FINAL REPORT

RESEARCH ON USING OIL HERDING AGENTS FOR RAPID RESPONSE *IN SITU* BURNING OF OIL SLICKS ON OPEN WATER

for:

U.S. Department of the Interior

Bureau of Safety and Environmental Enforcement

Oil Spill Response Research (OSRR) Program

Herndon, VA

by:

S.L. Ross Environmental Research Ltd.

Ottawa, ON

February 28, 2012

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Executive Summary

As a result of the experimental success to date thickening slicks for *in situ* burning in drift ice a research program was carried out to explore the use of herding agents for *in situ* burning in open water conditions as a rapid-response technique for oil spills offshore. The research was conducted in two parts: the first involved laboratory testing to identify the best herding agent(s) for warmer water conditions; the second involved experiments at Ohmsett to quantify the persistence of the herder monolayer in waves.

The suitability of the best cold-water herders for warmer water conditions was investigated in the SL Ross laboratory. A series of comparative tests was conducted with hydrocarbon-based and silicone herders in 1-m^2 pans, a 10-m^2 pool, small pans mounted on a rocking shaker, and in the SL Ross wind/wave tank, including small *in situ* burn tests.

In the warm-water laboratory herding tests the Silsurf A108, Silsurf A004-D and non-proprietary hydrocarbon-based US Navy cold-water (USN) herders performed best. The two silicone-based herders retained a small burning crude oil slick and achieved burn efficiencies as good as or better than the USN herder.

Surfactant film persistence (i.e., how long the monolayer generated by a specific herding agent will last as a function of sea state) and to what degree periodically replenishing the film can counteract this was investigated in an 8-day test program at Ohmsett. The experiments took advantage of the facility's newly upgraded wave making capabilities.

A total of 11 experiments were completed with three herding agents (USN, Silsurf A108 and Silsurf A004-D) in three wave conditions: calm, swell, and breaking waves.

Based on visual observations of the Ohmsett tests the following conclusions were drawn:

- The monolayer of each of the two best herders will survive for 45 minutes or more in a calm sea (the Ohmsett tests were terminated when the slicks reached the end of the tank).
- The presence of breaking or cresting waves rapidly disrupts the herder monolayer and the oil slick resulting in many small slicklets.
- The monolayer survives for considerable periods of time in a swell condition, but the constant stretching and contracting of the herded slick results in elongating the oil slick and slowly breaking the slick into smaller segments.
- The Silsurf A108 herder performed noticeably better than the other two herders in all test conditions.

It was recommended that:

- Work should begin on commercializing the USN and silicone herders, including getting them listed on the EPA NCP Product Schedule and developing suitable application systems.
- The use of the USN and silicone herders to thicken slicks in open water conditions for *in situ* burning should be included in plans for future offshore oil spill response field trials.
- Consideration should be given to how herders could be employed to improve offshore skimming encounter rates in suitable open water conditions.

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1 Introduction

Since 2004, the main goal of the research on herders has been to determine their efficacy at enhancing *in situ* burning in ice concentrations too low for natural containment of the oil slick by the ice itself (ice concentrations between <10% and 60%). In the research on herders in drift ice a considerable number of the experiments were performed in open water or trace ice conditions as an integral part of each experiment matrix, certainly enough to know that they will work quite well to thicken slicks for efficient *in situ* burning on quiescent cold seawater.

Herders were studied in the 1970's as an open water oil spill response technique but the goal then was to provide containment for mechanical recovery. In this original application herders were limited to relatively calm conditions because the herder itself dissipated quickly in higher energy sea conditions allowing the slick to re-spread. This dissipation occurred over periods of tens of minutes, which wasn't enough time to allow skimming of the herded slick. *In situ* burning is a process that only requires minutes to initiate and complete. Once ignited, the air being drawn into a large *in situ* oil fire by the combustion process will also contain the burning slick and thicken it further. Thus, there is great potential that *in situ* burning enhanced by chemical herders can be a very effective rapid response technique in ice-free waters – both in the Arctic and in temperate regions. However, there remain two issues requiring further study:

1. Suitability of herders for warmer water conditions; and,
2. Surfactant film persistence in open water sea conditions.

2 Background

The use of specific chemical surface-active agents, sometimes called oil herders or oil collecting agents, to clear and contain oil slicks on an open water surface is well known (Garrett and Barger, 1972; Rijkwaterstaat, 1974; Pope *et al.,* 1985; MSRC, 1995). These agents have the ability to spread rapidly over a water surface into a monomolecular layer, as a result of their high spreading coefficients, or spreading pressures. The best herding agents have spreading pressures in the mid-40 mN/m range, whereas most crude oils have spreading pressures in the 10 to 20-mN/m range. Consequently, small quantities of these surfactants (about 5 L per kilometre or 50 g/m^2) will quickly clear thin films of oil from large areas of water surface, contracting the oil into thicker slicks.

