



Liquid Waveguide Capillary Cell

For measurement of very low concentration samples

www.wpiinc.com

INSTRUCTION MANUAL

Serial No. _____

040208

World Precision Instruments, Inc.



General Warnings and Cautions

CAUTION: Opening the chassis will invalidate the warranty. Components inside are very fragile and are not user-serviceable. Contact WPI's technical department immediately if you have trouble with the instrument.

NOTE: Fluid pressure fluctuation, cross-contamination and the introduction of small air bubbles can cause baseline variations and measurement inaccuracies. Use the optional **Sample Injector Kit** (WPI #58006) to minimize the development of small air bubbles.

NOTE: WPI's **Waveguide Cleaning Kit** (WPI #501609) is recommended for cleaning the LWCC between uses and sample runs.



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Introduction

Liquid Waveguide Capillary Cells (LWCC) are optical sample cells that combine an increased optical pathlength (2-500 cm) with small sample volumes (5-1250 μ L). They can be connected via optical fibers to a spectrophotometer with fiber optic capabilities. 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) 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 WPI's patented aqueous waveguide technology. The LWCC can be connected directly to a pump, a chromatography column, a Sample Injector (WPI #58006), or can even be filled using a syringe. The LWCC is designed for use with WPI's **TIDAS II** spectrophotometer system. Further, modular sample systems can be assembled using WPI's PDA (**TIDAS I**) or CCD (**SPECTRAUSB**) based spectrometer modules and WPI's range of UV/VIS light sources **D₂H™**, **D₂Lite™**, and **FO-6000**.



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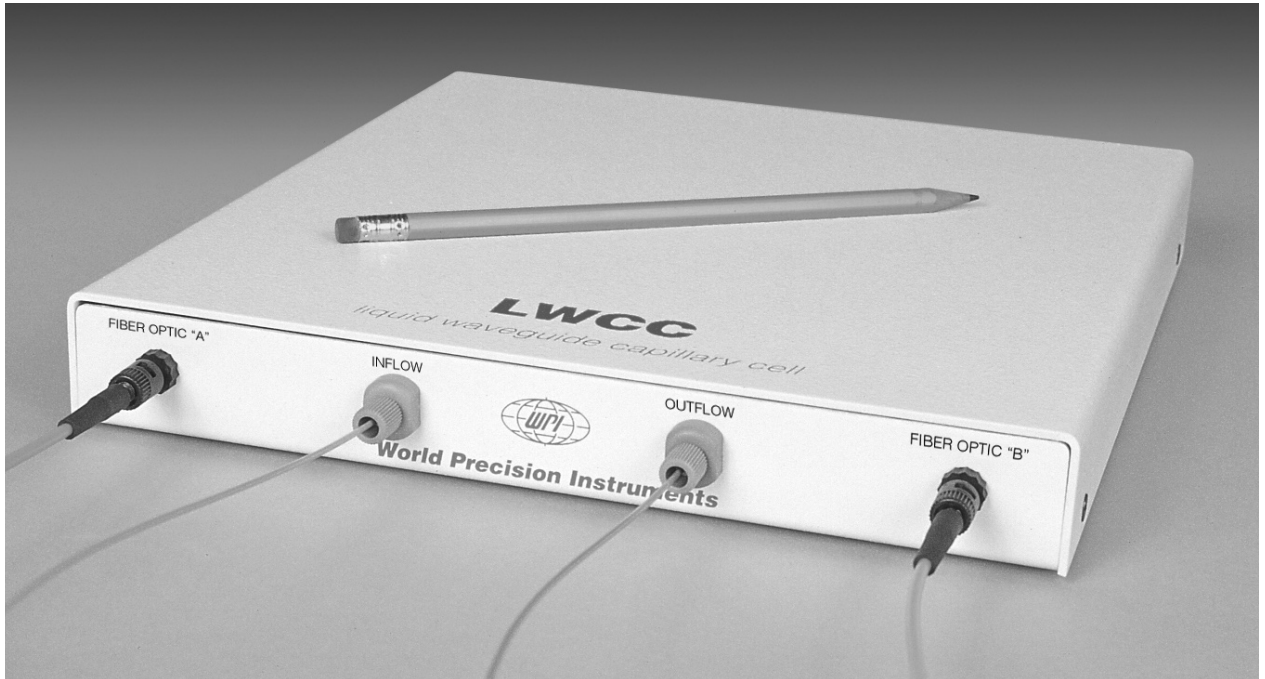


