TD-4100XD Calibration for Produced Water Applications

Correlation Calibration

Introduction

The TD-4100XD uses a linear calibration equation to convert its Raw Fluorescence readings into oil concentration. For produced water applications, the calibration equation must be determined by correlating the instrument's Raw Fluorescence response to the oil concentration measured by a TDHI-approved laboratory analysis method. Also, it is absolutely essential to base the correlation on the Raw Fluorescence and oil concentration of "live" process samples. "Live" samples are those flowing through the instrument directly from the water system process piping. Lab-prepared samples, made by adding crude oil to produced water, are spectroscopically quite different from live process fluids, and therefore cannot be used for calibrating the TD-4100XD in produced water service.

Overview

To perform a correlation calibration, you must record the Raw Fluorescence response of the TD-4100XD while collecting a series of water samples from the sample port. The oil concentrations of the samples are measured by the lab. Several samples are required, typically 10-20, with oil concentrations covering at least 50% of your desired monitoring range.

There are three ways to obtain samples with a range of oil concentration. First, you can simply wait for the system to yield different oil concentrations during the normal course of operations. Second, you can temporarily adjust process parameters to create samples with a variety of oil concentrations. Typical parameters include such things as treating chemical concentration, flotation cell skim levels, number of active agitators, etc. Third, you may also be able to blend variable percentages of upstream fluids (with high oil concentration) with the effluent of your final oil/water separator. This approach works best when the oil concentration of the upstream fluids is no more than 2-3 times the oil concentration of the final effluent.

After Raw Fluorescence and oil concentration values are available for several samples, a linear regression is performed to produce an equation relating oil concentration to Raw Fluorescence. A typical set of data is shown in graphical form on the next page.

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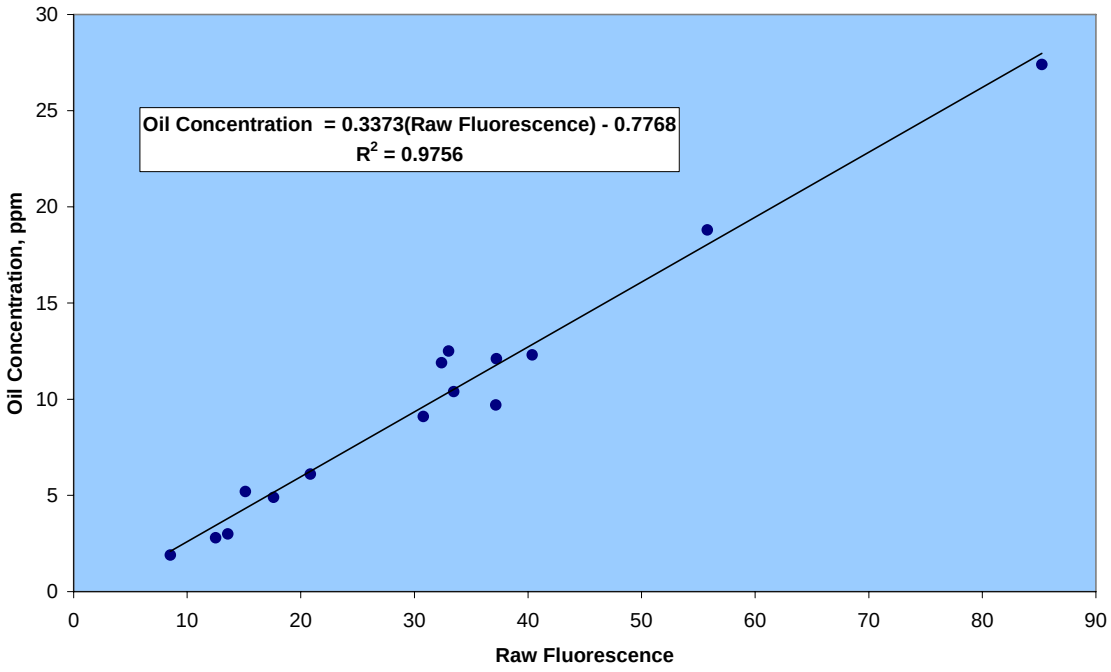

The regression also produces a correlation coefficient, R^2, which is used to determine the quality of the correlation. RP^2 ranges from 0 to 1. An R^2 value of 0 indicates no correlation between the two measured values. An R^2 value of 1 indicates perfect correlation. You should be able to obtain an R^2 value of 0.95 or greater. TDHI recommends a minimum R^2 value of 0.80. If your measurements do not achieve this, collect additional samples over a broader range of oil concentration. You may perform the correlation calculations with most spreadsheet programs. A custom Excel workbook (TD-4100XD Correlation Calibration.xls) is also available from TDHI.

