



WORLD
PRECISION
INSTRUMENTS

INSTRUCTION MANUAL

3000 Series LWCC

Liquid Waveguide Capillary Cell

*Measure low concentration samples with UV/VIS
absorbance spectroscopy*

Serial No. _____

www.wpiinc.com

022219

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ABOUT THIS MANUAL

The following symbols are used in this guide:



This symbol indicates a **CAUTION**. Cautions warn against actions that can cause damage to equipment. Please read these carefully.



This symbol indicates a **WARNING**. Warnings alert you to actions that can cause personal injury or pose a physical threat. Please read these carefully.

NOTES and **TIPS** contain helpful information.



Fig. 1 LWCC available with 50, 100, 250 and 500cm pathlength

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

INTRODUCTION

Liquid Waveguide Capillary Cells (LWCC) are optical sample cells that combine an increased optical pathlength (50-500cm) with small sample volumes (125-1250 μ L) for ultraviolet (UV) and visible (VIS) absorbance spectroscopy. 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. Using WPI's patented aqueous waveguide technology, a 1mAU signal (from a standard 1cm cell) is enhanced 50-fold with a 50cm cell to 50mAU.

The LWCC can be connected directly to a pump, a chromatography column, a Sample Injector Kit (WPI #**58006**) or the LWCC Injection System (WPI #**89372**). The LWCC is designed for use with WPI's optical systems or **LEDspec**. Modular sample systems can be assembled using WPI's **TIDAS** spectrometer modules and a UV/VIS/NIR light source like the **D4H™** and **FO-6000**.

General Warnings and Cautions



CAUTION: Opening the chassis will invalidate the warranty. Components inside are extremely fragile and are not user-serviceable. Contact WPI's technical support department immediately if you have trouble with the instrument at 941.371.1003 or technicalsupport@wpiinc.com.

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**) or the LWCC Injection System (WPI #**89372**) 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.

INSTRUMENT DESCRIPTION

Parts List

After unpacking, verify that there is no visible damage to the instrument. Verify that all items are included:

(1) **LWCC**

(1) Luer-fitted PEEK connectors with caps (package of 2)

(1) Instruction Manual

(1) Quality Control documentation

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 damage should be reported at once to the carrier and an inspection requested. Please read the section entitled "Claims and Returns" on page i19 of this manual. Please contact WPI Customer Service if any parts are missing at 941-371-1003 or customerservice@wpiinc.com.

Returns: Do not return any goods to WPI without obtaining prior approval (RMA # required) and instructions from WPI's Returns Department. Goods returned (unauthorized) by collect freight may be refused. If a return shipment is necessary, use the original container, if possible. 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 100mm (four inches) of shock absorbing material. For further details, please read the section entitled "Claims and Returns" on page i19 of this manual.

