

WARRANTY

Products sold by us, unless otherwise specified, are warranted for a period of two year from date of shipment or delivery to be free of defects in materials and workmanship. If any defects should occur in the product during this period of warranty, we will repair or replace the defects parts or product free of charge

This warranty shall not apply to defects resulting from following actions:

- 1) Improper or inadequate operation, maintenance, adjustment or calibration.
- 2) Unauthorized modification or misuse.
- 3) Use of parts that are not supplied by us.
- 4) Disaster.
- 5) Consumable parts such as fuse, battery and fittings.

The warranty period for all parts and repairs supplied under this warranty expires with the warranty period of the original product. For inquiries concerning repair service, contact your supplier after confirming the model name and serial number of your instrument. The contents of this manual are subject to change without notice in accordance with product improvements.

This operation manual describes the operation over the life of this instrument, carefully read this manual to obtain a through understanding of the operation of the unit before attempting to use it.

Special consideration and precautions for safe and efficient use are also described throughout the manual. These appear in the following forms;

WARNING ! : Warns potentially hazardous situations and outlines the correct procedures or practices required to prevent from personal injury.

CAUTION ! : Alert the operator to the correct operating or maintenance procedures required to prevent instrument failure, or damage.

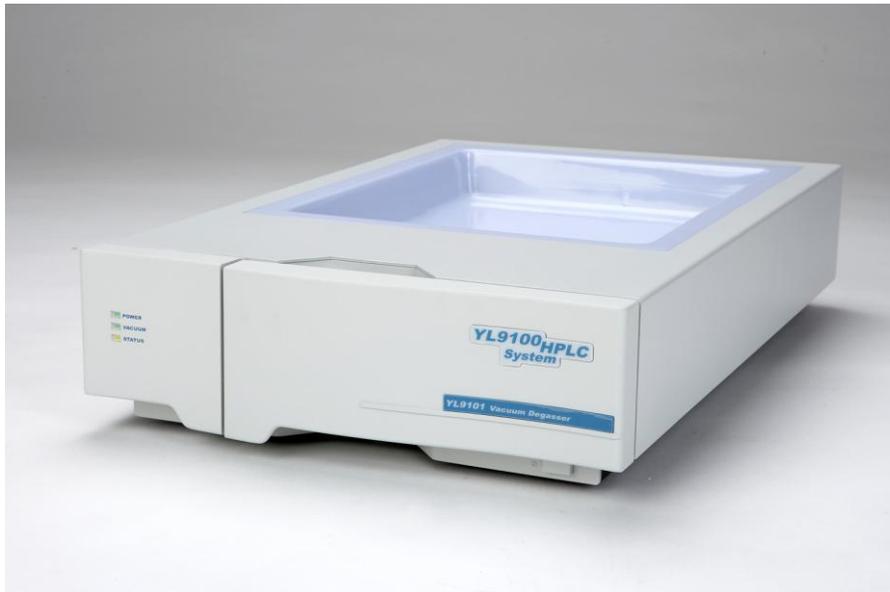
NOTE ! : Provides additional information for operator to obtain the best performance from the instrument.

Pressurized and hazardous solvents are used in high performance liquid chromatography. Take care to follow proper laboratory procedures to insure operator safety. Always wear eye, skin and clothing protection when operating the instrument, especially during sample injection, the opening of valves, etc.

YL9100 HPLC SYSTEM

YL9101 VACUUM DEGASSER

USER MANUAL



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Chapter 1. Introduction

It is not easy but important to select and handle mobile phase solvent in HPLC(High Performance Liquid Chromatography). YL9101 Vacuum Degasser is connected a high productive vacuum degassing module which is built-in to provide optimum solvent degassing to HPLC user.

Degassing of mobile phase solvent

Solvent is degassed when passing through the vacuum degasser, solvent preparation is greatly simplified and the degassed solvent is ready to use for analysis. With an in-line vacuum degasser, you can realize excellent environment for HPLC analysis.

YL9101 Vacuum Degasser consists of four channels for degassing and each channel removes efficiently air and oxygen in the solvent. YL9101 Vacuum Degasser provides the best optimized solvent.

1-1. Specifications

- 1) Number of channel: 4 CHs
- 2) Maximum flow rate : 10ml/min per channel
0-2.0ml/min per channel for 70% Gas Removed from Methanol
- 3) Internal volume per channel : 925ul per channel
- 4) Materials in contact with solvent : Teflon[®] AF and PEEK
- 5) Safety & maintenance : Error detection
- 6) Dimensions : 385 X 80 X 565mm (width X height X depth)
- 7) Line Voltage : 110 or 220VAC, $\pm 10\%$, automatic voltage selection
- 8) Line frequency : 50/60Hz, $\pm 5\%$
- 9) Power consumption : 20W

Chapter 2. Installation

2-1. Inspection and site preparation

Site requirement of YL9101 Vacuum Degasser

- 1) Room with 20°C temperature with variation $\pm 5^\circ\text{C}$ with and 60% humidity
- 2) Where no direct and straight sunlight
- 3) Plain floor without carpet
- 4) Where having spare space of 20cm or more
- 5) Where there are 10% or less voltage change, no frequency change and 100 Ω or less grounding point
- 6) Where there is no generation of corrosive gas and ventilation is well done
- 7) Where stable power of 110 or 220V is supplied, $\pm 10\%$
- 8) Where not receive electromagnetic induction from large transformer, high frequency heater, UPS, etc.
- 9) Within 2500 m above sea level(storage within 4600m)

Please check the following before you install the system.

- 1) Keep the ventilation as normal state.
- 2) Install on the stable place. Avoid the places as like near to air conditioner and heater, direct sun light, near to window.
- 3) Keep the place without dust and vibration.
- 4) Maintain voltage variation within 5% of proper voltage.
- 5) Avoid high frequency or strong magnetic field environment.
- 6) Avoid from the source of fire(spark, flame).
- 7) Keep the proper ground for electricity.
- 8) Check the place of water supply for emergency.

Caution ! : Keep distance with CRT at least 50cm.

2-2. Connection of power

Confirm supplying voltage, electric power, socket, and type of connector are correct. Before connect power cable on the pump, please turn off the power switch of YL9101 Vacuum Degasser.

Warning ! : The electric power should be matched with description on the rear side of instrument. If not, the instrument can be damaged or it can be a reason of fire.

Caution ! : Confirm the ground is correct. Water pipe is not recommended in case of non-metal pipe. Gas pipe should not use because of safety. If the ground is not correct, the electric shock can be happened.

2-3. Connection of tubing

2-3-1. Connecting of mobile phase solvent

YL9101 Vacuum Degasser can connect 4 kinds of solvent. If you do not use less than 4 solvents, you must plug up the solvent port, which is not used. Open a dust cover in the front, you will see 8 ports. 4 ports left are for input and right 4 ports are for output. Use a plug screw supplied to plug up un-used ports.

There is solvent reservoir in the upper part of YL9101 Vacuum Degasser. Place solvent bottles on the reservoir and connect input tubes. All input tubes have in-line 10 μm solvent filter in the end to prevent minute particles from entering into pump.

2-3-2. Connecting of Solvent delivery pump

Connect the degassed solvent tube to the input of Gradient Pump. The degassed solvent tube is located in right middle of YL9101 Vacuum Degasser. Open prim/purge valve by turning in counter-clock wise and setting mixing rate as 100% for each solvent and pull out solvent by a syringe. Check if all tubes are filled with solvent. Do not pull out quickly nor strongly, it will damage the in-line vacuum degasser. Pull out slowly until when tubes are will with solvent.

Chapter 3. Operation

There are three LEDs in front of YL9101 Vacuum Degasser.

	POWER	LED turns ON if main power turns on
	VACUUM	LED turns ON during vacuuming
	STATUS	LED turns ON if there is error during vacuum degassing

3-1. Degassing of the solvent

Power on YL9101 Vacuum Degasser by pressing power switch, which is located in the front. When you power on, check if pump is stopped, otherwise stop pump before turning on YL9101 Vacuum Degasser. YL9101 Vacuum Degasser will start immediately to degas solvent. If normally operational, green LED (VACUUM) located far left is on.

3-2. Operating of solvent delivery pump

Set mixing rate constant for all solvents and start pump at flow rate of 1.0 ml/min. Air bubble will be coming when start. To remove the bubble, open the prime/purge valve and pull out air bubble by syringe slowly until the bubbles are removed. After remove, close the valve for normal use of pump.

3-3. Replacing solvents

When necessary to replace solvents during analysis, stop pump and replace solvents and re-start pump. But do not turn off YL9101 Vacuum Degasser. If you want to change solvent filled inside if YL9101 Vacuum Degasser completely, set mixing rate at 100% for the solvent, pull out more than 15 ml solvent by syringe or start up pump and wait until solvent is completely changed.

3-4. Keeping method after use

If you do not use YL9101 Vacuum Degasser more than 1 day, use ultra pure water and flow into vacuum degasser and rinse with alcohol completely and keep it. Specially when use with buffering solution, be sure to rinse YL9101 Vacuum Degasser, otherwise precipitation can be occurred. In the case YL9101 Vacuum Degasser is kept lower than 0°C, dry completely after rinse.

Chapter 4. Maintenance

4-1. Filtration of solvent

Be sure to filter solvent by $<5 \mu\text{m}$ when specially use buffering solution. Keep filtered solvent in the bottle, which is cleared from minute particles. When use filtered solvent, which kept more than 1 week, please filter again before use.

4-2. Caution

- 1) Do not pressurize the vacuum degasser module. More than 120 kPa pressure will damage degassing membrane inside. When you put solvent bottles 2 m higher than degasser, the pressure is approximately 120 kPa. So place the bottles less than 2 m lower.
- 2) Be sure to prevent from any contamination when you connect or disconnect tubes. Entering fine dust will reduce degassing efficiency greatly.
- 3) If you want to increase degassing efficiency, make parallel connection of input port to output port. Do not connect it by serial way.
- 4) Can not use YL9101 Vacuum Degasser for output of solvent delivery pump.

4-3. Troubleshooting

- 1) Error and malfunctioning

Errors of YL9101 Vacuum Degasser are occurred when

- a) Not approach normal vacuum state 15 minutes after power on.
- b) Rapid pressure drops by solvent leak during normal operation.

When above errors occurred, degasser will turn red LED. Open above solvent reservoir and check if vacuum lines are installed correctly. If you cannot find any thing wrong, please contact our representative or us. In order to re-start, turn off and wait for more than 5 seconds and turn on.

- 2) Continuous air bubbling

If air bubbles are continuously happened after turning YL9101 Vacuum Degasser on more than 1 hour, check if input and output port and valve fitting are completed connected. Do not tighten the screw. Fitting and joint part can be damaged. If the problem is not solved, please contact our representative or us.

YL9100 HPLC SYSTEM

YL9110 QUATERNARY SOLVENT DELIVERY PUMP

USER MANUAL



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Chapter 1. Introduction

YL9110 Solvent delivery pump is a pump for High performance liquid chromatograph. It provides high performance and functions as a HPLC pump, and controlled by software.

YL9110 Quaternary pump uses specially designed cam and pulse damper for stable solvent delivery and has a compressibility compensation function for accurate and precise solvent delivery.

And the pump has a automatic rinsing function to prolong the life time of pump head. Using UHMWPE seal, it provides extended life time of high pressure seal even though use buffer solution as a mobile phase. On the outlet of pump, there is a in-line filter to prevent small particles come into the column and also protect column by the pressure limitation setup. YL9110 Quaternary pump consists of two independent dual carrier assembly to provides high pressure gradient operation. YL9110 Quaternary pump provides auto prime/purge function for easy operation and fast exchanging of solvent. This instrument manual includes basic principle, installation and operation method, and troubleshooting to use YL9110 Quaternary pump properly.

1-1. Specifications

- 1) Operating principle : Parallel dual-plunger pump, low pressure gradient
- 2) Compressibility compensation : Automatic
- 3) Flow range : 0.001-10ml/min
- 4) Flow rate accuracy : $\leq \pm 1\%$ at 1ml/min
- 5) Flow rate precision : 0.1% RSD at 1ml/min
- 6) Maximum pressure : 6000 psi
- 7) Operating range : 0-6000 psi up to 5ml/min
- 8) Operating range : 0-3000 psi up to 10ml/min
- 9) Pressure pulsation : $\leq \pm 1\%$ at 1ml/min
- 10) Composition Precision : $<0.1\%$
- 11) Composition Accuracy : $<0.5\%$
- 12) Semi-Auto prime/purge
- 13) Communications : LAN
- 14) Safety & maintenance : Leak detection, Diagnostics, Error detection
- 15) Dimensions : 385 X 160 X 565 mm (width X height X depth)
- 16) Line Voltage : 110 or 220 VAC, $\pm 10\%$
- 17) Line frequency : 50/60Hz, $\pm 5\%$
- 18) Power consumption : 70W

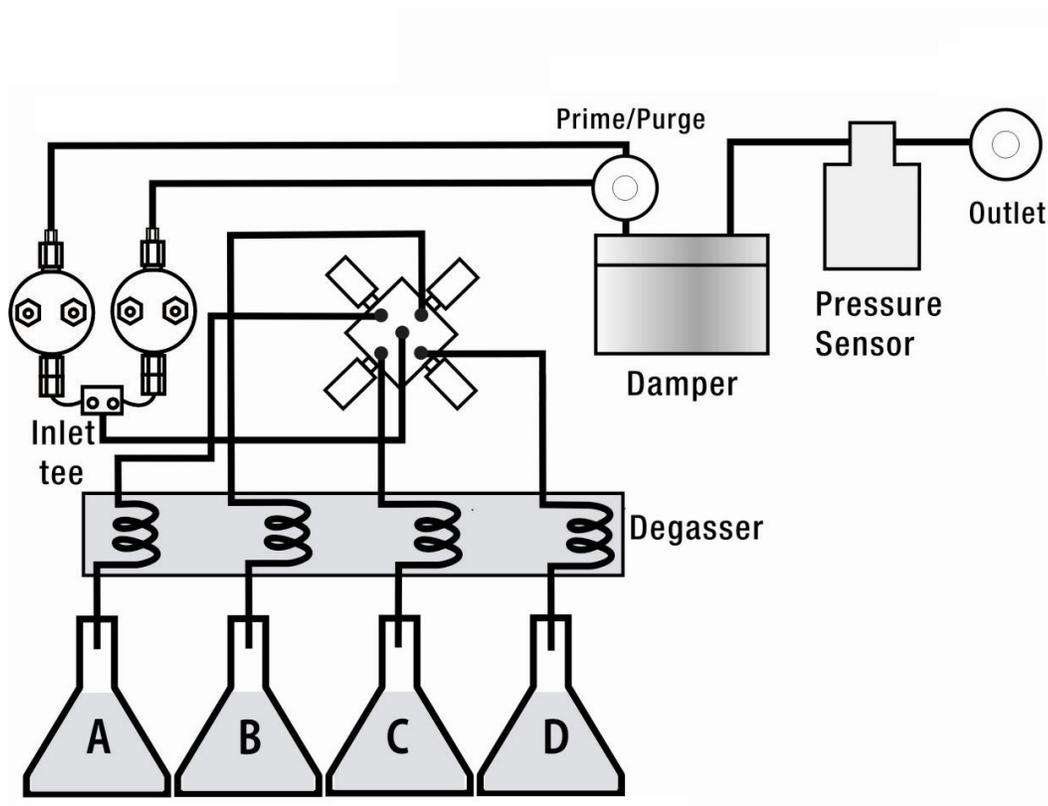
[Optional specification]

Head type	Narrow Bore	Analytical	Semi prep
Head volume(μ l/each)	25	64	144
Settable Flow range(ml/min)	0.001~5	0.001~10	0.01~50
Flow range(ml/min)	0.005~5	0.01~10	0.05~25
Pressure(psi)	5000psi	6000psi	3500psi
In-line filter (micron)	2	2	10
Material	Zirconium, Ruby, PEEK UHMWPE, PTFE	SS316, Zirconium, PTFE, Sapphire, UHMWPE	SUS316, Sapphire, Ruby, UHMWPE, PTFE

Chapter 2. Configuration and Principle

2-1. Configuration of flow path

YL9110 Quaternary pump consists as same with figure 1. It uses Teflon tubing between pump inlet and inlet check valve, SUS316 or PEEK tubing from outlet check valve.



[Fig. 1] Flow diagram of YL9110 pump

- **Solvent Filter**

The solvent filter is used for protecting the system from the particles in solvent. This filter removes particles from solvent to prolong the life time of high pressure seal and prevent damage on the column. It is recommended to use the solvent filter when you use YL9110 Quaternary pump with or without degassing module. Select proper filter depending on the column and flow rate.

- **Pump Head Assembly**

Pump head is real working part to deliver solvent by piston movement and check valve. It consists of plunger, check valve, high pressure seal, low pressure seal and rinsing port. YL9110 Quaternary pump built in automatic rinsing port to clean the head assembly efficiently if uses buffer solution.

- **Auto Rinsing Pump**

Auto rinsing pump delivers cleaning solvent from the rinsing solvent bottle to the inside of pump head. YL9110 Quaternary pump rinse the system every 3 minutes automatically.

- **Prime/purge valve**

This valve is used for priming the pump. Fill the solvent inside of tubing from the solvent bottle if you use the pump for the first time or the tubing lines are empty. Remove a plug on the prime/purge valve and suck the solvent using syringe, or click "Prime Start" button on the software to operate the micro pump.

- **Prime micro pump**

Using this pump, the pump circulates rinse solvent into the pump head.

- **Solenoid valve**

It is mixing valve for low pressure gradient. YL9110 Quaternary pump controls this valve for gradient according to solvent ratio and pump speed.

- **Mixer**

It is static mixer to improve mixing efficiency of solvent.

- **Pulse damper**

YL9110 Quaternary pump reduces pulse from the cam operation by the diaphragm damper. YL9110 Quaternary pump provides constant and pulseless flow using compressibility compensation and pulse damper, so the detector that affected by flow stability can be used with YL9110 pump.

- **Pressure transducer**

It checks real time system pressure. The pump uses this pressure to protect system and to operate compressibility compensation and even compensation. YL9110 Quaternary pump uses continuous flow path type pressure sensor.

- **In-line filter**

It removes fine particles that are not filtered by solvent filter or made by worn of high pressure seal.

2-2. Operation

There are essential parameters on the pump as like flow accuracy, precision, and reproducibility to get the reliance of analysis data and low detection limit. YL9110 Quaternary pump uses high pressure resistant dual pump head, controls microprocessor to monitor the phase of cam to remove pulse, so fulfill the necessities of solvent delivery pump.

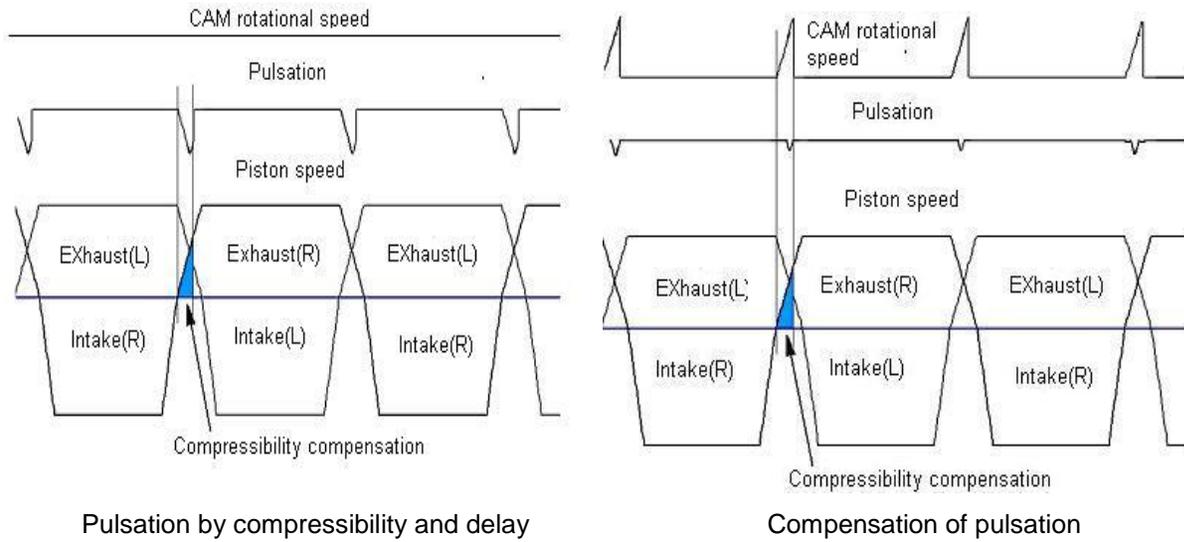
Diaphragm pulse damper reduces pulse from the cam operation more than 90% between low and high pressure range by internal elastic body of damper, and also works well as a mixer for gradient elution. The elastic body of Diaphragm pulse damper improves flow accuracy through compression, expansion procedure to make variance of kinetic energy by flow constant. The amount of contact solvent with pulse damper is around 1.5ml at 3000psi, which ensures that flow path is completely cleared away.

YL9110 Quaternary pump was designed so that integrated flow rate may realize no-pulse operation using specially designed cam. However, pulse incapable of being neglected is caused actually due to compressibility of mobile phase proportional to pressure and elasticity of high pressure seal, so the pump is controlled in real time so that occurrence of pulse proportional to pressure may be depressed. Control method of the Pump uses supervision of pressure and control of location simultaneously, so it has advantage to improve precision and accuracy of flow rate without being affected by range of pressure and flow rate. There is a part for microprocessor control to realize various function of pump including stepping motor control. Stepping motor control processor operates motor as a micro step, so can achieve constant motor speed at low flow rate with low noise.

Operation mechanism is for transmission of kinetic energy from step motor to piston. This mechanism includes specially designed cam, stepping motor, carrier, carrier housing, and phase sensor.

2-3. Compressibility compensation

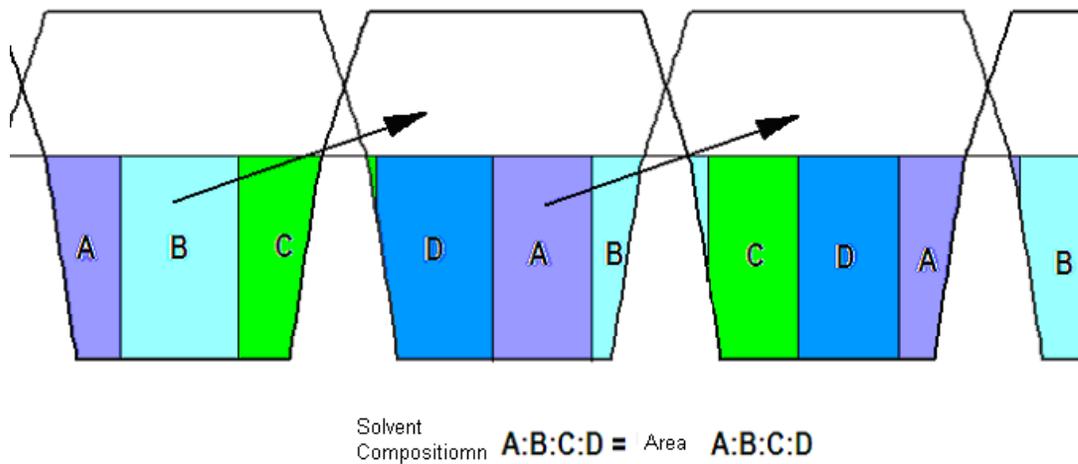
Most of pumps for HPLC analysis are used at high pressure. However, pulse occurs in high pressure due to compressibility of liquid and elasticity ratio of seal, so flow rate is also reduced. Occurrence of pulse due to this reduces precision and accuracy of pump flow rate, so compensation is necessary for this. YL9110 Quaternary pump monitors actual pressure and calculates compensation value for this; compressibility compensating operation to control angular velocity of cam with this value reduces occurrence of pulse flow remarkably as well as improves accuracy of flow rate largely.



[Fig. 2] Delivery mechanism of YL9110 Quaternary pump

2-4. Solenoid valve control

YL9110 Quaternary pump controls open/close signal of solenoid valve to perform low pressure gradient elution. For accuracy and precision of solvent mixing, YL9110 Quaternary pump controls valve opening and closing time with real time compensation according to time gap of valve cycling and the phase speed of cam.



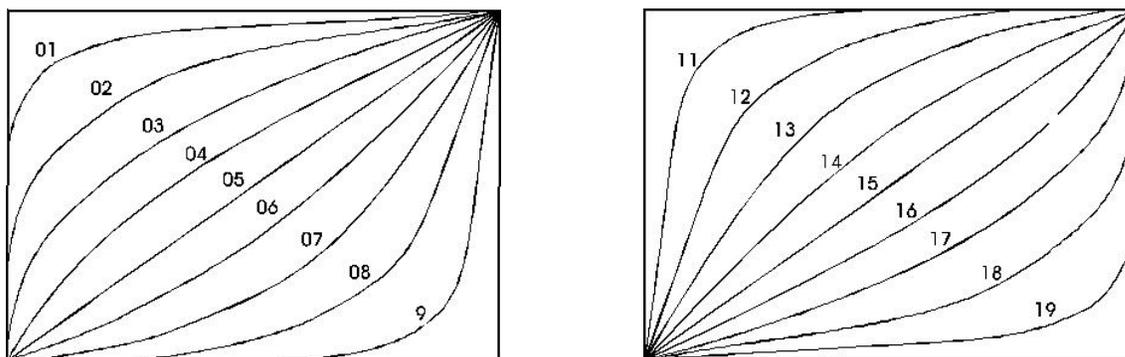
[Fig. 3] Compensation of solenoid valve time gap

2-5. Gradient curve

Solvent A and solvent B are mixed with flow rate F_b ,
 Mixed solvent flows to the column with flow rate F_{ab} ,
 Solvent volume V of solvent A vs Concentration of solvent B

$$C(\%) = 100 \times \left\{ 1 - (F_{ab} - F_b) / V \times t \right\}^{(F_b / F_{ab} - F_b)}$$

The solvent ratio difference between present row and next row increases or decreases as a exponential curve. In general, the gradient elution is used for separation of duplicated peaks. YL9110 Quaternary pump has a exponential curve program, but use gradient elution by linear curve when it operates with YLClarity software.



[Fig. 4] Gradient curve

2-6. Rinse port

When using buffer solution, salts are generated on back side of high pressure seal and these deposits wear pump seal to cause shortening of seal life, which has bad effect on pump.

Rinse port enables to insert proper solvent in back side of high pressure seal to prevent salts from being deposited and activated. Mixed solution(20% MeOH) of water and methanol is used as cleaning solution, and life of seal is extended with lubrication action in general analysis..

Chapter 3. Installation

3-1. Inspection and site preparation

YL9110 Quaternary Pump is delivered along with the following parts when being shipped. Before opening transportation package, perform inspection for trace of shock or mistake, and if there is abnormality, do not open the contents and inform this company of it. And, if contents are opened, perform inspection for existence of shock in the contents and contact with this company when trace of shock is found.

YL9110 Quaternary Pump is a delicate instrument, so use original box and buffer material as far as possible when re-packing it to transport instrument. If it is impossible to use original box; wrap pump with several layers of buffer material, and fill the bottom, top and all other sides of pump with buffer material in order to make pump endure shock or vibration during transportation.

Standard configuration of YL9110 Quaternary pump

- 1) Main body of instrument
- 2) Power cord and fuse
- 3) Tubing 60cm,
 - A. Bio Narrow Bore : ID 0.01" , OD 1/16" PEEK
 - B. Analytical : ID 0.01" , OD 1/16" SUS316
 - C. Semi-prep : ID 0.02" , OD 1/16" SUS316
- 4) Installation kit
- 5) Manual

Site requirement of YL9110 Quaternary pump

- 1) Room with 20℃ temperature with variation $\pm 5^\circ\text{C}$ with and 60% humidity
- 2) Where no direct and straight sunlight
- 3) Plain floor without carpet
- 4) Where having spare space of 20cm or more
- 5) Where there are 10% or less voltage change, no frequency change and 100 Ω or less grounding point
- 6) Where there is no generation of corrosive gas and ventilation is well done
- 7) Where stable power of 110 or 220V is supplied, $\pm 10\%$
- 8) Where not receive electromagnetic induction from large transformer, high frequency heater, UPS, etc.

- 9) Within 2500 m above sea level(storage within 4600m)

Please check the following before you install the system.

- 1) Keep the ventilation as normal state.
- 2) Install on the stable place. Avoid the places as like near to air conditioner and heater, direct sun light, near to window.
- 3) Keep the place without dust and vibration.
- 4) Maintain voltage variation within 5% of proper voltage.
- 5) Avoid high frequency or strong magnetic field environment.
- 6) Avoid from the source of fire(spark, flame).
- 7) Keep the proper ground for electricity.
- 8) Check the place of water supply for emergency.

Caution ! : Keep distance with CRT at least 50cm.

3-2. Connection of power

Confirm supplying voltage, electric power, socket, and type of connector are correct. Before connect power cable on the pump, please turn off the power switch of YL9110 Quaternary pump.

Warning ! : The electric power should be matched with description on the rear side of instrument. If not, the instrument can be damaged or it can be a reason of fire.

Caution ! : Confirm the ground is correct. Water pipe is not recommended in case of non-metal pipe. Gas pipe should not use because of safety. If the ground is not correct, the electric shock can be happened.

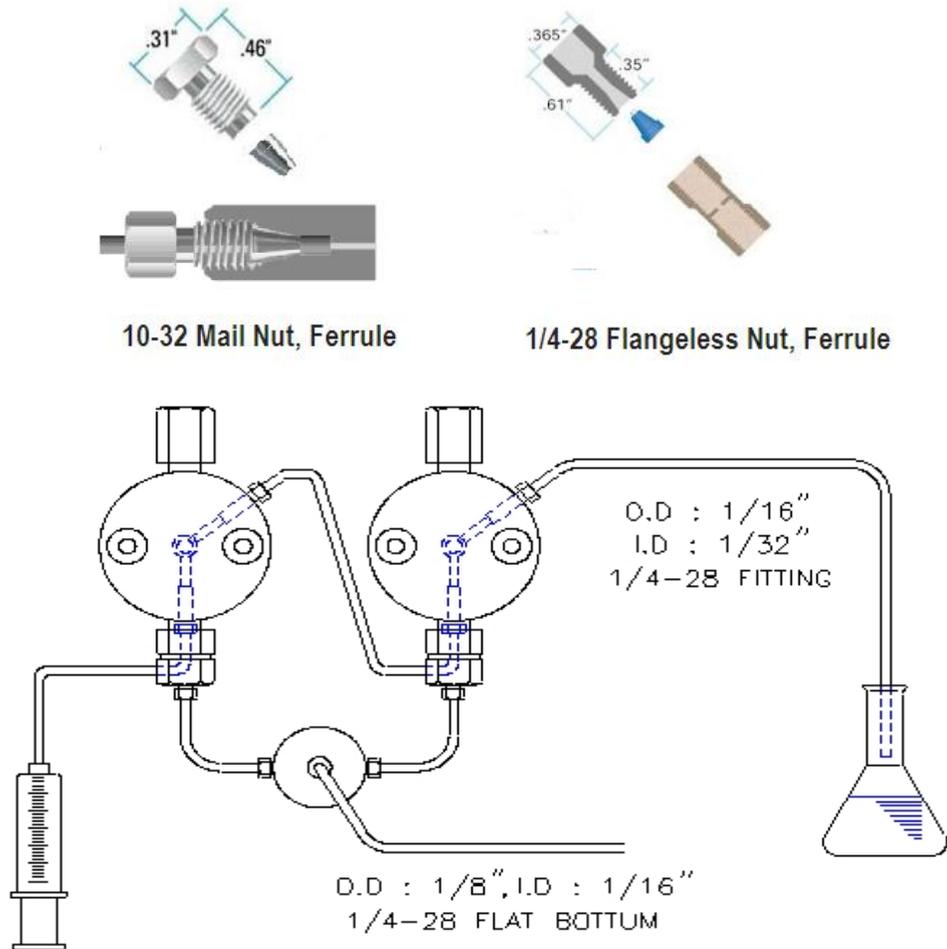
3-3. Connection of tubing

YL9110 Quaternary pump uses following fittings for connection.

Flow path		Material	OD	ID	Fitting(UNF)
Inlet Tee		Teflon	1/8"	1/16"	1/4-28(flat type)
Inlet Tee ~inlet check valve		Teflon	1/16"	1/32"	1/4-28(flat type)
Outlet check valve~ In-line filter	Narrow bore	PEEK	1/16"	0.02"	10-32
	Analytical	SUS316	1/16"	0.02"	10-32
	Semi prep	SUS316	1/16"	0.03"	10-32

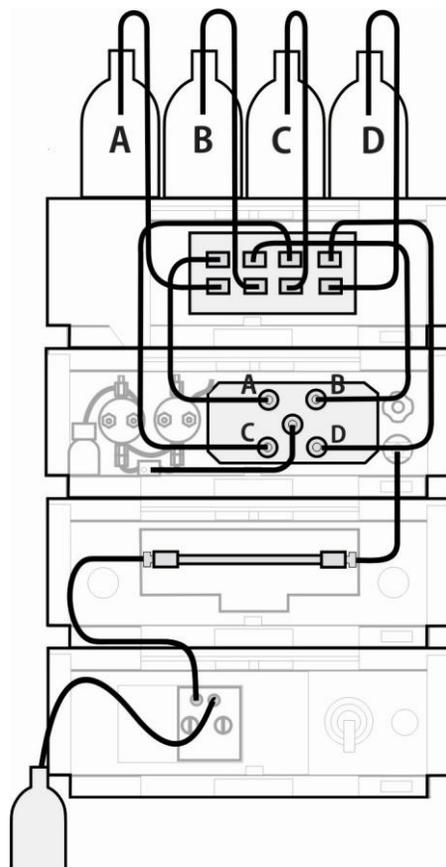
The cut surface of tubing should be cut at right angle without dust, tube should not be contracted, and middle inner diameter shall not be blocked.

In order to cut stainless steel tubing, tubing cutter should be used, plastic tubing cutter or shaving cutter should be used for teflon and similar material of tubing, and the surface should be clean and have no crumbling.



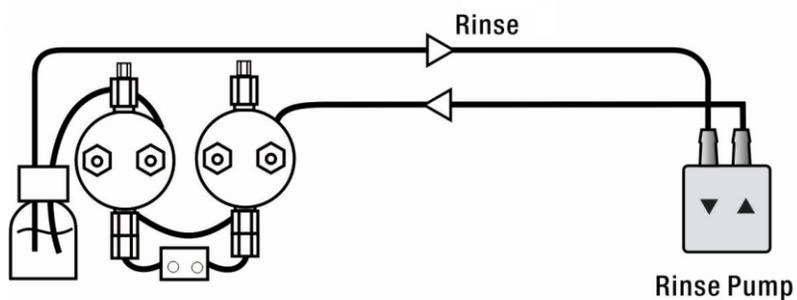
[Fig. 5] Fitting for 1/16" OD tubing

Inlet tubing of pump connects with degassing module using 1/4-28 fitting or with solvent bottle directly, the outlet tubing connects with injector. The fitting for injector is different depending on the injector type.



[Fig. 6] Connection between YL9110 Quaternary pump and YL9100 HPLC system

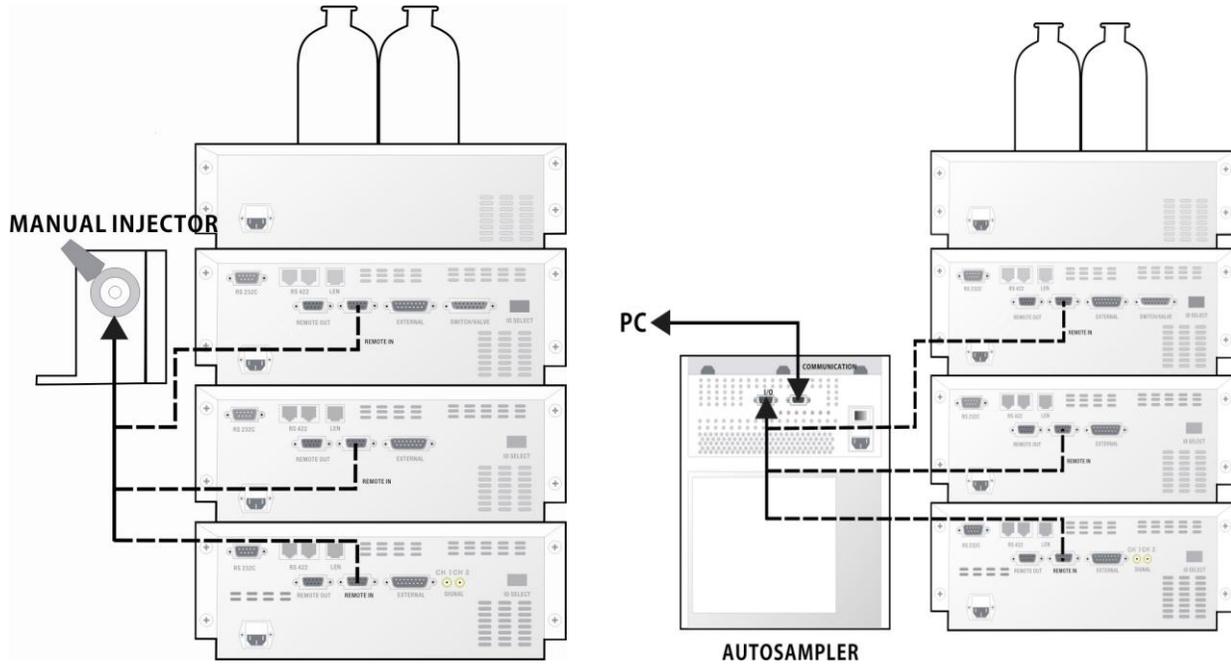
Rinse port tubing connects on the each pump head using 1/4-28 fitting as same as [Fig. 7], and fill the 20~50% Methanol. The rinse pump inside of YL9110 Quaternary pump circulates the rinse solvent into the pump head every minute. Check and replace rinse solvent once a week at least.



[Fig. 7] Tubing connection of rinse port

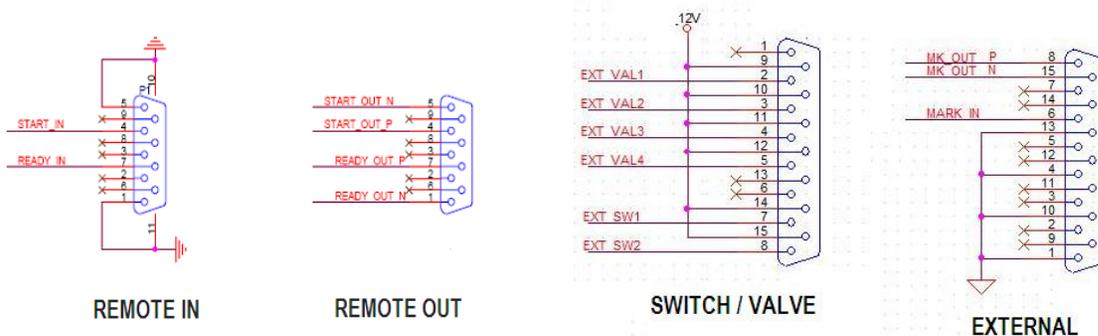
3-4. Connection of remote cable

YL9110 Quaternary pump has connection terminals for remote input/output, external solenoid valve, and marker input/output. The remote cable from the injector(manual or autosampler) has to be connected on the Remote In terminal on the rear side of YL9110 Quaternary pump to collect data at the moment of injection.



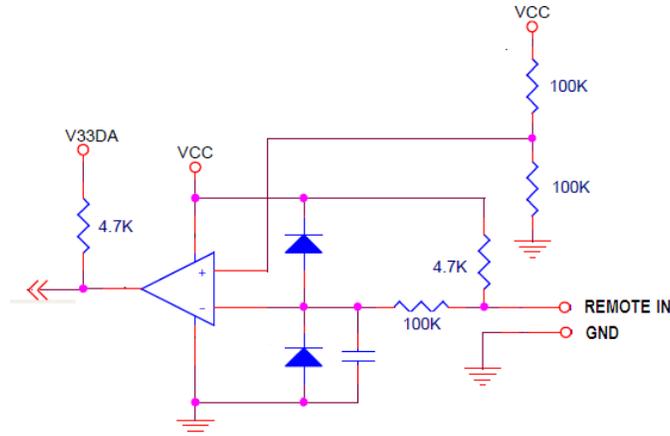
[Fig. 8] Connection of remote cable between YL9110 pump and injector

Notice ! : Please do not connect wires between cables at your discretion. If you want to connect with the other instrument, please check input/output information and confirm with YL9110 Quaternary pump terminal configuration.

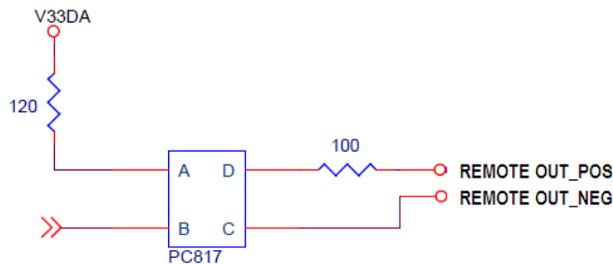


[Fig. 9] Pin configuration of each terminal

[Fig. 10] and [Fig. 11] are the diagram of remote and the other terminal input/output. In between YL9100 series modules, connect directly and confirm the configuration with the other modules.

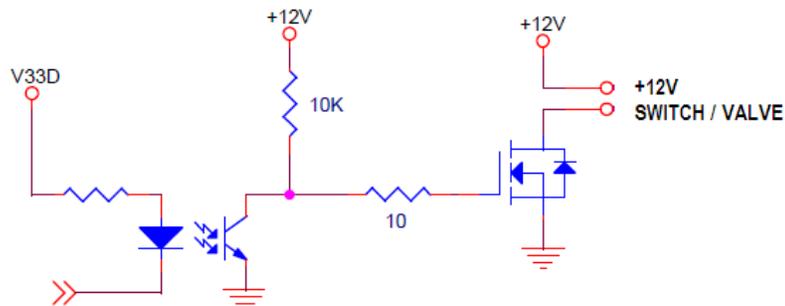


[Fig. 10] Diagram of Remote and Marker input



[Fig. 11] Diagram of Remote and Marker output

YL9110 Quaternary pump provides output signal(12V 500mA) to operate external solenoid valve as like [Fig 12].



[Fig. 12] Diagram of solenoid output

[Remote operation]

START-IN : Operate instrument, and start running of gradient program.

If you connect it with autosampler or external valve, automatic running is available.

START-OUT : If the signal input on the START-IN terminal, the signal pulse output through this port. It can be used for synchronization of remote start with the other instrument.

MARK-IN : To control event program or operate additional operation.

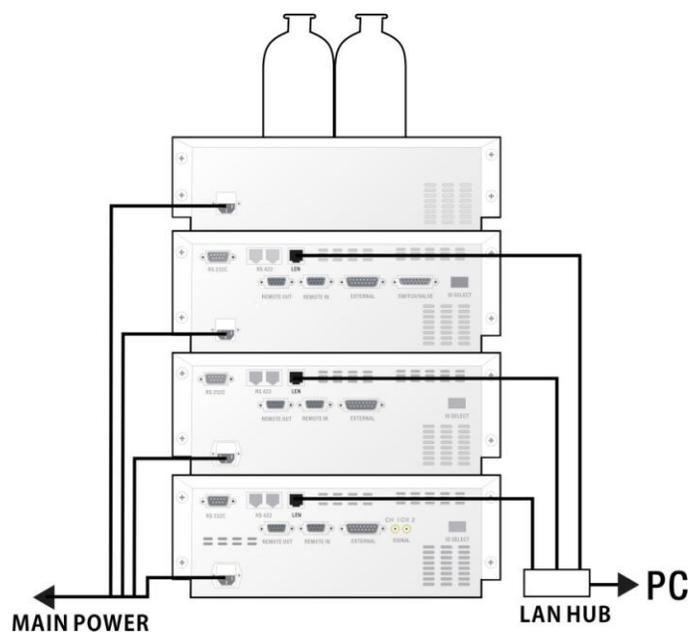
MARK-OUT : To control time event program output.

READY-IN : To change error state and stop operation if there is a input.

READY-OUT : When YL9110 Quaternary pump is not ready state because of running status, output error signal is indicated if there is a leak.

3-5. Connection of communication cable

YL9110 Quaternary pump provides TCP/IP internet protocol as a standard. The IP address of YL9100 pump series is 10.10.10.10, if DIP SW settings on the rear side are On position. If you change the IP address using control software, the DIP SW has to be set OFF.



[Fig. 13] Connection of communication cable

Notice ! : The LAN HUB used for cable connection on the PC must use switching mode module.

Chapter 4. Operation

There are four LEDs in front of YL9110 Quaternary pump.

	POWER	LED turns ON if main power turns on
	CONNECTED	LED turns ON if communication is connected, LED blinks during connection
	READY/RUN	LED turns ON before analysis, LED blinks during analysis
	ERROR	LED turns ON if there is error

4-1. Before Start

When using pump for the first time, initialize it through the following process in order to clean flow path and condition high pressure seal. This process is necessary in case instrument is installed newly or is not used for long time.

- 1) Prepare iso-propanol of HPLC grade.
- 2) Remove residual air bubble within instrument by turning prime/purge valve in counter clockwise and loading iso-propanol of at least 50ml by Prime Start button.
- 3) Separate pump outlet tubing.
- 4) Press the sucked iso-propanol into syringe with prime/purge valve and discharge more than 5ml to outlet of in-line filter.
- 5) Operate pump with instrument outlet open for 2-3 hours at 0.2ml/min flow rate and for 1 hour at 1.0ml/min flow rate using iso-propanol.
- 6) Perform process of 2) ~ 5) using solvent which will be used as a mobile phase.
- 7) Remove inside residual iso-propanol by operating it at 1ml/min flow rate for 30 minutes with instrument outlet open.
- 8) Form flow path by connecting injector, column, and detector tubing mutually.

4-2. Mobile phase filter and bottle

Solvent vessel should be positioned at higher location than pump and not be positioned below pump, and inlet tubing length should be as short as possible. This can minimize pressure drop caused at inlet of pump during suction.

When using solvent having high vapor pressure as hexane, formation of air bubble is caused due to large pressure drop in suction part in high flow rate; so particular care should be taken, and mobile phase should be maintained after air separation, filtration and air-tightening.

Mobile phase filter of 10 μ m porosity is connected into inlet tubing in order to prevent entering of small particles. Mobile phase filter is blocked if mobile phase is bad or is used for long time, it is necessary to clean or change filter in this case.

4-3. Preparation of solvent

Proper solvent prevents various problems happened during actual analysis.

Solvent gas removal and filtration are necessary because they have great effect on result of analysis and maintenance of instrument.

4-3-1. Degassing

Solvent gas removal is performed in order to remove gas such as nitrogen or oxygen contained in mobile phase. Contained gas should be removed by air separation before mobile phase is used or while mobile phase is used, and the most practical technology for air separation is to insert helium into solvent.

Helium is easily separated from HPLC solvent, so other gases contained in solvent may be easily removed due to diffusion of helium gas.

When mixing organic solvent such as methanol or acetonitrile into water, this mixture contains very small quantity of gas as compared to the quantity of pure composition; so it has more strong tendency to discharge gas. Back pressure regulator attached to outlet of detector prevents formation of noise in base line due to air bubble, and mobile phase vessel should be pressurized under 2-3psi pressure with helium if it is desired to reduce gas discharge due to solvent mixing.

4-3-2. Filtration

Solvent should be necessarily filtered through 0.45 μ m or less filtering membrane before use. Removal of small particles is necessary to compensate reliable operation of piston seal, and is necessary measure for reliability of other components in liquid chromatograph.

Filtration process is necessary after mixing of solvent, and is more necessary in case of buffer to which un-dissolved impurities are source of deposits. After filtration, solvent should be keep in air-tight bottle from which small particles are removed; once solvent has been filtered, it is not necessary to filter this solvent everyday unless reaction produce bacteria or indissoluble material occurs. If solvent is kept in storage vessel for more than one week, it is desired to filter it again before use.

4-3-3. Solvent effect on the instrument

All parts of the Gradient Pump contacting with mobile phase is manufactured from 316 stainless steel, ruby, sapphire, zirconium, or fluorine carbon polymer. Most of these materials are sensitive to chloride, and it is desired to avoid use of solvent which contains even small quantity of chloride. Main solvents that should be avoided especially are as follows.

Aqua Regia	Hydrochloric Acid(HCL) (20%)
Bromine	HCL (37%)
Chlorine Anhydrous	HCL (50%)
Copper Chloride	HCL (20%)
Ferric Chloride	HCL (75%)
Ferrous Chloride	Hydrofluorsilicic Acid (20%)
Freon 12	Hydrogen Peroxide
Guanidine	Lodine
Hydrochloride (6M)	Mercuric Chloride
Hydrobromic (20%)	(Dilute Solution)

In addition, it should be avoided to leave chloroform, carbon tetrachloride, etc. in instrument for long time, and use of ammonium hydroxide should be avoided because it has effect on stator and rotor of injector even though it has no effect on pump. When not using it for long time, keep it with iso-propanol filled with in flow path.

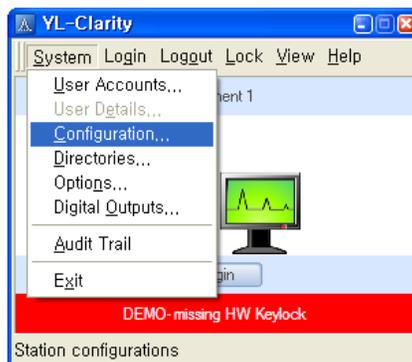
4-3-4. Measures when not uses for long time

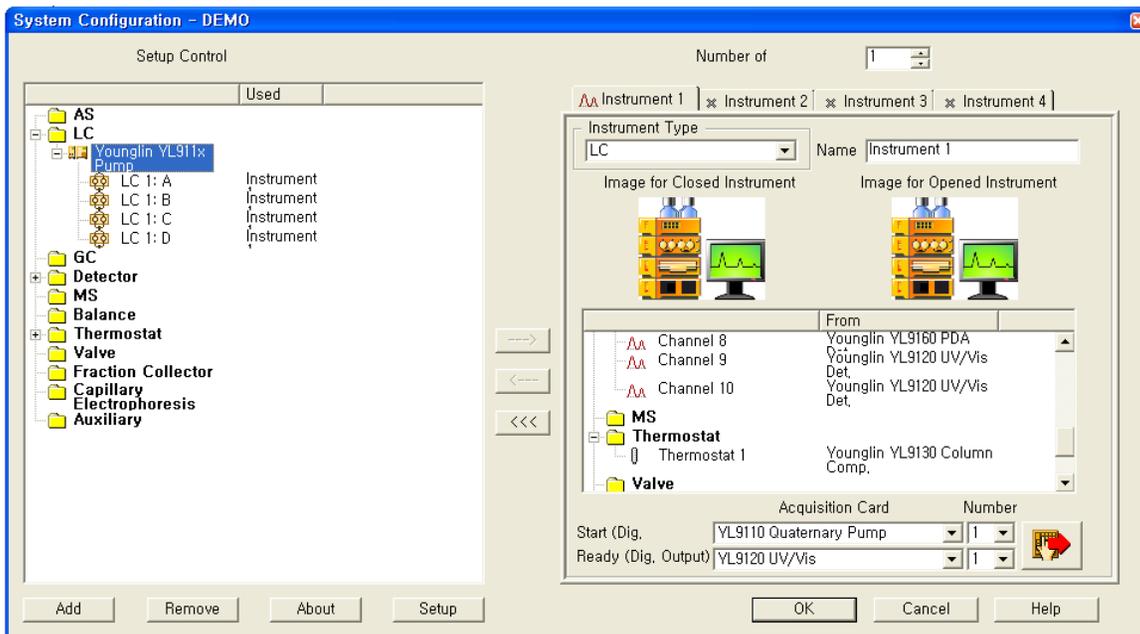
- 1) Prepare iso-propanol for analysis.
- 2) Open prime/purge pump and suck iso-propanol of at least 50ml into instrument.
- 3) Separate outlet tubing of pump.
- 4) Press out iso-propanol sucked into syringe in prime/purge valve and discharge at least 5ml into outlet of in-line filter.
- 5) Separate mobile phase filter assembly and block discharge hole and suction hole with cap.

4-4. YL-Clarity Chromatograph software

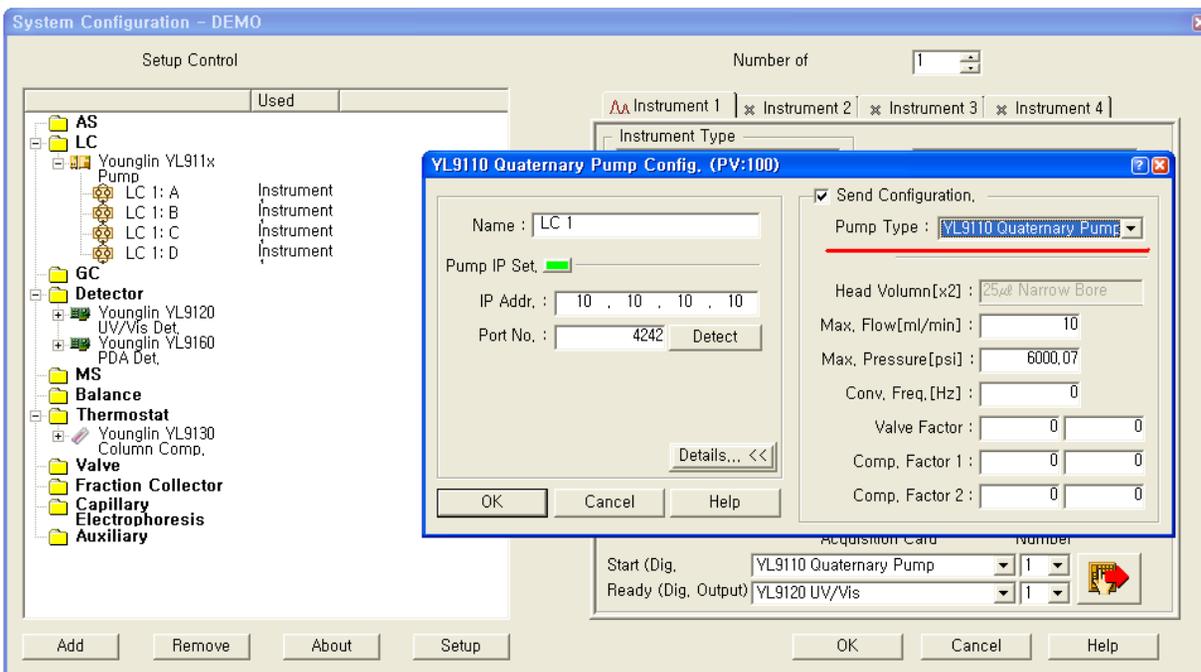
4-4-1. Installation of pump

Open YL-Clarity software and select Configuration on the main window. On the system configuration window, click [ADD] button and select YL911x.



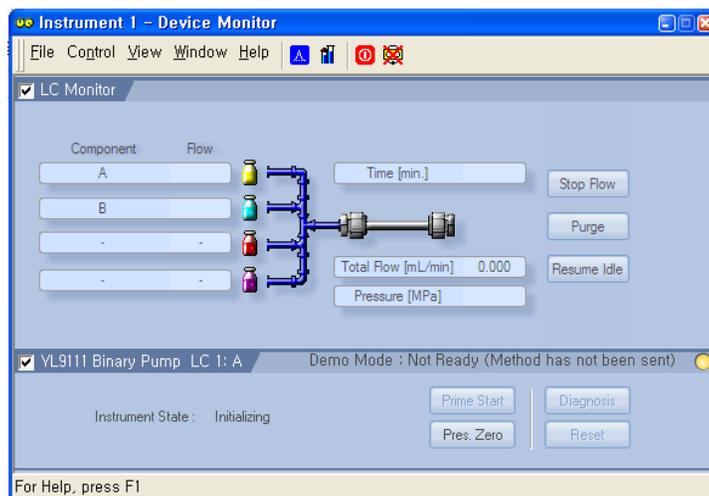


After select YL911x pump on the left window, click arrow button to move this on the right window. Click red arrow button on the right bottom side and select the pressure unit. Double click YL911x pump on the right window, and check IP address of pump. Click "Details" button to select pump type as YL9110 Quaternary pump.



4-4-2. Device Monitor

After configure the pump on the configuration window, log in to open main control window. On the main control window, click Device monitor and then Device Monitor window pops up as below. In this window, can control the pump and monitor instrument status as like flow and pressure.



[Control button]

Stop Flow : To stop the pump operation.

Purge : To run the pump initially. If you click this button, the window for setup solvent and flow pops up.

The pump starts according to the solvent ratio and flow value inputted on this window.

Resume Idle : If you click this button, the pump goes to idle state.

Prime Start : If you click this button, the pump runs at high speed with prime pump to fill the solvent into the lines. This function works only when the pump is not running. The prime pump inside pump runs when you click Prime start button.

Pressure Zero : To set present pressure to zero. Because the offset value of pressure sensor can be changed according to the temperature and using time, the pressure zero is necessary. Before you set the pressure zero, you should drop the pressure completely. This function works only when the pump is not running.

Diagnosis : To self test of instrument.

Reset : To release the pump status from the error.

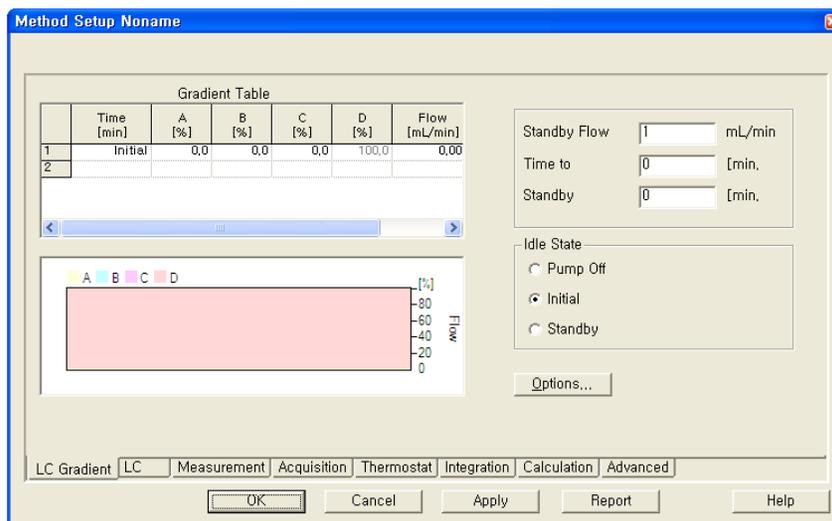
[Status message]

Initializing : It is displayed during initialization.

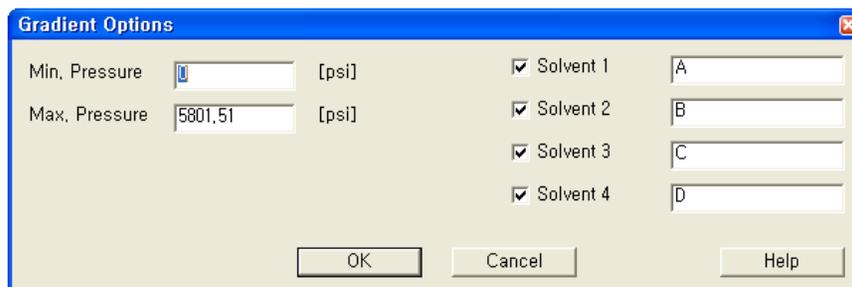
- Ready : It is displayed when the pump is ready.
- Prime : It is displayed during prime/purge status.
- Run : It is displayed during analysis.
- Fault : It is displayed if there is error on the pump.
- Halt : It is displayed if the pump stops.
- Diagnosis : It is displayed during self test.

4-4-3. Method Setup

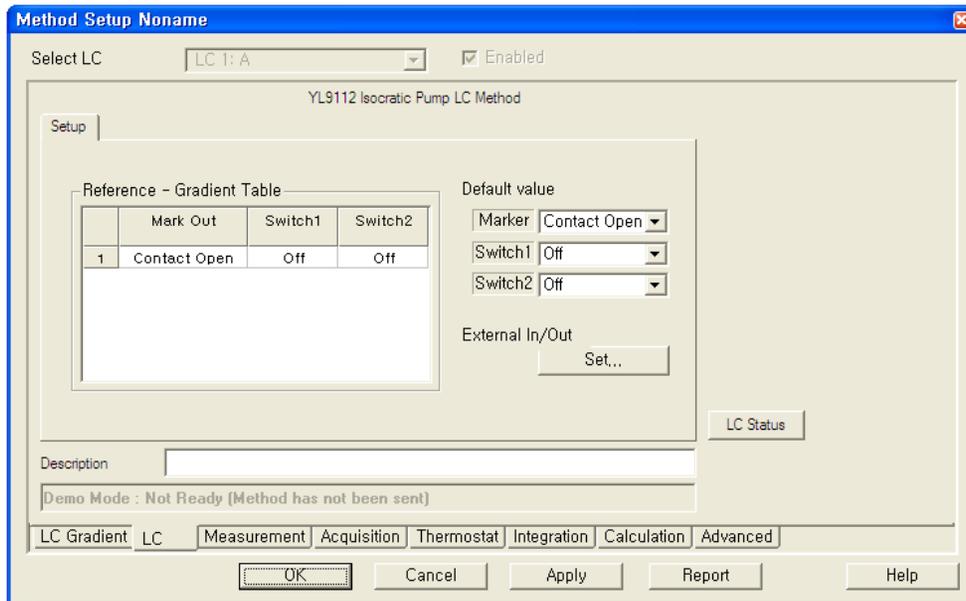
In this window, edit time program table, and setup the pump status during idle state.



Click Options button to setup Max. and Min. pressure limit values to protect column and system. In this window, you can type the name of solvent you will use.



On the below window, make a program for output signal of switch terminal on the rear side of YL9110 Quaternary pump.



If the signal is opposite when you use with the other device, change it on the External In/Out Set.



4-4-4. Error message



If there are errors on the pump caused by pressure limit, control value range, and leak, the pump stops operation with error message.

Chapter 5. Maintenance

In the event that problem occurs or it is necessary to change part due to wear of seal in using YL9110 Quaternary pump, perform maintenance for instrument by referring to the following items.

5-1. Caution

In order to protect instrument, take care for the following items in using it.

- 1) After using solvent with sediment such as buffer solution, replace solvent with pure water at first and then methanol or iso-propanol and make it flow for 30 minutes using each solvent at 1.0ml/min flow rate.
- 2) Do not use solvent to corrode stainless steel material that is less than pH 2.3.

