic Cleaner>



- Move the solvent into the container. In case of mixture solvent, stir the solvent thoroughly.
  Without stirring the mixture solvent, the reproducibility of the chromatogram may be lost, because the lost of the component may occurs.
- Solvent such as sealed containers without lids, and a lid on things such as Kleenex for good ventilation.

If the seal is inflated when the mixing temperature ultrasonic, solvent containers may be destroyed.

- Dip the container in the Ultra sonic cleaner. Ultra sonic cleaner should be half full with water.
- •Operate an ultra sonic cleaner in 10 to 15 min. When there is no bubbling, degassing is complete.

When bubbles stuck on the wall of solvent container, blow N2 or He2 gas in the solvent easy to remove the bubbles. After blowing, operate an ultra sonic cleaner in few minutes.

In case of the solvent mixture, keep in short time operation of the ultra sonic cleaner less than 30 min.

Vaporization of the solvent to change composition of the mixture occurs in long operation of the ultra sonic cleaner.

•Wipe the drops out of the container and place the container on the solvent stage in the LC-92XX NEXT.

Dissolvent of the air into the solvent occurs soon after degassing. Do degassing just before use the solvent.

<Degassing with Ultra Sonic Cleaner Plus Vacuum>



- Move the solvent into the container. In case of mixture solvent, stir the solvent thoroughly.
  Without stirring the mixture solvent, the reproducibility of the chromatogram may be lost, because the lost of the component may occurs.
  The solvent container must be vacuum tight. A conical beaker or conical flask may be damaged at decompression.
- Vapor from the solvent contains organic vapor. To prevent spill of the organic vapor to swage, use closed system aspirator to make vacuum.
   Waste water should be disposed properly according to laws and regulations in the same manner as organic solvents.
- Place the container in the ultra sonic cleaner, and seal it with a rubber stopper that is fitted into the container mouth. Set a vacuum line to the rubber stopper, and connect its one end to the aspirator.
- Perform the ultra sonic cleaning, and operate the aspirator to reduce the pressure of the container. Operate both the ultra sonic cleaner and a water aspirator in 5 min.

Solvent component will be change while the vacuum-filtration. Keep the condition of the filtration

time and vacuum condition. Keep the vacuum degree and time in constant.

- •Wipe the drops out of the container and place the container on the solvent stage in the LC-92XX NEXT.
- Dissolvent of the air into the solvent occurs soon after degassing. Do degassing just before use the solvent.

<20	Jiventi	roper	ty LIS	ι>							
Solvent	ε°(Al2O3)	Viscosity (mPa s; 20°C)	UV limit to use (nm)	Refractive index (20°C)	Boiling point (°C)	Solvent	ε°(Al2O3)	Viscosity (mPa s; 20°C)	UV limit to use (nm)	Refractive index (20°C)	Boiling point (°C)
Pentane	0.00	0.24	200	1.357	36.1	Acetone	0.56	0.30	330	1.359	56.3
Hexane	0.01	0.31	200	1.375	68.7	Methyl ethyl ketone	0.51	0.42	329	1.379	79.6
Heptane	0.01	0.42	200	1.388	98.4	iso-Butyl methyl ketone	-	0.54	334	1.396	116.5
2,2,4-triMethylpentane	0.01	0.50	215	1.391	99.2	Diethyl ether	0.38	0.24	218	1.352	34.6
Nonane	-	0.72	200	1.405	150.8	Diisopropyl ether	0.28	0.37	220	1.369	69.0
decane	0.04	0.92	210	1.412	174.1	Tetrahydrofuran	0.45	0.55	212	1.407	66.0
Cycropentane	0.05	0.47	210	1.409	49.3	1,4-Dioxane	0.56	1.44	215	1.442	101.3
Cycrohexeane	0.04	0.98	200	1.426	80.7	Propylene carbonate	-	-	-	1.419	240.0
Benzene	0.32	0.65	278	1.501	80.1	Methanol	0.95	0.55	205	1.328	64.7
Toluene	0.29	0.59	284	1.497	110.6	Ethanol	0.88	1.20	210	1.361	78.3
o-Xylene	0.26	0.81	288	1.505	144.4	1-Propanol	0.82	2.26	210	1.386	97.2
m- Xylene	0.26	-	290	1.496	138.5	2-Propanol	0.82	2.86	205	1.377	82.3
Dichloromethane	0.42	0.45	233	1.424	39.8	2-Methoxyethanol	-	1.72	210	1.402	124.6
Chloroform	0.40	0.58	245	1.446	61.2	1-Butanol	-	2.95	215	1.399	117.7
Carbon tetrachloride	0.18	0.97	263	1.460	76.8	2-Butanol	-	4.21	260	1.397	99.6
1,2-Dichloroethane	0.49	0.79	230	1.445	83.4	Isobutanol	-	4.70	200	1.396	107.7
Trichloroethylene	-	0.57	273	1.477	87.2	2- Ethoxyethanol	-	2.05	210	1.408	135.6
Tetrachloroethene	-	0.93	295	1.506	121.2	Acetonitrile	0.65	0.34	190	1.344	81.6
n-Butyl chloride	-	0.47	220	1.402	78.4	Diethylamine	0.63	0.38	275	1.387	56.0
Chlorobenzene	0.30	0.80	287	1.525	131.7	N,N-Dimethylformamide	-	0.92	268	1.430	153.0
o-Dichlorobenzene	-	1.32	295	1.551	180.5	N, N-Dimethylacetamide	-	2.14	268	1.438	166.1
Disulfide	0.15	0.37	380	1.626	46.3	Pyridine	0.71	0.95	330	1.510	115.3
Methyl acetate	0.60	0.37	260	1.362	56.3	N- Methyl -2-Pyrrolidone	-	1.67	285	1.488	202.0
Ethyl acetate	0.58	0.46	256	1.372	77.1	Dimethylsulfoxide	0.62	2.20	286	1.478	189.0
Butyl acetate	-	0.73	254	1.394	126.1	acetic acid	大	1.31	-	1.372	117.9
Methoxyethyl acetate	-	-	254	1.402	144.5	Water	大	1.00	-	1.333	100.0

#### Solvent Property Lists

Property of typical organic solvent





Replace solvent for a column should be easy soluble chemical against the solvent pre-used. Make sure that a solubility is good each other. When changing solvents are poor solubility each other, select the intermediate solvent which has good solubility to each other.

In case salt contained solvent, the intermediate solvent which has a good solubility to salt, then replace the final solvent.

#### **Micro particle elimination**

To come in Micro particles such as dust and insoluble, the LC-09XX and the column may be damaged. Such micro particles should be eliminated before the application.

<Elimination of Micro Particles in Solvent>



- •With Membrane filter of 0.45µm, filtrate the solvent using aspirator.
- Vapor from the filtering flask contain organic solvent. To prevent spill of the organic vapor to swage, use closed system aspirator to make vacuum.
  Waste water should be disposed properly according to laws and regulations in the same manner as organic solvents.

In case clear appearance, some time the solvent contain micro particles, so make sure to eliminate the micro particles.