Hardware Description

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

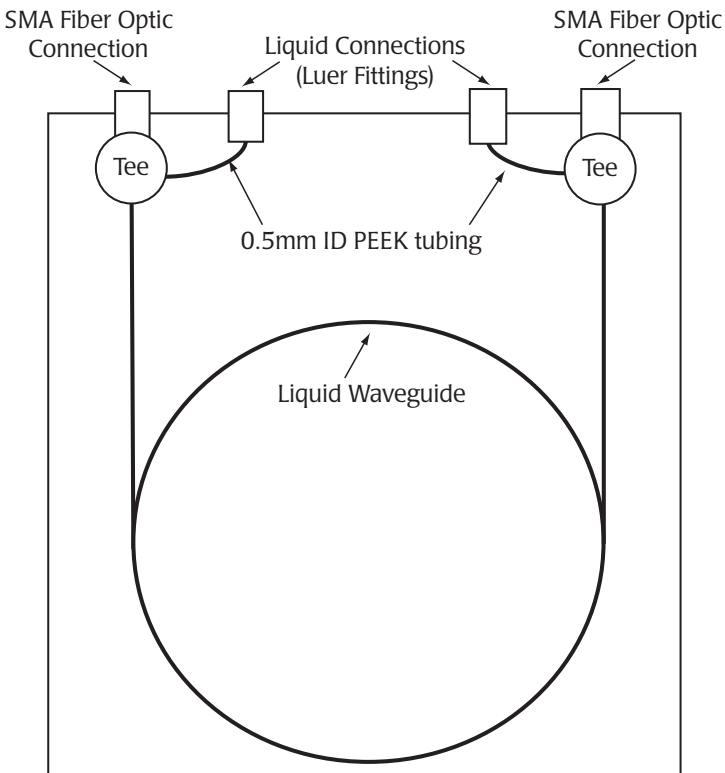


Fig. 2 LWCC Setup Schematic

LWCC

50, 100, 250, 500cm
 Type II

FIBER OPTIC CABLE

SMA 905 terminated
 for example, WPI #**FO-500-SMA1M**

The light source and detector can be connected to the LWCC via two SMA-terminated fiber optic cables with a core diameter of 500µm. For convenience only, each LWCC has the fiber optic connections marked as Light In and Light Out (**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. Use these fiber optic connections to 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. It makes no difference which port is the inflow and which port serves as the outflow. However, if an in-line filter is to be installed, connect it on the input side of the liquid flow path. For convenience only, each LWCC has the ports identified as "SAMPLE IN" and "SAMPLE OUT" (**Fig. 1**). Install the luer-fitted connectors to the ports.

NOTE: 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 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 SAMPLE IN connector.

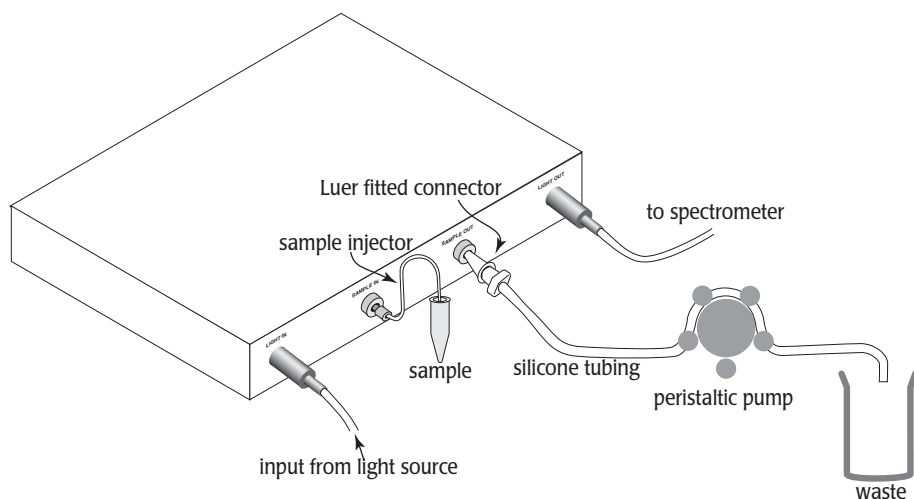


Fig. 3 Typical LWCC experimental setup with Sample Injector Kit

OPERATING INSTRUCTIONS



CAUTION: Materials exposed to fluid in the LWCC are PEEK, FEP 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). See “Appendix A: Properties of the LWCC” on page 14.



CAUTION: Keeping the LWCC clean is essential for a stable result. See “Instrument Maintenance” on page 9.

Measuring in a Continuous Flow

This is the recommended method.

1. To help prevent clogging the ports, filter all samples using a 0.2µm vacuum filtration disc before injection into the LWCC.
2. Using fiber optic cables, connect the LWCC to a light source and a detector.
3. Clean the LWCC using the standard cleaning procedure described in “Instrument Maintenance” on page 9.
4. 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) OD tubing, if necessary. A pressure of approximately 1.5–3.0 PSI is necessary to run liquid through the LWCC.
5. At a rate of 1mL/min., flush the LWCC with de-ionized water or experimental buffer solution using a pump or a syringe. Observe the light intensity or absorbance baseline on the detector. Continue flushing until the signal is stable. See “Troubleshooting” on page 11 if the signal does not stabilize.

Measuring Discrete Samples Using a Syringe

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. When injecting a sample or reference solution, apply consistent hand pressure to the syringe. Small variations in baseline levels may result from use of this method.

