FINAL REPORT

EXAMINING THE FATE OF EMULSION BREAKERS USED FOR DECANTING

by

SL Ross Environmental Research Ltd.
Ottawa, ON

for

Minerals Management Service
Technology Assessment and Research Division
Herndon, VA

and

Alaska Clean Seas
Prudhoe Bay, AK

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The project described in this report was funded by the U.S. Minerals Management Service (MMS) through Purchase Order 0103PO73457.

SUMMARY

In U.S. Territorial waters mechanical recovery (using containment booms and skimmers) is the preferred method used to clean up oil spills. Skimmers operating in waves often recover a large amount of water, both in the form of emulsions and free water. Recovered water dramatically reduces the temporary storage capacity available for oily fluids offshore. This report describes the latest study of an ongoing, multi-year program to research decanting of water from recovered oil spill fluids offshore.

The objective of this study was to research the partitioning of emulsion breakers injected into an oil spill recovery system at both lab-scale (at the SL Ross Environmental Research wave tank in Ottawa, ON) and mid-scale (at Ohmsett, the National Oil Spill Response Test Facility in Leonardo, NJ). The experiments were designed to simulate the conditions in an offshore oil spill recovery operation. The ability of emulsion breaker addition to reduce water contents of the recovered fluid and the effects of demulsifier addition of the oil content of decanted water were also assessed. The efficiency of emulsion breaking chemicals in resolving water-in-oil emulsions is highly parent oil/surfactant specific. The results are strictly valid only for the combinations of demulsifiers (Alcopol O 70% PG, Breaxit OEB-9, Exxon Nalco EC 2085 and Unichem RNB 60425) and emulsions used (50% salt water in either a blend of 80% Hydrocal 300/5% No. 6 Fuel Oil/15% diesel, or fresh Endicott crude).

The formation of micelles by the surfactants in the water at high concentrations and the resulting limitations of the analytical technique used to measure the concentration of the demulsifiers in the decanted water make definitive conclusions about the partitioning of the demulsifier between oily and water phases impossible. The following general conclusions could be made:

- A large fraction of the demulsifier injected into the recovered fluid stream appears to end up in the decanted water.
- The concentrations of demulsifier in the decanted water are well in excess of 100 ppm and could be as high as in the 1000's of ppm.

The use of a demulsifier injected into a recovery system, combined with decanting, substantially reduced the volume of water in temporary storage tanks and the water content of emulsions for disposal/recycling. The efficacy of the demulsifier was a strong function of free water content: if the free water content exceeded approximately 55%, the effect of the surfactant was substantially reduced.

The degree of emulsion breaking achieved increased with increasing mixing energy applied to the fluid. Increasing the flow rate (and hence turbulence level) and increasing the length of the flow path both resulted in increased emulsion breaking.

Primary break occurred in only a few minutes (2 to 5 in the lab tests, less than 30 for the Ohmsett tests). The application of demulsifier did not appear to affect the time required. The Ohmsett results indicated that the use of a demulsifier increased oil droplet concentrations in the decanted water by approximately a factor of two.

The implication of this research for oil spill response is that it may be possible to greatly reduce downtime for offshore skimming operations caused when the available temporary storage systems are filled with fluids containing large amounts of water. The legislated requirements for onsite temporary storage systems could also ultimately be reduced by the use of these results, resulting in considerable savings in operating and disposal costs for Oil Spill Response Organizations (OSROs). Knowing that the separated water can be decanted quickly will optimize onsite recovery operations and greatly reduce the volume of fluids requiring disposal. In fact, the removal of most of the free and emulsified water from the recovered product would greatly enhance the likelihood that it could be recycled, as opposed to requiring disposal. The Net Environmental Benefit of using demulsifiers and decanting water offshore should be addressed

ACKNOWLEDGEMENTS

The project described in this report was funded by the U.S. Minerals Management Service (MMS) through Purchase Order 0130PO73457. Alaska Clean Seas also provided major funding for the work. The authors of this report were Ian Buist and Alun Lewis. The author would like to acknowledge the contributions of Mr. Bob Smith and Mr. Joe Mullin, with MMS, Mr. Lee Majors and Mr. Ken Linderman with Alaska Clean Seas, the other staff of Alaska Clean Seas, and the staff of MAR, Inc. who operate Ohmsett, particularly Alan Guarino and Sue Cunneff.

DISCLAIMER

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1. INTRODUCTION

In 2003 the Minerals Management Service (MMS) and Alaska Clean Seas (ACS) jointly funded a research program to examine the fate of chemical emulsion breakers (also known as demulsifiers) when they are injected into a recovered fluid stream from a skimmer used in open water containment and recovery operations. The demulsifiers are used to aid in decanting of recovered emulsified water to conserve temporary storage capacity. The primary objective was to quantify the amount of the emulsion breaker that ends up in the decanted water, and how much stays with the oily phase.

1.1 Background

The preferred approach to cleaning up an offshore oil spill is to contain and thicken the oil slick(s) with booms and then place skimmers in the oil or emulsion to recover it. The recovered fluids are placed in temporary storage containers for transfer to larger storage vessels or for direct input into waste recycling and disposal systems. The most common type of high-capacity skimmer in use today is the weir skimmer. These skimmers often recover a large amount of water, both in the form of emulsified water and free water, when operating in waves. In some cases, the transfer pump built into the skimming system can impart enough energy to cause additional emulsification of the recovered fluids. The problem is that the recovered water (both emulsified and free) dramatically reduces the temporary storage space available at the site of skimming operations; this can result in having to stop skimming prematurely when the storage capacity is reached and having to wait until empty, temporary storage containers arrive at the response site.

Over the last six years a series of lab-scale and mid-scale tests with and without the use of emulsion breakers were completed that give some quantitative insight into the oil/water separation processes occurring in temporary storage devices (SL Ross 1998, 1999 and 2002). The objective of these earlier tests was to determine the optimum time to decant the water and maximize the available on-site storage space during a skimming operation as well as the efficacy of adding emulsion breakers into the recovery stream to allow decanting of emulsified water. The results indicated that “primary break” (the initial separation of the recovered fluid into a layer containing most of the oil and a layer containing most of the free water) occurred within a few minutes to one hour, depending on the physical characteristics of the oil. Rapidly decanting this free water layer, in appropriate situations, produced immediate increases of 200 to 300% in available temporary storage space. The addition of emulsion breakers increased the amount of water that could be decanted, in the same time frame. Addition of the emulsion breaker increased the oil content of the separated water significantly. During the last decanting experiments at Ohmsett using emulsion breakers (SL Ross 2002), the separated water foamed easily when agitated, providing strong qualitative evidence that it contained significant amounts of surfactant.

A significant potential impediment to the application of emulsion breakers to extend temporary storage capacity is the ultimate fate of the emulsion breaking chemical(s). If the demulsifier remains with the oil, there should be no problem with their use; however, if demulsifier components partition significantly into the separated water, they will be discharged into the environment when the water is decanted.

Demulsifiers are surface-active, or surfactant, chemicals that can be added to ‘break’ or ‘resolve’ the emulsion back into separate oil and water phases. Demulsifiers function by destabilizing or disrupting the film of precipitated asphaltenes and/or resins that are known to stabilize water-in-oil emulsions. For a demulsifier to function effectively, it must be able to come into intimate contact with the oil-water interface around the water droplets in emulsified oil. The surfactant chemicals within a demulsifier therefore need to be introduced into the emulsified oil and thoroughly mixed with it.

Being surfactants, the active ingredients of demulsifiers are not truly soluble in either water or oil; the minimum surface free energy is achieved when the surfactant molecules are orientated at an oil/water interface. This property results in their surface-active nature. The molecules of surfactants can orientate into “micelles” or “reverse micelles” to accommodate their dissolution in either water or oil. These are less preferred arrangements than orientation at an interface, but it is critical to the behaviour of these chemicals. It is therefore possible for surfactants to be present in bulk in either the water or oil phases, as well as at the oil/water interface. This tendency is known as ‘partitioning’. Of course, if a demulsifier is effective, it greatly reduces the amount of oil/water interface originally in a water-in-oil emulsion, and much of the surfactant would move back into the bulk liquid phases. The proportion of surfactant that will be present in the oil or water phases depends on the relative proportion of oil and water phases that are available for them to be dissolved in as well as the surface-active properties of the demulsifier itself.

The use of surfactants in demulsifiers for breaking recovered emulsified oils is therefore quite complex. The surfactants in demulsifiers are normally in the form of a concentrated solution blended in a solvent. The solvent in the blend allows the surfactants to transfer into the emulsified oil (in an oil spill emulsion the oil is the continuous phase that contains droplets of water). In the inevitable presence of free water during oil recovery operations some surfactant may move directly into the free water and will not perform its intended function of breaking the emulsion. This tendency can be minimised if the proportion of free water is kept to a minimum. The transfer of surfactants into the emulsified oil can be difficult because of the highly viscous nature of many emulsified oils. Once inside the bulk of the emulsified oil, the surfactants need to be able to contact the oil/water interface at the surface of the entrained water droplets. Some surfactant may orientate to form reverse micelles within the oil – this is effectively ‘lost’ from the emulsion-breaking process unless mechanical agitation introduces it to the oil/water interface.

The surfactants within demulsifiers can therefore partition into any of the phases that they may encounter during spilled oil recovery:

- Into the free water
- Into the oil phase
- Into the emulsified water phase that is subsequently separated by gravity

If the bulk of the surfactants in the demulsifier remain with the oil, there should be no problem with their use; the recovered oil will be collected and disposed of. However, if the majority of the surfactants partition into the separated water (either initially free or emulsified water), they will be discharged into the environment if the separated water is decanted overboard. Some partitioning is an inevitable consequence of surfactant behaviour. The relative tendency to partition, either as individual molecules or as micelles and reverse micelles between oil and water is very dependent on molecular structure.

Some demulsifiers, such as sodium diisooctyl sulfosuccinate, (the active ingredient in Alcopol, aka Drimax) are strong ionic surfactants that have a relatively high toxicity to some marine organisms. If a recovered fluid consists of 50% free water and 50% of an emulsion containing 75% water and all the emulsion breaker used to treat it (typically dosed at 1:400 demulsifier:recovered fluids) transfers into the water, the decanted water could contain some 1400 ppm of demulsifier. Discharge regulations in some jurisdictions would not permit the decanting of such water to the ocean in normal circumstances. Other demulsifiers, such as the EO/PO (ethylene oxide/propylene oxide) coplymers are non-ionic, and tend to be much less toxic.

Some emulsions are easier to break with ionic surfactants, and some are easier to break with non-ionic surfactants. The environmental consequences of demulsifier use will depend on:

- Their effectiveness in breaking emulsions
- Their partitioning behaviour into the different water and oil phases
- Their toxicity to marine organisms
- The potential for dilution of the decanted water in the receiving water body

The intention of this study was to research the partitioning of different emulsion breakers injected into a recovery system at both lab-scale (at SL Ross) and mid-scale (at Ohmsett). A series of small-scale tests with a scale-model piping system simulating a weir skimmer recovery system (used in the previous decanting study – SL Ross 2002) was completed in the summer of 2003 to determine the effects of several variables on the concentration of demulsifier in decanted water. In addition, a technique for determining the concentration of demulsifier in the decanted water was perfected. In the fall of 2003 a series of mid-scale experiments was conducted at Ohmsett.

1.2 Objective

The objective of the proposed study was to determine the partitioning of different chemical emulsion breakers between oily and water phases when they are used to enhance decanting of recovered water from offshore skimming operations.

2. LAB-SCALE TESTING

This section describes the development of an analytical test to measure the concentration of demulsifiers in water, the decanting laboratory test apparatus and results. The tests were conducted at the SL Ross laboratory in Ottawa, ON.

2.1 Analytical Test for Demulsifier in Water

Prior to carrying out the small-scale research program in the laboratory, it was necessary to develop a simple, inexpensive test to measure the concentration of demulsifier in decanted water. The approach taken was to adapt a technique developed to measure the concentration of dispersants in Ohmsett tank water (SL Ross 2003). This method involves measuring the interfacial tension between a highly refined mineral oil (USP, or pharmaceutical grade) and the water containing the surfactant using a DuNouy ring apparatus (ASTM –D971). The interfacial tension value obtained is compared to a plot of interfacial tension vs. concentration of prepared aqueous solutions of the demulsifier in question to obtain an estimate of the concentration of the demulsifier.

Figures 2-1 and 2-2 show the calibration curves prepared for the four demulsifiers considered for use in the lab-scale tests. Although the interfacial measurement technique gives a reasonable fit of the data for most of the demulsifiers to a power law relationship of the form:

$$\mathbf{Concentration = C_1(IFT)^{C_2}} \qquad \mathbf{(1)}$$

Where: C_1 and C_2 are demulsifier-specific constants

It is clear from Figure 2-1 that the relationships will not give very accurate results at concentrations of demulsifier above about 100 ppm. This is because there is very little change in interfacial tension with a large change in demulsifier concentration above this point, most likely due to the fact that the demulsifier has exceeded its Critical Micelle Concentration (CMC) and the oil/water interface is saturated with surfactant molecules at demulsifier concentrations. A difference of only 0.3 dynes/cm in interfacial tension in the 1.5-dyne/cm range (the DuNouy ring is a notoriously finicky apparatus to use and repeatability at this level would be quite good) results in a 300+ ppm difference in calculated demulsifier concentration.

Despite its shortcomings, the interfacial tension technique was used as the analytical method for determining the concentrations of demulsifier in the decanted water for this study. This was primarily because the other available techniques (High Pressure Liquid Chromatography [HPLC], complex titrations, etc.) are very expensive and time consuming.

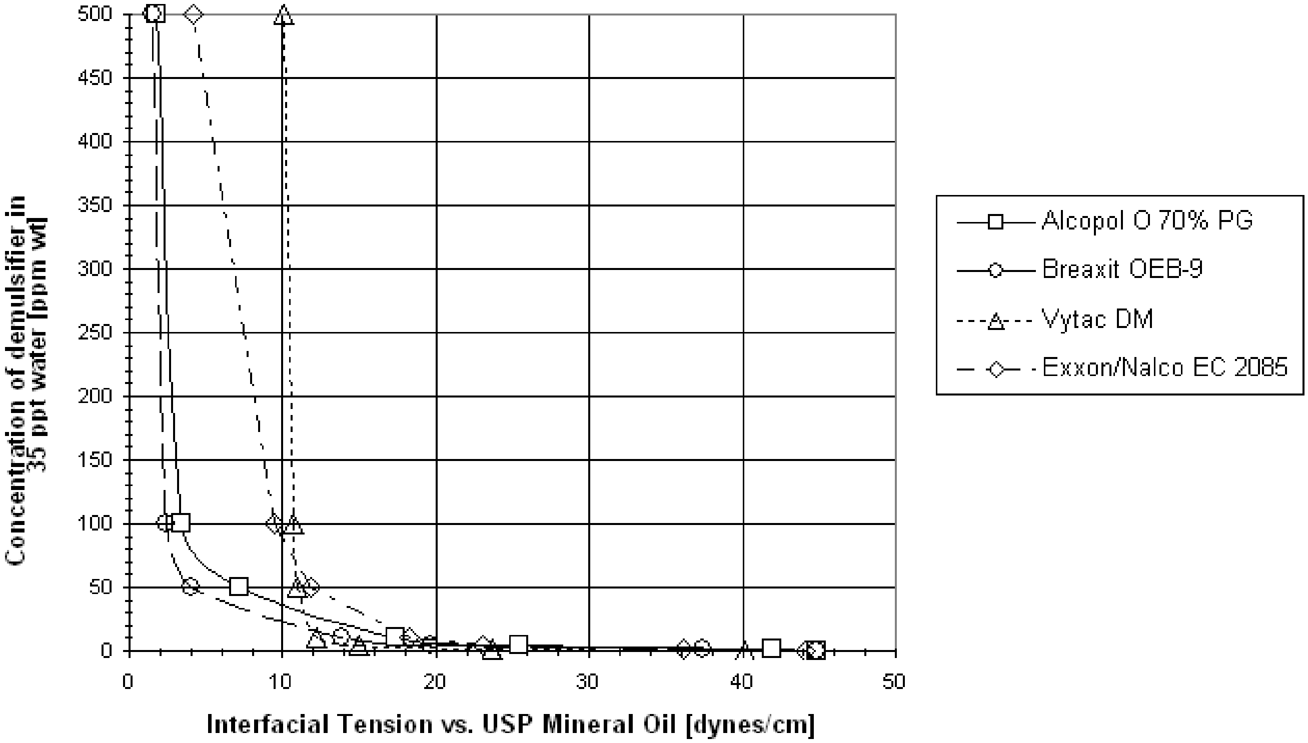


Figure 2-1. Calibration curve of interfacial tension vs. demulsifier concentration.

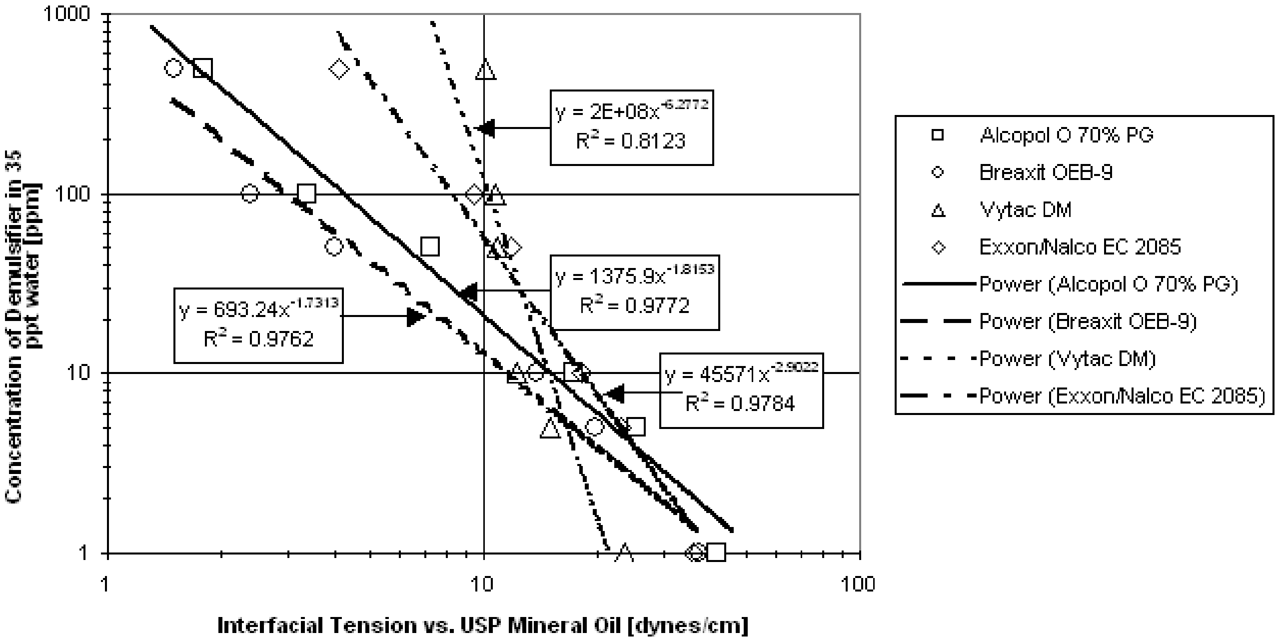


Figure 2-2. Least-squares fits to power law relationship for various demulsifiers.

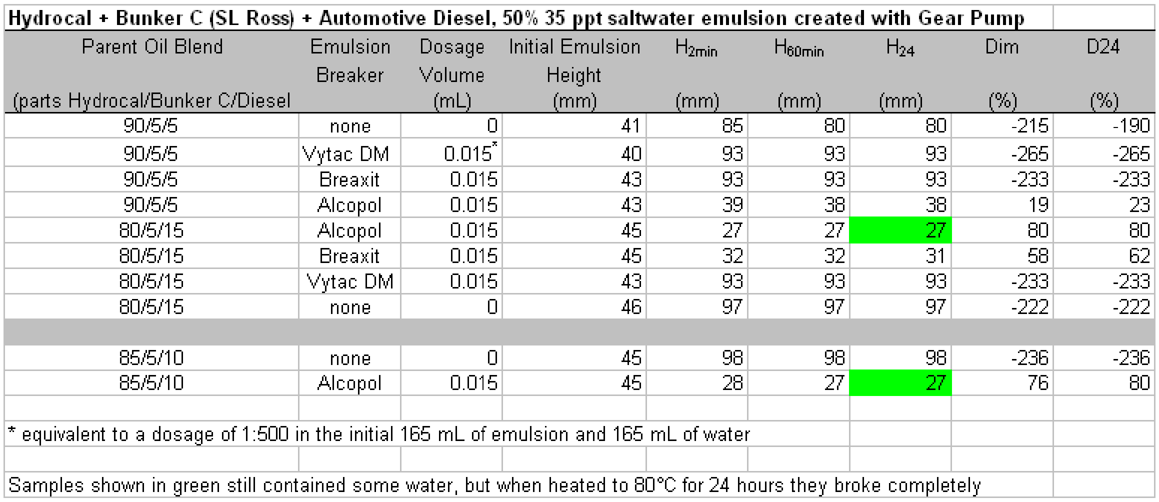
2.2 Parent Oil Blend for Emulsions

In the previous series of tests using demulsifiers (SL Ross 2002) it was observed that the demulsifiers could not completely resolve the emulsions created using a blend of 95% Hydrocal and 5% No.6 fuel oil (to add asphaltenes). This was presumed to be because this parent oil contained no aromatic compounds (Hydrocal is a de-aromatized lube stock) to act as a sink for the asphaltenes displaced from the water/oil interface by the demulsifier surfactant. As such, a series of emulsion stability tests with various mixtures of Hydrocal, No. 6 Fuel Oil (2.5 or 5 % by volume) and automotive diesel (5, 10 or 15% by volume) were conducted to select a mixture that would form a stable, 50 % salt water emulsion that could be completely resolved by the demulsifiers to be used.

Full results are contained in Appendix A. Figure 2-3 shows the stability test results (fraction of oil not creamed out of the emulsion) over four day settling periods for emulsions created using a high-speed hand blender and the gear pump used to create the emulsions for the tests (see Section 2.3 below). Only the 90/5/5 (Hydrocal/No. 6/diesel) parent oil created by the hand blender met the standard criteria for a stable emulsion, but all three emulsions created with the gear pump met the criteria.

Table 2-1 shows the results of the emulsion breaker effectiveness tests (the method of Hokstad *et al.* 1993 was used) with a variety of demulsifiers on emulsions created with the gear pump using the three blends of parent oils that passed the stability test discussed above.

Table 2-1. Demulsifier effectiveness tests with four emulsion breakers and three oils.



Hydrocal + Bunker C (SL Ross) + Automotive Diesel, 50% 35 ppt saltwater emulsion created with Gear Pump

Parent Oil Blend (parts Hydrocal/Bunker C/Diesel	Emulsion Breaker	Dosage Volume (mL)	Initial Emulsion Height (mm)	H_{2min} (mm)	H_{60min} (mm)	H_{24} (mm)	Dim (%)	D24 (%)
90/5/5	none	0	41	85	80	80	-215	-190
90/5/5	Vytac DM	0.015*	40	93	93	93	-265	-265
90/5/5	Breaxit	0.015	43	93	93	93	-233	-233
90/5/5	Alcopol	0.015	43	39	38	38	19	23
80/5/15	Alcopol	0.015	45	27	27	27	80	80
80/5/15	Breaxit	0.015	45	32	32	31	58	62
80/5/15	Vytac DM	0.015	43	93	93	93	-233	-233
80/5/15	none	0	46	97	97	97	-222	-222
85/5/10	none	0	45	98	98	98	-236	-236
85/5/10	Alcopol	0.015	45	28	27	27	76	80

* equivalent to a dosage of 1:500 in the initial 165 mL of emulsion and 165 mL of water

Samples shown in green still contained some water, but when heated to 80°C for 24 hours they broke completely

Based on the stability results and the demulsifier effectiveness tests, the parent oil blend was selected to be 80% Hydrocal, 5% No. 6 Fuel Oil (aka Bunker C) and 15 % automotive diesel. For some tests, emulsion created using fresh Endicott crude (which met the stability criteria), from Alaska, was also used. The three demulsifiers selected for testing in the lab-scale tests were: Alcopol O 70% PG (aka Drimax), Breaxit OEB-9 and Exxon Nalco EC2085, an older product specifically blended as a generic production emulsion breaker for Alaska North Slope crudes.

