Acknowledgements

- Funding for the project (Project No. 12121-6301-03) is provided through the “Ultra-Deepwater and Unconventional Natural Gas and Other Petroleum Resources Research and Development Program” authorized by the Energy Policy Act of 2005.
- National Energy Technology Laboratory (NETL)
- Research Partnership to Secure Energy for America (RPSEA)
- Working Project Group Members
  
  ExxonMobil (Project Champion)
  Anadarko
  BG
  Fluor
  OneSubsea
  Petrobras
  Statoil
  Total

- Presenters and Participants at Both Workshops
- Project Team
This presentation was prepared by Clearview Subsea LLC as an account of work sponsored by the Research Partnership to Secure Energy for America, RPSEA. Neither RPSEA members of RPSEA, the National Energy Technology Laboratory, the U.S. Department of Energy, nor any person acting on behalf of any of the entities:

a. MAKES ANY WARRANTY OR REPRESENTATION, EXPRESS OR IMPLIED WITH RESPECT TO ACCURACY, COMPLETENESS, OR USEFULNESS OF THE INFORMATION CONTAINED IN THIS DOCUMENT, OR THAT THE USE OF ANY INFORMATION, APPARATUS, METHOD, OR PROCESS DISCLOSED IN THIS DOCUMENT MAY NOT INFRINGE PRIVATELY OWNED RIGHTS, OR

b. ASSUMES ANY LIABILITY WITH RESPECT TO THE USE OF, OR FOR ANY AND ALL DAMAGES RESULTING FROM THE USE OF, ANY INFORMATION, APPARATUS, METHOD, OR PROCESS DISCLOSED IN THIS DOCUMENT.

THIS IS AN INTERIM PRESENTATION. THEREFORE, ANY DATA, CALCULATIONS, OR CONCLUSIONS REPORTED HEREIN SHOULD BE TREATED AS PRELIMINARY.

REFERENCE TO TRADE NAMES OR SPECIFIC COMMERCIAL PRODUCTS, COMMODITIES, OR SERVICES IN THIS REPORT DOES NOT REPRESENT OR CONSTITUTE AND ENDORSEMENT, RECOMMENDATION, OR FAVORING BY RPSEA OR ITS CONTRACTORS OF THE SPECIFIC COMMERCIAL PRODUCT, COMMODITY, OR SERVICE.
Outline

- Overview of RPSEA 12121-6301-03 Project
- Subsea Produced Water Sensor Requirements
- Technology Gap Analysis and Ranking
- Proof of Concept on Confocal Laser Fluorescence Microscopy
- Plan for Phase 2
- Forecast and Actual Cost
Overview of Project

- **Project: RPSEA 12121-6301-03, Subsea Produced Water Development**

- **Project Goal**
  - Progress Subsea Sensors to TRL 3 - performance tested (API 17N definition)
  - Focus on PWD Sensor
    - Developing technology toward enabling measurement of PW quality for regulatory compliance reporting

- **Project Schedule: September 2014 to September 2016**
Project Scope

- **Phase 1 (9 Months: September 2014 – June 2015)**
  - Develop Subsea Produced Water Sensor Requirements by Collecting Industry and Regulatory Input
  - Analyze the Technology Gaps in the Current Sensors
  - Proof of Concept on Confocal Laser Fluorescence Microscopy (CLFM)
  - Select up to 3 Sensors for Further Development in Phase 2
    - 2 Sensors from Established (Surface Operation) Technologies
    - CLFM as New Technology if Concept Is Proven in Phase 1

- **Phase 2 (15 Months: July 2015 – September 2016)**
  - Design and Construct Prototypes
  - Bench-Scale Testing of the Prototypes
Outline

- Overview of RPSEA 12121-6301-03 Project
- Subsea Produced Water Sensor Requirements
- Technology Gap Analysis and Ranking
- Proof of Concept on Confocal Laser Fluorescence Microscopy
- Plan for Phase 2
- Forecast and Actual Cost
Subsea PW Sensor Requirements

- Industry Workshop Held on December 16, 2014
  - Collected participant input
  - Attended by representatives from operators, subsea system suppliers, engineering companies and consultants, standard organizations

- Preliminary Requirements Developed from Workshop Discussions

- Regulatory Agency Input
  - Contacted BSEE and EPA
  - Response from BSEE
  - Response from EPA and discussions
Scope of Sensor functions

- Measure the oil and grease content in PW for NPDES compliance (or alternative compliance) reporting with readings equivalent to those from the EPA 1664 method.
- Be periodically validated or re-calibrated for reporting oil and grease content which is equivalent to EPA 1664.
- Provide information to the control system on the trending of produced water quality.
- Provide warnings and alarms to the control system if the quality of the produced water becomes worse than pre-designated levels.
  - The alarms shall have the required, operator-specific confidence level for the alarms to be used as input for the decision on process shutdown or flow diversion.
Outside of Sensor Functions Scope

- **Toxicity Testing**
- **Sand and Solids**
  - However, the sensors are required to properly account for the oil and grease associated with suspended sand and solids
- **Free oil discharge requirement**
  - However, the requirement for regulatory compliance is to be determined pending regulatory agency input
- **Subsea Process Monitoring**
  - No specific requirements
Preliminary Sensor Requirements

