

HM-900 Oil-in-Water Analyzer Operation Manual



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The Pyxis warranty term is thirteen (13) months ex-works. In no event shall the standard limited warranty coverage extend beyond thirteen (13) months from original shipment date.

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Damaged or dysfunctional instruments may be returned to Pyxis for repair or replacement. In some instances, replacement instruments may be available for short duration loan or lease.

Pyxis warrants that any labor services provided shall conform to the reasonable standards of technical competency and performance effective at the time of delivery. All service interventions are to be reviewed and authorized as correct and complete at the completion of the service by a customer representative or designate. Pyxis warrants these services for 30 days after the authorization and will correct any qualifying deficiency in labor provided that the labor service deficiency is exactly related to the originating event. No other remedy, other than the provision of labor services, may be applicable.

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A Repair Authorization Number (RA) must be obtained from Pyxis Technical Support before any product can be returned to the factory. Pyxis will pay freight charges to ship replacement or repaired products to the customer. The customer shall pay freight charges for returning products to Pyxis. Any product returned to the factory without an RA number will be returned to the customer.

Pyxis Technical Support

Contact Pyxis Technical Support at service@pyxis-lab.com.

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1 Introducing the Pyxis HM-900 oil-in-water analyzer

The Pyxis HM-900 is a handheld portable fluorometer that measures the oil-in-water concentration in a sample. Oil is a complex mixture of organic compounds. The term "oil" can be referred to as a range of different classes of chemical compounds, such as vegetable oil and petroleum oil. Almost all oil substances fluoresce under UV or near UV excitation in a different degree because invariably, all oil contains aromatic or other fluorescent compounds.

<u>Two Extraction Methods</u> - The HM-900 offers measurement of oil content though extraction to hexane and other organic solvents as preferred by the user. The fluorescence intensity of the solvent sample containing oil is then analyzed by the HM-900. The HM-900 offers two extraction methods that can be utilized as outlined in this manual.

- <u>24mm Vial Extraction Method</u> Enables the user to self-prepare their own hexane extracted sample for measurement.
- <u>16mm Vial Extraction Method</u> Enables the user to utilize the Pyxis 16mm prepared nonflammable solvent vial and adapter for a safer and more convenient measurement reducing sample preparation steps.

<u>Built In Default Curves</u> - The HM-900 has three default built-in calibration curves for Marine Crude Oil, #1 Diesel/Kerosene, and Heavy Fuel Oil. The user may select which of these three formats are desired for final measurement display. Users may also create up to 7 customized/user defined calibration curves for the specific oil desired.

<u>Common Alternative Methods</u> - Oil-in-water analysis by fluorescence is a widely used field-testing method but is fundamentally different from other methods. Commonly used alternative methods include the following:

- EPA 1664 gravimetric method measures the amount of oil extracted to a solvent by weight.
- The Infrared method (ASTD D7678) measures the absorption of oil extracted to cyclohexane in the range of 1370–1380 cm-1 by methyl group in the oil, using an IR spectrophotometer to qualify the oil concentration.

<u>Solvent Extraction Method - User Defined Calibration Curves</u> - Due to the HM-900 portable fluorometer utilizing the fluorescent signature of the oil in water, the concentrations measured by the HM-900 versus other methods for a given water sample "may" agree with each other in a relative sense, but this is not always a guarantee. As such, the HM-900 enables users to create their own "user defined" calibration curves based on the specific organic solvent and vial size desired. This manual covers the procedures for integrating a user defined calibration curve for future calibration.

<u>Secondary Standard for Rapid Calibration</u> – Preparation of a true oil in water calibration standard is both time consuming and subject to error. As such, the HM-900 can also be calibrated using Pyxis synthetic OIW-500 (oil in water) secondary standard. This approach enables users to utilize a synthetic oil in water standard for rapid device calibration, improved accuracy and extended storage / shelf life of the calibration solution.

<u>Direct Read Method For Dissolved or Emulsified Oil</u> - The HM-900 can be used to directly analyze oil in water content without the need for extraction using UV fluorescence methodology. This method is commonly utilized for samples containing dissolved or lightly emulsified oil.

