# Light Gas Condensate in Produced Water Measurement
# New TD-560 Field Sample Analyzer Demonstration

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## Background

A European oil company, which operates many oil and gas production facilities around the globe, either discharges produced water from its facilities to a local sea or injects it into a production or disposal formation, depending on the local requirements. The discharged water quality at these facilities must be monitored for oil concentration to control process upsets and environmental issues.

The company stated the need for a field method for use in its production facilities. It requested a demonstration of the Turner Designs Hydrocarbon Instruments (TDHI) oil in water online monitors or sample analyzers to learn how they can be used for its water processes. The light natural gas condensate produced in the company's Adriatic production fields is the primary challenge and what was requested to be used in the demonstration. Initially, the company suggested that a sample of produced water could be shipped to TDHI for test and analysis. Due to the challenges of shipping samples and maintaining the water chemistry during shipment, TDHI determined that any testing should be performed in the company's home country, under controlled conditions.

Oil concentration in water can be measured using many different methods. Local government regulations usually require a specific laboratory method, which is difficult to perform in the field. Each method used will demonstrate a different sensitivity and bias to the hydrocarbon mixture that makes the oil. Additionally, only under controlled conditions will two water samples have the same oil concentration. For these reasons, when comparing the measurement of oil concentration in water samples, field and laboratory methods will produce different responses. Most methods produce a linear response to the oil concentration, allowing for a correlation curve to compare the different methods.

Demonstrating a field method for measuring oil in water concentrations requires producing samples with known oil concentrations. The produced water process is inherently unstable because oil and water don't mix. This instability causes variations in oil concentration between two different water samples. Thus, collecting and preparing two identical oily water samples from a process is extremely difficult.

Demonstrating a sample analyzer is also complicated because sample preparation methods require accurate manual measurement of the water sample volume, a known volume of an extraction solvent added, and then identical shaking so the solvent extracts the oil in the same manner for both samples. Each of these steps can cause variation in the result. The demonstration method must reduce the chance for error in the results caused by sample preparation.

TD-560

Demonstrating online monitors is often cost prohibitive for a preliminary test because they require careful and safe installation. A sample analyzer, however, has no installation requirements and is significantly easier to demonstrate. Both the online monitors and sample analyzers from TDHI use the same UV Fluorescence technology and have similar measurement capabilities. Any performance capabilities of the sample analyzers will be similar to the online monitors.

### Test and Demonstration Setup

During the discussions prior to the demonstration, the oil company was given a written set of procedures. These procedures were intended to be a guide and allow the company to modify them as needed for its use. These procedures are in *Appendix A* of this report.

Field analytical methods always include a water sample preparation step. Oil and water will naturally separate in a sample bottle, so by the time the water sample is analyzed, much of the oil is floating on the water surface or stuck, as solids, on the bottle or lid.

The first phase of demonstration was started by manufacturing samples with known oil concentrations in an extraction solvent or surfactant. The oil company chose two different light oil samples and three different sample preparation methods. These prepared samples were analyzed, using the TD-560 Oil in Water Sample Analyzer and a GC-FID or infrared analyzer, as appropriate for the solvent used.



The second phase of the demonstration used a produced water sample split into separate bottles to demonstrate how water samples can be analyzed and how much variability can be expected between water samples. After splitting the samples, a known oil volume was added to each of the separate bottles. Each sample bottle was prepared for sample analysis by the TD-560 and GC-FID or infrared analyzer. The details of measurement and sample preparation learned in the first phase allowed for simplification of this second phase by eliminating a few samples and preparation methods.

The final phase was the correlation of the field method to the laboratory method. The correlation of the field method to the GC FID requires collection and analysis of multiple samples that are as similar to each other as possible. In a typical correlation study, eight samples are collected in quick sequence so they will be as similar as possible. Four will be analyzed by the field analyzer, and four by the laboratory (GC FID). The GC FID analysis usually requires shipping samples to an accredited laboratory and waiting for the results. When the measured concentrations of the four samples from the two different methods are averaged, a correlation ratio is calculated to know how the two methods compare.

Because this demonstration was performed in the company's laboratories, the GC FID analysis and field analyzers were in the same location. This allowed for easy coordination of the samples analyzed. For this study the extracted solvent from the produced water samples was analyzed by the TD-560 as well as the GC FID, instead of two separate sample sets.

The TD-560 sample analyzer uses UV Fluorescence to measure BTEX, light gas condensates, refined fuels, and crude oil from concentrations less than 1 ppm to more than 1000 ppm in some cases. The TD-560 has Deep UV and Near UV measurement channels. The Deep UV is more sensitive to BTEX and natural gas condensates and is ideal for low concentrations. The Near UV is best for heavier fuels and crude oil and is ideal for higher concentrations. In the first phase of the test, the ideal channel will be determined.

The TD-560 is compatible with all commonly used extraction solvents and the TDHI No Solvent Method, using the OIW surfactant. The oil company has preferred to use a non-flammable solvent, S-316, for the field analysis. However, the GC-FID uses hexane for the extraction solvent. Because the No Solvent Method is less toxic and flammable than hexane the company also wanted to test it for field analysis. For these reasons, samples in hexane, S316 and the OIW surfactant were manufactured.

The suggested demonstration procedure supplied instructions on how to make samples of 50, 100, 150, and 200 ppm. However, because the concentrations typically measured in the field are below 50 ppm, the company adjusted the samples to 10, 50, 100, and 150 ppm.

## Sample Manufacturing and Analysis

The sample preparation began with a single oil sample described as Gasoline B. Stock standards of 10,000 ppm Gasoline B in Hexane and S-316 as well as a 1000 ppm Gasoline B in OIW surfactant and deionized water were manufactured. Typical water preparation procedures use a 10:1 water to solvent extraction procedure. 10,000 ppm oil in solvent is equivalent to 1000 oil in surfactant and water.