- **For NPDES Permit Compliance**
  - BSEE or EPA has not provided direct comments on the sensor requirements. Protocol for approving New Method for Method Defined Parameters under development
  - Assumed requirements from industry input
    - Sensors will measure oil in water concentration, and convert to Oil and Grease amount
    - Accuracy of 10 - 15% as compared with EPA 1664, or statistically equivalent to EPA 1664. To be determined pending future protocol or specific requirements by EPA.
      - EPA 1664 Method precision is 8.7% - 11%.
    - Once correlation is proven, the sensor readings should be considered as the actual oil and grease amount for compliance reporting
    - Periodical validation/verification of the correlation needed. Method to be determined.
For NPDES Permit Compliance

- Toxicity: Not Measured. Tests will be by sampling

- Sheen: Not Measured. No Sheening due to discharge at seabed

- Water Soluble Organics: Not Directly Measured.
  - The oil and grease contribution would be accounted for by the correlation with EPA 1664 measurements
  - Most WSOs are fatty acids

- Sand and Solids: No Requirements
  - Whether the oil and grease on sand and solids is properly measured would be reflected in the accuracy of correlation with EPA 1664
Preliminary Sensor Requirements (3)

- **For Operations**
  - Alarming for flow diversion/process shutdown
    - Frequency of reporting measurements: hourly minimum
    - Acceptably low amount of false alarms
    - Need also to comply with operator-specific procedures
  
  - Redundancy is important
    - May likely need multiple sensors/technologies – Voting and redundancy.
    - May combine inline and online technologies
  
  - Detecting process upsets: continuous measurements
    - Spikes of oil and grease concentration may be caused by problems in separation or PW treatment system
### Preliminary Sensor Requirements (4)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oil Concentration</td>
<td>0 – 100 mg/L</td>
<td>Typical 15 ppm</td>
</tr>
<tr>
<td>Solid Concentration</td>
<td>0 – 100 mg/L</td>
<td>Oil and grease measurement accuracy may be affected. Measurement of solids not required.</td>
</tr>
<tr>
<td>Gas</td>
<td>0.5% Volume Fraction</td>
<td>Gas out of solution in treatment system</td>
</tr>
<tr>
<td>Accuracy</td>
<td>10-15%, or Statistically equivalent</td>
<td>Comparison with EPA 1664 Measurements</td>
</tr>
<tr>
<td>Water Depth</td>
<td>Up to 10,000 ft</td>
<td></td>
</tr>
</tbody>
</table>
| Seawater Temperature       | 33 F                               | 28 F as next step  
EPA Region 10 (Alaska)  
- No PW discharge in North Slope general permit  
- No PW discharge in Arctic general permit Beaufort and Chukchi seas (Exploration only) |
## Preliminary Sensor Requirements (5)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Design Temperature</td>
<td>33 – 300 F</td>
<td>350 F as next step</td>
</tr>
<tr>
<td>Operating/Service Temperature</td>
<td>38 – 200 F</td>
<td>Also refer to EPA/company limits</td>
</tr>
<tr>
<td>Design Pressure</td>
<td>10,000 psig</td>
<td>15,000 psig as next step</td>
</tr>
<tr>
<td>Operating/Service Pressure</td>
<td>0 – 5000 psig</td>
<td></td>
</tr>
<tr>
<td>Flow Velocity</td>
<td>Max: Up to 15 ft/s</td>
<td>Each sensor manufacturer to determine the specific limit</td>
</tr>
<tr>
<td></td>
<td>desired</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Min: To be determined</td>
<td>Min flow limit is for low PW flow in early life</td>
</tr>
<tr>
<td>Oil Density</td>
<td>20 – min 35, maybe 60</td>
<td>Even though Lower Tertiary fields can have oil gravity as low as 11 API, subsea separation below 20 API will be very challenging, maybe even not feasible. Upper limit for condensate.</td>
</tr>
<tr>
<td></td>
<td>API</td>
<td></td>
</tr>
</tbody>
</table>
### Preliminary Sensor Requirements (6)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Produced Water Salinity</strong></td>
<td>0 – 250,000 ppm</td>
</tr>
<tr>
<td><strong>Chemicals in Produced Water</strong></td>
<td>Typical deepwater chemicals and concentrations. Completion fluids including ZnBr.</td>
</tr>
<tr>
<td><strong>Response Time</strong></td>
<td>Hourly or faster</td>
</tr>
<tr>
<td><strong>Design Service Life</strong></td>
<td>25 years</td>
</tr>
<tr>
<td><strong>Mean Time Between Failure</strong></td>
<td>5 years minimum</td>
</tr>
<tr>
<td><strong>Maintenance by ROV</strong></td>
<td>Max quarterly or annually (as toxicity test schedule)</td>
</tr>
<tr>
<td><strong>Repair by Retrieval</strong></td>
<td>Max once every 5 years; Max weight - light intervention class</td>
</tr>
<tr>
<td><strong>Shock</strong></td>
<td>Per ISO 13628-6: Q1 (5 g) or Q2 (10 g) as applicable</td>
</tr>
<tr>
<td><strong>Vibration</strong></td>
<td>Per ISO 13628-6</td>
</tr>
</tbody>
</table>
Preliminary Sensor Requirements (7)

