



Multiple pathlength sample cell for absorbance spectroscopy with extended dynamic range

C.C0	SYSTEM GUIDE for visible (UPVIS) and ultraviolet (UPUV) systems
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General Warnings and Cautions

Read this manual before you attempt to use this instrument.

All warnings on the unit and in these operating instructions should be adhered to.

Warning: Do not look directly into the light output of the light source. Light radiation may damage your eyes.

Warning: Do not remove any safety devices installed. This will void your warranty and create an unsafe operating condition.

Warning: Dangerous voltages are present. No user serviceable parts inside unit. Instrument should be serviced by qualified service personnel only.

Warning: The UltraPath sampling system contains cleaning solutions. Only qualified personnel should use these solutions. Please refer to the MSDS Sheets for details on the solutions.

Warning: Before using the instrument for the first time, check for transport damage.



Introduction

UltraPath is a unique high-performance spectrophotometer offering user-selectable optical path lengths of 2, 10, 50 and 200 cm. Designed for the detection of low absorbing species in solution, UltraPath is an ideal tool for any study requiring precise and highly sensitive spectroscopic determination of analytes, either in the lab or in the field. The instrument operates in the wavelength range of 370 to 730 nm (UPVIS) or 250 to 730 nm (UPUV) and has an exceptional dynamic range allowing reliable absorbance measurements of between 5µAU/cm to 1 AU/cm to be routinely made.

UltraPath was developed by WPI under a collaborative agreement with NASA (Stennis



Space Center) for the spectroscopic determination of colored dissolved organic matter (CDOM) in seawater and fresh water environments, and was designed for use in the laboratory and in the field (*i.e.*, at sea). CDOM concentrations vary significantly between open ocean samples with low CDOM (*e.g.*, 0.007 m-1 at 380 nm), and high CDOM freshwater environments (*e.g.*, 10-20 m-1 at 380 nm).

To address these problems the design requirements of UltraPath mandated the

development of a rugged portable system capable of highly sensitive measurements across a wide dynamic range.

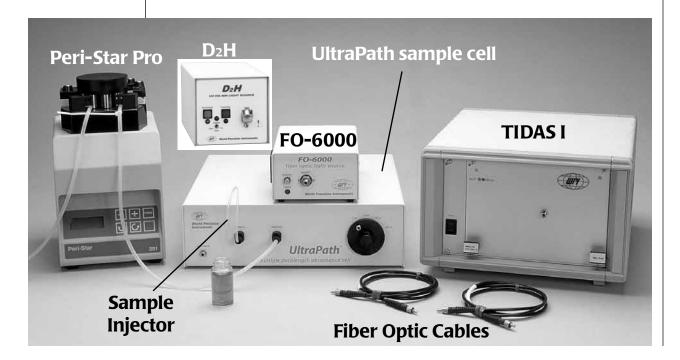
The UltraPath sample cell has four optical pathlengths contained within a single sample cell (i.e., 2 cm, 10 cm, 50 cm and 200 cm). The pathlengths are user-selectable, offering a very high sensitivity and an extended dynamic range for VIS absorbance measurements (a UV version of UltraPath is available on request). The fluid path of the sample cell is optimized to produce a laminar flow that is virtually free of interference from trapped air bubbles and adherence of dissolved substances to the cell wall. In particular, the design greatly minimizes the problems commonly found with flow cells of long optical pathlengths: the risk of trapping dust particles, fibers or particulate matter inside the cell.



Instrument Description

The UltraPath system includes a photodiode array-based spectrometer module (**TIDAS I**) to measure the absorbance at the selected pathlength. Absorption of light is measured between 370 and 730 nm (UPVIS) or 250 to 730 nm (UPUV) with a resolution of 5 nm (FWHM) and a noise level below 0.2 mAU. A color corrected tungsten light source, WPI's **FO-6000** (UPVIS), or a deuterium/tungsten light source, WPI's **D2H** (UPUV), delivers light via fiber optic cables to the **UltraPath** sample cell. A peristaltic pump (**Peri-Star Pro**) is utilized to evenly draw sample up into the **Sample Injector** and through the UltraPath sample cell.

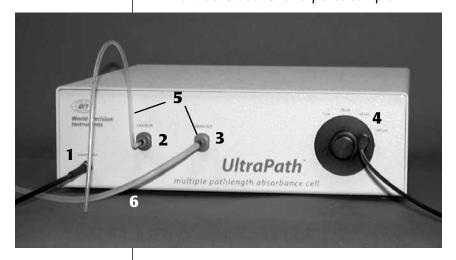
A standard PC or laptop (not included) is connected to the detection system via a RS232 interface. The **TIDASDAQ/SpectraView** software (included) displays and performs data collection and processing. High precision absorbance or transmittance spectra can be obtained within seconds. Key features of the software package include ease-of-use, simple spectra acquisition, as well as tools for sample qualification, quantification, and data extraction. (Refer to the TIDASDAQ & SpectraView manuals for a detailed description of the software supplied with the instrument.)