With the stock standards made, samples of 100, 500, 1000 and 1500 ppm in solvent were then manufactured. The details of the sample manufacturing are included in *Appendix A*. As an example, to manufacture a 500 ppm sample, 5 ml of the stock standard was added to a flask with 95 ml of solvent. The 500 ppm oil in solvent is equivalent to a 50 ppm oil in water sample.
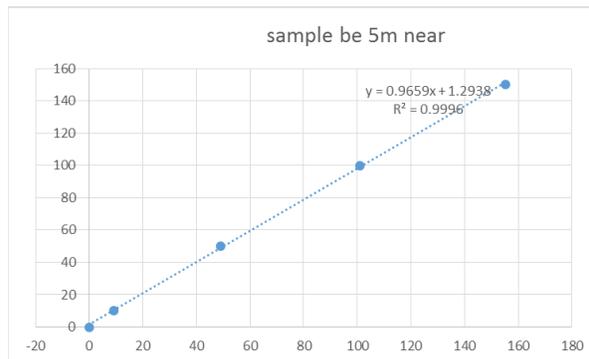
Ultimately, four samples of the target oil in hexane, S316 and OIW surfactant and deionized water were manufactured. The 50 ppm oil in water equivalent standards were used to calibrate the TD-560 for both channels. Each calibration was stored in the TD-560 and labeled according to the measurement channel and solvent.

With the calibration completed, all four manufactured samples and a blank sample were analyzed by the TD-560 for oil concentration. Because the TD-560 records each sample analyzed and allows for the operator to record notes, the data was transferred by USB cable to a laptop for graphing and protecting the data. The full calibrations and data are listed in *Appendices B and C* of this report.
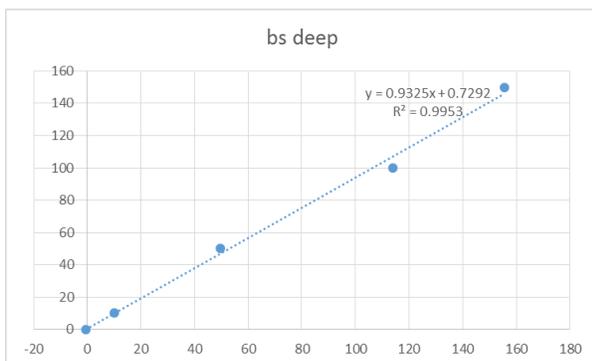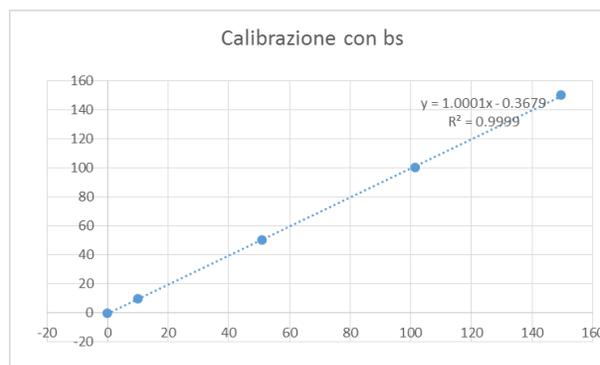
## Sample Analysis
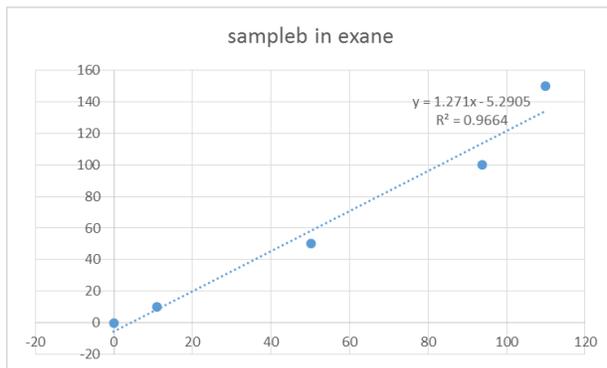

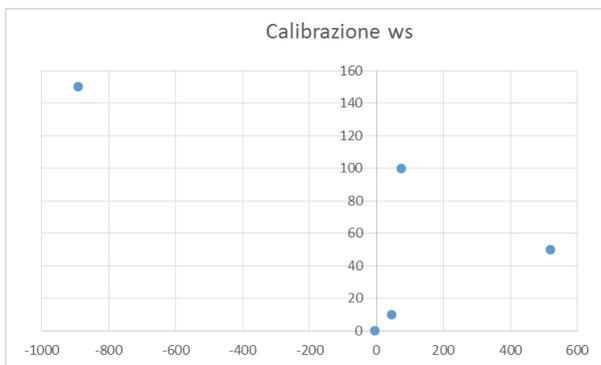
**Graph 1: Sample B in Hexane with Deep UV Channel**

Chart title: sampleb in exane

$y = 1.271x - 5.2905$
$R^2 = 0.9664$



**Graph 2: Sample B in Hexane with Near UV Channel**

Chart title: sample be 5m near

$y = 0.9659x + 1.2938$
$R^2 = 0.9996$



**Graph 3: Sample B in S316 with Deep UV Channel**

Chart title: bs deep

$y = 0.9325x + 0.7292$
$R^2 = 0.9953$



**Graph 4: Sample B in S316 with Near UV Channel**

Chart title: Calibrazione con bs

$y = 1.0001x - 0.3679$
$R^2 = 0.9999$



**Graph 5: Sample B in OIW Surfactant with Deep UV Channel**

Chart title: Calibrazione ws
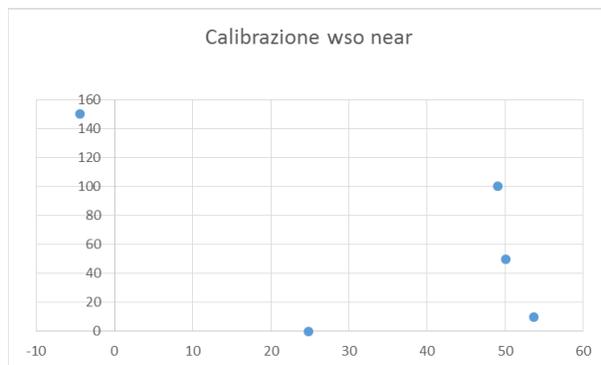


**Graph 6: Sample B in OIW Surfactant with Near UV Channel**

Chart title: Calibrazione wso near

The data in Graphs 1,2,3,4 show that the UV Fluorescence with both a deep UV light source and a near UV light source can produce good linear results for the Gasoline B oil sample from 0 to 150 ppm. The Near UV channel of the TD-560 had better linearity for both the hexane and S316 solvents, while the Deep UV channel showed the beginning of a non-linear response above 100 ppm.

