Oil Measurement in SAGD Steam Generation

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Abstract

The steam injection enhanced oil recovery method used primarily in Alberta, Canada, also known as Steam Assisted Gravity Drainage (SAGD), pushes energy in the form of steam into the formation to liquefy very heavy oil known as bitumen so it can be recovered and lifted to the surface. This method causes the continuous release of water soluble organic material more commonly known as WSO's into the produced water. The injected steam is recovered and recirculated for process efficiency and water conservation. In traditional industrial steam recycle processes the free oil concentration must be below 1 part per million to prevent boiler and other component fouling. Detection of oil with an on line fluorescence monitor at this concentration can achieve reliable protection for the boilers or boiler feed water pretreatment systems. In SAGD operations, the fluorescence response of the WSO's is similar to bitumen. Because the fluorescence signal of the WSO's is significantly higher than the bitumen, operators must have a reliable measurement method for Free Oil to protect the Once Through Steam Generators (OTSG). A study was conducted to find a viable solvent extraction procedure which would extract and measure the free oil from samples of produced water and which would leave the WSO's behind. Measurement of the bitumen in the extracted solvent provides the needed process information to protect the OTSGs in the SAGD plants. Determining an effective solvent to accomplish this was important because the bitumen is not readily extracted from the produced water in commonly used solvents such as Hexane. Field experience has shown that toluene is a more effective solvent than hexane at extracting the bitumen from the produced water.

Background

SAGD is used extensively in the Athabasca Oil Sands field, along with the Cyclic Steam and surface mining techniques. Because of the high steam demand for these processes, the recycling of the water for reuse is required for energy and water conservation.

In most industrial processes, collection and recycling of steam is common and well understood. The Power Industry is the best example of this, where virtually all steam generated is recovered and reused to save energy costs and water resources. However, in many other industries like Food Processing, Paper Mills, Steel and Aluminum Mills, etc., not all the steam can be recovered because it is consumed by the process or could be contaminated and not suitable as boiler feed water.

ASME has published a document, Consensus on Operating Practices for the Control of Feed Water and Boiler Water[1] which states oil concentration in industrial boiler feed water should be below 1 Part per Million for a low pressure boiler (less than 450 PSI), with lower concentrations required with higher pressure. Organic material in the water can cause water foaming and coking on the boiler tubes.

To avoid potential damage to the boilers or to avoid the cost of advanced water treatment / oil removal systems, industrial companies often decide to dispose of potential oil contaminated water. Other companies rely on advanced water treatment processes and oil detection equipment for steam condensate recovery and reuse.

Steam Assisted Gravity Drainage

Typical SAGD water cycle

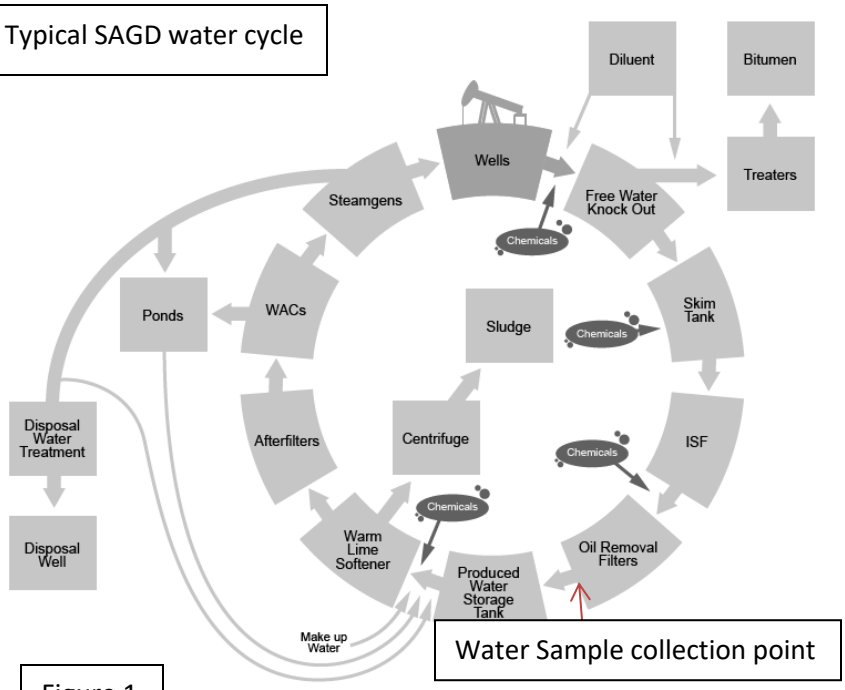


Figure 1

Steam Assisted Gravity Drainage is a commonly used enhanced oil recovery technique used in the Athabasca Oil Sands field. The oil contained in the Oil Sands is heavy oil or bitumen which must be heated to reduce the viscosity so that it can flow and be recovered.

SAGD involves two horizontal parallel drilled wells with high pressure and temperature steam injected into the Oil Sands formation. The oil flows by gravity to the lower well and is pumped with the liquid steam condensate to the surface. Once on the surface, the two liquid phases and any sand / sediment are separated.