Material	Solvents to avoid
PEEK	Carbon Tetrachloride, Liquid Chlorine Methylene Chloride, Tetrahydrofuran
Teflon(PTFE)	Dimethyl Formamide, Diethylamine
SS316	Phosphoric Acid(Conc, Rm Temp)

- 3) Do not install instrument where corrosive gas is generated or where there is carpet on floor.
- 4) Do not change flow rate rapidly in order to prevent from wrong operation of instrument, damage to column and damage to damper.
- 5) Do not operate instrument with excessive force.

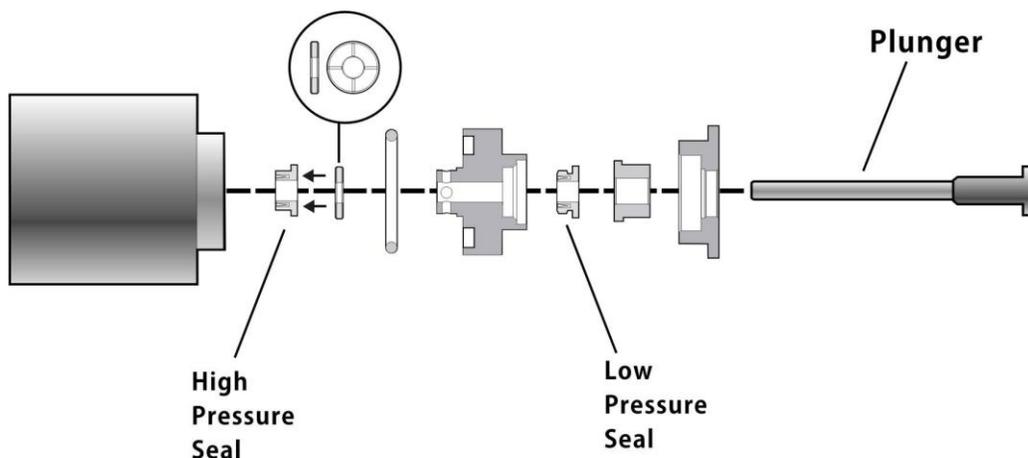
5-2. Replacement of high pressure seal

If instrument is used for long time, high pressure seal is worn out to produce leakage of solution. In this case, after replacing it with new seal, it is necessary to condition it in order to make seal be used for long time at high pressure. Change and condition high pressure seal in the following method. In case instrument is used for the first time after purchasing, it is desired to perform training and it is better to

change all seals of both head when change seal due to long use. Leaked solvent flows out through washing port.

Change of high pressure seal

- 1) Loosen tubing of inlet check valve and outlet check valve of pump head.
- 2) Loosen two head nut for each head using spanner.
- 3) Separate pump head assembly from instrument.
At this time, plunger is left at the place where pump head assembly was loosened. When loosening it, pull it carefully in pump head guide direction and take care not to damage plunger.
- 4) If backup washer in back side of pump head is pulled out, low pressure seal assembly appears. Use seal insertion/removal tool to separate low pressure seal assembly. Then, high pressure seal appears inside of head.
- 5) Remove worn seal with seal insertion/removal tool and insert new high pressure seal prepared at that place using seal insertion/removal tool in the same manner. Direction of seal should be such that the direction to see O-ring is toward front of head. Be careful not to change direction.
- 6) Insert low pressure seal assembly and backup washer.
- 7) Arrange pump head so that plunger left at the place where it is loosened be inserted into center hole of pump head assembly, and then press pump head to main body by inserting pump head by hand. When pressing it, press it carefully so that pump head may be maintained horizontal.
- 8) Tighten head nut in pressed condition. Tighten it so that left side and right side may be same, and tighten it until it is tightened no more by hand while confirming status of tightening finally.
- 9) Replace high pressure seal by applying the process of 1) to 8) to pump head of opposite side.



[Fig. 14] Replacement of high pressure seal

Conditioning of high pressure seal

- 1) Prepare organic solvent such as iso-propanol or methanol necessary. In order to conditioning it, use organic solvent only and do not use buffer solution and base solution.
- 2) Mix iso-propanol or methanol with water by 50:50, and fill instrument with it using the prime/purge valve. And plug the outlet of pump.
- 3) Set the high pressure limit to 2000psi and make flow rate be 0.2ml/min at Quaternary mode, and do not make air bubble be present inside using prime/purge valve.
- 4) Start pump. The pressure will increase upto 2000psi and then the pump will be stopped with a high pressure limit message. Repeat this procedure 2-3 times and then conditioning of seal is completed.

5-3. Replacement of plunger

If piston plunger is used for long time, it should be replaced due to wear.

The worn piston causes leakage of liquid as well as shortens life of high pressure seal. Piston wear is not well observed visually, so care should be taken when observing it.

- 1) Loosen tubing of inlet check valve and outlet check valve of pump head.
- 2) Press pump head to main body of instrument by hand, and loosen head nut.
- 3) Separate pump head assembly from instrument. Then, plunger is left in the place where pump head assembly is loosened. When loosening it, pull it carefully to the direction of pump head guide so that high pressure seal may not be damaged due to eccentricity.
- 4) Pull plunger, replace it with new one at same location, and insert it.
- 5) If there are contaminants on surface of plunger, remove contaminant by applying methanol on cloth without dust.
- 6) Arrange plunger in the manner that plunger may enter the center hole of loosened pump head assembly, and then press pump head by hand so that head may be pressed into body. When pressing it, press it carefully and take care so that pump head may be maintained vertical.
- 7) Tighten head nut with it pressed. Tighten it so that left and right sides may be same in turn, and tighten it until it may be tightened no more by hand while confirming tightening status finally.

5-4. Replacement of check valve cartridge

If check valve is not well operated due to contamination, pressure change is severe during operation and pump does not operate properly. Many problems of check valve are caused by small impurity that interferes with operation of check valve. Therefore, if impurity is prevented from entering inside of pump head using mobile phase filter, malfunction of check valve is almost not caused. The cleaning of the check valve cartridges using sonication can solve this problem.

- 1) Separate tubing connected to pump head.
- 2) Loosen inlet and outlet check valve housing of pump head using spanner.
- 3) Wash check valve cartridge in separated check valve housing for about 30 minutes using ultrasonic cleaner with 10% nitric acid solution.
- 4) Using pure water, rinse check valve cartridge to remove the nitric acid used for cleaning.
- 5) Assemble loosened check valve in the reverse order.

5-5. Replacement of low pressure seal

Wearing of the low pressure seal is caused when pump has been used for long time without using washing port. In order to prevent wear of low pressure seal, it is desired to use washing port, and it is more desirable in case of using buffer solution. Leakage of liquid due to wearing of the low pressure seal is caused between pump head and body.

- 1) Separate pump head with reference to 5.2.
- 2) Separate washer, and pull out low pressure seal assembly from pump headbody with seal insertion/removal tool.
- 3) Replace low pressure seal attached to low pressure seal assembly with new one.
- 4) Assemble pump head in reverse order by referring to 5.2.

5-6. Cleaning of flow path within pump

In order to prevent occurrence of problem in instrument, remove impurity accumulated in instrument, and it is better to clean flow path when it is not used for long time. Clean inside of flow path in the following

method, and be careful when treating strong acid and strong base.

- 1) Separate column inlet tubing connected to column.
- 2) Orient column inlet tubing toward waste bottle.
- 3) Set flow rate at 1ml/min.
- 4) If injector is installed, turn it to injection position.
- 5) Pump 100% iso-propanol through pump and injector for 10 minutes.
- 6) Pump distilled water filtered through pump and injector for 10 minutes.
- 7) Pump 10% nitric acid solution for 5 minutes.
- 8) Wash pump and injector with distilled water filtered for at least 10 minutes.
- 9) Pump 100% iso-propanol through pump and injector for 5 minutes.

Now, pump is prepared for use of mobile phase or for the period not being used for short time or long time. If pump is not used for long time or there is contamination in flow path due to use of impure solvent, it is desired to separate pump head assembly and wash it with ultrasonic cleaner. In order to wash pump head, separate pump head into parts in the same manner as seal change process of 5.2, wash it with ultrasonic cleaner, and assemble each part again. At this time, the high pressure seal is damaged, so replace it with new one.

5-7. Supply of lubricant

YL9110 Quaternary pump necessitates supply of proper lubricant into piston drive part for smooth operation of instrument. It is desired to use lubricant or low viscosity grease for piston carrier and pump housing and small amount of grease such as 630-AA for bearing of cam shaft and piston carrier. Care should be taken because pumping action is interfered with if lubricant is attached to surface of piston. Because shortening of pump life is caused where powder or dust is much generated, install instrument where surrounding environment is good.

5-8. Replacement of solvent filter and in-line filter

In case instrument is used for long time or mobile phase is bad, mobile phase filter and in-line filter is blocked due to small particles contained in solvent. If filter is blocked, pressure within flow path of pump is largely reduced when solvent is sucked to generate air bubble, make flow rate reduced and make

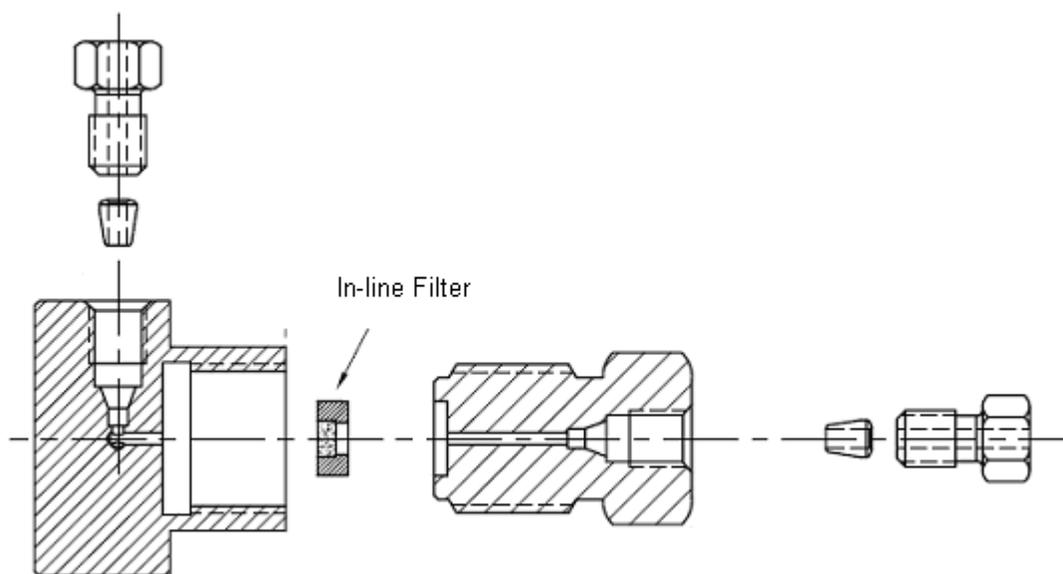
precision reduced ; so it is desired to check it periodically. Main cause of mobile phase filter blocking is growth of bacteria, and two causes to block inlet filter is growth of bacteria and use of solvent containing impurity. In order to prevent growth of bacteria, use at least 10-20% organic solvent or solvent containing growth depressing component. If pure water or soluble solution without interfering material is used, many bacteria will grow in mobile phase filter though it is replaced with fresh solution everyday. Therefore, use solvent of HPLC grade filtered well at all times for mobile phase. Blocking of in-line filter is caused by accumulation of small particles generated due to wear of high pressure seal by using of impure solvent and long use of instrument. In case mobile phase filter and in-line filter are contaminated, condition of filter may be improved by washing it by ultrasonic cleaner with 10% nitric acid solution for 30 minutes. If it is not improved by ultrasonic wave cleaning, replace it with new filter.

Change and cleaning of mobile phase filter

- 1) Separate mobile phase filter from tubing. As the surface of teflon tubing of mobile phase filter of insertion type is slippery, separate it with tubing held avoiding slippage using #1000 sand paper.
- 2) In case of performing ultrasonic wave cleaning, wash head part by ultrasonic cleaner with 10% nitric acid solution for 30 minutes, wash it again by ultrasonic cleaner with pure water for about 10 minutes, then dry it. In order to replace filter with new one, prepare new mobile phase filter of same size.
- 3) If washing has been completed, assemble filter to be replaced newly again. Hold teflon tubing using sand paper and insert mobile phase filter of insertion type with center adjusted into middle hole.

Changing and cleaning of in-line filter

- 1) Separate connected tubing from in-line filter assembly using spanner.
- 2) Separate head part of in-line filter assembly from body using spanner.
- 3) In case of performing ultrasonic wave cleaning, perform ultrasonic wave cleaning to head part for 30 minutes with 10% nitric acid solution, perform ultrasonic wave cleaning for 10 minutes with pure water, and then dry it. In order to replace filter with new one, separate filter located at back side of head part.
- 4) In case of replacing it with new filter, replace it with new in-line filter located at the location where it was separated ; and in case ultrasonic wave cleaning has been completed, re-assemble head part of dried assembly.
- 5) Using spanner, tighten head part sufficiently so that there may be no leakage of liquid even at 6000 psi.



[Fig. 15] Replacement of in-line filter.

5-9. After use system

If you keep the pump without cleaning, the pump can be damaged by the crystallization of salt, growth of microorganism, contamination. To maintain the pump properly, run the pump using iso-propanol for 30 minutes and the plug the outlet of pump, and keeps clean the solvent filter. Especially, after use buffer solution, run the pump using 100% water first then iso-propanol.

5-10. Troubleshooting

In case general problem occurs as the following table, confirm the possible causes regarding this first, and then take proper countermeasures. The following table is countermeasure in case general problems occur.

Problem	Cause	How to fix
Pressure upper limit is loaded.	<ul style="list-style-type: none"> -Tubing inside is blocked. -Check valve is blocked. -Solvent is changed. 	<ul style="list-style-type: none"> -Replace blocked tubing by loosening to be from tail side in turn. -Replace outlet check valve. -Wait until solvent is completely changed.
Pressure increases or decreases.	<ul style="list-style-type: none"> -Change of solvent is incomplete. -Column is unstable. 	<ul style="list-style-type: none"> -Wait until change is completely performed. -Wait until pressure is stable.
Solvent is not flowed out	<ul style="list-style-type: none"> -Air bubble is in pump head. -Air separation status of solvent is bad. -Check valve is not good. -Liquid containing oil flows into head part. -High pressure seal was worn. 	<ul style="list-style-type: none"> -Perform prime/purge again. -Take measures so that air separation condition of solvent may be good, and perform prime/purge again. -Wash or replace check valve. -Remove oil in head sufficiently with strong organic solvent using prime/purge port. -Replace the high pressure seal.
Pressure is unstable.	<ul style="list-style-type: none"> -Check valve is bad or defected. -Air separation or mixing conditions of solvent is bad. -Compression compensating reference value of configuration mode is wrong. -Cam shaft is loosened. -High pressure seal was worn. -Pump head was loosened. 	<ul style="list-style-type: none"> -Wash or replace check valve. -Use mobile phase with well mixed solvent, and improve air separation condition. -Initialize compensation value or re-input proper value. -Tighten wrench bolt of cam shaft -Replace high pressure seal. -Tighten nut of pump head.

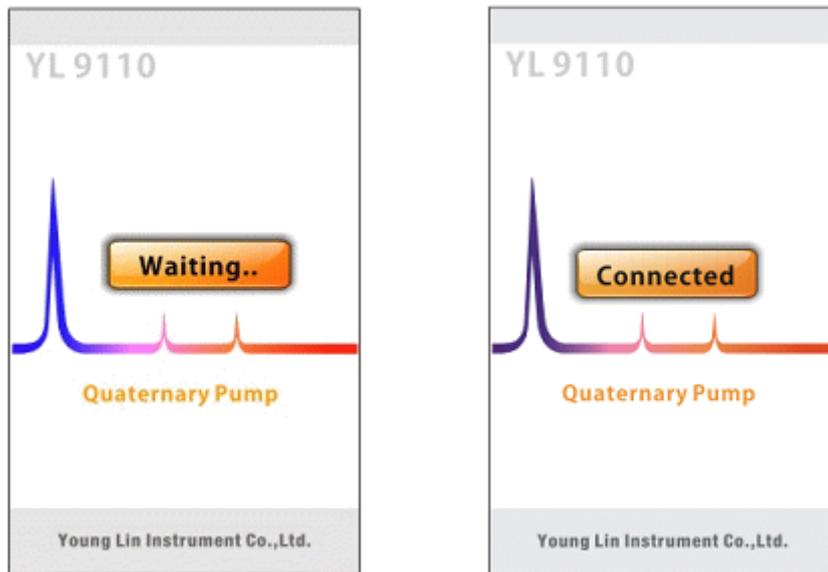
<p>Liquid is leaked or pressure reduces when cleaning port is not used</p>	<ul style="list-style-type: none"> -High pressure seal was worn. -Plunger was worn. 	<ul style="list-style-type: none"> -Replace high pressure seal. -Replace plunger.
<p>Liquid is leaked from inside of instrument.</p>	<ul style="list-style-type: none"> -Fitting in instrument was loosened. -Damper was damaged. -Low pressure seal was worn. 	<ul style="list-style-type: none"> -Tighten fitting in instrument. -Replace. -Replace low pressure seal.
<p>Pump is not operated after input of power.</p>	<ul style="list-style-type: none"> -Power voltage is unstable or low. -Strong induction voltage is generated in the surroundings. 	<ul style="list-style-type: none"> -Use stable, proper DC power source. -Close induction power source in surroundings, or install instrument away from power source.
<p>Noise is too high</p>	<ul style="list-style-type: none"> -Load is caused to piston carrier. -Timing belt is loose. -Motor is in defect. 	<ul style="list-style-type: none"> -Supply lubricant in carrier body. -Reduce clearance above belt by adjusting guide location of timing belt. -Inspect connection status of motor cable, and replace motor if noise is severe only in high pressure.

YL9110 series Pump Touch Pad control

1. Turn ON

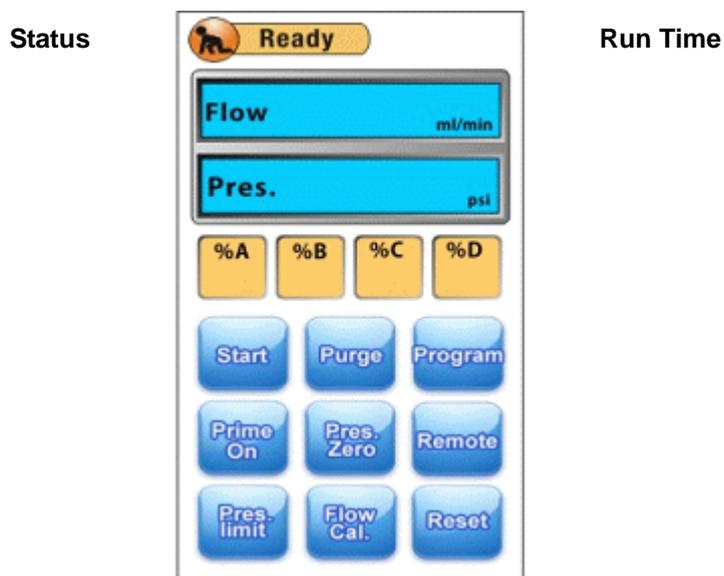
Turn on the power of pump, then the Model name and [Waiting] sign are displayed on the LCD. After connected with pump, it shows [Connected] sign.

If the model name is different, please contact to YL instruments or your local supplier.



2. Main window

If you touch the [Connected], the display is changed Menu window.



3. The pump status

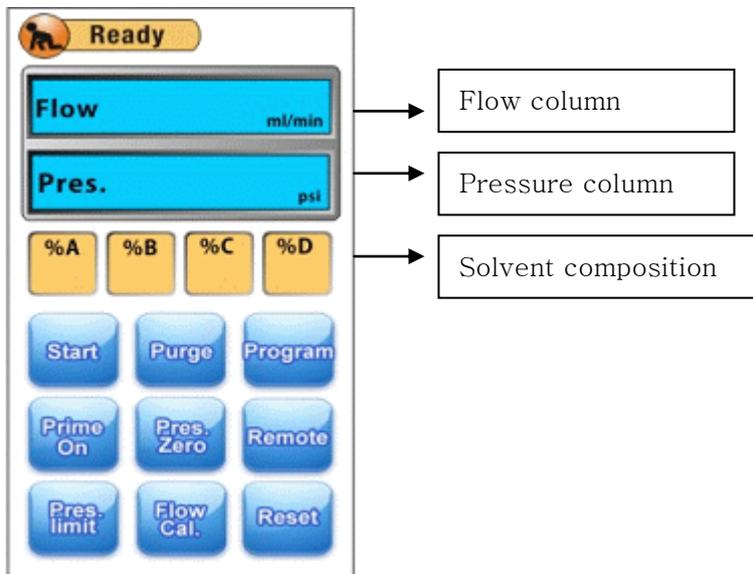
Ready, Running, Stop, Error are displayed on the left top of LCD and the Run time is displayed on the right top during running.

4. Display

The Flow column shows the flow rate at present.

The Pressure column shows the actual pressure of system.

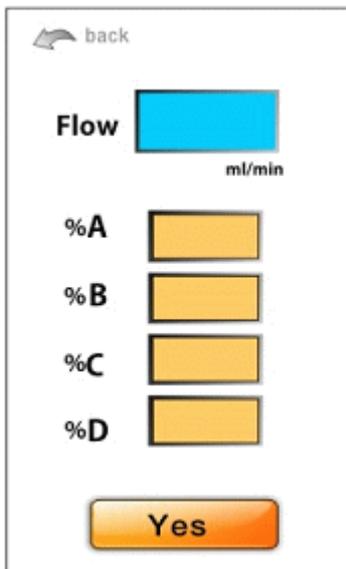
A, B, C, D column shows the solvent composition.



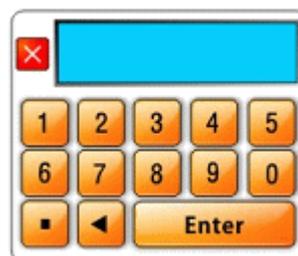
5. Function key

-[Start] : Start the pump or Stop the operation. If you press one time it works as a Start key. If you press this button during operation, it works as a Stop key.

-[Purge] : Used for control of Solvent composition and Flow rate. If you press this button, it shows as below.



If you click Flow column(blue box), the following window is displayed. Input the values want to use and select Enter.



To input the solvent composition, select each column(%A, %B, %C, %D) and input the ratio value. If the sum of these values is not 100%, it can not move to next step.

-[Program] : It is used for gradient table setup.

No.	1	2	3
Time (min)			
Flow (ml/min)			
%A			
%B			
%C			
%D			

Input the values on the blank column of Time, Flow, and Solvent ratio. The sum of Solvent ratio should be 100%. If it is not 100%, can not save the gradient table.

The maximum number of input line for gradient table is 12 and this table is saved as a Method by [Save] button.

The number of method can be saved is upto 6.

-[Prime On] : It is used for priming and purging solvent into the pump.

-[Pres. Zero] : It is used for reset the pressure zero value.

Pressure zero

Do you want to apply Pressure zero?

Yes

If you click  button to reset the pressure zero, the message pop up on the left is displayed. If you click Yes, the present pressure value is changed. By the  icon, close this window without reset the pressure.

-[Remote] : To disconnect the communication between the pump and touch pad.

Remote

Do you want to apply Remote?

Yes

If you want to use Remote mode, just click .

-[Press Limit] : To setup the high pressure limit value.
The default value is set on the factory.

6000

1 2 3 4 5

6 7 8 9 0

Enter

-[Flow Cal.] : To calibrate the actual flow rate by input the multiplying value.



The default value is set on the factory

6. Error message



1) Leak



If there is a leak on the pump, this window is displayed.

After resolve the leak on the pump, click . Then this

window is disappeared and shows



2) High press.



If the system pressure is higher than high pressure limit, this window is displayed. After remove the reason of high pressure,

Click . Then this window is disappeared and shows



YL9100 HPLC SYSTEM

YL9111 BINARY PUMP

USER MANUAL



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Chapter 1. Introduction

YL9111 Binary Solvent delivery pump is a pump for High performance liquid chromatograph. It provides high performance and functions as a HPLC pump, and controlled by software.

YL9111 Binary pump uses specially designed cam and pulse damper for stable solvent delivery and has a compressibility compensation function for accurate and precise solvent delivery.

And the pump has a automatic rinsing function to prolong the life time of pump head. Using UHMWPE seal, it provides extended life time of high pressure seal even though use buffer solution as a mobile phase. On the outlet of pump, there is a in-line filter to prevent small particles come into the column and also protect column by the pressure limitation setup. YL9111 Binary pump consists of two independent dual carrier assembly to provides high pressure gradient operation. YL9111 Binary pump provides auto prime/purge function for easy operation and fast exchanging of solvent. This instrument manual includes basic principle, installation and operation method, and troubleshooting to use YL9111 Binary pump properly.

1-1. Specifications

- 1) Operating principle : X2 Parallel dual-plunger pump, high pressure gradient
- 2) Compressibility compensation : Automatic
- 3) Flow range : 0.001-10ml/min
- 4) Flow rate accuracy : $\leq \pm 1\%$ at 1ml/min
- 5) Flow rate precision : 0.1% RSD at 1ml/min
- 6) Maximum pressure : 6000 psi
 - A. Operating range : 0-6000 psi up to 5ml/min
 - B. Operating range : 0-3000 psi up to 10ml/min
- 7) Pressure pulsation : $\leq \pm 1\%$ at 1ml/min
- 8) Composition Precision : $< 0.1\%$
- 9) Composition Accuracy : $< 0.5\%$
- 10) Auto prime/purge
- 11) Communications : LAN
- 12) Safety & maintenance : Leak detection, Diagnostics, Error detection
- 13) Dimensions : 385 X 160 X 565mm (width X height X depth)
- 14) Line Voltage : 100-240VAC, $\pm 10\%$, automatic voltage selection
- 15) Line frequency : 50/60Hz, $\pm 5\%$
- 16) Power consumption : 140W

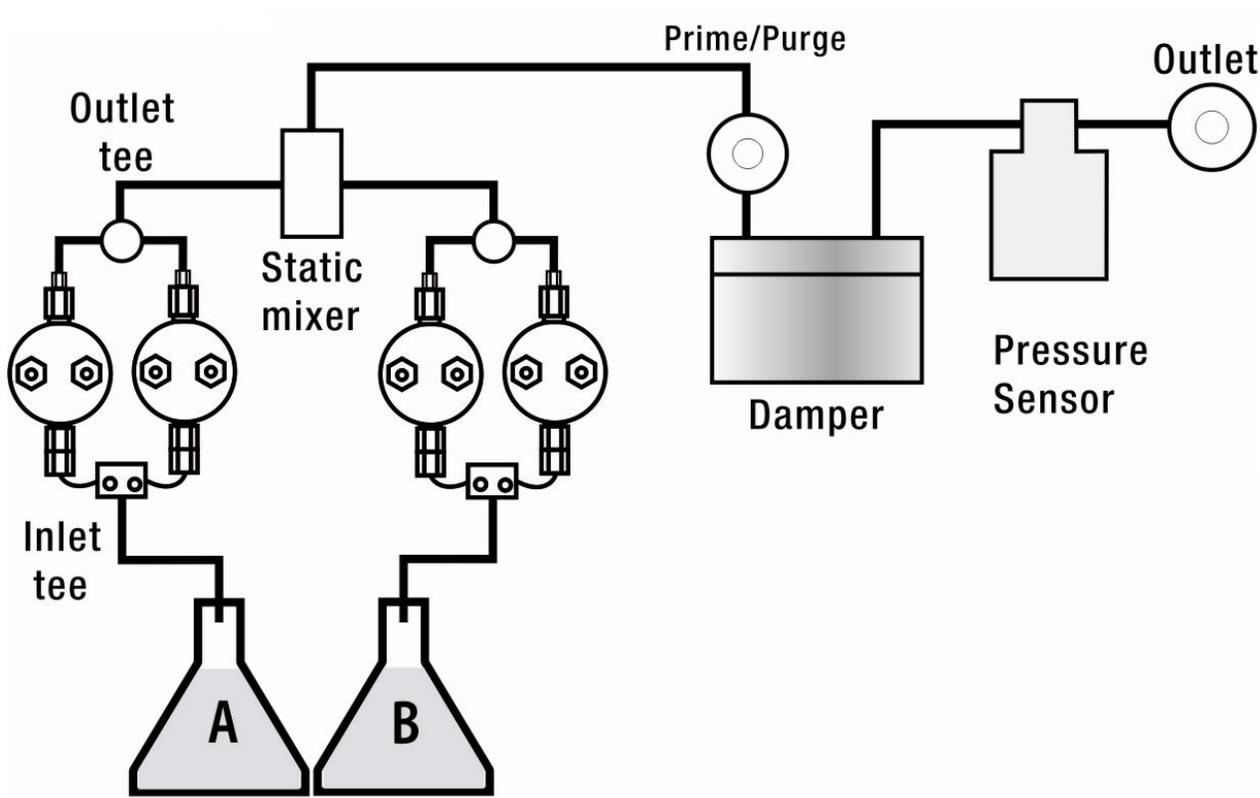
[Optional specification]

Head type	Narrow Bore	Analytical	Semi prep
Head volume(μ l/each)	25	64	144
Flow range(ml/min)	0.001~5	0.001~10	0.01~50
Flow range(ml/min)	0.005~5	0.01~10	0.05~25
Pressure(psi)	5000psi	6000psi	3500psi
In-line filter (micron)	2	2	10
Material	Zirconium, Ruby, PEEK UHMWPE, PTFE	SS316, Zirconium, PTFE, Sapphire, UHMWPE	SUS316, Sapphire, Ruby, UHMWPE, PTFE

Chapter 2. Configuration and Principle

2-1. Configuration of flow path

YL9111 Binary pump consists as same with figure 1. It uses Teflon tubing between pump inlet and inlet check valve, SUS316 or PEEK tubing from outlet check valve.



[Fig. 1] Flow diagram of YL9111 pump

- **Solvent Filter**

The solvent filter is used for protecting the system from the particles in solvent. This filter removes particles from solvent to prolong the life time of high pressure seal and prevent damage on the column. It is recommended to use the solvent filter when you use YL9111 Binary pump with or without degassing module. Select proper filter depending on the column and flow rate.

- **Pump Head Assembly**

Pump head is real working part to deliver solvent by piston movement and check valve. It consists of plunger, check valve, high pressure seal, low pressure seal and rinsing port. YL9111 Binary pump built in automatic rinsing port to clean the head assembly efficiently if uses buffer solution.

- **Auto Rinsing Pump**

Auto rinsing pump delivers cleaning solvent from the rinsing solvent bottle to the inside of pump head. YL9111 Binary pump rinse the system every 3 minutes automatically.

- **Prime/purge valve**

This valve is used for priming the pump. Fill the solvent inside of tubing from the solvent bottle if you use the pump for the first time or the tubing lines are empty. Remove a plug on the prime/purge valve and suck the solvent using syringe, or click "Prime Start" button on the software to operate the micro pump.

- **Prime micro pump**

Using this pump, the pump circulates rinse solvent into the pump head.

- **Mixer**

It is static mixer to improve mixing efficiency of solvent.

- **Pulse damper**

YL9111 Binary pump reduces pulse from the cam operation by the diaphragm damper. YL9111 Binary pump provides constant and pulseless flow using compressibility compensation and pulse damper, so the detector that affected by flow stability can be used with YL9111 pump.

- **Pressure transducer**

It checks real time system pressure. The pump uses this pressure to protect system and to operate compressibility compensation and even compensation. YL9111 Binary pump uses continuous flow path type pressure sensor.

- **In-line filter**

It removes fine particles that are not filtered by solvent filter or made by worn of high pressure seal.

2-2. Operation

There are essential parameters on the pump as like flow accuracy, precision, and reproducibility to get the reliance of analysis data and low detection limit. YL9111 Binary pump uses high pressure resistant dual pump head, controls microprocessor to monitor the phase of cam to remove pulse, so fulfill the necessities of solvent delivery pump.

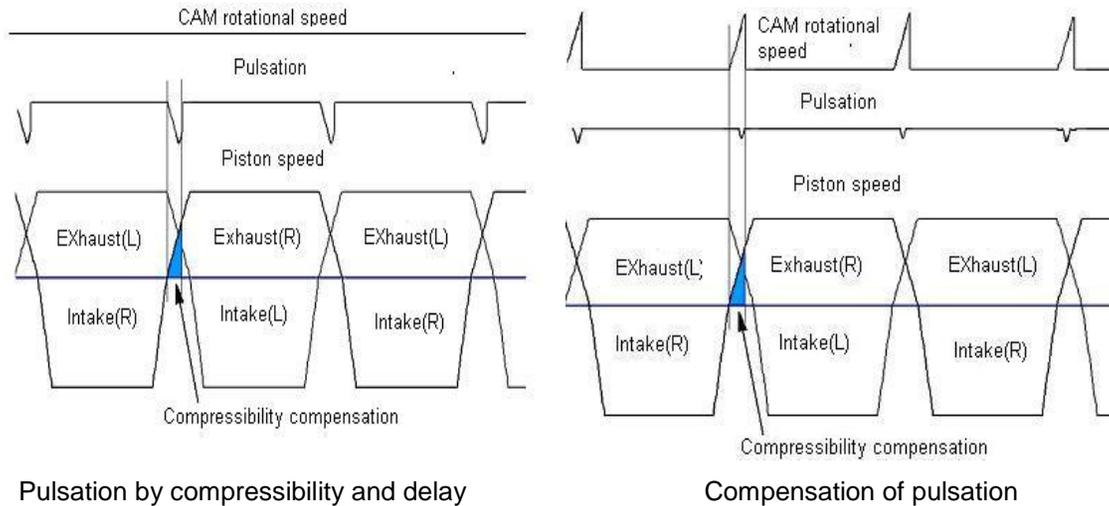
Diaphragm pulse damper reduces pulse from the cam operation more than 90% between low and high pressure range by internal elastic body of damper, and also works well as a mixer for gradient elution. The elastic body of Diaphragm pulse damper improves flow accuracy through compression, expansion procedure to make variance of kinetic energy by flow constant. The amount of contact solvent with pulse damper is around 1.5ml at 3000psi, which ensures that flow path is completely cleared away.

YL9111 Binary pump was designed so that integrated flow rate may realize no-pulse operation using specially designed cam. However, pulse incapable of being neglected is caused actually due to compressibility of mobile phase proportional to pressure and elasticity of high pressure seal, so the pump is controlled in real time so that occurrence of pulse proportional to pressure may be depressed. Control method of the Pump uses supervision of pressure and control of location simultaneously, so it has advantage to improve precision and accuracy of flow rate without being affected by range of pressure and flow rate. There is a part for microprocessor control to realize various function of pump including stepping motor control. Stepping motor control processor operates motor as a micro step, so can achieve constant motor speed at low flow rate with low noise.

Operation mechanism is for transmission of kinetic energy from step motor to piston. This mechanism includes specially designed cam, stepping motor, carrier, carrier housing, and phase sensor.

2-3. Compressibility compensation

Most of pumps for HPLC analysis are used at high pressure. However, pulse occurs in high pressure due to compressibility of liquid and elasticity ratio of seal, so flow rate is also reduced. Occurrence of pulse due to this reduces precision and accuracy of pump flow rate, so compensation is necessary for this. YL9111 Binary pump monitors actual pressure and calculates compensation value for this; compressibility compensating operation to control angular velocity of cam with this value reduces occurrence of pulse flow remarkably as well as improves accuracy of flow rate largely.



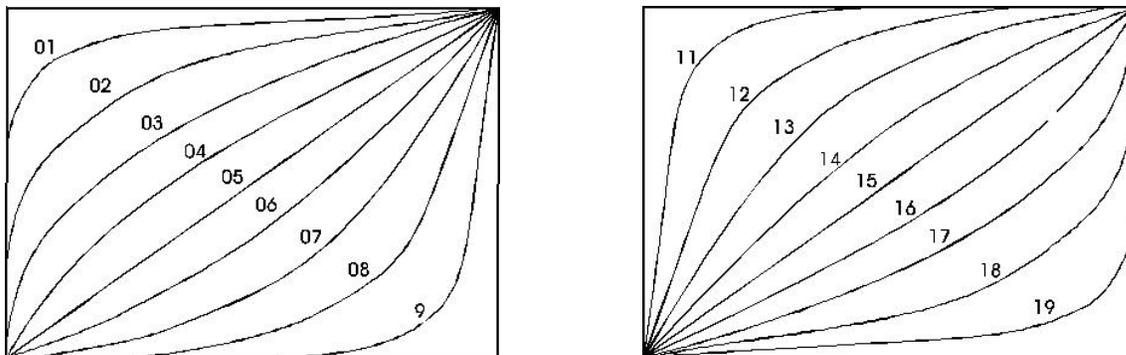
[Fig. 2] Delivery mechanism of YL9111 Binary pump

2-4. Gradient Curve

Solvent A and solvent B are mixed with flow rate F_b ,
 Mixed solvent flows to the column with flow rate F_{ab} ,
 Solvent volume V of solvent A vs Concentration of solvent B

$$C(\%) = 100 \times \{1 - (F_{ab} - F_b) / V \times t\}^{(F_b / F_{ab} - F_b)}$$

The solvent ratio difference between present row and next row increases or decreases as a exponential curve. In general, the gradient elution is used for separation of duplicated peaks. YL9111 Binary pump has a exponential curve program, but use gradient elution by linear curve when it operates with YLClarity software.



[Fig. 3] Gradient curve

2-5. Rinse port

When using buffer solution, salts are generated on back side of high pressure seal and these deposits wear pump seal to cause shortening of seal life, which has bad effect on pump.

Rinse port enables to insert proper solvent in back side of high pressure seal to prevent salts from being deposited and activated. Mixed solution(20% MeOH) of water and methanol is used as cleaning solution, and life of seal is extended with lubrication action in general analysis..

Chapter 3. Installation

3-1. Inspection and site preparation

YL9111 Binary Pump is delivered along with the following parts when being shipped. Before opening transportation package, perform inspection for trace of shock or mistake, and if there is abnormality, do not open the contents and inform this company of it. And, if contents are opened, perform inspection for existence of shock in the contents and contact with this company when trace of shock is found.

The Gradient Pump is a delicate instrument, so use original box and buffer material as far as possible when re-packing it to transport instrument. If it is impossible to use original box; wrap pump with several layers of buffer material, and fill the bottom, top and all other sides of pump with buffer material in order to make pump endure shock or vibration during transportation.

Standard configuration of YL9111 Binary pump

- 1) Main body of instrument
- 2) Power cord and fuse
- 3) Tubing 60cm,
 - A. Bio Narrow Bore : ID 0.01" , OD 1/16" PEEK
 - B. Analytical : ID 0.01", OD 1/16" SUS316
 - C. Semi-prep : ID 0.02", OD 1/16" SUS316
- 4) Installation kit
- 5) Manual

Site requirement of YL9111 Binary pump

- 1) Room with 20 °C temperature with variation $\pm 5^{\circ}\text{C}$ with and 60% humidity
- 2) Where no direct and straight sunlight
- 3) Plain floor without carpet
- 4) Where having spare space of 20cm or more
- 5) Where there are 10% or less voltage change, no frequency change and 100 Ω or less grounding point
- 6) Where there is no generation of corrosive gas and ventilation is well done
- 7) Where stable power of 110 or 220V is supplied, $\pm 10\%$
- 8) Where not receive electromagnetic induction from large transformer, high frequency heater, UPS, etc.
- 9) Within 2500 m above sea level(storage within 4600m)

Please check the following before you install the system.

- 1) Keep the ventilation as normal state.
- 2) Install on the stable place. Avoid the places as like near to air conditioner and heater, direct sun light, near to window.
- 3) Keep the place without dust and vibration.
- 4) Maintain voltage variation within 5% of proper voltage.
- 5) Avoid high frequency or strong magnetic field environment.
- 6) Avoid from the source of fire(spark, flame).
- 7) Keep the proper ground for electricity.
- 8) Check the place of water supply for emergency.

Caution ! : Keep distance with CRT at least 50cm.

3-2. Connection of power

Confirm supplying voltage, electric power, socket, and type of connector are correct. Before connect power cable on the pump, please turn off the power switch of YL9111 Binary pump.

Warning ! : The electric power should be matched with description on the rear side of instrument. If not, the instrument can be damaged or it can be a reason of fire.

Caution ! : Confirm the ground is correct. Water pipe is not recommended in case of non-metal pipe. Gas pipe should not use because of safety. If the ground is not correct, the electric shock can be happened.

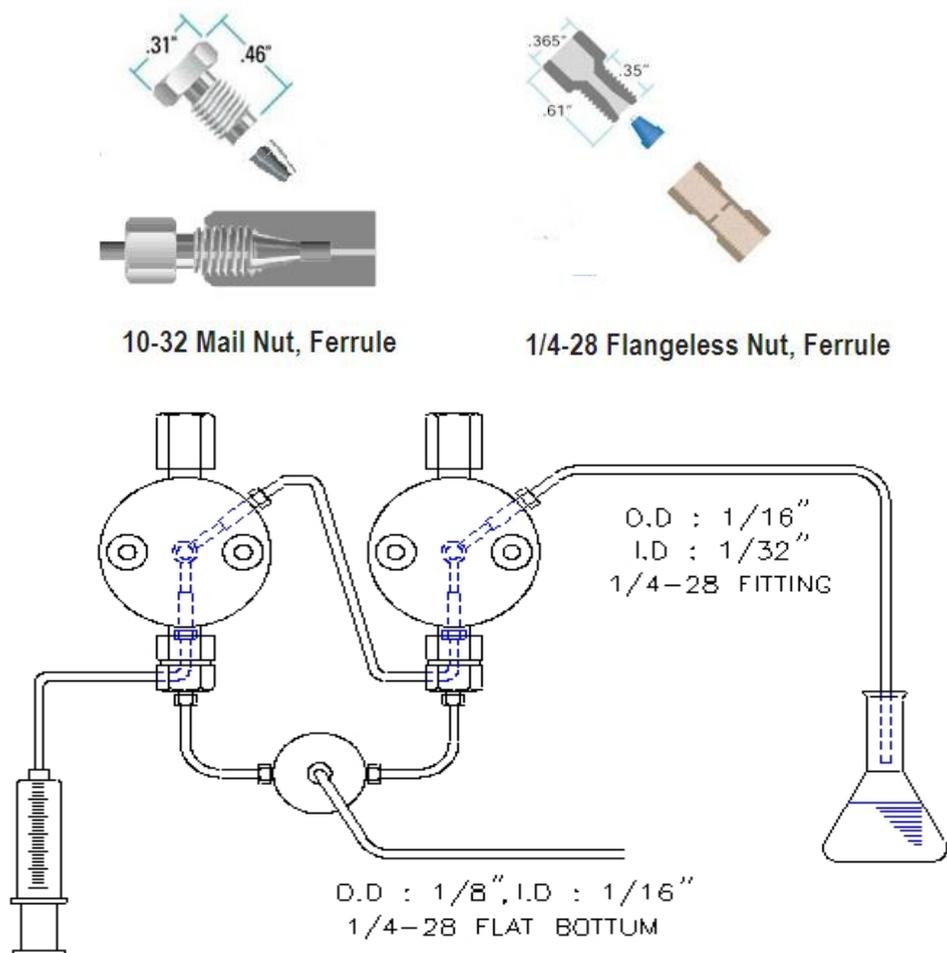
3-3. Connection of tubing

YL9111 Binary pump uses following fittings for connection.

Flow path		Material	OD	ID	Fitting(UNF)
Inlet Tee		Teflon	1/8"	1/16"	1/4-28(flat type)
Inlet Tee ~inlet check valve		Teflon	1/16"	1/32"	1/4-28(flat type)
Outlet check valve~ In-line filter	Narrow bore	PEEK	1/16"	0.02"	10-32
	Analytical	SUS316	1/16"	0.02"	10-32
	Semi prep	SUS316	1/16"	0.03"	10-32

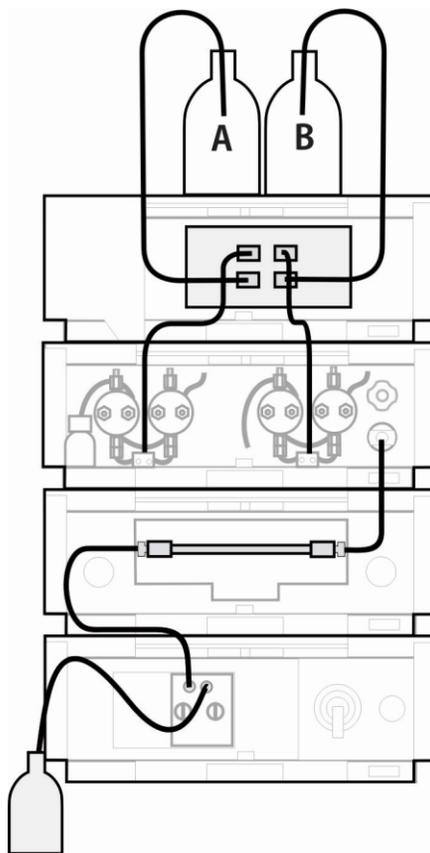
The cut surface of tubing should be cut at right angle without dust, tube should not be contracted, and middle inner diameter shall not be blocked.

In order to cut stainless steel tubing, tubing cutter should be used, plastic tubing cutter or shaving cutter should be used for teflon and similar material of tubing, and the surface should be clean and have no crumbling.



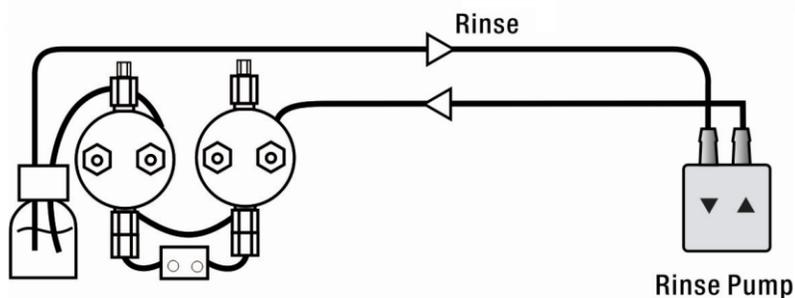
[Fig. 4] Fitting for 1/16" OD tubing

Inlet tubing of pump connects with degassing module using 1/4-28 fitting or with solvent bottle directly, the outlet tubing connects with injector. The fitting for injector is different depending on the injector type.



[Fig. 5] Connection between YL9111 Binary pump and YL9100 HPLC system

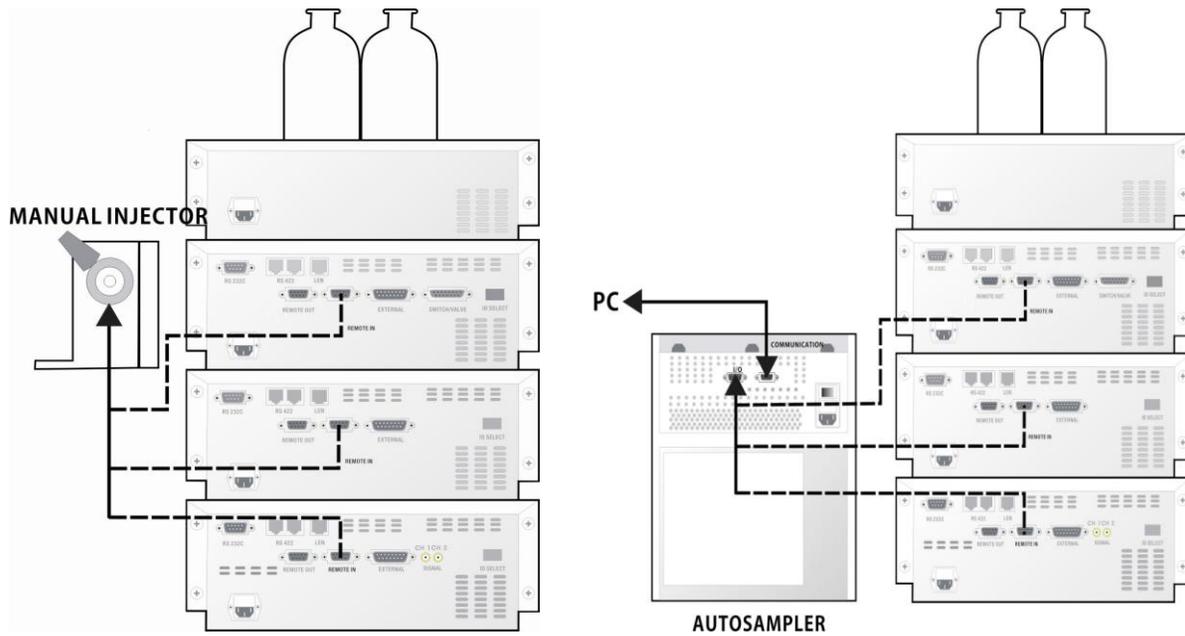
Rinse port tubing connects on the each pump head using 1/4-28 fitting as same as [Fig. 7], and fill the 20~50% Methanol. The rinse pump inside of YL9111 Binary pump circulates the rinse solvent into the pump head every minute. Check and replace rinse solvent once a week at least.



[Fig. 6] Tubing connection of rinse port

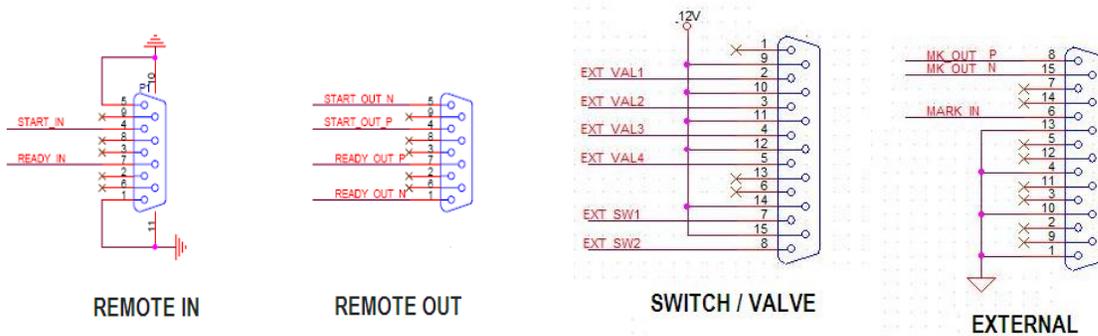
3-4. Connection of remote cable

YL9111 Binary pump has connection terminals for remote input/output, external solenoid valve, and marker input/output. The remote cable from the injector(manual or autosampler) has to be connected on the Remote In terminal on the rear side of YL9111 Binary pump to collect data at the moment of injection.



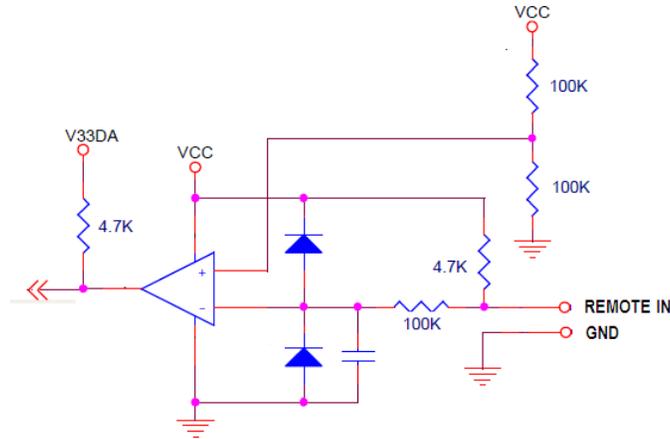
[Fig. 7] Connection of remote cable between YL9111 pump and injector

Notice ! : Please do not connect wires between cables at your discretion. If you want to connect with the other instrument, please check input/output information and confirm with YL9111 Binary pump terminal configuration.

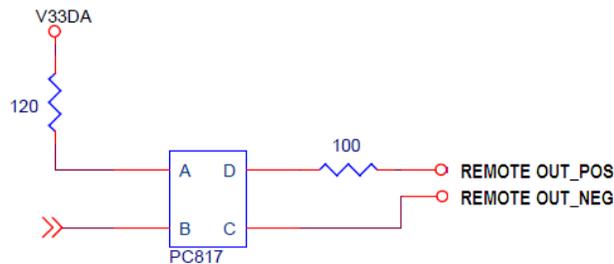


[Fig. 8] Pin configuration of each terminal

[Fig. 10] and [Fig. 11] are the diagram of remote and the other terminal input/output. In between YL9100 series modules, connect directly and confirm the configuration with the other modules.

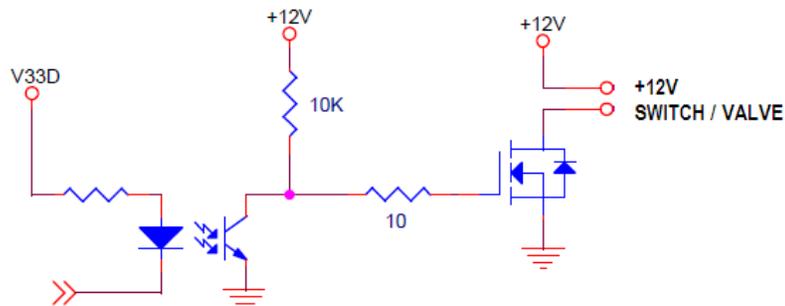


[Fig. 9] Diagram of Remote and Marker input



[Fig. 10] Diagram of Remote and Marker output

YL9111 Binary pump provides output signal(12V 500mA) to operate external solenoid valve as like [Fig 12].



[Fig. 11] Diagram of solenoid output

[Remote operation]

START-IN : Operate instrument, and start running of gradient program.

If you connect it with autosampler or external valve, automatic running is available.

START-OUT : If the signal input on the START-IN terminal, the signal pulse output through this port. It can be used for synchronization of remote start with the other instrument.

MARK-IN : To control event program or operate additional operation.

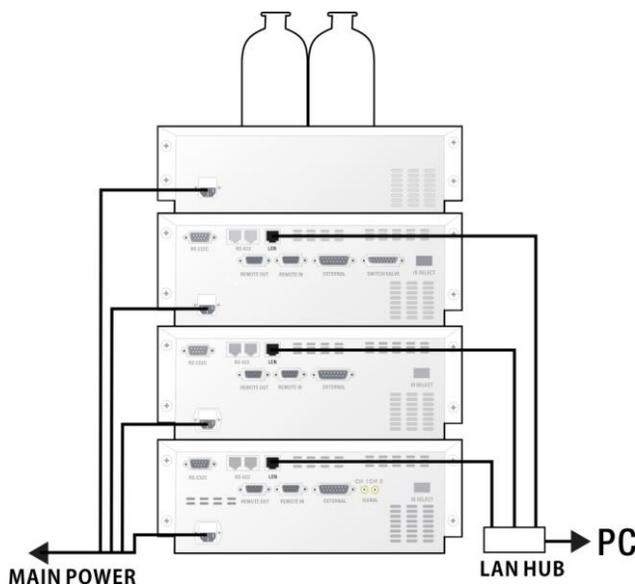
MARK-OUT : To control time event program output.

READY-IN : To change error state and stop operation if there is a input.

READY-OUT : When YL9111 Binary pump is not ready state because of running status, output error signal is indicated if there is a leak.

3-5. Connection of communication cable

YL9111 Binary pump provides TCP/IP internet protocol as a standard. The IP address of YL9100 pump series is 10.10.10.10, if DIP SW settings on the rear side are On position. If you change the IP address using control software, the DIP SW has to be set OFF.



[Fig. 12] Connection of communication cable

Notice ! : The LAN HUB used for cable connection on the PC must use switching mode module.

Chapter 4. Operation

There are four LEDs in front of YL9111 Binary pump.

	POWER	LED turns ON if main power turns on
	CONNECTED	LED turns ON if communication is connected, LED blinks during connection
	READY/RUN	LED turns ON before analysis, LED blinks during analysis
	ERROR	LED turns ON if there is error

4-1. Before Start

When using pump for the first time, initialize it through the following process in order to clean flow path and condition high pressure seal. This process is necessary in case instrument is installed newly or is not used for long time.

- 1) Prepare iso-propanol of HPLC grade.
- 2) Remove residual air bubble within instrument by turning prime/purge valve in counter clockwise and loading iso-propanol of at least 50ml by Prime Start button.
- 3) Separate pump outlet tubing.
- 4) Press the sucked iso-propanol into syringe with prime/purge valve and discharge more than 5ml to outlet of in-line filter.
- 5) Operate pump with instrument outlet open for 2-3 hours at 0.2ml/min flow rate and for 1 hour at 1.0ml/min flow rate using iso-propanol.
- 6) Perform process of 2) ~ 5) using solvent which will be used as a mobile phase.
- 7) Remove inside residual iso-propanol by operating it at 1ml/min flow rate for 30 minutes with instrument outlet open.
- 8) Form flow path by connecting injector, column, and detector tubing mutually.

4-2. Mobile phase filter and bottle

Solvent vessel should be positioned at higher location than pump and not be positioned below pump, and inlet tubing length should be as short as possible. This can minimize pressure drop caused at inlet of pump during suction.

When using solvent having high vapor pressure as hexane, formation of air bubble is caused due to large pressure drop in suction part in high flow rate; so particular care should be taken, and mobile phase should be maintained after air separation, filtration and air-tightening.

Mobile phase filter of 10 μ m porosity is connected into inlet tubing in order to prevent entering of small particles. Mobile phase filter is blocked if mobile phase is bad or is used for long time, it is necessary to clean or change filter in this case.

4-3. Preparation of solvent

Proper solvent prevents various problems happened during actual analysis.

Solvent gas removal and filtration are necessary because they have great effect on result of analysis and maintenance of instrument.

4-3-1. Degassing

Solvent gas removal is performed in order to remove gas such as nitrogen or oxygen contained in mobile phase. Contained gas should be removed by air separation before mobile phase is used or while mobile phase is used, and the most practical technology for air separation is to insert helium into solvent.

Helium is easily separated from HPLC solvent, so other gases contained in solvent may be easily removed due to diffusion of helium gas.

When mixing organic solvent such as methanol or acetonitrile into water, this mixture contains very small quantity of gas as compared to the quantity of pure composition; so it has more strong tendency to discharge gas. Back pressure regulator attached to outlet of detector prevents formation of noise in base line due to air bubble, and mobile phase vessel should be pressurized under 2-3psi pressure with helium if it is desired to reduce gas discharge due to solvent mixing.

4-3-2. Filtration

Solvent should be necessarily filtered through 0.45 μ m or less filtering membrane before use. Removal of small particles is necessary to compensate reliable operation of piston seal, and is necessary measure for reliability of other components in liquid chromatograph.

Filtration process is necessary after mixing of solvent, and is more necessary in case of buffer to which un-dissolved impurities are source of deposits. After filtration, solvent should be keep in air-tight bottle from which small particles are removed; once solvent has been filtered, it is not necessary to filter this solvent everyday unless reaction produce bacteria or indissoluble material occurs. If solvent is kept in storage vessel for more than one week, it is desired to filter it again before use.

4-3-3. Solvent effect on the instrument

All parts of the Gradient Pump contacting with mobile phase is manufactured from 316 stainless steel, ruby, sapphire, zirconium, or fluorine carbon polymer. Most of these materials are sensitive to chloride, and it is desired to avoid use of solvent which contains even small quantity of chloride. Main solvents that should be avoided especially are as follows.

Aqua Regia	Hydrochloric Acid(HCL) (20%)
Bromine	HCL (37%)
Chlorine Anhydrous	HCL (50%)
Copper Chloride	HCL (20%)
Ferric Chloride	HCL (75%)
Ferrous Chloride	Hydrofluorsilicic Acid (20%)
Freon 12	Hydrogen Peroxide
Guanidine	Lodine
Hydrochloride (6M)	Mercuric Chloride
Hydrobromic (20%)	(Dilute Solution)

In addition, it should be avoided to leave chloroform, carbon tetrachloride, etc. in instrument for long time, and use of ammonium hydroxide should be avoided because it has effect on stator and rotor of injector even though it has no effect on pump. When not using it for long time, keep it with iso-propanol filled with in flow path.

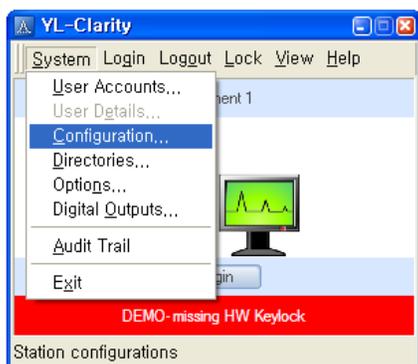
4-3-4. Measures when not uses for long time

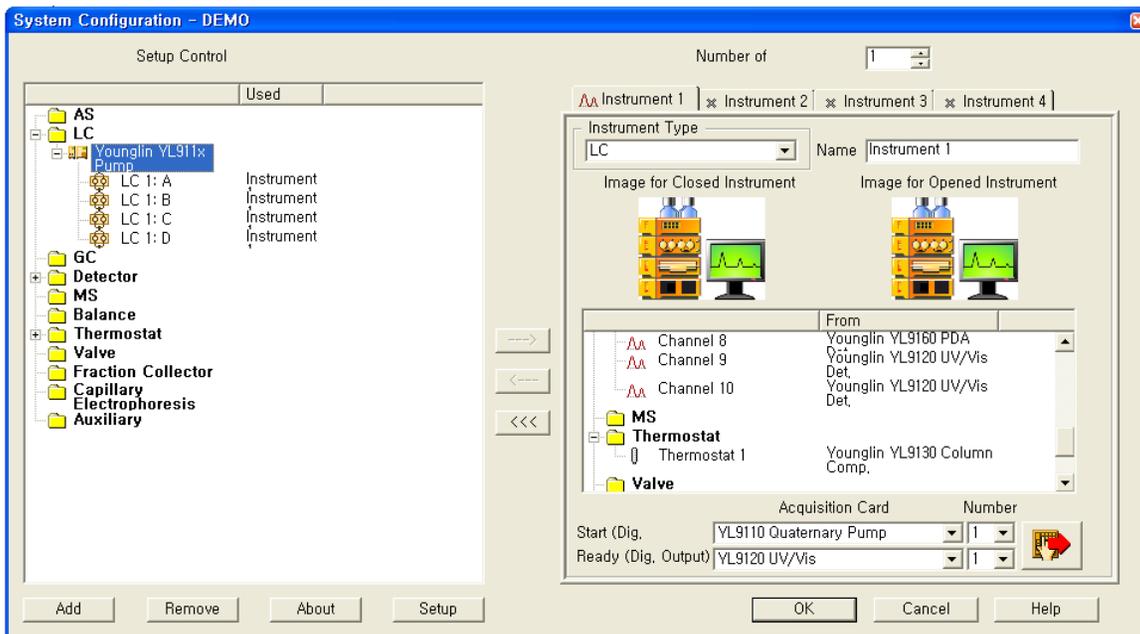
- 1) Prepare iso-propanol for analysis.
- 2) Open prime/purge pump and suck iso-propanol of at least 50ml into instrument.
- 3) Separate outlet tubing of pump.
- 4) Press out iso-propanol sucked into syringe in prime/purge valve and discharge at least 5ml into outlet of in-line filter.
- 5) Separate mobile phase filter assembly and block discharge hole and suction hole with cap.

