Shodex RI-101
Refractive Index Detector
Service and Maintenance Manual

High Sensitive Refractive Index Detector
for High Performance Liquid Chromatography

Publication No. _____

SHOWA DENKO

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Important Notice

The information contained in this manual is subject to change without notice.
SHOWA DENKO K.K. (SHOWA DENKO) assumes no responsibility for any errors that may appear in this manual. This manual is believed to be complete and accurate at the time of publication. In no event shall SHOWA DENKO be liable for incidental or consequential damages in connection with or arising from the use of this manual.

Spare Parts Availability

It is the policy of SHOWA DENKO to provide maintenance spare parts for a period of seven (7) years after the final production of the instrument. Spare parts may be available after the seven (7) year period but only on an "as available" basis.

The following is a Federal Communication Commission advisory:

WARNING:

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

Operating Instructions

This manual is provided to help you establish operating conditions that will make safe and efficient.
Special considerations and precautions are also described in this manual that shall appear in the form of WARNING, CAUTION and NOTE as follows. It is important that you operate and service equipment in accordance with this manual and any additional information that may be provided by Showa Denko from time to time.

WARNING — ***Alerts you to potentially hazardous situations that could result in serious injury, and how to avoid these situation***

CAUTION — ***Alerts you to situations that may cause moderate injury and/or equipment damage, and how to avoid these situations.***

note — ***Information to help you to achieve optimal performance from your equipment.***

note — ***The following Trademarks an Registered Trademarks are found in this manual.***

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E.I. du Pont de Nemours and Company.

Before Starting:

Shodex RI-101 Refractive Index Detector is designed to be an analytical device for pure research purpose and may not be suitable for in vitro diagnostic analysis. To operate Shodex RI-101 Refractive Index Detector properly, you are strongly recommended to go through this Operator's Manual. Improper use of this detector shall be dangerous and may cause a hazardous result.

Limited Warranty Policy

SHOWA DENKO warrants its products against defects in materials and workmanship for the period of twelve (12) month, from the date of shipment of its factory.

SHOWA DENKO will, at its option, repair or replace products that are proved to be defective.

The aforementioned warranty policy shall not be applied to defects being caused by:

(1) Improper or Inadequate maintenance, adjustment, calibration or operation by the user(s);

(2) User-supplied software, hardware, interfacing or consumable;

(3) Unauthorized modification or misuse;

(4) Operation outside of the environmental and electrical specifications for the product;

(5) Improper site preparation and maintenance; or

(6) User induced contamination or leaks.

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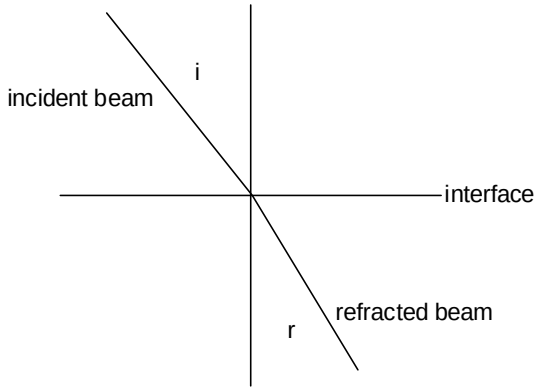
Section 1. Introduction

1-1. Principle of refractive index detection

Shodex RI-101 Refractive Index Detector (called as “Shodex RI-101” hereafter) is a high-performance universal detector designed for analyses requiring the continuous monitoring of the refractive index of a flowing liquid with respect to a reference.

Shodex RI-101 is a deflection or Snell type refractive index detector. Snell's law states that a parallel light beam, when passing through a dielectric interface separating two media of different refractive index at an angle of incidence greater than zero, will be refracted as a function of the magnitude of difference of the refractive indices of the two media.

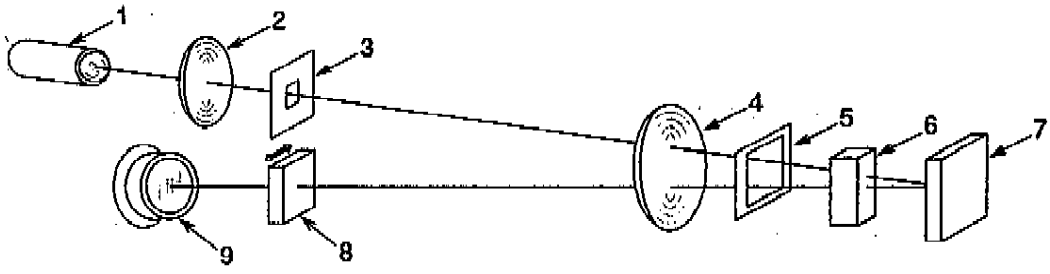
Snell's Law



$$n = \frac{\sin i}{\sin r} = \frac{v}{v^1}$$

n = index of refraction
i = angle of incidence
r = angle of refraction
v = velocity of light in first medium

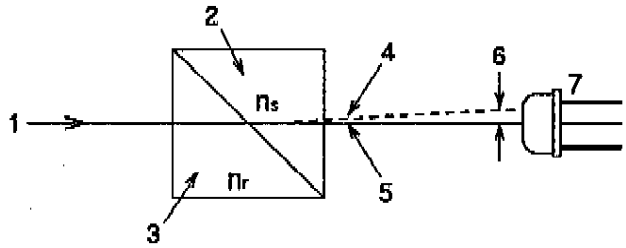
Optical System of Shodex RI-101



(1) tungsten lamp (2) condense lens (3) First Slit (4) Collimator Lens
(5) Second Slit (6) Flow Cell (7) Mirror (8) Null Glass (9)Photodiode

Light from a low-power, long-lifetime tungsten lamp is collimated by a lens and slit and passed through reference and sample cells, reflected off a mirror, passed back through the optical cells, and focused by lenses onto a pair of photo sensing diodes (photo sensor).

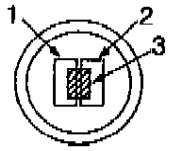
During operation, Shodex RI-101's reference and sample cells are filled with mobile phase. The reference cell is then isolated from the flow path and mobile phase flows through the sample cell only. As long as no difference exists between the refractive indices of the media of the two cells, there is no refraction of the light passing through them.



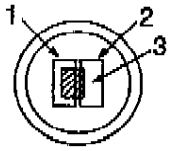
(1) Light Beam (2) Sample Cell (3) Reference Cell (4) Light Axis (Ns>Nr) (5) Light Axis (Ns=Nr) (6) Distance between (4)&(5) at the photo sensor (7)Photo sensor

Ns: Refractive Index of mobile phase in Sample Cell
Nr: Refractive Index of mobile phase in Reference Cell

The light shines on a pair of photodiodes, each of which gives an electrical signal; these signals are amplified and the difference between the two signals is measured. Zero refraction should generate a zero-volt difference in these signals. An electrically controlled mechanical linkage allows the user to optimize the photodiodes' outputs for zero deflection via a refractive lens in the optical path. Additional circuitry enables the user to easily correct the signal output to electronic zero.



Ns = Nr



Ns > Nr

(1) photo sensor A (2) photo sensor B (3) light beam

When a change occurs in the refractive index of the mobile phase, the light passing through the interface between the sample and reference cells is refracted, causing the light intensity on one photodiode to increase and on the other to decrease. This difference gives a signal having both amplitude and polarity; the signal is amplified to drive a chart recorder or integrator.

1-2. Specifications

● Construction	Deflection type
● Refractive Index Range	1.00 to 1.75
● Range	1/4 to 512 micro-RIU
● Linearity	600 micro-RIU
● Noise	=/<2.5 nRIU(Response : 1.5 seconds)
● Response Time	0.1, 0.25, 0.5, 1.0, 1.5, 2, 3, 6 sec.
● Polarity	Positive/Negative
● Auto Zero	Optical & Electrical Auto-Zero
● Auto Zero Range	All Refractive Index Range
● Auto Zero Resolution	=/<1 (@2mV/micro-RIU) / 4 (@8mV/micro-RIU) nRIU
● Offset Range	0 to 500mV (same with Integrator output sensitivity)
● Offset Resolution	10mV (same with Integrator output sensitivity)
● Integrator Output	0 to 1V/FS (Sensitivity: 2mV/micro-RIU, 8mV/micro-RIU)
● Recorder Output	0 to 10mV/FS
● Event Marker	Marker out: 10% of FS
● External Signal Input (Contact Closure)	Auto Zero, Marker, Polarity, Purge On/Off (Capacity: >/=DC24V 0.1A)
● Signal Output (Contact Closure)	(1) Ready (2) Solvent Leak (3) Error (One of following error occurred) Overheating Low Light Intensity Null Glass Home Position Error Lost Parameters Optical Balance
● Temperature Control Fuse	OFF , 30 to 50deg-C(1deg-C increment) : 77deg-C Temp.
● Operator Support	Automatic Start Up (Start Up Sequence) Span/Validation Guide Real Time Baseline Monitor
● External Communication	RS-232C
● Cell Volume	8micro-l
● Maximum Flow Rate	10 ml/min (mobile phase: pure water)
● Pressure Rating	50 kPa (0.5 kgf/cm^2)
● Internal Volume	Inlet Port / Flow Cell: approx. 60micro-l Flow Cell / Outlet Port: approx. 520micro-l Total: approx. 590micro-l
● Wetted Material	SST316, Teflon, Quarts Glass
● Power Requirement	AC100 to 240V +/- 10%: 50/60Hz
● Power Consumption	150VA maximum
● Dimensions	260mm(W) x 200mm(H) x 400mm(D)
● Weight	13 kgs (27 lbs)

Section 2. Unpacking and Installation

2-1. Installation Site Requirements

To install Shodex RI-101, please make sure that any of followings shall be kept away from it to prevent interference on your analysis. Refractive Index Detector, in general, is very sensitive to the change in ambient temperature and airflow that results a drift of baseline.

- Air fan (air-conditioner, cooler, heating equipment and ventilation)
- Open doors or windows
- Direct sunlight
- Dart or dusts
- Vibration source
- Electro-Magnetic Wave or High Frequency

2-2. Power Requirements

Shodex RI-101 requires 50 or 60 Hz single-phase power source capable of providing AC 100/240 +/- 10%.

CAUTION

Running Shodex RI-101 on a voltage other than the correct single-phase supply will void the warranty.

CAUTION

Shodex RI-101 is designed to operate with single-phase (phase-neutral) power ONLY.
If your facility provides only phase-phase (i.e., three-phase) power, consult our local representative in your area or SHOWA DENKO.

2-3. Unpacking and Inspection

This section describes in details how to install Shodex RI-101. This detector is designed to be operator installed.

CAUTION

Each steps of the installation site preparation must meet local safety, electrical, and building codes. These codes take precedence over any recommendations in these instructions, and compliance to them is the responsibility of the customer.

CAUTION

Shodex RI-101 weighs 13 kgs (27 lbs). Use proper lifting techniques to avoid potential injuries.

To remove the detector from the box, hold the bottom of the cabinet. Never attempt to lift by front panel or terminals.

CAUTION

Shodex RI-101 is packed with a number of accessories. Do not discard the packing material until all parts are accounted for.

Check the detector carefully for evidence of shipping damage.

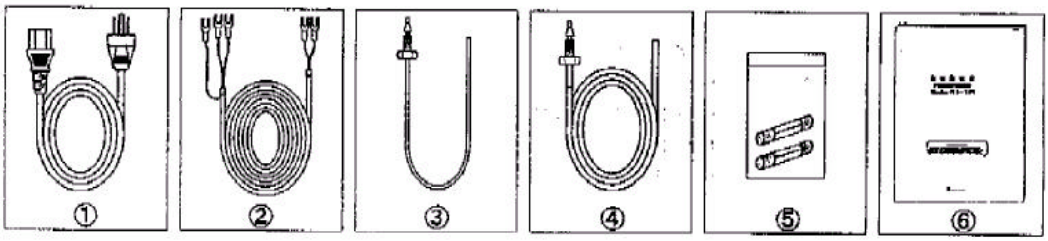
If there is any evidence of damage, any item(s) missing in the carton or discrepancies, please notify that to the carrier immediately and to our local representative in your area or SHOWA DENKO at following address.

SHOWA DENKO K.K.
Specialty Chemical Division / Shodex Group

5-1, Ohgi-mati, Kawasaki-ku, Kawasaki-shi,
Kanagawa 210-0867 Japan
Phone Japan(81) 44-329-0733
Facsimile Japan(81) 44-329-0794

2-4. Standard Accessories

1. P/N 4300120	Power Cord	1ea
2. P/N 4303190	Output Signal Cable	1ea
3. P/N 8809160	Inlet Tube (SST: 1.6 x 0.25 x 1000mm with nut & ferrule)	1ea
4. P/N 8809170	Outlet Tube (PTFE: 2.5 x 1.5 x 1500mm with nut & ferrule)	1ea
5. P/N 2401190	Fuse 3.15A(T3.15AL/250V)	2ea
6. P/N 9408990	Operator's Manual	1ea



CAUTION

The power cord for the US and other AC 120V, 60 Hz applications is terminated in a 3-prong parallel blade plug.

Outside of the 120V, 60 Hz areas, the detector shall be accompanied with a power cord (_____). Be sure using appropriate power cord for your power source.

2-5. Loosing the locking screws

To prevent damage may occur during the shipment, the optical block is fixed by two (2) locking screws (5mm Allen bolts). As you are installing Shodex RI-101, please make sure to loosen these screws.

1. Move Shodex RI-101 to the side of a bench to make the locking screws accessible while supporting it to prevent it from falling.

2. Loosen the locking screws, but do not remove them, using the 5mm hexagonal key

3. Move the detector securely back onto the bench.

Once the locking screws were loosened, the rubber insulator equipped with the optical block becomes functional and absorb an external shock or vibration.

CAUTION

Do not remove the screws. Do not run them out so far that the detector's weight rests on the screw heads instead of its feet.

note

Detector will not stabilize if locking screws are not loosened.

2-6. Making a connection

2-6-1. Power Line

The power switch is located on the lower left of front panel as viewed in 4-1. The power should be OFF before connecting the power cord to the detector. The receptacle for the power cable is located on the rear of the detector. Refer to 4-2.

Connect the end of grounding cable to the ground terminals on the back panel and to a known ground.

WARNING:
SHOCK HAZARD

Electrically conducting spills can occur when conductive HPLC solvents are spilled on or in the instrument.

Ground the instrument properly to protect the operator from electrical shock. Proper grounding also protects the instrument from power line noise.

Verify that the instrument is properly grounded through the power line ground terminal. Do not remove or otherwise disable the power cord's ground prong.

2-6-2. Replacing Fuse

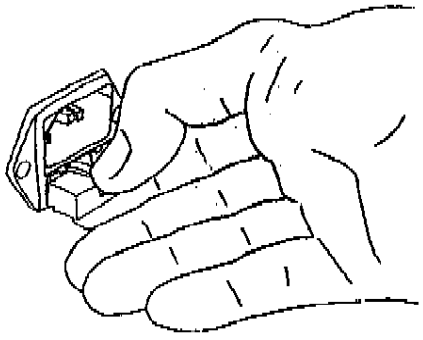
WARNING:
FIRE HAZARD

Replace only with same type and rating of fuse.

Fuse for Shodex RI-101: P/N 2401190

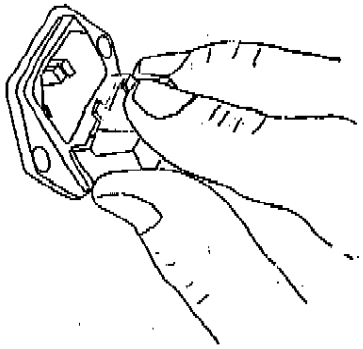
3.15A(T3.15AL/250V)

To remove fuse holder



Turn off the power of detector.
Remove the power cord.
Push the lever located just above of Fuse holder cover. Pull the holder out as you hear clicking sound.

To re-install fuse holder



Replacing fuse.
Push the fuse holder in until clicking Sound is heard.
Make sure the holder is locked.

2-6-3. Tubing Connections

- **Connection to the Inlet**

CAUTION

Shodex RI-101 must be the last component in your LC system. No detector or backpressure regulator can follow it. Do not use narrow-bore tubing for the outlet

Hook up the narrow-bore stainless steel tubing of the Standard Accessory (0.25 mm ID, 1M long) with the inlet(IN) port of front panel.

- **Connection on the Outlet Side**

Hook up Teflon® tubing (1.5 mm ID, 1.5 m long) of the Standard Accessory with the outlet (OUT) port of front panel. Lead the opposite end of tubing into drain bottle.

CAUTION

Do not expose the purge valve against backpressures greater than 50 kPa (7 psi). Also, never subject the reference and sample cells to back pressures greater than 700 kPa (100 psi). High backpressures can break the cells, which are difficult to replace.

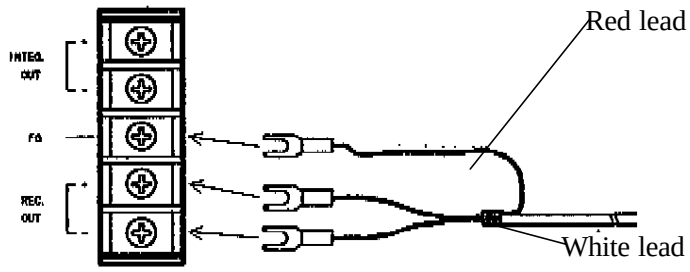
2-6-4. Cable Connections

CAUTION

The GROUND terminals, on the lower right side of the rear panel (see), is not the same as the recorder ground or Integrator ground terminals. Do not interchange these connections.

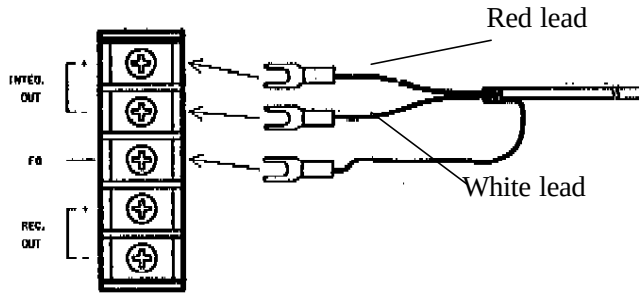
- **Recorder Connection**

Using the output signal cable supplied in the Standard Accessory, connect one end to the chart recorder, white lead to recorder negative (–) terminal and red lead to recorder positive (+). Connect the opposite end to the recorder's 10 mV terminal. Connect the third lead to the GROUND (FG).



