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## **Final report**

# Subsurface oil releases – Verification of dispersant effectiveness under high pressure

A scaled experimental approach using the SINTEF Tower Basin and SwRIs 90" high pressure chamber.

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The instrumentation being lowered into the SINTEF Tower Basin for initial testing and comparison at ambient conditions (5 meters depth).

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## $SwRI^{\circ}$



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#### ABSTRACT

The main objective with this project was to study possible pressure dependency of droplet formation in case of a subsea blow out of oil and the effectiveness of subsea dispersant injection (SSDI).

The droplet sizes documented by the SINTEF Silhouette Camera from comparable experiments (nozzle, oil type, flow rates, injection techniques and dispersant product) at ambient conditions (5 meters depth) and high pressure conditions (175 bar or 1750 meters depth) show no significant difference in droplet sizes as a function of pressure.

This lack of a pressure effect was observed for both formation of large droplets from untreated oil and formation of smaller droplets by dispersant injection (1 and 2% dispersant dosage). This strongly indicates that SSDI effectiveness is not significantly influenced by hydrostatic pressure.

These experiments were performed using stabilized dead oil without gas. Experiments with recombined oil & natural gas ("live oil") were performed in a study later in 2015 (Brandvik et al., 2016b).

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#### 1 Introduction

SINTEF has during 2012-16 performed several projects for the American Petroleum Institute (API) regarding subsea oil and gas releases and the effectiveness of dispersant injection. These projects have been a part of a Joint Industry Task Force organised by API. An overview of the projects within SSDI effectiveness within this JITF is given in Brandvik et al., 2016a. SINTEFs projects within this JITF have focused on initial droplet formation versus release conditions and the effectiveness of subsea dispersant injection as a function of dispersant type (Brandvik et al., 2014), dispersant dosage and oil properties (Brandvik et al., 2015). The main part of this work is performed in SINTEFs Tower basin in Trondheim, which is 6 meters high, 3 meters wide and holds 42 m<sup>3</sup> of natural sea water. A description of the Tower basin is given in Appendix B and in Brandvik et al., 2013b.

The study described in this report focus on droplet formation and dispersant effectiveness as a function of increasing water depth and hydrostatic pressure. South West Research Institute (SwRI) in San Antonio, TX, US has very suitable hyperbaric chambers for such studies. The largest has dimensions which are comparable to the SINTEF Tower Basin (75% of the volume). A description of the facilities at SwRI and the experimental procedures used are given in Appendix A.

After discussions between API, SwRI and SINTEF, during initialisation of this project, the following research activities were given priority:

#### API D3 Phase-III: Studying droplet formation at high pressure

Replicate experiments done at SINTEF API Phase-I and II. The most important parameters were:

- One oil type (Oseberg blend) and dispersant (Corexit 9500)
- One nozzle diameter
- Two pressures (Ambient and high pressure (2500 PSI or 172 bar))
- With and without dispersant injection
- Two different dosages of dispersant

#### API D3 Phase-V: Studying the effect of live-oil and natural gas at high pressure

Replicate some of the experiments done during SINTEF API Phase-III adding experiments to study the effect of combined releases (oil & gas) and "live oil". The most important parameters will be:

- Combined releases of oil and natural gas (NG)
- Release recombined oil and gas "Live-oil"

This report describes the experimental work from Phase-III. Initial work was performed at SINTEF and at SwRI in January 2014 (described in Appendix C). Additional work with new sensors for quantifying droplet sizes (SINTEF Silhouette camera) was performed in January - March 2015 and this work is the main focus for this report.

The work with combined releases of oil and gas and with "live-oil" (Phase-V) was performed at SINTEF in August-September 2015 and at SwRI in October 2015 and is described in Brandvik et al., 2016b.



#### 2 Objectives

Perform experiments in a hyperbaric chamber at SwRI to verify the main conclusions from API D3 subsurface effectiveness project (Phase-I and II) performed at SINTEF with aim of determining if deep water pressure affects droplet formation when dispersant are applied subsea at the wellhead. Specifically:

- Oil droplet formation and
- Oil droplet formation at different dosages of dispersant



#### 3 Experimental

The experimental part of this project has been performed in two periods. Initial work was done at both SINTEF (ambient conditions) and at SwRI facilities in San Antonio, Texas (ambient and HP conditions) in 2014. This work is described in Appendix C.

The results from this initial work were not conclusive and additional work was needed. This work was started at SINTEF in January 2015 and compared different instrument platforms (LISST-DEEP, LISST 100X and the new SINTEF Silhouette camera). This work verified the earlier hypothesis that the design of the path reduction module (PRM) of the LISST DEEP caused discrimination of larger oil droplets. However, the development of a high pressure version of the new Silhouette Camera (SilCam) made it possible to perform new high pressure experiments at SwRI in 2015. These experiments are the main focus for this report.

The high pressure (HP) testing at SwRI simulates deep water conditions at approximate 1700 meters depth and was performed at 2500 psi or 172 bars. It was conducted in a 90-inch (2.29 meter) inside diameter by 230-inch (5.84 meter) deep pressure chamber at SwRI. This chamber is rated for 4000 psi (275 bar) and can be cooled to maintain an inside water temperature of approximately 40 degrees Fahrenheit (4 degrees Celsius). A description of the SwRI 90" hyperbaric chamber modified for these experiments (oil release, salt water mixing, dispersant injection and droplet monitoring) is given in Appendix A, and a similar description of the SINTEF Tower Basin in Appendix B.

#### 3.1 Experimental work at SwRI February-March 2015

The following experimental design was selected for the experiments at SwRI. The experiments were performed in the period from 21. February to 6. March 2015.

Table 3.1:Experimental matrix for the second round of work at SwRI Hyperbaric chamber.<br/>Ambient experiment is performed with all three instruments (LISST-DEEP, LISST-<br/>100X and a Silhouette Camera). The LISTT 100X is only rated for 300 m and cannot<br/>be used for the high pressure experiment.

| Test<br>Tank<br>Set | Medium<br>Nozzle<br>(1.5 mm) | Pressure | Instrumentation  | Flow<br>rate<br>(L/min) | Oil<br>alone | Low<br>DOR<br>(1:100) | High<br>DOR<br>(1:50) |
|---------------------|------------------------------|----------|------------------|-------------------------|--------------|-----------------------|-----------------------|
|                     | Х                            | Amb      | DEEP/100X/SilCam | 1.2                     | Х            |                       |                       |
| 1                   | Х                            | Amb      | DEEP/100X/SilCam | 1.2                     |              | Х                     |                       |
|                     | Х                            | Amb      | DEEP/100X/SilCam | 1.2                     |              |                       | Х                     |
|                     | Х                            | HP       | DEEP/SilCam      | 1.2                     | Х            |                       |                       |
| 2                   | Х                            | HP       | DEEP/SilCam      | 1.2                     |              | Х                     |                       |
|                     | Х                            | HP       | DEEP/SilCam      | 1.2                     |              |                       | Х                     |

The Silhouette camera (SilCam) was the only instrumentation able to generate data for all experimental conditions used in this project. The LISST 100X could not be used during the HP experiments at SwRI. The LISST DEEP did not generate reliable data due to saturation (very low light transmittance, due to high oil concentration and small droplets) for many of the experimental settings. The LISST DEEP was operated without the path reduction module due to possible

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discrimination of large droplets (see Figure 4.3). For this reason the data obtained with the SilCam are the main focus of this report.

#### 3.2 Selection of oil type

To replicate the work previously performed in SINTEF Tower Basin in API D3 JITS Phase I and II the same oil was used during the work at SwRI (Oseberg blend). The oil was delivered to SINTEF from the oil terminal at Sture and shipped to San Antonio. One barrel of Oseberg blend was shipped in 2014 and another in 2015 to SwRI from SINTEF for this project (ID: 2013-0439 and 2015-0014). No significant differences in chemical composition were detected between these two batches.

|                                 | Macondo<br>MC252 | Oseberg<br>blend 2014<br>(2013-0439) | Oseberg<br>blend 2015<br>(2015-0014) |
|---------------------------------|------------------|--------------------------------------|--------------------------------------|
| Specific gravity (kg/l)         | 0.833            | 0.828                                | 0.826                                |
| Pour Point (°C)                 | -27              | -36                                  | -36                                  |
| Viscosity (mPas at 40°C)        | 4                | 3.0                                  | 2.7                                  |
| Asphaltene (wt%)                | 0.2              | 0.2                                  | 0.2                                  |
| Waxes (wt%)                     | 1.6              | 2.5                                  | 2.3                                  |
| 150°C – Evaporative loss (vol%) | 27               | 22                                   | $22^*$                               |
| 200°C – Evaporative loss (vol%) | 39               | 34                                   | 34*                                  |
| 250°C – Evaporative loss (vol%) | 50               | 45                                   | $45^{*}$                             |

Table 3.2: Properties of MC252 oil and Oseberg blend.

<sup>\*</sup> True boiling point curve or weathering data was not available, but based on the density the differences from earlier batches in evaporative loss are not expected to be large.

#### **3.3** Selection of dispersants

Corexit 9500 was selected for this study since it has been the main dispersant in earlier API studies at SINTEF (Brandvik et al., 2014 and Brandvik et al., 2015). The dispersant was delivered directly from Nalco to SwRI in 2014 and used in as received.

#### 3.4 Dispersant injection

Two injection methods for dispersant were selected for this project, Upstream injection and Simulated insertion tool. The release and injection arrangements were made similar to what have been used at SINTEF for earlier API studies. Both the injection nozzle and dispersant mixing T were delivered by SINTEF. With simulated injection tool (SIT) dispersant was injected 6 release diameters (1.5 x 6 = 9 mm) before the oil release opening. With upstream injection, dispersant were mixed into the oil line 2000 nozzle diameter before the oil release opening. Further details are given in Appendix A.

