

TITLE Dual-Wavelength Optical Density Analyses

Effective Date: _____

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Annual Review			
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Table of Contents

1.0 Purpose	3
2.0 Scope	3
2.1 Detection Limit	3
3.0 Summary of Method	3
3.1 Method Interferences	4
3.2 Personnel Qualifications	4
3.3 Deviations from the Method	5
4.0 Materials.....	6
5.0 Safety	6
6.0 Procedures	7
6.1 Start-Up.....	7
6.2 Instrument Performance Check.....	7
6.3 Calibration	8
6.4 Routine Operation	9
6.5 Shut-Down	10
6.6 Calculations.....	10
6.7 Data Analysis	11
6.8 Quality Control	11
7.0 References	11
8.0 Attachments	12
Figures	
Figure 1 Magee Scientific SootScan™ Model OT21 Transmissometer (a) and Filter Holder Assembly (b)	4
Figure 2. Flow diagram for light absorption (b_{abs}) of PM _{2.5} filter samples by Optical Transmissometer.....	5

1.0 Purpose

The purpose of the Standard Operating Procedure (SOP) is to provide consistent guidance to Wood Environment & Infrastructure Solutions, Inc. (Wood) laboratory personnel for the determination of black carbon (BC) using a Transmissometer for the Chemical Speciation Network (CSN).

2.0 Scope

Environmental Protection Agency (EPA) selected 47 mm Teflon (polytetrafluorethylene - PTFE) filters with an aerodynamic diameter less than or equal to 2.5 micrometers (PM_{2.5}) will be analyzed on a Magee SootScan™ Transmissometer. The transmissometer measures optical absorption (attenuation of light transmitted) through a filter at two wavelengths relative to a blank reference filter. The results are used to convert the light absorption into mass.

2.1 Detection Limit

The instrument manufacturer's recommended method detection limit (MDL), based on three standard deviations of the average blank, is 0.35 µg/filter, assuming 11.78 cm² deposit area of a 47 mm Teflon-membrane filter.

3.0 Summary of Method

The absorption of light (b_{abs} or optical attenuation) passing through a filter has long been used as a measure for black carbon (BC) in the aerosol deposit. Even through some mineral species also appear black, their absorption cross-section is small compared to that of BC. Light transmittance is measured on the blank filter (a reference laboratory blank) and the exposed filter after sampling. A Teflon-membrane filter is nearly transparent and gives a much more accurate measure of b_{abs} than an opaque quartz-fiber filter that scatters light internally rather than responding to the surface aerosol deposit.

PM_{2.5} b_{abs} will be measured by a dual-wavelength optical transmissometer (SootScan™ Model OT21). The OT21 shown in Figure 1, is equipped with a movable tray with two filter holder slots. Light transmittance is simultaneously measured for reference (blank) filter (inner tray Position 1) and sample (outer tray Position 2). An instrument performance check is performed at the beginning and end of each analytical session with a set of certified photometric standard glass disks. Each batch of 10 samples includes a QC check of one photometric standard. Replicate analyses are performed on 10% of the samples. See Section 6.8 for Quality Control (QC) requirements. A flow diagram of optical absorption calibration and measurement is shown in Figure 2. A standard glass disk is placed on top of a T60 diffuser filter for calibration to improve transmission sensitivity. A Teflon-membrane blank or sample is also placed on top of a diffuser filter for each analyses.

High correlation is expected between the optical BC and thermal Elemental Carbon (EC). Least-squares regression analysis can be used as part of Level II data validation performed external to the laboratory.