- **Integration with Subsea Control System**
  - Compliant with Subsea Instrument Interface Standard;
  - Provide readings in engineering values, e.g. oil and grease in mg/L
  - Power consumption limit – TBD, but as low as possible. MPFM as reference
  - Allow the control system to update the correlation parameters
  - Other typical requirements (company/project specific)
    - Allow the control system to direct its operation
    - Allow the control system to query its status and download stored data
    - Allow the control system to update end-user parameters such as measurement frequency, reporting frequency, quality levels for alarms
    - Allow the control system to set or change the data to be transferred to the control system, the frequency or timing of the transfer, and the storage of data to be transferred
    - Report regularly to the control system on the upcoming maintenance needs, applicable inventory levels, and other necessary operation and maintenance information for the sensor
BSEE and EPA Input

- **BSEE**
  - Formally assess new technology when at TRL 8-9
    - TRL per DOE/NASA/DOD definitions, e.g., TRL8 is *Technology is proven to work - Actual technology completed and qualified through test and demonstration.*
  - Technologies historically provided to BSEE as part of an OCS Operator Plan

- **EPA**
  - Two categories of changes from methods in permit: Alternative Test Procedure (ATP) and New Method
  - Subsea PWD Sensor use is New Method
  - Oil and Grease is a Method Defined Parameter (MDP), for which EPA’s current protocol for approving New Method does not apply
  - Consequently, approval of sensor use not expected at present
  - EPA working group is studying the protocol for approving ATP and New Methods on MDP. However, no decision yet.
Outline

○ Overview of RPSEA 12121-6301-03 Project
○ Subsea Produced Water Sensor Requirements
○ **Technology Gap Analysis and Ranking**
○ Proof of Concept on Confocal Laser Fluorescence Microscopy
○ Plan for Phase 2
○ Forecast and Actual Cost
Evaluation and assessment are only for the purpose of selecting sensors for participation in Phase 2

- Evaluations of sensors are based on available information provided by vendors and/or from public literature
- Assessment and scoring are subjective
- Scoring and weighting factors are heavily biased to subsea application
- Evaluations, assessments and results are NOT endorsements or exceptions for any vendor’s product or track record
Gap Analysis

- **Industry Workshop held on 23 February 2015, Houston**
  - Collected industry input on technology gaps and how to close gaps
  - Discussed sensor technical requirements and gap analysis methodology
  - Attended by over 40 representatives from operators, subsea system suppliers, engineering companies, consultants, standard organizations and vendors

- **Gap Analysis**
  - Developed the gap analysis methodology
  - Listed sensors / vendors for inclusion in the gap analysis
  - Contacted with vendors
  - Conducted the gap analysis
  - Reporting
Gap Analysis – Workshop

- **Key Gaps Identified**
  - Regulations for subsea discharge
  - Performance: low accuracy and measurement range
  - Reliability: low availability, MTBF (key issue: fouling)
  - Representative subsea sampling: verification / measurement
  - Testing: reference methods, lack of purposefully built testing facilities
  - Standards: qualification testing and instrument design

- **Closing the Gaps**
  - Information / results sharing / operators investing
  - Involvement of regulators / help better define requirements
  - Vendors to have an adviser on marinization / system integration
  - Better definition of oil and grease
  - Credible test program
  - Continue development on fouling mitigation technologies
Technologies for Gap Analysis

- Advanced Sensors (LIF)
- Digitrol (Light Scattering)
- J M Canty (Microscopy)
- Jorin (Microscopy)
- Mirmorax (Focused Ultrasonic Acoustic)
- ProAnalysis (LIF)
- Turner Design Hydrocarbon Instruments (Conventional UV Fluorescence)
Technologies Included in Gap Analysis

**Laser Induced Fluorescence**  
*Graph Source: ProAnalysis*

**Microscopy**  
*Graph Source: J M Canty*

**Ultrasonic**  
*Graph Source: Roxar / Mimorax*

**Light Scattering**  
*Graph Source: Deckma*
Technologies Included in Gap Analysis

UV Fluorescence
Graph Source: Turner Design Hydrocarbon Instruments
Gap Analysis – Methodology

- Current status vs requirements
- Current status vs TRLs
- Current status on specific technical aspects
Gap Analysis – Methodology

- Based on three elements
  - How well does a technology meet the requirements? (A)
  - How well is a technology placed in the API 17N TRL table? (B)
  - How well is a vendor prepared to develop a subsea sensor? (C)

- Each of the elements given a weighting factor, respectively X, Y, Z (initial values suggested X = 55%; Y = 10%; Z = 35%)

- Ranking point calculation
  - R = AX + BY + CZ (or R = 0.55A + 0.1Y + 0.35Z)
Gap Analysis – Results

- **Little or no gaps in**
  - Oil concentration range
  - Oil density coverage
  - Salinity coverage
  - Response time

- **Substantial gaps (with an exception Digitrol)**
  - MTBF
  - Marinization
  - Environmental tests
  - System integration
Gap Analysis – Results

- **Small or no gaps in**
  - Temperature (design and operating)
  - Maximum velocity
  - Ability to deal with chemicals

- **Bigger gaps**
  - Pressure (design and operating, internal and external)