The OIW surfactant used in the No Solvent Method has a high back ground compared to the response of Gasoline B oil sample. For this reason, the calibration data result was not useable for this oil.

At this time, the results of the GC FID analysis of the hexane and IR analysis of the S316 have not been received. Once they are, the data (Graph 5,6) will be added to this graph for linearity, sample manufacturing quality and sensitivity comparison. It is expected that the GC FID and IR analysis will show linear results to the known oil concentrations. However, any errors in sample manufacturing will appear as non-linear results.

The following day, a stock standard and manufactured oil in solvent samples were made using the Gasoline C oil and the same procedures as the Gasoline B samples. Because the Deep UV channel of the TD-560 produced less linear results for Gasoline B, it was decided to only calibrate and test the Gasoline C on the Near UV channel.
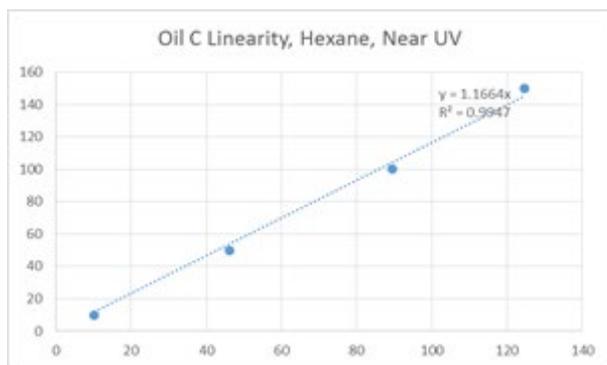


Graph 7
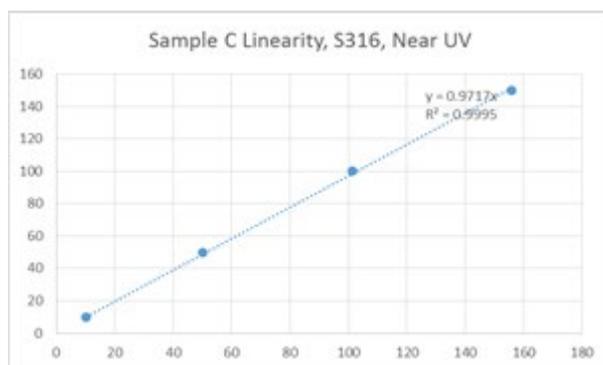


Graph 8
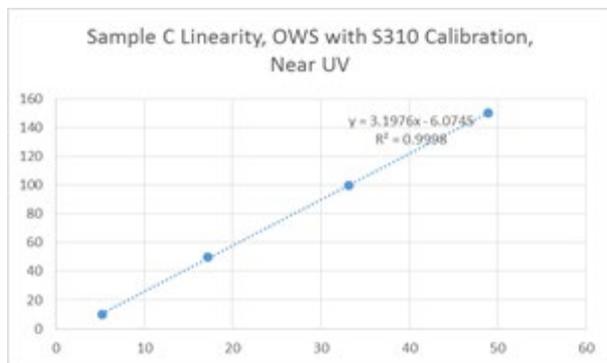


Graph 9

The data also shows a good linear result for the hexane and S316 solvents (Graph 7, 8). The OWS (No Solvent Method) samples (Graph 9) were accidently tested with the S316 calibration. Interestingly, the Gasoline C data shows a linear response from 10 – 150 ppm.

## Produced Water Sample Manufacturing

With completion of the manufactured samples in solvent, actual produced water samples collected from an oil platform were used for manufacturing water samples. For this phase, a 900 ml water sample (B1) was collected. This sample was split into eight different 100 ml water samples, with the water shaken vigorously between each split. Because the oil and water samples will separate quickly, it is extremely difficult to split a water sample into samples of identical oil concentration.

It is expected that the original produced water sample had some oil present. Four of the split samples had known oil volumes added to add 10, 50, 100 and 150 ppm to the original concentration. One sample

had no oil added. For instance, 5 micro-liters of the target Gasoline B oil was added to 100 ml of produced water for a 50 ppm concentration addition.

The first set of water samples was tested by adding 10 ml of hexane to the water bottles. Each bottle was shaken vigorously for 2 minutes. The hexane was carefully collected from the bottle for analysis using the Near UV Channel. The data shows that the measurement is linear, with good sensitivity to the oil in the produced water. As expected, due to the challenges manufacturing and preparing the samples, this data is not as linear as the samples manufactured directly in solvent. The GC-FID data should be linear with the TD-560 results once the results are returned.

