

Infrared Analysis – A Valuable Technique for Measuring Oil Levels in Industrial Wastewater

Fixed filter infrared (IR) analysers have been used to test oil content levels in produced water on offshore drilling platforms around the world for over 45 years. EPA methods 418.1 and 413.2 were used extensively until the Montreal Protocol called for Freon (the solvent used in the analysis) to be phased out. Hexane/infrared extraction as well as ASTM D-7066 S-316 (dimer/trimer of chlorotrifluoroethylene)/infrared extraction are now widely used for offshore oil in water testing.

“Infrared analysis can be a valuable measurement method for industrial wastewater professionals to help ensure discharges meet regulatory requirements.”

For industrial wastewater professionals, a rapid on-site infrared test can be invaluable—saving money, time and SSOs (Sanitary Sewer Overflows) by catching wastewater that is over the regulatory limit. On the industrial side, operators of a pretreatment system can test how their oil separators work under different loading conditions. For the treatment facility that is imposing the limits, an on-site test allows them to catch offenders. By sending only the samples over the limit for further testing, a significant savings on laboratory testing costs is achieved, as well as avoiding a more costly SSO by diverting effluent loaded with fats, oil and grease (FOG) that causes the blockages.

Individuals involved in industrial wastewater testing can benefit from the highly regulated, time-tested, field-proven choice of using infrared analysis to verify their oil in water levels.

Reduce Costs and Sampling Time

Infrared analysis provides an alternative to gravimetric testing which will significantly reduce costs and save time in the following ways:

1. 90% less hexane required for solvent extraction

When using EPA method 1664, a litre sample requires 100ml of hexane for the extraction. The amount of solvent cannot be reduced with this method as the weight of the residual oil would be so low it would be less accurate for lower levels of oil and grease. With the hexane/infrared extraction method, only 50 µl of extract are required for analysis and the sample size can be reduced to 100 ml for a fairly well mixed waste stream. This 100 ml volume only requires 10 ml of hexane for the extraction. In addition to a cost savings, reduced solvent usage means less waste, exposure and volatile fumes.



Photo 1: Hexane layer on top of sample

The hexane/infrared method is easy:

- Add hexane to the sample and shake for 2 minutes
- Allow sample to partition
- Take 50 µl from the top layer of hexane extract and deposit on sample plate (Photo 1)
- Press the “run” and measurement result is displayed

2. 10-15 minutes versus 2 hours for analysis

The hexane/gravimetric method is time consuming and labour intensive -- taking up to 2 hours before a final result. The hexane/infrared method takes less than 10-15 minutes. This means quick sample turnaround and less laboratory technician time.

3. Analysis can be done on-site

Fixed filter infrared analysers, like the Wilks InfraCal Analysers (photos 2 and 3) are compact (less than 6” square), light weight (less than 5 lbs) and be operated from a 12 volt power supply allowing them to be operated from a vehicle. Wastewater effluent testing can be done at the site -- making it easier to catch high oil and grease offenders. By screening for out-of-compliance effluent discharges, the number of samples collected, transported and ultimately tested in the laboratory can be reduced. The same can apply for in-laboratory testing. Samples can be quickly screened and the effluent samples that are over the oil and grease limit can be tested by the EPA 1664 method—saving time, solvent, and labour costs.

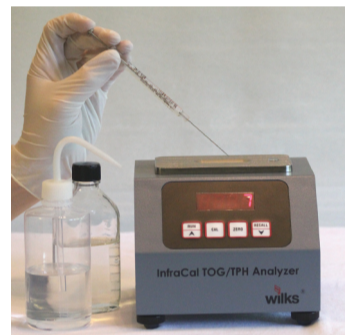


Photo 2: InfraCal Oil in Water Analyzer



Photo 3: New InfraCal 2 Oil in Water Analyzer

Matching Regulatory Methods

An often-asked question is will the infrared oil in water analysis method match the regulatory method?

While the amount of oil in water is highly regulated -- it can also be a challenging measurement. It is complicated by the fact that oil comes in many forms and the measurement is defined by the particular regulatory method. When EPA 1664 is the regulatory method, the “oil” is anything that is extracted into hexane and remains after the hexane has been evaporated and shows up as weight. In regions where infrared analysis is the defining method, the “oil” is whatever is extracted into the solvent and has carbon-hydrogen bonds that absorb infrared light at a specific frequency. Each method is looking at different properties of oil and can potentially give different results.