4-4. YL-Clarity Chromatograph software

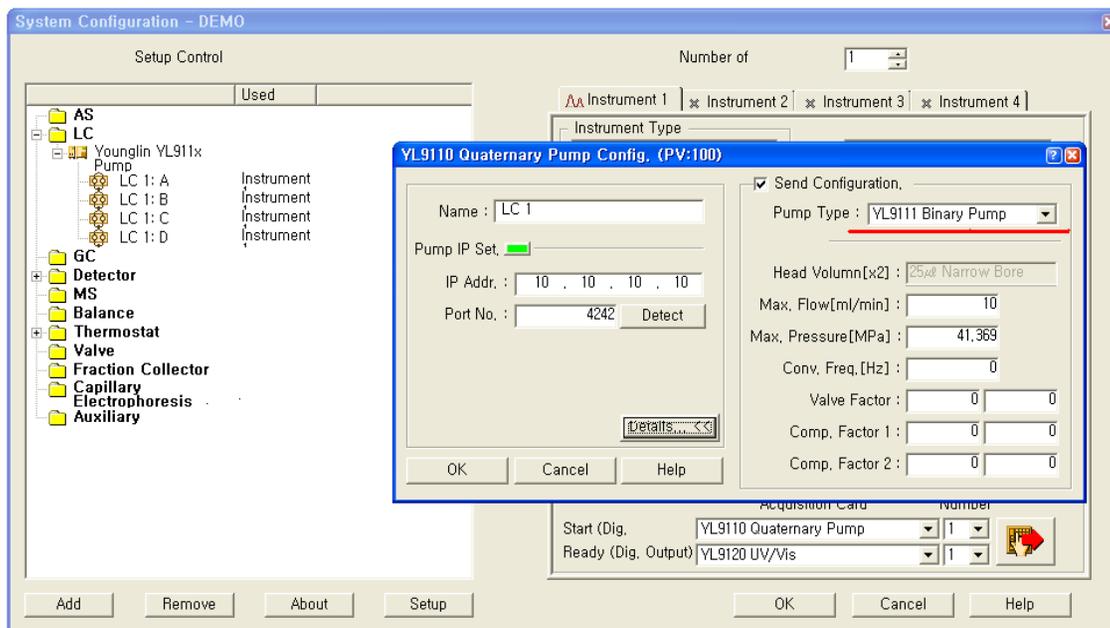
4-4-1. Installation of pump

Open YL-Clarity software and select Configuration on the main window. On the system configuration window, click [ADD] button and select YL911x.



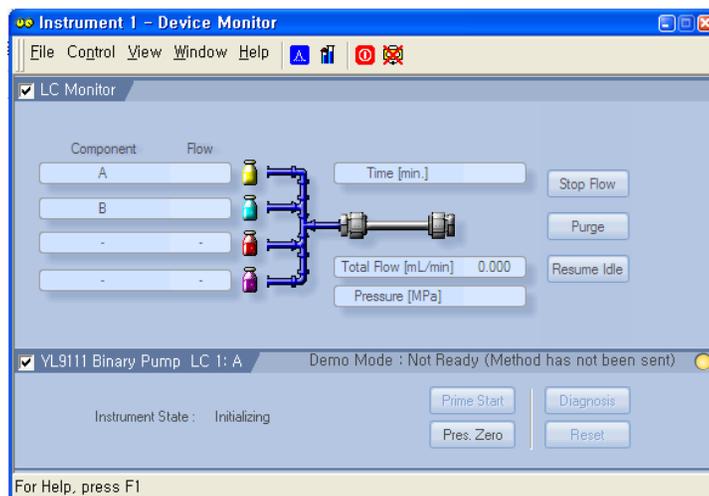


After select YL911x pump on the left window, click arrow button to move this on the right window. Click red arrow button on the right bottom side and select the pressure unit. Double click YL911x pump on the right window, and check IP address of pump. Click "Details" button to select pump type as YL9111 Binary pump.



4-4-2. Device Monitor

After configure the pump on the configuration window, log in to open main control window. On the main control window, click Device monitor and then Device Monitor window pops up as below. In this window, can control the pump and monitor instrument status as like flow and pressure.



[Control button]

Stop Flow : To stop the pump operation.

Purge : To run the pump initially. If you click this button, the window for setup solvent and flow pops up.

The pump starts according to the solvent ratio and flow value inputted on this window.

Resume Idle : If you click this button, the pump goes to idle state.

Prime Start : If you click this button, the pump runs at high speed with prime pump to fill the solvent into the lines. This function works only when the pump is not running. The prime pump inside pump runs when you click Prime start button.

Pressure Zero : To set present pressure to zero. Because the offset value of pressure sensor can be changed according to the temperature and using time, the pressure zero is necessary. Before you set the pressure zero, you should drop the pressure completely. This function works only when the pump is not running.

Diagnosis : To self test of instrument.

Reset : To release the pump status from the error.

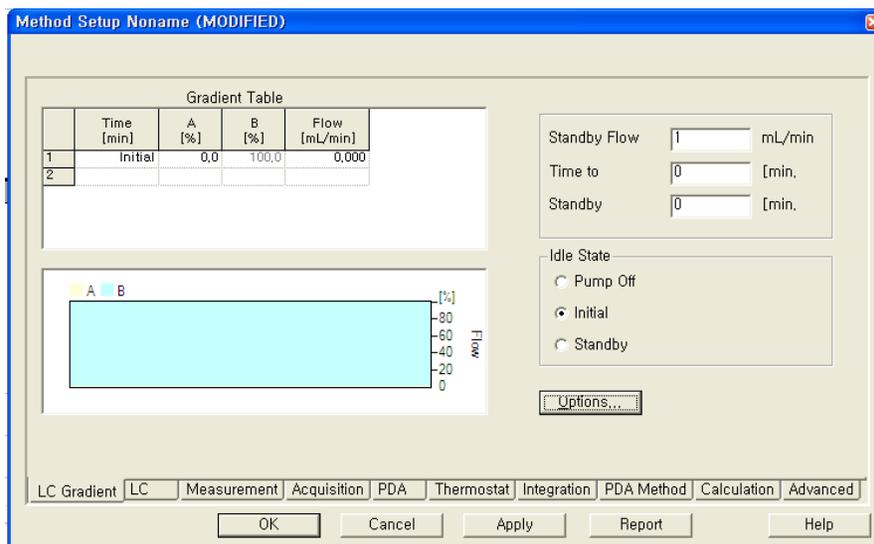
[Status message]

Initializing : It is displayed during initialization.

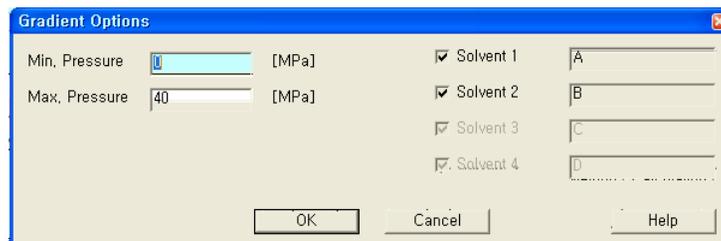
- Ready : It is displayed when the pump is ready.
- Prime : It is displayed during prime/purge status.
- Run : It is displayed during analysis.
- Fault : It is displayed if there is error on the pump.
- Halt : It is displayed if the pump stops.
- Diagnosis : It is displayed during self test.

4-4-3. Method Setup

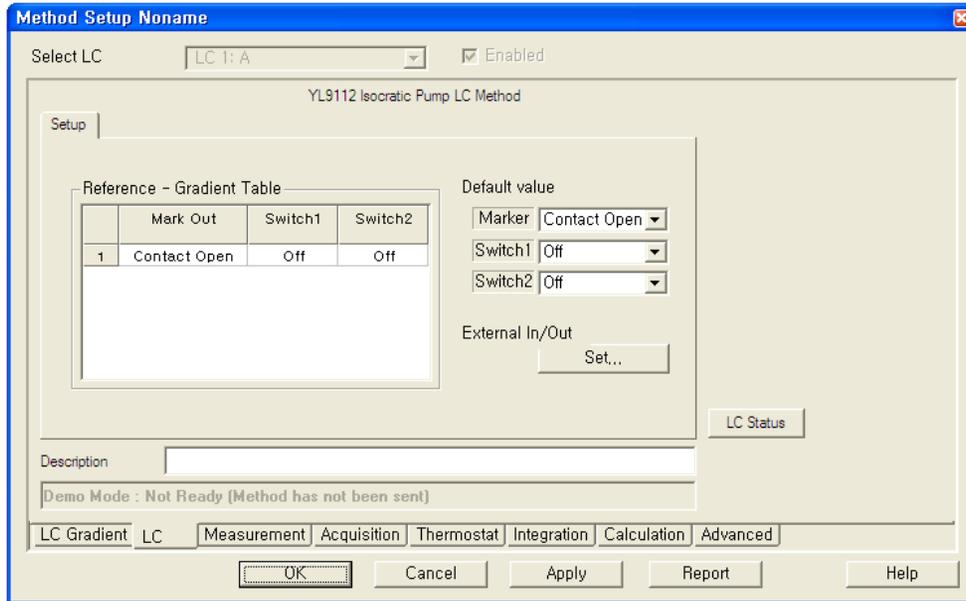
In this window, edit time program table, and setup the pump status during idle state.



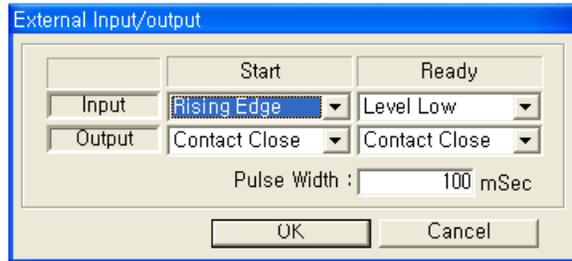
Click Options button to setup Max. and Min. pressure limit values to protect column and system. In this window, you can type the name of solvent you will use.



On the below window, make a program for output signal of switch terminal on the rear side of YL9111 Binary pump.



If the signal is opposite when you use with the other device, change it on the External In/Out Set.



4-4-4. Error message



If there are errors on the pump caused by pressure limit, control value range, and leak, the pump stops operation with error message.

Chapter 5. Maintenance

In the event that problem occurs or it is necessary to change part due to wear of seal in using YL9111 Binary pump, perform maintenance for instrument by referring to the following items.

5-1. Caution

In order to protect instrument, take care for the following items in using it.

- 1) After using solvent with sediment such as buffer solution, replace solvent with pure water at first and then methanol or iso-propanol and make it flow for 30 minutes using each solvent at 1.0ml/min flow rate.
- 2) Do not use solvent to corrode stainless steel material that is less than pH 2.3.

Material	Solvents to avoid
PEEK	Carbon Tetrachloride, Liquid Chlorine Methylene Chloride, Tetrahydrofuran
Teflon(PTFE)	Dimethyl Formamide, Diethylamine
SS316	Phosphoric Acid(Conc, Rm Temp)

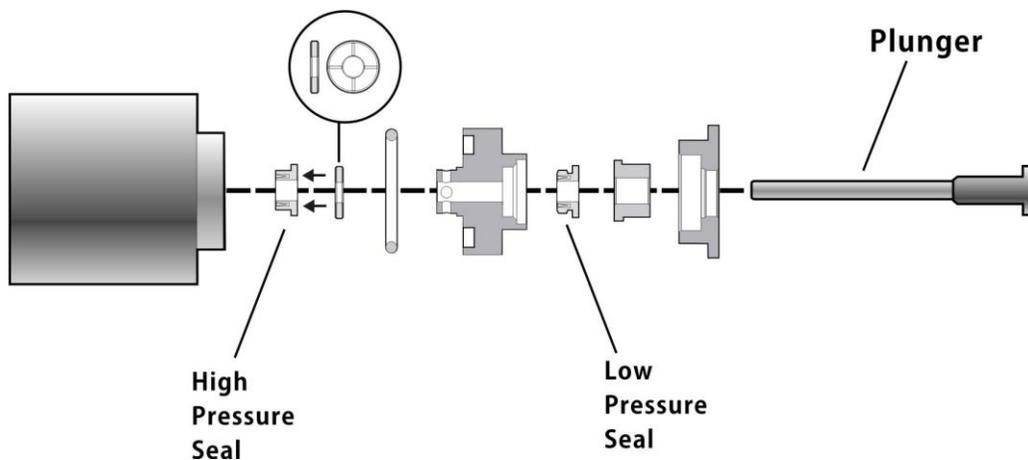
- 3) Do not install instrument where corrosive gas is generated or where there is carpet on floor.
- 4) Do not change flow rate rapidly in order to prevent from wrong operation of instrument, damage to column and damage to damper.
- 5) Do not operate instrument with excessive force.

5-2. Replacement of high pressure seal

If instrument is used for long time, high pressure seal is worn out to produce leakage of solution. In this case, after replacing it with new seal, it is necessary to condition it in order to make seal be used for long time at high pressure. Change and condition high pressure seal in the following method. In case instrument is used for the first time after purchasing, it is desired to perform training and it is better to change all seals of both head when change seal due to long use. Leaked solvent flows out through washing port.

Change of high pressure seal

- 1) Loosen tubing of inlet check valve and outlet check valve of pump head.
- 2) Loosen two head nut for each head using spanner.
- 3) Separate pump head assembly from instrument.
At this time, plunger is left at the place where pump head assembly was loosened. When loosening it, pull it carefully in pump head guide direction and take care not to damage plunger.
- 4) If backup washer in back side of pump head is pulled out, low pressure seal assembly appears. Use seal insertion/removal tool to separate low pressure seal assembly. Then, high pressure seal appears inside of head.
- 5) Remove worn seal with seal insertion/removal tool and insert new high pressure seal prepared at that place using seal insertion/removal tool in the same manner. Direction of seal should be such that the direction to see O-ring is toward front of head. Be careful not to change direction.
- 6) Insert low pressure seal assembly and backup washer.
- 7) Arrange pump head so that plunger left at the place where it is loosened be inserted into center hole of pump head assembly, and then press pump head to main body by inserting pump head by hand. When pressing it, press it carefully so that pump head may be maintained horizontal.
- 8) Tighten head nut in pressed condition. Tighten it so that left side and light side may be same, and tighten it until it is tightened no more by hand while confirming status of tightening finally.
- 9) Replace high pressure seal by applying the process of 1) to 8) to pump head of opposite side.



[Fig. 13] Replacement of high pressure seal

Conditioning of high pressure seal

- 1) Prepare organic solvent such as iso-propanol or methanol necessary. In order to conditioning it, use organic solvent only and do not use buffer solution and base solution.
- 2) Mix iso-propanol or methanol with water by 50:50, and fill instrument with it using the prime/purge valve. And plug the outlet of pump.
- 3) Set the high pressure limit to 2000psi and make flow rate be 0.2ml/min at Binary mode, and do not make air bubble be present inside using prime/purge valve.
- 4) Start pump. The pressure will increase upto 2000psi and then the pump will be stopped with a high pressure limit message. Repeat this procedure 2-3 times and then conditioning of seal is completed.

5-3. Replacement of plunger

If piston plunger is used for long time, it should be replaced due to wear.

The worn piston causes leakage of liquid as well as shortens life of high pressure seal. Piston wear is not well observed visually, so care should be taken when observing it.

- 1) Loosen tubing of inlet check valve and outlet check valve of pump head.
- 2) Press pump head to main body of instrument by hand, and loosen head nut.
- 3) Separate pump head assembly from instrument. Then, plunger is left in the place where pump head assembly is loosened. When loosening it, pull it carefully to the direction of pump head guide so that high pressure seal may not be damaged due to eccentricity.
- 4) Pull plunger, replace it with new one at same location, and insert it.
- 5) If there are contaminants on surface of plunger, remove contaminant by applying methanol on cloth without dust.
- 6) Arrange plunger in the manner that plunger may enter the center hole of loosened pump head assembly, and then press pump head by hand so that head may be pressed into body. When pressing it, press it carefully and take care so that pump head may be maintained vertical.
- 7) Tighten head nut with it pressed. Tighten it so that left and right sides may be same in turn, and tighten it until it may be tightened no more by hand while confirming tightening status finally.

5-4. Replacement of check valve cartridge

If check valve is not well operated due to contamination, pressure change is severe during operation and pump does not operate properly. Many problems of check valve are caused by small impurity that interferes with operation of check valve. Therefore, if impurity is prevented from entering inside of pump head using mobile phase filter, malfunction of check valve is almost not caused. The cleaning of the check valve cartridges using sonication can solve this problem.

- 1) Separate tubing connected to pump head.
- 2) Loosen inlet and outlet check valve housing of pump head using spanner.
- 3) Wash check valve cartridge in separated check valve housing for about 30 minutes using ultrasonic cleaner with 10% nitric acid solution.
- 4) Using pure water, rinse check valve cartridge to remove the nitric acid used for cleaning.
- 5) Assemble loosened check valve in the reverse order.

5-5. Replacement of low pressure seal

Wearing of the low pressure seal is caused when pump has been used for long time without using washing port. In order to prevent wear of low pressure seal, it is desired to use washing port, and it is more desirable in case of using buffer solution. Leakage of liquid due to wearing of the low pressure seal is caused between pump head and body.

- 1) Separate pump head with reference to 5.2.
- 2) Separate washer, and pull out low pressure seal assembly from pump headbody with seal insertion/removal tool.
- 3) Replace low pressure seal attached to low pressure seal assembly with new one.
- 4) Assemble pump head in reverse order by referring to 5.2.

5-6. Cleaning of flow path within pump

In order to prevent occurrence of problem in instrument, remove impurity accumulated in instrument, and it is better to clean flow path when it is not used for long time. Clean inside of flow path in the following

method, and be careful when treating strong acid and strong base.

- 1) Separate column inlet tubing connected to column.
- 2) Orient column inlet tubing toward waste bottle.
- 3) Set flow rate at 1ml/min.
- 4) If injector is installed, turn it to injection position.
- 5) Pump 100% iso-propanol through pump and injector for 10 minutes.
- 6) Pump distilled water filtered through pump and injector for 10 minutes.
- 7) Pump 10% nitric acid solution for 5 minutes.
- 8) Wash pump and injector with distilled water filtered for at least 10 minutes.
- 9) Pump 100% iso-propanol through pump and injector for 5 minutes.

Now, pump is prepared for use of mobile phase or for the period not being used for short time or long time. If pump is not used for long time or there is contamination in flow path due to use of impure solvent, it is desired to separate pump head assembly and wash it with ultrasonic cleaner. In order to wash pump head, separate pump head into parts in the same manner as seal change process of 5.2, wash it with ultrasonic cleaner, and assemble each part again. At this time, the high pressure seal is damaged, so replace it with new one.

5-7. Supply of lubricant

YL9111 Binary pump necessitates supply of proper lubricant into piston drive part for smooth operation of instrument. It is desired to use lubricant or low viscosity grease for piston carrier and pump housing and small amount of grease such as 630-AA for bearing of cam shaft and piston carrier. Care should be taken because pumping action is interfered with if lubricant is attached to surface of piston. Because shortening of pump life is caused where powder or dust is much generated, install instrument where surrounding environment is good.

5-8. Replacement of solvent filter and in-line filter

In case instrument is used for long time or mobile phase is bad, mobile phase filter and in-line filter is blocked due to small particles contained in solvent. If filter is blocked, pressure within flow path of pump is largely reduced when solvent is sucked to generate air bubble, make flow rate reduced and make

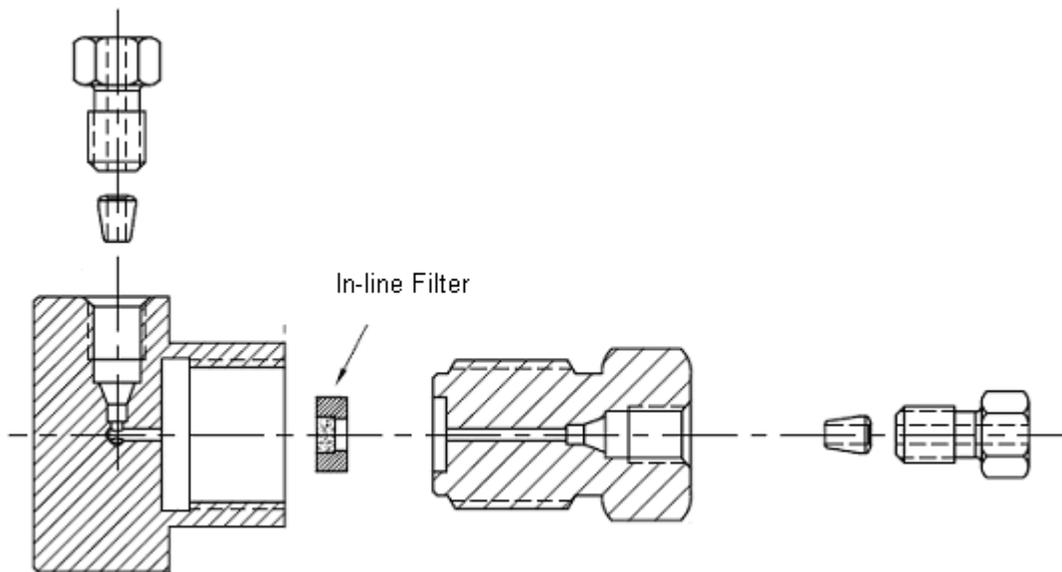
precision reduced ; so it is desired to check it periodically. Main cause of mobile phase filter blocking is growth of bacteria, and two causes to block inlet filter is growth of bacteria and use of solvent containing impurity. In order to prevent growth of bacteria, use at least 10-20% organic solvent or solvent containing growth depressing component. If pure water or soluble solution without interfering material is used, many bacteria will grow in mobile phase filter though it is replaced with fresh solution everyday. Therefore, use solvent of HPLC grade filtered well at all times for mobile phase. Blocking of in-line filter is caused by accumulation of small particles generated due to wear of high pressure seal by using of impure solvent and long use of instrument. In case mobile phase filter and in-line filter are contaminated, condition of filter may be improved by washing it by ultrasonic cleaner with 10% nitric acid solution for 30 minutes. If it is not improved by ultrasonic wave cleaning, replace it with new filter.

Change and cleaning of mobile phase filter

- 1) Separate mobile phase filter from tubing. As the surface of teflon tubing of mobile phase filter of insertion type is slippery, separate it with tubing held avoiding slippage using #1000 sand paper.
- 2) In case of performing ultrasonic wave cleaning, wash head part by ultrasonic cleaner with 10% nitric acid solution for 30 minutes, wash it again by ultrasonic cleaner with pure water for about 10 minutes, then dry it. In order to replace filter with new one, prepare new mobile phase filter of same size.
- 3) If washing has been completed, assemble filter to be replaced newly again. Hold teflon tubing using sand paper and insert mobile phase filter of insertion type with center adjusted into middle hole.

Changing and cleaning of in-line filter

- 1) Separate connected tubing from in-line filter assembly using spanner.
- 2) Separate head part of in-line filter assembly from body using spanner.
- 3) In case of performing ultrasonic wave cleaning, perform ultrasonic wave cleaning to head part for 30 minutes with 10% nitric acid solution, perform ultrasonic wave cleaning for 10 minutes with pure water, and then dry it. In order to replace filter with new one, separate filter located at back side of head part.
- 4) In case of replacing it with new filter, replace it with new in-line filter located at the location where it was separated ; and in case ultrasonic wave cleaning has been completed, re-assemble head part of dried assembly.
- 5) Using spanner, tighten head part sufficiently so that there may be no leakage of liquid even at 6000 psi.



[Fig. 14] Replacement of in-line filter.

5-9. After use system

If you keep the pump without cleaning, the pump can be damaged by the crystallization of salt, growth of microorganism, contamination. To maintain the pump properly, run the pump using iso-propanol for 30 minutes and the plug the outlet of pump, and keeps clean the solvent filter. Especially, after use buffer solution, run the pump using 100% water first then iso-propanol.

5-10. Troubleshooting

In case general problem occurs as the following table, confirm the possible causes regarding this first, and then take proper countermeasures. The following table is countermeasure in case general problems occur.

Problem	Cause	How to fix
Pressure upper limit is loaded.	-Tubing inside is blocked. -Check valve is blocked. -Solvent is changed.	-Replace blocked tubing by loosening to be from tail side in turn. -Replace outlet check valve. -Wait until solvent is completely changed.
Pressure increases or decreases.	-Change of solvent is incomplete. -Column is unstable.	-Wait until change is completely performed. -Wait until pressure is stable.
Solvent is not flowed out	-Air bubble is in pump head. -Air separation status of solvent is bad. -Check valve is not good. -Liquid containing oil flows into head part. -High pressure seal was worn.	-Perform prime/purge again. -Take measures so that air separation condition of solvent may be good, and perform prime/purge again. -Wash or replace check valve. -Remove oil in head sufficiently with strong organic solvent using prime/purge port. -Replace the high pressure seal.
Pressure is unstable.	-Check valve is bad or defected. -Air separation or mixing conditions of solvent is bad. -Compression compensating reference value of configuration mode is wrong. -Cam shaft is loosened. -High pressure seal was worn. -Pump head was loosened.	-Wash or replace check valve. -Use mobile phase with well mixed solvent, and improve air separation condition. -Initialize compensation value or re-input proper value. -Tighten wrench bolt of cam shaft -Replace high pressure seal. -Tighten nut of pump head.

<p>Liquid is leaked or pressure reduces when cleaning port is not used</p>	<ul style="list-style-type: none"> -High pressure seal was worn. -Plunger was worn. 	<ul style="list-style-type: none"> -Replace high pressure seal. -Replace plunger.
<p>Liquid is leaked from inside of instrument.</p>	<ul style="list-style-type: none"> -Fitting in instrument was loosened. -Damper was damaged. -Low pressure seal was worn. 	<ul style="list-style-type: none"> -Tighten fitting in instrument. -Replace. -Replace low pressure seal.
<p>ump is not operated after input of power.</p>	<ul style="list-style-type: none"> -Power voltage is unstable or low. -Strong induction voltage is generated in the surroundings. 	<ul style="list-style-type: none"> -Use stable, proper DC power source. -Close induction power source in surroundings, or install instrument away from power source.
<p>Noise is too high</p>	<ul style="list-style-type: none"> -Load is caused to piston carrier. -Timing belt is loose. -Motor is in defect. 	<ul style="list-style-type: none"> -Supply lubricant in carrier body. -Reduce clearance above belt by adjusting guide location of timing belt. -Inspect connection status of motor cable, and replace motor if noise is severe only in high pressure.

YL9100 HPLC SYSTEM

YL9112 ISOCRATIC PUMP

USER MANUAL



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YL9112

Chapter 1. Introduction

YL9112 Solvent delivery pump is a pump for High performance liquid chromatograph, it provides high performance and functions as a HPLC pump, and controlled by software.

YL9112 isocratic pump uses specially designed cam and pulse damper for stable solvent delivery and has a compressibility compensation function for accurate and precise solvent delivery.

And the pump has a automatic rinsing function to prolong the life time of pump head. Using UHMWPE seal, it provides extended life time of high pressure seal even though use buffer solution as a mobile phase. On the outlet of pump, there is a in-line filter to prevent small particles come into the column and also protect column by the pressure limitation setup. YL9112 isocratic pump provides auto prime/purge function for easy operation and fast exchanging of solvent. This instrument manual includes basic principle, installation and operation method, and troubleshooting to use YL9112 isocratic pump properly.

1-1. Specifications

- 1) Operating principle : Parallel dual-plunger pump
- 2) Compressibility compensation : Automatic
- 3) Flow range : 0.001-10ml/min
- 4) Flow rate accuracy : $\leq \pm 1\%$ at 1ml/min
- 5) Flow rate precision : 0.1% RSD at 1ml/min
- 6) Maximum pressure : 6000 psi
 - A. Operating range : 0-6000 psi up to 5ml/min
 - B. Operating range : 0-3000 psi up to 10ml/min
- 7) Pressure pulsation : $\leq \pm 1\%$ at 1ml/min
- 8) Composition Precision : $< 0.1\%$
- 9) Composition Accuracy : $< 0.5\%$
- 10) Auto prime/purge
- 11) Communications : LAN
- 12) Safety & maintenance : Leak detection, Diagnostics, Error detection
- 13) Dimensions : 385 X 160 X 565mm (width X height X depth)
- 14) Line Voltage : 100-240VAC, $\pm 10\%$, automatic voltage selection
- 15) Line frequency : 50/60Hz, $\pm 5\%$
- 16) Power consumption : 70W

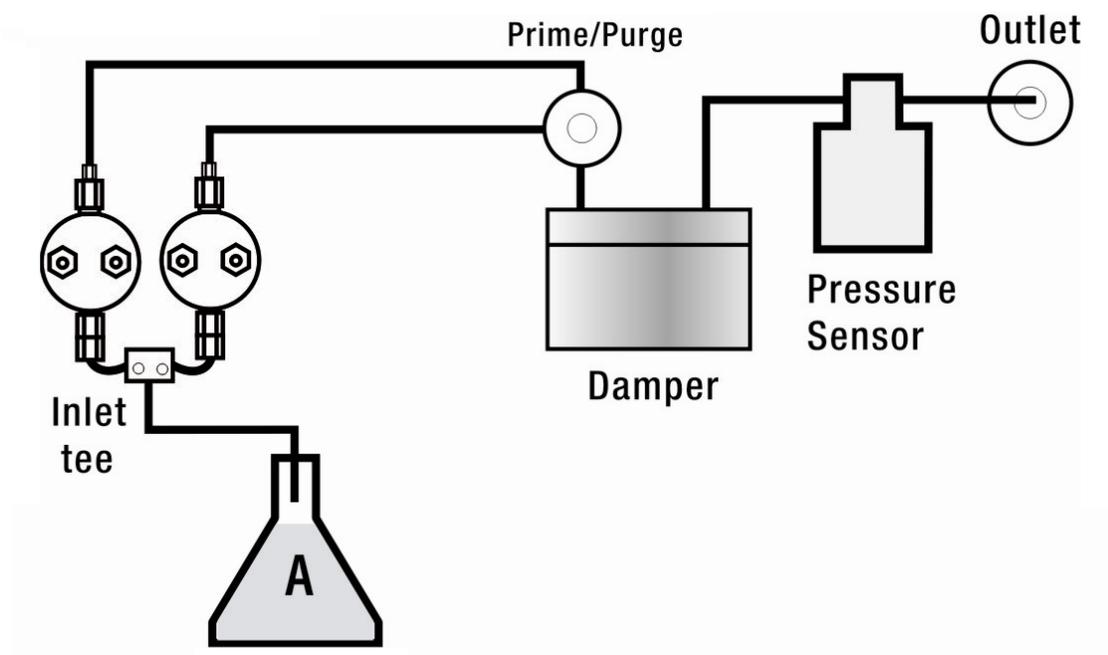
[Optional specification]

Head type	Narrow Bore	Analytical	Semi prep
Head volume(μ l/each)	25	64	144
Flow range(ml/min)	0.001~5	0.001~10	0.01~50
Flow range(ml/min)	0.005~5	0.01~10	0.05~25
Pressure(psi)	5000psi	6000psi	3500psi
In-line filter (micron)	2	2	10
Material	Zirconium, Ruby, PEEK UHMWPE, PTFE	SS316, Zirconium, PTFE, Sapphire, UHMWPE	SUS316, Sapphire, Ruby, UHMWPE, PTFE

Chapter 2. Configuration and Principle

2-1. Configuration of flow path

YL9112 isocratic pump consists as same with figure 1. It uses Teflon tubing between pump inlet and inlet check valve, SUS316 or PEEK tubing from outlet check valve.



[Fig. 1] Flow diagram of YL9112 pump

- **Solvent Filter**

The solvent filter is used for protecting the system from the particles in solvent. This filter removes particles from solvent to prolong the life time of high pressure seal and prevent damage on the column. It is recommended to use the solvent filter when you use YL9112 isocratic pump with or without degassing module. Select proper filter depending on the column and flow rate.

- **Pump Head Assembly**

Pump head is real working part to deliver solvent by piston movement and check valve. It consists of

plunger, check valve, high pressure seal, low pressure seal and rinsing port. YL9112 isocratic pump built in automatic rinsing port to clean the head assembly efficiently if uses buffer solution.

- **Auto Rinsing Pump**

Auto rinsing pump delivers cleaning solvent from the rinsing solvent bottle to the inside of pump head. YL9112 isocratic pump rinse the system every 3 minutes automatically.

- **Prime/purge valve**

This valve is used for priming the pump. Fill the solvent inside of tubing from the solvent bottle if you use the pump for the first time or the tubing lines are empty. Remove a plug on the prime/purge valve and suck the solvent using syringe, or click "Prime Start" button on the software to operate the micro pump.

- **Prime micro pump**

Using this pump, the pump circulates rinse solvent into the pump head.

- **Mixer**

It is static mixer to improve mixing efficiency of solvent.

- **Pulse damper**

YL9112 isocratic pump reduces pulse from the cam operation by the diaphragm damper. YL9112 isocratic pump provides constant and pulseless flow using compressibility compensation and pulse damper, so the detector that affected by flow stability can be used with YL9112 pump.

- **Pressure transducer**

It checks real time system pressure. The pump uses this pressure to protect system and to operate compressibility compensation and even compensation. YL9112 isocratic pump uses continuous flow path type pressure sensor.

- **In-line filter**

It removes fine particles that are not filtered by solvent filter or made by worn of high pressure seal.

2-2. Operation

There are essential parameters on the pump as like flow accuracy, precision, and reproducibility to get the reliance of analysis data and low detection limit. YL9112 isocratic pump uses high pressure resistant dual pump head, controls microprocessor to monitor the phase of cam to remove pulse, so fulfill the necessities of solvent delivery pump.

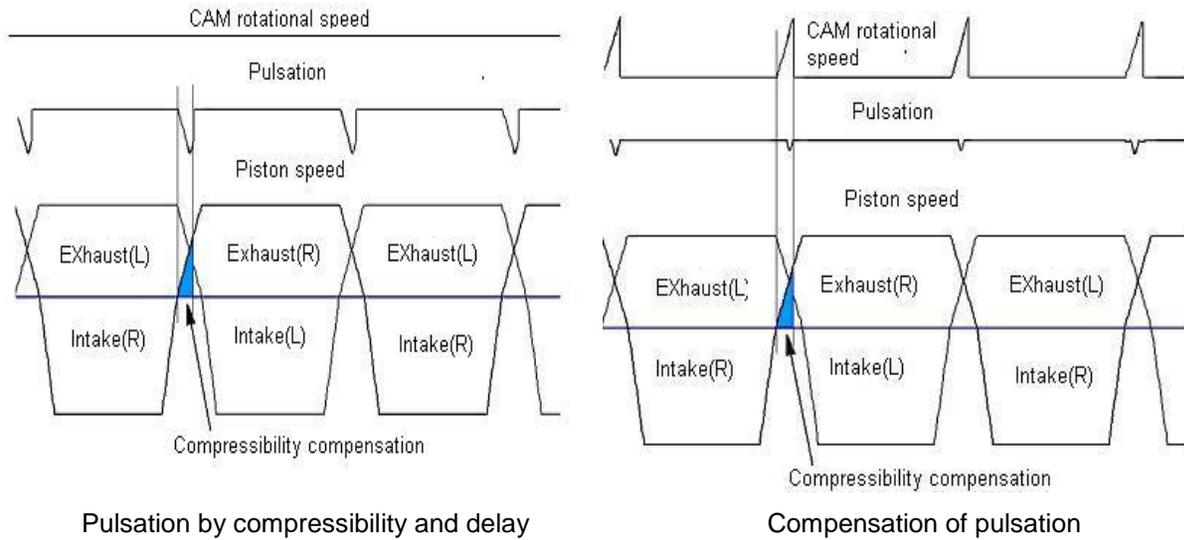
Diaphragm pulse damper reduces pulse from the cam operation more than 90% between low and high pressure range by internal elastic body of damper, and also works well as a mixer for gradient elution. The elastic body of Diaphragm pulse damper improves flow accuracy through compression, expansion procedure to make variance of kinetic energy by flow constant. The amount of contact solvent with pulse damper is around 1.5ml at 3000psi, which ensures that flow path is completely cleared away.

YL9112 isocratic pump was designed so that integrated flow rate may realize no-pulse operation using specially designed cam. However, pulse incapable of being neglected is caused actually due to compressibility of mobile phase proportional to pressure and elasticity of high pressure seal, so the pump is controlled in real time so that occurrence of pulse proportional to pressure may be depressed. Control method of the Pump uses supervision of pressure and control of location simultaneously, so it has advantage to improve precision and accuracy of flow rate without being affected by range of pressure and flow rate. There is a part for microprocessor control to realize various function of pump including stepping motor control. Stepping motor control processor operates motor as a micro step, so can achieve constant motor speed at low flow rate with low noise.

Operation mechanism is for transmission of kinetic energy from step motor to piston. This mechanism includes specially designed cam, stepping motor, carrier, carrier housing, and phase sensor.

2-3. Compressibility compensation

Most of pumps for HPLC analysis are used at high pressure. However, pulse occurs in high pressure due to compressibility of liquid and elasticity ratio of seal, so flow rate is also reduced. Occurrence of pulse due to this reduces precision and accuracy of pump flow rate, so compensation is necessary for this; YL9112 isocratic pump monitors actual pressure and calculates compensation value for this; compressibility compensating operation to control angular velocity of cam with this value reduces occurrence of pulse flow remarkably as well as improves accuracy of flow rate largely.



[Fig. 2] Delivery mechanism of YL9112 isocratic pump

2-4. Rinse port

When using buffer solution, salts are generated on back side of high pressure seal and these deposits wear pump seal to cause shortening of seal life, which has bad effect on pump.

Rinse port enables to insert proper solvent in back side of high pressure seal to prevent salts from being deposited and activated. Mixed solution(20% MeOH) of water and methanol is used as cleaning solution, and life of seal is extended with lubrication action in general analysis..

Chapter 3. Installation

3-1. Inspection and site preparation

YL9112 isocratic Pump is delivered along with the following parts when being shipped. Before opening transportation package, perform inspection for trace of shock or mistake, and if there is abnormality, do not open the contents and inform this company of it. And, if contents are opened, perform inspection for existence of shock in the contents and contact with this company when trace of shock is found.

The Gradient Pump is a delicate instrument, so use original box and buffer material as far as possible when re-packing it to transport instrument. If it is impossible to use original box; wrap pump with several layers of buffer material, and fill the bottom, top and all other sides of pump with buffer material in order to make pump endure shock or vibration during transportation.

YL9112

Standard configuration of YL9112 isocratic pump

- 1) Main body of instrument
- 2) Power cord and fuse
- 3) Tubing 60cm,
 - A. Bio Narrow Bore : ID 0.01" , OD 1/16" PEEK
 - B. Analytical : ID 0.01" , OD 1/16" SUS316
 - C. Semi-prep : ID 0.02" , OD 1/16" SUS316
- 4) Installation kit
- 5) Manual

Site requirement of YL9112 isocratic pump

- 1) Room with 20℃ temperature with variation $\pm 5^{\circ}\text{C}$ with and 60% humidity
- 2) Where no direct and straight sunlight
- 3) Plain floor without carpet
- 4) Where having spare space of 20cm or more
- 5) Where there are 10% or less voltage change, no frequency change and 100 Ω or less grounding point
- 6) Where there is no generation of corrosive gas and ventilation is well done
- 7) Where stable power of 110 or 220V is supplied, $\pm 10\%$
- 8) Where not receive electromagnetic induction from large transformer, high frequency heater, UPS, etc.

- 9) Within 2500 m above sea level(storage within 4600m)

Please check the following before you install the system.

- 1) Keep the ventilation as normal state.
- 2) Install on the stable place. Avoid the places as like near to air conditioner and heater, direct sun light, near to window.
- 3) Keep the place without dust and vibration.
- 4) Maintain voltage variation within 5% of proper voltage.
- 5) Avoid high frequency or strong magnetic field environment.
- 6) Avoid from the source of fire(spark, flame).
- 7) Keep the proper ground for electricity.
- 8) Check the place of water supply for emergency.

Caution ! : Keep distance with CRT at least 50cm.

3-2. Connection of power

Confirm supplying voltage, electric power, socket, and type of connector are correct. Before connect power cable on the pump, please turn off the power switch of YL9112 isocratic pump.

Warning ! : The electric power should be matched with description on the rear side of instrument. If not, the instrument can be damaged or it can be a reason of fire.

Caution ! : Confirm the ground is correct. Water pipe is not recommended in case of non-metal pipe. Gas pipe should not use because of safety. If the ground is not correct, the electric shock can be happened.

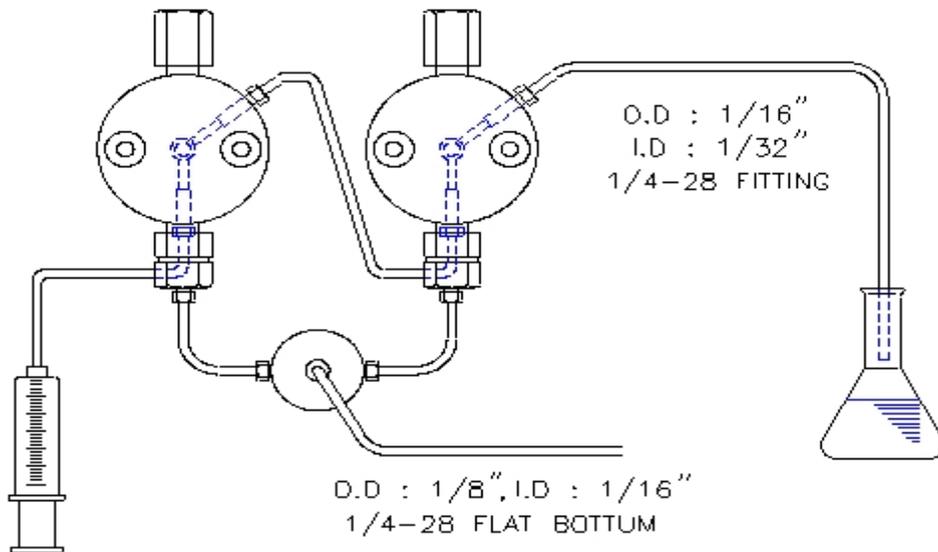
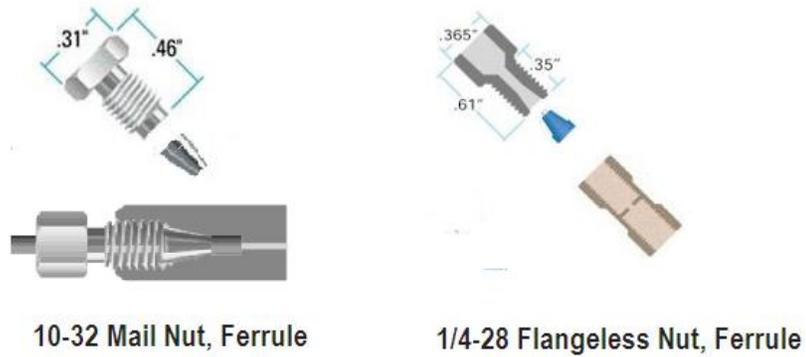
3-3. Connection of tubing

YL9112 isocratic pump uses following fittings for connection.

Flow path		Material	OD	ID	Fitting(UNF)
Inlet Tee		Teflon	1/8"	1/16"	1/4-28(flat type)
Inlet Tee ~inlet check valve		Teflon	1/16"	1/32"	1/4-28(flat type)
Outlet check valve~ In-line filter	Narrow bore	PEEK	1/16"	0.02"	10-32
	Analytical	SUS316	1/16"	0.02"	10-32
	Semi prep	SUS316	1/16"	0.03"	10-32

The cut surface of tubing should be cut at right angle without dust, tube should not be contracted, and middle inner diameter shall not be blocked.

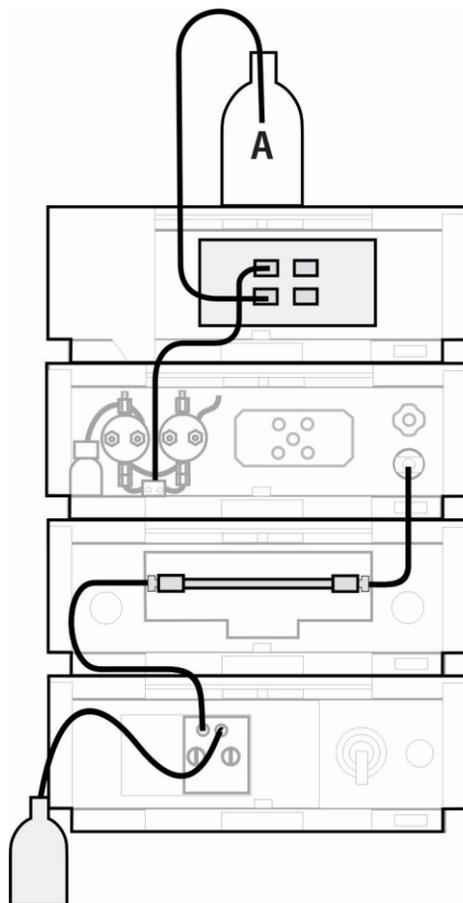
In order to cut stainless steel tubing, tubing cutter should be used, plastic tubing cutter or shaving cutter should be used for teflon and similar material of tubing, and the surface should be clean and have no crumbling.



[Fig. 3] Fitting for 1/16" OD tubing

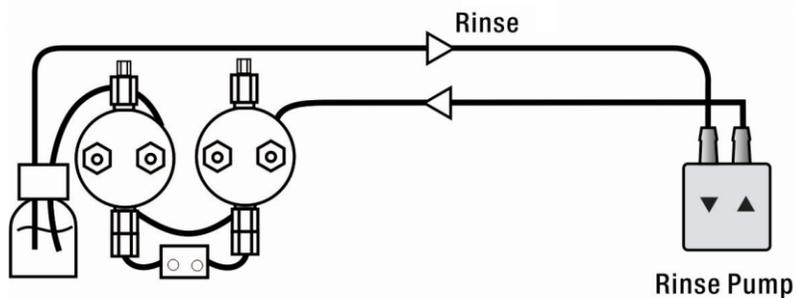
Inlet tubing of pump connects with degassing module using 1/4-28 fitting or with solvent bottle directly, the outlet tubing connects with injector. The fitting for injector is different depending on the injector type.

YL9112



[Fig. 4] Connection between YL9112 isocratic pump and YL9100 HPLC system

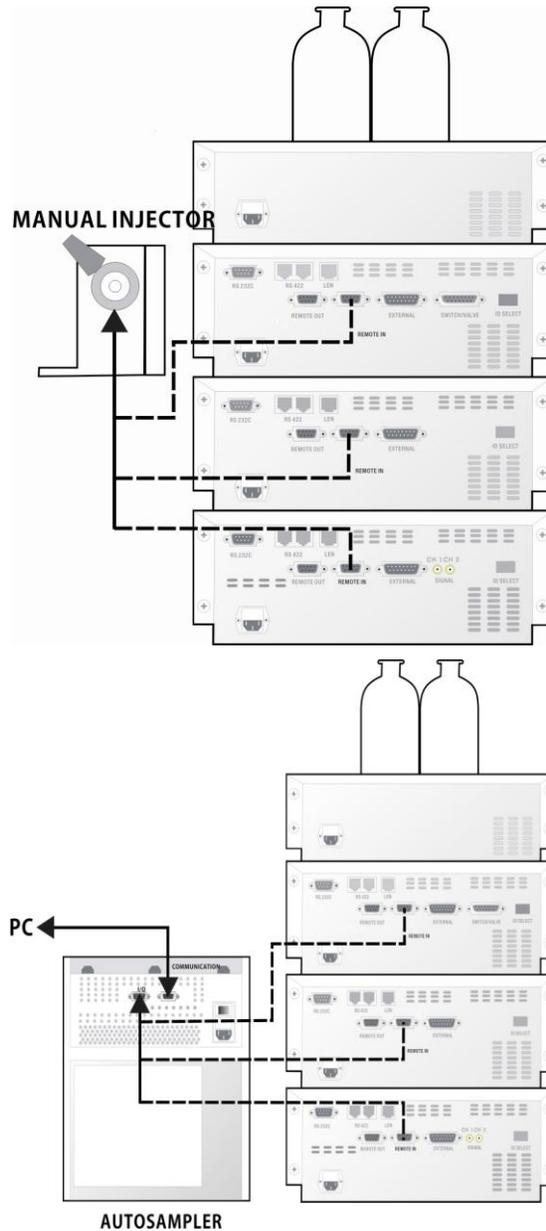
Rinse port tubing connects on the each pump head using 1/4-28 fitting as same as [Fig. 7], and fill the 20~50% Methanol. The rinse pump inside of YL9112 isocratic pump circulates the rinse solvent into the pump head every minute. Check and replace rinse solvent once a week at least.



[Fig. 5] Tubing connection of rinse port

3-4. Connection of remote cable

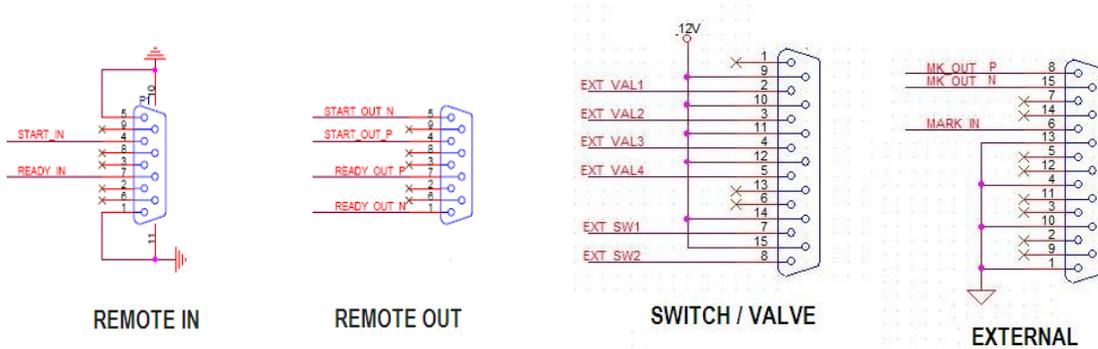
YL9112 isocratic pump has connection terminals for remote input/output, external solenoid valve, and marker input/output. The remote cable from the injector(manual or autosampler) has to be connected on the Remote In terminal on the rear side of YL9112 isocratic pump to collect data at the moment of injection.



YL9112

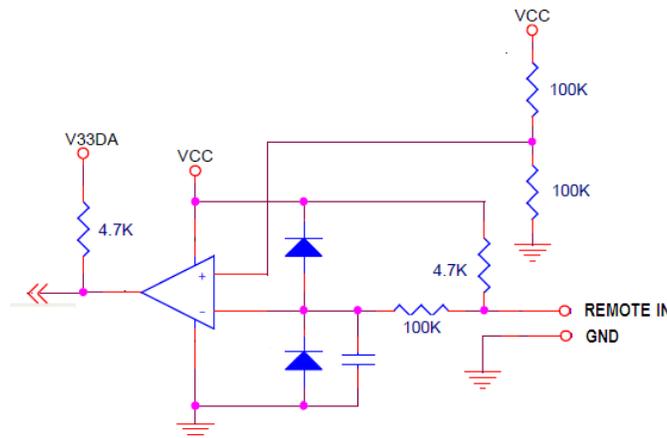
[Fig. 6] Connection of remote cable between YL9112 pump and injector

Notice ! : Please do not connect wires between cables at your discretion. If you want to connect with the other instrument, please check input/output information and confirm with YL9112 isocratic pump terminal configuration.

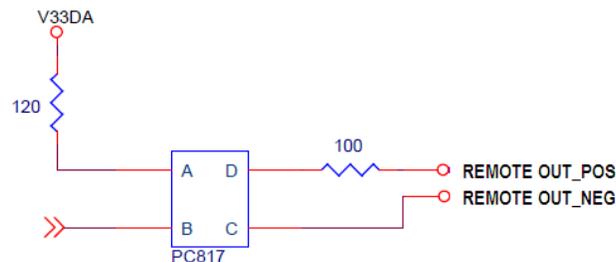


[Fig. 7] Pin configuration of each terminal

[Fig. 10] and [Fig. 11] are the diagram of remote and the other terminal input/output. In between YL9100 series modules, connect directly and confirm the configuration with the other modules.

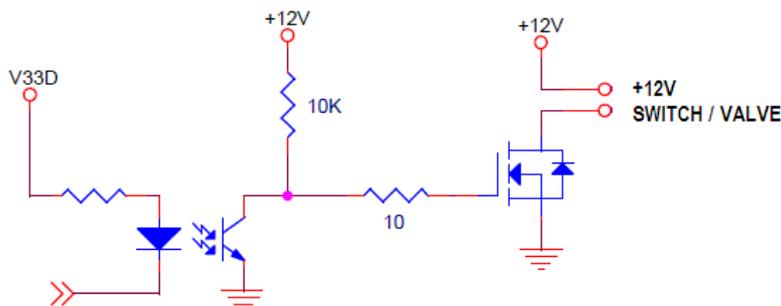


[Fig. 8] Diagram of Remote and Marker input



[Fig. 9] Diagram of Remote and Marker output

YL9112 isocratic pump provides output signal(12V 500mA) to operate external solenoid valve as like [Fig 12].



[Fig. 10] Diagram of solenoid output

[Remote operation]

START-IN : Operate instrument, and start running of gradient program.

If you connect it with autosampler or external valve, automatic running is available.

START-OUT : If the signal input on the START-IN terminal, the signal pulse output through this port. It can be used for synchronization of remote start with the other instrument.

MARK-IN : To control event program or operate additional operation.

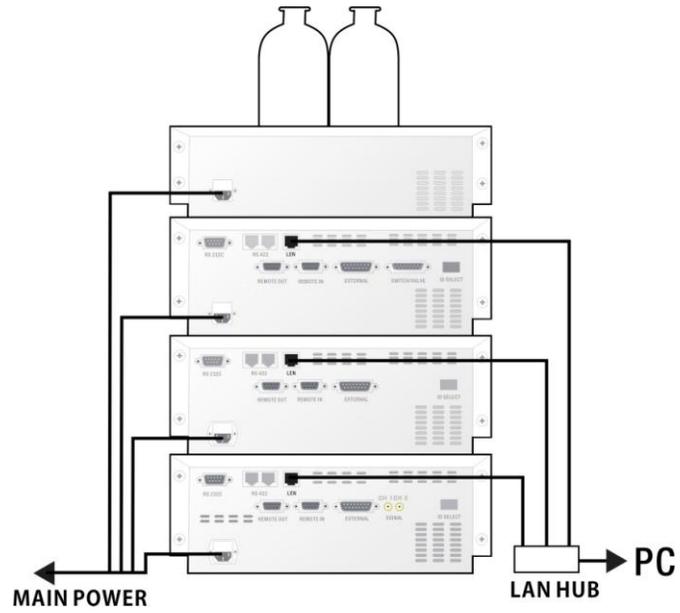
MARK-OUT : To control time event program output.

READY-IN : To change error state and stop operation if there is a input.

READY-OUT : When YL9112 isocratic pump is not ready state because of running status, output error signal is indicated if there is a leak.

3-5. Connection of communication cable

YL9112 isocratic pump provides TCP/IP internet protocol as a standard. The IP address of YL9100 pump series is 10.10.10.10, if DIP SW settings on the rear side are On position. If you change the IP address using control software, the DIP SW has to be set OFF.



[Fig. 11] Connection of communication cable

Notice ! : The LAN HUB used for cable connection on the PC must use switching mode module.

Chapter 4. Operation

There are four LEDs in front of YL9112 isocratic pump.

	POWER	LED turns ON if main power turns on
	CONNECTED	LED turns ON if communication is connected, LED blinks during connection
	READY/RUN	LED turns ON before analysis, LED blinks during analysis
	ERROR	LED turns ON if there is error

4-1. Before Start

When using pump for the first time, initialize it through the following process in order to clean flow path and condition high pressure seal. This process is necessary in case instrument is installed newly or is not used for long time.

- 1) Prepare iso-propanol of HPLC grade.
- 2) Remove residual air bubble within instrument by turning prime/purge valve in counter clockwise and loading iso-propanol of at least 50ml by Prime Start button.
- 3) Separate pump outlet tubing.
- 4) Press the sucked iso-propanol into syringe with prime/purge valve and discharge more than 5ml to outlet of in-line filter.
- 5) Operate pump with instrument outlet open for 2-3 hours at 0.2ml/min flow rate and for 1 hour at 1.0ml/min flow rate using iso-propanol.
- 6) Perform process of 2) ~ 5) using solvent which will be used as a mobile phase.
- 7) Remove inside residual iso-propanol by operating it at 1ml/min flow rate for 30 minutes with instrument outlet open.
- 8) Form flow path by connecting injector, column, and detector tubing mutually.

4-2. Mobile phase filter and bottle

Solvent vessel should be positioned at higher location than pump and not be positioned below pump, and inlet tubing length should be as short as possible. This can minimize pressure drop caused at inlet of pump during suction.

When using solvent having high vapor pressure as hexane, formation of air bubble is caused due to large pressure drop in suction part in high flow rate; so particular care should be taken, and mobile phase should be maintained after air separation, filtration and air-tightening.

Mobile phase filter of 10 μ m porosity is connected into inlet tubing in order to prevent entering of small particles. Mobile phase filter is blocked if mobile phase is bad or is used for long time, it is necessary to clean or change filter in this case.

4-3. Preparation of solvent

Proper solvent prevents various problems happened during actual analysis.

Solvent gas removal and filtration are necessary because they have great effect on result of analysis and maintenance of instrument.

4-3-1. Degassing

Solvent gas removal is performed in order to remove gas such as nitrogen or oxygen contained in mobile phase. Contained gas should be removed by air separation before mobile phase is used or while mobile phase is used, and the most practical technology for air separation is to insert helium into solvent.

Helium is easily separated from HPLC solvent, so other gases contained in solvent may be easily removed due to diffusion of helium gas.

When mixing organic solvent such as methanol or acetonitrile into water, this mixture contains very small quantity of gas as compared to the quantity of pure composition; so it has more strong tendency to discharge gas. Back pressure regulator attached to outlet of detector prevents formation of noise in base line due to air bubble, and mobile phase vessel should be pressurized under 2-3psi pressure with helium if it is desired to reduce gas discharge due to solvent mixing.

4-3-2. Filtration

Solvent should be necessarily filtered through 0.45 μ m or less filtering membrane before use. Removal of small particles is necessary to compensate reliable operation of piston seal, and is necessary measure for reliability of other components in liquid chromatograph.

Filtration process is necessary after mixing of solvent, and is more necessary in case of buffer to which un-dissolved impurities are source of deposits. After filtration, solvent should be keep in air-tight bottle from which small particles are removed; once solvent has been filtered, it is not necessary to filter this solvent everyday unless reaction produce bacteria or indissoluble material occurs. If solvent is kept in storage vessel for more than one week, it is desired to filter it again before use.

4-3-3. Solvent effect on the instrument

All parts of the Gradient Pump contacting with mobile phase is manufactured from 316 stainless steel, ruby, sapphire, zirconium, or fluorine carbon polymer. Most of these materials are sensitive to chloride, and it is desired to avoid use of solvent which contains even small quantity of chloride. Main solvents that should be avoided especially are as follows.

Aqua Regia	Hydrochloric Acid(HCL) (20%)
Bromine	HCL (37%)
Chlorine Anhydrous	HCL (50%)
Copper Chloride	HCL (20%)
Ferric Chloride	HCL (75%)
Ferrous Chloride	Hydrofluorsilicic Acid (20%)
Freon 12	Hydrogen Peroxide
Guanidine	Lodine
Hydrochloride (6M)	Mercuric Chloride
Hydrobromic (20%)	(Dilute Solution)

In addition, it should be avoided to leave chloroform, carbon tetrachloride, etc. in instrument for long time, and use of ammonium hydroxide should be avoided because it has effect on stator and rotor of injector even though it has no effect on pump. When not using it for long time, keep it with iso-propanol filled with in flow path.

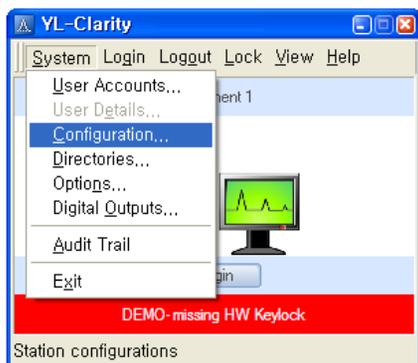
4-3-4. Measures when not uses for long time

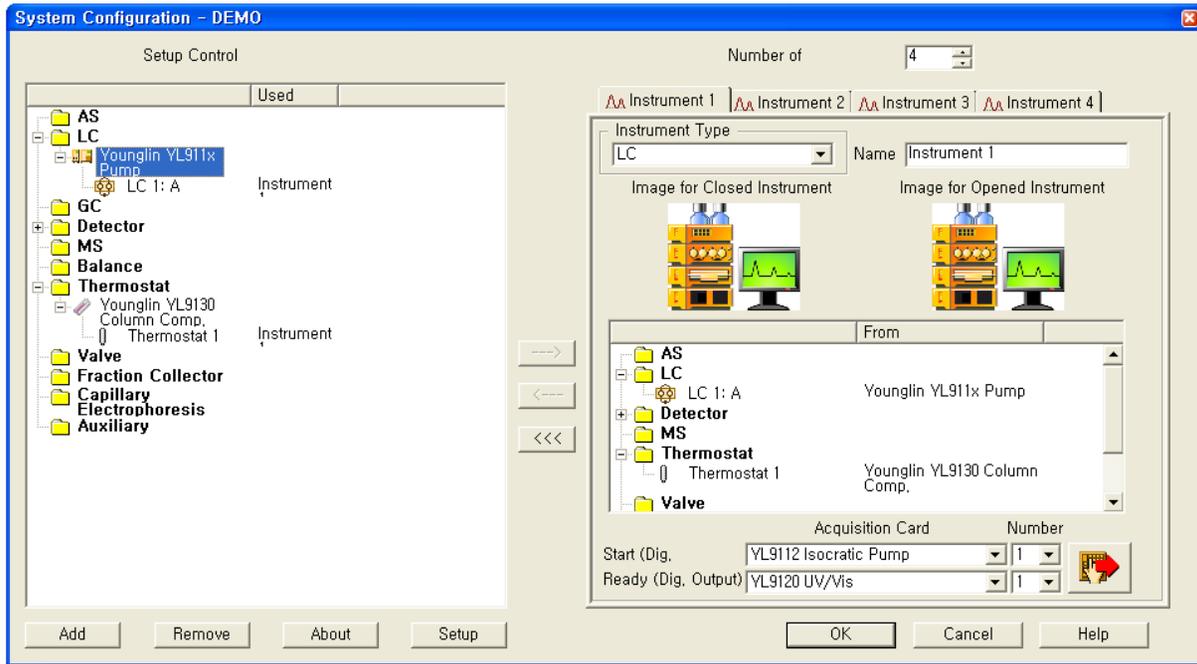
- 1) Prepare iso-propanol for analysis.
- 2) Open prime/purge pump and suck iso-propanol of at least 50ml into instrument.
- 3) Separate outlet tubing of pump.
- 4) Press out iso-propanol sucked into syringe in prime/purge valve and discharge at least 5ml into outlet of in-line filter.
- 5) Separate mobile phase filter assembly and block discharge hole and suction hole with cap.

