112 UV Detector User's Guide

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A List of Expendable Parts

B Flow Cell Pressures in PSI

The Gilson 112 UV Absorbance Detector continuously monitors column effluent at a single wavelength.

Simple, front-panel controls let you set the operating sensitivity and time constant. With the touch of a button, you can reset the baseline to zero or send an event mark to your chart recorder.

Your 112 UV Detector can monitor at 254 or 280 nm and is equipped with the lamp. You can switch between those two wavelengths with the touch of a button.

With a choice of flow cell assemblies, you can use the 112 for all types of low pressure and HPLC separations: preparative, analytical and microbore.

The 112 is a compatible component of a Gilson modular HPLC system, but also functions well with other commercially available systems.

Before you use your new detector, you must follow the instructions in *Chapter 2, Installation*. Then refer to *Chapter 3, Operation* to set the front panel controls accurately.

Chapter 4, Maintenance describes how to keep your detector operating at maximum efficiency. If you do encounter detection problems, refer to the problem/solution chart in *Chapter 5, Troubleshooting* to isolate and correct the problem.

Unpack the Gilson 112 and its accessories carefully from the carton.

1

Cross-check the contents against the equipment checklist shown below and against your packing slip to verify that all parts are included and are undamaged.

The flow cell and air reference aperture specified in your order are already mounted in the flow cell assembly.

Do this now, even if the 112 will not be used immediately. Many carriers must receive concealed damage claims within seven days of delivery.

Checklist

The Gilson 112 accessory package includes:

- event marker/autozero cable (1)
- Pomona recorder cable (1)
- Upchurch[®] finger-tight column fitting (1)
- Luer fittings (1 male, 1 female)
- 1/4-28 couplers (1 package of 5)
- power cord (1)
- spare fuse (1)
- restrictor tubing assembly, 10 ft. (1, none on prep models)
- replacement outlet tubing assembly (1)

Chapter A, List of Expendable Parts lists the part numbers associated with the above.

Customer Service

Gilson, Inc. and its worldwide network of authorized representatives provide customers with four basic types of assistance: sales, technical, applications, and instrument repair.

If you need assistance, please contact your regional Gilson representative or call the Gilson Customer Service Department, toll-free at 800-445-7661 or 608-836-1551. You can also contact Gilson Customer Service via its e-mail address: service@gilson.com

Outside the U.S., contact your Gilson representative.

To help us serve you quickly and efficiently, please refer to the **Before calling us** section on page 5-6.

1

Technical Specifications

Following are specifications for the 112 UV Detector. The noise, drift, and temperature sensitivity listed below are maximums. Under most circumstances, you can expect performance that surpasses the specified limits.

Note: Included with your detector is an information sheet listing its test results obtained from the factory checkout procedure.

Technical Specification	Definition
Autozero range	-0.5 to +1 AU, to within 5 x 10^{-5} AU. Activated from front panel or by remote contact closure.
Band pass	12 nm when using phosphor-coated lamps
Detector type	UV silicon photodiode
Dimensions (w x d x h)	32 x 32 x 13 cm 12.6 x 12.6 x 5.2 in.
Display	31/2 digit LCD display shows percent of full-scale output
Drift	3.0 x 10 ⁻⁴ AU/hour
Event marker	Negative deflection on trace. Activated from front panel or by remote contact closure.

Introduction

Technical Specification	Definition					
Flow cells (fused quartz)	Volume	Path length	Sensitivity range	Application		
	11 µL	10 mm	0.001 AU-1 AU	LC/HPLC		
	40 µL	10 mm	0.001 AU-1 AU	LC		
	1.3 μL	5 mm	0.002 AU-2 AU	microbore		
	10 µL	2 mm	0.005 AU-5 AU	preparative		
	2.5 μL	0.5 mm	0.02 AU-20 AU	preparative		
	0.3 µL	0.1 mm	0.1 AU-100 AU	preparative		
	All flow cells are	rated to 70 kg/cn	n² (1000 psi)			
Lamp	Mercury (254 nm)/Phosphor-coated Mercury (280 nm) low pressure lamp					
Lamp warm-up time	Approximately 1 hour, to monitor at high sensitivity					
Low sensitivity	<4 x 10 ⁻⁴ AU/mL/min (ASTM test)					
Manufacturing standards	Meets applicable Safety and EMC certification standards; UL and CE certified					
Minimum detection limit	<8 x 10 ⁻⁵ AU, peak-to-peak					
Noise at	Short term, peak	-to-peak: 4.0 x 10 ⁻⁵	AU/cm			
254 nm (air block installed)	Long term, peak-to-peak: 6.0 x 10 ⁻⁵ AU/cm					
Noise at	Short term, peak	-to-peak: 6.0 x 10 ⁻⁵	AU/cm			
280 nm (air block installed)	Long term, peak-to-peak: 6.0 x 10 ⁻⁵ AU/cm					
Operating temperature	5-40°C					

Technical Specifications

1

Introduction

Technical Specification	Definition
Optics	Dual beam with interference filter, to monitor at a single wavelength
Power requirement	Frequency: 50–60 Hz Voltage: 100–120 or 220–240V; mains voltage fluctuations not to exceed ±10% of the nominal voltage Current rating: 1.0A for 100–120V or 0.5A for 220–220V
Contact control	 Choose from three analog output channels: One output supplies a 10 mV full scale, variable-sensitivity signal to a recorder. One output supplies a 100 mV full scale, variable-sensitivity signal to a recorder. One output supplies a 10 mV full scale, fixed-sensitivity signal. The SENSITIVITY setting of this output channel is fixed at 1.0 AUFS (Absorbance Units Full Scale). Event marks do not appear on this channel.
Reference chamber	air
Sensitivity	0.001, 0.002, 0.005, 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1 AUFS with standard flow cell installed
Temperature sensitivity	3.0 x 10 ⁻⁴ /°C
Time constant	0.1, 0.2, 0.5, 1, 2, or 5 seconds: selectable on front panel
Voltage sensitivity	${<}2x10^{-5}AU/1\%$ line variation with standard power supply
Wavelengths	254/280 nm
Weight	Net 8 kg (18 lb.)
	Gross 12 kg (28 lb.)