2 Features

- Preprogrammed calibration curves for primary oil classes
 - Marine Crude Oil / #1 Diesel-Kerosene / Heavy Fuel Oil
- Dual Channel Wavelengths of Fluorescence enable automatic range selection
- Both 24 mm and 16 mm sample vials may be used with HM-900
- Easy to follow calibration steps displayed on the screen
- Suitable for solvent extraction methods
- Suitable for direct read dissolved/emulsified oil methods

3 Specification

Item	Parameter	
Power Supply	4 alkaline batteries	
Wavelength 365/470 (ex/em) and 470/650 (ex/em)		
Detection Limit 0.1 ppm (Marine offshore oil)		
Range	0.1 – 1000 ppm, auto channel switch	
Battery Life	2 months	
Display	192 x 128 LCD, readable under direct sunlight	
Weight 1.1 pound (510 g)		
Dimension	L 265 mm W 88 mm H 62 mm	
Temperature	Working: 14 - 122 °F (-10 - 50 °C) / Storage: -4 - 158 °F (-20 - 70 °C)	
Enclosure Rating	IP65	
Regulation	CE	

4 Unpackaging the Instrument

Remove the instrument and accessories from the shipping container and inspect each item for any damage that may have occurred during shipping. Verify that all items listed on the packing slip are included. If any items are missing or damaged, please contact Pyxis Customer Service at <u>service@pyxis-lab.com</u>.

4.1 Standard Accessories

- Two 10mL Sample Vials 24mm Diameter (P/N MA-24)
- One 16 mm Vial OIW Adapter (P/N 52213)
- Four AA Alkaline Batteries
- Instrument Manual, also available from www.pyxis-lab.com

Item	16mm Solvent Filled Extraction Vial	Oil-In-Water Secondary Standard Solution
Product	16mm-OIW	OIW-500
Part 52211 Number		21036
Range* 0-1000ppm		500ppm as Oil in Water
Shelf Life	6 months	6 months
Container	16mm Extraction Vial Prefilled with non- flammable solvent	16 oz/500mL Nalgene Amber Narrow Mouth Sample Bottle
Volume (net)	12 vials /~5mL each	510 +/- 10mL
Weight (net)	12 vials /~5g each	510 +/- 10g

4.2 **Optional Accessories**



* Undiluted Range is specified above.

5 Sample Vial Compartment

The sample vial compartment is shown in Figure 1, along with a 10 ml sample vial. When the sample vial is inserted into the sample vial compartment, the triangular mark on the sample vial should be aligned approximately with the 6 o'clock position of the sample vial compartment or any position consistently.

The sample vial compartment should be kept clean. A small amount of foreign material could significantly affect turbidity and fluorescence measurement results. Use a soft cloth or lint free paper tissue to clean sample vial compartment periodically. Remove debris, scale, and deposit promptly.

6 Light Shield Cover

The light shield cover is shown in Figure 1. The light shield cover can be conveniently slid between open and closed positions. The light shield cover is held firmly in these positions by permanent magnets.

The light shield cover should be in the closed position during storage, transportation, and measuring, especially while performing fluorescence measurements. When turned on, the HM-900 performs a self-diagnosis including a check of the performance of the optical devices. The light shield door should always be in the closed position to shield interference from ambient light during self-diagnosis. Care should be taken to avoid water or debris being trapped in the track of the light shield door.

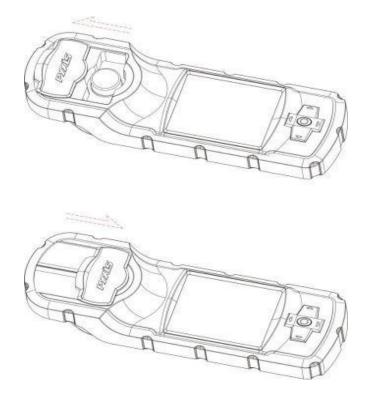


Figure 1. Open and Close the Light Shield Cover

Warning

Magnetic sensitive devices, including but not limited to, credit cards, watches, hard disks, should be kept at a distance of at least 2 inches from the Light Shield to avoid possible damage and/or loss of stored data.

7 Start The HM-900

7.1 Battery Installation

The HM-900 is powered by four AA-size alkaline batteries. <u>Do not use rechargeable nickel cadmium</u> (NiCad) batteries or any AA-size lithium batteries. A set of batteries typically lasts for three months. When the battery capacity is low, the HM-900 will prompt a LOW BATTERY warning. Replace all four batteries to resume operation of the HM-900 after the battery warning.

The HM-900 battery compartment, shown in Figure 2, is on the back side of the instrument. Insert a small pad underneath the screen area to make the back-surface level when the instrument is turned upside down. Install batteries as following:

- 1. Remove the battery compartment cover by loosening four screws.
- 2. Insert four batteries into the battery holder as shown in Figure 2. Make sure the positive battery polarity marker (+) is aligned with the positive marker (+) on the battery holder.
- 3. Replace the battery compartment cover, making sure that the sealing O-ring is lying flat on the battery holder and tighten the four screws.