Herders sprayed onto the water surrounding an oil slick result in formation of a monolayer of surfactants on the water surface. These surfactants reduce the surface tension of the surrounding water significantly (from 73 mN/m to 25-30 mN/m). When the surfactant monolayer reaches the edge of a thin oil slick it changes the balance of interfacial forces acting on the slick edge and causes the oil/water and oil/air interfacial tensions to contract the oil into thicker layers. Herders do not require a boundary to "push against" and work in open (boundary free) water. A conceptual drawing of the herding process in pack ice is shown in Figure 1. Although commercialized in the 1970s herders ultimately they were not used offshore because they only worked in very calm conditions: physical containment booms were still needed to hold or divert slicks in wind speeds above 2 m/s and breaking waves disrupted the herder layer.

2.1 Past Research on Herders to Thicken Oil for Burning

The key to effective *in situ* burning is thick oil slicks. Concentrated pack ice can enable *in situ* burning by keeping slicks thick. In loose pack ice conditions oil spills can rapidly spread to become too thin to ignite. Fire booms can collect and keep slicks thick in open water; however, field deployment tests of booms and skimmers in pack ice conditions in the Alaskan Beaufort

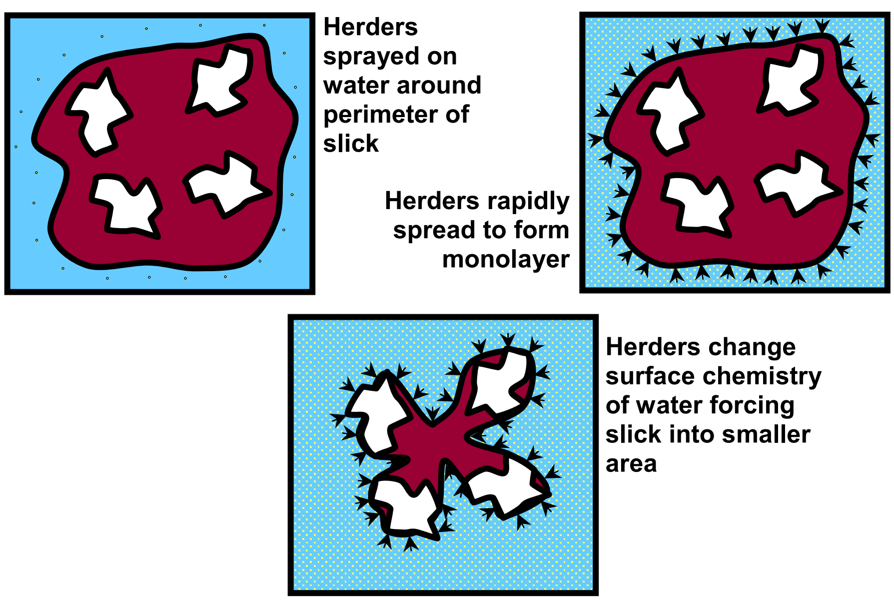

Figure 1. Conceptual drawing depicting the herding process in drift ice.

Sea highlighted the severe limitations of booms in even trace concentrations of drift ice (Bronson *et al.,* 2002). If slicks could be thickened to the 2- to 5-mm range in drift ice (less than 6 to 7 tenths coverage), even with no possibility of physical booming, effective burns could be carried out (SL Ross 2003). For application in loose pack ice, the intention is to herd freely-drifting oil slicks to a burnable thickness, then ignite them with a Helitorch. The herders will work in conjunction with the limited containment provided by the ice to allow a longer window of opportunity for burning.