1

Technical Specifications

Installation

This section describes how to set up your 112 UV Detector.



Gilson 112 UV Detector (Front View)

Installation

Operating Environment

The detector can be used in normal laboratory and cold room environments. Take care to keep the detector away from drafts, smoke, and other UV-absorbing vapors.

2

Electrical Setup

Electrical connections are clearly marked on the detector's rear panel.



Voltage Selector and Fuse

You **must** verify that the voltage selector and fuse were set according to the AC voltage specified on your order.

The fuse is located behind a small, clear plastic door on the 112 rear panel. The voltage selector is a small printed circuit (PC) board located just below the fuse.



2

Check PC board

Slide the plastic door to the left to reveal the small PC board.

When facing the rear panel, you will see a number on the top left side of the PC board. The number should be the same as the input AC voltage in your area (100, 120, 220, or 240V).

- If the voltage is correct, slide the door to the right and continue the electrical setup with **Output Channels** on page 2-5.
- If the visible number does not correspond to your voltage requirement, you must remove and reorient the PC board. Use the following instructions.

Disconnect the power cord.

Push the plastic door to the left.

Push the small, black lever marked "FUSE PULL" to the left and remove the fuse.

Carefully pull the PC board straight out of the 112. Use a needle-nosed pliers to remove the PC board.

Re-insert the PC board so the desired voltage is visible at the left side.

Re-insert the correct fuse as described in **Fuse** selection.

Fuse selection

The fuse the detector uses depends on the input voltage. Use a 1 amp fuse for 100/120V AC or a 0.5 amp fuse for 220/240V AC.

Installation

Both types of fuses are 3 AG, Slo-Blo[®]-type (1/4" x 11/4").

- 1 Return the "FUSE PULL" lever to its original position.
- 2 Replace the fuse with one of the proper amperage.
- 3 Slide the clear, plastic door to the right.

Output Channels

Description

When using the 112 UV Detector, you can choose from three analog output channels:

- One output supplies a 10 mV full scale, variablesensitivity signal to a recorder.
- One output supplies a 100 mV full scale, variablesensitivity signal to a recorder.
- One output supplies a 10 mV full scale, fixedsensitivity signal. The SENSITIVITY setting of this output channel is fixed at 1.0 AUFS (Absorbance Units Full Scale). Event marks do not appear on this channel.





The output voltages mentioned above, 10 mV and 100 mV, are the values at full-scale deflection of a recorder pen. When the pen moves off-scale, the output voltage is higher than the full-scale value.

2

See *Chapter 3, Operation* for an explanation of the relationship between the SENSITIVITY setting and AUFS (Absorbance Units Full Scale).

Some useful output configurations are described in the sections that immediately follow Making connections.

Making connections

You can direct output from the detector to a chart recorder or stand-alone integrator.

To connect an output channel, use a 5-foot shielded Pomona recorder cable from your accessory kit. One end of the cable has a dual-pronged plug. The other end has two tinned wires.

Insert the dual-pronged plug into one of the detector's output channels. (Insert the prong with the GNDlabeled tab into the black jack and the adjacent prong into the red jack.)

Connect the black wire to the other device's "–" or "low" input. Connect the red wire to the device's "+" or "high" input.

If the other device requires a dual-pronged plug, use a Pomona cable adapter (part number 6374022611) to make the necessary connections.

Installation

To reduce recorder noise, you may need to connect a ground strap between the recorder's GND and "–" input connectors.



1-pen recorders

Most recorders require either a 10 mV or 100 mV input signal. Check your recorder to determine its input voltage requirement.

- Connect the 10 mV variable-sensitivity output to the recorder's input if the recorder requires a 10 mV signal.
- Connect the 100 mV variable-sensitivity output to the recorder's input if the recorder requires a 100 mV signal.

Use the SENSITIVITY knob to set the full-scale sensitivity of the variable-sensitivity outputs.

2-pen recorders

If you connect the 10 mV fixed-sensitivity output and the 10 mV variable-sensitivity output to a 2-pen, 10 mV recorder, you can record at 1.0 AUFS (fixed) and at another sensitivity (variable).

This setup lets you see both low and high concentration peaks on one chart. Use the SENSITIVITY knob to set the sensitivity of the variable output.

Data device

If your system acquires and analyzes data, you can connect a 112 output channel to the data analysis device.

Remote Event and Autozero

To send an event mark to the recorder or to reset the baseline to zero, you could press the front panel EVENT and AUTOZERO buttons, respectively. Alternatively, the event and autozero functions can be activated by other devices that provide no-voltage contact closure switches.

Warning! Do not, under any circumstances, apply a voltage to these terminals.

Remote event

If your fraction collector or injector provides contact closure outputs, then event marks can be sent to the 112 UV Detector. The detector can, in turn, send those event marks to your recorder.

When you connect the remote event input cable to your fraction collector, you can note tube advance as an event mark on your chart recorder.

Note: With an appropriate connecting cable, the event marker can be connected to many kinds of fraction collectors.

If you need a fraction collector event cable, ask your Gilson representative or the Gilson Customer Service department.

When you connect the 112 remote event input cable to a properly-equipped auto-injector, you can note the start of injection as an event mark on your chart recorder.

