INSTRUCTION MANUAL

LWCC-M

Measure Low Volume Samples with UV/VIS/NIR Absorbance Spectroscopy

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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—The **LWCC-M** *is available with 10, 50 and 100mm pathlengths.*

INTRODUCTION

The **Micro Liquid Waveguide Capillary Cell** (WPI #**LWCC-M-xxx**) is a fiber optic, low volume flow cell for UV/VIS/NIR absorbance analysis; xxx represents the pathlength in mm. The **LWCC-M** is available with 10mm, 50mm and 100mm pathlengths. Based on WPI's established liquid core waveguide technology, the analyte solution functions as the core of a fluid-filled light waveguide. Wetted parts in the sample cell light path are PEEK, fused silica and Teflon. Optical fibers are used to transport light to and from the sample cell. The cell can be used in biochemistry for DNA, RNA and protein quantification, colorimetric nutrient and trace metal analysis, drug discovery and dissolution testing, process control and HPLC analysis.

Similar to optical fibers, light is confined within the (liquid) core of an **LWCC-M** 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.

General Warnings and Cautions

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-M** 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-M**
(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 13 of this manual. Please contact WPI Customer Service if any parts are missing at 1 (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 13 of this manual.

Hardware Description

Required But Not Provided (see Accessories)

- SMA-terminated, 600 μm core fiber optic cables (2)
- Detection System including either a spectrophotometer or a fiber optic spectrometer and a fiber optic 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

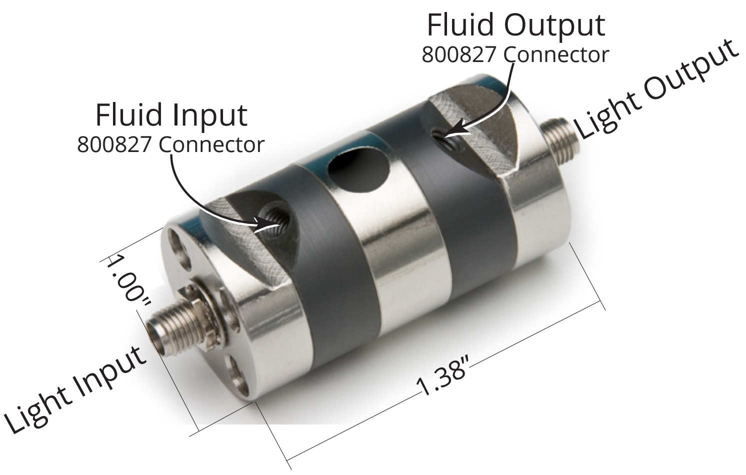


*Fig. 2—**LWCC-M**-10 is shown. The **LWCC-M**-50 and **LWCC-M**-100 are longer.*

The **LWCC-M** (10, 50, 100mm) has SMA 905 terminated (WPI #**505195**) fiber optic connections. The light source and detector can be connected to the **LWCC-M** via two SMA-terminated fiber optic cables with a core diameter of 600 μm. 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-M** 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. Use standard HPLC type 10-32 coned PEEK 1/16th tubing fittings (WPI #**800827**) to make connections to the pump and waste receptacle.

OPERATING INSTRUCTIONS

CAUTION: Materials exposed to fluid in the **LWCC-M** are PEEK, FEP and fused silica. Any chemical that could attack these substances should not be used in the **LWCC-M**. 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-M** clean is essential for a stable result. See "Instrument Maintenance" on page 6.

Transmission Verification

1. Connect one 500μm diameter fiber optic assembly to a light source and one 500μm diameter fiber optic assembly to the detector.
2. Use an SMA bulkhead adapter (WPI #**13395**) to butt-couple the fibers. This is the reference for the measurement of transmission.
3. Remove the SMA bulkhead adapter and insert the **LWCC-M**.
4. Record the percent transmission. Values for the 254nm and 540nm should exceed 40% and 50% respectively.