The steam temperature can start as high as 296 C and pressure as high as 10,000 kPa[2]. As of 2013, the most efficient operation for SAGD used 2 barrels of non-potable water (brackish) for every barrel of oil produced. There is approximately 900,000 barrels of oil produced by SAGD every day, with at least 1,800,000 barrels of water injected as steam. Approximately 90% of the injected steam is recovered[3].

Because the water is directly in contact with the Crude Oil produced in this oil field, the water recirculated as steam will always have oil contamination. If the operators chose to protect their boilers from this oil contaminated water by using an alternate source of water, the energy and water treatment costs would be higher, probably causing the projects to be economically unfeasible.

Oil in Water Detection in Boilers

Many measurement technologies have been attempted for the detection of or indication of oil in boiler feed water. These include Turbidity, Light Scatter, UV Absorption, pH, Residual Chlorine and Fluorescence. Turbidity and Light Scatter technologies give incorrect oil concentration measurements when interfered with by particles (Solids, Oil Droplets and Gas Bubbles) greater than about 5 microns in water. UV Absorption instruments also give incorrect oil measurement with organic material in the water processes as well as other particles that can absorb ultraviolet light. pH and Residual Chlorine instruments can indicate the presence of oil if the water chemistry is correct and all other factors are constant. Fluorescence measurement can be optimized to exclusively measure oil concentrations in water with little interferences from particles, bubbles or other water chemistry.

Hydrocarbon Fluorescence

Aromatic fractions of dispersed oil and water-soluble organics (WSO) found in industrial water can be stimulated to emit fluorescent light. The process is illustrated in Figure 2 Excitation. Light is directed to a sample at a wavelength, λEX. The aromatic molecules in the sample absorb the excitation light and jump from their normal energy level (E0, ground state) to an excited energy state, E2. The excited molecules then lose some of their absorbed energy by a variety of mechanisms (relaxation) and go to a lower energy state, E1. The molecules then drop back down to E0 by emitting a photon of fluorescent light at a wavelength λEM. The energy emitted by fluorescence (E1 – E0) is lower than the energy gained by absorption (E2 – E0). Since light energy is inversely proportional to wavelength, the wavelength of the fluorescent light, λEM, is always longer than the wavelength of the excitation light, λEX. The intensity of the fluorescence emission, IF, is proportional to the concentration of the fluorescent molecules in the sample.

1. Molecules (M) are excited to high energy state E_2 by the absorption of light.
2. Excited molecules (M) relax to lower energy excited state E_1.
3. Molecules return to ground state E_0 by emitting fluorescence.
4. Intensity of fluorescence emission I_F is proportional to the concentration of fluorescent molecules.

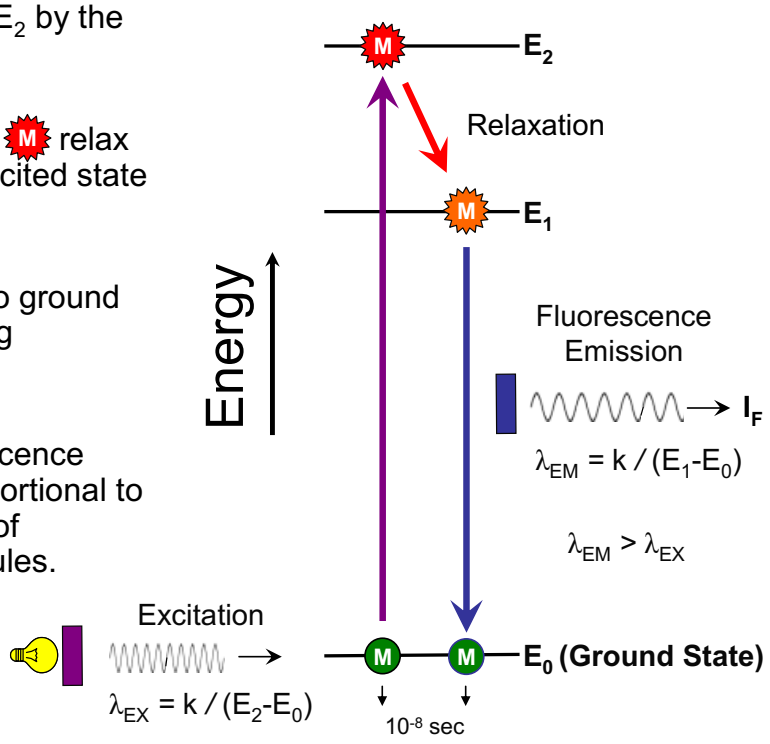


Figure 2

Oil in water monitors using Fluorescence measurement technology are able to detect oils as low as 5 parts per billion in a steam condensate. The technology used in continuous monitoring applications does not use sample preparation such as chemical additions, or homogenization. It has no significant interference from solid particles, gas bubbles or water treatment chemicals. The fluorescence response is immediate. The very low detection limits, immunity to solids and gas bubbles, make this technology reliable and appropriate for protecting boilers from oil contamination in the boiler feed water and steam condensate. Oil in water monitors using Fluorescence measurement technology are able to detect oils as low as 5 parts per billion in a steam condensate. The technology used in continuous monitoring applications does not use sample preparation such as chemical additions, or homogenization. It has no significant interference from solid particles, gas bubbles or water treatment chemicals. The

fluorescence response is immediate. The very low detection limits, immunity to solids and gas bubbles, make this technology reliable and appropriate for protecting boilers from oil contamination in the boiler feed water and steam condensate.