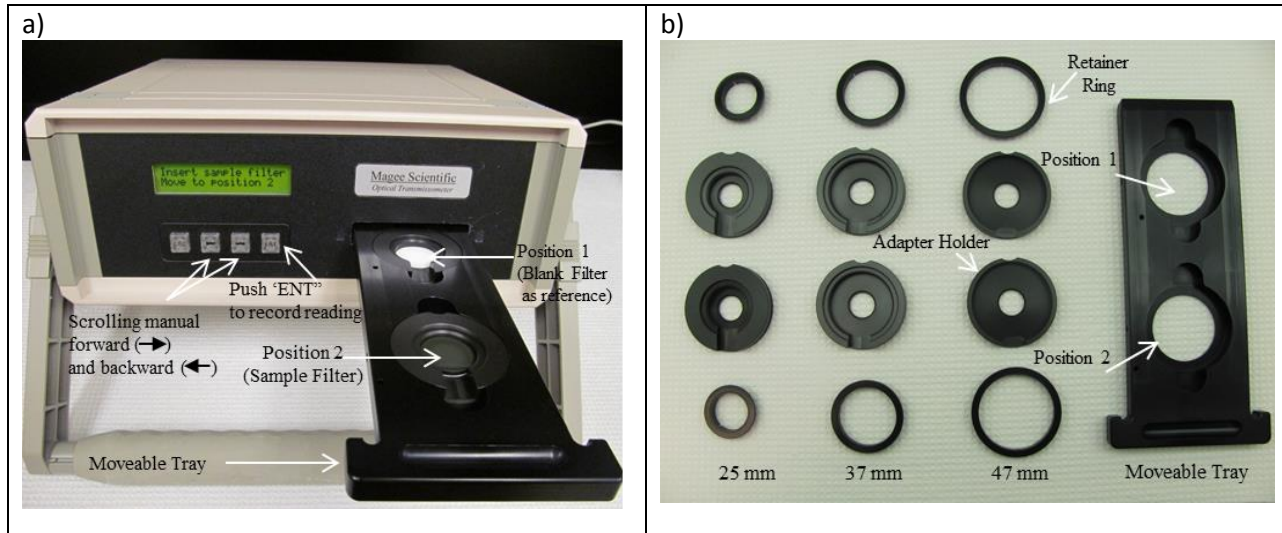


Figure 1. Magee Scientific SootScan™ Model OT21 Transmissometer (a) and Filter Holder Assembly (b)

3.1 Method Interferences

Representative blank filters from the same batch as the exposed samples need to be used as reference for the b_{abs} measurement to prevent bias.

The OT21 is sensitive to changes in the environment. Significant changes in ambient temperature (± 20 degrees Celsius) can alter measurement values. Measurements should not be performed if the temperature exceeds this range.

Keep the instrument away from dust, liquids and heat.

3.2 Personnel Qualifications

Personnel employed to perform optical density measurements must have a minimum of an associate's degree with at least 6 months experience in laboratory sample handling and record-keeping practices. Training is conducted by a lead analyst with a minimum of a bachelor's degree in a science related field and at least 6 months of experience with laboratory instrumentation (a minimum of 10 years laboratory instrumentation experience may be substituted for the degree requirement).

3.3 Deviations from the Method

A general overview of the steps described in this SOP is depicted in Figure 2. Deviations from the analytical method described in this SOP are not permitted.