Installation

Remote autozero

Your auto-injector or system controller can send a remote autozero signal to the detector before injecting a sample. This remote function replaces the manual baseline reset described in *Chapter 3, Operation*.



Making connections

To make connections to a system controller, autoinjector or fraction collector, use the remote event/ zero cable in the accessory package.

That cable has a 4-pin socket that mates with the remote/zero receptacle on the detector rear panel. The other end of the cable contains four tinned wires.

Event

Pins 1 and 2 are the event mark contacts.

- The red wire is connected to pin 1.
- The black wire is connected to pin 2.

Pin 2 is circuit ground.

Autozero

Pins 3 and 4 are the autozero contacts.

- The white wire is connected to pin 3.
- The green wire is connected to pin 4.

Pin 4 is circuit ground.

Installation

Connect these wires to the contact closure outputs of the controller, auto-injector or fraction collector according to that instrument's user's guide.

2

Plumbing Setup



Inlet Tubing

The inlet tubing provided is made of either stainless steel (ss) or Teflon (Tef), depending on the type of flow cell in the detector. The table below lists the inlet tubing that Gilson provides with each flow cell.

Application	Cell volume	In	let tubing	ID
LC/HPLC	11 µL	.010"	(.25 mm)	[ss]
LC	40 µL	.031"	(.8 mm)	[Tef]
microbore	1.3 µL	.007"	(.18 mm)	[ss]
preparative	10 µL	.031"	(.8 mm)	[Tef]
preparative	2.5 μL	.031"	(.8 mm)	[Tef]
preparative	0.3 µL	.031"	(.8 mm)	[Tef]

2

You can connect the stainless steel inlet tubing:

• to an HPLC column outlet, using the Upchurch Finger-tight fitting provided. This fitting accommodates most existing female column outlets.

Insert the Upchurch Finger-tight fitting onto the stainless steel tubing as shown below. Screw the fitting into the column outlet, then finger tighten.



Alternatively, you could connect other fittings as required by your column.

• to another detector in the system. Use a coupler that is compatible with the fittings from both detectors, to ensure a leak-free connection.

Teflon

You can connect the Teflon inlet tubing from the detector:

- to a low-pressure column. Connect the female column outlet to the inlet tubing using Omnifit-type fittings.
- to another detector in the system. Use a coupler that is compatible with the fittings from both detectors, to ensure a leak-free connection.

Instructions for replacing the inlet tubing and its fittings are found in *Chapter 4, Maintenance*.

Outlet Tubing

The outlet tubing provided is made of Teflon. Depending on the flow cell installed, the outlet tubing varies in inner diameter. The table below lists the outlet tubing that Gilson provides with each of the available flow cells.

Application	Cell volume	Outle	t tubing ID
LC/HPLC	11 µL	.012"	(.3 mm)
LC	40 µL	.031"	(.8 mm)
microbore	1.3 µL	.006"	(.15 mm)
preparative	10 µL	.031"	(.8 mm)
preparative	2.5 μL	.031"	(.8 mm)
preparative	0.3 µL	.031"	(.8 mm)

You can direct the detector's Teflon outlet tubing in one of three ways:

- to another type of detector where you can monitor a second characteristic of your sample. Use an appropriate coupler to make the connection.
- to the inlet of your fraction collector if you want to collect the column effluent. Use an appropriate coupler to make the connection.
- to drain if you don't need to save the column effluent.

When using an HPLC flow cell, you may need additional back pressure on the outlet tubing to reduce bubble formation.

- If effluent is diverted to a second detector, connect the restrictor tubing to the second detector outlet.
- If effluent is going to a fraction collector, use the restrictor tubing to connect the detector outlet to the fraction collector inlet.

In place of the 10-foot restrictor tubing you can use a mechanical regulator (part number 36070905), available from Gilson.

Note: You will not need the restrictor tubing or a regulator valve when a prep flow cell is installed.

Instructions for replacing the inlet tubing and its fittings are found in *Chapter 4, Maintenance*.

Power Connection

Connect the power receptacle to an AC power source with the supplied power cord. You can purchase a power strip from Gilson for use with your Gilson HPLC System. The 112 UV Detector is a rugged, economical, fixed-wavelength detector for analytical or preparative HPLC and LC. A front panel knob allows selection of the wavelength to monitor the absorbency of almost any sample.

Operation

Start Up

After you have made the connections described in *Chapter 2, Installation*, press the front panel MAINS power switch to activate the 112.

The 112 requires a 1 hour warm up time to operate at maximum sensitivity. At lower sensitivities, you can use the 112 after less than 1 hour.

After the 112 is warmed up, it is ready to begin detection. However, you must set the parameters described in **Before the Run** on page 3-3. Set these parameters in the order shown on the following pages.

Before the Run

Wavelength

The lamp/filter assembly that you specified on your order was installed at the factory.

Your detector is equipped with the 254/280 nm lamp. Decide which monitor wavelength you wish to use. Use the front panel knob to select a wavelength:

- push the knob in to monitor at 254 nm
- pull the knob out to monitor at 280 nm.

Instructions for changing lamp filter assemblies are found in *Chapter 4, Maintenance*.

Recorder Baseline

Set the SENSITIVITY knob on the front panel to ZERO.

Using the Zero control on your recorder, set the recorder baseline position. Gilson recommends a 10% offset from the bottom of the chart.

Sensitivity

Adjust the 112 SENSITIVITY knob to the desired operating sensitivity. The setting that you choose dictates the full-scale height of a trace on a recorder.

To set the sensitivity, you must consider two factors – flow-cell path length and sample concentration.

At a constant SENSITIVITY setting, the maximum full-scale height (AUFS) increases with decreasing path length. As you decrease the flow cell's path length from 10 mm, it takes increasing amounts of material to achieve comparable peak heights.