Fig. 1 – LWCC available with 2, 5, 10, 50, 100, 200 and 500 cm pathlength.

NOTE: The effective optical pathlength of the cell is given in the Quality Control Document provided with each instrument.

Set-up Instructions

Parts List

- LWCC
- Luer-fitted PEEK connectors with caps (package of 2)
- LWCC Instruction Manual
- Quality Control Document

Unpacking

Upon receipt of this instrument, make a thorough inspection of the contents and check for possible damage. 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. Please read the section entitled “Claims and Returns” on the Warranty page of this manual. Please call WPI Technical Support if any parts are missing.



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Returns: Do not return any goods to WPI without obtaining prior approval and instructions from our Service Department. Goods returned (unauthorized) by collect freight may be refused. If a return shipment is necessary, use the original container. If the original container is not available, use a suitable substitute that is rigid and of adequate size. Wrap the instrument in paper or plastic surrounded with at least 100 mm (four inches) of shock absorbing material. Please read the section entitled "Claims and Returns" on the Warranty page of this manual.

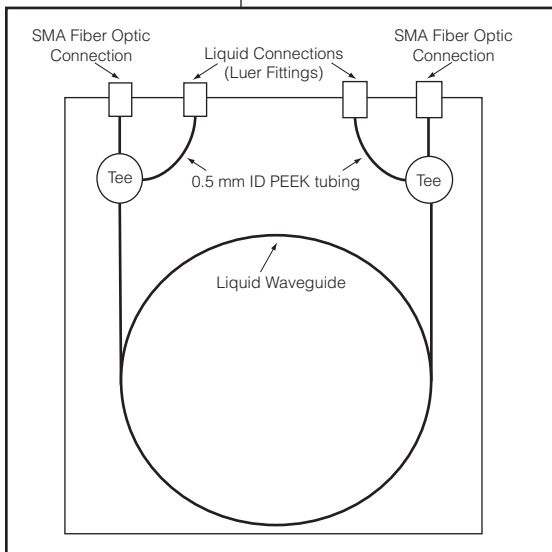


Fig. 2

Required But Not Provided (see Accessories)

- Fiber Optic Cables (2)
- Detection System including either a spectrophotometer or a spectrometer and light source

Assembly

Caution: Unlike electrical 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 may result.

External Fiber Optic Cable Connections

LWCC	FIBER OPTIC CABLE
Fig. 2 2, 5, 10, 50, 100, 200 cm Type II	SMA terminated <i>e.g.</i> , WPI # FO-400-SMA1M

Light source and detector can be connected 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" (Fig. 1). The fiber optic connections are interchangeable in that either connector can be used to connect to the light source or to the spectrometer.

Connect the LWCC to a light source and spectrometer (detector) module of your spectrophotometer system.



Liquid Ports

As with the fiber optic connectors, the liquid ports are also interchangeable, *i.e.*, it makes no difference which port is set-up as inflow and which port serves as the outflow. However, if an in-line filter is to be installed, it should be connected on the input side of the liquid flow path. For convenience only, each LWCC has the ports identified as “INFLOW” and “OUTFLOW” (Fig. 1).

Install the Luer-fitted connectors to the ports. These connectors may be removed if the LWCC is to be connected to a fluid injection analysis system or a liquid chromatography column.

Luer fittings may be replaced with other $\frac{1}{16}$ -inch (1.64 mm) OD tubing; loosen the small nut at the base of the luer fitting, remove the fitting, insert new tubing and tighten the nut. If a Sample Injector Kit is to be used, it will take the place of the inflow connector.

