Liquid Waveguide Capillary Cell

LPC-1 and LPC-5

Installation and Operation Manual

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Important Safety Notices

1. Do NOT use perfluorinated solvents with the LWCC because the amorphous fluoropolymer tubing is soluble in these chemicals.

2. Do NOT exceed a fluid pressure of 45 PSI.

3. LWCC will function with most liquids (one exception: perfluorinated solvents) having a refractive index >1.30.

4. Ensure that the plumbing fittings through the front panel are tight and free of leaks.

5. Minimize the injection of bubbles into the LWCC since they will cause erratic results. Continuous pumping will typically flush the bubbles through the system.

6. At the end of each experimental session, flush the system with solvent and then pump dry. Avoid leaving fluid in the LWCC for extended periods of time. Ocean Optics' Waveguide Cleaning Kit (LPC-CLEANKIT) is recommended for cleaning the LWCC between uses and sample runs.

7. Opening the chassis invalidates the warranty. Components inside are very fragile and are not user-serviceable. Contact Ocean Optics immediately if you have trouble with the instrument.

8. **WARNING: THIS EQUIPMENT IS NOT DESIGNED OR INTENDED FOR USE ON HUMANS.**

Warranty

WPI (World Precision Instruments, Inc.) warrants to the original purchaser that this equipment, including its components and parts, shall be free from defects in ma te ri al and workmanship for a period of one year* from the date of receipt. WPI's obligation under this warranty shall be limited to repair or replacement, at WPI's option, of the equipment or defective components or parts upon receipt thereof f.o.b. WPI, Sarasota, Florida U.S.A. Return of a repaired instrument shall be f.o.b. Sarasota.

The above warranty is contingent upon normal usage and does not cover products which have been modified without WPI's approval or which have been subjected to unusual physical or electrical stress or on which the original identification marks have been removed or altered. The above warranty will not apply if adjustment, repair or parts replacement is required because of accident, neglect, misuse, failure of electric power, air conditioning, humidity control, or causes other than normal and ordinary usage.

To the extent that any of its equipment is furnished by a manufacturer other than WPI, the foregoing warranty shall be applicable only to the extent of the warranty furnished by such other manufacturer. This warranty will not apply to appearance terms, such as knobs, handles, dials or the like.

WPI makes no warranty of any kind, express or implied or statutory, including without limitation any warranties of merchant ability and/or fitness for a particular purpose. WPI shall not be liable for any damages, whether direct, indirect, special or consequential arising from a failure of this product to operate in the manner desired by the user. WPI shall not be liable for any damage to data or property that may be caused directly or indirectly by use of this product.

Electrodes, batteries and other consumable parts are warranted for 30 days only from the date on which the customer receives these items.

Claims and Returns

Inspect all shipments upon receipt. Missing cartons or obvious damage to cartons should be noted on the delivery receipt before signing. Concealed loss or damage should be reported at once to the carrier and an inspection requested. All claims for shortage or damage must be made within 10 days after receipt of shipment. Claims for lost shipments must be made within 30 days of invoice or other notification of shipment. Please save damaged or pilfered cartons until claim settles. In some instances, photographic documentation may be required. Some items are time sensitive; WPI assumes no extended warranty or any liability for use beyond the date specified on the container.

- WPI cannot be held responsible for items damaged in shipment en route to us. Please enclose merchandise in its original shipping container to avoid damage from handling. We recommend that you insure merchandise when shipping. The customer is responsible for paying shipping expenses including adequate insurance on all items returned.
- Do not return any goods to WPI without obtaining prior approval and instructions (RMA#) from our returns department. Goods returned unauthorized or by collect freight may be refused. The RMA# must be clearly displayed on the outside of the box, or the package will not be accepted. Please contact the RMA department for a request form.
- Goods returned for repair must be reasonably clean and free of hazardous materials.
- A handling fee is charged for goods returned for exchange or credit. This fee may add up to 25% of the sale price depending on the condition of the item. Goods ordered in error are also subject to the handling fee.
- Equipment built as a special order cannot be returned.
- Always refer to the RMA# when contacting WPI to obtain a status of your returned item.
- For any other issues regarding a claim or return, please contact the RMA department:

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Table of Contents

About This Manual

Document Purpose and Intended Audience

This document provides the LWCC user with instructions for setting up, running and maintaining the LWCC device.