- **Integrator Connection**

Using the output signal cable supplied in the Standard Accessory, connect one end to the integrator, white lead to integrator negative (–) and red lead to integrator positive (+).
Connect the third lead to the GROUND (FG).

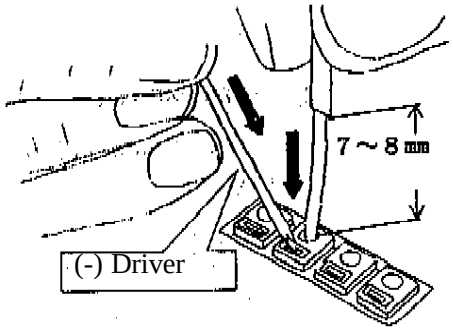


- **Other Cable Connection**

For the following cable connection, please use duplex lead wire.

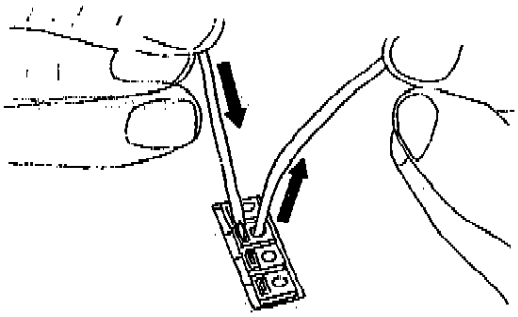
External Signal Input: Auto Zero, Marker, and Purge On/Off
Signal out: Ready, Solvent Leak, and Error

To connect the wire:



Scrape the sleeve off for about 7 to 8 mm. Inserts the end of lead wire while pressing the button with (-) driver. Then, remove the (-) driver to lock the wire. Make sure the wire is firmly grabbed.

To release the wire:



Press the button with (-) driver to release the lock while pulling the wire out.

CAUTION

Those terminals (Auto Zero-In, Marker-In, Purge On/Off-In, Ready-Out, Solvent Leak-Out, and Error-Out) are for contact closure. Never apply voltage to those.

2-7. Solvent Recommendations

General Recommendations

Grade of Solvents

HPLC grade solvents are recommended for better instrument performance and chromatographic data.

Solvent Filtering

All solvents, including deionized water, should be filtered by in-line solvent filter.

Solvent Degassing

Solvents should be properly degassed prior to use. Solvents that are not properly degassed may cause bubble formation in pump heads and in the detector cells potentially causing pressure flow problems and a noisy chromatographic baseline.

Degassing is especially critical when the column temperature is elevated above room temperature, or with solvents that have a high gas solubility, e.g., methanol. Analysis times that run greater than 5 to 6 hours will also require degassing of solvents.

On-line (continuous) solvent degassing is most ideal to have a constant state of mobile phase solvent over time, as the degassed solvent would start reintroducing the air.

Some solvents may corrode the wetted surfaces of the detector, if they are left in the detector after operation. The quartz cell window is easily etched by strong bases. It is recommended that some solvents be rinsed from the detector for overnight and weekend storage.

The solvents used with the detector are limited by the materials used for the wetted parts they come in contact with (e.g., quartz glass, Teflon® and 316 stainless steel). These limitations should be considered as you are choosing mobile phase solvents.

When an organic solvent that contains halogens, such as chloroform and methylene chloride, is used, flush out entire flow path with a solvent compatible with your chromatographic condition. (for example, hexane or another hydrocarbon)

For isocratic work with a normal phase column, the alcohols methanol or 2-propanol, a non-carcinogenic aromatic such as the xylenes, acetone, or an ether that is non-volatile and not a ready producer of peroxides can be used. DO NOT use ethyl ether.

If a solvent listed below is in use, flush out all the flow path sufficiently with an inert solvent that is compatible with your chromatographic system. Buffers, acids, and other highly ionic aqueous solutions should be flushed out with large amounts of water (5-10 times the volume of liquid from pump head to detector outlet If you neglect this flushing, the pump, injector, and column may become corroded and badly damaged.

Sulfuric Acid, Boric Acid, Citric Acid, Acetic Acid, Lactic Acid, Acetic Anhydride, KOH, NaOH, Hydrazine, Sodium formate
Ammonium salts: -formate, -perchlorate, - nitrate, -citrate, -oxalate, -sulfate, -H_2PO_4, K_2CO_3
K, Na salts: -bicarbonate, -chlorate, -nitrite

Following solvents should be avoided.

Hydrohalogenic, Metal Halides >2M, KCl, Ammonium Halides, Ammonium Formate, All hypochlorites, Tetrachloromethane
Acids: HCl, HF, etc.

CAUTION

Fluorocarbon solvents will alter Teflon® over long exposure. Flush with pentane or another light hydrocarbon.

note

If you want to replace one solvent with an immiscible one, flush out the existing mobile phase with an intermediary solvent inter-miscible with your initial and final solvents. For example, you want to replace water in your HPLC with chloroform; water and chloroform are immiscible. Replace the water in your system with 2-propanol, which is freely miscible with water and chloroform. When you are certain all water is removed, replace the 2-propanol with chloroform. See the Miscibility Chart in Appendix.

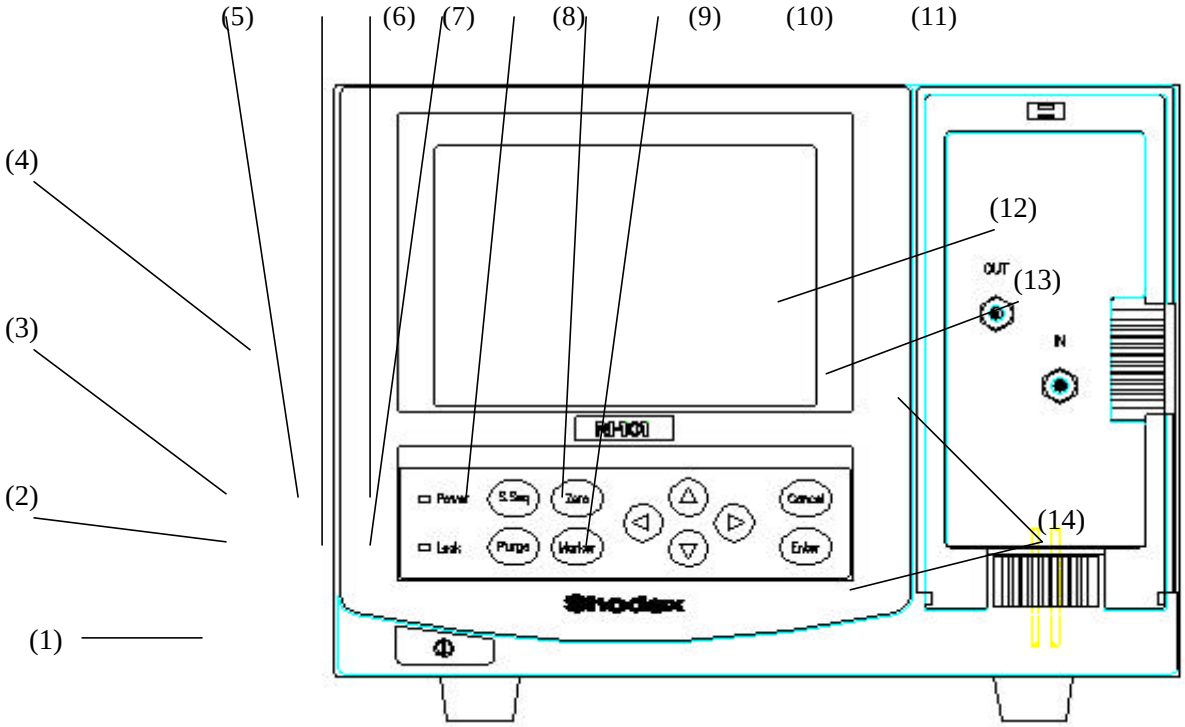
Characteristics of commonly used mobile phase

(Those in bold should not be employed)

	Polarity $E^2(Al_2O_3)$	Viscosity $_CP20deg-C$	Refractive Index	UV Cut-off (nm)	Flash Point (deg-C)	Fire Point			Vapor Density	Boiling Point (deg-C)	Gravity
						(deg-C)	Upper	Lower			
Fluoroalkanes	-0.25		1.25								
n-Pentane	0.00	0.23	1.358	210	<-40	308.9	1.5	7.8	2.5	**36.1**	0.6
Hexane	0.00		1.375	210	-21.7	233.9	1.2	7.5	3.0	68.9	0.7
Isooctane	0.01		1.404	210							
Petroleum ether	0.01	0.3		210							
n-Decan	0.04	0.92	1.412		46.1	207.8	0.8	5.4	4.9	173.9	0.7
Cyclohexane	0.04	1.00	1.427	210	-20	260	1.3	8	2.9	81.7	0.8
Cyclopentane	0.05	0.47	1.406	210							
Diisobutylene	0.06		1.411	210							
i-Pentene	0.08		1.371		-17.8	272.8	1.5	8.7	2.4	**30**	0.7
Carbon disulfide	0.15	0.37	1.626	380	-30	100	1.3	44	2.6	**46.1**	1.3
Carbon tetrachloride	0.18	0.97	1.466	265							
Amyl chloride	0.26	0.43	1.413	225	12.8	343.3	1.6	8.6	3.7	106.1	0.9
Busy chloride	0.26		1.436	220	-9.4	460	1.8	10.1	3.2		
					o-17.2	463.9	1.6	6.0		144.4	
Xylene	0.26	0.62-0.8 to 1.50		290	m-25	527.8	1.1	7.0	3.78	138.9	0.9
					p-25	528.9	1.1	7.0		138.3	
i-Propyl ether	0.28	0.37	1.368	220	-27.8	443.3	1.4	21	3.5	68.9	0.7
i-Propyl chloride	0.29	0.33	1.378	225	-32.2	593.3	2.8	1037	2.7	**35**	0.9
Tolune	0.29	0.59	1.496	285	4.4	536.1	1.4	6.7	3.1	110.6	0.9
n-Propyl-chloride	0.30	0.35	1.389	225	<-17.8		2.6	11.1	2.7	**46.1**	0.9
Chlorobenzene	0.30	0.80	1.525		32.2	637.8	1.3	7.1	3.9	132.2	1.1
Benzene	0.32	0.65	1.501	280	-11.1	562.2	1.4	7.1	2.8	80	0.9
Ethyl bromide	0.37		1.424			511.1	6.7	11.3	3.8	**37.8**	1.4
Ethyl ether	0.38	0.23	1.353	220	-45	180	1.9	48	2.6	**35**	0.7
Ethyl sulfide	0.38	0.45	1.442	290							
Chloroform	0.40	0.57	1.443	245							
Methylene -chlolride	0.42	0.44	1.424	245	-50	518.9	3.8	15.4	2.2	**38.5**	0.9
Methyl I-butyl ketone	0.43		1.394	330							
Tetrahydrofurane	0.45		1.408	220	-14.4	321.1	2	11.8	2.5	66.1	0.9
Ethylen dichloride	0.49	0.79	1.445	230	13.3	412.3	6.2	16	3.4	83.9	1.3
Methyl ethyl ketone	0.51		1.381	330	-6.1	515.6	1.8	10	2.5	80	0.8
i-Nitropropane	0.53		1.400	380	48.9	420.6	2.6		3.14	131.1	1.0
Acetone	0.56	0.32	1.359	220	-17.8	537.8	2.6	12.8	2.0	56.7	0.8
Dioxane	0.56	1.54	1.422	260	12.2	180	2.0	22	3.0	101.1	1.0
Ethyl acetate	0.58	0.45	1.370	260	4.4	460	1.8	8	3.5	90	0.9
Methyl acetate	0.60	0.37	1.362	210	-10	501.7	3.1	16	2.6	60	0.9
Amyl alcohol	0.61	4.1	1.410		32.8	300	1.2	10.0	3.0	137.8	0.8
Dimethyl sulfoxide	0.62	2.24									
Aniline	0.62	4.4	1.586		70	617.2	1.3		3.2	184.4	1.0
Dimethyl amine	0.63	0.38	1.387	275	<-17.8	312.2	1.8	10.1	2.5	56.7	0.7
Nitromethane	0.64	0.67	1.394	380	35	418.3	7.3		2.1	101.1	1.1
Acetonitrile	0.65	0.37	1.344	210	5.6				1.4	81.7	0.8
Pyridine	0.71	0.94	1.510	305	20		1.8	12.4	2.7	115	1.0
Butyl cellosolve	0.74			220							
i-Propanol n-Propanol	0.82	2.3	1.38	210	11.7	398.9	2.0	12	2.1	82.8	0.8
Ethanol	0.88	1.20	1.361	210	12.8	422.8	4.3	19	1.6	78.3	0.8
Methanol	0.95	0.60	1.329	210	11.1	463.9	7.3	36	1.1	63.9	0.8
Ethylene glycol	1.11	19.9	1.427	210	111.1	412.8	3.2			197.2	1.1
Acetic acid	large	1.26	1.372								

Section 3. Functional Keys and Displays

3-1. Front View

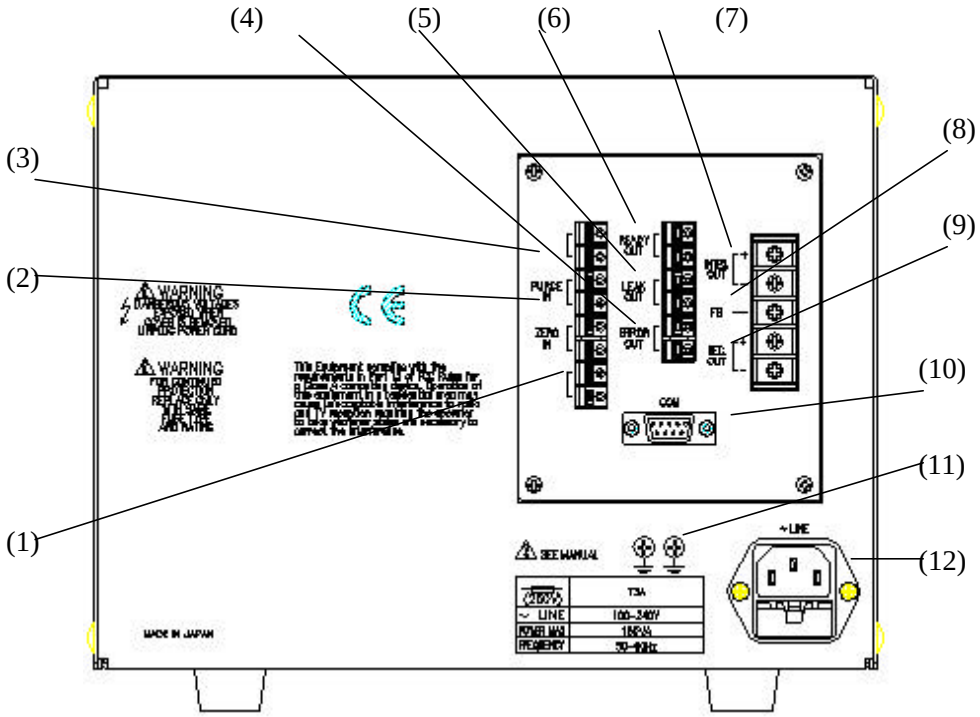


(1)Power Switch
(2)Solvent Leak Indicator
(3)Power Indicator (LED)
(4)Liquid Crystal Display (LCD Screen)
(5)Start Up Sequence Key
(6)Purge Key
(7)Auto Zero Key
(8)Marker Key
(9)Arrow Keys
(10)Cancel Key
(11)Enter Key
(12)Outlet Port
(13)Inlet Port
(14)Tube Holders