Noise Determination

Noise is determined using the ASTM E685-93 standard as reapproved in 2000. Use methanol as the solvent at a flow rate of 1.0mL/min. Using the WPI test setup, noise values are below 35μAU. See www.astm.org/Standards/E685.htm to obtain a copy of the standards document.

Refractive Index

The refractive index between methanol and cyclohexane is measured using a flow rate of 3.0mL/min. Because of the immiscibility of the solvents, ethanol is used between the solvents to prevent unwanted interaction between methanol and cyclohexane. At the 280nm wavelength, RI values shown below are acceptable:

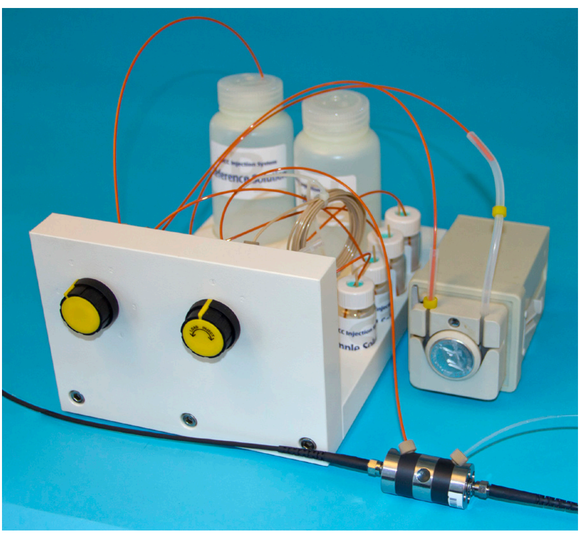
- **LWCC-M-10** 0–7
- **LWCC-M-50** 0–14
- **LWCC-M-100** 0–25

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-M**.
2. Using fiber optic cables, connect the **LWCC-M** to a light source and a detector.
3. Clean the **LWCC-M** using the standard cleaning procedure described in "Instrument Maintenance" on page 6.
4. Connect the liquid source to the **LWCC-M** system. A pressure of approximately 1.5–3.0 PSI is necessary to run liquid through the **LWCC-M**.
5. At a rate of 1mL/min., flush the **LWCC-M** 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 "Part Number Description" on page 7 if the signal does not stabilize.

Measuring Discrete Samples with WPI's Injection System



The sample measurement procedure can be improved using the optional LWCC Injection system (WPI # **89372**) as discrete, reproducible sample volumes can be introduced to the **LWCC-M** 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-M**), provides an efficient, continual flow for injecting a sample through the **LWCC-M**.

Fig. 3—The LWCC Injection System is shown with MINISTAR peristaltic pump and ***LWCC-M****. Pump and* ***LWCC-M*** *are sold separately.*

INSTRUMENT MAINTENANCE

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

Standard Cleaning

With normal use, the **LWCC-M-10** will rarely need cleaning. If measurement results diminish, clean your **LWCC-M-10**. Use the cleaning kit for liquid waveguides (WPI #**501609**).

1. Connect the exit tubing (for example, silicon or equivalent) from a fluid port (output) of the **LWCC-M** 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. Inject 1cc “Cleaning Solution 1” into the opposite **LWCC-M** fluid port (input).
4. Inject 1cc “Methanol Solution 2” into the fluid port (input) of the **LWCC-M**.
5. Inject 1cc “HCl Solution 3” into the fluid port (input) of the **LWCC-M**.
6. Flush out the 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.
8. If the UV readings are still low after cleaning, swab the fiber optic surfaces of the sample cell with a fiber optic Q-tip dipped in isopropyl alcohol.

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

Advanced Cleaning

1. Connect the exit tubing (for example, silicon or equivalent) from a fluid port (output) of the **LWCC-M** 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-M** 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-M**.

Storage

CAUTION: Do not leave the **LWCC-M** "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-M** and then remove all liquid using a peristaltic pump or syringe. Seal the fluid ports using either the caps provided or an alternative.