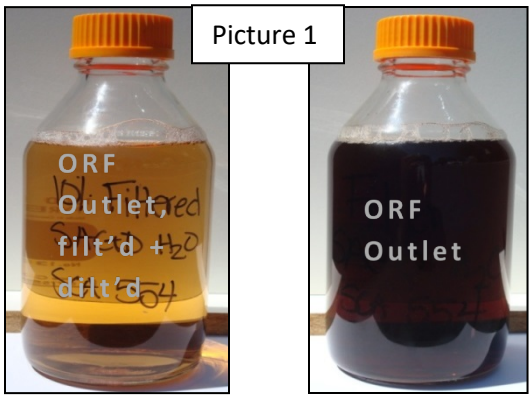
SAGD Produced Water Quality

The produced water returned from the oil production well is separated from the oil in a series of gravity separators (Free Water Knock Outs and Skim Tanks, flotation vessels, etc.). It is then treated by an Oil Removal Filter (ORF), which is often referred to as a Walnut Shell Filter in other locations. At each stage of separation more and more oil is removed from the water. The technologies used in each stage have a specific ability to remove oil droplets that are smaller than the previous stage. The final result after the ORF (See Figure 1) is typically water with a less than 20 parts per million free oil.

While there is no universal definition of Free Oil, it is generally defined as the oil droplets that are large enough to be efficiently removed by Gravity Separation and ORFs. The Oil that cannot be efficiently removed by these separation systems is typically called Dispersed Oil, and Water Soluble Organics (WSOs).

Because the WSOs are not removed by these processes, the concentration is steady and a function of the type of oil production and water chemistry. In traditional oil production the signal from the WSO concentrations is similar to Free Oil between 10 and 20 ppm. In some heavy oil fields of California with a long history of steam flooding, the WSO concentration is almost 0 ppm. However, in the SAGD fields, the WSO signals have been seen as high as 2000 ppm.

Picture 1



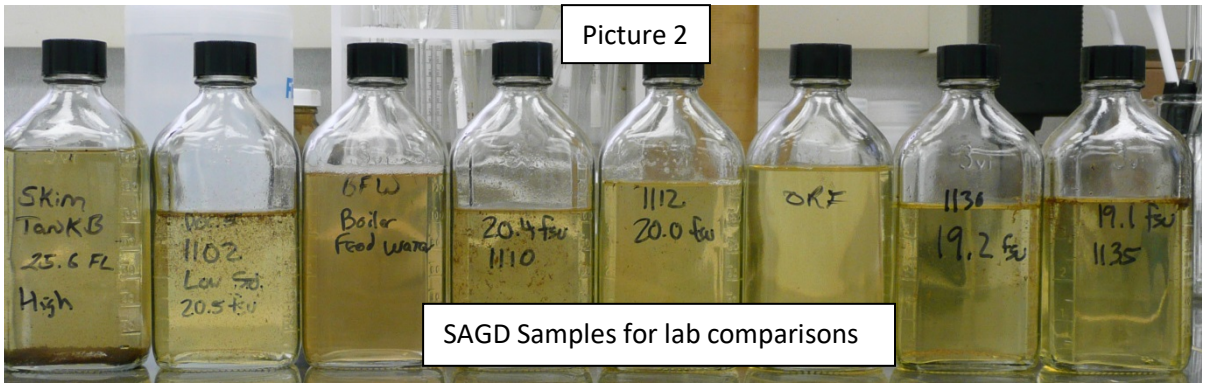
Oily Water Samples from Outlet of ORF

In the experiment, samples were collected to illustrate the possible concentrations of Water Soluble Organics in the SAGD water. In picture 1, the Bottle on the right is water collected directly from the outlet of an ORF. The sample was probably collected during an upset condition. The sample was first filtered with a 0.2 micron filter, and then diluted to 10% with clean water. In some operations any droplets that are below 0.2 microns are going to be Water Soluble Organics. By observing the remaining color seen in the photo on the left the water sample had a high concentration of Water Soluble Organics or other light absorbing compounds.

Picture 2 shows a number of water samples that were collected from a SAGD water process showing 'normal' water quality after the ORF and the Boiler Feed Water (as shown in Figure 1). The oil concentration was not determined for these samples. It can be seen that the Boiler Feed Water and ORF Water are not clean and with some free, soluble oil and / or asphaltenes present. This Boiler Feed Water is probably not acceptable in most other industries, but is tolerated in SAGD.

Operationally, SAGD boilers have a tolerance for Dissolved Oil and Water Soluble Organics. The needed measurement for SAGD boilers is the Free Oil independent of the Water Soluble Organics. Water Soluble Organics will tend remain in the water phase of the boiler and are flushed out during the blow downs However, Free Oil is likely to stick to these boiler tube surfaces to form coke, which can cause damage to the boilers.