UltraPath Sample Cell

- 1. Fiber optic input connector
- 2. Liquid Input connector
- **3. Liquid Output Connector.** Sample is removed from the UltraPath sample cell via the Liquid Output Connector with vacuum suction of the Peri-Star Properistaltic pump.
- **4. Fiber Optic Switch & Output Connector.** The optical pathlength of the UltraPath can be switched between 2, 10, 50 and 200 cm. Pull the knob and turn to the selected pathlength
- **5. UltraPath Sample Injector Kit.** The Sample Injector (WPI **#72100**) is connected to the Liquid Input connector (7). Sample is drawn into the UltraPath sample cell with the Sample Injector using vacuum suction of the Peri-Star Pro peristaltic pump.
- 6. Silicone pump tubing (WPI# 500320).
- **7. Waste Bottle** for expelled sample.

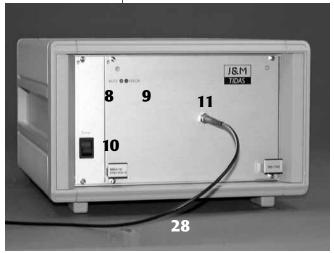


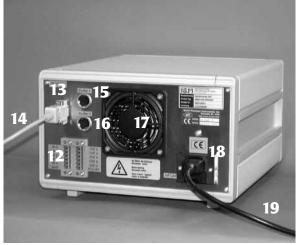




TIDAS I Spectrometer

- **8. Busy LED Indicator (Green).** The Busy LED shows communication of the TIDAS I and the computer via the RS232 interface.
- **9. Error LED Indicator (Red).** The Error LED lights up when the communication between TIDAS I and the computer is not functioning properly.
- **10. Power Switch** (ON/OFF). When the power switch is turned ON, power is supplied to the TIDAS I spectrometer module.
- **11. Fiber optic input.** The Fiber optic input connects to the light output of the UltraPath sample cell (4).
- **12. TTL Input/Output.** 5-Pin Phoenix receptacles (mating connectors supplied). For further instructions refer to TIDAS I instruction manual.
- **13. RS232 interface connection (9-Pin Sub D connector).** The TIDAS I is connected to a computer with a serial cable via the RS 232 interface connection.
- 14. RS232 interface cable.
- **15. Shutter L**: Two-Pin DIN socket for first light source
- **16. Shutter S**: Two-Pin DIN socket for second light source







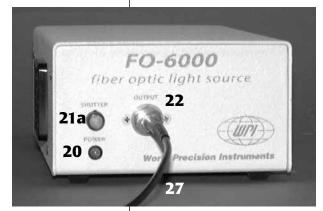
- **17. Cooling Fan.** The fan dissipated the heat generated by the power supply and electronics of the TIDAS I. To ensure proper ventilation, the unit should be situated away from walls or panels. Do not obstruct the cooling fan openings.
- **18. Input terminal for power cord and fuse.** The TIDAS I accepts voltages from 80 to 240 VAC, 50-60Hz. Use correct cord and fuse, as described in the manual.
- 19. Power cord for TIDAS I.

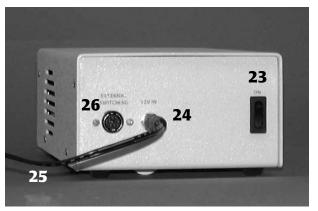
Fiber Optic Light Source

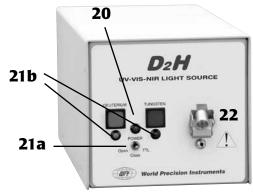
20. Power On LED Indicator Light (FO-6000 & D2H)

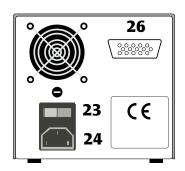
21a. Shutter Push button

UPVIS (FO-6000): Upon Pressing the Shutter button, the button illuminated, the shutter opens and light exits the SMA fiber optic connector (22).











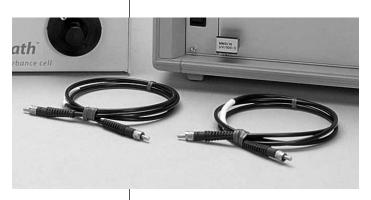
UPUV (**D**₂**H**): Upon moving the switch to "Open Position" the shutter opens and light exits the SMA fiber optic connector (22).

- **21b. Deuterium & Tungsten Enable switches** (UPUV: **D**₂**H** only)
- **22. Light output.** SMA Fiber Optic Connector (collimator) with adjacent mounting screws.
- **23. Power Switch** on/off, when power switch is turned ON, power is supplied to the unit. The tungsten lamp is switched on and starts to warm up. The Power ON LED indicator (20) is lighted at this time.
- **24. Power Input** for included 12-volt power supply (UPVIS: **FO-6000**) or for standard power cable (UPUV: **D**₂**H**).
- **25. Power cable to 12-volt power supply** (supplied with **FO-6000**).
- **26. TTL connection** permits external control of lamp shutter.