Solvent component will be change while the vacuum-filtration. Keep the condition of the filtration time and vacuum condition. Keep the vacuum degree and time in constant.

<Micro Particle Elimination in Sample Solution>



The sample may contain the micro particles. Make sure to eliminate the micro particles.

#### Needle port cleaning

The overflowed sample solution may contaminate the needle port of the injector when injecting the sample with the manual injector.

After injecting the sample with the manual injector, be sure to rinse and clean the needle port described as follows.



- •Remove the Syringe Needle from the syringe and attach Needle Port Guide.
- Suck pure solvent in the syringe and clamp the needle port guide on the needle port of the manual injector.
- Set the manual injector knob at the [INJECT] position to perform needle port cleaning.
- •Repeat 3 times this cleaning.

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## Volume 3 Installation Manual

Check list of accessories, inspection sheet and shipping data

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## Preface

This manual is for JAI staff to install the LC-92XX NEXT series properly.

Please be advised that installation and relocation by users are not suggested.

The installation and relocation by users self are unwarrantable.

For more details of operation, information, and maintenance of the instrument, please refer to other volumes, "Daily Operation Manual", "General Information Manual", and "Maintenance Manual" on the tabs.

There are checklist of accessories, shipping data and inspection sheet attached at the end of this chapter.



# Chapter 1 Place to Setup

Preparation to set up LC-92XX NEXT series

## **Installation Location**

Place the LC-92XX NEXT series at the location and environment that satisfy the following conditions for correct operation.

#### Installation Location

The LC-92XX NEXT requires space at least more than 500 mm (W) x 600 mm (H) x 600 mm (D). Depth may need 750mm for an air vent and power supply parts at the back of the body. For left and right of the body, may need extra 100mm each for enough space.

The table to place the body has to be horizontally stable and capable to hold the weight of the body.

Please prepare an enough space for optional accessories and attachments as well.

#### Environment

Indoor temperature must be between at 4 to 35 degree (c) with no dew condensation.

•Must not have temperature changes during operation.

Indoor humidity must be within 25 to 85%.

- ·Must be well ventilated.
- •Must not produce corrosive gas nor be filled with it.
- Should not be exposed to direct sunlight and direct wind.
- Should be supplying the same working voltage without sudden change.
- •Must not give tremor and or shock.
- •Must not have strong magnetic field interference.
- ·Should avoid dusts around.

## **Installation Conditions**

Place the LC-92XX NEXT series at the location that satisfies the following installation conditions for correct operation.

Power voltage:	AC 100V to 240V (within 10% variation voltage)
Frequency:	50Hz or 60Hz
Power supply:	Requires 500VA at minimum and 800VA at maximum.
	For additional business machines, prepare enough power supply.
Electrical sockets:	It has a 3 pins plug with a ground connection (Type A). In case of none ground connection socket or different type of plug, please be advised to use an extension and/or adapter to make it 2 pins plug and connect ground terminal.



# Chapter 2 Pipe Lines/Wiring Connections

Pipe lines/wiring connections of the LC-92XX NEXT series

## **Serial Number Check**

<Serial Numbers>

 $\cdot$  Check out the serial numbers of the body and detectors.

## **Pipe Line Connections**

Follow the instructions to connect the pipes appropriately to use the LC-92XX NEXT series.

Install the detector(s).>

Front Door
Install the detector(s).>
Install the detector(s)...
Install the dete

• Pull the front door to open.

Drain Valve

• Take out the plastic bag from inside of the body and unscrew all the seal plugs.

3. RI Detector Tray

4. RI Screws

- Draw out the UV detector tray.
- Place an UV detector on the tray and retain the UV detector by the UV screws.
- Push back the UV Detector tray into the body thoroughly.
- Draw out the RI detector tray.
- Place a RI Detector on the tray. Try to avoid the drain valve.
- Retain the RI Detector on the tray by the RI screws.
- Push back the UV Detector tray into the body thoroughly; make sure the other pipe lines.

#### <Pipe Connection>

<A line: Pump out tee joint - Pump drain valve>



Connect the A pipe between the tee joint of the pump out and the pump drain valve.

<B Line: Pump drain valve - FROM PUMP union>

FROM PUMP union

TO UV union



Connect the B pipe between the FROM PUMP union connector and the right side of the drain valve.

Pump Drain Valve

<UV detector- TO UV union>



Connect a pipe between the UV detector cell (the inlet port / lower part) and the TO UV union.



Pipe lines set up view

<UV detector cell (up part) – UV-RI union (IN)>

<C line: UV-RI union - RI detector (IN)>



- Connect a pipe line between the UV detector cell (up part) and the UV-RI union.
- Connect the C pipe between the UV-RI union (lower part) and the RI detector (IN).

<D line: Valve union behind drain valve - RI (out) >

<E line: Manual injector drain port - Manual injector>

<F line: Pump drain valve - Pump drain port>



- Connect the D pipe line between the upper part of the valve union behind the drain valve and the RI (out).
- Connect the E pipe line between the manual injector and the manual injector drain port of the siphon box.
- Connect the F pipe from underneath the pump drain valve to the pump drain port of the siphon box.

Pump Drain Port

(Behind the Pump Drain Valve)



Pipe lines set up view

<Solvent supply port – solvent suction line (with line filter)>



- <Total waste a waste reservoir>
  - Connect the solvent suction line (with line filter) to the Solvent supply port.

Connect a tube from underneath of the siphon box into a waste reservoir.

## Wiring Connections

Follow the instructions to connect the wiring appropriately to use the LC-92XX NEXT series.

<Main Body to AC Power>



•Put the power cable into [AC INLET 100  $\sim$  240 V] at the back of the body.

#### <Main Body to Integrator (or Recorder)>



#### Top of Output Terminal Block



- Connect each signal cable to the corresponding output port as shown in the above table, by referring the description below.
- To connect, use a flat head screwdriver and put it into a wire release hole. Insert a cable into a wire insert hole.
- Remove the screwdriver from the wire release hole and make sure that the wire end is locked tight in the wire insert hole.

\*Minus signals at recycle signal(12), collect valve(14), and siphon counter signal(16) are the same. In use of an electronic device such as JDS series to read several signals at once, joint the outputs with a jumper wire and connect as one output port.

<ul> <li>Connect an integrator (or a recorder) signal</li> </ul>	
cable to the output port (16 terminals).	

Port No	Description
1 011110.	
1	UV +Output for an Integrator
2	UV -Output for an integrator
3	RI +Output for an integrator
4	RI -Output for an integrator
5	UV +Output for a recorder
6	UV -Output for a recorder
7	RI +Output for a recorder
8	RI -Output for a recorder
9	START +
10	START -
11	Recycle signal +
12	Recycle signal -
13	Collect valve +
14	Collect Valve -
15	Siphon counter +
16	Siphon counter -

<Connect to JDS Series (Sold as optional accessory)>

<Connect to a JDS signal cable>



- •Connect the cable between the D subminiature connector and the JDS signal inputs [H] and [L].
- In use of 2 different detectors, connect another signal inputs [H] and [L] to output port + (3) and – (4) as well.

