

**Dynamic Light Scattering
Particle Size Analyzer
LB-550**

Preface

This instruction manual describes the operation of LB-550.

Be sure to read this manual before using the product to ensure proper and safe operation of the instrument.

Also safely store the manual so it is readily available whenever necessary.

Product specifications and appearance, as well as the contents of this manual are subject to change without notice.

■ Warranty and Responsibility

The product delivered to you is covered by HORIBA's warranty for a period of one (1) year.

If any malfunction or damage attributable to HORIBA's responsibility should occur during this period, necessary repairs or replacement of parts shall be made free of charge by HORIBA.

The warranty does not cover the following:

- Any malfunction attributable to improper operation
- Any malfunction attributable to repair or modification by any party not authorized by HORIBA
- Any malfunction attributable to the use in an improper environment
- Any malfunction attributable to natural disasters, or accidents or mishaps not involving HORIBA
- Any malfunction attributable to violation of the instructions in this manual
- Any malfunction attributable to operations in the manner not specified in this manual
- Any deterioration in appearance attributable to corrosion, rust, and so on.
- Consumables and replacement of consumables
- Products of other companies

HORIBA shall not be liable for any damages resulting from any malfunctions of this product, any erasure of data, or any other uses of this product.

■ Trademarks

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Generally, other company names and brand names are either registered trademarks or trademarks of the respective companies.

■ Copyrights

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HANDLE WITH CARE: FDA & FCC Regulations Require Proper Labeling.

FDA WARNING

→ This instrument is a Class 1 laser product complying with CFR 21, Chapter 1, Paragraph J.

→ The laser specifications are as follows:

Laser diode: Model NDL3321ST

Output: 5 mW

Wavelength: 650 nm

→ Although this equipment is encased in a protective housing to prevent leakage of the laser beam, there is a danger of exposure to laser radiation if the protective housing is removed. There is no need to open the protective housing during either ordinary operation or maintenance.

→ This warning plate has been attached to the instrument:

CAUTION-Laser radiation when open and interlock defeated.
DO NOT STARE INTO BEAM OR VIEW DIRECTLY WITH OPTICAL INSTRUMENTS.

FDA Label

Certification/Identification Label
This product complies with 21 CFR Chapter 1 Subchapter J.
Manufacturer of record: Horiba Instruments, Inc.
Address: 17671 Armstrong Avenue, Irvine, California 92614
Phone: 949-250 4811
*1 Date manufactured:
*2 Model:
*3 Serial Number

*1: MM.DD.YYYY

*2: LB-550

*3: 10-digit numerals

FCC WARNING

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and radiates radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

FCC Label

THIS DEVICE COMPLIES WITH PART 15 OF THE FCC RULES. OPERATION IS SUBJECT TO THE FOLLOWING TWO CONDITIONS : (1) THIS DEVICE MAY NOT CAUSE HARMFUL INTERFERENCE, AND (2) THIS DEVICE MUST ACCEPT ANY INTERFERENCE RECEIVED, INCLUDING INTERFERENCE THAT MAY CAUSE UNDESIRED OPERATION.

INSTALLATION ENVIRONMENT

This product is designed for the following environment:

- Installation Categories II
- Pollution degree 2

LIMITATION OF LIABILITY FOR DAMAGES

HORIBA will not accept responsibility for damage or malfunction that may occur as a result of operation or situation not recommended in this manual. HORIBA shall not be liable for Customer's incidental, consequential or special damages, or for lost profits or business interruption losses, in connection with the operation of the Manufactured Parts, CPU hardware, disk drives or Software.

CE MARKING



LB-550 Series conforms with the following directives and standards

Directives the EMC Directive 89/336/EEC in accordance with the Article 10(1) of the directive, the Low Voltage Directive 73/23/EEC

Standards EN61326:1997+A1:1998+A2:2001 (EMISSION: Class B, IMMUNITY Category: Minimum Requirements)
EN61010-1:1993+A2:1995
EN61000-3-2:2000
EN61000-3-3:1995+A1:2001

For Your Safety Policy

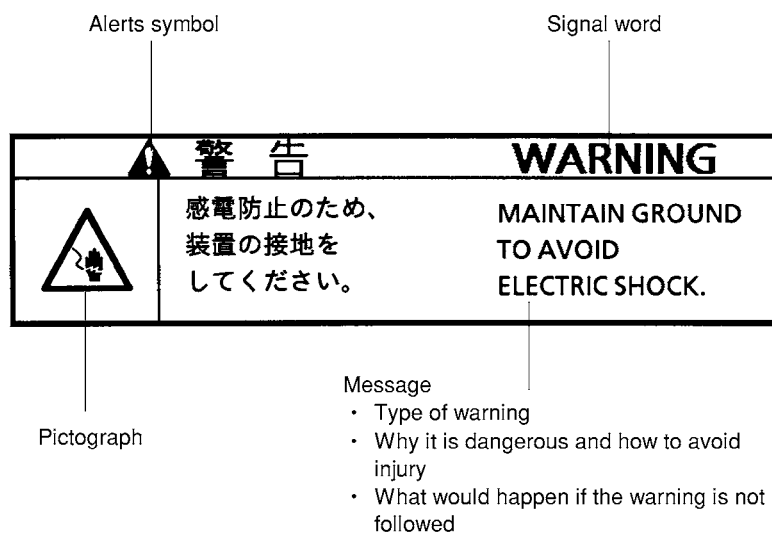
■ Warnings and Warning Labels

We arrange warning labels on our products, and describe notes and cautions in this manual. Make sure to follow these instructions for your safety.

● Warning Labels

Each warning label consists of the following parts:
an alert symbol, pictograph, signal word, and messages.

See the following pages for the labels and their locations on the product.



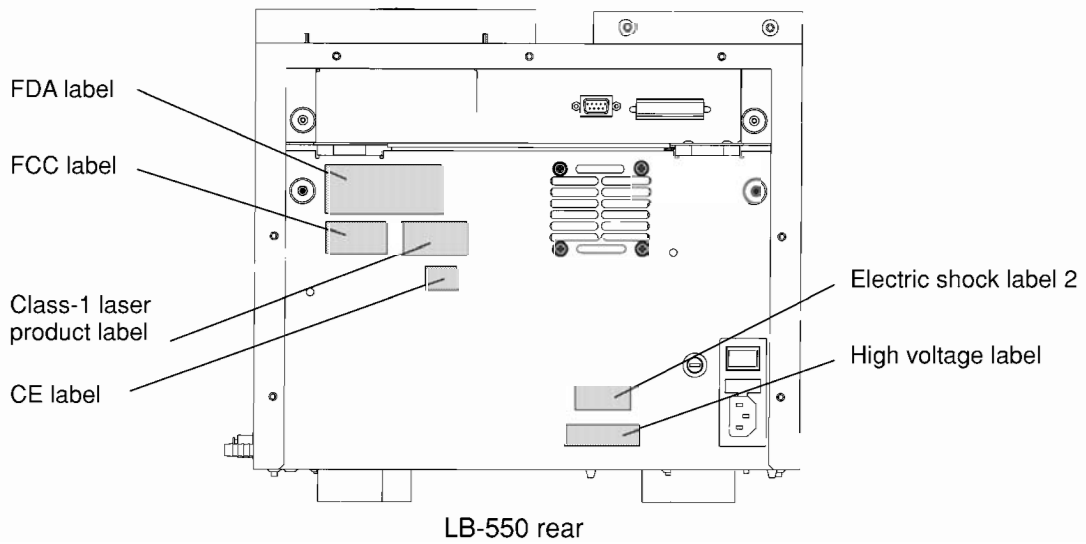
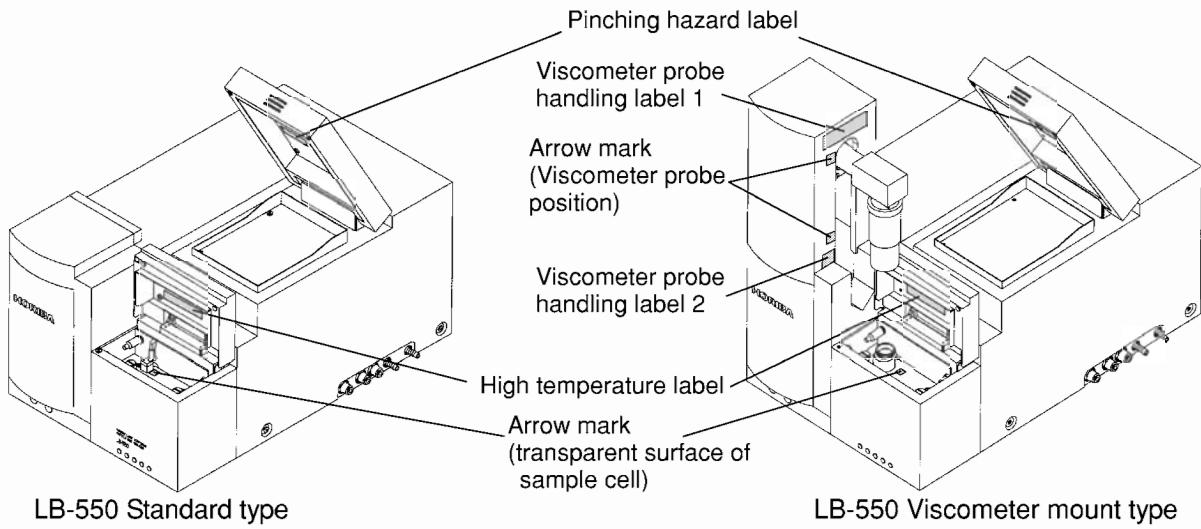
Meanings of the signal words are as follows:

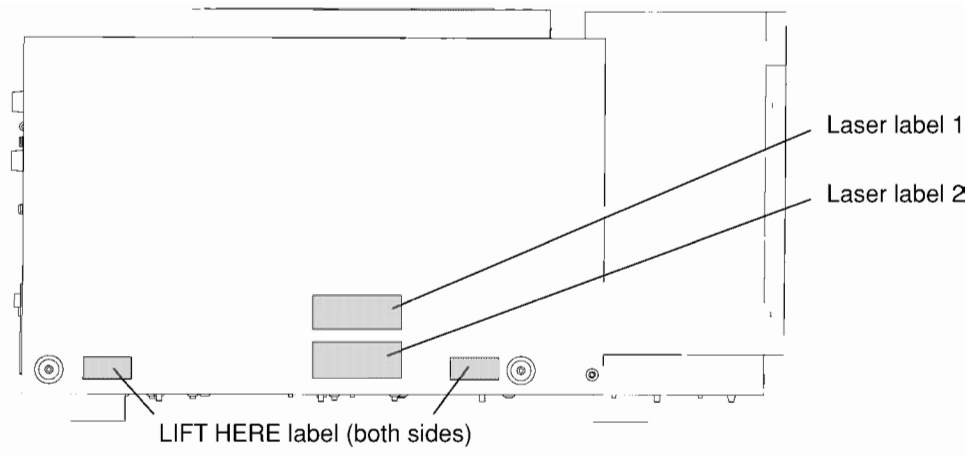
- **DANGER:**
This indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury. This signal word is to be limited to the most extreme situations.
- **WARNING:**
This indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.
- **CAUTION:**
This indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. It may also be used to alert against unsafe practices.

■ Labels and Location

● Label Location

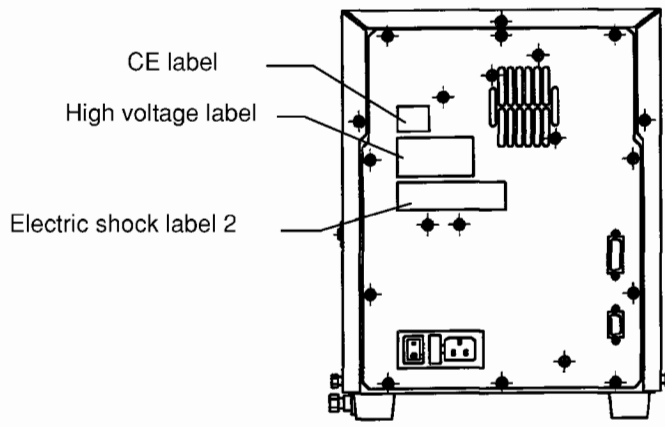
Dynamic light scattering particle size analyzer LB-550



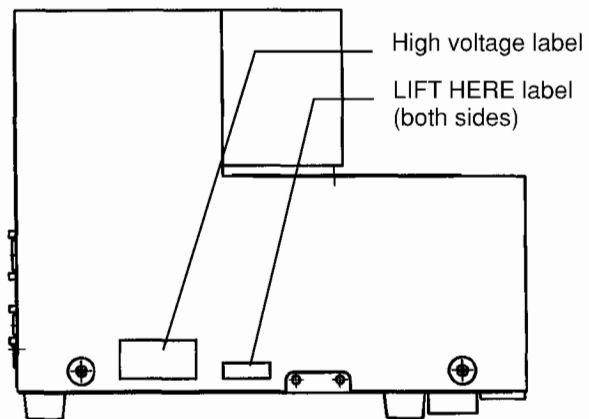


LB-550 side

Preparation unit LY-501

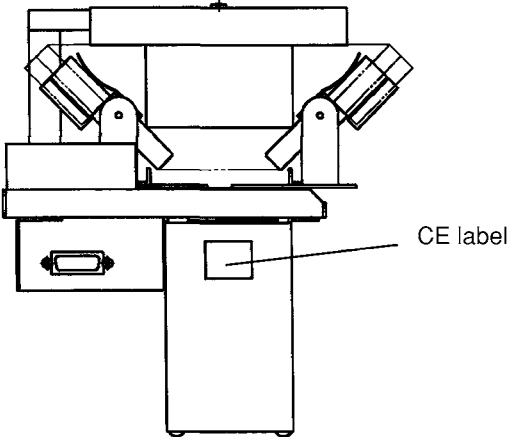


LY-501 rear

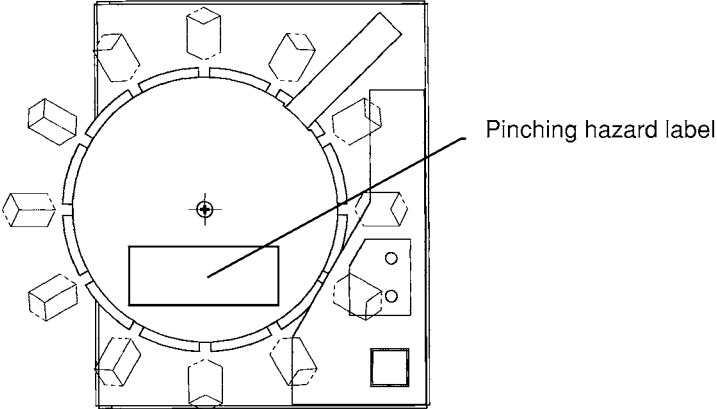


LY-501 side

Auto-Sampler LY-502



LY-502 front



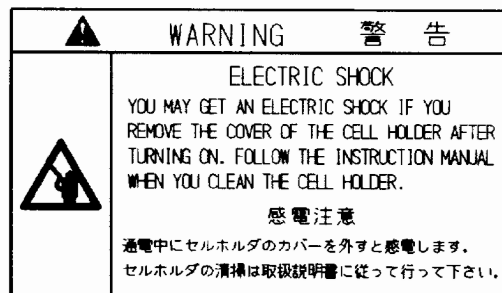
LY-502 top

- Labels

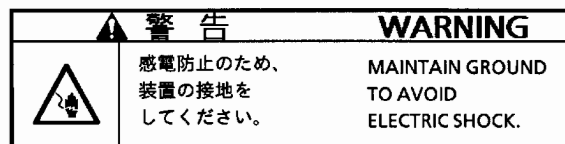
High voltage label



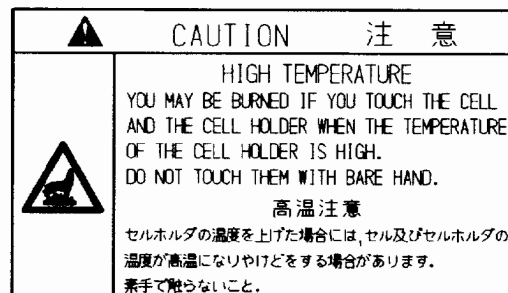
Electric shock label 1



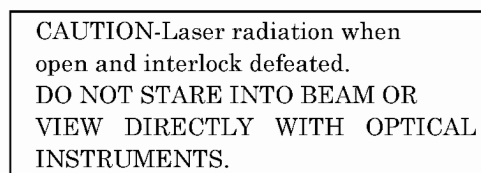
Electric shock label 2



High temperature label



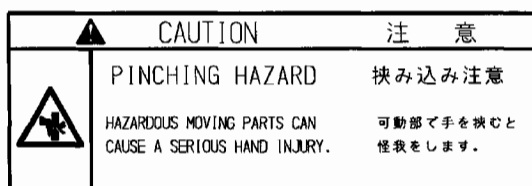
Laser label 1



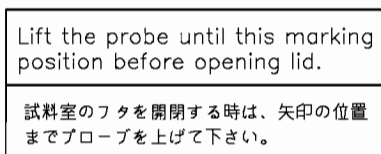
Laser label 2



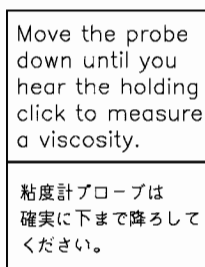
Pinching hazard label



Viscometer probe handling label 1



Viscometer probe handling label 2



Class-1 laser product label



- **Other labels**

CE label



FCC label

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Phone: 949-250 4811
*1 Date manufactured:
*2 Model:
*3 Serial Number

*1: MM.DD.YYYY
*2: LB-550
*3: 10-digit numerals

LIFT HERE label



Arrow mark (transparent surface of sample cell/ Viscometer probe position)



■ Precautions on Safety and Use

Please observe the following instructions to ensure that measurements are taken safely and accurately using the LB-550.

The operating site should meet the requirements stated in "Installation conditions" (page I-11).

- Do not shake the LB-550, nor give a shock to it.
- Be sure to ground the LB-550 using the dedicated power cord (with a 3-pin plug).
- The LB-550 uses precision optical parts, a laser light source and a high-voltage power source. Therefore, it should be handled carefully.
- After turning ON the power, warm up the LB-550 for about 20 minutes until the laser light has become stable.
- Do not remove any parts from inside the LB-550, nor modify the unit.
- If a volatile dispersant fluid is used, it may catch fire due to a spark from the motor or any other similar component. The LB-550 is not designed as an explosion-proof unit.
- If a dangerous dispersant fluid or sample is used, take care for its spillage and drainage.
- It is inhibited that the LB-550 is operated in such a manner as is not instructed in this instruction manual. This may cause the LB-550 to deteriorate and lead to any damage of its parts.
- Avoid such handling as may damage the surface of the cell. For example, do not:
 - Leave the cell immersed in detergent;
 - Immerse the cell on acidic or alkalic solution; or
 - Scrub the surface of the cell with anything hard
- During use of both the standard type and viscometer mount type, let the flow rate of the cooling water keep approximately 0.4 L/min.
- Back up all the existing software on your personal computer in accordance with the instruction manual that came with your personal computer. HORIBA is not responsible for backing up software.
- Tubes have their respective service life according to the type. For details, refer to "Service Life of Components" (page I-31).
- Do not touch the equipment while the auto-sampler is in operation. It is dangerous.
- When an acid, alkaline or organic solvent is used in the pre-processing unit, use the tube material suitable for each solvent. A PharMed tube (made by Masterflex) and Fluran tube (made by Norton) are included with the product.

Contents

Part I Getting Started

OVERVIEW	I-1
OPERATION	I-12
MAINTENANCE	I-17
APPENDIX	I-23

Part II Operation

START-UP AND SHUTDOWN	II-1
SETTINGS	II-2
MEASUREMENT	II-9
SAVE	II-12
CLEANING THE CELL	II-13
MEASUREMENT RESULT	II-15
SAMPLING & DISPERSION	II-18
VALIDATION	II-22
LIST OF THE REFRACTIVE INDEX VALUES	II-23
OPTIONAL UNIT	II-27

Part III Software

INSTALLING AND STARTING LB-550	III-2
PULL-DOWN MENUS	III-15
SYSTEM ADMINISTRATION	III-103
PRINT	III-113
SPECIFICATIONS	III-126
ADDITIONAL	III-128

Part I Getting Started

- OVERVIEW..... I-1
 - Description of CommunicationI-2
 - Names and FunctionsI-3
 - Installation ConditionsI-11
- OPERATION..... I-12
 - Preparation.....I-12
 - Start-up and ShutdownI-16
- MAINTENANCE..... I-17
 - Daily Care.....I-17
 - Cleaning The Cell.....I-17
 - Cleaning The Cell HolderI-18
 - Exchange of Pump TubeI-19
 - Troubleshooting.....I-20
 - Replacing The Fuse.....I-21
 - Other PrecautionsI-21
 - StorageI-21
- APPENDIX..... I-23
 - Specifications.....I-23
 - Dimensional OutlineI-26
 - AccessoriesI-30
 - Service Life of ComponentsI-31
 - Measurement PrinciplesI-32

OVERVIEW

The measurement of particle size distribution is used for fundamental studies and quality control in production processes in many diverse fields such as fine ceramics, cement, pharmaceuticals, metal powders, industrial minerals and ores, explosives, solid fuels, food, drugs and beauty care emulsions, micelles, polymers, coatings and adhesives, pigments and dyes, carbon black and other mineral additives, and fillers used in the rubber, plastics and paper industries.

Particle sizes ranging from 1 nm to 6 μm are measured. Measurement time is adjustable, but usually 2 minutes is sufficient.

The LB-550 comes in two types: the standard type and the viscometer mount type.