3-2. Functional Keys and Displays

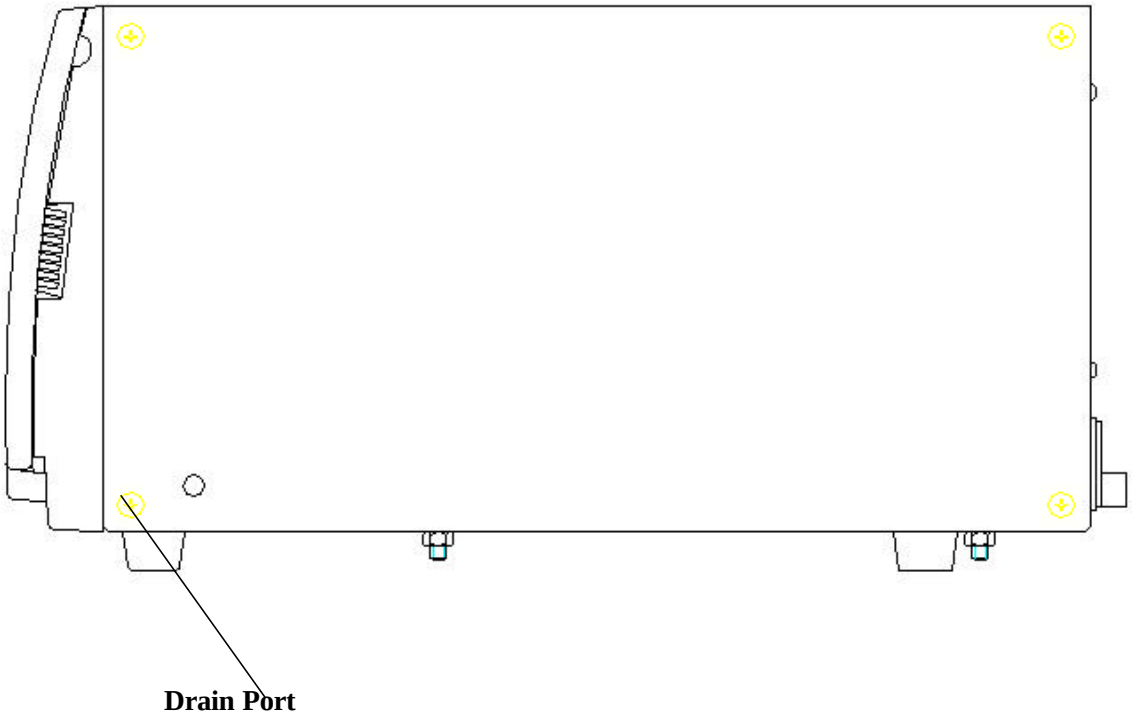
(1)Power Switch	Press this key once to turn on or off the unit.
(2)Leak LED	Illuminated when solvent leak is detected.
(3)Power LED	Illuminated when power is on.
(4)LCD Display	Liquid Crystal Display
(5)Start Up Sequence Key	Press S.Seq key once to call Start-Up Sequence. If S.Seq key is pressed, Dialog box request to confirm whether you really want to do the sequence. Press ENTER key to start the sequence. S.SEQ sign shall come up on the status bar. Press Cancel key if you don't want the sequence. Pressing S.Seq or Cancel key during the sequence shall suspend the sequence.(See "Start-Up Sequence - Exit")
(6)Purge Key	Press Purge key to turn purge valve on or off to change flow path. When the valve is on, PURGE sign is highlighted by yellow background and solvent flows through reference side of flow cell instead of sample side. Press Purge to turn off the valve. Purge key isn't functional during the Start-Up Sequence.
(7)Auto Zero Key	Press Zero key to command "Auto-Zero". Zero key doesn't function during the sequence.
(8)Marker Key	Press Marker key to generate an event marker signal. (10% of Full Scale)
(9)Arrow Keys [up] [down]	Press Arrow keys to move cursor or to edit values. Unless remarked specifically, cursor shall scroll circularly.
(10)Cancel Key	Press Cancel key to scrap the edited value(cursor returns to the tab.) or to cancel the command.
(11)Enter Key	Press Enter key to save the edited value(cursor returns to the tab) or to confirm the command.
(12)Outlet Port	Solvent passing through the flow path is expelled
(13)Inlet Port	Tubing from separation column outlet is connected.
(14)Tube Holder	Inlet Tube and Outlet Tube holders

3-3. Rear View



(1)External Signal In (Marker)
(2)External Signal In (Auto Zero)
(3)External Signal In (Purge On)
(4)Signal Out (Error)
(5)Signal Out (Solvent Leak)
(6)Signal Out (Ready)
(7)Integrator Our
(8)Ground for signal cable
(9)Recorder Out
(10)RS-232C Port
(11)Ground Terminal
(12)Power Inlet

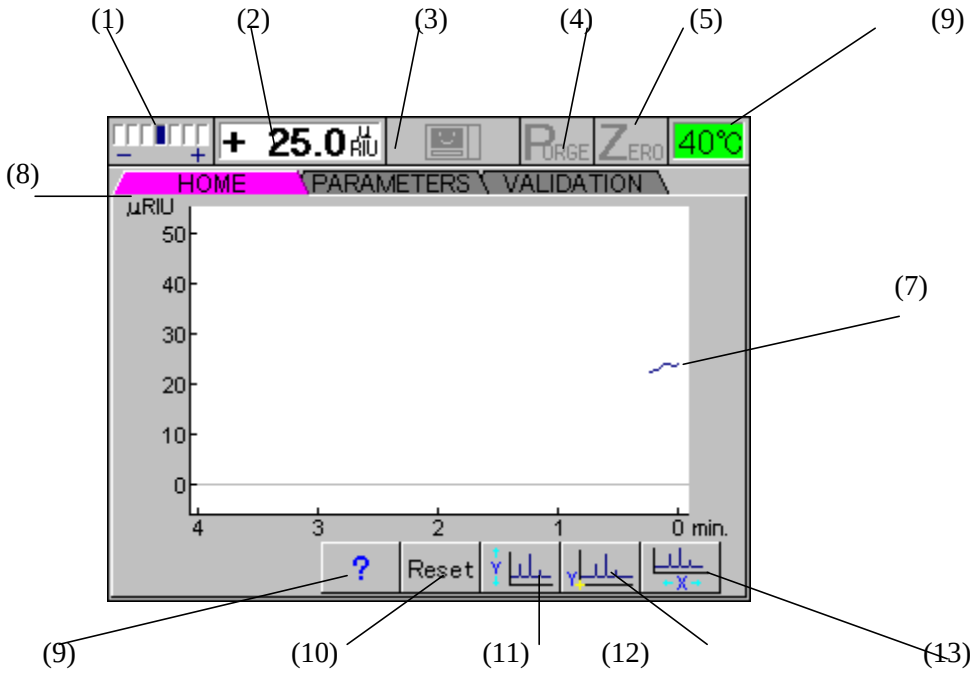
3-4. Side View



Drain Port

In case of internal solvent leak, solvent will be expelled from this port. Connect Teflon® tubing (6mm OD, 4mm ID). Lead the tubing to a safe waste disposal container always.

3-5. HOME Screen (Indicators and On-Screen Buttons)



(1)Optical Balance
This indicates current state of optical axis balance by the position of blue color box. Normally, the position of blue box should be in the center. If it isn't, press Zero key to do "Auto-Zero". If it doesn't return to the center still, please contact our local representative in your area.

(2)Refractive Index
This shows refractive indexes real time basis. When **OVER** sign comes on, refractive indexes are out of following RI range. This could be happened when sample and reference cells do not contain identical solutions or sample or reference cell contains air bubbles. Flush sample and reference cells with mobile phase. Press Zero Key to do "Auto-Zero". If **OVER** sign doesn't go off still, please contact our local representatives in your area

Analytical/Semi-micro
(Integrator range)
-600 to +600micro-RIU(512micro-RIU/V)
-150 to +150micro-RIU(125micro-RIU/V)
Preparative
(Integrator range)
-6,000 to +6,000micro-RIU(5,120RIU/V)
-1,500 to +1,500micro-RIU(1,250RIU/V)

(3)Status Bar
Showing general conditions of Detector S.SEQ sign comes on when the sequence starts.READY sign comes on when the sequence is completed satisfactory.

(4)Purge
PURGE sign will be highlighted when purge valve is on.

(5)Zero **ZERO** sign will be highlighted during Auto-Zero.

(6)Temperature
This shows the temperature reading real time basis. When the difference becomes greater than + or – 1 degree, the background color changes from green to yellow.

(7)Baseline Showing baseline real time basis

(8)Tab Showing available menus to choose.
Following is a list of tab that you can choose.

Choose desired tab by [-->] or [<--] key. Then, press [up arrow] or [down arrow] key to go into the screen.

- HOME
- PARAMETERS (Parameter Setting)
  - OPERATING: Operating Condition
  - S. SEQUENCE: Start Up Sequence
- VALIDATION

(9)Help Move cursor by [-->] or [<--] key and press Enter key to go to Help screen.

Each screen has [?] button. Choose [?], and press Enter key to jump to the help topics corresponding to current screen. Keep pressing [up arrow] or [down arrow] key to view other topics.

(10)Reset When rudders and origin of chart is changed, the default setting for the chart is cancelled. Move cursor to Reset by [-->] or [<--] key and press Enter key to resume the default setting.

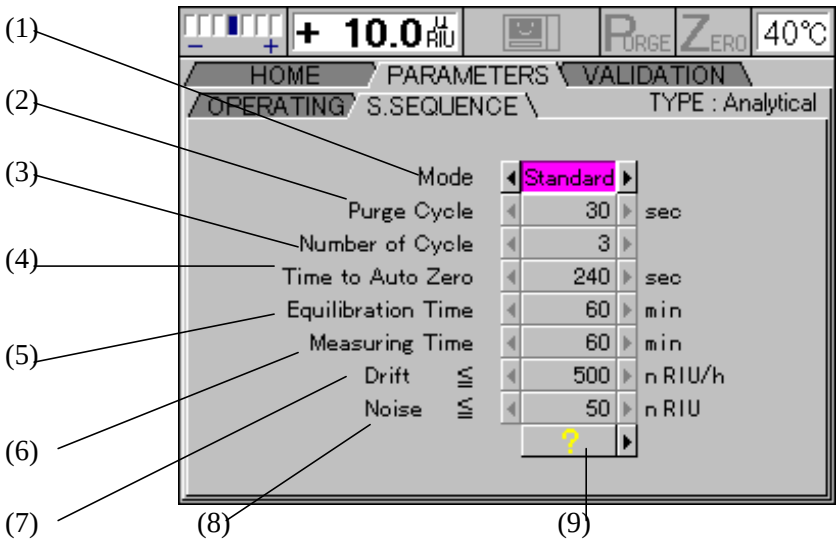
(11)Y axis Choose "Y axis" icon by [-->] or [<--] key. Then, press [up arrow] or [down arrow] key to set the maximum span.

(12)Y axis Choose "Y axis Origin" icon by [-->] or [<--] key. Then, press [up arrow] or [down arrow] to set the origin.
Minimum and maximum value should be within Refractive Index range.

(13)X axis Choose "X axis" icon by [-->] or [<--] key. Then, press [up arrow] or [down arrow] key to set the maximum span between 4 and 60 minutes."X axis" data for the last 60 minutes from present is always stored for your review.

To exit from HOME screen, press [up arrow] , [down arrow] or Cancel key to move cursor back to the tab.

3-6. PARAMETERS Screen (Start Up Sequence)



(1)Mode Choose one of built-in sequences (Fine, Standard, Coarse) from the menu or make your own (Custom). Move cursor by [up arrow] or [down arrow] key to "Mode". Press [-->] or [<--] key to choose the mode.

(2)Purge Cycle This will repeat on/off of purge solenoid valve to remove air bubbles trapped in the flow cell. The term of Purge Cycle is used for the length of one cycle. The default setting is 30 seconds. With this setting, the valve will be on for 15 seconds and off for another 15 seconds. Choose preferred setting (Custom) if necessary.

(3)Number of Cycle This will define number of purge cycle.

(4)Time to Auto Zero At the end of purge cycles, Auto-Zero should be made. This defines the length of pausing period between the end of purge cycle and Auto-Zero. The default setting is 240 seconds. Choose preferred setting (Custom) when necessary.

(5)Equilibration Time This is a period on which Detector is equilibrated for the measurement of drift and noise performance. Choose one from the menu (80, 60 and 40 minutes) or enter Custom value between 15 and 300 minutes.

(6)Measuring Time This is a period on which drift and noise performance is measured after Equilibration. Choose one from the menu (80, 60 and 40 minutes) or enter Custom value between 15 and 300 minutes.

(7)Drift Set the target drift value here.
(8)Noise Set the target noise value here.

(9)? Help

To exit from this screen, press Enter key to save those changes that you have made. Press Cancel key to cancel those changes. Both case, the cursor will move back to the tab.

🕮note

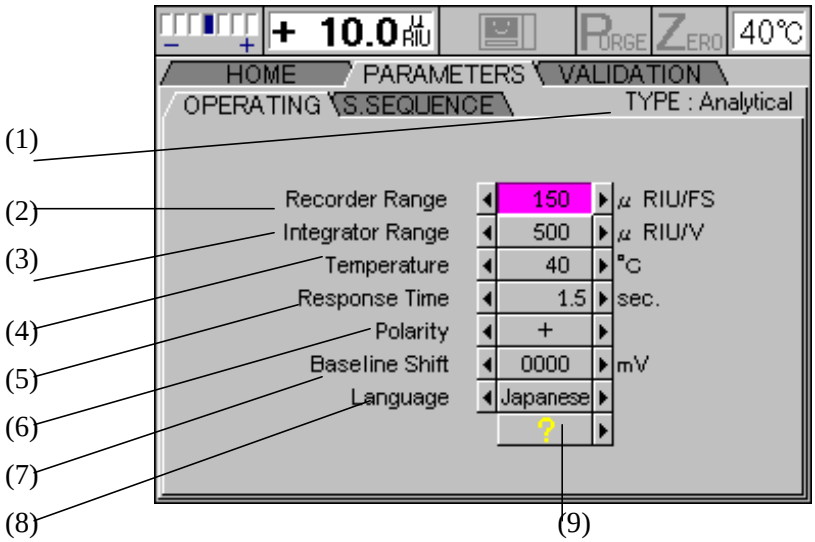
Following table shows you these built-in sequences and the option range that you may choose as your custom sequence.

	Fine	Standard (Default)	Coarse	Custom
Purge Cycle	30 sec.			10 to 990 sec.
Number of Cycle	3			3 to 9
Time to Auto Zero	240 sec.			60 to 990 sec.
Equilibration Time	80 min.	60 min.	40 min.	12,15 to 300 min.
Measuring Time	80 min.	60 min.	40 min.	12,15 to 300 min.
Drift	100 nRIU/h	500nRIU/h	2500nRIU/h	50 to 9990nRIU/h
Noise	50 nRIU			3 to 998nRIU

🕮note **Drift & Noise Advisory**

If, after the selected measuring time, elapses and the drift and noise specifications still have not been met, you will get a warning "Detector did not reach a setting condition. We suggest to repeat Start Up Sequence procedure once more". You can select Ignore or Repeat. (Refer to 4-2. Start Up)

3-7. PARAMETERS Screen (Operating Parameters)



(1)TYPE "Analytical" analytical type: Flow Rate: Do not Exceed 10ml/min!
"Semi-micro" semi-micro type: Flow Rate: Do not Exceed 1ml/min!
"Preparative" preparative type:Flow Rate:Do not Exceed 150ml/min!

(2)Recorder Range Recorder range setting. Choose the field by [up arrow] or [down arrow] key and edit by [-->] or [<--] key.

(3)Integrator Range Integrator range setting. Choose the field by [up arrow] or [down arrow] Key and edit by [-->] or [<--] Key.

(4)Temperature Temperature setting. Choose the field by [up arrow] or [down arrow] Key and edit by [-->] or [<--] Key

(5)Response Time Response time setting. Choose the field by [up arrow] or [down arrow] Key and edit by [-->] or [<--] Key.

(6)Polarity Polarity selection. Choose the field by [up arrow] or [down arrow] Key and edit by [-->] or [<--] Key.

(7)Baseline Shift Baseline shifting. Choose the field by [up arrow] or [down arrow] Key and edit by [-->] or [<--] Key

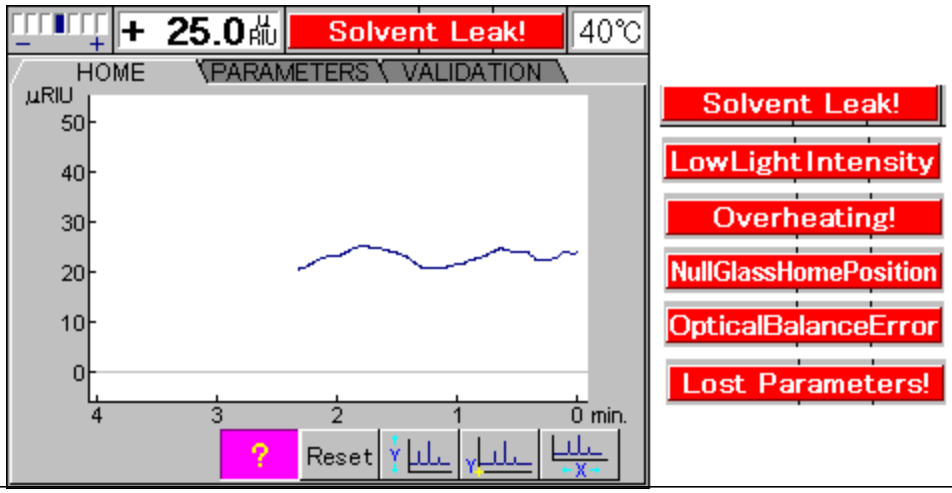
(8)Language Language Selection.(English or Japanese))Choose the field by [up arrow] or [down arrow] Key and edit by [-->] or [<--] Key

(9)? Help

To exit from this screen, press Enter Key to save those changes that you have made. Press Cancel Key to cancel those changes. Both case, the cursor will move back to the tab.

3-8. Error Messages

To alert you about the situation that is hazardous or may cause a deterioration of your analysis, Shodex RI-101 flashes following six different error messages.



Beep

When any invalid command or editing is made, Detector beeps once for 0.5 second. When following errors occur, Detector beeps until any key is pressed to acknowledge those errors.

(1)**Solvent Leak!** This indicates solvent leak within the flow path.

CAUTION

When this error message comes on, power off the detector at once.

Please double-check if there is any solvent leak. In case you cannot fix problems, please contact our local representatives in your area.

(2)**Low Light Intensity** This indicates inadequate light intensity. There are several possible cause for this error as follow.

1. **Different state of solvents between sample flow cell and reference flow cell. Purge reference side flow path with fresh solvent.**
2. **Air bubble in flow cells. Repeat purge on and off with flow to remove the air bubble.**
3. **Optical axis is off from the center. Press Zero key to do "Auto-Zero".**

(3)**Overheating!** This indicates overheating of optical block.

Please contact our local representatives in your area.

(4)**Null Glass Home Position** This indicates the null glass doesn't come back to its home position.

If you are commanding Auto-Zero via external control devise, refer to the procedure for "Optical Balance Error.

If you have this error as you are turning on Shodex RI-101, please contact our local representatives in your area.

(5)**Optical Balance Error** This indicates that Auto-Zero isn't successful to get optical zero point.

It may be caused by insufficient solvent exchange of reference flow path. Try purging procedure to fill reference flow path with fresh solvent at once.

If you cannot solve the problem still, please contact our local representatives in your area.

(6)**Lost Parameters** This indicates that the parameters are lost.

Please contact our local representatives in your area.

Section 4. Getting Started

4-1. *Just in Case !*

Prior to operation, check the following one more time.

.

- Locking screws are loosened
- Mobile phase solvent is freshly made and degassed well
- All wetted parts are chemically compatible to the mobile phase solvent
- Mobile phase flushed through the entire flow path; all incompatible or immiscible solvents have been flushed out
- Cable connections are properly made to chart recorder, integrator, data system, or other external equipment.
- All tubing connections are properly made and checked for leaks.
- Power cord is plugged into appropriate power receptacle.
- Proper fuse is installed.
- Drain tube is installed.
- Power switch is ON.