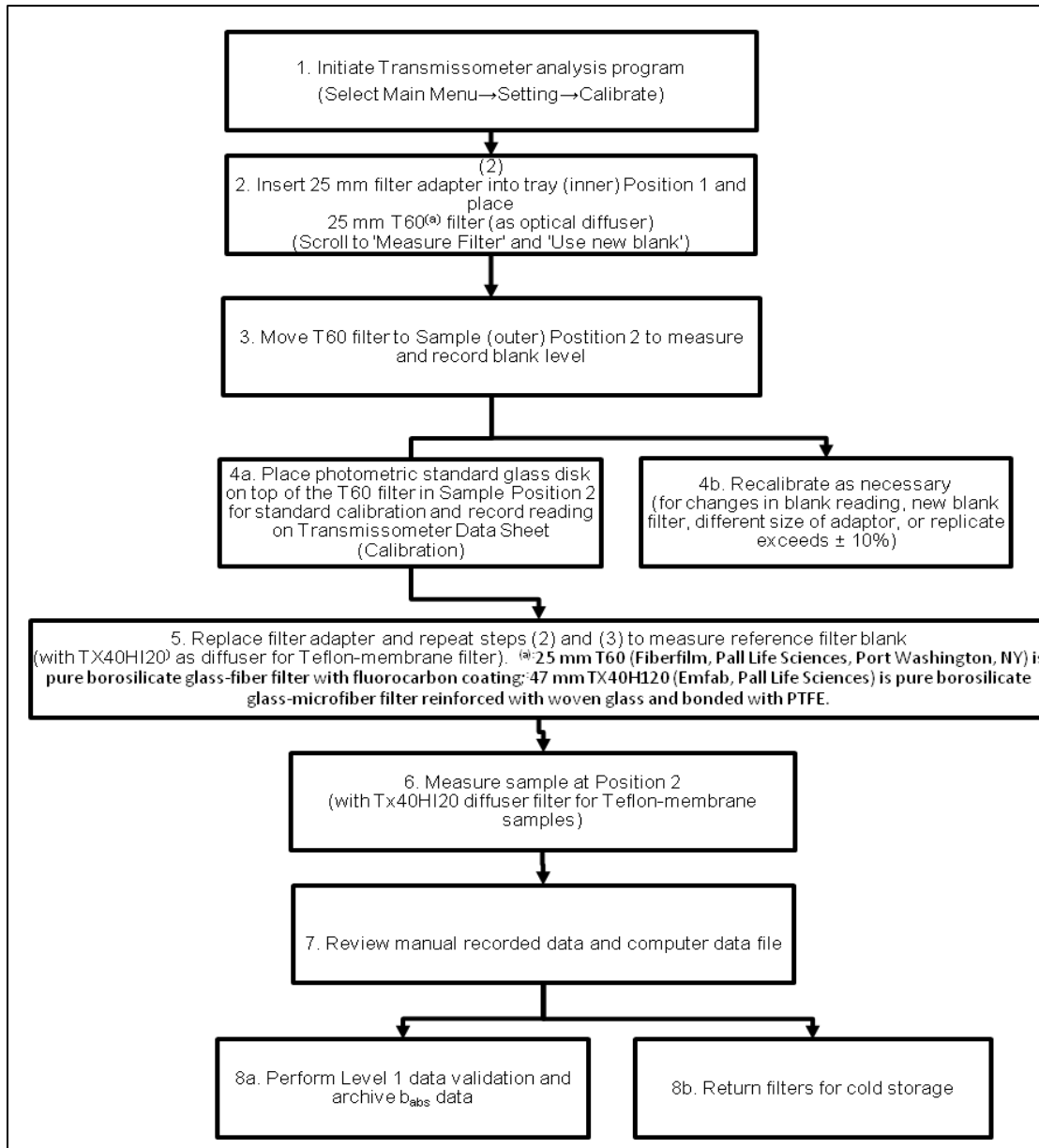


Figure 2. Flow diagram for light absorption (b_{abs}) of $PM_{2.5}$ filter samples by Optical Transmissometer

4.0 Materials

- Magee Scientific SootScan™ Model OT21 Transmissometer, Magee Scientific, Berkeley, CA
- Filter holder assembly for the Model OT21 that accommodates 25, 37, or 47 mm filters
- Transmissometer Neutral Density Filter Kit, Magee Scientific (certified photometric standard glass disks in the range of 0.15, 0.3, 0.4, and 0.6. Standard b_{abs} values for 370 nm (ultraviolet, UV) and for 880 nm (Infrared, IR) are supplied with each set.
- Reagent grade methanol. CAS# 67-56-1.
- Optical diffuser filters (47 mm TX40HI20)
- Teflon filters. 2- μm pore size PTFE (polytetrafluoroethylene) 47-mm membrane filters for $\text{PM}_{2.5}$
- Pall Membrane Filters (T60A20)
- Powder-free gloves
- Lab coat
- Clean, smooth (non-serrated) stainless steel, flat-tipped forceps

5.0 Safety

Proper laboratory procedures should be followed at all times to prevent accidents and to minimize sample contamination. No food or drinks are allowed in the area where the analysis is performed.

If the analyst is not familiar with the hazards associated with the methanol being used, the Safety Data Sheets (SDS) must be consulted. The SDS by chemical and brand can be found in the Wood laboratory or at the [SDS/MSDS search web site at https://www.msdonline.com/msds-search/](https://www.msdonline.com/msds-search/) or using the CAS number.

The SootScan™ OT21 light source contains an ultraviolet (UV) and infrared (IR) light emitting diode (LED). The LED sources radiates UV and IR light during operation. Precautions must be taken to prevent looking directly at the light source with unaided eyes. Never touch or stare directly into the OP21 light source.

6.0 Procedures

Exposed samples are received from the CSN Filter Shipping and Handling Unit (FiSH) with an internal chain of custody (see Attachment C) which provides the site information including the date collected and date received at Wood.

6.1 Start-Up

- Turn on the OT21 and allow machine to warm up for 15 minutes.
- Open computer icon labeled Transmissometer.

6.2 Instrument Performance Check

A Neutral Density (ND) check is performed at the beginning and the end of all measurements.