Document Summary

Chapter	Description
Chapter 1: *Introduction*	Contains descriptive information about the LWCC and its properties.
Chapter 2: *Setup*	Contains a list of package contents and unpacking instructions. Also provides instructions for making fiber and plumbing connections.
Chapter 3: *Operation*	Contains instructions for measuring a continuous flow and discrete samples with the LWCC.
Chapter 4: *Maintenance*	Provides tips for avoiding contamination, as well as cleaning instructions and storage recommendations. Also contains a troubleshooting table.
Appendix A: *Specifications*	Provides technical specifications for the LWCC.

Product-Related Documentation

You can access documentation for Ocean Optics products by visiting our website at **http://www.oceanoptics.com**. Select *Technical* → *Operating Instructions*, then choose the appropriate document from the available drop-down lists. Or, use the **Search by Model Number** field at the bottom of the web page.

Engineering-level documentation is located on our website at *Technical* → *Engineering Docs.*

Upgrades

Occasionally, you may find that you need Ocean Optics to make a change or an upgrade to your system. To facilitate these changes, you must first contact Customer Support and obtain a Return Merchandise Authorization (RMA) number. Please contact Ocean Optics for specific instructions when returning a product.

Chapter 1

Introduction

Overview

The Liquid Waveguide Capillary Cell (LWCC) from Ocean Optics features optical sample cells that combine an increased optical pathlength (2–500 cm) with small sample volumes (5–1250 µL) with fiber optic capabilities. The LWCC connects with optical fibers to Ocean Optics' high-sensitivity fiber optic spectrometers and compact light sources via SMA terminations for simple, efficient measurement of low-volume or low-concentration (ppb-ppt) aqueous samples. The LWCC functions with most liquids (with the exception of perfluorinated solvents) having a refractive index $\geq$1.30.

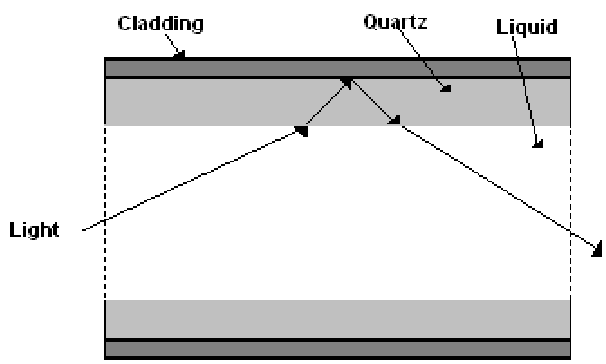
Similar to optical fibers, light is confined within the (liquid) core of an LWCC by total internal reflection at the core/wall interface. Ultra-sensitive absorbance measurements can be performed in the ultraviolet (UV), visible (VIS) and near-infrared (NIR) ranges to detect low sample concentrations in a laboratory or process control environment. According to Beer's Law, the absorbance signal is proportional to chemical concentration and light path length. Compared with a standard 1-cm cell, a 1-mAU signal is enhanced fifty-fold with a 50-cm cell to 50 mAU using the LWCC's patented aqueous waveguide technology.

The LWCC is available in 1-meter (LPC-1) and 5-meter (LPC-5) pathlengths for absorbance measurements.

LWCC Properties

Similar to optical fibers, light is confined within the (liquid) core of an LWCC by total internal reflection at the core/wall interface. Optical fibers are then used to transport light to and from the sensor cell. Designed for use with fiber optics, both LWCCs require only small sample volumes and have a high optical throughput.

