

***TD-500D***<sup>TM</sup>  
**Handheld Oil-in-Water Meter**

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**User's Manual**

Revision: C  
P/N 100668

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# PART-I

## I.1 Introduction

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### I.1.1 Description

The TD-500D™ is a dual-channel, handheld fluorometer designed for quick, easy and reliable measurements of crude oil, fuel oil, lube oil, diesel, some gas condensates and refined hydrocarbons in water or soil. When properly calibrated with a correlation method or a known standard, the TD-500D™ can be used to measure the hydrocarbon concentrations of water samples in less than 4 minutes.

The TD-500D™ is not designed to measure gasoline, BTEX, aromatic solvents, or BTEX-component gas condensate in water or soil. Contact us about the TD-3100™ bench top oil-in-water analyzer for measurement of gasoline, phenol, BTEX, or aromatic chemicals in water. Please contact [sales@oilinwatermonitors.com](mailto:sales@oilinwatermonitors.com) for more information.

The dual-channel design makes the TD-500D™ applicable to a wide range of hydrocarbon types and concentration ranges. Channel A provides the extreme sensitivity required to monitor very low hydrocarbon concentrations and for detecting weakly fluorescent hydrocarbons such as some gas condensates, diesel fuel and refined petroleum products. Channel B was designed for those applications where a wide concentration range is more important than high sensitivity. Channel B is ideal for the analysis of produced water samples where crude oil concentrations can vary from low levels to over one thousand parts-per-million.

The TD-500D™ is normally used with a solvent-extraction method such as the Oil-in-water Analysis Procedure described in Part II of this manual. In this method, the target hydrocarbon is extracted into a suitable solvent. The TD-500D™ measures the hydrocarbon content of the extract, and calculates the hydrocarbon content in water.

**NOTE:** The TD-500D™ is not explosion-proof or intrinsically safe. It is not designed in any way to be used in an explosion-proof designated area.

## **I.1.2 Getting Started**

### **I.1.2.1 Incoming Inspection**

Upon receiving your instrument, please inspect everything carefully and make sure all ordered accessories are present. All shipments include:

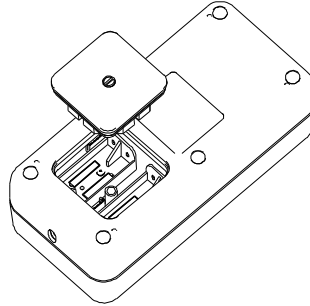
- TD-500D™ Handheld Oil-in-Water Meter
- 4 AAA Batteries
- User's Manual
- Recommended Accessories (as ordered):
  - Carrying Case
  - CheckPOINT™ Solid Standard
  - Appropriate cuvettes and adapters.

### **I.1.2.2 Battery Installation**

1. The battery compartment is located at the back of the instrument. Loosen the screw and remove the battery compartment cover (See *Figure 1*).
2. Install the 4 AAA batteries. Orient the batteries as indicated by the diagrams inside the battery compartment.

3. Replace the battery compartment cover and tighten the screw. The cover has an o-ring, which creates a watertight seal. The battery compartment cover may be difficult to install if there is no lubrication on the o-ring. If necessary, use silicon-based grease to lightly lubricate the o-ring.

**Back of the TD-500D™ showing battery cover removed**



*Figure 1. TD-500D™ Battery Compartment*

## I.2 Operation

### I.2.1 Principle of Operation

The TD-500D™ responds to the fluorescent aromatic compounds in the target hydrocarbon. The instrument must be calibrated by measuring the intensity of fluorescent light that is generated by a known concentration of hydrocarbon. Once calibrated, the instrument converts the fluorescent light intensity from an unknown sample into units of concentration.

Figure 2 shows the general relationship between fluorescence intensity and concentration.

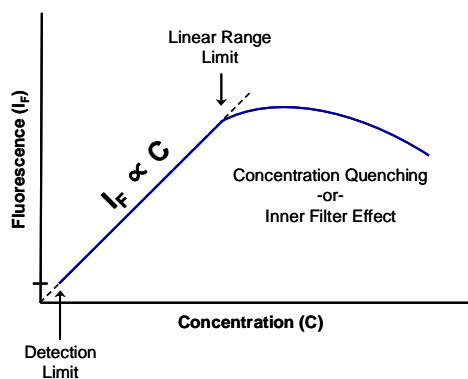


Figure 2. Fluorescence vs. Concentration

Every hydrocarbon will have a detection limit and a linearity limit. The detection limit is the lowest concentration that the instrument can detect. The linearity limit varies with each type of hydrocarbon. The linear range is defined by the concentration span from the detection limit to the linearity limit. As concentration increases beyond the linearity limit, the



slope of the line begins to reduce. At very high concentrations, the slope may become negative. For most crude oils the linearity limit is well beyond 1000 ppm.

The TD-500D™ must be operating in the linear range to display accurate results. The linear range can be optimized for any monitoring application by selecting the best combination of measurement channel (A or B) and cuvette size (mini-cell or 8 mm cuvette). See **Section I.3** for configuration guidelines.

After calibration you must perform a simple test to determine if your calibration standard is in the linear range. First select a channel and proper cuvette size and calibrate the TD-500D™ with a standard solution of known concentration. Then prepare a new standard that is half the concentration of the standard solution by mixing equal volumes of the original calibration standard and pure blank. Place the diluted standard in a sample cell, place the cell in the TD-500D™ and press <READ>. If the displayed result is greater than half the original standard concentration, allowing for some error due to dilution, the TD-500D™ calibration is beyond the linear range. The linear range is dependent upon the sample cuvette size and the optical channel of the TD-500D™; thus using a smaller cuvette size and/or changing the optical channel can extend the linear range.

### **I.2.2 Channel Selection Guidelines:**

Channel A is generally used where high sensitivity is required. Channel A is ideal for weakly fluorescent hydrocarbons or for very low concentrations of hydrocarbons. Channel B is used whenever Channel A gives poor linearity over the concentration range of interest or if channel A is over range for the calibration standard. Proper channel selection depends on the

type of hydrocarbon and the desired concentration range. The proper channel is determined when the calibration on the particular channel and using a particular cuvette size produces a diagnostic reading of %FS-Std > 2, but less than 100, while at the same time maintaining linearity over the measurement range. See **Sections I.3.3** and **I.3.4** for further details.

**Channel A:**

In most cases, use channel A for measuring following hydrocarbons in water or soil:

- Gas Condensate
- Diesel
- Marine Diesel
- Fuel Oil #6 (< 1 ppm)
- Lube Oil
- Crude Oil in Produced Water (< 5 ppm)

**Channel B:**

In most cases, use channel B for measuring:

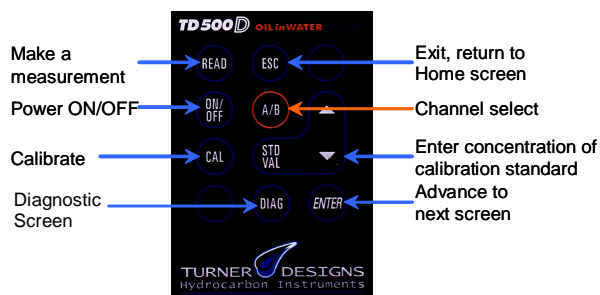
- Crude Oil in Produced Water (0 > 100 ppm)
- Heavy crude oils (higher concentrations)
- Heavy fuel oils (higher concentrations)

**I.2.3 Cuvette Selection Guidelines:**

Use the smaller mini-cell cuvettes (P/N 50952) for measuring heavy crude oil and other highly fluorescent oils in water. Use the larger 8 mm diameter cuvettes (P/N 50957) for diesel, gas condensates, lube oil and other refined hydrocarbons in water or for very low concentrations of crude oil-in-water. The cuvette size is determined when the calibration on the particular channel size produces a diagnostic reading of %FS-Std > 2, but less than 100, while at the same time

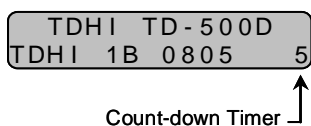
maintaining linearity over the desired measurement range. See **Sections I.3.3** and **I.3.4** for further details.