- On the accuracy requirement, few have completed and published any comparison between the sensors to the EPA 1664 method.
<table>
<thead>
<tr>
<th>Sensor Technical Requirements</th>
<th>Weighing factor</th>
<th>Technology</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Advanced Sensors (LIF)</td>
<td>ProAnalysis (LIF)</td>
</tr>
<tr>
<td>Oil density 20 – 35 (60)°API</td>
<td>4%</td>
<td>No issue</td>
</tr>
<tr>
<td>Salinity Coverage 0 – 280,000 ppm</td>
<td>4%</td>
<td>No issue</td>
</tr>
<tr>
<td>Max. Velocity Up to 15 (ft/s)</td>
<td>4%</td>
<td>33 (inline)</td>
</tr>
<tr>
<td>Sea water Temp. 33 (°F)</td>
<td>4%</td>
<td>Ambient (-4 to 131)</td>
</tr>
<tr>
<td>Design Temp 33-300 (°F)</td>
<td>4%</td>
<td>392 (Max)</td>
</tr>
<tr>
<td>Design Pres 10,000 (psig)</td>
<td>4%</td>
<td>2610 (Optional EX1000P)</td>
</tr>
<tr>
<td>Water Depth Up to 10,000 (ft)</td>
<td>4%</td>
<td>N/A</td>
</tr>
<tr>
<td>Operating Temp 38-200 (°F)</td>
<td>4%</td>
<td>392 (Max)</td>
</tr>
<tr>
<td>Operating Pres 6-5000 (psig)</td>
<td>4%</td>
<td>2610 (Optional EX1000P)</td>
</tr>
<tr>
<td>Oil conc. 0-100 mg/l</td>
<td>4%</td>
<td>0-20,000</td>
</tr>
<tr>
<td>Accuracy ±10% to EPA 1664 Method</td>
<td>4%</td>
<td>Yes, but ±1% [EX1000P]</td>
</tr>
<tr>
<td>Response time Hourly or faster</td>
<td>4%</td>
<td>1 sec</td>
</tr>
<tr>
<td>Design Service Life: 20 years</td>
<td>4%</td>
<td>Yes?</td>
</tr>
<tr>
<td>MTBF Min: 5 years</td>
<td>10%</td>
<td>N/A</td>
</tr>
<tr>
<td>Fouling Mitigation</td>
<td>20%</td>
<td>Ultrasonic</td>
</tr>
<tr>
<td>Ability to deal with chemicals</td>
<td>4%</td>
<td>Yes, with Spectrometry</td>
</tr>
<tr>
<td>Marinization</td>
<td>4%</td>
<td>Not yet</td>
</tr>
<tr>
<td>Environmental Tests</td>
<td>4%</td>
<td>Not for subsa PED 97/23/EC</td>
</tr>
<tr>
<td>System Integration</td>
<td>4%</td>
<td>No for subsa</td>
</tr>
<tr>
<td>Sensor Technical Requirements</td>
<td>Weighing factor</td>
<td>Technology</td>
</tr>
<tr>
<td>-------------------------------</td>
<td>----------------</td>
<td>----------------------------</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Advanced Sensors (LIF)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ProAnalysis (LIF)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Canty (Microscopy)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Jorin (Microscopy)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Mirmorax (Untrasound)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Digitrol (L Scattering)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Turner Design (Conv. UV)</td>
</tr>
<tr>
<td>Fluid properties</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Oil density</td>
<td>20 – 35 (60)° API</td>
<td>10</td>
</tr>
<tr>
<td>Saltiness Coverage</td>
<td>0 - 250,000 ppm</td>
<td>10</td>
</tr>
<tr>
<td>Max. Velocity</td>
<td>Up to 15 (ft/s)</td>
<td>10</td>
</tr>
<tr>
<td>Sea water</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Temp. 33 (°F)</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Design Temp</td>
<td>33-300 (°F)</td>
<td>10</td>
</tr>
<tr>
<td>Design Pres</td>
<td>10,000 (psig)</td>
<td>10</td>
</tr>
<tr>
<td>Water Depth</td>
<td>Up to 10,000 (ft)</td>
<td>7</td>
</tr>
<tr>
<td>Operating Temp</td>
<td>38-200 (°F)</td>
<td>7</td>
</tr>
<tr>
<td>Operating Pres</td>
<td>0-5000 (psig)</td>
<td>7</td>
</tr>
<tr>
<td>Oil conc.</td>
<td>0-100 mg/l</td>
<td>10</td>
</tr>
<tr>
<td>Accuracy</td>
<td>±10% to EPA 1664</td>
<td>10</td>
</tr>
<tr>
<td>Response time</td>
<td>Hourly or faster</td>
<td>10</td>
</tr>
<tr>
<td>Design Service Life</td>
<td>20 years</td>
<td>10</td>
</tr>
<tr>
<td>MTBF</td>
<td>5 years</td>
<td>10</td>
</tr>
<tr>
<td>Fouling Mitigation</td>
<td>20%</td>
<td>10</td>
</tr>
<tr>
<td>Ability to deal with chemicals</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Marination</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Environmental Texts</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>System Integration</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Overall A value</td>
<td>5.06</td>
<td>4.81</td>
</tr>
</tbody>
</table>