Boiler Feed Water ORF



Measurement of Low Oil Concentrations in Boiler Feed Water

With the ability to measure oil concentrations below 1 part per million, continuous on-line fluorescence monitors are commonly used to measure water quality for boilers. Because no sample preparations with chemicals or homogenizers are used and the measurement is continuous, a change in process conditions can be detected quickly. With the optimized optical arrangement, fluorescence measurement of oil can be independent of other organics in the water. Typically boiler feed water processes for industrial boilers are clean with oil present. In Refineries, or other industrial facilities the boiler feed water could be recovered Steam Condensate or Cooling Water from an oil process. If a leak occurs in the heat exchanger it must be detected early, at low concentrations (below 1 ppm).

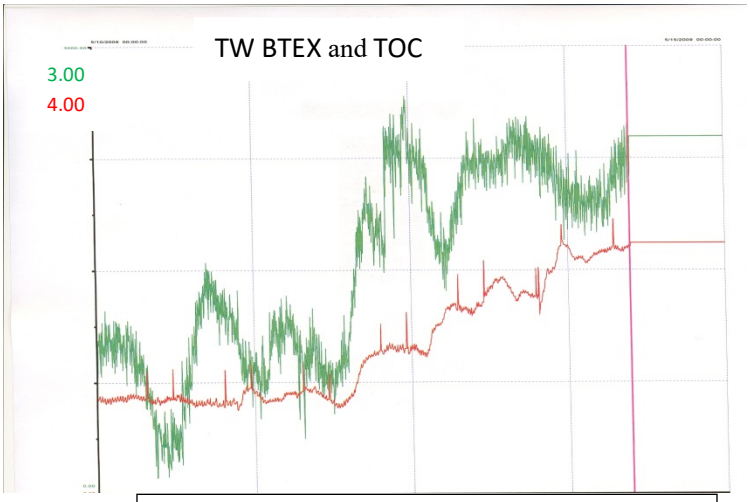


Figure 3: Boiler Feed Water measured by Total Organic Carbon Analyzer (Green) and Fluorescence Monitor (Red).

Scale: 0-3 ppm - TOC; 0-4 ppm - Fluorescence

In Figure 3, steam condensate is returned from a refining process and is mixed with makeup water from a local lake. The Total Organic Carbon measurement is responsive to both the naturally occurring organics like algae, decomposed plant and animal material, etc. in the lake water and the hydrocarbons which come from the refinery. The fluorescence monitor in this installation is configured to respond only to refined hydrocarbons. The left side of Figure 3 shows the TOC analyzer's measurement of organics in the lake water. However, once a leak occurs in a heat exchanger within the refinery, the TOC and the Fluorescence monitor both respond clearly indicating the presence of a hydrocarbon in the steam instead of a high organic load from the lake water.

The fluorescence response is on a molecular level. Soluble oils and free oil droplets will both produce a signal. The SAGD Water Soluble Organic signal has been seen as 2000 ppm. Yet the Free Oil concentration must be measured at less than 1 ppm for ideal boiler feed water quality. Even though the

concentration of WSOs is relatively constant, it can be difficult to have enough resolution to indicate when the Free Oil Concentration has changed.

An alternative method for measurement is needed for SAGD boiler feed water processes.