#### 3.5 IFT analysis - Spinning drop method

For the interfacial tension measurements by spinning drop method (Khehifa and So, 2009), the Dataphysics Spinning Drop Tensiometer SVT-20N with control and calculation software SVTS 20 IFT was used. The Julabo F12-ED Refrigerated and Heating Circulator were used for temperature control. Disposable 1ml plastic syringes were used to inject the oil sample into the SVT 20N capillary tube. Prior to each measurement, the capillary tube was rinsed three times with dichloromethane (DCM), acetone and deionized water, dried with nitrogen gas, and then rinsed

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three times with the sea-water. The capillary was carefully filled with sea-water (outer phase liquid) to ensure the absence of air bubbles. Depending on the oil sample, the capillary may be stationary or rotating when the drop of oil is injected and the rotation speed may also vary. Measurements of IFT were taken as soon as the drop elongation was stable.

#### 3.5.1 Oil sampling for IFT measurements

During different injection sequences of dispersants into the oil, oil/water samples were taken from 0.8 meters above the nozzle after 60 seconds of each dispersant injection. Oil/water samples were collected in 1 litre long necked measuring flasks. Upon collection, oil appeared as droplets in seawater, with droplet sizes dependant on DOR and method of application. Oil settled as a layer in the narrow neck of the bottle and was collected for IFT measurements after 24 hours. The settling time was important for collecting the smaller droplets in experiments with high dispersant effectiveness. The collected oil samples were stored in a dry and cool place overnight. No homogenization or heating was done before the IFT measurements. All IFTs were measured at 13°C.

During the high pressure experiments the oil-water sample had to be depressurized (from 172 bar to ambient). The mixing of oil and water during this pressure drop produced in some cases very stable water-in-oil emulsions which complicated the IFT measurements.



#### 4 Results

#### 4.1 Overview of experiments

Table 4.1: Overview of experiments performed at SwRI in February-March 2015.

|                 |                      | Type of experiment |                    |  |  |                  |  |  |
|-----------------|----------------------|--------------------|--------------------|--|--|------------------|--|--|
| Exp. Date<br>no |                      | Rate<br>(L/min)    | Type<br>experiment | Disp<br>GOR<br>Injection                         | Comments   | Water<br>samples |  |  |
| 1               | <u>270215</u>        | 1.2                | Ambient            | 1:100-50<br>Simulated<br>Injection<br>Tool (SIT) | Problems due to salt/density<br>gradients in tank. Only the largest<br>droplets penetrated through the<br>density layers and reached the<br>instrumentation. | 0                |  |  |
| 2               | <mark>020</mark> 315 | 1.2                | Ambient            | 1:100-50<br>Simulated<br>Injection<br>Tool (SIT) | Problems with dispersant injection.  | 0                |  |  |
| 3               | 040315               | 1.2                | High Pressure      | 1:100-50<br>Simulated<br>Injection<br>Tool (SIT) | Successful experiments   | 7                |  |  |
| 4               | 060315               | 1.2                | Ambient            | 1:100-50<br>Simulated<br>Injection<br>Tool (SIT) | Successful experiments   | 7                |  |  |

Red: Not successful experiment Yellow: Partly successful experiment Green: Completely successful experiment

#### **Type of experiment:**

Ambient pressure – Lid on, no pressurization High Pressure – Tank pressurized to 2500 Psi or 172 bars

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Table 4.2:Interfacial tension (IFT) measurements of oil samples taken from the hyperbaric<br/>chamber during 2015 experiments. The water/oil sampling intervals are indicated, as<br/>coloured bars, in Figure 4.4 and Figure 4.6.

| Experiment 040615     | SINTEF ID | IFT    |
|-----------------------|-----------|--------|
| (High Pressure – SIT) |           | (mN/m) |
| 3-Oil alone-1         | 2015-0478 | 22,4   |
| 3-DOR100-1b           | 2015-0479 | 5,5    |
| 3-DOR50-1b            | 2015-0480 | 0,7    |
| 3-Oil alone-2         | 2015-0481 | 9,1    |
| 3-DOR100-2            | 2015-0482 | 0,5    |
| 3-DOR50-2             | 2015-0483 | 0,8    |
| 3-Oil alone-3         | 2015-0484 | 4,4    |
|                       |           |        |
| Experiment 060315     | SINTEF ID | IFT    |
| (Ambient3 – SIT)      |           | (mN/m) |
| 4-Oil alone-1         | 2015-0485 | 19,2   |
| 4-DOR100-1            | 2015-0486 | 0,8    |
| 4-DOR50-1             | 2015-0487 | 0,8    |
| 4-Oil alone-2         | 2015-0488 | 8,0    |
| 4-DOR100-2            | 2015-0489 | 0,8    |
| 4-DOR50-2             | 2015-0490 | 0,01   |
| 4-Oil alone-3         | 2015-0491 | 9,2    |

#### Sample name legends:

- "Oil alone": Untreated oil
- "DOR100": Treated with C9500 DOR 1:100 (1%)
- "DOR50": Treated with C9500 DOR 1:50 (2%)





#### 4.2 Comparison of different analytical platforms - SINTEF

During the initial work done at both SINTEF and SwRI in 2014 (Appendix C) the results were not conclusive. The main challenge was thought to be discrimination of large droplets caused by the path reduction module (PRM) in the LISST DEEP. This module was necessary to handle the high oil concentrations and small droplets used in these experiments.

This section presents data from testing of alternative instruments for measuring oil droplet sizes at high pressure. Initial testing was performed in the SINTEF Tower Basin to compare the performance of alternative instrumentation and verify the performance of the LISST DEEP with and without the PRM.

The three instruments (LISST-100X, LISST-DEEP and the Silhouette camera, see Figure 4.1) were tested simultaneously in the SINTEF Tower Basin, see Figure 4.2.



Figure 4.1: Illustrations of the instruments LISST-100X, LISST-DEEP and the Silhouette camera. The two first units are commercially available from Sequoia and used as delivered, the last unit is designed and constructed by SINTEF. The unit used is a HP version rated for 300 bars.

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Figure 4.2: Images showing the configuration of the instrument (LISST-100X, LISST-DEEP and the Silhouette camera) inside the SINTEF Tower Basin. Instruments are seen both from above (A) and from below (B). The first images show instrument configuration without oil (A1 & B1) and A2 & B2 show the instruments surrounded by the plume of dispersed oil. White arrows indicate positions of the instrument sample volumes.

Two experiments were performed in the Tower Basin to test the LISST-DEEP with and without the path reduction module since the configuration of the PRM were suspected to be a possible source for discrimination of large droplets. All three instruments were mounted into a frame to ensure that their optical sensors (see white arrows in Figure 4.2) are positioned at an equal radial distance from the plume centreline.

The silhouette camera operates using the principle of backlighting to create silhouettes of particles suspended between the light and the camera. 15 images are taken per second and the number of droplets per image in these experiments is usually several hundred or more. Particle dimensions are quantified and used to determine droplet sizes and size distribution. The number of droplets processed per distribution is usually over 1-million. The silhouette camera used is a high pressure version rated for 300 bar. Further details regarding the SINTEF SilCam can be found in Davies et al., 2016.

The results from these two experiments comparing the three different instruments are presented in Figure 4.3.

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Droplet diameter (µm)

Figure 4.3: Droplet size distribution (5-500 μm) from the two experiments with the three instruments (LISST-100X, SINTEF Silhouette camera, and the LISST-DEEP with and without the 80% PRM). Conditions: 1.5 mm nozzle, flow rate 1.2 L/min with Oseberg blend.

The experiments without the PRM were performed at a longer distance from the nozzle to obtain an increased dilution of the plume. Operating LISST instruments without a PRM significantly shorten the experimental time available due to the high concentrations in the Tower Basin, even with these low flow rates. The LISST-100X used in this study has a permanent 80% PRM.

#### 4.3 Ambient experiments - SwRI

The experiments performed in 2015 replicated the initial experiments performed in 2014 (Appendix C). The only experimental difference was that all experiments were performed with one dispersant injection technique (Simulated Injection Tool – SIT).

The oil flow rate, tank pressure and water/oil sampling intervals are presented Figure 4.4. Intervals for oil samples and monitoring of droplet sizes are indicated on the figure as coloured bars. The droplet size distributions monitored by the SilCam are presented in Figure 4.5. Colours on the graphs are similar to colours used for the initial experiments performed in 2014 (Appendix C).







Figure 4.4: Ambient Experiment 3 (6. March 2015): Flow-log data showing oil flow (blue line), oil temperature (red line). Areas for oil sampling and monitoring droplet sizes (Figure 4.5) are indicated by bars, oil alone (grey), DOR 1:100 (red) and DOR 1:50 (blue).



Figure 4.5: Ambient experiment 3 (6. March 2015): Droplet size distribution for oil alone (black) and treated oil (red: DOR 1:100 and blue DOR 1:50). All droplet size data obtained with the SINTEF SilCam.

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#### 4.4 High Pressure Experiments - SwRI

The high pressure experiments performed in 2015 replicated the initial experiments performed in 2014 (Appendix C). The only experimental difference was that all experiments were performed with one dispersant injection technique (Simulated Injection Tool – SIT) and that the SINTEF Silhouette Camera was included together with the LISST.

The oil flow rate, tank pressure and water/oil sampling intervals are presented in Figure 4.6. Intervals for oil samples and monitoring of droplets are also indicated on the figures as coloured bars. The droplet size distributions monitored by the SilCam are presented in Figure 4.7. Colours on the graphs are similar to colours used for the initial experiments performed in 2014 (Appendix C).







Figure 4.6: High pressure experiment 1 (4. March 2015): Flow-log data showing oil flow (blue line), oil temperature (red line). Areas for monitoring droplet sizes (Figure 4.7) are indicated by bars, oil alone (grey), DOR 1:100 (red) and DOR 1:50 (blue).





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#### 4.5 Comparison of results

This section presents data from the previous figures plotted together in various combinations to compare the droplet sizes from the ambient experiments with the HP experiments.



4.5.1 HP versus Ambient conditions – oil alone (SwRI)

Figure 4.8: Comparison of ambient experiment (black) and high pressure experiment (yellow) at SwRI. Droplet size distributions are measured with the SINTEF SilCam.  $d_{50}$  from the cumulative distribution are given in the figure with colours and line types corresponding to the figures. Data are from figures Figure 4.5 and Figure 4.7.