A comprehensive, multi-year, multi-partner research program to study the use of chemical herding agents to thicken oil slicks in order to ignite and burn the oil *in situ* in loose pack ice has been underway since 2004. The program has included:

1. A very small scale (1 m^2) preliminary assessment of a shoreline-cleaning agent with oil herding properties was carried out to assess its ability to herd oil on cold water and among ice (SL Ross 2004).
2. Small-scale experiments to explore the relative effectiveness of three oil-herding agents in simulated ice conditions; larger scale (10 m^2) quiescent pan experiments to explore scaling effects; small-scale (2 to 6 m^2) wind/wave tank testing to investigate wind and wave effects on herding efficiency; and, small ignition and burn tests (SL Ross 2005).
3. Experiments at the scale of 100 m^2 in the indoor Ice Engineering Research Facility Test Basin at the US Army Cold Regions Research and Engineering Laboratory (CRREL) in November 2005 (SL Ross 2005).
4. Experiments at the scale of 1000 m^2 at Ohmsett in artificial pack ice in February 2006 (SL Ross 2005).
5. A series of 20 burn experiments at the scale of 30 m^2 with herders and crude oil in a specially prepared test basin containing broken sea ice in November 2006 at the Fire Training Grounds in Prudhoe Bay, AK (SL Ross 2005).
6. Field tests of a herder in pack ice in the Barents Sea in 2008 (Buist *et al*. 2010a)
7. Studies on better herding surfactants (Buist *et al*. 2010b).
8. Research on improving other marine spill countermeasures with herders (Buist *et al*. 2010c).

The U.S. Navy cold-water herder formulation (65% Span-20 and 35% 2-ethyl butanol) used in the earlier experiments (SL Ross 2004, 2005 and 2007) proved effective in significantly contracting fluid crude and refined oil slicks in brash and slush ice concentrations of up to 70% ice coverage. Slick thicknesses in excess of 3 mm, the minimum required for ignition of weathered oil *in situ*, were routinely achieved. The presence of frazil ice restricted the spreading of the oil and the effectiveness of the herder. Short, choppy waves in the test ice caused a herded slick to break up into small slicklets, although this may be an artifact of the relatively small volumes of oil used in the experiments. Longer, non-breaking waves, simulating a swell in pack ice, did not appear to cause a herded slick to break up, and in fact may have assisted the process by promoting spreading of the herder over water to the slick's edge.

Application of the herder to the water prior to the oil being spilled resulted in thicker slicks than post-spill application. This approach might be used in the event of a chronic spill event in pack ice conditions, such as a blowout or a pipeline leak.

Otherwise unignitable crude oil slicks that were contracted by the USN herder could be ignited and burned *in situ* in both brash and slush ice conditions at air temperatures as low as –17°C. Measured oil removal efficiencies for herded slicks averaged 50% for 7.5-L slicks and 70% for 15-L slicks. The efficiencies measured for the herded slicks were only slightly less than the theoretical maximums achievable for equivalent-sized, mechanically contained slicks on open water. The type of ice (brash or slush) did not significantly affect the burn efficiency.

When ignited, the herded slicks did spread slightly, but once the flames began to die down, the residue was re-herded by the agent remaining on the water surrounding the slick. Generally, it was not possible to reignite re-herded residue. Steeper, cresting waves detracted from the burn efficiency while longer, non-breaking waves did not. The oil removal rate for the slicks was in the range expected for equivalent-sized, mechanically contained slicks on open water.

In the spring of 2008 a field trial was carried out in pack ice east of Svalbard involving the release of 630 L of fresh crude onto water in a large lead. The free-drifting oil was allowed to spread for 15 minutes until it was far too thin to ignite, and then USN herder was applied around the slick periphery. The slick contracted and thickened for 20 minutes at which time the upwind end was ignited. A 9-minute burn ensued that consumed an estimated 90% of the oil (Sørstrøm *et al.* 2010).

In an attempt to further improve the herder technique, in 2008 and 2009, a series of laboratory tests and ice-basin tests were carried out to compare the efficacy of several silicone-based surfactants (superwetters) as potential oil slick herding agents to the results obtained with the USN (Buist *et al*. 2010b). Silicone surfactants are known to be more effective than hydrocarbon-based surfactants in other applications.

The best silicone-based herder of the three tested significantly outperformed the USN herder in most tests with similar conditions. These tests only evaluated the herding capability of the silicone herders – the slicks weren't ignited. Recent experiments in the lab confirmed the ability of the silicone herders to hold a burning slick as well as the USN herder does.