UPVIS (FO-6000): 7-pin sub-miniature DIN connector.

UPUV (D2H): 15-pin D-Sub connector (cable supplied).

Fiber Optic Cables

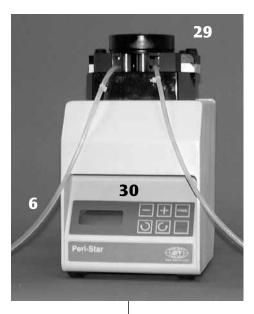


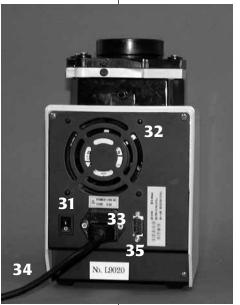
- **27. Fiber optic cable** Light is coupled into the fiber optic input connector (1) of the UltraPath sample cell from the FO-6000 light output (22) with a 600 µm core fiber (**FO-600-SMA1M**).
- **28. Fiber optic cable** Light is coupled from the output of the UltraPath fiber optic switch (4) into the TIDAS I spectrometer input (11) with a 600 µm core fused silica fiber (**FO-600-SMA1M**).

Peri-Star Pro Peristaltic Pump

- **29. Peri-Star Pro Pump Head.** Two channels and eight rollers.
- **30. Control Panel.** The Control Panel allows to start the pump clockwise and counter clockwise, to increase and decrease pump speed between 0.1 rpm



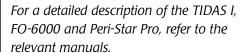




- and 100 rpm, to stop the pump and to prime the pump (Refer to the Peri-Star Pro manual for details).
- **31. Power Switch** (ON/OFF). When the power switch is turned ON, power is supplied to the Peri-Star Properistaltic pump
- **32. Cooling Fan.** The fan dissipated the heat generated by the power supply, motor and electronics of the Peri-Star pump. To ensure proper ventilation, the unit should be situated away from walls or panels. Do not obstruct the cooling fan openings.
- **33. Input terminal for power cord.** USA version: 110-115 VAC, 60Hz (WPI#**500291**), European Version: 230-240 VAC, 50 Hz (WPI#**500304**).
- 34. Power cord for Peri-Star Pro.
- **35. External Control socket.** DB-9 connection to control speed and direction of the pump head. Refer to the instruction manual for details.

Cleaning Kit

36. Waveguide Cleaning Kit. The waveguide cleaning kit contains three solutions specifically developed to keep the UltraPath sample cell clean and ensure a high measurement accuracy and repeatability.







Setting up UltraPath

UltraPath is a modular spectrophotometer system. Its main components are: UltraPath sample cell, TIDAS I spectrometer module, **FO-6000** light source (optional **D2H-2**). The assembly of the UltraPath system is described below.

Parts List

- UltraPath cell and UltraPath System Guide
- UltraPath Sample Injector Kit (WPI #**72100**)
- Peristaltic silicone tubing (WPI #500320)
- Waste bottle
- Waveguide cleaning kit (WPI #**501609**)
- TIDAS I spectrometer, including Power Cord, RS232 serial cable, UltraPath software CD, UltraPath Access disk and instruction manual (on UltraPath CD).
- Light source **FO-6000**, power supply and instruction manual (UPVIS); or Light source **D2H**, power cord, safety goggles and instruction manual (UPUV).
- Fiber optic cables (2), WPI #FO-600-SMA1M).
- Peri-Star Pro pump (WPI **#PERIPRO-4L** (110V, 60Hz) or **PERIPRO-4L-220** (220V, 50Hz) with power cord and instruction manual.

Please note: The following manuals are included as hardcopy: UltraPath System Guide, FO-6000, Peri-Star Pro, Waveguide Cleaning Kit. The software manuals to operate the TIDAS I are included on the UltraPath software CD.

Opening the package

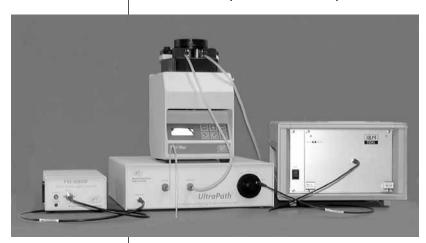
Upon receipt of this system, make a thorough inspection of the contents and check for possible damage. Concealed loss or damage should be reported at once to the carrier and an inspection requested. Please read the section entitled "Claims and Returns" on the warranty page of this manual. Please call WPI Technical Support if any parts are missing.



The following instructions describing the assembly of an UltraPath system for visible applications (UPVIS) are very similar to those for the ultraviolet system (UPUV).