3

Path length	Maximum full-scale sensitivity*			
10 mm	1 AUFS			
5 mm	2 AUFS			
2 mm	5 AUFS			
0.5 mm	20 AUFS			
0.1 mm	100 AUFS			
* with front panel SENSITIVITY set to 1				

Choose one of the ten front-panel SENSITIVITY settings if your 112 contains a standard 10 mm path length flow cell. If a different flow cell is installed, consult the table on page 3-4 to find the adjusted fullscale sensitivity (AUFS) for each of the front-panel settings.

	Flow Cell Path Length				
		5 mm	2 <i>mm</i>	0.5 mm	0.1 mm
Front Panel Setting	1	2 AUFS	5 AUFS	20 AUFS	100 AUFS
	0.5	1	2.5	10	50
	0.2	0.4	1	4	20
	0.1	0.2	0.5	2	10
	0.05	0.1	0.25	1	5
	0.02	0.04	0.1	0.4	2
	0.01	0.02	0.05	0.2	1
	0.005	0.01	0.025	0.1	0.5
	0.002	0.004	0.01	0.04	0.2
	0.001	0.002	0.005	0.02	0.1

Full-Scale Sensitivity of Optional Flow Cells

You can ensure that your peaks appear on-scale by running test chromatograms at various SENSITIVITY settings.

When you set the SENSITIVITY knob to a small number, the 112 will monitor at high sensitivity. As you turn the knob clockwise, the sensitivity increases.

Consider the examples and diagrams on the following pages as illustrative guides for setting SENSITIVITY.

3

- At the same SENSITIVITY setting, a sample with an Absorbance of greater than 1.0 produces a peak height that is greater than full scale.
- At the same SENSITIVITY setting, full-scale recorder pen deflection occurs when the sample flowing through the cell has an Absorbance of 1.0 AU.
- At a SENSITIVITY setting of 1.0 AUFS, a sample with an Absorbance of less than 1.0 AU produces a maximum peak height that is less than full scale.



Three samples charted at a single sensitivity. Note that the peak height varies as the sample concentration changes.

Operation

At a constant sample concentration, the peak height varies as the SENSITIVITY setting changes.

- At a SENSITIVITY setting of 1.0 AUFS, a sample with an Absorbance of 0.1 AU produces a maximum peak height that is less than full scale.
- At a SENSITIVITY setting of 0.1 AUFS, that same sample causes full-scale pen deflection.
- At a SENSITIVITY setting of 0.01 AUFS, that same sample produces a maximum peak height that is greater than full scale.



A single sample charted at three sensitivities. Note that the peak height varies as the sensitivity setting changes.

Detector Baseline

Fill the detector's flow cell by pumping pure mobile phase through the inlet tubing.

When mobile phase is flowing and all air bubbles have been removed from the inlet tubing and flow cell, press the AUTOZERO button on the front panel. The digital meter will display "00.0" (±0.1). The recorder should still be set to the baseline.

Note: You should be sure that mobile phase is flowing well before you press AUTOZERO because air bubbles, drift and noise will interfere with the autozero process.

Time Constant

The TIME CONSTANT is a measure of the amount of electronic filtering used to reduce baseline noise effects.

If you accurately set the TIME CONSTANT, you'll obtain traces with low baseline noise and good peak shape. A value of 0.5 or 1.0 (seconds) works well under most conditions.

- As you increase the TIME CONSTANT value, increased filtering results in a smoother baseline. When the time constant is too large, reduction of baseline noise may be offset by decreased peak heights.
- As you decrease the TIME CONSTANT value, sharper peaks are detected with good resolution.

Operation

To approximate the time constant value:

- set the TIME CONSTANT to 0.5 or 1.0
- perform a typical separation using your sample
- measure the width of an average peak at half-height
- the desired time constant will be roughly 1/20-1/10 of the measured peak width value.

Operation

During the Run

After setting the baseline, the SENSITIVITY, and the TIME CONSTANT, you are ready to begin the run. Start the flow through the column and inject the sample.

Digital Display

The digital display at the left side of the front panel shows the percent of full-scale recorder pen deflection.

To calculate the Absorbance of your samples, you must know the full-scale sensitivity (see **Sensitivity**) and the path length of the flow cell. You must also monitor the digital display.

Use the following formula to calculate Absorbance:

$$Abs = sensitivity(AUFS) \times \frac{DigitalReadout}{100} \times \frac{10}{Pathlength(mm)}$$

Consider the examples and diagrams on the following pages as illustrative guides for interpreting the digital display.

A change in the sample concentration affects the digital readout and the peak height if the SENSITIVITY setting and path length are held constant:

SENSITIVITY = 1 AUFS path length = 10 mm

• Effluent that causes the display to show "150.0" (percent full scale) has an absorbance of 1.5 AU. The maximum peak height recorded is greater than full-scale height.
- Effluent that causes the display to show "15.0" (percent full scale) has an absorbance of 0.15 AU. The maximum peak height recorded is 15% of fullscale height.
- Effluent that causes the display to show "1.5" (percent full scale) has an Absorbance of 0.015 AU. The maximum peak height recorded is 1.5% of full-scale height.

The readout can display up to 2X full-scale (199.9%).



A single sample charted at three sensitivities. Note that the peak height and display vary as the sensitivity setting changes.

3

Maximum Absorbance = .015 AU path length = 10 mm

- At a SENSITIVITY setting of 1.0 AUFS, the display shows "1.5" (percent full scale) and the maximum peak height recorded is 1.5% of full-scale height.
- At a SENSITIVITY setting of 0.1 AUFS, the display shows "15" (percent full scale) and the maximum peak height recorded is 15% of full-scale height.
- At a SENSITIVITY setting of 0.01 AUFS, the display shows "150" (percent full scale) and the maximum peak height recorded is greater than full-scale height.