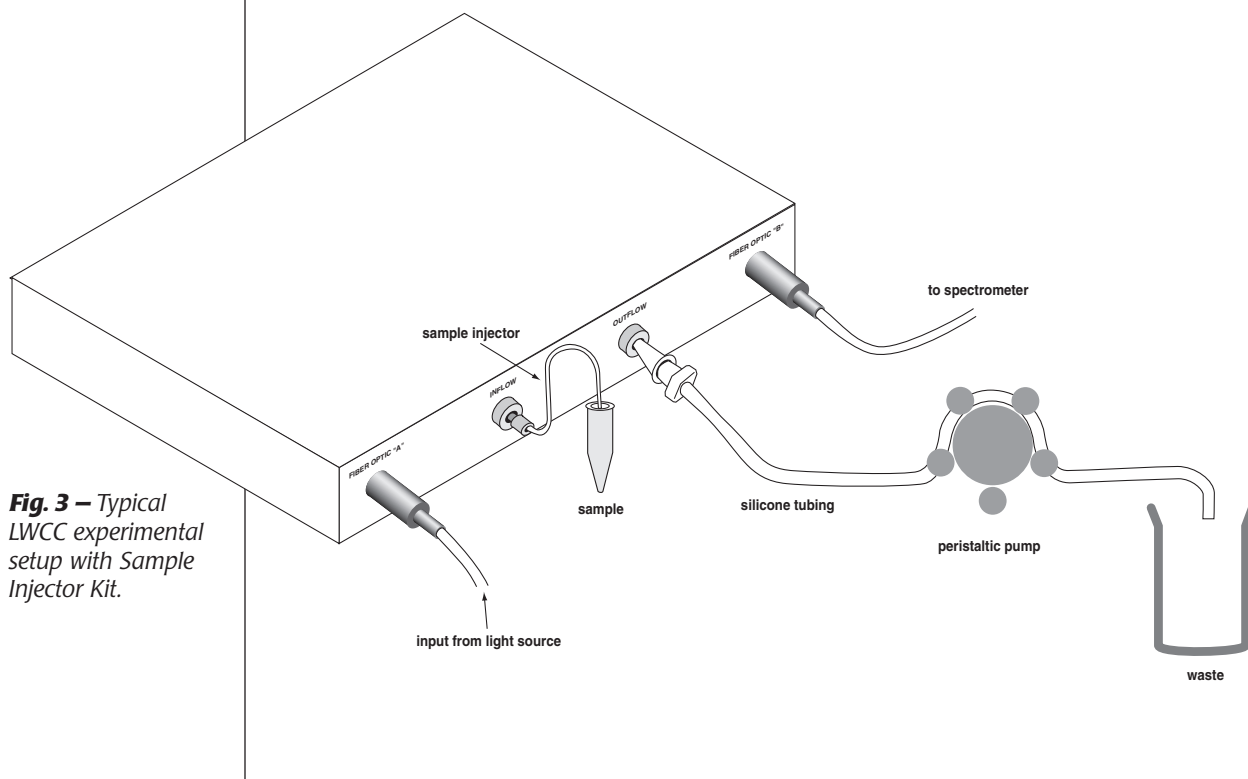


Fig. 3 – Typical LWCC experimental setup with Sample Injector Kit.



Operating Instructions

CAUTION: Materials exposed to fluid in the LWCC are CTFE, PEEK and fused silica. Any chemical that could attack these substances should not be used in the LWCC. For example, hydrofluoride (HF) will dissolve silica and PEEK will be damaged by concentrated sulfuric and nitric acids (40% w/w or greater). Type I waveguides will also be damaged by perfluorinated solvents. (See Appendix A.)

CAUTION: Keeping the LWCC clean is essential for a stable result. See the Instrument Maintenance Section.