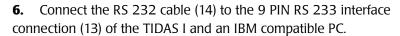
Assembling the Ultrapath system

1. Prepare a clear workspace for all components.



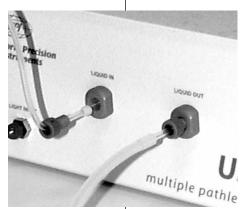
- **2.** From left to right place the following components on the workspace: FO-6000 Light source, Ultrapath sample cell, Peri-Star Pro peristaltic pump, Tidas I spectrometer and IBM compatible personal computer.
- **3.** Use a 600 µm core diameter fiber optic cable (27) to connect the light output (22) of the FO-6000 to the fiber optic input connector (1) of the UltraPath sample cell.
- **4.** Use a 600 μ m core diameter fiber optic cable (28) to connect the light output (4) of the UltraPath Fiber Optic Switch to the fiber optic input (11) of the TIDAS I spectrometer.





- **7.** Connect the Power cord (19) to the Input Terminal (18) of the TIDAS I and main.
- **8.** Assemble the sample injector loop of the UltraPath Sample Injector Kit (5) by sliding on a nut and a ferrule and then screwing the assembly into the bulkhead fitting labeled "liquid in" (2) of the UltraPath sample cell. Note: there are two ways the yellow ferrule will go onto the tubing; the wider side should face

the end of the assembly. Finger tighten only! (See picture for details.) Place the injector into a sample vial filled with de-ionized water.





- **9.** Use the silicone pump tubing (6) to connect the liquid output port (3) of the UltraPath sample cell via the Peri-Star peristaltic pump to the Waste bottle (11) (See Peri-Star instruction manual for details).
- **10.** Turn on the FO-6000 (or D2H) allow lamp to warm up at least 15 minutes before sampling. Turn on the Peri-Star Pro and the TIDAS I.

Installing the software

Software Requirements

The TIDASDAQ and SpectraView software packages require Windows XP or Vista.

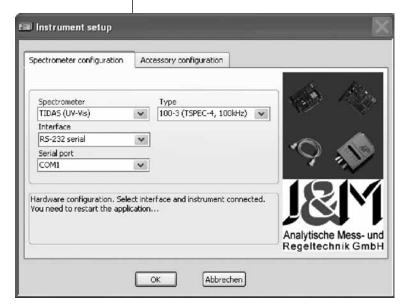
Hardware Requirements

- PC with Windows XP or Vista.
- VGA graphics adapter, minimum resolution 1024 x 768
- Minimum 150 MB of free hard disk space.
- CD-ROM Drive
- RS232 interface (Com 1 or Com 2) or USB-RS232 adapter.

Installation Steps

The Setup program prompts you through the installation process. To install the software, insert the CD labeled **UltraPath** into your CD-ROM drive.

TIDASDAQ:



- In the TIDASDAQ directory, run setup.exe to install TIDASDAQ. Follow the instructions on the screen. After the the installation is complete, copy the TIDASDAQ.lic file into the TIDASDAQ sub-directory under Program Files on your local hard drive.
- Ensure that the TIDAS I is connected to the COM1 port of your computer and switch it ON.
- Start TIDASDAQ. At the first start,



the Instrument setup window of the TIDASDAQ software is displayed. Select:

Spectrometer: "Tidas (UV-Vis)" Interface: "RS232 serial"

Serial port: "COM 1" or your active port Type: "100-3 (TSPEC-4, 100kHz)"

• Then, press ok to finish the instrument setup.

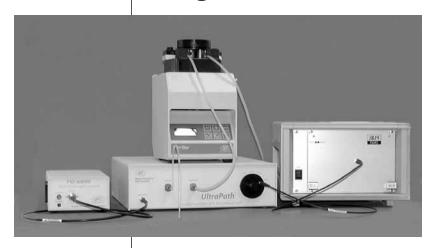
SpectraView:

- In the LabView_8.2_Runtime_Engine directory, run setup.exe to install the LabView 8.2 Runtime Engine. Follow the instructions on the screen.
- Restart the computer.
- In the SpectraView directory, run setup.exe to install SpectraView. Follow the instructions on the screen.

See the TIDASDAQ and the SpectraView manuals on the UltraPath CD for details on the software packages. For further details and changing the serial communication port from COM 1 to COM 2, refer to the TIDAS I manual.



Using UltraPath



Ultrapath is a very sensitive device for measuring low absorbing substances in solution. The following instructions will familiarize you with using and cleaning the Ultrapath system.

• Switch the light source ON (23) and allow approximately 15 minutes warming up time for the lamp to stabilize its temperature drift (<0.5mAU/h). For test purposes, the lamp may be used earlier. Open the shutter (20)

at the light source to take measurements. Close the shutter, when a dark current has to be taken.

- Switch the pump (Peri-Star Pro) ON (31) and set the pump speed to 40 RPM (12mL/min) at the control panel (30). The sample is drawn into the waveguide by vacuum suction.
- Switch the TIDAS I ON (10) and start the TIDASDAQ software.
- Select a pathlength (2, 10, 50, 200 cm) at the fiber optic switch (4) of the UltraPath sample cell.

How to fill the UltraPath sample cell

- Place the injector (5) into a sample vial. The sample is drawn with the peristaltic pump (Peri-Star Pro) into the UltraPath sample cell by vacuum suction.
- Press the forward button at the control panel (30) of the Peri-Star Pro to start drawing sample solution into the cell.