⚠ CAUTION

Before activating the purge valve (LED light OFF), pump about 10 mL of liquid through the cell.

This will flush possible dust or particulate matter and reduce the possibility of damaging the valve seals.

4-2. Start Up

4-2-1. Unattended Start Up

1. Set the parameter on operating parameter-setting screen (Refer to 3-8).
2. Set the parameter on start-up sequence parameter-setting screen (Refer to 3-7).

📖note

Start pumping mobile phase solvent at flow rate 1 ml/min. If you can pump only at lower flow rate, you may set longer Purge Cycle.

3. Press S.Seq key once to call Start-Up Sequence. If S.Seq key is pressed, Dialog box request to confirm whether you really want to do the sequence. Press ENTER key to start the sequence. S.SEQ sign shall come up on the status bar.

Elapsed time from the sequence start and total sequence time are displayed on the screen.

🕮note

If you want to stop the sequence for whatever reasons, press S.Seq key to suspend Start-Up sequence. In the event, Dialog box requests to confirm whether you really want to abort it or not. Press Enter key to abort the sequence. Press Cancel key to resume the sequence.

4. Before measurement results become available, "Equilibrate" and "Measuring" is displayed at Equilibration Time and at Measuring Time column respectively. As they become available, drift and noise value shall be displayed real time basis together with their target values.

5. If drift or noise didn't meet target values within defined period (Equilibration Time), Detector will hold the valve at on position.
6. Dialog box requests your command. Choose Repeat or Ignore button by [-->] or [<--] key. Confirm your preferred option by ENTER Key. Choose Repeat to redo this equilibration normally. Otherwise, you may disregard results and let Detector goes onto Measuring by Ignore button.
7. If drift or noise didn't meet the target value within the defined period (Measuring Time), Dialog box requests your command. Choose either Repeat or Ignore button by [-->] or [<--] Key and confirm your preferred option by ENTER Key.
8. Choose Repeat to redo the measuring normally. Otherwise, choose Ignore to abort the sequence. Though the READY sign doesn't come up, Detector is functional for analysis.
9. As soon as drift and noise met the target values during Measuring Time, the sequence is completed and READY sign comes up on the status bar.

Shodex RI-101 is ready for the analysis.

4-2-2. Manual Start

1. Set the parameter on operating parameter-setting screen (Refer to 4-7).
2. Start pumping mobile phase solvent at flow rate 1 ml/min to reference cell (Purge On).
3. Press Purge key in every 10 seconds to on/off the purge valve for few minutes.
4. Keep pumping mobile phase solvent to reference cell for about 20 minutes from the above step(2).
5. Press Purge key to turn off the valve. Mobile phase solvent flows to sample cell.
6. Wait until the baseline is stabilized.
7. Press Zero key to do Auto Zero.

WARNING:
CHEMICAL HAZARD

Wearing protective gloves and goggles is advised.

Section 5. Maintenance

5-1. Cell Cleaning

In many cases, performance degradation in sensitive instruments equipped with flow-through cells is caused by cell contamination. The use of filtered solvents with in-line solvent filters will protect the cell from contamination and reduce the amount of cleaning required. However, contamination from trapped particulates or bubbles, from precipitates, or from thin films of residues can still occur.

5-1-1. Preparations

To introduce cleaning solution into Shodex RI-101 by solvent delivery pump, connect a tubing line directly from the pump to Shodex RI-101 inlet port bypassing the column. Some cleaning solutions should be injected, however, directly into the flow cells by syringe due to their high corrosiveness or safety concern

CAUTION

Remember that flow cells can stand with only up to 70 kPa (100 psi). So, gently flush the cells under all conditions. If you encounter a high backpressure in Shodex RI-101, use extreme caution to proceed. You will be risking flow cell rupture, and flow cell assembly replacement is not a recommended customer procedure.

note

Clean all internal lines of Shodex RI-101 by injecting cleaning solution with PURGE OFF, and inject cleaning solution again with PURGE ON.

Particulate matter can be removed by forcing liquid through the cell using the syringe. Sometimes it helps to reverse flow and inject in the outlet port.

5-1-2. Cleaning

Depends on the solvents in use, the cleaning procedure is varied. Following is a procedure for typical.

[**Organic Solvent**]

1. Disconnect inlet tubing and press to turn on the valve.
2. By syringe, inject 30 to 50ml of solvents (in order of acetone, THF, chloroform, methanol and acetone).
3. Press Purge key in every 10 seconds to on/off the purge valve while injecting those solvents
4. Or, fill the flow cell(s) with acetone and leave it overnight.

[**Binding of Proteins, Salts or Sugars**]

1. Connect detector with contamination free solvent delivery pump.
2. Press Purge key to turn on the valve.
3. Pump deionized water at 1ml/min flow rate and leave it overnight.
4. You may want to wash the flow path with 0.1M NaOH priory if you know there is a potential of proteins binding.

[**Remark**]

You may want to repeat the above procedures when the result isn't quite satisfactory.

[**Real Heavy-Duty Cleaning**]

1. If the above whole procedure didn't work, you may apply 15% Nitric Acid solution.
2. Please make sure to flush the flow path with deionized water before and after of the above.
3. Apply the Nitric Acid solution by syringe and make sure the solution shall not stay more than 5 minutes.

[**Commonly Employed and Successful Procedure**]

1. Inject cleaning solution (acetone) by syringe from the inlet port (10 ml).
2. Inject deionized water by syringe from the inlet port (10 ml).
3. Inject nitric acid solution (15%) by syringe from the inlet port (10 ml).
4. Expel nitric acid solution completely by flowing deionaized water adequately.
5. Exchange deionized water with the mobile phase solvent.

If buffers or solutions of high salt content have been in use, the cells may be contaminated by precipitated salt. Large amounts of distilled, deionized water, such as 1 mL/min, for up to several hours, is the simplest clean-up procedure.

An elevated cell temperature will speed dissolution. The water wash can be acidified, if the precipitated salt is more soluble in acidic solutions. However, do not use strongly basic (pH 10 or higher) solutions, as these will etch the refractive index cells.

If contamination is suspected when a non-aqueous solvent is in use, flush the cells with a solvent that is (1) miscible with your mobile phase, (2) a good solvent for the predicted contaminant, and (3) generally of greater polarity than your mobile phase.

WARNING:
EXPLOSION HAZARD

Do not allow nitric acid to contact methanol. An explosion could result. Completely rinse the flow cell with water following cleaning with nitric acid.

WARNING:
CHEMICAL HAZARD

Corrosive acids are used. Use extreme caution to avoid spillage on skin, clothing, or the instrument. Protective gloves are advised.

CAUTION

Never put hydrochloric acid in the cell. This acid in any concentration will corrode the cell.

Diluted (10-20%) or concentrated nitric acid is a good cleaning solution.
The sample and reference cells should be filled with water or air (blown dry) before proceeding.

Filled the syringe with cleaning solution and connect to the inlet port of the Shodex RI-101. Carefully inject the cleaning solution. For safety, make sure that outlet tubing is led to drain bottle.

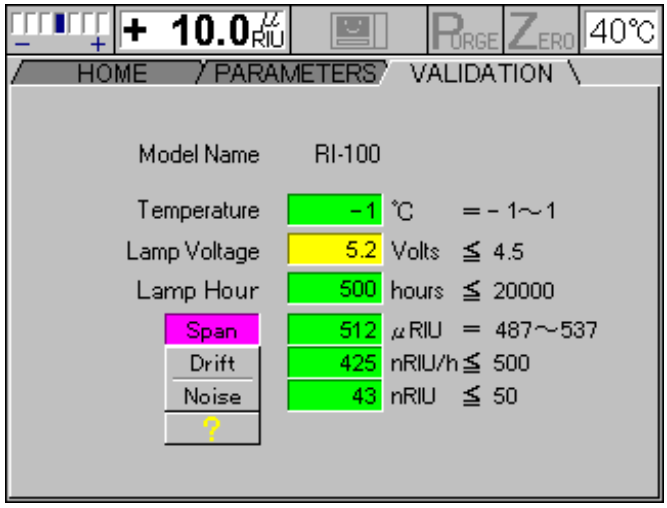
Flush acid cleaning solutions from the cells with large amounts of water, such as 1 mL/min. Flush for 15 to 30 minutes.

note

For shutdown and storage, please review Section 7. Shutdown Procedure.

5-2. Validation (Primarily for Customer Use)

From time to time, you are recommended to validate your HPLC system to keep an accuracy and credibility of your analysis. Shodex RI-101 has a very unique and user-friendly feature for this cumbersome mission.



(1)Temperature — This indicates the difference between temperature setting and actual temperature in Celsius. When the difference becomes greater than + or – 1 degree background color of this will be changed from green to yellow.

(2)Lamp Voltage — This indicates applied voltage to the lamp. As it exceeds 4.5V, the background color changes from green to yellow. Purge the reference side flow path with fresh solvent at once. (Applied voltage to the lamp is automatically raised as light intensity from the flow cell drops. At many cases the reduction of intensity is due to dirt in the reference flow cell.)

(3)Lamp Hour — This indicates accumulated service hours of lamp. When it exceeds 20,000 hours, background color of this will be changed from green to yellow. Lamp replacement is recommended.

(4)Span — Move cursor to Span by [up arrow] or [down arrow] key. Press Enter key to go on "SPAN VALIDATION" screen. (For the detail of span adjustment, please refer to the Operating Manual!) Press Cancel key to return to "VALIDATION" screen.

(5)Drift & Noise — Move cursor to Drift&Noise by [up arrow] or [down arrow] key. Push Enter key to call Start-Up Sequence to validate Detector according to the current parameters. Dialog box requests you if you really want to do this. Press Enter or Cancel key to confirm your preferred option. As the sequence starts, drift and noise are measured and shown on "VALIDATION" screen.

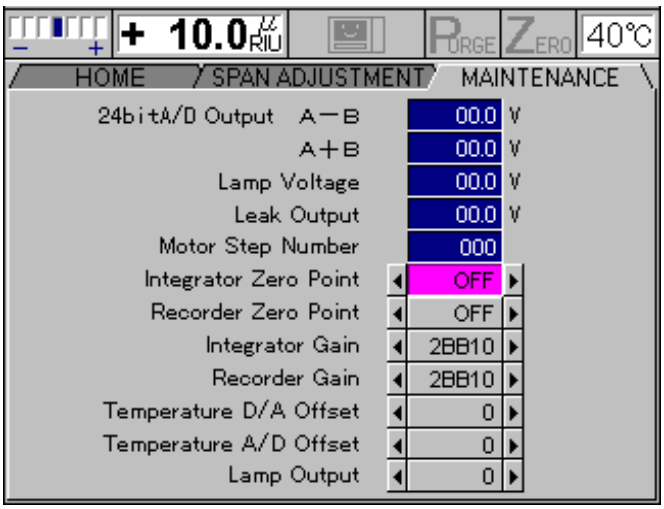
(6)Exit — To exit "VALIDATION" screen, press Cancel key. Cursor returns to the tab.

5-3. Maintenance Screen (Service Use Only)

note

To activate the maintenance screen, you have to do a special key entry. Do not make customer possible to do unless it is only needed.

1. ***Select "Home" screen.***
2. ***Press*** [up arrow] , [down arrow] ***and*** Enter ***keys simultaneously.***
3. ***Press and hold those keys for 5 seconds.***
4. ***The screen will be changed to Span Adjustment screen.***
5. ***Choose Maintenance screen by using*** [-->] ***or*** [<--] ***key.***

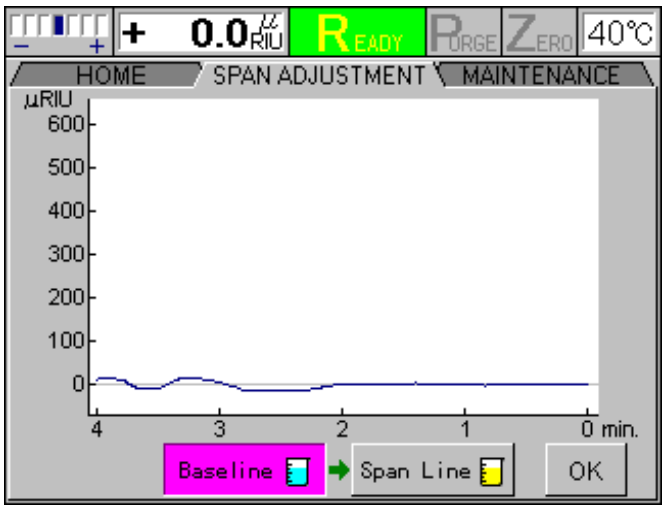


Items	Display	Remarks
24bit A/D Output A-B	__._V	
24bit A/D Output A+B	__._V	
Lamp Voltage	__._V	
Leak Output	__._V	Currently No Real Use
Motor Step Number	___	Currently No Real Use

Items	Steps	Actual Display	How to use.
Integrator Zero Point	512	OFF,3F000 to 40FF0	Chose a step giving 0 +/- 0.1mV output as monitoring Integrator
Recorder Zero Point	512	OFF,3F000 to 40FF0	Chose a step giving 0 +/- 0.1mV output as monitoring recorder
Integrator Gain	1280	29000 to 2E000	Chose a step giving 1024 +/- 1mV output as monitoring Integrator
Recorder Gain	1280	29000 to 2E000	Chose a step giving 10 +/- 0.1mV output as monitoring recorder
Temperature D/A Offset	40	-20 to +20	Currently No Real Use
Temperature A/D Offset	20	-10 to +10	Currently No Real Use
Lamp Output	256	0 to 255	Chose a step making A+B is -2.5 +/- 0.1V

Press Enter key to confirm the change. Cursor will move back to the "Maintenance" tab. If you press Cancel key no matter where cursor resides, all data will be deleted and cursor will move back to the "Maintenance" tab.

5-4. Span Adjustment Screen (Service Use Only)



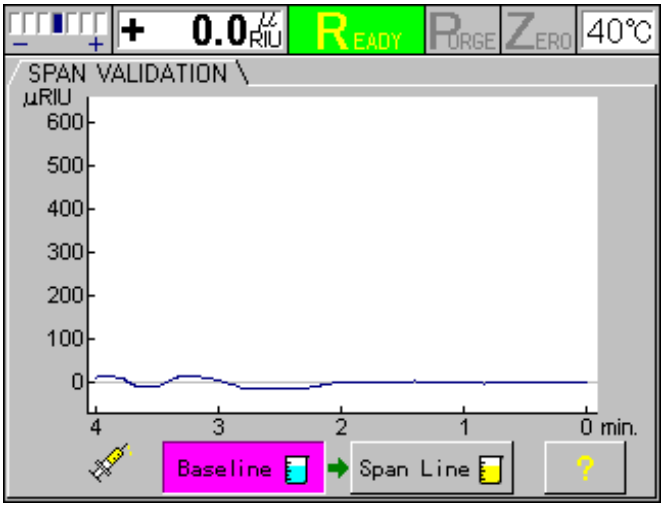
To do Span Adjustment, follow the below procedure.

1. Preparation of standard sucrose solution:
   Weigh out 350 mg sucrose and transfer quantitatively to a volumetric flask.
   Dissolve in the 100 ml deionized, filtered, degassed water and dilute to flask's mark.

note
Use freshly made sucrose solution always.

2. Equilibrate Shodex RI-101 by pumping deionized water through both reference and sample cells. Use the same deionized water as that used to prepare the sucrose standard solution. Start pumping at flow rate to 1 mL/min and do Start-Up Sequence.
3. Make sure that the baseline is stabilized and the drift is equal or less than 500 nRIU/h.
4. Make sure that the purge valve is off.
5. Press Zero key to do Auto Zero.
6. Move cursor to **Baseline** and press Enter key to memorize original(0micro-RIU)baseline. Cursor will move to **Span Line** automatically after storing original baseline information.
7. Disconnect and remove tubing from the inlet port of Shodex RI-101.
8. Make sure that the purge valve is off.
9. Filled the syringe with standard sucrose solution and gently inject from the inlet port.
10. Make sure that the baseline is stabilized
11. As you make sure that cursor is on **Span Line** , press Enter key to memorize the baseline(512micro-RIU). Cursor will move to **OK** automatically. Press Enter key to accept this baseline information as span. (If you press Cancel key no matter where cursor resides, all data will be deleted and cursor will move back to the "Span Adjustment" tab.)
12. After storing the baseline information, cursor will move back to the "Span Adjustment" tab.

5-5. Span Validation (Primarily for Customer Use)



To do Span Validation, follow the below procedure.

1. Preparation of standard sucrose solution:

2. Weigh out 350 mg sucrose and transfer quantitatively to a volumetric flask. Dissolve in the 100 ml deionized, filtered, degassed water and dilute to flask's mark.

note

Use freshly made sucrose solution always.

3. Equilibrate Shodex RI-101 by pumping deionized water through both reference and sample cells. Use the same deionized water as that used to prepare the sucrose standard solution. Start pumping at flow rate to 1 mL/min and do Start-Up Sequence.
4. Make sure that the baseline is stabilized and the drift is equal or less than 500 nRIU/h.
5. Press Zero key to do Auto Zero.
6. Move cursor to **Baseline** and press Enter key to memorize original(0micro-RIU)baseline. Cursor will move to **Span Line** automatically after storing original baseline information.
7. Disconnect and remove tubing from the inlet port of Shodex RI-101.
8. Make sure that the purge valve is off.
9. Filled the syringe with standard sucrose solution and gently inject from the inlet port.
10. As the baseline is stabilized, press Enter key to measure.
11. Validation screen will come on with the measuring result.
12. The result should be within 487 to 537(512micro-RIU +/-5%)

Section 6. Shutdown Procedure

6-1. Corrosive Solvents

Some solvents may corrode the detector, if they are left in the detector and should be thoroughly flushed from the entire system, including the reference and sample flow cell

The quartz flow cell window, in particular, is easily etched by strong bases. Do not turn power to Shodex RI-101 off without rinsing these solvents from the detector.