- The standard type and the viscometer mount type allow you to control the cell holder at any specific temperature in a range between 5°C and 70°C. When the specified temperature is under 20°C or the temperature is lowered for 20°C or more range, send approximately 0.4 L/min of cooling water controlled at a temperature of 25°C through the pipe for circulated water, which is required for improving the cooling efficiency of the electronic cooler.
The cell may not be cooled again in the following cases.
 - The specified temperature is under 20°C or the temperature is lowered for 20°C or more range without using circulated water.
 - The water at the temperature of 25°C or higher is used for circulated water.
- When temperature is controlled under 20°C, feed nitrogen gas of approximately 0.2 kPa from the side joint so that condensation does not occur on the cell surface. And make sure to keep good ventilation during the use of nitrogen gas for your safety.

For the connection section for circulating water, refer to "Sample Chamber" (page I-8).

LB-550 Getting Started

Description of Communication

The LB-550 performs communications with a personal computer via the SCSI port. It is controlled by the personal computer to take measure samples and send data.

When the optional units, Preparation Unit LY-501 or Auto-Sampler LY-502, are connected, the LB-550 and LY-501 are communicated via the RS-232C and the LY-501 and LY-502 are communicated via I/O.

To use the pH meter when the LY-501 is connected, set the COM port to be used for communications with the pH meter at the system administration on the LB-550 software. Then, connect the set COM port at the rear of the personal computer and the pH meter with a furnished RS-232C cable.

The viscometer unit (LY-554) can be mounted on LB-550. The LB-550 and LY-554 are communicated via the RS-232C.

Note:

- To use the pH meter connected to the personal computer, LY-501 must be connected. To connect the pH meter with the personal computer, a separately available adapter is necessary. For details, refer to the operation manual of the pH meter in your possession.

Note:

- Mounting the viscometer unit, LY-554, needs shop work. The LB-550 should be returned to us for remodeling the standard type into the viscometer mount type.

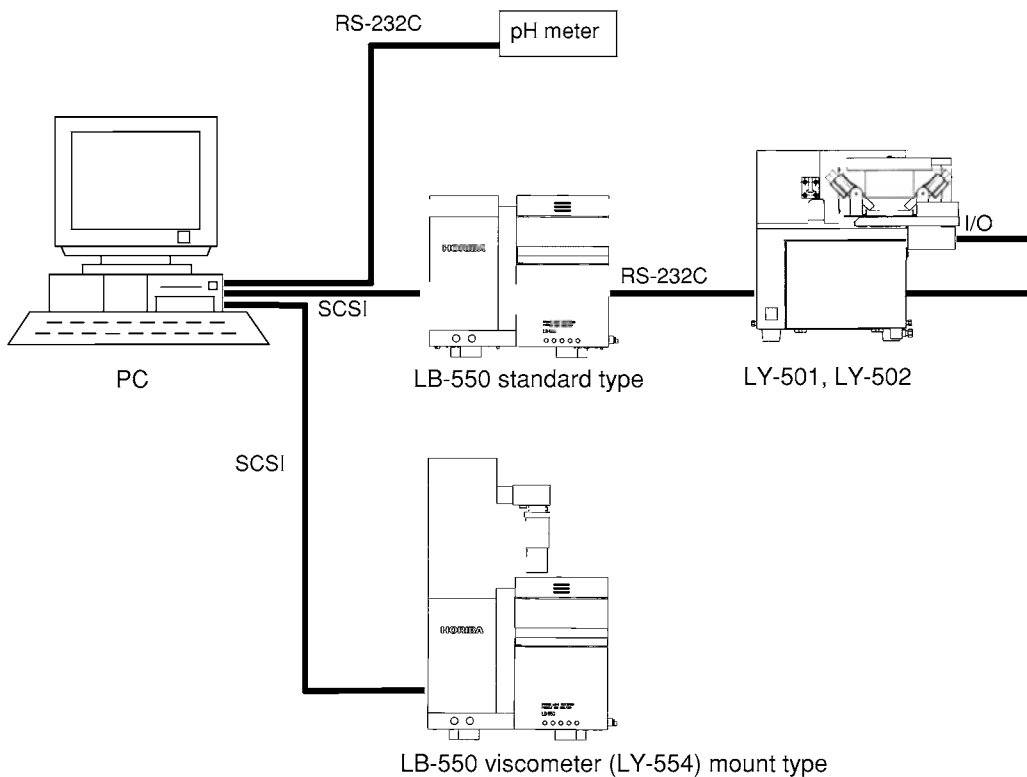


Figure 1: Communication System

Note:

- Consult us in case of connecting the pH meter to the LB-550.

Names and Functions

Front Panel of Main Unit

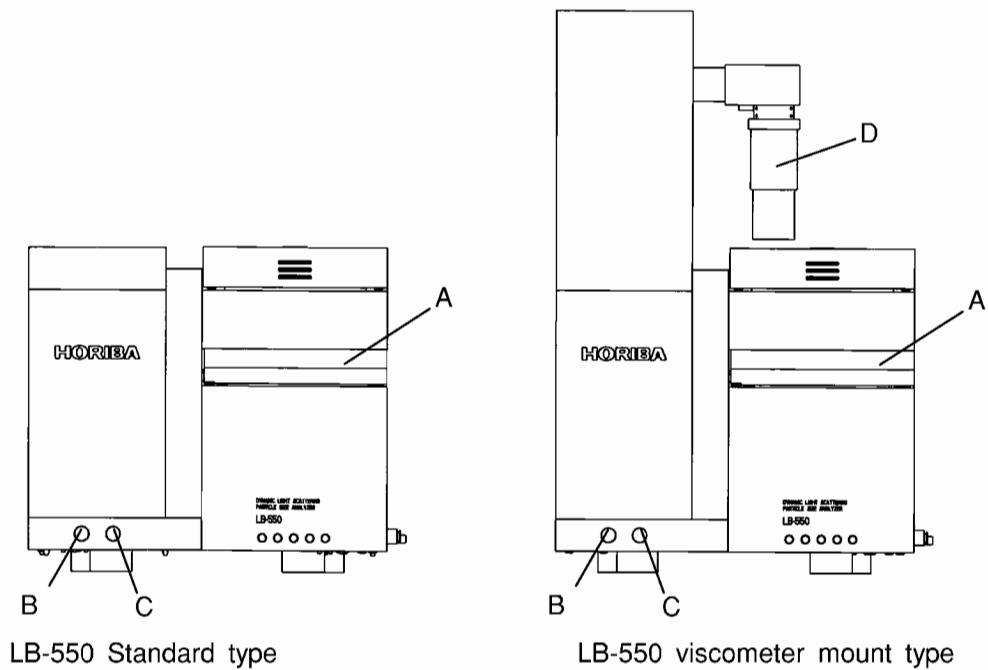


Figure 2: Front View

A: Upper door of sample chamber:

Opened and closed when the cell is changed.

B: Power indicator:

Lights up when the power switch is turned ON.

C: Laser indicator:

Lights up when the laser is activated.

D: Viscometer probe:

Move it manually in the downward direction when viscosity is measured.

LB-550 Getting Started

Rear Panel of Main Unit

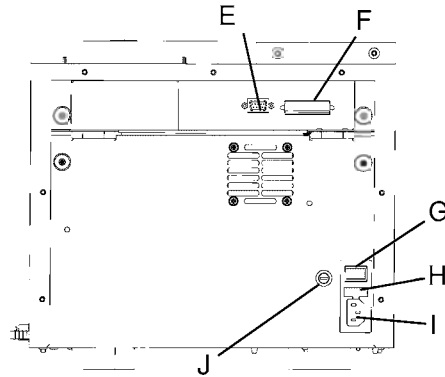


Figure 3: Rear View

E: Connector for Preparation Unit LY-501:

Available for connecting a signal cable for an Preparation unit. Currently not used.

F: Connector for communications use:

A signal cable for communication with the personal computer is connected here.

G: Power Switch:

Used to turn the LB-550 ON/OFF.

H: 1 A Fuse holder:

Contains a time-lag fuse with a rating of 1 A for AC power supply.

I: AC power connector:

The power cable is connected here.

J: Fuse holder

Contains a fuse with a rating of 5 A for the temperature controller.

LB-550 Getting Started

Preparation Unit LY-501

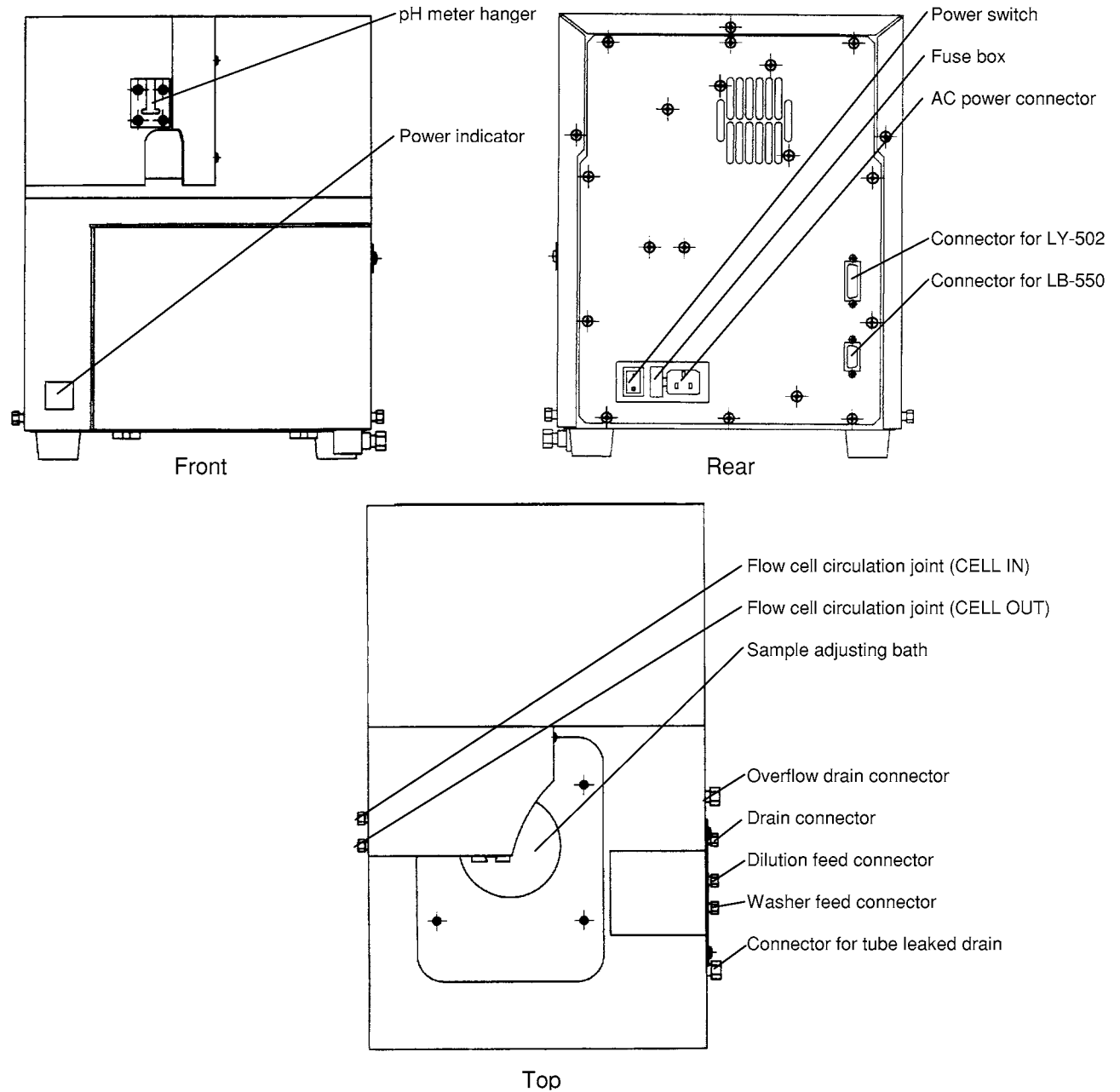


Figure 4: Front, Rear, and Top View

pH meter hanger:

When the pH meter is connected, install the electrode on this hanger.

Power indicator:

Lights up when the power switch is turned ON.

Power switch:

Turns ON/OFF the power for this system.

Fuse box:

Contains a time-lag fuse with a rating of 2 A for AC power supply.

LB-550 Getting Started

AC power connector:

The power cable is connected here.

Connector for LY-502:

Available for connecting a I/O signal cable for communications with LY-502.

Connector for LB-550:

Available for connecting a RS-232C signal cable for communications with LB-550.

Flow cell circulation joint (CELL IN):

Sample liquid returns from the flow cell. (I.D. 3 mm)

Flow cell circulation joint (CELL OUT):

Supplies sample liquid to the flow cell. (I.D. 3 mm)

Sample adjusting bath:

Bath for adjusting the sample liquid.

Overflow drain connector:

When the sample liquid is in excess of the bath capacity, the overflowed sample liquid is drained. (I.D. 6 mm)

Drain connector:

Connector for sample liquid drain. (I.D. 3 mm)

Dilution feed connector:

Connector for feeding dilution. (I.D. 3 mm)

Washer feed connector:

Connector for feeding dilution. (I.D. 3 mm)

Connector for tube leaked drain:

Connector for liquid drains leaked from the pump tube. (I.D. 6 mm)

Auto-Sampler LY-502

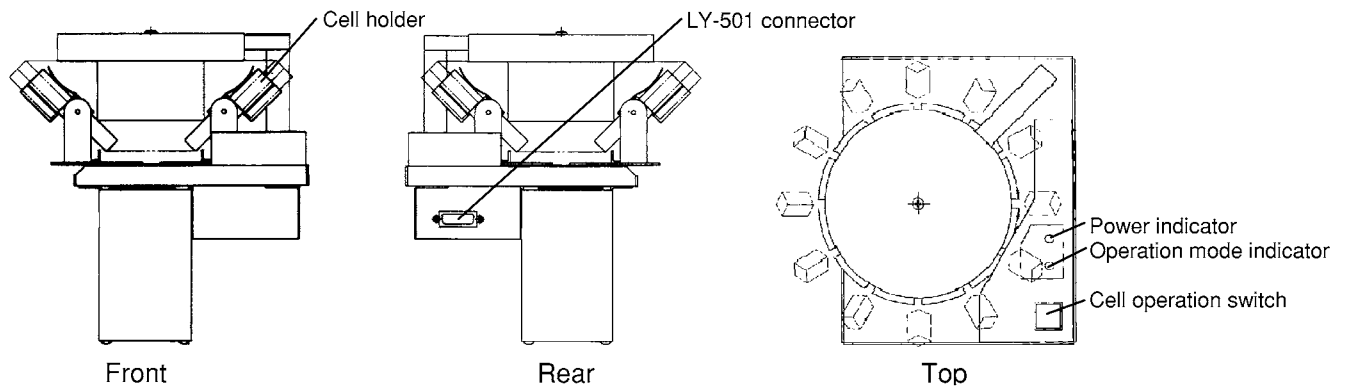


Figure 5: Front, Rear, and Top View

Cell holder:

Sample cell is set.

LY-501 connector:

I/O cable is connected to communicate with LY-501.

Power indicator:

Upon switching ON the power, green lamp lights up.

Operation mode indicator:

Operating status is displayed.

- ON: Remote control mode
- OFF: Local control mode
- Flickering: At error occurrence

Cell operation switch:

This switch is used to move the cell in local control mode.

LB-550 Getting Started

Sample Chamber

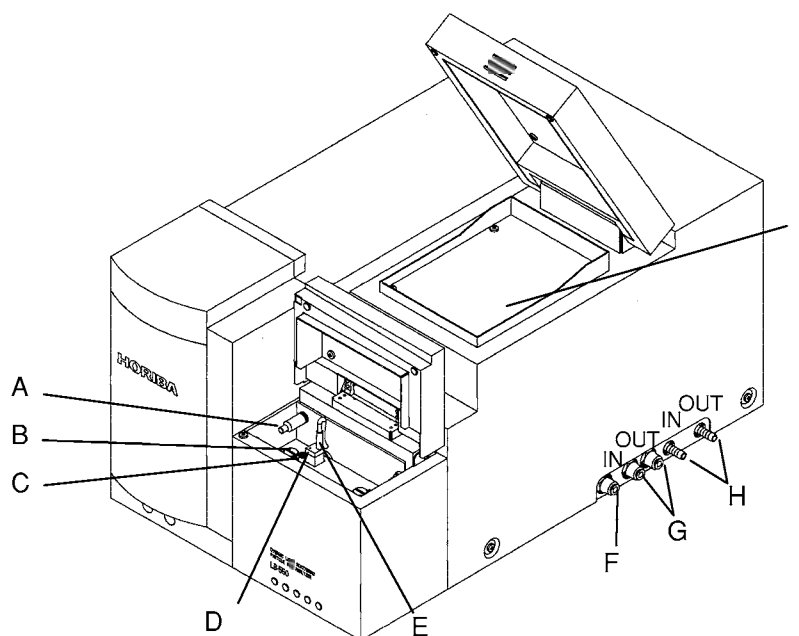


Figure 6: Sample Chamber

A: Connector for temperature sensor:

Used to install each temperature sensor: temperature sensor of the cuvette cell, the vial for standard type, the vial for viscometer mount type, or the flow cell.

B: Fixing screw for sample cell holder:

Used to secure the cell holder cover to the base of the cell holder.

C: Cell holder:

Secures the sample cell.

D: Sample cell:

Holds the sample liquid.

E: Cell cover with temperature sensor:

Used to measure the temperature of the sample liquid.

F: Joint for nitrogen purge of cell holder:

Used to supply nitrogen gas when temperature is controlled under 20°C, for prevention of cell condensation.

Used to attach a tube for nitrogen gas supply.

G: Joint for flow cell circulation:

Used for sample circulation to attach the supplied tube of LY-501 to connect the flow cell and LY-501.

H: Pipe for circulated water (outer diameter: 8 mm):

Used to connect the hose in which the cooling water (tap water, etc.) for cooling the built-in temperature controller flows.

Inner diameter: 6.4 mm to 7.0 mm (soft resin tube)

5 mm to 7 mm (soft rubber tube)

Use wire band etc., to avoid falling out the tube at the connecting part.

I: Equipment box:

Used to store the equipment. The cover can be opened.

LB-550 Getting Started

Note:

- When using the Preparation Unit LY-501 and Viscometer Unit LY-554, the cell unit must be changed to a dedicated cell unit.

LB-550 Getting Started

Sample Cell

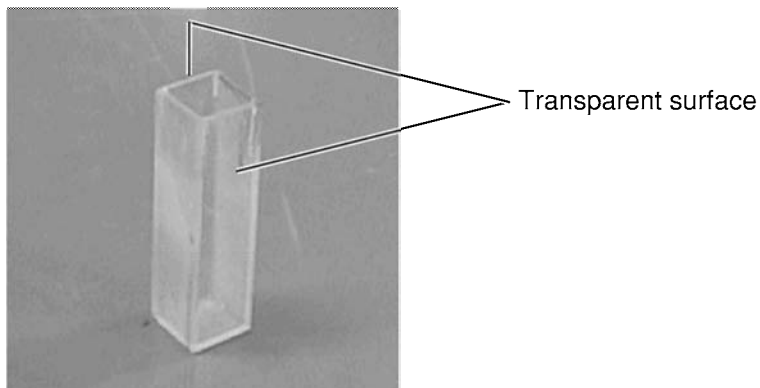


Figure 7-1: Glass cuvette cell



Figure 7-2: Vial (For Viscometer Unit LY-554 use)

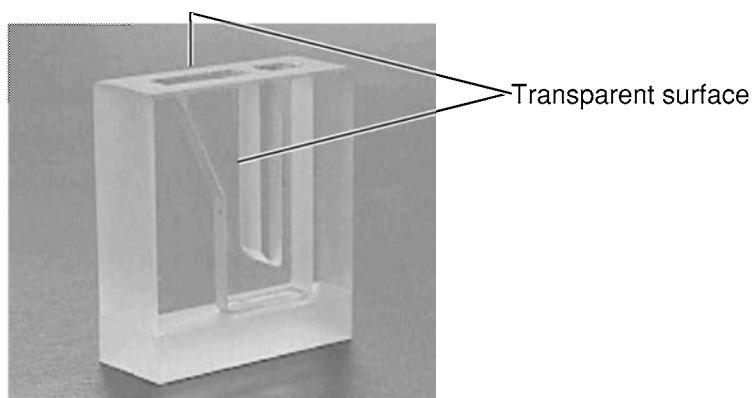


Figure 8: Glass flow cell (For Preparation unit LY-501 use)

Note:

- When handling the sample cell, take care not to directly touch the transparent surface with your fingers and not to damage it.

Installation Conditions

Note:

If the LB-550 is installed in a location where any of the following requirements is not met, it may not operate normally. This also may cause performance deterioration or failure.

- The range of power voltage shall be 100 V/120 V/230 V AC $\pm 10\%$, and the range of power frequency shall be 50 Hz/ 60 Hz ± 1 Hz. Use a power source without voltage fluctuation. The power capacity shall be 150 V AC minimum.
- When the Preparation Unit LY-501 is used, the supply voltage is 100 V/ 120 V AC and the power frequency range is 50 Hz/60 Hz ± 1 Hz. Use the power capacity of more than 120 VA without fluctuations.
- The LB-550 shall be connected to the ground with 3-pin plug.
- The operating ambient temperature shall be 15°C to 35°C. It is desirable that the temperature fluctuations during a measurement fall within a range of $\pm 1^\circ\text{C}$.
- Avoid installing the LB-550 in a location where the temperature fluctuates greatly, e.g., because the unit is exposed to direct sunlight or direct wind from an air-conditioner.
- Condensation occurs at high humidity, causing a measurement failure or malfunction of an electronic part. The LB-550 shall be used at relative humidity of 85% or less under condensation-free conditions.
- Use the LB-550 in an environment free from excessive dust and corrosive gasses.
- Avoid using the LB-550 in a place where it is exposed to or is likely to be exposed to strong vibrations.
- The LB-550 shall be installed on a stable base.
- The rear of the LB-550 has a ventilation grille for the power source cooling fan. Do not close this grille.
- Keep the LB-550 away from any source with powerful magnetic line of force, electric field, or high frequency.
- The LB-550 is designed for use in the installation environment classified as installation category (overvoltage category) II and pollution degree 2 (applicable standard: IEC61010-1). Do not use the LB-550 in an environment where impulse voltage exceeding this level may be applied.
- For the installation conditions applicable to the personal computer and peripheral devices, refer to their respective instruction manuals.
- Make sure to keep good ventilation during nitrogen-gas purge for your safety.
- Avoid arranging the LB-550 where the power switch operation might be difficult.

