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## SYSTEM GUIDE

for visible and ultraviolet (UPUV-2) systems

# UltraPath 2

*Multiple pathlength sample cell for absorbance  
spectroscopy with extended dynamic range*

Serial No. \_\_\_\_\_

[www.wpi-europe.com](http://www.wpi-europe.com)

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**CONTENTS**

GENERAL WARNINGS AND CAUTIONS..... 3

INTRODUCTION ..... 4

INSTRUMENT DESCRIPTION ..... 5

    UltraPath 2 Sample Cell..... 6

    TIDAS S 300 UV/VIS Spectrometer ..... 7

    Fiber Optic Cables ..... 8

    Peri-Star Pro Peristaltic Pump ..... 8

    Cleaning Kit (Available US Only) ..... 9

    LWCC Injection System..... 9

Setting up UltraPath 2 ..... 10

    Parts List..... 10

    Opening the package ..... 10

    Assembling the UltraPath 2 system ..... 12

    Installing the software ..... 13

Setting up QFT2 Glass Fiber Filter (GF/F) & cuvette holder ..... 14

Using UltraPath 2..... 15

    How to fill the UltraPath 2 sample cell..... 15

    Acquiring Data with TIDASDAQ – a typical measurement cycle..... 16

    Refractive index sensitivity of the UltraPath 2 sample cell ..... 18

    - salinity matched reference solutions to avoid baseline offsets ..... 18

    Measurement reliability..... 18

    Flow rate and maximum pressure..... 18

    Effective pathlength and linearity ..... 19

Using the QFT2 cuvette holder for particulate absorption measurements ..... 20

    Background ..... 20

    Taking particulate absorption measurements with a GF/F pad ..... 21

    Taking cuvette measurements ..... 21

    Relevant Literature for particulate absorption measurements ..... 22

Instrument Maintenance..... 23

Self-Test..... 25

Troubleshooting ..... 26

Storage ..... 28

Accessories ..... 29

Specifications..... 30

References..... 31

WARRANTY ..... 33

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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 2 sampling system contains cleaning solutions. only qualified personnel should use these solutions. Please refer to the SDS Sheets for details on the solutions.

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

## INTRODUCTION

The UltraPath 2 sampling system is a unique high-performance spectrophotometer offering user-selectable optical path lengths of 10, 50 and 200 cm. Designed for the detection of low absorbing species in solution, UltraPath 2 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 220 to 720 nm (UPUV-2) and has an exceptional dynamic range allowing reliable absorbance measurements between  $5\mu\text{AU}/\text{cm}$  to  $1\text{ AU}/\text{cm}$  to be routinely made.

The UltraPath system 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 freshwater environments. It 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.*,  $0.007\text{ m}^{-1}$  at 380 nm), and high CDOM freshwater environments (*e.g.*,  $10\text{--}20\text{ m}^{-1}$  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.