4-4. YL-Clarity Chromatograph software

4-4-1. Installation of pump

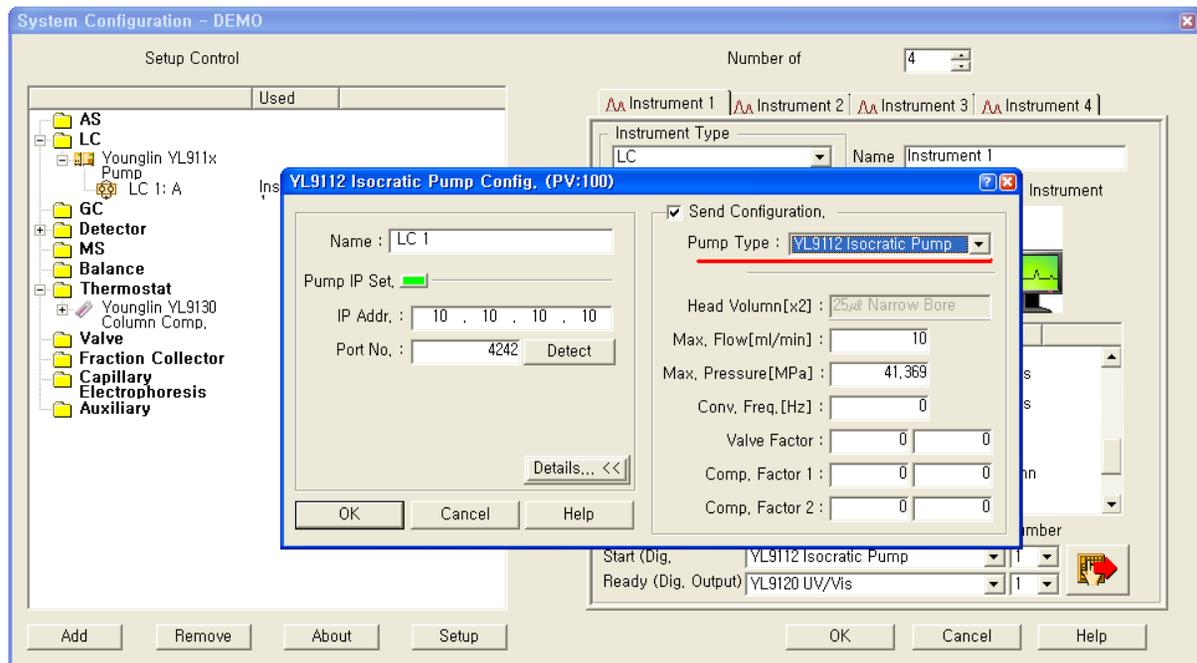
Open YL-Clarity software and select Configuration on the main window. On the system configuration window, click [ADD] button and select YL911x.





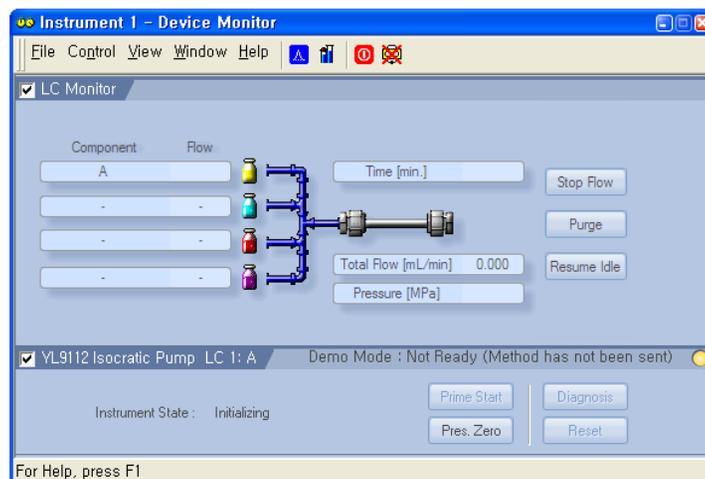
YL9112

After select YL911x pump on the left window, click arrow button to move this on the right window. Click red arrow button on the right bottom side and select the pressure unit. Double click YL911x pump on the right window, and check IP address of pump. Click "Details" button to select pump type as YL9112 isocratic pump.



4-4-2. Device Monitor

After configure the pump on the configuration window, log in to open main control window. On the main control window, click Device monitor and then Device Monitor window pops up as below. In this window, can control the pump and monitor instrument status as like flow and pressure.



[Control button]

Stop Flow : To stop the pump operation.

Purge : To run the pump initially. If you click this button, the window for setup solvent and flow pops up.

The pump starts according to the solvent ratio and flow value inputted on this window.

Resume Idle : If you click this button, the pump goes to idle state.

Prime Start : If you click this button, the pump runs at high speed with prime pump to fill the solvent into the lines. This function works only when the pump is not running. The prime pump inside pump runs when you click Prime start button.

Pressure Zero : To set present pressure to zero. Because the offset value of pressure sensor can be changed according to the temperature and using time, the pressure zero is necessary. Before you set the pressure zero, you should drop the pressure completely. This function works only when the pump is not running.

Diagnosis : To self test of instrument.

Reset : To release the pump status from the error.

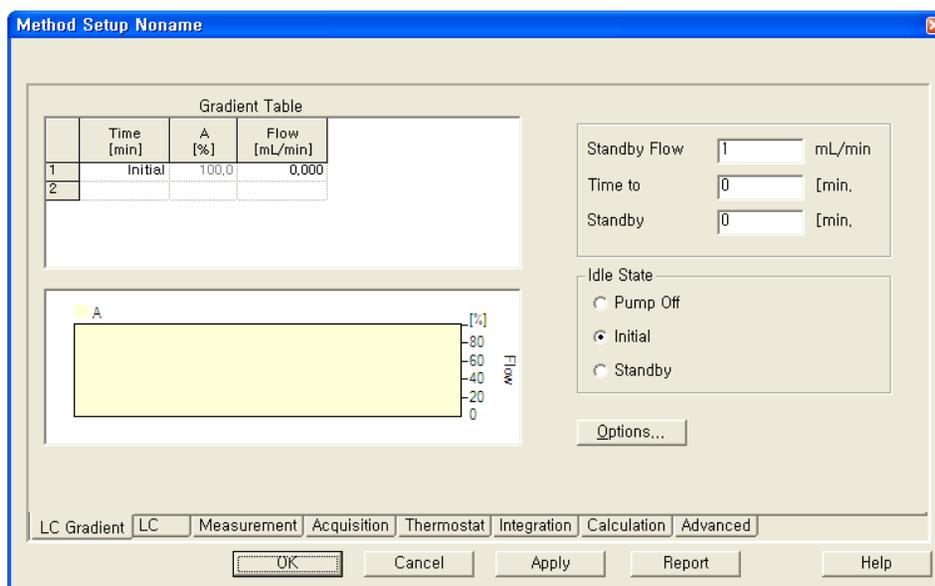
[Status message]

Initializing : It is displayed during initialization.

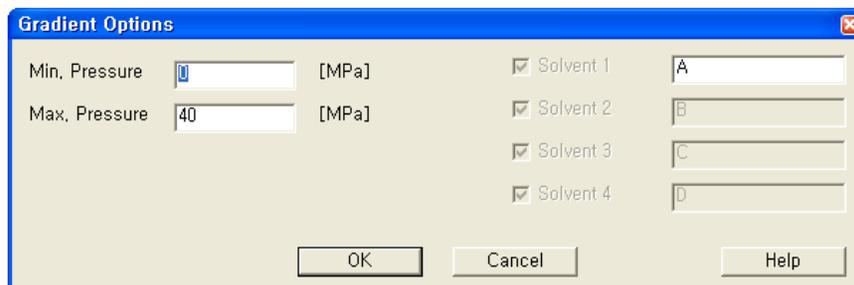
- Ready : It is displayed when the pump is ready.
- Prime : It is displayed during prime/purge status.
- Run : It is displayed during analysis.
- Fault : It is displayed if there is error on the pump.
- Halt : It is displayed if the pump stops.
- Diagnosis : It is displayed during self test.

4-4-3. Method Setup

In this window, edit time program table, and setup the pump status during idle state.

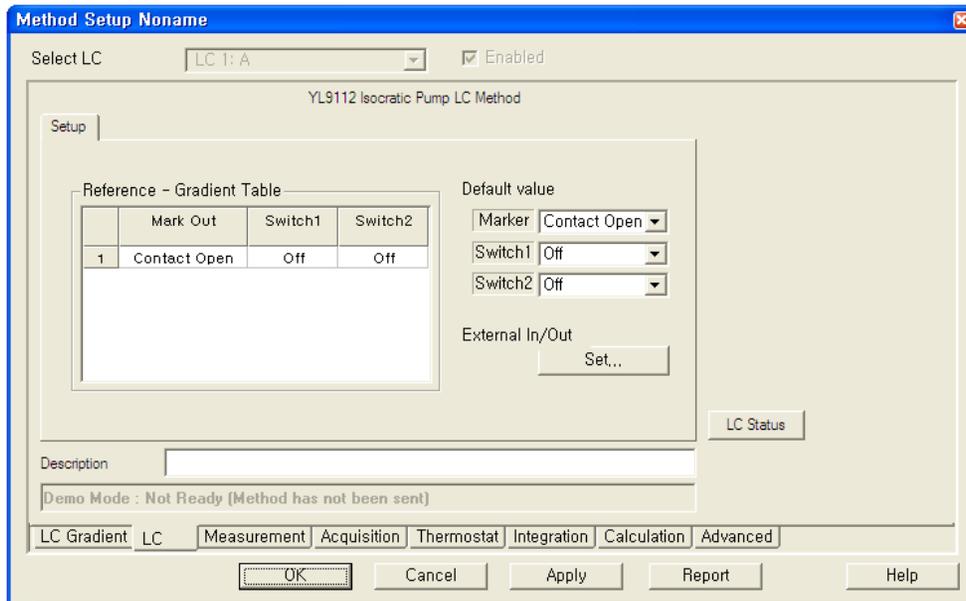


Click Options button to setup Max. and Min. pressure limit values to protect column and system. In this window, you can type the name of solvent you will use.



On the below window, make a program for output signal of switch terminal on the rear side of YL9112 isocratic pump.

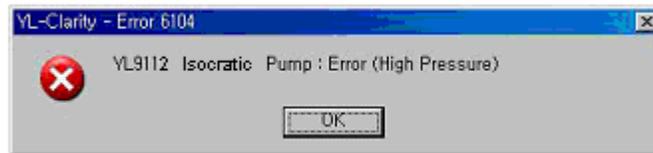
YL9112



If the signal is opposite when you use with the other device, change it on the External In/Out Set.



4-4-4. Error message



If there are errors on the pump caused by pressure limit, control value range, and leak, the pump stops operation with error message.

Chapter 5. Maintenance

In the event that problem occurs or it is necessary to change part due to wear of seal in using YL9112 isocratic pump, perform maintenance for instrument by referring to the following items.

5-1. Caution

In order to protect instrument, take care for the following items in using it.

- 1) After using solvent with sediment such as buffer solution, replace solvent with pure water at first and then methanol or iso-propanol and make it flow for 30 minutes using each solvent at 1.0ml/min flow rate.
- 2) Do not use solvent to corrode stainless steel material that is less than pH 2.3.

Material	Solvents to avoid
PEEK	Carbon Tetrachloride, Liquid Chlorine Methylene Chloride, Tetrahydrofuran
Teflon(PTFE)	Dimethyl Formamide, Diethylamine
SS316	Phosphoric Acid(Conc, Rm Temp)

- 3) Do not install instrument where corrosive gas is generated or where there is carpet on floor.
- 4) Do not change flow rate rapidly in order to prevent from wrong operation of instrument, damage to column and damage to damper.
- 5) Do not operate instrument with excessive force.

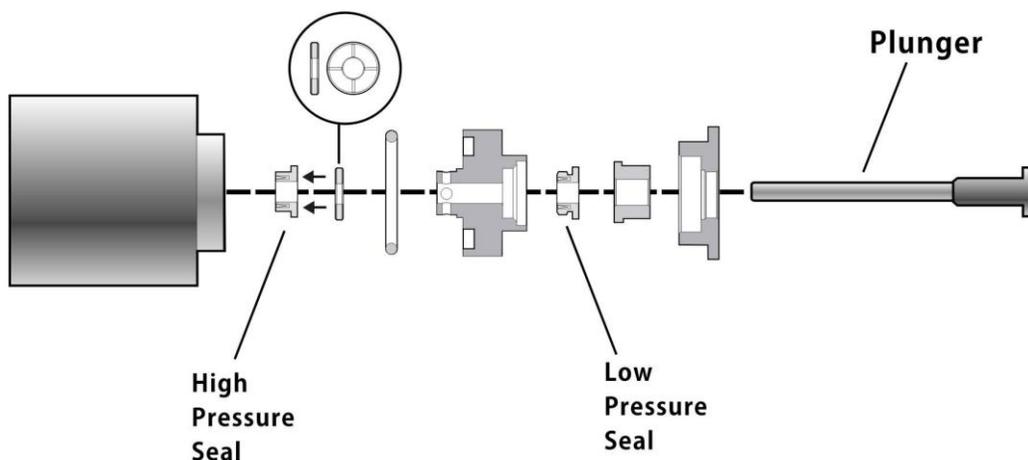
5-2. Replacement of high pressure seal

If instrument is used for long time, high pressure seal is worn out to produce leakage of solution. In this case, after replacing it with new seal, it is necessary to condition it in order to make seal be used for long time at high pressure. Change and condition high pressure seal in the following method. In case instrument is used for the first time after purchasing, it is desired to perform training and it is better to

change all seals of both head when change seal due to long use. Leaked solvent flows out through washing port.

Change of high pressure seal

- 1) Loosen tubing of inlet check valve and outlet check valve of pump head.
- 2) Loosen two head nut for each head using spanner.
- 3) Separate pump head assembly from instrument.
At this time, plunger is left at the place where pump head assembly was loosened. When loosening it, pull it carefully in pump head guide direction and take care not to damage plunger.
- 4) If backup washer in back side of pump head is pulled out, low pressure seal assembly appears. Use seal insertion/removal tool to separate low pressure seal assembly. Then, high pressure seal appears inside of head.
- 5) Remove worn seal with seal insertion/removal tool and insert new high pressure seal prepared at that place using seal insertion/removal tool in the same manner. Direction of seal should be such that the direction to see O-ring is toward front of head. Be careful not to change direction.
- 6) Insert low pressure seal assembly and backup washer.
- 7) Arrange pump head so that plunger left at the place where it is loosened be inserted into center hole of pump head assembly, and then press pump head to main body by inserting pump head by hand. When pressing it, press it carefully so that pump head may be maintained horizontal.
- 8) Tighten head nut in pressed condition. Tighten it so that left side and right side may be same, and tighten it until it is tightened no more by hand while confirming status of tightening finally.
- 9) Replace high pressure seal by applying the process of 1) to 8) to pump head of opposite side.



[Fig. 12] Replacement of high pressure seal

Conditioning of high pressure seal

- 1) Prepare organic solvent such as iso-propanol or methanol necessary. In order to conditioning it, use organic solvent only and do not use buffer solution and base solution.
- 2) Mix iso-propanol or methanol with water by 50:50, and fill instrument with it using the prime/purge valve. And plug the outlet of pump.
- 3) Set the high pressure limit to 2000psi and make flow rate be 0.2ml/min at isocratic mode, and do not make air bubble be present inside using prime/purge valve.
- 4) Start pump. The pressure will increase upto 2000psi and then the pump will be stopped with a high pressure limit message. Repeat this procedure 2-3 times and then conditioning of seal is completed.

5-3. Replacement of plunger

If piston plunger is used for long time, it should be replaced due to wear.

The worn piston causes leakage of liquid as well as shortens life of high pressure seal. Piston wear is not well observed visually, so care should be taken when observing it.

- 1) Loosen tubing of inlet check valve and outlet check valve of pump head.
- 2) Press pump head to main body of instrument by hand, and loosen head nut.
- 3) Separate pump head assembly from instrument. Then, plunger is left in the place where pump head assembly is loosened. When loosening it, pull it carefully to the direction of pump head guide so that high pressure seal may not be damaged due to eccentricity.
- 4) Pull plunger, replace it with new one at same location, and insert it.
- 5) If there are contaminants on surface of plunger, remove contaminant by applying methanol on cloth without dust.
- 6) Arrange plunger in the manner that plunger may enter the center hole of loosened pump head assembly, and then press pump head by hand so that head may be pressed into body. When pressing it, press it carefully and take care so that pump head may be maintained vertical.
- 7) Tighten head nut with it pressed. Tighten it so that left and right sides may be same in turn, and tighten it until it may be tightened no more by hand while confirming tightening status finally.

5-4. Replacement of check valve cartridge

If check valve is not well operated due to contamination, pressure change is severe during operation and pump does not operate properly. Many problems of check valve are caused by small impurity that interferes with operation of check valve. Therefore, if impurity is prevented from entering inside of pump head using mobile phase filter, malfunction of check valve is almost not caused. The cleaning of the check valve cartridges using sonication can solve this problem.

- 1) Separate tubing connected to pump head.
- 2) Loosen inlet and outlet check valve housing of pump head using spanner.
- 3) Wash check valve cartridge in separated check valve housing for about 30 minutes using ultrasonic cleaner with 10% nitric acid solution.
- 4) Using pure water, rinse check valve cartridge to remove the nitric acid used for cleaning.
- 5) Assemble loosened check valve in the reverse order.

5-5. Replacement of low pressure seal

Wearing of the low pressure seal is caused when pump has been used for long time without using washing port. In order to prevent wear of low pressure seal, it is desired to use washing port, and it is more desirable in case of using buffer solution. Leakage of liquid due to wearing of the low pressure seal is caused between pump head and body.

- 1) Separate pump head with reference to 5.2.
- 2) Separate washer, and pull out low pressure seal assembly from pump headbody with seal insertion/removal tool.
- 3) Replace low pressure seal attached to low pressure seal assembly with new one.
- 4) Assemble pump head in reverse order by referring to 5.2.

5-6. Cleaning of flow path within pump

In order to prevent occurrence of problem in instrument, remove impurity accumulated in instrument, and it is better to clean flow path when it is not used for long time. Clean inside of flow path in the following

method, and be careful when treating strong acid and strong base.

- 1) Separate column inlet tubing connected to column.
- 2) Orient column inlet tubing toward waste bottle.
- 3) Set flow rate at 1ml/min.
- 4) If injector is installed, turn it to injection position.
- 5) Pump 100% iso-propanol through pump and injector for 10 minutes.
- 6) Pump distilled water filtered through pump and injector for 10 minutes.
- 7) Pump 10% nitric acid solution for 5 minutes.
- 8) Wash pump and injector with distilled water filtered for at least 10 minutes.
- 9) Pump 100% iso-propanol through pump and injector for 5 minutes.

Now, pump is prepared for use of mobile phase or for the period not being used for short time or long time. If pump is not used for long time or there is contamination in flow path due to use of impure solvent, it is desired to separate pump head assembly and wash it with ultrasonic cleaner. In order to wash pump head, separate pump head into parts in the same manner as seal change process of 5.2, wash it with ultrasonic cleaner, and assemble each part again. At this time, the high pressure seal is damaged, so replace it with new one.

5-7. Supply of lubricant

YL9112 isocratic pump necessitates supply of proper lubricant into piston drive part for smooth operation of instrument. It is desired to use lubricant or low viscosity grease for piston carrier and pump housing and small amount of grease such as 630-AA for bearing of cam shaft and piston carrier. Care should be taken because pumping action is interfered with if lubricant is attached to surface of piston. Because shortening of pump life is caused where powder or dust is much generated, install instrument where surrounding environment is good.

5-8. Replacement of solvent filter and in-line filter

In case instrument is used for long time or mobile phase is bad, mobile phase filter and in-line filter is blocked due to small particles contained in solvent. If filter is blocked, pressure within flow path of pump is largely reduced when solvent is sucked to generate air bubble, make flow rate reduced and make

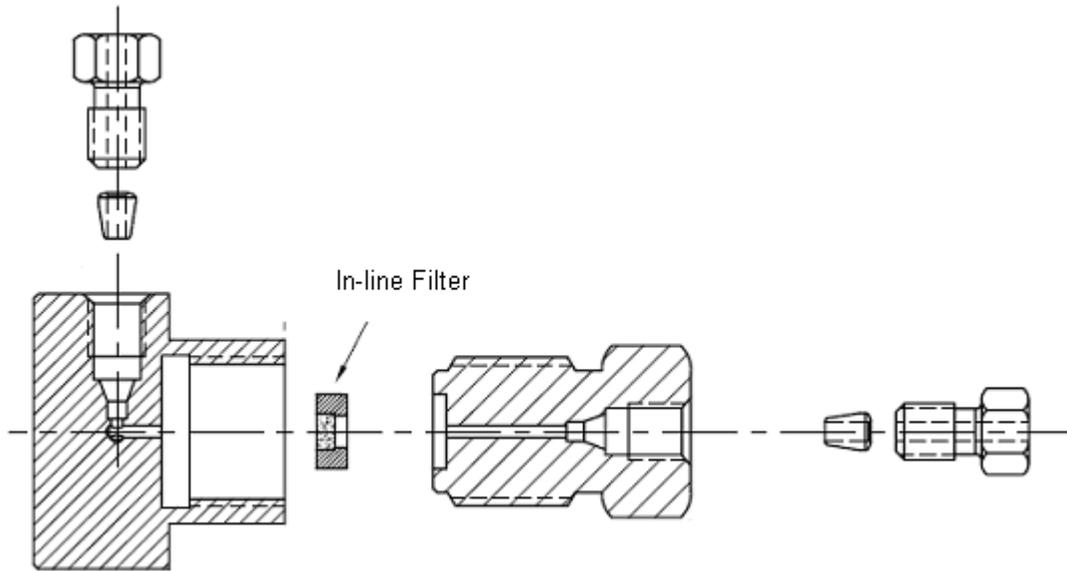
precision reduced ; so it is desired to check it periodically. Main cause of mobile phase filter blocking is growth of bacteria, and two causes to block inlet filter is growth of bacteria and use of solvent containing impurity. In order to prevent growth of bacteria, use at least 10-20% organic solvent or solvent containing growth depressing component. If pure water or soluble solution without interfering material is used, many bacteria will grow in mobile phase filter though it is replaced with fresh solution everyday. Therefore, use solvent of HPLC grade filtered well at all times for mobile phase. Blocking of in-line filter is caused by accumulation of small particles generated due to wear of high pressure seal by using of impure solvent and long use of instrument. In case mobile phase filter and in-line filter are contaminated, condition of filter may be improved by washing it by ultrasonic cleaner with 10% nitric acid solution for 30 minutes. If it is not improved by ultrasonic wave cleaning, replace it with new filter.

Change and cleaning of mobile phase filter

- 1) Separate mobile phase filter from tubing. As the surface of teflon tubing of mobile phase filter of insertion type is slippery, separate it with tubing held avoiding slippage using #1000 sand paper.
- 2) In case of performing ultrasonic wave cleaning, wash head part by ultrasonic cleaner with 10% nitric acid solution for 30 minutes, wash it again by ultrasonic cleaner with pure water for about 10 minutes, then dry it. In order to replace filter with new one, prepare new mobile phase filter of same size.
- 3) If washing has been completed, assemble filter to be replaced newly again. Hold teflon tubing using sand paper and insert mobile phase filter of insertion type with center adjusted into middle hole.

Changing and cleaning of in-line filter

- 1) Separate connected tubing from in-line filter assembly using spanner.
- 2) Separate head part of in-line filter assembly from body using spanner.
- 3) In case of performing ultrasonic wave cleaning, perform ultrasonic wave cleaning to head part for 30 minutes with 10% nitric acid solution, perform ultrasonic wave cleaning for 10 minutes with pure water, and then dry it. In order to replace filter with new one, separate filter located at back side of head part.
- 4) In case of replacing it with new filter, replace it with new in-line filter located at the location where it was separated ; and in case ultrasonic wave cleaning has been completed, re-assemble head part of dried assembly.
- 5) Using spanner, tighten head part sufficiently so that there may be no leakage of liquid even at 6000 psi.



[Fig. 13] Replacement of in-line filter.

5-9. After use system

If you keep the pump without cleaning, the pump can be damaged by the crystallization of salt, growth of microorganism, contamination. To maintain the pump properly, run the pump using iso-propanol for 30 minutes and the plug the outlet of pump, and keeps clean the solvent filter. Especially, after use buffer solution, run the pump using 100% water first then iso-propanol.

5-10. Troubleshooting

In case general problem occurs as the following table, confirm the possible causes regarding this first, and then take proper countermeasures. The following table is countermeasure in case general problems occur.

YL9112 Isocratic Pump

Problem	Cause	How to fix
Pressure upper limit is loaded.	<ul style="list-style-type: none"> -Tubing inside is blocked. -Check valve is blocked. -Solvent is changed. 	<ul style="list-style-type: none"> -Replace blocked tubing by loosening to be from tail side in turn. -Replace outlet check valve. -Wait until solvent is completely changed.
Pressure increases or decreases.	<ul style="list-style-type: none"> -Change of solvent is incomplete. 	<ul style="list-style-type: none"> -Wait until change is completely performed. -Wait until pressure is stable.
Solvent is not flowed out	<ul style="list-style-type: none"> -Air bubble is in pump head. -Air separation status of solvent is bad. -Check valve is not good. -Liquid containing oil flows into head part. -High pressure seal was worn. 	<ul style="list-style-type: none"> -Perform prime/purge again. -Take measures so that air separation condition of solvent may be good, and perform prime/purge again. -Wash or replace check valve. -Remove oil in head sufficiently with strong organic solvent using prime/purge port. -Replace the high pressure seal.
Pressure is unstable.	<ul style="list-style-type: none"> -Check valve is bad or defected. -Air separation or mixing conditions of solvent is bad. -Compression compensating reference value of configuration mode is wrong. -Cam shaft is loosened. -High pressure seal was worn. -Pump head was loosened. 	<ul style="list-style-type: none"> -Wash or replace check valve. -Use mobile phase with well mixed solvent, and improve air separation condition. -Initialize compensation value or re-input proper value. -Tighten wrench bolt of cam shaft -Replace high pressure seal. -Tighten nut of pump head.

<p>Liquid is leaked or pressure reduces when cleaning port is not used</p>	<ul style="list-style-type: none"> -High pressure seal was worn. -Plunger was worn. 	<ul style="list-style-type: none"> -Replace high pressure seal. -Replace plunger.
<p>Liquid is leaked from inside of instrument.</p>	<ul style="list-style-type: none"> -Fitting in instrument was loosened. -Damper was damaged. -Low pressure seal was worn. 	<ul style="list-style-type: none"> -Tighten fitting in instrument. -Replace. -Replace low pressure seal.
<p>Pump is not operated after input of power.</p>	<ul style="list-style-type: none"> -Power voltage is unstable or low. -Strong induction voltage is generated in the surroundings. 	<ul style="list-style-type: none"> -Use stable, proper DC power source. -Close induction power source in surroundings, or install instrument away from power source.
<p>Noise is too high</p>	<ul style="list-style-type: none"> -Load is caused to piston carrier. -Timing belt is loose. -Motor is in defect. 	<ul style="list-style-type: none"> -Supply lubricant in carrier body. -Reduce clearance above belt by adjusting guide location of timing belt. -Inspect connection status of motor cable, and replace motor if noise is severe only in high pressure.

YL9112

YL9100 HPLC System

YL9120 UV/Vis Detector

USER MANUAL



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Chapter 1. Introduction

The YL9120 UV/Vis Detector is the most sensitive and versatile absorbance detector available for HPLC. It features dual wavelength capability, improved signal-to-noise performance, minimized drift and reduced optical bandwidth. The YL9120 UV/Vis Detector is designed to provide higher performance in UV/VIS detection for HPLC application such as development, analytical research and QA/QC.

1-1. Features of YL9120

- 1) Higher light intensity by Seya-Namioka optic structure and wide wavelength range
- 2) Lower drift by the blazed holographic concave grating
- 3) Better designed flow cell assembly by compensated RI effect
- 4) Improved baseline stability with heat exchange effect
- 5) Great Usability by mounting lamps and flow cell assembly on the front
- 6) Dual wavelength detection and spectrum scanning

YL9120

1-2. Specifications

- 1) Wavelength Range : 190-900 nm
- 2) Data collection rate : up to 50Hz
- 3) Light Source : Deuterium arc lamp & tungsten lamp
- 4) 2-Order filter : Automatic filter switching
- 5) Bandwidth : 5.5 nm
- 6) Wavelength Accuracy : ± 1 nm
- 7) Wavelength Precision : ± 0.1 nm
- 8) Linearity : >99.5% at 2.5 AU (acetone, 254nm)
- 9) Noise level : $< \pm 0.5 \times 10^{-5}$ Abs/min , 254nm, dry cell
- 10) Drift : $< 1 \times 10^{-4}$ Abs/hour
- 11) Dispersion element : Concave grating
- 12) Warm-up period : 1 hour
- 13) Flow Cell Design : Cone type

- 14) Path Length : 10 mm (Analytical cell)
- 15) Cell Volume : 10 uL (Analytical cell)
- 16) Pressure limit : 1500psi (Analytical cell)
- 17) Wetted Materials : 316 stainless steel, Quartz, Teflon
- 18) Analog Outputs
 - A. Two, software selectable : Absorbance Energy, Sample Energy, Reference Energy
- 19) Communications : LAN
- 20) Power-up Diagnostics : Optics and electronic diagnostic routine
- 21) Power-up Wavelength Verification : Automatic on power up via internal holmium filter and D2 lamp
- 22) 5-Point Wavelength Calibration : On demand via internal holmium filter and D2 lamp
- 23) Safety & maintenance : Leak detection, Diagnostics, Error detection
- 24) Dimensions : 385 X 160 X 565mm (width X height X depth)
- 25) Line Voltage : 110 or 220 VAC, $\pm 10\%$
- 26) Line frequency : 50/60Hz, $\pm 5\%$
- 27) Power consumption : 100W

Chapter 2. Installation

2-1. Inspection and site preparation

Carefully unpack the detector from the shipping box and inspect both the unit and packing for any signs of damage. If any damage is noted, contact the shipping company immediately. In addition to this manual, the shipping box contains a power cord, and any options which you ordered. Carefully check the packing list against the contents of the container. If anything is missing, check the packing materials carefully for the overlooked items. If items are missing, contact us or your supplier. Place the detector on the bench where it will be used and familiarize yourself with the location and function of the controls and connections.

Site requirement of YL9120 UV/Vis Detector

- 1) Room with 20 °C temperature with variation ± 5 °C with and 60% humidity
- 2) Where no direct and straight sunshine
- 3) Plain floor without carpet
- 4) Where having spare space of 20cm or more
- 5) Where there are 10% or less voltage change, no frequency change and 100 Ω or less grounding point
- 6) Where there is no generation of corrosive gas and ventilation is well done
- 7) Where stable power of 110 or 220V is supplied, $\pm 10\%$
- 8) Where not receiving electromagnetic induction from large transformer, high frequency heater, UPS, etc.

Place the detector on a laboratory benchtop in close proximity to the HPLC column outlet. Allow at least 5 inches of clear space between the rear panel of the unit and any wall or obstruction. This provides both access to the rear panel connections and a free flow of air

In addition to the detector itself, you will need the following items for setup and initial operation :

- 1) YL-Clarity software or Chromatograph Data System.
- 2) Pump
- 3) Column
- 4) Standard test mixture

- 5) Appropriate solvents, reagents, etc
- 6) Nuts, ferrules, appropriate to the column end-fittings being used
- 7) Wrenches appropriate to column end-fittings
- 8) Connecting tubing and union (if column cannot be connected directly to the cell).

2-2. Connection of power

Confirm supplying voltage, electric power, socket, and type of connector are correct. Before connect power cable on the pump, please turn off the power switch of YL9120 UV/Vis Detector.

Warning ! : The electric power should be matched with description on the rear side of instrument. If not, the instrument can be damaged or it can be a reason of fire.

Caution ! : Confirm the ground is correct. Water pipe is not recommended in case of non-metal pipe. Gas pipe should not use because of safety. If the ground is not correct, the electric shock can be happened.

2-3. Connection of a column

The length of tubing between the inlet of flow cell assembly and outlet of column should be connected as short as possible. It is the ideal that you connect the tubing directly between these two ends. If this is not possible, you should use a minimum length of narrow bore (0.010 inch I.D.) connecting tubing and a zero dead volume union. Because different columns use different fittings, the detector is supplied with a bare tube end to allow connection to any column accepting 1/16 inch O.D. tubing. You should use nuts and ferrules suitable to your column.

NOTE: Tubing size and position is different for the adjustable path length preparative flow cells, high pressure narrow-bore flow cell, off column capillary flow cell, and on column capillary flow cell. See their owner's manuals for details.

Connect the cell outlet (the upper of the two tubes which protrude from the rear wall of the cell compartment) to a line leading to an appropriate waste reservoir. If bubble formation in the detector cell causes problems, you may wish to connect the cell outlet to a restrictor or back pressure device providing 20-60 psi back-pressure.

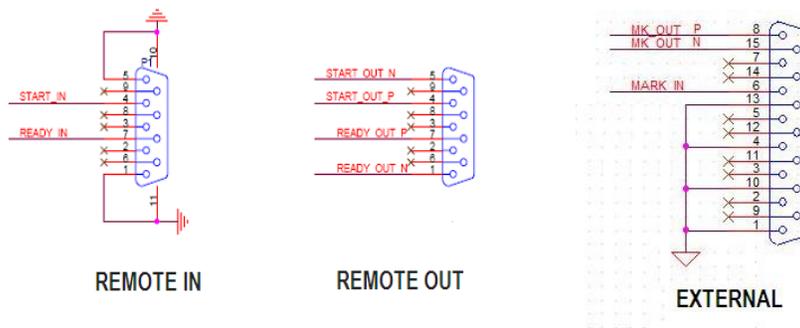
NOTE: Before connecting any new tube or column to the detector, flow several mL of clean solvent

through the new tube to a waste reservoir. This will clean any particulates or oil that may be residing in the tube that could clog the heat exchanger or contaminate the sample cell of the detector.

2-4. Connection of remote cable

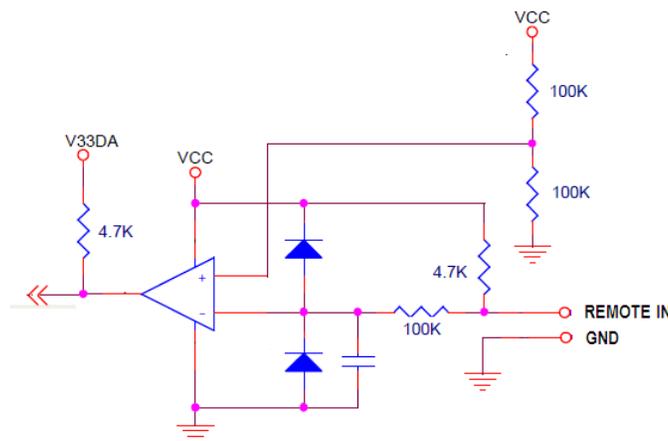
YL9120 UV/Vis detector has connection terminals for remote input/output, and marker input/output.

Note : Please do not connect wires between cables at your discretion. If you want to connect with the other instrument, please check input/output information and confirm with the terminal configuration of YL9120 UV/Vis detector.



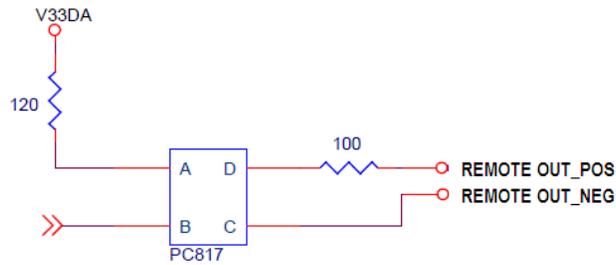
[Fig. 1] Pin configuration of each terminal

Fig. 2 and Fig. 3 are the diagram of remote and the other terminal input/output. In between YL9100 series modules, connect directly and confirm the configuration with the other modules.



[Fig. 2] Diagram of Remote and Marker input

YL9120



[Fig. 3] Diagram of Remote and Marker output

[Remote operation]

START-IN : Operate instrument, and start running of gradient program.
 If you connect it with autosampler or external valve, automatic running is available.

START-OUT : If the signal input on the START-IN terminal, the signal pulse output through this port. It can be used for synchronization of remote start with the other instrument.

MARK-IN : To control event program or operate additional operation.

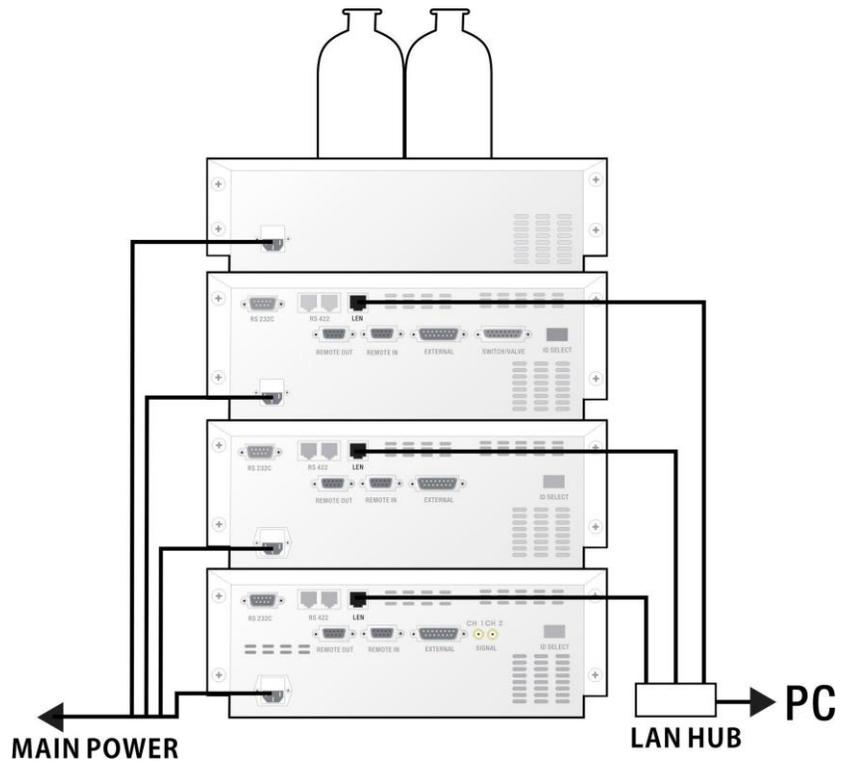
MARK-OUT : To control time event program output.

READY-IN : To change error state and stop operation if there is a input.

READY-OUT : When YL9120 UV/Vis Detector is not ready state because of running status, output error signal is indicated if there is a leak.

2-5. Connection of communication cable

YL9120 UV/Vis detector provides TCP/IP internet protocol as a standard. The IP address of YL9100 series is 10.10.10.20, if DIP SW settings on the rear side are On position. If you change the IP address using control software, the DIP SW has to be set OFF.



[Fig. 4] Connection of communication cable

Note : The LAN HUB used for cable connection on the PC must use switching mode module.

Chapter 3. Operation

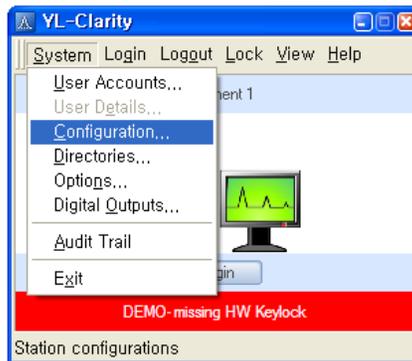
There are four LEDs in front of YL9120 UV/Vis detector.

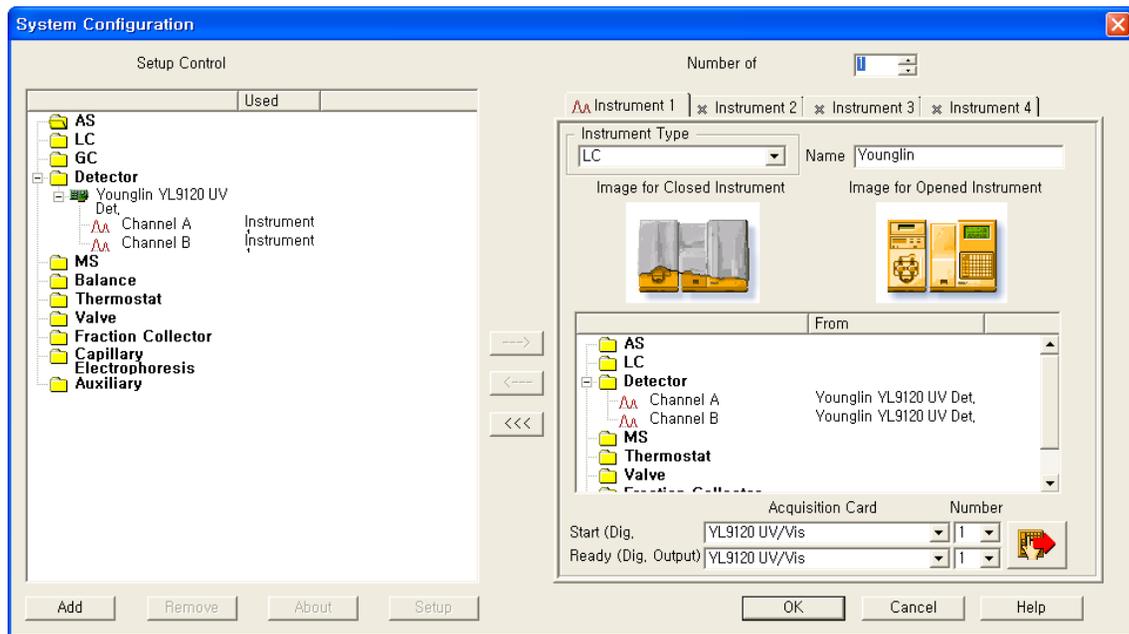
	POWER	LED turns ON if main power turns on
	CONNECTED	LED turns ON if communication is connected, LED blinks during connection
	READY/RUN	LED turns ON before analysis, LED blinks during analysis
	ERROR	LED turns ON if there is error

3-1. YL-Clarity Chromatograph software

3-1-1. Installation of UV/Vis detector

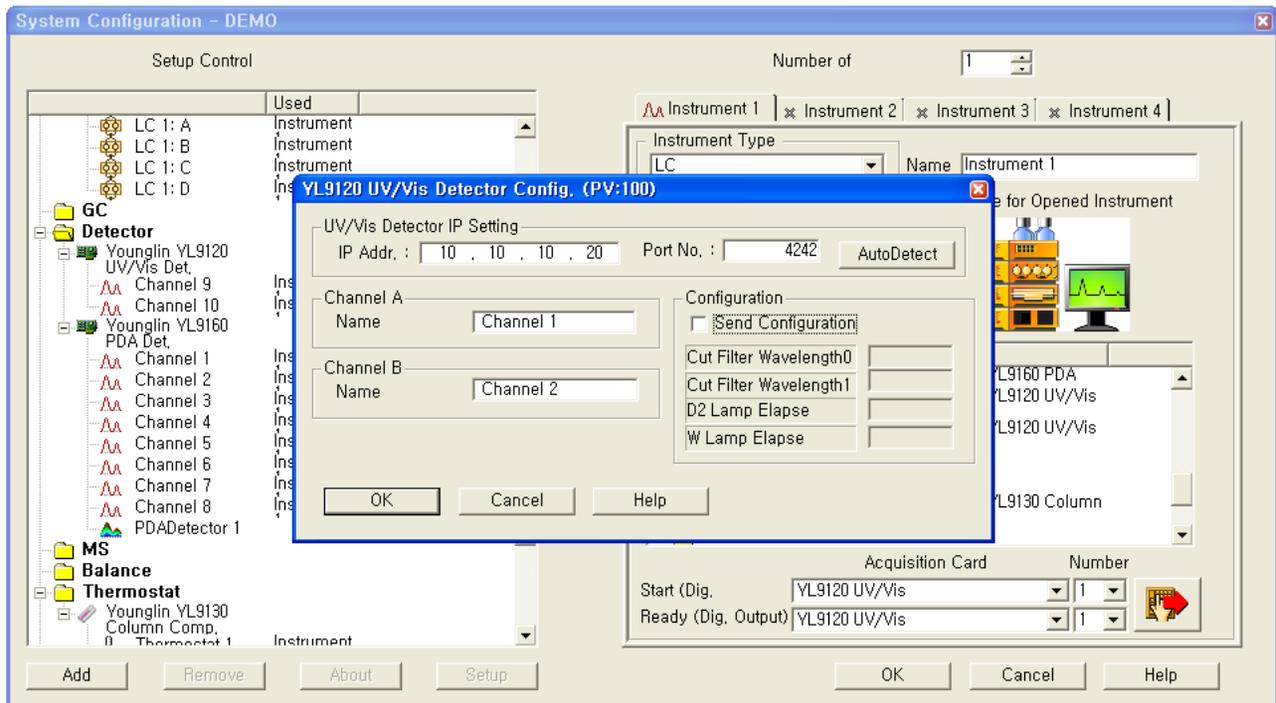
Open YL-Clarity software and select Configuration on the main window. On the system configuration window, click [ADD] button and select YL9120.





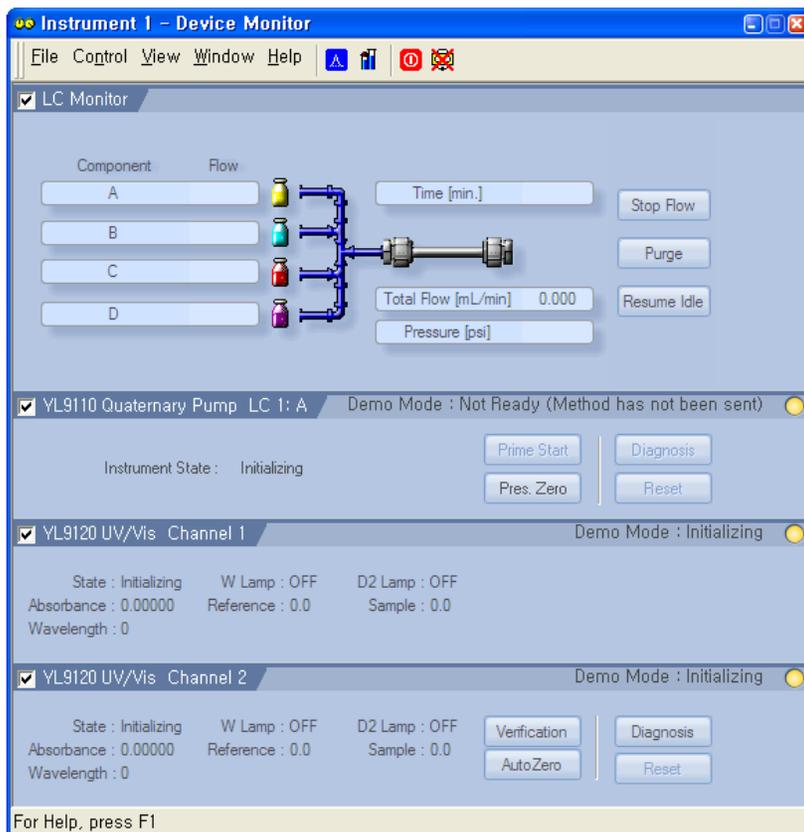
Double click YL9120 UV/Vis detector on the right window, and check IP address of the detector.

YL9120



3-1-2. Device Monitor

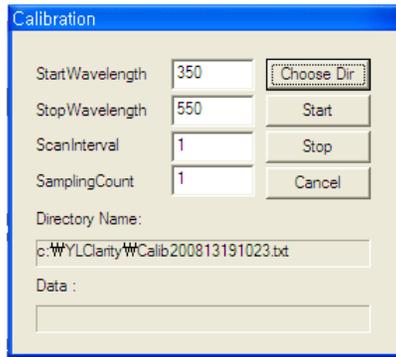
After configure the UV/Vis detector on the System Configuration window, log in to open main control window. On the main control window, click Device monitor and then Device Monitor window pops up as below. In this window, you can control the UV/Vis Detector and monitor instrument status as like lamp on/off, wavelength selection, sampling rate, etc.



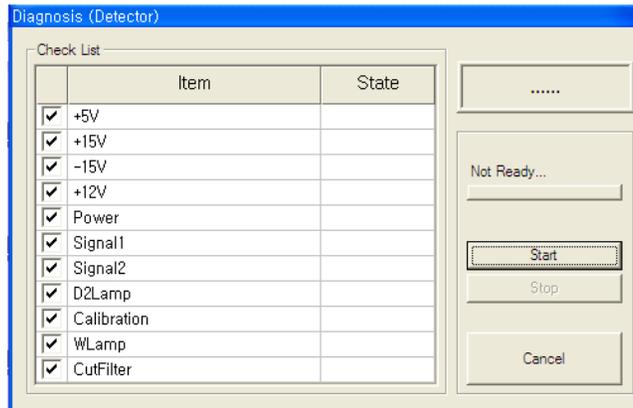
The Device Monitor shows the state, lamps on/off, the light intensity of reference and sample, absorbance, and wavelength.

Descriptions of control buttons

Verification : To inspect wavelength accuracy of the YL9120 as scanning of a specified wavelength.



Diagnosis : To Inspect hardware condition of the YL9120.



Autozero : To change the absorbance to Zero “0”

Reset : To release the UV/Vis detector status from the error.

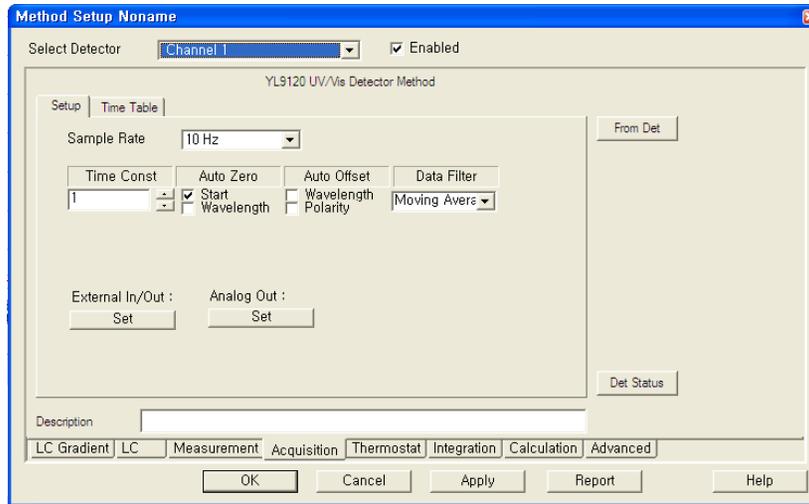
[Status message]

- Initializing : It is displayed during initialization.
- Ready : It is displayed when the pump is ready.
- Run : It is displayed during analysis.
- Fault : It is displayed if there is error on the pump.
- Diagnosis : It is displayed during self test.

3-1-3. Method Setup



In the table below, edit programming of time table, and setup the detector status during idle state



Time Constant : To input the value of digital filter.

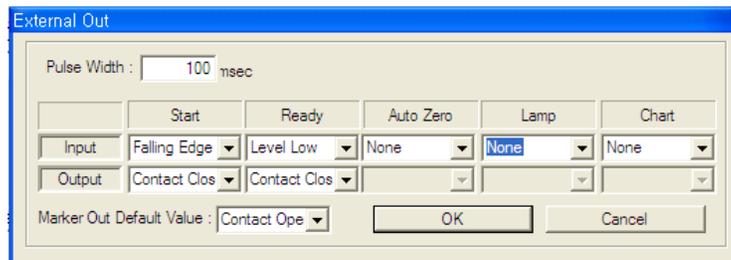
If value is larger, the baseline is smoother while peak width is wider.

Auto Zero : To change the absorbance to zero

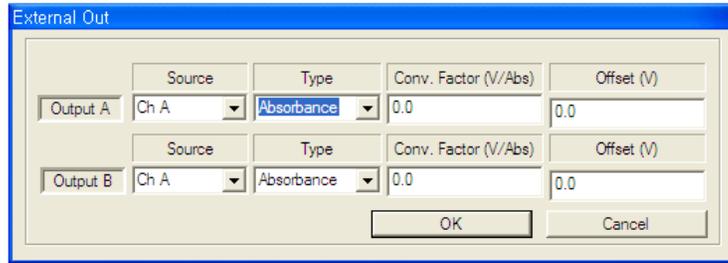
Auto Offset : To prolong the baseline by adjustment automatically, If either wavelength or polarity is changed.

Data Filter : To select which filter you use.

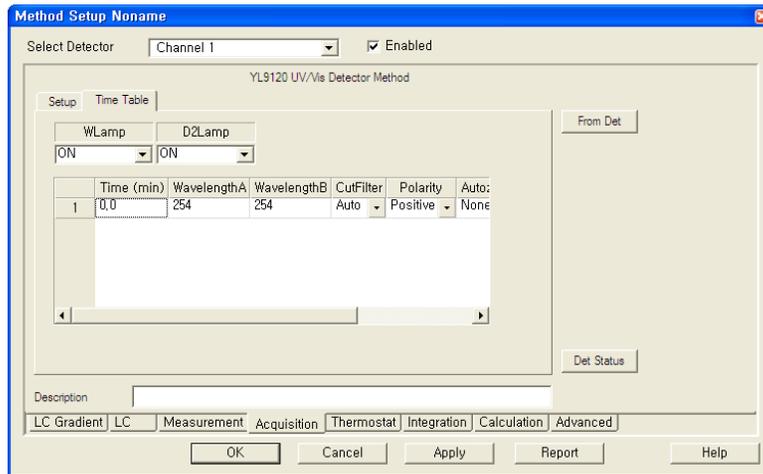
If the signal is opposite when you use with another device, change it on the External In/Out.



If you use an A/D converter for data acquisition, setup the each value of source, type, conversion factor and offset.



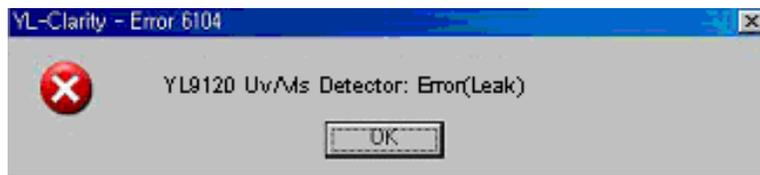
Setup the Time Table, choose lamps on/off and edit the time table such as wavelength A/B, CutFilter, Polarity, etc. according to time programming.



YL9120

3-1-4. Error message

If there is a leak from the detector, it stops operation with error message.



Chapter 4. Maintenance

4-1. D2 lamp

The Deuterium Lamp (D₂) is covered from 190 to 600 nm of wavelength. The replacement time is either in case you use the lamp more than 2000 hours or in case the light intensity is a half (50%) of the original intensity when you installed the lamp at first. An using time of D2 lamp is counted in the system indicating the total hours of operation after lamp on.

Check the lamp intensity as follows:

- 1) Power on the unit if it is not already on. Wait for a period of approximately 10 minutes.
- 2) Set the wavelength to 254 nm by YL-Clarity software
- 3) Select the Reference Light Intensity.
- 4) If the displayed value is less than a half of the original intensity, the lamp should be replaced to new one. Generally, you have to consider the lamp exchange when the light intensity of reference energy is less than 50 nA.

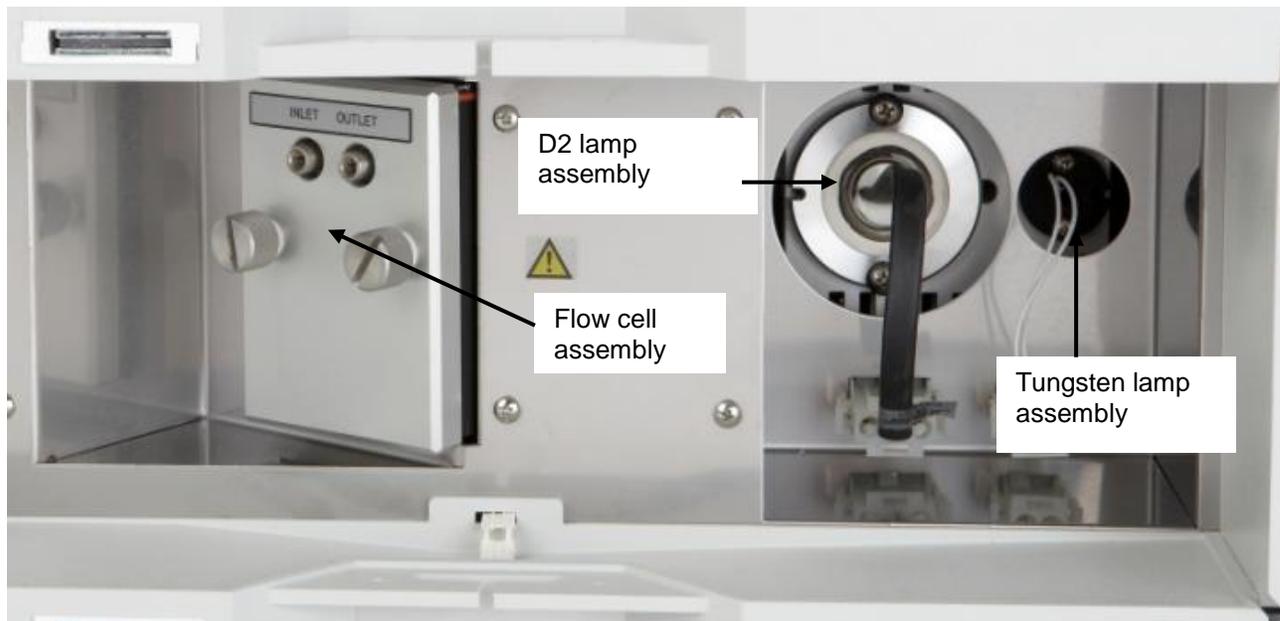
4-1-1. How to remove the D₂ lamp

- 1) Make sure that the power cord is disconnected from the rear panel of the detector.
- 2) Unscrew the screws and remove the lamp assembly on the right front panel.
- 3) **CAUTION** ; UV light can damage eyes and skin. Always disconnect the power cord before working in the vicinity of the lamp. The D₂ lamp gets quite hot. Care must be taken while handling it to prevent from burning. Always allow the lamp to cool before removing it.
- 4) Disconnect the UV lamp from the detector by gently pulling it straight back toward you. DO NOT twist the connector while pulling.
- 5) Unscrew the two thumbscrews holding the lamp mount in place, and pull the lamp mount straight back
- 6) towards you. Be careful not to lose the two thumbscrews. Be careful not to get fingerprints on the lamp.

4-1-2. How to install a new D₂ lamp

- 1) Insert the new lamp assembly onto the lamp housing on the right front panel.
- 2) Use the thumbscrews to attach the lamp assembly to the detector.
- 3) Connect the lamp lead to the lower of the two terminals in the lamp compartment.

CAUTION ; NEVER loosen the screw holding the lamp to the mount, and never attempt to rotate or move the lamp up or down in the mount. The lamp is provided as a pre-aligned assembly.



[Fig. 5] Front parts of YL9120 UV/Vis detector

4-2. Tungsten lamp (W)

The replacement time of the tungsten lamp is approximately 1,500 hours. To check the W lamp intensity:

- 1) Power on the unit if it is not already on. Wait approximately 10 minutes.
- 2) Set the wavelength to 720nm by YL-Clarity software.
- 3) Select the Reference Light Intensity.

- 4) If the displayed value is less than a half of the original intensity, the lamp should be replaced to new one. Generally, you have to consider the lamp exchange when the light intensity of reference energy is less than 5 nA.

4-2-1. How to remove the Tungsten lamp

- 1) Make sure that the power cord is disconnected from the rear panel of the detector.
- 2) Unscrew the screws and remove the lamp assembly on the right front panel.

CAUTION ; The light of Tungsten lamp can damage eyes and skin. Always disconnect the power cord before working in the vicinity of the lamp. The W lamp gets quite hot. Care must be taken while handling it to prevent from burning. Always allow the lamp to cool before removing it.

- 3) Disconnect the Tungsten lamp from the detector by gently pulling it straight back toward you. DO NOT twist the connector while pulling.
- 4) Unscrew the two thumbscrews holding the lamp mount in place, and pull the lamp mount straight back towards you. Be careful not to lose the two thumbscrews. Be careful not to get fingerprints on the lamp.



[Fig. 6] Tungsten lamp assembly

4-2-2. How to install a new Tungsten lamp

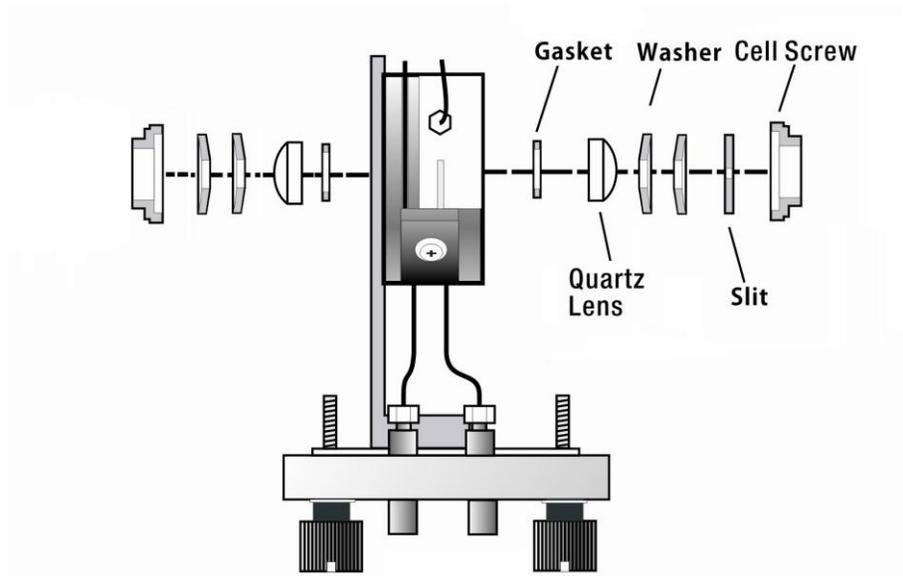
- 1) Insert the new lamp assembly onto the lamp housing on the right front panel.
- 2) Use the thumbscrews to attach the lamp assembly to the detector.
- 3) Connect the lamp lead to the lower of the two terminals in the lamp compartment.