Measurement by Single Grab Sample Analysis

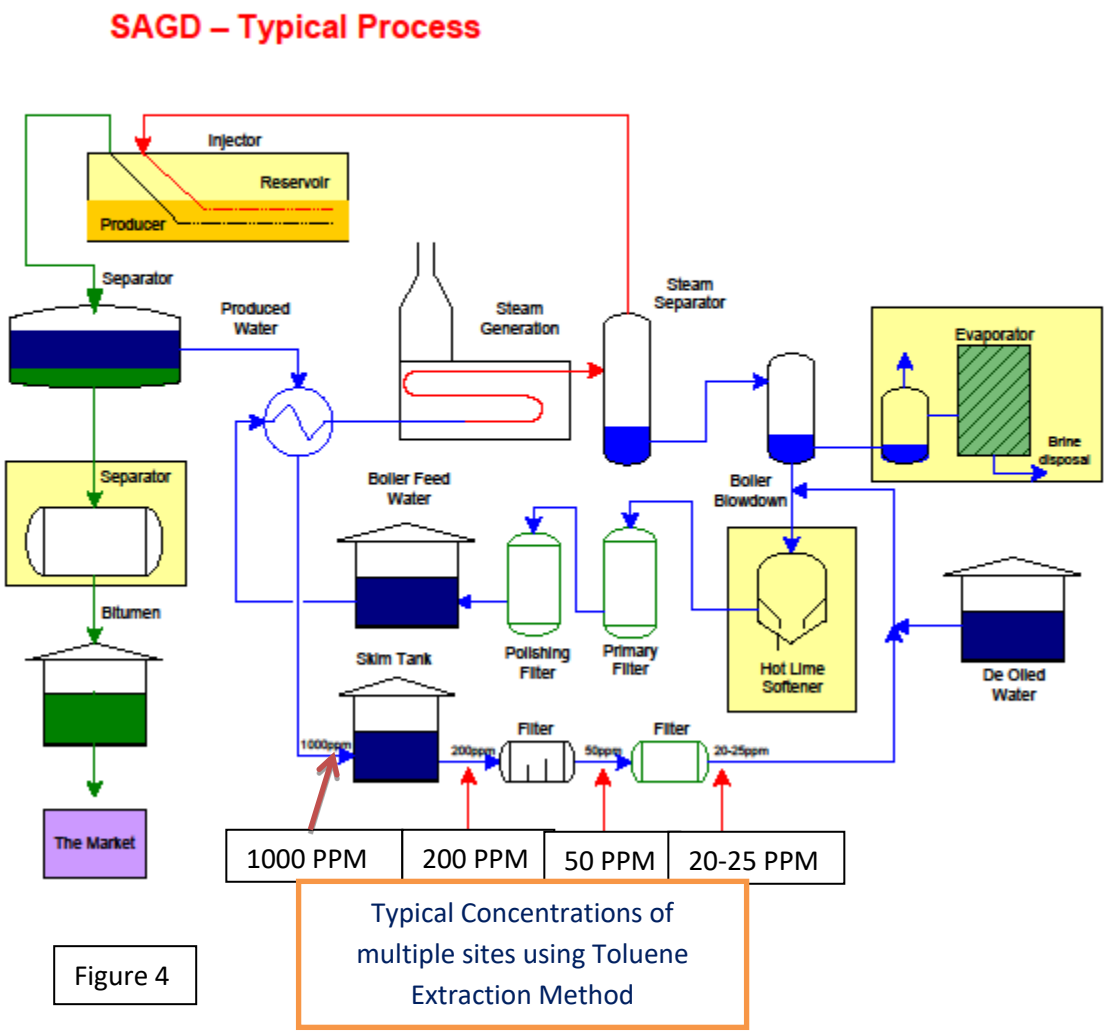


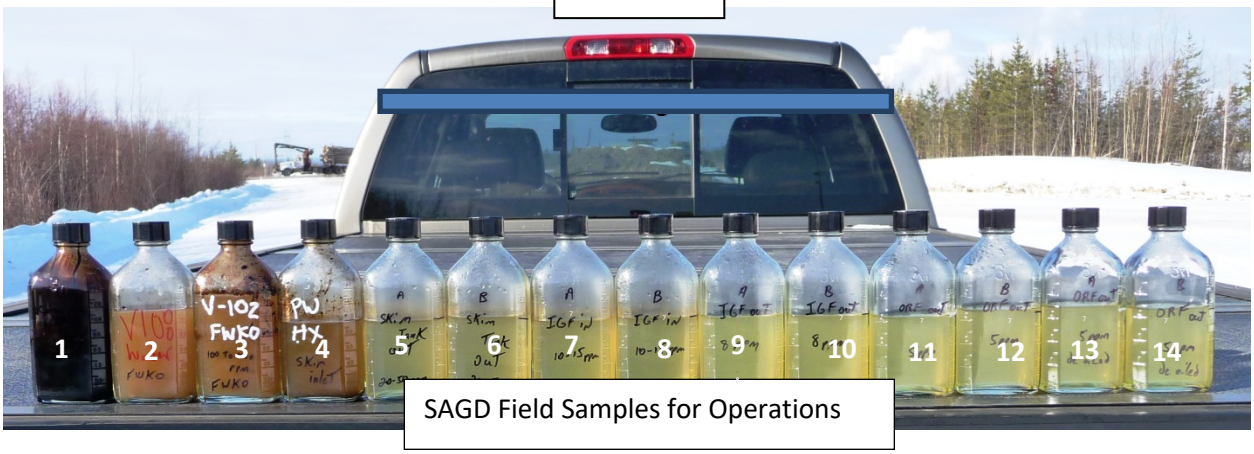
Figure 4

Because on line measurement of SAGD processes does not have the needed resolution needed for Free Oil Measurement, an alternate method was developed to prepare a water sample so that only the Free Oil would be measured. When oily water is collected in a sample bottle, separation of the oil and water begins immediately. Also, oil starts to stick to the glass and oily solids can settle to the bottom. To collect a sample of the water which accurately represents the original water sample, a procedure must be performed to produce homogeneous sample.

A common field preparation method for sample analysis of Produced Water includes collecting a 100 ml water sample, acidifying the sample to pH < 2.0 and then extracting the sample with Hexane. Because

most Water Soluble Organics are acidic, by reducing the pH these WSOs are better extracted by the solvent. After the extraction a sample of the solvent is collected, placed in a cuvette for measurement with a Fluorimeter. This produces a Total Oil Concentration that reliably correlates with laboratory analysis methods like the EPA 1664A. However, because only the Free Oil Concentration in SAGD is important, the acidification step is eliminated from the sample preparation process. Also, because Hexane is not an efficient extraction solvent for the Bitumen produced in SAGD, Toluene is used as the extraction solvent. The full details of the solvent can be found in Appendix A of this paper.