### I.2.4 TD-500D™ Keypad

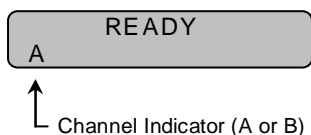


### I.2.5 Instrument Power-up

Turn the instrument face-up and press the <ON/OFF> button. The instrument should turn on and display an introduction screen with a count-down indicator in the lower right corner:



After 5 seconds, when the count-down indicator reaches zero, the TD-500D™ displays the “Home” screen and is ready for use:



The channel indicator will display the channel that was active during the instrument’s previous use.

If the display does not activate, check the batteries. Make sure they are fresh and installed in the proper orientation. If the display shows meaningless characters, replace the batteries. The TD-500D™ will automatically turn off if it is idle for 3 minutes.

Fresh alkaline batteries will provide adequate power for over 1000 measurements.

### **I.2.6 Calibration**

There are many ways to calibrate the TD-500D™. The most popular calibration method in the USA offshore industry is by correlation to the standard US EPA method. For details about this method and alternatives see **Section II.4**. Another way to calibrate is using a known standard made from the target oil and a blank solvent. This method is generally used in the field for quick analysis or when an official laboratory is not available. For a discussion regarding the various philosophies of calibration please contact [service@oilinwatermonitors.com](mailto:service@oilinwatermonitors.com) or visit our web site.

Before the TD-500D™ can be used to analyze a sample, you must calibrate it with a blank and a calibration standard containing your target hydrocarbon. If you are following a solvent-extraction method, use the pure extraction solvent for the blank. Prepare the calibration standard by dissolving a known amount of hydrocarbon in a known volume of extraction solvent. The standard solution will then have a known concentration. This is the standard value (STD VAL). The TD-500D™ can also be calibrated by correlating the instrument readings to another oil-in-water analysis method i.e. US EPA 1664.

**NOTE:** Make sure that you use the same size cuvette for blank, calibration solution and sample analysis. For example, if you use the mini-cell cuvettes for blank and calibration solution then use must mini-cell cuvettes for sample analysis also. Using different size cuvettes for calibration and sample analysis will result in incorrect readings.

### I.2.7 Calibration Standard Value

Assign a numeric value between 1 and 9999 for your known standard solution. This is done after performing the calibration as the last step of the calibration procedure. Make sure the instrument is set to the proper channel (A or B), then use the up and down arrows to adjust the standard value. Holding this button down will activate faster scrolling. When finished, press <ESC> or <ENT> to accept the value and return to the Home screen.

**NOTE:** No units are shown on the screen.

### I.2.8 Diagnostic Information

Press <DIAG> to access the diagnostic screens. Record the %FS-BLK and %FS-STD values shown on the screen for future reference. The %FS-BLK is the fluorescence response of the TD-500D for the blank i.e. clean extraction solvent. The %FS-STD is the fluorescence response of the TD-500D<sup>TM</sup> for the calibration standard. For best performance the %FS-STD should be greater than 2. The %FS-STD reading is the fluorescence value of the standard above the fluorescence value of the blank. The maximum values are 100 %FS. Press <ESC> when finished to return to the Home screen.

### I.2.9 Channel Selection

Press <A/B> for switching the channels on the TD-500D™. There are two channels on the TD-500D™, Channel A and Channel B. Please refer to **Section I.2.2** for channel selection guidelines. You may switch channels by pressing <A/B> only when the “HOME” screen is displayed.

**NOTE:** Each channel will store a separate calibration.

**NOTE:** Analyze your samples on the calibrated channel only. For example if your TD-500D™ is calibrated on Channel B, then use Channel B for sample analysis. Using a different channel for sample analysis will give incorrect readings.

## **I.3 General Considerations for Analysis**

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### **I.3.1 Sample Analysis**

1. Turn on the instrument. Make sure the proper channel (A or B) is displayed.
2. Insert your sample in a clean cuvette and put the cuvette in the appropriate cuvette adapter. Be sure to wipe any oil or finger prints from the outside of the cuvette.
3. Insert the cuvette adapter in the TD-500D™ adapter well and press <READ>. The instrument will measure and average the signal over a 5 second interval.
4. The result will be displayed on the screen.
5. The top left corner will display “WAIT” for 5 seconds. Once “WAIT” disappears, another sample reading can be performed.

### **I.3.2 General Information and Precautions**

- The adapter well with the appropriate adapter accepts either the mini-cell cuvettes or the 8 mm round cuvettes. Consult factory for questions regarding use of various cuvettes.
- Take care not to spill samples into the adapter well. Promptly wipe any spills. Simple alcohol rinse of the adapter well should neutralize any potential chemical attack.
- The TD-500D™ is very sensitive and even small amounts of material from a previous sample may result in errors. Use a clean cuvette for each reading. Wipe the outside of the cuvette with a Kimwipe or similar laboratory tissue. Do not use paper towels or kitchen wipes because the optical brighteners in the paper may cause a false reading.

- Fill the cuvette at least 75% full; significant error will result if the cuvette is not properly filled. Use a minimum of 75  $\mu\text{L}$  (0.075 mL) in a mini-cell cuvette, and a minimum of 1 mL in an 8 mm diameter cuvette for best results.
- The cuvette **MUST BE CLEAN AND DRY** on the outside when taking readings. Moisture and condensation or fingerprints on the outside can result in error. Use laboratory grade tissues or wipes for wiping the cuvettes.
- Avoid air bubbles in the cuvette. Bubbles in samples may cause drifting of the reading. Slight tapping on the outside cuvette wall should help remove any bubbles.
- Avoid transferring emulsions to the cuvette since bubbles of water in the extract will cause incorrect measurements.
- Always make sure to analyze the samples on the calibrated channel only. Using a different channel for sample analysis will give incorrect readings.

### **I.3.3 Linear Range**

The linear range is the concentration range within which the readout of the TD-500D<sup>TM</sup> is directly proportional to the concentration of the hydrocarbon. You must make sure the instrument is calibrated in the linear range (see **Section I.2.1**).

The linear range is dependent upon the sample cuvette size and the optical channel of the TD-500D<sup>TM</sup>; thus using a smaller cuvette size and/or changing the optical channel can extend the linear range. The glass mini-cell cuvettes provide a smaller cell diameter than the 8 mm round cuvettes.



For heavy and highly fluorescent oils, we recommend using the mini-cells with channel B for the widest possible linear range.

The linear range must always be checked after the first calibration by diluting the calibration solution half with blank and then measuring the concentration on the TD-500D™. If the displayed result is greater than half the original standard concentration, allowing for some error due to dilution, the TD-500D™ calibration is beyond the linear range.

#### **I.3.4 Sensitivity**

The instrument's sensitivity is measured by the %FS-STD value on the diagnostic screen. For best results, the %FS-STD should be greater than 2. The sensitivity should be checked when initially calibrating the TD-500D™.

The calibration and operation guidelines mentioned in this manual work with most oils. However, some refined oils and condensates do not have enough fluorescence (%FS-STD < 2). If the %FS-STD is less than 2 or if the calibration screen says "Standard < Blank, Recalibrate", then follow the steps below:

1. Make sure you are using clean solvent and clean cuvettes.
2. If the TD-500D™ is set to channel B, then switch to channel A, and/or;
3. Use larger cuvette, and/or;
4. Use higher concentration of oil in solvent for calibrating the TD-500D™.
5. If the above suggestions do not help, contact Turner Designs Hydrocarbon Instruments' Service Department (service@oilinwatermonitors.com).

### **I.3.5 Temperature Considerations**

Perform all calibrations and sample analysis at ambient temperature.

### **I.3.6 Data Quality**

The TD-500D™ is only as accurate as the standards that are used to calibrate it and the accuracy of the measurement of the standards into the solvent. This is why it is important to take care when preparing standards, samples, and blank. One should follow good laboratory practices when handling reagents and preparing standard solutions. Also, follow all solvent handling rules for the specific solvent and the specific facility. Use safety glasses with side shields or splash goggles while handling acids and solvents. Use nitrile examination gloves to protect your hands. Consult your company's safety guidelines for detailed safety instructions.

## I.4 Troubleshooting

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The following messages can appear on the TD-500D™ display screen:

**Message 1:** “Low Battery”

**Cure 1:** Replace the batteries with 4 new AAA batteries.

**Message 2:** “Circuit Failure”

**Cure 2:** Contact Turner Designs Hydrocarbon Instruments, Inc. according to directions in **Section I.5.2**.

**Message 3:** “High Blank”

**Cure 3:** Recalibrate the TD-500D with a clean blank.

**Message 4:** “Standard too High: Recalibrate”

**Cure 4:** a) Use lower concentration of Calibration solution; and/or  
b) Use smaller cuvette size for blank, calibration standard and for sample analysis; and/or  
c) Switch to channel B and recalibrate.