4-3. Flow cell cleaning

We do not recommend disassembly of the flow cell assembly for routine cleaning purposes. Most of the cell assembly can be adequately cleaned by flushing with several milliliters of appropriate solvent. We recommend the following solvents for this purpose:

- 1) Methanol
- 2) Tetrahydrofuran
- 3) Methylene Chloride
- 4) HPLC Grade Water
- 5) 6 N Nitric Acid followed by flushing with HPLC Grade Water

NOTE : Use only HPLC grade solvents.



[Fig. 7] The diagram of Flow cell assembly

4-4. Troubleshooting

Most problems with HPLC detectors are, in fact, caused by other parts of the system. Noisy and drifting baselines, poor reproducibility in quantitative analysis, and similar problems are more often the result of dissolved air bubbles, contaminated eluents, dirty samples, or damaged columns rather than of actual problems with detector hardware. In order to focus more effectively on troubleshooting detector problems, we will first discuss on-board diagnostic tips and later present a troubleshooting table organized by symptom, cause and how to fix.

Problem	Cause	How to fix
1. Unstable Baseline	Bubbles passing through cell.	Degas solvent and/or supply back pressure to the sample cell, also check all high pressure fittings for leaks(both liquid and gasses)
	External triggering device is creating electrical noise.	Check electrical lines for good connection and/or interference from broad cast radiation. Check for ground loops.
	Extremely large supply voltage transient on the AC line	Remove systems that consume high power from the AC line.
2. Irregular Baseline Noise	Sample cell windows are contaminated.	Flush cell with solvents(methanol, acetone, water, nitric(6N) acid, water) and check for leaks.
	Sample input line has a leak.	Check all lines from the output of the column to the input of the sample cell for leaks.
	Bubble trapped in the sample cell.	Increase flow rate and/or back pressure on cell.
	Recorder or integrator is grounded and is causing a "ground loop" problem.	Check recorder with voltmeter to see if either of the signal inputs is grounded to case or earth ground.
	Photodiode window is dirty or not held down properly to the cell holder.	Remove and clean photodiode window.
	Sample cell is not screwed down to the main unit.	Check sample cell mounts and cell holder assembly.
	Output span of the detector does not match input range of integrator.	Press event mark to see if the "spike" is approximately 20% of scale.
3. Baseline Drift	External triggering device is causing a ground loop problem.	Use only triggering device with ground isolated from earth ground.
	Contamination of sample cell windows has occurred.	Clean cell by flushing with solvents (methanol, acetone, water, nitric(5N) acid). Inspect cell and photodiode for fingerprints and smudges and clean if necessary.

	<p>The absorption of solvent in the column has been changed.</p>	<p>Column is filled with UV absorbers that are bleeding-replace column; impure solvent is equilibrating with the column-replace solvent with more pure grade, switch to a longer wavelength so that background absorption does not fluctuate as much.</p>
	<p>Leakage in the lines from column to flow cell.</p>	<p>Check lines for leakage.</p>

YL9100 HPLC SYSTEM

YL9131 COLUMN COMPARTMENT

USER MANUAL



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Chapter 1. Introduction

YL9131 column compartment is a thermostat to control column temperature of HPLC system. YL9131 column compartment uses RTD sensor to read temperature and PID control to control peltier device, it provides wide temperature range and excellent temperature accuracy. The followings are standard specification of YL9131 column compartment.

1-1. Specifications

- 1) Temperature range : 4°C (Cooling) - 90 °C
- 2) Temperature stability : $\pm 0.05^{\circ}\text{C}$
- 3) Temperature accuracy : $\pm 0.5^{\circ}\text{C}$ with 2-point temp. calibration
- 4) Temperature precision : $\pm 0.1^{\circ}\text{C}$
- 5) Temperature programs : 40 Steps
- 6) Column capacity : Total 3 columns up to 300mm length (max OD: up to 18mm)
- 7) Column switching : Automatically 6-port valve (optional) up to 2 ea
- 8) Heat-up time : 16 minutes from 4°C to 90°C
- 9) Cool-down time : 13 minutes from 90°C to 4°C
- 10) Preheat : Heat exchanger tube ID is 0.01inch
- 11) External input : Start, Ready
- 12) External output : Start, Ready Mark out
- 13) Communications : LAN
- 14) Safety & maintenance : Leak detection, Diagnostics, Error detection
- 15) Dimensions : 385 X 160 X 565mm (width X height X depth)
- 16) Line Voltage : 110 or 220 VAC, $\pm 10\%$
- 17) Line frequency : 50/60Hz, $\pm 5\%$
- 18) Power consumption : 150W

Chapter 2. Installation

2-1. Inspection and site preparation

YL9131 column compartment is delivered along with the following parts when being shipped. Before opening transportation package, perform inspection for trace of shock or mistake, and if there is abnormality, do not open the contents and inform this company of it. And, if contents are opened, perform inspection for existence of shock in the contents and contact with this company when trace of shock is found.

The column compartment is a delicate instrument, so use original box and buffer material as far as possible when re-packing it to transport instrument. If it is impossible to use original box; wrap pump with several layers of buffer material, and fill the bottom, top and all other sides of pump with buffer material in order to make pump endure shock or vibration during transportation.

Standard configuration of YL9131 column compartment

- 1) Main body of instrument
- 2) Power cord and fuse
- 3) Installation kit
- 4) Manual

Site requirement of YL9131 column compartment

- 1) Room with 20℃ temperature with variation $\pm 5^{\circ}\text{C}$ with and 60% humidity
- 2) Where no direct and straight sunlight
- 3) Plain floor without carpet
- 4) Where having spare space of 20cm or more
- 5) Where there are 10% or less voltage change, no frequency change and 100 Ω or less grounding point
- 6) Where there is no generation of corrosive gas and ventilation is well done
- 7) Where stable power of 110 or 220V is supplied
- 8) Where not receive electromagnetic induction from large transformer, high frequency heater, UPS, etc.
- 9) Within 2500 m above sea level(storage within 4600m)

Please check the following before you install the system.

- 1) Keep the ventilation as normal state.
- 2) Install on the stable place. Avoid the places as like near to air conditioner and heater, direct sun light, near to window.
- 3) Keep the place without dust and vibration.
- 4) Maintain voltage variation within 5% of proper voltage.
- 5) Avoid high frequency or strong magnetic field environment.
- 6) Avoid from the source of fire(spark, flame).
- 7) Keep the proper ground for electricity.
- 8) Check the place of water supply for emergency.

Caution ! : Keep distance with CRT at least 50cm.

Caution ! : Column compartment emits heat from the rear side, keep distance 10cm from the wall.

2-2. Connection of power

Confirm supplying voltage, electric power, socket, and type of connector are correct. Before connect power cable on the pump, please turn off the power switch of YL9131 column compartment.

Warning ! : The electric power should be matched with description on the rear side of instrument. If not, the instrument can be damaged or it can be a reason of fire.

Caution ! : Confirm the ground is correct. Water pipe is not recommended in case of non-metal pipe. Gas pipe should not use because of safety. If the ground is not correct, the electric shock can be happened.

2-3. Installation of switching valve and column

YL9131 column compartment has two positions for column switching and sample injection valve. These valves can be controlled by YL-Clarity software.

YL9131 Column Compartment

Choose either the Pos1 or Pos2 of Valves.

The screenshot shows the 'Method Setup 111' dialog box with the 'Setup' tab selected. The 'YL9131 Column Comp. Method' section is active. Under 'Not Controlled', the 'Set Temp. [°C]' is 35, 'Min. Temp. [°C]' is 4, and 'Max. Temp. [°C]' is 90. The 'Status after Analysis Termination' is set to 'Initial temp.'. In the 'Default value' section, 'Marker' is 'Contact Open', 'Valve1' is 'Pos. 1', and 'Valve2' is 'Pos. 1'. The 'External In/Out' button is labeled 'Set...'. The 'Status' field shows 'Not Ready (Method has not been sent)'. The bottom navigation bar includes 'Event Table', 'LC Gradient', 'LC', 'Measurement', 'Acquisition', 'Thermostat', 'Integration', 'Calculation', and 'Advanced'. Buttons at the bottom are 'OK', 'Cancel', 'Send method', 'Report', 'Audit Trail', and 'Help'.

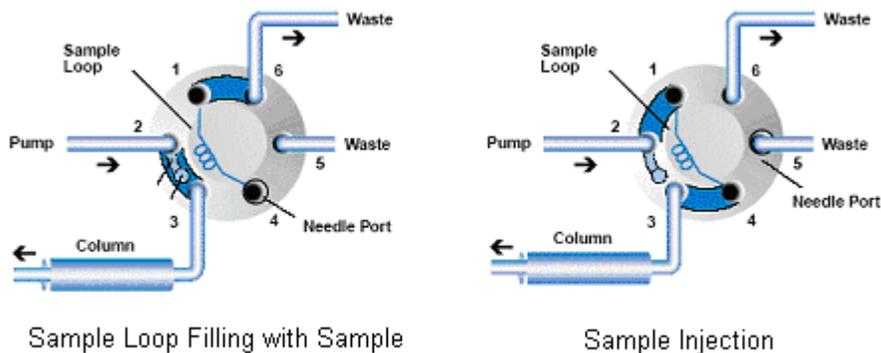
Change the valve position on the tab of time table.

The screenshot shows the 'Method Setup 111' dialog box with the 'Time Table' tab selected. The 'YL9131 Column Comp. Method' section is active. A table is displayed with the following data:

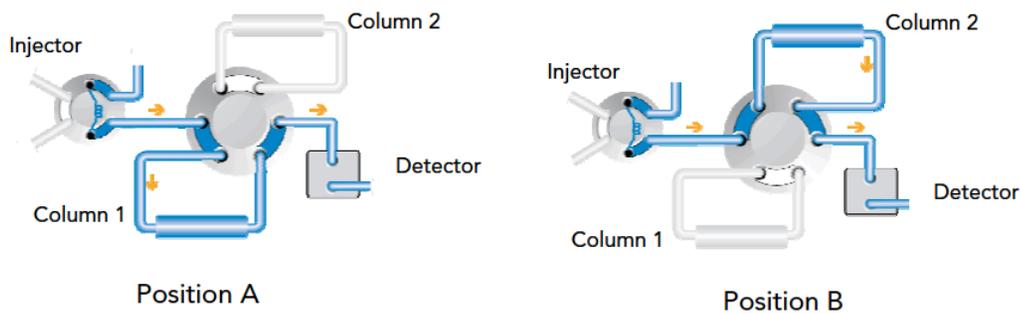
	Time (min)	Temp. (°C)	Marker	Valve1	Valve2
1	Initial	35	Contact Open	Pos. 1	Pos. 1
2	0.0				

The 'Reset' button is visible next to the table. The 'Status' field shows 'Not Ready (Method has not been sent)'. The bottom navigation bar and buttons are the same as in the previous screenshot.

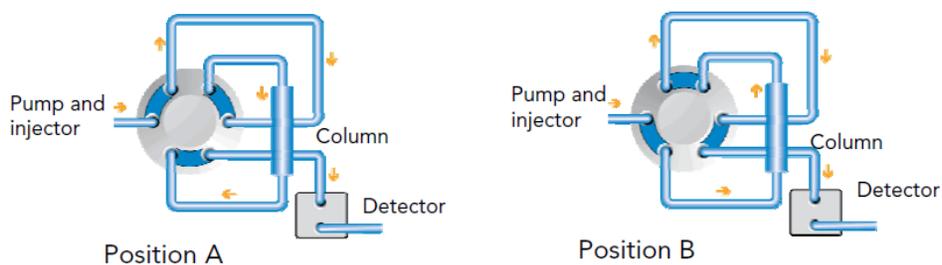
If you configure the YL9131 column compartment with switching valve, use fittings supplied with system. Followings are valve configurations.



[Fig. 1] Configuration of injection valve



[Fig. 2] Configuration of switching valve



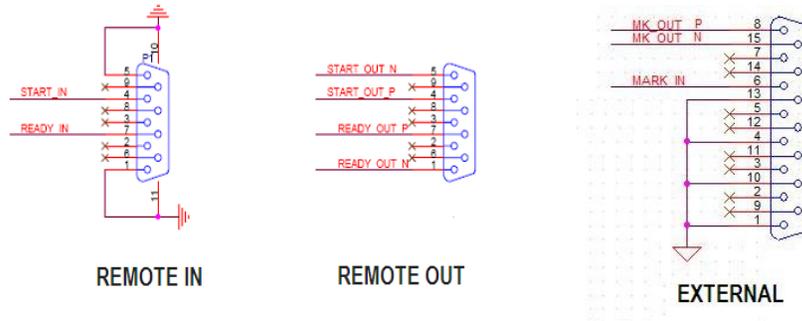
[Fig. 3] Configuration of Back Flush Elution for pre-column

YL9131

2-4. Connection of remote cable

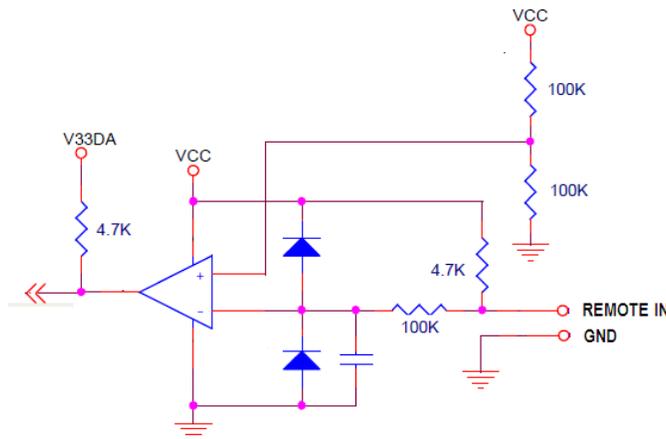
YL9131 column compartment has connection terminals for remote input/output, external solenoid valve, and marker input/output. The remote cable from the injector(manual or autosampler) has to be connected on the Remote In terminal on the rear side of YL9131 column compartment to collect data at the moment of injection.

Notice ! : Please do not connect wires between cables at your discretion. If you want to connect with the other instrument, please check input/output information and confirm with YL9131 column compartment terminal configuration.

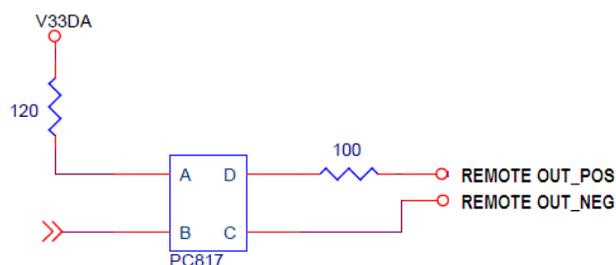


[Fig. 4] Pin configuration of each terminal

<Fig 6> and <Fig 7> are the diagram of remote and the other terminal input/output. In between YL9100 series modules, connect directly and confirm the configuration with the other modules.



[Fig. 5] Diagram of Remote and Marker input



[Fig. 6] Diagram of Remote and Marker output

[Remote operation]

START-IN : Operate instrument, and start running of gradient program.

If you connect it with autosampler or external valve, automatic running is available.

START-OUT : If the signal input on the START-IN terminal, the signal pulse output through this port. It can be used for synchronization of remote start with the other instrument.

MARK-IN : To control event program or operate additional operation.

MARK-OUT : To control time event program output.

READY-IN : To change error state and stop operation if there is a input.

READY-OUT : When YL9131 column compartment is not ready state because of running status, output error signal if there is a leak.

2-5. Connection of communication cable

YL9131 column compartment provides TCP/IP internet protocol as a standard. The IP address of YL9100 series is 10.10.10.30, if DIP SW settings on the rear side are On position. If you change the IP address using control software, the DIP SW has to be set OFF.

Chapter 3. Operation

3-1. Before Start

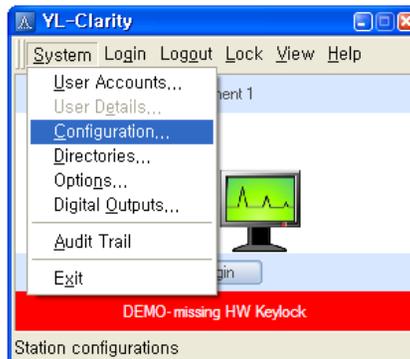
There are four LEDs in front of YL9131 Column Compartment.

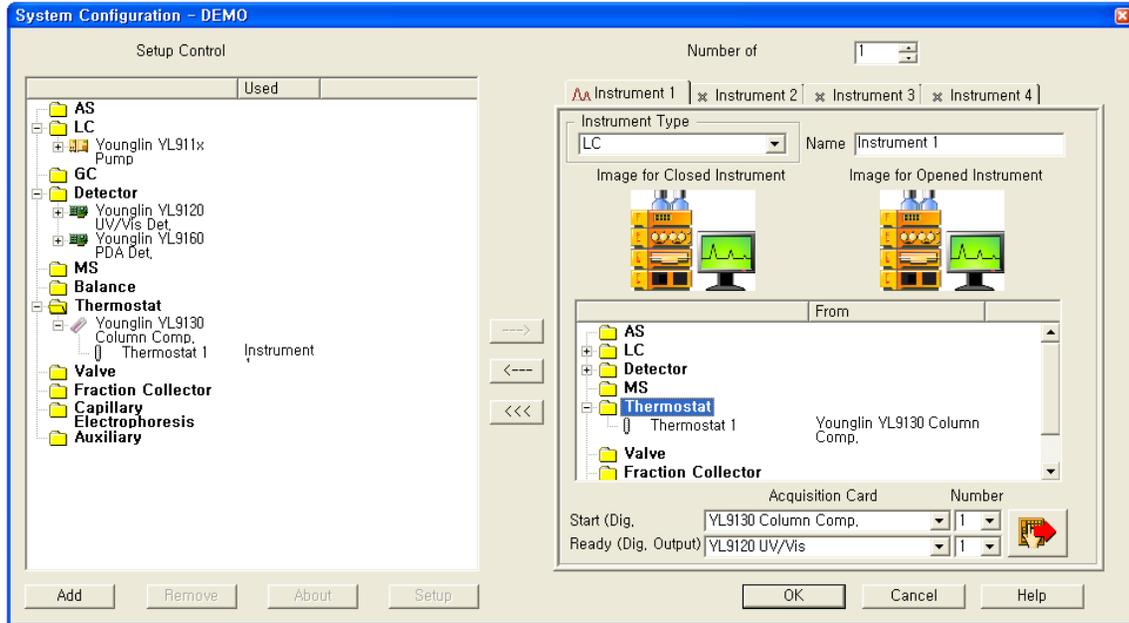
	POWER	LED turns ON if main power turns on
	CONNECTED	LED turns ON if communication is connected, LED blinks during connection
	READY/RUN	LED turns ON before analysis, LED blinks during analysis
	ERROR	LED turns ON if there is error

3-2. YL-Clarity Chromatograph software

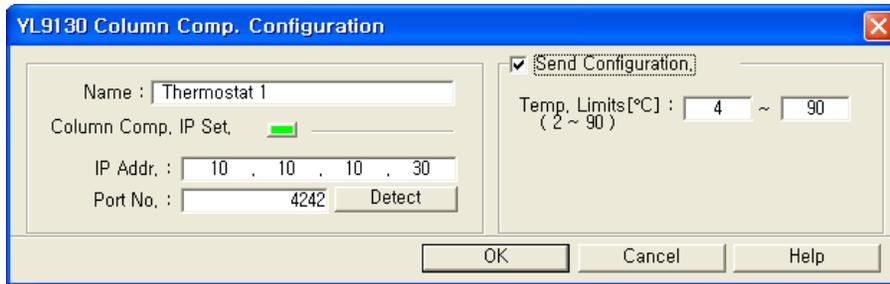
3-2-1. Installation of column compartment

Open YL-Clarity software and select Configuration on the main window. On the system configuration window, click [ADD] button and select YL913x.



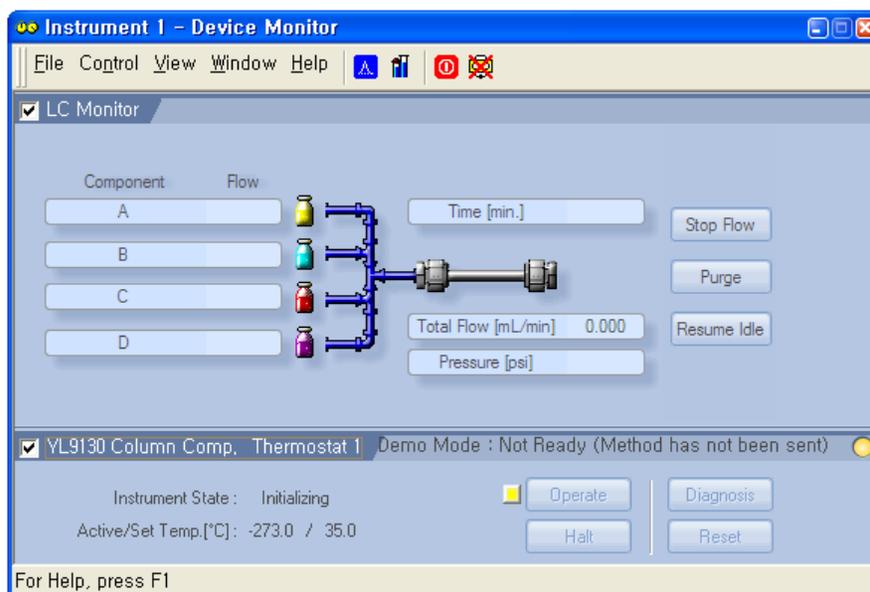


After select YL913x column compartment on the left window, click arrow button to move this on the right window. Click red arrow button on the right bottom side and select the temperature unit. Double click YL913x column compartment on the right window, and check IP address of pump. Click "Detect" button to check the connection.



3-2-2. Device Monitor

After configure the pump on the configuration window, log in to open main control window. On the main control window, click Device monitor and then Device Monitor window pops up as below. In this window, can control the column compartment and monitor instrument status as like temperature.

**[Control button]**

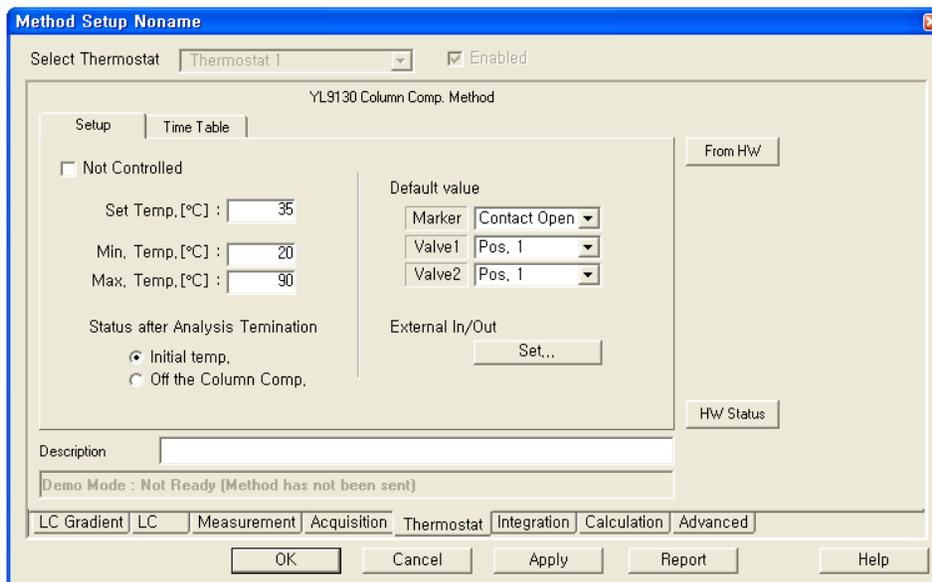
- Operate : To run the column compartment at set temperature.
- Halt : To stop the operation of column compartment.
- Diagnosis : To self test of instrument.
- Reset : To release the column compartment status from the error.

[Status message]

- Instrument State : To display the present status.
- Act/Set Temp : To set actual and set temperature.

3-2-3. Method setup

In this window, edit time program table, and setup the switching valve program.



Not Controlled

It can be checked if you do not want to use YL9131 column compartment for analysis. If you select “Not Controlled”, it is displayed on the device monitor window and all the function to control YL9131 Column Compartment are deactivated.

Set Temp.

Set the initial temperature of YL9131 Column compartment.

Min. Temp., Max Temp.

Set the minimum and maximum temperature. The initial temperature and temperature on the Time Table are limited by these temperatures. If the actual temperature is over this limitation, the error shows.

Status after Analysis Termination

Set the status of column compartment after finished analysis.

- Initial temp.
Return to initial temperature.
- Off the Column Comp.
Turn off the temperature control of Column compartment.

Default value

Set Initial Marker output and switching valve position.

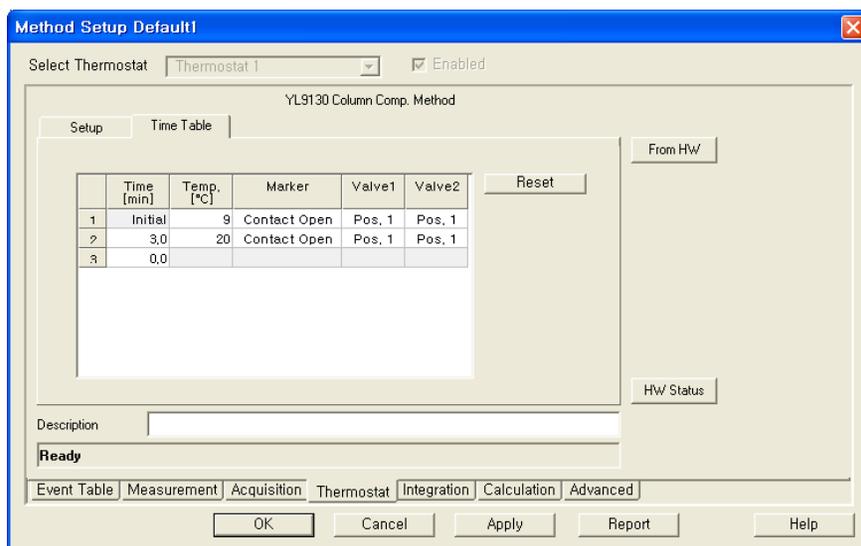
External In/Out

Set external input and output signal. If the signal is opposite when you use with the other device, change it on the External In/Out Set.



Pulse Width

If you select "Pulse" as a external in/out method, set the time of maintaining output signal. The setup range is 100 ~ 100000 mSec.



Time Table

It is a table to program temperature, Marker output, Valve position according to time. The temperature value is limited by limit temperature and consider the temperature unit(°C, °F, K) selected on the System Configuration window.

Reset

Delete the lines except first line. First line is same with initial temperature on the Setup Tab.

Chapter 4. Maintenance

YL9131 shows the error message, please refer to the following,

Error	Cause	How to fix
High Temp Limit!	Temperature is higher than Max. limit	Increase max. temperature limit Decrease operating temperature
Solvent Leak!	Solvent leaks	Remove the leaks on the leak sensor and find the leak position

The followings are the remedy for problem.

Problem	Cause	How to fix
Actual temp. is much higher than Setup temp.	Electronic problem	Ask to manufacturer
Temperature does not increase	Heater	
Instrument stops operation	1) Over heat 2) Fuse 3) Electronic problem	1) Turn off for 10minutes 2) Replace fuse 3) Ask to manufacturer

YL9100 HPLC SYSTEM

YL9150 AUTOSAMPLER

USER MANUAL



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Chapter 1. About this guide

This guide is written for laboratory technicians who use the Alias autosampler for execution of analytical runs.

This guide offers the following information:

- Chapter 2 describes the Alias autosampler and injection principles
- Chapter 3 describes maintenance procedures for the Alias autosampler
- Chapter 4 describes trouble shooting
- Chapter 5 describes installation and upgrading procedures for the Alias autosampler and Alias Service Manager software
- Appendix A lists specifications of the Alias autosampler
- Appendix B provides information on Control I/O
- Appendix C lists error messages
- Appendix D lists accessories and spare parts available for the Alias autosampler.

An index has been provided for easy reference.

Pictorials used in this manual

The following pictorials are used in this guide:



The danger sign warns about a hazard. It calls attention to a procedure or practice which, if not adhered to, could result in injury or loss of life.

Do not proceed beyond a danger sign until the indicated conditions are fully understood and met.



The warning sign denotes a hazard. It calls attention to a procedure or practice which, if not adhered to, could result in severe injury or damage or destruction of parts or all of the equipment. Do not proceed beyond a warning sign until the indicated conditions are fully understood and met.



The caution sign denotes a hazard. It calls attention to a procedure or practice which, if not adhered to, could result in damage or destruction of parts or all of the equipment. Do not proceed beyond a cautions sign until the indicated conditions are fully understood and met.



The attention sign signals relevant information. Read this information, as it might be helpful.



The note sign signals additional information. It provides advice or a suggestion that may support you in using the equipment.

Safety practices

The following safety practices will ensure safe operation of the autosampler and should only be executed by authorized personnel:



Removal of some panels exposes potentially dangerous voltages. Disconnect the instrument from all power sources before removing protective panels.

Replace blown fuses with size and rating indicated on the fuse panel or holder and as listed in the list of accessories and spares (appendix D) in this manual.

Replace or repair faulty or frayed insulation of power cords.

Check actual line voltage to confirm it is the value for which this instrument is wired. Make sure power cords are plugged into the correct voltage sources.



Perform periodic leak checks on supply lines.

Do not allow flammable and/or toxic solvents to accumulate. Follow a regulated, approved waste disposal program. Never dispose of such products through the municipal sewage system.

Chapter 2. Introduction

The Alias autosampler is a high throughput autosampler of robust design, developed to meet the challenge of the modern analytical laboratory. It is a very complete autosampler that needs little bench space; the Alias is designed for indoor use. The Alias autosampler features among other things:

- PASA™ **injection concept** (see "Injection principles").
- High-resolution syringe control; this ensures very high precision for injection and reagent addition.
- Internal standard addition, sample dilution or derivatization can simply be programmed.
- PC control ensures easy-to-understand operation; context-sensitive online help is available with every window and dialog.
- Special attention has been paid to ensure a service-friendly design.
- To enhance safety, speed of operation of the Alias will decrease when the door is opened.
- Optional sample cooling ensures consistent results.

Read this chapter to help identify parts of the Alias autosampler, and to learn more about injection principles.

2-1. Instrument description

The Alias is a complete autosampler that requires very little bench space. Standard high or low well plates or vial trays can be used. The sampling compartment of the Alias can house two different well plates. Any combination of well plates is allowed, except for 384 Low on the left and 96 High on the right.

The Alias autosampler is standard fitted with:

- 15 µL injection needle
- 500 µL syringe
- 1000 µL buffer tubing
- 100 µL sample loop.

All replaceable parts are easily accessible. Refer to the List of **accessories and spares** (see "List of accessories and spares") for more information.

To open the door, execute the following steps:

- 1 Get hold of the door handle.

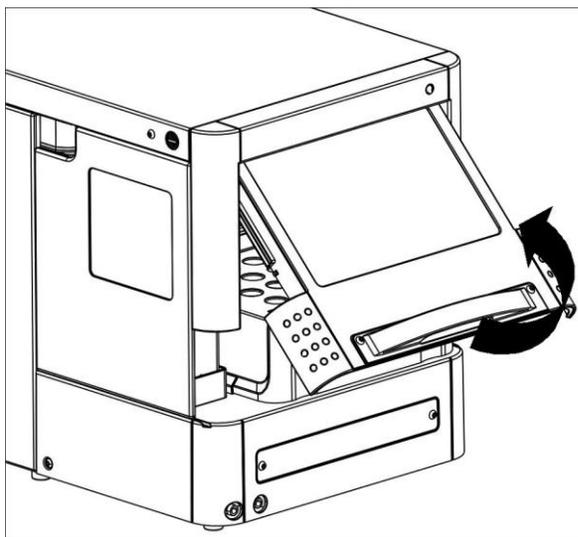


Figure 1: Open the door

- 2 Gently pull it towards you and push it upward until it is in horizontal position.

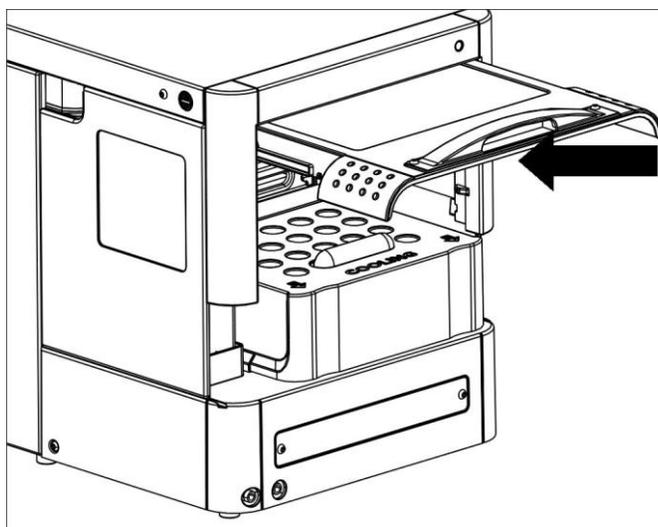


Figure 2: Push the door upward

- 3 Slide the door into the autosampler.

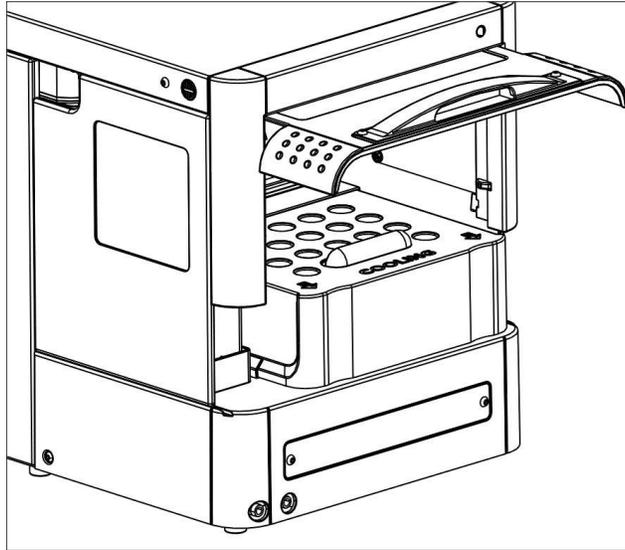


Figure 3: Slide the door into the autosampler

For easier access, you can remove the cover of the Alias:

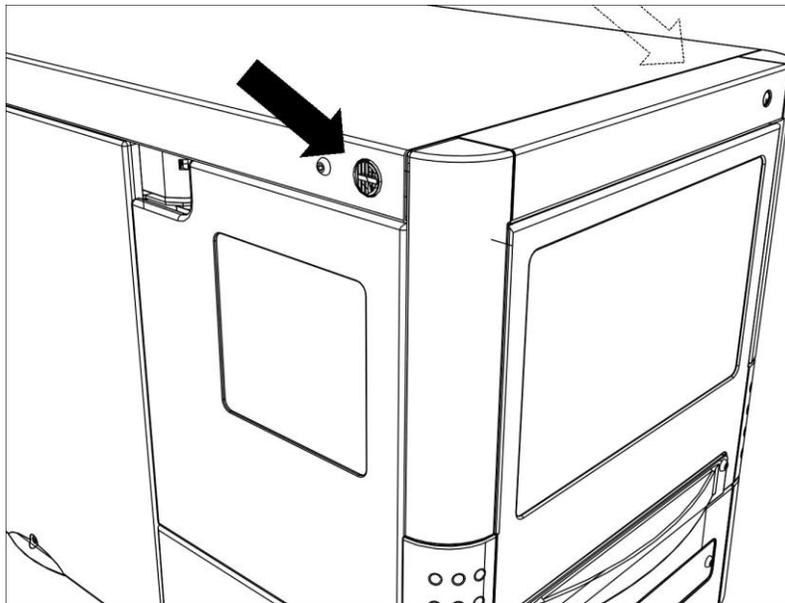


Figure 4: Location of Black push buttons

To remove the cover of the Alias:

- 1 Press the two black buttons on either side (top) of the autosampler simultaneously.
- 2 Gently pull the cover towards you.

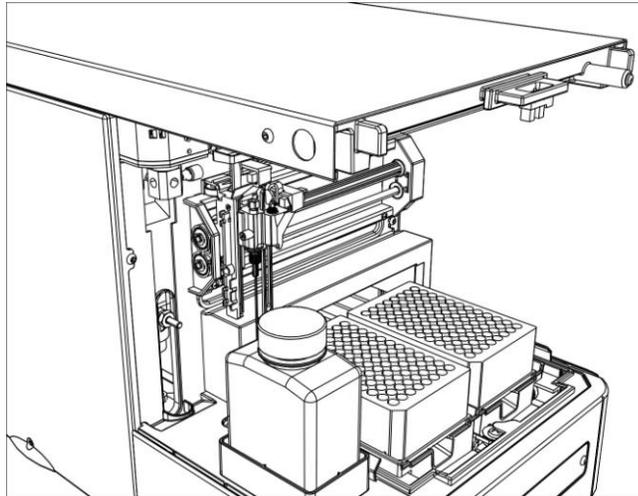


Figure 5: Alias without cover

If the cooling option is installed: slide out the cooling cover by pulling it gently towards you. You can now place well plates.

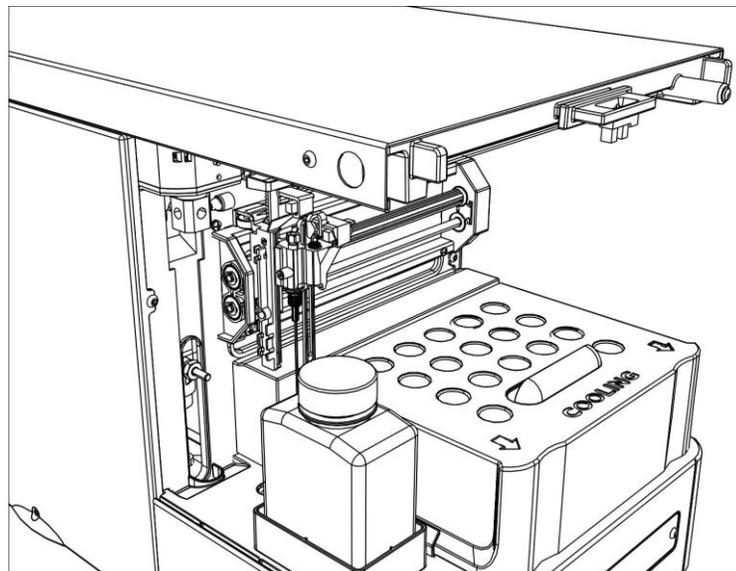


Figure 6: Alias with cooling cover

Alias autosampler - front

The Alias sampling compartment houses the following parts:

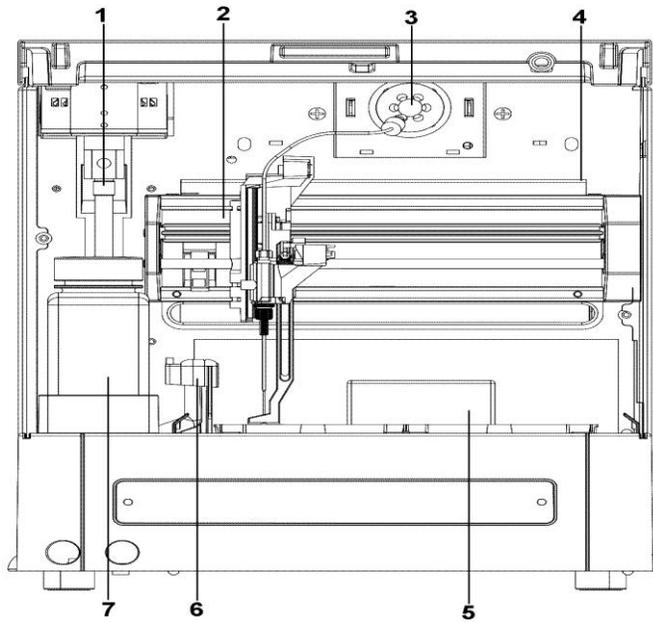


Figure 7: Alias sampling compartment

- 1 Syringe
- 2 Needle arm
- 3 Injection valve
- 4 Valve leak bin
- 5 Sample compartment
- 6 Needle wash position
- 7 Wash liquid bottle

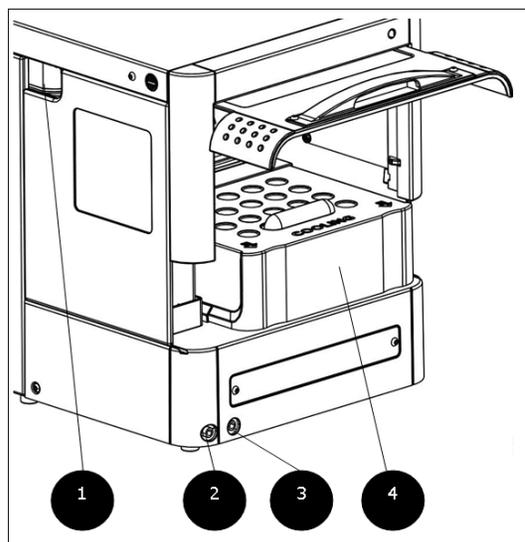


Figure 8: Alias with cooling

- 1 Tubing guide
- 2 Wash/waste
- 3 Condensed water/leakage
- 4 Cooling cover

Alias autosampler - back

The back of the autosampler has the following items:

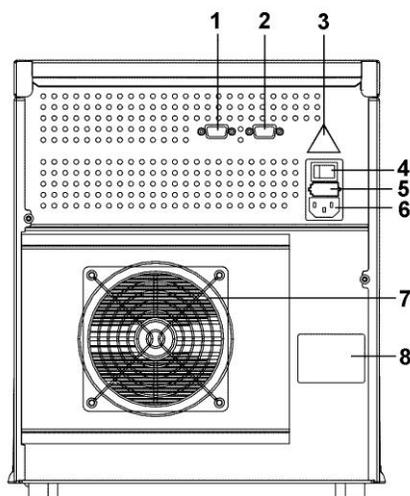


Figure 9: Back of Alias

- 1 9-pin male connector (inputs/output)

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- 2 9-pin female connector (serial interface)
- 3 **warning label** (see "Control I/O connections")
- 4 on/off switch
- 5 fusebox
- 6 power connector
- 7 cooling fan (if cooling option is installed; do not obstruct!)
- 8 type label

2-2. Options

The following **factory-installed** options are available for the Alias autosampler:

- Cooling: if installed, a cooling fan is visible at the back of the autosampler, and a cooling cover is installed inside the sampling compartment.
- Prep version: Alias suitable for large volume sampling. Because larger volumes must be injected for the **Prep mode** (see "Specifications Prep version"), Alias is fitted with a 2500 μL syringe and a 10000 μL sample loop.

The following **user-installable** options are available:

1) Bio-compatible sample flow path and valve	Inert sample needle (Silco steel) and bio-compatible valve (PEEK)
2) Prep Kit	2.5 mL syringe, Prep valve, 10 mL sample loop, LSV needle and sample tray for 10 mL vials
3) Air needles	6 different types of air needles are available for the Alias, each for a different type of well/vial plate. However, it is not just the type of well/vial plate that determines which air needle must be used. Refer to the section on Air needles (see "Air needles for Alias") for more information.
4) Valve unit	Special valve unit that can be replaced quickly and easily.

2-3. Injection principles

Three injection modes can be used:

- **Full loop injections:** for maximum precision
- **Partial loopfill injections :** for maximum flexibility
- **µL Pickup injections :** for zero sample loss.

These three injection modes accommodate use of a wide variety of applications.

For all injection modes loop injection with Pressure-Assisted Sample Aspiration (PASA™) is selectable.

It is a proven concept that combines high precision with simplicity and reliability:

- no moving around with the sample needle
- reduced risk for bubbles in the sample line
- no needle port that wears and contaminates.

There is only intelligent valve switching and highly accurate syringe control.

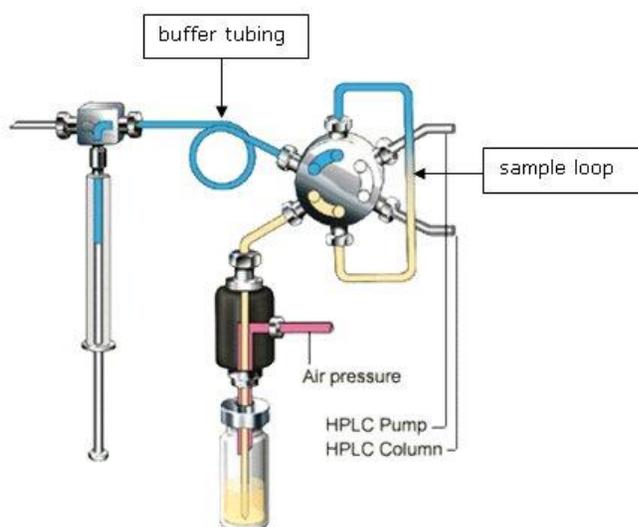


Figure 10: PASA injection concept

The syringe is used to aspirate the sample from a vial into the sample loop. Buffer tubing between the syringe and the injection valve prevents contamination of the syringe. Wash solvent is used:

- to remove the sample from the buffer tubing and sample needle
- to rinse the buffer tubing and sample needle.

2-3-1. Syringe and buffer tubing

Two sizes of syringes are available for the Alias: 500 and 2500 μL . The 2500 μL syringe is installed in the **Prep version** (see "Specifications Prep version") of the Alias. The 500 μL syringe is the standard syringe; combined with the standard 1000 μL buffer and the standard 100 μL sample loop, the following injection volume range is available for the various injection modes:

- Full loop : 100 μL
- Partial loopfill : 0 - 50 μL
- μL pick-up : 0 - 27 μL

The maximum injection volumes are calculated with the following formulas:

- Full loop : injection volume = loop volume
- Partial loopfill : max. inj. volume = $\frac{1}{2}$ x of loop volume
- μL Pick up : max. inj. volume = (loop volume - 3 x needle volume)/2

Full loop gives maximum possible reproducibility < 0.3%, but not maximum accuracy, since the loop volume is specified with an accuracy of $\pm 10\%$. Minimum sample loss = 230 μL (2 x loop overfill + flush

volume for 15 μL needle).

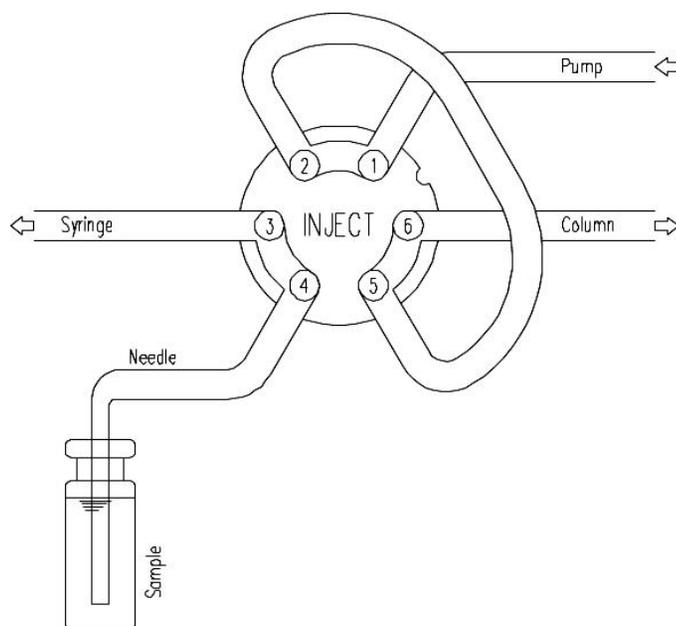
Partial loopfill gives maximum accuracy plus reproducibility better than 0.5% RSD for injection volumes > 10 μL . Minimum sample loss (Flush volume) = 30 μL . 30 μL is the recommended minimum flush volume, smaller flush volumes can be programmed, but will result in decreasing performance.

μL Pick-up offers no sample loss, maximum accuracy (same as partial loopfill), but slightly lower reproducibility: RSD better than 1% for injection volumes > 10 μL .

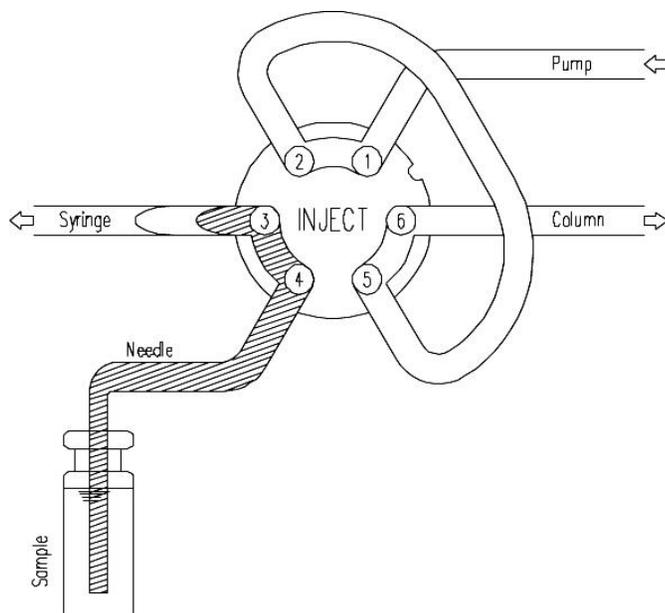
2-3-2. Full loop injections

The sample loop is completely filled (quantitatively) with sample. This type of injection results in extremely good reproducibility.

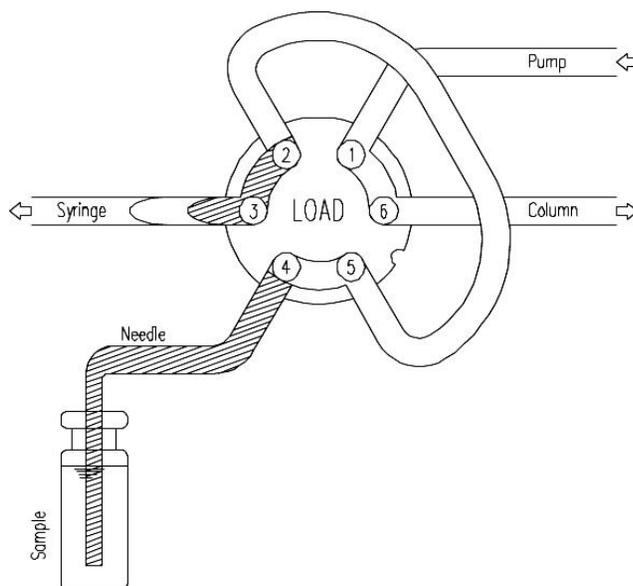
- 1 The initial situation: the injection valve is in INJECT position. The sample needle with air needle has entered the well or vial. Headspace pressure, applied through the air needle, ensures that no air or vapor bubbles are formed during sample aspiration.



- 2 The syringe dispenser aspirates the "flush volume" from the sample well/vial to fill the sample line with sample and remove wash solvent.



- 3 The injection valve is switched to LOAD position, placing a distinct sample plug at the inlet of the sample loop.

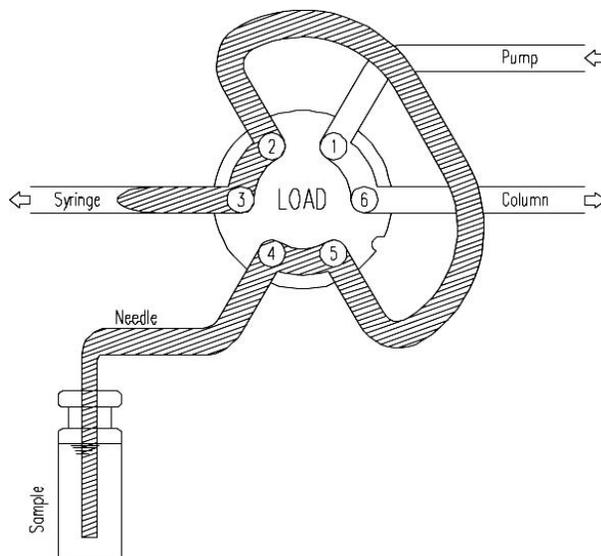


- 4 The sample loop is quantitatively filled by transporting a number of times the loop volume through the loop, depending on the volume of the loop.

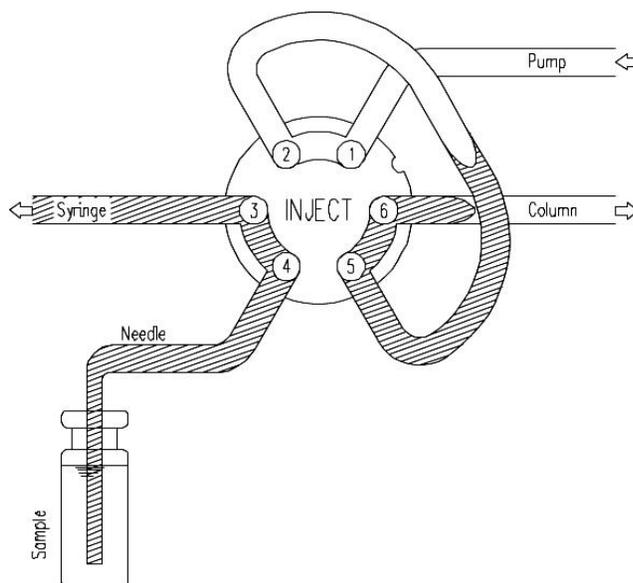
$$3 \times \text{loop volume for loop} \leq 100 \mu\text{L}$$

2 x loop volume for loops 100 μ L - 500 μ L

1.5 x loop volume for loop > 500 μ L



- 5 The injection valve switches to the INJECT position. The sample loop is not part of the HPLC mobile phase flow path: sample is transported to the column. The analysis starts.

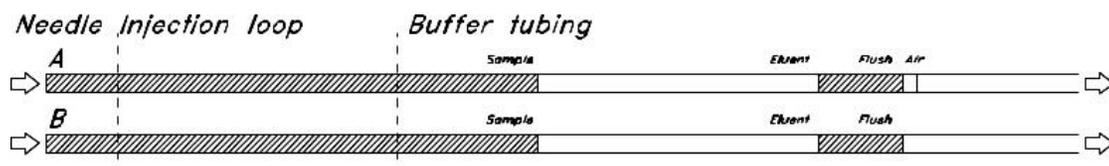


A wash routine is performed after each injection.

Air segment with Full loop injections

An air segment of 5 μ L can be used to reduce the amount of flush volume. This air segment is at the front of the flush volume and will not be injected.

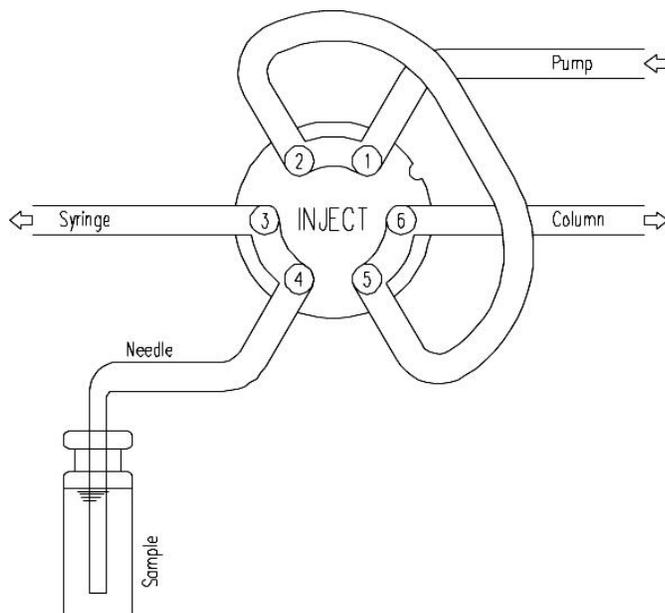
With a standard needle, the flush volumes must be a minimum of 30 μL for injections with air segment, and 35 μL for injections without air segment. If samples are highly viscous it may be necessary to program larger flush volumes and reduce the syringe speed for better performance.



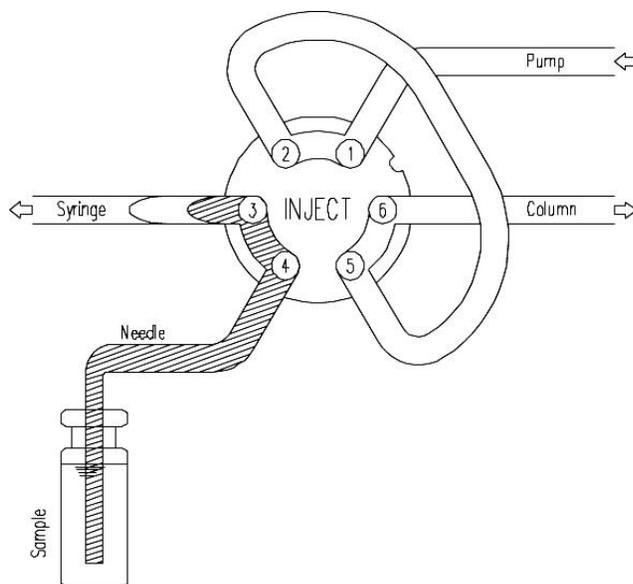
2-3-3. Partial loopfill injections

The switching sequence for a partial loopfill injection is:

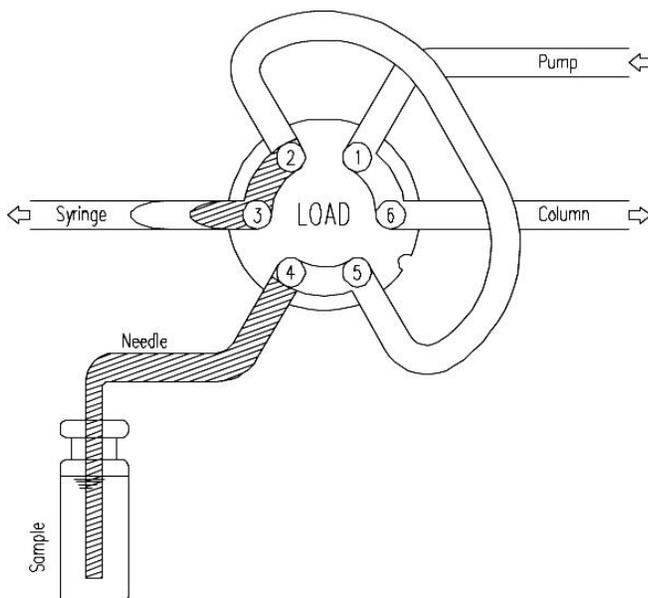
- 1 The initial situation: the injection valve is in the INJECT position. The sample needle with air needle has entered the vial/well. Headspace pressure, applied through the outer air needle, ensures that no air or vapor bubbles are formed during sample aspiration.



- The syringe dispenser aspirates the "flush volume" from the sample vial to fill the sample line with sample and remove wash solvent.

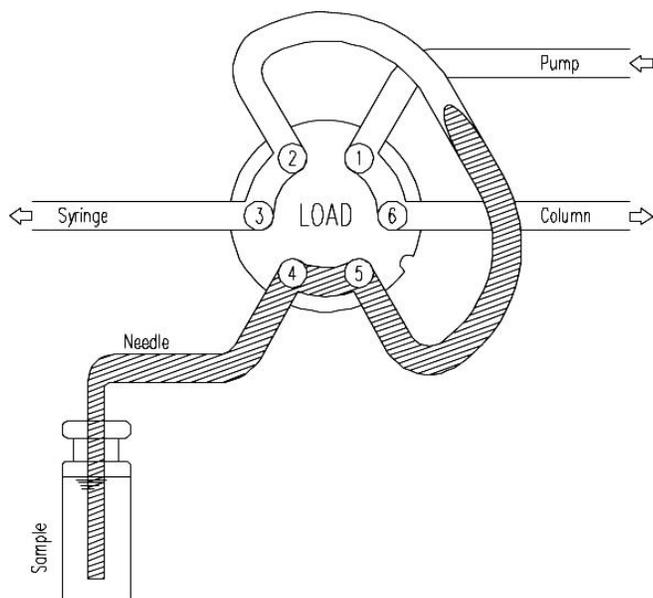


- The injection valve switches to LOAD, placing a distinct sample plug at the beginning of the sample loop.

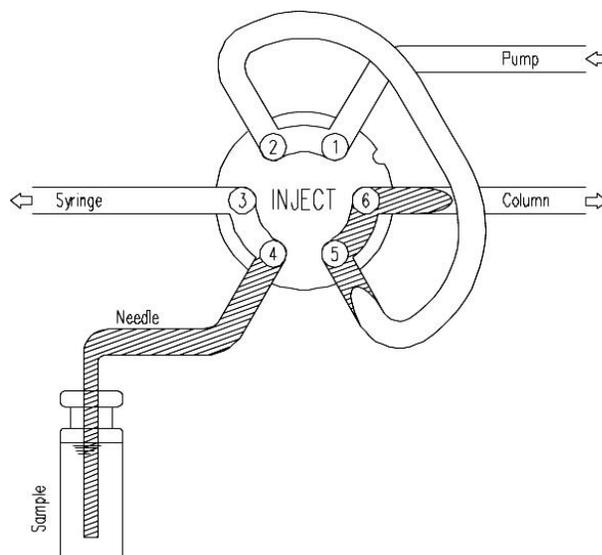


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- 4 The programmed injection volume is now aspirated into the sample loop.



- 5 The injection valve switches to INJECT. The sample loop is now part of the HPLC mobile phase flow path: sample is transported to the column. The analysis starts.

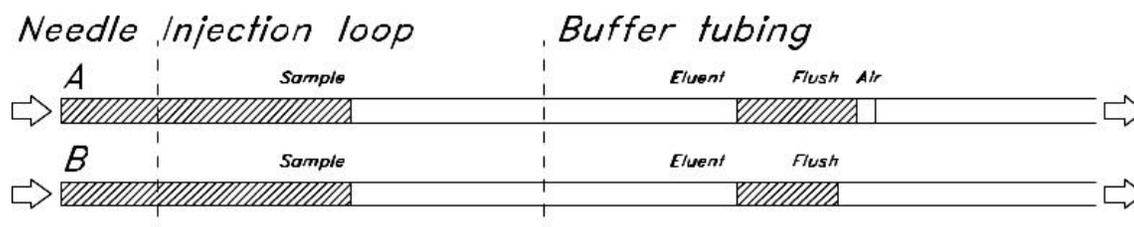


If an injection from the same vial and no wash routine is programmed, the next injection sequence will start with a flush of 50% of the programmed flush volume. Otherwise, it will start with a flush of the programmed flush volume. If the withdrawal of sample for the next injection exceeds the total volume of the sample buffer tubing, the buffer tubing is rinsed before the next injection. The next injection will start with the programmed flush.

Air segment with Partial loopfill injections

An air segment can be used to reduce the amount of flush volume. The air segment is at the front of the flush volume and will not be injected.