- Allow machine to warm up for 15 minutes.
- Clean sample tray and 25mm Filter Adapter holders with methanol.
- Insert 25mm Filter Adapter holders in sample tray.
- Scroll (using arrow buttons) through Main Menu to Settings. Press ENT button.
- Scroll through Settings to Calibrate. Press ENT. Screen will read “Calibrate: Press ENT...” Press ENT. Screen will read “Insert blank filter. Move to position 1.”
- Place 25mm T60 filter in reference holder. Gently push sample tray to position 1. Screen will read “Calibrating IR. Please wait...” then “Calibrating UV. Please wait...” When calibration is done, screen will return to Main Menu.
- Obtain a Dual-Wavelength Neutral Density Data Sheet (see Attachment A).
- Scroll to Measure Filter. Press ENT. Screen will read “Use new blank.” Press ENT. T60 filter is still in position 1. Screen will read “IR WARMING UP...” then “Measuring: BLANK filter IR...” Screen will read “UV WARMING UP...” then “Measuring: BLANK filter UV...” Screen will read “Insert sample filter. Move to position 2.”
- In computer enter “ND Disks” in field next to Use Description button. Click Use Description button. Entered description will appear in Description column. Enter “No ND disk” in Blank Filter ID column. Click box next to Use previous Blank Filter ID (a check mark will appear in box). Enter “No ND disk” in Filter ID column. If Description, Filter ID and Blank Filter ID columns are not filled in, the data will not go in the computer.
- On transmissometer screen will read “Insert sample filter. Move to position 2.” Remove T60 filter from reference holder using tweezers and place in sample holder. Push sample tray into position 2. Screen will read “IR WARMING UP...” then “Measuring: SAMPLE filter IR...” Screen will read “UV WARMING UP...” then “Measuring: BLANK filter UV...” When done measuring, screen will read UV ATTENUATION: 0000 and IR ATTENUATION: 0000. Values in computer should

be near zero. Record data from computer in columns ATN IR and ATN UV in ND Nominal Density table (at bottom of data sheet) in corresponding row.

- Pull sample tray out to expose sample holder. (Screen will still show “UV ATTENUATION: ##### and IR ATTENUATION: #####” until ENT is pressed.) Press ENT. Screen will read “Insert sample filter. Move to position 2.” Using disk tweezers, place ND disk on top of T60 filter in sample holder. Do not push tray in yet.
- In computer enter ND disk number (shown in ND table) in Filter ID column. (The thinner the disk, the lower the number.) Press Enter. Description and Blank will fill in.
- Push sample tray into position 2. Screen will read “IR WARMING UP...” then “Measuring: SAMPLE filter IR...” Screen will read “UV WARMING UP...” then “Measuring: Sample filter UV...” Screen will read “UV ATTENUATION: ##### and IR ATTENUATION: #####.” Record data from computer in columns ATN IR and ATN UV in ND table in corresponding row.
- Repeat for all ND disks.
- Remove ND disk and measure No ND disk again.
- To save file: First highlight the data by clicking the empty box next to Description to highlight all rows. Click Accept Values box. Values will appear in Accepted Values Table. File>Save. Save in project directory. Open file to check data is there. Delete data in transmissometer program.
- Calibration is verified if the average ND filter calculated test results are below 10.
- Keep ND disks in their box between usages.
- Press ESC to return to Main Menu.

6.3 Calibration

A calibration is performed at the beginning of using each new lab blank.

- After a ND Nominal Optical Density check is preformed, replace 25mm Filter Adapter holders with clean 47 mm Filter Adapter holders.
- Calibrate with a blank filter on top of a Tx40H120 (TX) filter shiny side up.
- Scroll (using arrow buttons) through Main Menu to Settings. Press ENT button.
- Scroll through Settings to Calibrate. Press ENT. Screen will read “Calibrate: Press ENT...” Press ENT. Screen will read “Insert blank filter. Move to position 1.”
- Place TX filter in reference holder. Place blank filter on top of the TX filter in reference holder. Blank filter is the corresponding lot blank. Gently push sample tray to position 1. Screen will read “Calibrating IR. Please wait...” then “Calibrating UV. Please wait...” When calibration is done, screen will return to Main Menu.
- On transmissometer data sheet place a check in the C column on the far left of the sheet to indicate the calibration was done.

- Scroll to Measure Filter. Press ENT. Screen will read “Use new blank.” Press ENT. Blank filter and TX filter is still in position 1. Screen will read “IR WARMING UP...” then “Measuring: BLANK filter IR...” Screen will read “UV WARMING UP...” then “Measuring: BLANK filter UV...” Screen will read “Insert sample filter. Move to position 2.”
- Recalibrate each time you change to a different blank filter or different size of adaptor.