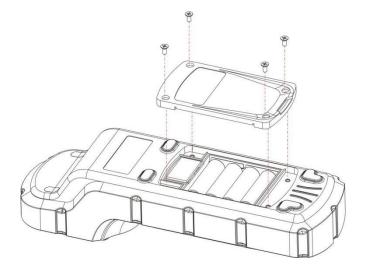


Figure 2. Replace batteries

7.2 Description of the Navigational Control Pad

The HM-900 navigational control pad consists of five keys as shown in Figure 1. The left, right, up, and down keys are navigational keys that are used to select an icon, a button, or other items in various pages. The center key is the OK key. Press the OK key on a selected item to launch the action associated with the selected item. The OK key is also used to accept the current selection, like the ENTER key on a computer keyboard.

7.3 Turning on the HM-900

After new batteries are installed, the HM-900 <u>will not be automatically turned on</u>. To turn on HM-900, press the OK key, and release the OK key when the LCD is lit.

You can navigate the main page menu and launch an operation by pressing on an icon. If battery voltage is too low for the instrument to work properly, the HM-900 will show a low battery warning message when it is being turned on. If this happens, replace all four batteries as outlined in Section 7.1.

7.4 Main Page

The HM-900 provides intuitive icon driven user operations. On the main page, four major feature groups are illustrated as below:

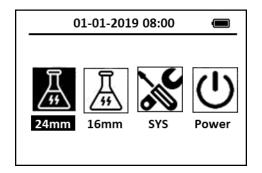


Figure 3. Main Page

A brief description of each feature group is given in Table 1. Detailed operation instructions can be found in the following chapters.

Title	Description
24 mm	24 mm Vial Extraction
16 mm	16 mm Vial Extraction
SYS	Diagnosis Information
STBY	Turning ON and OFF HM-900

Table 1 Feature Groups on Main Menu

7.5 Turning Off The HM-900

Turn the HM-900 off by navigating to Power icon and press the OK key. Alternatively, you can turn off the HM-900 by pressing OK key for 5 seconds in any menu.

7.6 The HM-900 Auto Power Off

To conserve battery life, the HM-900 automatically turns itself off with no-key activity for a given period, except during a measurement.

7.7 Auto LCD Power Saving

The HM-900 automatically turns LCD backlight off with no-key activity. Pressing any key will turn on the LCD backlight. Under normal ambient lighting conditions, icons and other contents shown on the LCD screen are readable without the backlight being on.

8 Oil-in-Water Measurement

The HM-900 has three default built-in calibration curves for Marine Crude Oil, Kerosene (#1 Diesel) and Heavy Fuel Oil. The user can measure the raw fluorescence signal for a sample with a known oil-in-water concentration and create up to 6 user-defined methods.

8.1 24mm Vial Extraction Procedure

- 1. Adjust the sample pH down to between 1 and 2 using diluted sulfuric acid or hydrochloric acid.
- Pour 100 ml of the pH adjusted sample to a 250 ml glass separatory funnel. Using a pipette, add 10 ml hexane solvent to the separatory funnel. *NOTE* The sample volume, funnel size, and the hexane volume should be user determined according to the oil concentration of the sample being measured as listed in Table 2.
- 3. Shake the separatory funnel for two minutes. After shaking, allow the upper hexane layer in the separatory funnel <u>to float and separate</u> from the bottom aqueous layer for a period of one minute. Pure the separated upper hexane layer into a 10 ml sample vial. This will be your reacted sample to be tested.



Hold stopper and stopcock

- 4. Pour another 10 ml of hexane <u>solvent only</u> into second 10 ml sample vial to be used as the solvent blank. Place the solvent blank vial to the HM-900 sample compartment and press the OK key on the 24 mm icon. Cycle through the oil type list using the left or right key to select an oil type (i.e. a calibration curve) desired for this test. Press the OK key on Zero to measure the solvent blank.
- Place the 10 ml sample vial containing the hexane extraction (reacted sample from the separatory funnel in step 3) into the HM-900 sample compartment and press the OK key on Read. The oil-in-water concentration in hexane extraction will be displayed as seen in Figure 4.
- 6. Multiply the final reading by the multiplication factor according to the hexane and sample volume ratio listed in Table 2 to calculate the oil concentration in the sample.