Produced Water Sample B1 with B Oil Added, Hexane Extraction, Near UV

$y = 0.8166x + 0.1786$
$R^2 = 0.9802$
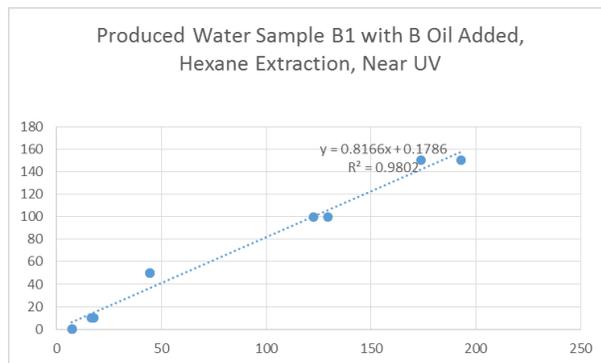
Graph 10

There are a few possible causes of the difference between the samples manufactured directly in solvent and those produced water. First, because the oil and water samples cannot be separated with identical initial oil concentrations, it is not known how much oil is in each sample bottle before the known oil volume is added. Second, because the volumes of the oil added is very small (1, 5, 10 and 15 microliters), the volumetric measurement may not be accurate. Additionally, the transfer pipette used for collecting the extracted samples was plastic. To make sure the plastic does not interfere with the analysis, the sample collection was repeated with a glass pipette. The data shows that the results are the similar for both types of transfer pipettes. (Note: The standard pipettes supplied with the TD-560 are made of a plastic that does not interfere with analysis.)

Using the same process, 3 water samples were manufactured from the same produced water samples for analysis by S316 extraction. To reduce the possible errors caused by small volume measurement, only samples of 0, 10 and 15 microliter addition were analyzed. This data shows a similar high quality linear response over the expected range of oil in water concentrations.

The following day the remaining water from this produced water sample was analyzed by extracting the sample with hexane, then measuring the remaining water volume. After correcting for water volume, the concentration measured was 6.4 ppm.

Produced Water Sample B2 with oil added, Hexane Extraction, Near UV

$y = 0.9835x - 13.795$
$R^2 = 0.9825$
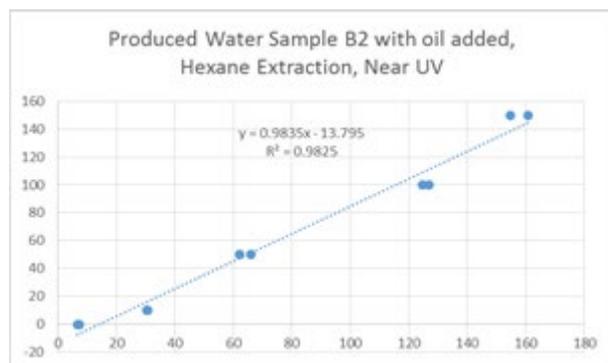
Graph 11

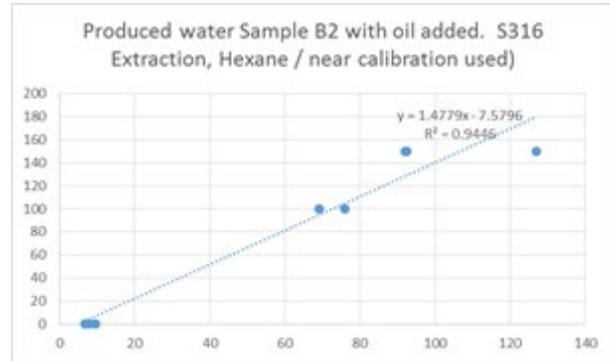Next, a second produced water sample (B2) was similarly split into multiple bottles and known oil volumes added. The samples were extracted using Hexane.

The data again shows good linearity and response over the range of oil concentrations. The samples were collected by plastic and glass pipettes to make sure the plastic had no effect on the response, and the smaller concentrations were not used for both sets of analysis.

Lastly, the B2 produced water sample was separated into sample bottles with known oil volumes added and extracted with the S316 solvent. Accidently the TD-560 analyzed the samples using the Hexane calibration. The values shown are not correct, the slope of the line is significantly different than expected. However the response is again linear over the range of measurement as expected. Had the correct calibration been used, it is expected that this data would be just as accurate as the previous data set.



Produced water Sample B2 with oil added. S316 Extraction, Hexane / near calibration used)

$y = 1.4779x - 7.5796$
$R^2 = 0.9446$

Graph 12

Reviewing the entire set of data from the samples manufactured by adding oil to the produced water illustrates a few key points. First, there is some variation in the quality of results between the samples manufactured directly in the solvent and those directly from the produced water. This variation was caused by the challenges in manufacturing the sample not the analysis of oil in the water sample. When the GC-FID and infrared analysis results are available, the TD-560 results should be validated.

Second, when comparing the results of the produced water samples split into different bottles with no oil added, two of the challenges are illustrated. The two samples from produced water B1 with no oil added and extracted with hexane had 7.25 and 6.4 ppm measured. This variability was due to splitting the water sample. Even though the produced water bottle was shaken vigorously between the samples being split there was enough separation that oil concentrations were different. Also, when comparing the results of the B1 water samples with no oil added and extracted with S316 and those extracted with Hexane, the results are also different. The hexane extracted samples averaged 6.8 ppm and the S316 average was 22.4 ppm. This variation was due to either different extraction efficiencies between the two solvents or concentration variation due to sample splitting. When comparing the similar no oil added samples from produced water B2 sample, the measured results are more similar, 7.9 and 8.7 ppm. Because the 22.4 ppm result is significantly larger than any of the other hexane and S316 samples, it is probable that the sample was not split with a similar oil concentration as the other samples. This variation in the concentration after splitting the sample illustrates how the process oil is a free oil which will separate in the bottle and why sample preparation with solvent extraction is needed.

## Produced Water Sample Analysis

The oil company wanted to understand how an operator would prepare a water sample for analysis in the field. With the TD-560 already calibrated to the target oil in hexane, 2x 900 ml produced water samples (C1 and C2) were prepared by extraction with 90 ml of hexane. The results of these samples were 4.1 and 4.2 ppm.

## Correlation

With just two water samples (C1 & C2), when the results from the GC FID analysis is returned, the measured values of the TD-560 can be correlated to match the GC-FID. The original procedure called for four water samples to be collected and their results averaged to produce a correlation. With four samples a technician is able to perform a correlation even if one of the samples is damaged, lost or for some reason has a significantly different result than the others. While this study only had two samples,

the results are similar and the correlation between the GC-FID and TD-560 will be valid. An example of instructions for sample collection is listed in *Appendix D*.