Picture 3



SAGD Field Samples for Operations

	Bottle Label	Measurement
2	V108 – FWKO	
3	V102 – FWKO	100 – 1000 PPM
4	PW HX Skim Inlet	
5	Skim Tank Out A	20 – 50 PPM
6	Skim Tank Out B	20 – 50 PPM
7	IGF In A	10 – 15 PPM
8	IGF In B	10 – 15 PPM
9	IGF Out A	8 PPM
10	IGF Out B	8 PPM
11	ORF Out A	5 PPM
12	ORF Out B	5 PPM
13	ORF Out A (DeOil In)	5 PPM
14	ORF Out B (DeOil In)	5 PPM

The water sample bottles in the above photo are typical from SAGD water processes that are analyzed by the Toluene extraction method. In most produced water processes, the Water Soluble Organic concentration is constant as the water passes through each separation step. The chart on the left shows the measurement of Free Oil that is typically reported in the SAGD process.

For instance, The V102 FWKO bottle will typically have 100 – 1000 PPM oil. This is supported in Picture 3, V102 is not as dark as is shown in right bottle in Picture 1 above. By observing no significant visible difference between the Skim Tank Bottles, the IGF In, IGF Out and the ORF Out Bottles because the Dissolved Oil / Water Soluble Organic concentrations are similar through all the water treatment systems. However, the Free Oil Concentration, as measured by the toluene extraction procedure is lower in each subsequent water treatment process, because the Free Oil is being removed but no Water Soluble Organics are removed.

Conclusion

Fluorescence measurement is capable and well suited to monitor most Boiler Feed Waters for the hydrocarbons at low concentrations as needed for proper Boiler operations. Although the ideal measurement of oil in the SAGD water would be continuous, due to the unique nature of the water chemistry, it is not possible at this time. A suitable grab sample alternate method has been developed and implemented in the SAGD production fields. Measurement interference from high Water Soluble Organic concentrations can be avoided through the non-acidified Toluene Extraction Method used for Sample Analysis. The measurement of water samples will provide an indication of process upsets and help the plant operators improve process control.

References

1. *Consensus Operating Practices for Control of Feedwater/ Boiler Water Chemistry in Modern Industrial Boilers*. ASME 1994

2. Elliot,K.T. & Kovsek, A.R. (1999). A Numerical Analysis of Single Well SAGD Processes. *20th Annual Workshop & Symposium Collaborative Project on Enhanced Oil Recovery International Energy Agency.* Petroleum Engineering Department. Stanford University.

3. Alberta Government. "Talk About SAGD". 11-2014. http://www.energy.alberta.ca/OilSands/pdfs/FS_SAGD.pdf

Appendix

Method Overview

Developed by Benchmark Instrumentation and Analytical Systems, Inc.

Water samples are extracted with toluene. The dual channel fluorescence sample analyzer measures the fluorescence of the toluene extracts and converts the fluorescence values (RFU, relative fluorescence units) to oil concentration (ppm) with an internal linear calibration. The slope of the calibration function (ppm/RFU) depends upon the fluorescence properties of the bitumen blend. These properties can vary with the composition of the diluent and the ratio of bitumen to diluent. Therefore, the fluorescence sample analyzer should be calibrated frequently to minimize analysis errors. Experience has shown that daily recalibrations give the best results.

The toluene extracts can be read with either Channel A or Channel B. Channel A fluorescence is linear with oil-in-water concentration from 0-5 ppm. Channel B fluorescence is linear with oil-in-water concentration from 0-50 ppm.

> **Note:** These linear ranges are typical for SAGD facilities. They may differ somewhat from site-to-site depending on bitumen composition, diluent composition and bitumen/diluent ratio.

Calibration of each channel is performed with a toluene blank and a standard solution of sales oil dissolved in toluene. Water samples are extracted with 1 part toluene for every 10 parts water. Because of this, oil-in-water concentration from a water sample is represented by 1/10 the oil-in-toluene concentration (i.e. A 10 ppm oil-in-water concentration becomes 100 ppm when extracted into toluene).

Equipment

Dual Channel Fluorescence Oil-in-Water Analyzer
Minicell Adaptor
Minicells
100 mL volumetric flasks (2)
Volumetric pipette, 0-100 µL
Pipette tips, 0-100 µL
Glass pipette, 10 mL
Glass pipette bulb
Transfer pipettes
Glass prescription bottles, 180 mL, graduated
Teflon caps
Toluene-resistant safety goggles
Nitrile gloves
Proper PPE
Fume hood
Toluene
Beaker, 50 mL

Preparation of Calibration Standards

1. Collect a fresh sample of sales oil (bitumen/diluent mixture).
2. Turn on the fume hood. All of the remaining steps should be carried out in the fume hood.
3. Fill a 100 mL volumetric flask to the bottom of the neck with toluene.
4. Pipette 20 µL of sales oil into the volumetric flask.
5. Add toluene to the flask to bring the total volume up to the 100 mL fill line.
6. Cap the flask and shake it vigorously to mix. Invert the flask several times until the sales oil is completely dissolved and uniformly distributed. This will make a 200 PPM Oil-in-Toluene Standard (analytically equivalent to 20 ppm oil-in-water).
7. Attach the pipette bulb to the end of the 10 mL glass pipette.
8. Pipette 10 mL of the 200 ppm oil-in-toluene standard into the second flask.
9. Fill the flask to the 100 mL fill line with toluene.
10. Cap the flask and shake it vigorously to mix. Invert the flask several times until the liquids are completely mixed. This will make a 20 ppm Oil-in-Toluene Standard (analytically equivalent to 2 ppm oil-in-water).

