4000 Series LWCC

Liquid Waveguide Capillary Cell

INSTRUCTION MANUAL

Serial No._____

112014

World Precision Instruments

Other WPI Favorites

World's Smallest Fiber Optic Dipping Probe

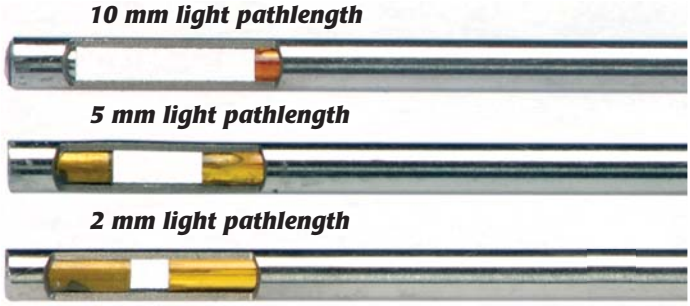
Mini DipTip™ is a miniature transmission probe for microliter spectroscopic sampling. Mini DipTip's tip diameter is only 1.5 mm—the size of a 17-gauge needle. It will fit into all micro centrifuge tubes on the market and is a useful tool for measuring protein and DNA samples. It can also be used for a dissolution system.

Microliter samples can be analyzed cost effectively when you combine the Mini DipTip with one of the following:

- The Fiber optic-based spectrometer (Tidas I) and a light source (D4H and FO-6000)
- WPI's biophotometric detection system (LEDspec)

The **Mini DipTip** is ideal for multi-channel applications.

- Compatible with many standard spectrophotometers (600µm fiber optic coupler connections)

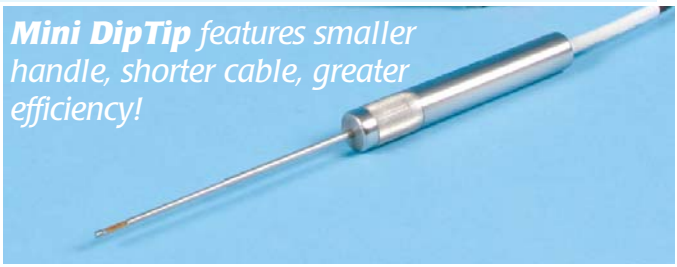


for UV/Vis Spectroscopy

DIPTIP SPECIFICATIONS

	DIP-UV-MINI
TIP DIAMETER	1.5 mm
LIGHT PATHLENGTH	2, 5, 10mm
WAVELENGTH RANGE (nm)	200-1000
SAMPLE VOLUME REQUIRED	20-50 µL
DISTANCE FROM TIP TO UPPER EDGE OF SAMPLE WINDOW	7 mm
FIBER LENGTH	1.0 m
FIBER OPTIC CONNECTION	SMA 905
LAUNCH FIBER BUNDLE (7 x 200µm)	680 µm*
RETURN FIBER BUNDLE (7 x 200µm)	680 µm*

**Circular packaging of the fiber bundle results in an active area equivalent to a fiber with a core diameter of 680 µm. Using a 600 µm connection is recommended and will result in negligible light loss.*



CONTENTS

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 10, 50 and 100cm pathlengths

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 (10–100cm) with small sample volumes (0.31–3.14mL) 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 or an UltraPath injector Kit (WPI **#72100**). 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 invalidates the warranty. Components inside are extremely fragile and are not user-serviceable. If you have trouble with the instrument, contact WPI's technical support department immediately 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 UltraPath Injector Kit (WPI **#72100**) 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) **72100** UltraPath Injector Kit
(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 15 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 15 of this manual.

Hardware Description

Required But Not Provided (see Accessories)

- (2) Fiber Optic Cables, like the **FO-600-SMA1M**
- 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