Once an acceptable correlation is obtained, the correlation equation is used to compute calibration values. Two data pairs are required, which must then be entered into the instrument's memory. The first data pair consists of a relatively high oil concentration and its corresponding Raw Fluorescence. The second data pair consists of a lower oil concentration and its corresponding Raw Fluorescence reading. It is important to emphasize that the values of Raw Fluorescence and oil concentration for the two data pairs are computed from the final correlation equation. They are <u>not</u> the values associated with individual high and low concentration samples. The TD-4100XD Correlation Calibration workbook performs all of the appropriate calculations.

The oil concentration values are entered into the instrument's memory using the keypad. Raw Fluorescence cannot be entered with the keypad. Rather, the Raw Fluorescence values must be actually read by the instrument. The Raw Fluorescence readings required for calibration are read by the instrument while you are collecting "High" and "Low" Standard samples as described below.

Procedure

A. Initial Setup and Sensitivity Adjustment

1. Make sure that the instrument is properly installed and operating within specifications. (Make sure that the optical windows are clean and dry and that the process water is flowing through the instrument at the recommended rate. Make sure the instrument has been powered up for at least 24 hours.)

2. Set the instrument to display Raw Fluorescence. This is done by pressing the <HOME> key, then the <*> key, then the <ENT> key.

   Note: The instrument will display Raw Fluorescence for 15 minutes, and then will automatically go back to the HOME screen. If this happens, press <HOME>, <*>, <ENT> to return to the Raw Fluorescence screen.

3. Flow "live" produced water through the instrument. "Live" produced water is the water coming directly from the water system process piping. Continue with this procedure when you are sure that the process is running normally, and water quality is not in an upset condition.

4. Remove the threaded plug at the base of the lamp housing to access the sensitivity adjustment screw. Adjust the sensitivity screw to obtain a Raw Fluorescence reading of approximately 28. Once set, replace the threaded plug, and do not change the position of the sensitivity screw again. Also, do not make sensitivity adjustments with the up and down arrow keys. It is important to keep the instrument at this sensitivity setting for the rest of this calibration procedure, and during monitoring when calibration is complete.

5. Allow the instrument to stabilize for at least 60 minutes before proceeding.

6. Record the Raw Fluorescence reading in the space provided on the “Data” worksheet of the TD-4100XD Correlation Calibration workbook. Enter the reading in the Raw Fluorescence column of the “Setup” row (red background).

B. Perform a Calibration Reset

1. Press <HOME> then <ENT> then <1>.

2. If the instrument asks for an ID, enter your User ID using the number keys. See page 3-1 of the Operating Manual. Then press <ENT>, <3> and skip to step 4.

3. If the unit does not ask for an ID, press <ENT> then press <3>.

4. Press <*> 5 times to reset the calibration to the factory default settings.

5. Press <HOME> to return to the HOME screen.

C. High Standard Measurements

1. Set the instrument to display Raw Fluorescence: press <HOME> then <*> then <ENT>.

   Note: The instrument will display Raw Fluorescence for 15 minutes, and then will automatically go back to the HOME screen. If this happens, press <HOME>, <*>, <ENT> to return to the Raw Fluorescence screen.

2. Wait for the process to yield a fairly stable Raw Fluorescence response that is 10-20% higher than the initial reading obtained after performing the sensitivity adjustment. If necessary, you might consider a slight manipulation of the water treatment process parameters to obtain this response.

3. Press <HOME>, <ENT>, <1>, enter your User ID, if necessary, <1> and then <ENT>. The High Standard Raw Fluorescence will be displayed.

4. When the reading is fairly stable, press <*> and immediately collect a water sample from the sample port on the bubble trap. The instrument will beep and "WAIT" will appear in the lower right-hand corner of the screen while the High Standard Raw Fluorescence is stored in memory. Press <ENT> and then <HOME>.

5. Label the water sample "High STD" and submit the sample to the lab for the oil concentration measurement.

D. Low Standard Measurements

1. Set the instrument to display Raw Fluorescence: press <HOME> then <*> then <ENT>.

   Note: The instrument will display Raw Fluorescence for 15 minutes, and then will automatically go back to the HOME screen. If this happens, press <HOME>, <*>, <ENT> to return to the Raw Fluorescence screen.