## Conclusions

The TD-560 produced quality, linear results using both the DUV and NUV channels and both hexane and S316 solvents. The NUV channel had better results and is able to measure at higher concentrations than the DUV. The NUV was determined to be appropriate for the target oils in this study.

The E09 TD-4100XD online oil in water monitor uses a similar optical configuration and sensor as the TD-560. The results determined by the TD-560 demonstration illustrates that the E09 TD-4100XD will have similar quality.

The OIW surfactant used in the No Solvent Method can have a high background response compared to the response of light gas condensates. The calibration data for the OIW surfactant was not good for Gasoline B, but looked better for Gasoline C, and the method was not used for this study. If the No Solvent Method is desired for use in fields that produce heavier crude oil (API 45 and heavier), the OIW surfactant could be used. The company should replicate this study with a crude oil and the OIW surfactant to verify the use.

The light gas condensate used in this study easily separates from the produced water. Using a single water sample subsequently split into multiple samples can cause some variation in the sample concentration. When comparing two different analytical methods for reliability and accuracy, care must be taken that the oil concentration is known at the start of the comparison.

## Appendix A: TD-560 Demonstration Procedures

**Supplies**

- 20 – 40 ml dry process oil / natural gas condensate

- 4 liters reagent grade hexane

- 5x 100ml volumetric flask

- 100 ml graduated cylinder

- 10 ml graduated cylinder

- 10-100 microliter digital micro pipette

- 5x 180 ml graduated sample bottles

- TD-560, with 10 mm square quartz cuvette

- Disposable transfer pipettes

**Preparation**

1. Prepare a 10,000 ppm oil in hexane solution but adding 1 ml target oil to 100 ml graduate flask, and fill to 100 ml mark. (10,000 ppm oil in hexane is equivalent to 1,000 ppm oil in process water)

2. Prepare 4 samples at different oil in hexane concentrations that will span normal process and upset conditions, such as 50, 100, 150 and 200 ppm oil in water.

     - 500 ppm standard - Add 5 ml stock standard to a 100 ml graduated flask, and fill to line with Hexane. 500 ppm oil in hexane is equivalent to 50 ppm oil in water.
     - 1000 ppm standard – Add 10 ml stock standard to a 100 ml graduated flask, and fill to line with Hexane.
     - 1500 ppm standard – Add 15 ml stock standard to a 100 ml graduated flask, and fill to line with Hexane.
     - 2000 ppm standard – add 20 ml stock standard to a 100 ml graduated flask, and fill to line with Hexane.

3. Calibrate TD-560 with 1000 ppm oil in hexane solution. Following calibration procedures, set the calibration standard to 100 ppm (Oil in Water), and blank hexane.

4. Measure 500, 1000, 1500 and 2000 ppm standards and record values.

**Process Water Testing**

Collect 4 liters process water in a single container.

1. Vigorously shake process water container, and fill five sample bottles to 100 ml mark.

2. Add 5, 10, 15 and 20 microliters oil to separate sample bottles. Leave one bottle with no oil added. 5 microliters in 100 ml is 50 ppm.

3. Add 10 ml hexane to each of the five bottles, shake vigorously for 2 minutes. A strong acid can be added to the water sample before hexane if Water Soluble Organics or Polar Organic Acids are to be included in measurement.

4. Once the sample bottle has settled, collect extracted solvent from the bottle and measure with TD-560.

**Correlation Calibration**

1. In quick succession, collect four process water samples in 1 liter bottles following standard procedures for sample analysis.

2. Immediately after collecting 1 liter samples, quickly collect four process water samples in 100 ml bottles

3. Delivery the four 1 liter samples to a laboratory for analysis of oil concentration, following standard procedures.

4. Analyze 100 ml samples with TD-560 using hexane extraction solvent.

   (If any of the measured values from the eight samples return significantly different from the others, a decision can be made if the value can be eliminated from the others. This may occur if something happened to the sample that is not normal, such as improper collection or analysis techniques)

5. Calculate the average value returned from the laboratory and the average value returned from the TD-560.

6. Calculate ratio of Lab average value / TD-560 average value. Multiply this value by 100. Change calibration value of TD-560 to this new number.

**Testing of Water Treatment Chemicals**

If any water treatment chemicals are used in the process, and the operators want to know the effect of the chemical on the response. Samples of the chemical in a water sample can be analyzed at this time.

1. Add sufficient volume of the water treatment chemical to a sample bottle to make a representative concentration of the actual process conditions.

2. Fill the sample bottle to 100 ml mark with tap water.

3. Add 10 ml hexane to the sample bottle, shake for 2 minutes and let the bottle settle.

4. Collect extracted hexane and present to previously calibrated TD-560 for measurement.

### Appendix B: TD-560 Calibration History

| Date | RFUs | Name | Channel | Cal Std | Units | Cuvette (1 = Square) | Extraction (3 = 10:1, 4= NS) | Blank RFU | Standard RFU |
|---|---|---|---|---|---|---|---|---|---|
| 1/28/2019 6:28 | 113.48298 = 0.0 | | | | | | | | |
| 1/28/2019 6:29 | 3946.0579 = 0.0 | | | | | | | | |
| 1/28/2019 6:29 | | sampleb in exane | Deep UV | 500 | PPM | 1 | 3 | 113.4 | 3946 |
| 1/28/2019 6:43 | 9.451136 = 0.0 | | | | | | | | |
| 1/28/2019 6:44 | 148.11577 = 0.0 | | | | | | | | |
| 1/28/2019 6:45 | | sample be 5 ml near | Near UV | 500 | PPM | 1 | 3 | 9.451 | 148.1 |
| 1/28/2019 6:57 | 3.411937 = 0.0 | | | | | | | | |
| 1/28/2019 6:58 | 67.60232 = 0.0 | | | | | | | | |
| 1/28/2019 6:58 | | Calibrazione con bs | Near UV | 500 | PPM | 1 | 3 | 3.411 | 67.6 |
| 1/28/2019 7:17 | 65.458565 = 0.0 | | | | | | | | |
| 1/28/2019 7:18 | 482.37326 = 0.0 | | | | | | | | |
| 1/28/2019 7:19 | | bs deep | Deep UV | 500 | PPM | 1 | 3 | 65.45 | 482.3 |
| 1/28/2019 7:37 | 233.9651 = 0.0 | | | | | | | | |
| 1/28/2019 7:38 | 217.26912 = 0.0 | | | | | | | | |
| 1/28/2019 7:38 | | calibrazione ws | Deep UV | 50 | PPM | 1 | 4 | 233.9 | 217.2 |
| 1/28/2019 7:48 | 88.718094 = 0.0 | | | | | | | | |
| 1/28/2019 7:49 | 14.275693 = 0.0 | | | | | | | | |
| 1/28/2019 7:49 | | calibrazione wso nea | Near UV | 50 | PPM | 1 | 4 | 88.71 | 14.27 |