Measurement	
Offshore oil	
265.3 ppm	ZERO
	READ
	CONF
	ABSB
	EXIT

Figure 4. Final Reading Display – Multiply by Multiplication Factor from Table 2.0

Operation Manual	

1.0

Table 3 - Final 16mm Measurement Conversion	n Table Based on Sample Size
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concentration in the extraction solvent.

the final oil concentration in the sample.

6

Sample volume (ml)	Multiplication factor	
3	0.5	

Pyxis HM-900

Estimated Sample Oil Concentration (ppm) Recommended Sample Volume (ml)		Recommended Hexane Volume (ml)	Multiplication Factor
0 - 10	1000	10	0.01
10 - 100	100	10	0.1
100 - 1000	100	100	1

Table 2 - Measurement Conversion Table Based on Sample Size & Hexane Volume Used (24mm Vial)

8.2	16mm	Vial	Extraction	Procedure
0.2	TOULUU	viai		FIUCEUUIE

For easier more efficient testing, this method requires the 16mm vial/test tube adapter (P/N - 52213) and Pyxis 16mm prepared extraction vials (P/N - 52211) with pre-filled extraction solvent as shown in Figure 5. The solvent used in this method is <u>3M ethoxy-nonafluorobutane</u>. It is a non-flammable, low toxicity solvent.

- Insert the 16 mm vial adapter (P/N 52213) into the sample compartment as shown in Figure 6.
- Using a pipette add 3 or 6 ml of sample to be tested to a Pyxis 16 mm extraction vial with pre-filled extraction solvent. Shake the vial for 2 minutes and let the vial settle for one minute. This will be your prepared sample vial for final measurement. *NOTE* Users may choose 3 or 6 ml of sample depending on the projected concentration of oil. (Example For higher concentration samples use 3 ml.)
- 3. Using a pipette add the same amount DI Water to another Pyxis 16 mm extraction vial with pre-filled extraction solvent. Shake the vial for two minutes and let the vial settle for one minute. This will be your solvent blank vial.
- 4. Place the extraction solvent blank vial to the 16 ml vial adapter. Press the OK key on the 16 mm icon to launch the method. Cycle through the oil type list to select an oil type (a calibration curve) using the left or right key. Press the OK key on Zero to measure the solvent blank.

5. Place the sample vial into the adapter and press the OK key on Read to measure the oil

6. Multiply the displayed value according to the sample volume listed in Table 3 to calculate





Figure 5. – Pyxis 16 mm extraction vial prefilled with solvent (P/N – 52211) 16mm Vial Adapter (P/N – 52213)

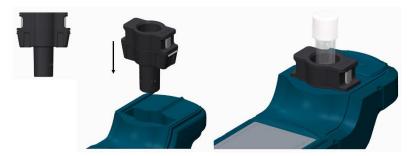


Figure 6. 16 mm adapter

Cautions

- Use a glass container to transfer oil-in-water samples.
- Avoid bulk surface oil when taking a sample.
- Analyze the sample within 3 hours after taking it.
- Take at least 1000 ml sample to be representative.
- Make sure the separatory flask is well-sealed to prevent hexane solvent evaporation during the extraction process.

<u>Over-Range Warning</u> - The HM-900 automatically selects the proper oil concentration range according to the selected calibration curve. If the oil concentration is too high and over the range of the selected calibration curve, a warning message will be displayed. Dilute the sample or use a different sample-to-solvent ratio listed in tables 2 and 3 in the extraction process.

Figure 7. Over Range Warning

Measurement	
Offshore oil	
WARNING:	ZERO
Dilute sample	READ
	CONF
	ABSB
	EXIT

8.3 Create User Defined Calibration Curves

When creating your own user defined calibration curves, please use hexane or other organic solvents that are not contaminated by oil. Please prepare a series of calibration solutions (a minimum of two calibration solutions are required to include the solvent blank). Use the solvent and the target oil type desired.

1. Press the 24 mm or 16 mm lcon and use the left or right key to select a user defined method among 7 templates (named User 1 to 7). Press the OK key on the highlighted icon CONF as shown in figure 8.

Measurement	
USER DEFINE 1	7500
	ZERO
	READ
	CONF
	ABSB
	EXIT



2. Place the vial containing the solvent blank into the HM-900 sample compartment. Press the OK key on the highlighted icon CALC to measure and accept the solvent blank as shown in figure 9.

Configure	
USER DEFINE 1	
Input: Blank	CALC
	SAVE
Calc OK	EXIT

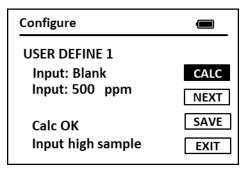


3. Place the calibration standard into the HM-900 sample compartment. Input the standard oil concentration using the four keys around the central OK key as shown in figure 10.