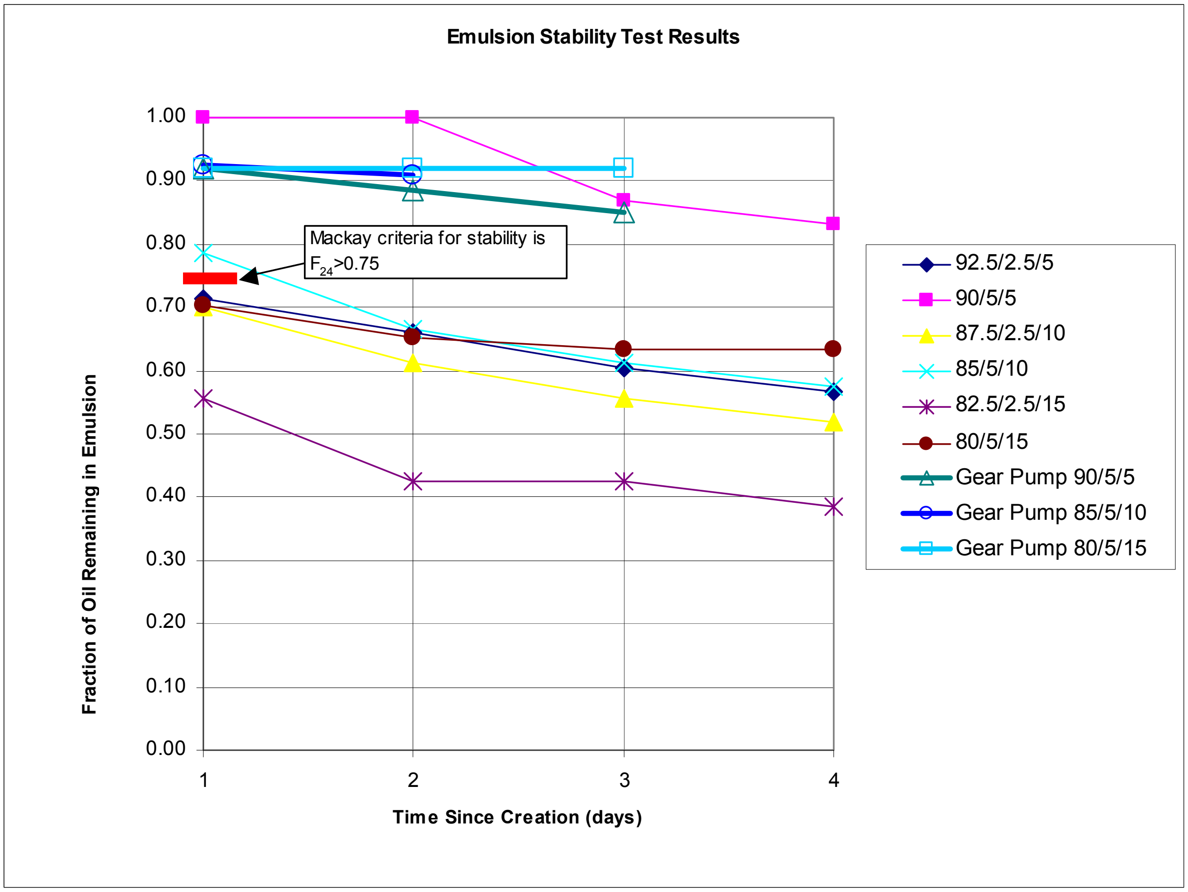


Figure 2-3. Emulsion stability test results for 50% salt water emulsions with various parent oil blends.

2.3 Laboratory Test Methods

The apparatus and most of the procedures used in the laboratory tests were the same as those used in the previous decanting tests with demulsifiers and are described in detail in that report (SL Ross 2002). The following is a brief summary.

2.3.1 Test Loop

The lab-scale test system schematic is given in Figure 2-4. A photograph of the setup is shown in Figure 2-5. The scale-model piping system was designed to mimic the pumping, mixing and flow processes that occur in an offshore oil recovery system. Pre-mixed 50% emulsion and nominally 50% free water were pumped separately, at measured, pre-determined rates, to the suction of the progressing cavity pump, representing the pump type used in most weir skimmers. The fluid was directed through a static in-line mixer, down either a 6-foot or 36-foot length of ½” ID plastic tubing and then to six cylindrical receiving tanks where samples were taken at different intervals to characterize the separation of the aqueous phase and the dehydration of the emulsion. Demulsifier was injected, at different dosages, into the system before the main pump using a chemical metering pump.

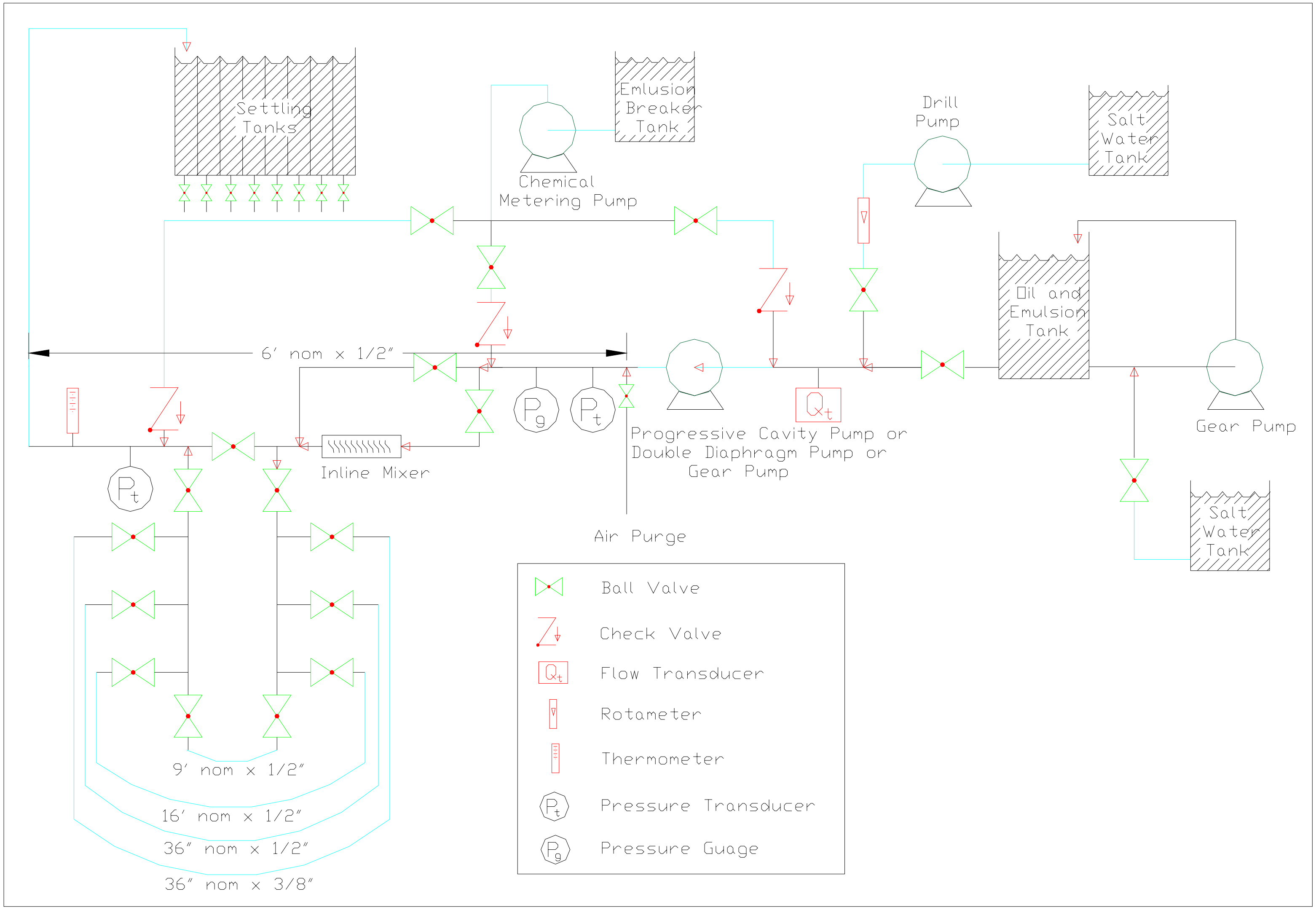


Figure 2-4. Schematic of laboratory scale-model piping layout

2.3.2 Test Procedures

1. Mix enough salt water for emulsion formation and free water injection
2. Mix emulsion
   - Add Hydrocal, No. 6 and diesel separately to emulsion tank
   - Add salt water to blend tank
   - Recirculate oil with gear pump
   - Bleed in water slowly (approximately 2 L of salt water per minute)
   - Measure gear pump output rate by timing fill of bucket and adjust to desired flow rate with valve
3. Open valves
4. Start chemical metering pump (if using)
5. Start water pump
6. Start main pump
7. Start emulsion pump
8. Discharge fluid into waste tank until fluid appears consistent
9. Discharge into sample tanks; start stopwatch; record time of day
10. Fill tanks to 5 L or 2.5 L, depending on free water content; note time on stopwatch when each tank is filled
11. When 6th tank is filled, direct discharge to waste tank
12. Stop main pump, emulsion pump, water pump and chemical pump
13. Decant water from sample tanks into graduated pitchers and record volume after appropriate settling times (2, 5, 10, 15, 30 and 60 minutes); take 30 mL samples of water from all tanks for IFT analysis
14. After decanting, mix oil remaining in sample tanks with spatula
15. Withdraw 20 mL with syringe and transfer to 30 mL glass bottle for water content analysis
16. Empty tanks in preparation for next test

2.3.3 Emulsion Sample Analysis

The emulsion samples in 30 mL glass vials withdrawn from each tank were treated with a few drops of Alcopol emulsion breaker, shaken vigorously, and then placed in a constant temperature bath at 80°C for at least 24 hours to separate. The vials were then removed from the bath, wiped and the heights of water and oil in the vials measured with a steel rule. The water content of the emulsion remaining after decanting could then be estimated.

2.3.4 Demulsifier Concentration in Decanted Water

The water samples taken from each recovery tank were subjected to the interfacial tension test described in Section 2.1 to estimate the concentration of demulsifier that they contained.

Figure 2-5. Photograph of laboratory scale-model piping system.

2.4 Laboratory Scale-model Test Results

A total of 25 test runs were completed using the laboratory scale model piping setup. The complete results may be found in Appendix B. The matrix of test variable target values is given in Table 2-2.

Table 2-2. Matrix of laboratory test variable targets.

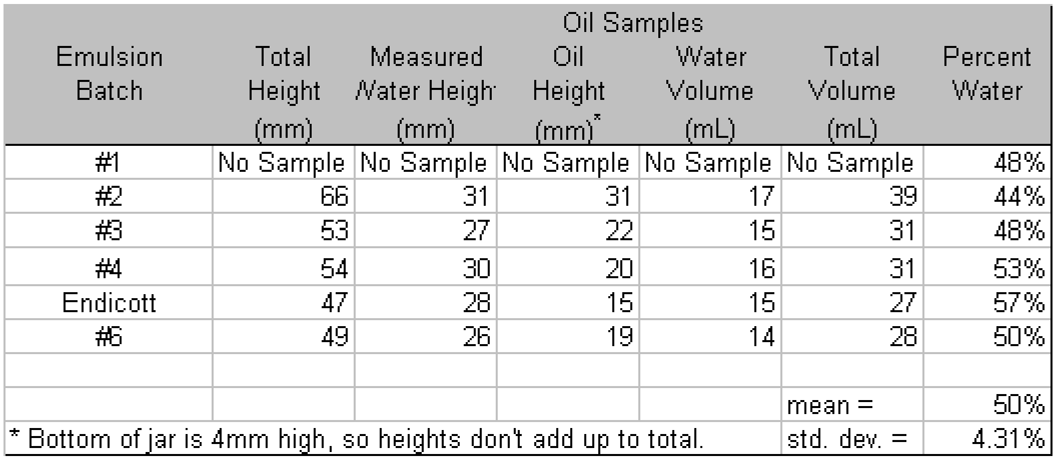
Test Number	Oil	Free Water (%)	Breaker	Dose Rate	Inline Mixer (Y/N)	Circuit Length (ft)
1	Hydrocal 80/5/15	50	None	0	Yes	36
2	Hydrocal 80/5/15	50	Alcopol	High	Yes	36
3	Hydrocal 80/5/15	50	Alcopol	High	Yes	36
4	Hydrocal 80/5/15	33	Alcopol	Low	Yes	36
5	Hydrocal 80/5/15	33	Alcopol	High	Yes	36
6	Hydrocal 80/5/15	50	Alcopol	High	Yes	36
7	Hydrocal 80/5/15	50	Alcopol	Low	Yes	36
8	Hydrocal 80/5/15	66	Alcopol	Low	Yes	36
9	Hydrocal 80/5/15	50	Alcopol	High	Yes	36
10	Hydrocal 80/5/15	50	Alcopol	High	Yes	6
11	Hydrocal 80/5/15	50	Alcopol	Low	Yes	6
12	Hydrocal 80/5/15	0	Alcopol	Low	Yes	6
13	Hydrocal 80/5/15	0	Alcopol	High	Yes	6
14	Hydrocal 80/5/15	66	Alcopol	High	Yes	36
15	Hydrocal 80/5/15	66	Alcopol	Low	Yes	36
16	Hydrocal 80/5/15	50	Exxon Nalco	High	Yes	36
17	Hydrocal 80/5/15	50	Exxon Nalco	Low	Yes	36
18	Hydrocal 80/5/15	0	Exxon Nalco	High	Yes	6
19	Endicott	50	Exxon Nalco	High	Yes	36
20	Endicott	50	Exxon Nalco	Low	Yes	36
21	Endicott	50	Alcopol	High	Yes	36
22	Hydrocal 80/5/15	50	Breaxit	Low	Yes	36
23	Hydrocal 80/5/15	50	Breaxit	High	Yes	36
24	Hydrocal 80/5/15	0	Breaxit	Low	Yes	36
25	Hydrocal 80/5/15	0	Breaxit	High	Yes	36

The tests were conducted with: two parent oils (the 80/5/15 Hydrocal/No. 6/diesel blend and fresh Endicott crude); free water contents ranging from 0 to 66%; and, three demulsifiers (Alcopol, Breaxit and Exxon Nalco) at two dosages (low or high). All runs involved flow through the inline mixer, and the 36-foot tubing circuit was used for most.

2.4.1 Emulsion Water Content

Table 2-3 shows the water contents measured for the six batches of emulsion created for the tests. All were close to the target of 50% water by volume. It is more likely that the variation between batches is due to the inherent error in the water content determination, than an actual variation in the emulsion water content of the batch.

Table 2-3. Water content of emulsion batches* for laboratory tests.



	Oil Samples					
Emulsion Batch	Total Height (mm)	Measured Water Heigh (mm)	Oil Height (mm)*	Water Volume (mL)	Total Volume (mL)	Percent Water
#1	No Sample	No Sample	No Sample	No Sample	No Sample	48%
#2	66	31	31	17	39	44%
#3	53	27	22	15	31	48%
#4	54	30	20	16	31	53%
Endicott	47	28	15	15	27	57%
#6	49	26	19	14	28	50%
					mean =	50%
* Bottom of jar is 4mm high, so heights don't add up to total.					std. dev. =	4.31%

* Note that the water content for Batch #1 comes from the results from Test 1.

2.4.2 Test Results

The complete data set for each test can be found in Appendix B. The ability of emulsion breaking chemicals to resolve water-in-oil emulsions is highly parent oil/surfactant specific. The results are strictly valid only for the combinations of demulsifiers (Alcopol O 70% PG, Breaxit OEB-9 and Exxon Nalco EC 2085) and emulsions used (50% salt water in either a blend of 80% Hydrocal 300/5% No. 6 Fuel Oil/15% diesel, or fresh Endicott crude).

Table 2-4 shows the full results obtained for Test 1, with no demulsifier injected, and Table 2-5 shows the results for Test 3, a run with Alcopol injected at 2654 ppm (a dose rate of 1 part demulsifier in 375 parts of fluid) into the suction side of the progressing cavity pump with a free water injection rate of approximately 50%. The cells with the shading are those into which data from the test was entered.

The results for Test 1 show that, without demulsifier, only free water is decanted, and that the water content of the emulsion remains approximately 50%. The IFT measurements on the water samples show that there are negligible amounts of demulsifier present in the decanted water.

The results for Test 3 show the effects of demulsifier addition. More water is decanted than free water was injected, and the water content of the remaining emulsions is greatly reduced. The emulsion dehydration (the percent reduction in the volume of water in the emulsion[1]) ranges from 45% in the early samples to 63% in the 60-minute sample (i.e.,

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[1] A reduction in emulsion water content from 50% to 20% equates to an emulsion dehydration of 60%

Table 2-4. Results for Test 1

T Test #1

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hy Hydrocal 80/5/15	No	0	0	54%	Yes	36 ft	0

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	1200	1550	3450	No Sample	No Sample	No Sample	No Sample	0	58	No Sample	17.0	8.03	
2	5	5000	1200	1550	3450	No Sample	No Sample	No Sample	No Sample	0	58	No Sample	No Sample	No Sample	
3	10	4800	1000	1350	3450	No Sample	No Sample	No Sample	No Sample	0	52	No Sample	39.0	1.78	
4	15	5000	1150	1500	3500	No Sample	No Sample	No Sample	No Sample	0	56	No Sample	No Sample	No Sample	
5	30	5000	1250	1600	3400	59	30	25	48%	0	59	4.0	31.1	2.68	
6	60	5000	1150	1500	3500	42	22	16	48%	0	56	3.1	34.3	2.25	
									average:	0	56	3.5			
									minimum:	0	52	3.1			
									maximum:	0	59	4.0			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
67	7.1	3.8	4.0	2.0	5.8

Table 2-5. Results for Test 3

Test #3 (redo of #2)

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	70	0.015491823	48%	Yes	36 ft	2654

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	4588	2200	2550	2037.5	41	13	24	26%	29	116	48.2	2.5	260.74	Filled to 2.5cm low
2	5	5000	2450	2800	2200	39	13	22	27%	31	117	45.3	2.1	357.82	
3	10	4835	2500	2850	1985	42	12	26	23%	42	123	54.7	1.8	473.36	Filled to 1cm low
4	15	4670	2500	2850	1820	35	11	20	25%	50	127	51.0	1.8	473.36	filled to 2 cm low
5	30	5000	2500	2850	2150	39	11	24	22%	35	119	56.5	2.1	357.82	
6	60	5000	2600	2950	2050	40	10	26	18%	42	123	63.1	1.9	429.11	No further sep'n after 24 hours for Tank 6
									average:	38	121	53.1			
									minimum:	29	116	45.3			
									maximum:	50	127	63.1			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
79	5.8	2.8	4.0	2.0	4.8

the final water content of the emulsion remaining after settling has been reduced from 50% to 18%). Note that the tank containing the 60-minute sample was left undisturbed for an additional 24 hours, and no further separation occurred. The IFT measurements gave demulsifier concentrations in the decanted water in the 260 to 475 ppm range. Given the lack of sensitivity of the IFT analytical technique at demulsifier concentrations above 100 ppm it was not possible to ascertain what fraction of the 2654 ppm of demulsifier injected into the fluid ended up in the decanted water. Suffice to say that a considerable portion of the demulsifier did end up in the water, and at concentrations in the hundreds to thousands of ppm.

Table 2-6 summarizes the key results from the laboratory tests. Note that fluid flow rate was not an independent variable: all the tests involving free water resulted in a measured flow rate of approximately 23 L/min (6 US gpm) and all the runs with no free water had a flow rate of approximately 11 L/min (3 US gpm).

Primary Break

In almost all of the tests, primary break occurred in two to five minutes.

Partitioning of the Demulsifiers

The formation of micelles by the surfactants in the water at high concentrations and the resulting limitations of the analytical technique used to measure the concentration of the demulsifiers in the decanted water make definitive results impossible. As such, it was not possible to discern any trends in the partitioning of the demulsifiers between the decanted water and the oily phase. The exception may be that more of the Exxon Nalco product ended up in the decanted water than either the Breaxit or Alcopol. The following general observations can be made:

- A large fraction of the demulsifier injected into the recovered fluid stream appears to end up in the decanted water.
- The concentrations of demulsifier in the decanted water are well in excess of 100 ppm and could be as high as in the 1000's of ppm.

Effectiveness of the Three Demulsifiers in Breaking Emulsions of the Two Oils

Overall, it was apparent that the Alcopol demulsifier was the best of the three demulsifiers tested on 50% salt water emulsions made from both parent oils (the Hydrocal blend and the fresh Endicott crude). The next most effective demulsifier on the Hydrocal blend emulsions was Breaxit. The Alcopol was better than the Exxon Nalco demulsifier on the fresh Endicott emulsions, and seemed to work as well with the Endicott as it did with the Hydrocal blend (comparing test 3 to test 21). The effect of the Exxon Nalco product seemed to be to create a very fine dispersion of oil droplets in the water, which made subsequent separation of the oil and water very slow.

Effect of Demulsifier Dose Rate

Comparing tests 4 to 5, 2 to 8 and 6 to 7 and 9, it can be seen that a higher Alcopol dose rate (ca. 2600 ppm) provided better resolution of the emulsion than did a lower rate (ca. 900 ppm). The same was true for the Breaxit demulsifier (comparing tests 22 to 23 and

Table 2-6. Summary of laboratory test results.

Test Number	Oil	Free Water (%)	Demulsifier	Demulsifier Concentration (ppm)	Inline Mixer (Y/N)	Circuit Length (ft)	Emulsion Dehydration (%)			Concentration of Demulsifier in Decant Water (ppm)			Comments
							2 or 5 min.	10 or 15 min.	60 min.	2 min.	10 min.	60 min.	
1	Hydrocal 80/5/15	54	None	0	Yes	36	No Sample	No Sample	3	8	2	2	Timer fouled up & start estimated
2	Hydrocal 80/5/15	55	Alcopol	3058	Yes	36	7	45	47	329	261	329	Water ran out and test divided into 2 & 2'
2'	Hydrocal 80/5/15	0	Alcopol	5408	Yes	36	22	42	50	No Sample	358	473	
3	Hydrocal 80/5/15	48	Alcopol	2654	Yes	36	48	55	63	261	473	429	Redo of #3
4	Hydrocal 80/5/15	32	Alcopol	989	Yes	36	22	32	90	391	473	329	
5	Hydrocal 80/5/15	33	Alcopol	2680	Yes	36	45	38	41	303	659	525	
6	Hydrocal 80/5/15	47	Alcopol	2606	Yes	36	48	32	No Sample	586	747	No Sample	Water ran out after third cylinder
7	Hydrocal 80/5/15	49	Alcopol	989	Yes	36	11	20	30	473	659	525	
8	Hydrocal 80/5/15	55	Alcopol	983	Yes	36	16	9	22	429	429	227	
9	Hydrocal 80/5/15	48	Alcopol	2639	Yes	36	38	45	48	227	261	227	
10	Hydrocal 80/5/15	53	Alcopol	2671	Yes	6	22	30	13	187	261	329	
11	Hydrocal 80/5/15	50	Alcopol	915	Yes	6	6	14	20	429	429	473	
12	Hydrocal 80/5/15	0	Alcopol	1929	Yes	6	3	3	52	No Sample	No Sample	329	
13	Hydrocal 80/5/15	0	Alcopol	4398	Yes	6	No Sample	3	36	No Sample	429	391	
14	Hydrocal 80/5/15	62	Alcopol	2671	Yes	36	25	22	38	329	525	391	
15	Hydrocal 80/5/15	64	Alcopol	1014	Yes	36	27	30	40	243	358	303	
16	Hydrocal 80/5/15	49	Exxon Nalco	2737	Yes	36	-37	-33	30	878	1022	661	Cylinders filled with oil/cloudy water, very slow to separ
17	Hydrocal 80/5/15	47	Exxon Nalco	1014	Yes	36	-33	-23	26	1022	759	759	Oily phase in cylinders still brown.
18	Hydrocal 80/5/15	0	Exxon Nalco	5239	Yes	6	-5	3	1	No Sample	No Sample	No Sample	
19	Endicott	49	Exxon Nalco	2737	Yes	36	No Sample	-10	-2	No Sample	1201	946	Oil/water mixture of low viscosity in cylinders
20	Endicott	49	Exxon Nalco	1038	Yes	36	-50	16	32	No Sample	759	381	
21	Endicott	48	Alcopol	2639	Yes	36	30	40	64	243	149	142	May have run out of emulsion.
22	Hydrocal 80/5/15	48	Breaxit	1001	Yes	36	17	13	9	164	177	209	
23	Hydrocal 80/5/15	47	Breaxit	2606	Yes	36	9	20	21	277	307	307	
24	Hydrocal 80/5/15	0	Breaxit	2077	Yes	36	8	3	1	No Sample	No Sample	No Sample	
25	Hydrocal 80/5/15	0	Breaxit	4789	Yes	36	1	1	9	No Sample	No Sample	No Sample	May have run out of demulsifier at end

24 to 25). In one case with the Exxon Nalco product (test 19 vs. 20), the lower dose rate resulted in better breaking of the Endicott crude emulsion than the higher dose did.