How one type of oil in water measurement compares with different regulatory methods is not always simple and straightforward. Listed below are four factors that need to be considered.

1. Precision and bias for each method

There are acceptable errors for each method typically expressed in the precision and bias statement for the method. EPA Method 1664 states in their “Ongoing precision & recovery” (section

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InfraCal	InfraCal	Lab 1	Lab 2	Lab 3
51	49	67	23	51
62	61	74	43	47
71	--	76	70	49
63	53	66	55	40
3	1	14	7	<5

Table 1: Comparison of a five way sample split with 3 different labs performing EPA 1664 and 2 infrared analysers

17.0) that for a 100 ppm sample the acceptable range is 78 -114 ppm. If the test includes the silica gel treatment (SGT) to remove the polar organics, the acceptable range is 64 – 132 ppm. Therefore, if the result from a laboratory for a silica gel treated sample is 65 ppm and the alternate method result is 130 ppm, they are within the acceptable range.

2. Operator errors

Even the same method can give significantly different results. With any method where there is sample preparation, the human factor is added in. If a solvent/sample mix is only shaken for one minute rather than the required two minutes, the amount of oil extracted into the solvent will be significantly less. Table 1 shows a comparison of a five way sample split analysed on two infrared InfraCal TOG/TPH Analyzers and at three laboratories performing the EPA 1664 method. The lab results vary significantly.

3. Sampling

Sample collection and handling can cause differences for comparative testing. In order to have an objective comparison, the samples should be identical. If there is variability in the waste stream, this can be a difficult task. The old adage that oil and water do not mix holds true for wastewater as well.

Oil also likes to stick to glass. If sample collection containers are being reused, they should get a final solvent rinse after cleaning to remove any residual oil. For sample analysis, the solvent should either be blended in the sample container or if

the sample is to be transferred to another container, the sample container should be rinsed with a portion of the solvent that will be used for the extraction. If not, any oil on the container surface will be excluded from the reading.

4. Sample Disparities

Not all oil in water samples are alike which makes it difficult when comparing analytical methods that look at different properties of the oil. Oil is a mix of chemical components that changes from one location to another and can even change at the same location.

For example, samples often contain a mix of aromatic and aliphatic hydrocarbons. UV fluorescence only detects aromatic hydrocarbons while infrared will detect both aromatic and aliphatic hydrocarbons. The two analytical methods could give different results if the aromatic/aliphatic ratio changes.

Samples that contain volatile hydrocarbons could also show different results between EPA 1664 and an infrared transmission method. With the 1664 gravimetric method, any volatile oils below the boiling point of hexane will be evaporated off with the solvent. With an infrared method that uses S-316 or perchloroethylene and a transmission cell, the oil is measured directly in the solvent without evaporation and the volatile hydrocarbons will be retained. This will make a transmission infrared reading higher than an EPA 1664 result if the sample has volatile oils. Like 1664, the hexane/ infrared extraction method has an evaporation step and volatile hydrocarbons will not be included in the measurement.

InfraCal Model HATR-T2	EPA 1664
31	33
23	29
15	30
20	34
10	12
12	14
12	13
2	14
25	24
19	11
17	21
17	19

Table 2: Comparison of samples tested with EPA 1664 and an infrared analyser

Infrared Analysis is a Valuable Technique for Wastewater Professionals

All of the considerations listed above may make it look like any correlation could be difficult. However, by using careful analytical procedures, understanding the composition of the waste stream and knowing the limits of each measurement system, useful information can be generated. Table 2 shows samples from an oil rig in the North Sea that were tested by a laboratory using EPA 1664 and the InfraCal TOG/TPH Analyzer, Model HATR-T2, which uses hexane as an extraction solvent.

This example clearly shows that infrared analysis can be a valuable measurement method for industrial wastewater professionals to help ensure discharges meet regulatory requirements. Industrial pretreatment operators have the advantage of taking samples before and after treatment to see how a system functions with different loads and regulators can catch high FOG levels before it stops sewer lines.