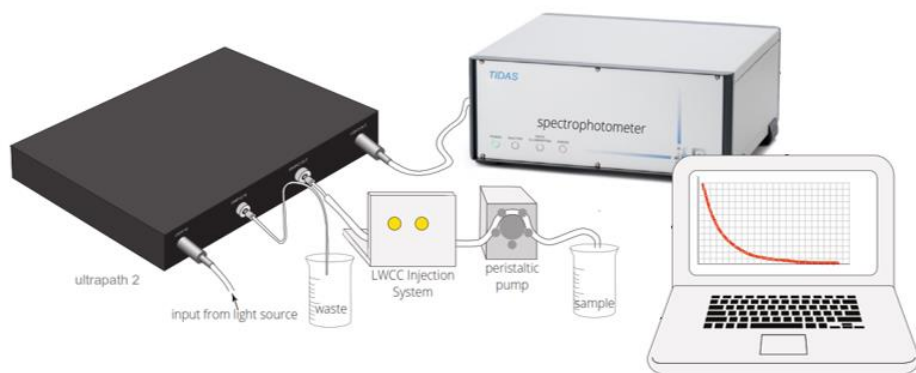


The UltraPath 2 sample cell has three optical pathlengths contained within a single sample cell (10 cm, 50 cm and 200 cm). The pathlengths are user selectable, offering a very high sensitivity and an extended dynamic range for VIS and UV absorbance measurements. The fluid path of the sample cell is optimized 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 2 system includes a photodiode array-based spectrometer module (**TIDAS S 300 UV/VIS**) to measure the absorbance at the selected pathlength. Absorption of light is measured between 190 to 720 nm with a resolution of 4 nm (FWHM) and a noise level below 0.2 mAU. The spectrometer includes an integrated deuterium/halogen light source which delivers light via the supplied fiber optic cables to the **UltraPath 2** sample cell. A peristaltic pump (**Peri-Star Pro**) is utilized to evenly draw sample up into the **Sample Injector** and through the UltraPath 2 sample cell.

The spectrophotometer is connected to the network via an ethernet port. The **TIDASDAQ/SpectraView** software (included) is installed on a standard Windows PC or laptop (not included) and connects to the TIDAS via the network. The TIDASDAQ/SpectraView software 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 on the USB drive for a detailed description of the software supplied with the instrument.)



## UltraPath 2 Sample Cell

1. **Fiber optic input connector**
2. **Liquid Input connector**
3. **Liquid Output Connector.** Sample is removed from the UltraPath 2 sample cell via the Liquid Output Connector with vacuum suction of the Peri-Star Pro peristaltic pump.
4. **Fiber Optic Switch & Output Connector.** The optical pathlength of the UltraPath 2 can be switched between 10, 50 and 200 cm. Pull the knob and turn to the selected pathlength
5. **UltraPath 2 Sample Injector Kit.** The Sample Injector (WPI #58006) is connected to the Liquid Input connector (7). Sample is drawn into the UltraPath 2 sample cell with the Sample Injector using vacuum suction of the Peri-Star Pro peristaltic pump.
6. **Silicone pump tubing** (WPI# 500320 – part of the sample injector assembly kit WPI# 58006).
7. **Waste Bottle** for expelled sample (included with WPI# 89372-2 LWCC Injection Kit).



## TIDAS S 300 UV/VIS Spectrometer

8. **Status LED.** LED's indicating the TIDAS S 300 UV/VIS state.
9. **Error LED Indicator (Red).** The Error LED lights up when the TIDAS S 300 UV/VIS is in a fault state. On system start-up the error LED is on for up to 3 seconds.
10. **Front Power Switch (ON/OFF).** When the power switch is turned ON, power is supplied to the TIDAS S 300 UV/VIS spectrometer module and this LED will be lit.
11. **Fiber optic input (SMA type).** The Fiber optic input connects to the light output of the UltraPath 2 sample cell (4).
12. **Debug RS-232.** Interface for system diagnostics (connectors supplied). For further instructions refer to TIDAS S 300 UV/VIS instruction manual.
13. **LAN RJ45 ethernet communication interface.** The TIDAS S 300 UV/VIS is connected to a network via the supplied ethernet cable. For further instructions refer to TIDAS S 300 UV/VIS instruction manual.
14. **Shutter Button:** Pushing the shutter button the shutter is toggled. If the shutter is open the LED is lit.



15. **Cooling Fan.** The fan dissipated the heat generated by the power supply and electronics of the TIDAS S 300 UV/VIS. To ensure proper ventilation, the unit should be situated away from walls or panels. Do not obstruct the cooling fan openings.
16. **Input terminal for power cord and fuse.** The TIDAS S 300 UV/VIS accepts voltages from 80 to 240 VAC, 50-60Hz. Use correct cord and fuse, as described in the manual.

17. **Light output.** SMA Fiber Optic Connector (collimator) with adjacent mounting screws.

## Fiber Optic Cables

18. **Fiber optic cable** — Light is coupled into the fiber optic input connector (1) of the UltraPath 2 sample cell from the TIDAS S 300 UV/VIS light output (22) with a 600  $\mu\text{m}$  core fiber (**WVLXDUV-S-0600-SMA**).
19. **Fiber optic cable** — Light is coupled from the output of the UltraPath 2 fiber optic switch (4) into the TIDAS S 300 UV/VIS spectrometer Input (11) with a 600  $\mu\text{m}$  core fused silica fiber (**WVLXDUV-S-0600-SMA**).