Some solvents can be left in the cells at the end of an operation. For example, water, acetonitrile, 2-propanol, the xylenes, and paraffinic hydrocarbons are quite innocuous. They may be left in Shodex RI-101 overnight or over a weekend.

6-2. No Flow Shutdown Versus Reduced Flow Shutdown

A continuous slow flow through Shodex RI-101 is the preferable shutdown procedure if the situation permits. (especially if buffers, tetrahydrofuran and organohalocarbons are in use.)

Reduced flow may be 0.5 mL/min to 0.01 mL/min but the HPLC pump must be able to stay primed at reduced flow.

Buffers:

Even if the buffer is non-corrosive, it is better to keep the solvent flowing at a reduced rate to eliminate the possibility of salt precipitation in the flow cells and tubing.

Tetrahydrofuran:

Because THF does oxidize, you may find that, if you keep solvent flowing at a reduced rate, the chromatographic system takes less time to re-stabilize upon start-up. Generally a reduced-flow shutdown procedure will minimize re-stabilization time; the time saved is noticeable with THF as the solvent.

Organohalocarbons, such as Methylene chloride and Chloroform:

Keep a small amount of flow to keep down the amount of corrosive chloride impurities in the cell.

6-3. Long-Term Storage

If Shodex RI-101 will not be used for a week or more, the sample and reference cells should be blown dried.

If the detector is to be exposed to sub-freezing temperatures, an antifreeze flush, such as methanol must be used. Ideally, you are suggested to expel residual solution out of RI-101 and to blown dry.

Section 7. Troubleshooting

7-1. Introduction

Malfunctions within Shodex RI-101 can arise from three general sources:

- Shodex RI-101 itself can be dirty, or operating non-optimally.
- The HPLC system can have a broken, dirty, or non-optimally operating component, but the problem is manifesting itself in Shodex RI-101.
- A mobile phase and/or column problem, which by its very nature is spread throughout the HPLC system but appears as a malfunction of Shodex RI-101.

To troubleshoot, you must be able to isolate the performance of Shodex RI-101 within the HPLC system from its performance outside the HPLC system. Therefore, this section begins with guidelines for testing Shodex RI-101as a stand-alone.

Following is the Troubleshooting Table that lists the observed phenomenon with the possible cause and the suggested solution.

7-2. Make sure how the detector itself is working.

You must know how well the detector performs by itself before you can troubleshoot it in an HPLC system.

To perform the tests, disconnect all cables from the detector except one signal cable from recorder output to a calibrated, functioning recorder. The recorder should match input to the output of Shodex RI-101 (10 mV).

Before proceeding, verify also that the locking screws on the optics module have been loosened; that the correct voltage is supplied and the correct fuse installed; and that degassed water is in the reference cell, the sample cell, and the entire flow path.

7-3. To isolate the cause of problem

As you go through the Troubleshooting Table, you will have occasional instructions for HPLC systems. Generally, however, the rule of thumb is to add one component at a time back into the HPLC system so that, should the condition arise again, the component causing the problem is indicated.

You will begin by adding the pump to Shodex RI-101 first and you will add the column last.
If another type of detector is available, it is a good idea to use it before Shodex RI-101 to aid in troubleshooting as reference. Let's assume that you have a UV detector before Shodex RI-101 and only Shodex RI-101 has a noisy baseline.

One possible implication is that the noise arises from pressure fluctuation, to which the refractive index detector is more sensitive. On the other hand, if both detectors are showing noise, a power line current may be indicated.

If both detectors show anomalous baseline performance, such as huge peaks that continue indefinitely, a bleed-off problem (material from the column or immiscible solvents trapped in the system) is more likely.

7-4. Troubleshooting (Q and A)

Problem	Possible Cause	Solution
Solvent Leak	Loosely connection at Inlet and Outlet ports.	Check if there is any solvent leak
	Broken Cell	Remove cabinet cover and check if you see a liquid drained out of optical block. Also check ports of solenoid valve for a leak.
	Damaged Solenoid Valve	
	False (Faulty) Error	Check the voltage at test point
Overheating	Malfunction of temperature sensor (Thermister).	No matter whether the system is really overheated or not, this error message shows up and applied voltage is shut off automatically. In this case, the temperature of front panel screen shall be “zero”.
	Damaged circuit or parts within the heating system.	With an extra caution, turn on the power of unit and monitor if temperature of front panel screen raise or not? It should be static if system has a broken part.
Null Glass **Home Position**	Sample and reference cells do not contain identical solutions.	Flush sample and reference cells with mobile phase.
	Sample or reference cell(s) contains air bubbles.	Flush reference cell with mobile phase. Or, inject 10ml of IPA or Alcohol from Inlet port
	Broken limit switch	Remove cabinet cover and check if the motor shaft rotates or not as the unit is powered on. The shaft extends out of optical block (right hand side of front end of optical block). If the shaft rotates into the optical block all the way and does not return, the limit switch is malfunctioned. Replace the switch.

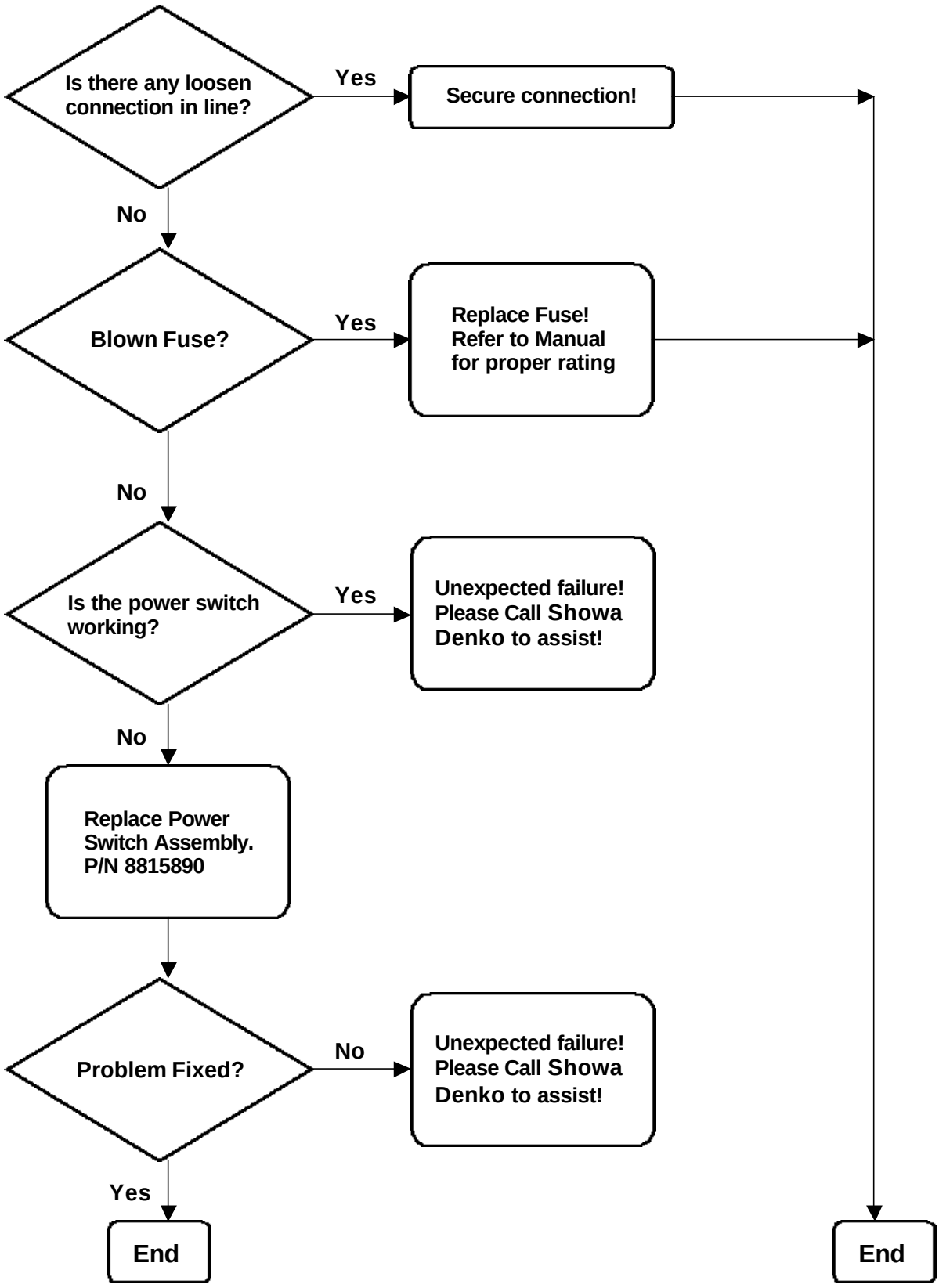
Problem	Possible Cause	Solution
Null Glass Home Position (Continued)	Stepping Motor (Motor-Lock) or Motor Malfunction	Remove cabinet cover and check if the motor shaft rotates or not as the unit is powered on. The shaft extends out of optical block (right hand side of front end of optical block). If the shaft doesn't rotate, powered off the unit and rotate manually. If you can rotate easily, there may be a loosely connection in a circuit. Disconnect the connector of motor at once and re-connect to check if problem reside with the connector connection. If you cannot, motor should be burned out. Replace the motor.
Optical Balance Error	Sample and reference cells do not contain identical solutions.	Flush sample and reference cells with mobile phase.
	Sample or reference cell(s) contains air bubbles.	Flush reference cell with mobile phase. Or, inject 10ml of IPA or Alcohol from Inlet port
	Optical axis is off.	Remove optical block cover and check if the light beam arrives in the center of sensor photodiodes. If it doesn't arrive in center evenly, carry out the optical axis adjustment procedure.
Low Light Intensity	Lamp is burned out.	Check maintenance screen and check if "A+B" voltage. If it indicates 0 volt, remove optical block cover and check if lamp is on or not. If lamp is on, refer to the below "Optical axis is off".
	Sample and reference cells do not contain identical solutions.	Flush sample and reference cells with mobile phase.
	Sample and reference cell contains air bubbles.	Flush reference cell with mobile phase. Or, inject 10ml of IPA or Alcohol from Inlet port
	Flow cell(s) dirty.	Clean flow cells. Refer to Cell Cleaning.
	Optical axis is off.	Remove optical block cover and check if the light beam arrives in the center of sensor photodiodes. If it doesn't arrive in center evenly, carry out the optical axis adjustment procedure.

Problem	Possible Cause	Solution
Noise	Bubble in the pump.	Purge pump heads. Use only premixed/degassed mobile phase.
	Bubble in the detector.	Elevate drain reservoir above the level of the flow cells to create slight backpressure. Premix and degas mobile phase well.
	Dirty flow cell(s).	Clean flow cells. Refer to Cell Cleaning.
	Weak lamp.	Check validation screen.
	Ambient temperature fluctuations.	Move detector to a more stable environment.
	Vapor pressure of the mobile phase is too high for the detector.	Reduce or turn temperature control off. Modify method to exclude or decrease concentration of troublesome solvent.
	Electrical transients from power line or radio frequency source.	Isolate the detector power source from other heavy equipment, motors, etc. Ground the detector to earth.
Noise appears in 8-10 hours of operation.	Formation of gases in the mobile phase reservoir.	Consider using on-line solvent degasser(R).
Cyclic Noise	Ambient temperature fluctuation.	Move the detector to a more stable environment. Place a cover over the detector
	Bubbles in reference cell.	Flush detector with Purge on/off
Cyclic Noise matching pump stroke frequency	Drain tube is too small (narrow bore).	Verify that correct exit line is installed. Check outlet tubing for crimps
Drift	Brown Temperature Fuse	Check temperature display on front panel screen. If it doesn't rise after power on, the fuse may be brown off.
	Flow cell(s) dirty.	Clean reference and sample cells. Refer to Cell cleaning procedure
	Flow cell(s) damaged.	Check for liquid in rear drain tube indicating a broken cell..
	Contamination from HPLC System.	Flush HPLC system with a solvent stronger than the mobile phase (less polar for reverse phase, more polar for normal phase, etc.) until contaminant disappears.

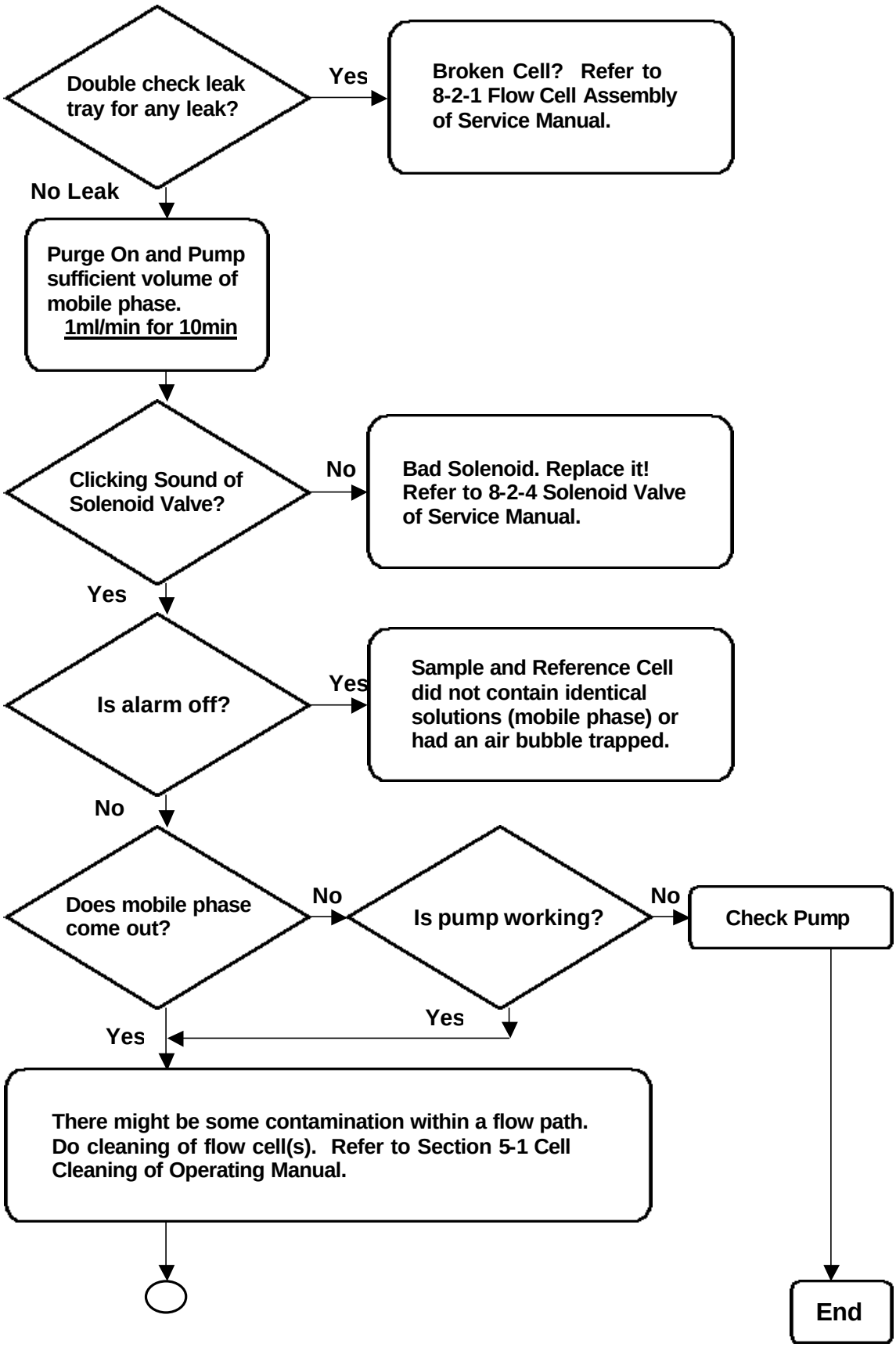
Problem	Possible Cause	Solution
	Contaminated or non-HPLC grade solvents.	Prepare fresh mobile phase (premixed/degassed)
	Vapor pressure of mobile phase is too high for operating temperature causing bubble formation in reference cell.	Reduce or turn temperature control off. Modify method to exclude or decrease concentration of troublesome solvent.
	Tetrahydrofuran (THF) in the mobile phase will oxidize in the reference cell.	Add an antioxidant to stabilize the THF, if compatible with other chromatographic requirements. Allow >2 hours stabilization time for oxidation in reference cell to reach a steady state condition.
Baseline drift occurs in few hours of start.	Reference cell solvent has aged and deteriorated.	Flush reference cell with mobile phase.
Baseline will not zero	Sample and reference cells do not contain identical solutions.	Flush sample and reference cells with mobile phase.
	Reference cell contains air bubbles.	Flush sample and reference cells with mobile phase.
	Flow cell(s) dirty.	Clean flow cell. Refer to Cell Cleaning procedure.
	Flow cell(s) damaged.	Check for liquid in drain tube indicating a broken cell.
	Deteriorating lamp or lamp out of adjustment.	Check validation screen.