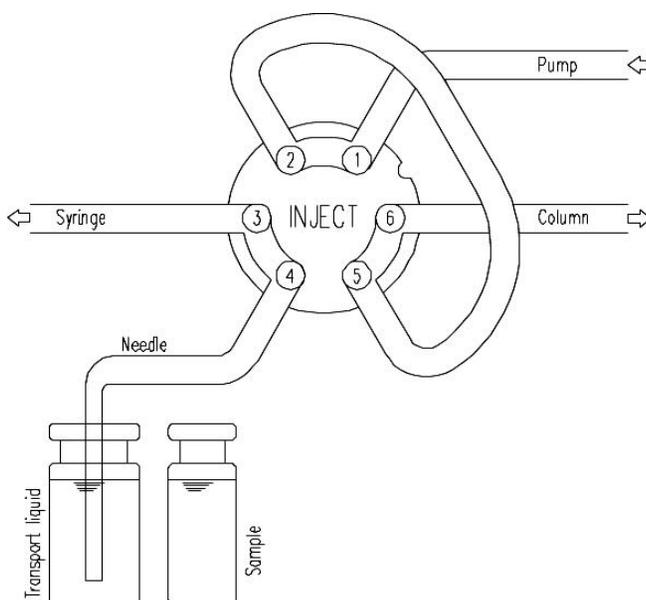
With a standard needle, the flush volumes must be a minimum of 30 μL for injections with air segment and 35 μL for injections without air segment. If the samples are highly viscous, it may be necessary to program larger flush volumes and reduce the syringe speed for better performance.



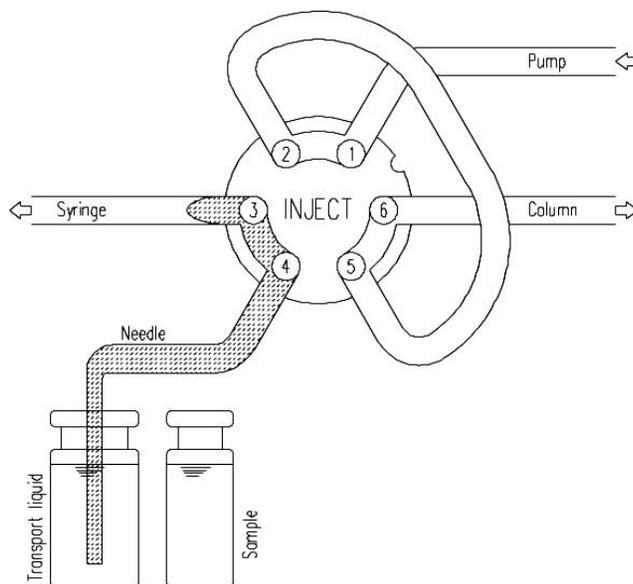
2-3-4. μL Pickup injections

The switching sequence for μL pickup injections is:

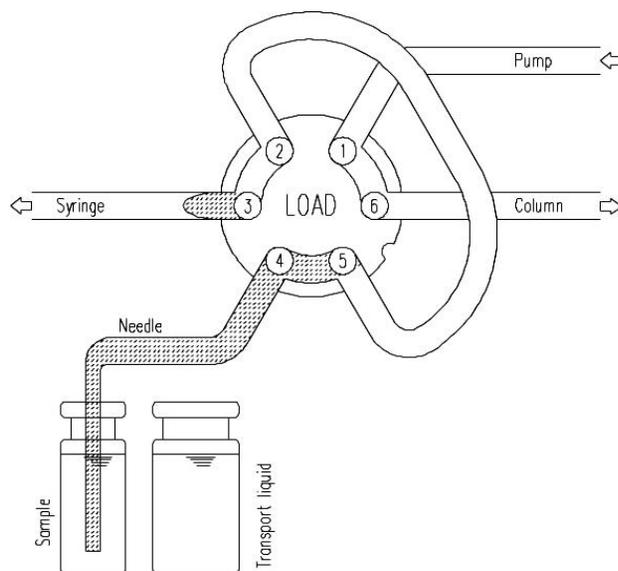
- 1 In the initial situation, the injection valve is in INJECT position. The sample needle has entered the transport position.



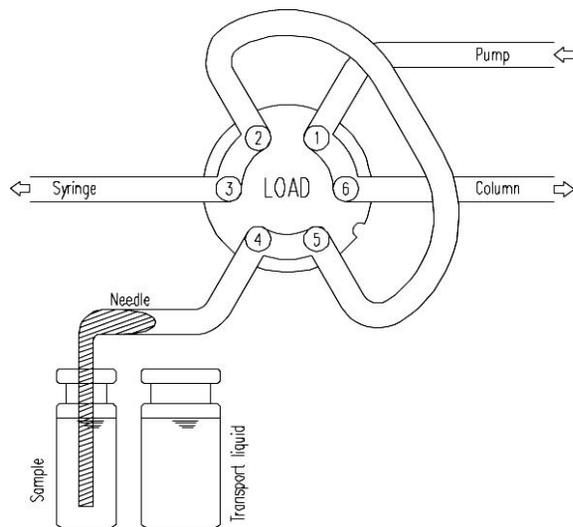
- 2 For the first injection (after a wash or after emptying of the buffer tubing), the syringe dispenser aspirates a transport plug from the transport position to fill the sample line with transport liquid and remove wash solvents.



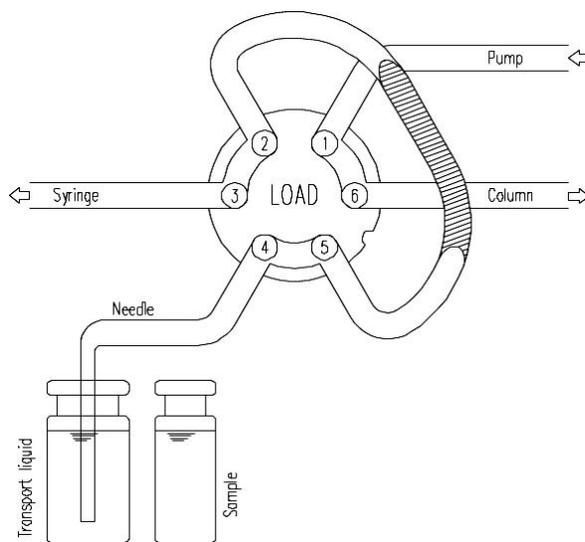
- 3 The needle moves from the transport position to the sample vial. The injection valve switches to LOAD position.



- 4 The programmed injection volume is aspirated from the sample vial.

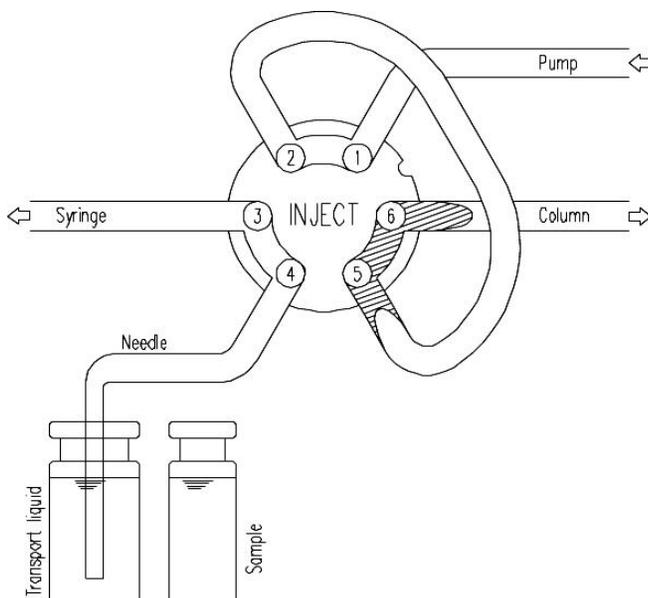


- 5 The sample needle moves back to the transport position. A second transport plug is aspirated. The sample is quantitatively transported into the loop.



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6 The injection valve switches to INJECT. The sample loop is now part of the HPLC mobile phase flow path: sample is transported to the column. The analysis timer starts.



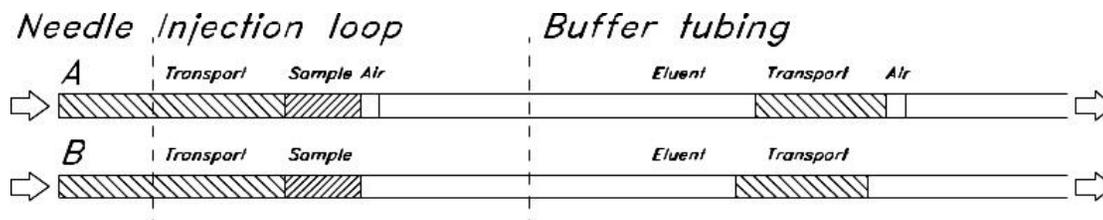
The sequence is repeated for each injection.

Air segment with μL Pickup injections

If an air segment has been programmed, it appears at the front of the first plug of transport liquid and at the front of every sample plug.

In this injection mode:

- the air segment at the front of the sample plug is injected into the HPLC system
- no headspace pressure can be applied on vials/wells in this mode to avoid sample errors due to air expansion during exchange from the sample vial/well to the transport position.



2-4. Mix & dilute

A Mix & Dilute routine can be created for the Alias. This routine allows you to process the sample before injection. You can program three different types of actions:

- **Add** the indicated volume from the Sample/Reagent A/Reagent B/Wash position and dispense it to the Sample/Destination position.



To prevent cross-contamination, the Alias will aspirate an additional volume of 25% of the programmed volume to flush the tubing and needle.

The aspirate and dispense speed depends on the selected syringe and the programmed syringe speed.

Example: ADD 200 uL from Reagent A to Destination will result in the following actions:

- 1 Aspirate an air segment of 5 μ L to separate the wash solvent in the buffer tubing from Reagent A.
- 2 Aspirate 50 μ L Reagent A to flush the tubing and needle.
- 3 Empty the syringe to the syringe-waste position.
- 4 Aspirate 200 μ L Reagent A and dispense it to the destination vial.

Rinse buffer tubing and needle with wash solvent.

- **Mix** (aspirate and dispense) a number of the times the programmed volume from the destination vial. If no destination vial is available, the mix is performed in the sample vial.

Example: Mix 3 times with 250 μ L will result in the following actions:

- 1 Aspirate an air segment of 50 μ L to separate the wash solvent in the buffer tubing from the solvent to be mixed.
- 2 Empty the syringe to the syringe-waste position.
- 3 Aspirate 250 μ L solvent and dispense it back into the vial/well.
- 4 Repeat step number 3 twice.
- 5 Rinse buffer tubing and needle with wash solvent.

The mix is performed from the destination position when the previous ADD action is TO DESTINATION. When the previous ADD action is TO SAMPLE, the mix is performed from the sample position.

- **Wait** the programmed period of time before continuing with the next step (reaction time).
A maximum of 15 steps can be created for the Mix & Dilute routine.

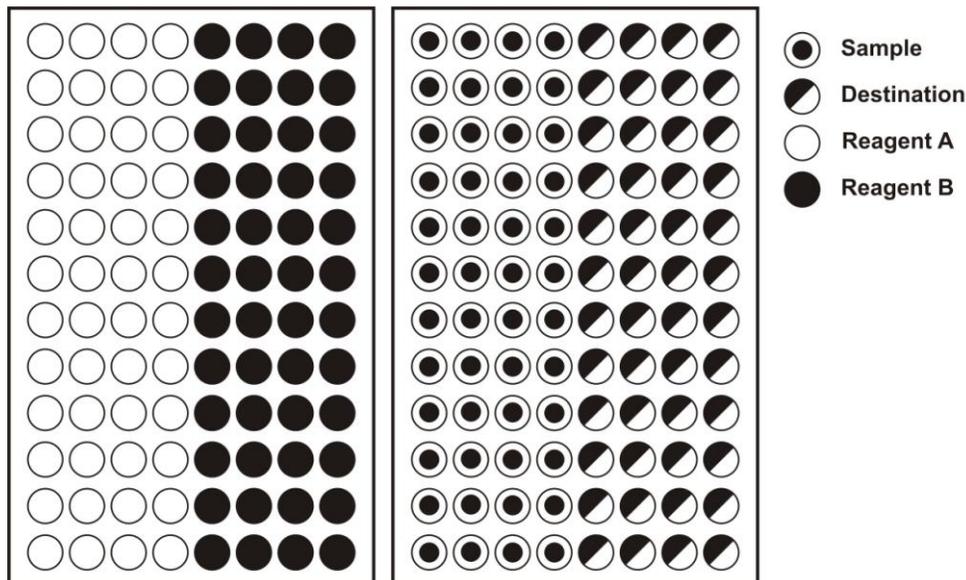
2-4-1. Sample positions Mix & Dilute



If a mix is programmed, the reagent solvents, destination and sample positions in the trays are as follows:

- 1) Reagent A: Left plate
- 2) Reagent B: Left plate
- 3) Samples: Right plate
- 4) Destination: Right plate

If you chose to process plates in rows, the following positions are available for sample, destination, reagent A and reagent B:



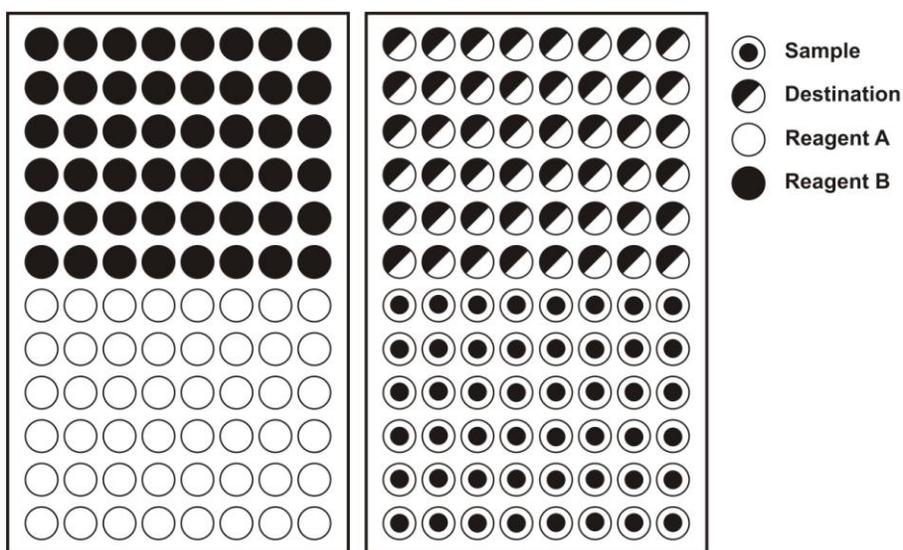
For example, you can program the following:

- two 96-low plates, processed **in rows**
- first line: first sample position 2A1, last sample position 2A1, destination position 2E1, Reagent A 1A1 and Reagent B 1E1
- second line: first sample position 2A2, last sample position 2A2, destination position 2E2, Reagent A 1A2 and Reagent B 1E2
- third line: first sample position 2A3, last sample position 2A3, destination position 2E3, Reagent A 1A3, Reagent B 1E3.

However, if you were to use a 12-vial tray for the Reagent, you could use reagent from the same vial a number of times. For example:

- first line: first sample position 2A1, last sample position 2A1, destination position 2E1, Reagent A 1A1 and Reagent B 1E1
- second line: first sample position 2A2, last sample position 2A2, destination position 2E2, Reagent A 1A1 and Reagent B 1E1
- third line: first sample position 2A3, last sample position 2A3, destination position 2E3, Reagent A 1A1, Reagent B 1E1, etc.

If you chose to process plates in columns, the following positions are available for sample, destination, reagent A and reagent B:



For example, you can now program the following:

- two 96-low plates, processed in **columns**
- first line: first sample position 2A1, last sample position 2A1, destination position 2A7, Reagent A 1A1 and Reagent B 1A7
- second line: first sample position 2B1, last sample position 2B1, destination position 2B7, Reagent A 1B1 and Reagent B 1B7
- third line: first sample position 2C1, last sample position 2C1, destination position 2C7, Reagent A 1C1, Reagent B 1C7, etc.

The mix method can be executed before the injection method.

- The injection is performed from the destination position when a mix method is programmed and TO DESTINATION is the last step in the mix method.
- The injection is performed from the sample position when a mix method is programmed and TO SAMPLE is the last step in the mix method.

The mix method can also be executed without an injection method.

Chapter 3. Maintenance

For all maintenance procedures:

- 1 Open the door of the Alias.
- 2 If the cooling option is installed: remove the cooling cover by sliding it towards you.
- 3 Press the two buttons at the top sides of the Alias simultaneously.
- 4 Remove the cover by pulling it towards you.



You need not disconnect the Alias from the power source for any of the maintenance procedures. In this way software control will still be possible. Use the Direct Control function in the Alias Service Manager (ASM) software to check operation of the various parts of the autosampler.

3-1. Cleaning

In general, the Alias autosampler needs very little maintenance. You can clean the outside with a damp cloth with non-aggressive cleaning liquid. Other items that may need periodic cleaning:

- **valve leak bin** (see "Alias autosampler - front" on page 7): a special leak bin is installed underneath the injection valve. You can clean this bin with a damp cloth with non-aggressive cleaning liquid.
- sample tray: if sample has been spilled on the sample tray, clean the tray with a damp cloth with non-aggressive cleaning liquid.
- drain tubing: regularly flush the drain tubing with solvent to prevent clogging and to ensure that liquids and condensate are disposed of.

3-2. Injection valve and rotor seal

The Alias is equipped with an injection valve, either with quick-connect mounting, or with fixed mounting.

Execute the following steps to remove the injection valve:

- 1 Disconnect all tubing from the valve. Only the sample loop can stay in place.

- 2 Remove the valve.
- 3 Remove the screws from the stator part of the valve.
- 4 Gently open the valve and take out the rotor seal. Clean and/or replace the seal.
- 5 Place the stator back on the rotor and fasten the screws.
- 6 Hold the valve for mounting with port 1 pointing upward.
- 7 Place the valve into its slot and fasten it.
- 8 Reconnect all tubing to the valve.
- 9 In Direct control, click **Initialize** to make sure that the valve is in Inject position.
- 10 Perform a standard wash (Direct control - Initial wash group box).

The Alias is now ready for use.

3-3. Sample loop

The Alias is standard fitted with a 100 µL sample loop. A different sample loop size can be installed, but note that you will need the proper combination of **syringe and tubing** (see "Syringe and buffer tubing") to ensure good results.

Take the following into account when you have installed a sample loop:

- Connect the loop between ports 2 and 5 of the injection valve
- Go to the configuration settings and adapt settings in the flowpath group box if you have installed a loop with a different volume.



Remember that the maximum injection volumes are calculated with the following formulas:

full loop: injection volume = loop volume

partial loopfill: maximum injection volume = 50% of the loop volume

µL pickup: maximum injection volume = (loop volume - 3x needle volume)/2

3-4. Replacing the sample needle

Execute the following steps to replace the sample needle:

- 1 Open Direct control (Alias Service Manager).
- 2 Click **Exchange** in the Needle group box. The needle moves to exchange position.
- 3 Loosen the needle connection nut (number 3).
- 4 Loosen the nut (number 1) that connects the tubing (number 2) to port 4 of the injection valve.
- 5 Remove the sample needle by pulling it out of its fitting by the tubing.
- 6 Install a new needle assembly; make sure that the air seal is around the needle.
- 7 Tighten the needle assembly with the needle connection nut.
- 8 Connect the other end of the needle connection tubing to port 4 of the injection valve. Do not tighten too much as this may block the tubing.
- 9 Click **Initialize** in Direct control. The sample needle moves back to home position.
- 10 Perform a wash routine to clean the new needle by clicking **Start** in the Initial wash group box of Direct Control. Click **Stop** to end the wash routine.
- 11 Use the Alias/Adjustments option to adapt Needle - Tray settings.



If you use trays with 12 vials or 48 vials, make sure that the needle height settings is > 2mm to prevent the needle from touching the bottom of the vials.

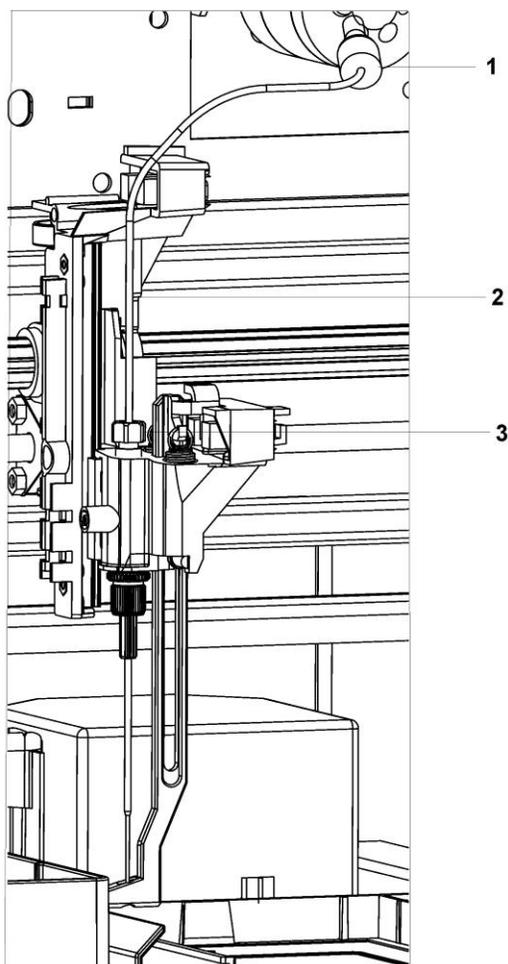


Figure 11: Replacing the sample needle

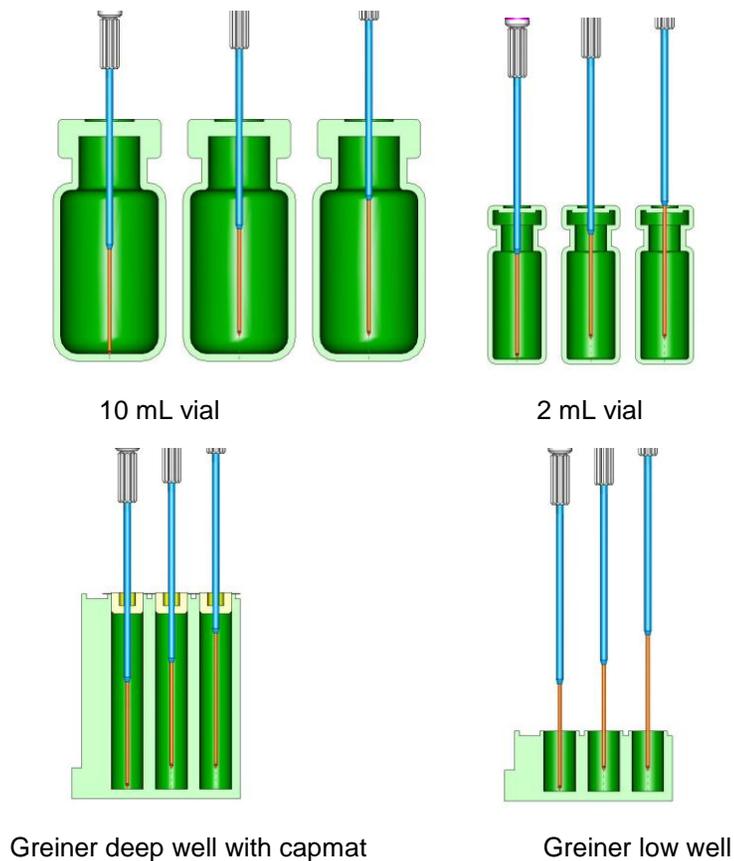
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3-5. Air needles for Alias

Six types of air needles are available for the Alias autosampler, all different in length (difference of 6 mm). These air needles are required to accommodate use of different plate heights in the Alias. For every well/vial plate the correct air needle is available. Apart from the 6 mm difference in length between the air needle types, the needle holder allows for an extra 6 mm variation in needle height.

Standard air Needle

The standard air needle is a 62 mm needle (no. 0045.505). This air needle accommodates use of a wide range of high and low plates. See the illustrations below for the puncturing depth of the needle:



Note that no PASA™ should be used for low wells: as the sample needle sufficiently punctures the seal to prevent vacuum, the function of the air needle will be insignificant for the low well plates.

If the 10 mL vials are used, the air needle is lowered pretty far into the vial. If the vial is not filled for more than 60%, the air needle can be applied as usual. The same applies for the deep wells.

If you need to deviate from these standard settings, use one of the optional needle types.

Which air needle for which titre plate or vial

To determine which air needle to use, the following dimensions need to be considered:

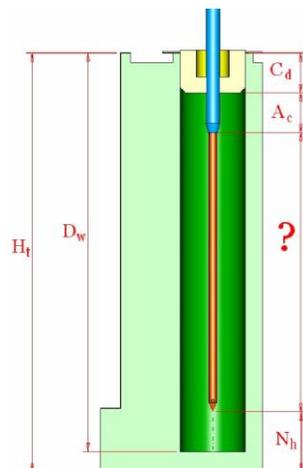
- The height of the titre plate in mm: H_t
- Well depth in mm: D_w
- Thickness of capmat or seal in mm: C_d
- Set needle height in mm: N_h
- Distance air needle point through the capmat or seal in mm, min. 2 mm: A_c

The following must be true:

$H_t - D_w$ must be between 2 and 6 mm

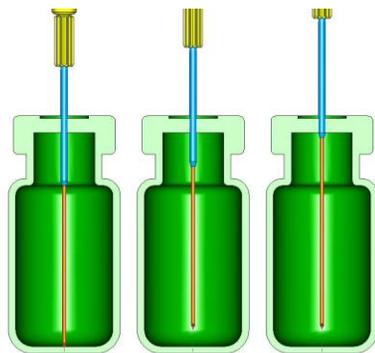
If this is true, the protrusion length of the sample needle can be calculated; this is the distance between the point of the sample needle and the point of the air needle. It can be calculated as follows:

$$\text{Protrusion length} = H_t - C_d - N_h - A_c$$

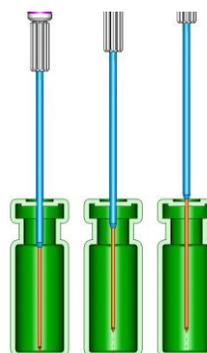


You can select the most suitable air needle on the basis of the protrusion length:

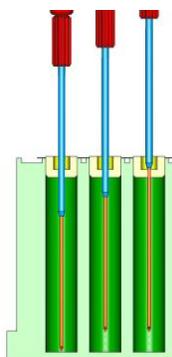
Air needle type	Protrusion length	
	from	to
50 mm, yellow	34	40
56 mm, red	28	34
62 mm, white (standard needle)	22	28
68 mm, blue	16	22
74 mm, green	10	16
80 mm black	4	10



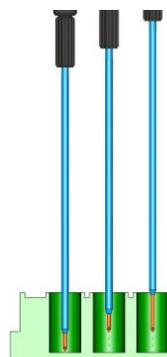
10 mL vial - 50 mm air needle



2 mL vial - 62 mm air needle



Greiner deep well M53000,



Greiner low well - 80 mm air needle

with capmat - 56 mm air needle

Example

You have a Greiner deep well with Micronic capmat M53000; the Alias has a standard needle height setting. Calculations will be as follows:

Ht = 41.4 mm	The following is true:
Dw = 37.8 mm	$41.4 - 37.8 = 3.6$ (is between 2 and 6 mm)
Cd = 3.8 mm	Protrusion length = $41.4 - 3.8 - 6.0 - 2.0 = 29.6$
Nh = 6.0 mm (standard)	
Ac = 2.0 mm (minimum)	

An air needle of 56 mm is required.

3-5-1. Air needle replacement

Execute the following steps to replace the air needle:

- 1 **Remove** (see "Replacing the sample needle") the sample needle.
- 2 Unscrew the chrome locking nut to remove the air needle.
- 3 Unscrew the chrome locking nut from the adjustment nut.
- 4 Get the new air needle.
- 5 Screw the height adjustment nut to the chrome locking nut (thread of the height adjustment nut must be level with the lower part of the locking nut). Make sure the O-ring seal is in the locking nut.
- 6 Install the air needle.
- 7 Install the sample needle.
- 8 Program the proper needle height for the new needle in the ASM settings window. Go to Adjustments to adapt Needle - Tray settings, if necessary.



If you use trays with 12 vials or 48 vials, make sure that the needle height settings is > 2mm to prevent the needle from touching the bottom of the vials.

- 9 Do an initial wash from Direct control to rinse the needle.

3-6. Syringe dispenser

The Alias is standard supplied with a 500 μ L syringe, but a 2500 μ L syringe can also be installed for the Prep version.

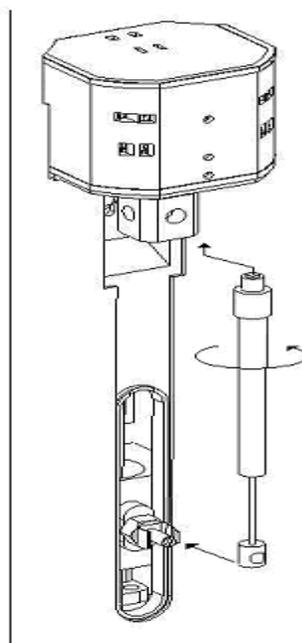


Figure 12: Syringe

Execute the following steps to install a different syringe:

- 1 In Direct Control, click **Exchange** in the Syringe group box.
 - 2 Unscrew the syringe from syringe valve, but make sure that the connector in the valve remains in place.
 - 3 Disconnect the plunger from the syringe drive.
 - 4 Fill the new syringe with wash solvent, preferably IPA (isopropanol). Make sure that most air bubbles are removed from the syringe.
 - 5 Connect the plunger of the filled syringe to the syringe drive and connect the syringe with the connector at the syringe valve.
 - 6 Screw the syringe firmly into the connector.
 - 7 In Direct control, click **Home** in Syringe group box. The syringe moves to home position and its content will be dispensed to syringe waste.
 - 8 If there is still some air in the syringe, click **End** again in Direct control. The syringe is filled with wash solvent. Use IPA.
 - 9 Click **Home** again to dispense the wash solvent to waste.
- If there is still air in the syringe, repeat steps 8 and 9 and gently tap the syringe as the wash solvent is

dispensed to syringe waste.

Perform a standard wash routine (Direct control: click **Start/Stop** in de Initial wash group box). All tubing connected to the syringe valve will be refilled and flushed.

3-7. Syringe plunger & plunger tip

Execute the following steps to replace the plunger or plunger tip:

- 1 Direct control, click **Exchange** in the Syringe group box.
- 2 Remove the syringe.
- 3 Slide the plunger out of the glass part of the syringe.
- 4 With pliers: remove the tip.
- 5 Dampen the new tip with for example isopropanol.
- 6 Mount the new tip on the plunger.
- 7 Insert the plunger in the glass part the syringe.
- 8 Install the syringe in the autosampler again.

3-8. Replacing the Syringe dispenser valve

The syringe valve is a 4-port selection valve. Ports are assigned as follows:

Waste	Use this port as a drain for the syringe dispenser.
Wash	Use this port to aspirate wash liquid from the wash bottle (or in case of multiple wash liquids: connect it to the solvent selection valve)
Needle	Connect the buffer tubing to this port

All connections to the syringe valve must be made using fingertight fittings. An exception can be made for the waste outlet (the port on the rear of the valve).

Execute the following steps to replace the syringe dispenser valve:



Place the syringe valve in waste position before you replace the syringe valve. In this position, the mounting screws are opposite/in line with the holes.

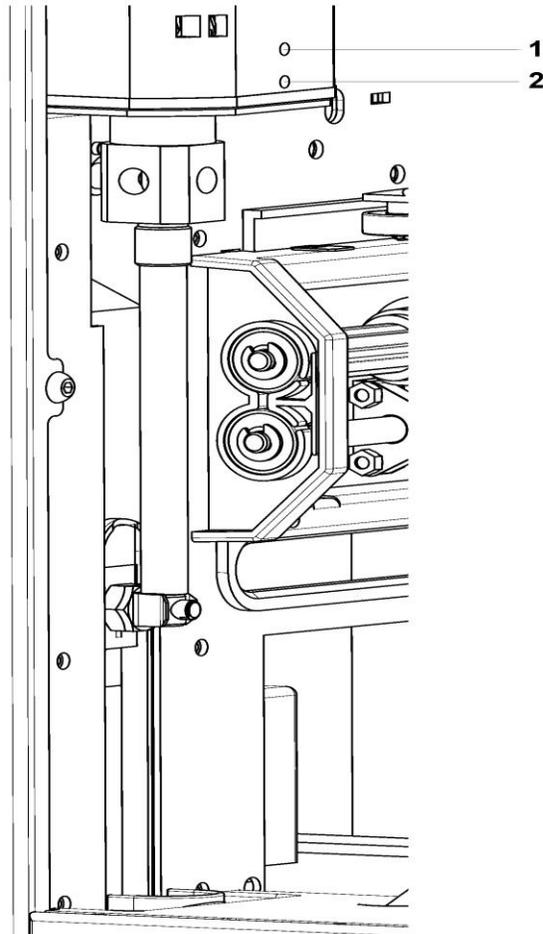


Figure 13 : Replacing the syringe dispenser valve

- 1 Move the syringe to exchange position (use the Exchange button in Direct control).
- 2 Loosen the lower socket-head screw (number 2).
- 3 Loosen the upper socket head screw (number 1).
- 4 Remove the syringe: pull out the top of the syringe first.
- 5 Remove the syringe valve and install a new one.
- 6 Install the new syringe.
- 7 Fasten the two socket-head screws again (fingertight + 1/4 turn).

3-9. Fuses

Fuses of the following types are installed in the Alias:

2 x 2.5A



Disconnect the Alias from its power source if you need to replace fuses.

If you need to replace the fuses, make sure that you install fuses of the same type and rating.

Fuses are in the fusebox at the back of the autosampler.



Contact Service if problems with fuses are recurring.

Chapter 4. Trouble shooting

Even though great care was taken in the design of the Alias, problems may occur:

- **instrument errors**: these can be caused by a variety of reasons.
- **software errors**: usually caused by faulty communication between instruments, or by faulty installation of the software.
- **analytical problems** (see "Analytical trouble shooting"): these may occur e.g. as a result of wear of parts, errors in injection settings and methods, or a wrong combination of sample loop, buffer tubing and syringe.

Alias Service Manager contains a Service option (select Alias/Service). Note that an access code is required for this option, and that the service option is intended for service engineers only.

Contact your supplier if a problem occurs that you cannot solve.

4-1. Instrument errors

Incidental fault conditions may occur in any instrument. The Alias will generate an instrument error message with an error number, a short description of the error and instructions on how to proceed.

In most cases, you will be asked to either initialize the system, or to switch the system off and then on again. Always click **OK** and follow the instructions to resolve the error status. Use Alias/Direct control in Alias Service Manager to monitor the error. Initialize the system in the Alias/Direct control window.



Make sure Alias is connected to a grounded power source.

If the LED is not lighted, a fuse may have blown.

Checking a valve implies that you remove the valve and check all parts for wear and dirt. Execute the following steps after any problem with a valve has been resolved:

- 1 Select Alias/Direct control. The Direct control window appears.
- 2 Click **Initialize**.
- 3 In the Initial wash group box, click **Start** to start the wash.
- 4 Click **Stop** to end the wash.
- 5 Click **Close** to exit the Direct control window.

Execute the following steps if you are asked to initialize the system:

- 1 Select Alias/Direct control. The Direct control window appears. From this window you can control separate parts of the autosampler to check whether they function as intended.
- 2 Click **Initialize** to reset the system and prepare it for normal use.

Execute the following steps if you are asked to switch the system off, and then on again:

- 1 Check that the communication cable between Alias and PC is properly installed.
- 2 Turn the instrument off with the on/off switch at the back of the autosampler.
- 3 Turn the system on again with the on/off switch. The system is initialized and is now ready for use.

4-2. Software errors

Software errors usually are caused by faulty installation of the software, or by faulty communication between instruments; you will be asked to re-install the software on the PC that controls the system.

If a software error message appears, first check if it may be caused by faulty communication between instruments:

- 1 Check all cable connections between instruments.
- 2 Open Alias Service Manager.
- 3 Select Alias/Direct Control.
- 4 Click **Initialize**.

4-3. Analytical trouble shooting

Analytical problems like bad reproducibility or carry-over may occur in any HPLC system. It may be hard to find the cause; you may have to try out several procedures. The first thing to do is to determine whether the problem is caused by the autosampler or by the rest of the system:

- 1 Replace the valve by a manual injection valve to discriminate between valve problems and other problems.
- 2 Do a number of Full loop injections. If the results are fine, the fault is in the autosampler; if not, check the rest of the HPLC system.

Please bear in mind that analytical problems may also be caused by external influences like temperature or light-sensitive samples. Make sure that the application was running trouble-free before and that no changes have been made to the system.

A number of causes and possible solutions for analytical problems is listed below. Contact service if you need further help.

If **reproducibility** is not according to specifications, check the following possible causes:

Causes:	Solutions:
Air in flow path.	Do an initial wash (select Alias/Direct Control in Alias Service Manager)
Leaking syringe.	If leakage occurs at the top of the syringe, check whether it has been properly mounted. If leakage occurs at the bottom of the syringe, replace plunger tip or syringe.
Leaking syringe valve.	Check or replace valve.
Rotor seal worn out.	Replace seal. Check stator.
Dead volumes in tubing connections.	Redo connections with new ferrules and nuts.

If a **blank gives a peak that is too high** for your criteria:.

Causes:	Solutions:
Solubility problem.	You can either modify your sample, or accept carry-over.

<p>Bad match between sample characteristics and hardware.</p>	<p>Check hardware:</p> <p>Needle: either use an extra wash (to wash the inside and outside needle), or install a different type of needle (Steel or Silica-coated)</p> <p>Valve: replace rotor in valve by Valco E or H type.</p> <p>Tubing: install different tubing (Steel, Peek) between autosampler and column, or use different wash solvents</p>
<p>The blank you use has been soiled.</p>	<p>Use a new blank.</p>
<p>Cause not clear.</p>	<p>Check if you can solve the problem by using more variation in solvents.</p>

If **no injection** takes place:

Causes:	Solutions:
<p>Blockage in flow path</p>	<ol style="list-style-type: none"> 1 Disconnect needle from valve. 2 Start a manual wash. 3 If solvent flows from the injection port, check the needle; if no solvent flows from the injection port, disconnect buffer tubing from valve. 4 Start a manual wash. 5 If solvent flows from open end: check rotor seal; if not: disconnect buffer tubing from syringe valve. 6 Start a manual wash. 7 If solvent flows from syringe valve: check buffer tubing; if not, check for over-tightened connections in the entire flow path and check the syringe valve.

Leakage in the injection valve

- 1 Disconnect the needle tubing and buffer tubing.
- 2 Connect port 1 to an HPLC pump.
- 3 Block port 6.
- 4 Start the pump at a low flow.
- 5 Observe ports 3 and 4 for leakage.
- 6 If leakage occurs at ports 3 and 4: check rotor seal; if not: recheck with manual valve.



Observe the maximum allowed pressure of 350 bar to prevent leakage in the valve!

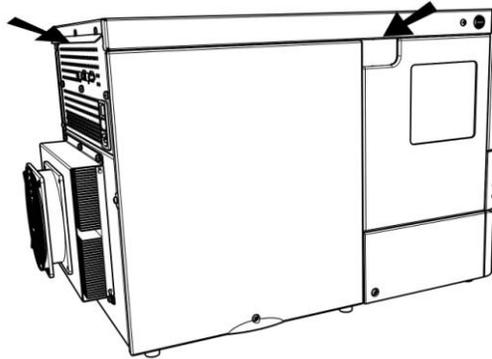
Chapter 5. Installation

Check that all items (refer to packing list) are present.

5-1. Alias autosampler

Execute the following steps for initial installation of the Alias autosampler:

- 1 Lift the Alias from its packaging using both hands at the marked position:



- 2 With both hands *under* the instrument, lift the Alias to its operating location. Keep the instrument upright.



Make sure that the ventilation holes at the back of the autosampler are not blocked. Note that if the ventilation holes are blocked, this may influence performance and cooling capabilities of the autosampler.

If objects are placed on top of the Alias, this may also influence the cooling capabilities.

Objects can be placed on any side of the Alias; however, make sure these objects are placed at a distance of:

- 5 cm from the Alias, if objects are placed at only **one** side of the Alias
- 10 cm from the Alias, if objects are placed on **more than one side** of the Alias

Do not place the Alias in an area subject to excessive dust or shocks. Use the Alias indoors only. Do not place it near a source of heat or in direct sunlight, as this may influence the cooling capabilities of the system.

- 1 Leave the Alias to adopt ambient temperature for at least one hour.
- 2 Install **Alias Service Manager** (see "ASM software") on your PC.
- 3 Check that fuses and voltage range on the rear side of the instrument match that for the power outlet to be used.
- 4 Connect the Alias to the PC COM-port with the cable provided with the Alias.
- 5 Connect the power cable between the Alias and the power outlet.
- 6 Switch on the Alias.
- 7 On your PC, open Alias Service Manager and enter the required settings.
- 8 Connect the drain tubing to the waste outlet.
- 9 Fill the wash solvent bottle inside the sampling compartment of the Alias with distilled water and propanol (80/20 v/v%) or mobile phase. Only water or organic solvents should be used. Do not use crystalline or buffer solutions, as these may block the system and cause severe damage. Degas the wash solvent to prevent air bubbles from forming in the syringe.

10 Fill the wash solvent tubing, syringe and buffer tubing by washing the system two or three times.

Use 100% IPA for better degassing or removing of air bubbles.

11 Check if air bubbles are trapped in the syringe; remove them by gently tapping the syringe.

12 Connect your HPLC pump to port 1 of the injection valve and the column (or the capillary) to port 6 of the injection valve. Check for leakage and let the system stabilize for at least 5 minutes.

5-2. ASM software

A software package is supplied with the Alias autosampler: Alias service manager (ASM). Alias Service Manager allows you to upload Alias firmware. It offers the following functionality:

- You can define a port through which you want to do the update.
- You can access the Communication Settings, Direct control, Service and Adjustment windows for Alias.
- You can access ASM help and About.

Execute the following steps to install ASM:

- 1** Insert the CD into the CD drive of your PC. If the autorun feature is active, the installation wizard will appear. If autorun is not active, use the browser to go to the CD drive and double click install.exe to start the installation wizard.
- 2** Answer all questions that pop up in the wizard; click **Next** to go to the next step in the installation procedure.
- 3** Click **Finish** to end the installation procedure.

The software is now installed. Refer to online help of ASM for more information.

5-2-1. Upload notification

If the upload notification is displayed, the ASM has discovered a problem in PC settings that prevents correct uploading of the new files. This problem may be solved by adapting the size of the serial ports FIFO. Execute the following steps to adapt settings:

- 1** Open the Windows Device Manager (Configuration settings/System settings/Communication port)
- 2** In the COM port properties, select the Port settings tab.

- 3 Click **Advanced**.
 - 4 Use the sliders to change the size of the receive buffer. The default values for Receive buffer high (14) and Transmit buffer high (16) should be sufficient. However, decrease the values to 8 if buffer overflow errors appear.
 - 5 Click **OK** and close the Windows Device Manager.
 - 6 Restart your PC.
 - 7 Open ASM and try to upload again.
- Contact service if there still are problems during uploading.

5-3. Alias fluid connections

When all items have been installed, the following fluid connections are in place:

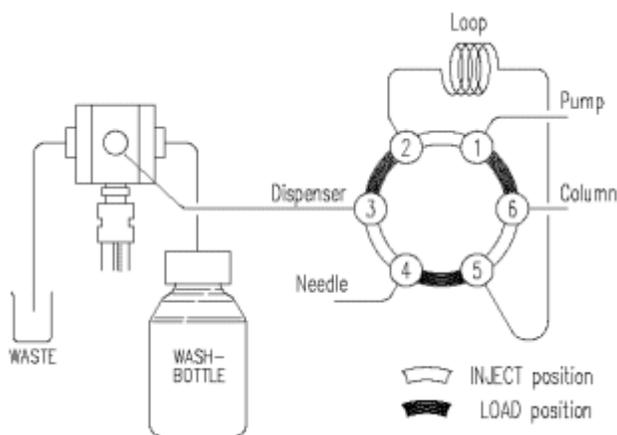


Figure 14 : Alias fluid connections

The Alias is factory-installed with:

- 500 μ L syringe
- 100 μ L sample loop
- 1000 μ L buffer tubing
- 15 μ L stainless steel sample needle.



Make sure that the following are correctly connected:

HPLC pump to port 1 of the injection valve.

HPLC column to port 6 of the injection valve.

5-3-1. Alias tubing

The Alias is standard fitted with the following tubing:

Tubing	Materials/Dimensions
Standard sample needle and tubing (label 15 μ L)	SS: 97 mm x 0.8 mm OD x 0.25 mm ID
Buffer tubing from high-pressure valve to syringe valve (label 1000 μ L)	ETFE (Tefzel): 200 mm x 1/16" OD x 0.25 mm ID
Tubing syringe valve to wash solvent bottle	ETFE (Tefzel): 1275 mm x 1/16" OD x 1.0 mm ID
Tubing syringe valve to waste	PTFE: 400 mm x 1/8" OD x 1.6 mm ID
	PTFE: 400 mm x 1/8" OD x 1.6 mm ID

Note the following if you need to install new tubing:

- insert tube ends always flush with ferrule ends
- do not overtighten nuts, as this may cause blockage in the flow path
- make sure that you always use tubing volumes that are suitable for use with the other items in the flow path.

Tubing guide

To prevent that the wash tubing obstructs the horizontal movement of the needle unit, use the tubing

guide integrated in the leakage drain:

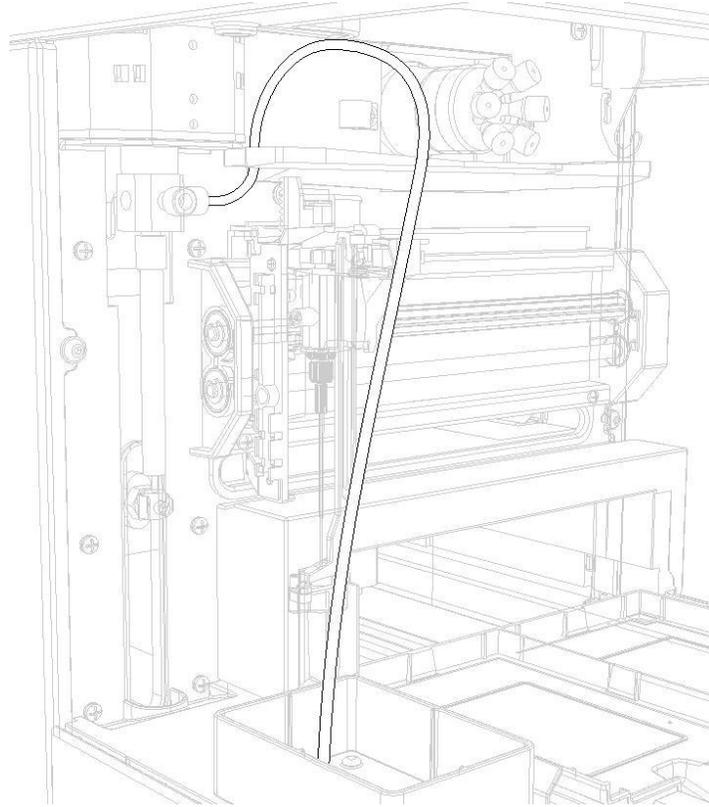


Figure 15 : Tubing guide

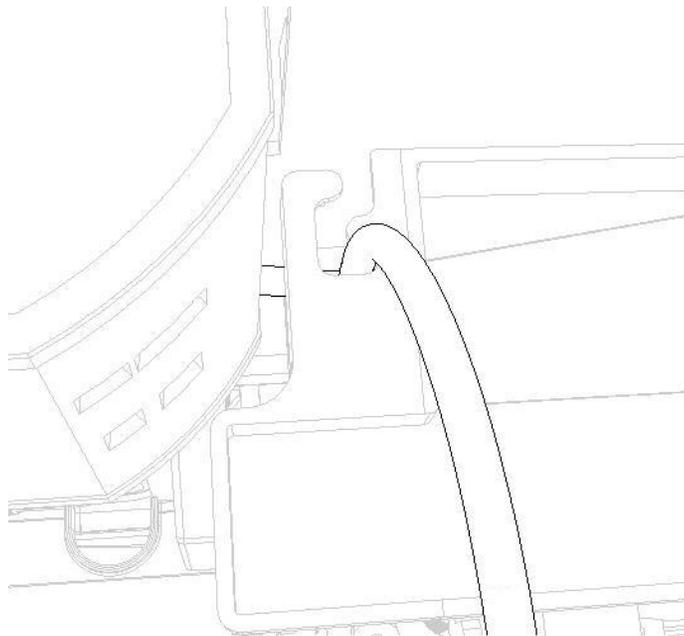


Figure 16: Top view of tubing guide

5-3-2. Waste tubing

Make the following connections for disposal of waste liquids:

- General waste: connect the drain tubing (in the shipkit of the Alias) to the right-hand drain hose connector (see figure 8, number 2). Place the other end in a bottle for waste (on the floor). Through this drain all the liquid dispensed to the wash position is removed. Sample liquid that is not injected is also removed through this tubing.
- Condensation water and leakage drain: through the left-hand hose connector (see figure 8, number 3) all leaked solvents and condensation (from cooling) are drained. If the cooling is used, you are advised to connect this hose connector (in the shipkit of the Alias) to a waste container on the floor.

Make sure that none of the drain or waste tubes is twisted; this might obstruct the flow path.

5-3-3. Wash solvent and syringe rinse

Use a clean bottle for the wash solvent and place it on the left-hand side of the Alias. You are recommended to use a mixture of distilled water and isopropanol (80 /20%) or mobile phase as wash solvent. Before using the wash solvent, degas the solvent with helium or an ultrasonic bath. Do not use salts or buffer solutions; crystals may block or damage the system.

To fill the wash solvent tubing execute the following steps:

- 1 Place the end of the wash solvent tubing in the filled wash solvent bottle.
- 2 Open Direct Control in Alias Service Manager.
- 3 In the Syringe group box, click **End**. A syringe volume of wash solvent is aspirated from the wash solvent bottle and the wash solvent tubing is filled.
- 4 Click **Home**. The syringe contents is dispensed to syringe waste.
- 5 Repeat steps 3 and 4 until the wash solvent tubing and the syringe are completely filled.
- 6 When wash solvent tubing and syringe are completely filled, click **Start** in the Initial wash group box to perform a standard wash routine. All tubing connected to the syringe valve will be rinsed with wash solvent.
- 7 Click **Close** to leave the Direct control screen. The Alias is initialized.

5-3-4. Syringe

A 500 μ L syringe is standard installed in the Alias. However, it is also possible to install a 2500 μ L syringe. Refer to **Syringe dispenser** for more information on how to replace the syringe.

Note that the Alias will give the best results if all air is removed from the syringe. Execute an extra wash to remove air from the syringe.

5-3-5. Sample handling

Take the following into account when handling samples:

- Standard vials can best be filled by means of a narrow-end pipette to allow air to escape when filling the vial.
- Do not fill vials/wells to the edge. If you do, sample will be forced into the air needle, risking cross-contamination of samples and soiling the needles.
- It is important that seals and capmats are airtight to prevent air bubbles from forming and to block evaporation of volatile samples. We recommend use of the following seal types:
 - for standard (low) well plates: sealing tape
 - for deep well plates: pierceable capmats (Pre-slit or silicon) or sealing tape
 - for vials: standard septa (thin types); do not use vials with hard caps that are not designed for being pierced by an injection needle (do not use e.g. Eppendorf SafeLock micro test tubes).
- When you use uncapped vials/wells, injection performance may not be to specification.

Appendix A. Specifications

General:

Sound pressure level	LeAq < 70 dB
Working temperature	10 - 40°C (indoor use only)
Storage temperature	-25 - +60°C
Humidity	20 - 80% RH
Safety and EMC compatibility	According to EC-directives; CSA (UL) approved
Installation class	II
Pollution degree	2
Altitude	up to 2000 m
Dimensions	300 mm x 510 mm x 360 mm (without cooling option) 300 mm x 575 mm x 360 mm (with cooling option)
Weight	19 kg (without cooling) 21 kg (with cooling)
Max. weight that can be placed on top of Alias	65 kg
Power requirements	95 - 240 Volt AC \pm 10%; 50 - 60 Hz; 200VA
Viscosity range	0.1 - 5 cP

Sampling:

Sample capacity	2 Micro Titre Plates according to SBS standards; 96-well high/low and 384-well low formats, 48-vial or 12-vial trays; any combination of plates is allowed, except for 384 Low left and 96 High right.
Vial/Plate dimensions (incl. cap)	Max. plate/vial height: 47 mm (incl. septa or capmat)
Loop volume	1 - 5000 μ L, 10 mL loop optional
Dispenser syringe	500 μ L standard or 2500 μ L for Prep option
Vial detection	Missing vial/well plate detection by sensor
Headspace pressure	Built-in compressor, but only for vials with septa
Switching time injection valve	Electrically < 100 msec
Piercing precision needle	\pm 0.6mm
Wash solvent	Integrated wash solvent bottle
Wetted parts in flow path	SS316, PTFE, TEFZEL, VESPEL, Glass, Teflon. Optional: PEEK
Injection cycle time	< 60 sec. in all injection modes for 1 injection \leq 100 μ L including 300 μ L wash

Analytical performance:

Injection modes	Full loop, partial loopfill and μ L pickup mode, PASA™ (pressure-assisted sample aspiration)
-----------------	--

Reproducibility (valid at 1.0 cP)	RSD \leq 0.3% for full loop injections RSD \leq 0.5% for partial loopfill injections, injection volumes > 10 μ L RSD \leq 1.0% for μ L pickup injections, injection volumes > 10 μ L
Memory effect	< 0.05% with programmable needle wash

Programming:

Interface	via Alias Service Manager software
Injection methods	Full loop, partial loopfill and μ L pickup
Injection volume	0 μ L – 9,999 μ L (with 1 μ L increment), depending on system settings
Max. injection volume	<ul style="list-style-type: none"> ● Full loop = loop volume ● Partial loopfill = $\frac{1}{2}$ x of loop volume ● μL Pick up = (loop volume - 3 x needle volume)/2
Injections per vial/well	max. 9 injections
Analysis time	max. 9 hr, 59 min, 59 sec
Wash	Programmable: Wash between injections and Wash between vials
Timed events	Programmable: 4 x AUX ON/OFF
Priority sample	Programmable

Communication:

Outputs	1 programmable relay output, programmable as Inject marker (default), Auxiliary, Alarm
Inputs	2 programmable TTL inputs, programmable as Next injection input (default), Freeze input, Stop input

Serial communication port RS232C standard

Options (factory installed):

Sample tray cooling	Built-in Peltier cooling Range: 4°C to Ambient - 3°C Temp: air temperature in sample compartment: 4°C ± 2°C (at temperature sensor) (Temperature at relative humidity of 80% and ambient temperature of 25°C)
Communication hardware	On request: RS422/485, CAN, Ethernet

Options (user-installable):

Bio-compatible sample flow path and valve	Inert sample needle (Silco steel) and bio-compatible valve (PEEK)
Prep Kit (see specifications Prep version) (see "Specifications Prep version" on page 56)	2.5 mL syringe, Prep valve, 10 mL sample loop, LSV needle and sample tray for 10 mL vials

A-1. Specifications Prep version

Note that this specification only lists items that are different from the standard Alias specification. The Prep version of Alias is designed for Large Sample Volumes (LSV).

Sampling

Sampling capacity	24 vials of 10 mL (LSV)
Vial dimensions (cap included):	Maximum vial height: 47 mm Minimum vial height: 32 mm
Loop volume	Not programmable, injection volume determines the aspirated sample volume

Dispenser syringe	2500 μ L syringe
Injection volume	0 μ L – 19,999 μ L, with 1 μ L increments
Valve	Valco 0.75 bore valve
Sample loop	10 mL SS sample loop, 1/8" tubing with 1/16" tubing ends and fittings (Valco)
Buffer tubing	2 mL
Needle	LSV needle with LSV air needle Promis and seal

Analytical performance

Injection method	Partial loopfill injection mode
Reproducibility	RSD 1.0% for partial loopfill injections, injection volumes >10 μ L up to 50% of the installed sample loop
Viscosity range	0.1 – 5 cP
Memory effect	< 0.1% with programmable needle wash

Appendix B. Control I/O connections

The Alias has two I/O connections:

- RS232 connector for serial communication using the SparkLink protocol.
- Contact closures output and TTL inputs connector.



The manufacturer will not accept any liability for damages directly or indirectly caused by connecting this machine to instruments which do not meet relevant safety standard.

The IO connector contains active high or active low TTL inputs and one contact closure output, user definable in the System Settings. The two inputs can be programmed as *Next Injection Input*, *Freeze Input* or *Stop Input*. The *Next Injection Input*, *Freeze Input* and *Stop Input* can be used to control the Alias by other devices.

The contact closure output can be programmed as *Inject Marker*, *Auxiliary* or *Alarm output*.

Table: IO connector - Contact closure output and TTL inputs

Pin no:	Description:	Cable colors:
1	Output - Common	RED (3-wired)
2	Output - Normally open	BLACK (3-wired)
3	Input 1	RED (4-wired)
4	Input 2	BLACK (4-wired)
5	GND	
6	Output - Normally closed	BROWN (3-wired)
7	GND	
8	GND	ORANGE (4-wired)
9	GND	BROWN (4-wired)

Contact closure output:

- Inject Marker Output (default): an Inject marker output will be generated when the injection valve switches from LOAD to INJECT. Status duration of the Inject Marker is the same as for the setting for the SparkLink Inject marker pulse. Range of the adjustment of the inject marker pulse is 0.1 - 2.0 seconds.
- Alarm Output: the Alarm Output will be activated whenever an error occurs, see appendix C for a description of the error codes of the Alias.
- Auxiliary: the contact closure output can be used as an Auxiliary which can be programmed on a time base up to 4 times On/Off.

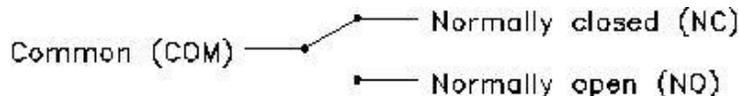


Figure 11: Contact closure output



Contact closure output: $V_{max} = 28 \text{ Vdc / Vac}$, $I_{max} = 0.25 \text{ A}$

TTL inputs:

- Next Injection Input (default): this input will start the next injection sequence After finishing the injection sequence the Alias will wait for the Next Injection Input.
- Freeze input: the Alias will freeze the analysis time for the time this input is active. If the Freeze Input is activated while the analysis time is not running, the Alias will perform all programmed pre-injection sample handling (sample loop). But the Alias will wait with injecting the sample until the Freeze Input is no longer active.
- Stop Input: with this input the run of the Alias is immediately aborted.

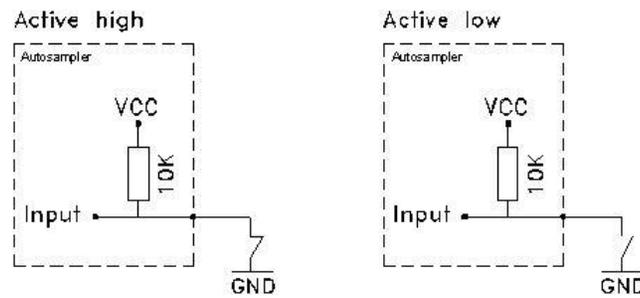


Figure 17: TTL input

Appendix C. List of errors

The following errors may occur with Alias:

Tray unit	
ERROR 294	Home sensor not reached.
ERROR 295	Deviation of more than +/-2mm towards home.
ERROR 296	Home sensor not de-activated.
ERROR 297	Home sensor activated when not expected.
ERROR 298	Tray position is unknown.
Needle unit	
ERROR 303	Horizontal: needle position is unknown.
ERROR 304	Horizontal: home sensor not reached.
ERROR 306	Horizontal: home sensor not de-activated.
ERROR 307	Horizontal: home sensor activated when not expected.
ERROR 312	Vertical: needle position is unknown.
ERROR 313	Vertical: home sensor not reached.
ERROR 315	Vertical: home sensor not de-activated.

ERROR 317	Vertical: stripper did not detect plate (or wash/waste).
ERROR 318	Vertical: stripper stuck.
ERROR 319	Vertical: The sample needle arm is at an invalid position.

Syringe dispenser unit

ERROR 324	Syringe valve did not find destination position.
ERROR 330	Syringe home sensor not reached.
ERROR 331	Syringe home sensor not de-activated.
ERROR 334	Syringe position is unknown.
ERROR 335	Syringe rotation error.

Injection valve

ERROR 340	Destination position not reached.
ERROR 341	Wear-out limit reached.
ERROR 342	Illegal sensor readout.

Cooling unit

ERROR 347	Temperature above 48°C at cooling ON.
-----------	---------------------------------------

Electronics

ERROR 280	EEPROM write error.
ERROR 282	EEPROM error in settings.
ERROR 283	EEPROM error in adjustments.
ERROR 284	EEPROM error in log counter.
ERROR 290	Error occurred during initialization, the Alias cannot start.

Appendix D. List of accessories and spares

The standard shipkit for the Alias contains:

- a shipkit list
- a silicon drain hose
- shipkit for the injection valve (Valco, Rheodyne or other)
- 2 x 2.5A TL 250V fuse for power connection
- Wash bottle
- CD with user manual
- Needle for high titre plates (needle for low titre plates is in instrument)
- IO cable
- serial cable

The following accessories are available for the Alias autosampler:

- Vial adapter 2 mL vials
- Vial adapter 10 mL vials
- Various types of titre plates
- Capmats
- Syringes: 4400.500 Syringe 500 μ L
 4400.255 Syringe 2500 μ L (Prep)
- Sample needles: 0840.303 STD with Rheodyne connections
 0840.304 BIO with Rheodyne connections
 0840.313 STD with Valco connections
 0840.314 BIO with Valco connections
 0840.319 serum sample needle with Valco connections
- 6 different types of air needles for different heights of titre plates or vials:

		Protrusion length	
	Air needle type	from	to
0045.503	50 mm, yellow	34	40
0045.504	56 mm, red	28	34
0045.505	62 mm, white (standard needle)	22	28

0045.506	68 mm, blue	16	22
0045.507	74 mm, green	10	16
0045.508	80 mm black	4	10

YL9100 HPLC SYSTEM

YL9160 PDA Detector

USER MANUAL



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Chapter 1. Introduction

YL9160 PDA detector is the photo diode array absorbance HPLC detector covering very wide wavelengths from 190~950nm which can analyze simultaneous spectrum at very high rate and sensitivity. It features very low noise, drift level, variable bandwidth selection and very high rate data acquisition in order to meet most of the requirements for a HPLC detector.