Measuring in a Continuous Flow

- Connect the LWCC to a light source and a detector with fiber optic cables.
- Clean the LWCC using the standard cleaning procedure described in the Instrument Maintenance Section.
- Connect the liquid source to the LWCC system. The standard Luer fitting at the LWCC input and output can be replaced with $\frac{1}{16}$ -inch (1.64 mm) tubing if necessary. A pressure of approx. 1.5 - 3.0 PSI will be necessary to run liquid through the LWCC.
- 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 the Troubleshooting Section 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 approx. 1.5 - 3 times the cell volume are necessary to fill the LWCC.

- Using fiber optic cables, connect the LWCC to a light source and a detector.
- Clean the LWCC using the standard cleaning procedure described in the Instrument Maintenance Section.



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- 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.
- Introduce the sample *slowly* into the LWCC with a syringe using steady pressure to avoid generating air bubbles.

Optional Sample Injector Kit

The use of the Sample Injector Kit, in conjunction with a pump connected to the outflow tubing of the LWCC, permits the continual draw of sample into and through the LWCC at a constant flow rate and pressure. The laminar flow of liquid eliminates the production of air bubbles often associated with the turbulent liquid flow caused by variations in syringe pressure. The replacement of the Luer fitted PEEK connector with the PEEK nut and ferrule fitting on the Inflow port, greatly minimizes the area or “dead space” in which potential sample mixing can occur. In addition, the hydrophobic nature of the Teflon PFA tubing used for the Sample Injector Assembly minimizes sample retention and the possibility of cross-contamination when moving the tubing between one sample and another. Syringes used for sample injection are often a source of air bubbles despite repeated attempts to purge the syringe of air. The Sample Injector Assembly provides a bubble-free uptake into the LWCC due

to the nature of the Teflon PFA tubing and the constant uptake pressure from the pump.

Fig. 4 – Sample Injector Kit





Instrument Maintenance

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

Cleaning

Cleaning of the LWCC will depend on the type of contamination. The following methods have been found effective.

- **Standard Cleaning Procedure** – Before and after each usage of the LWCC, it is recommended that cleaning be performed. Use cleaning kit for liquid waveguides (WPI Part #501609).

Follow these steps:

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

The sample injector assembly kit (WPI Part #58006) can be used to conveniently fill all LWCC models, unlike syringe type injection methods described above. Further, using the sample injector, the injection volume of the cleaning solutions per cycle can be reduced to the internal volume of the LWCC (*e.g.*, 0.26-mL for a 100 cm LWCC).



Instrument Storage

CAUTION: Do not leave the LWCC “half dried” and 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 may 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 – the user may need to develop new skills in handling both the equipment and the samples being examined.

Typical Contamination Effects

Phenomena: Transmission in both UV and visible ranges becomes low or very unstable

Possible cause: A contamination layer (such as biofilm) sticking to the LWCC wall; or a particle trapped in the LWCC.

Solution 1a: Flush cell for 30 seconds each of each of the three cleaning solvents in the waveguide cleaning kit (WPI Part #501609).

Solution 1b: Prepare a 5% Surfactant using Ultrasonic cleaning solution concentrate (WPI Part #15807), followed by HPLC grade Methanol and HPLC grade 2N HCl solution.

Phenomena: Transmission in UV is low. Visible range is OK and stable.

Possible cause: Optic fiber and silica tubing are coated by a layer of metal corrosion.

Solution: Flush with 1 N HCl

Possible cause: Optic fiber and silica tubing is coated by a layer of organics.

Solution: Flush with an organic solvent, such as acetonitrile



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Phenomena: Transmission below 250 nm is low. Visible range is OK and stable

Possible cause: Contamination of fiber optic cable end-faces with a metal film generated during repeated connection attempts.

Solution: Wipe all fiber optic end-faces, fiber connections (on the LWCC box front) using a fiber optic foam swab (WPI part #800490) dipped in methyl alcohol. Note: Use of cotton swabs (Q-Tips) not recommended.