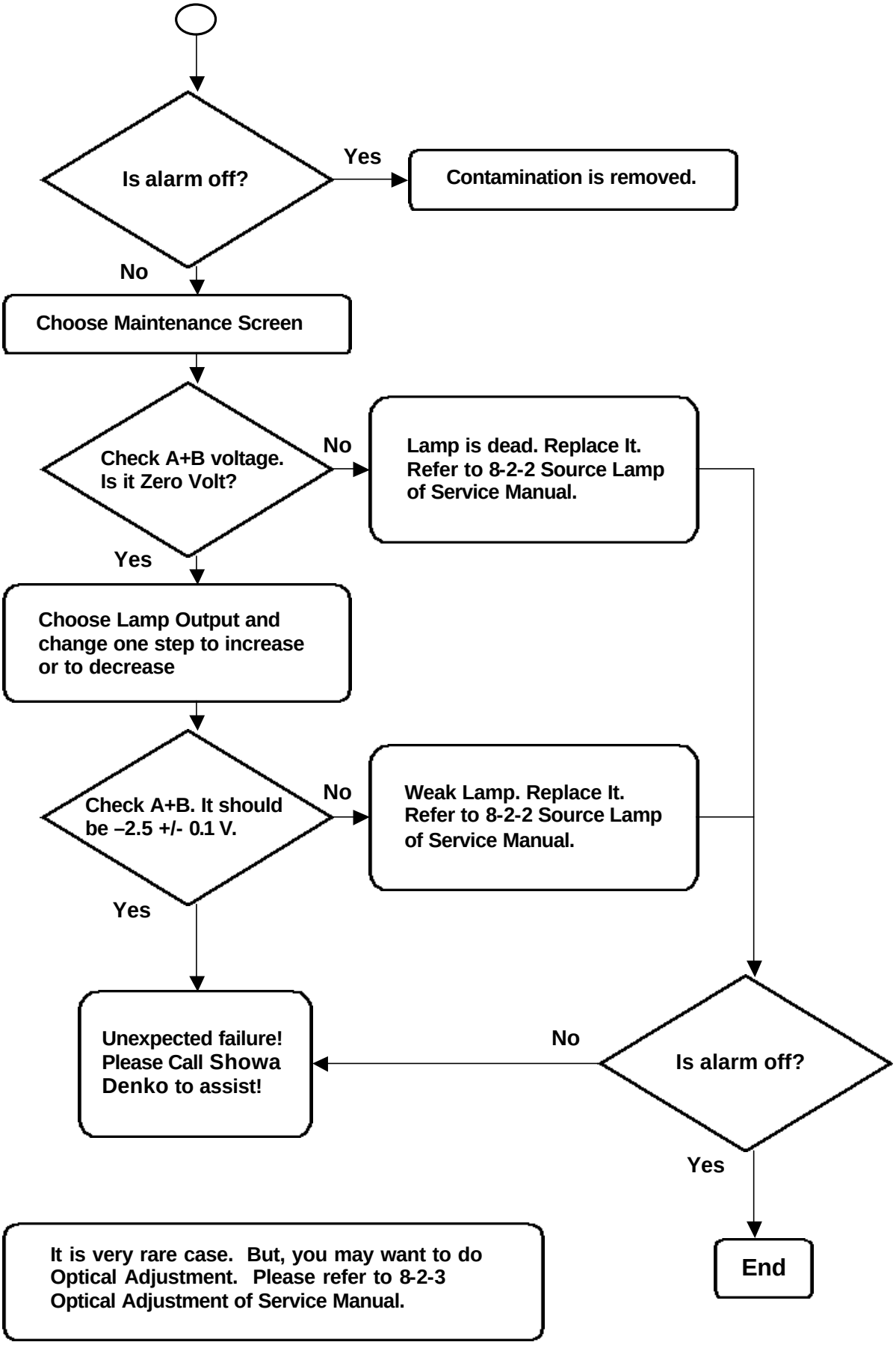
7-5. Troubleshooting (Diagnosis Flow Chart)

All Displays Are Off

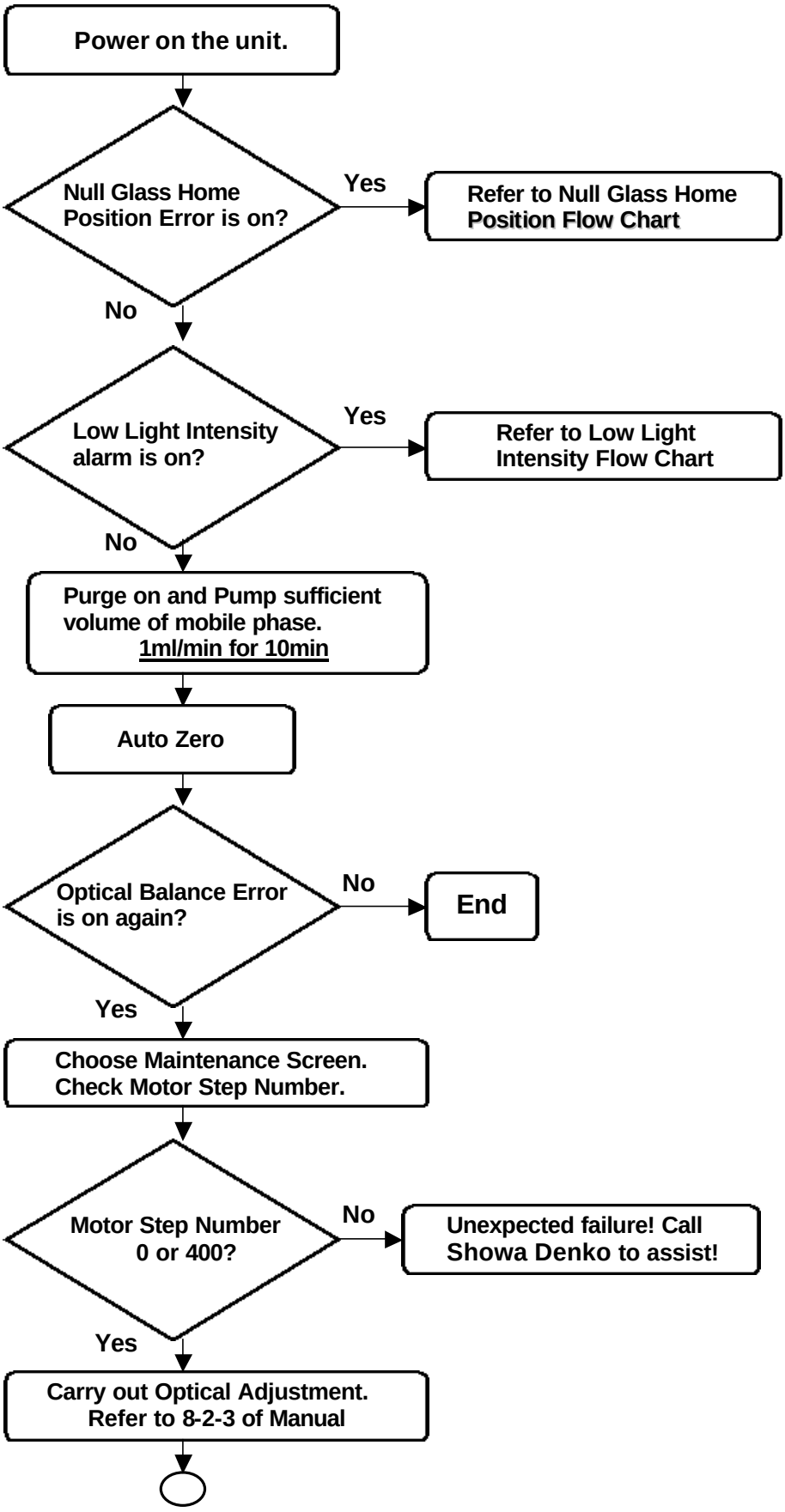


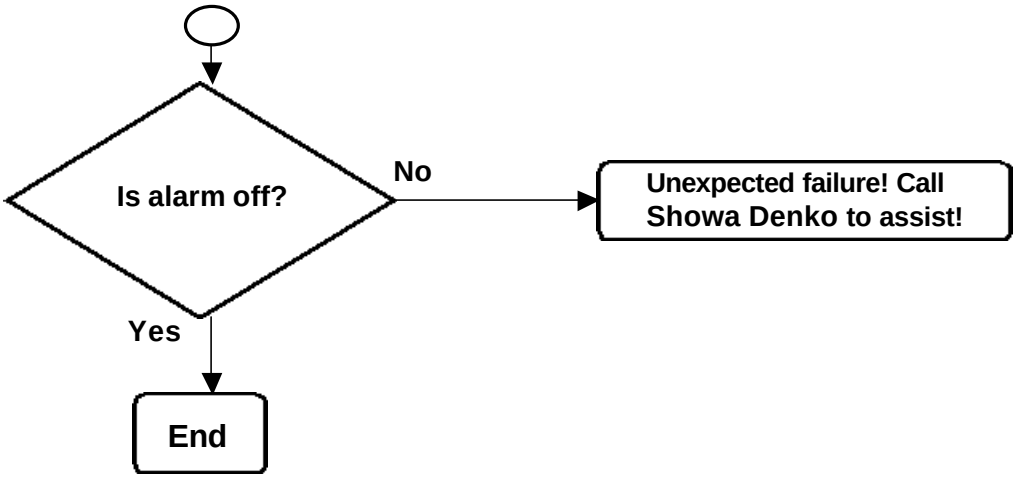
Low Light Intensity



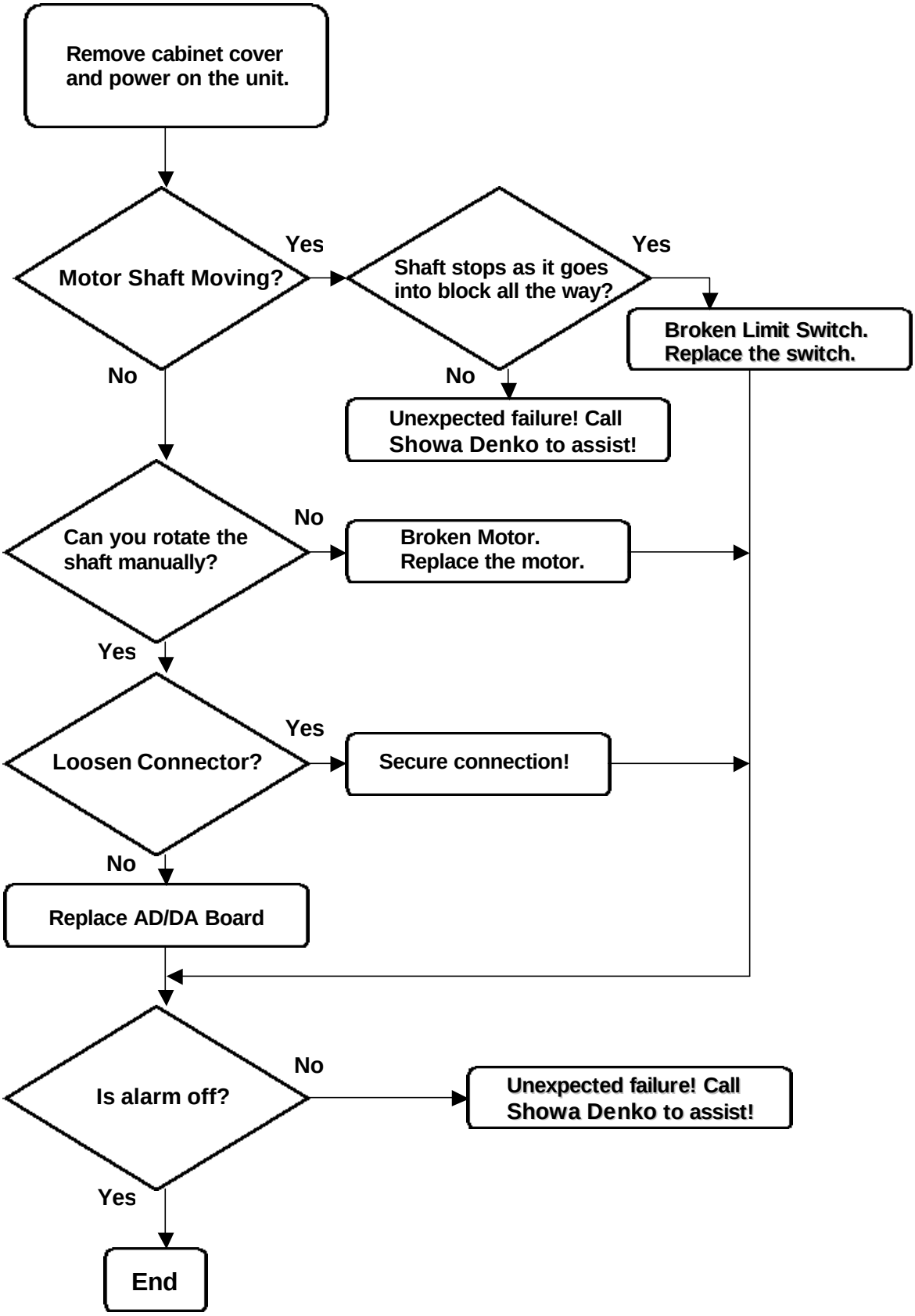

Is alarm off?
Yes
Contamination is removed.
No
Choose Maintenance Screen
Check A+B voltage.
Is it Zero Volt?
No
Lamp is dead. Replace It.
Refer to 8-2-2 Source Lamp
of Service Manual.
Yes
Choose Lamp Output and
change one step to increase
or to decrease
Check A+B. It should
be –2.5 +/- 0.1 V.
No
Weak Lamp. Replace It.
Refer to 8-2-2 Source Lamp
of Service Manual.
Yes
Unexpected failure!
Please Call Showa
Denko to assist!
No
Is alarm off?
Yes
It is very rare case. But, you may want to do
Optical Adjustment. Please refer to 8-2-3
Optical Adjustment of Service Manual.
End

Optical Balance Error

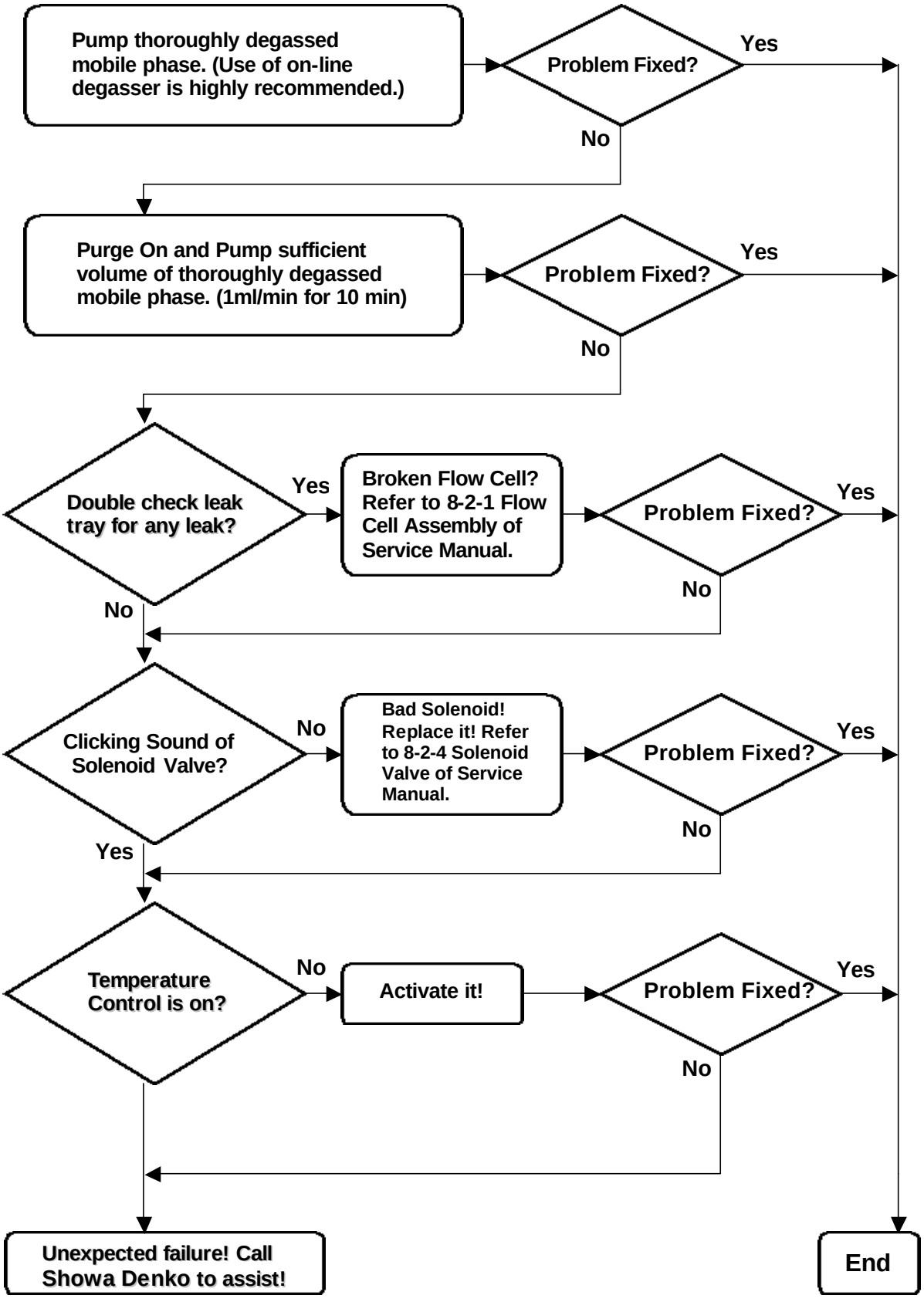



Is alarm off?
No
Unexpected failure! Call Showa Denko to assist!
Yes
End

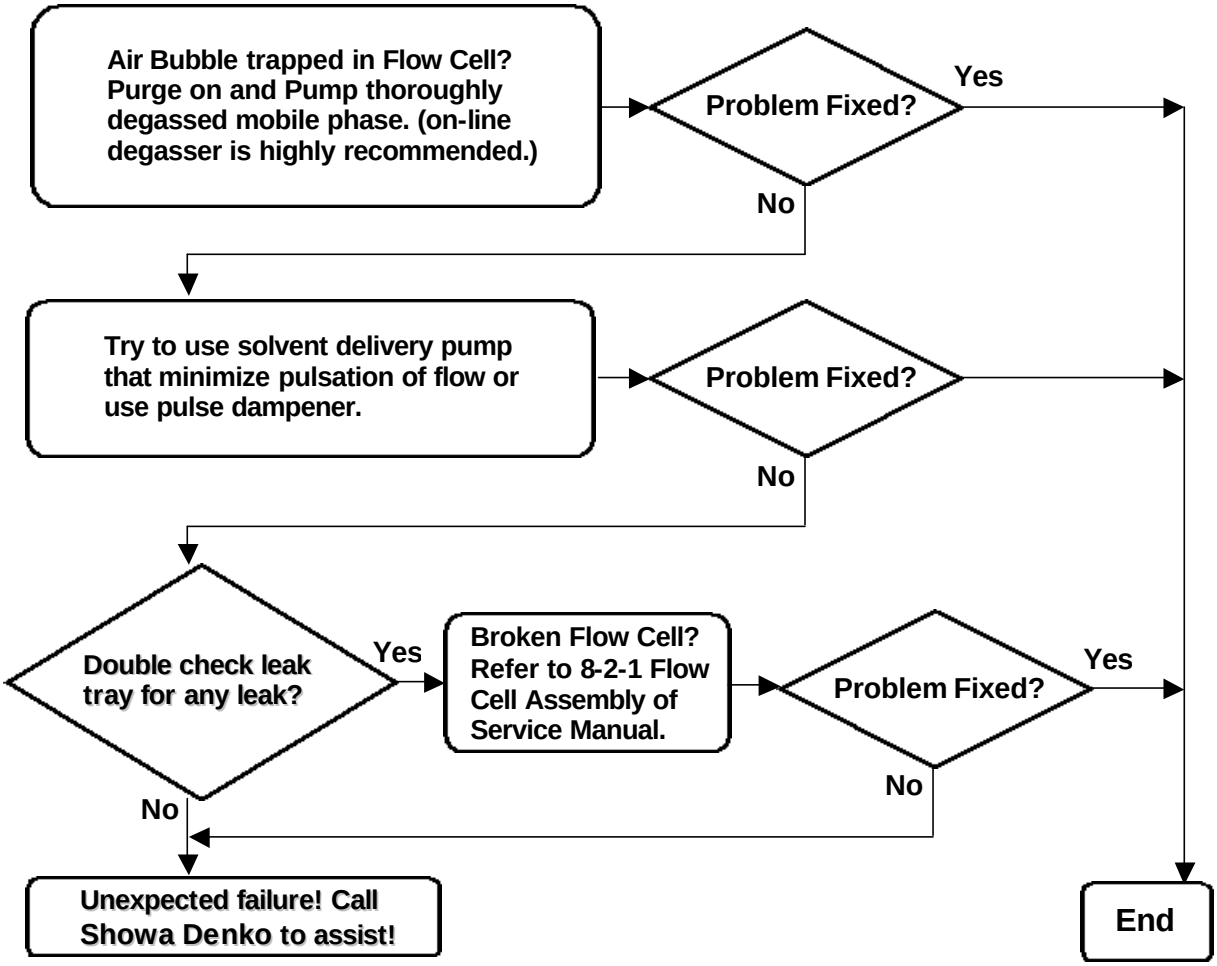
Null Glass Home Position



Excessive Drift



Excessive Noise



7-6. Test Points on PC Boards

7-6-1. A/D-D/A Board (28-5025)