The LWCC is made from fused silica tubing with an outer coating of a low refractive index polymer. The core liquid is contained by the synthetic silica tube coated with the low refractive index cladding material. Placing the refractive surface outside the silica protects it from undesirable effects of the liquid. In addition, the fused silica wall is impermeable to gases.



Lateral Section of LWCC

Effective Pathlength and Linearity

Effective pathlength and linearity have been extensively studied with the LWCC. Effective pathlength is defined as the equivalent pathlength of the cell, assuming the LWCC strictly follows Beer's law. Although there have been several reports in the literature in which calculation of effective pathlength has been performed, the theoretical basis by which to calculate the effective pathlength of the LWCC has not yet been established. It is, therefore, currently determined experimentally. The effective optical pathlength was determined to be slightly shorter than the physical pathlength (0.94± 0.01 times of its physical pathlength), dependent on the LWCC's inner diameter and wall thickness. This is caused by the fact that light is partially traveling in the fused silica wall of the LWCC. By Beer's Law, the absorption of a liquid sample in the LWCC bears a linear relationship to the concentration of an analyte. The LWCCs were extensively tested and proved to be linear over a range of 0.01 to 2.0 AU (limited only by noise and stray light from the measuring spectrophotometer). A detailed analysis of the effective pathlength and linearity of the LWCCs has been published (Belz *et al.,* 1999).*

* Mathias Belz, Peter Dress, Aleksandr Sukhitskiy and Suyi Liu, "Linearity and effective optical pathlength of liquid waveguide capillary cells," Part of the SPIE Conference on Internal Standardization and Calibration Architectures for Chemical Sensors, Boston, Massachusetts, September 1999, SPIE Vol. 3856, 271–281.

Pressure and Flow Rate

The applied pressure and fluid flow rate through the LWCC obeys the Hagen-Poiseuille relationship. Flow is proportional to pressure and to the fourth power of the diameter of the fluid capillary, as well as reciprocal to the length of the capillary and fluid viscosity. A one-meter length of 550 µm ID waveguide requires approximately 1.5 PSI for water flow of 1 mL/min.

Mechanical Properties

Maximum hydrostatic pressure that the LWCC can withstand has not yet been determined. It has been operated at 100 to 200 PSI without observed malfunction (a silica capillary with a similar structure has been reported to withstand pressures of at least 2000 PSI).

Chapter 2

Setup

Overview

Setting up your LWCC involves unpacking the device and making fiber and plumbing connections.

Unpacking the LWCC

Upon receipt of this instrument, make a thorough inspection of the contents and check for possible damage. Note any missing cartons or obvious damage to cartons on the delivery receipt before signing. Report concealed loss or damage at once to the carrier and request an inspection (see *Claims and Returns* for information on returns). Call Ocean Optics if any parts are missing.

Contents

Your LWCC package should contain the following:

- LWCC unit
- Luer-fitted PEEK connectors with caps (package of 2)
- Quality Control document – Contains the effective optical pathlength of the cell for this LWCC unit

Other Accessories Not Provided

The following accessories for your LWCC are required, but must be ordered separately:

- Fiber optic cables (2)
- Detection system including either a spectrophotometer, or a spectrometer and a light source

In addition, an optional Waveguide Cleaning Kit (LPC-CLEANKIT) is available from Ocean Optics. This kit is recommended for cleaning the LWCC between uses and sample runs.

Visit our website at **http://www.oceanoptics.com** for a complete list of Ocean Optics products.

Connections

The following connections must be made to use your LWCC:

- Plumbing (Liquid Port) Connections
- Fiber Connections

Plumbing Connections

On the front panel, there are two plumbing feed-through (liquid) ports. It does not matter which one is used for the plumbing input or the plumbing output. However, for experimental consistency, once you have assigned which fitting will be the plumbing input and which one will be the plumbing output, try not to switch the plumbing configuration. The tubing goes over the plumbing fittings, which are standard ¼-28 threads. The tubing should fit snugly over the fittings and be free of leaks.