Effect of Free Water

As was the case with the previous series of tests (SL Ross 2002), when the free water content in the treated fluid exceeded 55%, the efficiency of the demulsifier was reduced. When the demulsifier was injected into a fluid stream that contained only emulsion, the separation initially was much poorer than in tests where the free water was less than 50%, but after 60 minutes, the demulsifier effectiveness was about the same for both cases. In the case of the Breaxit demulsifier, it seemed to work much better with 50% free water in the treated fluid than it did when there was no free water present. The same was true for the Exxon Nalco demulsifier when applied to the Hydrocal blend emulsion.

Effect of Tubing Length

Pumping the treated fluid down either a 6-foot or 36-foot long length of ½”-tubing was the only variation in mixing level used in this test series. As was observed in the earlier demulsifier tests (SL Ross 2002), better resolution of the emulsion was obtained when the treated fluid was pumped through the 36-foot length than the 6-foot length (tests 9 vs. 10, 7 vs. 11 and 2’ vs. 13). This was likely related to greater mixing of the demulsifier and the emulsion in the longer length of tubing.

3. OHMSETT TESTING

This section describes the procedures and results for the meso-scale tests carried out at Ohmsett. These are based on the previous demulsifier tests, and are described fully in the report (SL Ross 2002). Only a summary of the equipment and procedures is given here. The tests were completed during the week of the 9th through the 14th of November 2003. The Ohmsett Test Plan may be found in Appendix C.

3.1 Ohmsett Test Equipment and Methods

3.1.1 Preparations

The preparations for the tests included:

- Installing the skimmer, hoses, Globe boom and instrumentation
- Conducting required safety checks, calibrations and notifications.

Test Set-up and Instrumentation

All tests were conducted in a stationary position (i.e., no towing down the tank). A schematic layout of the test equipment is given in Figure 3-1.

The test area consisted of 11.5 m (37.5 feet) of 24-inch Globe boom deployed in a triangle (12.5' per side) between the Auxiliary Bridge and the Main Bridge (Figure 3-2). The boomed area was approximately 6.2 m^2 (67 ft^2). The Desmi Terminator skimmer was placed in the test area and operated from the deck. The skimmer discharge was directed to four of the oil recovery tanks on the Auxiliary Bridge (Figure 3-3) via 3-inch flexible hose. For all tests, the skimmer discharge was directed through a Lightnin Series 45 Model 4 Type 12H in-line mixer. The separated water from the oil recovery tanks was directed to a temporary holding tank (Figure 3-4) for water sampling, and then sent to a holding tank for eventual treatment and return to the tank.

Demulsifiers (Alcopol O 70% PG, aka Drimax 1235B, and Unichem RNB-60425, an emulsion breaker specifically designed for Endicott crude) were injected using a fixed-rate (0.25 gpm) peristaltic pump directly into the skimmer weir. For all tests the decanted water was sent to the sampling tank, where it was mixed thoroughly, allowed to settle and then sampled for oil content and IFT analysis. Oil or emulsion from the recovery tanks was pumped to the Ohmsett oily water processing system then stored for disposal.

Waves were generated at the south end of the tank and controlled by the bridge operator in the Control Tower at the north end. The wave profiles were recorded using a Datasonics ultrasonic distance meter. The signal from the wave meter was recorded and analyzed to confirm the wave characteristics.

Two wave conditions were generated during this test series. Their nominal characteristics are defined in Table 3-1.

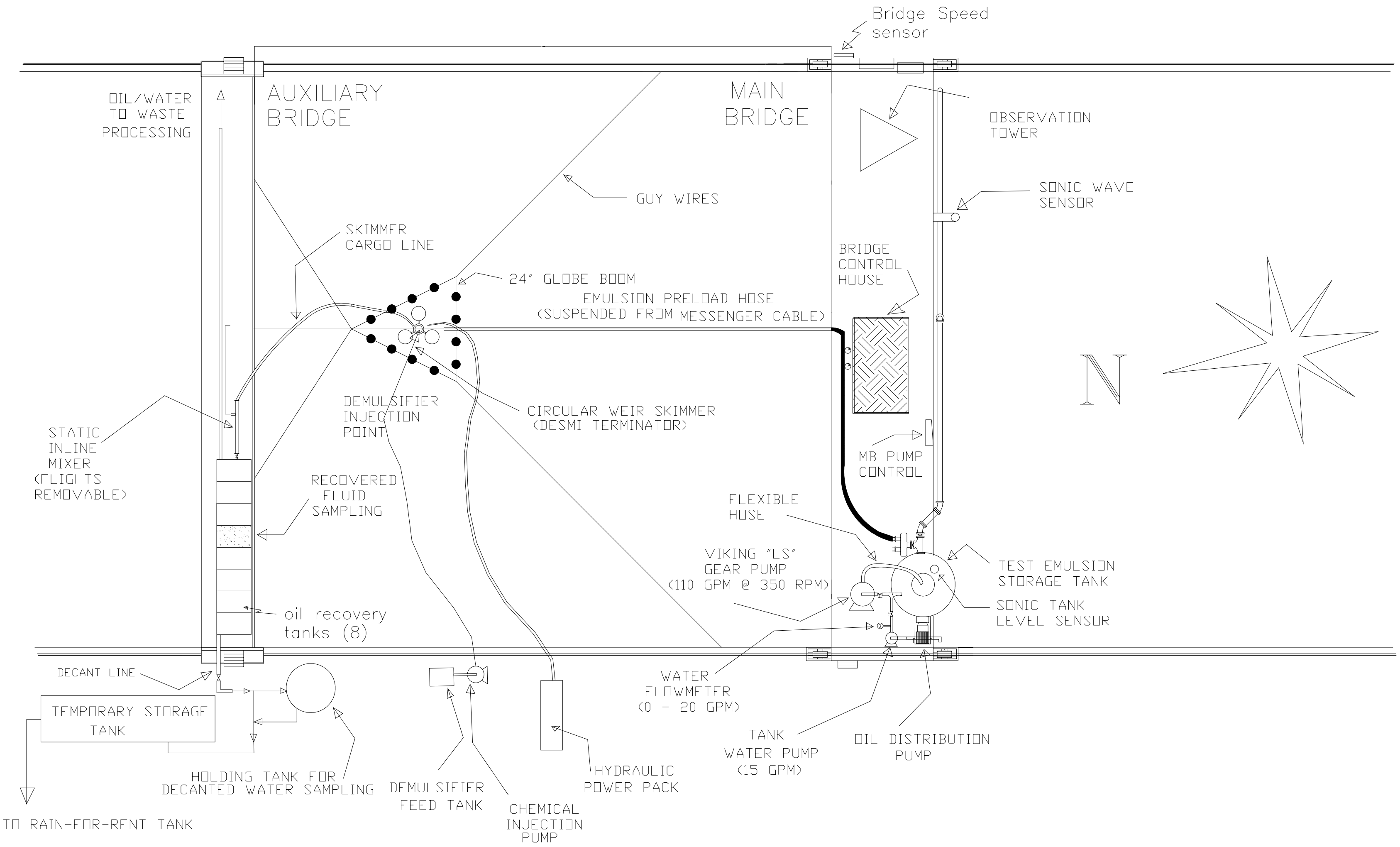


Figure 3-1. Layout of equipment in tank at Ohmsett

Figure 3-2. Photo of boom triangle and Desmi Terminator skimmer in water.

Figure 3-3. Photo showing power pack on deck, skimmer discharge hose to recovery tanks and inline mixer.

Table 3-1. Nominal Wave Characteristics.

Wave No.	Stroke (in.)	CPM	Type	Nominal $H^{1/3}$ (in.)	Wave Length (ft.)	Period (sec)
#1	3	22	Sinusoidal	16.5	37	2.8
#2	3	35	Sinusoidal	15	15	1.7

Emulsion Preparation

At the beginning of the tests, and subsequently as required, batches of emulsion were prepared. A gear pump was used to prepare the emulsion, since large quantities of a consistent quality were required on a daily basis. The use of high-speed pumps, including gear pumps, to create emulsions for equipment testing is well known and widely utilized in North America and Europe (eg., Gåseidnes 1993, DNV 2002). A blend of 80% Hydrocal 300/5% IFO 380/15% automotive diesel was used as the parent oil for most of the tests. Fresh Endicott crude was used as the parent oil for two tests. A sample of the first batch of Hydrocal blend emulsion prepared was allowed to sit for 24 hours, and showed no signs of breaking. The target property of the emulsion was 50% (vol.) water content. A 50% water content was chosen because it could be prepared reasonably quickly using the gear pump technique with little risk of inverting the emulsion, as can occur with higher water contents. The emulsion batches were prepared, using the Viking gear pump plumbed to the Main Bridge oil tank as shown schematically in Figure 3-5, using the same procedures as described in detail in SL Ross 2002. Samples of each batch were taken and subjected to BS&W analysis (see Section 3.1.4 below). The sample from the first batch of Hydrocal blend emulsion was misplaced, but the water content of the first sample from the second baseline test (i.e., Test 2 - no demulsifier) was 50%. The second batch had a water content of 45% and the batch of emulsion created with the fresh Endicott crude had a water content of 53%.

3.1.2 Test Procedures

The following procedures were followed for each test:

Before each test the Emulsion Recovery Rate (ERR) for the skimmer was estimated and the volume of emulsion removed from the boomed area during the previous test calculated. The aim was to pump emulsion into the boomed area at the same rate that the skimmer removed it so that a constant thickness of emulsion was being presented to the skimmer. Then:

1. The required volume of test emulsion was added to the boomed area to make up the desired slick thickness (approximately 100 mm, see Test Matrix below).
2. The Main Bridge distribution pump speed was set to supply fresh test emulsion at the ERR estimated for the test.
3. The waves were turned on at the desired setting and allowed to come to apparent steady state (this required about two minutes). The data acquisition system was started.

Figure 3-4. Decanted water temporary storage tank (foreground) and water sampling tank (background)

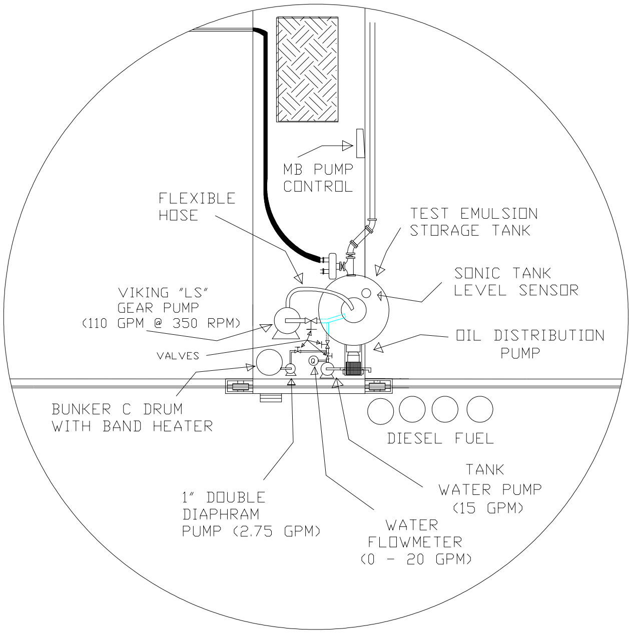


Figure 3-5. Schematic of emulsion formation system

4. The emulsion distribution pump was started and the skimmer turned on, with its discharge directed to recovery tanks #8 and #7. The chemical injection pump was started with flow to the desired location at the desired demulsifier flow rate (nominally 1/400th or 1/1000th of the Fluid Recovery Rate).
5. When the cargo line was purged, the skimmer discharge was directed to the recovery tank cells sequentially (i.e., fill cell #6, then #5, etc.). The target volume of emulsion (exclusive of free water) in each cell was 180 L.
6. The time when filling each tank cell was started and finished was recorded. The depth of fluid in each cell was measured and recorded.
7. After the last tank cell (#3) was filled, the emulsion distribution pump, demulsifier injection pump, skimmer and waves were stopped.
8. Simultaneously with the filling operation, two minutes after tank cell #6 was filled, the separated water was decanted until the discharge from the bottom was "black". The water was sent to a temporary storage tank and not poured back into the test basin. Note that cells #8 and #7 were also decanted to the temporary storage tank for processing.
9. All the test cells in each run were decanted directly to a Nalgene temporary holding tank on the deck beside the Auxiliary Bridge. When all water from a given test cell was transferred, the contents of the temporary holding tank were thoroughly mixed with an electric, bladed mixer and allowed to settle for five minutes to allow large droplets of emulsion to surface. A small water sample, for oil content analysis, was taken when half the water had drained. The purpose of this was to estimate the average concentration of "permanently dispersed" oil in the decanted water - i.e., the droplets that would not rise out and re-coalesce with the slick if the decanted water was discharged back into a boomed area. A second water sample for IFT analysis to measure demulsifier concentration was obtained at the same time.
10. The remaining emulsion recovery tank cells were decanted in sequence at 10, 30 and 60 minutes after the time they were filled. The purpose of this was to determine the time required for "primary break" of the skimmer discharge product. "Primary break" is the point at which the bulk of the lower density phase has risen to the top and the higher density phase has settled to the bottom; both phases typically contain small droplets of the other phase at this point.
11. The depth of fluid remaining in each cell was measured (these depths, combined with the initial depths, were used to calculate the volumes of recovered product, decanted water and emulsion remaining).
12. Each recovery tank cell was mixed and sampled to determine the water content of the oily fluid remaining.
13. The contents of the recovery tank cells were transferred for waste processing.

3.1.3 Test Matrix

The following variables were involved in constructing the test matrix:

- One circular weir skimmer (representative of OSRO stockpiles)
  - Desmi Terminator (USCG/Ohmsett) - nominal ORR in waves 20 m^3/hr (90 USgpm)

- One slick thickness (representing a thickness typically expected for large-scale offshore boom/skimmer operations)
  - 100 mm (requires 625 L - 165 US gallons - preload in triangular boom)
- One demulsifier injection point
  - into the skimmer mouth
- Two Wave Conditions
  - wave #1, $H^{1/3}$= 15" with λ = 37'
  - wave #2, $H^{1/3}$= 15" with λ = 15'
- Three demulsifier dose rates (1:400, 1:1000 and 1:2500)
- Three parent oil/demulsifier combinations (Hydrocal/Alcopol, Hydrocal/Unichem RNB-60425 and two tests with Endicott/Unichem RNB-60425)
- Two control tests (no demulsifier injected)

One duplicate test was conducted. Table 3-2 gives the preliminary test matrix.

Table 3-2. Planned Test Matrix.

Test No.	Emulsion Parent Oil	Demulsifier	Demulsifier Dose	Wave No.
1	Hydrocal 80/5/15	none	none	1
2	Hydrocal 80/5/15	none	none	2
3	Hydrocal 80/5/15	Alcopol O 70% PG	1:400	1
4	Hydrocal 80/5/15	Alcopol O 70% PG	1:400	2
5	Hydrocal 80/5/15	Alcopol O 70% PG	1:1000	1
6	Hydrocal 80/5/15	Alcopol O 70% PG	1:1000	2
7 (duplicate)	Hydrocal 80/5/15	Alcopol O 70% PG	none	none
8	Hydrocal 80/5/15	Alcopol O 70% PG	1:2500	2
9	Endicott crude	Unichem RNB-60425	1:400	1
10	Endicott crude	Unichem RNB-60425	1:400	2

3.1.4 Sample Analyses

Each test involved collecting 4 water samples for TPH analysis, 4 emulsion samples for BS&W, and 4 water samples for IFT analysis to determine demulsifier concentrations. The oil-in-water samples were sealed in glass jars until such time as they could be analysed. The emulsion and IFT samples were stored in Nalgene jars. In addition, a sample of each batch of emulsion was subjected to a BS&W analysis.

Bottom Solids and Water

The water content of the emulsion samples was determined using the procedures specified in ASTM D1796. The method involved splitting a well-shaken, 100-mL emulsion sample into two aliquots. Each aliquot was poured into a graduated, centrifuge tube containing 50 mL of toluene, filling the tube to the 100-mL mark. The tube was shaken vigorously, warmed and then placed in the centrifuge and spun for 10 minutes. The volume of water in the tubes was read directly from the graduations. For water volumes in the 10 to 25 mL range (20% to 50% water content emulsions) the reading error was on the order of 1 mL (2%); for higher water content emulsions the error was likely in the 3 to 5 mL range (6% to 10%).

Total Petroleum Hydrocarbons

The decanted water samples were extracted with a solvent and then analysed with a gas chromatograph at the US Army Fort Monmouth Environmental Testing Laboratory. The techniques used followed those specified in NJ-DEP OQA-QAM-025,. The limit of detection of this method was 1 to 5 ppm. The technique also detects the dissolved hydrocarbons in the tank water from previous testing. Generally, the “background” TPH level in the tank is 3 to 5 ppm. During the extraction process, the solvent could also remove some portion of the demulsifier that is dissolved in the water. It is not certain what this portion would be.

IFT Analysis for Demulsifier

The water samples taken from each recovery tank were subjected to the interfacial tension test described in Section 2.1 to estimate the concentration of demulsifier that they contained. Calibration curves (IFT against USP mineral oil vs. concentration of demulsifier in Ohmsett tank water) were constructed for both the Alcopol (aka Drimax) and Unichem demulsifiers.

3.2 Ohmsett Test Results

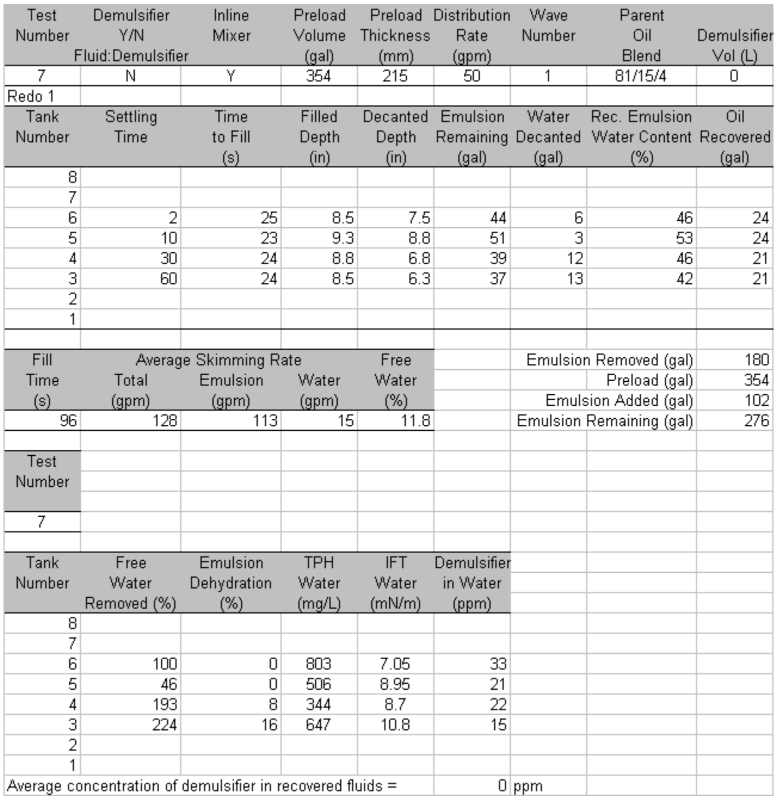
The complete data set for each test can be found in Appendix D. The ability of emulsion breaking chemicals to resolve water-in-oil emulsions is highly parent oil/surfactant specific. The results are strictly valid only for the combinations of demulsifiers (Alcopol O 70% PG, aka Drimax, and Unichem RNB-60425) and emulsions used (50% salt water in either a blend of 80% Hydrocal 300/5% IFO 380/15% diesel, or fresh Endicott crude).

Table 3-3 shows the full results obtained for Test 7, with no demulsifier injected, and Table 3-4 shows the results for Test 3, a run with Alcopol injected at 1413 ppm (a dose rate of 1 part demulsifier in 700 parts of fluid) into the hopper of the Desmi Terminator skimmer.

The results for Test 7 show that, without demulsifier, only free water is decanted, and that the water content of the emulsion remains approximately 50%. The IFT measurements on the water samples show that there are negligible amounts of demulsifier present in the decanted water, although there may be traces left from a previous test. The TPH data shows initial oil concentrations in the decanted water in the 800-ppm range, declining as the settling proceeds.

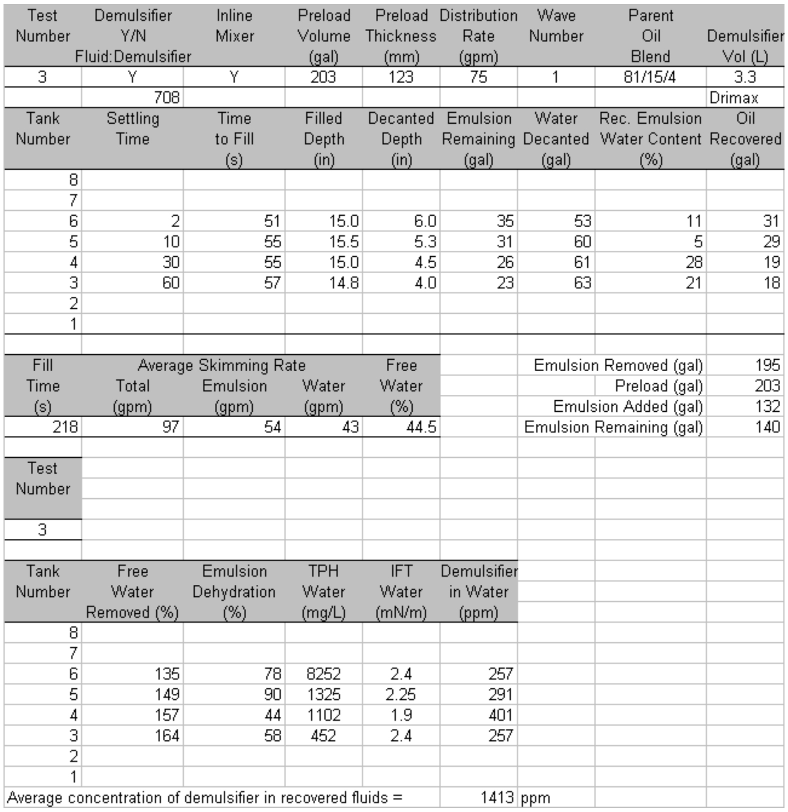
The results for Test 3 show the effects of demulsifier addition. More water is decanted than free water was injected, and the water content of the remaining emulsions is greatly

Table 3-3. Data spreadsheet for Test No. 7 – No Demulsifier.



Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
7	N	Y	354	215	50	1	81/15/4	0
Redo 1								
Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	25	8.5	7.5	44	6	46	24
5	10	23	9.3	8.8	51	3	53	24
4	30	24	8.8	6.8	39	12	46	21
3	60	24	8.5	6.3	37	13	42	21
2								
1								
Fill	Average Skimming Rate			Free			Emulsion Removed (gal)	180
Time	Total	Emulsion	Water	Water			Preload (gal)	354
(s)	(gpm)	(gpm)	(gpm)	(%)			Emulsion Added (gal)	102
96	128	113	15	11.8			Emulsion Remaining (gal)	276
Test Number								
7								
Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)			
8								
7								
6	100	0	803	7.05	33			
5	46	0	506	8.95	21			
4	193	8	344	8.7	22			
3	224	16	647	10.8	15			
2								
1								
Average concentration of demulsifier in recovered fluids =					0	ppm		

Table 3-4. Data spreadsheet for Test No. 3 – 1400 ppm Alcopol.



Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
3	Y	Y	203	123	75	1	81/15/4	3.3
	708							Drimax
Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	51	15.0	6.0	35	53	11	31
5	10	55	15.5	5.3	31	60	5	29
4	30	55	15.0	4.5	26	61	28	19
3	60	57	14.8	4.0	23	63	21	18
2								
1								
Fill	Average Skimming Rate			Free			Emulsion Removed (gal)	195
Time	Total	Emulsion	Water	Water			Preload (gal)	203
(s)	(gpm)	(gpm)	(gpm)	(%)			Emulsion Added (gal)	132
218	97	54	43	44.5			Emulsion Remaining (gal)	140
Test Number								
3								
Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)			
8								
7								
6	135	78	8252	2.4	257			
5	149	90	1325	2.25	291			
4	157	44	1102	1.9	401			
3	164	58	452	2.4	257			
2								
1								
Average concentration of demulsifier in recovered fluids =					1413	ppm		

reduced. The emulsion dehydration (the percent reduction in the volume of water in the emulsion[1]) ranges from 45% to 90%. Given the lack of sensitivity of the IFT analytical technique at demulsifier concentrations above 100 ppm it was not possible to ascertain what fraction of the 1400 ppm of demulsifier injected into the fluid ended up in the decanted water. Suffice to say that a considerable portion of the demulsifier did end up in the water, and at concentrations in the hundreds to thousands of ppm. The concentrations of oil in the decanted water were over 8000 ppm initially, declining to 450 ppm after 60 minutes.

Table 3-5 summarizes the key results from the Ohmsett tests.

Primary Break

Figure 3-6 shows the rate of separation of water from the recovered fluids in the recovery tank. The baseline test results (no demulsifier was used in Tests 1, 2 and 7) are shown with larger symbols and thicker lines. In each test the four recovery tanks were filled to the same level, within an inch or so (5.84 gallons per inch is the factor for the recovery tanks) at a steady recovery rate. The exceptions were Tests 6 and 9, during which there were problems with the skimmer hydraulic power pack that resulted in a change in the "steady state" conditions for the test. It is clear that, in most cases, primary break is achieved in 30 minutes or less. This is entirely consistent with the results of both previous decanting test series (SL Ross 1999 and 2002).

Partitioning of the Demulsifiers

The formation of micelles by the surfactants in the water at high concentrations and the resulting limitations of the analytical technique used to measure the concentration of the demulsifiers in the decanted water make definitive results impossible. As such, it was not possible to discern any trends in the partitioning of the demulsifiers between the decanted water and the oily phase. The following general observations can be made:

- A large fraction of the demulsifier injected into the recovered fluid stream appears to end up in the decanted water.
- The concentrations of demulsifier in the decanted water are well in excess of 100 ppm and could be as high as in the 1000's of ppm.

Effectiveness of the Two Demulsifiers in Breaking Emulsions of the Two Oils

Figure 3-7 shows the measured dehydration of the samples from the recovery tanks as a function of time since the tank was filled (i.e., the time at which they were decanted). Again, the results from the baseline tests (2 and 7) are shown with larger symbols and thicker lines.

Without the addition of demulsifier, there was no dehydration in the emulsions recovered in Wave 1 conditions, and an increase in the water content of the emulsions (from 50% at 2 min. to 65% at 60 minutes) in Wave 2 conditions. Note that the 30-minute and 60-minute samples from test 7, the repeat of the baseline test 1 in Wave 1, did show a low level of dehydration, possibly related to contamination of the skimmer, hoses and recovery tanks from previous tests with demulsifier. The extra mixing energy added to

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[1] A reduction in emulsion water content from 50% to 20% equates to an emulsion dehydration of 60%

Table 3-5. Summary of Ohmsett test results.

Test Number	Oil	Wave Number	Total Fluid Flow Rate (gpm)	Free Water (Avg %)	Demulsifier	Demulsifier Concentration (ppm)	Recovered Emulsion Water Content (Avg %)	Emulsion Dehydration (%)			
								2 min.	10 min.	30 min.	60 min.
1	Hydrocal 80/5/15	1	67	12	None	0	No Sample	No Sample	No Sample	No Sample	No Sample
2	Hydrocal 80/5/15	2	104	19	None	0	60	0	0	0	0
3	Hydrocal 80/5/15	1	97	44	Alcopol	1413	16	78	90	44	58
4	Hydrocal 80/5/15	2	80	45	Alcopol	1194	29	44	48	42	36
5	Hydrocal 80/5/15	1	117	16	Alcopol	888	24	54	50	38	64
6	Hydrocal 80/5/15	2	80	49	Alcopol	1354	23	70	62	38	46
7	Hydrocal 80/5/15	1	128	12	Alcopol	0	47	0	0	8	16
8	Hydrocal 80/5/15	2	224	58	Alcopol	425	19	62	58	62	64
9	Endicott	1	135	18	Unichem	1380	34	26	30	32	44
10	Endicott	2	95	34	Unichem	1647	35	42	28	30	20

Test Number	Concentration of Oil In Decanted Water (ppm)				Concentration of Demulsifier in Decanted Water (ppm)			
	2 min.	10 min.	30 min.	60 min.	2 min.	10 min.	30 min.	60 min.
1	No Sample	212	206	918	No Sample	No Sample	2	2
2	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample
3	8252	1325	1102	452	257	291	401	257
4	3491	799	430	979	445	267	267	257
5	2189	25330	740	22209	140	592	229	401
6	22547	2816	14572	923	630	470	401	445
7	803	506	344	647	33	21	22	15
8	4649	1680	990	1249	279	317	279	364
9	11239	3338	1347	934	241	223	175	171
10	4515	4184	2143	491	301	149	175	156

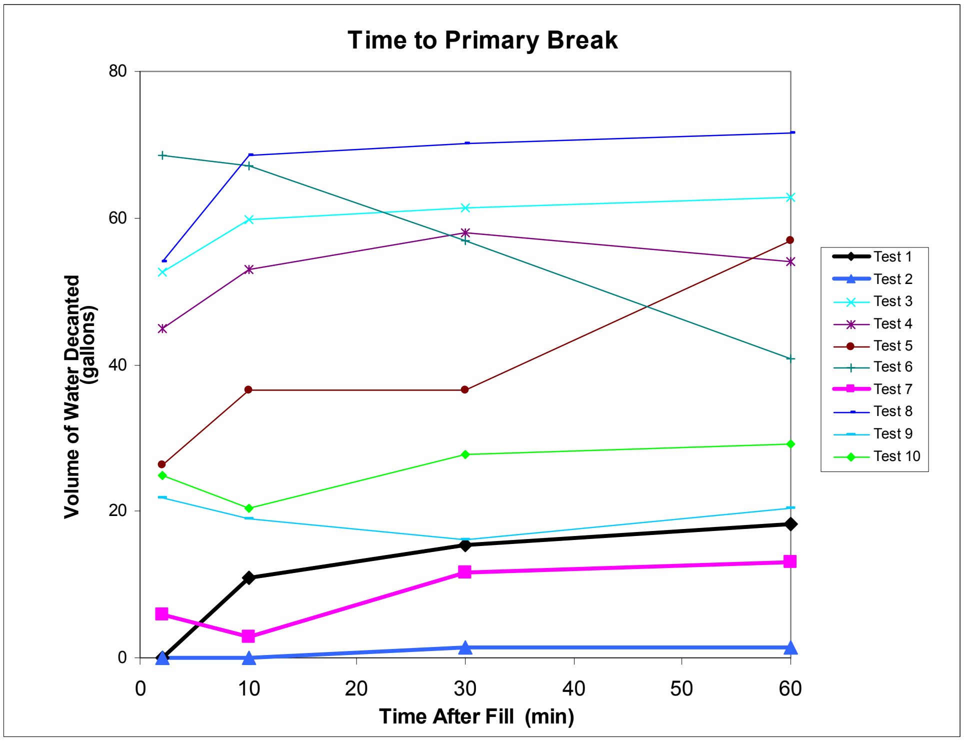

Figure 3-6. Separation of recovered fluid as a function of time.

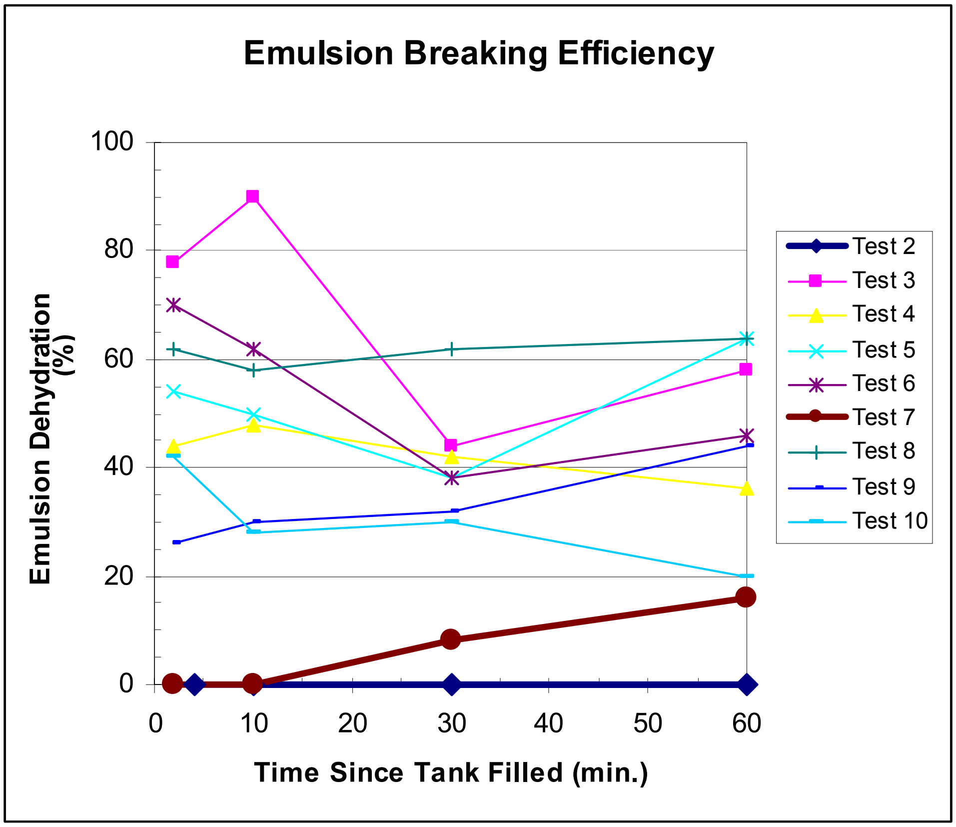

Figure 3-7. Emulsion breaking efficiency test results.

the slick by the steeper Wave 2 conditions caused additional emulsification of the oil (as observed in the previous tests – SL Ross 2002).

The addition of demulsifier caused significant amounts of water to separate from the treated emulsions. In Wave 1 conditions almost ⅔^{rds} of the emulsion water was removed and decanted; in Wave 2 conditions, a lesser degree of emulsion dehydration was calculated; however, these calculations are based on the assumption that the emulsion has a water content of 50%. If, as is likely based on the results of Test 2, the emulsion water content was upwards of 65% by the end of a test in Wave 2 conditions, the dehydration efficiencies for Tests 4 and 6 would be closer to 60%, rather than 36% and 46% respectively.

The best dehydration obtained was for Test 8, run in Wave 2 with the lowest dose rate of Alcopol of all, but with a recovery rate almost twice that of any other test. The 60-minute dehydration result of 64% (72%, if a 65% water content emulsion was being skimmed) was a testament to the fact that mixing energy is very important for effective emulsion breaking, even more so than dose rate.

The results obtained at Ohmsett were consistent with those from the lab tests with free water contents of less than 50%.

The efficiency of the Unichem demulsifier on the emulsions of fresh Endicott crude was not as high as the Alcopol with the Hydrocal blend emulsions, but the results were encouraging nonetheless. This is because the demulsifier is not an oil spill demulsifier, but a product designed for oil field production purposes (and hence, stored in large quantities in Alaska at the oil fields). In Wave 1 conditions (Test 9), 44% dehydration was achieved in the 60-minute sample. In Wave 2 (Test 10), 20% dehydration was calculated after 60 minutes (40%, if the emulsion was 65% water, not 50%).

Oil Content of the Decanted Water

Figure 3-8 shows the measured oil content of the decanted water from the various tests. The results from the two baseline tests (#1 and #7) are shown with larger symbols and thicker lines. Several of the samples from tests using demulsifier had measured TPH values in the 10,000 to 25,000 ppm range, much higher than expected. It is possible that these samples contained one or two very large oil droplets that biased the result. One oil droplet with a diameter of 2.67 mm would result in a concentration of 20,000 ppm in a 500-mL water sample.

In general, the concentration of oil in the decanted water declined from values in the thousands of ppm after two minutes, to the high hundreds of ppm after 60 minutes.. The baseline results were generally similar to those obtained in the previous test series, with TPH values in the 200 to 1000 ppm range. The TPH values measured with tests involving demulsifier on Hydrocal blend emulsions were general higher than those obtained in the previous test series. This could be due to different analytical techniques (the previous series used extraction/IR analysis for TPH) but is more likely due to the addition of 15% diesel to the parent oil blend for this test series. This would make the parent oil

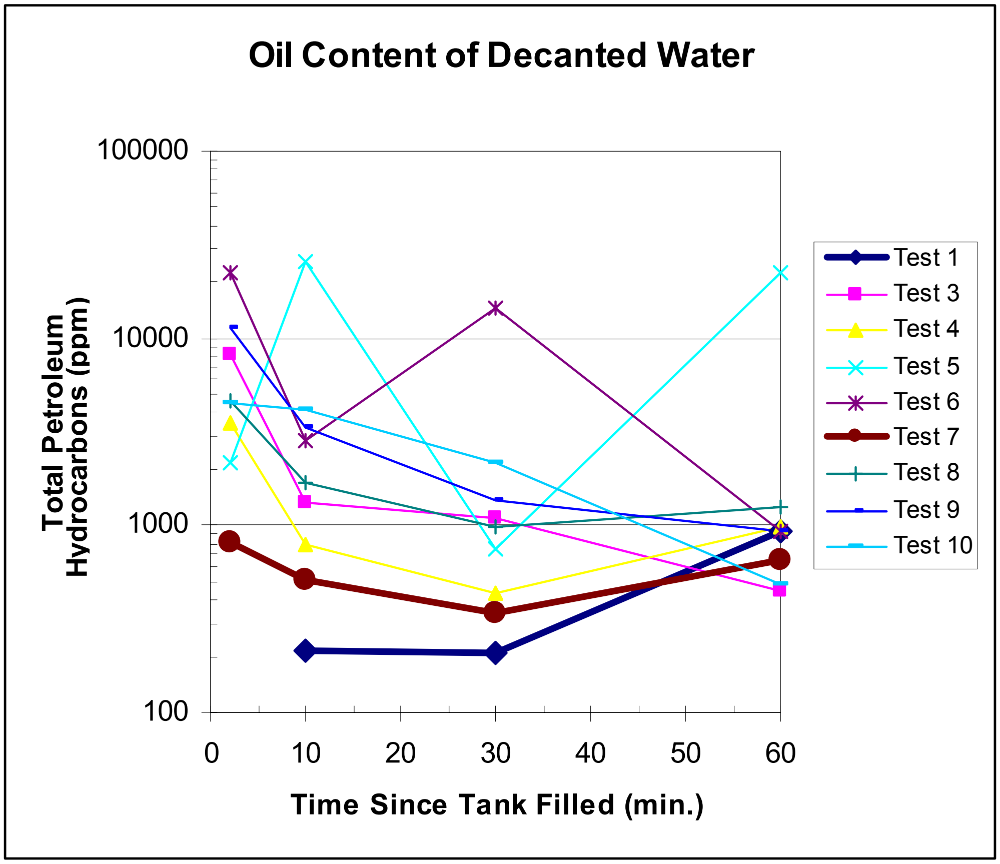

Figure 3-8. Concentrations of oil in decanted water.

significantly less viscous, and hence easier to shear into very small droplets that take longer to rise out of the water.

The TPH results for the Endicott emulsions treated with the Unichem demulsifier were in the same range as the results for the Hydrocal emulsion treated with the Alcopol demulsifier.

4. CONCLUSIONS AND RECOMMENDATIONS

The efficiency of emulsion breaking chemicals in resolving water-in-oil emulsions is highly parent oil/surfactant specific. The conclusions drawn from the results are strictly valid only for the combinations of demulsifiers (Alcopol O 70% PG, Breaxit OEB-9, Exxon Nalco EC 2085 and Unichem RNB 60425) and emulsions used (50% salt water in either a blend of 80% Hydrocal 300/5% No. 6 Fuel Oil/15% diesel, or fresh Endicott crude).

The major implication of this research for oil spill response is that it may be possible to greatly reduce downtime for offshore skimming operations caused when the available onsite temporary storage systems are filled with fluids containing large amounts of water; however, it is likely that much of the demulsifier used will be contained in the decanted water. The legislated requirements for onsite temporary storage systems could also ultimately be reduced by the use of these results, resulting in considerable savings in operating and disposal costs for OSRO's. Knowing that the separated water can be decanted quickly will optimize onsite recovery operations and greatly reduce the volume of fluids requiring disposal. In fact, the removal of most of the free and emulsified water from the recovered product would greatly enhance the likelihood that it could be recycled, as opposed to requiring disposal.

4.1 Conclusions

- The use of a demulsifier injected into a recovery system, combined with decanting, substantially reduced the volume of water in temporary storage tanks and the water content of emulsions for disposal/recycling.

- The formation of micelles by the surfactants in the water at high concentrations and the resulting limitations of the analytical technique used to measure the concentration of the demulsifiers in the decanted water make definitive conclusions about the partitioning of the demulsifier between oily and water phases impossible. The following general conclusions could be made:
    1. A large fraction of the demulsifier injected into the recovered fluid stream appears to end up in the decanted water.
    2. The concentrations of demulsifier in the decanted water are well in excess of 100 ppm and could be as high as in the 1000's of ppm.

- The efficacy of the demulsifier was a strong function of free water content. In these tests, if the free water content exceeded about 55%, the effect of the surfactant was substantially reduced.

- The degree of emulsion breaking achieved increased with increasing mixing energy applied to the fluid. Increasing the flow rate (and hence turbulence level) and increasing the length of the flow path both resulted in increased emulsion breaking.

- Primary break occurred in only a few minutes (2 to 5 in the lab tests, less than 30 for the Ohmsett tests). The application of demulsifier did not appear to affect the time required.
- The Ohmsett results indicated that the use of a demulsifier increased TPH concentrations in the decanted water.
- The efficiency of the Unichem demulsifier on the emulsions of fresh Endicott crude was not as high as with the Alcopol, but the results were encouraging nonetheless. This is because the demulsifier is not an oil spill demulsifier, but a product designed for oil field production purposes (and hence, stored in large quantities in Alaska at the oil fields).

4.2 Recommendations

- It is apparent that the decision to decant recovered water offshore to increase available on-site temporary storage will involve trade-offs. The ability to continue skimming and remove oil from the water surface must be compared as quantitatively as possible to the potential effects of discharging the decanted water overboard. Ideally, a Net Environmental Benefit (NEB) comparison should be performed.
- The North Slope of Alaska utilizes a number of different emulsion breakers for production operations in a number of fields of varying oil characteristics and age. The use of an existing stockpile of production demulsifiers to treat emulsions recovered during spill response operations is very attractive. It is recommended that the various demulsifier chemicals stockpiled on the Slope be tested with emulsions of crude oils to determine which work best with which crudes.
- Consideration should be given to modifying the IFT technique to better measure demulsifier concentrations above 100 ppm. It is suggested that serial dilution of the samples, to reduce the demulsifier concentrations to the range in which the IFT technique gives reasonable results, could be a simple solution to the problem brought about by demulsifier micelle formation. Serial dilution will ultimately reverse the surfactant micellisation, but it can be relatively slow - hence IFT readings from a just-diluted sample will change over time as the individual surfactant molecules separate from the micelles. As long as the diluted solution is left to equilibrate for a while (a time period that will need to be determined experimentally – but is likely minutes, not seconds, but probably not hours) it will achieve a new micelle-molecule equilibrium.

- A standard emulsion for use in testing at Ohmsett should be developed. This effort should entail developing techniques to consistently “build” emulsions with water contents in the 70% to 80% range in order to achieve the high viscosities typical of oil spill emulsions at sea.

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APPENDIX A - OIL BLEND AND DEMULSIFIER TEST RESULTS

STABILITY OF EMULSIONS CREATED FROM VARIOUS PARENT OIL BLENDS (HYDROCAL/BUNKER C (SL ROSS)/AUTOMOTIVE DIESEL)

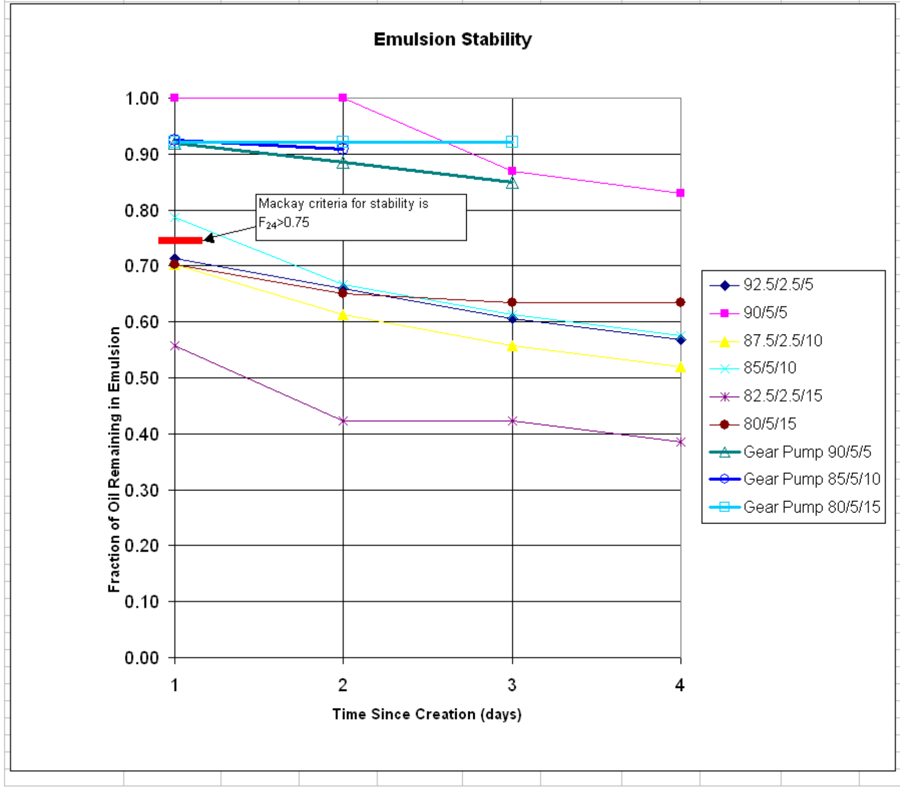
50% WATER EMULSIONS CREATED WITH HAND BLENDER USING 35 PPT SALTWATER AT 28°C

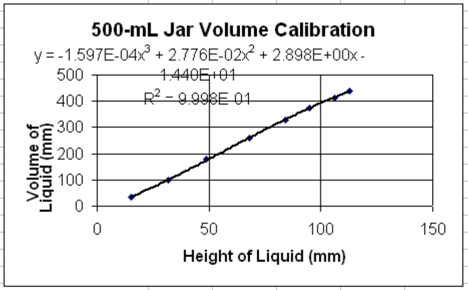
							Height of Emulsion						Fraction of Oil Remaining in Emulsion			
Parent Oil Composition			Initial Emulsion Amount		After 24 h		After 48 h		after 96 h		after 120 h					
Parts Hydrocal 300	Parts Bunker C	Parts Diesel	Height (mm)	Volume (mL)	Height (mm)	Volume (mL)	Height (mm)	Volume (mL)	Height (mm)	Volume (mL)	Height (mm)	Volume (mL)	24 h	48 h	96 h	120 h
92.5	2.5	5	113	437.1	95	374.5	92	362.8	89	350.8	87	342.7	0.71	0.66	0.61	0.57
90	5	5	127	474.2	127	474.2	127	474.2	115	443.1	112	434.0	1.00	1.00	0.87	0.83
87.5	2.5	10	114	440.1	95	374.5	90	354.8	87	342.7	85	334.4	0.70	0.61	0.56	0.52
85	5	10	114	440.1	100	393.3	93	366.7	90	354.8	88	346.7	0.79	0.67	0.61	0.58
82.5	2.5	15	114	440.1	87	342.7	80	313.3	80	313.3	78	304.7	0.56	0.42	0.42	0.38
80	5	15	117	448.9	97	382.1	94	370.6	93	366.7	93	366.7	0.70	0.65	0.63	0.63