Test Point #	Item	Voltage
TP 1	A-B (x1) 500micro RIU/V	
TP 2	Pre-Amp Signal Output B	0 to –2.5V
TP 3	Pre-Amp Signal Output A	0 to –2.5V
TP 4	A-B (x4) 125micro RIU/V	
TP 5	ADC Reference Voltage	3V
TP 6	A+B	-2.5V +/- 0.1V
TP 7	N/A	
TP 8	N/A	
TP 9	Power Supply (24V)	24V
TP 10	Power Supply (12V)	12V
TP 11	Power Supply (analog 5V)	5V
TP 12	Analog Ground	0V
TP 13	Power Supply (-12V)	-12V
TP 14	Power Supply (analog –5V)	-5V
TP 15	Power Supply (5V)	5V
TP 16	Digital Ground	0V
TP 17	Integrator Input Signal	-1.5V to 1.5V
TP 18	Recorder Input Signal	-1.5V to 1.5V
TP 19	Lamp Voltage (Control Signal)	=/<1V
TP 20	Lamp Voltage (Reference)	=/<1V
TP 21	Lamp Voltage	=/<4.5V
TP 22	Leak Sensor Output Signal	=/<2V
TP 23	Integrator Input	-3V to 3V
TP 24	Integrator Input (PWM)	=/<5V
TP 25	Recorder Input	-3V to 3V
TP 26	Recorder Input (PWM)	=/<5V
TP 27	Motor Pulse Signal A	=/<10Hz
TP 28	Motor Pulse Signal B	=/<10Hz
TP 29	Solenoid Valve Voltage	0V when power is on
TP 30	Heater Voltage (Driver)	24V when power is off
TP 31	Temperature	=/<5V
TP 32	Temperature (Reference)	=/<5V

7-6-2. Main Board

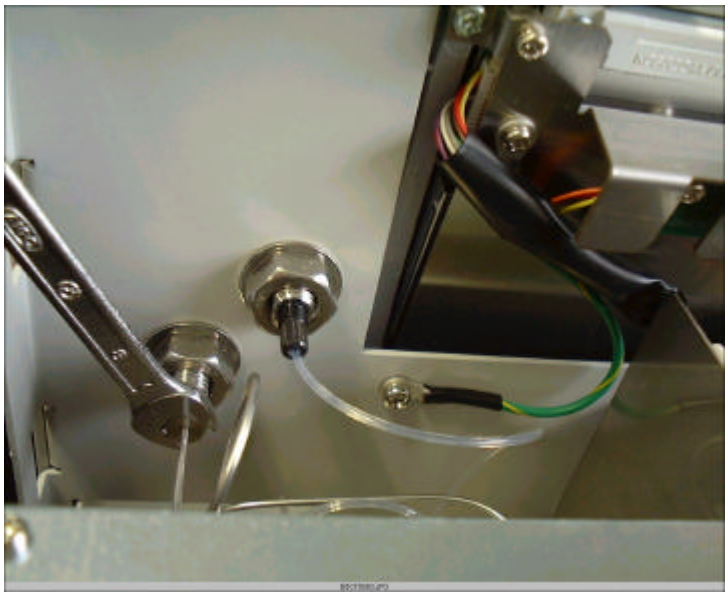
Test Point #	Item	Voltage
TP 1	Power Supply (24V)	24V
TP 2	Power Supply (5V)	5V
TP 3	Digital Ground	0V

Section 8. Repair Services

8-1. Optical Block

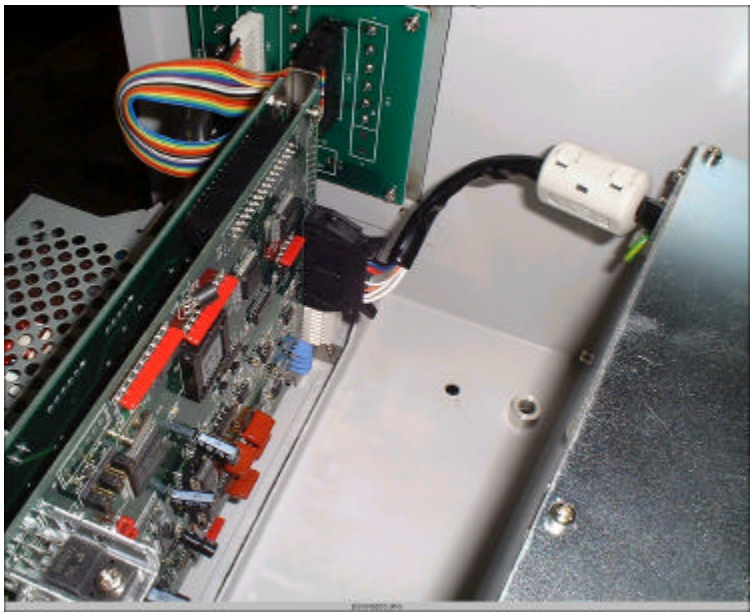
8-1-1 Dismount the optical block

1. Power off the unit
2. Remove the cabinet cover
3. Disconnect Inlet and Outlet tubing that are fixed bulkhead unions on the front panel.



4. Place the unit (optical block side as bottom) carefully as follow and remove M10 screws (4 ea) that fix the optical block onto the chassis.

5. Dismount the optical block from the chassis.

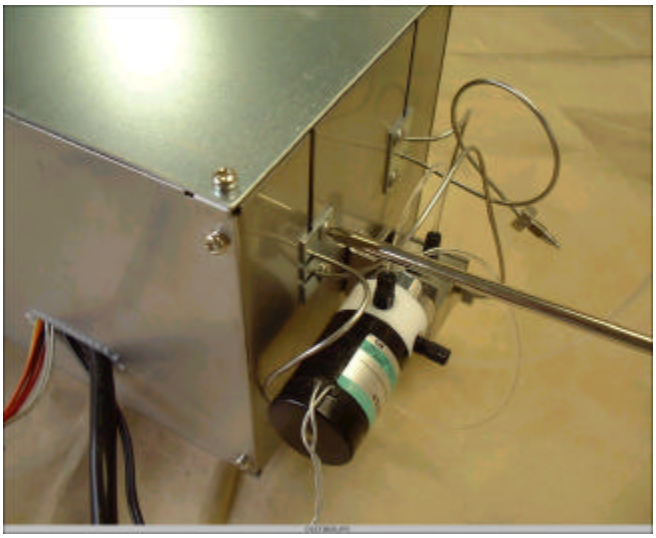


6. Detach connectors that connect the optical block and A/D-D/A board.

8-1-2 Dismantle the optical Block

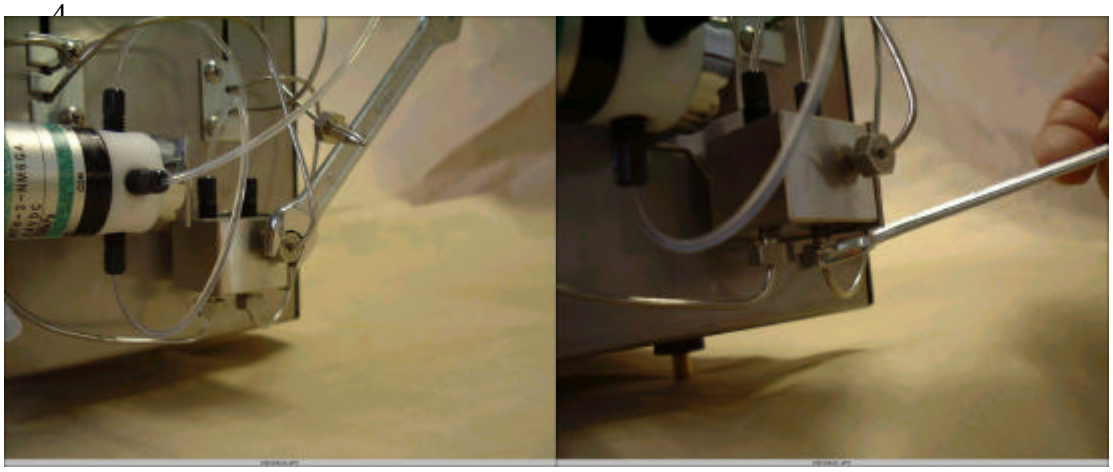
1 Loosen M3 screws (6 ea) and remove the optical block cover.

2 Loosen screws and remove brackets that hold Inlet and Outlet Capillary tubing.

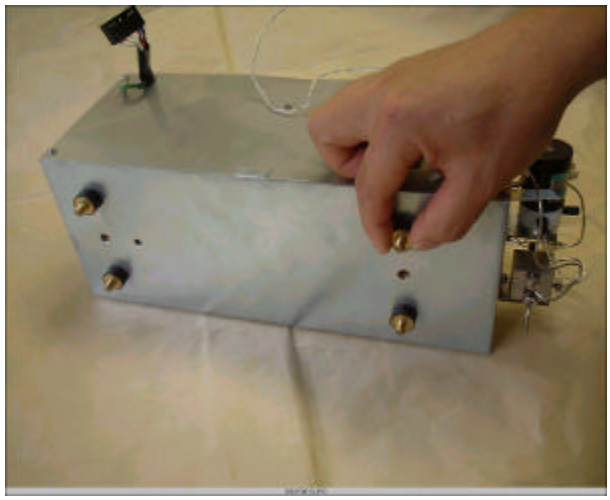


3 Loosen screws (3 ea) and remove capillary tubing from the joint block.

4



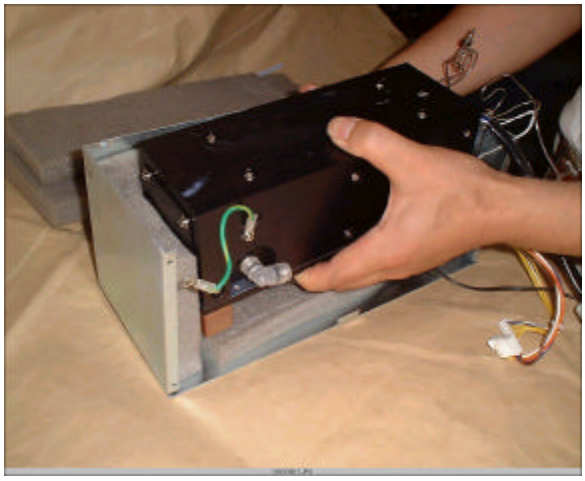
4. Loosen shock absorbing rubber foot (4 ea)



5 Loosen screws (4 ea) that fix side cover. Remove the side cover.

6 Remove heat-insulating foam. Detach drain tube from the elbow joint. (Press or squeeze the coupler edge to hatch the locking mechanism).

7 Pull out the optical block out of optical block cabinet.

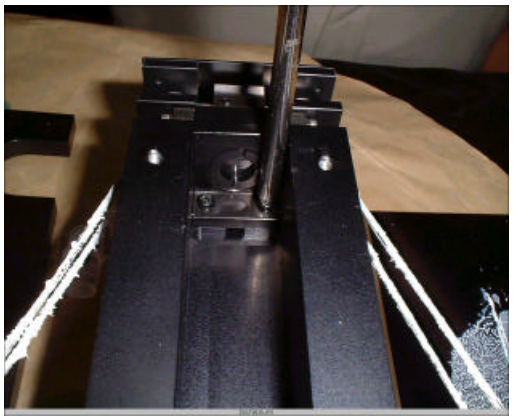


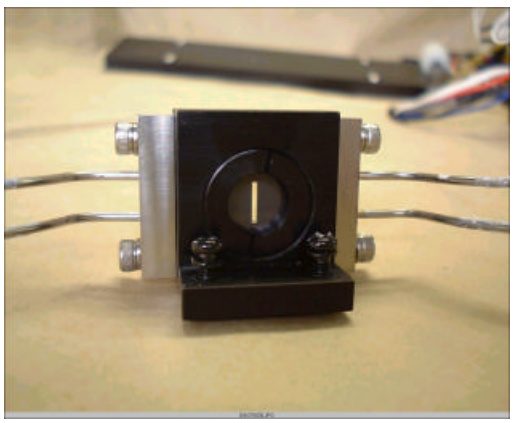
8-2 Replacement

8-2-1. Flow Cell Assembly

1. Loosen screws (4 ea) and remove the top cover of optical block.
2. Loosen M4 screws (12 ea) and remove heat-exchange block plates (both side of optical block)

3. Loosen M3 screws (2 ea) that fix the Flow Cell Assembly. Carefully dismount the flow cell assembly for not scratching or damaging the mirror sits in the rear of the flow cell assembly.

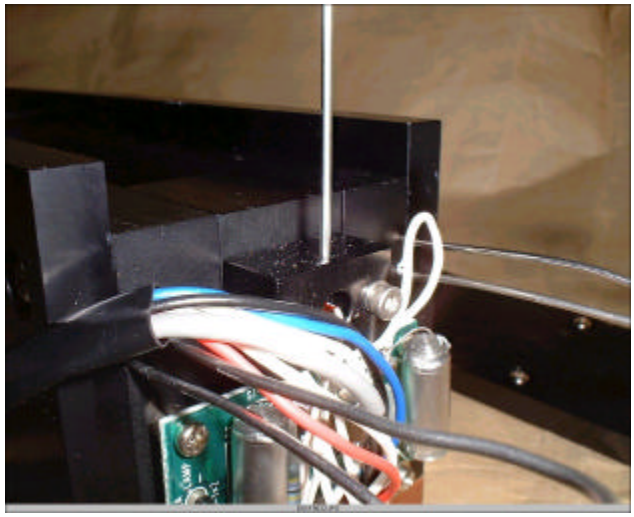




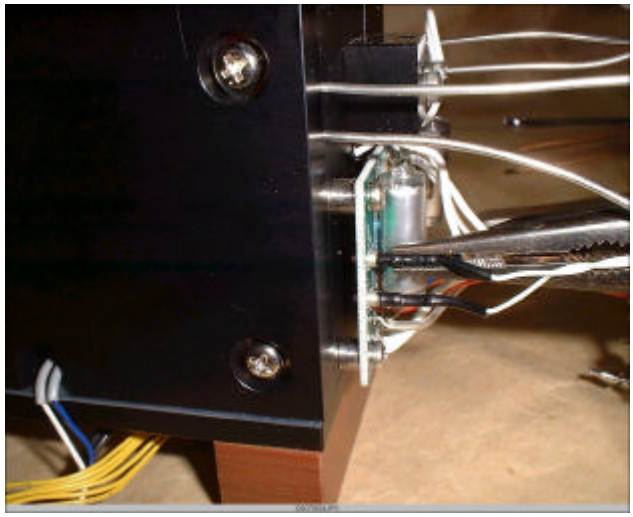
4. Mount new flow cell assembly back to the optical bench and rebuild the optical block by reversal procedure.

8-2-2. Source Lamp

1. Loosen screws (4 ea) and remove the top cover of optical block.
2. Loosen M3 set screw that mount source lamp to the lamp holder and carefully pull the source lamp out.



3. Pull off wires (2 ea) out of Pre-Amp Board.

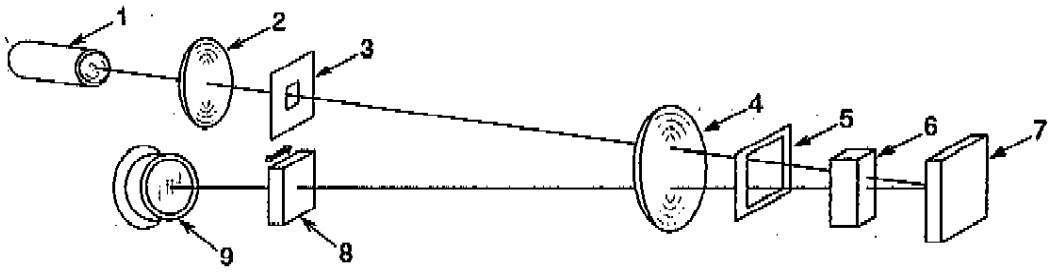


4. Mount new source lamp back to the source lamp holder by reversal procedure.
5. Make sure that filament should be positioned horizontally.