ACCESSORIES

Part Number	Description
505195	1 M, SMA/600 µm core, anti-solarization
15807	Cleaning solution concentrate–100 g (Solution 1)
58450	External syringe adapters
89372	LWCC Injection System
501609	Waveguide cleaning kit
500320	Silicone tubing for Peri-Star pump. 1.6mm ID x 1.6W
505155	Qualified Optical Fiber, Deep UV/NIR*
505160	Qualified Optical Fiber, UV-NIR*
800490	Fiber optic foam swabs (5)
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
D4H	D4H™ deuterium/halogen light source
FO-6000	Tungsten light source

*A variety of configurations are available with different core diameters, length and connectors. These fibers can be custom made to order.

RELATED PRODUCTS

Part Number	Description
504717	Tidas-E Base Series Photo Diode Array Spectrometer, UV
504718	Tidas-E Base Series Photo Diode Array Spectrometer, UV/VIS
504719	Tidas-E Base Series Photo Diode Array Spectrometer, NIR

TROUBLESHOOTING

The **LWCC-M** 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-M** 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-M** wall	Flush cell for 30 seconds each of each of the three cleaning solvents in the wave-guide 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-M**	Attach 1–5cc syringes with luer fittings to the SAMPLE IN and SAMPLE OUT ports on the **LWCC-M**. Fill one syringe with distilled water and inject it into the **LWCC-M**. Use the other syringe to push the water back through the **LWCC-M** in the opposite direction. Continue using both syringes to move the liquid rapidly through the **LWCC-M** 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-M** 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-M**:

- 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-M**.
- 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-M** 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-M**. If trapped in the **LWCC-M**, particles can scatter light and may cause unstable spectrometer readings. The **LWCC-M** contains two potential "bottlenecks" at the fiber-capillary interface. Due to the diverse applications of **LWCC-M**, 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-M** if the solution contains large particles. When the **LWCC-M** 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:

	M-10	M-50	M-100
Optical Pathlength	10 mm	50 mm	100 mm
Internal Volume	2.4 µL	12 µL	24 µL
Wavelength Range	UV-NIR (200 – 1200 nm)		
Fiber Connection (SMA)	600 µm		
Transmission @ 254 nm *	> 25%	> 20%	>15%
Transmission @500 nm *	> 40%	> 35%	>30%
Maximum Pressure	1000 PSI		
Refractive Index @ 280 nm**	< 15 mAU	<20 mAU	<30 mAU
Wetted Material	PEEK, Fused Silica, PTFE (Other materials are available		

* Reference: Two 600 µm fibers, butt-coupled
** Measured using ASTM E 685 - 93, includes intrinsic solvent absorbance
WPI U.S. Patents: 5,444,807; 5,570,447; 5,604,587; 6,603,556; 6,385,380.

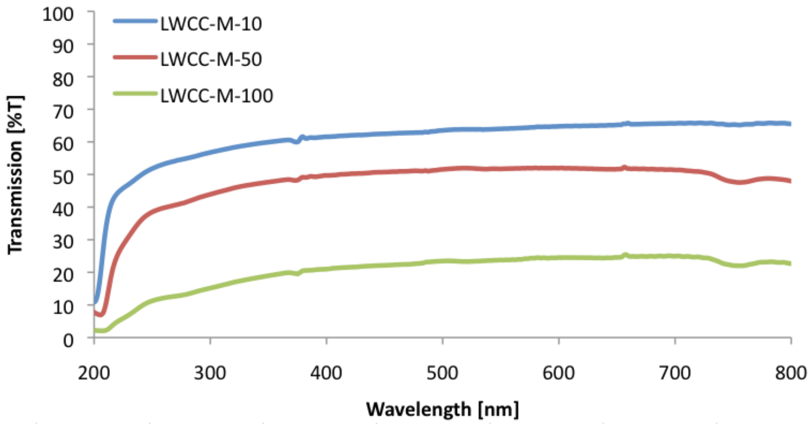


Fig. 4—This graph show the percentage of light transmission at various wavelengths. The higher the light transmission percentage, the greater the resolution.

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

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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