## Peri-Star Pro Peristaltic Pump

20. **Peri-Star Pro Pump Head.** Two channels and eight rollers.
21. **Control Panel.** The Control Panel allows to start the pump clockwise and counterclockwise, 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).



22. **Power Switch (ON/OFF).** When the power switch is turned ON, power is supplied to the Peri-Star Pro peristaltic pump



- 23. **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.
- 24. **Input terminal for power cord.**
- 25. **External Control socket.** DB-15 connection to control speed and direction of the pump head. Refer to the instruction manual for details.

## Cleaning Kit (Available US Only)

- 26. **Waveguide Cleaning Kit.** The waveguide cleaning kit contains three solutions specifically developed to keep the UltraPath 2 sample cell clean and ensure a high measurement accuracy and repeatability.



26

## LWCC Injection System

- 27. **6 Port Injection Valve**
- 28. **6 Port Selection Valve**
- 29. **LWCC Injection System Connection Kit** to connect between the switch and samples, reference solutions and cleaning solutions on the LWCC injection system.



For a detailed description of the TIDAS S 300 UV/VIS and Peri-Star Pro, refer to the relevant manuals.

## Setting up UltraPath 2

UltraPath 2 is a modular spectrophotometer system. Its main components are: UltraPath 2 sample cell and TIDAS S 300 UV/VIS spectrometer module. The assembly of the UltraPath 2 system is described below.

### Parts List

Part Number	Quantity	Description
UPATH2-CELL	1	UltraPath 2 Cell
TIDAS S 300 UV/VIS	1	UV/VIS Spectrometer system (190-720nm) including software, DH Lightsource.
WVLUXDUV-S-0600-100-SMA	2	DUV Fiber (200-1200nm) 600um 100cm SMA
WVLUXDUV-S-0200-100-SMA	1	DUV Fiber (200-1200nm) 200um 100cm SMA
13395	2	SMA Bulkhead Feed Thru Connector, D-hole
58006	1	Sample Injector Assembly for LWCC
58450	1	Kit, Adapter Syringe, LWCC
89372-2	1	LWCC Injection System
LOOP-5ML	1	Sample Loop Kit, 5 mL (2.5m PFA Tubing 1/16" ID x 1/8" OD + 2 x PEEK fittings 1/8" OD Tubing x 1/4"-28 UNF)
PERIPRO-4LS	1	Peristaltic Pump, 4 Channel, low speed
QFT2*	1	25mm GFF Filter and 10mm Cuvette Holder, SMA
CUV2012-1*	1	Quartz Cuvette, Style C, 12.4x12.4x45mm, 10mm, 3.5ml

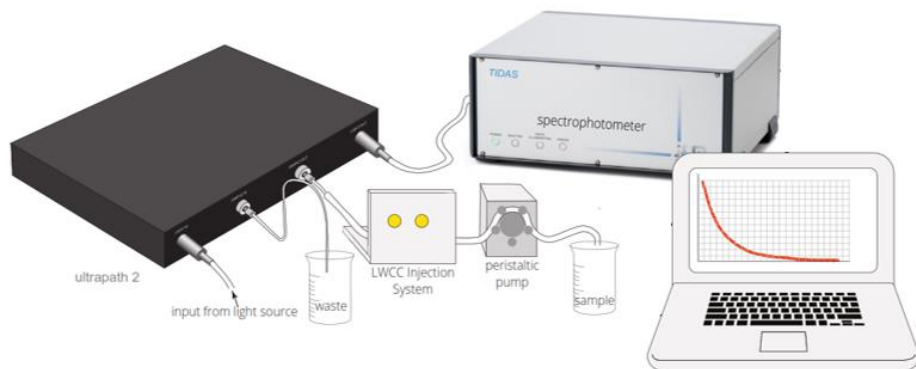
**\* Used when you need to measure particulate absorption on glass fiber filter pads (see Setting up QFT2 Glass Fiber Filter section) – not used with the UltraPath 2 System setup.**