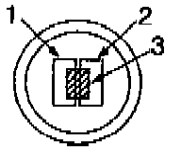
8-2-3. Optical Adjustment

After changing either flow cell assembly or source lamp, you have to do optical adjustment as follow. It is better to carry out the procedure at a place where you can control the brightness of room or area to have a better visibility of light beam.

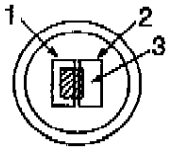
1. Connect the wire of Pre-Amp Board with connector (CN-1) of Main Board.
2. Plug in the detector to main power and power on.
3. As soon as the unit is powered, light intensity alarm shows on and beep will start. Press any key on front panel to stop the alarm beep.
4. Do not command Auto-Zero. (Let Null Glass stay at default Home Position.



5. Manipulate two setscrews (both side) of source lamp holder to adjust a position of lamp so that light beam can cover the second slit (#5 of the above figure) completely.
6. Center the light beam on photodiodes as below. **Diagonal area represents a light beam.**

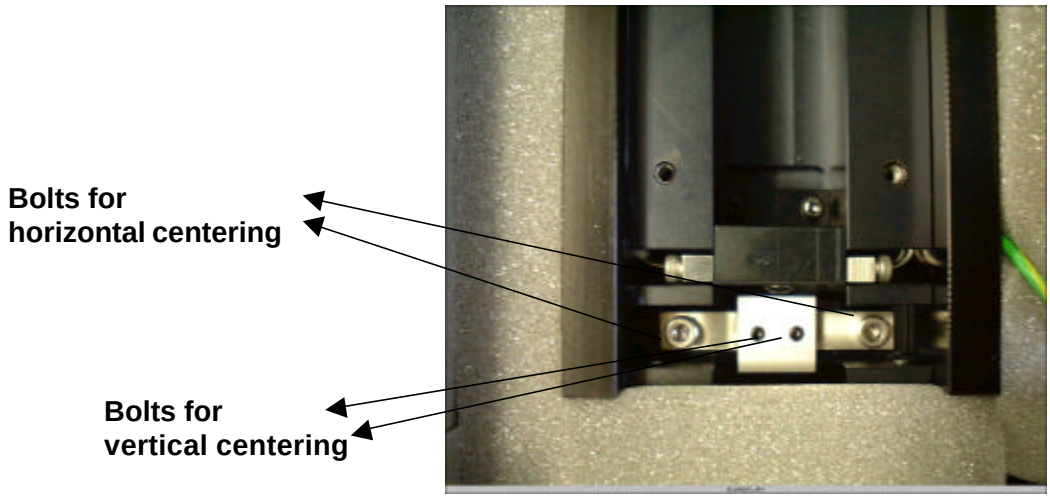


Centered



Off

7. To make the mirror position adjustable by slightly loosening two bolts (both end of mirror shaft) by Allen key (2.5mm).

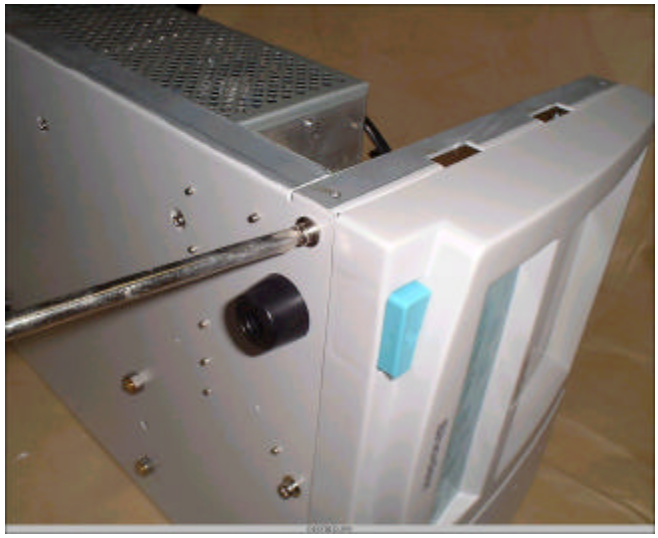


8. Adjust mirror to position the light beam in horizontal center on photodiode surface.
9. Firmly tighten them again after horizontal centering is successfully done.
10. If vertical centering is necessary, slightly loose those two bolts by Allen key (1.5mm)
11. Firmly tighten them again after vertical centering is successfully done.
12. Put the top cover back to the optical block.
13. Choose "Maintenance" screen (**Refer to 5-3. Maintenance Screen**)
14. Choose Lamp Output cell by [up arrow] or [down arrow] key and edit it by [-->] or [<--] key in order to make "24bit A/D Output A+B" is **–2.5V +/- 0.1V (it should be in 50 +/- 10 resion).**
15. Press Enter key to confirm the change. Cursor will move back to the "Maintenance" tab. If you press Cancel key no matter where cursor resides, all data will be deleted and cursor will move back to the "Maintenance" tab.

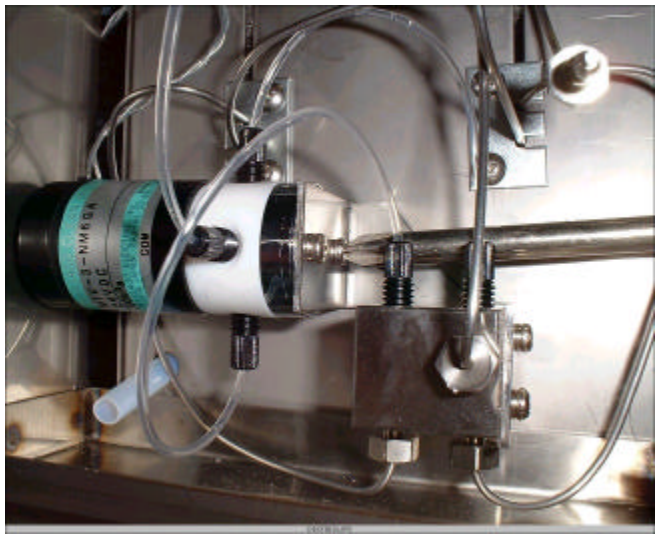
After the above procedure is done successfully, do span adjustment (**Refer to 5-4. Span Adjustment**).

8-2-4. Solenoid Valve

1. Refer to the procedure **8-1-1 Dismount the optical block** ((1) to (3))
2. Detach front panel from chassis by removing M4 screws (3 ea) as follow.



3. Dismount solenoid valve from the mounting plate by removing M3 screws (2 ea)



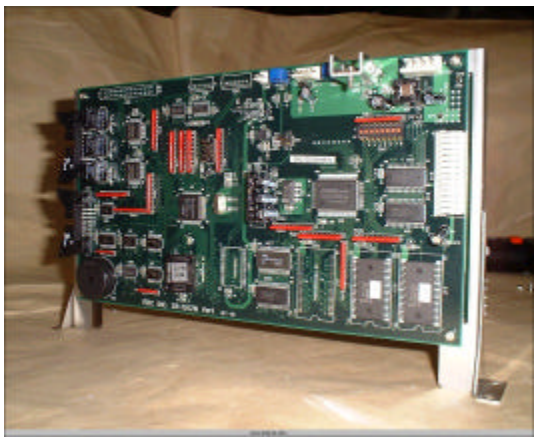
4. Loosen finger tight fittings and disconnect plastic tubing from solenoid valve as follow.

5. Detach wires of solenoid valve from A/D-D/A Board (Connector CN-10)

6. Mount new solenoid valve back by reversal procedure.
7. Make sure that tubing (in and out) are connected with right port.

8-2-5. Main Board and A/D-D/A Board

8. Refer to the procedure 8-1-1 Dismount the optical block ((1) to (3))
9. Detach front panel from chassis by removing M4 screws (3 ea)…Optional
10. Detach board stand from chassis by loosening M3 screws (4 ea) and disconnecting



cables.

11. Dismount Boards from board stand by loosening M3 screws (4 ea).
12. Mount new Boards back to board stand by reversal procedure.
13. Mount the board stand back to chassis by reversal procedure.

8-2-6. Pre-Amp Board

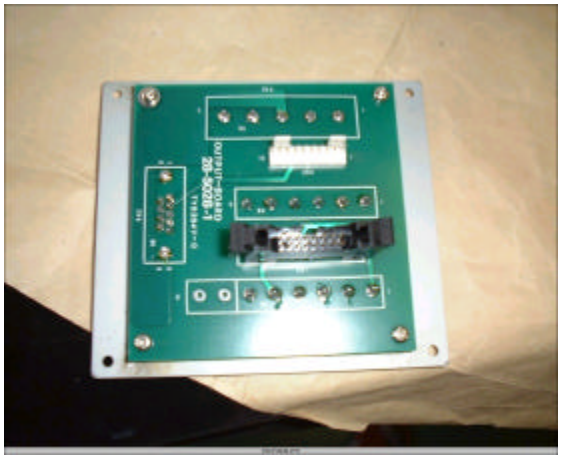
1. Refer to 8-1-1 Dismount the optical block, 8-1-2 Dismantle the optical block and 8-2-2. Source Lamp
2. Disconnect wires from source lamp.
3. Detach three wires (for photodiode) soldered onto the Pre-Amp Board.
4. Loosen M3 screws (4 ea)
5. Remove Pre-Amp Board
6. Mount new Pre-Amp Board backs to by reversal procedure.

8-2-7. Output Board

1. Detach output panel of rear panel by loosening M3 screws (4 ea) as follow.

2. Detach ribbon cable that connects with CN-1 and CN-4

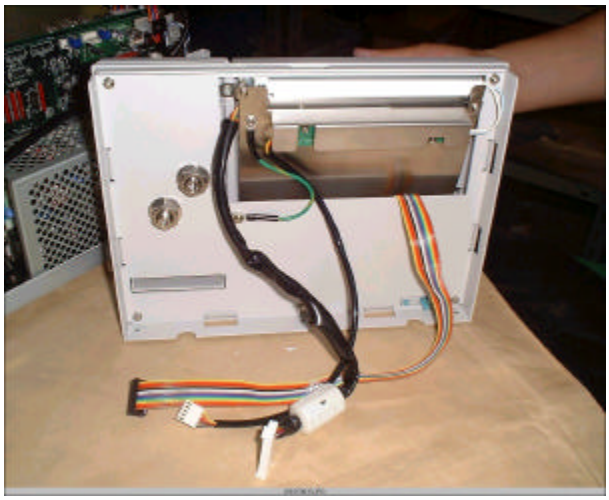
3. Detach Output Board from Output Panel by loosening M3 screws (4 ea).



4. Mount new Output Board back to by reversal procedure.

8-2-8. Front Panel Assembly and its components

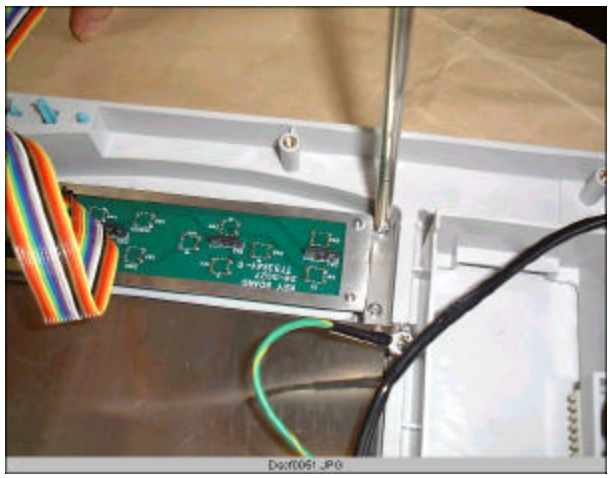
1. Refer to 8-2-4. Solenoid Valve ((1) to (2))
2. Detach cables from Main Board (CN-4, CN-5 and CN-8).



3. Detach Ground Cable from front panel (sheet metal)
4. Detach front panel (sheet metal) from the front frame by loosening M4 screws (5 ea).
5. Detach LCD Display (with mounting panel) from front frame by loosening screws (4 ea)

6. Detach LCD Display from mounting panel by loosening M3 screws (4 ea) as follow.

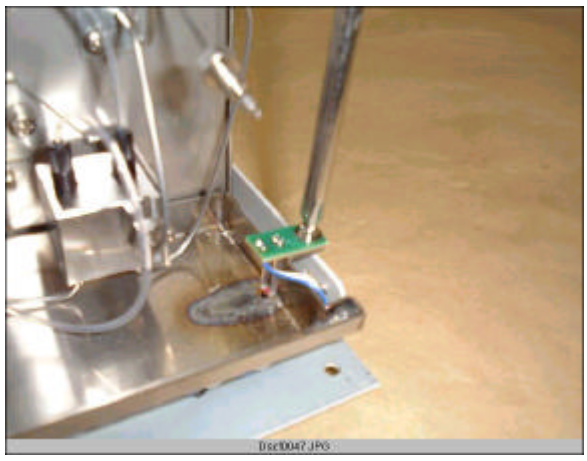
7. Detach Key Assembly from front frame by loosening screws (4 ea) as follow.



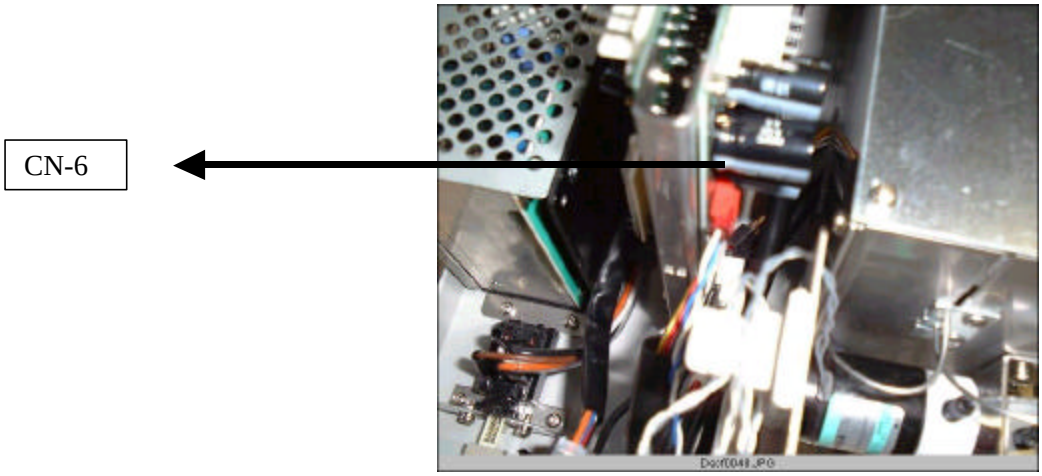
8. Mount either new Front Panel Assembly or its new components back to by reversal procedure respectively.

8-2-9. Leak Sensor

1 Refer to 8-2-4. Solenoid Valve ((1) to (2))
2 Detach Leak Sensor from metal leak tray by loosening a screw.



3 Disconnect CN-6 of A/D-D/A Board.



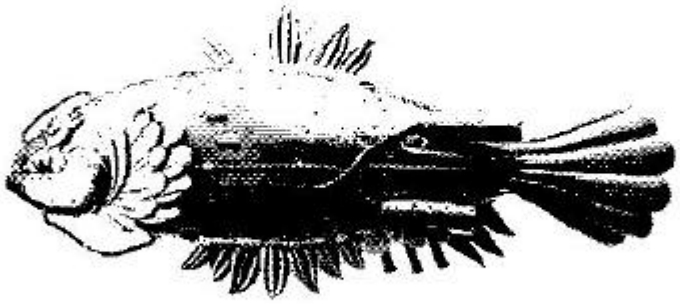
Appendix 1. Shodex RI-101 Components and Subassemblies

Item/Description	Part Number
RI-101 Refractive Index Detector Operator's Manual	9409000
A/D-D/A Board (28-5025)	8815220
Main Board (28-5026)	8815210
Pre Amplifier Board	8815470
Back-light inverter (PH-BLC08-K3)	4908230
Liquid Crystal Display (KCS057QV1AJ-A26)	4908240
Front Panel Assembly	8815180
Operation Sheet (DB69-705-1/1-0)	6970510
Membrane Key Panel (28-5027)	8810040
Lamp Assembly	8814310
Flow Cell Assembly	8815570
Optical Unit Assembly	8815050
Motor Unit	6500845
Output Board (28-5028):with rear panel	8815250
Time Lag Fuse (ET-3.15A)	2401190
Solvent Leak Sensor	8815160
Power Entry Module	2401200
Inlet Tube	8809160
Outlet Tube	8809170
Switching regulator (ECW-007d)	4908340
Accessory Kit	8815510
Signal Cable	4303190
Drain Tube	9316220
Solenoid Valve Assembly	8815090
Internal Tubing Kit (A)	9907660
Internal Tubing Kit (B)	9907670
Thermister	3800720
Bulkhead Union (Inlet Port)	6973910
Bulkhead Union (Outlet Port)	6974010
Power Cord (UC-904-J10)	4300120
Power Switch Assembly	8815890

Appendix 2. Miscibility Chart of Solvents

	Acetic Acid	Acetonitrile	Chloroform	Cyclohexane	Methylene Chloride	Dimethyl Formamide	Dioxane	Ethyl Ether	Hexane	Methanol	Methyl t-Butyl Ether	Trimethylpentane	Pentane	Propanol-2	Tetrahydrofuran	Water
Acetic Acid																
Acetonitrile																
Chloroform																
Cyclohexane		X														
Methylene Chloride																
Dimethyl Formamide				X												
Dioxane																
Ethyl Ether																
Hexane		X				X										
Methanol				X					X							
Methyl t-Butyl Ether																
Trimethylpentane		X				X				X						
Pentane		X				X				X						
Propanol-2																
Tetrahydrofuran																
Water			X	X	X			X	X		X	X	X			

X = Immiscible



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