3 Objective

The objective of this research was to evaluate the feasibility of using herders to enable *in situ* burning as a rapid-response technique in open water. Specifically, the goals of the research were to:

1. Experiment in the laboratory with the USN herder formulation and the best silicone herder formulation to find the most effective product for warmer water temperatures.
2. Conduct experiments at Ohmsett to determine the persistence of the herder monolayer in realistic waves.

4 Study Approach

The research was conducted in two parts: the first involved laboratory testing to identify the best herding agent(s) for warmer water conditions; the second involved experiments at Ohmsett to quantify the persistence of the herder monolayer in waves.

4.1 Herding Agents for Warmer Open Water

The suitability of the best cold-water herders for warmer water conditions was investigated in the SL Ross laboratory. They were tested against reportedly superior surfactant/solvent combinations at room temperature identified in the original US Navy study (Garret and Barger 1972) and a previously available hydrocarbon-based herder recipe (Exxon's Oil Collector 5, or OC-5). Performance data on the silicone-based herders in warmer waters was also collected. A series of comparative tests was conducted with hydrocarbon-based and silicone herders in 1-m^2 pans (Figure 2); a 10-m^2 pool (Figure 3); Dynamic Film Performance (DFP) tests in small pans on a rocking shaker (Figure 4); and, small *in situ* burn tests in a wind/wave tank (Figure 5). Overhead digital photographs were taken and analyzed by computer to determine herder effectiveness. Small-scale (≈ 40 cm diameter) burn tests were also conducted with the best herders identified for warm water to confirm their suitability.

Figure 2. Lined 1-m^2 steel pans for experiments.

Figure 3. 10-m^2 pool with liner before filling.

Figure 4. Rocking shaker for DFP tests.

Figure 5. Herded slick burning in wind/wave tank

4.2 Ohmsett Testing of Herder Persistence in Waves

Surfactant film persistence (i.e., how long the monolayer will last as a function of sea state) and to what degree periodically replenishing the film can counteract this were investigated in an experimental program at Ohmsett, taking advantage of the facility's newly upgraded wave making capabilities. Although waves generated in the Ohmsett tank do not exactly mimic ocean waves, recent studies of Ohmsett waves show that they can be related to seas in the early stages of development (Veron *et al.* 2009). The results of experiments on herder monolayer persistence in different wave environments at Ohmsett, combined with historic offshore experiment data, will be useful in predicting persistence at sea.

5 Laboratory Experiments

The goal of the laboratory tests was to experiment in the laboratory with the USN herder formulations, OC-5 and the best silicone herder formulations to find the most effective product for warmer water temperatures.

5.1 Laboratory Experiment Methods

The laboratory test plan may be found in Appendix 1. The first series of experiments involved thirty 1-m^2 pan tests to evaluate the various herders' performance on warm water. Salinity was included as a variable because it can have an effect on herder effectiveness. The following parameters were varied in these experiments:

- Five candidate warm-water herders
    1. The original USN cold water blend (65% sorbitan monolaurate [aka Span-20] and 35% 2-ethyl butanol)
    2. A warm-water herder blend suggested in the original US Navy study, denoted here as USN_{smo} (75% sorbitan monooleate [aka Span-80] and 25% 2-ethyl butanol)
    3. Silsurf A108
    4. Silsurf A004D
    5. OC-5 [the old Exxon Oil Collector]
- One ambient temperature (≈ 20°C)
- Two water salinities (0 and 35‰)
- Three oil types: Kuparuk and ANS crudes and a No. 2 fuel oil (dyed with 2% Bunker C for better visibility).

The general procedure for a 1-m^2 pan experiment was:

1. Place 20 L (a depth of 2 cm) of room-temperature water in each 1-m^2 pan (Figure 2 above) lined with freshly rinsed (with tap water) new plastic film.
2. Take a sample of the water from the surface using a Petri dish and measure the water-air interfacial tension (IFT) using a DuNuoy Ring Tensiometer. If the IFT reading is less than 60, replace the water and film and retry.
3. Carefully pour 1 L of the crude on the water; making sure that it doesn't stick to the bottom of the tray while being poured.
4. Allow the oil to spread to equilibrium and take a digital photograph from overhead for subsequent oil area analysis.
5. Apply prescribed amount (150 µL) of herding agent to open water area with micropipette.
6. Allow the oil to contract and take another digital photograph after one minute, 10 minutes, 30 minutes and 1 hour.
7. Empty water from pan, remove plastic film, dry with paper towels.