2. Wait for the process to yield a fairly stable Raw Fluorescence response that is 10-20% lower than the High Standard value. If necessary, you might consider a slight manipulation of the water treatment process parameters to obtain this response.

3. Press <HOME>, <ENT>, <1>, enter your User ID, if necessary, <2> and then <ENT>. The Low Standard Raw Fluorescence will be displayed.

4. When the reading is fairly stable, press <*> and immediately collect a water sample from the sample port on the bubble trap. The instrument will beep and "WAIT" will appear in the lower right-hand corner of the screen while the Low Standard Raw Fluorescence is stored in memory. Press <ENT> and then <HOME>.

5. Label the water sample "Low STD" and submit the sample to the lab for the oil concentration measurement.

E. Retrieve the High and Low Raw Fluorescence Measurements from Memory

1. Press <HOME>, <ENT>, and then <2> to display the Raw Fluorescence values for the High and Low Standards. Record the values.

2. Press <HOME>.

3. Note that the readings on the HOME screen are not yet displayed as oil concentration values.

F. Enter High and Low Standard Values into the Worksheet

1. Enter the High Standard Raw Fluorescence value on the "High STD" row (yellow background) of the worksheet.

2. Enter the Low Standard Raw Fluorescence value on the "Low STD" row (green background) of the worksheet.

3. Enter the oil concentration values (measured by the lab) for the High and Low Standards.

Note: As soon as you enter the oil concentration values, the worksheet will calculate an initial correlation equation and display an initial set of calibration values. At this point, you could enter the values into the TD-4100XD as described in Section H below. However, because the correlation is now based on data from only two samples, the calibration equation may give poor accuracy at higher and lower oil concentrations. To achieve suitable accuracy over your desired concentration range, you must build a better correlation equation with data from additional samples.

G. Additional Sample Measurements

1. Set the instrument to display Raw Fluorescence: press <HOME> then <*> then <ENT>.

   Note: The instrument will display Raw Fluorescence for 15 minutes, and then will automatically go back to the HOME screen. If this happens, press <HOME>, <*>, <ENT> to return to the Raw Fluorescence screen.

2. Record the Raw Fluorescence reading obtained while collecting a water sample from the sample port on the bubble trap. Enter the value in the Raw Fluorescence column of the next blank row of the worksheet.

3. Label the sample with the Sample number of the row and submit the sample to the lab for the oil concentration measurement. When the oil concentration is available, enter the value in the Oil Concentration column of the same row.

4. The worksheet will recalculate the correlation results as soon as you enter an additional set of Raw Fluorescence and Oil Concentration values.

5. Repeat steps 2-4 until you have an R^2 value of 0.8 or greater (preferably 0.95 or greater), based on samples with oil concentrations that span at least 50% of your desired range. TDHI recommends at least 3 low-concentration samples, 3 mid-range samples, and 3 high-concentration samples.

H. Enter Calculated TD-4100XD Concentration Values for High and Low Standards

1. Press <HOME> and then <0> to display the concentration input menu.
2. Press <1> to display the “Hi Std Conc” sub-menu.
3. Press <ENT> to display the data input screen. You may also need to enter your User ID.
4. Press the number keys to enter the Hi Std Conc value calculated by the worksheet (cell J23).
5. Press <ENT> to store the value in memory.
6. Press <ESC>, <ESC> to return to the concentration input menu.
7. Press <2> to display the “Low Std Conc” sub-menu.
8. Press <ENT> to display the data input screen.
9. Press the number keys to enter the Low Std Conc value calculated by the worksheet (cell J27).
10. Press <ENT> to store the value in memory.
11. Press <HOME>.
12. Note that oil concentration values are now displayed on the HOME screen.

I. TD-4100XD Analog Output (4-20 mA) Scaling

1. Press <HOME> and then <8> to display the 20 mA (full scale) oil concentration.
2. Press <ENT> to display the data input screen.
3. Press the number keys to enter the desired 20 mA oil concentration.
4. Press <ENT> to store the value in memory.
5. Press <HOME> and then <7> to display the 4 mA (baseline) oil concentration.
6. Press <ENT> to display the data input screen.
7. Press the number keys to enter the desired 4 mA oil concentration.
8. Press <ENT> to store the value in memory.
9. Press <HOME> to resume normal monitoring.