1-1. Features of YL9160

- 1) Wide wavelength range and high rate data acquisition
- 2) Improved baseline stability by using high inner pressure cell assembly having RI-compensated heat exchange
- 3) Reduced drift level by improved heat exchange
- 4) Improved user access by front-mounting of lamps and cell
- 5) External valve controls for solvent recovery and sample preparation.

1-2. Specifications

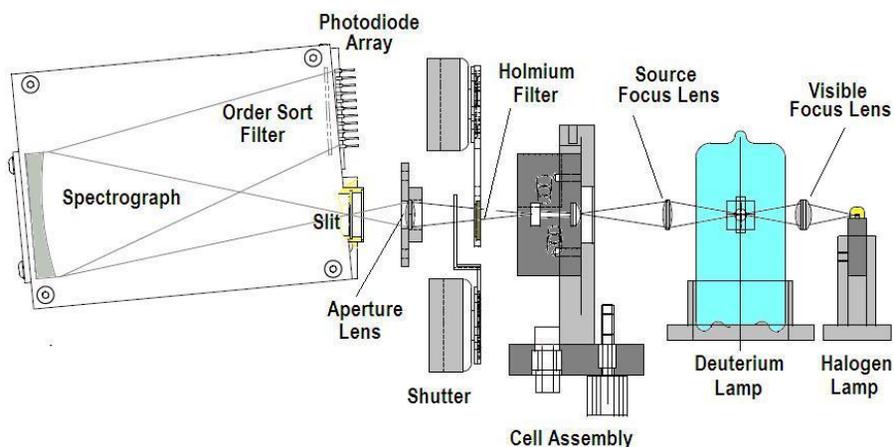
- 1) PDA AD Resolution : 16bit
- 2) Slit Bandwidth : 1.7nm
- 3) Pixel Resolution : 0.9nm
- 4) No. of PDA Channel : 1024
- 5) Wavelength : 190~950
- 6) Shield Optics
- 7) Analytical Cell
 - Path-length : 10mm
 - Pressure : >1500psi
 - Volume : 13ul
- 8) Dimension : 385 X 160 X 565 mm (width X height X depth)
- 9) Weight : 15Kg
- 10) Noise Level : $\pm 2 \times 10^{-5}$ AU (Empty Cell, 1sec Rise Time, 254nm)
- 11) Drift : $< 2 \times 10^{-4}$ AU/hr (Baseline Correction), 0.001AU/hr (Room Temp)
- 12) Wavelength Accuracy : < 1nm (HY-1 Holmium Oxide Filter)

- 13) Sampling Rate : Max. 50Hz 1~16 nm Spectral Bandwidth, Wide Spectral Range, Alternative Spectral Function
- 15) GLP Compliance:
 - Photometric Accuracy, Linearity, Noise Level, Drift
 - System Check,
- 16) Analog Output : 2 channel (Replaceable Dual Wavelength Detector)
- 17) Valve Output : 2ch, Programmable Sampling (Fraction Collector Function Available)
- 18) Trigger Input : 2ch
- 19) Communication : TCP/IP
- 20) Simultaneous Data Channel : 8 or Full Scan
- 21) Filtering : Bessel, RC, Aver. 0.01~10Hz

[Selection of cells]

	Microbore	Analytical	Semi-prep
Path length	10mm	5mm	3mm
Cell volume	13ul	6ul	5.3ul
Tube ID	0.007"	0.01"	0.02"
Cell pressure(psi)	1500	1500psi	1000psi
Flow contact materials	PEEK, teflon, Quartz	SS316, Teflon, Quartz	

1-3. Optics Design



[Fig. 1] YL9160 PDA detector optics design

Optics design of YL9160 PDA detector is shown as [Fig. 1]. A deuterium lamp is used for ultraviolet

range and a tungsten-halogen lamp is used for visible range. Visible lights pass through the arc hole of a D2 lamp so that all range of lights transmit to the 50 um slit. After projected to the slit, lights go to 1,024 photo diode array by a grating. Order sort filter is located in the front of PDA to remove 2nd light.

All parts are very precise materials, so it is prohibited for a user to open the optics for repair. Optics should be opened by a trained service engineer only. Only remained lights after absorbed by sample arrives at the photo diode array and photo-electricity exchange occurs in proportion to arrived light amounts at the PDA . The voltage is digitalized by A/D converter and calculated logarithmic and filtered at YL9160 microprocessor. The value is given as voltage output on a output terminal or transmitted through communication.

Chapter 2. Installation

2-1. Inspection and site preparation

Carefully unpack the detector from the shipping box and inspect both the unit and packing for any signs of damage. If any damage is noted, contact the shipping company immediately. In addition to this manual, the shipping box contains a power cord, and any options which you ordered. Carefully check the packing list against the contents of the container. If anything is missing, check the packing materials carefully for the overlooked items. If items are missing, contact us or your supplier. Place the detector on the bench where it will be used and familiarize yourself with the location and function of the controls and connections.

Site requirement of YL9160 PDA Detector

- 1) Room with 20°C temperature with variation $\pm 5^\circ\text{C}$ with and 60% humidity
- 2) Where no direct and straight sunshine
- 3) Plain floor without carpet
- 4) Where having spare space of 20cm or more
- 5) Where there are 10% or less voltage change, no frequency change and 100 Ω or less grounding point
- 6) Where there is no generation of corrosive gas and ventilation is well done
- 7) Where stable power of 110 or 220V is supplied, $\pm 10\%$
- 8) Where not receiving electromagnetic induction from large transformer, high frequency heater, UPS, etc.

Place the detector on a laboratory benchtop in close proximity to the HPLC column outlet. Allow at least 5 inches of clear space between the rear panel of the unit and any wall or obstruction. This provides both access to the rear panel connections and a free flow of air

In addition to the detector itself, you will need the following items for setup and initial operation :

- 1) YL-Clarity software or Chromatograph Data System.
- 2) Pump
- 3) Column
- 4) Standard test mixture

- 5) Appropriate solvents, reagents, etc
- 6) Nuts, ferrules, appropriate to the column end-fittings being used
- 7) Wrenches appropriate to column end-fittings
- 8) Connecting tubing and union (if column cannot be connected directly to the cell).

2-2. Connection of power

Confirm supplying voltage, electric power, socket, and type of connector are correct. Before connect power cable on the pump, please turn off the power switch of YL9160 PDA Detector.

Warning ! : The electric power should be matched with description on the rear side of instrument. If not, the instrument can be damaged or it can be a reason of fire.

Caution ! : Confirm the ground is correct. Water pipe is not recommended in case of non-metal pipe. Gas pipe should not use because of safety. If the ground is not correct, the electric shock can be happened.

2-3. Connection of a column

The length of tubing between the inlet of flow cell assembly and outlet of column should be connected as short as possible. It is the ideal that you connect the tubing directly between these two ends. If this is not possible, you should use a minimum length of narrow bore (0.010 inch I.D.) connecting tubing and a zero dead volume union. Because different columns use different fittings, the detector is supplied with a bare tube end to allow connection to any column accepting 1/16 inch O.D. tubing. You should use nuts and ferrules suitable to your column.

NOTE: Tubing size and position is different for the adjustable path length preparative flow cells, high pressure narrow-bore flow cell, off column capillary flow cell, and on column capillary flow cell. See their owner's manuals for details.

Connect the cell outlet (the upper of the two tubes which protrude from the rear wall of the cell compartment) to a line leading to an appropriate waste reservoir. If bubble formation in the detector cell causes problems, you may wish to connect the cell outlet to a restrictor or back pressure device providing 20-60 psi back-pressure.

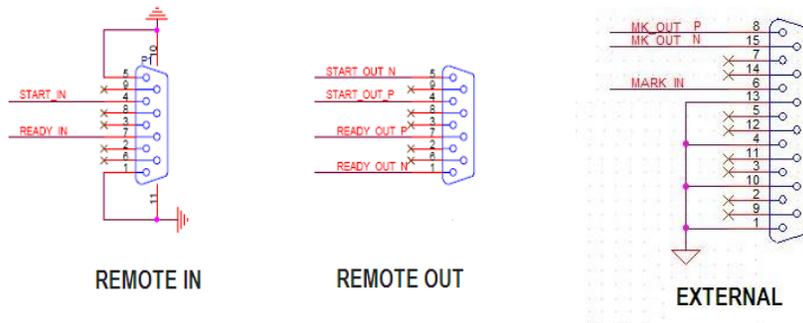
NOTE: Before connecting any new tube or column to the detector, flow several mL of clean solvent

through the new tube to a waste reservoir. This will clean any particulates or oil that may be residing in the tube that could clog the heat exchanger or contaminate the sample cell of the detector.

2-4. Connection of remote cable

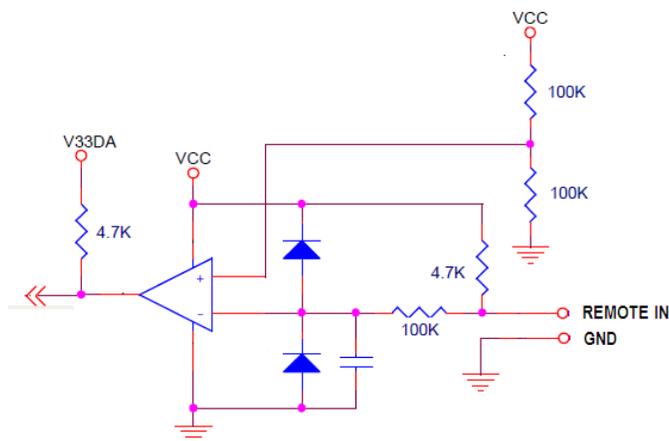
YL9160 PDA detector has connection terminals for remote input/output, and marker input/output.

Note : Please do not connect wires between cables at your discretion. If you want to connect with the other instrument, please check input/output information and confirm with the terminal configuration of YL9160 PDA detector.

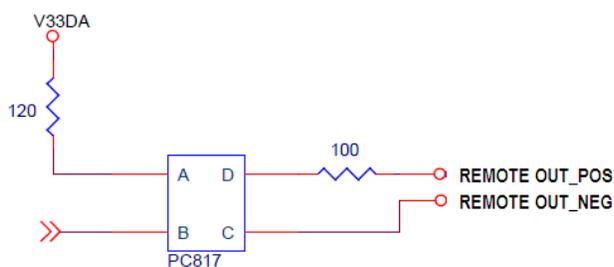


[Fig. 2] Pin configuration of each terminal

[Fig. 3] and [Fig. 4] are the diagram of remote and the other terminal input/output. In between YL9100 series modules, connect directly and confirm the configuration with the other modules.



[Fig. 3] Diagram of Remote and Marker input



[Fig. 4] Diagram of Remote and Marker output

[Remote operation]

START-IN : Operate instrument, and start running of gradient program.
 If you connect it with autosampler or external valve, automatic running is available.

START-OUT : If the signal input on the START-IN terminal, the signal pulse output through this port. It can be used for synchronization of remote start with the other instrument.

MARK-IN : To control event program or operate additional operation.

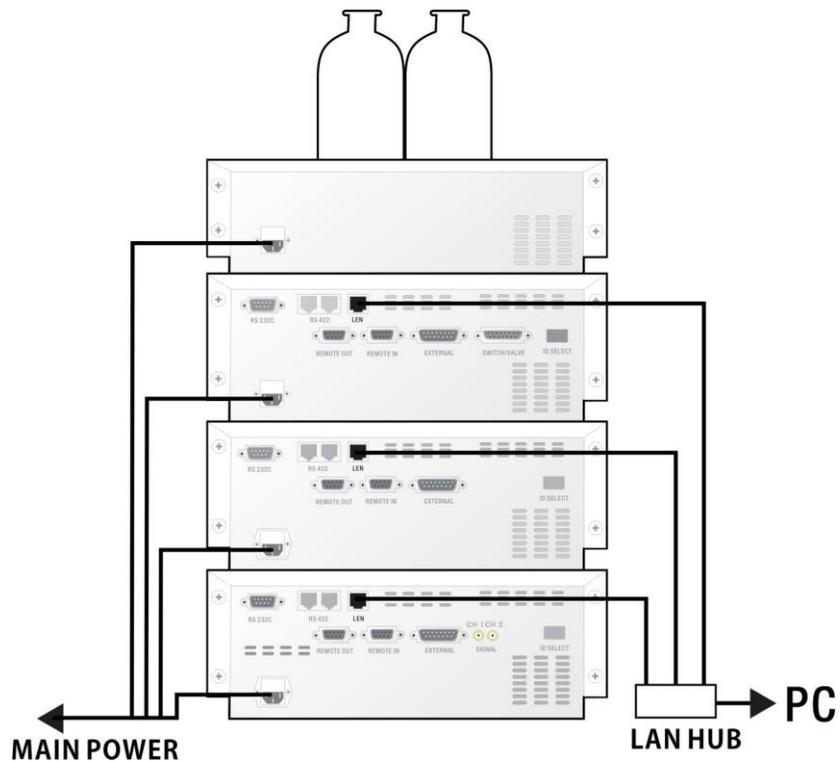
MARK-OUT : To control time event program output.

READY-IN : To change error state and stop operation if there is a input.

READY-OUT : When YL9160 PDA detector is not ready state because of running status, output error signal is indicated if there is a leak.

2-5. Connection of communication cable

YL9160 PDA detector provides TCP/IP internet protocol as a standard. The IP address of YL9100 series is 10.10.10.60, if DIP SW settings on the rear side are On position. If you change the IP address using control software, the DIP SW has to be set OFF.



[Fig. 5] Connection of communication cable

Note : The LAN HUB used for cable connection on the PC must use switching mode module.

Chapter 3. Operation

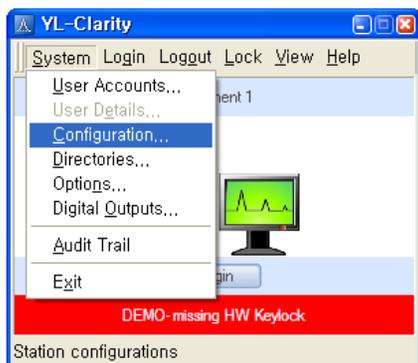
There are four LEDs in front of YL9160 PDA detector.

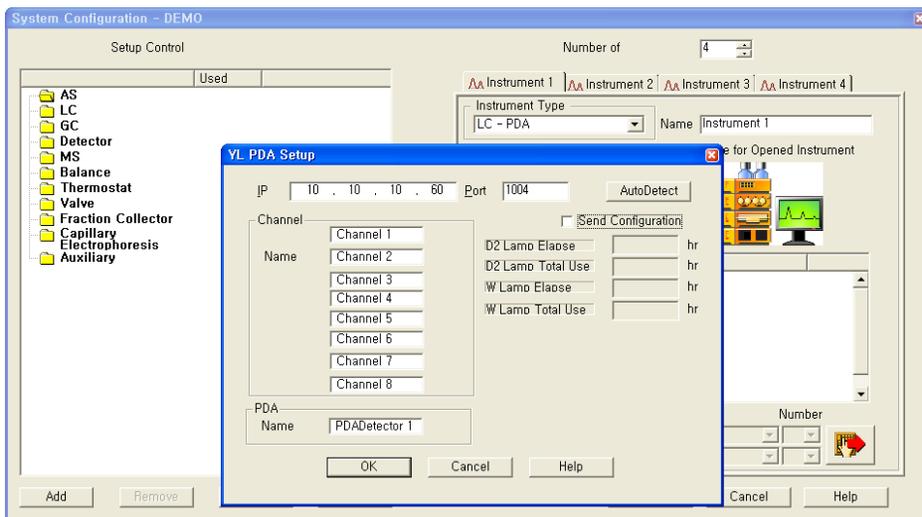
	POWER	LED turns ON if main power turns on
	CONNECTED	LED turns ON if communication is connected, LED blinks during connection
	READY/RUN	LED turns ON before analysis, LED blinks during analysis
	ERROR	LED turns ON if there is error

3-1. YL-Clarity Chromatograph software

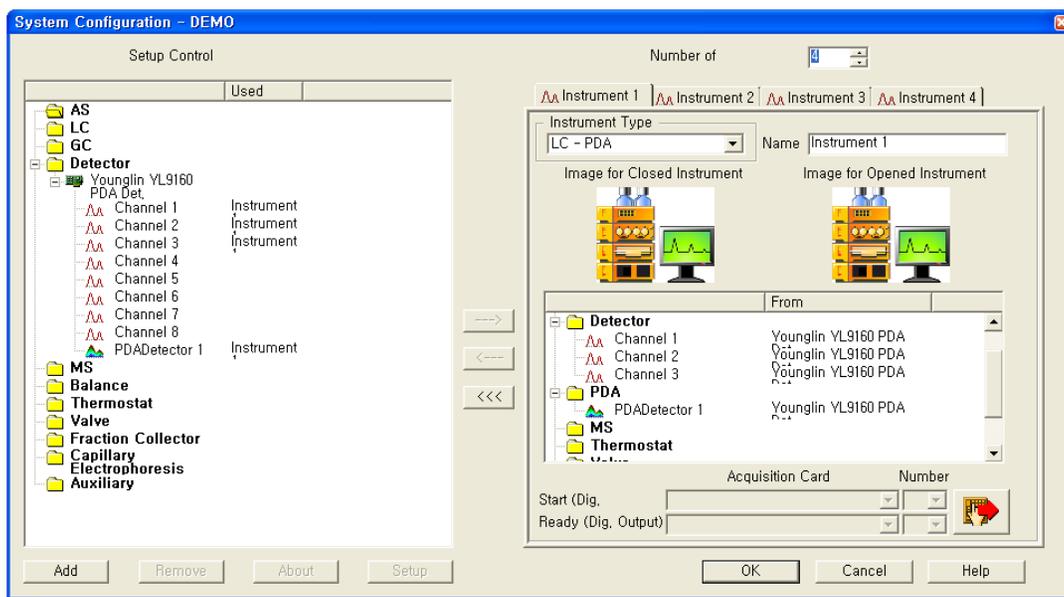
3-1-1. Installation of PDA Detector

After execute the YL-Clarity software, select Configuration at the first menu to open Setup Window and select LC-PDA.



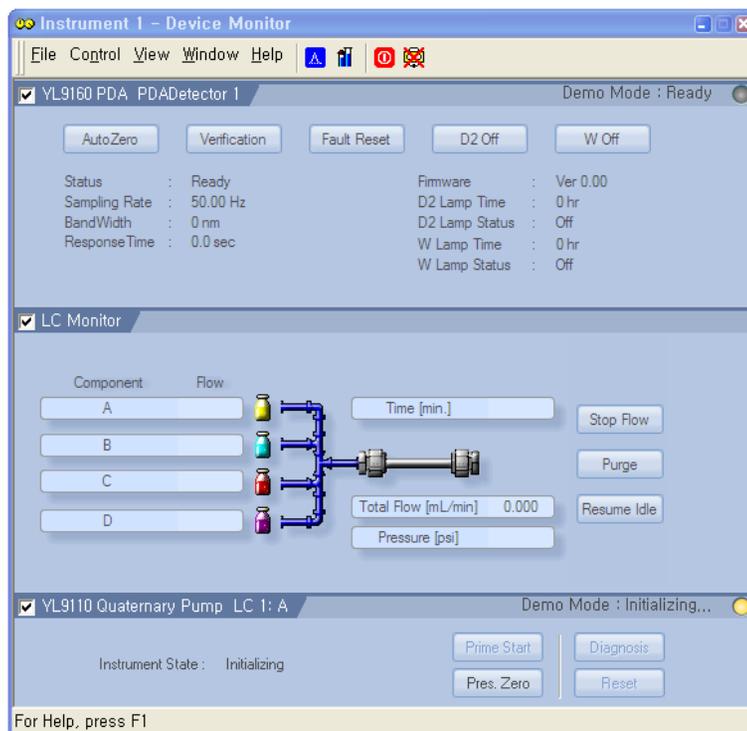


Click Add button to add YL9160 PDA Detector to pop up Setup window. Check IP address and add acquisition channels and PDA module.



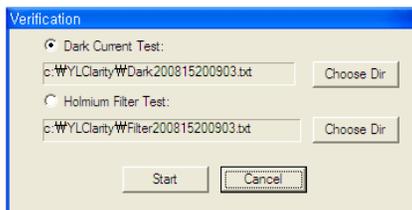
3-1-2. Device monitor

After setting the PDA, log in the software then following device monitor windows will come up. On this window, can check detector status and control.



Auto Zero : Automatically zero absorbance value.

Verification : Verify wavelength accuracy and dark current. Test result will be saved as a text file format in a designated folder.



Fault Reset : Release Fault status when error happens.

D2 On/Off : Turn on/off D2 lamp.

W On/Off : Turn on/off Halogen-tungsten lamp.

Status : Display of YL9160 status. Ready, Initializing, Run or any errors

Sampling Rate : Display acquisition rate selected.

Bandwidth : Display bandwidth selected.

Response Time : Display filter response time of acquisition data.

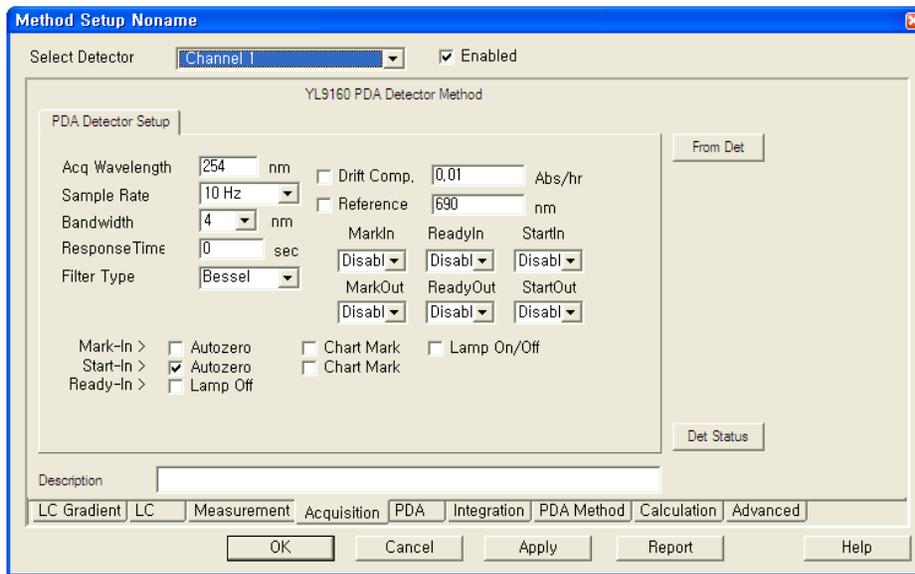
Firmware : Display firmware version.

D2 Lamp Time : Display used time of D2 lamp.

D2 Lamp Status : Display whether D2 lamp is on or off.

W Lamp Time : Display used time of W lamp..
 W Lamp Status : Display whether W lamp is on or off.

[Setup of acquisition]



In this window, can select acquisition wavelength for each channel. Can specify up to 8 channels and other parameters except wavelength are applied to all channels.

- Acq Wavelength : specify wavelength that want to acquire.
- Sampling Rate : input sample rate.
- Bandwidth : select bandwidth.
- Response Time : input response time for the acquisition date from 0.1 ~ 9.9.
- Filter Type : select filter type applied to the response time.
- Drift Comp : select if want to compensate drift. If selected, automatically compensate the drift within the specified valve.
- Reference : select if want to compensate drift and specify the wavelength to use for drift compensation.
- Mark-In > : Select Able if want to use Mark-in. Also select Autozero, Char Mark and Lamp on/off if want to perform any of them when marker is inputted.
- Start-In > : Select Able if want to use Start-in. Also select Autozero and Chart Mark when start signal is inputted.
- Ready-In> : Select Lamp Off when Ready-in signal is inputted.

Chapter 4. Maintenance

4-1. D2 lamp

The Deuterium Lamp (D₂) is covered from 190 to 600 nm of wavelength. The replacement time is either in case you use the lamp more than 2000 hours or in case the light intensity is a half (50%) of the original intensity when you installed the lamp at first. An using time of D2 lamp is counted in the system indicating the total hours of operation after lamp on.

Check the lamp intensity as follows:

- 1) Power on the unit if it is not already on. Wait for a period of approximately 10 minutes.
- 2) Set the wavelength to 254 nm by YL-Clarity software
- 3) Select the Reference Light Intensity.
- 4) If the displayed value is less than a half of the original intensity, the lamp should be replaced to new one. Generally, you have to consider the lamp exchange when the light intensity of reference energy is less than 50 nA.

4-1-1. How to remove the D₂ lamp

- 1) Make sure that the power cord is disconnected from the rear panel of the detector.
- 2) Unscrew the screws and remove the lamp assembly on the right front panel.

CAUTION ; UV light can damage eyes and skin. Always disconnect the power cord before working in the vicinity of the lamp. The D₂ lamp gets quite hot. Care must be taken while handling it to prevent from burning. Always allow the lamp to cool before removing it.

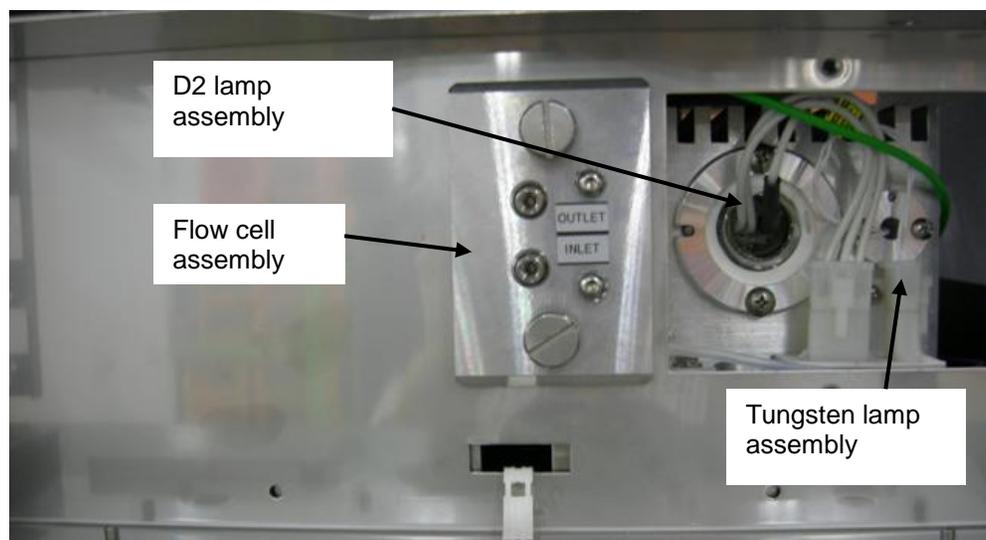
- 3) Disconnect the UV lamp from the detector by gently pulling it straight back toward you. DO NOT twist the connector while pulling.
- 4) Unscrew the two thumbscrews holding the lamp mount in place, and pull the lamp mount straight back

- 5) towards you. Be careful not to lose the two thumbscrews. Be careful not to get fingerprints on the lamp.

4-1-2. How to install a new D₂ lamp

- 1) Insert the new lamp assembly onto the lamp housing on the right front panel.
- 2) Use the thumbscrews to attach the lamp assembly to the detector.
- 3) Connect the lamp lead to the lower of the two terminals in the lamp compartment.

CAUTION ; NEVER loosen the screw holding the lamp to the mount, and never attempt to rotate or move the lamp up or down in the mount. The lamp is provided as a pre-aligned assembly.



[Fig. 5] Front of YL9160 PDA Detector

4-2. Tungsten lamp (W)

The replacement time of the tungsten lamp is approximately 1,500 hours. To check the W lamp intensity:

- 1) Power on the unit if it is not already on. Wait approximately 10 minutes.
- 2) Set the wavelength to 720nm by YL-Clarity software.
- 3) Select the Reference Light Intensity.
- 4) If the displayed value is less than a half of the original intensity, the lamp should be replaced to new one. Generally, you have to consider the lamp exchange when the light intensity of reference energy is less than 5 nA.

4-2-1. How to remove the Tungsten lamp

- 1) Make sure that the power cord is disconnected from the rear panel of the detector.
- 2) Unscrew the screws and remove the lamp assembly on the right front panel.

CAUTION ; The light of Tungsten lamp can damage eyes and skin. Always disconnect the power cord before working in the vicinity of the lamp. The W lamp gets quite hot. Care must be taken while handling it to prevent from burning. Always allow the lamp to cool before removing it.

- 3) Disconnect the Tungsten lamp from the detector by gently pulling it straight back toward you. DO NOT twist the connector while pulling.
- 4) Unscrew the two thumbscrews holding the lamp mount in place, and pull the lamp mount straight back towards you. Be careful not to lose the two thumbscrews. Be careful not to get fingerprints on the lamp.



[Fig. 6] Tungsten lamp assembly

4-2-2. How to install a new Tungsten lamp

- 1) Insert the new lamp assembly onto the lamp housing on the right front panel.
- 2) Use the thumbscrews to attach the lamp assembly to the detector.

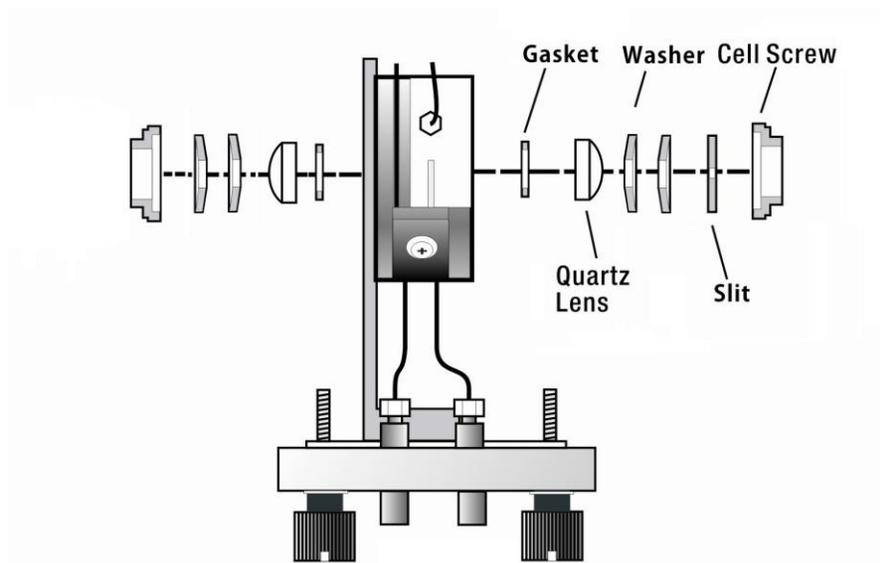
- 3) Connect the lamp lead to the lower of the two terminals in the lamp compartment.

4-3. Flow cell cleaning

We do not recommend disassembly of the flow cell assembly for routine cleaning purposes. Most of the cell assembly can be adequately cleaned by flushing with several milliliters of appropriate solvent. We recommend the following solvents for this purpose:

- 1) Methanol
- 2) Tetrahydrofuran
- 3) Methylene Chloride
- 4) HPLC Grade Water
- 5) 6 N Nitric Acid followed by flushing with HPLC Grade Water

NOTE : Use only HPLC grade solvents.



[Fig. 7] The diagram of Flow cell assembly

4-4. Troubleshooting

Most problems with HPLC detectors are, in fact, caused by other parts of the system. Noisy and drifting baselines, poor reproducibility in quantitative analysis, and similar problems are more often the result of

dissolved air bubbles, contaminated eluents, dirty samples, or damaged columns rather than of actual problems with detector hardware. In order to focus more effectively on troubleshooting detector problems, we will first discuss on-board diagnostic tips and later present a troubleshooting table organized by symptom, cause and how to fix.

Problem	Cause	How to fix
1. Unstable Baseline	Bubbles passing through cell.	Degas solvent and/or supply back pressure to the sample cell, also check all high pressure fittings for leaks(both liquid and gasses)
	External triggering device is creating electrical noise.	Check electrical lines for good connection and/or interference from broad cast radiation. Check for ground loops.
	Extremely large supply voltage transient on the AC line	Remove systems that consume high power from the AC line.
2. Irregular Baseline Noise	Sample cell windows are contaminated.	Flush cell with solvents(methanol, acetone, water, nitric(6N) acid, water) and check for
	Sample input line has a leak.	Check all lines from the output of the column to the input of the sample cell for leaks.
	Bubble trapped in the sample cell.	Increase flow rate and/or back pressure on cell.
	Recorder or integrator is grounded and is causing a "ground loop" problem.	Check recorder with voltmeter to see if either of the signal inputs is grounded to case or earth ground.
	Photodiode window is dirty or not held down properly to the cell holder.	Remove and clean photodiode window.
	Sample cell is not screwed down to the main unit.	Check sample cell mounts and cell holder assembly.
	Output span of the detector does not match input range of integrator.	Press event mark to see if the "spike" is approximately 20% of scale.
	External triggering device is causing a ground loop problem.	Use only triggering device with ground isolated from earth ground.
3. Baseline Drift	Contamination of sample cell windows has occurred.	Clean cell by flushing with solvents (methanol, acetone, water, nitric(5N) acid). Inspect cell and photodiode for fingerprints and smudges and clean if necessary.
	The absorption of solvent in the column has been changed.	Column is filled with UV absorbers that are bleeding-replace column; impure solvent is equilibrating with the column-replace solvent with more pure grade, switch to a longer wavelength so that background absorption does not fluctuate as much.
	Leakage in the lines from column to flow cell.	Check lines for leakage.

YL9100 HPLC System

YL9170 RI Detector

USER MANUAL



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Chapter 1. Introduction

The YL9170RI Detector(Refractive Index detector) is used for detection of polymer(PE, PP, PVC, etc.), polymer additive and bio-polymer(Glucose, Sucrose, Fructose, Maltose, Lactose, etc.) as detector used for HPLC and GPC.

1-1. Features of YL9170

- 1) High Analysis Sensivity
- 2) Broad Linearity
- 3) High Stability
- 4) Internal temperature program and control
- 5) Thermal Isolation Optical Design
- 6) Preparative or Semi-micro application(Optional)

1-2. Specifications

- 1) Detection method: Deflection
- 2) RI Range: 1.00 to 1.75 RIU
- 3) Typical flow rate : 0.2 ~ 3.0 ml/min
- 4) Noise : $\leq 5 \times 10^{-10}$ RIU
- 5) Auto zero range : 40×10^{-5} RIU
- 6) Auto zero resolution: to 5×10^{-10} RIU
- 7) Recorder output: ± 1 V
- 8) Linear dynamic range : 80×10^{-5} RIU
- 9) Drift rate : 4×10^{-8} RIU
- 10) Flow rate range : 0.2 ~ 3.0 mL/min
- 11) Cell volume : 9 μ l
- 12) Cell pressure: 6 kg/cm²(84 psi)
- 13) Volume into cell : 24 μ l
- 14) Temperature control: 35 °C ~ 55 °C
- 15) Digital input : Purge, Auto Zero
- 16) Dimension : 385 X 160 X 565mm (width X height X depth)
- 17) Communications : RS232C
- 18) Line Voltage : 110 or 220 VAC, $\pm 10\%$

19) Line frequency : 50/60Hz, $\pm 5\%$

20) Weight : 11 Kg

Chapter 2. Installation

2-1. Inspection and site preparation

Carefully unpack the detector from the shipping box and inspect both the unit and packing for any signs of damage. If any damage is noted, contact the shipping company immediately. In addition to this manual, the shipping box contains a power cord, and any options, which you ordered. Carefully check the packing list against the contents of the container. If anything is missing, check the packing materials carefully for the overlooked items. If items are missing, contact to local supplier or us. Place the detector on the bench where it will be used and familiarize yourself with the location and function of the controls and connections.

Site requirement of YL9170 RI Detector

- 1) Room with 20°C temperature with variation $\pm 5^\circ\text{C}$ with and 60% humidity
- 2) Where no direct and straight sunshine
- 3) Plain floor without carpet
- 4) Where having spare space of 20cm or more
- 5) Where there are 10% or less voltage change, no frequency change and 100 Ω or less grounding point
- 6) Where there is no generation of corrosive gas and ventilation is well done
- 7) Where stable power of 110 or 220V is supplied, $\pm 10\%$
- 8) Where not receiving electromagnetic induction from large transformer, high frequency heater, UPS, etc.

Place the detector on a laboratory benchtop in close proximity to the HPLC column outlet. Allow at least 5 inches of clear space between the rear panel of the unit and any wall or obstruction. This provides both access to the rear panel connections and a free flow of air

In addition to the detector itself, you will need the following items for setup and initial operation :

- 1) YL-Clarity software or Chromatograph Data System.

- 2) Pump
- 3) Column
- 4) Standard test mixture
- 5) Appropriate solvents, reagents, etc
- 6) Nuts, ferrules, appropriate to the column end-fittings being used
- 7) Wrenches appropriate to column end-fittings
- 8) Connecting tubing and union (if column cannot be connected directly to the cell).

2-2. Connection of power

Check the supplying voltage, electric power, socket, and types of connector are correct. Before connect power cable on the pump, please turn off the power switch of YL9170 RI Detector.

Warning ! : The electric power should be matched with description on the rear side of instrument. If not, the instrument can be damaged or it can be a reason of fire.

Caution ! : Confirm the ground is correct. Water pipe is not recommended in case of non-metal pipe. Gas pipe should not use because of safety. If the ground is not correct, the electric shock can be happened.

2-3. Connection of a column

The length of tubing between the inlet of flow cell assembly and outlet of column should be connected as short as possible. It is the ideal that you connect the tubing directly between these two ends. If this is not possible, you should use a minimum length of narrow bore (0.010 inch I.D.) connecting tubing and a zero dead volume union. Because different columns use different fittings, the detector is supplied with a bare tube end to allow connection to any column accepting 1/16 inch O.D. tubing. You should use nuts and ferrules suitable to your column.

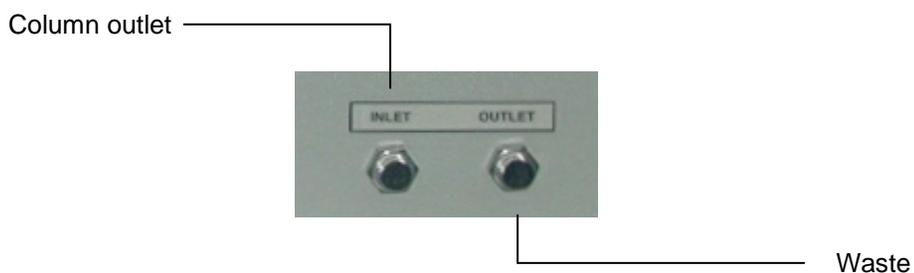
NOTE: Tubing size and position is different for the adjustable path length preparative flow cells, high pressure narrow-bore flow cell, off column capillary flow cell, and on column capillary flow cell. See their owner's manuals for details.

Connect the cell outlet (the upper of the two tubes which protrude from the rear wall of the cell compartment) to a line leading to an appropriate waste reservoir. If bubble formation in the detector cell causes problems, you may wish to connect the cell outlet to a restrictor or back pressure device providing

20-60 psi back-pressure.

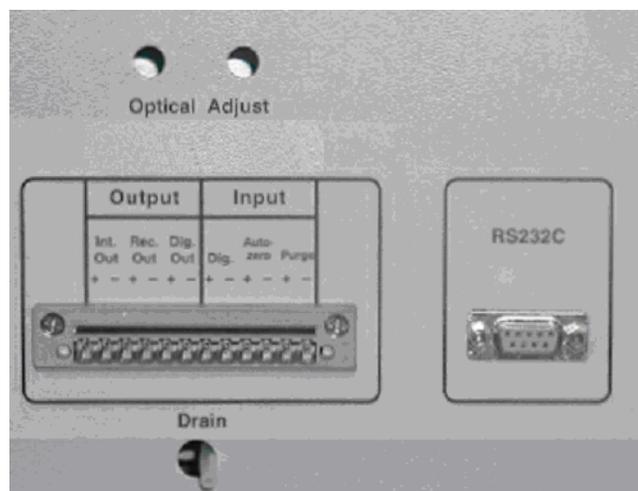
NOTE: Before connecting any new tube or column to the detector, flow several mL of clean solvent through the new tube to a waste reservoir. This will clean any particulates or oil that may be residing in the tube that could clog the heat exchanger or contaminate the sample cell of the detector.

Connect the capillary, coming from the HPLC column with the INLET port of RI Detector. Use the stainless steel capillary tubing with the Teflon tubing and connect the tubing to your waste reservoir.



2-4. Connection of cables

If you acquire the data with an integrator, data system or recorder, connect the signal cable, using one of the 2pin-connectors with the analog output of YL9170 RI detector at the rear side of the detector, using **INT-Out or Rec-Out**.



Chapter 3. Operation

There are three LEDs in front of YL9170 RI detector.

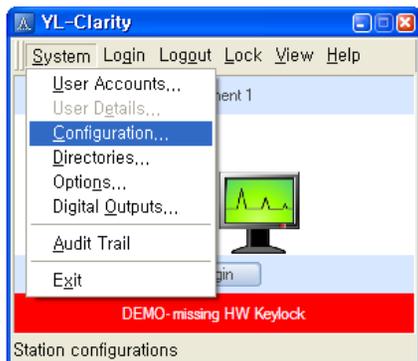
	POWER	LED turns ON if main power turns on
	PURGE	LED turns ON during cell purging
	PORALITY	LED indicates the polarity of signal output

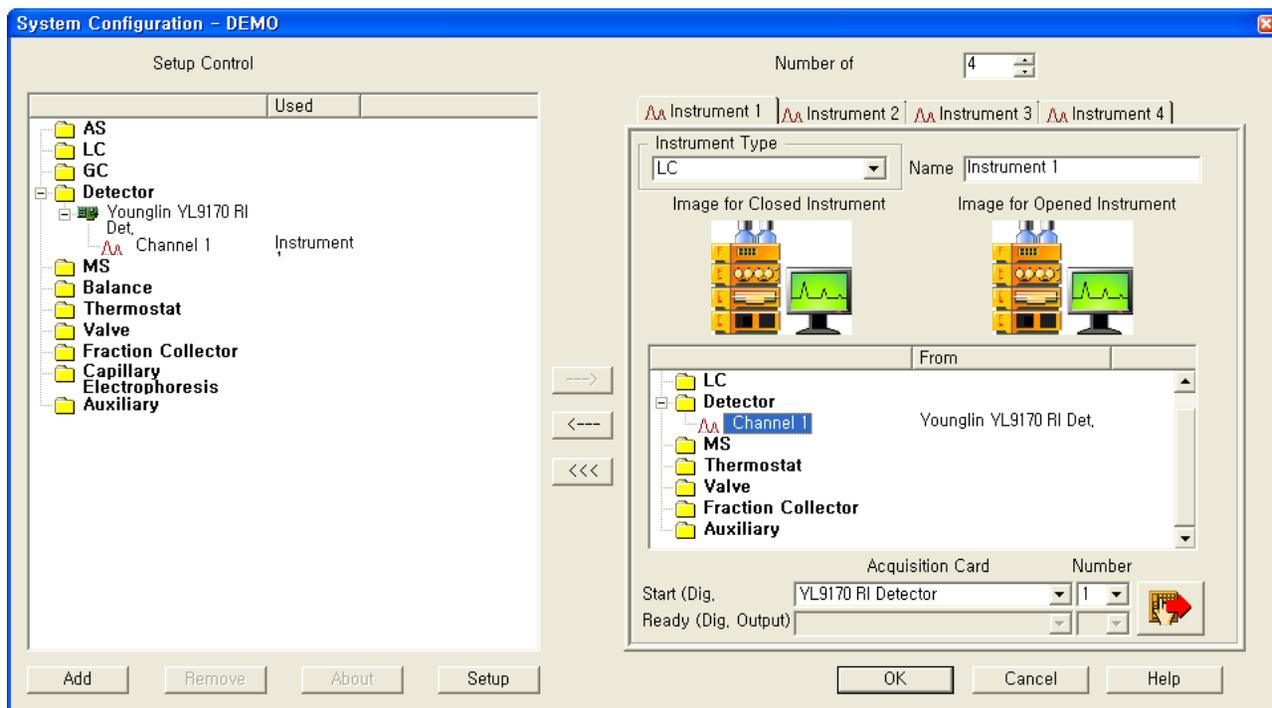
About 2 minutes later after power-up, you can control and operate YL9170 RI detector by YL-Clarity.

3-1. YL-Clarity Chromatograph software

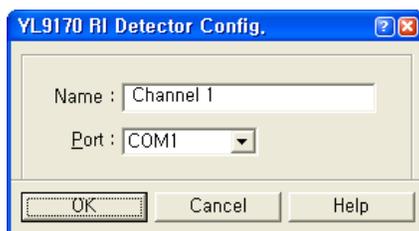
3-1-1. Installation of YL9170 RI detector

Open YL-Clarity software and select Configuration on the main window. On the system configuration window, click [ADD] button and select YL9170.



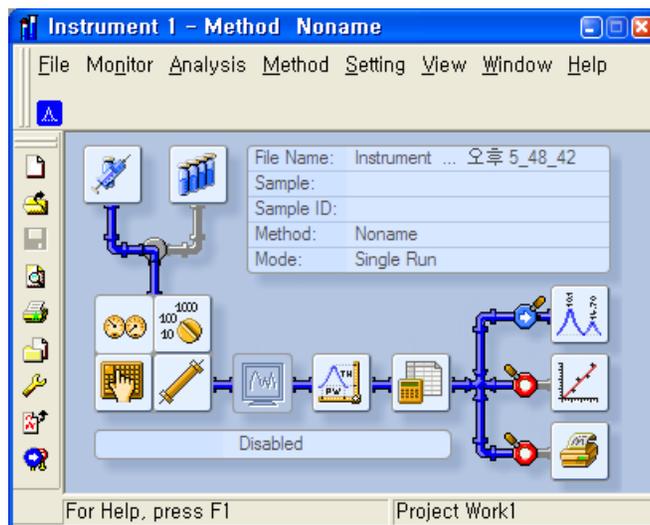


Double click YL9170 RI detector on the right window, and select the communication port that RI detector is connected.

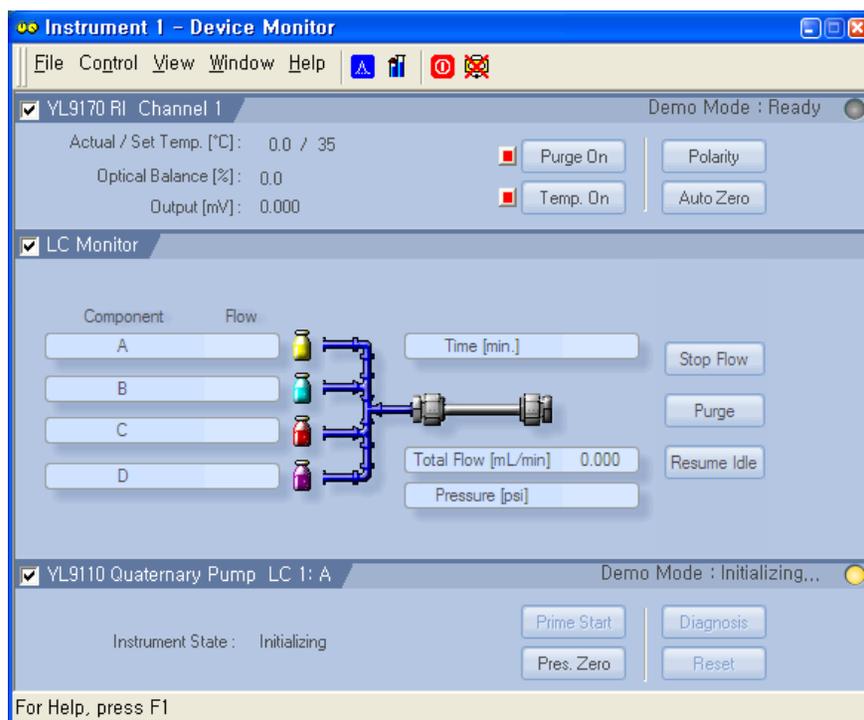


3-1-2. Device Monitor

After configure the RI detector on the System Configuration window, log in to open main control window. On the main control window, click Device monitor and then Device Monitor window pops up as below. In this window, you can control the RI Detector and monitor instrument status as like lamp on/off, wavelength selection, sampling rate, etc.



The Device Monitor shows the temperature of cell, optical balance and out signal as well.



Descriptions of control buttons

- Purge : Start/Stop flushing cells with solvents.
- Temp On/Off : Set-up On/Off for the cell temperature.
- Polarity : Reverse the polarity of signal.
- Autozero : Auto-zero the detector signal.

3-1-3. Method Setup

In the table below, edit programming of time table, and setup the detector status during idle state.

The screenshot shows the 'Method Setup' dialog box for the YL9170 RI Detector. The 'Select Detector' is set to 'Channel 1' and is 'Enabled'. The 'Setup' tab is selected, showing the following settings:

- Range[mV]: 10000.0
- Sample Rate[Hz]: 2
- Option: Autozero at a acquisition.
- Temperature: Off
- Enable Analysis: With any temp.
- Set 35 °C
- In range +/- 5 °C

At the bottom of the dialog, there are tabs for 'LC Gradient', 'LC', 'Measurement', 'Acquisition', 'Integration', 'Calculation', and 'Advanced'. Below the tabs are buttons for 'OK', 'Cancel', 'Apply', 'Report', and 'Help'.

Range : Set-up for the maximum limit of voltage range.

Sample Rate : Set-up for the sample rate.

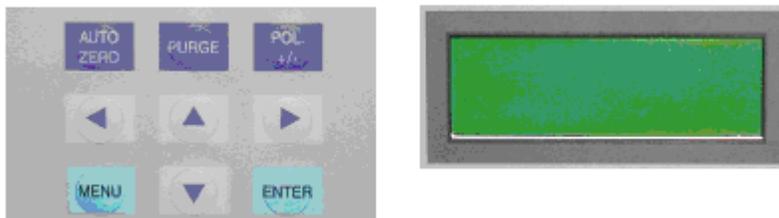
Autozero : Set-up for autozero at a data acquisition.

Temperature : Set-up for the cell temperature

Enable Analysis : Set-up for the enable analysis depending on cell temperature.

Chapter 4. Maintenance

For use of service and maintenance, there is a keyboard and LCD display inside of YL9170 RI detector.



LCD Display: The RI Detector has a four line display.
 The first line shows the actual temperature of the optical bank.
 The second line shows the optical balance in %.
 The third line shows the signal of the Integrator Output
 The fourth line shows the output range of the recorder.
 The recorder output is set to 1 by default.

Autozero: Use of this button autozeros the detectors signal.

Purge : Pressing this button, flow- and reference cell will be flushed with solvent. (LED is highlighted)
 Pressing the purge button again (LED is turned off). Solvent runs only through the sample cell.

Polarity: Can be used to reverse a signal.(LED is highlighted)

Enter: All changes in the menu must be confirmed with "**Enter**".

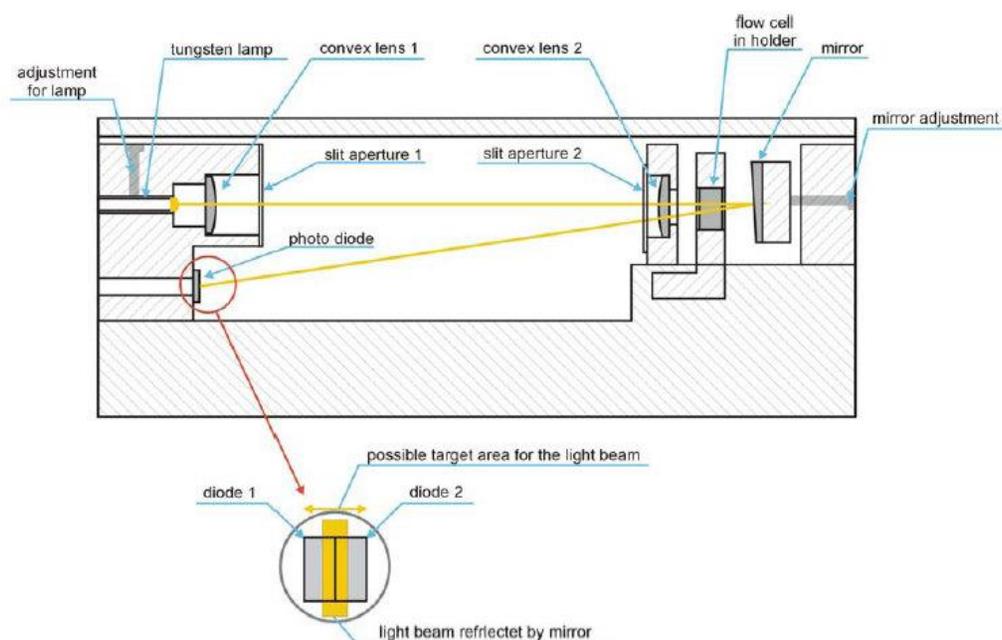
Menu: The fourth line in the display is the "MENU LINE". The Recorder range which is shown in this line, can be set by using the upper and lower arrows directly without pressing "**Menu**".

4-1. Adjusting the optical system

To perform the adjustment the sample and reference chamber of the flow cell have to be purged with distilled water. When both chambers contain the same liquid theoretically the same light intensity should reach the sample and reference side of the light sensor. In this case the optical balance should be zero.

The adjustment of the optical system is performed manually. During this procedure the position of the light beam on the photo sensor is changed by adjusting the mirror.

During the procedure you can follow the changes by observing the change of the detector signal or more detailed by observing the difference and sum voltage.



To adjust the optical system follow the steps listed below.

- Switch to purge mode, make sure the red purge LED is on.
- Flush both chambers of the flow cell with clean mobile phase for several minutes.
- Switch back to measuring mode, make sure the red purge LED is off.
- Put the hex-wrench into one of the opened hole on the rear side.
- Adjust the screws until the optical balance reaches a value of $0 \pm 20 \%$.

There are two adjustment screws for the mirror. Keep in mind that you might have to turn the other screw. Anyway you should turn the screws not more than 1/2 or 3/4 turns.

4-2. Lamp exchange and adjustment of the lamp

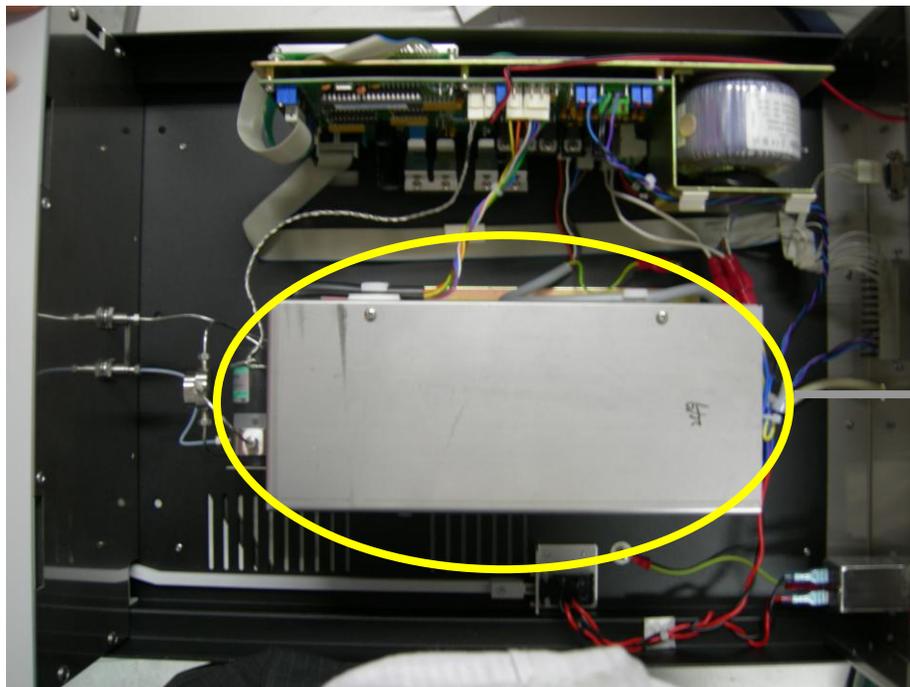
It might be possible that you need to re-adjust the light source due to some changes in the used light bulb due to the transport or you need to replace a burned out lamp. The adjustment of the light source is only possible when the housing is opened. To prepare for adjustment do the following steps :

- Switch off the instrument
- Unplug the main cable to prevent electrical shock

NOTE

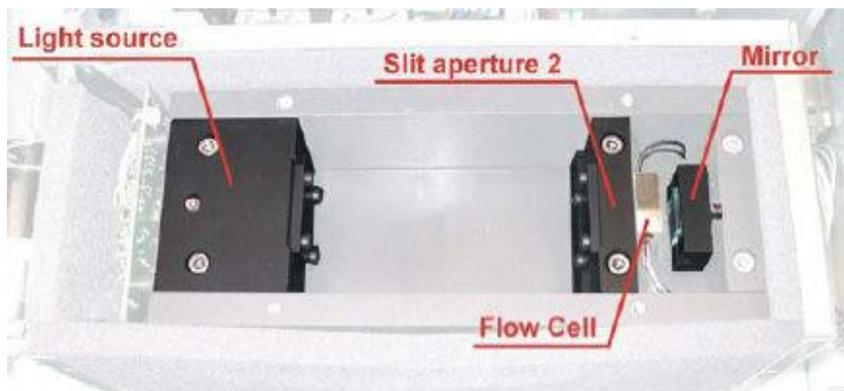
Some operations during adjustment have to be done when the instrument is in operation. So you need to reconnect the main cable and to switch on the YL9170 RI detector. When the instrument is operated with opened housing make sure that you DO NOT TOUCH any electrical component!

- 1) Open the outer housing of the optical bench
- 2) Open the inner black housing of the optical bench.



Optical bench of
YL9170 RI Detector

[Interior and optical bench of RI Detector]



[Inside of optical bench]

- 3) Loosen the holding screw of the light source and remove the lamp
- 4) Loosen the screw connector of the lamp's power supply at the pre-amplifier board
- 5) Place a new lamp in the holder and tighten the holding screw
- 6) Connect the power wires of the lamp with the screw connector on the pre-amplifier board
- 7) Connect the main cable to the detector and switch on the instrument

NOTE

The following steps need to be performed on the running instrument. There is the danger of electrical shock when touching electrical components.

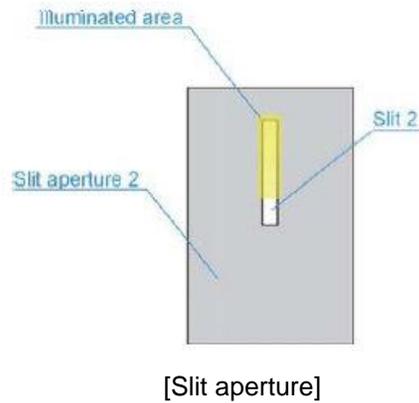
Use a voltmeter to check the lamp voltage at the test points on the circuit board. The label of this test point depends on the version of this board. (lampe and GND or LampeULmp and GND) The lamp voltage should be $3.3 \pm 0.3V$. If needed you can adjust this voltage by turning the potentiometer R19 at the main board.

In the following two steps the light bulb is positioned for optimal operation.

Step 1 : Place the lamp in the holder resulting in a sharp picture of the illuminated area at the slit aperture 2. Usually the metal cover of the light bulb ends with the holder, sometimes the metal cover stands about 1 or 2mm out of the metal block.

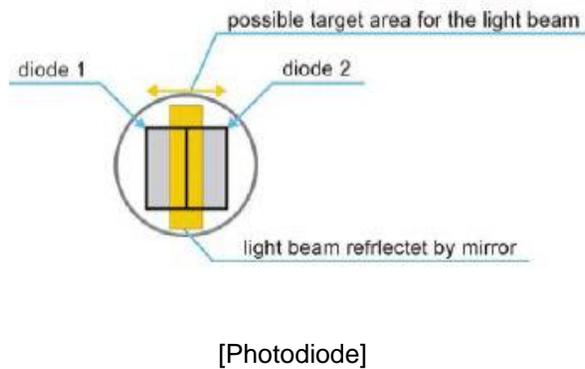
Step 2 : Turn the light bulb that the picture of the filament is parallel to the slit of the second slit aperture.

Make sure that only 2/3 of the slit are illuminated by the light source. This is illustrated in the following figure.



NOTE

The projected picture at the light sensor should be a sharp one. The full height of the photodiodes has to be illuminated by the light beam. This is shown in the following figure.



4-3. Checking Sum and Difference voltages

This chapter describes the check of the sum and difference voltages. These voltages result from different intensities reaching the sample side and the reference side of the light sensor.

To perform the check follow the steps listed below:

Make sure the instrument is switched on and both chamber of the flow cell are filled with distilled water without bubbles. In this case the optical balance should be $0 \pm 20.0\%$. To ensure a stable temperature the instrument should be switched on for several hours before performing this test.

- 1) Press the Purge button. Make sure the purge mode is activated.
- 2) Use a disposable syringe to press approx. 5mL distilled water through the flow cell.
- 3) The display will show the current detector signal.
- 4) Activate the service mode. The display will change to the following

```
Temp:                +0023.500 °C
Opt. Bal:            +0013.400 %%
Signal:              +0015.000
                    mV
ServMode >>        Menu,↵
```

- 5) Press the MENU button until the status line changes to ViewFine. Now press the ENTER button. The display will show four values.

```
Check  Source  Unit ↵
0044  1636   0840   0796
Signal:      + 250.668 mV
ServMode           Viewine
```

To find out which value is displayed in which column press the ENTER button. The display will change to the following view:

```
Check  Source  Unit
Diff  Summ   Smpl  Rfrn
Signal:      + 250.668 mV
ServMode           Viewine
```

- 1) Use a multimeter to check the lamp voltage at the test point on the circuit board. The lamp voltage should be set $3.3 \pm 0.3V$.
- 2) If the sum voltage is only a few millivolt the light bulb does not work. Check the power cables of the light source to make sure that they are connected properly.
- 3) The sum voltage should be in the region of $5000 \pm 500mV$. If this is not the case adjust the lamp voltage by turning the potentiometer R19 until the sum voltage is in the region of $5000 \pm 500mV$.
- 4) Due to the adjustment of the light intensity it is necessary to check the lamp voltage again.

4-4. Check and replacement of the valve

If the valve which is used to switch the path of the mobile phase in the normal mode and purge mode needs to be replaced follow the following steps:

- Switch off the YL9170 and unplug the main cable.
- Remove the stainless steel capillaries from the valve body.

NOTE

Remind the position of the capillaries for proper reconnection.

- 1) Loosen the control cable of the valve at the main circuit board.
- 2) Loosen the two screws holding the valve body.
- 3) Remove the valve body and replace with the new one.
- 4) Tighten the holding screws and reconnect the capillaries in the correct positions.
- 5) Connect the signal cable to the contact labelled **Valve** on the main board.
- 6) Start the pump to flush mobile phase
- 7) Check the capillary connections for any leakage.
- 8) Close the instrument's housing.