### 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.

## Assembling the UltraPath 2 system



1. Prepare a clear workspace for all components.
2. From left to right place the following components on the workspace: UltraPath 2 sample cell, LWCC injection system, Peri-Star Pro peristaltic pump and TIDAS S 300 UV/VIS spectrometer.
3. Use a 600  $\mu\text{m}$  core diameter fiber optic cable (18) to connect the light output (17) of the TIDAS S 300 UV/VIS spectrometer to the fiber optic input connector (1) of the UltraPath 2 sample cell.
4. Use a 600  $\mu\text{m}$  core diameter fiber optic cable (19) to connect the light output (4) of the UltraPath 2 Fiber Optic Switch to the fiber optic input (11) of the TIDAS S 300 UV/VIS spectrometer.
5. Connect the TIDAS S 300 UV/VIS to the network via the RJ45 connector.
6. Connect the Power cord supplied with the to the TIDAS S 300 UV/VIS to the Input Terminal (16) of the TIDAS S 300 UV/VIS and the mains.
7. Assemble the sample injector loop of the UltraPath 2 Sample Injector Kit (5) by sliding on a nut and a ferrule and then screwing the assembly into the bulkhead fitting labelled "liquid in" (2) of the UltraPath 2 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!  
Place the injector into a sample vial filled with de-ionized water.
8. Use the silicone pump tubing (6) to connect the liquid output port (3) of the UltraPath 2 sample cell via the Peri-Star Pro peristaltic pump to the Waste bottle (11) (See Peri-Star Pro instruction manual for details).
9. Turn on the Peri-Star Pro and the TIDAS S 300 UV/VIS — allow lamp to warm up at least 15 minutes before sampling.

Note:

The 89372-2 LWCC Injections system together with the LOOP-5ML can be used in the setup to provide an efficient, continual flow for injecting a sample through the UltraPath 2. It minimizes contamination and the formation of tiny bubbles that interfere with spectroscopic data recording. (See the LWCC Injection System Manual for connection details)

The QFT2 and CUV2012-1 are not part of the standard setup. They are used when you need to measure particulate absorption on glass fiber filter pads (see section on Setting up QFT2 Glass Fiber Filter (GF/F) & cuvette holder)

## **Installing the software**

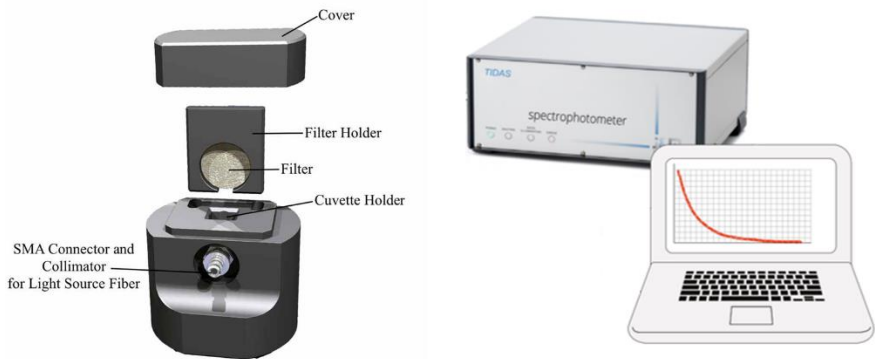
For hardware and software installation of the TIDAS S300, please refer to included TIDAS S User Manual and the TIDAS DAQ3 User Manual.

# Setting up QFT2 Glass Fiber Filter (GF/F) & cuvette holder

The components required to measure particulate absorption on glass fiber filter pads are:

TIDAS S 300 UV/VIS	1	UV/VIS Spectrometer system (190-720 nm) including software, DH Lightsource.
WVLUXUVIS-S-1000-100-SMA	1	UVIS Fiber (260-1200 nm) 1000 $\mu$ m 100 cm SMA
WVLUXUVIS-S-0600-100-SMA	1	UVIS Fiber (200-1200nm) 600 $\mu$ m 100 cm SMA
QFT2	1	25 mm Glass Fiber Filter (GF/F) and 10mm Cuvette Holder
CUV2012-1	1	Quartz Cuvette, Style C, 12.4x12.4x45 mm, 10 mm, 3.5 ml

The light output of the TIDAS spectrophotometer is connected to one optical port of the QFT2 using the UVIS Fiber with a core diameter of 1000  $\mu$ m. The second port of the QFT2 is connected to the detector input of the TIDAS spectrophotometer with the UVIS Fiber with a core diameter of 600  $\mu$ m.