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 “Instrument Maintenance” on page 9.
3. Flush the LWCC with de-ionized water or experimental buffer solution using a syringe. 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.

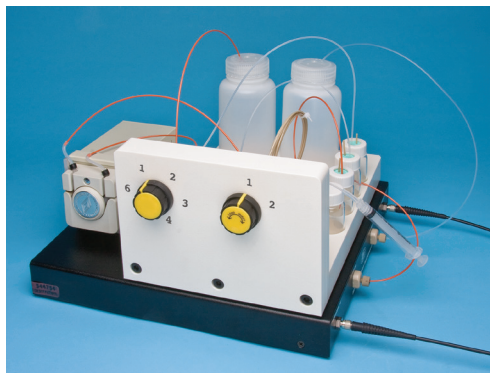
Optional Sample Injector Kit

The use of the Sample Injector Kit (WPI #58006), in conjunction with a pump connected to the SAMPLE OUT 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 SAMPLE IN 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 Kit minimizes the 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 Kit 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

Measuring Discrete Samples with WPI's Injection System



The sample measurement procedure can be further improved using the optional LWCC Injection system (WPI # **89372**) as discrete, reproducible sample volumes can be introduced to the LWCC with a seamless transition from the baseline solution to sample at a continuous flow rate. The LWCC Injection System, together with a pump and liquid waveguide capillary cell (LWCC), provides an efficient, continual flow for injecting a sample through the LWCC.

Fig. 5 The LWCC Injection System is shown with MINISTAR peristaltic pump and LWCC. Pump and LWCC are sold separately.

INSTRUMENT MAINTENANCE

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

Cleaning

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

Standard Cleaning

Clean the LWCC before and after each use. Use the cleaning kit for liquid waveguides (WPI #**501609**).

1. Connect the exit tubing (silicon or equivalent) from the SAMPLE OUT 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 1cc syringe with "Cleaning Solution 1" and inject it into the LWCC's SAMPLE IN via the luer fitting adapters provided with the LWCC.
4. Fill a 1cc glass type syringe with "Methanol Solution 2" and inject it into the SAMPLE IN of the LWCC.
5. Fill a 1cc syringe with "HCl Solution 3" and inject it into the SAMPLE IN of the LWCC.
6. Flush out the cleaning solutions with distilled, Ultra Pure, reverse osmosis or equivalent quality water and take an absorbance reading.
7. Repeat steps 3–6 until a stable absorbance signal can be obtained.

NOTE: The sample injector kit (WPI #**58006**) can be used to conveniently fill all LWCC models. Using the sample injector, the injection volume of the cleaning solutions per cycle can be reduced to the internal volume of the LWCC (for example, 0.26mL for a 100cm LWCC).

Advanced Cleaning

1. Connect the exit tubing (silicon or equivalent) from the SAMPLE OUT 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 the LWCC with a 10% solution of Contrad-NF (available from Decon Labs, Inc. – www.deconlabs.com part #6002). Allow the solution to sit inside the cell for 20 minutes.
4. Flush out the Contrad-NF solution with distilled, Ultra Pure, reverse osmosis or equivalent quality water and take an absorbance reading.
5. Repeat steps 3–4 until a stable absorbance signal can be obtained.

NOTE: For removal of persistent contamination, the 10% Contrad-NF solution can be heated to 60°C prior to injection into the LWCC.

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 remove all liquid using a peristaltic pump or syringe.

ACCESSORIES

Table 1: Accessories

Part Number	Description
FO-500AS-SMA	1M, SMA/500µm core, anti-solarization
FO-500-SMA1M	1M, SMA/500µm core, UV-enhanced
15807	Cleaning solution concentrate–100g (Solution 1)
58006	Sample Injector Kit
58450	Spare set of external syringe adapters
89372	LWCC Injection System
501609	Waveguide cleaning kit
500320	Silicone tubing for Peri-Star pump. 1.6mm ID x 1.6W
800490	Fiber optic foam swabs (5)
LWCC-CAL	LWCC Calibration Solution
MINISTAR	Miniature DC Peristaltic Pump
PERIPRO-8LS	Peri-Star™ 8-channel, low rate peristaltic pump
PERIPRO-4LS	Peri-Star™ 4-channel, low rate peristaltic pump
TIDAS-1	Tidas I high performance photodiode array spectrometer module
TIDAS-BASIC	Tidas E high performance fiber optic based photodiode array Spectrophotometer
D4H	D4H™ deuterium/halogen light source
FO-6000	Tungsten light source