4-5. Checking and cleaning the flow cell

In some cases it might be necessary to clean the flow cell inside the YL9170 RI detector. You should try to wash away possible contaminations by purging both chambers of the flow cell with fresh mobile phase.

Possible reasons to open the optical bench for checking the flow cell:

- 1) Drop of the sum voltage below 4500mV. Make sure that your sample chamber is washed and filled with distilled water before checking the sum voltage.
- 2) Noise baseline
- 3) Constant drift of the baseline

- 4) To check the flow cell inside detector follow the steps listed below:
- 5) Switch off the instrument and unplug the power cable. Open the detector housing.
- 6) Open the outer housing of the optical bench.
- 7) Remove the heat insulation on top of the optical bench.
- 8) Open the inner(black) housing.
- 9) Loosen the two hex-screws holding the slit aperture 2 and remove the aperture carefully.
- 10) Use a flash light to check the flow cell for contaminants or damages. Make sure that no bubbles are inside the flow cell.
- 11) If there are contaminants in the flow cell purge it with a suitable solvent. If you are working with aqueous systems distilled water is convenient. If you use organic solvent try solvents like acetone, tetrahydrofuran or chloroform. After purging the cell with solvent to clean for a longer time you should purge it with your mobile phase.

NOTE

In the case of dangerous solvents make sure that they are disposed correctly.

NOTE

In the case of aqueous solvents it is possible that algae grow inside your system. For that reason it is not recommended to store the detector for a long time with aqueous solvents inside. For long time storage it is recommended to purge the flow cell with ethanol followed by air.

If possible we recommend the addition of a small amount of organic solvent(isopropanol or methanol).

If it is not possible to remove the contaminant from the cell it might be necessary to replace it with a new one.

After clean or replace the flow cell reassemble the detector. Perform a measurement to check the problem is solved.

4-6. The heating circuit of the Detector

In the following chapter the complete specification of the heating circuit is put together.

- 1) In the normal mode the temperature is displayed with one digit(e.g. 35.0 °C). If you want a more detailed information you can switch to the service mode where the temperature is displayed with three digit(e.g. 35.012 °C).
- 2) The temperature sensor will be detected automatically when the instrument is switched on.
- 3) If the heating is switched off the optical bench is operated at a temperature of about 6 °C above ambient temperature due to heat irradiated of electronic components.
- 4) The heat cartridge has an internal resistance of 750Ω
- 5) If the temperature sensor does not work or is not connected to the main board, the error message **noTS** will be displayed.
- 6) If the current temperature of the optical bench is below 9 °C or beyond 65 °C, the error message „!“ will show up and an acoustic signal occurs.
- 7) If the heating is not switched off properly by the firmware a thermal fuse will switch off the heating at a temperature of 72 °C.

YL9100 HPLC System

YL9180 ELS Detector

USER MANUAL



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Chapter 1. ELSD Principle of Operation

1-1. Introduction

ELSDs are near universal detectors, primarily used in High Performance Liquid Chromatography(HPLC) though they have been used successfully in other types of chromatography as well. Their principal requirement is that the analyte be less volatile than the mobile phase. An ELSD cannot detect highly volatile analytes. However, most analytes of interest are less volatile than the eluting solvents.

Evaporative light scattering detectors are replacing ultraviolet (UV) detection because they can detect most analytes, even those that do not absorb UV radiation, are stable during gradient elutions, and respond to the relative mass of the analyte -an important feature that is useful when detecting unknown materials. The ELSD is superior to the refractive index detector (RID), it can be used with gradient chromatography, it is not susceptible to ambient temperature changes, and it does not produce negative peaks, which can be difficult to quantify. The ELSD does not respond to the mobile phase disruption seen as solvent front peaks in the void volume with UV and RI detectors, so early eluting analytes can be easily quantified. Mass spectrometry (MS) detection is also a universal detector, but its high cost and complexity have kept it from being widely used. In fact, the operation requirements of MS closely match that of the ELSD. This allows the less expensive and complicated ELSD to be used as a method development detector for methods to be used on the MS systems.

1-1-1. Specifications

- 1) Dimensions: 250 x 460x 290mm (width X depth X height)
- 2) Weight: 10.5 kg
- 3) Display: 2 line x 20 character per line VFDL
- 4) User Interface: Two multi-function buttons
- 5) Operating Conditions:
 - Intended for indoors operation only. 60°F to 85°F and <90% R.H. non condensing
- 6) Evaporative Zone Temperature: Ambient to 120°C
- 7) Nebulization Chamber Temperature: Ambient to 60°C
- 8) Gas Requirement: 65psi \pm 5 psi Nitrogen or other inert gas
- 9) Gas Consumption: Approximately 2.5 SLPM
- 10) Liquid Flow Rate: 0.25 ml/min. to 3 ml/min.
- 11) Electrical Requirement: 120 V AC, 50/60 Hz or 240 V AC, 50/60 Hz 600 Watts
- 12) Wetted Materials: Stainless Steel, glass, Anodized Aluminum, Teflon

- 13) Light Source: 670 nM Laser Diode, <5 m W
- 14) Detector: Hermetically Sealed Photo-Diode/Operational Amplifier
- 15) Output Signal: 0-5 VDC
- 16) Communications: RS-232

1-2. Operation

The YL9180 ELSD employs a unique method of detection. The process involves the nebulization of the column eluent, transforming it into an aerosol cloud. As this cloud travels through a heated zone within the instrument, the more volatile mobile phase evaporates, leaving a smaller cloud of analyte particles. These particles pass through a beam of light, scattering some of the light, which is converted into an electronic signal.

1-2-1. Nebulization

Nebulization transforms the liquid phase leaving the column into an aerosol cloud of fine droplets. The size and uniformity of the droplets are extremely important in achieving sensitivity and reproducibility. The YL9180 ELSD uses a special concentric flow nebulizer and a constant flow of an inert gas to ensure a narrow droplet size distribution. Our nebulizer is constructed entirely from Teflon®, which accumulates deposits less than either glass or stainless steel.

To handle flow rates and mobile phases common in HPLC, all ELSDs need a way to divert part of the aerosol cloud to waste. YL9180 uses a patent pending **Thermo-Split technology**. Our **Thermo-Split** chamber combines a gentle bend with temperature controlled walls. When the aerosol exiting the nebulizer encounters a cool environment, it partially condenses into larger particles whose momentum carries them into the wall and down the drain.



Fig.1. With cooling, the particles condense and increase in size. They are carried into the walls of the bend and exit via the drain.



Fig.2. With heat, the particles decrease in size and all pass the bend in the Thermo-Split chamber.

With the Temperature control option installed, the temperature of the Spray Chamber may be elevated. As the aerosol traverses the chamber, it partially evaporates, shifting the particle size distribution low enough for essentially all the particles to negotiate the bend. These operating conditions may be useful for special applications. Under these conditions a majority of the aerosol particles pass through the chamber and are carried into the evaporative zone.

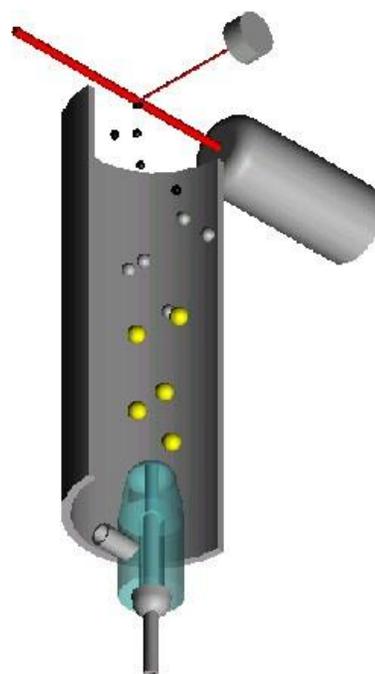
1-2-2. Evaporation

After passing through the nebulization chamber the aerosol cloud is propelled through the heated evaporation tube assisted by the carrier gas. In the evaporation tube the solvent is volatilized to produce particles or droplets of pure analyte.

The temperature of the drift tube is set at the temperature required to evaporate the solvent. The temperature is kept as low as possible to avoid particle shape distortion, evaporation of the analyte or when working with thermally sensitive compounds.

1-2-3. Detection

The particles emerging from the evaporation tube enter the optical cell, where the sample particles pass through the light emitted by a low power laser. The particles scatter the light, which is detected by a silicon photodiode located at a 90° angle from the laser. A light trap is located opposite the laser to collect the light not scattered by particles. The quantity of light detected is proportional to the solute concentration and solute particle size distribution. The photodiode produces a signal, which is sent to the outputs for collection.



Chapter 2. Unpacking and Installation

What you will need

In addition to the ELSD and its accompanying accessories you will need:

Gas Supply: Clean, dry, inert gas regulated to 65psi \pm 5 psi is needed for nebulization. Either argon or nitrogen is acceptable. Do not use gases that support combustion.

Exhaust Device: The carrier gas containing volatilized mobile phase and sample components will exit the ELSD. You should provide a means of removing this from the laboratory. The ELSD should be located close to a fume hood or other ventilation device. Tubing, fittings and tools to connect your HPLC system to the ELSD.

2-1. Unpacking

Unpack carefully and confirm that all the items are present. YL9180 ELSDs come in a high quality shipping container, engineered to avoid damage in transit. Save the shipping container and packaging for future shipments.

2-2. Connection

2-2-1. Power connection

The ELSD operates at either 120V or 240V, 50/60 Hz. Confirm that the YL9180 ELSD is configured for the correct voltage before plugging it into line voltage. If the power input module is set to the wrong voltage, unplug the power cord from the module. Open the fuse compartment by gently prying the cover from the module with a flat blade screwdriver. Remove the fuse block. Remove the voltage configuration card from the module by gently pulling with pliers or tweezers. Rotate the plastic voltage selector until the correct voltage appears on the side opposite the voltage selector. Replace the card and fuse block. Confirm that the correct voltage is indicated through the cover before securing the cover. Plug the modular power cord provided into the power input module on the back of the detector.

2-2-2. Exhaust

The ELSD exhaust is a 1/2" O.D. stainless steel tube. Use 1/2" I.D. tubing to connect to a fume hood

during operation. Alternately, connect tubing and direct to a cooled collection vessel for later disposal.

2-2-3.Communication

An output jack can be found on the back of the ELSD for connection to a chart recorder, computing integrator or computerized data system. The maximum signal output is 5V.

2-2-4.I/O Connections

Pin 1 and Pin 2: A contact closure output. Pins are open when an error conditions exists.

Pin 3 and pin 4: A contact closure input to turn on/off gas remotely. Gas control toggles with electrical continuity between these two pins. Maintain connectivity for a minimum of 500mS. A subsequent toggle command within 2 seconds may be ignored.

Pin 5 and 6: A TTL output for instrument status. A logical high indicates the detector is in standby.

Pin 7 and pin 8: A contact closure input to reset baseline remotely. Maintain connectivity for a minimum of 500mS. A subsequent toggle command within 2 seconds may be ignored.

2-2-5.Gas Connections

Connect a supply of clean, dry, inert gas regulated to 65psi \pm 5 psi to the GAS INLET port on the back of the unit. The internal gas regulator maintains the gas pressure at the factory set value displayed on the front panel.

2-2-6.Fluid Connections

Connect the outlet from your column to the LIQUID INLET on the front of the unit. The length and volume between the column outlet and the detector inlet should be kept as short as possible to avoid unnecessary band broadening.

2-2-7.Nebulization Chamber Drain

The instrument's internal "P" trap must be full during operation. Restricting the drain port with liquid is very important to ensure detection sensitivity. Pump mobile phase at 1mL/min without gas flow for 10 minutes or until you see liquid coming out of the drain. Alternately, you may introduce 10 mL of mobile phase into the drain using a squirt bottle or syringe.

Place the ¼" stainless steel drain adapter, provided in the accessory kit, on the drain port located on the front of the instrument. Direct the outlet of the tube to a collection vessel. Watch the liquid level in the vessel during operation and empty when full.

Chapter 3. Operation

3-1. Instrument Controls

The ELSD is controlled via two multi-function buttons on the front panel labeled POWER and AUTOZERO. The ELSD features a 2 line vacuum florescent display and a row of status lights to aid in the operation of the detector.

3-1-1. Status Indicator Lights

The status lights below the display provide a visual indication of the conditions of the ELSD.

From left to right the function of the status lights are:

1. Spray Chamber Heater: The yellow light is illuminated when the heater is active, blinks when the spray chamber is being controlled at the set temperature, and off when the spray chamber is cooling to a lower temperature.
2. Drift Tube heater: The yellow light is illuminated when the heater is active, blinks when the drift tube is being controlled at the set temperature, and off when the drift tube is cooling to a lower temperature.
3. Optic Cell Heater: The yellow light is illuminated when the heater is active, blinks when the optical cell is being controlled at the set temperature, and off when the optics cell is cooling to a lower temperature.
4. Laser power: The green light is illuminated when the laser is on and stable and off when the laser is off. The light blinks when laser power is low or unstable.
5. Gas Supply: The green light is on when the nebulizer gas pressure is within factory set limits, and the gas valve is open. The green light will blink if a pressure error exists. The green light will be off if the gas valve has been turned off.
6. Ready Condition: The green light is illuminated when all operating conditions are met and no error conditions exist.

3-1-2. Power Button

The POWER key is a multi function button. An audible beep is heard when the key is activated. When the instrument is off, press the POWER key to turn the ELSD on. Note that when the power cord is plugged in the POWER key is dimly illuminated from behind with a blue light. When the POWER key is used to turn on the ELSD, the backlighting expands to the AUTOZERO key. Once the ELSD is powered up the POWER button is used to view error conditions, enter standby mode, enter parameter modification

screens, and turn the power off.

Error condition exist when the ready light is not illuminated and when the * appears in the upper right corner of the display. Press the POWER button and a description of the error is displayed in the window. The display will cycle through all error conditions.

Pressing the POWER key once when no error conditions exist and twice if an error condition exists enters the stand-by count down screen. Stand-by mode should be used when the system is being shutdown. When the detector enters stand-by count down mode a timer is started. After the time had expired the gas solenoid is closed, shutting off the flow of the nebulization gas, the laser is turned off, and the heaters are disabled.

To turn the power off, press and hold the POWER key for 5 seconds. Wait at least 10 seconds before turning the instrument on, after it has been turned off.

3-1-3. Autozero Button

When the AUTOZERO button is depressed the ELS Detection signal will reset to about 20mV. An audible beep is heard when the key is activated.

3-1-4. Display Screens

The ELSD uses multiple screens to aid in the operation of the detector.

Start Up Screen (only visible on power up)

Home Screen

Error Screen (only visible when errors exist)

Standby Countdown Screen

Standby Screen

Spray Chamber Modification Screen

Drift Tube Modification Screen

Filter Type and Weight Modification Screen

Startup Screen

```

Program Loading
>>>>>>>>

```

The “>” moves from left to the right while the program is loaded. The process ends when “>” reaches the far right.

After loading the program, the system begins running diagnostics. Pressing the POWER key exits the

start-up process without completing the diagnostics.

Running Diagnostics Checking Laser Power

When the ELSD finishes the start-up process it enters the stand-by mode. Pressing the POWER key to exit and enter the HOME Screen.

ELSD is in Standby Mode Diagnostics Complete

Push PWR to exit Laser Power OK

Home Screen

ELS Detection	XX
20.000mV	20.0°C

The ELSD will automatically circulate the XX section to review operation condition.

XX = SC, DT, BLN, GAS, FLT or BFT, CL, GX

SC = Spray Chamber (setpoint: 25°C to 70°C; readout range: 25.0°C to 120.0°C)

DT = Drift Tube (setpoint: 25°C to 120°C; readout range: 25°C to 120.0°C)

BLN = Baseline reading (5000.000 – 0.000mV)

GAS = Gas (0.0 to 100.0psi)

FLT or BFT = Baseline Filter (OFF, weight 1 to 10, FLT or BFT)

CL = Calibration (20% to 200%)

GX = Gain (Normal or EDR)

FS = Full Scale (5V or 10mV)

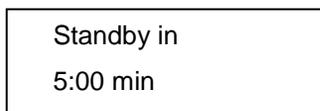
The displayed gas pressure is the nebulizer gas pressure, as controlled by the internal regulator. This display will increase with increased gas pressure supplied to the gas inlet on the rear panel. Do not exceed specified inlet pressure. Ensure that the incoming gas pressure is 65psi ± 5 psi.

If there is a "*" at the upper right corner, pushing POWER key will bring up a screen to explain the error.

Error Condition Screen

If any of the detector parameters are not met, the Ready status indicator will not illuminate and the * will appear in the upper right corner of the home screen. Press the POWER key for more information about the affected parameter.

Standby Countdown Screen



Pressing the POWER key once when no error conditions exist and twice if an error condition exists enters the standby countdown screen. It is recommended that the standby mode be activated when shutting down the detector. The timer allows enough time for the vapor to be expelled from the detector before the gas is turned off eliminating the possibility of condensation in the optics cell. After the time had expired the gas solenoid is closed, shutting off the flow of the nebulization gas, the laser is turned off, and the heaters are disabled. If there are no leaks between the gas source and the detector, the user will not need to return to the system after the standby sequence has started.

The timer default value is 5 minutes and is the recommended time. The time can be increased or decreased by using the AUTOZERO key. The up and down arrows (▲▼) next to Standby Mode alternate between up and down and indicate which way the set point will be modified.

To change the Standby delay time:

1. Push the AUTOZERO key when the appropriate arrow is illuminated (▲ to increase the time ▼ to decrease the time)
2. A beep is sounded each time the key is pressed.
3. Continue to press the key until the desired time is displayed.

Detector Parameter Modification Screens

To change the operating conditions, press the POWER key repeatedly to scroll through the screens until the desired modification screen is visible. If key is not pressed within 5 seconds, the display returns to the Home screen, except in the Standby countdown screen.

Spray Chamber Modification Screen

SPRAY CHAMBER	SET ▲▼
XX.X°C	XX°C

The current spray chamber temperature is displayed on the left; the current set point is displayed on the right. The up and down arrows (▲▼) next to SET alternate between up and down and indicate which way the temperature set point will be modified.

To change the Spray Chamber set point

4. Push the AUTOZERO key when the appropriate arrow is illuminated (▲ to increase the temperature ▼ to decrease the temperature)
5. A beep is sounded each time the key is pressed.
6. Continue to press the key until the desired temperature is displayed.
7. Push the POWER key to proceed to the Drift Tube modification screen.

Drift Tube Modification Screen

DRIFT TUBE	SET ▲▼
XX.X°C	XX°C

The current drift tube temperature is displayed on the left; the current set point is displayed on the right. The up and down arrows (▲▼) next to SET alternate between up and down and indicate which way the temperature set point will be modified.

To change the Drift Tube set point

1. Push the AUTOZERO key when the appropriate arrow is illuminated (▲ to increase the temperature ▼ to decrease the temperature)
2. A beep is sounded each time the key is pressed.
3. Continue to press the key until the desired temperature is displayed.
4. Push the POWER key to proceed to the Filter Weight modification screen.

Filter Type and Weight Modification Screen

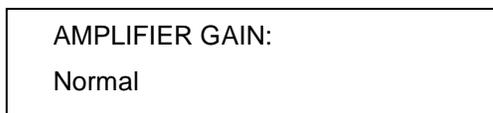
OFF, 1 to 10, FLT or BFT

The current filter weight is displayed. The up and down arrows (▲▼) alternate between up and down and indicate which way the filter weight will be modified.

To change the Filter Weight:

1. Push the AUTOZERO key when the appropriate arrow is illuminated (▲ to increase the filter ▼ to decrease the filter)
2. A beep is sounded each time the key is pressed.
3. Continue to press the key until the desired value is displayed.
4. Push the POWER key to proceed to the Amplifier Gain modification screen.

There are two filter types to choose from FLT and BFT. The filter type is selected at the top of the menu after weight 10. Select FLT or BFT and push the POWER KEY to enter. Then enter the filter menu again and selected the appropriate filter weight. Filter weight is the level of baseline noise filtration. OFF indicates no filtration. 10 is maximum filtration. In most cases, select BFT for baseline filtering. The FLT setting applies a RC filter to the entire signal. For high-speed chromatography, less than a 5 sec peak width, select BFT and turn the weight OFF. If the peak widths are 5 to 30 seconds use the BFT filter with a weight of 1 to 10. For peak widths greater than 30 seconds, select the FLT setting with a weight of 1 to 10. When the baseline filter is on, a dramatic operation condition change (e.g. turning on/off the HPLC pump) may upset the filter and cause baseline drifting. The baseline will stabilize again in a few minutes.

Detector Gain Page

Normal, Low

To change the Amplifier Gain:

1. Push the AUTOZERO key when the appropriate arrow is illuminated (▲ to increase the filter ▼ to decrease the filter)
2. A beep is sounded each time the key is pressed.
3. Continue to press the key until the desired value is displayed.
4. Push the POWER key to return to the Home screen.

There are two gain settings: Norm and Low. Use Normal setting for all analytical scale analysis. Use the Low setting for semi-preparative or preparative applications or when the signal exceeds the output at the normal setting.

3-2. Operating Conditions

3-2-1. Start Up Procedure

If this is the first time you have operated the detector, please refer to the Quick Start Guide. It is very important that you reproduce the QC tests found in the Quick Start Guide before beginning your analysis.

1. Make all connections; gas, liquid, power, communications, exhaust, and drain as described in Chapter 2.
2. Turn on the power to the ELSD. Allow the system to run through the start-up sequence and then push the POWER button to enter the Home screen.
3. Allow the Drift Tube and Spray Chamber temperatures to reach thermal equilibrium as indicated by the blinking of the corresponding status lights.
4. While waiting for the temperatures to reach the setpoints, start the HPLC Pump to deliver the mobile phase to the detector. Don't turn on the gas yet. Monitor the Thermo-Split drain on the front of the unit. It may take up to 10 minutes (flowrate=1.0ml/min) to see liquid coming out of the drain tube.
5. When liquid begins to flow out of the drain tube, turn on the regulated gas flow and gradually increase the gas pressure to 65psi \pm 5 psi. If you use an on-off valve to turn on the gas, make sure the upstream gas pressure is below 70psi before you turn on the valve. Gas pressure higher than 70psi may permanently damage the detector.
6. Begin data collection system and monitor the baseline. A "*" may be blinking at the upper right corner indicating the detector is not ready yet. You can depress POWER key to check what is not

ready.

7. Press the AUTOZERO key to rest the baseline to approx. 20mV. Repeated autozeros may be necessary until the baseline stabilizes.
8. When the baseline is stable (variation is less than 1.0mV when FS is set as 5V) and the “*” has disappeared the Ready status light will be illuminated.
9. Inject your standard or sample and begin analysis.

3-2-2. Shut Down Procedure

1. Depress the power key to enter the standby countdown screen. Stop the flow of mobile phase to the system.
2. Allow the detector to count down with the 5-minute timer.
3. If there are no leaks between your gas source and the ELSD, you do not have to turn the gas off at the source.

3-2-3. QC Test Conditions

Please refer to the QC report shipped with your ELSD for the exact conditions used to test your ELSD. The general conditions are:

1.0 ml/min of 50/50 Water/Methanol
Spray Chamber 30°C
Drift Tube 60°C
Filter 5
Injected Standard: 1000ng Sodium Benzoate in water

3-2-4. Choosing Operating Conditions

The drift tube temperature and the Thermo-Split spray chamber temperature are selected to provide the maximum detector response with minimum baseline noise. The temperatures are selected based on the solvent volatility and mobile phase flowrate. Some experimentation will be required to optimize the ELSD. When setting the ELSD temperatures for a new method, select 30°C for spray chamber temperature and 60°C for drift tube temperature. These temperatures should then be adjusted for the best signal to noise ratio during method optimization. For the best performance, a mobile phase that is highly organic and volatile requires an ambient or elevated spray chamber temperature and moderately high drift tube temperature. When highly aqueous or high boiling point organic mobile phases with the detector, the best performance will be at low spray chamber temperatures and moderate drift tube temperatures.

Thermo-Split Spray Chamber Temperature

The Thermo-Split Spray Chamber can operate from ambient to 60°C. The Spray Chamber temperature controls the vapor phase split ratio. For an easily evaporated mobile phase, the split ratio can be set low. To achieve this, the Thermo-Split chamber is heated. As the aerosol traverses the chamber, it partially evaporates, shifting the particle size distribution low enough for essentially all the particles to negotiate the bend. So, when highly organic mobile phases are used, the Thermo-Split chamber is used at ambient or elevated temperatures. Under these conditions a majority of the aerosol particles pass through the chamber and are carried into the evaporative zone.

For difficult to evaporate mobile phases, or high flow rates the split ratio needs be high so the Thermo-Split chamber is not heated. When the aerosol exiting the nebulizer encounters a cooler environment, it partially condenses into larger particles whose momentum carries them into the wall 16 and down the drain. By making the walls cooler, some of an aqueous stream can be diverted away from the evaporative zone.

Drift Tube Temperature

The drift tube temperature can be set from ambient to 120°C. The drift tube temperature is set at a temperature high enough to evaporate the mobile phase and not vaporize the analyte. A higher drift tube temperature may give result in a quieter baseline but smaller peak. The drift tube temperature should always be higher than the spray chamber temperature but only as high as needed to achieve a quiet baseline.

A drift tube temperature setting that is too high could vaporize the analyte and cause a loss of sensitivity. If the analyte is thermally labile, use a lower temperature to improve sensitivity.

However, there will be a point where the temperature is not high enough to evaporate the mobile phase and the increase in noise will negate the increase in signal. Optimize for the best signal to noise ratio.

Mobile Phase Flowrate

Mobile phase flowrate will also affect the optimum temperature set point. The higher the flowrate of an aqueous mobile phase, the lower the spray chamber temperature will need to be. High flowrates of volatile mobile phases may require a higher drift tube temperature.

Gradient Separations

For gradient separations, select the system temperatures required for the least volatile segment of the gradient program.

3-2-5. Mobile Phase Considerations

Selecting a solvent

High purity mobile phase solvents with low boiling points are recommended for use with the ELSD.

Solvents should be spectral or HPLC grade. Dirty or contaminated solvents will cause baseline noise and drift, blocked fluid paths, and a build up in the detector. All solvents used should have less than 1ppm of residue after evaporation and filtered to less than 0.45 μ m. Solvents can be evaluated by pumping them directly into the detector, and comparing the noise to other known solvents. We have found that not all HPLC grade solvents are acceptable for use with an ELSD. Preservatives commonly used in Tetrahydrofuran (THF), will increase the noise level. If unstabilized THF is used, ensure that it is fresh. THF can contain peroxides that can increase noise and are potentially explosive if taken to dryness.

Mobile Phase Flowrate and Composition

The recommended flowrate for the ELSD is 0.25mL/min to 3mL/min. The mobile phase flowrate will affect baseline noise. In general, more baseline noise will be generated by higher flowrate of a mobile phase. The ELSD will operate with common HPLC solvents that are volatile enough to form a vapor under the operating conditions. This includes common HPLC solvents such as water, methanol, acetonitrile, acetone, isopropyl alcohol, and THF. Normal phase solvents such as dichloromethane and hexane may also be used. Note that solvents with higher boiling points will generally result in more baseline noise. These should be used in limited percentages or at a lower flowrates.

Buffer Compatibility

The ELSD is not compatible with mobile phase modifiers that are not volatile, such as salts. Some modifiers are volatile and can be used. These include but are not limited to acetic acid, trifluoroacetic acid (TFA), formic acid, triethylamine, and ammonia. The concentration of buffer in the mobile phase should be as low as possible.

Column Pre-Treatment

Chromatographic columns may introduce particles into the mobile phase, which may lead to increased noise and blocked fluid paths. It is recommended that the chromatographic column be flushed with at least 10 column volumes before it is connected to the ELSD.

Chapter 4. Maintenance & Troubleshooting

The ELSD does not require regular user maintenance. There are no user serviceable components inside the ELSD. Opening the case will void your warranty. Please call your local agent before attempting any service.

The ELSD should be cleaned and calibrated once a year by a qualified technician.

For troubleshooting, get an assistance and helpful advice for the operation of your ELSD through your local agent.

YL9100 HPLC System

YL9181 ELS Detector

USER MANUAL



 YL INSTRUMENT CO., LTD.

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Chapter 1. ELSD Principle of Operation

1-1. Introduction

ELSDs are near universal detectors, primarily used in High Performance Liquid Chromatography(HPLC) though they have been used successfully in other types of chromatography as well. Their principal requirement is that the analyte be less volatile than the mobile phase. An ELSD cannot detect highly volatile analytes. However, most analytes of interest are less volatile than the eluting solvents.

Evaporative light scattering detectors are replacing ultraviolet (UV) detection because they can detect most analytes, even those that do not absorb UV radiation, are stable during gradient elutions, and respond to the relative mass of the analyte -an important feature that is useful when detecting unknown materials. The ELSD is superior to the refractive index detector (RID), it can be used with gradient chromatography, it is not susceptible to ambient temperature changes, and it does not produce negative peaks, which can be difficult to quantify. The ELSD does not respond to the mobile phase disruption seen as solvent front peaks in the void volume with UV and RI detectors, so early eluting analytes can be easily quantified. Mass spectrometry (MS) detection is also a universal detector, but its high cost and complexity have kept it from being widely used. In fact, the operation requirements of MS closely match that of the ELSD. This allows the less expensive and complicated ELSD to be used as a method development detector for methods to be used on the MS systems.

1-1-1. Specifications

- 1) Dimensions: 250 x 460x 290mm (width X depth X height)
- 2) Weight: 11.3 kg
- 3) Display: 2 Line x 20 Character per line VFD
- 4) User Interface: Four multi-function keys
- 5) Evaporative Zone Temperature: Ambient to 120°C
- 6) Thermo-Split™ Chamber Temperature: 10°C to 70°C
- 7) Liquid Flow Rate: 0.2mL/min to 5mL/min
- 8) Gas Requirements: 65psi ± 5 psi Nitrogen or other inert gas
- 9) Gas Consumption: ~ 2.5 SLPM
- 10) Operating Conditions:
 - Intended for indoor use only, 60°F to 85°F and <90% R.H. non condensing
- 11) Electrical Requirements: 120 VAC, 50/60 Hz or 240 VAC, 50/60 Hz; 600 watts
- 12) Wetted Materials: Stainless steel, glass, anodized aluminum, Teflon™

- 13) Light Source: 670 nm Laser Diode, <5mW
- 14) Detector: Hermetically sealed photo-diode/operational amplifier
- 15) Output Signal: 0 - 5 VDC
- 16) Communications: RS232

1-2. Operation

The YL9181 ELSD employs a unique method of detection. The process involves the nebulization of the column eluent, transforming it into an aerosol cloud. As this cloud travels through a heated zone within the instrument, the more volatile mobile phase evaporates, leaving a smaller cloud of analyte particles. These particles pass through a beam of light, scattering some of the light, which is converted into an electronic signal.

1-2-1. Nebulization

Nebulization transforms the liquid phase leaving the column into an aerosol cloud of fine droplets. The size and uniformity of the droplets are extremely important in achieving sensitivity and reproducibility. The YL9181 ELSD uses a special concentric flow nebulizer and a constant flow of an inert gas to ensure a narrow droplet size distribution. Our nebulizer is constructed entirely from Teflon®, which accumulates deposits less than either glass or stainless steel.

To handle flow rates and mobile phases common in HPLC, all ELSDs need a way to divert part of the aerosol cloud to waste. YL9181 uses a patent pending **Thermo-Split technology**. Our **Thermo-Split** chamber combines a gentle bend with temperature controlled walls. When the aerosol exiting the nebulizer encounters a cool environment, it partially condenses into larger particles whose momentum carries them into the wall and down the drain.



Fig.1. With cooling, the particles condense and increase in size. They are carried into the walls of the bend and exit via the drain.



Fig.2. With heat, the particles decrease in size and all pass the bend in the Thermo-Split chamber.

With the Temperature control option installed, the temperature of the Spray Chamber may be elevated. As the aerosol traverses the chamber, it partially evaporates, shifting the particle size distribution low enough for essentially all the particles to negotiate the bend. These operating conditions may be useful for special applications. Under these conditions a majority of the aerosol particles pass through the chamber and are carried into the evaporative zone.

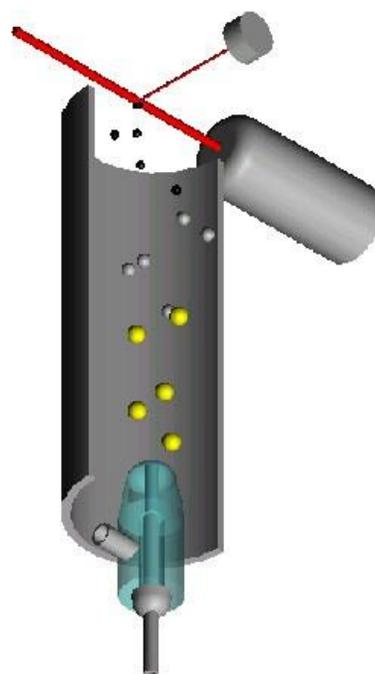
1-2-2. Evaporation

After passing through the nebulization chamber the aerosol cloud is propelled through the heated evaporation tube assisted by the carrier gas. In the evaporation tube the solvent is volatilized to produce particles or droplets of pure analyte.

The temperature of the drift tube is set at the temperature required to evaporate the solvent. The temperature is kept as low as possible to avoid particle shape distortion, evaporation of the analyte or when working with thermally sensitive compounds.

1-2-3. Detection

The particles emerging from the evaporation tube enter the optical cell, where the sample particles pass through the light emitted by a low power laser. The particles scatter the light, which is detected by a silicon photodiode located at a 90° angle from the laser. A light trap is located opposite the laser to collect the light not scattered by particles. The quantity of light detected is proportional to the solute concentration and solute particle size distribution. The photodiode produces a signal, which is sent to the outputs for collection.



Chapter 2. Unpacking and Installation

What you will need

In addition to the ELSD and its accompanying accessories you will need:

Gas Supply: Clean, dry, inert gas regulated to 65psi \pm 5 psi is needed for nebulization. Either argon or nitrogen is acceptable. Do not use gases that support combustion.

Exhaust Device: The carrier gas containing volatilized mobile phase and sample components will exit the ELSD. You should provide a means of removing this from the laboratory. The ELSD should be located close to a fume hood or other ventilation device. Tubing, fittings and tools to connect your HPLC system to the ELSD.

2-1. Unpacking

Unpack carefully and confirm that all the items are present. YL9181 ELSDs come in a high quality shipping container, engineered to avoid damage in transit. Save the shipping container and packaging for future shipments.

2-2. Connections

2-2-1. Power connection

The ELSD operates at either 120V or 240V, 50/60 Hz. Confirm that the YL9181 ELSD is configured for the correct voltage before plugging it into line voltage. If the power input module is set to the wrong voltage, unplug the power cord from the module. Open the fuse compartment by gently prying the cover from the module with a flat blade screwdriver. Remove the fuse block. Remove the voltage configuration card from the module by gently pulling with pliers or tweezers. Rotate the plastic voltage selector until the correct voltage appears on the side opposite the voltage selector. Replace the card and fuse block. Confirm that the correct voltage is indicated through the cover before securing the cover. Plug the modular power cord provided into the power input module on the back of the detector.

2-2-2. Exhaust

The ELSD exhaust is a 1/2" O.D. stainless steel tube. Use 1/2" I.D. tubing to connect to a fume hood

during operation. Alternately, connect tubing and direct to a cooled collection vessel for later disposal.

2-2-3.Communication

An output jack can be found on the back of the ELSD for connection to a chart recorder, computing integrator or computerized data system. The maximum signal output is 5V.

2-2-4.I/O Connections

Pin 1 and Pin 2: A contact closure output. Pins are open when an error conditions exists.

Pin 3 and pin 4: A contact closure input to turn on/off gas remotely. Gas control toggles with electrical continuity between these two pins. Maintain connectivity for a minimum of 500mS. A subsequent toggle command within 2 seconds may be ignored.

Pin 5 and 6: A TTL output for instrument status. A logical high indicates the detector is in standby.

Pin 7 and pin 8: A contact closure input to reset baseline remotely. Maintain connectivity for a minimum of 500mS. A subsequent toggle command within 2 seconds may be ignored.

2-2-5.Gas Connections

Connect a supply of clean, dry, inert gas regulated to 65psi \pm 5 psi to the GAS INLET port on the back of the unit. The internal gas regulator maintains the gas pressure at the factory set value displayed on the front panel.

2-2-6.Fluid Connections

Connect the outlet from your column to the LIQUID INLET on the front of the unit. The length and volume between the column outlet and the detector inlet should be kept as short as possible to avoid unnecessary band broadening.

2-2-7.Nebulization Chamber Drain

The instrument's internal "P" trap must be full during operation. Restricting the drain port with liquid is very important to ensure detection sensitivity. Pump mobile phase at 1mL/min without gas flow for 10 minutes or until you see liquid coming out of the drain. Alternately, you may introduce 10 mL of mobile phase into the drain using a squirt bottle or syringe.

Place the ¼" stainless steel drain adapter, provided in the accessory kit, on the drain port located on the front of the instrument. Direct the outlet of the tube to a collection vessel. Watch the liquid level in the vessel during operation and empty when full.

Chapter 3. Operation

3-1. Instrument Controls

The YL9181 ELSD is controlled via four multi-function keys on the front panel labeled MENU | POWER, ▲, ▼ and AUTOZERO. The ELSD features a 2 line vacuum florescent display and a row of status lights to aid in the operation of the detector.

3-1-1. Status Indicator Lights

The status lights below the display provide a visual indication of the conditions of the ELSD.

From left to right the function of the status lights are:

1. Spray Chamber Heater: The yellow light is illuminated when the heater is active, blinks when the spray chamber is being controlled at the set temperature, and off when the spray chamber is cooling to a lower temperature.
2. Drift Tube heater: The yellow light is illuminated when the heater is active, blinks when the drift tube is being controlled at the set temperature, and off when the drift tube is cooling to a lower temperature.
3. Optic Cell Heater: The yellow light is illuminated when the heater is active, blinks when the optical cell is being controlled at the set temperature, and off when the optics cell is cooling to a lower temperature.
4. Laser power: The green light is illuminated when the laser is on and stable and off when the laser is off. The light blinks when laser power is low or unstable.
5. Gas Supply: The green light is on when the nebulizer gas pressure is within factory set limits, and the gas valve is open. The green light will blink if a pressure error exists. The green light will be off if the gas valve has been turned off.
6. Ready Condition: The green light is illuminated when all operating conditions are met and no error conditions exist.

3-1-2. Status Alarm

The status alarm sounds when the detector status changes from not ready to ready or from ready to not ready. The alarm is 10 short beeps repeated every 30 seconds. Pressing the AUTOZERO key resets the alarm. The alarm can be disabled via the Control Page menu screen.

3-1-3. MENU|POWER Key

The MENU|POWER key is a multi-function key. An audible beep is heard when the key is activated. When the instrument is off, press the MENU|POWER key to turn the ELSD on. Note that when the power cord is plugged in the MENU|POWER key is dimly illuminated from behind with a blue light. When the MENU|POWER key is used to turn on the ELSD, the backlighting expands to the other keys. Once the ELSD is powered up the MENU|POWER key is used to view error conditions, cycle through the menu, accept changes to set points, enter standby mode, and turn the power off.

Error condition exist when the ready light is not illuminated and when the * appears in the upper right corner of the display. Press the MENU|POWER key and a description of the error is displayed in the window. The display will cycle through all error conditions.

Pressing the MENU|POWER key once, when no error conditions exist, or twice times, if an error condition exists, enters the menu screen.

Pressing the MENU|POWER key twice, when no error conditions exist, or three times, if an error condition exists, enters the stand-by count down screen. Stand-by mode should be used when the system is being shutdown. When the detector enters stand-by count down mode a timer is started. After the time had expired the gas solenoid is closed, shutting off the flow of the nebulization gas, the laser is turned off, and the heaters are disabled.

To turn the power off, press and hold the MENU|POWER key for 5 seconds. Wait at least 10 seconds before turning the instrument on, after it has been turned off.

3-1-4. Autozero Button

When the AUTOZERO key is depressed the ELS Detection signal will reset to about 20mV. An audible beep is heard when the key is activated. When the detector is in the menu mode, the AUTOZERO functions as an escape key, abolishing any changes made. The AUTOZERO key is also used to reset the status alarm.

3-1-5. Display Screens

The ELSD uses one of 4 screens to aid in the operation of the detector.

Startup Screen

Program Loading >>>>>>>>

The “>” moves from left to the right while the program is loaded. The process ends when “>” reaches the far right.

After loading the program, the system begins running diagnostics. Pressing the MENU|POWER key exits the start-up process without completing the diagnostics. If the MENU|POWER key is pressed before the completion of the diagnostics, the ELSD enters a special diagnostic mode.

Running Diagnostics Checking Laser Power

When the ELSD finishes the start-up process it enters the stand-by mode. Pressing the MENU|POWER key to exit and enter the HOME Screen.

ELSD is in Standby Mode Diagnostics Complete

Push PWR to exit Laser Power OK

Home Screen

ELS Detection XX 20.000mV 20.0°C

Pushing ▲ or ▼ keys will circulate XX section to review operation condition. SC, DT, OC and ET sections display their set points to the degree for 5 seconds when selected. Then they display the actual temperatures to the tenth of a degree.

Pushing MENU|POWER key will bring up the Menu screen. If the key is pushed within 5 seconds after an arrow key was pushed, the selection at XX will be displayed. Otherwise, the timer page will always be the first menu page displayed.

If there is a “*” at the upper right corner, pushing MENU|POWER key will bring up a screen to explain what’s wrong.

XX = SC, DT, OC, ET, RST, BLN, GAS, FLT or BFT, CL, GX, FS

- SC = Spray Chamber (setpoint: 10°C to 70°C; readout range: -10.0°C to 120.0°C)
- DT = Drift Tube (setpoint: 22°C to 120°C; readout range: -10.0°C to 120.0°C)
- OC = Optical Cell (setpoint: 22°C to 70°C; readout range: -10.0°C to 120.0°C)
- ET = Exhaust Tube (setpoint: 22°C to 70°C; readout range: -10.0°C to 120.0°C)
- RST= Reset baseline to about 20mV.
- BLN = Baseline reading (5000.000 – 0.000mV)
- GAS = Gas (0.0 to 130.0psi)
- FLT or BFT = Baseline Filter (OFF, weight 1 to 10, FLT or BFT)
- CL = Calibration (20% to 200%)
- GX = Gain (Normal or EDR)
- FS = Full Scale (5V or 10mV)

RST/BLN Screen

ELS Detection	RST
20.000mV	Baseline

ELS Detection	BLN
20.000mV	221.22

When RST is selected from the menu screen, it will display “RST Baseline” for 5 seconds. Pushing MENU|POWER key during this period will reset the ELS Detection signal to about 20mV. [Using the AUTOZERO key can also reset the baseline.] After 5 seconds, the display changes to BLN and displays the voltage offset by the autozero function. This value is important to note for each set of operating conditions. If the value is high, greater than 500mV, the mobile phase is not being evaporated completely and the temperature set points should be modified. Other contaminants in the system can also affect this reading.

Menu Screen

<<MENU>> [Spray Chamber Page]

Pressing the MENU|POWER key once, when no error conditions exist, or twice times, if an error condition exists, enters the menu screen.

Push ▲ or ▼ keys to select the menus.

Push MENU|POWER twice to return to the Home screen.

Available Menus

- [Spray Chamber Page] Sets Spray Chamber temperature.
- [Drift Tube Page] Sets Drift Tube, optical cell and exhaust tube temperatures.
- [Control Page] Sets Run/Standby mode, on/off Laser, on/off Alarm.
- [Timer] Sets duration of the shut down time.
- [Filter Page] Sets the type and weight of filter for noise filtration
- [Load Method Page] Loads one of nine methods.
- [Save Method Page] Saves a method
- [Calibration Page] Attenuates or amplifies the detector output
- [Detector Gain Page] Changes Gain
- [Full Scale Page] Changes Full Scale

Spray Chamber Page

SPRAY CHAMBER	SET
10.0°C	10°C

The reading under SPRAY CHAMBER is the current temperature. The reading under SET is the setpoint.

- Pushing ▲ or ▼ keys to change a setpoint at 1°C increment.
- Pushing MENU|POWER key to accept the change and go back to Home screen.
- Pushing AUTOZERO key to abolish the change and go back to Home screen.

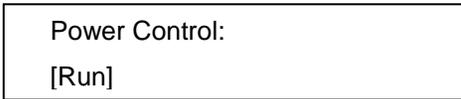
Drift Tube Page

DRIFT TUBE	SET
40.0°C	40°C

The reading under DRIFT TUBE is the current temperature. The reading under SET is the setpoint.
 The temperatures of the optical cell and the exhaust tube are controlled at the same temperature as that of DRIFT TUBE but their maximum temperature is limited.

- Pushing ▲ or ▼ keys to change a set point at 1°C increment.
- Pushing MENU|POWER key to accept the change and go back to Home screen.
- Pushing AUTOZERO key to abolish the change and goes back to Home screen.

Control Page



[Run], [Standby], [Laser Off] or [Laser On], [Turn off Sound] or [Turn on Sound]

Use ▲ or ▼ keys to select an action.

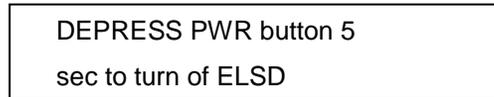
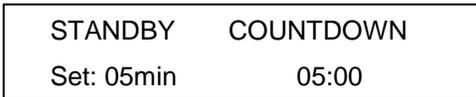
Push MENU|POWER key to take an action and go back Home screen.

Push AUTOZERO key to keep the current state and go back Home screen.

If the display reads Laser Off or Laser On, pushing MENU|POWER key will turn the laser off or on. The laser may take up to 30 minutes to stabilize when it has been turned off, and on again.

If the display reads Turn off Sound or Turn on Sound, pushing MENU|POWER key will turn the status alarm off or on. The status alarm sounds when the detector status changes from not ready to ready or from ready to not ready. The alarm is 10 short beeps repeated every 30 seconds. Pressing the AUTOZERO key resets the alarm.

Timer Page



1 min to 60 min

Use ▲ or ▼ keys to extend or reduce countdown interval.

Push MENU|POWER key to save the modified countdown interval but cancel countdown and return to home screen.

Push AUTOZERO key to cancel the countdown, keep the previous interval and go back Home screen.

The timer default value is 5 minutes and is the recommended time. The time can be increased up to 60 minutes or decreased down to 1min.

Filter Page

Filter Weight:

OFF, 1 to 10, FLT or BFT

Use ▲ or ▼ keys to select an action.

Push MENU|POWER key to take an action and go back Home screen.

Push AUTOZERO key to keep the current state and go back Home screen.

There are two filter types to choose from FLT and BFT. The filter type is selected at the top of the menu after weight 10. Select FLT or BFT and push the MENU|POWER KEY to enter. Then enter the filter menu again and selected the appropriate filter weight. Filter weight is the level of baseline noise filtration. OFF indicates no filtration. 10 is maximum filtration. In most cases, select BFT for baseline filtering. The FLT setting applies a RC filter to the entire signal. For high-speed chromatography, less than a 5 sec peak width, select BFT and turn the weight OFF. If the peak widths are 5 to 30 seconds use the BFT filter with a weight of 1 to 10. For peak widths greater than 30 seconds, select the FLT setting with a weight of 1 to 10. When the baseline filter is on, a dramatic operation condition change (e.g. turning on/off the HPLC pump), may upset the filter and cause baseline drifting. The baseline will stabilize again in a few minutes.

Load Method Page

LOAD METHOD: 0 1 2 3 4 5 6 7 8 9

Method 0 is the current setup of the unit. It will be loaded upon power up. Use the ▲ or ▼ keys to select a method number, pushing MENU|POWER key will apply the method and update the Method 0.

If the number selected has no method saved with, "Method not available" will be displayed. If the non-volatile memory IC chip is damaged, the same message will be displayed.

Save Method Page

SAVE METHOD: 0 1 2 3 4 5 6 7 8 9

Up to 9 methods can be saved. Use the ▲ or ▼ keys to select a method number and the

MENU|POWER key to save the current conditions to the selected method number. A method includes Spray Chamber Temperature Setpoint, Drift Tube Temperature Setpoint, Filter Weight, Calibration Factor, and Detector Gain.

Calibration Page

Calibration: 100%

20% to 200% in 1% increments

Use ▲ or ▼ keys to select an action.

Push MENU|POWER key to take an action and go back Home screen.

Push AUTOZERO key to keep the current state and go back Home screen.

This feature scales the detector output. This feature is useful to match individual detectors to each other for standardization within a lab, or set the full scale output to a value other than 5V or 10mV. At 100%, the signal is neither amplified nor attenuated.

Detector Gain Page

AMPLIFIER GAIN: Normal

Normal, EDR

Use ▲ or ▼ keys to select an action.

Push MENU|POWER key to take an action and go back Home screen.

Push AUTOZERO key to keep the current state and go back Home screen.

There are two gain settings: Norm and EDR. Use Normal setting for all analytical scale analysis. Analyte quantities from 10ng to 10,000ng can be quantified on Normal. "EDR" or Extended Dynamic Range provides a greater dynamic range than the Normal setting. Analyte quantities from 20ng to 200,000ng can be quantified in this gain level.

Full Scale Page

FULL SCALE: 10mV

Another selection is 5V. Use whichever setting is appropriate for your data collection system. Using the Calibration parameter can also modify the full scale. See Calibration page section for details.

Error Condition Screen

If any of the detector parameters are not met, the Ready status indicator will not illuminate and the * will appear in the upper right corner of the home screen. Press the MENU|POWER key for more information about the affected parameter.

Standby Countdown Screen



Pressing the MENU|POWER key twice, when no error conditions exist, or three times, if an error condition exists, enters the standby countdown screen. Press MENU|POWER and hold for 5 seconds if you wish to turn off the detector immediately. It is recommended that the standby mode be activated when shutting down the detector. The timer allows enough time for the vapor to be expelled from the detector before the gas is turned off eliminating the possibility of condensation in the optics cell. After the time had expired the gas solenoid is closed, shutting off the flow of the nebulization gas, the laser is turned off, and the heaters are disabled. If there are no leaks between the gas source and the detector, the user will not need to return to the system after the standby sequence has started. The system runs the diagnostic tests, then enters standby mode. Press MENU|POWER to exit standby and begin running the detector.

3-2. Operating Conditions

3-2-1. Start Up Procedure

If this is the first time you have operated the detector, please refer to the Quick Start Guide. It is very important that you reproduce the QC tests found in the Quick Start Guide before beginning your analysis.

1. Make all connections; gas, liquid, power, communications, exhaust, and drain as described in Chapter 2.
2. Turn on the power to the ELSD. Allow the system to run through the start-up sequence and then push the MENU|POWER button to exit standby and enter the Home screen.

3. Allow the Drift Tube and Spray Chamber temperatures to reach thermal equilibrium as indicated by the blinking of the corresponding status lights.
4. While waiting for the temperatures to reach the setpoints, start the HPLC Pump to deliver the mobile phase to the detector. Don't turn on the gas yet. Monitor the Thermo-Split drain on the front of the unit. It may take up to 10 minutes (flowrate=1.0ml/min) to see liquid coming out of the drain tube. Stop the pump
5. When temperatures reach setpoints, turn on the regulated gas flow and gradually increase the gas pressure to 65psi \pm 5 psi. If you use an on-off valve to turn on the gas, make sure the upstream gas pressure is below 70psi before you turn on the valve. Gas pressure higher than 70psi may permanently damage the detector. Re-start the pump.
6. A "*" may be blinking at the upper right corner indicating the detector is not ready yet. You can depress MENU|POWER key to check what is not ready.
7. When the detector is ready, begin data collection system and monitor the baseline.
8. Press the AUTOZERO key to rest the baseline to approx. 20mV. Repeated autozeros may be necessary until the baseline stabilizes.
9. When the baseline is stable (variation is less than 1.0mV when FS is set as 5V) and the "*" has disappeared the Ready status light will be illuminated.
10. Inject your standard or sample and begin analysis.

3-2-2. Shut Down Procedure

1. Depress the MENU|POWER key to enter the standby countdown screen. Stop the flow of mobile phase to the system.
2. Allow the detector to count down with the 5-minute timer.
3. If there are no leaks between your gas source and the ELSD, you do not have to turn the gas off at the source.

3-2-3. Choosing Operating Conditions

The drift tube temperature and the Thermo-Split spray chamber temperature are selected to provide the maximum detector response with minimum baseline noise. The temperatures are selected based on the solvent volatility and mobile phase flowrate. Some experimentation will be required to optimize the YL9181 ELSD.

When setting the ELSD temperatures for a new method, select 25°C for spray chamber temperature and 55°C for drift tube temperature. These temperatures should then be adjusted for the best signal to noise ratio during method optimization. For the best performance, a mobile phase that is highly organic

and volatile requires an ambient or elevated spray chamber temperature and moderately high drift tube temperature. When highly aqueous or high boiling point organic mobile phases with the detector, the best performance will be at sub-ambient spray chamber temperatures and moderate drift tube temperatures.

Thermo-Split Spray Chamber Temperature

The Thermo-Split Spray Chamber can operate from 10°C to 70°C. The Spray Chamber temperature controls the vapor phase split ratio. For an easily evaporated mobile phase, the split ratio can be set low. To achieve this, the Thermo-Split chamber is heated. As the aerosol traverses the chamber, it partially evaporates, shifting the particle size distribution low enough for essentially all the particles to negotiate the bend. So, when highly organic mobile phases are used, the Thermo-Split chamber is used at ambient or elevated temperatures. Under these conditions a majority of the aerosol particles pass through the chamber and are carried into the evaporative zone.

For difficult to evaporate mobile phases, or high flow rates the split ratio needs to be high so the Thermo-Split chamber is cooled. When the aerosol exiting the nebulizer encounters a cooled environment, it partially condenses into larger particles whose momentum carries them into the wall and down the drain. By making the walls suitably cold, 99+% of an aqueous stream can be diverted away from the evaporative zone.

As an example, the following data was collected with 90% water and 10% methanol at 1ml/min, a more difficult to evaporate mobile phase. The recommended conditions for this mobile phase are 15°C Spray Chamber and 45°C Drift Tube. Figure 3.1 shows the effect of Spray Chamber temperature on the resulting peaks when the drift tube temperature is held constant.

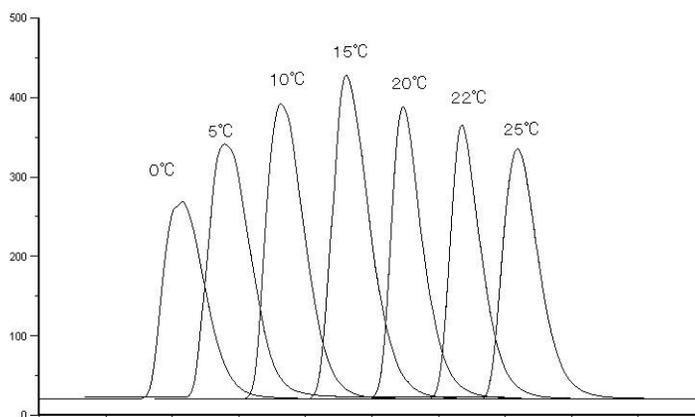


Figure 3.1: Spray Chamber Temperature Effects—Sub-ambient

As the Spray Chamber temperature increases from 0° to 15°C, more of the vapor phase goes to Drift Tube and not to the drain and the signal becomes larger. When the Spray Chamber is heated from 15°C

to 25°C the signal decreases because the vapor is partially evaporated in the spray chamber. When the SC temperature is higher than 30°C, more of the vapor is sent to the Drift Tube. A Drift Tube temperature of 45°C is not sufficient to evaporate the larger volume of vapor and results in more baseline noise.

When the mobile phase is changed to 90% Methanol and 10% water at 1 mL/min the recommended conditions are Spray Chamber 50°C and Drift Tube 70°C. In Figure 3.2, the drift tube was held constant at 70°C and the spray chamber was lowered. The signal height decreased as the spray chamber temperature decreased because more of the vapor was diverted from the Drift Tube and sent to the drain.

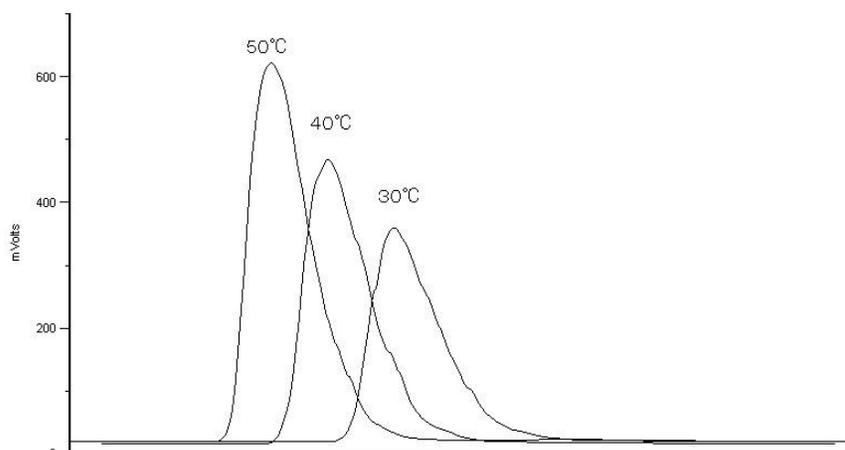


Figure 3.2: Spray Chamber Temperature Effects—Elevated

Drift Tube Temperature

The drift tube temperature can be set from ambient to 120°C. The drift tube temperature is set at a temperature high enough to evaporate the mobile phase and not vaporize the analyte. A higher drift tube temperature may give result in a quieter baseline but smaller peak. The drift tube temperature should always be higher than the spray chamber temperature but only as high as needed to achieve a quiet baseline.

In this example the mobile phase was 90% water and 10% methanol at 1ml/min and the spray chamber was held constant at 10°C. Figure 3.3 shows the effect of drift tube temperature on the signal.

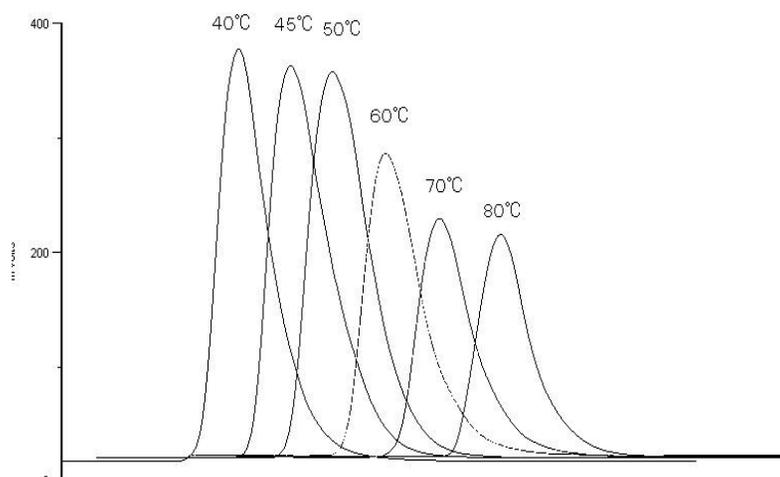


Figure 3.3: Drift Tube Temperature Effects

The effect of temperature on signal between 45°C and 50°C is small, but as the drift tube temperature increased above 60°C the signal height decreased.

A drift tube temperature setting that is too high could vaporize the analyte and cause a loss of sensitivity. If the analyte is thermally labile, use a lower temperature to improve sensitivity. However, there will be a point where the temperature is not high enough to evaporate the mobile phase and the increase in noise will negate the increase in signal. Optimize for the best signal to noise ratio.

Mobile Phase Flowrate

Mobile phase flowrate will also affect the optimum temperature set point. The higher the flowrate of an aqueous mobile phase, the lower the spray chamber temperature will need to be. High flowrates of volatile mobile phases may require a higher drift tube temperature. The YL9181 ELSD may perform best at sub-ambient spray chamber temperatures if the flowrate is extremely high, even for volatile mobile phases.

Gradient Separations

For gradient separations, select the system temperatures required for the least volatile segment of the gradient program.

Suggested Operating Temperatures

Solvent @ 1.0mL/min	Drift Tube Temperature, °C	Spray Chamber Temperature, °C
90/10 Water/METHANOL	45	15
90/10 Methanol/WATER	65	50
50/50 Methanol/Water	60	30
Acetonitrile	60	50
Methanol	60	50
Water	45	10

3-2-4. Mobile Phase Considerations**Selecting a solvent**

High purity mobile phase solvents with low boiling points are recommended for use with the ELSD.

Solvents should be spectral or HPLC grade. Dirty or contaminated solvents will cause baseline noise and drift, blocked fluid paths, and a build up in the detector. All solvents used should have less than 1ppm of residue after evaporation and filtered to less than 0.45µm. Solvents can be evaluated by pumping them directly into the detector, and comparing the noise to other known solvents. We have found that not all HPLC grade solvents are acceptable for use with an ELSD. Preservatives commonly used in Tetrahydrofuran (THF), will increase the noise level. If unstabilized THF is used, ensure that it is fresh. THF can contain peroxides that can increase noise and are potentially explosive if taken to dryness.

Mobile Phase Flowrate and Composition

The recommended flowrate for the ELSD is 0.25mL/min to 3mL/min. The mobile phase flowrate will affect baseline noise. In general, more baseline noise will be generated by higher flowrate of a mobile phase. The ELSD will operate with common HPLC solvents that are volatile enough to form a vapor under the operating conditions. This includes common HPLC solvents such as water, methanol, acetonitrile, acetone, isopropyl alcohol, and THF. Normal phase solvents such as dichloromethane and hexane may also be used. Note that solvents with higher boiling points will generally result in more baseline noise. These should be used in limited percentages or at a lower flowrates.

Buffer Compatibility

The ELSD is not compatible with mobile phase modifiers that are not volatile, such as salts. Some modifiers are volatile and can be used. These include but are not limited to acetic acid, trifluoroacetic acid (TFA), formic acid, triethylamine, and ammonia. The concentration of buffer in the mobile phase should be

as low as possible.

Column Pre-Treatment

Chromatographic columns may introduce particles into the mobile phase, which may lead to increased noise and blocked fluid paths. It is recommended that the chromatographic column be flushed with at least 10 column volumes before it is connected to the ELSD.

3-2-5. QC Test Conditions

Please refer to the QC report shipped with your ELSD for the exact conditions used to test your ELSD. The general conditions are:

1.0 ml/min of 50/50 Water/Methanol

Spray Chamber 30°C

Drift Tube 60°C

Filter 5

Injected Standard: 1000ng Sodium Benzoate in water

Chapter 4. Maintenance & Troubleshooting

The ELSD does not require regular user maintenance. There are no user serviceable components inside the ELSD. Opening the case will void your warranty. Please call your local agent before attempting any service.

The ELSD should be cleaned and calibrated once a year by a qualified technician.

For troubleshooting, get an assistance and helpful advice for the operation of your ELSD through your local agent.