Measuring Oil in Water: A Sanity Check

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Outline

• Common Methods Used – Advantages & Disadvantages
• Measurement Techniques Used in This Experiment
• Sample Preparation
• Measurement Results
• Conclusions – Things You Need to Know!
What is Oil in Water, and Why Measure it?

• “Oil in water” is not precisely defined
• Really only defined by a “method” of measurement!
• Initially driven by environmental concerns
• Now being driven more by economic concerns (extraction efficiency)
Two Major Types of Measurement Techniques

• “Indirect” - Most common techniques: Measure something that can be “correlated” to oil in water (IR absorption, UV fluorescence, etc.)

• “Direct” techniques: Directly measure oil in water (particle counters, imaging devices, ultrasound, etc.)
Common Measurement Methods 1: EPA 1664

- US Regulatory Method: the “yardstick”
- “Direct” method; chemical extraction & gravimetric
- Only measures organics soluble in hexane, therefore not ALL “oil in water”
- Limited to laboratory environment and skilled personnel
Common Measurement Methods 2: IR Absorption

- “Indirect” method: C-H bond common to organics absorbs InfraRed (IR)
- Cannot use water as solvent as it also absorbs IR, so must use other solvent
- Must be calibrated using known concentration samples
- Limited to laboratory environment and skilled personnel
Common Measurement Methods 3: UV Fluorescence

- “Indirect” method: aromatics absorb UV and fluoresce at different emission wavelength
- Amount of fluorescence proportional to amount of aromatics present
- Advantage over IR absorption: no solvent required
- Other compounds (e.g. Iron) also may fluoresce
New Measurement Techniques
1: Particle Counters

• “Direct” measurements
• Turbidity: too “coarse”, not precise or repeatable for sparse samples
• “Electrozone counters”: also limited to laboratory environment
• Cannot distinguish between “droplets” and other particulates (e.g. sand, etc.)
New Measurement Techniques

2: Imaging Particle Analysis

- “Direct” measurements
- Very rapid and repeatable, with potential to use in-situ or “at-line”
- Can differentiate between oil droplets and other particulates based upon shape
- Limited to $\geq 3\mu m$ in diameter due to optical considerations
Imaging Particle Analysis
How it Works:

Narrow Depth Flow Cell Restricts Location of Particles Perpendicular to the Optical Axis

Digital Camera

Microscope Objective

Optical Axis

Sample Flow

Light Source
Imaging Particle Analysis
How it Works:

- Oil Droplets
- Other Particulates
Imaging Particle Analysis

How it Works:

Oil Droplets
Sample Preparation

• SOP developed and closely followed
• Batches of sample mixed with known concentrations
  – 10, 25, 50, 100, 500, 1000 PPM
  – Each batch then separated into 4 identical samples for each test method
• 2\textsuperscript{nd} Batch also made with known quantity of sand added to test “separability”
Methods Tested

• All methods used on same samples
  – EPA 1664
  – IR Absorption
  – Two different imaging particle analysis systems
Results 1 (oil only):

Instrument Reading vs Actual Concentration

Actual Concentration (PPM)

Instrument Reading (PPM)

- EPA
- IR Device
- Opt Device 1
- Opt Device 2
Results 2 (oil + sand):

Instrument Reading vs Actual Concentration with Sand added to Oil Sample

Actual Concentration (PPM)

Instrument Reading (PPM)

- EPA
- Opt Device 1
- Opt Device 2
Discussion of Results

- Imaging particle analysis system #1 most closely tracked EPA 1664 results
- Imaging system #2 only reasonably accurate for concentrations >50 PPM
- IR absorption consistently higher by order of magnitude for concentrations <500 PPM
- Imaging system #1 only one that tracked closely after addition of sand
Things to Remember!

• All techniques have positives/negatives
• A firm understanding of how the measurement method works is key
• Any method should be validated and calibrated against known test samples
• Known calibration samples should be “representative” of the actual environment to be measured for best correlations
Questions?

Thank you!