The slicks (including any oil sheen) in the photographs were corrected for perspective, converted into black and white images using Paint Shop Pro (Figure 6) and then a computer program (Image J) was used to count the number of black pixels. The area of the slick in the photograph was then calculated by dividing the total number of black pixels by the number of pixels per square inch in the original image. Average slick thickness was estimated by dividing the volume of oil added originally by the calculated area.

The error in estimating area should be quite small, less than 5% taking into account parallax errors at the sides of the pans. Errors in average slick thickness would increase as time progressed, as evaporation losses were not taken into account, but in the quiescent lab environment over the period of an hour would not likely exceed 10% (SL Ross 2004 and 2005).

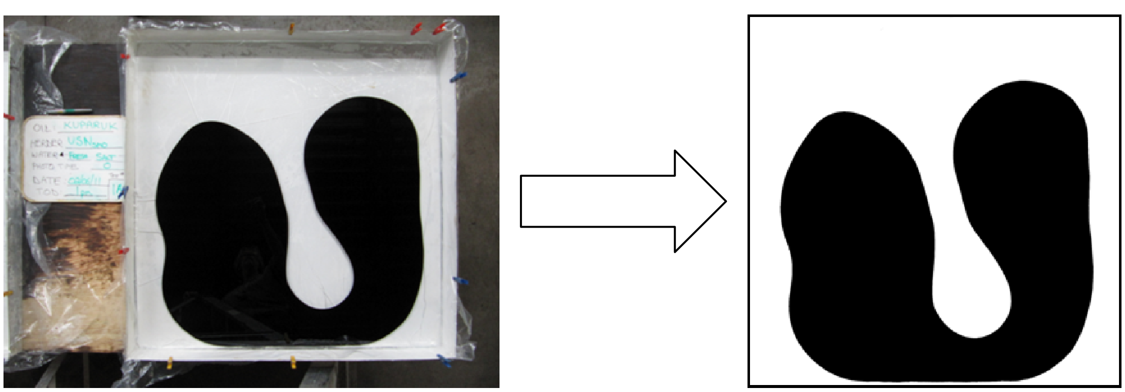

Figure 6. Determining slick area using overhead digital photos.

The next series of 8 tests are called Dynamic Film Performance (DFP) tests and were adapted from experiments of the same name conducted in the original US Navy study (Garret and Barger 1972). These tests were performed in a double Rocking Shaker (Figure 4 above) with a rocking angle of 6° and a frequency of 0.25 Hz. The test matrix involved four herders (the USN_{smo} herder was not tested due to its poorer performance in the 1-m^2 tests) with Kuparuk and Endicott crude on fresh water at room temperature. The procedure for a rocking shaker tray experiment was:

1. Place 1.115 L of room-temperature water (≈ 2 cm deep) in each of two trays (18 cm wide x 28 cm long) and allow them to equilibrate to the test temperature in the environmental chamber.
2. Take a sample of the water from the surface using a Petri dish and measure the water-air interfacial tension (IFT) using the DuNuoy Ring Tensiometer. If the IFT reading is less than 60, replace the water and retry.
3. Carefully place 50 mL of the test oil on the water, making sure that it doesn't stick to the bottom of the tray while being poured.
4. Allow the oil to spread to equilibrium and take a digital photograph from overhead for subsequent oil area analysis.
5. Apply 5 to 10 µL of herding agent to an open water area with a micropipette (the target dose for the herder is 7.5 µL based on a recommended treatment of 150 mg/m^2; however, it is impossible to deliver an accurate dose with the viscous herder which tends to form discrete droplets at the end of the micropipette).
6. Allow the oil to contract and take another digital photograph after one minute.
7. Carefully place the trays on the rocking shaker and start shaker and timer.
8. After 10 minutes and 30 minutes, stop the shaker, gently remove the trays re-photograph the slicks and replace the trays on the shaker.
9. Stop experiment after 1 hour, gently remove the trays re-photograph the slicks.
10. Remove and empty trays, clean with Alconox and hot water and rinse thoroughly with hot water.

The oil and herder combinations to be tested at a scale of 10 m^2 and in the SL Ross wind/wave tank were selected based on the results of the 1-m^2 and rocking shaker experiments.