## Using UltraPath 2



UltraPath 2 is a very sensitive device for measuring low absorbing substances in solution.

The following instructions will familiarize you with using and cleaning the UltraPath 2 system.

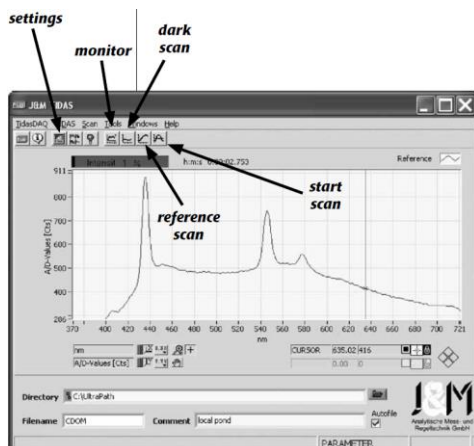
- Switch the TIDAS S 300 UV/VIS ON (10) and start the TIDASDAQ software. Allow its light source approximately 15 minutes warming up time for the lamp to stabilize its temperature drift ( $<0.5\text{mAU/h}$ ). For test purposes, the lamp may be used earlier. Open the shutter (14) at the light source to take measurements. Close the shutter when a dark current has to be taken.
- Switch the pump (Peri-Star Pro) ON (22) and with size #14 tubing set the pump speed to 60 RPM or 12mL/min via the control panel (21). The sample is drawn into the waveguide by vacuum suction.
- Select a pathlength (10, 50, 200 cm) at the fiber optic switch (4) of the UltraPath 2 sample cell.

### How to fill the UltraPath 2 sample cell

- Place the injector (5) into a sample vial. The sample is drawn with the peristaltic pump (Peri-Star Pro) into the UltraPath 2 sample cell by vacuum suction.
- Press the forward button at the control panel (21) 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 10cm or 50cm cell, a pumping time of approx. 30 seconds is necessary, using the 200cm path, a pumping time of approximately 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 190-720 nm

**Representation** to **Absorbance**,

**Scan Type** to **Time scan**, **Integration time** to **240 ms**

**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 S 300 UV/VIS. A maximum of 45000 cts should be observed, when using MilliQ water in the UltraPath 2 sample as a reference. Typical integration times are 100 to 2000ms.

### **Sample collection**

1. Close the shutter at the light source (14) and take a **Dark Scan**
2. Open the shutter (14) 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 2 sample cell (See “How to fill the UltraPath 2 sample cell” for details).
5. Introduce an air bubble of approximately the length of the Injector tubing at the liquid input of the UltraPath 2 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 2 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 2 sample cell**

### **- salinity matched reference solutions to avoid baseline offsets**

The UltraPath 2 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 than 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 2 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 2 sample cell are 12 mL/ min (equal to 60 RPM with the Peri-Star Pro pump using size #14 tubing). Drawing liquid into the cell via the injector fills the sample cell. The UltraPath 2 sample cell is designed for low pressure operations and should not be used at pressures greater than 75 PSI.

## Effective pathlength and linearity

Effective pathlength and linearity have been extensively studied with WPI's Liquid waveguide capillary cell technology and the UltraPath 2 system. "Effective pathlength" is defined as the equivalent pathlength of the cell if we assume the LWCC strictly follows Beer's law:

$$A = \varepsilon * C * \lambda$$

where A is absorbance,  $\varepsilon$  is the absorption coefficient, C is concentration and  $\lambda$  is the optical pathlength of the sample cell. UltraPath 2's three pathlengths (10, 50 and 200 cm) are manufactured with a tolerance of  $\pm 2$  mm and calibrated experimentally with a dye solution (Amaranth).