(The left image shows only the UV detector.)

<Connect to JDS 25P D sub-miniature connector>

Insert JDS 25P D sub-miniature connector to the JDS.

Top of Output Terminal Block



Connect the signal cables to the output ports of the LC-92XX NEXT series according to the numbers printed on the mark tubes.

·Check all cable connections.

\*Each signal cable is attached with a mark tube on which the number is printed.

Check Cable Connection	
Cable connection is completed	



# Chapter 3 Validation after Installation

LC-92XX NEXT series validation after the installation

## Preparation for the validation

The items listed below are required to validate that the LC-92XX NEXT series is installed correctly. \* 1 items are standard equipped. \* 2 items are prepared by JAI staff. Those without \*1 or \*2 should be prepared by customer side.

Upon customer's request, we can execute validation in a different way, after mutual agreement instead of the following standard validation.

- Solvent: Chloroform or Methanol 500ml or more.
- Waste container or bottle 3L capacity or bigger.
- A beaker 100 ml sized or bigger.
- Sample vial 2 pcs.
- Toluene Small amount
- An integrator or a recorder. (In case of purchasing our system with NEXT seriese, JAI staff will set it up at once.)
- \*1 Hamilton syringe 1 pc.
- •\*1 Luer lock syringe 1 pc.
- \*1 8 mm wrench spanner 1 pc.
- •\*2 A dummy pressure sensor 1 pc.

## Sample for Validation

Prepare the validation sample for the LC-92XX NEXT series.

- Measure 1.3ml toluene with a hamilton syringe.
- 0.3ml toluene out of 1.3ml to a sample vial with hamilton syringe.
- Prepare chloroform or methanol in a beaker.
- Take out chloroform or methanol from the beaker by hamilton syringe. 20ml of chloroform or methanol to the sample vial and dilute the toluene.

## Pipe Line and Start Cable Test

Check the pipe line connection and the start cable connection of the LC-92XX NEXT series.

- •Remove the column cover from the right side of the instrument, and connect a dummy pressure sensor between [TO COLUMN] and [FROM COLUMN] using a wrench.
- Dip the solvent filter into methanol or chloroform. Place the solvent container on Solvent table located upper shelf of LC-92XX NEXT series.
- •Refer to "Chapter 2 General Operation" in "Volume 1 Daily Operation Manual" to turn the power on and execute the [CLEAN UP] operation.
- •When [PUMP PURGE] is displayed on the screen during the [CLEAN UP] operation, check the solvent out from the pump drain out in the repeat injector box. In the LC-92XX NEXT, you must inhale the solvent by using a solvent suction attachment at the preparation for the [CLEAN UP] operation.

#### Settings

	Settings
Flow Rate	9.0 mL/min
Pressure Limiter	[MAXIMUM] 0 [MINIMUM] 0
Injector	3 ml (3 times injection)
Detector UV*1	254 nm In case of pen recorder, RECORDER SIGNAL RANGE x 1
Detector RI	TEMP. CONTROL ON (LED is ON) SIGNAL POLARITY POSITIVE (LED is ON) In case of pen recorder ,RECORDER SIGNAL RANGE (Methanol x 1, Chloroform x 4)

\*1 This is the case of the optical pass length of 0.5 mm. With other optical pass length, adjust the concentration of sample accordingly.

Start Pump.

• Make sure that the pump pressure is stable.

Check Column Pressure Stability	
Column pressure is stable.	

• Refer to the page 1-8 at Volume 1 to perform the zero adjustment of the detector.

•Set up the integrator or recorder.

- Set the acquisition time to 1.5 min with the integrator.
- Refer to the page 1-10 at Volume 1 to inject the sample.
- Check no leakage from flow line inside of the front door, connectors of fraction collector and connectors of dummy pressure sensor during the chromatogram acquisition.

Leak Check	
Inside of the front door	
Connectors of dummy pressure sensor	

•Repeat the operation three times.

•When using the integrator, check whether 3 chromatograms are obtained, and whether the peaks are similar in visual. When using the recorder, check that three peaks are obtained and that they are similar in visual.

Check Chromatogram	
Three chromatograms or peaks are obtained	
Peaks are similar in visual	

•Press the [ON/OFF] key in [PUMP] to stop the pump.

## Wiring Connections and Valve Validation

Check the wiring connections and valve operation of the LC-92XX NEXT series.

Refer to "Volume 1 Daily O	peration Manual" to enter the following values:
	Settings
Flow Rate	9.0 mL/min
Pressure Limiter	[MAXIMUM] 0
	[MINIMUM] 0
Injector	SAMPLE 1 ml
Detector UV*1	254 nm
	In case of pen recorder, RECORDER SIGNAL RANGE x 4
Detector RI	TEMP. CONTROL ON
	SIGNAL POLARITY POSITIVE
	In case of pen recorder, RECORDER SIGNAL RANGE (Methanol
	x 4 Chloroform x 8)

- Turn on the pump.
- Refer to the page 1-8 to perform the zero adjustment of the detector.
- Set the acquisition time to 30 min with the integrator.
- Refer to the page 1-10 at Volume 1 to inject the sample.
- 10 seconds after the start, move the manual sample injector to the [LOAD] position, and press in [RECYCLE] to monitor the chromatogram.
- · Make sure that peak is in recycling.

Check of Recycling Chromatogram		
Peak is recycling		
[R] mark appears on the Chromatogram (JDS acquisition)		

• After checking the recycling peak, press • In [RECYCLE] to stop the [RECYCLE] operation. Then press • In [FRACTION] and check that the eluent is flowing from the fraction nozzle.

Check Collect Chromatogram		
Check the collect valve		
[C1] mark appears on the Chromatogram (JDS acquisition)		

• After checking the collect valve, press in [FRACTION] and check that the eluent stops running from the fraction nozzle.

Check Collect Chromatogram		
Check the collect valve		
[D1] mark appears on the Chromatogram (JDS acquisition)		

• Press the [ON/OFF] key in [PUMP] to stop the pump.


### Volume 4 Maintenance Manual

Maintenance and trouble shooting of LC-92XX NEXT series (except for optional parts)

## Contents

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## Preface

Fundamental maintenance and trouble shooting of LC-92XX NEXT series are described in this manual.

Refer to the paragraph for your successfully operation. Prevent solvent leak and electrical shock during the maintenance.

Normally cut off the AC power. Be careful of electrical shock during the maintenance when power switch turn on.



# Chapter 1 Maintenance of LC-92XX NEXT

Description of LC-92XX NEXT series maintenance

### **Replacement of Column**

If the separation is required with the different type column, replacement of column is needed.

Surely stop the pump during the replacement of column to prevent solvent spill.

<Installing Column> Install the column as following.

•Turn the column reject valve to the [REJECT] position.

•Remove the column cover of the LC-92XX NEXT from the chassis.

 $\cdot$  Hold the column(s) on the column holder inside of the column cover.