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

Issue	Possible Cause	Solution
Transmission in both UV and VIS ranges becomes low or very unstable	A contamination layer (such as biofilm) sticking to the LWCC wall	Flush cell for 30 seconds each of each of the three cleaning solvents in the waveguide cleaning kit (WPI Part #501609). Prepare a 10% Surfactant using Contrad-NF concentrate followed by HPLC grade Methanol and HPLC grade 2N HCl solution.
	A particle trapped in the LWCC	Attach 1–5cc syringes with luer fittings to the SAMPLE IN and SAMPLE OUT ports on the LWCC. Fill one syringe with distilled water and inject it into the LWCC. Use the other syringe to push the water back through the LWCC in the opposite direction. Continue using both syringes to move the liquid rapidly through the LWCC in alternating directions.
UV transmission is low. VIS is fine and stable.	Optic fiber and silica tubing are coated by a layer of metal corrosion.	Flush with 1N HCl.
	Optic fiber and silica tubing is coated by a layer of organics.	Flush with an organic solvent, such as acetonitrile.
Transmission below 250nm is low. VIS is fine and stable	Contamination of fiber optic cable end-faces with a metal film generated during repeated connection attempts	Wipe all fiber optic end-faces, fiber connections (on the LWCC box front) using a fiber optic foam swab (WPI #800490) dipped in methyl alcohol. NOTE: Do NOT use cotton swabs (Q-Tips).

Additional Information on Contamination

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

- Most syringe filters contain some contaminants that absorb UV. More than likely, this is caused by the (plastic) mold release agent used in manufacturing. The first few milliliters of solution coming from a new filter will have some absorption in the UV range.
- 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.
- 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 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.
- 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 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. 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.

NOTE: If you have a problem/issue with that falls outside the definitions of this troubleshooting section, contact the WPI Technical Support team at 941-371-1003 or technicalsupport@wpiinc.com.

SPECIFICATIONS

This instrument conforms to the following specifications:

	LWCC-3050	LWCC-3100	LWCC-3250	LWCC-3500
Optical Pathlength	50cm	100cm	250cm	500cm
Internal Volume	125µL	250µL	625µL	1250µL
Fiber Connection	500µm SMA			
Transmission @254nm*	≥ 20%	≥ 10%	≥ 1%	-
Transmission @540nm*	≥ 35%	≥ 30%	≥ 30%	≥ 20%
Noise [mAU]**	<0.1	<0.2	<0.5	<1.0
Maximum Pressure	100 PSI			
Wetted Material	PEEK, Fused Silica, PTFE			
Liquid Input	Standard 10-32 Coned Port Fitting			

* Referenced using coupled 500µm fibers
** Measured using ASTM E685-93
*** A one-meter waveguide of 550µm internal diameter requires approximately 1.5PSI for water flow of 1.0mL/min.

APPENDIX A: PROPERTIES OF THE LWCC

LWCC Type II

Similar to optical fibers, in the LWCC, light is confined within the (liquid) core by total internal reflection at the core/wall interface. Optical fibers are then used to transport light to/from the sensor cell. The LWCC is designed for small sample volumes and high optical throughput. WPI's LWCC Type II replaced the original Type I and offers many advantages:

- Improved signal efficiency.
- Outer coating of a low refractive index polymer—The core liquid in Type II is contained by the synthetic fused silica tubing. Instead of using a low refractive index polymer tubing (Type I), the tubing has an outer coating of a low refractive index cladding material. Placing the refractive surface outside the silica isolates it from the liquid sample.
- No pH buffering necessary—Since the cladding material is on the outside, the CO_2 cannot penetrate it as in Type I LWCCs.
- No baseline shifts resulting from trapped organic vapor—The fused silica wall is impermeable to gases, eliminating effects observed in Type I that were due to the nature of the low refractive index material.
- Improved signal stability—The hydrophilic cell wall allows for easier removal of trapped air bubbles.