#### 4.5.2 Dispersant effectiveness - HP versus Ambient (SwRI)





Figure 4.10: Comparison of ambient (red) and HP experiments (yellow). All droplet sizes are measured with SINTEF SilCam. Dispersant DOR 1:50/Corexit 9500. d<sub>50</sub> from the cumulative distribution are given on figure with colours and line types corresponding to the figures. Data are from figures Figure 4.5 and Figure 4.7.

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#### 5 Discussion

This section contains a discussion of the data presented in the previous section.

#### 5.1 Ambient conditions - SINTEF

The results from the comparative testing at SINTEF shown in Figure 4.3 confirm the hypothesis that the PRM of the LISST-DEEP is a possible source for droplet discrimination.

The main deviation in the dataset measured with the LISST-DEEP at SwRI in January 2014, from data generated earlier at SINTEF, was the smaller droplets for untreated oil ( $d_{50}$  of 150 instead of 230-280 µm). This is consistent with the size distributions in Figure 4.3, where the LISST-DEEP (and PRM) gives a significant shift towards smaller droplets.

Based on these measurements it is our conclusions that the discrimination of the large droplets at SwRI in 2014 was caused by the PRM in the LISST-DEEP and that new experiments were necessary using the Silhouette camera to quantify droplet sizes of untreated oil.

#### 5.2 Ambient and HP conditions - SwRI

Operating the LISST-DEEP without the PRM in the SwRI tank was difficult due to the high oil concentrations during these experiments and the reduced tank volume (75% of the Tower basin). The experiments with dispersant injection were especially challenging due to the small droplet size giving a high optical density causing saturation (no transmittance of light to the LISST sensor). The LISST-100X cannot be used during the high pressure experiments, which results in the SINTEF SilCam being the main instrument used during the Ambient and HP experiments at SwRI.

The measured droplet sizes for the untreated oil (oil alone) and the reduction as a function of dispersant injection (DOR 1:100 and 1:50) in Figure 4.5 show trends similar to earlier experiments in API D3 Phase-I and II. A reduction from 230-280  $\mu$ m for the untreated oil to around 100  $\mu$ m for DOR 1:100 and around 80  $\mu$ m for DOR 1:50 are in the same ranges as seen in earlier experiments in the SINTEF Tower Basin (Brandvik et al., 2014 and 2015). The measured IFTs are comparable to similar experiments in the SINTEF Tower basin giving approximately 20 mN/m for untreated Oseberg versus 1 - 0.1 mN/m for treated oil (1 and 2% dispersant).

The new droplet size distributions from 2015 for the Oil alone experiments (Ambient and HP) are compared in Figure 4.8. The black graphs represent the Ambient conditions and the initial droplets sizes are around 230-280  $\mu$ m (oil alone 1). The reduction in droplet sizes for the later oil alone measurements (oil alone 2 and 3) are explained by the general lowering of the oil-water IFT due to the increasing amount of dispersant injected into the tank (see Figure 4.4). This is also seen in the measured IFT values from the oil samples taken inside the chamber during the experiments (see Table 4.2). The IFT for the three untreated oil samples oil alone 1, 2 and 3 (HP experiment) are 22, 9 and 4 mN/m. This effect is not seen so clearly in the SINTEF Tower Basin during experiments of similar duration, probably due to the larger water volume.

The initial values and the reduction in droplet sizes during the experiment are very comparable for the Ambient (black) and the HP (Yellow) experiments. The slightly smaller droplet sizes for the HP experiment could be influenced by the higher water & oil temperature that day (17°C) compared to the Ambient (10°C). The difference in viscosity of the Oseberg blend at these temperatures (4 versus 6 cP at shear rate 1000 s<sup>-1</sup>) should give a reduction on d<sub>50</sub> of approximately 5% (modified

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Weber scaling). However, this variation in droplet sizes between the experiments could also be within the expected experimental error.

The measured droplet sizes for the treated oil, both at DOR 1:100 (Figure 4.9) and DOR 1:50 (Figure 4.10) show very similar results when comparing Ambient and HP conditions. The droplet sizes for the two replicate experiments at both dispersant dosages are also very similar.

To simplify and clarify the comparison of the results from Ambient experiments (5 meters depth) and High Pressure experiments (1750 meters depth), only the first replicates are compared in Figure 5.1 below. This removes the uncertainties introduced by the increased surfactant concentrations in the experimental chamber as increasing amount of dispersants are injected into the chamber.



Figure 5.1: Comparison of the first replicates from both Ambient (Figure 4.5) and HP experiments (Figure 4.7). d<sub>50</sub> for the volume size distributions are given in brackets. All droplet sizes are measured with SINTEF SilCam. Dispersant DOR 1:100 (1%) and 1:50 (2%) with Corexit 9500.

The  $d_{50}$ , given in brackets in Figure 5.1, show slightly lower values for the High Pressure experiment (1750 m), compared to the Ambient experiment (5 m). As discussed earlier, this could probably be explained by the higher water & oil temperature for the HP experiment (17°C) compared to the Ambient experiment (10°C). These differences in oil & water temperature are due to different air temperatures on these two days at this outdoor facility (23 versus 17°C).



#### 6 Conclusions

Only the SINTEF Silhouette camera could be used to compare initial droplet formation at Ambient and HP conditions due to the high oil concentrations and the high pressure during the experiments.

The droplet sizes documented by the SilCam from comparable experiments (nozzle, oil type, flow rates, injection techniques and dispersant product) at Ambient pressure (5 meters depth) and High Pressure conditions (1750 meters depth) show no significant difference in droplet sizes as a function of water depth, represented by varying pressure.

This lack of a pressure effect was observed for both untreated oil and for the smaller droplets created as a function of dispersant injection (Figure 5.1). This strongly indicates that subsea dispersant injection effectiveness is not dependent on water depth and hydrostatic pressure.

These experiments were performed with stabilized dead oil without any presence of gas. Experiments with recombined oil & natural gas ("live oil") and additional natural gas were performed in separate study in November 2015 (API D3 Phase-V). Also this study showed no pressure effect on initial droplet formation and SSDI effectiveness, see Brandvik et al., 2016b for further details.

The dispersant injection method used (Simulated Insertion Tools) simulates operative dispersant injection as close as possible in such down-scaled laboratory experiments (Brandvik et al., 2014).

#### 7 Recommendations – Next phase of HP experimentation

The conclusions in this report are drawn from only the two pressure levels, additional data at multiple pressure levels would help to consolidate the conclusions in this report.

The conclusions from this study was used to design the next phase of this study (API D3 Phase-V) performed in October - November 2015. Experiments were performed with Oseberg blend recombined with natural gas under pressure to form "live oil". These experiments with "live oil" and natural gas (NG) were used to verify the results from the HP testing described in this report and findings from earlier phases in the API D3 JITF (combined oil & gas releases at ambient conditions).

Further details regarding experiments including multiple pressure levels and "live oil" are given in Brandvik et al., 2016b.



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#### A Appendix: Experimental description of HP & Ambient work performed at SwRI

This appendix describes the 90-inch chamber facilities at SwRI and the general experimental setup.

#### A.1 Basic facilities

The 90-inch Inner Diameter chamber at SwRI has been in service with the 1960's as a part of SwRI's Ocean Simulation Labs. It has been used for many purposes from initial hydrostatic testing of manned and unmanned submeribles to buoyancy modules, pipe, subsea Oil & Gas industry equipment and recently for research activity involving deep water oil releases.

The main specifications of the 90-Inch Chamber and test setup are:

- 1. The tank has approximately 18.25 feet (5.6 meters) of working length and has 90-inches (2.3 m) inner diameter with a hemispherical head on the bottom. The chamber holds 6,444 gallons (24.4 m<sup>3</sup>) of simulated salt water. The chamber is located outdoors and under cover.
- 2. The salt water is mixed in a separate tank to nearly 24% by weight concentration and is circulated through high capacity sand filters to remove impurities before mixing with chamber fresh water. Commercially available water softener salt with minimal additives is used.
- 3. A separate frame is installed within the chamber to hold SINTEF measurement equipment and tubing routed to the test nozzle.
- 4. An oil flow loop was setup external to the chamber that consisted of an accumulator, flow meter and control valves. Oil stream extraction equipment was placed on the chamber closure to withdraw oil/water sample from the plume during atmospheric and high pressure experiments.
- 5. A LabVIEW based control system recorded data and controlled the oil delivery. Dispersant delivery was controlled via a separate Insco Pump and control software.
- 6. SwRI's facilities include an effluent tank to hose the contaminated oil/water mixture. The waste fluid is filtered and disposed in accordance with local ordances and permits.

The overall configuration of the 90-inch chamber, control and oil/dispersant delivery system can be seen in Figure A - 1.



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Figure A - 1: Piping and Instrumenation Diagram for Phase III Amendment Testing

#### A.1.1 Monitoring during the experiments

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This chapter contains the description of the different monitoring techniques used during the release experiments. The main monitoring is performed in the center of the plume approximately 3 meters above the release point. SINTEF monitoring equipment statically affixed to the chamber internal frame as seen in Figure A - 2.



Figure A - 2: SINTEF Instruments Mounted to Test Frame

SwRI pressure and temperature monitoring instrumentation was mounted in accordance with Figure A - 1.

#### A.1.1.1 Oil/Water sampling

Water samples were taken below the position in the tank as droplets sizes were measured. The water is sampled through a long tube located on the internal moutnign frame. The water samples were captured and stored in flasks for SINTEF posttest evaluation.

#### A.1.1.2 Video documentation

One video camera was used to document the plume during experiments. This camera was used to monitor the plume and had pan/zoom capabilities to visualize the release nozzle and SINTEF equipment. Figure A - 3 shows the plume exiting the nozzle during a high pressure experiment.