Before You Begin:

The LB-550 unit should be installed on a hard, level bench top.

If the dispersant fluid used emits harmful gases or vapors, use appropriate safety measures, such as installation in a fume hood.

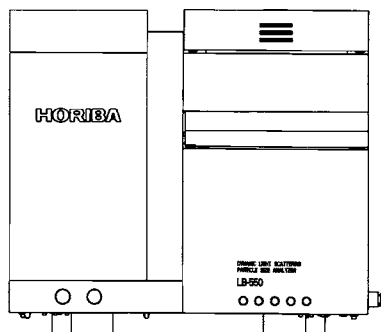


Figure 9: LB-550 Particle Size Analyzer

LB-550 Getting Started

OPERATION

Preparation

Connecting Cables, Piping

Connecting LB-550

Connect the cables and piping as follows. For connecting the personal computer to peripheral devices, refer to the corresponding instruction manuals.

1. Check that the power switches on the LB-550 and the personal computer are OFF.
2. Using the signal cable supplied with the LB-550, connect the communications connector on the rear of the LB-550 to the SCSI port on the personal computer. (Refer to Figure 3).

Note:

- **SCSI and connector type**
LB-550 supports a SCSI-II board which is made by ADAPTEC only. The communication speed is 10 MB/s. It has 50-pin connector (male).

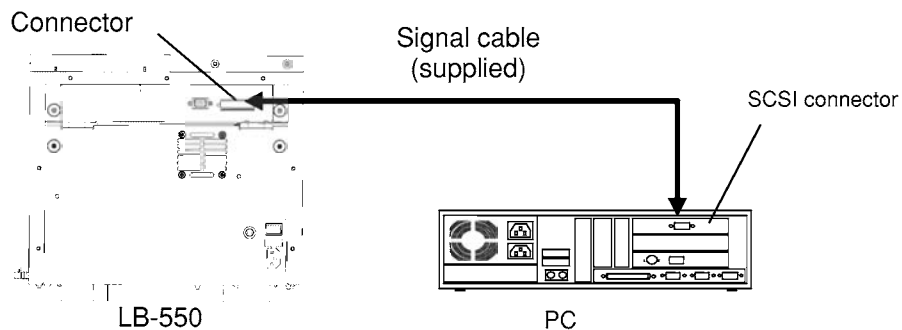


Figure 10: Wiring between the LB-550 and the personal computer

3. Plug the LB-550 and the personal computer into power outlets. Be sure to use the dedicated power cords (with a 3-pin plug) for the power source.

Warning:

Improper insulation may cause electric shock. Be sure to ground the LB-550 and the personal computer.

4. When the specified temperature is under 20°C or the temperature is lowered for 20°C or more range, connect the piping for cooling water to the pipe for circulated water.

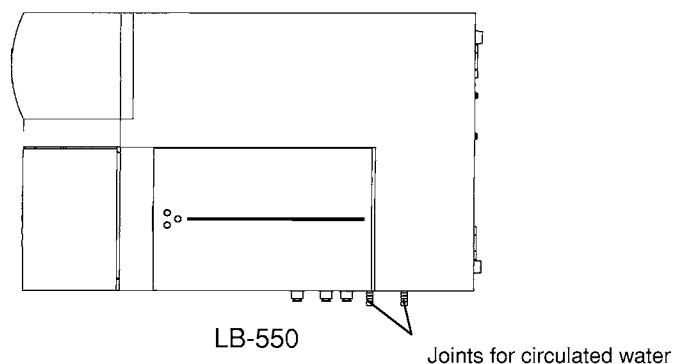


Figure 11: Piping of LB-550

Connecting the Preparation Unit (LY-501)

1. Remove the cell holder for LB-550.
 - ① Open the upper door of the sample chamber.
 - ② The cell holder is fixed to the main body with pan head screws (at 2 places). Remove the fixing screws with a screwdriver and take out the cell holder from the sample chamber.
2. Connect the LB-550 connector at the rear of the LY-501 and the RS-232C connector for optional unit at the rear of the LB-550 using a signal cable furnished.

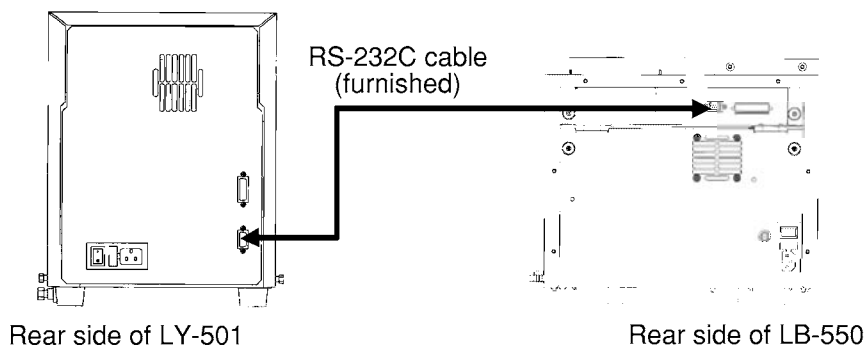


Figure 12: Wiring between LB-550 and LY-501

3. Fix the flow cell holder supplied with the LY-501 to the LB-550 sample chamber.
 - ① Mount the flow cell holder adjusting on the pins under the sample chamber and tighten the knurled screws (at 2 places).

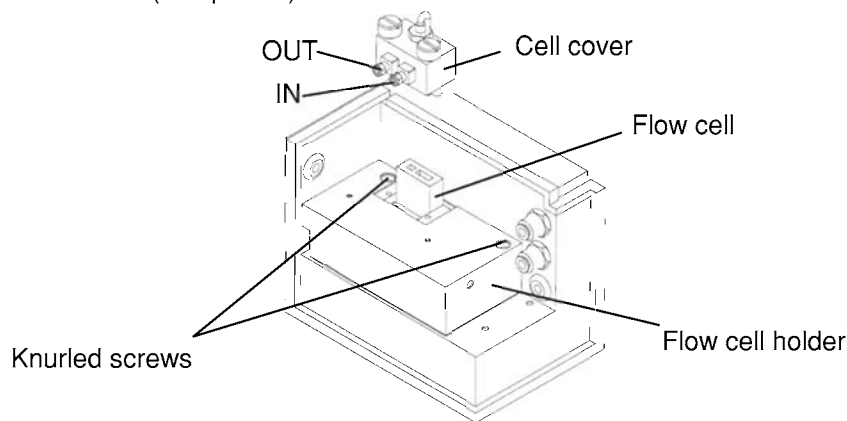


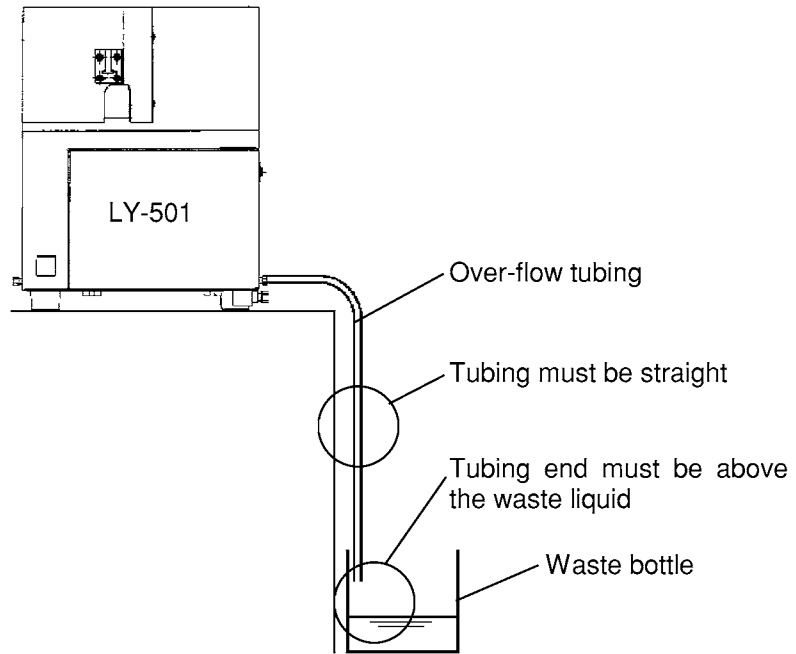
Figure 13: Fixing the flow cell holder

- ② Connect the piping for sample circulation. Connect it to the "CELL OUT" connector at the right side viewing to the side of the LY-501. Connect the other end of the tube to the right connector (IN) at the top of the cell holder through the "CELL IN" hole on the side cover of the LB-550. Connect to the "CELL IN" at the side of the LB-550 in the same procedure.
- ③ For the draining operation, contact a drain tubing to the LY-501 drain piping, and then put the other side of tubing into a waste bottle.
- ④ For the over-flow draining, contact a tubing to the LY-501 over-flow drain piping, and then put the other side of tubing into a waste bottle.

LB-550 Getting Started

Note:

- The over-flow drain tubing should be installed as following diagram.



Connecting the Auto-Sampler (LY-502)

1. Fix the LY-502 on the mounting plate at the side of LY-501 with screws.

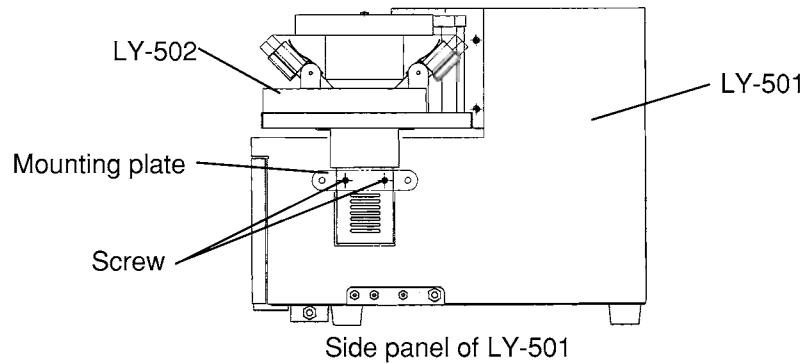


Figure 14: Mounting the LY-502

2. Connect the connector at the rear of the LY-502 and the LY-501 connector using a furnished I/O cable.

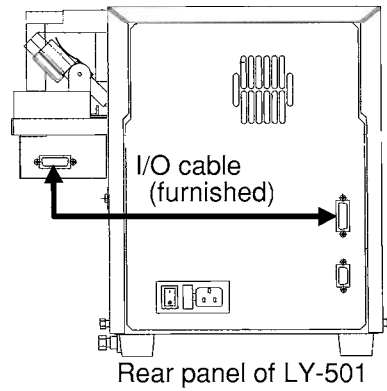


Figure 15: Wiring between LY-501 and LY-502

LB-550 Getting Started

Start-up and Shutdown

Start-up

1. Turn on the power switch of the LB-550. Wait approximately 20 minutes, before measurement, until the laser becomes stable.
2. Turn on the power switch of the personal computer. Windows will be activated automatically.
3. Double-click the icon for LB-550 to activate the application program.

Notes:

- Be sure to turn ON the LB-550 before starting the personal computer. If the LB-550 is turned ON after the personal computer has been started, the LB-550 will not operate.
- When connecting the Preparation Unit LY-501, turn ON the power for the LY-501 before turning ON the power for startup of the LB-550 software. The power switch of the LY-501 is located at the rear of the main unit.
- When measuring viscosity with the viscometer mount type, be sure to place a cell holder for vial into the sample chamber.

Shutdown

1. Quit the LB-550 Software. To do so, take one of the following actions:
 - ① Click on the "LB-550 Exit" command in the File menu.
 - ② Click on the Close button located at the right end of the title bar.
 - ③ Press the Alt and F4 keys simultaneously.
2. Shut down Windows.
Click on the Start button, and then click on the Shut Down command. The quitting options will be displayed. Select "Shut down the computer?" and then click on the Yes button, or press the Enter key. Windows will be shut down.
3. Turn OFF the LB-550 and the personal computer. The POWER switch for the LB-550 can be found on the rear of the main unit.
4. Remove the cell and clean it.

Note:

- If the LB-550 is left ON without using it for a long period of time, the laser will remain active and can shorten its service life.

MAINTENANCE

Daily Care

- If the LB-550 becomes dirty, wipe it with dry cloth or clean it by lightly wiping with gauze (or something similar) soaked with a small amount of neutral cleanser.
- Remove the sample cell from the sample chamber, clean it well, and then put it back in place.
- If a sample or dispersant is spilled in the cell holder, wipe it off using a cotton swab or something similar. If a large amount of sample or dispersant is spilled, perform the steps for cleaning the cell holder as described below:

Cleaning The Cell

1. Clean the cell with the neutral detergent. Use a cotton applicator or soft cloth for this purpose.

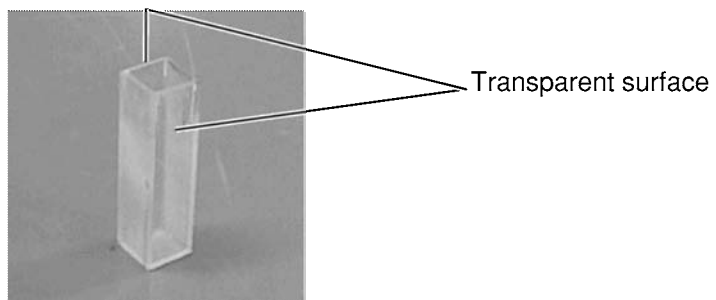


Figure 16-1: Cleaning the glass cuvette cell



Figure 16-2: Cleaning the vial (For Viscometer Unit LY-554 use)

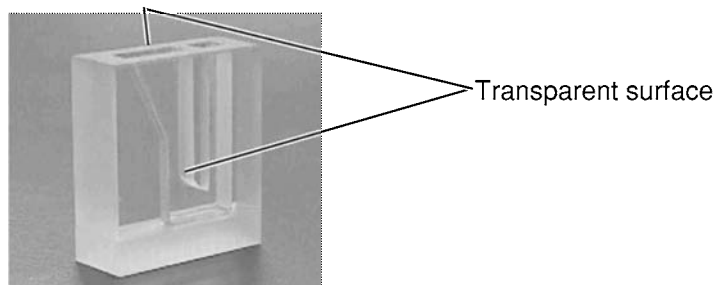


Figure 17: Cleaning the glass flow cell

! Caution:

- The transparent surface of the cell should be cleaned with a cotton applicator or soft professional, lens cleaning cloth. Never scrub it, nor touch it with anything hard.
- The flow cell is used when the Preparation Unit LY-501 is connected.
- A vial is used when the viscometer is connected. When using it repeatedly, wipe it lightly with a swab or soft cloth. Never scrub strongly or wipe with a hard object.

LB-550 Getting Started

Cleaning The Cell Holder

If a sample or dispersant is spilled in the cell holder, clean the cell holder as follows:

- 1 After turning OFF the power, allow the cell holder to cool down, and then shut off the column oven and cooling water.

Notes:

- Never touch any of the optical and electrical parts that are located inside the cell holder.
 - If the power is ON, you may get an electric shock. Be sure to check that the power is OFF before opening the sample chamber.
 - Touching the inside of the cell holder may result in a burn. Be sure to check that the cell holder has cooled down completely before starting the work.
- 2 Open the upper door of the sample chamber, and disconnect the tube for nitrogen purge.
 - 3 Remove the pan-head screws retaining the cover with a screwdriver, and then remove the cover.
 - 4 Clean the inside of the cell holder using a cotton swab, absorbent cotton, tweezers, or something similar.

Exchange of Pump Tube

When the service life message for the pump tube built in the Preparation Unit LY-501 is displayed, exchange it in the following procedure:

Reference:

- For the material of the tube to be exchanged, refer to "Chemically Resistant Table for Various Tube Materials" (page III-130).
1. Completely drain the sample liquid in the piping.
 2. Shift the peristaltic pump lever to the left.
The pump head opens.

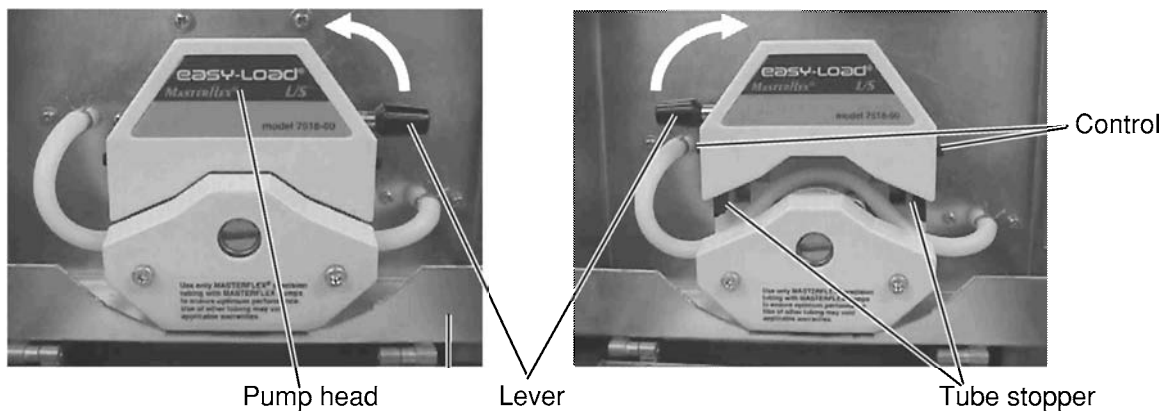


Figure 18: Exchange of pump tube

3. Remove the clamps at both ends of the tube and take out the tube.
4. Exchange the tube and set the clamps at both ends of the tube.
5. Place the tube under the tube stopper.
6. Shift the lever to the rightmost so that the tube should be securely held by the pump head.
7. Holding the control, adjust so that the height of the tube stopper is the same as the tube diameter.

Note:

- Verify that the tube is not bent or twisted.

LB-550 Getting Started

Troubleshooting

If you have encountered a trouble, check the possible causes for the symptom involved in that trouble before determining the trouble as a failure.

Table 1: Symptoms and remedies

Symptom	Possible cause	Action		
When the power switch is turned ON, the power indicator on the front of the main unit does not light up.	The power cable is not connected.	Turn OFF the power switch, and connect the cable properly.		
	The fuse is blown.	Replace the fuse.		
The alarm indicating the dead laser beam occurs.	The light source is broken.	The light source must be replaced. Contact our service station.		
The stability is low.	The LB-550 is influenced by changes in the room temperature.	Stabilize the room temperature.		
	The LB-550 is exposed to severe vibrations.	Eliminate the source of vibrations.		
	The cell is condensed.	Wipe vapor on the cell and use the LB-550 with the temperature condition of no condensation.		
The software of the LB-550 does not start.	The System Administration has started up.	Shut down the System Administration program.		
The System Administration does not start.	The software of the LB-550 has started up.	Shut down the software of the LB-550.		
LY-502 does not work.	Check the LED on the LY-502 body. Flickering of the red LED means the cell position error. If the red LED is not flickering, the cause may be the communication error.	When the red LED is flickering, move the cell to the normal position and verify that the flickering LED goes off. When the red LED is not flickering, verify that the I/O cable is properly connected and turn ON the power for LY-501 again.		
		* Lighting of LY-502 LED		
		Red LED:	Remote mode	ON
			Local mode	OFF
			Cell position error	Flickers
Green LED:	Power ON	ON		
	Power OFF	OFF		

The above table covers the troubles you may encounter with the hardware of the LB-550. If an error has occurred, refer to "Error Handling" (page III-128).

If your trouble is not solved after you have taken the corresponding action, contact our service station.

Notes:

- The ventilation fan stops during the measurement. This occurs to prevent noise during the measurement and is not a malfunction.
- The shutter to prevent laser beam works and the noise occurs at the beginning and during the measurement. This is not a malfunction.

Replacing The Fuse

If the fuse is blown, replace it with those delivered with the LB-550.

1. Turn off the power switch.
2. Disconnect the power cable.
3. Replace the fuse. The location of the fuse is shown in Figure 3 (page I-4). Never use any fuse that is not specified.
There are a 1 A and a 5 A fuses in LB-550. There is a 2 A fuse in LY-501.

Notes:

- Note that the fuse holder below the power switch should be a 1 A Time Lag, and the fuse holder for the temperature control should be a 5 A Time Lag.
4. Connect the power cable.
 5. Turn on the power switch.
 6. Check if the LB-550 operates normally.

! Caution:

If the fuse is still blown after the replacement, immediately turn off the power switch, disconnect the power cable, and then contact our service person. Never use any fuse other than the standard accessories. For the place to set the fuse, refer to Figure 3 (page I-4).

Other Precautions

Moving the LB-550

To move the LB-550, you need to fasten it in conjunction with the fixations of the optical bench and others. If you move the LB-550 without these fixations, this may cause a malfunction. Before moving the LB-550, contact our service center or sales personnel.

Storage

1. After carrying out the shut-down procedure described above, remove the cell and clean it, dry thoroughly, place the dust cover over the unit and store carefully.
2. The analyzer must be stored where it will not be exposed to temperatures exceeding a range of 5°C to 40°C (40°F to 104°F).
3. If the unit has been stored for a long time, be sure to carry out all the appropriate steps described above before attempting to conduct measurements.