Configure	
USER DEFINE 1	CALC
Input: Blank	SAVE
Input: 500 ppm	EXIT



4. After entering the known value of sample as input, press the OK key on the highlighted icon CALC to accept the first calibration solution. The first range calibration is successfully stored as shown in figure 11.





5. To continue the calibration entry points using the second calibration standard solution, place the second calibration standard into the HM-900 sample compartment and repeat the above process by entering in the known value of the second standard. Then press the OK key when highlighted on the CALC icon to accept the second calibration standard (Figure 12). If a second range calibration is not required, press the OK key on the icon SAVE to save the first range calibration, then press OK key on the icon EXIT to exit. *NOTE* - The concentration of the second calibration solution must be higher than the first calibration solution.

Configure	
USER DEFINE 1	
Input: Blank	CALC
Input: 1000 ppm	SAVE
Complete	EXIT



6. Once finishing the whole calibration process, a "Saved" message will be displayed on the screen. Press the OK key on the icon EXIT to exit the measurement interface as shown in figure 13.

Configure	
USER DEFINE 1 Input: Blank Input: 1000 ppm Saved	CALC SAVE EXIT

Figure 12

User Defined Calibration Curve Notes & Considerations

<u>Contaminated Extraction Solvent</u> - Make sure the extraction solvent is not contaminated. You can do this by using a preprogrammed default calibration curve to measure the solvent directly as though it is an extraction test. The results will determine if the solvent is contaminated with oil. If a significant oil concentration is measured, the solvent is likely being contaminated and should not be used for development of user defined calibration curves.

<u>Oil Concentration Too High For Measurement</u> - If the oil concentration in a user developed standard is too high, a warning message will be displayed. Please add more solvent to dilute the calibration standard to appropriate range of detection of the HM-900.

8.4 Calibration using a Secondary Standard

Technically a universal oil-in-water standard <u>does not exist</u>. The user can use the default three built-in, pre-calibrated methods to measure a sample. The user can create a user-defined method pertaining to a specific sample type as described in the above section. To avoid the need to prepare an oil-in-water standard, the user can conveniently use a synthetic secondary standard to calibrate the user defined methods. The equivalent concentration value corresponding to each "user defined method" must be determined after the "user defined method" is created and recorded for later use. The equivalent concentrations of the Pyxis OIW-500 secondary standard for the three built-in oil types (pre-calibrated methods) are listed below.

Default Oil Type Configured in HM-900	OIW-500 Secondary Standard Equivalent ppm Value
Offshore	25
#1 Diesel/Kerosene	100
Heavy oil	5

Table 4 – OIW-500 Secondary Standard Equivalent ppm using Pre-Configured Method

8.4.1 Determine the equivalent concentration of the Pyxis OIW secondary standard for a user-defined method

- 1. Repeat the steps in section 8.3 to define a user-defined method.
- 2. Repeat the steps in section 8.1 or 8.2 to determine the equivalent concentration of the Pyxis OIW secondary standard. <u>Record this concentration and the associated user defined method name on the secondary standard bottle for future calibrations.</u>

8.4.2 Calibrate a user defined method using the secondary standard

- 1. Press the OK key on the 24 mm or 16 mm Icon and select a method (default or user defined) using the left or right key.
- 2. Place a vial filled with the solvent blank to the sample compartment to ZERO
- 3. Place the vial containing the secondary standard to the sample compartment. Press the OK key on READ to read the concentration (figure 4).

- 4. Press the OK key on CONF to launch the calibration page and Press the OK key on CALC to start the calibration.
- 5. Adjust the equivalent concentration using the four keys around the central OK key as shown in figure 10. The equivalent concentration of the secondary standard corresponding to each default oil type is listed above. The equivalent concentration of the secondary standard corresponding to the user defined methods are determined in section 8.4.
- 6. Press OK key on SAVE to save the calibration.

8.5 Dissolved or Dispersed Oil Measurement

Water samples containing dissolved oil or lightly dispersed/emulsified oil can be measured directly without extracting oil to hexane or other organic solvents. A user defined calibration curve for the dissolved oil measurement can be created with the same procedure as shown in the section above. Calibration standards can be prepared by emulsifying a given amount of oil into deionized water. Alternatively, the oil concentration of a real water sample can be first analyzed by the extraction procedure and the sample can be directly used as a calibration standard to calibrate the direct non-extraction procedure.

Some non-homogenous water samples may need to be emulsified with an addition of surfactants.

Contact us

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