***The pathlength marked on the QS-Sheet supplied with UPATH-2 flowcell 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 2 sample cell and WPI's waveguide technology has been published in the following papers:

Richard L. Miller, Mathias Belz, Carlos Del Castillo, Rick Trzaska,

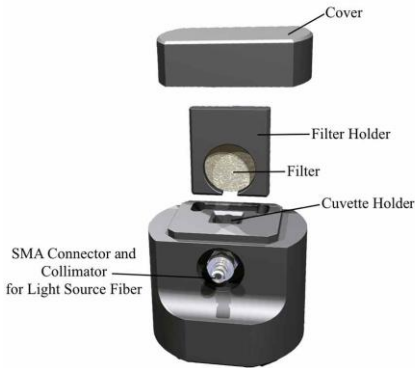
"Determining CDOM Absorption Spectra in Diverse Coastal Environments Using a Multiple Pathlength, Liquid Core Waveguide System", *Continental Shelf Research*, July 2002, 22:9, p 1301-1310.

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.

# Using the QFT2 cuvette holder for particulate absorption measurements

## Background

The rugged and portable QFT2 is specially designed for measuring absorption of particulate concentrated on Glass Fiber Filter (GF/F) Pads and for simple cuvette measurements in 10 mm cuvettes. Instead of collecting samples and transporting them to a laboratory, samples can be measured on-site.



Particulate absorption of fresh water and seawater can be determined by filtering a known amount of sample through a Glass Fiber Filter (GF/F) and measuring the particulate absorption coefficient  $a_p(\lambda)$  concentrated on the filter. This technique is called quantitative filter technique (QFT) and corrects for the pathlength amplification, an effect of scattering. The correction of the pathlength amplification and the correction of the non-linear relationship between the optical density of samples on a Whatman GF/F filter and in suspension are discussed in Mitchell (1990). (See "References" on page 9). Mitchell's report results in an equation to calculate the corrected particulate absorption coefficient  $a_p(\lambda)$  as follows:

$$a_p(\lambda) = 2.3 * \frac{a * a_p^*(\lambda) + b * (a_p^*(\lambda))^2}{\frac{V_f}{A_c}}$$

with

$a_p^*(\lambda)$  = uncorrected optical density of the collected sample on the GF/F pad

$V_f$  = filtered volume

$A_c$  = optical clearance area on the GF/F Pad

Coefficients a and b are filter-type dependent (Whatman GF/F filters: a = 0.392, b = 0.665).

### **Taking particulate absorption measurements with a GF/F pad**

- Wetten a GF/F Filter pad with de-ionized water and place it into the filter pad holder of the QFT2 and cover it with the lid
- Take a reference scan with the TIDAS S300 spectrophotometer and adjust the integration time to measure approx. 50.000 counts at the maximum intensity level, as described in the TIDAS DAQ3 user manual. This is typically achieved with an integration time of 3 seconds (3000 milliseconds).
- Filter a known amount of natural sea or fresh water sample through the GF/F filter pad, as described in Mitchell et al.
- Re-insert the filter pad in the filter holder of the QFT2 and take an absorbance reading, as described in the TIDAS DAQ3 user manual.
- Save the scan to your hard drive, import the data into e.g. Excel and calculate the corrected particulate absorption coefficient  $a_p(\lambda)$  with the equation described above.

### **Taking cuvette measurements**

- Fill your reference solution into the 10 mm fused silica cuvette supplied with the instrument.
- Verify that the filter pad holder has NO GF/F filter pad installed
- Insert the cuvette with reference solution into the space for the cuvette and cover it with the supplied lid
- Take a reference scan with the TIDAS S300 spectrophotometer and adjust the integration time to measure approx. 50.000 counts at the maximum intensity level, as described in the TIDAS DAQ3 user manual. Without a GF/F pad, this is typically achieved with an integration time below 100 milliseconds.
- Remove the cuvette, fill it with sample solution, re-insert it back into the cuvette holder and close the lid.
- Take an absorbance reading, as described in the TIDAS DAQ3 user manual.