50% WATER EMULSIONS CREATED WITH GEAR PUMP USING 35 PPT SALTWATER AT 28°C

90	5	5	108	421.2	103	404.1	101	396.9	99	389.6			0.92	0.88	0.85	
85	5	10	111	430.9	106	414.5	105	411.0					0.92	0.91		
80	5	15	109	424.5	104	407.6	104	407.6	104	407.6			0.92	0.92	0.92	

Mass Water (g)	Height in 500-mL Jar (mm)	
35.8	15	
98.1	32	
179.1	49	
260	68	
329.2	84	
375.6	95	top of cylindrical portion of jar
413.3	106	
437.8	113	





Emulsion-Breaker Effectiveness

Hydrocal + Bunker C (SL Ross) + Automotive Diesel, 50% 35 ppt saltwater emulsion created with Gear Pump

Parent Oil Blend (parts Hydrocal/Bunker C/Diesel	Emulsion Breaker	Dosage Volume (mL)	Initial Emulsion Height (mm)	H_{2min} (mm)	H_{60min} (mm)	H_{24} (mm)	Dim (%)	D24 (%)
90/5/5	none	0	41	85	80	80	-215	-190
90/5/5	Vytac DM	0.015*	40	93	93	93	-265	-265
90/5/5	Breaxit	0.015	43	93	93	93	-233	-233
90/5/5	Alcopol	0.015	43	39	38	38	19	23
80/5/15	Alcopol	0.015	45	27	27	27	80	80
80/5/15	Breaxit	0.015	45	32	32	31	58	62
80/5/15	Vytac DM	0.015	43	93	93	93	-233	-233
80/5/15	none	0	46	97	97	97	-222	-222
85/5/10	none	0	45	98	98	98	-236	-236
85/5/10	Alcopol	0.015	45	28	27	27	76	80

* equivalent to a dosage of 1:500 in the initial 165 mL of emulsion and 165 mL of water

Samples shown in green still contained some water, but when heated to 80°C for 24 hours they broke completely

APPENDIX B - LABORATORY-SCALE TEST RESULTS

Decant III

Lab Test Summary

Test Number	Oil	Free Water (%)	Breaker	Dose Rate	Inline Mixer (Y/N)	Circuit Length (ft)	Comments
1	Hydrocal 80/5/15	50	None	0	Yes	36	Timer fouled up & start estimated
2	Hydrocal 80/5/15	50	Alcopol	High	Yes	36	Water ran out and test divided into 2 & 2'
3	Hydrocal 80/5/15	50	Alcopol	High	Yes	36	Redo of #3
4	Hydrocal 80/5/15	33	Alcopol	Low	Yes	36	
5	Hydrocal 80/5/15	33	Alcopol	High	Yes	36	
6	Hydrocal 80/5/15	50	Alcopol	High	Yes	36	Water ran out after third cylinder
7	Hydrocal 80/5/15	50	Alcopol	Low	Yes	36	
8	Hydrocal 80/5/15	66	Alcopol	Low	Yes	36	
9	Hydrocal 80/5/15	50	Alcopol	High	Yes	36	
10	Hydrocal 80/5/15	50	Alcopol	High	Yes	6	
11	Hydrocal 80/5/15	50	Alcopol	Low	Yes	6	
12	Hydrocal 80/5/15	0	Alcopol	Low	Yes	6	
13	Hydrocal 80/5/15	0	Alcopol	High	Yes	6	
14	Hydrocal 80/5/15	66	Alcopol	High	Yes	36	
15	Hydrocal 80/5/15	66	Alcopol	Low	Yes	36	
16	Hydrocal 80/5/15	50	Exxon Nalco	High	Yes	36	Cylinders filled with oil/water mixture of low viscosity that was very slow to separate Separated water very cloudy
17	Hydrocal 80/5/15	50	Exxon Nalco	Low	Yes	36	Oily phase in cylinders still brown.
18	Hydrocal 80/5/15	0	Exxon Nalco	High	Yes	6	
19	Endicott	50	Exxon Nalco	High	Yes	36	Oil/water mixture of low viscosity in cylinders
20	Endicott	50	Exxon Nalco	Low	Yes	36	
21	Endicott	50	Alcopol	High	Yes	36	May have run out of emulsion.
22	Hydrocal 80/5/15	50	Breaxit	Low	Yes	36	
23	Hydrocal 80/5/15	50	Breaxit	High	Yes	36	
24	Hydrocal 80/5/15	0	Breaxit	Low	Yes	36	
25	Hydrocal 80/5/15	0	Breaxit	High	Yes	36	May have run out of demulsifier at end

Added "Volume Collected" column
Changed all "5000" references to actual "Volume Collected"
Added constant for "H2O Vol to top of outlet:"
Changed "Fluid Flow" to use SUM of "Volume Collected"
Incorporated 4mm sample bottle bottom into sample measures
Incorporated "IsNumber" tests to allow non-numerics
Added IFT columns including calc based on EXACT spelling of demulsifier

Emulsion Batch	Total Height (mm)	Measured Water Heigh (mm)	Oil Samples Oil Height (mm)*	Water Volume (mL)	Total Volume (mL)	Percent Water
#1	No Sample	No Sample	No Sample	No Sample	No Sample	48%
#2	66	31	31	17	39	44%
#3	53	27	22	15	31	48%
#4	54	30	20	16	31	53%
Endicott	47	28	15	15	27	57%
#6	49	26	19	14	28	50%
					mean =	50%
					std. dev. =	4.31%

* Bottom of jar is 4mm high, so heights don't add up to total.

mL=[(mm) - 3.2119]/1.6251

Decant III Lab Results

Constants:		
	H_2O Vol to top of outlet (ml):	350
	Sample bottle bottom height (mm)	4

Chemical Metering Pump Setting	Flowrate (gal/min)	(ml/min)
70%	0.0155	58.56
40%	0.0059	22.22

IFT/Concentration	Calibration Equatior	Slope	Exponent
Alcopol	y = 1375.9x-1.815	1375.9	-1.8153
Breaxit	y = 693.24x-1.731	693.24	-1.7313
Exxon/Nalco	y = 45571x-2.9022	45571	-2.9022

Test #1

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	No	0	0	54%	Yes	36 ft	0

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	1200	1550	3450	No Sample	No Sample	No Sample	No Sample	0	58	No Sample	17.0	8.03	
2	5	5000	1200	1550	3450	No Sample	No Sample	No Sample	No Sample	0	58	No Sample	No Sample	No Sample	
3	10	4800	1000	1350	3450	No Sample	No Sample	No Sample	No Sample	0	52	No Sample	39.0	1.78	
4	15	5000	1150	1500	3500	No Sample	No Sample	No Sample	No Sample	0	56	No Sample	No Sample	No Sample	
5	30	5000	1250	1600	3400	59	30	25	48%	0	59	4.0	31.1	2.68	
6	60	5000	1150	1500	3500	42	22	16	48%	0	56	3.1	34.3	2.25	
									average:	0	56	3.5			
									minimum:	0	52	3.1			
									maximum:	0	59	4.0			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
67	7.1	3.8	4.0	2.0	5.8

Test #2

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	70	0.015491823	55%	Yes	36 ft	3058

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	5	5000	1800	2150	2850	35	18	13	47%	0	78	7.0	2.2	328.84	
2	10	5000	2450	2800	2200	39	13	22	27%	3	101	45.3	2.5	260.74	
3	30	4380	2200	2550	1830	40	13	23	27%	13	105	46.8	2.2	328.84	Filled to 4cm below mark
									average:	5	95	33.0			
									minimum:	0	78	7.0			
									maximum:	13	105	46.8			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
45	5.1	2.8	4.0	2.0	4.8

Test #2'

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	70	0.015491823	0%	Yes	36 ft	5408

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
4	5	5000	1400	1750	3250	36	16	16	39%	70	none	22.0	No Sample	No Sample	Water ran out during fill; assumed 0
5	10	5000	200	550	4450	37	13	20	29%	22	none	42.1	2.1	357.82	
6	30	5000	1350	1700	3300	42	13	25	25%	68	none	49.5	1.8	473.36	No further sep'n after 24 and 48 hours for Tank 6
									average:	53	none	37.9			
									minimum:	22	none	22.0			
									maximum:	70	none	49.5			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
83	2.9	0	4.0	2.0	2.0

Test #3

(redo of #2)

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	70	0.015491823	48%	Yes	36 ft	2654

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	4588	2200	2550	2037.5	41	13	24	26%	29	116	48.2	2.5	260.74	Filled to 2.5cm low
2	5	5000	2450	2800	2200	39	13	22	27%	31	117	45.3	2.1	357.82	
3	10	4835	2500	2850	1985	42	12	26	23%	42	123	54.7	1.8	473.36	Filled to 1cm low
4	15	4670	2500	2850	1820	35	11	20	25%	50	127	51.0	1.8	473.36	filled to 2 cm low
5	30	5000	2500	2850	2150	39	11	24	22%	35	119	56.5	2.1	357.82	
6	60	5000	2600	2950	2050	40	10	26	18%	42	123	63.1	1.9	429.11	No further sep'n after 24 hours for Tank 6
									average:	38	121	53.1			
									minimum:	29	116	45.3			
									maximum:	50	127	63.1			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
79	5.8	2.8	4.0	2.0	4.8

Test #4

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	40	0.005878895	32%	Yes	36 ft	989

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	2350	2700	2300	41	18	19	39%	65	169	21.7	2.0	390.96	
2	5	5000	2450	2800	2200	42	17	21	36%	71	175	28.9	2	390.96	
3	10	5000	2600	2950	2050	41	16	21	34%	79	185	32.3	1.8	473.36	
4	15	5000	2400	2750	2250	41	16	21	34%	68	172	32.3	2.3	303.35	
5	30	5000	3000	3350	1650	41	9	28	15%	103	210	69.4	2.1	357.82	
6	60	5000	3000	3350	1650	40	5	31	5%	103	210	90.3	2.2	328.84	
									average:	81	187	45.8			
									minimum:	65	169	21.7			
									maximum:	103	210	90.3			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
80	5.9	1.9	4.0	2.0	3.9

Test #5

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	70	0.015491823	33%	Yes	36 ft	2680

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	2650	3000	2000	39	13	22	27%	81	183	45.3	2.3	303.35	
2	5	4835	2600	2950	1885	41	16	21	34%	84	186	32.3	1.5	659.07	Filled to 1cm low
3	10	4835	2550	2900	1935	38	14	20	31%	81	182	38.0	1.5	659.07	Filled to 1cm low
4	15	4835	2600	2950	1885	40	14	22	29%	84	186	41.4	1.4	747.00	Filled to 1cm low
5	30	5000	2750	3100	1900	40	14	22	29%	87	189	41.4	1.6	586.20	
6	60	4670	2500	2850	1820	40	14	22	29%	84	186	41.4	1.7	525.11	Filled to 2cm low
									average:	83	185	39.9			
									minimum:	81	182	32.3			
									maximum:	87	189	45.3			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
80	5.8	1.9	4.0	2.0	3.9

Test #6

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	70	0.015491823	47%	Yes	36 ft	2606

Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Oil Samples Total Height (mm)	Oil Samples Measured Water Height (mm)	Oil Samples Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	Water Samples IFT Reading (dynes/cm^2)	Water Samples Demulsifier Concentration (ppm)	Comments
1	2	5000	2600	2950	2050	41	13	24	26%	45	125	48.2	1.6	586.20	
2	5	5000	2600	2950	2050	41	14	23	29%	45	125	42.9	1.3	854.57	
3	10	5000	2600	2950	2050	41	16	21	34%	45	125	32.3	1.4	747.00	
									average:	45	125	41.1			Water ran out after 3rd cylinder
									minimum:	45	125	32.3			
									maximum:	45	125	48.2			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
40	5.9	2.8	4.0	2.0	4.8

Test #7

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	40	0.005878895	49%	Yes	36 ft	989

Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Oil Samples Total Height (mm)	Oil Samples Measured Water Height (mm)	Oil Samples Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	Water Samples IFT Reading (dynes/cm^2)	Water Samples Demulsifier Concentration (ppm)	Comments
1	2	5000	2250	2600	2400	41	20	17	44%	13	107	11.1	1.8	473.36	
2	5	5000	2200	2550	2450	41	19	18	42%	9	105	16.4	1.6	586.20	
3	10	5000	2400	2750	2250	40	18	18	40%	24	113	19.6	1.5	659.07	
4	15	5000	2450	2800	2200	41	17	20	36%	28	115	27.0	1.8	473.36	
5	30	5000	2500	2850	2150	41	17	20	36%	32	117	27.0	1.5	659.07	
6	60	5000	2500	2850	2150	40	16	20	35%	32	117	30.5	1.7	525.11	
									average:	23	112	22.0			
									minimum:	9	105	11.1			
									maximum:	32	117	30.5			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
80	5.9	2.9	4.0	2.0	4.9

Test #8

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	40	0.005878895	55%	Yes	36 ft	983

Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Oil Samples Total Height (mm)	Oil Samples Measured Water Height (mm)	Oil Samples Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	Water Samples IFT Reading (dynes/cm^2)	Water Samples Demulsifier Concentration (ppm)	Comments
1	5	5000	2600	2950	2050	41	19	18	42%	17	107	16.4	1.9	429.11	
2	10	5000	2450	2800	2200	40	20	16	46%	4	102	8.7	1.9	429.11	
3	30	5000	2600	2950	2050	39	18	17	41%	17	107	17.4	1.9	429.11	
4	60	5000	2600	2950	2050	41	18	19	39%	17	107	21.7	2.7	226.74	
									average:	14	106	16.1			QC check on ST of oil gives 33.9
									minimum:	4	102	8.7			
									maximum:	17	107	21.7			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
53	6.0	3.3	4.0	2.0	5.3

Test #9

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	70	0.015491823	48%	Yes	36 ft	2639

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	2700	3050	1950	41	15	22	31%	51	128	37.6	2.7	226.74	
2	5	5000	2800	3150	1850	40	13	23	27%	59	132	46.8	2.6	242.82	
3	10	5000	2800	3150	1850	39	13	22	27%	59	132	45.3	2.5	260.74	
4	15	5000	2550	2900	2100	39	12	23	25%	39	122	50.9	2.2	328.84	
5	30	5000	2700	3050	1950	39	14	21	30%	51	128	39.7	2.3	303.35	
6	60	5000	2850	3200	1800	45	14	27	26%	62	134	48.4	2.7	226.74	
									average:	53	129	44.8			QC check on IFT of tap water/USP mineral oil gives 40.3
									minimum:	39	122	37.6			
									maximum:	62	134	50.9			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
81	5.9	2.8	4.0	2.0	4.8

Test #10

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	70	0.015491823	53%	Yes	6ft	2671

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	2400	2750	2250	41	18	19	39%	7	103	21.7	3.0	187.27	
2	5	5000	2500	2850	2150	41	17	20	36%	15	107	27.0	2.6	242.82	
3	10	5000	2580	2930	2070	40	16	20	35%	22	110	30.5	2.5	260.74	
4	15	5000	2550	2900	2100	41	17	20	36%	20	108	27.0	2.5	260.74	
5	30	5000	2600	2950	2050	39	15	20	33%	24	110	34.1	2.5	260.74	
6	60	5000	2500	2850	2150	42	20	18	43%	15	107	13.4	2.2	328.84	
									average:	17	107	25.6			
									minimum:	7	103	13.4			
									maximum:	24	110	34.1			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
82	5.8	3.1	4.0	2.0	5.1

Test #11

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	40	0.005878895	50%	Yes	6ft	915

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	2450	2800	2200	41	21	16	47%	25	112	5.9	1.9	429.11	
2	5	5000	2450	2800	2200	41	20	17	44%	25	112	11.1	1.7	525.11	
3	10	5000	2525	2875	2125	40	19	17	43%	31	115	14.2	1.9	429.11	
4	15	5000	2400	2750	2250	41	18	19	39%	21	110	21.7	1.8	473.36	
5	30	5000	2450	2800	2200	41	18	19	39%	25	112	21.7	1.7	525.11	
6	60	5000	2500	2850	2150	40	18	18	40%	29	114	19.6	1.8	473.36	
									average:	26	113	15.7			
									minimum:	21	110	5.9			
									maximum:	31	115	21.7			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
74	6.4	3.2	4.0	2.0	5.2

Test #12

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	40	0.005878895	0%	Yes	6ft	1929

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	2500	0	0	2500	40	21	15	48%	0	none	3.3	No sample	No Sample	
2	5	2500	0	0	2500	39	22	13	52%	0	none	-5.0	No sample	No Sample	
3	10	2500	0	0	2500	40	21	15	48%	0	none	3.3	No sample	No Sample	
4	15	2500	0	0	2500	40	21	15	48%	0	none	3.3	No sample	No Sample	
5	30	2500	50	400	2100	40	20	16	46%	32	none	8.7	1.8	473.36	A small amount of decanted water, estimated at 50ml. Sample had lots of oil.
6	60	2500	500	850	1650	40	12	24	24%	68	none	52.2	2.2	328.84	
									average:	17	none	11.0			
									minimum:	0	none	-5.0			
									maximum:	68	none	52.2			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
78	3.0	0	4.0	2.0	2.0

Test #13

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	70	0.015491823	0%	Yes	6ft	4398

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	10	2500	0	0	2500	42	22	16	48%	0	none	3.1	No sample	No Sample	
2	15	2500	100	450	2050	41	19	18	42%	36	none	16.4	1.9	429.11	Lots of oil in sample
3	30	2500	100	450	2050	41	12	25	23%	36	none	53.5	2.4	280.80	
4	60	2500	250	600	1900	40	15	21	32%	48	none	35.9	2.0	390.96	
									average:	30	none	27.2			
									minimum:	0	none	3.1			
									maximum:	48	none	53.5			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
45	3.5	0	4.0	2.0	2.0

Test #14

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	70	0.015491823	62%	Yes	36ft	2671

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	3100	3450	1550	40	17	19	37%	37	111	25.0	2.2	328.84	
2	5	5000	3100	3450	1550	39	17	18	39%	37	111	22.9	1.9	429.11	
3	10	5000	no sample	no sample	no sample	no sample	no sample	No Sample	No Sample	No sample	Nosample	No Sample	No sample	No Sample	
4	15	5000	3100	3450	1550	36	16	16	39%	37	111	22.0	1.7	525.11	
5	30	5000	3100	3450	1550	42	14	24	28%	37	111	44.4	2.3	303.35	
6	60	5000	3100	3450	1550	38	14	20	31%	37	111	38.0	2.0	390.96	
									average:	37	111	30.5			
									minimum:	37	111	22.0			
									maximum:	37	111	44.4			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
82	5.8	3.6	2.9	1.5	5.1

Test #15

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Alcopol	40	0.005878895	64%	Yes	36ft	1014

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	2800	3150	1850	41	17	20	36%	0	99	27.0	2.6	242.82	
2	5	5000	2800	3150	1850	36	16	16	39%	0	99	22.0	2.2	328.84	
3	10	5000	2850	3200	1800	37	15	18	35%	1	100	30.2	2.1	357.82	
4	15	5000	2800	3150	1850	39	16	19	36%	0	99	28.5	2.2	328.84	
5	30	5000	3000	3350	1650	38	15	19	34%	18	105	32.2	2.2	328.84	
6	60	5000	3150	3500	1500	39	14	21	30%	34	110	39.7	2.3	303.35	
									average:	9	102	30.0			
									minimum:	0	99	22.0			
									maximum:	34	110	39.7			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
82	5.8	3.7	2.9	1.5	5.2

Test #16

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Exxon/Nalco	70	0.015491823	49%	Yes	36ft	2737

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	0	0	5000	no sample	no sample	No Sample	No Sample	0	0	No Sample	3.2	1558.27	Water sample: lots of oil. All cloudy.
2	5	5000	0	350	4650	38	27	7	68%	0	14	-36.8	3.9	877.61	Water sample: some oil. All cloudy.
3	10	5000	300	650	4350	39	27	8	66%	0	27	-32.9	3.7	1022.48	Water sample: little oil. All cloudy.
4	15	5000	425	775	4225	39	26	9	64%	0	32	-27.4	4.0	815.44	All cloudy.
5	30	5000	1200	1550	3450	36	21	11	54%	0	64	-8.5	4.1	759.04	All cloudy.
6	60	5000	1900	2250	2750	34	14	16	35%	0	93	29.9	4.3	661.05	All cloudy.
									average:	0	38	-15.1			
									minimum:	0	0	-36.8			Cylinders filled with oil/water mixture of low viscosity
									maximum:	0	93	29.9			that was very slow to separate
															Separated water very cloudy

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
84	5.7	2.75	4.0	2.0	4.8

Test #17

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Exxon/Nalco	40	0.005878895	47%	Yes	36ft	1014

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	0	0	5000	39	27	8	66%	0	0	-32.9	3.7	1022.48	Water sample: lots of oil. All cloudy.
2	5	5000	400	750	4250	39	26	9	64%	0	32	-27.4	4.0	815.44	All cloudy.
3	10	5000	800	1150	3850	37	24	9	62%	0	49	-23.0	4.1	759.04	All cloudy.
4	15	5000	1200	1550	3450	40	23	13	54%	0	65	-7.6	4.0	815.44	All cloudy.
5	30	5000	1800	2150	2850	39	23	12	55%	0	91	-10.6	4.4	618.39	All cloudy.
6	60	5000	2300	2650	2350	38	16	18	37%	21	112	26.5	4.1	759.04	All cloudy.
									average:	4	58	-12.5			
									minimum:	0	0	-32.9			Oily phase in cylinders still brown.
									maximum:	21	112	26.5			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
82	5.8	2.75	4.0	2.0	4.8

Test #18

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Exxon/Nalco	70	0.015491823	0%	Yes	6ft	5239

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm²)	Demulsifier Concentration (ppm)	Comments
1	2	2500	0	0	2500	41	23	14	52%	0	none	-4.7	no sample	No Sample	
2	5	2500	0	0	2500	40	21	15	48%	0	none	3.3	no sample	No Sample	
3	15	2500	0	0	2500	40	21	15	48%	0	none	3.3	no sample	No Sample	
4	30	2500	0	0	2500	40	21	15	48%	0	none	3.3	no sample	No Sample	
5	60	2500	0	0	2500	39	21	14	50%	0	none	0.6	no sample	No Sample	
									average:	0	none	1.1			
									minimum:	0	none	-4.7			
									maximum:	0	none	3.3			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
67	3.0	0	4.0	2.0	2.0

Test #19

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Endicott	Exxon/Nalco	70	0.015491823	49%	Yes	36ft	2737

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm²)	Demulsifier Concentration (ppm)	Comments
1	2	2500	0	350	2150	no sample	no sample	No Sample	No Sample	0	28	No Sample	no sample	No Sample	
2	10	2500	1050	1400	1100	41	24	13	55%	26	113	-10.0	3.5	1201.42	Water sample all cloudy.Oil sample had slime pods
3	30	2500	1300	1650	850	40	24	12	57%	65	133	-13.0	3.5	1201.42	Water sample all cloudy.Oil sample had slime pods
4	60	2500	1400	1750	750	38	21	13	51%	81	142	-2.3	3.8	946.33	Water sample all cloudy.
									average:	43	104	-8.4			
									minimum:	0	28	-13.0			
									maximum:	81	142	-2.3			
															Oil/water mixture of low viscosity in cylinders

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
28	5.7	2.8	4.0	2.0	4.8

Test #20

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Endicott	Exxon/Nalco	40	0.005878895	49%	Yes	36ft	1038

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm²)	Demulsifier Concentration (ppm)	Comments
1	2	2500	0	0	2500	39	30	5	75%	0	0	-49.7	no sample	No Sample	Water sample all cloudy.Oil sample had slime pods
2	10	2500	1400	1750	750	41	19	18	42%	81	142	16.4	4.1	759.04	Water sample all cloudy.
3	30	2500	1500	1850	650	34	11	19	25%	97	150	49.4	4.4	618.39	Water sample all cloudy.
4	60	2500	1600	1950	550	29	12	13	34%	113	158	31.8	5.2	380.81	Water sample all cloudy.
									average:	73	112	12.0			
									minimum:	0	0	-49.7			
									maximum:	113	158	49.4			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
28	5.7	2.8	4.0	2.0	4.8