TD-4100XD Correlation Calibration

Sample	Date	Time	Raw Fluorescence	Oil Concentration	Notes
Setup	12/28/04	13:00	29.52	NA	Initial Sensitiviy Setup
High STD	12/28/04	14:09	33.47	10.4	High Standard
Low STD	12/28/04	14:11	20.85	6.1	Low Standard
3	12/28/04	14:15	30.79	9.1	set floculant to 1.5 ppm
4	12/28/04	14:30	37.16	9.7	set floculant to 1.5 ppm
5	12/29/04	8:30	12.51	2.8	set floculant to 3.0 ppm
6	12/29/04	8:45	8.52	1.9	set floculant to 3.0 ppm
7	12/29/04	9:05	13.58	3	set floculant to 3.0 ppm
8	12/29/04	10:20	15.12	5.2	set floculant to 1.5 ppm
9	12/29/04	11:25	17.6	4.9	set floculant to 1.5 ppm
10	12/29/04	13:15	37.22	12.1	shut off floculant pump
11	12/29/04	13:45	85.24	27.4	shut off floculant pump
12	12/29/04	14:00	55.79	18.8	shut off floculant pump
13	12/29/04	14:30	40.37	12.3	shut off floculant pump
14	12/29/04	16:45	32.39	11.9	Normal ops, floculant: 0.5 ppm
15	12/29/04	17:00	33.02	12.5	Normal ops, floculant: 0.5 ppm
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Monitoring Range	
Min. Conc., ppm	0
Max. Conc., ppm	50
Desired Range, ppm	50
Sample Range, ppm	25.5
Range Coverage, %	51

Correlation Results	
ppm = m(Raw Fluorescence) + b	
# Samples	15
m	0.3372918
b	-0.7767679
R^2	0.9755924

TD-4100XD Calibration Values

Calculated High STD Concentration	
Raw Fluorescence	Hi Std Conc
33.47	10.512

Calculated Low STD Concentration	
Raw Fluorescence	Low Std Conc
20.85	6.256

For assistance, contact:

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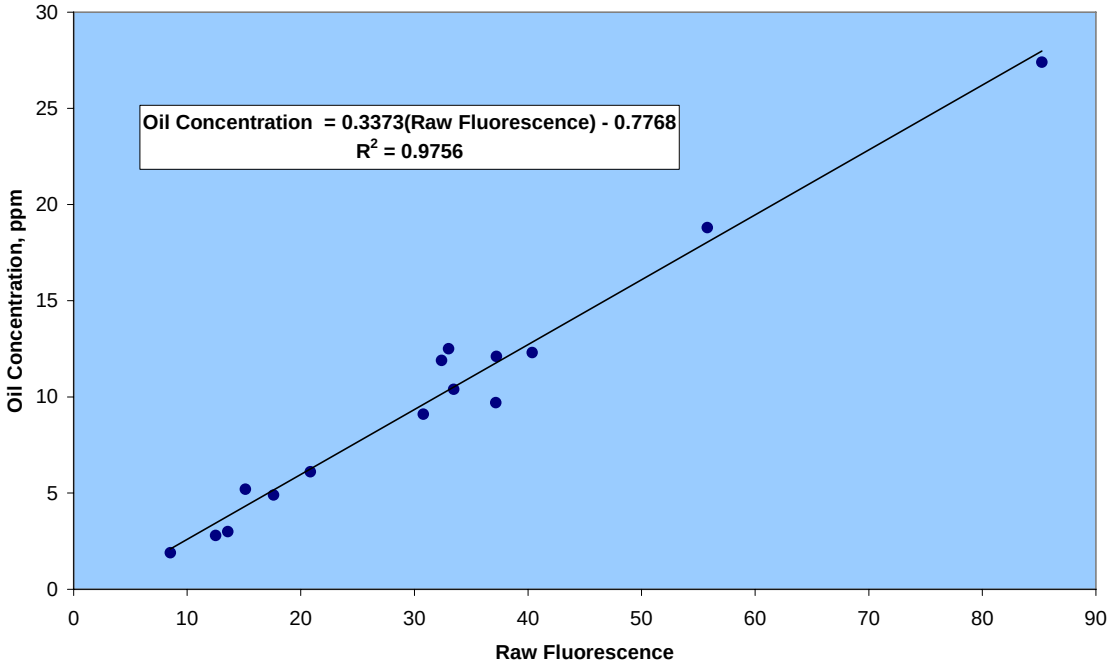
Correlation Scatter Plot



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