A single sample charted at three sensitivities. Note that the peak height varies as the sensitivity setting changes.

A change in the path length affects the digital readout and the peak height if the SENSITIVITY setting and sample concentration are held constant:

Maximum Absorbance = 1.5 AU SENSITIVITY = 1 AUFS

- In a flow cell with 10 mm path length, the display shows "150" (percent full scale) and the maximum peak height recorded is greater than full-scale height.
- In a flow cell with 2 mm path length, the display shows "30" (percent full scale) and the maximum peak height recorded is 30% of full-scale height.
- In a flow cell with 1 mm path length, the display shows "1.5" (percent full scale) and the maximum peak height recorded is 15% of full-scale height.



A single sample charted using three different flow cells. Note that the peak height and display vary as the path length changes.

Event Mark

When you press the EVENT button or send a remote event signal, a negative spike appears on the recorder trace on the two variable output channels. Event marks do not appear on the fixed output channel.

The remote event signal can be activated by a fraction collector, a hand-held switch or a properly equipped injector. To install a remote event switch, see *Chapter 2, Installation*.

Autozero

When you press the AUTOZERO button or send a remote autozero signal, the detector baseline is reset to zero.

The remote autozero signal can be activated by a system controller, a hand-held switch or a properly equipped injector. To install a remote event switch, see *Chapter 2, Installation*.

3-14

Mobile Phase Tips

Degas Solvents and Buffers

Always use degassed, HPLC-grade solvents (including water) and buffers for your mobile phase.

If the back pressure on the flow cell is sufficiently low, gas bubbles may still appear, even when using degassed solvents. This can be avoided easily by connecting the pressure-regulator tubing supplied with the detector to the outlet tubing. You can also purchase a back pressure regulator valve from Gilson.

Check UV Absorbance of Solvents

Impure solvents can absorb significant quantities of UV light even when the UV cutoff may be below the monitoring wavelength. This can result in an inability to accurately set the baseline to zero. Remember to use only HPLC-grade solvents.

Manufacturer's specifications list the cutoff value (the wavelength at which the solvent is opaque) below which a solvent or buffer should not be used as a mobile phase constituent.

Interference can still occur above the rated limit, particularly when the solvent composition changes during a run. This frequently appears as a gradual baseline shift.

The table below lists commonly used solvents with low UV cutoffs.

Solvent	UV cutoff		
Acetonitrile	190 nm		
Cyclohexane	200		
Cyclopentane	200		
Decahydronaphthalene	200		
Ethanol	210		

Solvent	UV cutoff
Heptane	200
Hexadecane	190
Hexane	195
Hexanes	210
Isooctane	197
Methanol	205
Methylene chloride	233
Pentane	205
i-Pentanol	208
i-Propanol	205
n-Propanol	240
Tetrahydrofuran	212
Water	<190

Follow these instructions if:

- the flow cell needs to be cleaned or replaced
- the lamp, filter or tubing needs to be replaced
- you wish to change the unit's monitor wavelength

You should not attempt any maintenance that is not described in this chapter. If you suspect other problems with the detector, contact Gilson or your regional Gilson representative.

If you will use several flow cells with this detector, you should purchase additional flow cell assemblies from Gilson. Then follow the flow cell removal/replacement instructions on page 4-4.

To change the monitor wavelength of your unit, you must purchase a complete lamp and filter assembly from Gilson. Then follow the lamp/filter replacement instructions on page 4-14.

Flow Cell Maintenance

Follow these instructions if you need to clean or replace the flow cell. You can purchase replacement flow cells from Gilson.

Be extremely careful when working with the flow cell and its fittings. Flow cells are made of quartz and are considered expendable. They are not covered by Gilson's warranty if damaged or broken during any maintenance procedure.

Flow Cell Cleaning (Interior)



There are two ways to clean a flow cell that has become coated with UV absorbing material:

- solvent cleaning
- acid cleaning

Solvent cleaning

If you suspect that droplets of an organic-soluble contaminant or immiscible solvent contaminate the flow cell, draw or pump a series of miscible solvents through the cell.

Either of these solvent series will remove contaminants with a wide range of solubilities:

- methanol, then tetrahydrofuran, then methylene chloride, and then methanol again
- hexane, then isopropanol, then methanol, then water

Acid cleaning

If you suspect that residual proteins contaminate the flow cell, draw 50% nitric acid through the cell. Acid cleaning is a more thorough technique, though much more care must be taken.

CAUTION! Take all necessary precautions to avoid contact with eyes, skin, clothing, and equipment surfaces.

Using a 1/4-28 coupler, connect a Luer-lock end fitting to the 1/4-28 fitting on the outlet tubing.

Attach a large glass syringe to the Luer-lock end.

Place the end of the inlet tubing into a reservoir of 50% nitric acid.

Draw nitric acid through the cell.

Note: Do not pump acid through the cell! You should never put acid under pressure. Use nitric acid. Do not use halogenated acid.

After the acid flush, draw HPLC-grade water through the cell in the same manner.

Reconnect the column to the inlet tubing. Redirect the outlet tubing to drain.

Finish by pumping HPLC-grade water to cleanse the last traces of acid.

To be sure that all acid has been removed, monitor the pH of the final outflow.



Proper maintenance of the detector includes periodic removal of the flow cell for cleaning. You should remove and clean the cell immediately if a leak occurs at the cell inlet or outlet.

Note: If you have experienced flow cell leakage, you should replace the ferrule on the end of the inlet tubing. See **Inlet Tubing Replacement** on page 4-9 for instructions.