- Replace the solvent which is able to use with the replaced column(s), with before used solvent, thoroughly in the flow line of LC-92XX.
   Before the solvent replacement, test the solubility between replaced solvent and before used solvent.
  - Use Auto-cleaning function for the solvent replacement in the LC.
- Connect the pipe on the [TO COLUMN connector]. Do not connect the pipe on the replaced column(s) yet.
- Set the pump flow rate to use with the replaced column(s).

• Cover the outlet of the pipe with a paper towel or thick paper for absorption of the over flowed solvent.

And start the pump for purging air in the pipe. Please make sure to protect the replaced column(s).

If the flow rate is large, solvent may flush and spray. Be careful.



- Once stop pump and remove the seals of the replaced column(s). Connect the pipe on inlet of the replaced column(s).
- The pipe is connected on [TO COLUMN connector]. Keep the flow of the replaced column(s) according arrow indicator [Flow seal] on the column. The flow of the replaced column(s) is important for the column performance. Do not connect the reverse direction.



- When the column is jointed series two or more, the air in the jumper pipe jointed columns also should be purged.
- Joint the jumper pipe on the first column outlet. Do not joint the pipe on the second column inlet on this point.
- Cover the outlet of the jumper pipe with a paper towel or thick paper for absorption of the over flowed solvent. And start the pump for purging air in the pipe. If the flow rate is large, solvent may flush and spray. Be careful.



• Connect the jumper pipe on the second column inlet. Connect the pipe from the column outlet on the [FROM COLUMN connector].



• Make sure that no leakage is found when the pump is started, then close the front door.

Pipes for LC-9210 NEXT		
Parts No.	Name	
911-001	Inlet pipe for Preparative Column	
911-002	Outlet pipe for Preparative Column	
	(twin column)	
911-003	Outlet pipe for Preparative Column	
	(single column)	
911-004	Jumper pipe for Preparative Columns	
Pipes for LC-9225 NEXT		
Parts No.	Name	
913-001	Inlet pipe for Preparative Column	
913-002	Outlet pipe for Preparative Column	
	(twin column)	
913-003	Outlet pipe for Preparative Column	
	(single column)	
913-004	Jumper pipe for Preparative Columns	

<Removing Column> To remove columns, refer to the instructions below.

- Turn the column reject valve to the [REJECT] position.
- Remove the LC-92XX NEXT column cover (see page 4-2).
- Remove the outlet pipe column. When the Column pressure is high, you may have dispersed solvent. Be careful.



• Put the seal plug into the column outlet. Tighten the seal plug 1/4 turns by hand, and then tighten by a wrench. Use a hand for a PEEK-type seal plug. Too strong tightening may damage the screws or column. Be careful.



- Disconnect the column inlet pipe.
- Put the seal plug into the column inlet.

Make sure that the column removed the pipe are tightly sealed without solvent leakage, keep in cool dark place.

- Connect with the U shape pipe between the [TO COLUMN connector] and [FROM COLUMN connector] of the LC that are removed the pipe from the Column.
- Close the column cover.

LC-92XX NEXT common part		
Parts No.	Name	
901-024	Seal plug : for 1.6φ pipe (column seal)	

### **Cleaning of Solvent Filter**

When the pump flow rate is not stable and, when no solvent is supplied at the autocleaning, the Solvent filter may cause clogging of dust or particles.

The Solvent filter is clogged with dirt and dust if the solvent without dust removal is fed to the solvent reservoir.

<Cleaning Solvent filter>

 Open the front door. Remove the solvent filter connected on the solvent supply port.





- Remove the Solvent filter and pipe from the solvent reservoir, put the Solvent filter and pipe into a beaker filling the solvent.
- Do clean for 10 minutes with the beaker dipped into an ultrasonic cleaner.



• Replace the filter pipe on the solvent supply port.

LC-92XX NEXT common part		
Parts No.	Name	
995-017	Solvent supply connector (for NEXT)	
995-018	Gallons bottle pipe (for NEXT)	

#### Maintenance of Manual Injector

After high concentration or low solubility sample injection, the Stator face, and the Rotor seal of the Injector may dirt or precipitate with the remained sample. If you do not wash with the needle port cleaner after the injection the symptom may occur. If solvent start leaking from Manual Injector, or if the ghost peaks appear, Manual Injector may be improved by maintenance. Rotor seal and Stator face should be replaced every year.

<Removing Manual Sample Injector>

- Turn the column reject valve to the [REJECT] position.
- Remove the Solvent filter and pipe from the solvent reservoir.
- Remove the column cover of the LC-92XX NEXT from the chassis.
- Remove pipes marked 2, 3, 5, 6 (marked by RHEODYNE Co.) from the back of a Manual Injector.
- Disconnect the connector of start signal cable.
  1, 4 are connected a Sample loop, and no need of removal during the maintenance.



• Loosen two socket set screws side of the manual injector knob at the front of the LC-92XX NEXT with a hexagonal wrench.



· Remove the manual injector knob.



• Remove retaining screws of the Manual Injector.



• Move apart the Manual Injector from the back of the LC panel.

<Replacing Rotor Seal and Stator Face>

• Remove Stator retaining screws. Remove Rotor seal, Stator ring, Stator face, and Stator face from the Manual Injector body.



- Install New Rotor seal. Place the Rotor seal to fit the tip of the needle port tube into the liquid passing hole, and insert pins into the pin holes. Insert as the mark of RHEODYNE co. on the Rotor seal faced toward Stator face.
- Install Stator face as 3 pins fit on each hales.
- Insert Stator ring into the Manual Injector body.
- Insert pins of Stator ring fit on the grooves.
- Tighten retaining screws of the Manual Injector tightly.

<Installing Manual Sample Injector>

- Place the Manual Injector on the original position at the back of the LC panel, and tighten the Manual Injector by retaining screws.
- Replace and tighten the Manual Injector knob. Check the smooth movement of Sample injection.
- If the movement of the Manual Injector knob is too hard or too loose, readjust the Manual Injector knob. Loosen two socket set screws side of the knob again. Readjust and insert the knob as fit perfectly the key into the groove of the knob.



- Readjust the knob to turn. Appropriately check the movement of the knob and readjust.
- Tighten the retaining socket set screws to install the Manual Injector knob.
- Connect each pipes restored on the back connection port of the Manual Injector.
- Connect the connector of start signal cable.
- Start Pump and flow solvent using Auto-cleaning function, and check that no leakage is appeared at any connector. If the leakage is found, tighten the connector additionally to stop.
- After the Auto-cleaning, Start Pump and flow solvent into the Column to pressurize the system and check that no leakage is appeared at any connector.
  - LC-92XX NEXT common partParts No.Name996-012Variable Injector(7725i)996-014Gauge needle for Variable Injector996-015Rotor seal for Variable Injector996-016Stator face Assembly for Injector
- Close the column cover.

#### **Maintenance of Pump**

The pump has the necessary cleaning supplies and replacement parts, after which period of time.

The pump seal replaced every four months, do cleaning the pump head in same time.

The check valve packing to exchange every six months, please replace with new ones every two years.

Around the time of consumption may be due to the property and frequency of the sample.

<Replacing Pump Seal>

- Turn the column reject valve to the [REJECT] position.
- Remove the Solvent filter and pipe from the solvent reservoir.
- Open the LC-92XX NEXT front door.