LB-550 Getting Started

Keeping the LB-550 out of service for long period of time

1. Remove the sample cell from the sample chamber.
2. Close the sample chamber lid.
3. After cleaning the sample cell, dry it well.
4. Wrap the sample cell with gauze while taking care not to damage its transparent surface, and store it.
5. Turn OFF the power switches located on the personal computer and the LB-550. If there are any peripheral devices to the personal computer or any special accessories for the LB-550, turn OFF all of them.
6. Disconnect the power cable and other wiring.
7. Store the accessories such as the cables and system disk in the same place as the personal computer, in order to prevent them from being missing.
Keep the disks away from the monitor and any other source with powerful magnetic line of force.

Disposing of the LB-550

The LB-550 uses no parts that require any special treatment for disposal. It may be disposed of as a normal industrial waste.

APPENDIX

Specifications

LB-550

		Standard type	Viscometer (LY-554) mount type
Principles of Measurement		Based on dynamic light scattering method	←
Range of Particle Size Displayed		1 nm to 6 μ m	←
Measurement Time		Approx. 2 minutes, normally from start of measurement to display of data.	←
Amount of Sample Liquid Required for Measurement		2 mL to 4 mL	20 mL to 30 mL
Principles of Thermostatic		Cartridge heater, Peltier element	←
Range of Thermostatic Temperature for Cell Holder		5°C to 70°C (When the specified temperature is under 20°C or the temperature is lowered for 20°C or more range, cooling water should be circulated. Cooling water flow: approx. 0.4 L/min) Note that nitrogen purge is required under 20°C to prevent dew condensation.	←
Viscosity range of measurement sample		N/A	0.40 to 10.0 mPa · s
Optical System	Light source	650 nm Laserdiode, 5 mW	←
	Photo-cell detector	Photo-multiplier tube	←
	Sample cell	Cuvette cell	Cuvette cell or Vial (Be sure to use a vial when measuring viscosity.)
Power		100 V /120 V /230 V AC \pm 10%, AC input range selection is fully automatic, 50 Hz/60 Hz Approx. 150 VA	←
Operation Temperature		15°C to 35°C	←
Humidity		Less than 85% (no condensation)	←
Dimensions		340 mm (W) x 565 mm (D) x 306 mm (H)	340 mm (W) x 565 mm (D) x 530 mm (H)
Mass		Approx. 28 kg	Approx. 36 kg

LB-550 Getting Started

Preparation Unit LY-501

	Standard type
Liquid feed method	Flow type (but, stationary state when measuring) method
Full liquid required time	Within 40 seconds (when using water)
Type of cell	Our glass flow cell (Liquid temperature sensor built-in type)
Required sample amount	60 mL Min. (Bath capacity; approx. 120 mL)
Principles of Thermostatic	Cartridge heater, Peltier element
Range of Thermostatic Temperature	20°C to 70°C, Cooling water required according to the environmental condition / set temperature condition. (Cooling water flow: Approx. 1 L/min)
Acceptable dispersion medium	Acid solvent, Alkali solvent, water, organic solvent
pH applicable for solvent	pH2 to pH12 (Tube material must be checked.)
Ultrasonic output	Approx. 20 W (Resonance frequency: 28 kHz)
Feed line	2 lines (1 line for washer, 1 line for dilution or sample solution) Proof pressure: 1×10^5 Pa Max.
Circulation velocity	280 mL/min (at water circulation)
Piping	Teflon
O-rings	Kalrez
Tube	PharMed (MasterFlex-make) and Fluran (Norton-make) supplied
Overflow sensor	Float type
Communications	RS-232C (between LY-501 and LB-550), RS-232C (between pH meter and PC)
Service temperature	15°C to 35°C
Humidity	Relative humidity 85% Max.
Cell holder	Available for connection of constant temperature water circulation system
Power	100 V/120 V/230 V AC, 50 Hz/60 Hz, 120 VA
Dimensions	250 mm (W) x 400 mm (D) x 320 mm (H) (without protrusion)
Mass	Approx. 17 kg

Optional measurement

pH measurement	Consult us in case of connecting the pH meter to the LB-550.
Conductivity measurement	

Auto-Sampler LY-502

Type of cell	Glass made round shape sampling tube
Number of samples	10 samples, Max.
Sample amount	Approx. 4 mL, Max.
Service temperature	15°C to 35°C
Humidity	Relative humidity 85% Max.
Dimensions	25 mm (W) x 220 mm (D) x 240 mm (H) (without protrusion)
Mass	Approx. 5 kg

Viscometer Unit LY-554

Measuring method	Rotational oscillation
Viscosity measurement range	0.40 mPa·s to 10.0 mPa·s
Use environment	Temperature: 10°C to 40°C, Relative humidity: 10% to 80%
Measurement accuracy	Measured value: $\pm 5\%$ (Room temperature, Fluid temperature: $23^{\circ}\text{C} \pm 3^{\circ}\text{C}$) Comparative calibration by means of JIS Z8809-1999 standard solution for viscosity meter calibration

LB-550 Getting Started

Dimensional Outline

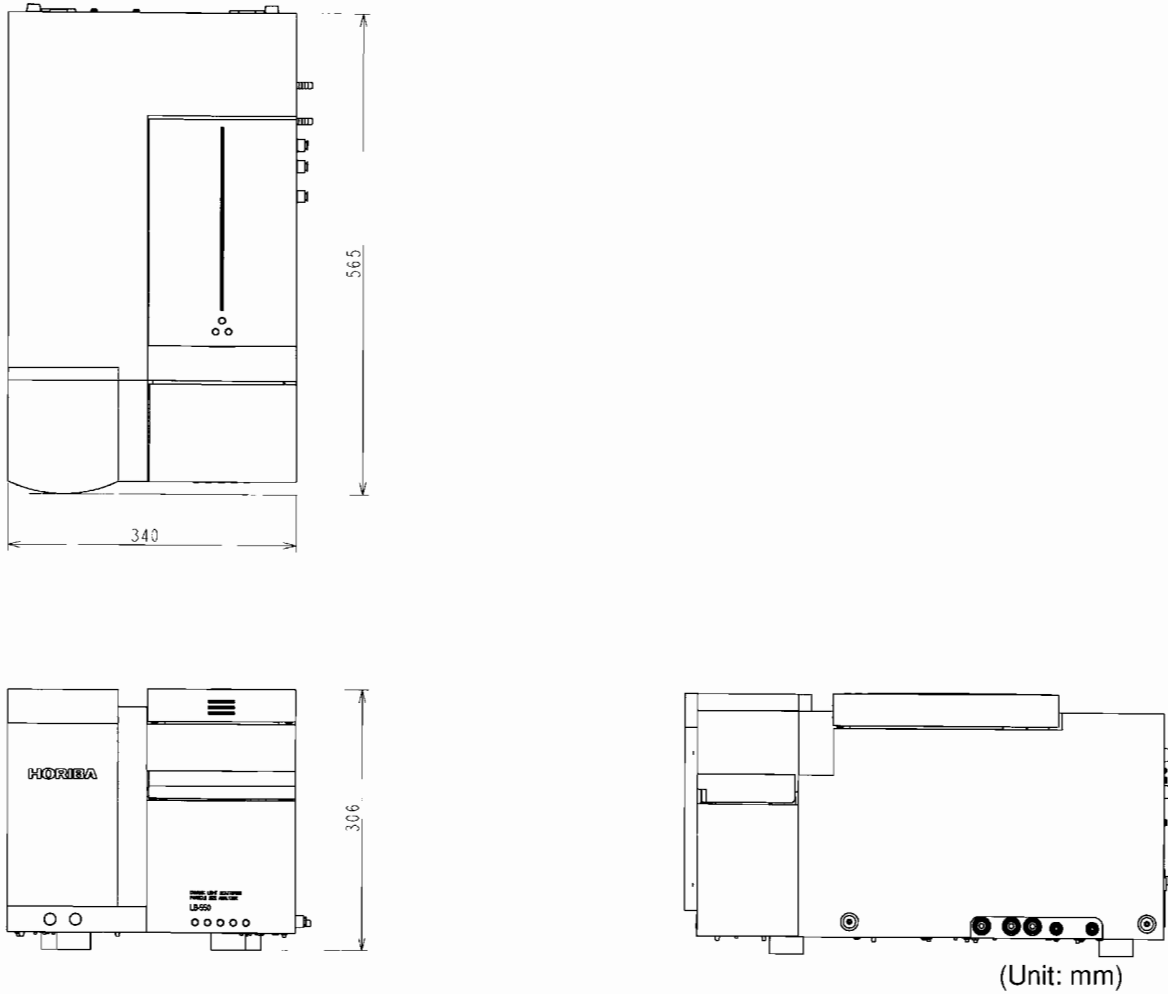


Figure 19: LB-550 (Standard Type) Dimensional Outline

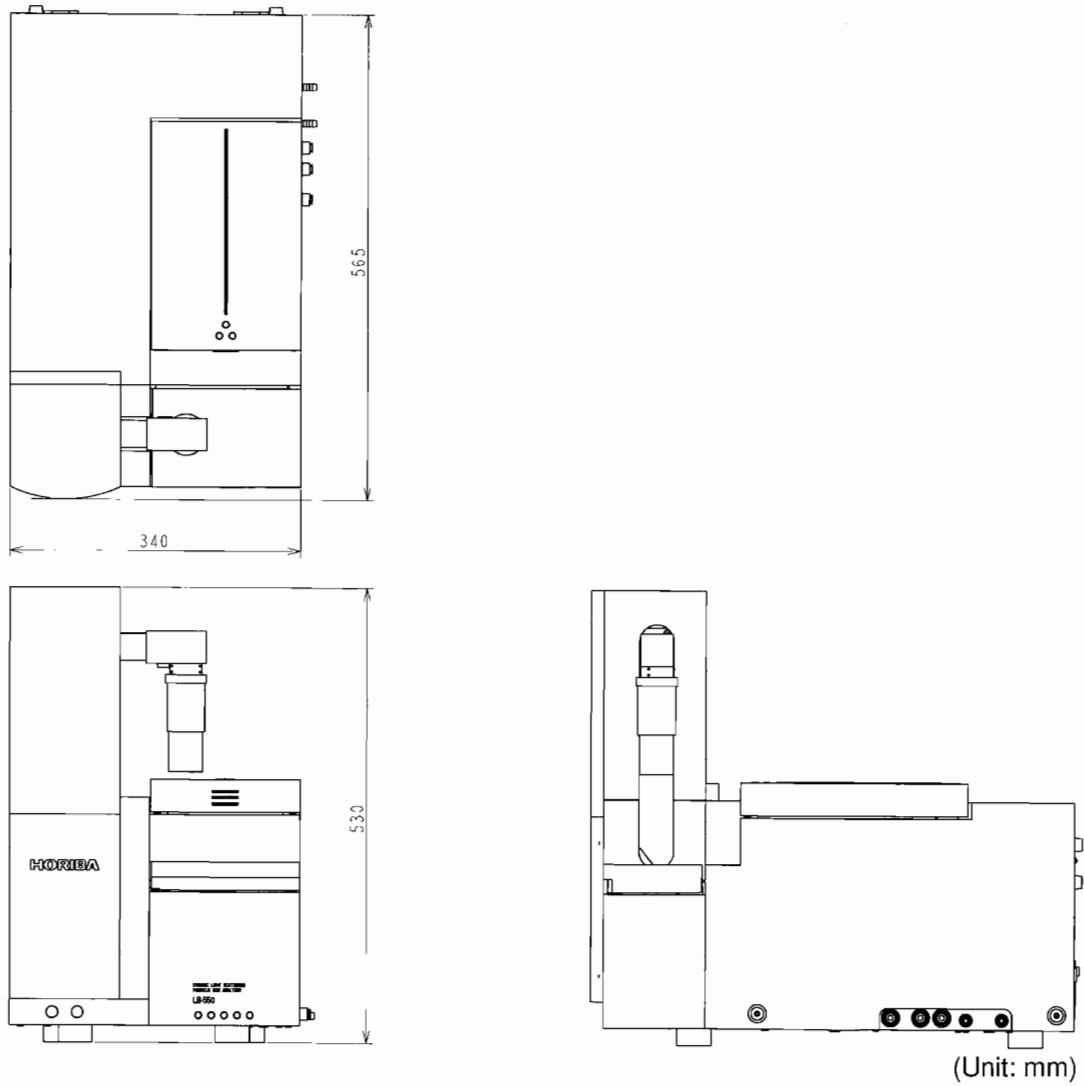
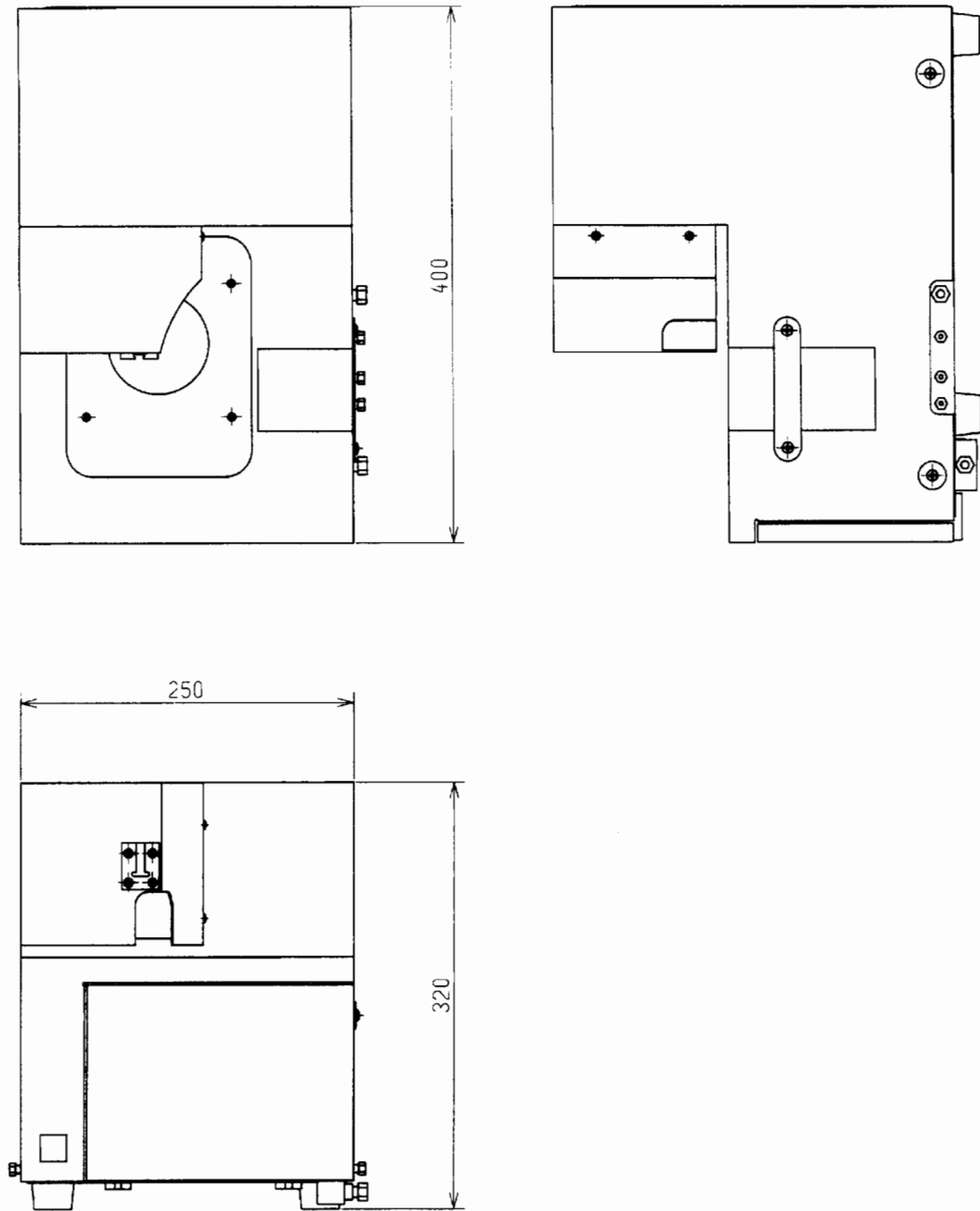


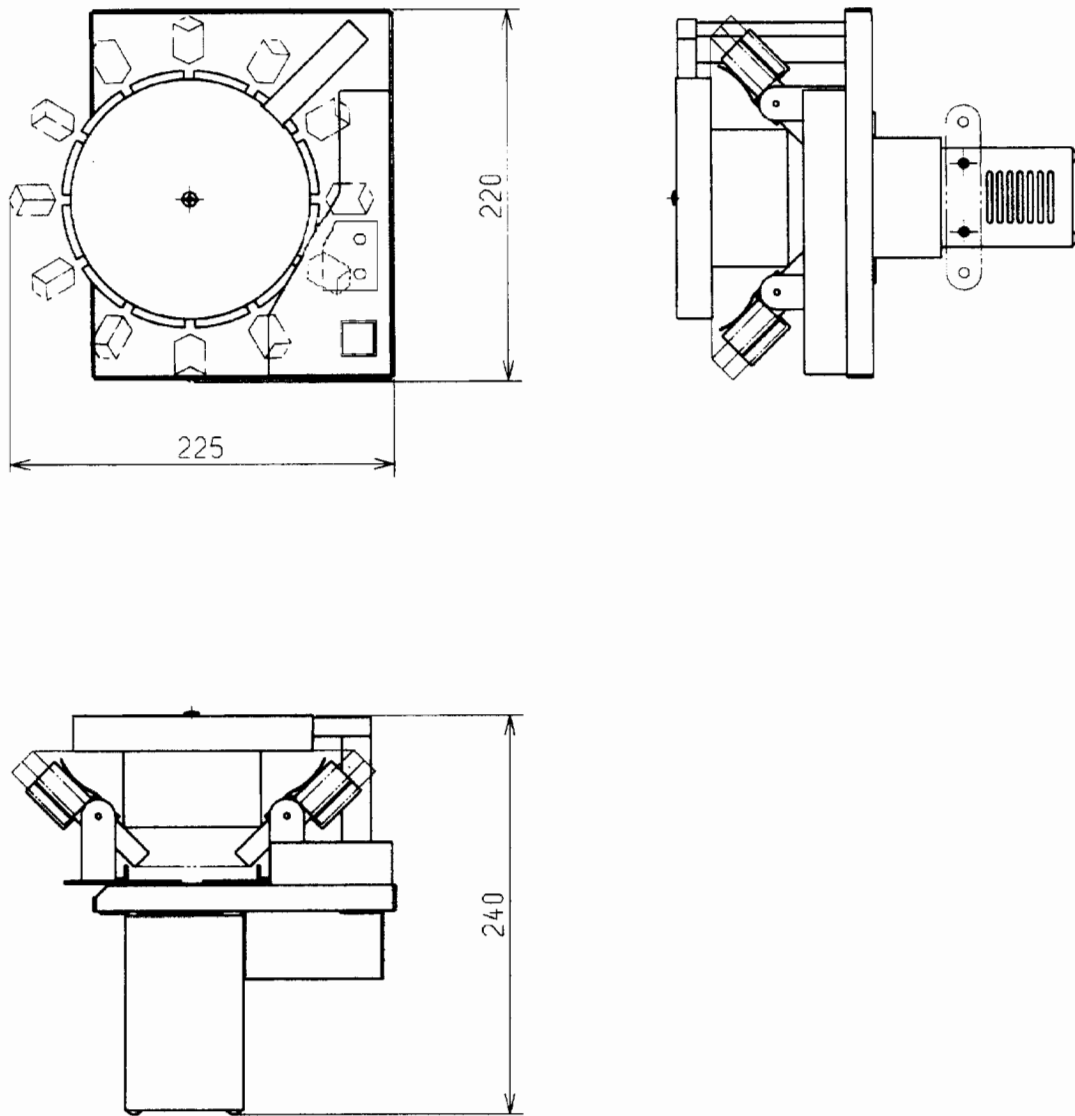
Figure 20: LB-550 (Viscometer Mount Type) Dimensional Outline

LB-550 Getting Started



(Unit: mm)

Figure 21: LY-501 Dimensional Outline



(Unit: mm)

Figure 22: LY-502 Dimensional Outline

LB-550 Getting Started

Accessories

The standard accessories are included in the upper section of the case. Check them with Table 2 below.

Table 2: LB-550 Accessory Kit

Parts Name	Quantity	Remark
Power cable	1 pc.	2 m (For 100 V/120 V)
	1 pc.	2 m (For 230 V)
SCSI cable with 50 pins (male)	1 pc.	For connecting to PC
Glass cuvette cell	1 pc.	
Cotton applicator	100 pcs.	
Fuse	1 pc.	1 A Time Lag
	1 pc.	5 A Time Lag
Instruction Manual	1 pc.	
LB-550 Relocation Instructions	1 pc.	
LB-550 CD-ROM	1 pc.	For software backup
ASPI file floppy disk	1 pc.	Install the ASPI file when the LB-550 software is operated on Windows 2000 or Windows XP.
Vial (for viscometer mount type)	55 pcs.	Inner bore 21 mm (dia.) × body 30 mm (dia.) × 63 mm (H) Glass: 1 mm thick
Holder for vial	1 pc.	