## Relevant Literature for particulate absorption measurements

A detailed description on measuring particulate absorption of fresh water and seawater with Glass Fiber Filters (GF/F) and determining the particulate absorption coefficient  $a_p(\lambda)$  concentrated on the filter has been published in the following papers:

Mitchell, B. G., "Algorithms for Determining the Absorption Coefficient of Aquatic Particles Using the Quantitative Filter Technique (QFT)", SPIE Vol. 1302 Ocean Optics X (1990), 137-148.

Sosik, H. M., "Storage of marine particulate samples for light-absorption measurements", Limnol. Oceanogr., 44(4), 1999, 1139-1141

Belz, M., Larsen, K., Klein, F. "Fiber optic sample cells for polychromatic detection of dissolved and particulate matter in natural waters", Proc. SPIE, Vol. 6377, Oct 2006, 63770X

# Instrument Maintenance

## Cleaning procedure for UltraPath 2

It is good practice to keep track of the light performance of the sensor cell by frequently storing the reference intensities of all three pathlengths (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 re-filled 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 three pathlengths are supplied with each UltraPath 2 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.

The optional cleaning kit (**WPI #501609**) can be used with the UltraPath 2 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 2 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 2 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 = 200cm cell as criteria for determining a clean system
5. Record light spectrum for each pathlength (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 2 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 2 sample system before sampling. Reference light intensity scans of all three pathlength are supplied with each UltraPath 2 sampling system. These scans were taken with Millipore de-ionized water after the cells were rigorously cleaned. To test the UltraPath 2 sample system:

- Switch on all instrumentation,
- Fill the UltraPath 2 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 both the UV bulb and VIS bulb is 3000 hours. After this time the light output will decrease to 50-70% of its original value.

### **Note:**

If the color balance of the TIDAS S 300 UV/VIS bulb is changed or the bulb is exchanged, the calibration sheets become invalid and new calibration sets should be prepared.

## Troubleshooting

FAULT	Possible Cause	Remedy
UltraPath 2 sample cell is leaking.	Nuts and ferrules are not tightened.	<ol style="list-style-type: none"> <li>1 . Check and tighten all Nuts and ferrules at the sample injector and the sample output of the UltraPath 2 cell. Ensure that the ferrules are installed properly; hand tighten the nuts.</li> <li>2 . If the ferrules are deformed, exchange them.</li> <li>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.</li> <li>4 . Exchange Injector if a tight seal cannot be obtained at the sample inlet.</li> <li>5 . Exchange the silicone tubing assembly, if no seal can be obtained at the sample outlet.</li> </ol>
Sample solution is not drawn into the UltraPath 2 sample cell.	Nuts and Fittings are not tightened.	Remove fittings and insert them into the bulkhead fittings and hand-tighten them again.
Sample solution is not drawn into the UltraPath 2 sample cell.	Silicone tubing is not pressed at the rollers in the pump head.	Silicone tubing is not properly set in the PeriStar Pro pump head. See PeriStar Pro instruction manual how to set ins tall 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 may stick to each other as the tubing gets older and if the tubing is not removed from the pump head, when not in use. Check and exchange the silicone pump tubing; further, ensure that the pressure applied by the pump head of the PeriStar Pro tubing is optimized (see PeriStar Pro manual for details).
There is no light or very little light at the TIDAS S 300 UV/VIS detector	The sample cell is not filled with solution	Fill the sample cell with reference solution and check again
There is no light or very little light at the TIDAS S 300 UV/VIS detector	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.