- Pump unit is installed in the upper left in the body.
- Disconnect the pipes A, B, C, D connected to the pump unit. For the pipes B and C, remove the pipes while fixing the check valve cases with a wrench.



- Loosen the screw of the pump head with a 2.5 mm hexagonal wrench. Loosen the screws alternately.
- Pull the pump head. Pull horizontally on the front. The tilted pull may damage the plunger. Be careful.



Remove the pump seal G.
 Remove the pump seal G by holding the flange part by hand.
 Remove the backup ring left in the main body of the pump unit by holding it with tweezers (you should also replace the backup ring).



• Loosen the screw of the pump head guide with a 2.5 mm hexagonal wrench. Loosen the screws alternately.





 Remove the pump seal F attached to the pump head guide by using the seal removal jig.



- Clean the pump head. Put Pump head into the beaker. The most frequently used solvent fills the beaker. Make ultrasonic cleaning for 5 minutes. Empty the solvent in the beaker. Fill a beaker of methanol. Make ultrasonic cleaning for 5 minutes. When methanol is non-miscible with used solvent, use ethanol instead of methanol. Wash the Pump head attached the check valve without any problems.
- Also wipe around the plunger with BEMCOT or soft gauze. In severe cases
  of contaminated, moisten the gauze with a strong solvent like as HFIP and
  wipe the Plunger with the moistened gauze.
- Install the pump seal F on the pump head guide by using the seal removal jig.



- Install the pump head guide on the pump unit. Insert the pump head guide horizontally, being careful not to damage the pump seal F with the plunger.
- Tighten the screws of the pump head guide lightly. Tighten two screws on the pump head alternately by 1/4 turns with a 2.5 mm hexagonal wrench to tightly fix it.

Install the pump head guide with its holes facing outward.



- Insert the backup ring into the plunger.
- Install the pump seal G on the pump head.
- Tighten the screws of the pump head lightly. Tighten two screws on the pump head alternately by 1/4 turns with a 2.5 mm hexagonal wrench to tightly fix it. Be careful not to turn upside down.
- Connect the pipes A, B, C, D on the pump head. For the pipes B and C, connect them while fixing the <u>check valve cases with a</u> wrench.

While fixing the check valve case with a 10 mm wrench, tighten the upper screw with a 8 mm wrench.



- Put the Solvent filter into the solvent reservoir. Fill the Pump head with the solvent to use the [AUTO CLEAN] operation.
- Make sure no leakage. Close the front door.

<Cleaning Check Valve, Replacing Check Valve Seal>

- Turn the column reject valve to the [REJECT] position.
- Remove the Solvent filter and pipe from the solvent reservoir.
- Open the LC-92XX NEXT front door.



- Pump unit is installed in the upper left in the body.
- Disconnect the pipes A, B connected to the pump unit. For the pipe B, remove the pipe while fixing the check valve case with a wrench.



• Remove the check valve inlet case and the check valve outlet case from the pump head. Loosen the check valve to turn anti-clockwise. Be careful not to lose inside parts. Remove the check valve outlet to turn anti-clockwise.



• The inside parts of the check valve is shown as following figure. Handle as a unit each as "check valve case", "seat", and "check ball", do not confuse the parts each other. Place each unit separately in the beakers.



- Fill each beakers of methanol. Make ultrasonic cleaning for 5 minutes.
- If dirt remains in the visual, remove the dirt with chloroform, toluene, or HFIP as strong solvent.
- Assemble the check valve. Assemble the check valve as shown below.



• Assemble the check valve inlet. Assemble I part on the on the paper. Cover gently Assemble part I with the check valve inlet case. Turn over gently by pinching the corners of the paper.



 Assemble the check valve outlet. Assemble the check valve II on the paper, holding your finger on the top of the parts, and turn over assembled check valve gently by pinching the corners of the paper. Put the assembled parts gently in the check valve case. Turn over the Cover with the assembled parts.



 Connect the check valve outlet case into the pump head inlet case, and tighten the case with a 10 mm wrench. The gasket B is fixed tightly into the case. Remove the case, and make sure that the check valve gasket B insertion is tight. Then connect the case to the pump head outlet side and tighten it with a 10 mm wrench.



Connect the check valve inlet case into the pump head inlet and tighten by a wrench anchor.



• The pipe B is connected to the pump unit as before. Tighten the screw of pipe B. The Check valve outlet case is fixed by a wrench while the screw is tightening.



- Use Auto-cleaning function for the solvent replacement in the Pump head.
- Make sure that no leakage is found. Close the front door.

Parts of LC-9210 NEXT (FX-10)	
Parts No.	Name
918-312	Pump seal G
918-013	Check valve inlet (Set)
918-014	Check valve outlet (Set)
918-217	Check valve
918-218	Valve seat case
918-219	Check valve seal A
918-220	Check valve seal B
918-011	Plunger
Parts of LC-9225 NEXT (FX-25)	
Parts No.	Name
928-312	Pump seal G
918-013	Check valve inlet (Set)
918-014	Check valve outlet (Set)
918-217	Check valve
918-218	Valve seat case
918-219	Check valve seal A
918-220	Check valve seal B
928-011	Plunger

#### **Pipe Connection**

Attached standard pipe PEEK and Stainless steel pipe is a consumable. Repeated connection and release of Ferrule will be occurred collapse or bite of the screws. Increased dead volume and leakage or damage of the screws may cause. Replace with new ones when the connector repeats three times of lock and release. PEEK pipe has life. If only organic solvent is used, PEEK pipe early decays. Solvent leakage or splash may occur from the decayed PEEK pipe. Please replace a new one a month in continuous operation.



<Pipe Line Connections>

• Insert a Male Screw, a Back ferrule, and a Front ferrule into the pipe.



In case of PEEK pipe, insert only D) Fitting for PEEK pipe into the pipe.

- Insert top of the pipe into a union connector. Pressing the pipe into the bottom of the connector, turn clockwise by hand.
- Pressing the pipe into the bottom of the connector, turn 3/4 clockwise by a wrench.



In case of PEEK pipe, turn clockwise Fitting for PEEK pipe only by hand, and do not use a wrench or other tool.

901-010	Nut for 1.6 pipe
901-011	Ferrule for 1.6φ pipe (pair)
901-023	Male Screw for 1.6 pipe
901-030	SUS 1.6φ pipe
901-075	PEEK pipe (o.d.1.5mm, i.d. 0.5mm)
901-076	PEEK pipe (o.d.1.5mm, i.d. 0.8mm)
901-077	Fitting for PEEK pipe

#### Adjustment of RI Detector

Optical zero of RI Detector may shift according solvent to use. After turning the power switch on, the output value that is displayed on the LCD display immediately after the auto-cleaning operation indicates the optical position. If the monitoring output is over  $\pm 200$ , the optical alignment is recommended with the re-alignment of the NUL glass.

<Optical Alignment of RI Detector>

- •Turning power switch on.
- Press the [CLEAN UP] key to execute the auto-cleaning.
- •After the [CLEAN UP] operation, check the RI output value on the LCD display.
- Remove the Column cover of LC-92XX NEXT from the chassis.