Instrument Calibration—Channel A

1. Fill a minicell about 2/3 full with pure toluene using a transfer pipette. This is the Blank.
2. Fill another minicell about 2/3 full with the 20 ppm oil-in-toluene standard using a transfer pipette.
3. Turn on the fluorescence sample analyzer. Place the minicell adaptor in the sample compartment.
4. Set the instrument to Channel A.
5. Wipe the Blank minicell with a Kimwipe and place it in the minicell adapter.
6. Close the sample compartment lid.
7. Press CAL.
8. Press ENT.
9. Press ENT.
10. Remove the BLANK minicell.
11. Wipe the minicell containing the 20 PPM Oil-in-Toluene Standard with a Kimwipe and place it into the minicell adapter.
12. Close the sample compartment lid.
13. Press ENT.
14. Press ENT.
15. Press DIAG and record the DIAG values.
16. Press STD VAL.
17. Press the up and down arrow keys until the STD VAL is set to 2.0 ppm oil-in-water.
18. Channel A is now calibrated to read 0 to 5 ppm oil-in-water.
19. Remove the minicell from the sample compartment.

CAUTION: Do not leave minicells containing toluene in the sample compartment for long periods. Liquid toluene and toluene vapors can damage the fluorescence sample analyzer's sample compartment and case.

Instrument Calibration—Channel B

1. Fill a minicell about 2/3 full with pure toluene using a transfer pipette. This is the Blank.
2. Fill another minicell about 2/3 full with the 200 ppm Oil-in-Toluene Standard using a transfer pipette.
3. Turn on the FLUORESCENCE SAMPLE ANALYZER. Place the minicell adaptor in the sample compartment.
4. Set the instrument to Channel B.
5. Wipe the Blank minicell with a Kimwipe and place it in the minicell adapter.
6. Close the sample compartment lid.
7. Press CAL.
8. Press ENT.
9. Press ENT.
10. Remove the BLANK minicell .
11. Wipe the minicell containing the 200 PPM Oil-in-Toluene Standard with a Kimwipe and place it into the minicell adapter.

12. Close the sample compartment lid.
13. Press ENT.
14. Press ENT.
15. Press DIAG and record the DIAG values.
16. Press STD VAL.
17. Press the up and down arrow keys until the STD VAL is set to 20 ppm oil-in-water.
18. Channel B is now calibrated to read 0 to 50 ppm oil-in-water.
19. Remove the minicell from the sample compartment.

CAUTION: Do not leave minicells containing toluene in the sample compartment for long periods. Liquid toluene and toluene vapors can damage the fluorescence sample analyzer's sample compartment and case.

Sample Analysis

1. Obtain a clean, 180 mL prescription bottle.
2. Mark the 100 mL line of the prescription bottle with a permanent marker ("Sharpie").
3. Open the sample valve so that water is flowing at a rapid rate. Allow the water to run for about 2 minutes to purge the tubing of deposited oil and solids.
4. If necessary, partially close the sample valve to reduce the water flow to a safe sampling rate.
5. Fill the prescription bottle to the 100 mL mark with process water.
6. Cap the bottle. Label the bottle with the sample location, date, and time of collection.
7. Allow the sample to cool to room temperature.
8. Add 10 mL of toluene to the prescription bottle.
9. Tightly cap the prescription bottle and shake it vigorously for 2 minutes.
10. Place the bottle on the counter to allow the toluene extract to separate from the water.
11. Wait for the toluene layer to at least partially clarify.
12. Using a transfer pipette, fill a minicell about 2/3 full with clear toluene extract. Make sure that no emulsion gets into the minicell.
13. Turn on the fluorescence sample analyzer.
14. Make sure the instrument is set to Channel B.
15. Clean the minicell with a Kimwipe and place into the fluorescence sample analyzer adapter.
16. Close the sample compartment lid.
17. Press READ.
18. If the Channel B reading is >5 ppm, record the value and go directly to **Linearity Check** (see below).
19. If the Channel B reading is <5 ppm, switch to Channel A, press READ to obtain a new reading and go directly to **Linearity Check** (see below).

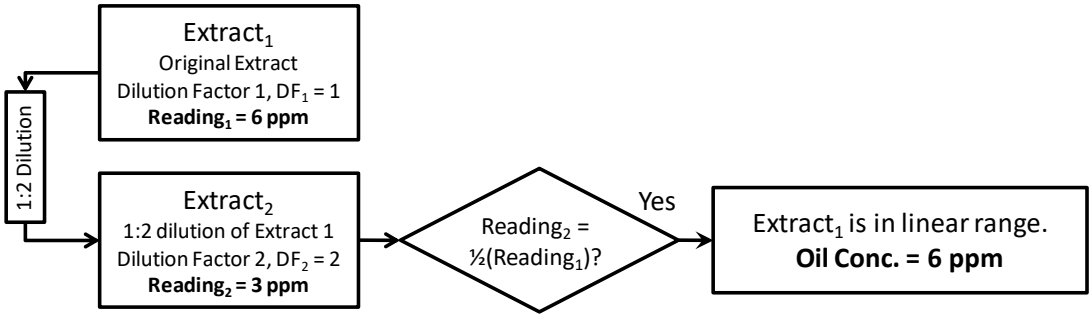
Linearity Check

When you first begin sampling from a particular process location, you should perform this procedure to ensure that your oil concentration is in the fluorescence sample analyzer's linear range. The procedure involves diluting the toluene extract 1:2 with pure toluene and reading the oil concentration again. If the reading decreases by approximately ½, then the oil concentration of the original extract was in the linear range. If not, dilute the already diluted extract 1:2 again and obtain another fluorescence sample analyzer reading. Continue diluting serially by 1:2 until the reading of the most diluted extract is close to