6.4 Routine Operation

- On the Dual-Wavelength Optical Transmissometer (OT) Data Sheet (see Attachment B), fill in appropriate project and filter identification. Also record environmental information.
- Wear gloves and handle the filters with flat-tipped tweezers. Hold the filters near the edge, as far as possible from the deposit area.
- Clear the aperture of all objects.
- Using forceps, place the filter in the filter holder. Exposed filters are placed with the deposit side up. Unexposed Teflon filters are loaded with the shiny support ring facing up.
- If necessary, place the retaining ring over the Teflon filters, being careful not to scratch the surface. The retaining ring serves to hold the filters flat.
- In computer enter I (initial), IR (initial replicate), F (final), or FR (final replicate) as appropriate in field next to Use Description button. Click Use Description button. Entered description will appear in Description column. Enter blank identification in Blank Filter ID column. Click box next to Use previous Blank Filter ID (a check mark will appear in box). Enter blank filter ID in Filter ID column. If Description, Filter ID and Blank Filter ID columns are not filled in, the data will not go in the computer. On data sheet record blank filter ID in Filter ID column in the first row with a B next to it.
- On transmissometer screen will read “Insert sample filter. Move to position 2.” (If not, scroll to Measure Filter. Press ENT. Scroll to “Use previous blank.” Press ENT.) Remove blank filter and TX filter from reference holder using tweezers and place both in sample holder. Push sample tray into position 2. Screen will read “IR WARMING UP...” then “Measuring: SAMPLE filter IR...” Screen will read “UV WARMING UP...” then “Measuring: BLANK filter UV...” When done measuring, screen will read UV ATTENUATION: 0000 and IR ATTENUATION: 0000. Values in computer should be near zero. Record data from computer in columns Initial IR and Initial UV or Final IR and Final UV on data sheet in corresponding row.
- Record the reading on the measurement form (see Attachment B). Insure that the top section of the form is completed and that the filter ID of the sample matches the one recorded on the form.
- Remove the retaining ring, if used, and replace the filter into its petri slide.

- Pull sample tray out to expose sample holder. (Screen will still show “UV ATTENUATION: ##### and IR ATTENUATION: #####” until ENT is pressed.) Press ENT. Screen will read “Insert sample filter. Move to position 2.” Using tweezers, remove blank filter and place sample filter (initial or final) on top of TX filter in sample holder. Do not push tray in yet.
- In computer enter sample filter ID in Filter ID column. Press Enter. Description and Blank will fill in. On data sheet record sample filter ID in Filter ID column.
- Push sample tray into position 2. Screen will read “IR WARMING UP...” then “Measuring: SAMPLE filter IR...” Screen will read “UV WARMING UP...” then “Measuring: Sample filter UV...” Screen will read “UV ATTENUATION: ##### and IR ATTENUATION: #####.” Record data from computer in columns Initial IR and Initial UV or Final IR and Final UV on data sheet in corresponding row.
- Repeat for all sample filters.
- After every 10 sample filters (and at the end of the session) re-measure blank filter in sample holder.
- Recalibrate each time you change to a different blank filter.
- Save file at least every 20 sample filters. First highlight the data by clicking the empty box next to Description to highlight all rows. Click Accept Values box. Values will appear in Accepted Values Table. File>Save. Save in project directory. Open file to check data is there. Delete data in transmissometer program.
- Press ESC to return to Main Menu.
- Record your work on the transmissometer worksheet.

6.5 Shut-Down

After all samples are measured for the day, perform ND Nominal Optical Density check as described in Section 6.2, recording all readings in the ND table. Turn the power to the instrument off and push sample tray to position 2.

6.6 Calculations

Measurements recorded on the transmissometer data sheet will be forwarded to the CSN Management group for further calculations.

Light transmission intensity (at 370 and 880 nm) through the loaded (T) and the blank filter (T₀) determines the b_{abs} , where:

$$b_{abs} = -\ln\left(\frac{T}{T_0}\right)$$

Brown carbon (BrC) can be estimated as:

$$b_{abs} (BrC) = b_{abs} (370 \text{ nm}) - b_{abs} (880 \text{ nm}) \times \frac{880}{370}$$

Although σ_{abs} varies by aerosol type and the age of the carbonaceous aerosol, σ_{abs} of 16.6 m²/g is commonly used to convert b_{abs} to BC for 880 nm channel. Assuming b_{abs} is proportional to BC concentration, the BC can be derived as:

$$BC \left(\frac{\mu g}{m^3} \right) = \frac{b_{abs}}{\sigma_{abs} \left(\frac{m^2}{g} \right)} \times \frac{deposit\ area\ (m^2)}{sample\ volume\ (m^3)} \times 10^6 \left(\frac{\mu g}{g} \right)$$