There is no light or very little light at the TIDAS S 300 UV/VIS detector	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
There is no light or very little light at the TIDAS S 300 UV/VIS detector	Shutter of the light source is not open	If there is still no light at detectable at the TIDAS S 300 UV/VIS output, follow the following test sequence:
There is still no light at the TIDAS S 300 UV/VIS detector	Optical fiber (18) used to couple light from the TIDAS S 300 UV/VIS light source to the UltraPath 2 sample is broken	If light exits the light source, detach the fiber optic cable at the input of the UltraPath 2 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 2 sample cell after this test.
There is still no light at the TIDAS S 300 UV/VIS detector	UltraPath 2 sample cell is contaminated	<p>Fill the UltraPath 2 with de-ionized water and disconnect the fiber at the fiber optic switch of the UltraPath 2. Check if light exits at the 10, 50 or 200 cm output. This light will be dimmer than the light coupled into the sample cell.</p> <p>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 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 2 sample cell is maintenance free and should not be opened by the end-user.</p> <p>Contact WPI if you expect damage inside the sample cell. Reconnect the optical fiber to the fiber optic output (switch) of the UltraPath 2 sample cell, if light can be seen in the light output.</p>
There is still no light at the TIDAS S 300 UV/VIS detector	Fiber optic cable (19) used to couple light from the UltraPath 2 sample cell to the TIDAS S 300 UV/VIS is defect	Detach the fiber optic cable at the TIDAS S 300 UV/VIS input connector. Check if light exits the optical fiber. Check if there are kinks in the fiber or scratches on the fiber end face. Replace the fiber if you suspect damage.

There is still no light at the TIDAS S 300 UV/VIS detector	TIDAS S 300 UV/VIS does not work properly	If light exits the optical fiber, set the integration time of the TIDAS S 300 UV/VIS to approx. 200 ms and take an intensity scan with the fiber optic input of the instrument pointed against your room light to ensure its functionality.
Using the QFT 2, there is no light at the TIDAS S 300 UV/VIS detector	Integration time set too low	Set integration time to approx. 3000 ms, take an intensity reading. Check if highest intensity is approx. 50000 cts and redo experiment with different integration times until successful.
Using the QFT 2, there is still no light at the TIDAS S 300 UV/VIS detector	Fibers not properly connected to TIDAS S300 and the QFT2	Check if fibers are broken and that all fiber optic connectors are screwed into the QFT and the spectrometers finger-tight.

### Where to go for more help

If you have questions about any aspect of UltraPath 2, you are welcome to contact WPI at the offices specified on the back of the manual.

## Storage

The UltraPath 2 system should be stored empty at temperatures between 10 °C to 60 °C, preferably at room temperature and moderate humidity. However, the UltraPath 2 system is supplied with a liquid cleaning kit, see for storage details of the solutions its manual.

# Accessories

Part Number	Description
WVLUXDUV-S-0600-100-SMA	DUV Fiber (200-1200nm) 600um 100cm SMA
WVLUXDUV-S-0200-100-SMA	DUV Fiber (200-1200nm) 200um 100cm SMA
13395	SMA Bulkhead Feed Thru Connector, D-hole
58006	Sample Injector Assembly for UltaraPath2
58450	Kit, Adapter Syringe, LWCC
500320	Silicone tubing, 1m length, 1.6 mm I.D., 1.6 mm wall thickness
CUV2012-1	Quartz Cuvette, Style C, 12.4x12.4x45mm, 10mm, 3.5ml
501609	Waveguide Cleaning Kit (Available in US Only)
LOOP-5ML	Sample Loop Kit, 5 mL (2.5m PFA Tubing 1/16" ID x 1/8" OD + 2 x PEEK fittings 1/8" OD Tubing x 1/4" - 28 UNF)

## Specifications

Dynamic range	0.002 m-1 – 230 m-1 (Absorption) 5 $\mu$ AU cm-1- 1 AU cm-1 (Absorbance)
Optical pathlengths	Selectable: 10 cm, 50 cm and 200 cm
Wavelength range	220 nm to 720 nm
Inner diameter	$\approx$ 1 mm
Cell volume	$\approx$ 2,4 mL
Sample in let/outlet	Standard 10-32 Coned Port Fitting
Fiber input	600 $\mu$ m core diameter, SMA
Fiber Output	600 $\mu$ m core diameter, SMA
Solvent resistance	Most organic and inorganic solvents
Shipping weight	34 kg

*For specifications of the spectrometer module (TIDAS S 300 UV/VIS) and peristaltic pump (Peri-Star Pro), refer to the specific manuals.*

## References

### UltraPath 2

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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) 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.

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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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