Remove faceplate

Unscrew the thumbscrews on the front panel faceplate.

Remove the entire flow cell assembly by sliding it towards you. You will feel some resistance as you remove it.

From the right side of the assembly, you will see:

- the flow cell, inserted into a rectangular hole
- the reference aperture, in a circular hole

Loosen fittings

Loosen the 1/4-28 inlet and outlet fittings located above and below the flow cell.

Remove/clean cell

Carefully invert the assembly until the cell drops into the palm of your hand. The reference aperture plate is secure and will not drop out.

Moisten the quartz windows with alcohol or a laboratory cleaning solution. Then wipe the windows with a piece of lens paper or lint-free towel.

Clean the inside of the flow cell mounting area with a lint-free towel soaked in alcohol.

Carefully dry all surfaces of the assembly.

Replace cell

Without touching the quartz windows, carefully insert the flow cell into its receptacle.

Note: One corner of the cell is beveled. Be sure to align that edge with the bevel on the receptacle.

Seat the flow cell firmly. Push the cell as far into the hole as it will go. A poorly aligned flow cell may restrict or block flow.

Stainless steel inlet tubing

Position a 1/4-28 fitting and a ferrule on the flow-cell end of the stainless steel tubing as shown in the illustration below. The tubing should extend about 1 mm beyond the ferrule.

4



Finger-tighten the inlet fitting. As you tighten the fitting, the tubing will be pushed flush with the end of the ferrule.

Teflon inlet tubing

Position a 1/4-28 fitting and an Omnifit gripper on the flow-cell end of the Teflon inlet tubing as shown in the illustration on the next page. The gripper should be flush with the end of the tubing.

Finger tighten the inlet fitting. Then firmly tighten the inlet fitting using a 5/16" wrench (1/8 turn).

Teflon outlet tubing

Position a 1/4-28 fitting and an Omnifit gripper on the flow-cell end of the Teflon outlet tubing as shown in the previous illustration.



Finger tighten the outlet fitting. Then firmly tighten the outlet fitting using a 5/16" wrench (1/8 turn).

Inspect flow cell

Inspect the quartz window again. If it is dirty, wipe once more with lens paper.

Check flow

Before you replace the flow cell assembly into the detector, test the integrity of the inlet and outlet fittings. Reconnect the inlet tubing to your system and initiate flow.

If you notice leakage from the inlet or outlet fittings, you must remove, clean, and re-install the flow cell. Carefully install the fittings as described previously.

Replace faceplate

When no leaks are found, replace the flow cell assembly into the detector. Tighten the thumbscrews on the flow cell assembly faceplate. Reconnect the inlet and outlet tubings to your system.

Flow Cell Unclogging

To try to eliminate a blockage from the cell or inlet or outlet tubing:

Remove faceplate

Unscrew the thumbscrews on the front panel faceplate.

Remove the entire flow cell assembly by sliding it towards you. You will feel some resistance as you remove it.

Pump solvent

Very carefully pump solvent backwards through the flow cell. Connect a high-pressure pump to the outlet and direct the inlet to waste.

CAUTION: Set the maximum pressure below 300 PSI (20 BAR)!

Beginning at a flow rate of 0.1 mL/min, gradually increase the flow rate, in increments of 0.1 mL/min.

A significant pressure drop indicates that the blockage has been removed.

Purchasing Other Flow Cells

The standard quartz flow cell contains an 11 μ L cavity with a 10 mm path length.

Additional flow cell/reference aperture assemblies are available from Gilson. With a choice of several flow cells, you can quickly and easily vary the detection limits and increase the versatility of the detector.

Assemblies contain a flow cell, a reference aperture plate and tubing of the appropriate inner diameter (ID).

Other flow cell assemblies available from Gilson are listed in *Chapter 1, Introduction*.

Inlet Tubing Replacement

Note: You should ensure that the inner diameter of your inlet tubing is appropriate for the flow cell that is installed.

You can purchase pre-bent stainless steel inlet tubing for use with the standard HPLC flow cell [ID 0.010" (0.25 mm)] or with the microbore flow cell [ID 0.007" (0.18 mm)].

Other flow cells require Teflon inlet tubing as specified in *Chapter 2, Installation*.

Remove old tubing

To remove old stainless steel or Teflon inlet tubing, remove the fitting from the column end.

Unscrew the thumbscrews on the front panel faceplate.

Remove the entire flow cell assembly by sliding it towards you. You will feel some resistance as you remove it.

Pull the old tubing through inlet hole on the faceplate.

Secure new tubing

Push the new tubing through the inlet hole on the rear of the faceplate.

Stainless steel inlet tubing

Locate a new Upchurch ferrule from the detector accessory package. It is identical to the ferrule you just removed.



Place the fitting and ferrule on the flow-cell end of the stainless steel tubing as shown in the illustration above. The tubing should extend about 1 mm beyond the ferrule.

Finger tighten the inlet fitting. As you tighten the fitting, the inlet tubing will be pushed flush with the end of the ferrule.

Teflon inlet tubing

Position a 1/4-28 fitting and an Omnifit gripper on the end of the inlet tubing as shown below. Use a razor blade to cut the tubing and a needle-nosed pliers to pull the tubing through the gripper.



Teflon outlet tubing

Finger tighten the outlet fitting. Then firmly tighten the outlet fitting using a 5/16" wrench (1/8 turn).

Check flow

Before you replace the flow cell assembly into the detector, test the integrity of the inlet and outlet fittings. Reconnect the inlet tubing to your system and initiate flow.

If you notice leakage from the inlet or outlet fittings, you must remove, clean, and re-install the flow cell. Carefully install the inlet fitting as described above.