Figure A - 3: Screen Capture of Video during High Pressure Test, Dispersant Introduced (1% C9500)

#### A.1.2 General description of a 90-Inch Chamber experiment

A short version of this procedure for a blow-out experiment in the Tower basin is given below:

- 1. Test Preparation:
  - a. Fill salt water mixing chamber approximately 2/3 full with fresh, chilled water. Turn on agitator and add predetermined amount of salt. Circulated high concerntraion salt water mix through chiller and filter. Skim off impurities in form of foam from tank top opening.
  - b. Flow high concerntation salf water and fresh chilled water through mixing chamber into 90-inch diameter chamber. Install sump pump at bottom of 90-inch chamber to continuously mix the water as it fills the chamber. Measure the salinity of the mix at regular intervals and adjust flow of salt and/or fresh water accordingly.
  - c. Once the chamber is filled, allow the sump to mix the water for several hours. Approximately 4 hours or more before testing, switch the flow to route the chamber water through filters. Clarity is needed for the camera.
  - d. Fill piston accumulator full with Oseberg blend oil and attach high pressure nitrogen on other side with regulator. Set nitrogen pressure appropriately for the test to be performed.
  - e. Load chamber with test frame holding the nozzle and instrumentation. Close chamber and make all necessary water, oil, dispersant, video and electrical connections. Check function of all items prior to beginning test.
- 2. Testing
  - a. Begin to flow oil alone at specificed rate. Begin plume oil/water sampling system. Extract oil/water sample once flow rate has stabilized.
  - b. Begin injection of dispersant at first speicified rate. Extract oil/water sample.
  - c. Repeat for all remaining dispersant injection rates required. Record video of the plume and/or other points of interest.

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- d. Stop flow of oil and dispersant. Depressurize chamber (if necessary) and disconnect necessary items in order to open the chamber.
- 3. Frame Removal and Chamber Reset
  - a. Remove test frame from chamber, use high pressure water spray to clean excess oil from frame and capture inside chamber.
  - b. Extract the oil layer from the top of the chamber water and route to effluent tank first. Wash down chamber internal surfaces while removing contaminated water and route all fluids to effluent tank.
  - c. Clean chamber and test frame. Remove all remaining oil residue from instrumentation surfaces.

A typical atmospheric or high pressure test takes one day with approximately 15 minutes of actual oil flow time before the chamber is too saturated for camera use and/or the small droplets saturate the oil droplet measurement equipment. Cleaning and chamber reset activities take one calendar day.



Figure A - 4: Chamber Ready for Test Prior to Frame Install





Figure A - 5: The path reduction module of the LISST-DEEP (round/conical prism) and the perforated plastic clip holding it in place used during the initial testing (see Appendix C for details). Seen form the side (upper) and from above (lower).



Figure A - 6: The path reduction module of the LISST-100X (round/conical prism). The LISST-100X and LISST DEEP were used during the initial testing (see Appendix C for details).



#### **B** Appendix: Experimental description of SINTEF Tower Basin

#### **B.1 Basic facilities**

The basic parts of the SINTEF Tower Basin were constructed and built in 2005 as a part of SINTEFs research activity within deep water releases. This was a follow-up activity of the DeepSpill field experiment in 2000. This basic infrastructure was financed by STATOIL ASA. Due to reduced focus on deep water releases and since our main ice basin at SeaLab was heavily booked for other project, the Tower Basin was not mounted and tested until January 2011.

The ice basin without the Tower Basin and the mounted Tower Basin during the first filling are seen in Figure A.1.A drawing showing the scaffolding/railing around the Tower Basin together with the ventilated hood and oil collecting system is shown in Figure A.2. The main components of the basin before the first experiment in March 2012 are shown in Figure A.3 and the principles of the experimental set-up for the Tower Basin are shown in Figure A.4.

The main specifications of the Tower Basin are:

- 7. The tank is 6 meters high, 3 meters wide and holds  $40 \text{ m}^3$  of natural sea water.
- 8. The sea water is rinsed through high capacity sand filters and holds a stable and high purity.
- 9. All release rates of oil and gas are remotely controlled and both set points and real values are logged on a central control system.
- 10. The tank has three remotely operated and programmable instrument platforms. The positions of these are logged during operation (depth and axial position).
- 11. To insure proper HSE working conditions a scaffolding/railing around the tower and a staircase to reach the top section is installed for inspection and sampling
- 12. A ventilated hood prevents light hydrocarbons to enter the laboratory hall. It is not necessary for the operators to wear any breathing protection.
- 13. A overflow system to skim off surfacing oil from the top of the tower ensure safe and efficient removal of surface oil.
- 14. A disposal system approved by the local environmental authorities is in place to take care of the surface oil and the large volume of oil containing water. Especially the chemical enhanced dispersion experiments will create very small droplets with very long settling times.

The principal overview of the experimental set-up (Figure A.4) shows the main features of the Tower Basin. Oil, gas and dispersants can be delivered over a wide range of flow rates and internal ratios. Both oil and gas are delivered through mass controllers and both the set points and the obtained values are logged during an experiment. We have two pressurized tanks (30 bar) for delivering the oil (25 and 100 Liters). The system is operated and monitored from a central computer through a program written in NI LabView<sup>®</sup>. A screen dump of the settings and obtained values for flow rate (oil) is also given in Figure A.4. The log of the actual values obtained during the experiments is important for further analysis of the data.

NB! This is a general text describing the capabilities of SINTEF Basin Tower and MiniTower. The resources allocated to a specific project (type of equipment, sampling frequency etc.) are scope dependent and described in the experimental plan of each project.



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Figure A.1: Ice basin without propellers and other equipment used for circulation showing the fundament for the Tower Basin (left) and the initial mounting of the Tower Basin in January 2011 (right).



Figure A.2: Principles for the scaffolding/railing around the tower, ventilated hood and overflow system to collect surface oil from the top of the tower.

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Figure A.3: The Tower Basin per March 2012 showing the scaffolding, staircase and the railings to ensure safe working conditions and the ventilated hood.



Figure A.4: Principle overview of the experimental set-up showing how oil, gas (air) and dispersant will be released during a Tower Basin experiments (P: Pressure gauge, F: Flow controller). An example of the set point for oil flow rate (L/min) and the obtained values are also given. This is a screen dump from the operator's computer during an experiment.

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Figur A.5: Left: Adjustment of camera equipment and the Vitrino 3D current profiler with all three instrument platforms in surface position. Right: All three instrument platforms lowered into the tank and ready to initiate an experiment. The squared release platform can be seen in the middle on the bottom of the tank.

#### **B.1.1 Monitoring during the experiments**

This chapter contains the description of the different monitoring techniques used during the blowout simulation experiments. The main monitoring is performed in the centre of the plume approximately 3 metres above the release point. A suit of instruments is mounted on a piston operated platform which is inserted into the plume. The platform is mounted on a slide on the inner wall of the basin and its vertical and radial position can be continuously adjusted, see Figure A.6. The instrument platform can continuously be lifted or lowered in the tank during an experiment to study variations in droplet size as a function of height. However, monitoring too close to the release could be difficult due to saturation of our instrumentation, especially the LISST instrument.

#### **B.1.2 Droplet size distribution**

Since documentation of oil droplet size distribution is central in many projects, three different approaches can be used to measure droplet size distribution of the rising oil droplets (see Figure A.6). How many and which methods used depends on the scope of the specific projects.

- 1. LIST 100X Particle size analyzer (2 500 μm)
- 2. In-situ macro camera with a green laser focusing plane  $(5 2500 \,\mu m)$
- 3. External particle Visual Microscope, Mettler Toledo PVM V819 (5 -1200 µm )



Figure A.6: The three systems for monitoring droplet size distribution shown during adjustment over the water surface and submerged.

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The LISST-100X and the in-situ macro camera is operated inside the Tower Basin (Figure A.6) and can do measurement at different locations with respect to the oil plume. The external camera (PVM) is located outside the tank, but the water is collected from a hose located together with the LIST 100X (Figure A.6). Images from PVM and In-situ camera are used to generate droplet size distributions that are complementary to distributions from the LISST-100X, since they can detect larger droplets.

#### B.1.2.1 Water sampling

Water samples can be taken in the same position in the tank as droplets sizes are measured. The water is sampled through a short flexible hose located on the moving sampling platform. The water samples can be analysed for (dependent on the scope of the projects):

- a. Oil content (Total hydrocarbons THC)
- b. Dispersant content (For dispersant experiments)

#### B.1.2.2 In-situ measurement of oil in water

The overflow hose (no pumping) used for water sampling above can also be used for monitoring of oil-in-water content (droplets and dissolved components). This is done by ultraviolet fluorescence (UVF) with an UviLux flow-through cell. The water flows through this cell before being sampled.

#### B.1.2.3 Oil sampling

The oil in the water samples taken from the plume can be analyzed for the following parameters:

- a. Interfacial tension (when dispersants are applied).
- b. Surfactant content (a part of the C9500 solvent package a glycol ether (DPnB) is used as reference, GC-MS analysis).

#### **B.1.2.4 Video documentation**

Several video cameras are used to control and document the operation of the Tower Basin during an experiment (operational cameras). These cameras are used to monitor the following locations:

- a. The release nozzle
- b. The use of injection tools for dispersant (wand, dispersant ring etc.)

The video footage from the operational cameras is stored as a part of the operational documentation, but is usually not used for further analysis.

The video recordings used for documenting and analysing the droplet sizes are taken by four HD cameras (1280 x 960 pixels) at four different adjustable heights over the release point (for example 0, 0.5, 1 and 1.5 meters). An example of such video is given in Figure A.7. Close-up or macro still or video cameras are also used to study details regarding injection of dispersants, turbulence around release nozzle etc. (Figure A.8).

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Figure A.7: An example of a composite video showing the four HD cameras covering the rising oil flume at 0.5 m intervals over the release point. This is from earlier experiments with a North Sea crude (Oseberg blend).



Figure A.8: Close-up images (video) of release nozzle with options for injection dispersant horizontally into the oil. A: Oil released alone, no dispersant, B: Dispersant injected.

#### **B.1.3 General description of a Tower Basin experiment**

This section gives a general description of a Tower Basin experiment. A more specific procedure for operating the Tower Basin is found in our internal operational procedures (Laboratory procedure no: 507). This procedure is a part of SINTEF general QA system for laboratory activities.