### Sensor Technical Requirements

<table>
<thead>
<tr>
<th>Sensor Technical Requirements</th>
<th>Weighing factor</th>
<th>Technology</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Advanced Sensors (LIF)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ProAnalysis (LIF)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Canty (Microscopy)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Jorin (Microscopy)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Mirmorax (Untrasound)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Digitrol (L Scattering)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Turner Design (Conv. UV)</td>
</tr>
<tr>
<td>Fluid properties</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Oil density</td>
<td>20 – 35 (60)° API</td>
<td>10</td>
</tr>
<tr>
<td>Saltiness Coverage</td>
<td>0 - 250,000 ppm</td>
<td>10</td>
</tr>
<tr>
<td>Max. Velocity</td>
<td>Up to 15 (ft/s)</td>
<td>10</td>
</tr>
<tr>
<td>Sea water</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Temp. 33 (°F)</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Design Temp</td>
<td>33-300 (°F)</td>
<td>10</td>
</tr>
<tr>
<td>Design Pres</td>
<td>10,000 (psig)</td>
<td>10</td>
</tr>
<tr>
<td>Water Depth</td>
<td>Up to 10,000 (ft)</td>
<td>7</td>
</tr>
<tr>
<td>Operating Temp</td>
<td>38-200 (°F)</td>
<td>7</td>
</tr>
<tr>
<td>Operating Pres</td>
<td>0-5000 (psig)</td>
<td>7</td>
</tr>
<tr>
<td>Oil conc.</td>
<td>0-100 mg/l</td>
<td>10</td>
</tr>
<tr>
<td>Accuracy</td>
<td>±10% to EPA 1664</td>
<td>10</td>
</tr>
<tr>
<td>Response time</td>
<td>Hourly or faster</td>
<td>10</td>
</tr>
<tr>
<td>Design Service Life</td>
<td>20 years</td>
<td>10</td>
</tr>
<tr>
<td>MTBF</td>
<td>5 years</td>
<td>10</td>
</tr>
<tr>
<td>Fouling Mitigation</td>
<td>20%</td>
<td>10</td>
</tr>
<tr>
<td>Ability to deal with chemicals</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Marination</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Environmental Texts</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>System Integration</td>
<td>4%</td>
<td>10</td>
</tr>
<tr>
<td>Overall A value</td>
<td>5.06</td>
<td>4.81</td>
</tr>
</tbody>
</table>
## Technology Readiness Level

<table>
<thead>
<tr>
<th>Sensor Technical Requirements</th>
<th>Technology Readiness Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>Advanced Sensors</td>
<td>ProAnalyis (Microscopy)</td>
</tr>
<tr>
<td>Canty (Microscopy)</td>
<td>Jorin (Microscopy)</td>
</tr>
<tr>
<td>Mirmorax (Untrasound)</td>
<td>Digitrol (L Scattering)</td>
</tr>
<tr>
<td>Turner Design (Conv. UV)</td>
<td></td>
</tr>
<tr>
<td>TRL</td>
<td>3</td>
</tr>
<tr>
<td>Overall B Value</td>
<td>7</td>
</tr>
</tbody>
</table>

TRL level referring to general subsea application, for comparison of sensors only. No sensor has reached TRL 3 for subsea produced water discharge application.
## Assessment of Vendors

<table>
<thead>
<tr>
<th>Parameters (Further considerations)</th>
<th>Weighing Factor</th>
<th>Advanced Sensors</th>
<th>ProAnalysis (Microscopy)</th>
<th>Canty (Microscopy)</th>
<th>Jorin (Microscopy)</th>
<th>Mirmorax (Untrasound)</th>
<th>Digitrol (L Scattering)</th>
<th>Turner Design (Conv. UV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Technology suitability/potential for subsea</td>
<td>30%</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>4</td>
<td>7</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td>Work done so far towards subsea</td>
<td>20%</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>7</td>
<td>10</td>
<td>10</td>
<td>4</td>
</tr>
<tr>
<td>Commitment to develop a subsea sensor</td>
<td>20%</td>
<td>7</td>
<td>10</td>
<td>10</td>
<td>4</td>
<td>4</td>
<td>10</td>
<td>4</td>
</tr>
<tr>
<td>Experience with Surface applications</td>
<td>15%</td>
<td>10</td>
<td>10</td>
<td>7</td>
<td>10</td>
<td>4</td>
<td>4</td>
<td>10</td>
</tr>
<tr>
<td>Involvement in other subsea OIW sensor development project</td>
<td>15%</td>
<td>10</td>
<td>7</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>1</td>
</tr>
<tr>
<td>Overall C Value</td>
<td></td>
<td>9.4</td>
<td>9.55</td>
<td>9.55</td>
<td>8.2</td>
<td>6.1</td>
<td>8.2</td>
<td>4.45</td>
</tr>
</tbody>
</table>

Parameters (Further considerations):

- **Technology suitability/potential for subsea**: Evaluates the vendor's capability to develop technology suitable for subsea applications, including potential for future developments.
- **Work done so far towards subsea**: Measures the vendor's progress in subsea technology development.
- **Commitment to develop a subsea sensor**: Assesses the vendor's dedication to subsea sensor research.
- **Experience with Surface applications**: Determines the vendor's experience in surface applications, which could be relevant for subsea technology transfer.
- **Involvement in other subsea OIW sensor development project**: Indicates the vendor's participation in other subsea projects, possibly providing insights into their expertise and resources.