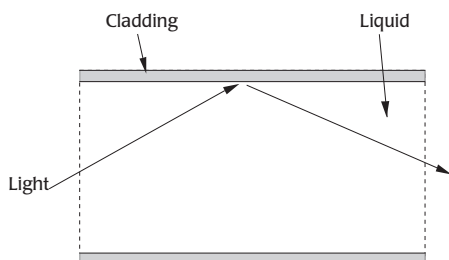


Fig. 6 (Left) Lateral section of **Type I** Liquid Waveguide Capillary Cell

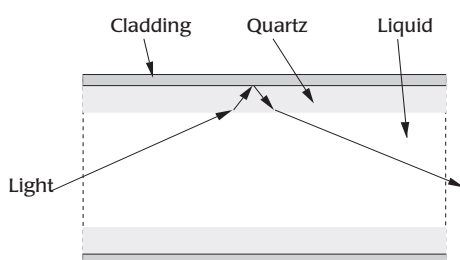


Fig. 7 (Right) Lateral section of **Type II** Liquid Waveguide Capillary Cell

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 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 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 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 2.0AU (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 μ m 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 200PSI without observed malfunction (a silica capillary with a similar structure has been reported to withstand pressures of at least 2000 PSI).

Light Throughput of FO Cables

The graph below compare three different fiber optic cables (400 μ m, 500 μ m and 600 μ m) used with the LWCC. The 500 and 600 μ m fibers are recommended to obtain maximum light throughput on the LWCC. While the 400 μ m fibers are compatible with an LWCC, you can expect an approximate 50% decrease in light throughput.

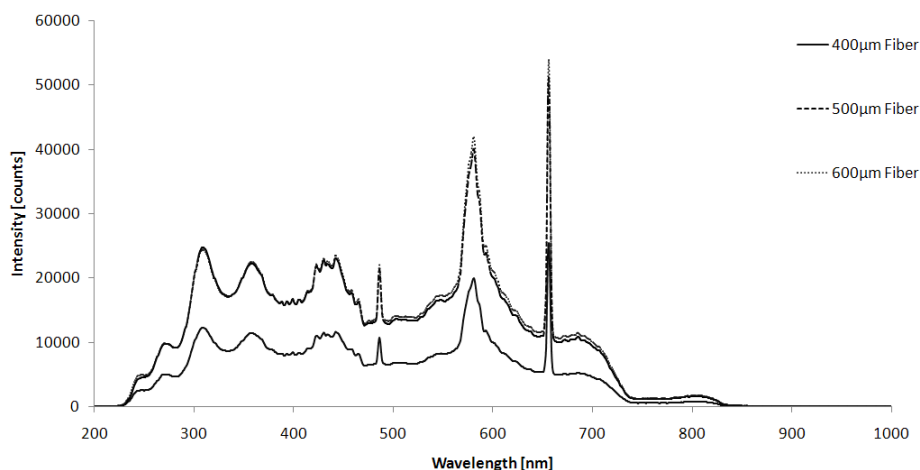


Fig. 8 When comparing light throughput versus wavelength of three fiber optic cables, the greater the diameter of the cable, the better the LWCC performance up to 500µm which is the input diameter of the SMA connector.

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) 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 30 days* 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 ten (10) days after receipt of shipment. Claims for lost shipments must be made within thirty (30) days of receipt of invoice or other notification of shipment. Please save damaged or pilfered cartons until claim is settled. 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

Do not return any goods to us without obtaining prior approval and instructions from our Returns Department. Goods returned (unauthorized) by collect freight may be refused. Goods accepted for restocking will be exchanged or credited to your WPI account. Goods returned which were ordered by customers in error are subject to a 25% restocking charge. Equipment which was built as a special order cannot be returned.

Repairs

Contact our Customer Service Department for assistance in the repair of apparatus. Do not return goods until instructions have been received. Returned items must be securely packed to prevent further damage in transit. The Customer is responsible for paying shipping expenses, including adequate insurance on all items returned for repairs. Identification of the item(s) by model number, name, as well as complete description of the difficulties experienced should be written on the repair purchase order and on a tag attached to the item.

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

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