Table 3: LY-501 Accessory Kit

Parts Name	Quantity	Remark
Power cable	1 pc.	2 m (For 100 V/120 V)
	1 pc.	2 m (For 230 V)
RS-232C cable	1 pc.	Between LB-550 and LY-501
	1 pc.	Between pH meter and PC
Tube (For pump)	3 pcs.	PharMed 6485-25
	3 pcs.	Fluran F-5500-A 3/16 × 5/16
Teflon tube	2 pcs.	0.4 m (O.D. 3 mm/I.D. 2 mm, for cell)
	3 pcs.	1.4 m (O.D. 3 mm/I.D. 2 mm, for inlet/outlet)
	1 pc.	1.5 m (O.D. 6 mm/I.D. 4 mm, for over flow)
	1 pc.	1.5 m (O.D. 6 mm/I.D. 4 mm, for drain)
Fuse	1 pc.	2 A Time Lag (For 100 V/120 V)
	1 pc.	1 A Time Lag (For 230 V)
Ball wrench	1 pc.	Opposite side 4.0 mm
Glass flow cell	1 pc.	H2011773001A

Table 4: LY-502 Accessory Kit

Parts Name	Quantity	Remark
I/O cable	1 pc.	
Disposal cell	100 pcs.	12.5 mm (W) × 12.5 mm (D) × 45 mm (H)
Sample tube	100 pcs.	16.5 mm (Dia.) × 40 mm (H)

Service Life of Components

LB-550

The median time fault (MTF) of the laser according to the manufacturer is approximately 11,000 hours for continuous use.

The laser shutter is actuated once when the measurement panel is opened. Its average life is 100,000 actuating times.

The above service lives are averages and may vary depending on systems.

They are given for your reference only, and are not guaranteed values.

Parts Name	Parts No.
Laser light source unit	G0000860

Note:

- For the cell, purchase a cell for use with a spectrophotometer (optical path length: 10 mm; 12.5 mm W x 12.5 mm D x 45 mm H) from your nearest distributor for products related to physics and chemistry.
In addition to a glass cell, a disposal cell can also be used.
The cell of other size as a micro cell can not be used. Refer to "Sample Cell" (page I-10).
Use a vial with inner bore diameter 21 mm x body diameter 30 mm x height 63 mm, and glass of 1 mm thick. Purchase it at your nearest distributor of physical and chemical appliance.

LY-501

The service life of the ultrasonic tip is approx. 70 hours (our tested value).

The service life of the Farmed is in case of the continuous 600 rpm, approx. 1000 hours (Manufacturer's reference value) when the water of 0 kPa is used at 21°C.

The service life of the Fulrun is in case of the continuous 600 rpm, approx. 150 hours (Manufacturer's reference value) when the water of 0 kPa is used at 21°C.

When the ultrasonic tip is exchanged, contact your nearest service station.

Purchase the tubes at your nearest agent for the physical and chemical equipment.

Note:

- Order the tubes for pump with the model specified in Remark of Table 3 (page I-30).

Parts Name	Parts No.
Ultrasonic tip	9035000300
Teflon tube (for cell/inlet/outlet)	F0020074600
Teflon tube (for over-flow/drain)	F0020032100
O-ring	9035000100
Flow cell	9035000200

Reference:

- In case of the continuous 600 rpm, the service life of the tubes is as follows provided the water of 0 kPa is used at 21°C. (Manufacturer's reference values)
 - Norprene: 1000 hours
 - Silicon, C-Flex: 100 hours
 - Tygon: 50 hours
 - Byton: 30 hours

LB-550 Getting Started

Measurement Principles

Overview of the LB-550

The LB-550 uses the dynamic light scattering theory to measure the entire particle size distribution range between 1 nm and 6 μm at one time. It allows you, normally, to complete the cycle from starting the measurement to displaying the result within two minutes.

The configuration of the LB-550 is shown below:

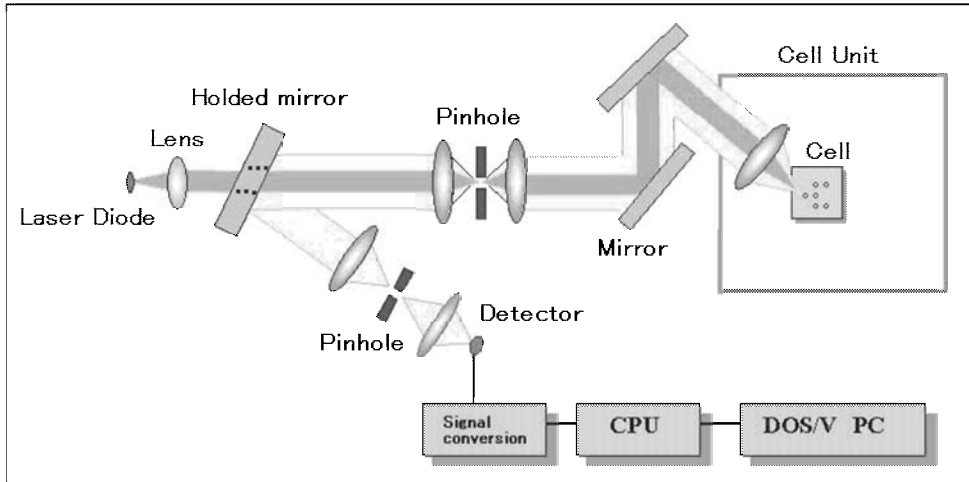


Figure 23: Configuration of the LB-550

The light emitted from the laser diode passes through the pinhole, and is radiated to the sample particles in the cell. The light scattered by the sample particles returns through the optical path for incoming light, is reflected by the holed mirror, go through the pinhole and enters the photo detector.

The detected analog signal is converted into a digital signal, and sent to the CPU. The converted digital signal is transmitted to the personal computer via the SCSI port, and then processed arithmetically.

Overview of arithmetic operations

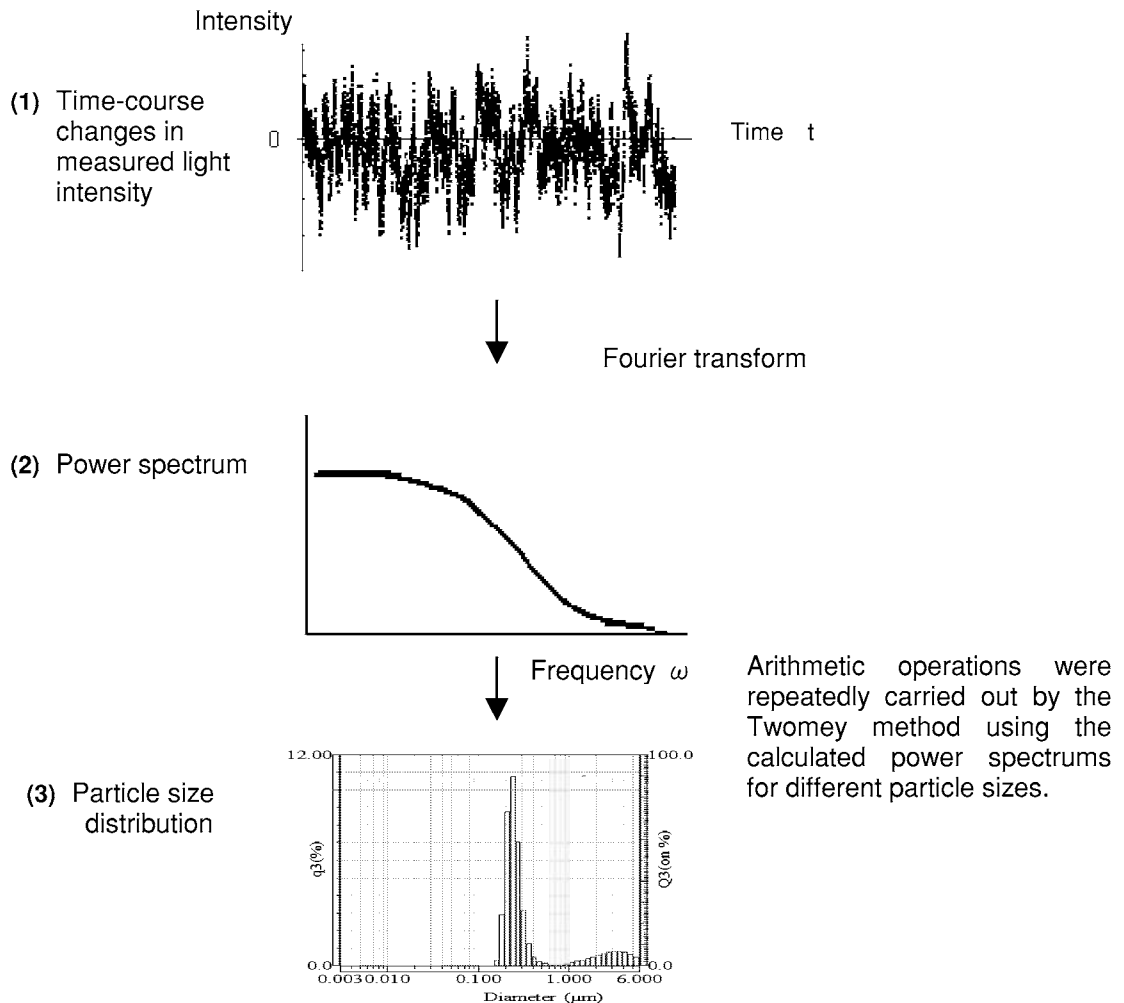


Figure 24: Measurement principle

The light scattered by the sample particles contains light that has a frequency distribution deviating from the incoming light frequency due to the Doppler effect resulting from the Brownian movement of the particles (1).

These scattered light rays interfere with each other. Thus, the light intensity detected by the detector causes fluctuations over time.

When the time-course change data for the detected light intensity is loaded to the personal computer, and processed by Fast Fourier transformation, the frequency distribution (power spectrum) for the changes in the light intensity can be obtained (2).

When this data is compared to and calculated using the previously calculated frequency distribution functions based on the intensity of the Brownian movement (scattering factor) for particles with different particle sizes, the particle size distribution of the particles contained in the sample can be obtained (3).

Part II Operation

START-UP AND SHUTDOWN	II-1
Start-up	II-1
Shutdown	II-1
SETTINGS	II-2
Set Conditions	II-3
MEASUREMENT	II-9
Sample Measurement	II-9
Measurement Results	II-11
Checking for Dirt.....	II-11
SAVE	II-12
File Operations.....	II-12
CLEANING THE CELL.....	II-13
Cleaning the Cell When it is Very Dirty.....	II-14
Removing the Flow Cell.....	II-14
MEASUREMENT RESULT.....	II-15
Result Data.....	II-15
Calculated Data.....	II-15
SAMPLING & DISPERSION	II-18
Sampling Methods.....	II-18
Dispersing Methods for Samples.....	II-20
VALIDATION	II-22
LIST OF THE REFRACTIVE INDEX VALUES.....	II-23
Inorganic Substances	II-23
Organic Substances.....	II-25
Dispersant Refractive Index.....	II-26
OPTIONAL UNIT	II-27
Preparation Unit LY-501	II-27
Auto-Sampler LY-502	II-30
Viscometer Unit LY-554	II-31

START-UP AND SHUTDOWN

Start-up

1. Turn on the power switch of the LB-550.
Wait about 20 minutes before measurement until the laser becomes stable.
2. Turn on the power switch of the personal computer.
Windows will be activated automatically.
3. Double-click the icon for LB-550 to activate the application program.
4. Once the program has been started, you may enter into a measurement immediately. For the detailed operations of the software, refer to Part III.

Notes:

- Be sure to turn ON the LB-550 before starting the personal computer. If the LB-550 is turned ON after the personal computer has been started, the LB-550 will not operate.
- When connecting the Preparation Unit LY-501, turn ON the power for the LY-501 before turning ON the power for startup of the LB-550 software. The power switch of the LY-501 is located at the rear of the main unit.
- When measuring viscosity with the viscometer mount type, turn on the power switch of the LB-550 after placing a cell holder for vial into the sample chamber.

Shutdown

1. Quit the LB-550 Software. To do so, take one of the following actions:
 - ① Click on "LB-550 Exit" command in the "File" menu.
 - ② Click on the Close button located at the right end of the title bar.
 - ③ Press the Alt and F4 keys simultaneously.
2. Shut down Windows.
Click on the Start button, and then click on the Shut Down command. The quitting options will be displayed. Select "Shut down the computer?" and then click on the Yes button, or press the Enter key. Windows will be shut down.
3. Turn OFF the LB-550 and the personal computer. The POWER switch for the LB-550 can be found on the rear of the main unit.

If the LB-550 is left ON without using it for a long period of time, the laser will remain active and can shorten its service life.

4. Remove the cell and clean it.

LB-550 Operation

SETTINGS

The LB-550 software has 2 types of window:

- Distribution window
- 3D window

Only one of them is displayed on the screen at a time.

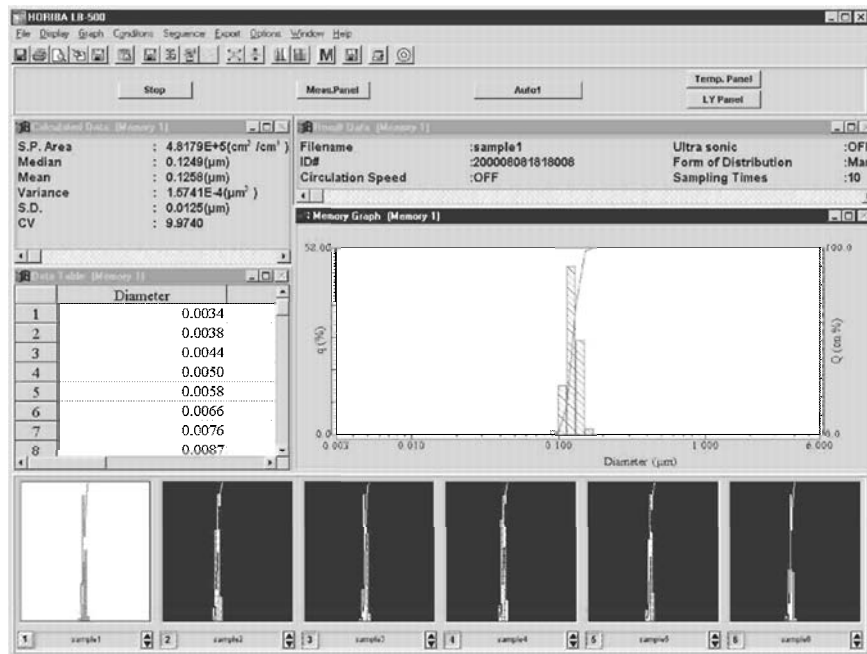


Figure 1: Distribution Window

You can choose the window by clicking the name of the window in the "Display" menu.

Use "Measurement panel" for measurement.

Measurement panel is displayed by clicking "Meas. Panel" in the "Display" menu, or clicking the "Meas. Panel" button on the screen.

"3D window" is used when you want to make a "3 dimensional graph".

Set Conditions

Set conditions before measurement.

These conditions can be saved as a "condition file" (click "condition files" in the "File" menu).

System Condition

Click "System Condition" in "Conditions" menu to open the following window.

Auto Functions

On this screen, set up the automatic operation that will be carried out before and after the measurement. It takes long time until there is no fluctuation when larger sample is measured.

"Wait time after case closed" is to wait stable state of sample.

In the viscometer mount type, it is possible to specify the wait time from when viscosity measurement is completed until measurement of particle size distribution is started.

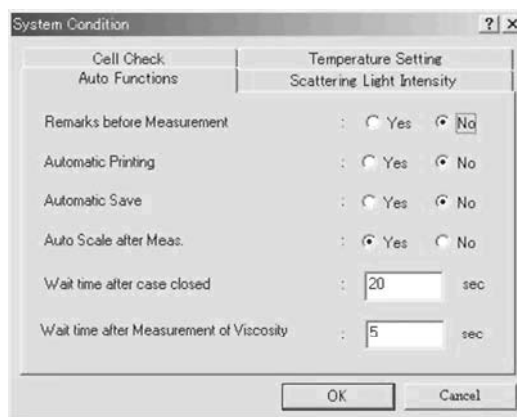


Figure 2: System Condition-Auto Function Tab

Temperature Setting

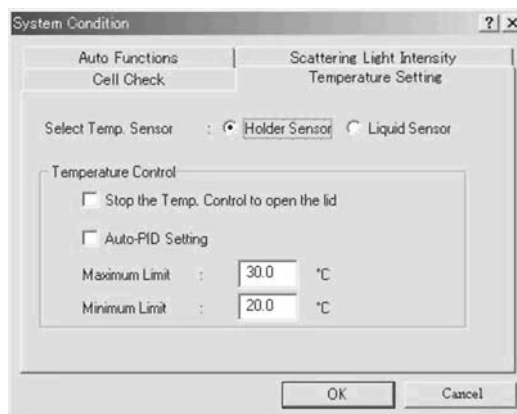


Figure 3: System Condition-Temperature Setting Tab

LB-550 Operation

Selecting a temperature sensor

Specify a temperature sensor.

The LB-550 comes with two temperature sensors: the holder sensor and the liquid sensor. Specify either of the two sensors, which will be used to measure the temperature at which particle size distribution is calculated.

The temperature measured by the specified sensor will be displayed on the measurement panel.

Select the liquid sensor when measuring the temperature exactly. Especially, there may be a great difference between the liquid temperature and the holder temperature when using a disposal cell.

Note:

- A holder sensor is always used to the temperature control.

Note:

- Do not bend cables of the temperature sensors by force, or pull them hard, as these could cause disconnection. Handle the sensors with great care.

Notes:

- When using the sensor-attached lid for the cell, make sure that the sensor cable is tight and untwisted. A slack or twist of the cable may obstruct the lid and cause leakage.
- The liquid sensor is covered with a protection tube to prevent the cable from bending and disconnection. Use the sensor-attached lid with the protection tube attached.

When connecting the optional units

Preparation Unit LY-501

The settings for Circulation / Ultrasonic ON/OFF, circulation time, ultrasonic time and pH / conductivity are available.

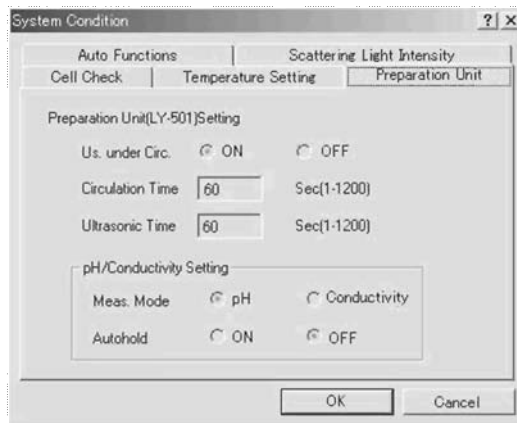


Figure 4: System Condition-Preparation Unit Tab

Note:

- Consult us in case of connecting the pH meter to the LB-550.

Auto-Sampler LY-502

Operation mode and wait time after sample supply are set.

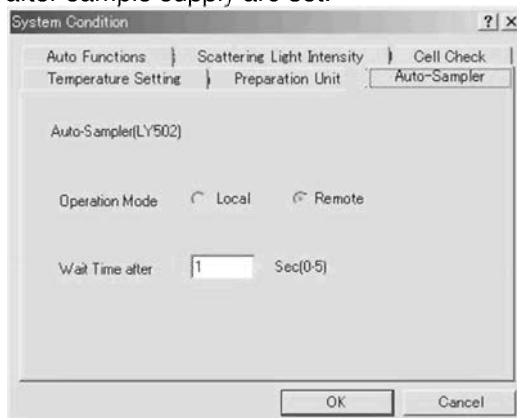


Figure 5: System Condition-Auto-Sampler Tab

Measure Conditions

Click "Measure Condition" in "Conditions" menu to open the following window.

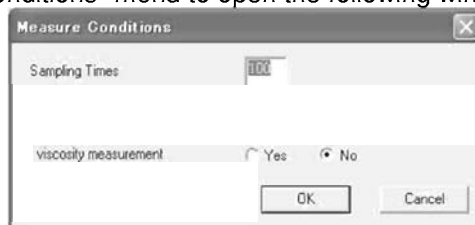


Figure 6: Measure Conditions

Set "Sampling Times"

Usually, "100" is a proper value for sampling times, which means data acquisition is carried out 100 times per measurement. It takes about 100 seconds. When your sample is very dilute, set a larger value to accumulate more scattered light intensity data.

In the viscometer mount type, it is possible to specify whether or not viscosity is measured before particle size distribution is measured.

Note:

- This condition cannot be changed after the measurement.

LB-550 Operation

Display Conditions

1. Click "Display Condition" in "Conditions" menu to open the following window.

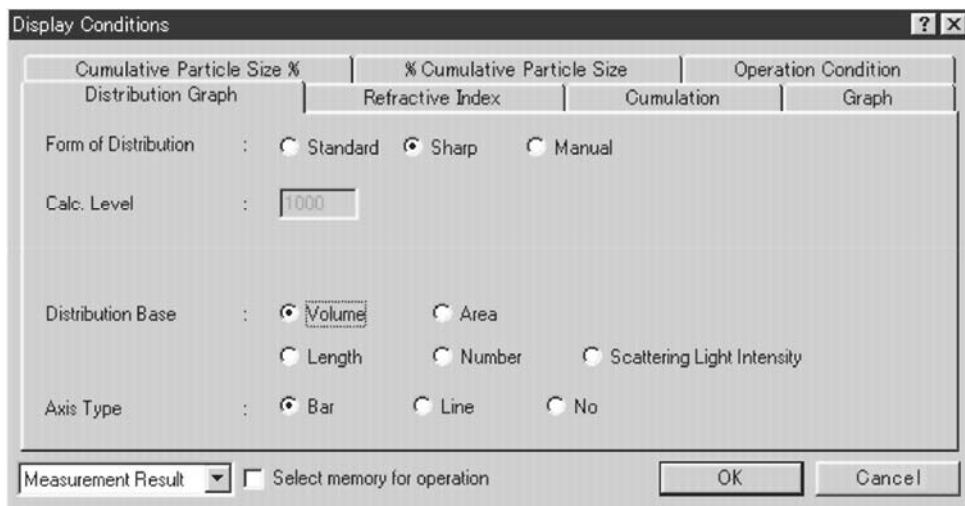


Figure 7: Display Conditions-Distribution Graph Tab

Display conditions can also be changed after measurements.