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A short version of this procedure for a blow-out experiment in the Tower Basin is given below:

- a. Test filling, release and control equipment.
- b. Fill Tower Basin with sea water and check for leaks.
- c. Check background values (particle/oil concentration, droplet size distribution, temperature).
- d. Determine and program test conditions (oil type and rates of gas/oil).
- e. Check and confirm status on monitoring equipment.
- f. Prepare for experiment, perform background monitoring (approximately 3 meters above release nozzle)
- g. Initiate experiment, start release of oil/gas/dispersant (dependent of experiment type)
- h. Monitoring of oil/gas/dispersant plume (0.5 3 min)
  - Video cameras (4 cameras at three different heights)
  - Oil droplet size distribution (LISST-100X, particle visual microscope (PVM) and in-situ macro camera/lasers).
  - UVF monitoring of oil content/dissolved components
  - Water and oil sampling, dependant on type of experiment.
- i. Stop release
- j. Collection, initial quality control of monitoring data and storage of data.
- k. Settling of oil droplets and removal/skimming of surface oil
- 1. Emptying/disposal of used water containing small oil droplet and dissolved oil components according to lab. Procedure.
- m. Cleaning and control of equipment.

A typical Tower Basin experiment consist of two days of preparation (filling of water, filling of oil in the pressurized tank, testing of release and monitoring equipment etc.), one day for the actual experiment and two days for settling of oil droplets and cleaning of the tank and monitoring equipment, QA, storage and initial treatment of data, chemical analysis etc.

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#### C Initial investigations performed in 2014 at SINTEF and SwRI

#### **C.1** Introduction

The main objective with this experimental work was to verify earlier findings obtained under ambient conditions (SINTEF Tower basin) regarding initial droplet formation and subsea dispersant effectiveness by performing similar experiments at high pressure at SwRI in San Antonio, TX, USA.

The instrumentation used to measure droplet sizes in the earlier studies (LISST 100X) cannot be used at high pressure (>30 bar), so the new HP version LISST DEEP was used for this work. This instrument is mainly designed for monitoring low oil concentrations (< 50 ppm??) in deep waters. However, the upper concentration tolerance is strongly dependent on droplet sizes and will be reduced for smaller droplets, for example, after subsea dispersant injection.

To make it possible for the LISST DEEP to handle the high oil concentration (200-500 ppm) and especially in combination with small droplet sizes after dispersant injection (10-100  $\mu$ m), a path reduction module (PRM) was constructed for SINTEF by Sequoia. This is not a standard option for the LISST DEEP.

#### C.2 Experimental

This work is performed both at SINTEF in Norway and at SwRI in USA. The work at SINTEF is performed in their Tower basin, which is 6 meters high, 3 meters wide and holds 40 m<sup>3</sup> of natural sea water. A description of the Tower basin is given in Appendix B and in Brandvik et al., 2013.

SwRI has very suitable hyperbaric chambers for such studies. The largest has dimensions which are comparable to the SINTEF Tower Basin. A description of their facilities the experimental procedures used is given in Appendix A.

#### C.2.1 Experimental work at SwRI January 2014

The experimental matrix is described in

Table C.1. The first tank test revealed that 2.8 L/min was a too high flow rate and gave a very short monitoring time. All later experiments were performed at 1.2 L/min.



| Test<br>Tank<br>Set                     | Medium<br>Nozzle<br>(1.5 mm) | Ambient<br>Pressure | 2500psi<br>(172 bar)<br>Pressure | Low<br>Flow<br>Rate <sup>*</sup> | High<br>Flow<br>Rate <sup>*</sup> | Oil alone | Low<br>(1:100)   | High<br>DOR<br>(1:50) |
|---|------------------------------|---------------------|----------------------------------|----------------------------------|-----------------------------------|-----------|--|-----------------------|
|   | Х                            | Х                   |                                  | Х                                |                                   | Х         |  |                       |
| 1                                       | Х                            | Х                   |                                  | Х                                |                                   |           | Х  |                       |
| Test<br>Tank<br>Set<br>1<br>2<br>3<br>4 | Х                            | Х                   |                                  | Х                                |                                   |           |  | Х                     |
|   | Х                            | Х                   |                                  |                                  | Х                                 | Х         |  |                       |
| 2                                       | Х                            | Х                   |                                  |                                  | Х                                 |           | Х  |                       |
|   | Х                            | Х                   |                                  |                                  | Х                                 |           | Low<br>(1:100)   x | Х                     |
|   | Х                            |                     | Х                                | Х                                |                                   | Х         |  |                       |
| 3                                       | Х                            |                     | Х                                | Х                                |                                   |           | Х  |                       |
| Set       1       2       3             | Х                            |                     | Х                                | Х                                |                                   |           |  | Х                     |
|   | X                            |                     | X                                |                                  | X                                 | X         |  |                       |
| 4                                       | X                            |                     | X                                |                                  | X                                 |           | X  |                       |
|   | X                            |                     | X                                |                                  | Х                                 |           |  | Х                     |

Table C.1: Original experimental matrix for Phase 1 high pressure experiments.

\* After initial testing it was decided that the tank was too small for the high flow rate experiments (too high droplet concentration). All experiments were for this reason performed at low flow rate (1.2 L/min) and two different injection techniques were used instead (Simulated Injection tool – SIT and upstream injection).



#### C.3 Results

|            |                      |                     |   | Ту   | pe of experiment  |                  |
|------------|----------------------|---------------------|---|--|---|------------------|
| Exp.<br>no | Date                 | Rate<br>(L/mi<br>n) | Type<br>experime<br>nt                        | Disp<br>GOR<br>Injection                         | Comments  | Water<br>Samples |
| 0          | 160114               | 1.2<br>+ 2.8        | Initial<br>testing (oil<br>and water<br>only) | non  | Testing at both pressures and flow<br>rates, short ramp times for oil flow<br>rates even at high pressure 2500<br>PSI. Calibrating flow meter with water<br>and oil.  | 0                |
| 1          | <mark>170</mark> 114 | 1.2<br>+ 2.8        | Ambient                                       | 1:100-50<br>Upstream<br>injection<br>C9500A      | Bubbles from air trapped in piping<br>interfered with LISST – limited results<br>Low LISST signal due to high optical<br>density. High flow rate <b>not possible</b> .  | <mark>5</mark>   |
| 2          | 210 <mark>114</mark> | 1.2                 | Ambient                                       | 1:100-50<br>Upstream<br>injection                | Dispersant leaking into mixing T from<br>dispersant pump/piping during first<br>part. Disconnected & connected line<br>manually later OK! Usable data from<br>second part? The oil alone data<br>can be compared with the later<br>experiments. | N                |
| 3          | <mark>240114</mark>  | 1.2                 | Ambient                                       | 1:100-50<br>Upstream<br>injection                | Good data, but small "Oil alone"<br>droplets ( $d_{50}$ =144µm). Very low water<br>transparency. Freezing outdoor<br>conditions, oil temp; 7.5 °C (45.5 F°)   | 5                |
| 4          | 270114               | 1.2                 | High<br>Pressure                              | 1:100-50<br>Upstream<br>injection                | Good data, but generally small "oil<br>alone" droplets (?). Data seems<br>comparable with Ambient<br>experiments! Water samples contain<br>small droplets due to pressure<br>reduction during sampling.   | 5                |
| 5          | 290114               | 1.2                 | High<br>Pressure                              | 1:100-50<br>Simulated<br>Injection Tool<br>(SIT) | Good data. Better/clearer shift in d <sub>50</sub><br>due to SIT injection. Very good and<br>reproducible "oil alone" distributions.<br>Very good video/Clear water!! Very<br>low oil temperature due to near<br>freezing conditions.           | Z                |
| 6          | 310114               | 1.2                 | Ambient                                       | 1:100-50<br>Simulated<br>Injection Tool<br>(SIT) | Good data. Again better/clearer shift<br>in d <sub>50</sub> due to SIT injection. Very good<br>and reproducible distributions. High<br>oil temperature (19 °C) due to very<br>warm weather.   | 7                |

Table C.1: Overview of experiments performed at SwRI in January 2014.

Red: Not successful experiment Yellow: Partly successful experiment Green: Completely successful experiment

#### **Type of experiment:**

Ambient pressure – Lid on, no pressurization High Pressure – Tank pressurized to 2500 Psi or 170 bars

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Table C2:Interfacial tension (IFT) measurements of oil samples taken from the hyperbaric<br/>chamber during 2014 experiments. The water/oil sampling interval is indicated on<br/>Figure C1, Figure C3, Figure C5 and Figure C7.

| Exper 210114 (Ambient – Upstream)       | SINTEF ID | IFT      |
|---|-----------|----------|
|   |           | (mN/m)   |
| 2-Oil alone-1C                          | 2014-0052 | 20,4±1,2 |
| 2-DOR100-A                              | 2014-0041 | 3,8±0,7  |
| 2-DOR50-A                               | 2014-0028 | 2,6±1,5  |
| 2-Oil alone-2C                          | 2014-0053 | 10,4±2   |
| Exper 240114 (Ambient – Upstream)       |           |          |
| 3-Oil alone-1                           | 2014-0054 | 20,9±1,9 |
| 3-DOR100                                | 2014-0043 | 4,7±1,1  |
| 3-DOR50                                 | 2014-0031 | 0,1±0,4  |
| 3-Oil alone-2A                          | 2014-0055 | 20,3±1,4 |
| 3-Oil alone-2B                          | 2014-0056 | 14,0±2,1 |
| Exper 270114 (High Pressure – Upstream) |           |          |
| 4-Oil alone-1                           | 2014-0057 | ND*      |
| 4-DOR100                                | 2014-0044 | 3,8±1,0  |
| 4-DOR50-1                               | 2014-0032 | 0,3±0,4  |
| 4-DOR50-2                               | 2014-0033 | 0,6±0,4  |
| 4-Oil alone-2                           | 2014-0058 | 10,1±2,5 |
| Exper 290114 (High Pressure – SIT)      |           |          |
| 5-Oil alone-1                           | 2014-0059 | 20,1±1,3 |
| 5-DOR100-1b                             | 2014-0046 | 4,2±0,9  |
| 5-DOR50-1b                              | 2014-0035 | 1,3±0,3  |
| 5-Oil alone-2                           | 2014-0060 | 9,2±2,6  |
| 5-DOR100-2                              | 2014-0047 | 4,0±1,0  |
| 5-DOR50-2                               | 2014-0036 | 1,8±0,4  |
| 5-Oil alone-3                           | 2014-0061 | 8,3±1,7  |
| Exper 310114 (Ambient – SIT)            |           |          |
| 6-Oil alone-1                           | 2014-0062 | 19,7±1,3 |
| 6-DOR100-1                              | 2014-0048 | 5,6±2,0  |
| 6-DOR50-1                               | 2014-0037 | 2,2±1    |
| 6-Oil alone-2                           | 2014-0063 | 7,5±2,3  |
| 6-DOR100-2                              | 2014-0049 | 3,6±0,6  |
| 6-DOR50-2                               | 2014-0038 | 1,4±0,7  |
| 6-Oil alone-3                           | 2014-0064 | 6,1±1,1  |
| SwRI oIL samples                        |           |          |
| 2- directly from drum                   | 2014-0025 | 18,0±1,9 |
| 4-dir from accumulator                  | 2014-0023 | 21,0±1,4 |
| 4- from nozzle after experiment         | 2014-0022 | 1,5±1    |
| 6-dir from accumulator                  | 2014-0024 | 19,6±1,7 |