## Appendix C: Test Data

TD-560 Reading History

| Dates | RFU | Units | Calibration | RFU | Concen. | Units | Actual | Notes |
|-------|-----|-------|-------------|-----|---------|-------|--------|-------|
| 1/28/2019 6:32 | 7295.023 PPM | PPM | sampleb in exane | 7295 | 93.69 | PPM | 100 | |
| 1/28/2019 6:35 | 8551.587 PPM | PPM | sampleb in exane | 8551 | 110 | PPM | 150 | |
| 1/28/2019 6:38 | 955.7577 PPM | PPM | sampleb in exane | 955.7 | 10.98 | PPM | 10 | |
| 1/28/2019 6:45 | 289.5219 PPM | PPM | sample be 5 ml near | 289.5 | 100.9 | PPM | 100 | |
| 1/28/2019 6:47 | 440.0316 PPM | PPM | sample be 5 ml near | 440 | 155.2 | PPM | 150 | |
| 1/28/2019 6:49 | 34.559753 PPM | PPM | sample be 5 ml near | 34.55 | 9.053 | PPM | 10 | |
| 1/28/2019 6:50 | 145.64981 PPM | PPM | sample be 5 ml near | 145.6 | 49.11 | PPM | 50 | |
| 1/28/2019 6:51 | 3958.856 PPM | PPM | sampleb in exane | 3958 | 50.16 | PPM | 50 | |
| 1/28/2019 7:08 | 3.3533454 PPM | PPM | Calibrazione con bs | 3.353 | -0.045 | PPM | | |
| 1/28/2019 7:10 | 68.00223 PPM | PPM | Calibrazione con bs | 68 | 50.31 | PPM | | |
| 1/28/2019 7:12 | 133.67596 PPM | PPM | Calibrazione con bs | 133.6 | 101.4 | PPM | 100 | |
| 1/28/2019 7:13 | 195.35385 PPM | PPM | Calibrazione con bs | 195.3 | 149.5 | PPM | 150 | |
| 1/28/2019 7:14 | 16.370865 PPM | PPM | Calibrazione con bs | 16.37 | 10.09 | PPM | 10 | |
| 1/28/2019 7:15 | 68.661446 PPM | PPM | Calibrazione con bs | 68.66 | 50.82 | PPM | 50 | |
| 1/28/2019 7:19 | 131.37273 PPM | PPM | Calibrazione con bs | 131.3 | 99.67 | PPM | | |
| 1/28/2019 7:19 | 1015.6036 PPM | PPM | bs deep | 1015 | 113.9 | PPM | 100 | |
| 1/28/2019 7:20 | 1364.2528 PPM | PPM | bs deep | 1364 | 155.7 | PPM | 150 | |
| 1/28/2019 7:21 | 148.05417 PPM | PPM | bs deep | 148 | 9.905 | PPM | 10 | |
| 1/28/2019 7:22 | 478.2808 PPM | PPM | bs deep | 478.2 | 49.5 | PPM | 50 | |
| 1/28/2019 7:41 | 209.07259 PPM | PPM | calibrazione ws | 209 | 74.54 | PPM | 100 | |
| 1/28/2019 7:42 | 531.9339 PPM | PPM | calibrazione ws | 531.9 | -892 | PPM | 150 | |
| 1/28/2019 7:44 | 218.96255 PPM | PPM | calibrazione ws | 218.9 | 44.92 | PPM | 10 | |
| 1/28/2019 7:46 | 60.51875 PPM | PPM | calibrazione ws | 60.51 | 519.4 | PPM | 50 | |
| 1/28/2019 7:50 | 95.31773 PPM | PPM | calibrazione wso nea | 95.31 | -4.432 | PPM | 150 | |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 1/28/2019 7:52 | 15.73469 PPM | PPM | calibrazione wso nea | 15.73 | 49.02 | PPM | 100 | |
| 1/28/2019 7:53 | 14.2335 PPM | PPM | calibrazione wso nea | 14.23 | 50.02 | PPM | 50 | |
| 1/28/2019 7:54 | 8.869779 PPM | PPM | calibrazione wso nea | 8.869 | 53.63 | PPM | 10 | |
| 1/28/2019 7:57 | 51.71563 PPM | PPM | calibrazione wso nea | 51.71 | 24.85 | PPM | 0 | |
| 1/28/2019 7:58 | 235.67781 PPM | PPM | calibrazione ws | 235.6 | -5.129 | PPM | 0 | |
| 1/28/2019 8:00 | 61.54768 PPM | PPM | bs deep | 61.54 | -0.469 | PPM | 0 | |
| 1/28/2019 8:00 | 3.3939118 PPM | PPM | Calibrazione con bs | 3.393 | -0.014 | PPM | 0 | |
| 1/28/2019 8:02 | 9.368806 PPM | PPM | sample be 5 ml near | 9.368 | -0.029 | PPM | 0 | |
| 1/28/2019 8:02 | 104.30079 PPM | PPM | sampleb in exane | 104.3 | -0.119 | PPM | 0 | |
| 1/29/2019 1:06 | 58.073597 PPM | PPM | sample be 5 ml near | 58.07 | 17.53 | PPM | 10 | B1 1 |
| 1/29/2019 1:07 | 132.76257 PPM | PPM | sample be 5 ml near | 132.7 | 44.46 | PPM | 50 | B1 2 |
| 1/29/2019 1:09 | 349.13467 PPM | PPM | sample be 5 ml near | 349.1 | 122.4 | PPM | 100 | |
| 1/29/2019 1:10 | 492.1366 PPM | PPM | sample be 5 ml near | 492.1 | 174 | PPM | 150 | B1 3 |
| 1/29/2019 1:11 | 29.559612 PPM | PPM | sample be 5 ml near | 29.55 | 7.25 | PPM | 0 | B1 5 |
| 1/29/2019 1:13 | 55.932247 PPM | PPM | sample be 5 ml near | 55.93 | 16.76 | PPM | 10 | B1 1 bis |
| 1/29/2019 1:14 | 133.31654 PPM | PPM | sample be 5 ml near | 133.3 | 44.66 | PPM | 50 | B1 2 bis |
| 1/29/2019 1:16 | 369.08685 PPM | PPM | sample be 5 ml near | 369 | 129.6 | PPM | 100 | B1 3 bis |
| 1/29/2019 1:17 | 545.0963 PPM | PPM | sample be 5 ml near | 545 | 193.1 | PPM | 150 | B1 4 bis |
| 1/29/2019 1:18 | 29.784876 PPM | PPM | sample be 5 ml near | 29.78 | 7.331 | PPM | 0 | B1 5 bis |
| 1/29/2019 1:46 | 142.85971 PPM | PPM | Calibrazione con bs | 142.8 | 108.6 | PPM | 100 | |
| 1/29/2019 1:47 | 254.1856 PPM | PPM | Calibrazione con bs | 254.1 | 195.3 | PPM | 150 | |
| 1/29/2019 1:48 | 151.49088 PPM | PPM | Calibrazione con bs | 151.4 | 115.3 | PPM | 100 | |
| 1/29/2019 1:50 | 32.75044 PPM | PPM | Calibrazione con bs | 32.75 | 22.85 | PPM | 0 | |
| 1/29/2019 1:51 | 158.4579 PPM | PPM | Calibrazione con bs | 158.4 | 120.7 | PPM | 100 | |
| 1/29/2019 1:53 | 261.16083 PPM | PPM | Calibrazione con bs | 261.1 | 200.7 | PPM | 150 | |
| 1/29/2019 1:54 | 31.61634 PPM | PPM | Calibrazione con bs | 31.61 | 21.96 | PPM | 0 | |
| 1/29/2019 2:32 | 94.27177 PPM | PPM | sample be 5 ml near | 94.27 | 30.58 | PPM | 10 | B2 1 |