*Weights:* 30% for Technology suitability/potential for subsea, 20% for Work done so far towards subsea, 20% for Commitment to develop a subsea sensor, 15% for Experience with Surface applications, and 15% for Involvement in other subsea OIW sensor development project.

*Overall C Value:* A composite score calculated from the individual parameter evaluations, helping to prioritize vendors based on their overall suitability for subsea applications.
## Assessment of Vendors

<table>
<thead>
<tr>
<th>Parameters (Further considerations)</th>
<th>Weighing Factor</th>
<th>Vendors</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Advanced Sensors</td>
<td>ProAnalysis (Microscopy)</td>
</tr>
<tr>
<td>Technology suitability/ potential for subsea</td>
<td>30%</td>
<td>10</td>
</tr>
<tr>
<td>Work done so far towards subsea</td>
<td>20%</td>
<td>10</td>
</tr>
<tr>
<td>Commitment to develop a subsea sensor</td>
<td>20%</td>
<td>7</td>
</tr>
<tr>
<td>Experience with Surface applications</td>
<td>15%</td>
<td>10</td>
</tr>
<tr>
<td>Involvement in other subsea OIW sensor development project</td>
<td>15%</td>
<td>10</td>
</tr>
<tr>
<td>Overall C Value</td>
<td>9.4</td>
<td>9.55</td>
</tr>
</tbody>
</table>
Overall Ranking

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Weighing Factor</th>
<th>Advanced Sensors (LIF)</th>
<th>ProAnalysis (LIF)</th>
<th>Canty (Microscopy)</th>
<th>Jorin (Microscopy)</th>
<th>Mirmorax (Untrasound)</th>
<th>Digitrol (L Scattering)</th>
<th>Turner Design (Conv. UV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Value A</td>
<td>55%</td>
<td>5.06</td>
<td>4.81</td>
<td>6.40</td>
<td>5.06</td>
<td>4.94</td>
<td>7.44</td>
<td>3.72</td>
</tr>
<tr>
<td>Value B</td>
<td>10%</td>
<td>7</td>
<td>7</td>
<td>7</td>
<td>7</td>
<td>7</td>
<td>10</td>
<td>4</td>
</tr>
<tr>
<td>Value C</td>
<td>35%</td>
<td>9.4</td>
<td>9.55</td>
<td>9.55</td>
<td>8.2</td>
<td>6.1</td>
<td>8.2</td>
<td>4.45</td>
</tr>
<tr>
<td>TOTAL</td>
<td></td>
<td>6.77</td>
<td>6.69</td>
<td>7.56</td>
<td>6.35</td>
<td>5.55</td>
<td>7.96</td>
<td>4.00</td>
</tr>
</tbody>
</table>

Ranking results are not significantly sensitive to weighting factors
Outline

- Overview of RPSEA 12121-6301-03 Project
- Subsea Produced Water Sensor Requirements
- Technology Gap Analysis and Ranking
- **Proof of Concept on Confocal Laser Fluorescence Microscopy**
- Plan for Phase 2
- Forecast and Actual Cost
Objectives for the Technology

- **Can Be Used as a Substitute of Lab Measurements (EPA 1664 etc) for Regulatory Compliance Reporting**
  - When frequent sampling for regulatory compliance reporting is not feasible or economical

- **High Accuracy Oil in Water Concentration Measurement**
  - Accuracy in 10 – 50 mg/L range
    - High resolution (0.3 micron) and 3-D capabilities
    - Laboratory tests successful using water from several to several hundred ppm
  - Accuracy for water with up to several percent of oil
    - 3-D capability enables accurate accounting of oil droplets behind other droplets

- **Online Application**
  - Especially subsea and other difficult to access installations

- **Free of Calibration when New Reservoir Is Added or When Processing Conditions Change**
Measurement Method

Scanning Disk Confocal

OR

Point Scanning Confocal

Graph Source: Olympus
CLFM Subsea PWD Sensor Schematic
Study on Oil Droplet Distribution

- **Onshore Field Produced Water Used**
  - “Medium” – From tank between 1st stage separator and discharge
  - “Clean” – From tank for discharge

- **3-D Imaged and Analyzed**

### Total Volume Probability of Droplets

<table>
<thead>
<tr>
<th>Water</th>
<th>Volume Averaged Size (microns)</th>
<th>Up to 1.0 Micron</th>
<th>Up to 1.6 Microns</th>
<th>Up to 2.0 Microns</th>
<th>Up to 4.0 Microns</th>
</tr>
</thead>
<tbody>
<tr>
<td>Medium</td>
<td>11.9</td>
<td>0.5%</td>
<td>2.2%</td>
<td>4.0%</td>
<td>18.7%</td>
</tr>
<tr>
<td>Clean</td>
<td>8.7</td>
<td>1.5%</td>
<td>4.1%</td>
<td>6.8%</td>
<td>25.5%</td>
</tr>
</tbody>
</table>
Notes:
Approx. 100 mg/L oil in water
Flow channel depth 0.8 mm. ibidi slide with Luer connectors.
10X dry objective.
Elongation of droplet due to refractive index mismatch. Can be restored in image processing.
Imaging Solids & Droplets Behind Others

Confocal Laser Fluorescence Image

Differential Interference Contrast Image

Cross Section View

3-D View
Laboratory imaging tests show that, for the measurements planned, the imaging is not sensitive to normal vibration.