► *Procedure*

1. Attach one end of the tubing to your pump. The pump used must not pump the solution so fast that the fluid pressure exceeds 45 PSI.

Note

Remember to always turn off the pump in-between taking a reference and taking sample measurements.

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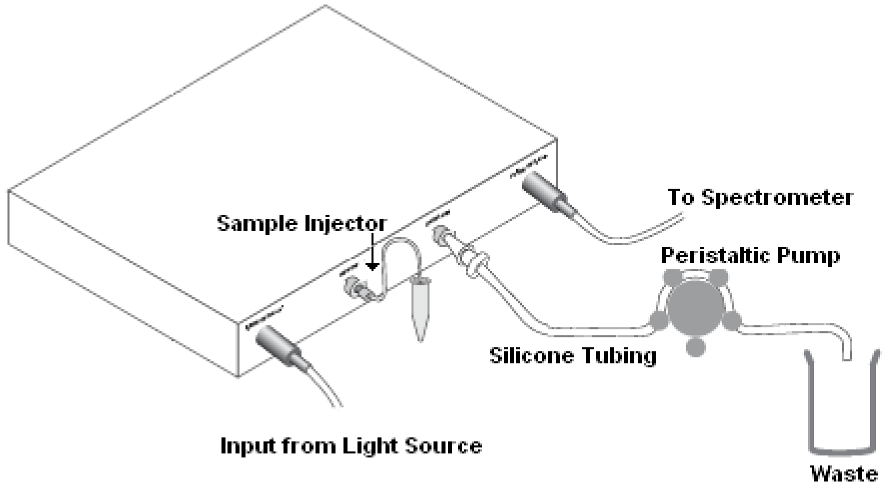
2. Make sure you have a proper waste receptacle for the other end of the tubing.



Example LWCC Experimental Setup

Tightening the Plumbing Connections

The plumbing connections inside the LWCC are standard industry fittings. No maintenance is required. However, if leaks develop, the plumbing connections need to be tightened.

► ***Procedure***

To tighten the plumbing connections,

1. Remove the back panel.
2. Carefully slide off the top cover, being careful not to damage or pinch the tubing or fiber.
3. Hand-tighten the fittings and reassemble the cover and back panel.

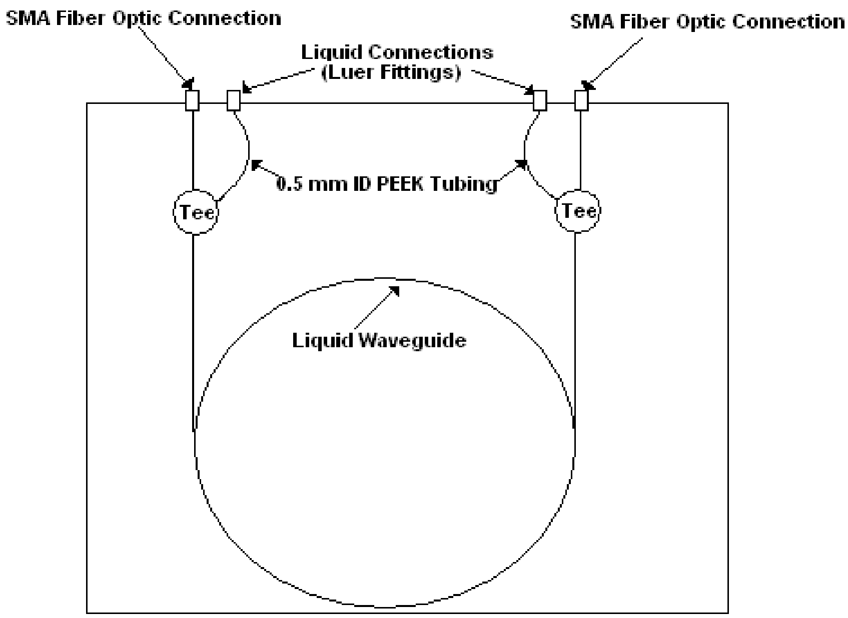
Fiber Connections

Caution

Unlike electric cables, fiber optic cables are fragile as they contain glass and are subject to breakage. Avoid sharp bends in the cables and protect them from impact or permanent damage can result.