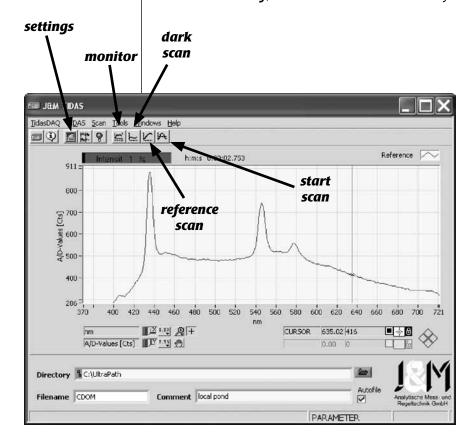


- Lift the sample injector between samples to allow for an air bubble to enter the injector tubing. In this way samples can be separated and cross contamination due to mixing minimized.
- Using the 2cm, 10cm or 50cm cell, a pumping time of approx. 30 seconds is necessary, using the 200cm path, a pumping time of approx. 60 seconds is necessary to fill the cell completely with sample solution.
- The cell is completely filled when the air bubble generated earlier exits the "Liquid out" connector (3) of the sample cell.

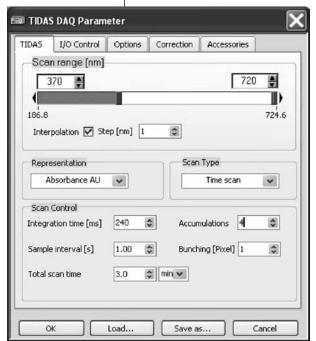
Acquiring Data with TIDASDAQ – a typical measurement cycle

After the light source is running for 15 minutes, the waveguide is filled with reference solution and the TIDASDAQ software started. Measurements can then be taken by the following steps.

1. Define **Directory**, **Filename** and **Comment** of your dataset.







2. Press the **Settings** icon to open the **TIDASDAQ Parameter** window.

Use the following typical parameters to get started:

Scan range to 370-720nm (UPVIS) or 250-720 nm (UPUV),

Representation to Absorbance,

Scan Type to Time scan,

Integration time to **240 m**s

Accumulations to 4

Sample Interval to 1 s

Total scan time to 3 min

(Adjust these settings later depending on your pathlength setting.)

Use the Monitor Parameter function icon to set integration time of the TIDAS I. A maximum of 45000 cts should be observed, when using MilliQ water in the UltraPath sample as a reference. Typical integration times are 100 to 2000ms.

Sample collection

- 1. Close the shutter at the light source (21) and take a **Dark Scan**
- **2.** Open the shutter (21) at the light source and press the **Reference Scan** icon to take a reference signal.
- **3.** Press the **Scan Start** icon to start a continuous scan.
- **4.** Start the pump and draw sample into the UltraPath sample cell (See "How to fill the UltraPath sample cell" for details).
- **5.** Introduce an air bubble of approximately the length of the Injector tubing at the liquid input of the UltraPath sample cell and draw the sample into the cell.
- **6.** A very high absorbance signal will be observed until the sample cell is filled with the sample solution, then a steady signal will be present. This may take between 15 seconds and 1 minute, depending on the pathlength used.



It is good practice to measure the baseline with reference solution at the beginning and the end of the measurement cycle. Thus, introduce a second air bubble and draw a second draw reference solution into the sample cell until a stable baseline signal has been reached. After the signal has returned to the baseline, stop the continuous measurement cycle by pressing the **Start Scan** icon.

NOTE:

1. Sample Cell Preparation

It is good practice to fill the UltraPath at the beginning of each measurement cycle with clean deionized water and measure the Light intensity throughput of the cell for quality reference. This allows the user to determine, if the sample cell is contaminated and needs cleaning.

2. Seawater

WPI highly recommends using salinity matched reference solutions, when working with seawater to avoid absorbance baseline offsets caused by refractive index variations when reference and sample solution have different salinities.

Refractive index sensitivity of the UltraPath sample cell – salinity matched reference solutions to avoid baseline offsets

The UltraPath sample cell is sensitive to variations in refractive index between reference and sample solution. When measuring in saline solutions, such as e.g. colored dissolved organic matter (CDOM) in seawater, it is recommended to use a reference/standard solution of matched salinity. These reference solutions should be filtered with the same filter material as the sample (e.g., 0.2-micron filters). Saline solutions have a slightly higher refractive index that pure water, resulting in a negative absorbance baseline shift compared to de-ionized water. For more information see:

Richard L. Miller, Mathias Belz, Carlos Del Castillo, Rick Trzaska, "Determining CDOM Absorption Spectra in Diverse Coastal Environments Using a Multiple Pathlength, Liquid Core Waveguide System", *Continental Shelf Research* (July 2002), **22**:9, p 1301-1310.