\* ND: Not possible to measure IFT due to formation of stable water-in-oil emulsion during sampling

#### Sample name legends:

- "Oil alone": Untreated oil
- "DOR100": Treated with C9500 DOR 1:100 (1%)
- "DOR50": Treated with C9500 DOR 1:50 (2%)



#### C.3.1 SwRI – Ambient experiments 2014

The oil flow rate, tank pressure and water/oil sampling interval are presented for both upstream injection (Figure C1) and for Simulated Injection Tool – SIT (Figure C3). Intervals for oil samples and monitoring of droplets are also indicated on the figures as coloured bars. The droplet size distributions monitored by the LISST-DEEP are presented in Figure C2 and Figure C. Colours on the graphs are similar to colours on bars in previous figures.



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Figure C1: Ambient experiment 3 Upstream (240114): Flow-log data showing the oil flow (blue line), oil temperature (red line). Areas for monitoring droplet sizes (Figure C2) are indicated by bars, oil alone (grey), DOR 1:100 (red) and DOR 1:50 (blue).



Figure C2: Ambient experiment 3 Upstream (240114): Droplet size distribution for oil alone (black) and treated oil (red: DOR 1:100 and blue DOR 1:50).

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Figure C3: Ambient Experiment 4 SIT (310114): Flow-log data showing the oil flow (blue line), oil temperature (red line). Areas for monitoring droplet sizes (Figure C4) are indicated by bars, oil alone (grey), DOR 1:100 (red) and DOR 1:50 (blue).



and treated oil (red: DOR 1:100 and blue DOR 1:50).

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#### C.3.2 Swri – High Pressure Experiments 2014

The oil flow rate, tank pressure and water/oil sampling interval are presented for both upstream injection (Figure C5) and for Simulated Injection Tool – SIT (Figure C7). Intervals for oil samples and monitoring of droplets are also indicated on the figures as coloured bars. The droplet size distributions monitored by the LISST-DEEP are presented in Figure C6 and Figure C8. Colours on the graphs are similar to colours on bars in previous figures.





Figure C5: High pressure experiment 1 Upstream (270114): Flow-log data showing the oil flow (blue line), oil temperature (red line). Areas for monitoring droplet sizes (Figure C6) are indicated by bars, oil alone (grey), DOR 1:100 (red) and DOR 1:50 (blue).



Figure C6: High Pressure experiment 1 Upstream (270114): Droplet size distribution for oil alone (black) and treated oil (red: DOR 1:100 and blue DOR 1:50).

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Figure C7: High pressure experiment 2 SIT (290114): Flow-log data showing the oil flow (blue line), oil temperature (red line). Areas for monitoring droplet sizes (Figure C8) are indicated by bars, oil alone (grey), DOR 1:100 (red) and DOR 1:50 (blue).



Figure C8: High Pressure experiment 2 SIT (290114): Droplet size distribution for oil alone (black) and treated oil (red: DOR 1:100 and blue DOR 1:50).

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#### C.3.3 Comparison of 2014 results

This section presents data from the previous figures plotted together in various combinations to compare across different variables (pressure, injection techniques, SwRI-SINTEF and LISST-DEEP and 100X).



#### C.3.3.1 Experiments with oil alone at ambient conditions SwRI - SINTEF

Figure C9: Comparison of ambient experiments at SwRI (black) and SINTEF (green, blue and red). Droplet size distributions from SwRI are measured with the LISST-DEEP, while distributions from SINTEF are measured with the LISST-100X. See later figures for comparison of LISST-DEEP and LISST-100X.

C.3.3.2 Upstream versus SIT at ambient conditions - SwRI



Figure C10: Comparison of ambient experiments at SwRI with two different injection techniques; Upstream injection (dotted lines) and Simulated Injection Tool (solid lines). Data are obtained with LISST-DEEP, Oseberg blend and Corexit 9500.





#### C.3.3.3 HP versus Ambient conditions - oil alone

Figure C11: Comparison of three ambient experiments (black) and two high pressure experiments (yellow) at SwRI. Droplet size distributions are measured with the LISST-DEEP.



Figure C12: Comparison of all three replicate experiments at ambient and HP experiments (SIT) at SwRI (black/yellow) and ambient experiments at SINTEF (green, blue and red). Droplet size distributions from SwRI are measured with the LISST-DEEP, while distributions from SINTEF are measured with the LISST-100X. Predictions of d<sub>50</sub> at different flow rates are given. All experiments in this study were performed at 1.2 L/min.

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#### C.3.3.4 Dispersants effectiveness - HP versus Ambient - SIT





Figure C14: Comparison of ambient and HP experiments at SwRI (blue/yellow) and ambient experiments at SINTEF (green). Droplet size distributions from SwRI are measured with the LISST-DEEP, while distributions from SINTEF are measured with the LISST-100X (API D3 JITS Phase-II report). Dispersant DOR 1:50/Corexit 9500.

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C.3.3.5 Dispersants effectiveness - HP versus Ambient - Upstream injection





experiments at SINTEF (green). Droplet size distributions from SwRI are measured with the LISST-DEEP, while distributions from SINTEF are measured with the LISST-100X (API D3 JITS Phase-II report). Dispersant DOR 1:50/Corexit 9500.

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#### C.3.3.6 Post experiments performed at SINTEF Tower Basin, March 2014

To further study the surprisingly small droplets for the untreated oil experiments at SwRI a series of experiments were performed with both the LISST-DEEP and 100X in the SINTEF Tower Basin. The results from these experiments are presented in the graphs in this chapter.







Figure C18: Comparison of ambient experiments at SwRI with the LISST-DEEP (solid lines) and ambient experiments at SINTEF with LISST-DEEP (dashed lines) and LISST-100X (dotted lines). Dispersant: Corexit 9500. All LISST DEEP experiments were performed with the PRM installed.

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Figure C19: Comparison of ambient experiments at SINTEF Tower Basin using both LISST-DEEP (upper figure) and LISST-100X (lower figure). This data was obtained during one experiment with the instruments mounted in the same height and distance from the centre of the plume from opposing sides. Dispersant: Corexit 9500 and oil type Oseberg blend.

#### C.4 Discussion

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This section contains a discussion of the data presented in the previous section.

#### C.4.1 Comparison of droplet size data obtained at SINTEF versus SwRI

The main difference between the droplet size data obtained at SwRI in January 2014 and at SINTEF are the different peak value of the distributions for the oil alone experiments (also called Mean Volume Diameter - MVD or  $d_{50}$ ). The data obtained at SwRI show generally a smaller  $d_{50}$  (typically 144 µm) compared to  $d_{50}$  from similar oil alone experiments performed at SINTEF (typically 280 µm). This can be seen in Figure C9 and Figure C12. The experiments are performed with the same oil and identical release arrangements/nozzles. When oil properties and release diameter are kept constant, droplet sizes are mainly dependent on; oil release rate, water properties (oil-water IFT) and oil temperature (oil viscosity). Below follows a discussion of possible causes for the SWRI-SINTEF oil alone data differences and findings.

#### Oil flow rate:

The flow meter used at SwRI was calibrated with water and oil at both ambient and high pressure conditions and showed stable and accurate readings (1.2 L/min  $\pm 3\%$  over 30 second periods). Flow meters at SINTEF have also been calibrated and showed stable and accurate readings ( $\pm 5\%$  over 30 second periods) See experimental description in Appendix A and B for further details.

If the observed shift in  $d_{50}$  (from 280 to 144  $\mu$ m) should be explained by variation in oil flow rate, a flow rate of 2.2 L/min would be needed (predicted by modified Weber scaling).

#### Quality of artificial salt water:

Experiments at SINTEF are performed with natural sea water corrected at 80 meters depth in the local fjord, while the experiments at SwRI were performed with artificial sea water. The sea water was made by using ordinary city water mixed with salt from a natural salt mine (evaporated sea water). The salt composition in this salt is very similar to what found in sea water and the salinity was 3.4% in both cases. See Appendix A for further details.

IFT between the artificial salt water and Oseberg blend was measured both at SwRI during the experimental period and later at SINTEF with samples of the artificial water brought back to SINTEF. They all showed values around 20 mN/m similar to values obtained with natural sea water at SINTEF.

#### **Oil temperature:**

The experiments at SwRI were performed in January at an outdoor facility with very variable temperatures. The outdoor temperature changed from freezing conditions to warm & nice working conditions (from -10 to 25 C<sup>o</sup>). The artificial sea water used was chilled and had a temperature in the 3-7 C<sup>o</sup> for all experiments, but the oil temperature was influenced by the outdoor temperature (temperature of holding tank, accumulator and pipelines in general). The oil temperature varied from 6-8 C<sup>o</sup> (coldest day) to 14-19 C<sup>o</sup> (warmest day). However, the difference in measured viscosity for the Oseberg blend at these temperatures (5 – 8 cP) is not sufficient to explain this shift in droplet sizes. The influence of viscosity (giving slightly smaller droplets) can clearly be seen in Figure C4 for the three replicate oil alone experiments as the oil heats up from 14 to 19 C<sup>o</sup> during the experiment (high outdoor temperature).