The light source and detector connect to the LWCC via two SMA-terminated fiber optic patch assemblies with a core diameter of 400 µm. For convenience only, each LWCC has the fiber optic connections marked as **FIBER OPTIC "A"** and **FIBER OPTIC "B"**. The fiber optic connections are interchangeable in that either connector can be used to connect to the light source or to the spectrometer. However, for experimental consistency, once you have assigned the fiber ports try not to switch the fiber configuration. You must then connect the LWCC to a light source and spectrometer (detector) module of your spectrophotometer system.

The fiber inside the LWCC has a core diameter of 200 µm. External coupling fibers should be 200 µm or larger for maximum coupling efficiency.



► *Procedure*

To connect the optical fibers,

1. Attach one end of an illumination fiber to a port on the LWCC.
2. Attach the other end of the fiber to your light source.
3. Attach one end of a read fiber to the second port on the LWCC.
4. Attach the other end of the fiber to your spectrometer.

Assessing if the LWCC is Free of Particles

Fluids need to be relatively particle-free. Particles larger than 20 μm can be trapped inside the tubing and can then block or scatter a significant amount of light.

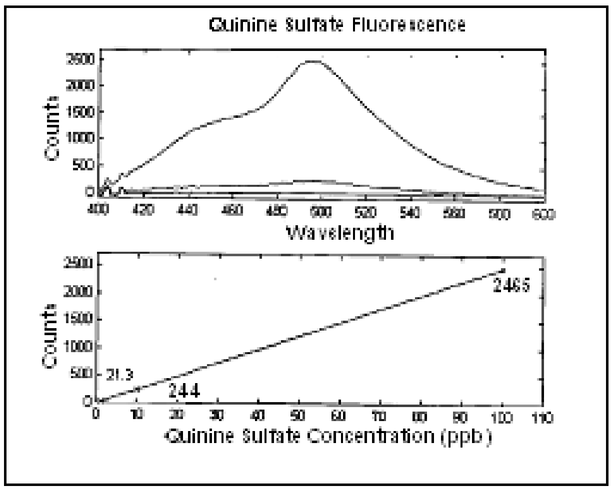
► *Procedure*

To rid the LWCC of particles, follow these steps:

1. Pump the sample fluid through the LWCC.
2. While in Scope Mode, save a dark spectrum with the light source off and a reference spectrum with the light source on.

3. Continue to pump the sample fluid and switch to the Absorbance Mode. Ideally, you should see a spectrally flat line (see *Example Spectra* below). Particle effects manifest themselves as an exponentially decreasing curve from shorter to longer wavelengths. The length of time that you pump the sample and the magnitude of the absorbance peak depend upon the time required and the minimum detectable absorbance value for your specific analyses. Prefiltering of the sample may be required to eliminate this exponentially decreasing absorbance spectrum if it is significant to your analyses.

The following sample fluorescence spectra of quinine sulfate solution were obtained with an S2000 Fiber Optic Spectrometer (100 µm slit, L2 Detector Collection Lens) and 1-meter LWCC with built-in 365-nm excitation source. The integration period was 500 milliseconds. Intensity was linear with concentration. The lowest detectable concentration of the sample was ~0.5 ppb.



Example Spectra

Chapter 3

Operation

Overview

Using your LWCC system you can measure liquids

- In a continuous flow, or
- Using discrete samples

Caution

Materials exposed to fluid in the LWCC are CTFE, PEEK and fused silica. Any chemical that could harm these substances should not be used in the LWCC. For example, hydrofluoride (HF) dissolves silica, and PEEK can be damaged by concentrated sulfuric and nitric acids (40% w/w or greater).

Keeping the LWCC clean is essential for a stable result. See *Cleaning* in Chapter 4: *Maintenance*.