Measurement reliability

Baseline absorbance measurements should be within \pm 1-2 mAU between baseline samples. If larger baseline variations are observed, the following problems may be present:

- 1. An air bubble is trapped inside the cell. Allow for more sample to be injected and scan again.
- 2. The cell is contaminated. Follow the cleaning protocol and take a new reference.
- 3. The salinity of the sample is not the same as the salinity of the reference solution. If working in saline environments, prepare saline-matched reference solutions to accommodate for baseline shifts.

See instrument maintenance and troubleshooting section on how to overcome these problems.

Flow rate and maximum pressure

The applied pressure and fluid flow rate through the LWCC/UltraPath obeys the Hagen-Poiseuille relationship. Flow is proportional to pressure and to the fourth power of the diameter of the sample cell, as well as reciprocal to the length of the cell and fluid viscosity. Typical flow rates of the UltraPath sample cell are 12 mL/min (equal to 40 RPM with the Peri-Star pump). Drawing liquid into the cell via the injector fills the sample cell. The UltraPath sample cell is designed for low pressure operations and should not be used at pressures greater than 100 PSI.

Effective pathlength and linearity

Effective pathlength and linearity have been extensively studied with WPI's Liquid waveguide capillary cell technology and the UltraPath system. "Effective pathlength" is defined as the equivalent pathlength of the cell if we assume the LWCC strictly follows Beer's law:

 $A=\mathbf{E}\cdot\mathbf{C}\cdot\lambda$

where A is absorbance, $\boldsymbol{\mathcal{E}}$ is the absorption coefficient, C is concentration and λ is the optical pathlength of the sample cell.



UltraPath's four pathlengths (2, 10, 50 and 200 cm) are manufactured with a tolerance of \pm 2 mm and calibrated experimentally with a dye solution (Phenol Red).

The pathlength marked on the back of the instrument's enclosure is its calibrated effective pathlength.

By Beer's Law, the absorption of a liquid sample in a long pathlength sample cell bears a linear relationship to the concentration of an analyte. WPI's sample cells based on Liquid Waveguide Technology were extensively tested and proved to be linear over a range 0.01 to 2.0 AU (limited only by noise and stray light from the measuring spectrophotometer).

Relevant Literature

A detailed analysis of the effective pathlength and linearity of WPI's UltraPath sample cell and WPI's waveguide technology has been published in the following papers:

Richard L. Miller, Mathias Belz, Carlos Del Castillo, Rick Trzaska, "Determining CDOM Absorption Spectra in Diverse Coastal Environments Using a Multiple Pathlength, Liquid Core Waveguide System", *Continental Shelf Research*, July 2002, **22**:9, p 1301-1310.

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



Instrument Maintenance

Cleaning procedure for Ultrapath

It is good practice to keep track of the light performance of the sensor cell by frequently storing the reference intensities of all four pathlengths (2, 10, 50 and 200 cm) — observed with Millipore water. If variations in the baseline of more than 5-10 mAU are observed between measurements of the same sample, the cell should be cleaned. The stored reference signals can then be used as a guideline for how clean the cell is and how much additional cleaning is necessary.

Reference light intensity scans of all four pathlengths are supplied with each UltraPath sampling system. These scans were taken with Millipore water after the cells were rigorously cleaned. It is recommended that the user confirm these after first receiving the instrument.

A cleaning kit (WPI **#501609**) is supplied with the Ultrapath system. It consists of three solutions numbered 1, 2, and 3. Organic contamination can be cleaned very efficiently using these solutions in sequence, as described below. The following cleaning cycle has been optimized for organic (*e.g.*, CDOM-type) contamination; however, depending on your contaminant, the cleaning cycle may have to be altered. For thorough cleaning cycles, the UltraPath sample cell should be switched to the 200 cm optical pathlength. This will ensure, that the complete cell is cleaned. For intermediate cleaning between samples, the sample cell should be switched to the pathlength used in the experiments.

Cleaning cycle:

- 1. Rinse the cell thoroughly using ultra pure water. Obtain a new reference intensity and take a baseline absorbance reading.
- 2. Inject 3 injector volumes, separated by air bubbles, of Solution 1 "Waveguide Cleaning solution" followed by Solution 2 "Methanol solution" and then Solution 3 "HCl solution"
- 3. Then re-fill the UltraPath with 1-2 cell volumes of Millipore water for reference and comparison with initial absorbance signal.



- 4. Repeat (2) until scans show "little" or no change. NOTE: Use scans for pathlength = 200 cm cell as criteria for determining a clean system.
- Record light spectrum for each pathlength (2, 10, 50, and 200 cm) in a separate file, record integration time for each pathlength.
 NOTE: Set integration times such that maximum counts do not exceed 45,000 to 50,000 counts to allow for negative baseline offsets in the 200 cm cell caused by sample salinity.

Note:

Experiments have shown that three to five cleaning cycles were sufficient to clean the cell or organic contamination. In extreme cases (for example, CDOM left in the UltraPath cell overnight), it was found that filling the waveguide with Solution 1 and letting it sit for several minutes (*e.g.*, 10 minutes) and then subsequently flushing with Solutions 2 and 3 were necessary to remove contamination at the cell wall.