Test #21

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Endicott	Alcopol	70	0.015491823	48%	Yes	36ft	2639

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	2500	0	350	2150	40	16	20	35%	0	29	30.5	2.6	242.82	Oil sample had slime pods on bottom
2	10	2500	1600	1950	550	39	14	21	30%	116	164	39.7	3.4	149.21	Oil sample had slime pods on bottom
3	30	2500	1650	2000	500	35	13	18	31%	124	168	38.4	2.9	199.16	Oil sample had slime pods on bottom
4	60	2500	1400	1750	750	47	11	32	18%	85	147	64.4	3.5	141.56	May have run out of emulsion, no slime pods.
									average:	81	127	43.3			
									minimum:	0	29	30.5			
									maximum:	124	168	64.4			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
27	5.9	2.8	4.0	2.0	4.8

Test #22

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Breaxit	40	0.005878895	48%	Yes	36ft	1001

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	2350	2700	2300	39	18	17	41%	24	113	17.4	2.3	163.92	
2	5	5000	2300	2650	2350	39	19	16	44%	20	111	11.8	1.7	276.64	
3	10	5000	2300	2650	2350	42	20	18	43%	20	111	13.4	2.2	177.03	
4	15	5000	2250	2600	2400	40	20	16	46%	16	109	8.7	2.3	163.92	
5	30	5000	2250	2600	2400	40	20	16	46%	16	109	8.7	2.0	208.79	
6	60	5000	2350	2700	2300	40	20	16	46%	24	113	8.7	2.0	208.79	
									average:	20	111	11.5			
									minimum:	16	109	8.7			
									maximum:	24	113	17.4			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
81	5.9	2.8	4.0	2.0	4.8

Test #23

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Breaxit	70	0.015491823	47%	Yes	36ft	2606

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	5000	2150	2500	2500	40	20	16	46%	11	106	8.7	1.7	276.64	
2	5	5000	2400	2750	2250	38	17	17	40%	30	117	20.7	1.8	250.57	
3	10	5000	2550	2900	2100	40	18	18	40%	41	123	19.6	1.6	307.25	
4	15	5000	2500	2850	2150	40	14	22	29%	37	121	41.4	1.6	307.25	
5	30	5000	2600	2950	2050	38	14	20	31%	45	125	38.0	1.3	440.16	
6	60	5000	2600	2950	2050	38	17	17	40%	45	125	20.7	1.6	307.25	
									average:	35	120	24.9			
									minimum:	11	106	8.7			
									maximum:	45	125	41.4			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
80	5.9	2.8	4.0	2.0	4.8

Test #24

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Breaxit	40	0.005878895	0%	Yes	36ft	2077

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	2500	0	0	2500	42	21	17	46%	0	none	8.3	no sample	No Sample	
2	5	2500	0	0	2500	41	21	16	47%	0	none	5.9	no sample	No Sample	
3	10	2500	0	0	2500	42	22	16	48%	0	none	3.1	no sample	No Sample	
4	15	2500	0	0	2500	41	21	16	47%	0	none	5.9	no sample	No Sample	
5	30	2500	0	0	2500	42	22	16	48%	0	none	3.1	no sample	No Sample	
6	60	2500	0	0	2500	39	21	14	50%	0	none	0.6	no sample	No Sample	
									average:	0	none	4.5			
									minimum:	0	none	0.6			
									maximum:	0	none	8.3			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
84	2.8	0	4.0	2.0	2.0

Test #25

Oil		Demulsifier Setting	Flow rate	Free Water	Inline Mixer	Circuit Length	Demulsifier Dose Rate (ppm)
Hydrocal 80/5/15	Breaxit	70	0.015491823	0%	Yes	36ft	4789

						Oil Samples							Water Samples		
Tank Number	Settling Time	Volume Collected (mL)	Decanted Water (mL)	Corrected Water (mL)	Emulsion Remaining (mL)	Total Height (mm)	Measured Water Height (mm)	Oil Height (mm)	Water Content (%)	Emulsified Water Removed (%)	Free Water Removed (%)	Emulsion Dehydration (%)	IFT Reading (dynes/cm^2)	Demulsifier Concentration (ppm)	Comments
1	2	2500	0	0	2500	43	23	16	50%	0	none	0.5	no sample	No Sample	
2	10	2500	0	0	2500	39	21	14	50%	0	none	0.6	no sample	No Sample	
3	30	2500	0	0	2500	39	20	15	47%	0	none	6.2	no sample	No Sample	
4	60	2500	0	0	2500	40	20	16	46%	0	none	8.7	no sample	No Sample	May have run out of demulsifier at end
									average:	0	none	4.0			
									minimum:	0	none	0.5			
									maximum:	0	none	8.7			

Fill Time (s)	Fluid Flow (gpm)	Water Flow (gpm)	Emulsion Flow (gpm)	Oil Input (gpm)	Water Input (gpm)
49	3.2	0	4.0	2.0	2.0

Test Number	Oil	Free Water (%)	Demulsifier	Demulsifier Concentration (ppm)	Inline Mixer (Y/N)	Circuit Length (ft)	Emulsion Dehydration (%)			Concentration of Demulsifier in Decant Water (ppm)			Comments
							2 or 5 min.	10 or 15 min.	60 min.	2 min.	10 min.	60 min.	
1	Hydrocal 80/5/15	54	None	0	Yes	36	No Sample	No Sample	3	8	2	2	Timer fouled up & start estimated
2	Hydrocal 80/5/15	55	Alcopol	3058	Yes	36	7	45	47	329	261	329	Water ran out and test divided into 2 & 2'
2'	Hydrocal 80/5/15	0	Alcopol	5408	Yes	36	22	42	50	No Sample	358	473	
3	Hydrocal 80/5/15	48	Alcopol	2654	Yes	36	48	55	63	261	473	429	Redo of #3
4	Hydrocal 80/5/15	32	Alcopol	989	Yes	36	22	32	90	391	473	329	
5	Hydrocal 80/5/15	33	Alcopol	2680	Yes	36	45	38	41	303	659	525	
6	Hydrocal 80/5/15	47	Alcopol	2606	Yes	36	48	32	No Sample	586	747	No Sample	Water ran out after third cylinder
7	Hydrocal 80/5/15	49	Alcopol	989	Yes	36	11	20	30	473	659	525	
8	Hydrocal 80/5/15	55	Alcopol	983	Yes	36	16	9	22	429	429	227	
9	Hydrocal 80/5/15	48	Alcopol	2639	Yes	36	38	45	48	227	261	227	
10	Hydrocal 80/5/15	53	Alcopol	2671	Yes	6	22	30	13	187	261	329	
11	Hydrocal 80/5/15	50	Alcopol	915	Yes	6	6	14	20	429	429	473	
12	Hydrocal 80/5/15	0	Alcopol	1929	Yes	6	3	3	52	No Sample	No Sample	329	
13	Hydrocal 80/5/15	0	Alcopol	4398	Yes	6	No Sample	3	36	No Sample	429	391	
14	Hydrocal 80/5/15	62	Alcopol	2671	Yes	36	25	22	38	329	525	391	
15	Hydrocal 80/5/15	64	Alcopol	1014	Yes	36	27	30	40	243	358	303	
16	Hydrocal 80/5/15	49	Exxon Nalco	2737	Yes	36	-37	-33	30	878	1022	661	Cylinders filled with oil/cloudy water, very slow to separate
17	Hydrocal 80/5/15	47	Exxon Nalco	1014	Yes	36	-33	-23	26	1022	759	759	Oily phase in cylinders still brown.
18	Hydrocal 80/5/15	0	Exxon Nalco	5239	Yes	6	-5	3	1	No Sample	No Sample	No Sample	
19	Endicott	49	Exxon Nalco	2737	Yes	36	No Sample	-10	-2	No Sample	1201	946	Oil/water mixture of low viscosity in cylinders
20	Endicott	49	Exxon Nalco	1038	Yes	36	-50	16	32	No Sample	759	381	
21	Endicott	48	Alcopol	2639	Yes	36	30	40	64	243	149	142	May have run out of emulsion.
22	Hydrocal 80/5/15	48	Breaxit	1001	Yes	36	17	13	9	164	177	209	
23	Hydrocal 80/5/15	47	Breaxit	2606	Yes	36	9	20	21	277	307	307	
24	Hydrocal 80/5/15	0	Breaxit	2077	Yes	36	8	3	1	No Sample	No Sample	No Sample	
25	Hydrocal 80/5/15	0	Breaxit	4789	Yes	36	1	1	9	No Sample	No Sample	No Sample	May have run out of demulsifier at end

APPENDIX C - OHMSETT TEST PROTOCOL

Test Plan for

Task Order No. 422

EXAMINING THE FATE OF EMULSION BREAKERS USED FOR DECANTING

Sponsors: U.S. DEPARTMENT OF THE INTERIOR
Minerals Management Service
Contact: Robert Smith COTR
(703) 787-1580

and

ALASKA CLEAN SEAS
Prudhoe Bay, AK
Contact: Lee Majors
(907) 659-3207

Client: S.L. Ross Environmental Research Ltd
Ottawa, ON

Time Frame: NOVEMBER 2003

September 30, 2003

Second Draft Test Plan

Test Plan for

EXAMINING THE FATE OF EMULSION BREAKERS USED FOR DECANTING

Task Order No. 422

Sponsors: US DEPARTMENT OF THE INTERIOR
Minerals Management Service
Contact: Bob Smith COTR
(703) 787-1580

and

ALASKA CLEAN SEAS
Prudhoe Bay, AK
Contact: Lee Majors
(907) 659-3207

Client: S.L. Ross Environmental Research Ltd.
200-717 Belfast Rd.
Ottawa, ON
K1G 0Z4

Time Frame: November 2003

Prepared By: ____________________________ and ______________________________
Dave DeVitis - Test Director Ian Buist - SL Ross Project Officer

APPROVALS:

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SL Ross: ________________________
Ian Buist - Project Manager

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1. INTRODUCTION

A test program is planned to examine the fate of chemical emulsion breakers (also known as demulsifiers) when they are injected into a recovered fluid stream from open water containment and recovery operations to aid in decanting of recovered water to conserve temporary storage capacity. The primary objective is to quantify how much of the emulsion breaker ends up in the decanted water, and how much stays with the oily phase.

2.1 Background

The preferred approach to cleaning up an oil spill is to contain and thicken the oil slick(s) with booms and then place skimmers in the oil or emulsion to recover it. The recovered fluids are placed in temporary storage containers for transfer to larger storage vessels or for direct input into waste recycling and disposal systems. The most common type of high-capacity skimmer in use today is the weir skimmer. These skimmers often recover a large amount of water, both in the form of emulsified water and free water, when operating in waves. In some cases, the transfer pump built into the skimming system can impart enough energy to cause additional emulsification of the recovered fluids. The problem is that the recovered water (both emulsified and free) dramatically reduces the temporary storage space available at the site of skimming operations; this can result in having to stop skimming prematurely when the storage capacity is reached and having to wait until empty, temporary storage containers arrive at the response site.

A series of lab-scale and mid-scale tests with and without the use of emulsion breakers were completed recently that give some quantitative insight into the oil/water separation processes occurring in temporary storage devices (SL Ross 1998, 1999 and 2002). The objective of these tests was to determine the optimum time to decant the water and maximize the available on-site storage space during a skimming operation as well as the efficacy of adding emulsion breakers into the recovery stream to allow decanting of emulsified water. The results indicate that "primary break" (the initial separation of the recovered fluid into a layer containing most of the oil and a layer containing most of the free water) occurs within a few minutes to one hour, depending on the physical characteristics of the oil. Rapidly decanting this free water layer, in appropriate situations, may offer immediate increases of 200 to 300% in available temporary storage space. The addition of emulsion breakers can increase the amount of water that can be decanted, in the same time frame. Addition of the emulsion breaker seems to increase the oil content of the separated water significantly. At least one technology exists that can rapidly and effectively remove this dispersed oil from the decanted water.

A significant potential impediment to the application of emulsion breakers to extend temporary storage capacity is the ultimate fate of the emulsion breaking chemical(s). If the demulsifier remains with the oil, there should be no problem with their use; however, if they partition significantly into the separated water, they will be discharged into the environment when the water is decanted. Some demulsifiers are strong ionic surfactants that have a relatively high toxicity (on the order of tens of ppm) because their surface activity can disrupt the gills of fish. If

a recovered fluid consists of 50% free water and 50% of an emulsion containing 75% water and all the emulsion breaker used to treat it (typically dosed at 1:400 demulsifier:emulsion) transfers into the water, the decanted water could contain some 1400 ppm of demulsifier. Discharge regulations in some jurisdictions would not permit the decanting of such water to the ocean. Other demulsifiers are non-ionic, and tend to be much less toxic. Some emulsions are easier to break with ionic surfactants, and some are easier to break with non-ionic surfactants.
During the July 2001 decanting tests at Ohmsett using emulsion breakers, the separated water foamed easily when agitated, providing strong qualitative evidence that it contained significant amounts of surfactant.

A series of small-scale tests with a scale-model piping system simulating a weir skimmer recovery system (used in the previous decanting study – SL Ross 2002) was completed this summer to determine the effects of several variables on the concentration of demulsifier in decanted water. As well, a technique for determining the concentration of demulsifier in the decanted water was perfected. This involves measuring the interfacial tension of the decanted water against a highly-refined mineral oil, such as USP grade (pharmaceutical) and comparing the measured interfacial tension with a calibration curve of prepared samples of different concentrations of the demulsifier in question).

The research idea here is to study the partitioning of different emulsion breakers injected into a recovery system at full-scale at Ohmsett. Experiments have been developed based on the lessons learned from the scale-model tests and the earlier decanting studies. They are designed to assess the fate of the demulsifier chemical(s).

1.2 Objectives

The objective of the proposed study is to determine the partitioning of different chemical emulsion breakers between oily and water phases when they are used to enhance decanting of recovered water from offshore skimming operations.

1.3 Organizations Participating in the Testing

All those who will be at the Ohmsett Facility are advised that they are subject to US Navy, Naval Weapons Station Earle (NWS-Earle) and Department of Interior, Minerals Management Service rules and regulations. The most obvious of those regulations involve health, safety, and security. All operational personnel must have 40-hour or 24-hour HAZWOPER training and an introductory Ohmsett Health & Safety training session. Access to the site is controlled by NWS-Earle. Use of a camera requires a permit issued by a NWS-Earle Base Security Officer. **Unless informed otherwise by the Site Manager, testing is on weekdays only, and begins at 0700.**

Minerals Management Service (MMS):

- Funds the operation of Ohmsett
- Provides the Work Order to MAR, Inc.
- Reviews and approves the Work Order Proposal
- Reviews and Approves the Final Report

SL Ross Environmental Research and Alun Lewis Oil Spill Consultancy:

- Prepares the Test Plan with MAR input
- Assists with the equipment assembly and checking
- Assists with the equipment operation
- Writes the final report

MAR, Inc:

- Prepares the Test Plan with SL Ross
- Provides the Desmi Terminator skimmer
- Prepares test fluids and confirms suitability
- Collects test data including oil distribution rates and volumes, volumes recovered, initial oil properties, and recovered oil and water analysis
- Collects background data including oil/water temperatures and wave data
- Photographs and videotapes the trials
- Provides raw data to SL Ross
- reviews the Draft Final Report

1.4 Test Personnel

The test personnel assignments are listed in Table 1.

Table 1: Test Personnel Assignments

Personnel	**Location**	**Duties**
Site Manager Bill Schmidt	Control Tower	Oversight
Test Engineer/Director Dave DeVitis	Test Basin	Overall supervision of testing
QA Engineer Alan Guarino	Roving	Monitors fluid sampling, data collection and test parameter accuracy.
Bridge Operator/Instrumentation Tech. Don Backer	Control Tower	Operates traveling bridge and data acquisition system
Chemical Technician Susan Cunneff	Oil Analysis Lab	Handles and analyzes fluid samples.
H&S Specialist Rich Naples	Roving	Monitors personnel safety.
Fluid Transfer Technician Dave Knapp	Main Tank Deck	Operates oil transfer system, Operates fill and off-loading pumps
Video Technician Rob Stewart	Roving	Operates hand-held video and digital still camera
Oil Recovery Technician Don Snyder	Auxiliary Bridge	Operates Ohmsett recovery tank valves, measure fluid recovery depths samples fluids
SL Ross Sr. Engineer Ian Buist	Roving	Provides advice on system operation and test suitability
Chemist Alun Lewis	Roving	Provide advice on tests and chemistry issues.
Writer/Editor Kathleen Nolan	Control Tower	Collate Raw Data and Deliver Test Documentation

2. TEST PROCEDURES

2.1 Preparation

The preparations for the tests include:

- Obtaining 1200 gallons of Hydrocal 300, 75 gallons of No. 6 Fuel Oil, 225 gallons of automotive diesel and 220 gallons of fresh Endicott crude, 1 gallon of Unichem RNB-60425 and 6 gallons of Alcopol O 70% PG (aka Drimax 1235B)
- Installing skimmer, hoses and Globe boom
- Conducting required safety checks and notifications.

2.1.1 Test Set-up and Instrumentation

All tests are to be conducted in a stationary position (i.e., no towing down the tank). A preliminary layout of the test equipment is given in Figure 1.

The test area will consist of 40 feet of 24"-Globe boom deployed in an isosceles triangle (11’10” base and 12’6” height) between the auxiliary bridge and the main bridge. The boomed area will be approximately 6.8 m^2 (73 ft^2). The smaller test area being used this time, compared to the earlier tests, is so that less oil is consumed in each test, allowing more tests to be conducted in the one-week test window. The Desmi Terminator skimmer will be placed in the test area and operated from the side of the tank or the Auxiliary Bridge. The skimmer discharge will be directed to the oil recovery tanks on the auxiliary bridge. The separated water from the oil recovery tanks will be directed to a temporary water sampling Nalgene tank, then to a temporary holding tank on the deck and finally, pumped to a Rain-for-Rent tank located by the filter for treatment. Oil or emulsion from the oil recovery tanks will be processed to remove as much water as possible, then stored for disposal. The Hydrocal cannot be re-used for testing at Ohmsett because it will contain some diesel fuel, Bunker “C” and residual demulsifier that would reduce it’s interfacial tension.

Portable video and digital still cameras will be used to record the testing from various perspectives.

Waves are generated at the south end of the Test Basin and controlled by the Bridge Operator in the Control Tower at the north end. A local readout of the wave generator cycles per minute is on the control console. The wave profiles will be recorded using a Datasonics ultrasonic distance meter. The signal from the wave meter will be recorded and analyzed after testing to confirm the wave characteristics.

2.1.2 Wave conditions

Two wave conditions will be generated during this test series. Their nominal characteristics are defined in Table 2.

Table 2. Nominal Wave Characteristics

Wave No.	Stroke (in.)	CPM	Type	Nominal $H^{1/3}$ (in.)	Wave Length (ft.)	Period (sec)
#1	3	22	Sinusoidal	16.5	37	2.8
#2	3	35	Sinusoidal	15	15	1.7

2.1.3 Emulsion Preparation

At the beginning of the tests, and subsequently as required, emulsions will be prepared. A gear pump will be used to prepare the emulsion, since large quantities of a consistent quality are required on a daily basis. The use of pumps to create emulsions for equipment testing is well known and widely utilized in North America and Europe (eg., Gåseidnes 1993). The capability of the Hydrocal 300 test oil doped with 5% No. 6 Fuel Oil and 15% automotive diesel to form a meso-stable emulsion that is completely broken by the Alcopol emulsion breaker has been confirmed by lab tests. In addition, this year, an Alaskan crude oil , Endicott, will be included in the tests. The lab tests have shown that it will form a stable emulsion with seawater at 20°C. The target properties of the emulsion are a 50% (vol) water content. A 50% water content was chosen because it can be prepared reasonably quickly using the pump technique with little risk of inverting the emulsion, as can occur with higher water contents. The Hydrocal-based emulsion will be prepared, using the Viking gear pump plumbed to the Main Bridge oil tank as shown in Figure 2, as follows:

1) Add 1840 L (560 gallons) of Hydrocal 300 to the 1500-gallon Main Bridge oil tank.
2) Warm No. 6 Fuel Oil to 40 to 45°C using electric band heater
3) Turn on Viking gear pump (at 350 rpm, nominally 110 gpm) and Moyno oil distribution pump (nominally 350 gpm) and recirculate tank contents.
4) Add 400 L (105 gallons) of automotive diesel to the Main Bridge oil tank.
5) Slowly (2.75 gpm) add 145 L (35 gallons) of warm No. 6 Fuel Oil to the suction side of the Viking gear pump while circulating.
6) Continue recirculating for 10 minutes after last No. 6 Fuel Oil added.
7) Stop Viking gear pump and Moyno oil distribution pump.
8) Record volume of oil in tank using ultrasonic probe.
9) Restart Viking gear pump and Moyno oil distribution pump and recirculate tank contents.
10) Open water valve and draw 57 L/min (15 gpm) of tank water into suction side of Viking pump.
11) Monitor tank level until a total of 2650 L (700 gallons) of tank water has been added (for a total volume in the tank of 5300 L (1400 gallons).
12) Continue recirculating for 30 minutes.

Each test will require an estimated 850 L (225 gallons) of emulsion, allowing approximately six runs per batch of Hydrocal-based emulsion.

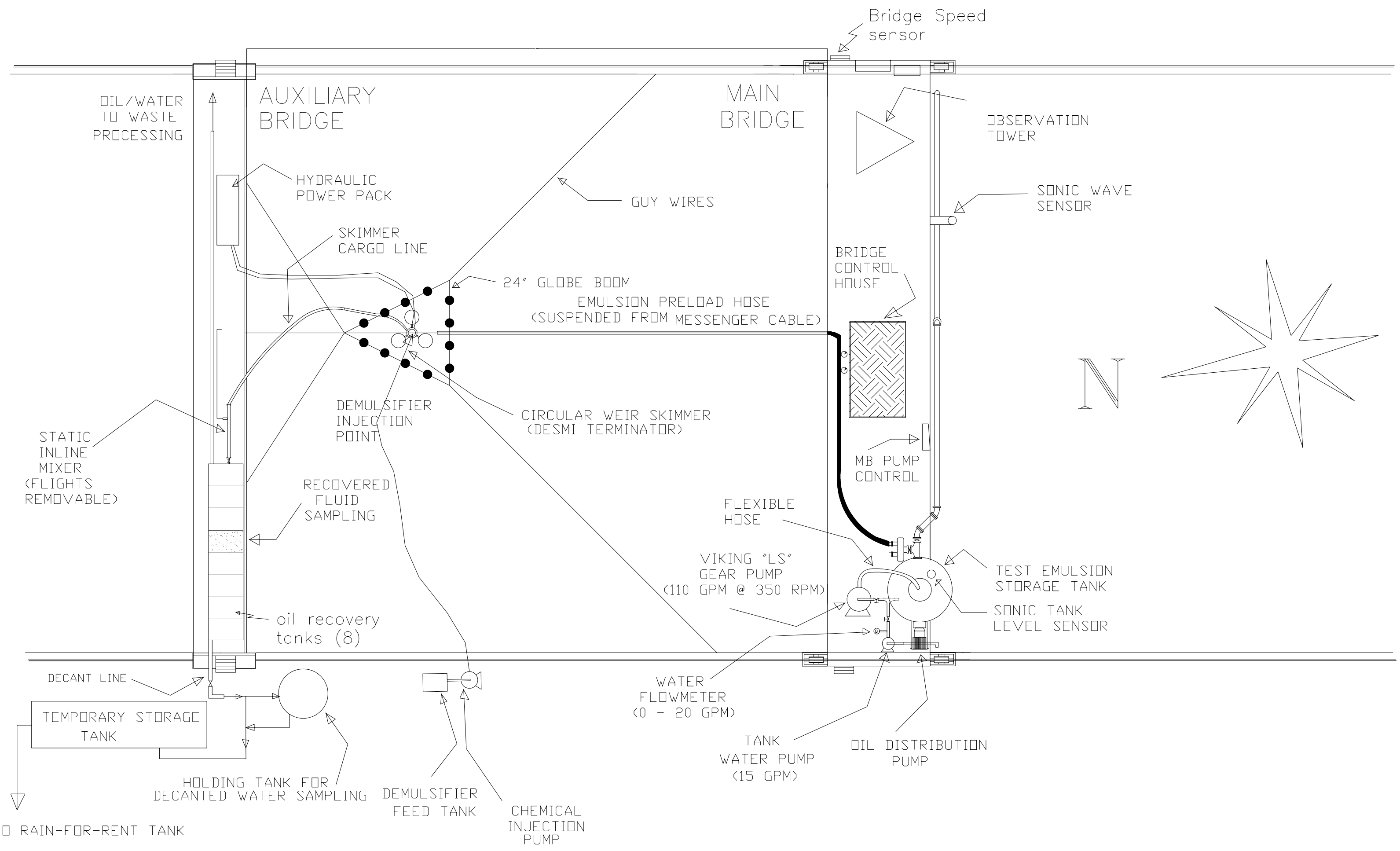

Figure 1. Test Set-up.