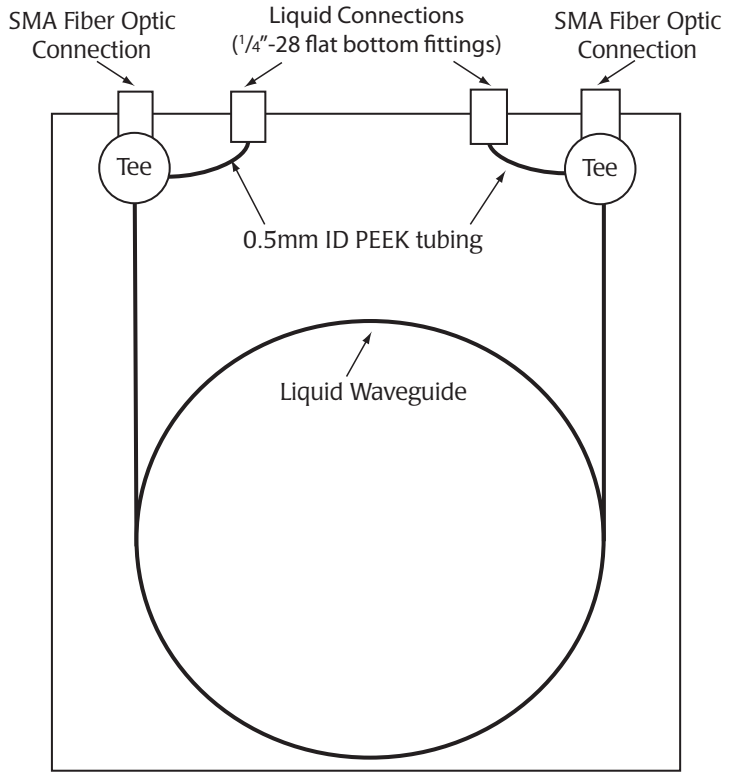


Fig. 2—LWCC Setup Schematic

LWCC	FIBER OPTIC CABLE
10, 50, 100cm	SMA 905 terminated, for example, WPI **#FO-600-SMA1M**

The light source and detector can be connected to the LWCC via two SMA-terminated fiber optic cables with a core diameter of 600µm. 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. 3**). Install the 1/4"-28 flat-bottom fittings to the ports.

NOTE: These connectors may be removed if the LWCC is to be connected to a fluid injection analysis system.

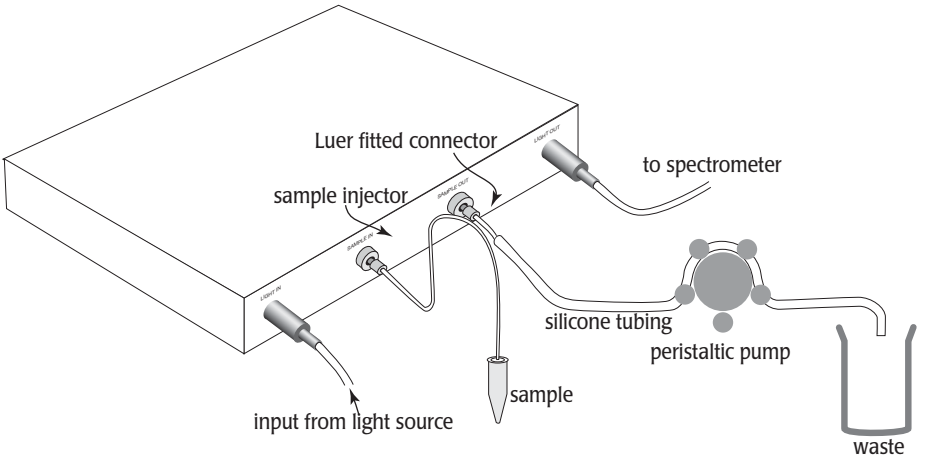


Fig. 3—Typical LWCC experimental setup with Sample Injector Kit

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).

CAUTION: Keeping the LWCC clean is essential for a stable result. See "Instrument Maintenance" on page 7.

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 7.
4. Connect the liquid source to the LWCC system. A pressure of approximately 1.5–3.0 PSI is necessary to run liquid through the LWCC.
5. At a rate of 12mL/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 8 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 conistent 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 7.
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.

NOTE: In the LWCC-4000 series, sample and reference solution may be separated by air pockets to minimize mixing of reference and sample solution.

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 10cc 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 10cc glass type syringe with "Methanol Solution 2" and inject it into the SAMPLE IN of the LWCC.
5. Fill a 10cc 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 UltraPath injector kit (WPI #**72100**) 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, 3.14mL 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 60ºC 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-600AS-SMA	1M, SMA/600µm core, anti-solarization
FO-600-SMA1M	1M, SMA/600µm core, UV-enhanced
15807	Cleaning solution concentrate–100g (Solution 1)
72100	UltraPath Injector Kit
501609	Waveguide cleaning kit
500320	Silicone tubing for Peri-Star pump. 1.6mm ID x 1.6W
800490	Fiber optic foam swabs (5)
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.

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 a 10cc 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.

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-4010	LWCC-4050	LWCC-4100
Optical Pathlength	10cm	50cm	100cm
Internal Volume	0.31mL	1.57mL	3.14mL
Fiber Connection	600µm SMA		
Wavelength Region	200–1,000nm		
Transmission @254nm*	20	10	5
Transmission @540nm*	35	30	25
Noise [mAU]	<0.1	<0.2	<0.5
Maximum Pressure	100 PSI		
Wetted Material	PEEK, Fused Silica, PTFE		
Liquid Input	Standard ¼"-28 Flat Bottom		

* Referenced using coupled 600µm fibers

APPENDIX A: PROPERTIES OF THE LWCC

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 2mm ID waveguide requires approximately 1.5 PSI for water flow of 1mL/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.

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 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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