All solvents used should be HPLC grade.



Self-Test

It is advisable to perform a self test of the UltraPath sample system before sampling. Reference light intensity scans of all four pathlength are supplied with each UltraPath sampling system. These scans were taken with Millipore de-ionized water after the cells were rigorously cleaned. To test the UltraPath sample system:

- Switch on all instrumentation,
- Fill the UltraPath sample cell with Millipore de-ionized water and
- Obtain reference intensities at the integration times given in the calibration sheets (See Operation Instructions for details).
- Confirm the results with the calibration sheets.
- If the light intensities are significantly lower than on the calibration sheets, clean the sample cells (see section Instrument maintenance) and repeat the Self-Test.

Note:

Light intensity output will degrade over time, as a function of bulb age. The average lifetime of the FO-6000 bulb is 3000 hours. After 3000 hours, the light output will decrease to 50-70% of its original value.

Note:

If the color balance of the FO-6000 light bulb is changed or the bulb is exchanged, the calibration sheets become invalid and new calibration sets should be prepared.



Troubleshooting

Fault	Possible Cause	R e m e d y
UltraPath sample cell is leaking.	Nuts and ferrules are not tightened.	Check and tighten all Nuts and ferrules at the sample injector and the sample output of the UltraPath cell. Ensure that the ferrules are installed properly; hand tighten the nuts. If the ferrules are deformed, exchange them.
		3. If the tubing end of the Injector is deformed and the fitting ferrule does not have a snug fit, shorten the tubing 2-4 mm using a sharp razor blade.
		4. Exchange Injector if a tight seal cannot be obtained at the sample inlet.5. Exchange the silicone tubing assembly, if no seal can be obtained at the sample outlet.
Sample solution is not drawn into the Ultrapath sample cell.	Nuts and fittings are not tightened.	Remove fittings and insert them into the bulkhead fittings and hand-tighten them again.
	Silicone tubing is not pressed at the rollers in the pump head.	Silicone tubing is not properly set in the PeriStar pump head. See PeriStar instruction manual how to set install silicone tubing in the pump head
The pump is not drawing liquid, even silicone tubing is inserted directly into the sample vial.	Silicone tubing walls stick to each other.	The walls of silicone tubing have a tendency to stick to each other, as the tubing gets older and if the tubing is not removed from the pump head, when it is not used. Check and exchange the silicone pump tubing; further, ensure that the pressure applied by the pump head of the PeriStar tubing is optimized (see PeriStar manual for details).
There is no light or very little light at the TIDAS I detector	The sample cell is not filled with solution	Fill the sample cell with reference solution and check again
	Integration time is not setup correctly	Check in the calibration sheets, which integration time is required for the selected pathlength and set the appropriate time in the Spectralys software.
	Fibers are not connected properly	Check if all fiber optic cables are connected properly and the nuts are tightened. Open shutter of the light source
	Shutter of the light source is not open	If there is still no light at detectable at the TIDAS I output, follow the following test sequence:





There is still no light at the TIDAS I detector	Light source does not work	Check if the shutter at the light source is open by detaching the fiber optic cable at the light output. If no light exits the fiber optic collimator, check if the light source is switched on (see FO-6000 or D2H manual for a more detailed error analysis). Reconnect the fiber to the light output after the test. Note: DO NOT look directly into the light output. The light output is strong and may damage your eyesight. Wear UV-blocking safety glasses or goggles when working with the D2H (UPUV).
	Optical fiber (27) used to couple light from the FO-6000 light source to the UltraPath sample is broken	If light exits the light source, detach the fiber optic cable at the input of the UltraPath and check if light is transmitted through the fiber. Look for kinks in the fiber and replace it, if broken. Reconnect the fiber to the fiber optic input of the UltraPath sample cell after this test.
	UltraPath sample cell is contaminated	Fill the UltraPath with de-ionized water and disconnect the fiber at the fiber optic switch of the UltraPath. Check if light exits at the 2, 10, 50 or 200 cm output. This light will be dimmer than the light coupled into the sample cell. Note: DO NOT look directly into the light output. The light output is strong and may damage your eyesight. If no light exits the sample cell at the 2, 10, 50 or 200 cm cell, the cell is most probably contaminated. Refer to the Maintenance section how to clean the cell. The inside of the UltraPath sample cell is maintenance free and should not be opened by the end-user. Contact WPI if you expect damage inside the sample cell. Reconnect the optical fiber to the fiber optic output (switch) of the UltraPath sample cell, if light can be seen in the light output.
	Fiber optic cable (28) used to couple light from the UltraPath sample cell to the TIDAS I is defect	Detach the fiber optic cable at the TIDAS I input connector. Check if light exits the optical fiber. Check if there are kinks in the fiber or scratches on the fiber endface. Replace the fiber if you suspect damage.
	TIDAS I does not work properly	If light exits the optical fiber, set the integration time of the TIDAS I to approx. 200 ms and take a intensity scan with the fiber optic input of the instrument pointed against your room light to ensure its functionality. Alternatively, connect the TIDAS I with the 400 mm fiber directly to the FO-6000. See

Where to go for more help

If you have questions about any aspect of Ultrapath, you are welcome to contact WPI at (941) 371-1003. Business hours are from 9 to 5 Eastern Standard Time, every weekday. WPI's spectroscopy e-mail address is spectroscopy@wpiinc.com.