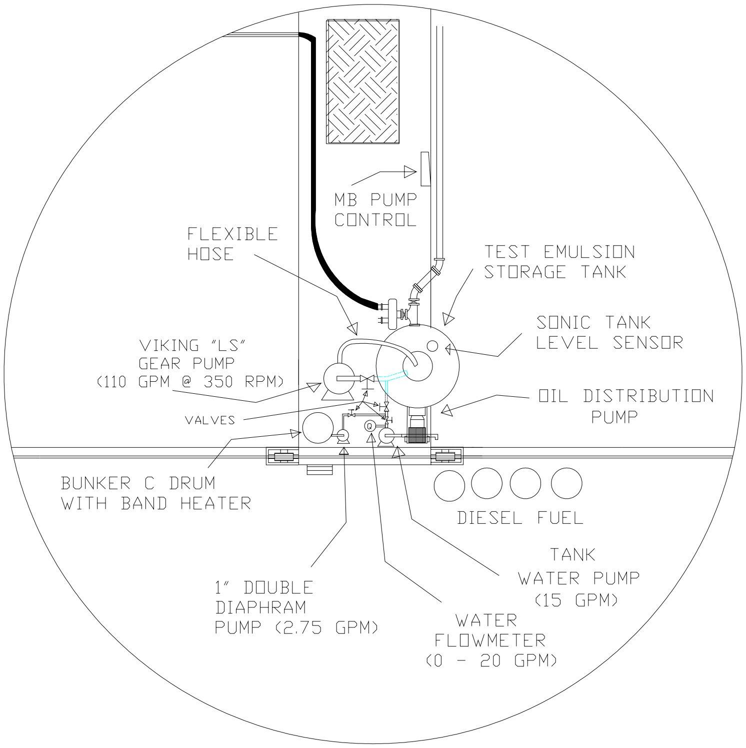

Figure 2. Plumbing for Main Bridge Oil Tank Emulsion Mixing and Oil Distribution.

The procedure for preparing the emulsions with Endicott crude will be similar:

1) Add 820 L (220 gallons) of fresh Endicott crude to the 1500-gallon Main Bridge oil tank.
2) Record volume of oil in tank using ultrasonic probe.
3) Turn on Viking gear pump (at 350 rpm, nominally 110 gpm) and Moyno oil distribution pump (nominally 350 gpm) and recirculate tank contents.
4) Open water valve and draw 57 L/min (15 gpm) of tank water into suction side of Viking pump.
5) Monitor tank level until a total of 820 L (220 gallons) of tank water has been added (for a total volume in the tank of 1640 L (440 gallons).
6) Continue recirculating for 30 minutes.

This will allow two full-scale tests with the Endicott-based emulsion.

2.2 Testing

2.2.1 Test Descriptions

The following procedures are suggested for each test:

Before each test the Emulsion Recovery Rate (ERR) for the skimmer will be estimated and the volume of emulsion removed from the boomed area during the previous test calculated. The aim is to pump emulsion into the boomed area at the same rate that the skimmer removes it so that a constant thickness of emulsion is being presented to the skimmer. The following procedures are then used:

1. The required volume of test emulsion is added to the boomed area (to a total of 180 gallons) to make up the desired slick thickness (100 mm).
2. The Main Bridge distribution pump speed is set to supply fresh test emulsion at the ERR estimated for the test.
3. The waves are turned on at the desired setting and allowed to come to apparent steady state (this requires about two minutes). The data acquisition system is started.
4. The emulsion distribution pump is started and the skimmer turned on, with its discharge directed to recovery tank #8. The chemical injection pump is started with flow to the skimmer hopper at the desired demulsifier flow rate (nominally 1/400th or 1/1000th of the Fluid Recovery Rate).

5. When the cargo line is purged, the skimmer discharge is directed to four recovery tank cells sequentially (i.e., fill cell #7, then #6, etc., ending with cell #4). The target volume of emulsion (exclusive of free water) in each cell is 150 L, or 40 gallons.
6. The time when filling each tank cell is started and finished is recorded. The depth of fluid in each cell is measured and recorded.
7. After the last tank cell (#4) is filled, the emulsion distribution pump, demulsifier injection pump, skimmer and waves are stopped.
8. Simultaneously with the filling operation, two minutes after tank cell #7 was filled, it is decanted until the discharge from the bottom is "black". The water is sent to a temporary sampling tank and not poured back into the test basin.
9. The remaining emulsion recovery tank cells are decanted in sequence at 10, 30 and 60 minutes after the time they were filled.
10. For each cell in each test, the decanted water is directed to a Nalgene temporary holding tank on the deck beside the auxiliary bridge. When all water from the selected cell is transferred, the contents of the temporary holding tank are thoroughly mixed with an electric, bladed mixer and allowed to settle for five minutes to allow large droplets of emulsion to surface. The surface emulsion is removed with a sorbent pad, and then the temporary sampling tank is drained to the temporary storage tank. Two small water samples, one for oil content analysis and one for dissolved demulsifier analysis, are taken when half the water had drained from the temporary sampling tank. Note that cell #8 is also decanted directly to the temporary storage tank for processing at the end of the test.
11. The depth of fluid remaining in each cell is measured (these depths, combined with the initial depths, are used to calculate the volumes of recovered product, decanted water and emulsion remaining).
12. Each recovery tank cell (#7 through #4) is mixed and sampled to determine the water content of the fluid remaining.
13. The contents of the recovery tank cells are transferred for waste processing.

2.2.2 Test Schedule

Test Matrix Variables

- One circular weir skimmer (representative of OSRO stockpiles)
    - Desmi Terminator (USCG/Ohmsett) - nominal ORR in waves 20 m^3/hr (90 USgpm)
- One slick thickness (representing a thickness typically expected for large-scale boom/skimmer operations)
    - 100 mm (requires 684 L - 180 US gallons - preload in triangular boomed area)
- One demulsifier injection point
    - into the skimmer mouth
- Two Wave Conditions

  - wave #1, $H^{1/3}$= 15" with λ = 37'
  - wave #2, $H^{1/3}$= 15" with λ = 15'
- Two demulsifier dose rates (1:400 and 1:1000)
- Three parent oil/demulsifier combinations (Hydrocal/Alcopol, Hydrocal/Unichem RNB-60425 and two tests with Endicott/Unichem RNB-60425)
- Two control tests (no demulsifier injected)

Varying all of these gives 9 individual tests. It is proposed that one duplicate test be run to bring the total to 10 test runs. Table 3 gives the preliminary schedule for the tests.

Table 3. Preliminary Schedule of Tests

Day	Test No.	Emulsion Parent Oil	Demulsifier	Demulsifier Dose	Wave No.
1	1	Hydrocal 80/5/15	none	none	1
2	2	Hydrocal 80/5/15	none	none	2
2	3	Hydrocal 80/5/15	Alcopol O 70% PG	1:400	1
2	4	Hydrocal 80/5/15	Alcopol O 70% PG	1:400	2
3	5	Hydrocal 80/5/15	Alcopol O 70% PG	1:1000	1
3	6	Hydrocal 80/5/15	Alcopol O 70% PG	1:1000	2
3	7 (duplicate)	Hydrocal 80/5/15	Alcopol O 70% PG	TBD	TBD
3	8	Hydrocal 80/5/15	Unichem RNB-60425	1:1000	2
4	9	Endicott crude	Unichem RNB-60425	1:1000	1
4	10	Endicott crude	Unichem RNB-60425	1:1000	2

2.2.4 Sample Analyses

Each test will involve 4 oil-in-water analyses (i.e., TPH with solvent extraction/IR), 4 water-in-oil analyses (Centrifuge -perhaps with a little demulsifier added - a well-mixed sample from each cell in the recovery tank after it has been decanted) and 4 interfacial tension measurements against USP mineral oil to determine the concentration of demulsifier in the decanted water. For the 10 tests this totals 40 TPH, 40 water-in-oil analyses and 40 IFTs. Duplicates (at 10%) would raise the totals to 44 for each. A total of about 10 IFT measurements will be required to construct calibration curves for the concentration of the two emulsion breakers in tank water.

In addition a rheological work up on each batch of emulsion prepared (viscosity at different shear rates with the Haake), a water content and a density is desired. It is estimated that about three batches of emulsion will be required.

3. DELIVERABLES

3.1 Test Data

Original data logs, computer generated data files, video and photos will be kept on file at Ohmsett. Copies or duplicates will be created and delivered to SL Ross to generate the final data report. The Ohmsett deliverable items will include:

- Raw computer generated data files.
- Observations on tests.
- All manually generated logs.
- Ohmsett laboratory oil-in-water, water-in-oil, rheology, density and interfacial tension analyses.

3.2 Video Documentation

High-resolution, commercial-grade videos (S-VHS) shall be produced with titles that clearly state the test name, time of day, date and test number. Video documentation will be duplicated in VHS format as deliverable items for SL Ross. Logs will accompany the videos specifying test number, date, time and location on the video tape. Photos, digital and 35 mm, will also be duplicated as deliverables. All original video documentation will be maintained at Ohmsett.

4. HEALTH AND SAFETY JOB HAZARD ANALYSIS

4.1 Introduction

A job hazard analysis is a means of preventing or controlling hazardous conditions associated with testing activity. Analysis begins by determining the basic tasks of a job. Each task is then analysed to identify potential hazards associated with it. It will then be possible to develop control measures for the hazards identified. Prior to any test activity, personnel involved with the test are informed of potential hazards and controls for an understanding of their health and safety responsibilities.

4.2 Hazardous Materials

Fuel:

- Hydrocal 300
- Automotive Diesel
- Endicott crude oil
- No. 6 Fuel oil

Other Products/Chemicals:

- Alcopol O 70% PG (aka Drimax 1235B) emulsion breaker
- Unichem RNB-60425
- Citrus (Cleaning Agent)
- Steam Cleaner (Cleaning Agent)
- Hydraulic Fluid

According to available product safety information, respiratory protection is not needed, as the evaporation rate of the oil is negligible, resulting in the off-gassing of little, if any, vapors.

All personnel involved in testing are informed of associated health hazards, as well as the proper personal protective measures required to eliminate exposure to the oil, in accordance with OSHA Hazard Communication Standard requirements. A Material Safety Data Sheet is maintained for test oils, chemicals or various products, and will be available to each employee involved in testing.

In addition to the above-mentioned products/chemicals, there are laboratory and sampling chemicals. Although specific to laboratory operations, Material Safety Data Sheets will be made available for review. They are: Carbon Tetrachloride, Toluene, Hydrochloric Acid, and Propane. In the event additional lab chemicals are utilized, MSDS sheets are available for review.

4.3 Generic Job Safety Analysis

The following table lists basic or generic tasks necessary for the “Extending Temporary Storage Capacity Offshore with Emulsion Breakers” test. Hazards associated with the tasks are listed with preventive measures to be followed by affected personnel.

Table 4. Task Hazard Prevention

TASK	HAZARDS	PREVENTION/CONTROL
1) Materials handling, general set-up	a) Lifting material(s) (muscle strains, back injuries)	a) Use proper lifting techniques; lift with your legs, not your back; get help for heavy loads, use mechanical devices (i.e., fork lift, job cranes).
	b) Forklift operations (objects striking)	b) Follow acceptable safe practices for operators.
	c) Jib crane(s) operations (objects striking)	c) Do not stand under raised loads. Do not exceed capacity of jib crane. Use one signal man.
	d) Mobile crane (contractor personnel, objects striking)	d) Only qualified crane operator and signal man will control lift operations. Do not stand under raised loads.
	e) Hand/power tools (muscle strains, pinch points, electrocution)	e) Use correct tool for the job, use correct PPE and proper body positioning when handling tools. Inspect all power tools to ensure no frayed or exposed wires exist, equipment is grounded and insulated and GFI’s extension cords etc. are functioning properly.
2) Boom assembly and placement into tank (set-up)	a) Rigging from work boat (falls)	a) Personnel on work boat MUST wear PFD’s. Evenly distribute weight and do not overload. Life preservers are in place as needed.
	b) Cable handling (pinch points)	b) Wear hand protection during rigging.
	c) Positioning bridges (objects striking)	c) Have appropriate lines of continual communication.
	d) Positioning boom equipment. Mobile crane operations (objects striking)	d) No one permitted under heavy loads. Only contract operator and signal man will control lift operations.

3) Oil transfer	a) Spilled oil/deck area (slip/fall hazard) b) Pressurized equipment/pumps/hoses/ lines (pressure release, objects striking)	a) Clean spills on deck/bridges immediately. Utilize spill equipment, as required. b) Inspect all equipment prior to use. Do not use damaged equipment. Replace cracked hoses, broken gauges prior to pressurization. Inspect for leaks. Use adequate PPE (hard hat, gloves, face shield).
4) Bridge operation positioning and movement	a) Bridge movement (objects striking, falls)	a) No personnel permitted on the deck, under moving cables or in motor perimeter while in operation. b) All guard rails must be in place and secured while working on moving bridge. c) Continued and open communications with bridge operator is mandatory. While testing, only authorized personnel involved with the test allowed in bridge control area (third floor).
5) Oil addition to test tank	a) Splashing/spraying oils while transferring to Test Tank. [Slips/falls, exposure (skin/eyes), exposure (inhalation)] b) Pressure release (object striking, pinch points)	a) Wear appropriate PPE (protective clothes, goggles/face shield, nitrile gloves). Air sample base line tests will be taken. Appropriate respirators will be worn as required. Technician will keep bridge/deck as oil-free as possible. b) Utilization of damaged hoses for faulty equipment is prohibited. Check all piping, hoses, hose connections, etc. prior to use. Bleed pressure prior to disconnect. Wear PPE to include protective clothes, goggles/face shield, hard hat, nitrile gloves.
6) Operations of skimmer system(s)	a) General operation for Collection/Skimming (high noise levels) b) General operations (hydraulics, striking objects) c) Deployment and general operations (testing)	a) Sound level readings will be taken and protective devices will be issued should action levels be reached. b) All hoses are to be inspected prior to use to ensure adequate rating. All fittings will be inspected to ensure adequate ratings. Hoses and fittings will be securely tightened. c) Wear appropriate PPE (protective clothes goggles/face shield, gloves, appropriate respirators will be worn as required.

7) Wave generation	a) Moving wave generating equipment (pinch points, objects striking).	a) No personnel permitted in wave generating room during operations. PPE must be utilized when adjusting mechanics of wave generation equipment. Use correct tools for the job and use them safely.
8) Removal of oil from test tank	a) Oil exposure (skin/eye contact) b) Falls, slips c) Drum skimmer power pack operations.	a) Wear protective clothing, goggles/face shields and nitrile gloves. b) When moving oil from the water with high pressure hose streams, avoid direct contact of oil with water stream. Clean any splashed oil from the deck with absorbent pads. c) Hearing protection is mandatory, (muffs or plugs)
9) Cleanup of equipment	a) Disassembly of rigging from work boat (falls). b) Pressurized water/water lines (objects striking) c) Hot water/steam wash (burns) d) Oil/cleaning agent exposure (skin, eye contact) e) Slippery surfaces from excess oil/cleaning agents (falls/slips)	a) Personnel on work boat must wear PFD's. Evenly distribute weight and do not overload. Life preservers are in place as needed. b) Inspect all equipment prior to use. Ensure hoses/fittings, etc. Are in good condition with no signs of deterioration/cracks damage. c) Wear appropriate PPE (face shield, goggles, gloves, protective clothes). d) Wear appropriate PPE (face shield, goggles, protective clothes, Sarnac or Tyvek suits, gloves). e) Keep deck as oil and soap free as possible, watch footing and remove obstacles. Creation of a decontamination zone will be mandatory.
10) Pack up	a) Fork lift operations (objects striking) b) Material handling (muscle strains, back injuries)	a) Follow acceptable safe practices for fork lift operations. b) Use proper lifting techniques, lift with your legs and not with your back, get help for heavy loads (i.e. fork truck, jib crane, etc.).

Finally, personal protective equipment guidelines (for items such as hard hats, steel toed boots, and the like) will be followed based on our Health & Safety Site Plan. The assessment is based only on generic or basic steps. Chemical Hazards will be discussed based on hazard communication standards with MSDS's reviewed.

Material Safety Data Sheets are available to participants at the Ohmsett Facility Office, Building R-26.

4.4 Personal Protective Equipment

The following personal protective equipment shall be available at all times. Specific use requirements may be found in Section 4.2.

- Work gloves
- Oil resistant gloves (neoprene, nitrile)
- Eye protection (safety glasses, goggles)
- Face protection
- Hardhats
- Safety shoes
- Personal flotation devices (for workboat operations) mandatory
- Respiratory protection (suitable for dusts, mists, vapors and fumes) if applicable
- Hearing protection, for power pack operation
- Life rings
- Splash suits, for boom clean up
- Fall-arrest system (life line, safety belt, tie-off point)

4.5 Communication Plan

Good communication is essential to the safe execution of the test. The following types of communication tools and skills will be available for use:

- Two-way radios
- Intercom system
- PA system
- Hand signals

4.6 Contingency Plan

In case of medical emergency, fire, major oil spill, or other emergency, it is necessary to notify Naval Weapons Station Earle. The OHMSETT Spill Response Plan shall be followed in the event of any oil spill.

A) Emergency Telephone Numbers:

• Naval Weapons Station Earle	X 2911
• Leonardo First-Aid	9 - 615 - 2100
• Riverview Medical Center	9 - 741 - 2700
• Bayshore Hospital	9 - 739 - 5900
• Poison Control Center	9 - 1 - (800) 962-1253

5. EXAMINING THE FATE OF EMULSION BREAKERS TEST QUALITY

5.1 Introduction

Examining the Fate of Emulsion Breakers Test Quality is the active application of The Ohmsett "General Quality Procedures and Documentation Plan Manual" and the "Examining the Fate of Emulsion Breakers Test Quality Checklist."

The Quality Checklist has a list of those items in the Examining the Fate of Emulsion Breakers Test Plan (see Section 5.2) that are deemed important elements in creating a quality test. This list will be used by the QA Engineer to record spot checks of key quality elements, along with appropriate comments, where necessary. A description of these key quality elements follows. The QA Checklist will be provided in the Final Test Plan.

5.2 Procedures

The Examining the Fate of Emulsion Breakers Test Quality Checklist is implemented as follows:

The Examining the Fate of Emulsion Breakers Test Quality Checklist consists of a complete list of Quality concern items that the QA Engineer uses to spot check items, and confirm adherence to the Test Plan. This checklist is used both before, during and after the test to make sure all areas of the test plan receive the same thorough Quality attention. These areas include:

A. Initial calibration data
B. Pre- and post-test checks and conditions
C. Test checks and conditions
D. Sampling
E. Significant occurrences/variations
F. Data reduction and validation
G. Data accuracy and precision
H. Documentation of the tests
I. Technical project report

5.3 Initial Calibration Data

A check is made to insure that data is available to show the initial source of calibration data for each piece of instrumentation used in the test. This includes any calibration information necessary to assure that the calibration data is current for this test.

5.4 Pre- and Post-Test Checks and Conditions

These are checks that are performed on the instrumentation and weather conditions each morning before testing starts and at the end of the day when testing stops. This is done on all days that testing occurs. Note is made of any unusual conditions that occur. These conditions must be evaluated before testing is started or if noted at the end of the day, the day's data is examined to determine its validity and whether the affected tests need to be repeated.

5.5 Test Checks and Conditions

These checks insure that the test plan's instructions on how the test is to be done are followed and that the records that are to be made during the test are completed accurately.

5.6 Sampling

Sampling will be checked for compliance with the instructions in this plan and the "Operating Manual for Ohmsett Laboratory Including Laboratory Procedures."

5.7 Significant Occurrences/Variations

This part of the Examining the Fate of Emulsion Breakers Test Quality checks will be concerned with recording any significant occurrences/variations that might occur during the tests. These will be immediately reported to the Project Officer.

5.8 Data Reduction and Validation

All data reduction and validation will be performed in accordance with approved and accepted methods. When non-standard methods are utilized, they shall be included in the Technical Project Report and sufficiently described so that they can be used by independent sources to duplicate the results. The treatment of data is described in Sections 3.

6. SCHEDULE

The following schedule is planned for conduct of the tests.

DATE	**EVENT**
September 30, 2003	Submit Draft Test Plan
November 10 to 14, 2003	Examining the Fate of Emulsion Breakers Tests
December, 2003	Deliver Raw and Processed Data, Observations and Photo Video Documentation
December 31, 2003	Submission of Final Report

7. REFERENCES

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Schulze, R. V. Keith and C. Purcell. 1995. World catalog of oil spill response products. Port City Press. Baltimore, MD

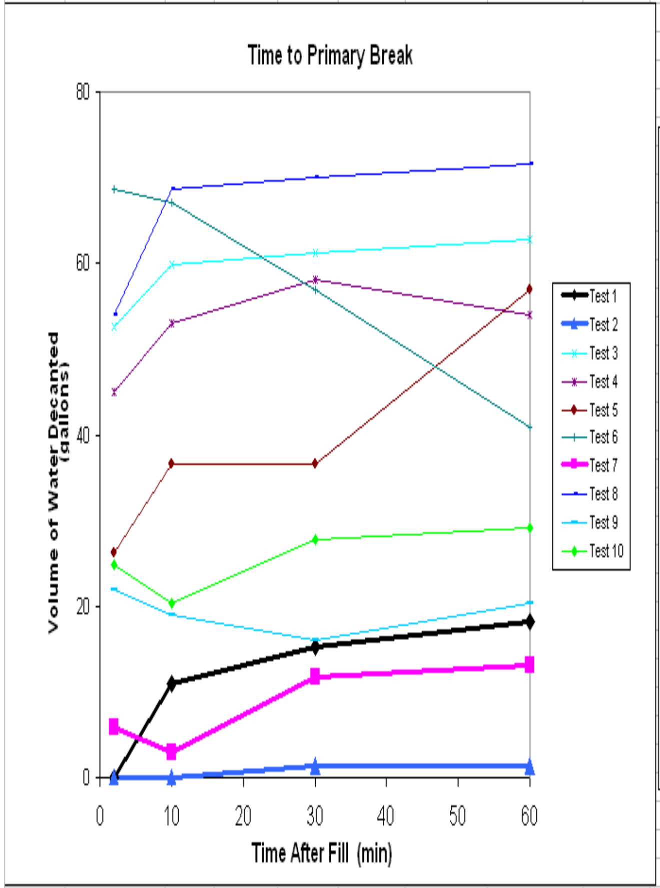
SL Ross Environmental Research Ltd. 1998. Modeling and lab-scale testing of water separation from fluids recovered by weir-type skimmers. Report to Alaska Clean Seas. Deadhorse, AK

SL Ross Environmental Research Ltd. 1999. Testing at Ohmsett to determine optimum times to decant simple temporary storage devices. Report to MMS and CCG. Herndon, VA

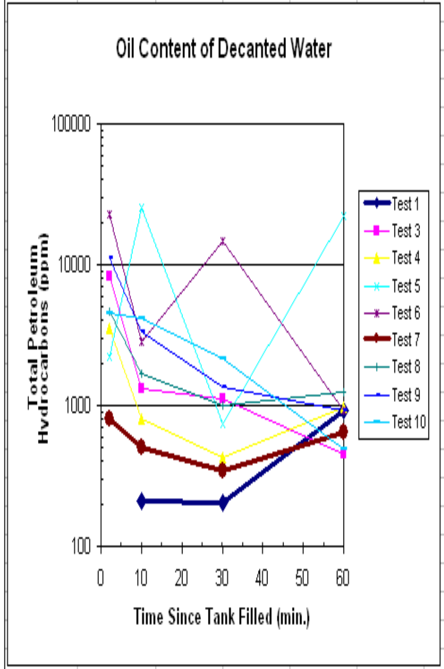
SL Ross Environmental Research Ltd. 2002. Extending temporary storage capacity offshore with emulsion breakers. Report to MMS and CCG. Herndon, VA.