**NOTE:** The optimum combination of channel and cuvette size will depend on the type of oil, detection limit and measurement range.

If the above suggestions do not help, please make a copy of the *Troubleshooting Worksheet* and then complete the worksheet with the requested information. The completed worksheet should be sent to Turner Designs Hydrocarbon Instruments ([service@oilinwatermonitors.com](mailto:service@oilinwatermonitors.com) or fax + 1 559 253-1090) or your local dealer.

## TD-500D™ Troubleshooting Worksheet

Customer Name: \_\_\_\_\_

Company Name: \_\_\_\_\_

Company Address: \_\_\_\_\_

Country: \_\_\_\_\_

Phone Number: \_\_\_\_\_

Fax Number: \_\_\_\_\_

E-Mail Address: \_\_\_\_\_

- 1) API or specific gravity of oil or condensate: \_\_\_\_\_
- 2) Extraction solvent : \_\_\_\_\_
- 3) Calibration method: (Prepared solutions or correlation) \_\_\_\_\_  
(If correlation is used, attached correlation data and/or graph)
- 4) Measurement channel used (A or B): \_\_\_\_\_
- 5) Size of cuvette used (mini-cell or 8 mm cuvettes): \_\_\_\_\_
- 6) Calibration solution concentration: \_\_\_\_\_
- 7) Standard value (STD VAL): \_\_\_\_\_
- 8) Diagnostic screen (DIAG) readings:  
%FS-Blk: \_\_\_\_\_  
% FS-Std: \_\_\_\_\_
- 9) Linearity test –  
Concentration of diluted solution \_\_\_\_\_  
Measured concentration of diluted solution \_\_\_\_\_

## **I.5 Warranty**

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### **I.5.1 Terms**

Turner Designs Hydrocarbon Instruments warrants the TD-500D™ Oil-in-water Meter and accessories to be free from defects in materials and workmanship under normal use and service for a period of one year from the date of shipment. The instrument and accessories must be installed, powered, and operated in compliance with the directions in this TD-500D™ User's Manual and the instructions accompanying the accessories. Additional 12 months warranty is available at the time of order. Please consult factory.

The following items are ***not*** covered under warranty:

- Damage resulting from contact with solvents or other corrosive liquids and gases.
- Damage resulting from contact with corrosive materials or atmosphere.
- Damage caused by modification of the instrument.
- Damage caused by improper handling and physical abuse of the instrument.

### **I.5.2 Service During and After Warranty Period**

To obtain service at any time, do the following:

1. Write or call the Turner Designs Hydrocarbon Instruments, Inc. Service Department and describe the nature of the problem. See contact information on page 23.

2. Make minor adjustments or tests as suggested by Turner Designs Hydrocarbon Instruments, Inc. Service Department personnel.
3. If the Service Personnel determine that the TD-500D™ must be returned to the factory, **OBTAIN AN RMA (Return Material Authorization) NUMBER BEFORE SHIPPING THE INSTRUMENT TO TURNER DESIGNS HYDROCARBON INSTRUMENTS, Inc. You must ship all items associated with the instrument, including the TD-500DTM, the appropriate adapter, the CheckPOINT™ Solid Standard and the carrying case.** After obtaining an RMA number, pack the instrument and accessories well, insure it, write the RMA # on the outside of the carton, and ship it to Turner Designs Hydrocarbon Instruments, Inc. prepaid. If the instrument and/or the problem are determined to be repairable under warranty, the instrument will be repaired; however, all shipping costs are for the customer's account.

Out of warranty repair for the TD-500D™ is billed on a flat rate basis regardless of the problem. Your bill will include return shipment freight charges.

**NOTE:** Please do not return the instrument without notice.

**Address for Return Shipments:**

Turner Designs Hydrocarbon Instruments, Inc.  
Attn: RMA#\_\_\_\_  
2023 N. Gateway Blvd., Suite 101  
Fresno, CA 93727  
United States of America

**Contact Information for Sales and Service**

Main Phone Number: +1 559 253-1414

Main Fax: +1 559 253-1090

Email (Sales issues only):

[sales@oilinwatermonitors.com](mailto:sales@oilinwatermonitors.com)

Email (Service issues only):

[service@oilinwatermonitors.com](mailto:service@oilinwatermonitors.com)

## I.6 Appendix A: Instrument Specifications

TD500D Hand Held Oil-in-water Meter Data Sheet							
<b>GENERAL</b>	1 Hydrocarbons in Water		Crude Oil, Condensate, diesel, lube oil, fuel oil, motor oil, diesel range organics				
	2 Ambient Temperature	Min F/°C	Max F/°C	41/5 104/40			
	3 Area Classification		General Purpose				
	4 Mounting		Hand / Table Top				
	5 Dimensions		1.75" x 3.5" x 7.25" (4.45 cm x 8.9 cm x 18.4 cm)				
	6 Weight		13.9 oz (0.4 kg)				
	7 Enclosure Material		Non-Metallic				
	8 IP Rating		Meets IP 67 standard; dustproof and waterproof				
	9 Power Supply Required		4 AAA Cells (1,000+ Samples)				
	10 Automatic Power Down		After 3 minutes of inactivity				
	11 Local Display		Yes, LCD				
	12 Output Signal		No				
	13 Warranty		1 Year Factory Parts and Labor				
<b>Technical Detail</b>	14 Principle		UV Fluorescence				
	15 Cuvettes	API	Type	Solvent	<45	Mini-cell	All
					>45	8mm	All
	16 Measurement Method		Solvent Extraction				
	17 Compatible Solvents		Hexane, Vertrel, AK-225, Xylene, Freon, Horiba, (most other extraction solvents, consult factory)				
	18 Total Elapsed Time for Measurement Method		< 4 min or owners preference				
	19 Accuracy		Better than 2% of Full Scale				
	20 Repeatability		Better than 2% of Full Scale				
	21 Linear Range		Up to 1000 ppm Hydrocarbon Dependent				
	22 Sensitivity		1 ppm				
23 Calibration		Single-point and blank					
24 Response Time		5 Seconds					
25 Alarms		Low battery, circuit failure, High blank					
26 Warm Up		5 seconds					



# ***TD-500D*<sup>TM</sup> User's Manual**



## **Oil-in-Water Analysis Procedure**

## **II.1 *fastHEX*<sup>TM</sup> Method Introduction**

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### **II.1.1 Introduction**

The *fastHEX*<sup>TM</sup> method for the TD-500D<sup>TM</sup> was developed to provide a simple, rapid, accurate, safe and environmentally friendly field procedure for the analysis of oil and grease in water. The TD-500D<sup>TM</sup> measures the oil content of a water sample by measuring the fluorescent light emitted by oil that has been extracted into hexane. Hexane is an inexpensive, non-fluorescent, organic solvent that efficiently extracts all types of crude oil, and separates out quickly on top of the water sample.

The *fastHEX*<sup>TM</sup> method has been in use for several years, and has been field-proven to give results that correlate well to US EPA Method 1664 and other official oil and grease methods. The entire procedure, from taking a water sample to recording results, takes less than 4 minutes. Because the method uses inexpensive, disposable bottles and cuvettes, solvent waste is reduced to a minimum. Also, unlike Freon and other halogenated or chlorinated solvents, hexane can be disposed of in most oil and gas facilities without worrying about physical exposure.

### **II.1.2 Other Methods:**

There have been many oil and grease analysis methods developed over the years and many of them are useful. If you have questions regarding the viability of any method please consult our Service Department or the appropriate department in your own company.

### **II.1.3 Alternative Solvents**

Although the *fastHEX*<sup>TM</sup> method was originally intended to use hexane, its solvent extraction and measurement procedures can also be applied to other lighter-than-water solvents (xylene, petroleum ether, etc.). In addition, the procedure can be modified allowing use of a variety of heavier-than-water solvents (Vertrel MCA, Ashikleen AK-225, perchloroethylene, etc.). Analysis procedures for lighter-than-water solvents and heavier-than-water solvents are given in **Section II.2**, and **Section II.3**, respectively.