Measuring in a Continuous Flow

► ***Procedure***

1. Connect the LWCC to a light source and a detector with fiber optic cables.
2. Clean the LWCC using the standard cleaning procedure described in Chapter 4: *Maintenance*.
3. Connect the liquid source to the LWCC system. The standard Luer fitting at the LWCC input and output can be replaced with 1/16-inch (1.64 mm) tubing if necessary. A pressure of approx. 1.5-3.0 PSI is necessary to run liquid through the LWCC.
4. Flush the LWCC with de-ionized water or experimental buffer solution using a pump or a syringe and observe the light intensity or absorbance baseline on the detector. Continue flushing until the signal is stable. (See *Troubleshooting* in Chapter 4: *Maintenance* if the signal does not stabilize appropriately.)

Measuring Discrete Samples

Discrete samples can be measured with the LWCC by introducing the sample with a syringe. Sample volumes approximately 1.5 – 3 times the cell volume are necessary to fill the LWCC.

► *Procedure*

1. Using fiber optic cables, connect the LWCC to a light source and a detector.
2. Clean the LWCC using the standard cleaning procedure described in Chapter 4: *Maintenance*.
3. Flush the LWCC with de-ionized water or experimental buffer solution using a syringe and observe the light intensity or absorbance baseline on the detector until the signal is stable.
4. Introduce the sample *slowly* into the LWCC with a syringe using steady pressure to avoid generating air bubbles.

Chapter 4

Maintenance

Overview

Thorough and consistent cleaning routines are essential for maintaining the instrument and ensuring optimal operation.

Tips to Avoid Contamination

The following information about contamination has been collected during the development and testing of the LWCC:

- Most syringe filters contain some contaminants that absorb UV (probably the plastic mold release agent). The first few milliliters of solution coming from a new filter will have some absorption in the UV range due to the mold release agent.
- The first two loads of solution from most new plastic syringes often have some contamination that absorbs UV. In addition, when plastic syringes are used to transfer organic solvents, the rubbery gasket material in the plunger absorbs some of the chemical. If the syringe is later used to transfer aqueous solution, the chemical will slowly leach out. Since most organic solvents have an absorbance in the UV range, the liquid initially released from the syringe might be found to have a different spectrum than the last of the liquid in the syringe, the latter having been contaminated by the chemical in the plunger. Some commonly used organic solvents with relatively low UV absorption that are suitable for UV detection in conventional spectrometers might not be problem-free when used in the LWCC.
- A beaker of freshly filtered water sitting overnight in open air will probably have an increased absorbance in the UV range because of dust from the air or growth of microorganisms.
- Some plastic tubing release a substance that absorbs UV. In lab tests, silicone tubing used in a peristaltic pump constantly released a contaminant even after a week of washing.
- A bubble in the LWCC will result in unstable readings. Additional liquid circulating through the device will usually push the bubble out. If the bubble doesn’t clear easily, try introducing a larger bubble followed by liquid. This will usually pick up a small bubble that may cling and cause problems.

- Avoid introducing particulate matter into the LWCC. If they are trapped in the LWCC, particles can scatter light and may cause unstable spectrometer readings. The LWCC contains two potential bottlenecks at the fiber-capillary interface. For the 550 µm ID waveguide, the bottlenecks are 75 µm-wide ring gaps. If a particle larger than 60 µm is forced into the waveguide and trapped there, it can take a lot of effort to remove it. Due to the diverse applications of LWCC, no in-line filter can be installed which will fit all users' needs. It is imperative, therefore, that a proper in-line filter be added to the LWCC if the solution contains large particles. When the LWCC is directly connected to a chromatography column, a filter might not be necessary.

Cleaning

Before and after each usage of the LWCC, it is recommended that cleaning be performed. A Waveguide Cleaning Kit made especially for the LWCC is available from Ocean Optics (LPC-CLEANKIT).