Note: The elongations and other non-droplet like features shown in images are due to refractive index mismatch and other optical characteristics, which can be eliminated by image processing.
EPA 1664 Method – Definition of Oil and Grease

- Materials that are extractable by *n*-hexane, not evaporated at 70°C

- EPA's method 1664A:
  - A liter of water is acidified to pH<2 and extracted using 3 volumes of *n*-hexane
  - The extracts are combined, dried, and distilled at 85°C

- Method 1664A accounts for:
  - Free oil: large droplets that can be removed by gravity separation methods
  - Dispersed oil: small droplets

- Method 1664 does not account for dissolved oil (and other similar material)
1664 Procedure

1. Sample
2. Acidification
3. Extraction
4. Distillation
5. HEM
1. Sample

- 950 mL nanopure water in brown glass bottle with PTFE lined cap
- Add known amount of tridecane using glass syringe with PTFE plunger
2. Acidification

- Test pH of sample using glass stir rod and pH paper
- Add 6N HCl until sample pH ≤ 2 (~2.5 mL)
- Mix thoroughly by shaking/inverting bottle
- Test pH of acidified sample using same glass stir rod and new pH paper
3. Extraction

- Pour sample into separatory funnel
- Add 10 mL hexane to sample bottle and shake vigorously (repeat 3x and pour hexane into separatory funnel after each 10 mL)
- Vigorously shake separatory funnel
- Allow to separate for $\geq$ 10 min
- Drain water into original sample bottle
- Drain organic layer through 10 g sodium sulfate and filter paper into boiling flask
- Repeat 3x
4. Distillation

- Connect boiling flask to distillation head using keck clamp and submerge in water bath (~85–90°C)
- Hexane vapor condenses by contact with condenser and ice bath, drips down adapter into collection flask
- Remove boiling flask when 2–3 mL HEM remains and store in desiccator
5. Hexane Extractable Material (HEM)

- HEM remains in boiling flask
- Record mass of boiling flask \( \geq 30 \text{ min} \) until difference in measurements is 4% or 0.5mg (whichever is smaller)
- Boiling flask with HEM is stored in desiccator except when measuring mass
Initial Precision and Recovery (IPR)

- 4 samples: precision and recovery (PAR) standard
  - Add 10mL spiking solution to 950 mL nanopure water
  - Spiking solution: 200mg stearic acid + 200mg hexadecane dissolved in 100mL acetone
- Average recovery ($x$) must be within 83-101%
- Standard deviation ($s$) must be <11%
Example with different oil concentrations
EPA 1664 Method – Distillation

Distillation set-up

Crude oil in hexane during distillation

HEM

HEM
Confocal Experimental Procedure

Use disperser mix for 3 min at 1000 rpm

2 mL of sample injected into flow cell

The sample was observed under confocal microscope 10X magnification

Stack was taken in 3 different spots of the flow cell and images were analyzed through matlab. Experiment repeated 3x

Data generated in matlab was copied and average and Standard Dev. were calculated with Excel
### Confocal Experiment Settings

#### Microscope Setting (only for the experiments to compare with EPA 1664)

<table>
<thead>
<tr>
<th>Setting</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Z Step Size</td>
<td>4 µm</td>
</tr>
<tr>
<td>Z Range</td>
<td>500 µm</td>
</tr>
<tr>
<td>Frame Accumulation</td>
<td>1</td>
</tr>
<tr>
<td>Frame Average</td>
<td>1</td>
</tr>
<tr>
<td>Number of Stacks</td>
<td>125</td>
</tr>
</tbody>
</table>
Matlab Analysis

- Calculate the volume of bright pixel
- Convert into mass of oil
- Calculate the volume of whole stack
- Calculate the oil concentration (m/V)

Grey Image  Binary Image  Calculate Concentration

Original Image  Matlab analyzed Image  3D stack
Comparison – Confocal vs EPA 1664 (1)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Prepared Conc. (mg/L)</th>
<th>EPA Method 1664</th>
<th>Confocal Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Measured Conc. (mg/L)</td>
<td>Standard Dev.</td>
</tr>
<tr>
<td>Synthetic</td>
<td>28.67</td>
<td>37.70</td>
<td>13.7</td>
</tr>
<tr>
<td>Synthetic</td>
<td>47.79</td>
<td>39.40</td>
<td>6.01</td>
</tr>
</tbody>
</table>
3 Field Produced Water Samples

“Clean”  Medium  “Dirty”
Confocal vs 1664 for Produced Water

<table>
<thead>
<tr>
<th>Produced Water Sample</th>
<th>EPA Method 1664</th>
<th>Confocal Analyses</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Measured Conc (mg/L)</td>
<td>Standard Deviation</td>
</tr>
<tr>
<td>“Clean”</td>
<td>6.56</td>
<td>0.78</td>
</tr>
<tr>
<td>“Medium”</td>
<td>28.53</td>
<td>0.28</td>
</tr>
<tr>
<td>“Dirty”</td>
<td>86.8</td>
<td>17.09</td>
</tr>
</tbody>
</table>
Conclusions from Produced Water Experiments