### **II.1.4 Important note regarding solvents:**

Some solvents, especially Vertrel MCA, Ashikleen AK-225 and chloroform, can damage the TD-500D<sup>TM</sup> if left in contact with the plastic parts. If you spill solvents on the case or inside the adapter well, flush the contacted area immediately with isopropyl alcohol and dry the area with a non-abrasive lab tissue.

Always follow safety rules for handling and disposing of solvents.

## **II.2 *fastHEX*<sup>TM</sup> Method: Analysis Procedure for Lighter than Water Extraction Solvents**

*(See page 36 for Calibration Procedure)*

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This procedure is for use with lighter than water extraction solvents such as:

Hexane  
Cyclohexane  
Heptane  
Octane  
Xylenes (may give high blank reading)  
Toluene (may give high blank reading)

**NOTE:** Please use this procedure even if your solvent is not listed above and you are using lighter than water extraction solvent.

**NOTE:** If you are using heavier than water extraction solvent then refer to **Section II.3** of this manual for heavier than water extraction solvent analysis procedure.

### **II.2.1 Analysis Procedure:**

1. Collect 100 mL of contaminated water in a clean, graduated prescription bottle or suitable graduated glass container.
2. Add 6 N hydrochloric acid until the pH of the water sample is < 2. Check the pH with pH paper.

**NOTE:** Depending on the method philosophy of the facility, the sample can be measured at normal pH.

3. Add 10 mL of extraction solvent and securely cap the bottle.

4. Extract oil from the water by vigorously shaking the bottle for 2 minutes.
5. Allow the extraction solvent to separate from the water for approximately 1 minute. If the extraction solvent is trapped in an emulsion; break the emulsion by agitating with a clean pipette. Allow the extract to sit for several minutes after agitating the emulsion. This will allow the solvent to separate from the water. The solvent extract will be floating on top of the water.
6. Carefully release the pressure from the bottle.
7. Fill the appropriate cuvette (mini-cell or 8 mm cuvette)  $\frac{3}{4}$  full with the solvent extract. Wipe the outside of the cuvette with a clean laboratory grade tissue or cloth to remove any liquid, dirt or oil that might be present.
8. Insert the cuvette into the TD-500D™ cuvette holder in the adapter well and close the lid.
9. If the TD-500D™ is off, press the <ON/OFF> button and allow the instrument to proceed to the end of the countdown period.
10. Make sure the proper channel (A or B) is shown in the lower left-hand corner of the display. If it is not, press the <A/B> button until the correct channel appears.
11. Press the <READ> button and then record the reading after the “WAIT” message disappears.
12. If the TD-500D™ was calibrated using a look-up table, use the look-up table to convert the reading to oil concentration.
13. Dispose of the water, extraction solvent, cuvette and prescription bottle in a safe, environmentally responsible manner, in accordance with your company’s procedures.

**NOTE:** If the TD-500D™ Oil-in-Water Meter reads “OVER”, or the sample reads higher than the correlation chart or look-up table, this means the oil and grease concentration is too high. Follow the steps below to overcome this issue:

1. Dilute the solvent extract 5 times by combining 1 mL of solvent extract with 4 mL of clean solvent and mix the diluted extract.
2. Measure the diluted extract (Steps 7 through 11 on page 29).
3. Record the concentration of the diluted sample extract using the correlation chart (Step 11 on page 29).
4. Multiply the result from Step 3 by five (5) to calculate the true oil and grease result in the water sample.

## **II.3 Analysis Procedure for Heavier than Water Extraction Solvents**

*(See page 36 for Calibration Procedure)*

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This procedure is for use with heavier than water extraction solvents such as:

Vertrel MCA  
Freon  
Ashikleen AK-225  
Chloroform  
Carbon Tetrachloride  
Horiba S-316  
Methylene Chloride

**NOTE:** Please use this procedure even if your solvent is not listed above and you are using heavier than water extraction solvent.

**NOTE:** If you are using lighter than water extraction solvent then refer to **Section II.2** of this manual for lighter than water extraction solvent analysis procedure.

### **II.3.1 Analysis Procedure:**

1. Collect 100 mL of produced water in a clean, graduated prescription bottle.
2. Add 6 N hydrochloric acid until the pH of the water sample is < 2. Check the pH with pH paper.
3. Add 10 mL of extraction solvent and securely cap the prescription bottle with a clean, Teflon-lined septum cap.
4. Extract oil from the water by vigorously shaking the prescription bottle for 2 minutes.

5. Allow the extraction solvent to separate from the water for approximately 1 minute. If the extraction solvent is trapped in an emulsion; break the emulsion by agitating with a clean pipette. Allow the extract to sit for several minutes after agitating the emulsion. This will allow the solvent to separate from the water. The solvent extract will be on the bottom of the bottle, underneath the water.
6. Place a blunt needle on the end of a 3 mL plastic syringe. Push the syringe plunger all the way in.
7. Turn the prescription bottle upside down and allow the solvent extract to settle on top of the Teflon-lined septum cap.
8. While holding the prescription bottle upside down, insert the needle through the Teflon-lined septum cap far enough for the end of the needle to be in the solvent extract.
9. By pulling the syringe plunger out, withdraw 1-1.5 mL of solvent extract.
10. Inject the solvent extract into the cuvette (mini-cell or 8 mm cuvette, the same type used for calibration) until the cuvette is  $\frac{3}{4}$  full.
11. Wipe the outside of the cuvette with a clean tissue or cloth to remove any liquid, dirt or oil that might be present.
12. Insert the cuvette into the TD-500D™ adapter well and close the lid.
13. If the TD-500D™ is off, press the <ON/OFF> button and allow the instrument to proceed to the end of the countdown period.
14. Make sure the proper channel (A or B) is shown in the lower left-hand corner of the display. If it is not, press the <A/B> button until the correct channel appears.



15. Press the <READ> button and then record the reading after the “WAIT” message disappears.
16. If the TD-500D™ was calibrated using a loop-up table, use the loop-up table to convert the reading to oil concentration.
17. Dispose of the water, extraction solvent, cuvette and prescription bottle in a safe, environmentally responsible manner, in accordance with your company’s procedures.

**NOTE:** If the TD-500D™ Oil-in-Water Meter reads “OVER”, or the sample reads higher than the correlation chart or look-up table, this means the oil and grease concentration is too concentrated.

The following steps should be performed to dilute the sample extract:

1. Dilute the solvent extract 5 times by combining 1 mL of solvent extract with 4 mL of clean extraction solvent and mix the diluted sample extract.
2. Measure the diluted sample extract using the TD-500D™ Oil-in-Water Meter (Steps 10 through 15 on page 32-33).
3. Record the concentration of the diluted sample extract using the correlation chart (Step 16 on page 33).
4. Multiply the result from Step 3 by five (5) to calculate the true oil and grease result in the water sample.

## **II.4 Calibration Procedure for the TD-500D™**

*(For all extraction solvents)*

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### **II.4.1 Option 1: Calibrating with Standard Solutions**

The TD-500D™ Oil-in-water Meter can be calibrated with a standard solution of oil in extraction solvent. The standard solution (known oil concentration) is prepared by dissolving a known amount of oil in a known volume of extraction solvent. The instrument is calibrated by relating measured fluorescence response to the concentration of oil in the standard.

#### **II.4.1.1 Calibration Standards**

Sample preparation for the TD-500D™ Oil-in-water Analysis Methods involves extracting the oil from a 100 mL water sample into 10 mL of solvent. Therefore, the oil concentration in the extraction solvent will always be 10 times greater than the oil concentration of the water sample. For example, a 100 ppm oil concentration in a water sample will give a 1000 ppm oil concentration in the solvent extract. This 10 times concentration factor is affected during calibration i.e. if you want to calibrate the TD-500D™ for 100 ppm oil in water, you would use a standard solution of 1000 ppm in the extraction solvent.

#### **II.4.1.2 Calibration Standard Preparation**

**Part 1**—Prepare a 10,000 ppm Stock Standard.

1. Pipette 1 mL (1000 µL) of oil into a 100 mL volumetric flask that is approximately half full of clean extraction solvent.

2. Cap the flask and shake it to dissolve the oil.
3. Fill the 100 mL volumetric flask to the mark with clean extraction solvent.
4. Cap and shake the flask to mix.