- Insert a blade of a screw driver and turn with slot of the alignment screw.
- •While checking the RI output value on the LCD display of the main body, turn the driver to perform zero adjustment.
- Remove the driver from the hole. Close the Column cover.

#### Manual Cleaning Instruction of Buffer Volume

The LC-92XX series has the Reverse flow protection during Recycling Separation as a standard. The Reverse flow protection system has a buffer-volume.

The buffer-volume is automatically cleaned in the CLEAN UP operation. When the LC-92XX has stopped in long term, or when the high-viscosity or low solubility sample is injected, the buffer-volume is contaminated and cannot be cleaned with the CLEAN UP operation.

If the noise from the buffer volume contamination is remarkable, following Manual Cleaning of the buffer volume is recommended.

<Manual Cleaning of Buffer Volume>

- Clean the buffer volume with the CLEAN UP operation.
- After the CLEAN UP operation, turn the Pump Drain Valve to the [DRAIN].

• Turn the Column reject valve in the middle between [REJECT] position and [CONNECT] position. (Refer to below figure.)



- Set the flow rate to maximum. Set 10 mL/min for LC-9210 NEXT and 25 mL/min for LC-9225 NEXT.
- Start the pump, and check that the solvent flows steadily from the pump drain out in the siphon box. If the flow from the Pump Drain is not appeared, stop the pump as soon as possible. If setting is abnormal in the Pump Drain Valve or the Column reject valve, the maximum flow may damage the column. Check the position of two valves, and re-start the pump.
- Press the **INVOFF** key in [RECYCLE].
- •One minute later, press , and then stop the pump to finish the cleaning maintenance.



# Chapter 2 Trouble Shooting

LC-92XX NEXT series mechanical failure, error message and remedy

### Chromatogram Acquisition Error

If chromatogram is abnormal, refer to following paragraph and to remedy. If abnormal chromatograph cannot be remedy, please contact us or our representative at the end of the book.

<Base Line Wander 1>



- ! Babble in the Reference cell of RI Detector!
- Solvent degassing is recommended.

For details on degassing, refer to the pages describing degassing in "General Information Manual".

Do the CLEAN UP operation with degassed solvent and wait that base line becomes stable after the column equilibrium.

Check the base line again.

If the wander cannot be remedy, go to the next step <Base Line Wander 2>.

#### <Base Line Wander 2>



- ! Babble in the Sample cell of RI Detector, babble in the eluent!
- Check the solvent filter line.
  - If the babble is found in the solvent filter line, use degassed solvent for the eluent.
  - For details on degassing, refer to the page 2-48 describing degassing in "General Information Manual".

Do the CLEAN UP operation with degassed solvent and wait that base line becomes stable after the column equilibrium. Check the base line again.

- ! Leakage of the UV Detector cell!
- Please contact us or our representative at the end of the book.
- ! Decay of UV Lamp!
- Please contact us or our representative at the end of the book.
- ! Leakage of the flow line!
- Check the connectors of pump-injector-column-detector.
   If the leakage at the connection of flow is found, tighten the screw further, or replace the new flow line.

- ! Loose connection of the Signal Cable!
- Check the connection of the Signal Cable.
   If the Loosen connection is found, re-tighten the connection in proper.
- ! Decay of Pump seal!
- Check the pump head.

If solvent leakage is found under the pump head, refer to Maintenance of Pump the page 4-10 of "Maintenance Manual" (this manual) for replacement of the pump seal to replace the pump seal if need.

#### <Base Line Drift>

- ! Environmental influence of the Laboratory!
- Check that wind hit the LC chassis directly, or that there is a temperature change in the laboratory.
   RI Detector is sensitive to fluctuation of temperature.
  - Fluctuation of temperature may cause the base line drift of the RI Detector.
- ! RI Detector temperature controller works not enough!
- Make sure that the time is over 30 to 100 min after the CLEAN UP operation. Wait that the temperature controller become stable.
- ! Solvent replacement is not enough!
- Wait that the Column become stable.
- ! Contamination of the column is continued!
- Column cleaning is required.

Refer to the column instruction and clean the column if need. In case of JAIGEL-H series, the solvent replacement is impossible. For the JAIGEL-H series, the addition of 0.1 - 0.5% tri-ethylamine in the solvent is recommended. The addition removes and purges the contamination on the packing material.

In case of using Chloroform, the addition of tri-ethylamine hydrochloride is recommended in concentration of 50 mM/L.

Before cleaning, test that the sample does not re-precipitate in the added solvent, and then do the column cleaning with the solvent.

- ! Leakage of the flow line!
- Check the connectors of pump-injector-column-detector. If the leakage at the connection of flow is found, tighten the screw further, or replace the new flow line.
- ! Decay of Pump seal!
- Check the pump head.

If solvent leakage is found under the pump head, refer to Maintenance of PumpMaintenance of Pump the page 4-10 of "Maintenance Manual" (this manual) for replacement of the pump seal to replace the pump seal.

- ! Leakage of the UV Detector cell!
- · Please contact us or our representative at the end of this manual.
- ! Decay of UV Lamp!
- Please contact us or our representative at the end of the book.
- ! Contamination of the RI cell!
- Make sure that the UV Detector signal has no noise.
   If base line drift cannot be remedied yet, RI detector cell might be dirty.
   The RI detector cell is built-in. Customers cannot disassemble and wash the RI detector cell. Please contact us or our representative at the end of the book.

<Base Line Drift during Recycling Operation>



- ! Contamination of the buffer volume!
- Clean the buffer volume with the CLEAN UP operation.
   When the LC-92XX is started and finished, clean the buffer volume with the CLEAN UP operation.
   When the CLEAN UP is neglected, the base line drift may occur from the

When the CLEAN UP is neglected, the base line drift may occur from the second-cycle of the recycle operation.

- ! Not suitable selection of the column!
- When the selection of the column against the sample is not suitable, the base line is drifting upward in the Recycling operation. Select suitable column(s) for the sample.



- ! Diffusion of the Recycled Sample in the Sample loop!
- When multiple times Recycling is done, turn over the knob to LOAD to eliminate the sample loop from the flow line because the recycled sample diffuse in the sample loop.

When you want to use the manual sample injector to execute the recycling separation, flow the solvent of 3 mL or more for LC-9210 NEXT or 10 mL or more for LC-9225 NEXT after the sample injection, and then turn the manual injector knob to the [LOAD] position.

- ! Not suitable separation conditions!
- When the selection of the column and solvent against the sample is not suitable, Poor Recycling efficiency appears in the Recycling operation.
   Select suitable column(s) and solvent for the sample.

#### Abnormal Pressure

Abnormal pressure may occur from decayed flow parts, sample property, or handling error. Refer to following paragraph and to remedy the abnormal pressure. When the abnormal pressure cannot be remedy, please contact us or our representative at the end of the book.