Replace faceplate

When no leaks are found, replace the flow cell assembly into the detector. Tighten the thumbscrews on the flow cell assembly faceplate. Reconnect the inlet and outlet tubings to your system.

Outlet Tubing Replacement

Remove old tubing

Unscrew the thumbscrews on the front panel faceplate.

Remove the entire flow cell assembly by sliding it towards you. You will feel some resistance as you remove it.

Pull the old tubing through the outlet hole on the faceplate.

Secure new tubing

Push the new tubing through the outlet hole on the rear of the faceplate.

Position a 1/4-28 fitting and an Omnifit gripper on the end of the outlet tubing as shown below. Use a razor blade to cut the tubing and a needle-nosed pliers to pull the tubing through the gripper.



Finger tighten the outlet fitting. Then firmly tighten the outlet fitting using a 5/16" wrench (1/8 turn).

Check flow

Before you replace the flow cell assembly into the detector, test the integrity of the inlet and outlet fittings. Reconnect the inlet tubing to your system and initiate flow.

If you notice leakage from the inlet or outlet fittings, you must remove, clean, and re-install the flow cell. Carefully install the inlet fitting as shown on page 4-9.

Replace faceplate

When no leaks are found, replace the flow cell assembly into the detector. Tighten the thumbscrews on the flow cell assembly faceplate. Reconnect the inlet and outlet tubings to your system.

Lamp Replacement

Caution! Never touch a hot lamp! After turning off the detector, wait 30 minutes or more before replacing the lamp.

Δ

Before performing the maintenance described below, you must unplug the detector from its power source. Do not remove the cover until you have done this.

Remove cover

Remove the cover by removing the four screws from the sides of the unit. Lift the cover straight up.

Place the detector in front of you, so you are facing the front panel.

Locate the lamp/filter assembly below and to the right of the white plastic knob (the filter-block release). Use the following drawing as a reference.



On the right side panel of the detector is a cut-out that eases access to the lamp assembly.

Disconnect connector

The cable from the lamp/filter assembly terminates with a 6-pin connector that is visible at the right side of the unit. Disconnect the 6-pin connector.

Remove lamp/filter assembly

To remove the lamp/filter assembly,

- unscrew and remove the 254/280 wavelength selection knob (standard units only)
- lift the white plastic knob and slide the lamp/filter assembly toward the rear of the unit

Detach old lamp (note old lamp type)

To detach a mercury (or phosphor-coated mercury or 254/280 nm) lamp from the lamp/filter assembly, carefully remove the two screws that secure the lamp to the filter block. Do not touch the surface of the lamp.

Note: If you need to install a new filter, you should do that now, while the unit is dissembled. Instructions for filter replacement are found on page 4-17.

Install new lamp (note lamp type)

To install a new 254/280 nm lamp:

- 1 Position the lamp so it is adjacent to the filter. Be sure that the lamp is not inverted relative to the filter block. (The new lamp is pre-aligned in a mounting block.)
- 2 Secure the lamp to the filter block with two screws
- 3 Replace the lamp/filter assembly (lift the white knob).
- 4 Reconnect the 6-pin connector.



Replace and secure the detector's cover using the four screws you removed earlier.

Filter Replacement

Before performing the maintenance described below, you must unplug the detector from its power source. Do not remove the cover until you have done this.

Note: To replace a filter, you will need internal C-ring pliers.

Unplug the 6-pin lamp connector and remove the lamp/filter assembly as described in Lamp Replacement on page 4-14.

Using C-ring pliers, remove the C-ring from the circular chamber on the filter block.

Invert the filter block and let the filter cartridge drop into the palm of your hand.

Taking care not to touch the optical surface, place the new filter into its chamber. Secure the new filter cartridge with the C-ring.

If you encounter a problem while operating the 112 UV Detector, refer to the following pages. If you cannot solve or isolate the problem, contact the Gilson Customer Service Department or your local Gilson representative. See **Before calling us** on page 5-6.

Troubleshooting Chart

Baseline Spikes

Eliminate bubbles that pass through the flow cell by degassing solvents.

5

Check fittings for a leak in system, repair as needed.

Voltage transients on a single circuit may interfere with detector output.

- ensure that the detector and recorder are connected to the same outlet
- eliminate voltage transients by connecting detector and recorder power lines to a constant voltage transformer or line filter

Reduce pump noise by changing compensation setting on pump and by examining check valves.

Baseline Noise

Allow lamp to warm up.

Check flow cell for contamination.

If you see baseline deflection when back pressure is exerted on the flow cell, you probably have bubbles trapped in the cell. Try one of these methods to eliminate bubbles:

- increase back pressure on the cell by connecting pressure regulator tubing to the flow cell outlet
- flush flow cell using a syringe filled with fresh solvent
- raise the collector vial to a level higher than the cell

Check grounds on all instruments; eliminate ground loops by plugging detector and recorder into the same outlet.

Troubleshooting

Eliminate drafty air currents.

Eliminate smoke from the environment.

Lamp may be weak.

Filter may be defective.

Baseline Drift

Eliminate changes in ambient temperature. If drift is dependent on temperature, eliminate drafts and avoid areas of direct sunlight.

Check flow cell for contamination.

Check for contaminated or "bleeding" column. If drift disappears after you stop the flow, then check the solvents and regenerate or replace column.

If you see baseline deflection when back pressure is exerted on the flow cell, you probably have bubbles trapped in the cell. Try one of these methods to eliminate bubbles:

- increase back pressure on the cell by connecting pressure regulator tubing to the flow cell outlet
- flush flow cell using a syringe filled with fresh solvent
- raise the collector vial to a level higher than the cell

Check pump or delivery system to determine that flow rate is constant.

Lamp may be weak.

Filter may be defective.