**Part 2—Prepare a Calibration Standard**

Select a concentration that is equal to the maximum anticipated in your water samples. Prepare a Calibration Standard by diluting the Stock Standard with clean solvent. Recipes for preparing several typical concentrations are given in *Table 1*. To prepare the Calibration Standard shown in column 1, pipette the Stock Standard volume given in column 3 into a 100 mL volumetric flask, then fill the flask to the 100 mL mark with clean solvent.

Calibration Standard, ppm	Oil-in-water Equiv., ppm	Stock Standard Volume
5,000	500	50 mL
2,500	250	25 mL
1,000	100	10 mL
500	50	5.0 mL
250	25	2.5 mL
100	10	1.0 mL

*Table 1. Calibration Standards*

**II.4.1.3 Calibrate the TD-500D™ Oil-in-Water Meter**

1. Before calibration begins, you must select a TD-500D™ channel (A or B) and a cuvette size (mini-cell cuvette or 8 mm cuvette). See **Section I.2.2** for channel selection guidelines.

See **Section I.2.3** for cuvette selection guidelines.

2. Prepare a Blank by filling a cuvette with clean extraction solvent. Wipe the outside of the cuvette with a clean laboratory grade tissue to remove any liquid, dirt or oil that might be present.
3. Insert the Blank cuvette into the TD-500D™ adapter well and close the lid.
4. If the TD-500D™ is off, press the <ON/OFF> button and allow the instrument to proceed to the end of the countdown period.
5. Make sure the proper channel (A or B) is shown in the lower left-hand corner of the display. If it is not, press the <A/B> button until the correct channel appears.
6. Press the <CAL> button and then press <ENT>.
7. Press <ENT> to read the response of the Blank.
8. Fill a new cuvette with the Calibration Standard. Wipe the outside of the cuvette with a clean laboratory grade tissue to remove any liquid, dirt or oil that might be present.
9. Insert the Calibration Standard cuvette into the TD-500D™ adapter well and close the lid.
10. Press <ENT> to read the response of the Calibration Standard.
11. Press <ENT> within 10 seconds to store the calibration.
12. Press the <STD VAL> button.
13. Enter the STD VAL as 1/10 of the concentration of the Calibration Standard. Adjust the Calibration Value using the <▲> and <▼> buttons. Press either button once for each increment of STD VAL. Holding either of these buttons down will activate faster scrolling to the desired value.
14. Press <ENT> to accept the STD VAL.

The calibration is now complete. The calibration will be stored and maintained when the instrument is turned off.

#### **II.4.1.4 Notes on error conditions and remedial procedures**

The following messages can appear on the TD-500D™ display screen while calibrating the TD-500D™:

**Message 1:** “High Blank”

**Cure 1:** Recalibrate the TD-500D™ with a clean blank.

**Message 2:** “Standard too High: Recalibrate”

**Cure 2:** a) Use lower concentration of calibration solution; and/or

b) Use smaller cuvette size for blank, calibration standard and for sample analysis; and/or

c) Switch to channel B

#### **II.4.2 Option 2: Calibrating by Correlation to an External Method (i.e. US EPA 1664) or other standard oil and grease method**

The TD-500D™ Oil-in-water Meter can be calibrated to give oil concentrations equivalent to an external analysis method. This method has been used extensively for offshore platforms in the Gulf of Mexico to give results equivalent to EPA Method 1664. The method is based on establishing a correlation between the TD-500D™ response of the extracted sample and the oil concentration determined

by the external method performed at an appropriate analysis laboratory.

Procedures for performing this type of calibration depend strongly on the type of oil, the desired analysis range and the nature of the external method. Please consult the factory for additional information and guidance.

## **II.5 Using the TD-500DTM Oil-in-water Meter CheckPOINT™ Solid Reference Standard**

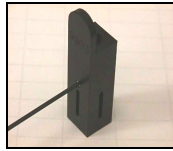
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The CheckPOINT™ Solid Reference Standard is used to verify the TD-500D™ Oil-in-water Meter calibration and performance prior to sample analysis procedure. The CheckPOINT™ Solid Standard contains a fiber that produces an adjustable signal that can be set to be equal to the calibration solution. The fiber is covered by an attenuation screw. Opening the attenuation screw (counter-clockwise rotation) will increase the exposure of the fiber and hence will increase the reading on the TD-500D™. Closing the attenuation screw (clockwise rotation) will decrease the readings on the TD-500D™.

The following procedure describes how a calibration value is assigned to the CheckPOINT™ Solid Standard. Once the CheckPOINT™ Solid Standard has a calibration value assigned to it, the TD-500D™ Oil-in-water Meter can be recalibrated in the field as necessary. The CheckPOINT™ Solid Standard is also used to verify instrument performance or calibration stability.

### **II.5.1 Adjusting the CheckPOINT™ Solid Standard**

1. Calibrate the TD-500D™ Oil-in-water Meter by Option 1 (Page 36) or by Option 2 (Page 40) mentioned in this manual.
2. Remove the cuvette and cuvette adapter.
3. Remove the CheckPOINT™ Solid Standard from its storage box. Using the 0.050" Allen wrench provided, loosen the locking setscrew on the back of the solid standard by turning it counterclockwise one turn.



Picture 1. Loosening the locking setscrew on the backside of the adjustable CheckPOINT™ Solid Reference Standard.

4. Insert the CheckPOINT™ Solid Standard with the “dog-ear” handle positioned closest to the back of the adapter well.



Picture 2. Placing the adjustable CheckPOINT™ Solid Reference Standard in the TD-500™.

5. If the TD-500™ Oil-in-water Meter is off, press the <ON/OFF> button and allow the instrument to proceed through the countdown period.
6. Press the <READ> button and then record the reading after the “WAIT” message disappears.
7. Open the lid and use the 3/32” Allen wrench provided to adjust the attenuation screw through the hole at the top of the solid standard to increase or decrease the value displayed on the screen. Turning the screw counter-clockwise will increase the reading.





Picture 3. Adjusting the value on the CheckPOINT™ Solid Reference Standard by turning the attenuation screw.

8. Repeat Steps 5 through 7 until the CheckPOINT™ Solid Standard reads the concentration value of interest.
9. At this point, remove the CheckPOINT™ Solid Standard and turn the locking setscrew until it just makes contact with the attenuation screw – do not over tighten.
10. Record the value of the CheckPOINT™ solid standard on the adhesive label on the bottom of the CheckPOINT™ Solid Standard.
11. Remove the CheckPOINT™ Solid Standard.
12. Recalibrate the TD-500D™ Oil-in-water Meter using the value of the CheckPOINT™ Solid Standard obtained in Step 10 as the Standard Value (STD VAL).

On a daily basis, prior to sample analysis, use the CheckPOINT™ to verify TD-500D™ Oil-in-water Meter calibration. If the CheckPOINT™ value has changed by greater than  $\pm 10\%$  of its assigned value, then, blank with clean extraction solvent and recalibrate the TD-500D™ Oil-in-Water Meter using the CheckPOINT™ Solid Standard.

## II.6 Extraction and Calibration

### Procedure Method Supplies

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The TD-500D™ can be purchased with the extraction kits for heavier and lighter than water solvents. These extraction kits have enough supplies for approximately 96 sample analysis each and they include necessary laboratory supplies and glassware required for the extraction procedure. Extraction solvent, hydrochloric acid, 2-propanol (also called isopropanol or isopropyl alcohol), and detergents and liquids are not provided in the extraction kit.

The following list of items are included in the extraction kits:

#### II.6.1 Extraction Starter Kit for Lighter than Water Solvents (P/N 100590)

Description	Quantity	Part No.
Disposable Glass Prescription Bottles with Caps, 180 mL	2 cases (48 per case)	100585
25 mL Glass Graduated Cylinder	2	100653
250 mL Plastic Squirt Wash Bottles	2	100631
pH Paper Indicator Strips, Universal 0-14	1 pack/100 strips per pack	100373
Glass Sample Vials 20 mL with Teflon Lined Caps	2	100621
Plastic Disposable Transfer Pipette	Pack of 100	100675

### II.6.2 Extraction Starter Kit for Heavier Than Water Solvents (P/N 100629)

Description	Quantity	Part No.
Disposable Glass Prescription Bottles with Caps, 180 mL	2 cases (48 per case)	100585
Teflon Lined Septum Caps	96	100632
Disposable Syringe, 3 mL Luer-Lok®	1 pack (100 per pack)	100651
Needles, SS with Blunt end	2	100652
25 mL Glass Graduated Cylinder	2	100653
250 mL Plastic Squirt Wash Bottles	2	100631
pH Paper Indicator Strips, Universal 0-14	1 pack/100 strips per pack	100373
Glass Sample Vials 20 mL with Teflon Lined Caps	2	100621
Plastic Disposable Transfer Pipette	Pack of 100	100675

The TD-500D™ Calibration Kit includes the necessary laboratory supplies required for preparing samples and for making known oil standards for calibrating the TD-500D™. The TD-500D™ Calibration kit is sufficient for 50 to 80 calibrations.