½ the reading of the previous extract. Keep track of the total serial dilution factor of each extract. Once you find an extract in the linear range, compute the oil concentration of the water sample by multiplying the next-to-last reading by the total serial dilution factor of the next-to-last extract. After you gain experience at a certain location, you can perform the appropriate dilution (if one is required) in one step to speed up the analysis procedure.

Example 1. Original Extract is in the linear range.

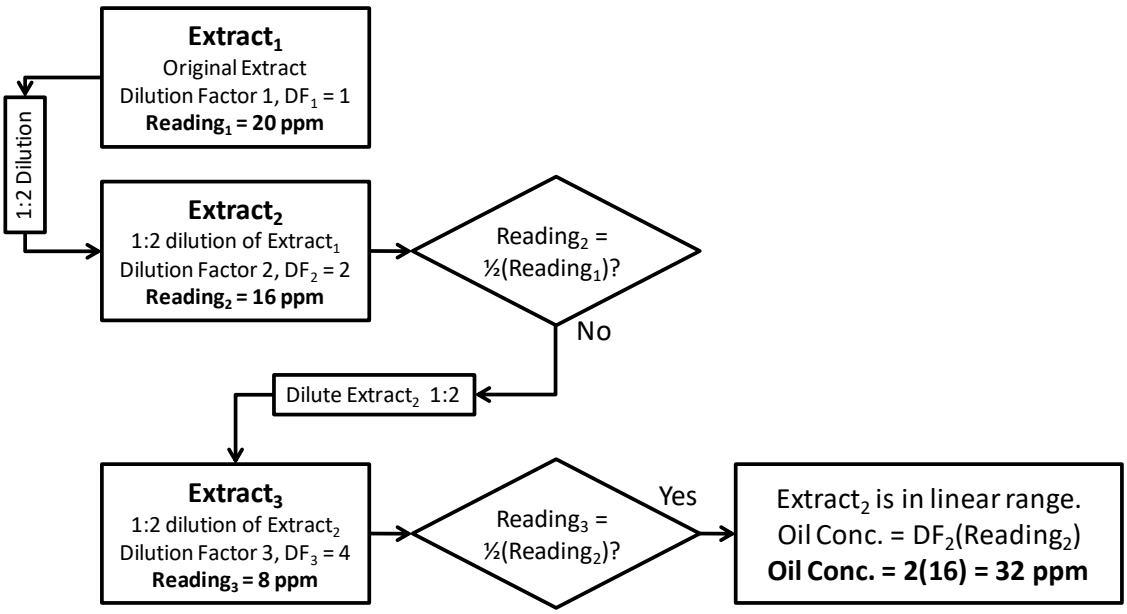
1. $Extract_1$ (original extract) gave a reading of 6 ppm.
2. The reading for $Extract_2$ (1:2 dilution of $Extract_1$) gave a reading of 3 ppm.
3. The $Extract_1$ was in the linear range and 6 ppm is the correct oil concentration.



Example 2. Original Extract is not in the Linear Range

1. The $Extract_1$ (original extract) gave a reading of 20 ppm.
2. The reading for $Extract_2$ (1:2 dilution of $Extract_1$) gave a reading of 16 ppm.
   Since 16 is > ½(20), another dilution is required.
3. The reading for $Extract_3$ (1:2 dilution of $Extract_2$) was 8 ppm.
4. Since 8 is equal to ½(16), $Extract_2$ is in the linear rangeOil concentration is computed by:

$$\text{Oil Conc.} = DF_2(\text{Reading for Extract 2}) = 2(16) = 32 \text{ ppm} \qquad (DF_2 = 2)$$



The procedures above can be put in general terms:

1. Extract the water sample. This is $Extract_1$, (N=1).
2. Obtain $Reading_1$ for $Extract_1$.
3. Serially dilute 1:2 until $Reading_N$ for $Extract_N$ is within 10% of ½($Reading_{N-1}$) for $Extract_{N-1}$.
4. Compute oil concentration by:

$$\textbf{Oil Conc.} = \mathbf{DF_{N-1}(Reading_{N-1})}$$

where,

$$\mathbf{DF_{N-1} = 2(N-1)}$$

A convenient procedure for accurately performing the serial dilutions is suggested below:

1. Using a volumetric pipette, dispense 100 µL of the original extract into a new minicell.
2. Using the same volumetric pipette, but with a new tip, add 100 µL of pure toluene to the new minicell.
3. While wearing clean nitrile gloves, place your finger over the minicell opening. Mix the diluted extract by repeatedly inverting the minicell. The oil concentration in the new minicell is ½ of the oil concentration of the original extract.

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