6.7 Data Analysis

1. The original Neutral Density Worksheet(s) and Transmissometer Data Sheet(s) are submitted to the Laboratory Operations Manager (LOM) for review.
2. After data review, if the results are acceptable, the LOM will transfer the filters and completed internal chain of custody back to the CSN FiSH group and prepare an excel data report containing the b_{abs} results to the CSN Program Manager. If the results are not acceptable, the filters will be reanalyzed.

6.8 Quality Control

1. The Neutral Density Optical Filters must be recertified annually by the manufacturer using National Institute of Technology (NIST) traceable standards. A Certification Certificate will be supplied with the date of analysis.
2. The results of the Neutral Density checks must be <10 for each wavelength. If the results are ≥ 10 , the test must be repeated. If an acceptable result cannot be obtained, the manufacturer will be contacted for service.
3. Calibration is performed before each new blank filter using a certified photometric standard glass disks. If the measurement varies by more than $\pm 5\%$, the optics are cleaned and the system is recalibrated before proceeding to sample analysis.
4. Each batch of 10 samples includes a QC check of one photometric standard. Results must be $\pm 5\%$. If the QC sample fails, recalibrate the instrument and reanalyze every sample since the last acceptable check standard.
5. Replicate analyses are performed on 10% of the samples. The replicate sample results must be $\pm 5\%$. If the results exceed 5%, repeat the analysis. If the results still do not pass the criteria, the data will be flagged in the report to the CSN Program Manager.

7.0 References

Magee Scientific SootScan™ Model OT21 Transmissometer User Manual, Version 5.2, March 2016.

Desert research Institute SOP – Dual Wavelength Optical Transmission Analysis, October 2014.

8.0 Attachments

Attachment A – Neutral Density Worksheet

Attachment B – Transmissometer Worksheet

Attachment C – Internal Chain of Custody

**Attachment A
 Neutral Density Worksheet**

Reference Set Serial #				Initial / Final
OD	0.15	0.3	0.4	0.6
ATN @ 370 nm	75	146	194	314
ATN @ 880 nm	43	78	105	193
Measured ATN				
370 nm	A1	B1	C1	D1
880 nm	A2	B2	C2	D2
diff @ 370	A3 = abs (A1-75)	B3 = abs (B1-146)	C3 = abs (C1-194)	D3 = abs (D1-314)
diff @ 880	A4 = abs (A2-43)	B4 = abs (B2-78)	C4 = abs (C2-105)	D4 = abs (D2-103)

Conformity	Results	Criteria	Pass
370 nm	Avg(A3:C3)	< 10	Yes / No
880 nm	Avg (A4:D4)	< 10	Yes / No

Attachment B Data Sheet for Dual-Wavelength Optical Transmissometer

Dual-Wavelength Optical Transmissometer Data Sheet

Initial IR/UV
 Date: _____
 Time: _____
 Temp(°C): _____
 RH (%): _____
 Tech: _____

Final IR/UV
 Date: _____
 Time: _____
 Temp(°C): _____
 RH (%): _____
 Tech: _____

File Saved As:
 I: _____
 IR: _____
 F: _____
 FR: _____

V:\Documentation\FORMS\LABimages\data-sheet1

Project Name:	Project Code:	Sampling Site:	Filter Type:	Filter Lot No.:	Equilibration:	Back Filter ID:	Initial ATN Test Values		Final ATN Test Values		Initial ATN Test Values	#2 Initial ATN Test Values		#2 Final ATN Test Values		
							IR	UV	IR	UV		IR	UV	IR	UV	IR
No ND Disk	0	0														
0.15	62	75														
0.3	96	144														
0.4	115	188														
0.6	211	302														
No ND Disk	0	0														

Date/Time:	Attenuation (ATN) Values		Initial ATN Test Values		Final ATN Test Values		#2 Initial ATN Test Values		#2 Final ATN Test Values	
	IR	UV	IR	UV	IR	UV	IR	UV	IR	UV
No ND Disk	0	0								
0.15	62	75								
0.3	96	144								
0.4	115	188								
0.6	211	302								
No ND Disk	0	0								

Filter ID	Initial IR	Initial UV	Final IR	Final UV	Flag	Comments
C						
B						
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
B						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
B						