The following list of items is included in the TD-500D™ Calibration Kit:

**II.6.3 TD-500DTM Calibration Kit (P/N 100367)**

Description	Quantity	Part No.
100 mL Glass Volumetric Flask with Teflon Lined Cap	3	100368
10-100 Microliter Digital Micropipette	1	100369
100 Microliter Disposable Plastic Pipette Tips	1 Pack of 96 tips	100370
10 mL Glass Graduated Cylinder-Hexagonal Base	2	100371
50 mL Glass Graduated Cylinder-Hexagonal Base	2	100372
pH Paper Indicator Strips, Universal 0-14	1 pack/100 strips per pack	100373
Plastic Disposable Transfer Pipette	Pack of 100	100675

**NOTE:** VWR Scientific is a recommended supplier for lab equipment, but alternative suppliers may be used if similar equipment is purchased. VWR Scientific's website address is [www.vwrsp.com](http://www.vwrsp.com).

Turner Designs Hydrocarbon Instruments Telephone Number	559-253-1414
VWR Scientific Telephone Number	800-932-5000

**NOTE:** The cuvette choice may be dictated by the application. Please perform the linearity and sensitivity checks described in **Section I.3.3** and **Section I.3.4** to verify the cuvette choice.

***TD-500D*<sup>TM</sup> User's Manual**

**PART-III**

**No-Solvent  
Oil-in-Water Analysis Procedure**

## **III.1 No-Solvent Method for Oil-in-Water Analysis with the TD-500D**

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### **III.1.1 Introduction**

The “No-Solvent Method” makes it possible to perform oil-in-water analyses by making fluorescence measurements directly on the produced water sample. No organic solvents are required. The method is based upon the addition of a proprietary surfactant (OIW Surfactant) to a produced water sample. OIW Surfactant is safe to handle with a minimum of personal protective equipment and is only slightly flammable, even under a direct flame. The US Department of Transportation does not consider it to be a hazardous material. It can be shipped without hazardous identification labels and can be carried on commercial airlines and helicopters without declaration.

The “No-Solvent Method” is designed to determine the dispersed oil content of a water sample. It also provides a measure of the fluorescent water-soluble organic (WSO) compounds. The analysis is based upon two samples taken from the same water stream. The first, called the “OIW” sample, is a sample of water that has been treated with the OIW Surfactant and filtered into a measurement cuvette. The “OIW” sample contains dispersed oil that has been solubilized by the OIW Surfactant, as well as the WSO present in the water. The second sample, called the “WSO” sample, contains no surfactant. It is simply filtered into a measurement cuvette to remove dispersed oil, leaving only WSO. Both the “OIW” and “WSO” cuvettes are measured by the TD-500D. The dispersed oil concentration is calculated by subtracting the “WSO” measurement from the “OIW” measurement. Dispersed oil is reported in units of parts-per-million (ppm). WSO is reported in units of dispersed-oil-

equivalents. One dispersed- oil-equivalent is the fluorescence intensity emitted by 1 ppm of dispersed oil.

The TD-500D has two measurement channels, A and B. With the “No-Solvent Method”, the linear range of Channel A measurements is 0–100 ppm dispersed oil for all crude oils. Many crude oils can be measured at oil concentrations below 1 ppm. Channel A measurements can be extended to 750 ppm and higher through the use of a non-linear calibration function. When the instrument is operating on Channel B, the “No-Solvent Method” can measure oil concentrations up to 10,000 ppm, eliminating the need to dilute highly concentrated oil-in-water samples prior to measurement. In practice, Channel A is best for low-concentration measurements. Channel B should only be used for extremely high concentration measurements that cannot be performed with Channel A.

The procedures given below will enable you carry out oil-in-water analyses using the “No-Solvent Method”. For a more detailed discussion of the analytical principles, please refer to the paper entitled “*No-Solvent” Oil-in-Water Analysis – A Robust Alternative to Conventional Solvent Extraction Methods*, which is available on the Turner Designs Hydrocarbon Instruments web site ([www.oilinwatermonitors.com](http://www.oilinwatermonitors.com)).

## III.2 Equipment

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The “No-Solvent Method” requires a TD-500D Oil-in-Water Analyzer and the auxiliary equipment listed in the following table. Part numbers are listed for items that are available from Turner Designs Hydrocarbon Instruments. Other common laboratory items can be purchased from a variety of sources (e.g. VWR International, LLC, [www.vwr.com](http://www.vwr.com)).

<b>Item Description</b>	<b>Quantity</b>	<b>Part Number</b>
8 mm cuvettes	Box of 400	50957
8 mm cuvette adapter, large aperture	1	102935
8 mm cuvette adapter, small aperture	1	102936
OIW Surfactant	500 mL	102933
OIW Surfactant Dispenser Bottles	Bag of 4	102932
Graduated sample bottles	Case of 48	100585
Teflon lined caps	Bag of 48	100632
3 mL Syringes	Bag of 100	102931
Syringe filters	Bag of 100	102930
1000 µL pipette	1	102951
10 mL pipette	1	
4 mL graduated vials with caps	Bag of 5	102937
Kimwipes	1 box	101396
600 mL beaker	1	
500 mL volumetric flask	1	
100 mL graduated cylinder	2	
Hot plate	1	
Bottle brush	1	



Distilled water		
Disposable beaker	Box of 100	102934
Heat-resistant gloves	1 pair	
Safety goggles	1 pair	

### III.3 Initial Preparation

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1. Label a 500 mL volumetric flask “BLANK”.
2. Label four (4) clean, dry sample bottles “1000 ppm”, “100 ppm”, “WSO”, and “OIW.”
3. Label four (4) 8mm cuvettes “100 ppm” “BLANK”, “WSO” and “OIW.” Confine the labels to the top 0.5” (13 mm) of the cuvette to keep the mark out of the measurement zone.
4. Place 250 mL of tap water in a 600 mL beaker. Place the beaker on a hot plate. Turn on the hot plate and heat the water until it is near boiling (~180°F, 82°C).
5. Turn on the TD-500D and wait 5 seconds for the warm-up count-down to expire. The word “READY” will appear on the first line of the display. Also, a letter (A or B) indicating the measurement channel will appear on the lower left.
6. Press the <A/B> button to select the desired measurement channel. Select Channel A for oil in-water measurements in the 0-100 ppm range. See **Sections III.6** and **III.7** for channel selection criteria for extended range measurements.
7. Place the small-aperture 8 mm cuvette adapter (PN 102936) in the adapter well.

## III.4 Calibration: 0-100 ppm Oil-in-Water

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### III.4.1 Prepare the Blank

1. Place 15 mL of OIW surfactant into the 500 mL volumetric flask labeled “BLANK.”
2. Add distilled water to the flask until the fluid level reaches the 500 mL mark.
3. Cap the flask and invert it several times until the OIW Surfactant is completely dissolved.
4. Fill an 8 mm cuvette marked “BLANK”  $\frac{1}{2}$  to  $\frac{3}{4}$  full with the “BLANK” solution.

### III.4.2 Prepare the Standards (1000 ppm and 100 ppm)

1. Place 3 mL of OIW Surfactant into the bottle marked “1000 ppm”.
2. Pipette 100  $\mu$ L of oil directly into the OIW Surfactant. Swirl the bottle until the oil is fully dispersed in the OIW Surfactant.

**NOTE:** It may be necessary to heat viscous oils for accurate pipetting. A temperature of 120 °F (49 °C) to 150 °F (66 °C) is usually sufficient. If you must heat the oil in order to pipette it, you should also warm the OIW Surfactant in the “1000 ppm” bottle before you add the oil. This will help the OIW Surfactant fully disperse the oil.