Storage

The UltraPath system should be stored empty at temperatures between $-20\,^{\circ}\text{C}$ to $60\,^{\circ}$ C, preferably at room temperature and moderate humidity. However, the UltraPath system is supplied with a liquid cleaning kit, see for storage details of the solutions its manual.





Accessories

500291 Peri-Star Peristaltic pump, 2 channels, 0.1-80 mL/min (110V)

500304 Peri-Star Peristaltic pump, 2 channels, 0.1-80 mL/min (240 V, CE)

500320 Silicone tubing, 1m length, 1.6 mm l.D., 1.6 mm wall thickness

72100 UltraPath injector kit

501609 Waveguide cleaning kit

15807 Cleaning solution concentrate (Refill for solution 1 of WPI# 501609)

FO-6000 FO-6000 fiber optic light source, 370 nm – 1700 nm

800120 Replacement lamp for FO-6000

D2H D2H Deuterium Halogen Light Source, 215 nm - 1700 nm

D2H-HB Halogen replacement lamp for D2H

D2H-DB Deuterium replacement lamp >215 nm

FO-400-SMA1M Fiber optic cable, 1m, 400 μm core diameter

FO-600-SMA1M Fiber optic cable, 1m, 600 μm core diameter

KIT-UPVIS-STARTUP FO-600-SMA1M, 501609, 72100, 800120, 15807

KIT-UPUV-STARTUP FO-600-SMA1M, 501609, 72100, D2H-DB, D2H-HP, 15807





Specifications

DYNAMIC RANGE 0.002 m⁻¹ – 230 m⁻¹ (Absorption)

5 μAU cm⁻¹- 1 AU cm⁻¹ (Absorbance)

OPTICAL PATHLENGTHS Selectable: 2 cm, 10 cm, 50 cm and 200 cm

WAVELENGTH RANGE

UPVIS 370 nm to 730 nm UPUV 250 nm to 730 nm

INNER DIAMETER ≈ 2 mm

CELL VOLUME ≈ 11 mL (at 200 cm pathlength)

SAMPLE INLET/OUTLET Quick Connect fittings, 1/8"

FIBER INPUT 600 µm core diameter, SMA

FIBER OUTPUT 600 µm core diameter, SMA

SOLVENT RESISTANCE Most organic and inorganic solvents

SHIPPING WEIGHT 33 lb (15 kg)

For specifications of the spectrometer module (TIDAS I), light source (FO-6000, D2H-2 for UV-upgraded version) and peristaltic pump (Peri-Star Pro), refer to the specific manuals.



References

Ultrapath

Richard L. Miller, Mathias Belz, Carlos Del Castillo, Rick Trzaska, "Determining CDOM Absorption Spectra in Diverse Coastal Environments Using a Multiple Pathlength, Liquid Core Waveguide System", Continental Shelf Research, [in press] (July 2002) Vol. 22, Issue 9, p 1301-1310.

Richard L. Miller, Mathias Belz and Su Yi Liu, "Measuring the absorption of CDOM in the field using a multiple pathlength liquid waveguide system", Ocean Optics XV, paper 1308, Monaco, October 2000.

Related 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.

- E. J. D'Sa, G. J. Kirkpatrick, S. Y. Liu, "Application of a long path-length aqueous capillary waveguide for seawater absorption spectral measurements", Ocean Sciences Meeting of the American Geophysical Union, San Diego, CA, EOS Transactions, (1998), 76(3):OS34.
- G. J. Kirkpatrick, O. M. Schofield, D. F. Millie, M. A. Moline, "Optical discrimination of phytoplankton species in mixed populations", Limnol Oceanogr., 45(2), 467-471.
- E. J. D'Sa, S. E. Lohrenz, C. L. Carroll, H. Fein, "Liquid waveguide capillary flow





cell for determining absorption of scattering suspensions: Comparison with an integrating sphere", to appear in Proceedings of the Society of Photo-optical Instrumentation Engineers (SPIE), Ocean Optics XIV, paper presentation: Hawaii, November 13, 1998.

P. K. Dasgupta, Z. Genfa, S. K. Poruthoor, S. Caldwell, S. Dong, and S.-Y. Liu, "High-Sensitivity Gas Sensors Based on Gas-Permeable Liquid Core Waveguides and Long-Path Absorbance Detection", Anal. Chem., (1998), Vol. 70 pp. 4661-4669.

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 UK Product Review, May 1997, page 9.



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

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

* 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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