<Abnormal High Pressure>

- If the maximum pressure limit is set, an error message screen appears to inform the emergency stop of the pump.
   Check that the [MAXMAM] pressure limit is set to monitoring pressure + 2.0 MPa.
- ! Clogging of the Column or Dirt of the Column Packing!
- Press the [OK] key, and turn the column reject valve to [REJECT] to start the pump.
- If the pressure is significant smaller than before, column clogging may occur.
- Stop the pump.
- Remove the column from the flow line. Turn the Column reject valve to [CONNECT] position. Re-start the Pump and check the pressure.
- If the pressure is significant large without the Column, the clogging may be between the LC chassis and the Column inlet. Replace flow line part between the LC chassis and the Column inlet.
- If the pressure is abnormally high with connection of the Column, replacement of the Column is need.
- ! High viscosity of the solvent!
- In case using reverse phase column or using high polarity column, the solvent replacement of the column may occur high pressure. While the solvent replacement is finished, set the maximum limiter at the suitable value.

<Abnormal Pressure Fluctuation>

- If the monitoring pressure abnormally fluctuates, the pressure fluctuation may occur the abnormal chromatogram or decay of the column. Stop the Pump, check the following items.
- ! Clogging of the check valve!
- Turn the pump drain valve to [DRAIN].
- Set the flow rate to 5 mL/min for LC-9210 NEXT and 15 mL/min for LC-9225 NEXT, and start the pump.
- Check that the solvent flows steadily from the pump drain out in the siphon box. If the flow from the Pump Drain is not constant or repeat stop and flow, the check valve cleaning is required. Refer to the check valve Cleaning at 4-14page in this manual.
- ! Leakage of the flow line!
- Check the connectors of pump-injector-column-detector.
   If the leakage at the connection of flow is found, tighten the screw further, or replace the new flow line.
- ! Decay of Pump seal!
- Check the pump head.

If solvent leakage is found under the pump head, refer to Maintenance of Pump the page 4-10 of "Maintenance Manual" (this manual) for replacement of the pump seal to replace the pump seal.

- ! Babble in the Sample cell of RI Detector, babble in the eluent!
- Check the solvent filter.
  - If the babble is found in the solvent filter line, use degassed solvent for the eluent.
  - For details on degassing, refer to the page 2-48 describing degassing in "General Information Manual".
  - Do the CLEAN UP operation with degassed solvent and wait that base line becomes stable after the column equilibrium. Check the base line again.
#### <Abnormal Low Pressure>

- If the minimum pressure limit is set, an error message screen appears to inform the emergency stop of the pump.
   Check that the [MINIMAM] pressure limit is set to monitoring pressure - 2 MPa or less.
- ! Babble in the Sample cell of RI Detector, babble in the eluent!
- Check the solvent filter.
  - If the babble is found in the solvent filter line, use degassed solvent for the eluent.
  - For details on degassing, refer to the page 2-48 describing degassing in "General Information Manual".
  - Do the CLEAN UP operation with degassed solvent and wait that base line becomes stable after the column equilibrium. Check the base line again.
- ! Clogging of the check valve!
- Turn the pump drain valve to [DRAIN].
- Set the flow rate to 5 mL/min for LC-9210 NEXT and 15 mL/min for LC-9225 NEXT, and start the pump.
- Check that the solvent flows steadily from the pump drain out in the siphon box. If the flow from the Pump Drain is not constant or repeat stop and flow, the check valve cleaning is required. Refer to the check valve Cleaning at 4-14page in this manual.
- ! Solvent loss in the Solvent container!
- Make sure that Solvent fills enough in the Solvent container.
- If the Solvent is lost in the container, the Pump occurs emergency stop to prevent air introduction into the column.

- ! Leakage of the flow line!
- Check the connectors of pump-injector-column-detector.
   If the leakage at the connection of flow is found, tighten the screw further, or replace the new flow line.

### Abnormal Signal

The decay of the consumable parts or the property and miss-operation, may occur the abnormal signal of the Detector.

When the abnormal signal cannot be remedy, please contact us or our representative at the end of the book.

<Fail Power>

- ! Disconnection of POWER Cable!
- Make sure the insertion of the POWER Cable at the rear of LC chassis.

<No Signal and No Event Mark>

- ! Disconnection of SIGNAL Cable!
- Make sure that the connection of signal cable on the Output terminal block at the left hand of LC.
  - Check the input cable connection as same.
- ! Failure of the Recorder or Integrator!
- Please carefully read the instruction manual for your attachment.

<UV Detector Abnormal Signal>

- ! Dirt in the UV Detector cell and/or Leakage of the cell!
- If the solvent leaks from the UV detector cell, the chromatogram may cause fluctuation of base line and the sensitivity loss may cause. In the Recycling separation, extreme low peaks may appear with the leakage.
  If above chromatogram is obtained, please contact us or our representative at the end of the book.
- ! Decayed UV Lamp!
- If the UV Lamp decays, the chromatogram may cause fluctuation of base line. The zero balance of the baseline may not achieve. or the sensitivity loss may cause. If above symptoms appear, please contact us or our representative at the end of the book.
- ! Selected UV wave length is not suitable for the sample!
- Each sample has suitable UV wave length for detection.
   Select suitable UV wave length for the sample.

<RI Detector Abnormal Signal>

- ! Babble in the Reference cell of RI Detector!
- Solvent degassing is recommended.
   For details on degassing, refer to the page 2-110 describing degassing in "General Information Manual". Do the CLEAN UP operation with degassed solvent and wait that base line becomes stable after the column equilibrium. Check the base line again.
- ! RI Detector is out of optical alignment!
- If RI Detector is extremely out of optical alignment, electrical zero may not achieve or the RI signal causes full- scale in tight range.
   Refer to p.4-20 described RI Detector Adjustment, and do re-alignment of RI Detector.

### **Error Message**

If a failure or wrong operation occurs in the LC-92XX NEXT, an error message appears on the LCD display. At the beginning of the error message, "ERROR" is displayed followed by a number. Based on this number, perform the following operations to resolve the cause of failure. If the error message appears again, please contact us or our representative at the end of the book.

<ERROR 01>



- ! This error occurs if the minimum pressure limit is set to a value larger than the maximum pressure limit when setting them.
- Set them so that the maximum pressure limit is larger than the minimum pressure limit.

<ERROR 02>

- ! This error occurs if the maximum pressure limit is set to a value equal to the minimum pressure limit when setting them.
- Set them again so that the maximum pressure limit is not equal to the minimum pressure limit.

#### <ERROR 03>

```
<ERROR 03>
PURGE MODE
(WAIT 60 SECOND)
PUSH "OK" KEY
```

- ! This error occurs if you press one of the [Co/Dr] key in [FRACTION], the [START] key in [CLEAN UP], the [ON/OFF] key in [RECYCLE], and the [FLOW SET] key in [PUMP], during the [PURGE] operation of [RI DETECTOR].
- These keys are unavailable during the [PURGE] operation. Use them after the [PURGE] operation.

<ERROR 04>

```
<ERROR 04>
RECYCLE ON MODE
(CHANGE RECYCLE OFF)
PUSH "OK" KEY
```

- ! This error occurs if you press one of the [START] key in [CLEAN UP], the [PURGE] key in [RI DETECTOR], and the [FLOW SET] key in [PUMP], during the [RECYCLE] operation.
- These keys are unavailable during the [RECYCLE] operation. Use them after the [RECYCLE] operation.

