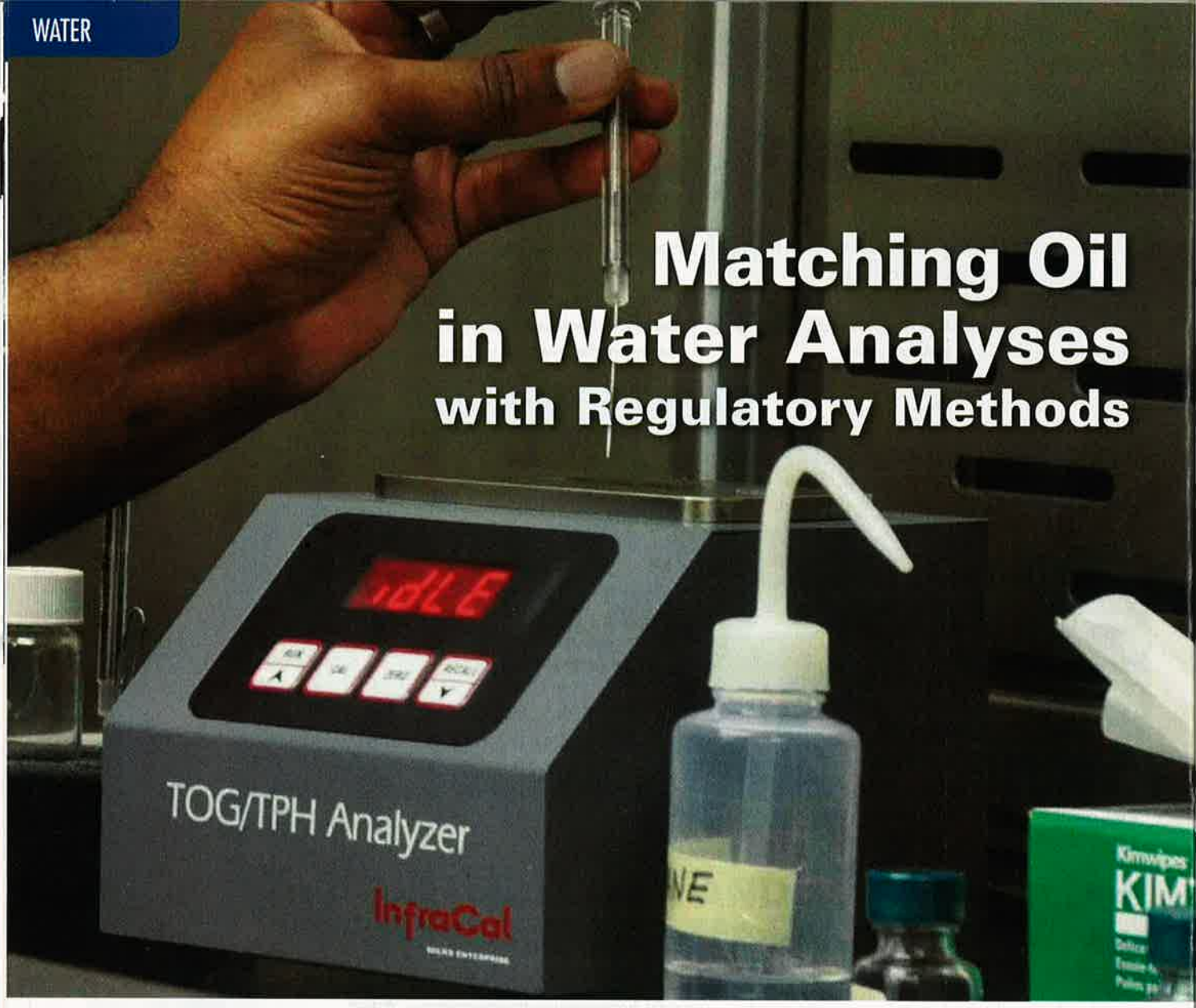

Matching Oil in Water Analyses with Regulatory Methods

When analyzing oil and grease in water while out in the field, it is important to use an EPA approved method and equipment.

>> BY SANDRA RINTOUL, PRESIDENT OF WILKS ENTERPRISE INC.

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 appears 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 reviews different properties of oil and can potentially yeild different results.

Therefore, the answer to the commonly asked question of 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.

Above: An infrared analyzer similar to the one shown above can provide results that meet the requirements of EPA Method 1664.

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. For example, EPA Method 1664 states in their Ongoing precision & recovery (section 17.0) that for a 100 ppm sample, the acceptable range is 78 to 114 ppm. If the test includes the silica gel treatment (SGT) to remove the polar organics, the acceptable range is 64 to 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**

With any method where there is sample preparation, the human factor is a contribution to be considered. 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. In some cases, it has been half the reading. **Table 1** *(on page 28)* shows a comparison of a five-way sample split analyzed on two InfraCal TOG/TPH Analyzers at three laboratories.

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.

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 in order to make the measurement. Oil is a mix of chemical components that changes from one location to another – it 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 yeild 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 using a transmission cell, the oil is directly measured in the solvent without evaporation and the volatile hydrocarbons will be retained. This will make a transmission infrared reading higher than the EPA Method 1664 result if the sample contains volatile oils.

While all of the considerations listed suggest any correlation could be difficult, by using careful analytical procedures, understanding compositions of the waste streams and knowing the limits of each measurement system, useful information can still be generated. **Table 2** *(on page 28)* shows samples from on an oil rig in the North Sea that were tested by a laboratory using EPA Method 1664 and an InfraCal TOG/TPH Analyzer, which uses hexane as an extraction solvent. This example demonstrates that infrared analysis, which has been used off-shore

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Wilks InfraCal	Customer's 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: Possible variation in sample results. Five samples were analyzed at three laboratories.

InfraCal Model HATR-T2	NTL-1664
31	33
15	32
17	27
23	29
15	30
20	34
10	12
12	14
12	13
2	14
25	24
19	11
13	21
10	21
17	21
17	19
15	19
13	14
13	8
13	10
14	11
13	12

Table 2: North Sea oil rig samples were tested on site and compared to results supplied by a laboratory using Method 1664

for over 40 years, can be a valuable tool in assessing if an oil separation system is performing to the required specifications.

So, the answer to the initial question as to whether one analysis method will match another is that most methods will typically correlate closely enough to provide operators with the information necessary to make sure their effluent does not exceed the regulatory limit. Will the numbers match exactly? Sometimes…

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