Additional Information on Contamination

The following notes regarding contamination have been collated at WPI during the development and testing of the LWCC:

1. 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.
2. 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 last of the liquid in the syringe, the latter having been contaminated by the chemical in the plunger. Some commonly used organic solvents which have “relatively low” UV absorption and are suitable for UV detection in conventional spectrometers might not be problem-free when used in LWCC.
4. 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.
5. Some plastic tubing will release a substance that absorbs UV. In WPI’s lab, silicone tubing used in a peristaltic pump constantly released a contaminant even after a week of washing.



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6. 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.

7. Avoid introducing particulate into the LWCC. If 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 the 60 μm is forced into the waveguide and trapped there, it might 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 a chromatography column, a filter might not be necessary.



Accessories

WPI Part No.	Item
KITLWCC**	LWCC sampling kit
FO-400AS-SMA	1M, SMA/400 um core, anti-solarization
FO-400-SMA1M	1M, SMA/400 um core, UV-enhanced
FO-400SMA/ST*	SMA/ST terminated, 400 um core, UV-enhanced
15807	Cleaning solution concentrate–100g (Solution 1)
58006	Sample Injector Kit
58450	Spare set of external syringe adapters
501609	Waveguide cleaning kit
500291	Peri-Star™ peristaltic pump-110V US CE
500304	Peri-Star™ peristaltic pump-220V Euro CE
500320	Silicone tubing for Peri-Star pump. 1.6mm ID x 1.6W
800490	Fiber optic foam swabs (5)
TIDAS I	Tidas I high performance photodiode array spectrometer module
TIDAS-BASIC	Tidas II high performance fiber optic based photodiode array Spectrophotometer
SPECTRAUSB	Miniature fiber optic spectrometer module, UV-VIS range
SPECTRAUSB-DBL	Dual channel fiber optic spectrometer module, UV-VIS range
D2LITE	D2Lite™ miniature UV-VIS light source – 200-1700nm
D2H	D2H™ deuterium/halogen light source – 215-1700nm
FO-6000	Tungsten light source – 380-1700nm

**Used on older LWCC units that have ST type front fiber optic connections.*

***Kit Includes 2 fibers (FO-400-SMA1M), cleaning kit (501609), peristaltic pump (500291), and sample injector assembly (58006).*

Appendix A: Properties of the LWCC

Type I versus Type II

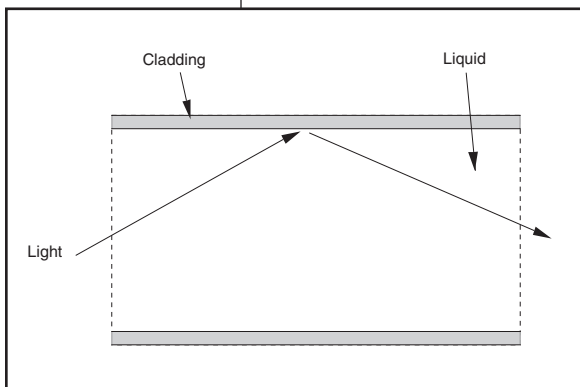


Fig. 5a – Lateral section of **Type I** Liquid Waveguide Capillary Cell (LWCC).

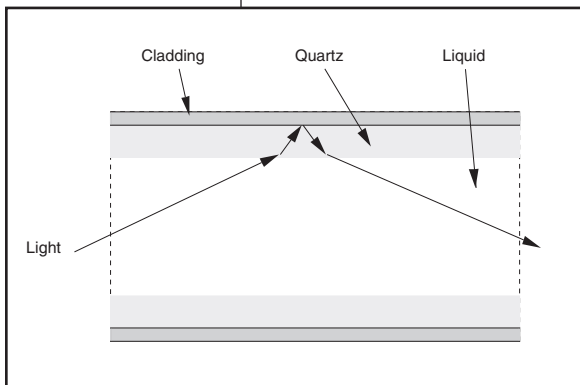


Fig. 5b – Lateral section of **Type II** Liquid Waveguide Capillary Cell (LWCC).