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Possible precipitation of waxes in oil accumulator and pipes at low temperatures and possible later dissolution on warmer days, has been discussed, and this could influence oil properties. However, the wax content in this paraffinic oil is not very high (2.4 %) and the pour point is low (-36  $C^{0}$ ). Analysis of wax composition in oil samples from each experiment (high temperature gas chromatography) showed no significant variation in wax composition.

Based on the investigations of oil flow rate, salt water quality, oil temperature and possible wax precipitation it was not possible to find a reasonable explanation of the observed shift in  $d_{50}$  (280 to 144  $\mu$ m) based on the experimental conditions. Further discussions of this shift are continued later in this appendix.

#### C.4.2 Comparison of different injection techniques

Due to the small droplets measured for untreated oil, a reduced shift as a function of dispersant treatment was observed. Since earlier experiments have shown that Simulated Insertion Tool (SIT) usually gives better effectiveness with Corexit C9500 (Brandvik et al., 2014a), this injection method was used for two of the experiments.

Droplet size distributions from ambient experiments at SwRI with both injection techniques (SIT & Upstream) are presented in Figure C10 and an increased effectiveness (larger shift) can be observed for SIT compared to upstream injection. The same trend is also observed in the later figures when the two injection techniques are discussed in more detail.

#### C.4.3 Comparison of high and ambient pressure

The results generated both at SwRI (ambient & high pressure) and SINTEF (ambient) are discussed in this section. The results for oil alone (untreated oil) and the effect of dispersants are discussed separately.

#### C.4.3.1 Untreated oil

The droplet size distributions for untreated oil are presented in Figure C11 (all experiments) and in Figure C12 (only SIT experiments). The main trend from these figures is that pressure has little influence on droplet formation, except for one of the HP experiments (290114). This deviating experiment is the HP SIT experiment and all three replicate measurements for the SIT experiments are presented in Figure C12 (HP and Ambient). The results presented in these two figures are not conclusive, regarding the effect of pressure, since;

- 1. Identical release arrangements were used for both injection techniques and should not influence droplet distributions for untreated oil.
- 2. However, for the deviating experiment (smaller droplets at HP), the replicate measurements show consistent results.

#### C.4.3.2 Dispersant effectiveness

Dispersant effectiveness (shift in  $d_{50}$ ) as a function of pressure is presented in Figure C13 and Figure C14 (DOR 1:100 and 1:50 - SIT) and Figure C15 and Figure C16 (DOR 1:100 and 1:50 - Upstream injection). These figures present droplet sizes for HP and ambient experiments at SwRI and ambient experiments at SINTEF. Although a small difference may be seen for the DOR 1:50 experiments (smaller droplets for HP experiments), no significant difference in droplet sizes can be seen between the ambient experiments (SINTEF & SwRI) and the HP experiments at SwRI.

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#### C.4.4 Comparison of LISST-DEEP and 100X results

The main objective for the experiments performed in the SINTEF Tower Basin after the experimental work at SwRI (January 2014) was to find the reason for the surprisingly small droplets for the untreated oil ( $d_{50} = 144 \ \mu m$ ) compared to prior results at SINTEF ( $d_{50} = 280 \ \mu m$ ). These experiments were performed with both the LIST DEEP and the 100X instrument.

The same trend as observed in SwRI with increased effectiveness of SIT compared to upstream injection can be observed in the SINTEF Tower Basin with the LISST-100X (Figure C17).

When results from SwRI (LISST-DEEP) and SINTEF (LISST-DEEP and 100X) are compared (Figure C18) we observe a good agreement between these experiments for the treated oils (DOR 1:100 and 1:50), but as discussed earlier the untreated experiments at SwRI produce smaller droplets. For the SINTEF LISST-DEEP results we can observe a bimodal distribution, which could offer an explanation to the small droplets observed at SwRI. This bimodal distribution could indicate a discrimination of larger droplets. To further investigate this, a separate experiment was performed using both the LISST-DEEP and 100X to monitor the droplet distribution.

This experiment was performed as a dosage experiment varying the rate of dispersant from oil alone, via DOR 1:1000 up to 1:25. The experiment was terminated with a new period with oil alone (Figure C19). These results show bimodal distributions for the LISST-DEEP for the largest droplets (oil alone and DOR 1:1000 and 1:500). This bimodal distribution contains peaks at approximately 280 and 144  $\mu$ m. The LISST-100X show distributions as observed in previous experiments at SINTEF, with d<sub>50</sub> = 280  $\mu$ m for oil alone and decreasing droplets down to d<sub>50</sub> = 45  $\mu$ m for DOR 1:25.

A possible explanation for the bimodal distribution and the additional peak at 144  $\mu$ m for the LISST DEEP (figure C19) might be found in the design of the path reduction module (PRM). The LISST-DEEP instrument is not originally delivered with a PRM, but Sequoia developed this on our request. The main difference of the PRM in the LISST-DEEP and 100X is illustrated in figure A1 and A2 in Appendix A. The perforated plastic clip holding the glass prism in place in the LISST-DEEP could interfere with the rising plume of water and oil droplets and create circulation patterns that differentiate different droplet size ranges.

This hypothesis was the only explanation found in 2014 to explain the small droplets for the oil alone experiments at SwRI. This hypothesis was verified by experiments performed in 2015 comparing the LISST DEEP (with/without PRM) with alternative instrumentation (see Figure 4.3).

#### C.5 Conclusions from preliminary investigations

Differences in droplets size data between experiments performed at SINTEF and SwRI for untreated oil are probably caused by discrimination of large droplets in the path reduction module used with the LISST-DEEP.

No significant effect of high pressure (175 bar or 1750 meter depth) was found on dispersant effectiveness (shift in droplet sizes). Similar droplet sizes were measured during experiments at high pressure (SwRI) and at ambient conditions (SwRI and SINTEF) at both 1 and 2% dispersant injection.

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#### **D** Droplet Size data for the figures in the report

Experimental data for figures in the report are given in this appendix. Data for figures in the Appendices C are available at SINTEF.

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#### Ambient experiments March 2015(Experiment 4 - 6. March 2015)

| Condition<br>Comments<br>Nozzle | Low or<br>SIT injo<br>1,50   | Low or ambient pressure experiment with dead oil<br>SIT injection, 1 and 2% C9500<br>1,50 |                     |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
|---------------------------------|--|---|---------------------|------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|--------|
| FlowRate                        | 1,20   |   |                     |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| ID:                             | Uil alone 1 Amb  |   |                     |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Sampling:                       | #Images  | S/Particles: 9  | 01/11/0964          | 4 (60 sec) |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| d <sub>50</sub> :<br>Start:     | 06 03 20   | 015 14.51.1   | 258                 |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Bin mid-siz                     | es (microns  | 2.72  | 3 21                | 3 79       | 4 47  | 5 27  | 6.22  | 7 35  | 8 67  | 10.23 | 12.07 | 14 24 | 16.81 | 19.84 | 23 41 | 27.62 | 32.60 | 38 47 | 45 39  |
| Din ning on                     | 53.57  | 63.21   | 74.60               | 88.03      | 103.9 | 122.6 | 144.7 | 170.7 | 201.4 | 237.7 | 280.5 | 331.0 | 390.7 | 461.0 | 544.0 | 642.0 | 757.6 | 894.0 | 1054.9 |
|                                 | 12445  | 1469  | 1734                | 2046       | 2414  | 2849  | 3362  | 3967  | 4682  | 5525  | 6520  | 7693  | 9079  | 10713 | 12643 | ··-,· | ,.    |       |        |
| Vol. Conc.                      | / bin (%):   | 0,0   | 0,0                 | 0,0        | 0,0   | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,03  | 0,11  | 0,21  | 0,41   |
|                                 | 0,66   | 1,15  | 1,75                | 2,86       | 4,13  | 5,62  | 7,47  | 9,58  | 11,50 | 12,97 | 12,92 | 11,66 | 9,14  | 4,83  | 2,05  | 0,75  | 0,20  | 0,00  | 0,00   |
|                                 | 0,00   | 0,00  | 0,00                | 0,00       | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |       |        |
| ID:                             | DOR 1:10   | 01 Amb  |                     | - \        |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Sampling:                       | ; #Images/raticles: 223/349/848 (1) sec)<br>Cumulative/Peak: 115/104 |   |                     |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| d <sub>50</sub> :<br>Start:     | Cumulativ  | e/Peak: 115<br>5 14-54-11   | /104                |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Vol Conc                        | / hin (%)  | 0   | 0                   | 0          | 0     | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.17  | 0.58  | 1 19  | 2 39   |
| voi. cone.                      | 3.73   | 5.71  | 7.62                | 9.71       | 12.08 | 11.51 | 11.77 | 11.57 | 8,17  | 5.96  | 4.29  | 1.46  | 1.14  | 0.93  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00   |
|                                 | 0,00   | 0,00  | 0,00                | 0,00       | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | -,    | .,    | -,    | -,     |
| ID:                             | DOR 1:50   | 1 Amb   |                     |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Sampling:                       | #Images/P  | articles: 450   | )/2337007 (3        | 30 sec)    |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| d <sub>50</sub> :               | Cumulativ  | e/Peak: 83/   | 104                 |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:                          | 06.03.2015   | 5 14:54:38  | 0.00                | 0.00       | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.04  | 0.44  | 0.04   |
| Vol. Conc.                      | / bin (%):   | 0,00  | 0,00                | 0,00       | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,08  | 0,24  | 0,44  | 0,84   |
|                                 | 1,51   | 2,18  | 3,28                | 5,14       | 0,84  | 9,01  | 11,14 | 12,81 | 12,32 | 0.00  | 10,50 | 7,30  | 2,92  | 1,15  | 0,10  | 0,62  | 0,00  | 0,00  | 0,00   |
| ID:                             | Oil alone  | 2 Amb   | 0,00                | 0,00       | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |       |        |
| Sampling:                       | #Images/P  | articles: 901   | /1651938 (6         | 50 sec)    |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| d <sub>50</sub> :               | Cumulativ  | e/Peak: 158   | /171                |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:                          | 06.03.2015   | 5 14:55:45  |                     |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Vol. Conc.                      | / bin (%):   | 0   | 0                   | 0          | 0     | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,42  | 1,30  | 2,13  | 3,63   |
|                                 | 4,78   | 6,80  | 8,85                | 11,25      | 13,09 | 13,20 | 11,51 | 9,25  | 6,71  | 3,96  | 1,62  | 0,91  | 0,26  | 0,33  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00   |
| ID.                             | 0,00<br>DOD 1-10   | 0,00  | 0,00                | 0,00       | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |       |        |
| ID:<br>Sampling:                | #Images/P  | o 2 Ano<br>articles: 45(  | )/2267155 (3        | 30 sec)    |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| dso.                            | Cumulativ  | e/Peak · 101  | /123                | 50 300)    |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:                          | 06.03.2015   | 5 14:56:57  |                     |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Vol. Conc.                      | / bin (%):   | 0   | 0                   | 0          | 0     | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,42  | 1,30  | 2,13  | 3,63   |
|                                 | 4,78   | 6,80  | 8,85                | 11,25      | 13,09 | 13,20 | 11,51 | 9,25  | 6,71  | 3,96  | 1,62  | 0,91  | 0,26  | 0,33  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00   |
|                                 | 0,00   | 0,00  | 0,00                | 0,00       | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |       |        |
| ID:                             | DOR 1:50   | 2 Amb   | 10046000 (          |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Sampling:                       | #Images/P  | articles: 45  | 1/2340932 (:<br>104 | SU sec)    |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| d <sub>50</sub> .<br>Start:     | 06.03.2014   | 5 14.58.08  | 104                 |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Vol. Conc.                      | / bin (%):   | 0   | 0                   | 0          | 0     | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.84  | 2.53  | 3.94  | 6.30   |
|                                 | 7,60   | 9,91  | 11,60               | 13,02      | 13,00 | 10,94 | 8,06  | 5,39  | 3,59  | 2,12  | 0,87  | 0,29  | 0,01  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00   |
|                                 | 0,00   | 0,00  | 0,00                | 0,00       | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |       |        |
| ID:                             | Oil alone  | 3 Amb   |                     |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Sampling:                       | #Images/P  | articles: 450   | )/580806 (29        | 9 sec)     |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| d <sub>50</sub> :               | Cumulativ  | e/Peak: 145   | /201                |            |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:<br>Vol. Corre            | 06.03.2015   | 0.00  | 0.00                | 0.00       | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.06  | 0.22  | 0.48  | 1.04   |
| voi. Cofic.                     | 1 74   | 2.94  | 4.43                | 6.83       | 8 70  | 9.00  | 13.00 | 13.03 | 14 76 | 11 41 | 6,00  | 3,60  | 0,00  | 0,00  | 0,00  | 0,00  | 0,25  | 0,48  | 1,04   |
|                                 | 0.00   | 2,74  | 4,45                | 0,05       | 0,79  | 9,90  | 0.00  | 0.00  | 0.00  | 0.00  | 0,09  | 0.00  | 0,98  | 0,07  | 0,08  | 0,00  | 0,00  | 0,00  | 0,00   |
|                                 | 0,00   | 5,00  | 0,00                | 0,00       | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |       |        |