Three 10-m^2 experiments were performed in a rectangular, plastic-lined wooden frame (2.98 m x 2.975 m x 9 cm high) lined with a new, rinsed sheet of 1-mil plastic film to ensure a clean, uncontaminated surface (Figure 3 above). White plastic signboard was attached to the bottom of the frame to increase the contrast in the photos between oil and water in the digital photos. Three experiments were conducted with the USN, A004-D and A108 herders on fresh water at room temperature using the Kuparuk crude.

The general procedure for a 10-m^2 pan experiment was:

1. Place 200 L (a depth of ≈ 2 cm) of room-temperature fresh water in the 10-m^2 pool lined with freshly rinsed (with tap water) new plastic film.
2. Take a sample of the water from the surface using a Petri dish and measure the water-air interfacial tension (IFT) using the DuNuoy Ring Tensiometer. If the IFT reading is less than 60, replace the water and film and retry.
3. Carefully pour 2 L of the oil on the water, making sure that it doesn't stick to the bottom of the pool while being poured.
4. Allow the oil to spread to equilibrium and take a digital photograph from the rafters overhead for subsequent oil area analysis.
5. Apply the prescribed amount (1.5 mL) of herding agent to open water area with micropipette.
6. Allow the oil to contract and take another digital photograph after one minute, 10 minutes, 30 minutes and 1 hour.
7. Pump water from pool, remove plastic film, and replace film for next experiment.

Following the 10-m^2 pool tests, the three candidate herders were tested in the wind/wave tank for monolayer persistence in waves. These were qualitative tests done in order to plan for the Ohmsett tests, involving visually observing the behaviour of a herder slick in increasing wave conditions. The general procedure for a wind/wave tank monolayer persistence experiment was:

1. Raise the floating barriers at either end of the test section and thoroughly clean the water surface with sorbent pads to remove any oil or herder traces.
2. Take a sample of the water from the surface using a Petri dish and measure the water-air interfacial tension (IFT) using the DuNuoy Ring Tensiometer. If the IFT reading is less than 60, reclean the test area.
3. Apply prescribed amount (400 µL) of herding agent to open water area with a micropipette (herder added first to minimize oil spreading differences between tests).
4. Turn on the video.
5. Carefully pour 400 mL of the crude on the water; making sure that it doesn't submerge while being poured.
6. Allow the oil to spread to its herded equilibrium area.
7. Turn on the wave generator to produce non-breaking waves and turn on the fan to counterbalance the wave drift.
8. After observing the slick behavior for several minutes, increase the wave generator to produce more energetic waves.
9. When the herded slick begins to re-spread and sheen, stop wave generator, recover oil and herder with sorbent pads.

The purpose of these tests was to compare a small-scale test burn using the silicone-based herders with one using the USN herder, which has been tested in the field (Sørstrøm *et al.* 2010). For the experiments, metal heat shields were installed along the inside walls of the wind/wave tank, and the metal fume hood was swung over the burn area (Figure 5 above). A test area was created by isolating an area of water surface with floating barriers stretched from one side of the tank to the other between the metal shields. The smoke from the burns was removed with a 200-m^3/min fan, through a 60-cm metal duct that is connected to the fume hood suspended 1 m above the water surface. Each test was videotaped to record burn times. One test burn was performed with each of three herders: USN, A004-D and A108. The general procedure for a test burn pan experiment was:

1. Place 400 mL of fresh Alaska North Slope (ANS) crude into a graduated cylinder and weigh it.
2. Apply 0.4 mL (150 mg/m^2) of herding agent to the contained water surface.
3. Carefully pour the oil from the graduated cylinder onto the treated water surface.
4. Reweigh the graduated cylinder to obtaining the mass of oil added.
5. Start the video and then ignite the herded oil gently with a propane soldering torch.
6. After the slick has extinguished, recover the residue with pre-weighed sorbent.
7. Allow the sorbent to dry overnight to evaporate water (herding agent reduces the hydrophobicity of oil sorbents) and weigh the sorbent to obtain the weight of residue recovered.
8. Estimate the burn efficiency using Equation 1.

$$\text{Burn Efficiency [mass \%]} = \frac{\text{(Initial Oil Mass - Residue Mass) x 100\%}}{\text{Initial Oil Mass}} \qquad \textbf{(1)}$$

5.2 Laboratory Experiment Results

Each series of laboratory tests is presented and discussed below. Raw data from the laboratory experiments may be found in Appendix 2.