<ERROR 05>

```
 < ERROR 05>
PUMP FLOW LIMIT
PUSH "OK" KEY
```

- ! This error occurs if you set a value greater than the maximum flow rate of the pump when changing the flow rate.
- Set a value less than the maximum flow rate of the installed pump. LC-9210 NEXT(FX-10) : 10.0 mL/min LC-9225 NEXT(FX-25) : 25.0 mL/min

<ERROR 06>

```
<ERROR 06>
CLEAN UP IS NOT MADE
PUSH "OK" KEY
```

- ! This error occurs when normal cleaning is not performed in three minutes during the buffer tube cleaning of the [CLEAN UP] operation.
- If the error is not solved, please contact us. Followings are possible causes of the error:
  - 1. The sensitivity of the sensor of the buffer tube is too low.
  - 2. The buffer tube is very dirty.

The error will be solved by replacing these parts.

<ERROR 07>

```
 < ERROR 07>
BEYOND THE LIMIT
PUSH "OK" KEY
```

- ! This error occurs if you set a value greater than or equal to 50.1 MPa in the maximum or minimum pressure limit setting screen.
- Set each pressure limit to a value less than 50.0 MPa.

<ERROR 08>

```
<ERROR 08>
THE PUMP IS
NOT OPERATING
PUSH "OK" KEY
```

- ! This error occurs if you press the [Co/Dr] key in [FRACTION], the [ON/OFF] key in [RECYCLE], or the [PURGE] key in [RI DETECTOR] when the pump is stopped.
- Press the [ON/OFF] key in [PUMP] to start the pump, and then press these keys.

<ERROR 09>



- ! This error occurs if you press the [ON/OFF] key in [PUMP] during the [RECYCLE] operation.
- Stop the [RECYCLE] operation, and then stop the pump. \*The pump is stopped when ERROR 09 is displayed.

<ERROR 10>



- ! This error occurs when the pressure exceeds 1.5 MPa during the [CLEAN UP] operation.
- Check that the column reject valve is located at the [REJECT] position.

<ERROR 11>

- ! This error occurs if you perform the injection operation using the manual sample injector (INJECT->LOAD->INJECT) when the pump is stopped.
- Start the pump before performing the injection.

<ERROR 12>



- ! This error occurs if you press an irrelevant key when setting a function, or if you press a key for setting a function when the function is running.
- This error occurs in the following situations:
  - 1. When you press a setting key ([MAXIMUM], [MINIMUM], or [RANGE]), while fraction collection is being executed by the [Co/Dr] key in [FRACTION], or while the solvent is running through the dead volume.
  - When you press the [Co/Dr] key in [FRACTION], while you are setting the function by using the setting keys ([MAXIMUM], [MINIMUM], [RANGE], or [DEAD VOL SET]).

After the operation finishes or the setting is completed, press the key.

<ERROR 13>

<error 13=""></error>	
THE PUMP IS	
OPERATING	
PUSH "OK" KEY	

- ! This error occurs if you press the [START] key in [CLEAN UP] when the pump is running.
- · Stop the pump, and then press the [START] key in [CLEAN UP].

<ERROR 14>

```
<ERROR 14>
PRESS OVER LIMIT
   (PUMP)
PUSH "OK" KEY
```

- ! This error occurs when an abnormality due to the pressure rise of the pump is detected.
- As the failure may occur on the parts in the pump, please contact us or our representative.

<ERROR 15>

- ! This error occurs when the monitoring pressure exceeds the specified maximum pressure limit.
- Refer to the page 4-30 of this manual to solve the problem according <Abnormal High Pressure>.

\*The pump is stopped when ERROR 15 is displayed.

<ERROR 16>

```
<ERROR 16>
FALL BELOW 01.0 MPa
(MINIMUM SET VALUE)
PUSH "OK" KEY
```

- ! This error occurs when the monitoring pressure falls below the specified minimum pressure limit.
- Refer to the page 4-33 of this manual to solve the problem according <Abnormal Low Pressure>.
  - \*The pump is stopped when ERROR 16 is displayed.

#### <ERROR 17>

```
<ERROR 17>
BURNOUT
(STOP THE PUMP)
PUSH "OK" KEY
```

- ! This error occurs when the temperature around the pump control board rises very high (over 60°C).
- Stop the pump for 30 minutes, and then start the pump again. If the error occurs again, please contact us or our representative.
  - \*The pump is stopped when ERROR 17 is displayed.

<ERROR 18>



- ! This error occurs if abnormal rotation is detected at the pump driving part when the pump is running.
- As the failure may occur on the parts in the pump such as board or sensor, please contact us or our representative.

#### <ERROR 19>

```
<ERROR 19>
SOLVENT ESCAPED
(CHECK THE PUMP)
PUSH "OK" KEY
```

! This error occurs if leakage is found around the suction pipe of the pump.

- Followings are possible causes of the error:
  - 1. Check that the connectors such as check valve and PEEK screw are tightened additionally.
  - 2. The leakage is caused by secular change of the pump seal. Refer to the page 4-10 in this manual to replace the pump seal.
  - 3. Remove the leakage sensor, wipe the liquid out of the sensor, and check the LED changes from red to green.

If the error is not solved, please contact us or our representative at the end of the book.

<ERROR 20>

```
<ERROR 20>
FRACTION MODE
COLLECT → DRAIN
PUSH ″OK″ KEY
```

- ! This error occurs, if you press the [PURGE] key in [RI DETECTOR], the [FLOW SET] key in [PUMP], or the [START] key in [CLEAN UP], while fraction collection is being executed by the [Co/Dr] key in [FRACTION].
- Press the [Co/Dr] key in [FRACTION] so that the eluent flows from the siphon, and then press the desired key.

#### <ERROR 21>

```
 <ERROR 21>
BEYOND THE LIMIT
(MAX MEMORY:33)
PUSH "OK" KEY
```

- ! This error occurs if you press the [Co/Dr] key in [FRACTION] 33 times continuously.
- If you press the key continuously while the solvent is running through the dead volume, the press timings are stored, and accordingly the valve works.
  33 times is the limit that can be saved while running through the dead volume. So do not press the [Co/Dr] key continuously over 33 times.

# **CUSTOMER SERVICE**

If you have any questions, or something is wrong with Recycling Preparative HPLC, please contact to the following.

208 Musashi, Mizuho, Nishitama, Tokyo 190-1213 Japan Japan Analytical Industry Co., Ltd Engineering Department TEL:+81 42-557-2332 FAX:+81 42-557-0894

# **Order for Accessories / Supplies**

Order for accessories / expendable supplies, please contact to the following.

208 Musashi, Mizuho, Nishitama, Tokyo 190-1213 Japan Japan Analytical Industry Co., Ltd Operation Department TEL: +81 42-557-2331 FAX: +81 42-557-1892

LC-92XX NEXT Series User's Manual (1.3 Edition)

JNIJapan Analytical Industry Co.,