API Subsurface dispersant HP testing Phase-III Appendix D - page 57

## **()** SINTEF

#### HP experiments March 2015 (Experiment 3 - 4. March 2015)

| Condition         | High pi           | ressure exp           | eriment wi           | th dead oil |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
|-------------------|-------------------|-----------------------|----------------------|-------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|--------|
| Nozzle            | 1.50              | ection, 1 ai          | IU 276 C950          |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| FlowRate          | 1,20              |                       |                      |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Sampling:         | #Images/P         | articles: 45          | 0/131239 (3          | 0 sec)      |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| d <sub>50</sub> : | Cumula            | tive/Peak: 2          | 200/238              |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:            | 04.03.2           | 015 19:25:4           | 1                    |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Bin mid-siz       | zes (microns      | s): 2,72              | 3,21                 | 3,79        | 4,47  | 5,27  | 6,22  | 7,35  | 8,67  | 10,23 | 12,07 | 14,24 | 16,81 | 19,84 | 23,41 | 27,62 | 32,60 | 38,47 | 45,39  |
|                   | 53,57             | 63,21                 | 74,60                | 88,03       | 103,9 | 122,6 | 144,7 | 170,7 | 201,4 | 237,7 | 280,5 | 331,0 | 390,7 | 461,0 | 544,0 | 642,0 | 757,6 | 894,0 | 1054,9 |
| W1G               | 12445             | 1469                  | 1734                 | 2046        | 2414  | 2849  | 3362  | 3967  | 4682  | 5525  | 6520  | 7693  | 9079  | 10713 | 12643 | 0.02  | 0.00  | 0.14  | 0.24   |
| Vol. Conc.        | / bin (%):        | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,03  | 0,09  | 0,14  | 0,34   |
|                   | 0,76              | 1,25                  | 1,50                 | 2,72        | 4,00  | 0,30  | 8,35  | 10,34 | 15,05 | 16,59 | 15,09 | 8,33  | 4,25  | 3,85  | 0,96  | 0,00  | 0,00  | 0,00  | 0,00   |
| т.                | 0,00<br>DOD 1.10  | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |        |
| Sampling:         | #Images/P         | articles: 45          | 0/3/9808 (3          | () sec)     |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| deo:              | Cumulativ         | e/Peak: 107           | 0/349000 (3<br>7/145 | 0 300)      |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:            | 04 03 201         | 5 19·28·40V           | /ol                  |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Conc. / bin       | (%):              | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,20  | 0,67  | 1,02  | 2,13   |
|                   | 4,21              | 6,01                  | 7,92                 | 11,30       | 13,97 | 14,01 | 15,42 | 13,15 | 6,36  | 2,64  | 0,39  | 0,56  | 0,06  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00   |
|                   | 0,00              | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |        |
| ID:               | DOR 1:50          | ) 1 HP                |                      |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Sampling:         | #Images/P         | articles: 45          | 1/2774582 (          | 30 sec)     |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| d <sub>50</sub> : | Cumulativ         | e/Peak: 82/           | 104                  |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:            | 04.03.201         | 5 19:30:33            |                      |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Vol. Conc.        | / bin (%):        | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,77  | 2,36  | 3,65  | 5,88   |
|                   | /,34              | 9,77                  | 11,64                | 13,14       | 13,41 | 12,12 | 9,07  | 5,92  | 3,05  | 1,36  | 0,45  | 0,07  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00   |
| т.                | 0,00<br>Oil alana | 0,00<br><b>2 HD</b>   | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |        |
| Sampling          | #Images/P         | 2 III<br>articles: 45 | 1/1249717 (          | 30 sec)     |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| deo:              | Cumulativ         | e/Peak · 164          | 1/1249717 (<br>1/201 | 30 300)     |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:            | 04.03.201         | 5 19:33:00            |                      |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Vol. Conc.        | / bin (%):        | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,08  | 0,27  | 0,50  | 0,93   |
|                   | 1,37              | 2,30                  | 3,45                 | 4,85        | 7,18  | 9,02  | 10,37 | 11,95 | 12,50 | 11,42 | 9,31  | 7,23  | 4,07  | 2,46  | 0,66  | 0,07  | 0,00  | 0,00  | 0,00   |
|                   | 0,00              | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |        |
| ID:               | DOR 1:10          | 0 2 HP                |                      |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Sampling:         | #Images/P         | articles: 45          | 0/2696144 (          | 30 sec)     |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| d <sub>50</sub> : | Cumulativ         | e/Peak: 93/           | 104                  |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:            | 04.03.201         | 5 19:34:22            | 0.00                 | 0.00        | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.52  |       | 2 60  | 1.00   |
| Vol. Conc.        | / bin (%):        | 00,00                 | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,53  | 1,65  | 2,68  | 4,39   |
|                   | 5,79              | 8,10                  | 10,16                | 12,41       | 14,11 | 13,93 | 11,92 | 8,06  | 3,82  | 1,95  | 0,41  | 0,10  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00   |
| т.                | 0,00<br>DOD 1.50  | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |       |        |
| Sampling:         | #Images/P         | articles: 45          | 1/2764136 (          | 30 sec)     |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| deo:              | Cumulativ         | e/Peak · 82/          | 1/2/04150 (          | 50 300)     |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:            | 04.03.201         | 5 19:35:41            | 10.                  |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Vol. Conc.        | / bin (%):        | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,78  | 2,36  | 3,68  | 5,90   |
|                   | 7,35              | 9,85                  | 11,72                | 13,53       | 14,26 | 12,10 | 9,06  | 5,50  | 2,65  | 0,96  | 0,29  | 0,01  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00   |
|                   | 0,00              | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |       |        |
| ID:               | Oil alone         | 3 HP                  |                      |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Sampling:         | #Images/P         | articles: 45          | 0/2057086 (          | 30 sec)     |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| d <sub>50</sub> : | Cumulativ         | e/Peak: 136           | 5/171                |             |       |       |       |       |       |       |       |       |       |       |       |       |       |       |        |
| Start:            | 04.03.201         | 5 19:37:16            | 0.00                 | 0.00        | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.00  | 0.15  | 0.40  | 0.02  | 1.50   |
| Vol. Conc.        | / bin (%):        | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,15  | 0,48  | 0,82  | 1,50   |
|                   | 2,10              | 3,51                  | 4,91                 | 6,92        | 9,17  | 11,61 | 12,96 | 13,05 | 11,73 | 9,17  | 6,26  | 5,06  | 1,/1  | 0,60  | 0,22  | 0,00  | 0,00  | 0,00  | 0,00   |
|                   | 0,00              | 0,00                  | 0,00                 | 0,00        | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  | 0,00  |       |       |       |        |

API Subsurface dispersant HP testing Phase-III Appendix D - page 58





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