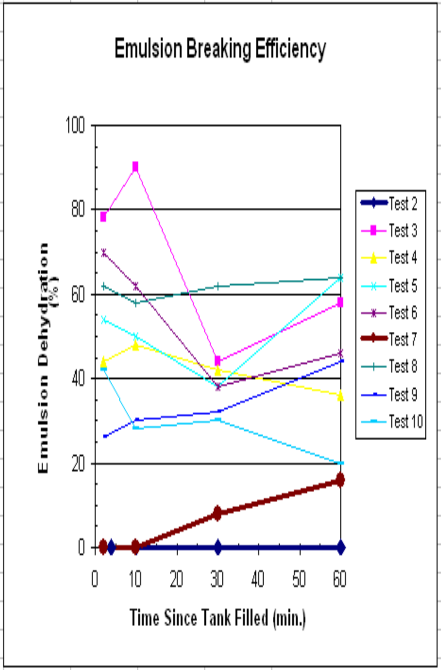
APPENDIX D - OHMSETT TEST RESULTS

Test Number	Oil	Wave Number	Total Fluid Flow Rate (gpm)	Free Water (Avg %)	Demulsifier	Demulsifier Concentration (ppm)	Recovered Emulsion Water Content (Avg %)	Emulsion Dehydration (%) 2 min.	10 min.	30 min.	60 min.	Concentration of Oil In Decanted Water (ppm) 2 min.	10 min.	30 min.	60 min.	Concentration of Demulsifier in Decanted Water (ppm) 2 min.	10 min.	30 min.	60 min.	Comments
1	Hydrocal 80/5/15	1	67	12	None	0	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	212	206	918	No Sample	No Sample	2	2	Neglected to take recovery tank samples, No water samples at 2 minutes, Forgot IFT sample at 10 min.
2	Hydrocal 80/5/15	2	104	19	None	0	60	0	0	0	0	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	No Sample	Not enough water for samples
3	Hydrocal 80/5/15	1	97	44	Alcopol	1413	16	78	90	44	58	8252	1325	1102	452	257	291	401	257	Creamed oil on surface of emulsion in boom, 10-min tank decanted at 12 min. 26 s
4	Hydrocal 80/5/15	2	80	45	Alcopol	1194	29	44	48	42	36	3491	799	430	979	445	267	267	257	10-min. tank decanted at 12 min. 2 s., Note tanks 3 and 4 times switched
5	Hydrocal 80/5/15	1	117	16	Alcopol	888	24	54	50	38	64	2189	25330	740	22209	140	592	229	401	Demulsifier injection pump had problems early in test.
6	Hydrocal 80/5/15	2	80	49	Alcopol	1354	23	70	62	38	46	22547	2816	14572	923	630	470	401	445	Skimmer paused during filling of recovery tank #3, 10-min tank decanted at 12 min. 51 s
7	Hydrocal 80/5/15	1	128	12	Alcopol	0	47	0	0	8	16	803	506	344	647	33	21	22	15	
8	Hydrocal 80/5/15	2	224	58	Alcopol	425	19	62	58	62	64	4649	1680	990	1249	279	317	279	364	Demulsifier injection pump turning slowly, 10-min tank decanted at 12 min. 14 s
9	Endicott	1	135	18	Unichem	1380	34	26	30	32	44	11239	3338	1347	934	241	223	175	171	Skimmer slow to start, 10-min tank decanted at 10 min. 48 s
10	Endicott	2	95	34	Unichem	1647	35	42	28	30	20	4515	4184	2143	491	301	149	175	156	Problems with skimmer HPU for first 5 minutes, then better,10-min tank decanted at 10 min. 40 s



2
10
30
60





Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
1	N	Y	193	117	151	1	81/14/5	0
	#DIV/0!							

Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	66	14.8	14.8	86	0	nm	#VALUE!
5	10	76	14.8	12.9	75	11	nm	#VALUE!
4	30	85	15.0	12.4	72	15	nm	#VALUE!
3	60	82	15.1	12.0	70	18	nm	#VALUE!
2								
1								

Fill Time (s)	Average Skimming Rate Total (gpm)	Emulsion (gpm)	Water (gpm)	Free Water (%)
309	67	59	8	11.9

Emulsion Removed (gal)	304
Preload (gal)	193
Emulsion Added (gal)	370
Emulsion Remaining (gal)	259

Test Number
1

Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)
8					
7					
6	#DIV/0!	#DIV/0!	nm	nm	
5	#DIV/0!	#DIV/0!	212.4	nm	
4	#DIV/0!	#DIV/0!	205.8	30.96667	2
3	#DIV/0!	#DIV/0!	917.9	30.75	2
2					
1					

Average concentration of demulsifier in recovered fluids = #DIV/0! ppm

Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
2	N	Y	259	157	101	2	81/14/5	0
	#DIV/0!							

Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	53	15.5	15.5	91	0	50	45
5	10	51	16.5	16.5	96	0	55	43
4	30	50	15.3	15.0	88	1	68	28
3	60	57	15.5	15.3	89	1	65	31
2								
1								

Fill Time (s)	Average Skimming Rate Total (gpm)	Emulsion (gpm)	Water (gpm)	Free Water (%)
211	104	84	20	19.3

Emulsion Removed (gal)	296
Preload (gal)	259
Emulsion Added (gal)	315
Emulsion Remaining (gal)	278

Test Number
2

Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)
8					
7					
6	0	0	nm	nm	nm
5	0	0	nm	nm	nm
4	8	0	nm	nm	nm
3	8	0	nm	nm	nm
2					
1					

Average concentration of demulsifier in recovered fluids = #DIV/0! ppm

Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
5	Y	Y	278	169	50	1	81/14/5	1.6
	1127							Drimax

Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	52	15.3	10.8	63	26	23	48
5	10	40	16.0	9.8	57	37	25	43
4	30	42	14.8	8.5	50	37	31	34
3	60	48	15.0	5.3	31	57	18	25
2								
1								

Fill Time (s)	Average Skimming Rate Total (gpm)	Emulsion (gpm)	Water (gpm)	Free Water (%)
182	117	99	18	15.5

Emulsion Removed (gal)	301
Preload (gal)	278
Emulsion Added (gal)	120
Emulsion Remaining (gal)	97

Test Number
5

Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)
8					
7					
6	190	54	2189	3.3	140
5	251	50	25330.2	1.55	592
4	273	38	739.7	2.55	229
3	418	64	22209.4	1.9	401
2					
1					

Average concentration of demulsifier in recovered fluids = 888 ppm

Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
6	Y	Y	97	59	100	2	81/14/5	3.1
	738							Drimax

Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	62	15.0	3.3	19	69	15	16
5	10	61	15.5	4.0	23	67	19	19
4	30	62	15.3	5.5	32	57	31	22
3	60	80	14.8	7.8	45	41	27	33
2								
1								

Fill Time (s)	Average Skimming Rate Total (gpm)	Emulsion (gpm)	Water (gpm)	Free Water (%)
265	80	41	39	48.9

Emulsion Removed (gal)	181
Preload (gal)	97
Emulsion Added (gal)	287
Emulsion Remaining (gal)	203

Test Number
6

Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)
8					
7					
6	160	70	22546.7	1.5	630
5	152	62	2816.4	1.75	470
4	131	38	14571.7	1.9	401
3	101	46	923	1.8	445
2					
1					

Average concentration of demulsifier in recovered fluids = 1354 ppm

Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
3	Y	Y	203	123	75	1	81/15/4	3.3
	708							Drimax

Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	51	15.0	6.0	35	53	11	31
5	10	55	15.5	5.3	31	60	5	29
4	30	55	15.0	4.5	26	61	28	19
3	60	57	14.8	4.0	23	63	21	18
2								
1								

Fill Time (s)	Average Skimming Rate Total (gpm)	Emulsion (gpm)	Water (gpm)	Free Water (%)
218	97	54	43	44.5

Emulsion Removed (gal)	195
Preload (gal)	203
Emulsion Added (gal)	132
Emulsion Remaining (gal)	140

Test Number
3

Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)
8					
7					
6	135	78	8252	2.4	257
5	149	90	1325	2.25	291
4	157	44	1102	1.9	401
3	164	58	452	2.4	257
2					
1					

Average concentration of demulsifier in recovered fluids = 1413 ppm

Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
4	Y	Y	238	145	100	2	81/15/4	2.3
	838							Drimax

Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	37	15.0	7.3	42	45	28	30
5	10	34	15.3	6.3	37	53	26	27
4	60	31	14.0	4.8	28	54	32	19
3	30	35	14.0	4.0	23	58	29	17
2	NB: Order of decanting switched for this test only							
1								

Fill Time (s)	Average Skimming Rate Total (gpm)	Emulsion (gpm)	Water (gpm)	Free Water (%)
137	149	81	68	45.4

Emulsion Removed (gal)	186
Preload (gal)	238
Emulsion Added (gal)	113
Emulsion Remaining (gal)	165

Test Number
4

Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)	
8						
7						
6	114	44	3491	1.8	445	
5	130	48	799	2.35	267	
4	146	36	979	2.4	257	60 min
3	157	42	430	2.35	267	30 min
2	NB: Order of decanting switched for this test only					
1						

Average concentration of demulsifier in recovered fluids = 1194 ppm

Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
8	Y	Y	262	159	75	2	81/15/4	0.5
	2354							Drimax

Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	25	15.3	6.0	35	54	19	28
5	10	23	16.3	4.5	26	69	21	21
4	30	24	15.0	3.0	18	70	19	14
3	60	24	14.8	2.5	15	72	18	12
2								
1								

Fill Time (s)	Average Skimming Rate Total (gpm)	Emulsion (gpm)	Water (gpm)	Free Water (%)
96	224	94	129	57.9

Emulsion Removed (gal)	151
Preload (gal)	262
Emulsion Added (gal)	51
Emulsion Remaining (gal)	162

Test Number
8

Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)
8					
7					
6	105	62	4649	2.3	279
5	125	58	1680	2.15	317
4	138	62	990	2.3	279
3	143	64	1249	2	364
2					
1					

Average concentration of demulsifier in recovered fluids = 425 ppm

Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
7	N	Y	354	215	50	1	81/15/4	0
Redo 1								

Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	25	8.5	7.5	44	6	46	24
5	10	23	9.3	8.8	51	3	53	24
4	30	24	8.8	6.8	39	12	46	21
3	60	24	8.5	6.3	37	13	42	21
2								
1								

Fill Time (s)	Average Skimming Rate Total (gpm)	Emulsion (gpm)	Water (gpm)	Free Water (%)
96	128	113	15	11.8

Emulsion Removed (gal)	180
Preload (gal)	354
Emulsion Added (gal)	102
Emulsion Remaining (gal)	276

Test Number
7

Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)
8					
7					
6	100	0	803	7.05	33
5	46	0	506	8.95	21
4	193	8	344	8.7	22
3	224	16	647	10.8	15
2					
1					

Average concentration of demulsifier in recovered fluids = 0 ppm

Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
9	Y	Y	100	61	75	1	Endicott	1.2
.	725							UNICHEM

Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	28	10.5	6.8	39	22	37	25
5	10	21	9.0	5.8	34	19	35	22
4	30	19	7.0	4.3	25	16	34	16
3	60	23	8.5	5.0	29	20	28	21
2								
1								

Fill Time (s)	Average Skimming Rate Total (gpm)	Emulsion (gpm)	Water (gpm)	Free Water (%)
91	135	111	24	17.7

Emulsion Removed (gal)	168
Preload (gal)	100
Emulsion Added (gal)	150
Emulsion Remaining (gal)	82

Test Number
9

Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)		
8							
7							
6	201	26	11239	5	241		26
5	204	30	3338	5.15	223		30
4	221	32	1347	5.65	175		32
3	232	44	934	5.7	171		44
2							
1							

Average concentration of demulsifier in recovered fluids = 1380 ppm

Test Number	Demulsifier Y/N Fluid:Demulsifier	Inline Mixer	Preload Volume (gal)	Preload Thickness (mm)	Distribution Rate (gpm)	Wave Number	Parent Oil Blend	Demulsifier Vol (L)
10	Y	Y	82	50	75	2	Endicott	1.8
.	607							UNICHEM

Tank Number	Settling Time	Time to Fill (s)	Filled Depth (in)	Decanted Depth (in)	Emulsion Remaining (gal)	Water Decanted (gal)	Rec. Emulsion Water Content (%)	Oil Recovered (gal)
8								
7								
6	2	44	8.5	4.3	25	25	29	18
5	10	40	9.25	5.8	34	20	36	21
4	30	22	9	4.3	25	28	35	16
3	60	24	8.5	3.5	20	29	40	12
2								
1								

Fill Time (s)	Average Skimming Rate Total (gpm)	Emulsion (gpm)	Water (gpm)	Free Water (%)
130	95	62	33	34.4

Emulsion Removed (gal)	135
Preload (gal)	82
Emulsion Added (gal)	144
Emulsion Remaining (gal)	91

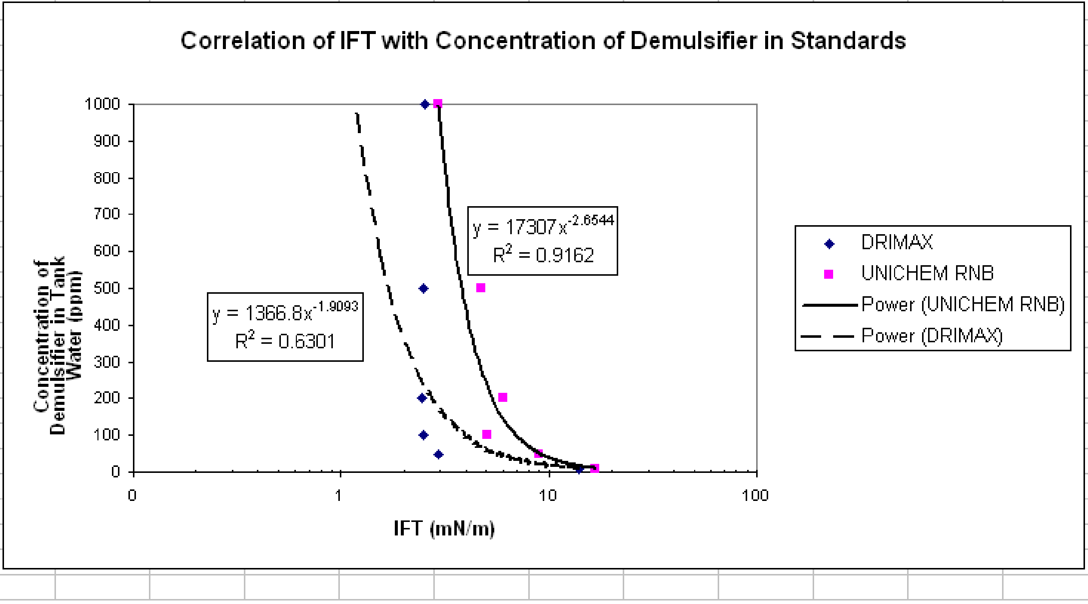
Test Number
10

Tank Number	Free Water Removed (%)	Emulsion Dehydration (%)	TPH Water (mg/L)	IFT Water (mN/m)	Demulsifier in Water (ppm)
8					
7					
6	145	42	4515	4.6	301
5	110	28	4184	6	149
4	153	30	2143	5.65	175
3	171	20	491	5.9	156
2					
1					

Average concentration of demulsifier in recovered fluids = 1647 ppm

DECANT 3 (TO 422)
DEMULSIFIER STANDARDS
W/ MINERAL OIL (USP)
FISHER TENSIOMAT RING 0043

STD NAME	CONC. (PPM)	IFT1	IFT2	IFT(AVG)
UNICHEM RNB	1000	2.92	2.94	2.93
	500	4.80	4.60	4.70
	200	6.10	6.00	6.05
	100	5.10	5.00	5.05
	50	8.90	9.00	8.95
	10	16.70	16.50	16.60
DRIMAX	1000	2.60	2.50	2.55
	500	2.50	2.50	2.50
	200	2.40	2.50	2.45
	100	2.50	2.50	2.50
	50	3.00	2.90	2.95
	10	13.90	13.80	13.85



TO-422 Examining the Fate of Emulsion Breakers Used for Decanting

Interfacial Tensions of Recovered Water vs Mineral Oil (Eckerd USP)

Feb. 6, 2004		**dynes/cm**	**dynes/cm**	**dynes/cm**	**Notes**
Test #1					
	2 min.	N/A			
	10 min	N/A			
	30 min	32.3	30.7	29.9	
	60 min	31.9	29.6		
Test #2	No samples collected				
Test #3					Straightened ring for rest of tests
	2 min.	2.5	2.3		
	10 min	2.3	2.2		
	30 min	1.9	1.9		Very cloudy soln.
	60 min	2.5	2.3		Very cloudy soln.
Test #4					
	2 min.	1.8	1.8		slightly cloudy soln
	10 min	2.4	2.3		
	30 min	2.5	2.2		
	60 min	2.4	2.4		
Feb. 7, 2004					
Test #5					
	2 min.	3.4	3.2		
	10 min	1.8	1.3		a lot of surf oil on sample
	30 min	2.8	2.3		
	60 min	1.9	1.9		
Test #6					
	2 min.	1.5	1.5		a lot of surf oil on sample
	10 min	1.9	1.6		
	30 min	1.9	1.9		solid layer of surface oil
	60 min	1.9	1.7		
Test #7					
	2 min.	7.2	6.9		slightly cloudy soln
	10 min	9.2	8.7		slightly cloudy soln
	30 min	11.1	6.2	4.2	slightly cloudy soln-unstable results
		6.1	3.9		slightly cloudy soln-unstable results
	60 min	10.9	10.7		slightly cloudy soln

TO-422 Examining the Fate of Emulsion Breakers Used for Decanting

Interfacial Tensions of Recovered Water vs Mineral Oil (Eckerd USP)

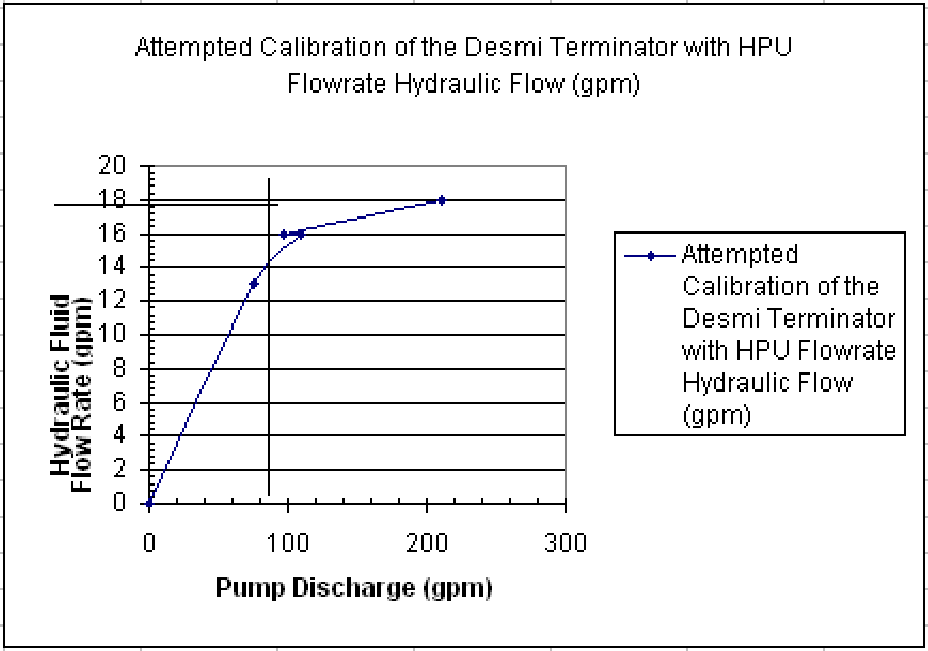
Feb. 8, 2004		**dynes/cm**	**dynes/cm**	**dynes/cm**	**Notes**
Test #8					
	2 min.	2.5	2.1		slightly cloudy soln
	10 min	2.2	2.1		slightly cloudy soln
	30 min	2.4	2.2		slightly cloudy soln
	60 min	2.0	2.0		slightly cloudy soln
Test #9					used Roberts USP for rest of tests*
	2 min.	5.1	4.9		oil covered-very cloudy-new duNuoy ring
	10 min	5.2	5.1		oil covered-very cloudy-new duNuoy ring
	30 min	5.8	5.5		oil covered-very cloudy-new duNuoy ring
	60 min	5.7			oil covered-very cloudy-new duNuoy ring
Test #10					
	2 min.	5.5	3.7	3.8	oil covered-very cloudy-new duNuoy ring
		3.6			oil covered-very cloudy-new duNuoy ring
	10 min	6.3	5.7	5.4	oil covered-very cloudy-new duNuoy ring
	30 min	5.8	5.5		oil covered-very cloudy-new duNuoy ring
	60 min	5.9	5.9		oil covered-very cloudy-new duNuoy ring

* Roberts USP Grade Mineral Oil is a Squibb Product. Tested this oil against the results of Test #7, 2 minutes. Results of 7.0 and 6.9 dynes/cm compared favorably.

Test #	Recovery Tank #	TPHC (mg/L)	MDL (mg/l)
1	5	212.4	1
	4	205.8	0.9
	3	917.9	1.4
3	6	8251.9	1.1
	5	1325.1	1.1
	4	1102.2	1.1
	3	451.5	1
4	6	3491.4	1.6
	5	798.7	1.1
	4	979.3	1
	3	429.6	1
5	6	2189	1.2
	5	25330.2	5
	4	739.7	1
	3	22209.4	3.1
6	6	22546.7	3
	5	2816.4	1
	4	14571.7	2.6
	3	923	1
7	6	802.5	0.9
	5	506	2
	4	344.4	2
	3	646.6	1.7
8	6	4649.4	2.1
	5	1680.3	2.1
	4	990.2	2.1
	3	1249.2	1.9
9	6	11238.6	2.2
	5	3338	2.1
	4	1347.4	2.2
	3	933.5	2.1
10	6	4515.4	2.1
	5	4184.2	2.2
	4	2142.7	2.1
	3	490.8	2

Attempted Calibration of the Desmi Terminator with HPU Flowrate

Hydraulic F	Pump Discharge Rate (gpm)
18	211
16	97
16	109.5
13	75
0	0



	Sample ID			
Date in '03	*Test #*	*Tank #*	*%' H2O*	*% Oil*
12-Nov	T2	TK3	65.0	35.00
12-Nov	T2	TK4	68.0	32.00
12-Nov	T2	TK5	55.0	45.00
12-Nov	T2	TK6	50.0	50.00
12-Nov	T3	TK3	21.0	79.00
13-Nov	T3	TK4	28.0	72.00
13-Nov	T3	TK5	5.0	95.00
13-Nov	T3	TK6	11.0	89.00
13-Nov	T4	TK3	29.0	71.00
13-Nov	T4	TK4	32.0	68.00
13-Nov	T4	TK5	26.0	74.00
13-Nov	T4	TK6	28.0	72.00
13-Nov	T5	TK3	18.0	82.00
13-Nov	T5	TK4	31.0	69.00
13-Nov	T5	TK5	25.0	75.00
13-Nov	T5	TK6	23.0	77.00
12-Nov	T6	TK3	27.0	73.00
12-Nov	T6	TK4	31.0	69.00
12-Nov	T6	TK5	19.0	81.00
12-Nov	T6	TK6	15.0	85.00
12-Nov	T7	TK3	42.0	58.00
12-Nov	T7	TK4	46.0	54.00
12-Nov	T7	TK5	53.0	47.00
12-Nov	T7	TK6	46.0	54.00
13-Nov	T8	TK3	18.0	82.00
13-Nov	T8	TK4	19.0	81.00
13-Nov	T8	TK5	21.0	79.00
13-Nov	T8	TK6	19.0	81.00
13-Nov	T9	TK3	28.0	72.00
13-Nov	T9	TK4	34.0	66.00
13-Nov	T9	TK5	35.0	65.00
13-Nov	T9	TK6	37.0	63.00
13-Nov	T10	TK3	40.0	60.00
13-Nov	T10	TK4	35.0	65.00
13-Nov	T10	TK5	36.0	64.00
13-Nov	T10	TK6	29.0	71.00
13-Nov	Emulsion Batch 2 0730		53.0	47.00
14-Nov	Endicott Emulsion Batch 3		45.0	55.00