3. Add distilled water to the “1000 ppm” bottle until the liquid level reaches the 100 mL mark.
4. Cap the bottle and shake it vigorously for at least 15 seconds.

5. Place the bottle in the hot water bath and heat it until the contents are uniformly milky-white in appearance.
6. Put on heat-resistant gloves suitable for handling glass bottles at boiling water temperature (212 °F, 100 °C).
7. Remove the bottle from the hot water and, while the contents are still milky-white, shake the bottle vigorously for at least 30 seconds.
8. Allow the “1000 ppm” standard to cool to room temperature.

**NOTE:** You may accelerate cooling by placing the “1000 ppm” standard in a refrigerator or a container of cold water.

9. Using a 10 mL pipette, add exactly 10 mL of “1000 ppm” standard to the “100 ppm” bottle.
10. Add the “BLANK” solution to the “100 ppm” bottle until the liquid level reaches the 100 mL mark.
11. Cap the “100 ppm” bottle and shake it until the contents are completely mixed.
12. Fill a 3 mL syringe with the “100 ppm” standard.
13. Attach an unused syringe filter to the end of the syringe.
14. Press the syringe plunger to filter the liquid into the 8 mm cuvette marked “100 ppm”. Continue until the cuvette is  $\frac{1}{2}$  to  $\frac{3}{4}$  full.

### **III.4.3 Calibrate the TD-500D**

1. Turn on the TD-500D and wait 5 seconds for the warm-up count-down to expire. The word “READY” will appear on the first line of the display. Also, a letter (A or B) indicating the measurement channel will appear on the lower left.

2. Make sure that the TD-500D is set to Channel A. If you see a “B” displayed instead of an “A”, press the <A/B> button until an “A” appears.
3. Wipe the “BLANK” and “100 ppm” cuvettes with a Kimwipe to remove any fingerprints, dirt or liquid droplets that might be present.
4. Press the <CAL> button.
5. Press the <ENTER> button.
6. When prompted for the “Blank”, place the “BLANK” cuvette in the adapter well and close the lid.
7. Press <ENTER>.
8. When prompted for the “Cal Soln”, remove the “BLANK” cuvette and place the “100 ppm” standard cuvette in the adapter well. Close the lid.
9. Press <ENTER>.
10. Press the <STD VAL> button.
11. Press <↑> or <↓> until the STD VAL is set to 100.0 ppm.
12. Press <ENTER>.
13. Press the <DIAG> button and record the values of %FS-BLK and %FS-STD.
14. If the %FS-STD value is below 3 %, switch to the large-aperture 8 mm cuvette adapter (PN 102935) and repeat steps 4 through 9.

**NOTE:** If you wish to make measurements greater than 100 ppm, save the unused portions of the “1000 ppm” and “100 ppm” standards for later use. See **Section III.6.**

## **III.5 Analysis: 0-100 ppm Oil-in-Water**

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### **III.5.1 Prepare the “WSO” Sample**

1. Open the process sample valve and allow water to run for at least 15 seconds.
2. Fill the bottle labeled “WSO” to the neck with process water.

**NOTE:** Make sure that the “WSO” bottle is clean and dry and that no OIW Surfactant has been added.

3. Cap the bottle and allow it to cool to room temperature.
4. Fill a 3 mL syringe with “WSO” water.
5. Attach a syringe filter to the end of the syringe.
6. Press the syringe plunger to force liquid through the filter and into the “WSO” cuvette. Continue until the cuvette is  $\frac{1}{2}$  to  $\frac{3}{4}$  full.

### **III.5.2 Prepare the “OIW” Sample**

1. Add 3 mL of OIW Surfactant to the “OIW” bottle.
2. Open the process valve and purge the sample valve by allowing the water to flow at a high rate for at least 1 minute.
3. Adjust the sample valve until the water is flowing at a rate of ~10 mL per second. Allow the water to flow at this rate for at least 15 seconds.
4. Allow water to flow into the “OIW” bottle until the liquid level reaches the 100 mL mark.
5. Cap the bottle and shake it vigorously for at least 15 seconds.

6. If the contents of the bottle are uniformly milky-white, it is not necessary to perform Steps 7 through 9. You may skip to Step 10.
7. Place the “OIW” bottle in the hot water bath and heat it until the contents of the bottle are uniformly milky-white.
8. Put on heat-resistant gloves suitable for handling glass bottles at boiling water temperature (212 °F, 100 °C).
9. Remove the bottle from the hot water.
10. While the contents are still milky-white, shake the bottle vigorously for at least 30 seconds.
11. Allow the “OIW” sample to cool until the contents change from milky-white to clear.

**NOTE:** You may accelerate cooling by placing the “OIW” bottle in a refrigerator or a container of cold water. You may also mix the sample thoroughly and immediately pour ~5 mL into a small disposable beaker. The small volume will cool more quickly than the original sample bottle.

12. Fill a 3 mL syringe with the “OIW” sample.
13. Attach an unused syringe filter to the end of the syringe.
14. Press the syringe plunger to filter the liquid into the 8 mm cuvette marked “OIW”. Continue until the cuvette is ½ to ¾ full.

### **III.5.3 Measurement**

15. Turn on the TD-500D and wait 5 seconds for the warm-up count-down to expire. The word “READY” will appear on the first line of the display. Also, a letter (A or B) indicating the measurement channel will appear on the lower left.

16. Make sure that the TD-500D is set to Channel A. If you see a “B” displayed instead of an “A”, press the <A/B> key until an “A” appears.
17. Make sure that the 8 mm cuvette adapter that is in the adapter well is the same one that was used for calibration.
18. Wipe the “OIW” and “WSO” cuvettes with a Kimwipe to remove any fingerprints, dirt or liquid droplets that might be present.
19. Place the “OIW” cuvette in the adapter well and close the lid.
20. Press the <READ> button.
21. Record the OIW Reading.
22. Place the “WSO” cuvette in the adapter well and close the lid.
23. Press the <READ> button.
24. Record the WSO Reading.

#### **III.5.4 Calculation**

The OIW Reading includes contributions from both dispersed oil and water-soluble organic compounds (WSO). The WSO Reading is due only to WSO. The dispersed oil concentration ( $C_{\text{dispersed}}$ ), in units of ppm, can be calculated by subtracting the WSO Reading from the OIW Reading, as shown in *Equation III.5.1*.

*Equation III.5.1:*

$$C_{\text{dispersed}} = \text{OIW Reading} - \text{WSO Reading}$$



## III.6 Extended Range Measurements— Channel A

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### III.6.1 Linear Range Determination

While all oils give linear response on Channel A up to a dispersed oil concentration of 100 ppm, many give linear response to higher oil concentrations. You can establish the linear range of your oil by preparing a few additional standards and measuring them with the TD-500D calibrated with the “100 ppm” standard (**Section III.4**).

Use the following procedure to establish the linear range up to 1000 ppm:

1. Make sure that the TD-500D is set to Channel A, and that you have already calibrated it with your “100 ppm” standard.
2. Prepare an additional “1000 ppm” standard as described in **Section III.4.2**, steps 1 through 8. Combine this 1000 ppm standard preparation with the unused portion of the original 1000 ppm standard that you used to calibrate the instrument. You need approximately 170 mL of 1000 ppm standard to complete this procedure.
3. Dilute the 1000 ppm standard with “BLANK” solution (**Section III.4.2**) to create 4 additional standards with concentrations of 750 ppm, 500 ppm and 250 ppm and 100 ppm. See *Table III.6.1* for the required volumes.

Table III.6.1

Standard	Volume of 1000 ppm Standard	Dilute with "BLANK" to:
750 ppm	75 mL	100 mL
500 ppm	50 mL	100 mL
250 ppm	25 mL	100 mL
100 ppm <sup>1</sup>	10 mL	100 mL

<sup>1</sup>You may also use the "100 ppm" standard used for calibration.

4. Fill a 3 mL syringe with the 1000 ppm solution.
5. Attach an unused syringe filter to the end of the syringe.
6. Press the syringe plunger to filter the 1000 ppm solution into an 8 mm cuvette. Continue until the cuvette is  $\frac{1}{2}$  to  $\frac{3}{4}$  full.
7. Label the cuvette "1000 ppm". Confine the labels to the top 0.5" (13 mm) of the cuvette to keep the mark out of the measurement zone.
8. Repeat steps 4 through 7 for the other standards listed in *Table III.6.1*, labeling each cuvette with the appropriate concentration. Also fill an 8 mm cuvette with "BLANK" solution, which represents an oil concentration of 0 ppm.
9. Turn on the TD-500D and wait 5 seconds for the warm-up count-down to expire. The word "READY" will appear on the first line of the display. Also, a letter (A or B) indicating the measurement channel will appear on the lower left.
10. Make sure that the TD-500D is set to Channel A. If you see a "B" displayed instead of an

“A”, press the <A/B> key until an “A” appears.