Check flow cell for leak.

Troubleshooting

Unable to Zero

Check connections to recorder. Refer to recorder's troubleshooting instructions.

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Check purity and UV-cutoff of solvents. Use only HPLC-grade solvents.

Eliminate bubbles trapped in flow cell. Increase flow rate and apply back pressure by using syringe or septum to momentarily block exit stream.

Check flow cell for contamination.

Check for contaminated column. If necessary, regenerate or replace column.

Check flow cell for leak. Tighten flow cell connections.

No UV Illumination

Fuse may be blown.

4-pin power supply connector and/or 6-pin lamp connector may be loose.

Lamp may be defective.

Decreased Response to Known Sample Concentration

Lamp may be weak.

Filter may be defective.

Check connections to recorder.

Verify sample concentration and solvent.

Unit not functioning

Check cord and power connection.

Fuse may be blown.

Repair and Return Policies

Before calling us

Gilson Customer Service personnel will be able to serve you more efficiently if you have the following information:

- the serial number and model number of the instruments involved. The serial number is visible on the back panel of the detector.
- the installation procedure you used
- list of concise symptoms
- list of operating procedures and conditions you were using when the problem arose
- list of other devices connected to the fraction collector and a description of those connections
- list of other electrical connections in the room

Warranty repair

Units covered under warranty will be repaired and returned to you at no charge. If you have any questions about applicability, please contact Gilson or your authorized representative.

Non-warranty repair

For out-of-warranty repairs, contact your local Gilson representative or the Gilson Customer Service Department. A Customer Service representative will discuss service options with you and can assist in making arrangements to return the equipment, if necessary.

Rebuilt exchange

For some units, rebuilt exchange components are available. Contact Gilson for details.

Troubleshooting

Return procedure

In the United States, contact the Gilson Customer Service Department to obtain authorization before returning any Gilson equipment. To return a piece of equipment:

- Carefully pack the unit to prevent damage in transit. Check with Gilson regarding proper method of shipment. No responsibility is assumed by Gilson for damage caused by improperly packaged instruments. Indicate the authorization on the carton and on the packing slip.
- Always insure for the replacement value of the unit.
- Include a description of symptoms, your name, address, phone number, and purchase order to cover repair costs, return and shipping charges, if your institution requires it. Ship to:

Gilson, Inc. Attention: Customer Service (indicate the authorization here) 3000 W. Beltline Highway Middleton, WI 53562

Outside the United States, contact your Gilson representative for return procedures.

Repair and Return Policies



Part Number	Description
030554	Lamp/Filter Assembly, 254/280 nm, mercury.
030726	Restrictor tubing for HPLC flow cell, 0.3 mm ID x 10 ft., Teflon
090731	Outlet tubing assembly for HPLC/LC flow cell, 0.3 mm ID, Teflon
090732	Outlet tubing assembly for microbore flow cell, 0.18 mm ID, Teflon
090738	Inlet/Outlet tubing assembly for prep flow cell, 0.8 mm ID, Teflon
100719	Restrictor tubing for LC flow cell, 0.5 mm ID x 10 ft., Teflon
100731	Autozero/Event Cable.
495018	Luer fitting, female
495019	Luer fitting, male
03032710	254/280 nm replacement Mercury lamp
03041501	Analytical flow cell, 11 μ L, 10 mm path length
03043001	LC flow cell, 40 µL, 10 mm path length
03044001	2 mm path length flow cell for preparative applications, 10 μ l volume.
03044501	0.5 mm path length flow cell for preparative applications, 2.5 μ l volume.
03045106	Inlet tubing for HPLC/LC flow cell, stainless steel, 0.25 mm ID

List of Expendable Parts

Part Number	Description
03045107	Inlet tubing for microbore flow cell, stainless steel, 0.15 mm ID
03046001	5 mm path length flow cell for microbore applications, 1.3 μ l volume.
03046501	0.1 mm path length flow cell for preparative applications, 0.3 μ l volume.
49041001	Upchurch Finger-tight column 10-32 fitting
49041011	Upchurch ferrule, 1/16"
49041012	Upchurch male nut for ferrule, 1/16"
49041034	PEEK ferrule for 1.6 mm OD tubing, anti-twist (Upchurch P-250)
490410115	LT-115, PEEK SHORT NUT 1/16"
6374022611	Pomona Cable Adapter, banana plugs to binding posts.
6750054007	Fuse, 0.5 amp, 3 AG Slo-Blo for 220/240V units
6750104007	Fuse, 1 amp, 3 AG Slo-Blo for 100/115V units
7080200202	Pomona Recorder Cable.
7080200203	RECORDER CABLE TINNED LEADS
7080316105	Power cord, 115V
7080316106	Power cord, 220/240V, European plug
03045106T	Titanium inlet tubing (no fittings).
F1410050	1/4-28 couplers for outlet tube fittings

The following table lists expected pressures when using the 112 UV Detector with different flow cells at different flow rates. To obtain these pressures, water was pumped through the detector's flow cell.

Path Length (µL Volume)

mL/min	0.1 mm (0.3 μL)	0.5 mm (2.5 μL)	2 mm (10 µL)	5 mm (1.3 μL)	10 mm (11 μL)	10 mm (40 μL)
40	3	0	0	0	0	0
60	17	0	0	4	0	0
80	22	0	0	12	5	0
100	36	0	0	20	7.5	0
120	43.5	0	0	24.5	8	2
140	55	0	1	27	8.5	2
160	69	1	1	32	8.5	2
180	88	1	1.5	41.5	10	2
200	117	3	3	53	14.5	4



If you were to chart the information, it would look like this.

Β

The above resulted from connecting the detector to a pump, manometric module, and pressure gauge, shown below.