2. Form of distribution: Click "Standard", "Sharp" or "Manual".

- "Standard" is used for measuring polydisperse samples, which contain particles with various diameters.
- "Sharp" is used for monodisperse samples, where all particles are essentially the same size.
- "Manual" is used to manually select the calculation level.

"Standard" makes the particle size distribution broad, and "Sharp" makes the distribution narrow.

3. "Refraction Index" tab

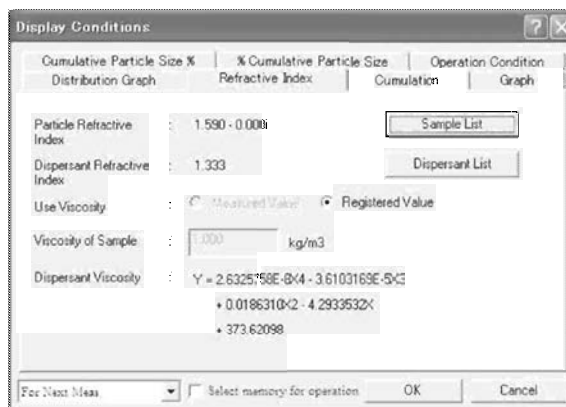


Figure 8: Display Conditions-Refractive Tab.

Specify refractive indexes of the sample and dispersant.

Click on the Sample List button, select refractive index data for the sample, and then click on the OK button.

Reference:

- Select "mono-polystyrene" from the sample list when measuring the standard PSL sample.

Click on the Dispersant List button, select a refractive index and viscosity for the dispersant, and then click on the OK button.

These conditions may be changed after the measurement. Since the measurement results greatly affect the conditions, check the refractive indexes of the sample being measured and the dispersant to be used, before specifying them.

In the viscometer mount type, it is also possible to use actual viscosity values as the viscosity data. For further details of the setting method, refer to Part III.

4. Click "Sample List" button.



Figure 9: Sample List

5. Choose one of the Refractive Index Files for the sample, and click "OK".
6. Click "Dispersant List" button.
7. Choose one of the Refractive Index, viscosity files for the dispersant, and click "OK".
8. Set the other settings in "Display Condition" window and click "OK".

Sample Information

1. Click "Sample Information" in "Conditions" menu to open the following window.

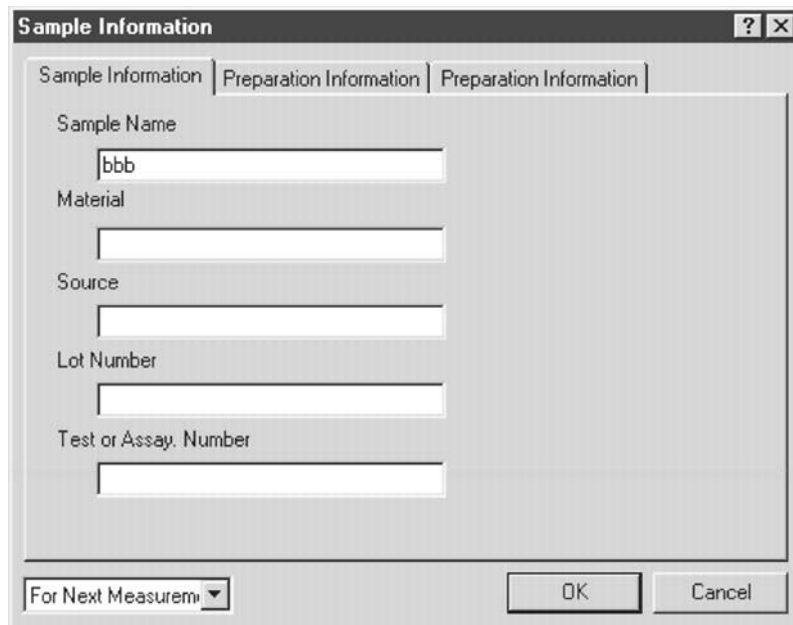


Figure 10: Sample Information

This data will be written to a data file.
It can be changed after the measurement.

LB-550 Operation

Display Item Settings

1. Click "Display Item Settings" in "Options" menu to open the following window.

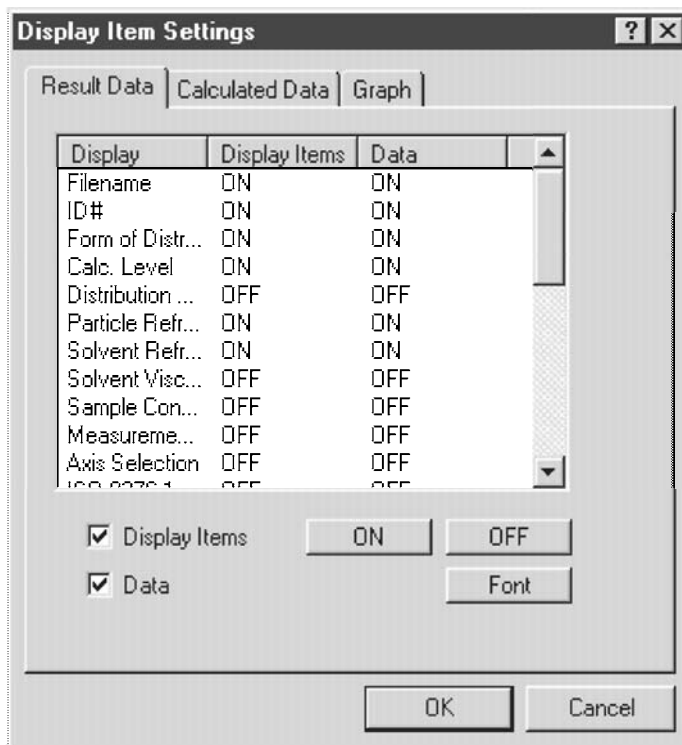


Figure 11: Display Item Settings

Click on the items you want to display along with the measurement result on the screen, and then click on the OK button.

Only the items for which ON has been specified will be displayed on the screen after the measurement.

The items can be changed after the measurement.

If the ON or OFF button is clicked on when the check mark for either Display Items or Data is removed, only the items or data can be displayed on the screen.

2. Select Items to be displayed on the screen.

MEASUREMENT

Sample Measurement

1. Under the "Conditions" menu on the Distribution window, specify the necessary measurement and display conditions.

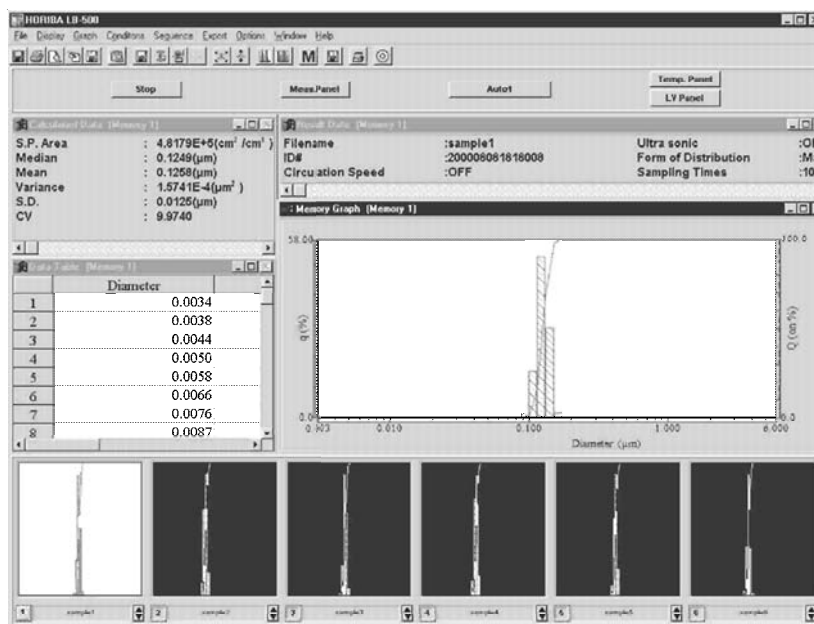


Figure 12: Distribution window

Note:

- The "LY operation panel" can be used only when the Preparation Unit LY-501 is connected. When the LY-501 feature is selected at installation of the LB-550 software, it is displayed.
2. Add the sample liquid to approximately the 80% level of the sample cell.
 3. Place the sample cell into the cell holder so that the transparent surface of the sample cell lines up with the arrow mark on the cell holder, and then close the upper door of the sample chamber.

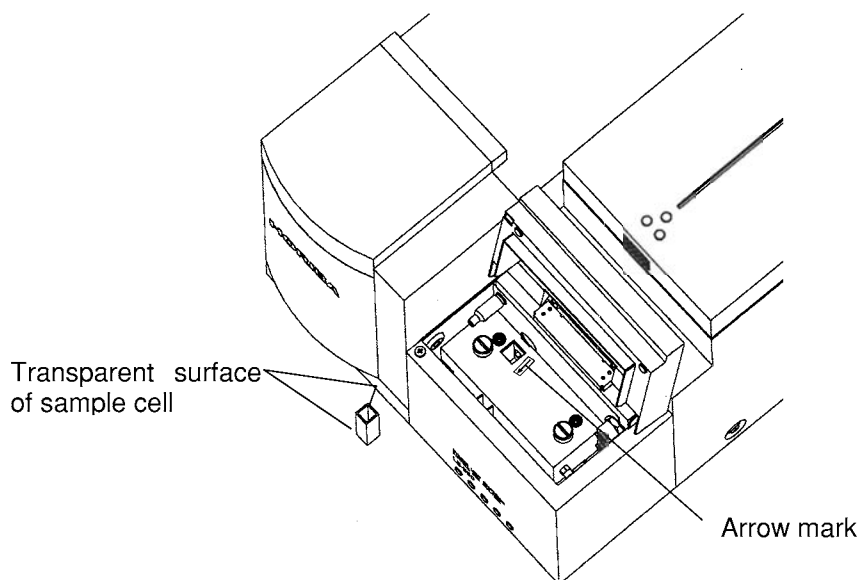


Figure 13: Inserting the sample cell

LB-550 Operation

4. Click on the Meas.Panel button to display the measurement panel.

Note:

- The "sample concentration auto-adjusting" button can be used only when the Preparation Unit LY-501 is connected. When the LY-501 feature is selected at installation of the LB-550 software, it is displayed.

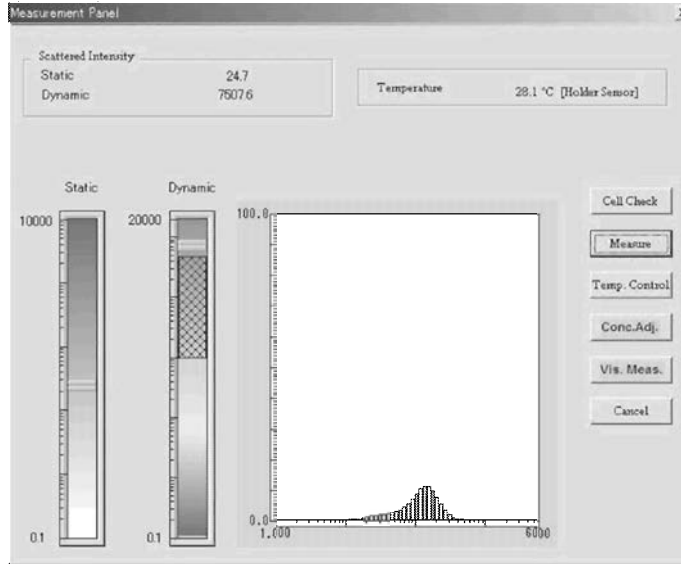


Figure 14: Measurement panel

5. To control the temperature, perform the following steps:

- ① Send approximately 0.4 L/min of the cooling water controlled at a temperature of 25°C.
- ② Click on the Temp. Control button to display the temperature control panel.
- ③ Enter a temperature, and then click on the Temp. Control button to start the temperature control. The measurement cannot be started until the specified temperature has been reached.

Note:

- When the specified temperature is under 20°C or the temperature is lowered for 20°C or more range, be sure to circulate cooling water.

Note:

- Do not bend cables of the temperature sensors by force, or pull them hard, as these could cause disconnection. Handle the sensors with great care.

Notes:

- When using the sensor-attached lid for the cell, make sure that the sensor cable is tight and untwisted. A slack or twist of the cable may obstruct the lid and cause leakage.
- The liquid sensor is covered with a protection tube to prevent the cable from bending and disconnection. Use the sensor-attached lid with the protection tube attached.

6. Using the sample concentration monitor on the Measurement Panel, check that the concentration falls within the proper range, and then click on the Measure button. The measurement for particle size distribution will be started, and the measurement result will be displayed on the Distribution window.

Note:

- When clicking the Measure button with the condition that the value of the sample concentration monitor is out of the proper range, the message "Concentration over the upper limit." or "Concentration under the lower limit." is displayed and the measurement cannot be performed. Adjust the sample concentration again to settle in the proper range.

Note:

- Scattered light intensity monitor graph (Dynamic) is displayed in the color gradation of green, yellow and red.
The level bar on the green colored range (100 to 20000) indicates that scattered light is sufficient for the measurement.
The level bar on the yellow colored range (2 to 100) indicates that scattered light is weak. Perform appropriate preparation such as filtering in order to remove impurities before measurement.
The level bar on the red colored range (0.1 to 2) indicates that the scattered light is very weak, and the light intensity is lowest limit for measurement.
7. The message "Delete" is displayed at the end of the measurement when the data is in the measurement result memory. Clicking "OK" is to save the new measured data on the previous data in the measurement result memory and the new data is displayed on the measurement result screen.

Note:

- It is possible not to display this message. Refer to "Customize" (page III-99).

Measurement Results

When the measurement for particle size distribution is finished, the measurement result graph, data table, measurement result data, and calculation result data will be displayed on the Distribution window.

For the setting of the displayed data and the meanings of the displayed values, see "Options Menu" (page III-88).

Checking for Dirt

Excessive dirt on the cell will affect the measurement accuracy of the LB-550.

For this reason, the LB-550 provides a function for checking the amount of dirt on the cell.

To check the dirt on the cell, put only the dispersant in the cell (without a sample), and click on the Cell Check button on the Measurement Panel.

A message whether the dirt level exceeds a given level will be generated.

This specified dirt level on the cell can be changed by clicking on the Cell Check tab on the System Condition under the "Conditions" menu.

The default value for the Cell Check is set to 30.

LB-550 Operation

Save

File Operations

Once the particle size distribution has been obtained after the measurement, the measurement data may be saved as a file.

To save a measurement data file, use Measurement Data File window.

The saving procedure is briefly described below. For further details, refer to Part III.

Operation procedure

1. Click on "Measurement Files" in the "File" menu on the Distribution window. The Measurement Files dialog box will open (Figure 15).
2. Click on the File Name text box. A vertical cursor will appear in the text box prompting you to enter a file name for the measurement data.
3. After you have entered a file name, click on the Save button in the dialog box. The measurement file will be saved under the file name entered in the text box.

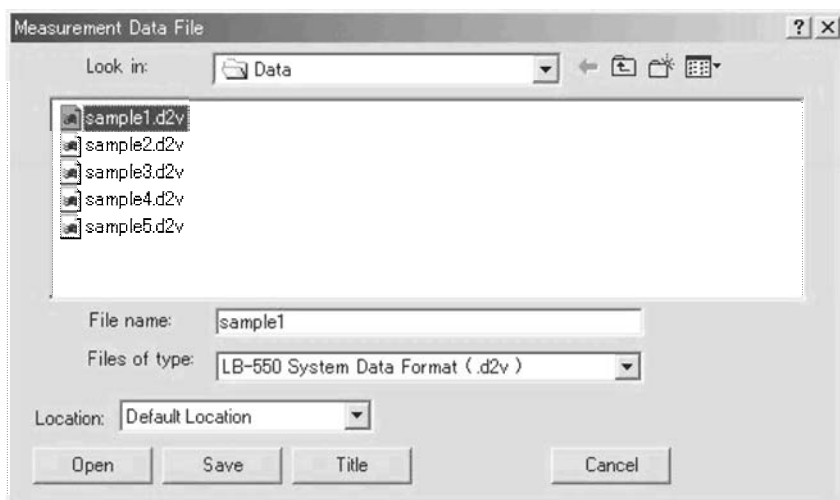


Figure 15: Measurement Files dialog box

To call up the saved measurement file, click on that file in the list displayed in the dialog box. The file name will be displayed in the text box. Click on the Open button.

The obtained particle size distribution data may be saved in the ASCII format so that it can be used in other commercial application software. Refer to "Export Menu" (page III-87).

Clicking on the Title button will enable you to modify the sample information and folders.

CLEANING THE CELL

Remove the cell from the LB-550 and clean the cell.
Clean the cell with a neutral detergent. Use a cotton applicator or soft cloth for this purpose.

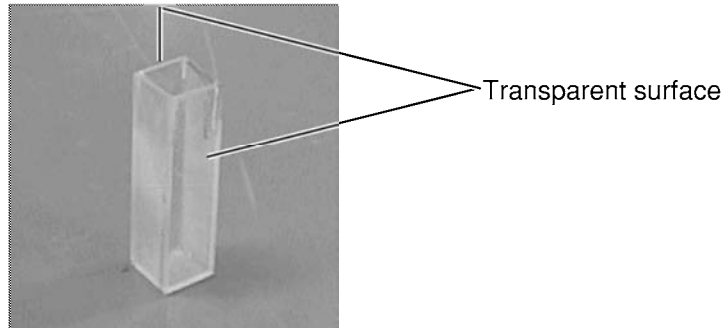


Figure 16-1: Cleaning the glass cuvette cell



Figure 16-2: Cleaning the vial (For Viscometer Unit LY-554 use)

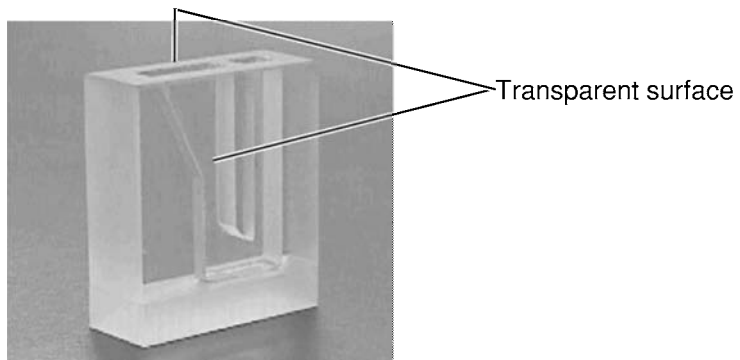


Figure 17: Cleaning the glass flow cell

Notes:

- When handling the sample cell, take care not to directly touch the transparent surface with your fingers and not to damage it.
- The flow cell is used when the Preparation Unit (LY-501) is connected.
- A vial is used when the viscosity meter is connected. When using it repeatedly, wipe it lightly with a swab or soft cloth. Never scrub strongly or wipe with a hard object.

LB-550 Operation

Cleaning the Cell When it is Very Dirty

Cleaning liquid:

- Aqueous solution of neutral detergent
- Aqueous solution of about 5% of hydrochloric acid

Cleaning method:

If the cell cannot be cleaned completely with a cotton swab, put the cell cleaning liquid in a beaker and clean the cell again by a cotton swab.

Notes:

- Do not clean the cell with hydrofluoric acid, strong phosphoric acid, or an alkaline solution.
- Do not put the cell directly in the cleaning bath, or use a high-output ultrasonic cleaner. Otherwise, the cell will be damaged.

Removing the Flow Cell

Remove the flow cell according to the following procedure:

1. Loosen the knurled head screws and remove the cell cover on the top of the flow cell holder.

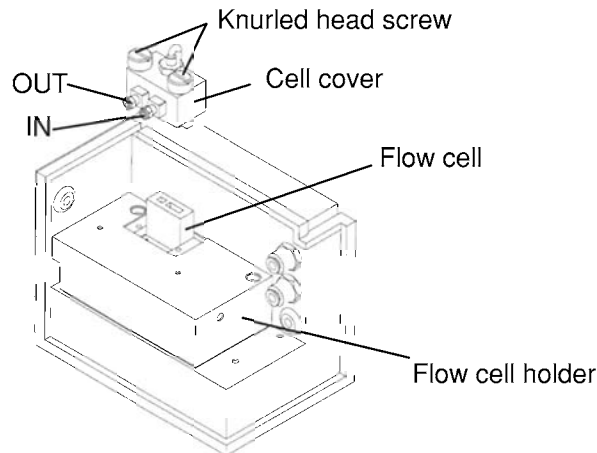
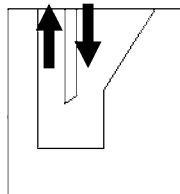


Figure 18: Removing the flow cell

2. Take out the flow cell with great care.
3. Wash the cell according to the "Washing Method" described in the previous page.
4. Set the flow cell in the cell holder so that the liquid inlet is positioned at the right side and the cell outlet at the left side viewing to the front of the LB-550.

Sample flow



Front side of LB-550

Figure 19: Flow cell

Note:

- The sensor cable is fixed on the side of the cell cover by the wire clamp to prevent the cable from bending and disconnection. Use the sensor-attached lid with the cable fixed by the wire clamp.

MEASUREMENT RESULT

When the particle size distribution measurement has been completed, a measurement result graph, a data table, measurement result data, and a calculated result are displayed as graphs or values on the Distribution window.

This chapter describes the displayed details of the particle size distribution graph obtained by the measurement.

Some information may not be displayed or the number of displayed items may be different depending on your display method.

Result Data

Filename:

Displayed within the title bar located at the top of this window. Where a file name is not entered yet, nothing is displayed.