► *Procedure*

To clean your LWCC, use the following procedure:

1. Connect exit tubing (silicon or equivalent) from the OUTFLOW port of the LWCC to a waste container.
2. Rinse the cell thoroughly using Ultra Pure water.
3. Obtain a new reference intensity and take a baseline absorbance reading.
4. Fill a 1 cc syringe with “Cleaning Solution 1” and inject it into the LWCC’s INFLOW port via the Luer fitting adapters provided with your LWCC.
5. Fill a 1 cc glass-type syringe with “Methanol Solution 2” and inject it into the INFLOW port of the LWCC.
6. Fill a 1 cc syringe with “HCl Solution 3” and inject it into the INFLOW port of the LWCC.
7. Flush out cleaning solutions with distilled Ultra Pure, reverse osmosis, or equivalent quality water and take an absorbance reading.
8. Repeat these cleaning cycles until a stable absorbance signal can be obtained.

Instrument Storage

Caution

Do not partially dry the LWCC and leave it open to the air. Oxygen in the air may facilitate the growth of microorganisms inside the device.

To store the instrument, clean the LWCC and then fill it with an 80/20 solution of distilled water/methyl alcohol. Seal the INFLOW and OUTFLOW ports using either the caps provided or an alternative.

Troubleshooting

The LWCC is a highly sensitive device, and it is extremely important to keep it clean. This is especially important when working in the ultraviolet range, where unexpected results can often be produced by contamination of the experimental solution. The high sensitivity of the LWCC may create some problems that can be easily overcome with care and forethought. Therefore, you may need to develop new skills in handling both the equipment and the samples being examined.

Typical Contamination Effect	Possible Cause(s)	Suggested Solution(s)
Transmission in both UV and visible ranges becomes low or very unstable.	A contamination layer (such as biofilm) is sticking to the LWCC wall. Or, a particle is trapped in the LWCC.	Flush the cell for 30 seconds each with each of the 3 cleaning solvents contained in the Waveguide Cleaning Kit. OR Prepare a 5% surfectant using Ultrasonic cleaning solution, followed by HPLC-grade Methanol and HPLC-grade 2N HCl solution.
Transmission in the UV range is low. Transmission in the visible range is OK and stable.	1. Optic fiber and silica tubing are coated by a layer of metal corrosion. 2. Optic fiber and silica tubing are coated by a layer of organics.	1. Flush with 1 N HCl. 2. Flush with an organic solvent, such as acetonitrile.
Transmission below 250 nm is low. Transmission in the visible range is OK and stable.	Contamination of fiber optic cable end-faces with a metal film generated during repeated connection attempts.	Wipe all fiber optic endfaces and fiber connections (on front of the LWCC) using a fiber optic foam swab dipped in methyl alcohol. Use of cotton swabs (Q-Tips) is *not* recommended.

Appendix A

Specifications

Specification	Value
Waveguide material	Fused silica tubing coated with a low refractive index polymer
Optical pathlength	2–500 cm
Inner diameter	550 µm
Internal volume	~5–1250 µL
Sample inlet/outlet compression fitting	1/16 in., 1/32 in.
Fiber core diameter	400 µm
Maximum temperature	160ºC
Gas permeability of cell	None
Minimum pressures	1.5–3 PSI
Maximum pressure	2000 PSI
Solvent resistance	Most organic and inorganic solvents
Shipping weight	3 lbs. (1.36 kg)
Path lengths	1-meter, 5-meter (standard); custom lengths also available
Tubing	Teflon® Amorphous Fluoropolymer 2400 ~560 µm inner diameter, ~800 µm outer diameter
Refractive index	1.29
Internal volume	250 µl/meter

Specification	Value
Chemical resistance	Tubing can be altered by perfluorinated solvents, FREON® 113, and Perclene®
Recommended optical fibers for coupling to spectrometers and light sources	400 µm illumination fiber (UV-VIS) 200 µm or 400 µm read fiber (UV-VIS)
Plumbing fittings	Standard ¼ in. x 28 chromatography fittings

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