11. Make sure that the 8 mm cuvette adapter that is in the adapter well is the same one that was used for calibration.
12. Wipe each standard cuvette with a Kimwipe to remove any fingerprints, dirt or liquid droplets that might be present.
13. Place the “BLANK” cuvette in the adapter well and close the lid.
14. Press the <READ> button.
15. Record the “BLANK” (0 ppm) reading.
16. Repeat Steps 13 through 15 for the 1000 ppm standard and each of the standards listed in *Table II.6.1*.

For the purpose of explanation, example readings are listed in *Table III.6.2*.

**NOTE:** The readings in *Table III.6.2* are for example only. Your readings will be somewhat different.

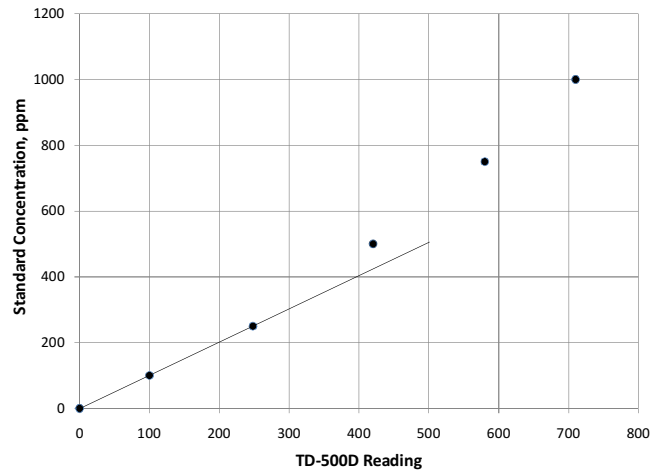
*Table III.6.2*

TD-500D Reading	Standard Concentration
710	1000 ppm
580	750 ppm
420	500 ppm
248	250 ppm
100	100 ppm
0	0 ppm (BLANK)

17. Enter the data from your measurements into a spreadsheet program (e.g. Microsoft Excel™).

18. Plot the data in a chart with the TD-500D Reading on the X-axis and the Standard Concentration on the Y-axis.
19. Draw a straight line that passes through the origin and as many data points as possible. Estimate the linear range. The example data from *Table III.6.2* is plotted in *Figure III.6.1*. The linear range is approximately 250 ppm.

**Figure III.6.1**



### III.6.2 Measurements Beyond the Linear Range

The TD-500D can also make accurate oil concentration measurements above the linearity limit through the use of a non-linear calibration function.

At oil concentrations above the linearity limit, TD-500D fluorescence response (R) follows the non-linear relationship shown in *Equation III.6.1*.

Equation III.6.1

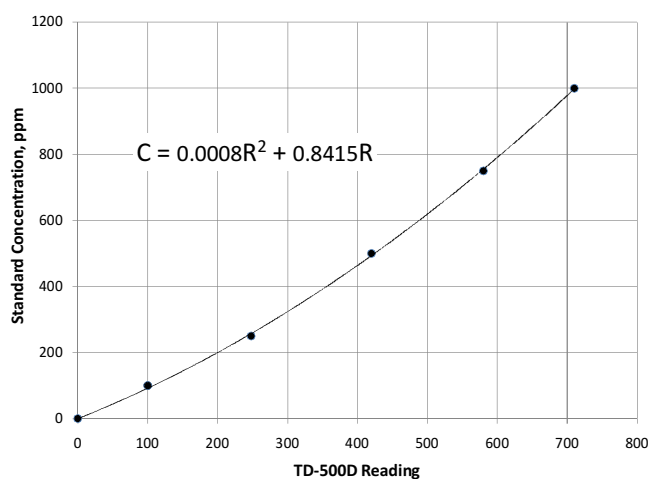
$$C_{\text{dispersed}} = aR^2 + bR$$

$C_{\text{dispersed}}$  is dispersed oil concentration and “a” and “b” are calibration constants.

The calibration constants “a” and “b” can be established by fitting the data from the linear range determination (Section 18, **Linear Range**

**Determination**) to a 2<sup>nd</sup>-order polynomial. This can be done with most spreadsheet programs (e.g. Microsoft Excel™). The 2<sup>nd</sup>-order polynomial that fits the example data in *Table III.6.2* is illustrated in *Figure III.6.2*. The fit was forced through the origin to eliminate the third term that normally results from a 2<sup>nd</sup>-order polynomial fit.

Figure III.6.2



In this example, the value of “a” is 0.0008 and the value of “b” is 0.8415.

The TD-500D cannot be calibrated directly with a 2<sup>nd</sup>-order polynomial function. Therefore, oil concentrations above the linearity limit must be computed externally from readings taken from the TD-500D display. Use the following procedure:

1. Calibrate the TD-500D using the “100 ppm” standard (**Section III.4**).
2. Perform the linear range determination (**Section III.6.1**). Record the linearity limit.
3. Use a spreadsheet program to fit the linear range data with a 2<sup>nd</sup>-order polynomial. Remember to force the fit through the origin (0,0). (In Microsoft Excel™ this is done by checking the “Set intercept to =” checkbox and entering the value of “0.0” in the adjacent field.)
4. Record the values of “a” and “b”. Refer to the example in *Figure III.6.2*.
5. Prepare an “OIW” sample and a “WSO” sample and record the OIW Reading and WSO Reading (**Section III.5**).
6. If OIW Reading is less than or equal to the linearity limit. Use *Equation III.5.1* to compute the dispersed oil concentration.
7. If the OIW Reading is greater than the linearity limit, compute the dispersed oil concentration using *Equation III.6.1*. Use *Equation III.6.2* below to compute the value of R.

*Equation III.6.2*

$$R = \text{OIW Reading} - \text{WSO Reading}$$

The following examples are based upon the example data presented above (*Table III.6.2*, *Figures III.6.1* and *III.6.2*), which result in the following values:

Linearity Limit = 250 ppm

a = 0.0008

b = 0.8415

#### Example 1 – Linear Range Measurement

Sample Readings:

OIW Reading = 210

WSO Reading = 10

Since the OIW Reading is less than the linearity limit ( $210 < 250$ ), use *Equation III.5.1* to compute the dispersed oil concentration:

$$C_{\text{dispersed}} = 210 - 10 = 200 \text{ ppm}$$

#### Example 2 – Extended Range Measurement

Sample Readings:

OIW Reading = 520

WSO Reading = 10

Since the OIW Reading is greater than the linearity limit ( $520 > 250$ ) use *Equation III.6.2* to compute the value of R.

$$R = 520 - 10 = 510$$

Then use *Equation III.6.1* to compute dispersed oil concentration.

$$C_{\text{dispersed}} = 0.0008(510)^2 + 0.8415(510) = 637 \text{ ppm}$$

### **III.7 Extended Range Measurements— Channel B**

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Channel B is provided for the measurement of extremely high concentration samples (up to 10,000 ppm for most crude oils). Use Channel B if your linear range determination with Channel A (**Section III.6.1**) gives a reading of “Over” for any standard that is within your required analytical range.

You will usually achieve the best analytical precision if you use Channel A whenever possible, even if you must make measurements beyond the linear range (**Section III.6.2**). Consult Turner Designs Hydrocarbon Instruments, Inc. for details.

**Section III.3**, steps 5 and 6 describe how to set the TD-500D to Channel B. All the procedures, equations and cuvette adapter selection guidelines described for Channel A also apply to Channel B.



### **III.8 Equipment Clean-up and Re-use**

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1. Clean the sample bottles with the brush and rinse them thoroughly with distilled water.
2. Drain all the rinse water from the sample bottles.
3. If necessary, the syringes can be rinsed with distilled water, dried and re-used.
4. Discard all used syringe filters.

**NOTE:** Although the sample bottles can be re-used from either the solvent or no-solvent procedures if properly cleaned, do not re-use syringes, pipette tips or transfer pipettes used in the solvent method.