ID#:

A serial number. This number is added to the data whenever a measurement is taken, and cannot be changed after the measurement.

Form of Distribution:

Form of distribution based on particle size distribution calculation.

Calc. Level:

Repetition count for particle size distribution calculation.

Distribution Base:

Distribution Base for displayed measurement data.

Particle Refractive Index:

Specified refractive index of the sample.

Dispersant Refractive Index:

Specified refractive index of the dispersant.

Dispersant Viscosity:

Specified viscosity of the dispersant.

ISO 9276-1 Format:

Shows ON if the graph is displayed in accordance with ISO or JIS.

Sample Name:

Information entered as sample information, which consists of:

Sample Name, Material, Source, Lot Number, Test or Assay Number, Sample Preparation, Dispersant, Dispersion Steps, Dispersion Verification, Remarks, Remarks 1, Remarks 2, Remarks 3, Remarks 4, and Remarks 5.

Calculated Data

S.P.Area:

A total (cm²) of surface areas of particles per cm³. This is calculated from the particle size distribution data. In general, it does not match with the surface area measured by other measurement methods.

Median:

Particle size equivalent to cumulative 50%.

LB-550 Operation

Diameter on %:

Up to ten particle sizes (% on Diameter) corresponding to the cumulative distribution value specified in Display Condition are displayed in accordance with the setting.

% on Diameter:

Up to ten cumulative distribution values corresponding to the particle size specified in Display Condition are displayed in accordance with the setting.

Mean:

A value obtained by arithmetically averaging the density distribution with the equation below:

$$\text{Mean} = \frac{\sum \{q(J) \times X(J)\}}{\sum \{q(J)\}}$$

where

J: Particle size division number
q(J): Density distribution value (%)
X(J): Typical size (μm) in the "J"th particle size range

Variance:

Distribution variance obtained using the equation below:

$$\text{Variance} = \sum \left[(X(J) - \text{Mean})^2 \frac{q(J)}{100} \right]$$

where

J: Particle size division number
q(J): Density distribution value (%)
X(J): Typical size (μm) in the "J"th particle size range
Mean: Mean value (μm)

S.D.:

Variance value without a root.

CV:

Standard deviation divided by Mean value.

Mode:

Particle size at the peak of the density distribution graph, which maximizes the density distribution value.

Span:

Distribution span obtained using the equation below. This is not displayed if neither of % on Diameter is specified.

$$\text{Span value} = (\text{Diameter on \% A} - \text{Diameter on \% B}) \div \text{Median}$$

where

Diameter on % A: First value to be entered in Display Condition
Diameter on % B: Second value to be entered in Display Condition

Geo. Mean:

Geometrically averaged density distribution obtained with the equation below:

$$\text{Geo. Mean} = 10^{\frac{\sum (\log X(J) \times q(J))}{\sum q(J)}}$$

where

J: Particle size division number
q(J): Density distribution value (%)
X(J): Typical size (μm) in the "J"th particle size range

Geo. Variance:

Distribution variance obtained with the equation below:

$$\text{Geometric variance} = \sum \left[(\log X(J) - \log(\text{Mean}))^2 \frac{q(J)}{100} \right]$$

where

- J: Particle size division number
- q(J): Density distribution value (%)
- X(J): Typical size (μm) in the "J"th particle size range
- Mean: Geo. Mean value (μm)

Geo. S. D.:

Geometric variance value without a root.

Diffusion Coefficient:

Diffusion coefficient delivered from the Stokes-Einstein's equation:

$$D = KT / 3\pi\eta d$$

where

- d: Diameter specified in the Operation Conditions screen

R Parameter:

Value showing the matching level between the calculated result of particle size distribution based on the refractive index used and actually measured value of data on scattered light. The matching level increases as it becomes closer to zero. This value is used to select a refractive index when a sample with an unknown refractive index is being measured.

R Parameter is obtained with the equation below:

$$R = \frac{1}{N} \sum_{i=1}^N \left\{ \frac{1}{y(x_i)} |y_i - y(x_i)| \right\}$$

where

- y_i : Actually measured data on scattered light
- $y(x_i)$: Data on scattered light obtained from refractive index file data and displayed particle size distribution
- N: Number of data point used for operation

Validation:

Validation Result: Shows whether the measurement result is included in the validation standard specified in Operation Condition on the Display Conditions screen (OK or NG). The defined content of the standard is also displayed. Nothing is displayed when the validation standard is defined.

SAMPLING & DISPERSION

Sampling Methods

A representative sample is required in order to measure the particle size distribution of powders or particles suspended in a liquid. This measurement sample must, therefore, have the same particle size distribution as the overall sample.

Obviously, when extracting measurement samples from a large reaction vessel, sample drawn from the top will have a different particle size distribution from samples drawn from the bottom.

Small and heavy particles collect at the bottom of the vessel. This is called "Segregation." Taking samples from the upper and lower levels of the vessel is appropriate if the degree of particle settling is to be measured, but for particle size distribution measurements, meaning-less, unreproducible data will be obtained unless suitable sampling techniques are employed.

Sampling methods for powders

- Take samples of equal weight from throughout the material.
- Use a twin divider or rotary splitter (see Figure 20).
- Use the flow division method. (see Figure 21).

Particles suspended in a liquid

- Do not dilute at once in a large amount of dispersant, but carry out the dilution over several smaller stages.
- Agitate well while diluting.
- Dilute in the same dispersant as the medium used in the original sample.
- Disperse evenly with ultrasonic energy or a stirrer.
- Draw off a measurement sample while agitating and pour it into the cell.

Although the concept is rather difficult to explain, the sampling method must be chosen to suit the properties of the sample, as some particles (particularly fine particles) adhere due to strong surface forces, or cluster together if they are made damp by humidity.

Particles in solids, e.g. pigments in plastics

Dilute the solid containing the particles in an appropriate dispersant

- Use the diluted samples as the measurement sample.
- Remove only the particle and measure.

Clean until the dissolved solid has completely been removed and extract the particles.
All further operations are the same as sampling methods for powder.

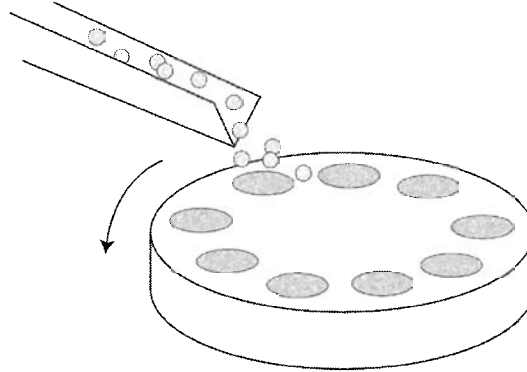


Figure 20: Rotary Splitter Method

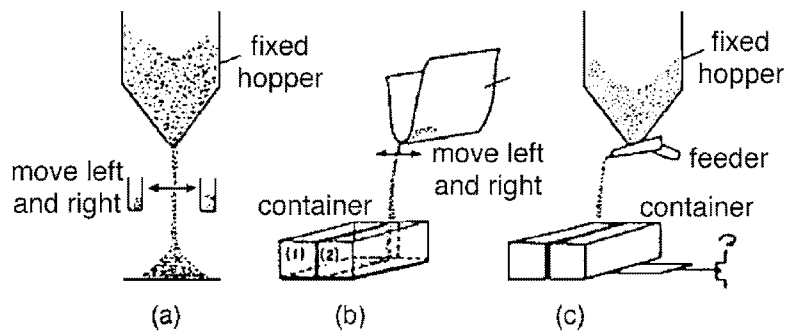


Figure 21: Flow Division Method

LB-550 Operation

Dispersing Methods for Samples

As the LB-550 carries out measurements with the particles in a diluted state, the dispersed state of the particles is an important factor in measurement accuracy.

Even if the sampling of powders is carried out correctly, large particles that should not exist are measured and the distribution is unnatural if particles agglomerate.

Alternatively, when a dispersant of the sort that dissolves the powder is used, particles are measured to be smaller than they are, and this error causes inaccurate results.

In order to carry out accurate measurement without any of the above problems, the dispersant used and the dispersion method must be chosen carefully.

Dispersion Methods

Samples with Stable Particles

Disperse the sample in a ultrasonic bath.

The ultrasonic time depends on the sample. If vibration is carried out for too long, the particles may coagulate or be damaged. However, the ideal ultrasonic time must be experimentally determined for each sample in order to thoroughly disperse the particles.

Samples with Unstable (Fragile) Particles

When measuring such particles, do not apply ultrasonic vibration.

Selecting the Dispersant

It is important that the dispersant properly "wets" the sample powder. The dispersant must be one that does not cause coagulation, dissolution, swelling or chemical reaction.

Water is used in most cases. However, in some cases, measurement may be carried out using organic fluids such as ethanol or ethylene glycol. Use fraction cell (option) for measurement with organic dispersant.

Effect and Type of Dispersant

Powder samples are generally charged, and may coagulate or may not be sufficiently dispersed due to the interaction with the dispersion fluid. The role of the dispersant is to increase this interaction and to disperse each and every one of the particles. Widely used dispersants are sodium polymetaphosphate and sodium pyrophosphate. Surfactants are also used for hydro-phobic (preferentially wet by oil or air) or lipophilic (preferentially wet by oil) particles.

However, if there is an excessive amount of surfactant, it might form micelles and agglomerate. Therefore, care must be taken regarding the concentration and the type of dispersant to be used. The concentration of the dispersant depends on the type of dispersant. A concentration of approximately 1 to 0.2% is widely used.

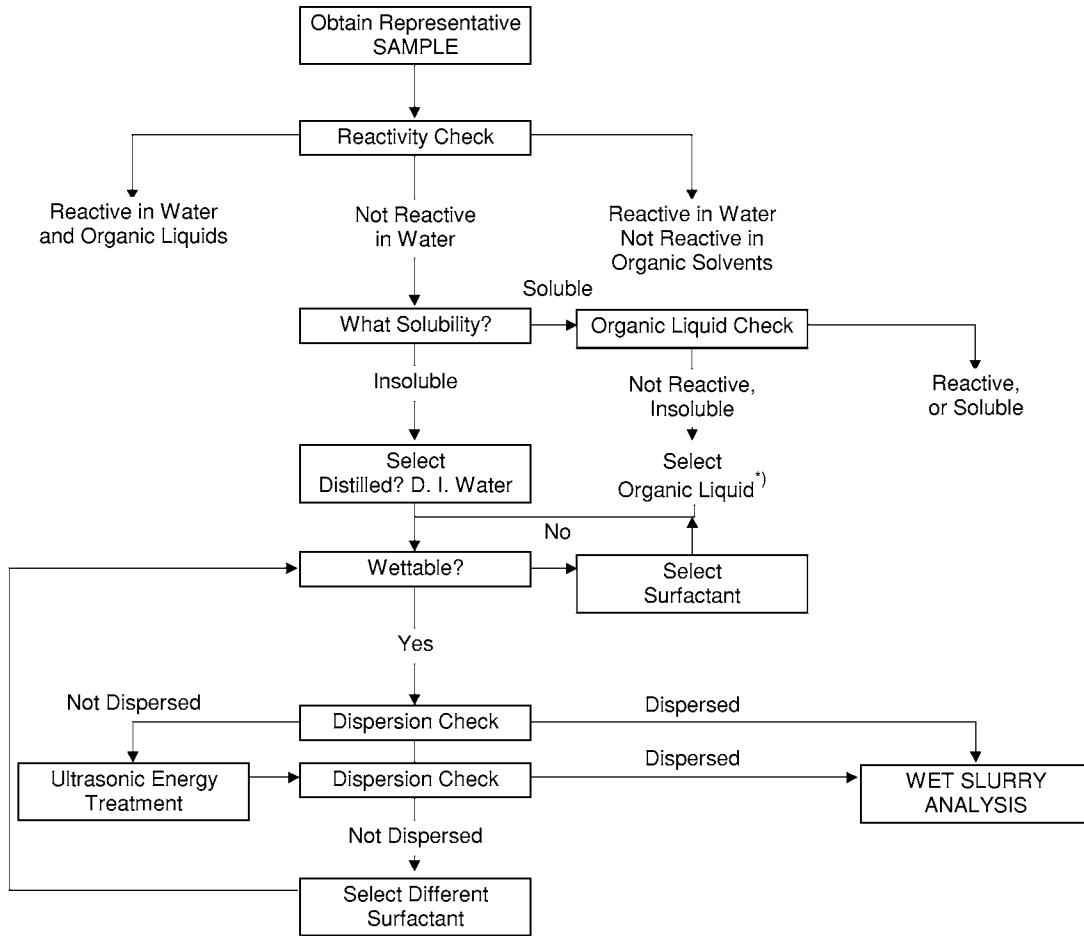


Figure 22: Selection procedure for dispersant when the properties of the sample are unknown

LB-550 Operation

VALIDATION

Measure the standard particle regularly and make sure that the value is in the standard value to confirm whether the instrument is running normally.

This validation can be used to control a quality of product or sample, too.

Reference:

- Polystyrene latex (PSL) is used as the standard particle at the shipment and the pure water is used as the dispersant for the inspection.

General process of the validation

1. Click "Display Condition" in "Conditions" menu.
2. Click "Operation Condition" tab.
3. Click "Setting" button.
4. Input the standard value of the validation.

Reference:

- PSL with 20 nm, 100 nm, 1 μ m in diameter is the inspection standard at the shipment. The standard particle whose median is 20 nm and 100 nm is $\pm 10\%$ of the official value, and the standard particle whose median is 1 μ m is $\pm 15\%$ of the official value.

5. Click "OK" and close "Validation Setting" dialog box.
6. Click "Refractive Index" tab.
7. Click "Sample List" button and select (or input) a refractive index of the standard particle.

Reference:

- Select "mono-polystyrene" of refractive index list when using monodisperse PSL. Don't use "mono-polystyrene" when mixed PSL are used, but select "multi-polystyrene". When selecting "mono-polystyrene", the repetitive frequency becomes 3,000 times, and other operation conditions are automatically set to the best value for the validation at PSL. For that reason, all the other condition settings are not needed.

8. Click "OK" and close "Sample List" dialog box.
9. Click "Dispersant List" and select (or input) a refractive index and viscosity of the dispersant.
10. Click "Distribution Graph" tab and select "Form of Distribution".
11. Select "Next Measurement" from the memory list at left down side of "Display Condition" dialog box.
12. Click "OK" and close "Dispersant List" dialog box.
13. Click "OK" and close "Display Condition" dialog box.
14. Click "Display item setting" in "Options" menu.
15. Click "Operation Result Data" tab and click "Validation Result", then click "OK" button.
16. Click "OK" button and close "Display item setting" dialog box.
17. Perform measurement of the standard sample following "Sample Measurement" on page II -8.
18. Either "OK" or "NG", the difference from the standard value (%), and the standard are displayed on the measurement result screen as the validation result.
19. Saving data and printing out should be performed according to the necessity.

LIST OF THE REFRACTIVE INDEX VALUES

Inorganic Substances

**Table 1: Taken from 1) "Handbook of Chemistry and Physics", C.R.C. Press,
2) "American Institute of Handbook", MaGraw-Hill and
3) "The Merck Index", MERCK & Co., Inc.**

Substance	Composition	Specific Gravity	Refractive index, n_1
Lead white	$Pb_3(CO_3)_2(OH)_2$	6.8	2.02
Zinc flower	ZnO	5.6	2.00
Titanium oxide(rutile)	TiO ₂	4.2	2.75
Titanium oxide(anatase)	TiO ₂	3.9	2.50
Zinc sulfide	ZnS	4.0	2.37
Lead titanate	PbTiO ₂	7.3	2.70
Zirconium oxide	ZrO	5.7	2.40
Barium sulfate	BaSO ₄	4.4	1.62
Barium carbonate	BaCO ₃	4.3	1.60
Calcium carbonate	CaCO ₃	2.8	1.58
Gypsum	CaSO ₄ · 2H ₂ O	2.4	1.55
Alumina	Al ₂ O ₃	-	1.66
Iron oxide	Fe ₂ O ₃	4.8	2.90
Red lead	Pb ₃ O ₄	8.9	2.42
Mercuric sulfide	HgS	8.0	2.95
Lead chromate	PbCrO ₄	6.0	2.40
Cadmium sulfide	CdS	4.4	2.42
Zinc yellow	ZnCrO ₄	3.5	1.87
Strontium yellow	SrCrO ₄	-	1.96
Barium yellow	BaCrO ₄	4.4	1.63
Chrome green	-	4.1	2.40
Emerald green	-	3.2	1.97
Chromium oxide	Cr ₂ O ₃	5.1	2.50
Cobalt green	CoO · xZnO	-	1.97
Ultra marine	Na ₇ Al ₆ Si ₆ O ₂₄ S ₃	2.4	1.57
Prussian blue	Fe ₄ [Fe(CH) ₆] ₃	1.8	1.56
Cobalt blue	CoO · xAl ₂ O ₃	3.8	1.74
Celuriene	CoO · xSnO ₂	-	1.84
Cobalt violet	Co ₃ (PO ₄) ₂	-	1.72
Manganese violet	(NH ₄) ₂ Mn ₂ (P ₂ O ₇) ₂	-	1.70
Silver chloride	AgCl	-	2.08
Fluorite	CaF ₂	-	1.43
Germanium	Ge	-	4.10
Potassium bromide	KBr	-	1.58
Potassium chloride	KCl	-	1.80
Lithium fluoride	LiF	-	1.39
Magnesium oxide	MgO	-	1.76
Silicon	Si	-	3.50
Quartz	SiO ₂	-	1.45
Rock crystal	SiO ₂	-	1.54
Diamond	C	-	2.41
Sapphire	Al ₂ O ₃	-	1.76
Magnesium fluoride	MgF ₂	-	1.37
Mica	KAl ₂ (AlSi ₃ O ₁₀)(OH) ₂	-	1.59
	BaCa ₂ (C ₃ H ₅ O ₂) ₆	-	1.45
Barium fluochloride	BaC ₁₂ · BaF ₂	-	1.64
Barium fluoride	BaF ₂	-	1.47

LB-550 Operation

Substance	Composition	Specific Gravity	Refractive index, n_1
Barium phosphate	BaHPO ₄	-	1.62
Barium sulfide	BaS	-	2.16
Calcium alminate	Ca ₃ Al ₂ O ₃	-	1.71
Calcium borate	CaO · B ₂ O ₃	-	1.60
Carbon	C	-	1.92-0.522
Chromium oxide	Cr ₂ O ₃	-	2.50
Copper oxide	Cu ₂ O	-	2.71
Copper sulfate	CuSO ₄	-	1.73
Magnesium orthoborate	3MgO · B ₂ O ₃	-	1.65
Potassium carbonate	K ₂ CO ₃	-	1.50
Potassium hydrogencarbonate	KHCO ₃	-	1.48
Potassium cyanide	KCN	-	1.41
Potassium chlorate	KClO ₃	-	1.62
Sodium bromide	NaBr	-	1.64
Sodium cyanide	NaCN	-	1.45
Sodium metasilicate	Na ₂ SiO ₃	-	1.52
Sodium sulfate	Na ₂ SO ₄	-	1.55
Strontium dichromate	SrCr ₂ O ₇ · 3H ₂ O	-	1.71
Strontium fluoride	SrF ₂	-	1.44
Strontium sulfide	SrS	-	2.11
Zinc metasilicate	ZnSiO ₃	-	1.62
Iron	Fe	-	2.4-1.4i
Zinc	Zn	-	2.4-5.5i
Gold	Au	-	0.34-3.2i
Silver	Ag	-	0.19-3.4i
Copper	Cu	-	0.6-3.6i
Aluminum	Al	-	1.6-5.4i
Antimony	Sb	-	3.2-5.0i
Magnesium	Mg	-	0.4-4.6i
Manganese	Mn	-	2.5-4.0i
Nickel	Ni	-	1.8-3.6i
Platinum	Pt	-	2.9-4.5i

* n_0 of this value is methanol.

Organic Substances

**Table 2: Taken from 1) "Handbook of Chemistry and Physics", C.R.C. Press,
2) "American Institute of Handbook", McGraw-Hill and
3) "The Merck Index", MERCK & Co., Inc.**

Substance	Composition	Specific Gravity	Refractive index, n_D
Asphalt	-	-	1.63
Ebonite	-	-	1.66
Opal	-	-	1.44
Canadian balsam	-	-	1.52
Amber	-	-	1.54
Ivory	-	-	1.54
Vinyl chloride resin	-	-	1.54
Vinylidene chloride resin	-	-	1.61
Vinyl acetate resin	-	-	1.46
Silicon oil	-	-	1.40
Tetrafluoroethylene resin	-	-	1.35
Nylon	-	-	1.53
Polyethylene	-	-	1.53
Polystyrene	-	-	1.60
Methylmethacrylic acid resin	-	-	1.49
Melamine resin	-	-	1.60

LB-550 Operation

Dispersant Refractive Index

Table 3: Taken from 1) "Handbook of Chemistry and Physics", C.R.C. Press, 2) "International Critical Tables", I.C.T. Press

Dispersant	Refractive index, n_1	Source book
Water	1.3330	I.C.T
Ethanol	1.3610	I.C.T
Isopropanol	1.3780	I.C.T
Methanol	1.3290	I.C.T
Hexane	1.3760	I.C.T
Cyclohexane	1.4273	I.C.T
Cyclohexanol	1.4606	I.C.T
Acetone	1.3591	I.C.T
Tetrahydrofuran	1.4040	C.R.C
o-Xylene	1.5058	I.C.T
m-Xylene	1.4973	I.C.T
p-Xylene	1.4956	I.C.T
Methyl Ethyl Ketone	1.3791	I.C.T
Dichloromethane	1.4237	I.C.T
Glycerin	1.4729	I.C.T
n-butanol	1.3993	I.C.T
sec-butanol	1.3970	I.C.T
tert-butanol	1.3870	I.C.T
isobutanol	1.3960	I.C.T
Cyclohexanone	1.4526	I.C.T
Decane	1.4209	I.C.T
Ethyl Acetate	1.3707	I.C.T
Ethylene Glycol	1.4290	C.R.C
Methyl Isobutyl Ketone	1.3959	I.C.T
Toluene	1.4962	I.C.T

OPTIONAL UNIT

Preparation Unit LY-501

LY operation panel

If you click the "LY operation panel" button on the main screen or select the "LY operation panel" on the "Display" menu, the LY operation panel is displayed.