| 1/29/2019 2:33 | 192.63914 PPM | PPM | sample be 5 ml near | 192.6 | 66.05 | PPM | 50 | B2 2 |
|---|---|---|---|---|---|---|---|---|
| 1/29/2019 2:35 | 438.71436 PPM | PPM | sample be 5 ml near | 438.7 | 154.7 | PPM | 150 | B2 3 |
| 1/29/2019 2:36 | 4595.548 PPM | PPM | sample be 5 ml near | 4595 | 1653 | PPM | | |
| 1/29/2019 2:36 | 4398.401 PPM | PPM | sample be 5 ml near | 4398 | 1582 | PPM | | |
| 1/29/2019 2:37 | 355.47995 PPM | PPM | sample be 5 ml near | 355.4 | 124.7 | PPM | 100 | B2 4 |
| 1/29/2019 2:39 | 29.397146 PPM | PPM | sample be 5 ml near | 29.39 | 7.192 | PPM | 0 | B2 5 |
| 1/29/2019 2:40 | 361.0279 PPM | PPM | sample be 5 ml near | 361 | 126.7 | PPM | | |
| 1/29/2019 2:42 | 94.90214 PPM | PPM | sample be 5 ml near | 94.9 | 30.81 | PPM | 10 | B2 1 Glass |
| 1/29/2019 2:44 | 181.9101 PPM | PPM | sample be 5 ml near | 181.9 | 62.18 | PPM | 50 | B2 2 Glass |
| 1/29/2019 2:45 | 455.19467 PPM | PPM | sample be 5 ml near | 455.1 | 160.7 | PPM | 150 | B2 3 Glass |
| 1/29/2019 2:46 | 3314.0776 PPM | PPM | sample be 5 ml near | 3314 | 1191 | PPM | | |
| 1/29/2019 2:47 | 361.93246 PPM | PPM | sample be 5 ml near | 361.9 | 127 | PPM | 150 | B2 4 Glass |
| 1/29/2019 2:49 | 28.129059 PPM | PPM | sample be 5 ml near | 28.12 | 6.734 | PPM | 0 | B2 5 Glass |
| 1/29/2019 2:59 | 201.05383 PPM | PPM | sample be 5 ml near | 201 | 69.08 | PPM | 100 | B2 3 s316 Glass |
| 1/29/2019 3:01 | 265.8862 PPM | PPM | sample be 5 ml near | 265.8 | 92.46 | PPM | 150 | B2 4 s316 Glass |
| 1/29/2019 3:02 | 31.518833 PPM | PPM | sample be 5 ml near | 31.51 | 7.957 | PPM | 0 | B2 5 S316 Glass |
| 1/29/2019 3:04 | 219.99068 PPM | PPM | sample be 5 ml near | 219.9 | 75.91 | PPM | 100 | B2 3 S316 BIS |
| 1/29/2019 3:06 | 264.97513 PPM | PPM | sample be 5 ml near | 264.9 | 92.13 | PPM | 150 | |
| 1/29/2019 3:07 | 35.95751 PPM | PPM | sample be 5 ml near | 35.95 | 9.557 | PPM | 0 | B2 5 S316 bis glass |
| 1/29/2019 6:22 | 135.66162 PPM | PPM | sample be 5 ml near | 135.6 | 45.5 | PPM | | |
| 1/29/2019 6:26 | 257.58957 PPM | PPM | sample be 5 ml near | 257.5 | 89.47 | PPM | 100 | CE 10 |
| 1/29/2019 6:30 | 355.06723 PPM | PPM | sample be 5 ml near | 355 | 124.6 | PPM | 150 | CE 15 |
| 1/29/2019 6:31 | 37.95306 PPM | PPM | sample be 5 ml near | 37.95 | 10.27 | PPM | 10 | CE 1 |
| 1/29/2019 6:32 | 137.22325 PPM | PPM | sample be 5 ml near | 137.2 | 46.07 | PPM | 50 | CE 5 |
| 1/29/2019 6:44 | 125.85912 PPM | PPM | ce 0_5 | 125.8 | 48.77 | PPM | 50 | c3 5 ml |
| 1/29/2019 6:46 | 247.97792 PPM | PPM | ce 0_5 | 247.9 | 100.2 | PPM | 100 | ce 10 ml |
| 1/29/2019 6:47 | 376.0969 PPM | PPM | ce 0_5 | 376 | 154.2 | PPM | 150 | CE 15 ml |