WPI pioneered two types of LWCCs during ten years of intensive research in this field. 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. Although both Type I and its successor Type II are suitable for most spectrometric analysis, they differ in their optical performance and ease-of-use. Type I is made of a low refractive index polymer tubing. Type II is made from fused silica tubing with an outer coating of a low refractive index polymer. The core liquid in Type II 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, Type I may require pH buffering of the sample due to CO₂ penetration through the cladding material. Baseline shifts may also be observed due to trapped organic vapor. These effects, which are only observed in Type I, are due to the nature of the low refractive index material. This effect is eliminated in Type II, since the fused silica wall is impermeable to gases. Type II also offers improved signal stability and easier removal of any air bubbles trapped at its hydrophilic cell wall.

Today, WPI offers exclusively Type II waveguides, which have been proven to be the superior cells in spectrophotometric analysis.



Effective Pathlength and Linearity

Effective pathlength and linearity have been extensively studied with Type I and Type II LWCCs. "Effective pathlength" is defined as the equivalent pathlength of the cell if we assume 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 WPI's LWCC has not yet been established. It is, therefore, currently determined experimentally. WPI has measured the effective pathlength of Type I and II LWCCs. Type I cells have an effective optical pathlength statistically indistinguishable from the physical pathlength. With Type II cells, 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 travelling in the fused silica wall of Type II LWCCs. By Beer's Law, the absorption of a liquid sample in the LWCC bears a linear relationship to the concentration of an analyte. WPI's 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 Type I and Type II LWCCs has been published (Belz *et al.*, 1999).

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).



Technical Specifications

WAVEGUIDE MATERIAL.....	Fused silica tubing coated with a low refractive index polymer
OPTICAL PATHLENGTH.....	2-500 cm
INNER DIAMETER	550 μ m
INTERNAL VOLUME.....	\approx 5-1250 μ L
SAMPLE INLET/OUTLET	
COMPRESSION FITTING	1/16", 1/32"
FIBER CORE DIAMETER.....	400 μ m
MAXIMUM TEMPERATURE	160°C
GAS PERMEABILITY OF CELL.....	None
MINIMUM PRESSURES.....	1.5 – 3 PSI
MAXIMUM PRESSURE.....	2000 PSI
SOLVENT RESISTANCE.....	Most organic & inorganic solvents
SHIPPING WEIGHT	3 lb (1.4 kg)



References

- E. D'Sa, *et al.*, "Determining optical absorption of colored dissolved organic matter in seawater with a liquid capillary waveguide", *Limnol, Oceanogr.* 44(4), 1999, 1142-1148.
- Jia-Zhong Zhang, "Shipboard automated determination of trace concentrations of nitrite and nitrate in oligotrophic water by gas-segmented continuous flow analysis using a liquid waveguide capillary flow cell", *Deep-Sea Research I*, Vol. 47, (2000), 1157-1171.
- 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.
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- Li. Song, S. Liu, V. Zhelyaskov, and M.A. El-Sayed, "Application of liquid waveguide to Raman spectroscopy in aqueous solution", *Appl. Spectrosc.*, (1998), Vol. 52, pp. 1364-1367.
- Su-Yi Liu, Ian R. Davies, "Testing drinking water with a very long path-length cell", *Nature*, May 1997, pp 9.



Related Patents

Micro Chemical Analysis Employing Flow Through Detectors, 1995, U.S. Patent No. 5,444,807.

Aqueous Fluid Core Waveguide, 1996, U.S. Patent No. 5,507,447.

Long Capillary Waveguide Raman Cell, 1997, U.S. Patent No. 5,604,587.

Chemical Sensing Techniques Employing Liquid-Core Optical Fibers, U.S. Patent No. 6,016,372



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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 material 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 merchantability 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.

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 which was 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.

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

Warning: This equipment is not designed or intended for use on humans.

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