The basic operations of the LY-501 can be controlled from this panel.

Note:

- When the measurement panel is open or during measurement, this LY operation panel cannot be opened.

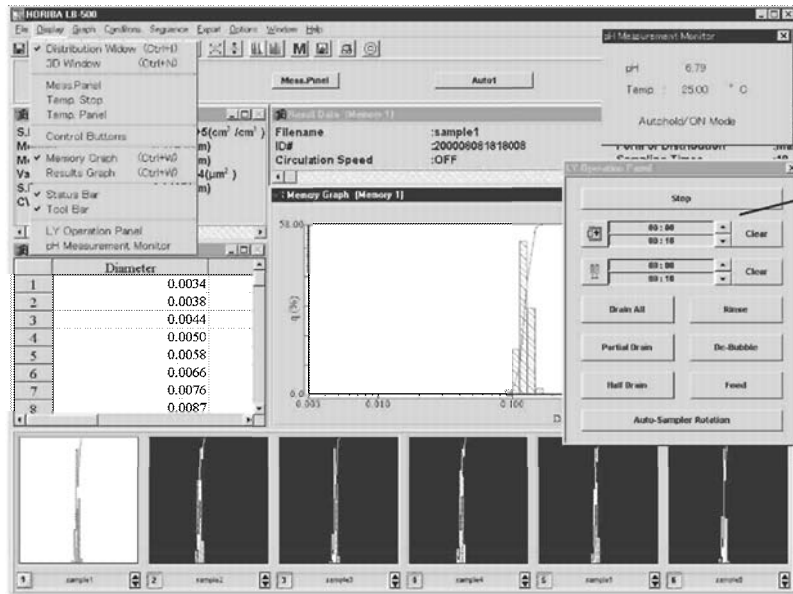


Figure 23: Distribution Window

The basic operation of each button on the LY operation panel is as follows:

"Stop" button

All operations are stopped.



(Circulation) button

Circulation is performed only for a set time. The upper row shows the total operating hours and the lower row shows the remaining operating hours. Pushing the "Clear" button will reset the upper row to zero and return the lower row to the default value.



(Ultrasonic) button

The ultrasonic operation is performed only for a set time. The upper row shows the total operating hours and the lower row shows the remaining operating hours. Pushing the "Clear" button will reset the upper row to zero and returns the lower row to the default value.

"Drain All" button

The sample liquid in the bath is completely drained.

"Half Drain" button

The sample liquid is drained to the middle level in the bath.

"Partial Drain" button

The sample liquid in the bath is drained for 3 seconds.

LB-550 Operation

"Rinse" button

Washing (rinse) operation is executed.

"De-bubble" button

De-bubbling in the cell is executed.

"Feed" button

Dilution is fed to the high water level.

"Auto-Sampler Rotation" button

The rotating plate of the Auto-Sampler (LY-502) is rotated by one cell in clockwise direction.

The circulation / ultrasonic operation can be set at the "Preparation Unit" of the "System Conditions" on the "Set" menu.

If "ON" is selected for the Ultrasonic ON/OFF at circulation, it is set to start the ultrasonic operation simultaneously with circulation. However, if the water level at circulation is below a fixed standard level (approx. 60 mL), the ultrasonic operation is automatically stopped.

In the settings for circulation time and ultrasonic time, the default values for circulation and ultrasonic operation in the operation panel can be changed. The setting for repetition frequency of the rinse operation can also be changed.

Note:

- For the sample concentration auto-adjusting" on the measurement panel, refer to "Sample Concentration Auto-Adjusting (only when the Preparation Unit LY-501 is Connected)" (page III-13).

pH measurement monitor

Set the type of the pH meter in the pH/conductivity panel at the system administration. Then, click the "pH measurement monitor" on the "Display" menu. The pH measurement monitor is displayed on the main screen.

The pH value or conductivity in the LY-501 bath as well as the temperature in the bath are displayed in real time on the pH measurement monitor.

The setting for the pH/conductivity measurement is performed at the "Preparation Unit" of the "System conditions" on the "Set" menu.

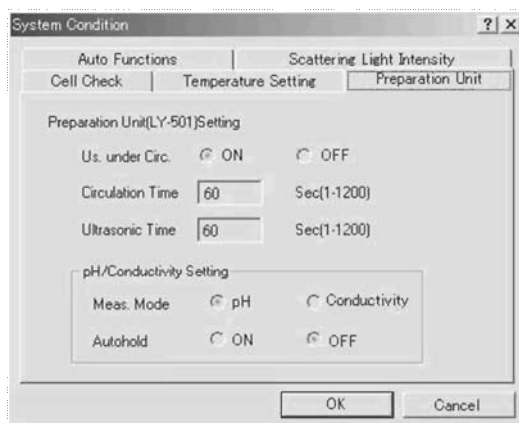


Figure 24: System conditions setting -Preparation Unit Tab

Meas. Mode

Select either pH measurement or conductivity measurement.

Autohold

Switch to the Autohold measurement (This selection is determined depending on the fluctuations of the value) to obtain measured values.

Notes:

- If the pH meter connection "NO" is selected in the "pH meter setting" panel at the system administration, the "pH measurement monitor" on the "Display" menu is not displayed. The area turns gray and inoperative for setting.
- In the setting for the type of the pH meter, if the "no connection of the pH meter" or "power OFF for the pH meter" is selected, all the items related to the pH/conductivity turn gray and inoperative for setting.

Note:

- Do not perform the pH measurement for high viscosity of sample. It will cause the liquid blocking of the pH electrode.

Note:

- Consult us in case of connecting the pH meter to the LB-550.

Before starting up the LB-550 software, connect the pH meter to the personal computer. Then, always start up the LB-550 software only after the power is turned ON.

The settings for "Port" and "Type" to be connected should be performed on the "pH meter setting" tab at the system administration.

Refer to the "pH Meter Settings with the LY-501" (page III-110).

Operation procedure

1. Perform the pH/conductivity setting at the "System conditions setting" on the "Set" menu.
2. If the "pH measurement monitor" on the "Display" menu is check marked, the monitor window as shown in Figure 25 is displayed.
On this monitor, an instantaneous value by either pH measurement or conductivity measurement, which has been set at the above 1, is displayed in real time.

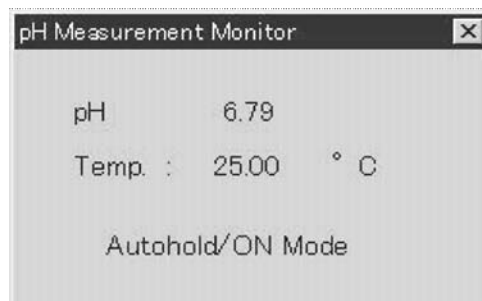


Figure 25: pH Measurement Monitor

3. When the measurement button is pushed, the pH measurement monitor is closed automatically. If the particle size distribution analysis is started here by pushing the measurement button, pH/conductivity is measured according to the Autohold ON/OFF selected at 1. When the Autohold is ON, the pH/conductivity measurement time is required for approx. 3 minutes at the maximum. If the Autohold measurement is continued even after the particle size distribution analysis, the message of "Autohold measuring" is displayed. When the Autohold is OFF, the pH/conductivity value last fetched during the fetch of the particle size distribution data is adopted as the result.

Notes:

- The pH measurement monitor is used to check the pH/conductivity value other than at the LB-550 measurement. It doesn't need to open by all means. Display the monitor as necessary.
- If you have anything unknown or unclear regarding the operation of the pH meter, refer to the operation manual for the pH meter in your possession.

Note:

- Consult us in case of connecting the pH meter to the LB-550.

LB-550 Operation

Auto-Sampler LY-502

The remote/local switching is performed on the "Auto-Sampler" tab at the "System conditions" of the "Set" menu.

The "wait time after sample supply" can also be set.

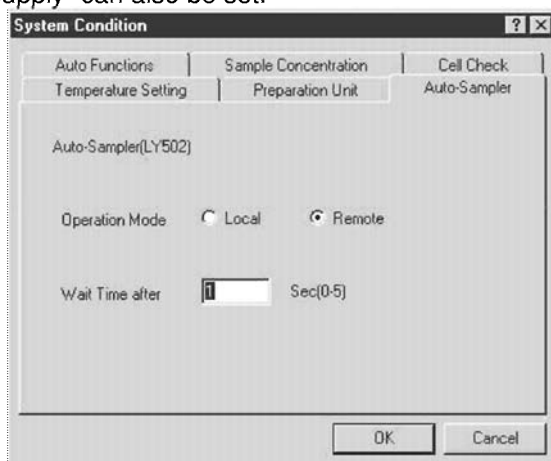


Figure 26: System conditions setting-Auto-Sampler Tab

Operation mode

The Auto-Sampler LY-502 is operated in "Remote" mode on the LB-550 software and in "Local" mode controlled with the switches on the LY-502 unit.

Wait time after sample supply

During this wait time, the LY-502 cannot perform the following operations. When using high viscosity of sample, the liquid dripping time from the cell after sample supply will be longer. Set a longer value (Default: 1 second).

Viscometer Unit LY-554

In the viscometer mount type, a viscosity measuring function can be used. When measuring viscosity, be sure to place a cell holder for vial.

Note:

- To prevent damage to the viscosity meter, do not keep the upper door of the sample chamber closed when moving the viscosity meter probe.

There are the following two modes for viscosity measurement:

- Sample viscosity measurement at the time of particle size distribution measurement:
Execute viscosity measurement when particle size distribution is measured. The actual viscosity measurement value is used as the viscosity parameter required for distribution calculation.
- Measurement of only sample viscosity:
Execute only sample viscosity measurement. Particle size distribution measurement is not executed.

Note:

- The viscosity measuring range is 0.40 mPa·s to 10.0 mPa·s.
Regarding the actual viscosity value used for particle size distribution measurement, however, recommended upper limit value is 3 mPa·s.
When a sample viscosity exceeds 3 mPa·s, particle size distribution may not be measured accurately.
When the viscosity value exceeds 3 mPa·s, a message, "Continue measurement?" is displayed.
If "Yes" is selected, particle size distribution is measured using the actual viscosity measurement value (3.01 mPa·s to 10.0 mPa·s).

Preparation for viscosity measurement

1. Remove the protecting cap of the oscillator.
Never twist or bend the oscillator, which might cause malfunctions.

Note:

- Keep the protecting cap, because it is used when relocating the LB-550.

2. Set the vial to "MEASUREMENT" of the vial holder.
3. Pour the sample and adjust the liquid level of the sample to the same level as the top surface of the holder, and then put the sample into the vial. Measure of difference between sample liquid level and the top surface of the holder is within ± 3 mm.

Note:

- If difference between sample liquid level and the top surface of the holder is out of ± 3 mm, the accuracy of viscosity measurement becomes low.

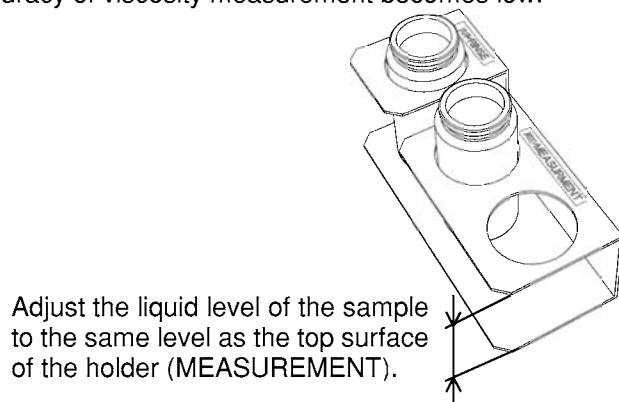


Figure 27: Holder for vial

LB-550 Operation

4. Open the upper door of the sample chamber, and place a vial containing a sample into the chamber.
5. Move the viscosity meter probe slowly in the downward direction until you hear it click.

Preparation for viscosity measurement is now complete.

Sample viscosity measurement at particle size distribution measurement

Setting

Select the execution of viscosity measurement as the condition for measuring particle size distribution.

1. Select [Measurement condition setting] from the [Setting] menu, and open the “measurement condition” dialog box.

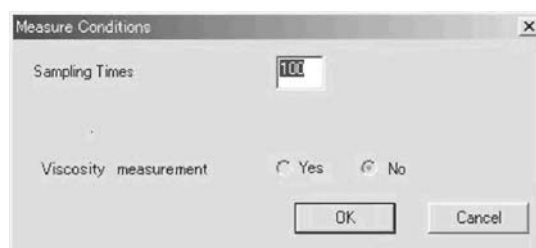


Figure 28: Measurement condition

2. Select “Yes” for Sample viscosity measurement, and then click [OK].
3. Select [Display condition setting] from the [Setting] menu, open the [Display condition] dialog box, and then click the [Refractive index] tab.

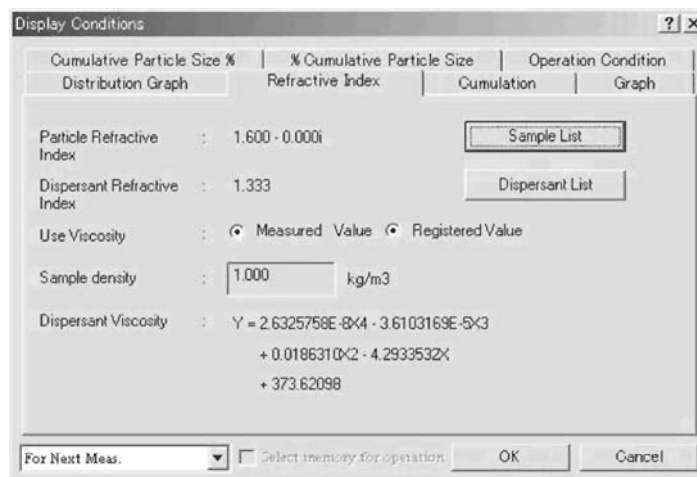


Figure 29: Display condition

4. Select “Next measurement” from Calculation memory at the bottom.
5. Make sure that the “actual viscosity measurement value” is used, and then input the sample density. The actual viscosity value divided by the sample density is used for calculation.

Note:

- Viscometer LY-554 is an oscillating type meter. It shows a viscosity value multiplied by density. (It shows the viscosity value regarding the liquid density as 1 g/cm³.) Therefore, the viscosity parameter used for calculating particle size distribution is the actual viscosity value divided by the sample density.
6. Click the [OK] button.

Execution of measurement

1. Click the [Measure] button on the main window. Then the “measurement panel” is displayed.

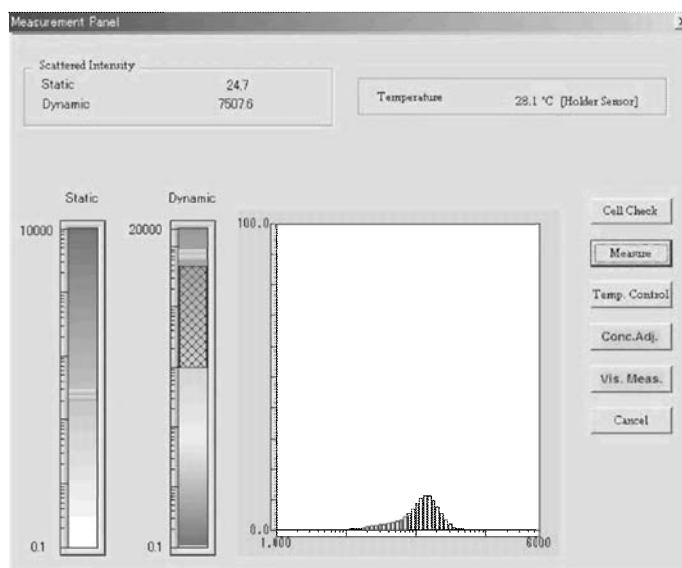


Figure 30: Measurement panel

2. Click the [Measure] button on the measurement panel. Measurement is then started. Initially, viscosity is measured. The viscosity measurement panel is displayed during viscosity measurement. The actual viscosity value and sample temperature which is read from the liquid sensor are displayed on the viscosity measurement panel. After the measurement, the viscosity measurement panel is automatically closed, and then particle size distribution is measured.



Figure 31: Viscosity measurement panel Under measurement

Note:

- The temperature displayed on the measurement panel is read from the liquid sensor. It cannot be changed to the temperature from the holder sensor.

Note:

- If the viscosity value has exceeded 3 mPa·s when viscosity measurement is completed, a message “Continue measurement?” is displayed. When “Yes” is selected, particle size distribution is measured using the actual viscosity value (3.01 mPa·s to 10.0 mPa·s). However, it should be noted that particle size distribution is not accurately measured in such a high-viscosity sample.

Note:

- If the sample temperature is unstable, the viscosity value may not settle and the measurement may not be completed. The timeout for viscosity measurement is 180 seconds. If water viscosity is measured while the sample temperature is stable, it usually takes approximately 20 seconds to complete the measurement.

3. After the measurement data is captured, distribution is calculated. At this time, the actual viscosity value divided by the sample density value is used as the viscosity value parameter. The actual viscosity value and sample density value are displayed on the measurement result panel.

LB-550 Operation

Measurement of only sample viscosity

1. Click the [Measure] button on the main window. Then the “measurement panel” is displayed

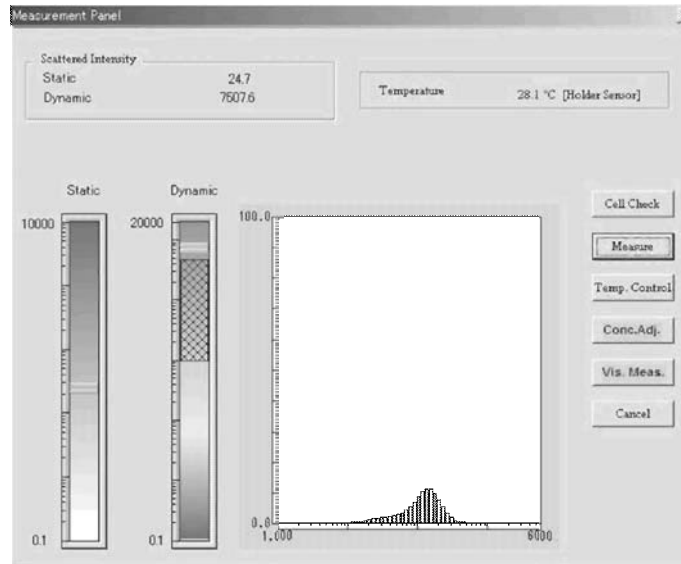


Figure 32: Measurement panel

2. Click the [Vis. Meas.] button on the measurement panel. Viscosity measurement is then executed. The viscosity measurement panel is displayed during the measurement.

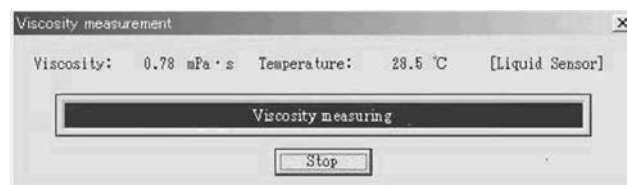


Figure 33: Viscosity measurement panel Under measurement

Note:

- The temperature displayed on the measurement panel is read from the liquid sensor. It cannot be changed to the temperature from the holder sensor.
3. When the measurement is completed, the message on the viscosity measurement panel is changed as shown below

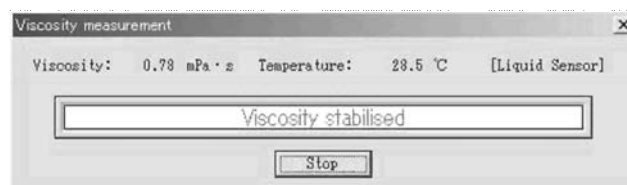


Figure 34: Viscosity measurement panel Viscosity stabilised

Note:

- 0.40 mPa · s to 10.0 mPa · s can be measured within the specification. If the actual viscosity value is out of the specification, a message “Viscosity value is out of specification.” is displayed.

Procedure after completion of viscosity measurement

1. After viscosity measurement is completed, lightly press the upper part of the probe downward, and then raise the probe to the upper arrow position manually.
2. Set the vial to "RINSE" of the vial holder, and pour acetone to the level about the top surface of the holder.

Adjust the liquid level of acetone to the same level as the top surface of the holder (RINSE).

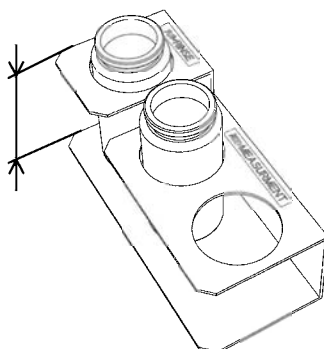


Figure 35: Holder for vial

3. Set the vial which acetone is poured to the cell holder of the LB-550.
4. Move the viscometer probe to the measurement position, and press the [Vis. Meas.] button. Dummy measurement for cleaning the detection terminal of the viscometer.
5. After lowest limit error is displayed and measurement is finished, move the probe to the arrow position and wipe it with cotton swab or soft cloth.

Note:

- When wiping the detection terminal, never twist or bend it strongly, which might cause malfunctions.

Note:

- When closing the upper door of the sample chamber, check that the viscosity probe is at the upper arrow position before closing the upper cover of the sample chamber.

Note:

- Never keep the sample adhering to the detection terminal for long time after measurement.