- The EPA method and the confocal method obtained similar results overall.
- Higher standard deviation for the “clean” and “medium” sample in confocal than 1664.
  - The results indicate that additional imaging stacks (currently 3-9 per sample) are needed for water with lower oil concentration.
- “Dirty” sample challenging to both methods as evidenced by higher standard deviation.
Initial Jet Cleaning Tests – Configuration

Jet Fluid Reservoir

Produced Water Reservoir

Valve for Switching Flow Direction

Jetting Fluid Tube

Jet Nozzles

Measurement Section (for jetting tests only)
Test04221501 – 4.5 Hours “Dirty Water”

- Beginning of Test
- 2 Hour Flowing
- 4.5 Hours Flowing
- After Jetting

Oil droplets
Air bubbles

Video
## Factors for Subsea Feasibility

<table>
<thead>
<tr>
<th>Factors</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>Functional Performances in Subsea</td>
<td>Conceptual design and initial laboratory testing indicate feasibility to provide 1 reading every 2 minutes, and the stability of the statistics after several readings.</td>
</tr>
<tr>
<td>• Imaging operation</td>
<td></td>
</tr>
<tr>
<td>• Image processing</td>
<td></td>
</tr>
<tr>
<td>Interfaces with Subsea System</td>
<td>Interfaces with subsea system (hardware, control system and chemical system) identified and considered feasible.</td>
</tr>
<tr>
<td>Constructability, Installability and Retrievability</td>
<td>• All key components except for objectives identified to be off-shelf products. Objective vendors can do custom design to extend the cover glass thickness correction to the 10,000/15,000 psi requirement.</td>
</tr>
<tr>
<td></td>
<td>• Preliminary dimension and weight developed and are considered well within offshore equipment limits</td>
</tr>
<tr>
<td></td>
<td>• Shock and vibration are conceptually considered as manageable and will be further developed during the next phase</td>
</tr>
<tr>
<td>Maintenance</td>
<td>Maintenance requirements are typical of normal subsea system operations.</td>
</tr>
<tr>
<td>Reliability</td>
<td>Reliability of each component considered. Conceptual design indicates ability to meet the 5-year MTBF requirement.</td>
</tr>
</tbody>
</table>
The Study Has Shown the Feasibility and Advantages of CLFM as a Subsea Produced Water Discharge Quality Sensor

- Confirmed capabilities as subsea PWD sensor
- Correlates well with EPA 1664 measurements
- Subsea conceptual design indicates suitability
- Potential advantages over existing sensor on accuracy for PWD applications

Future Work

- Prototype designs, construction, bench-scale testing
Outline

- Overview of RPSEA 12121-6301-03 Project
- Subsea Produced Water Sensor Requirements
- Technology Gap Analysis and Ranking
- Proof of Concept on Confocal Laser Fluorescence Microscopy
- Plan for Phase 2
- Forecast and Actual Cost
Plan for Phase 2

- **Prototypes**
  - Four sensor prototypes will be designed, constructed and tested
    - 3 of the top existing technologies, 1 new technology
  - Existing technologies
    - Top four ranked vendors contacted for proposals
    - Technology ranking as key factor of selection
    - 3 sensors to be tested (light scattering, imaging, laser induced fluorescence)
  - New technology
    - Confocal Laser Fluorescence Microscopy

- **Bench-Scale Testing**
  - 3 proposals received; short list in June 2015; selection in Oct 2015
  - Focus on performance testing
  - Possible inclusion of sensor cleaning at subsea pressure
Outline

- Overview of RPSEA 12121-6301-03 Project
- Subsea Produced Water Sensor Requirements
- Technology Gap Analysis and Ranking
- Proof of Concept on Confocal Laser Fluorescence Microscopy
- Plan for Phase 2
- Forecast and Actual Cost
## Forecast Costs

<table>
<thead>
<tr>
<th>PHASE 1</th>
<th>PHASE 2</th>
<th>TOTAL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Federal (RPSEA) Cost</td>
<td>$791,530</td>
<td>$2,872,008</td>
</tr>
<tr>
<td>Cost Share</td>
<td>$197,910</td>
<td>$721,830</td>
</tr>
<tr>
<td><strong>Total Cost</strong></td>
<td><strong>$989,440</strong></td>
<td><strong>$3,593,839</strong></td>
</tr>
</tbody>
</table>

| Duration (months) | 9                | 15               | 24               |

- **Technology Transfer Costs**
  - Project specific (1.5%): $68,749
  - Program (1%): $45,833
Planned and Actual Cost – Total
Planned and Actual Cost Share
Planned and Actual Cost – Technology Transfer
Contacts

Principal Investigator:
Jeff Zhang
Clearview Subsea LLC
jzhang@clearviewsubsea.com
832-528-7133

Project Manager:
Bill Fincham
NETL
William.fincham@netl.doe.gov
304-285-4268

Technical Coordinator:
James Pappas
RPSEA
jpappas@rpsea.org
281-690-5511
Thank You!