| 1/29/2019 6:48 | 35.72674 PPM | PPM | ce 0_5 | 35.72 | 10.77 | PPM | 10 | ce 1 ml |
|---|---|---|---|---|---|---|---|---|
| 1/29/2019 6:50 | 147.553 PPM | PPM | cs 0_5 | 147.5 | 86.74 | PPM | 100 | cs 10 ml |
| 1/29/2019 6:51 | 226.83765 PPM | PPM | cs 0_5 | 226.8 | 134.9 | PPM | 150 | cs 15 ml |
| 1/29/2019 6:52 | 19.061249 PPM | PPM | cs 0_5 | 19.06 | 8.697 | PPM | 10 | cs 1 ml |
| 1/29/2019 6:53 | 75.2338 PPM | PPM | cs 0_5 | 75.23 | 42.81 | PPM | 50 | cs 5 ml |
| 1/29/2019 6:57 | 74.12982 PPM | PPM | cs 0_5 | 74.12 | 50.06 | PPM | 50 | cs 5 ml |
| 1/29/2019 6:58 | 145.4008 PPM | PPM | cs 0_5 | 145.4 | 101.4 | PPM | 100 | cs 10 ml |
| 1/29/2019 6:59 | 220.6941 PPM | PPM | cs 0_5 | 220.6 | 155.8 | PPM | 150 | cs 15 ml |
| 1/29/2019 7:00 | 18.826838 PPM | PPM | cs 0_5 | 18.82 | 10.16 | PPM | 10 | cs 1 ml |
| 1/29/2019 7:06 | 50.6527 PPM | PPM | cs 0_5 | 50.65 | 33.12 | PPM | 100 | cws 10 ml |
| 1/29/2019 7:07 | 72.56613 PPM | PPM | cs 0_5 | 72.56 | 48.93 | PPM | 150 | cws 15 ml |
| 1/29/2019 7:08 | 12.054742 PPM | PPM | cs 0_5 | 12.05 | 5.276 | PPM | 10 | cws 1 ml |
| 1/29/2019 7:09 | 28.60974 PPM | PPM | cs 0_5 | 28.6 | 17.22 | PPM | 50 | cws 5 ml |
| 1/30/2019 1:16 | 24.289248 PPM | PPM | sample be 5 ml near | 24.28 | 5.35 | PPM | | |
| 1/30/2019 1:24 | 9.288248 PPM | PPM | ce 0_5 | 9.288 | -0.374 | PPM | | |
| 1/30/2019 1:27 | 11.548879 PPM | PPM | ce 0_5 | 11.54 | 0.578 | PPM | | |
| 1/30/2019 1:46 | 19.290428 PPM | PPM | ce 0_5 | 19.29 | 3.842 | PPM | | |
| 1/30/2019 1:54 | 10.23552 PPM | PPM | ce 0_5 | 10.23 | 0.025 | PPM | | |
| 1/30/2019 1:57 | 20.476234 PPM | PPM | ce 0_5 | 20.47 | 4.342 | PPM | | |
| 1/30/2019 1:59 | 20.083046 PPM | PPM | ce 0_5 | 20.08 | 4.176 | PPM | | |
| 1/30/2019 2:02 | 20.178434 PPM | PPM | ce 0_5 | 20.17 | 4.217 | PPM | | |

# Environmental Enterprises USA, Inc.

## Produced Water Sample Collection Instructions
### for
### IR / UV Meter Calibration *ONLY!*

In addition to the IR or UV meter, field personnel must collect and ship the following samples for Meter Calibration Service:

- 4, 1-liter Produced Water (PW) samples
- 4, 140-mL PW sample in 160-mL medicine bottles – FILL EACH WITH APPROXIAMTELY 140 mL Produced Water. "140-mL" GRADUATION IS MARKED ON EACH BOTTLE.

These 8 samples must be collected from the same collection point at the same time (one after the other).

- 1, 140-mL sample of crude oil in a 160-mL medicine bottle.

Environmental Enterprises (EE USA) has supplied a preprinted Chain-of-Custody (COC) and labeled containers

### *The 4, 1-liter jars contain acid preservative – non-protocol samples.*

1) Collect the PW samples (8) and crude oil sample (1) filling each container: DO **NOT OVERFILL** or **OVERRUN** the sample containers.
2) **Complete the COC and container labels** – remember to date, sign, and indicate when and where the samples were collected. **Each person that takes custody of the samples must complete his part on the COC (collected by & relinquished by or received by & relinquished by).**
3) **Tightly** cap the sample containers and store at ambient temperature until packaged for transport.

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