5.2.1 1-m^2 Pan Tests

Figures 7, 8 and 9 present the 1-m^2 pan test results using Kuparuk crude, ANS crude and Fuel Oil No. 2 respectively. Note that on Figure 7 there is no initial average slick thickness for the A004-D herder in salt water due to camera failure.

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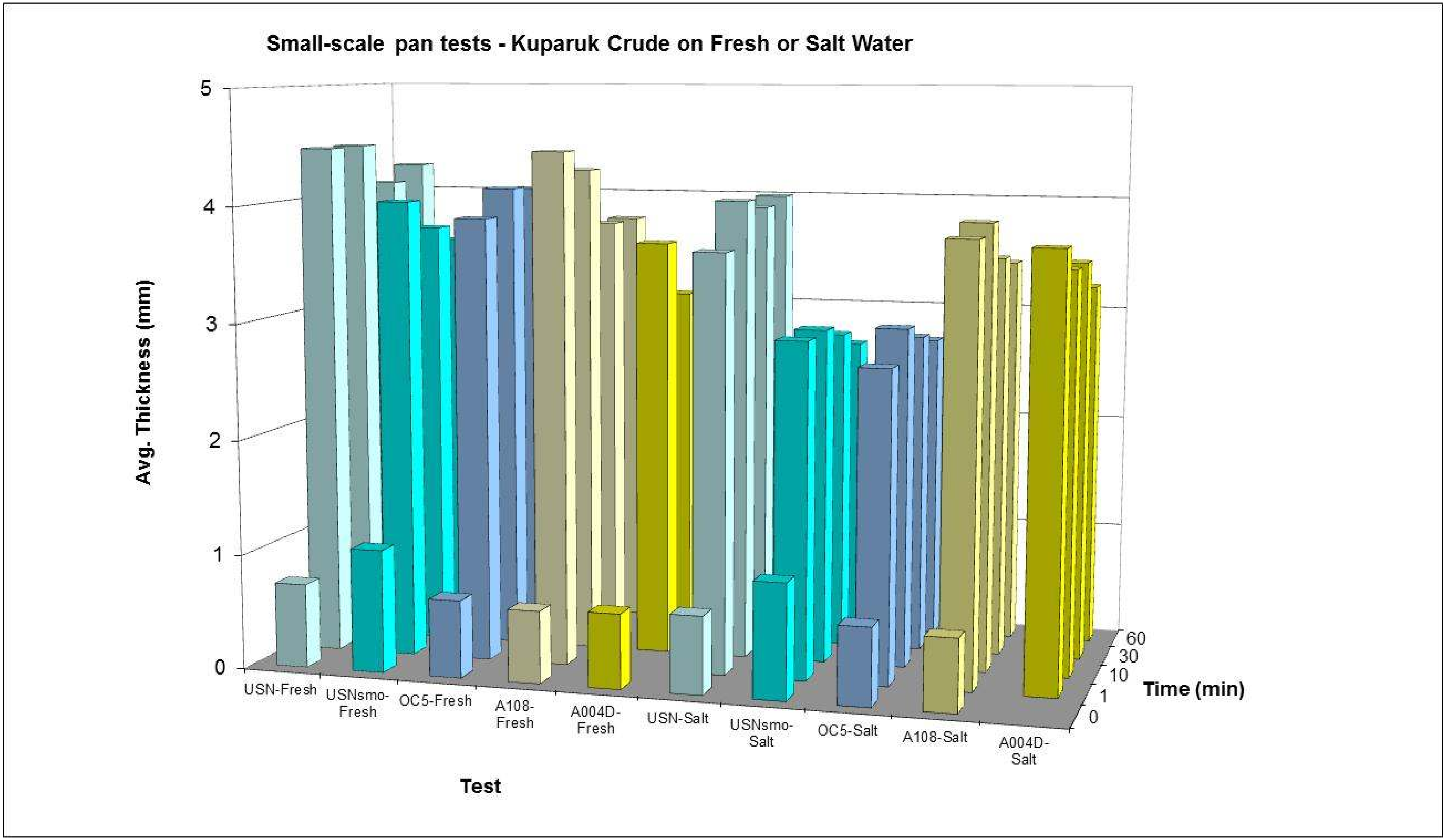

Figure 7. Results of 1-m^2 pan herding tests using Kuparuk crude.

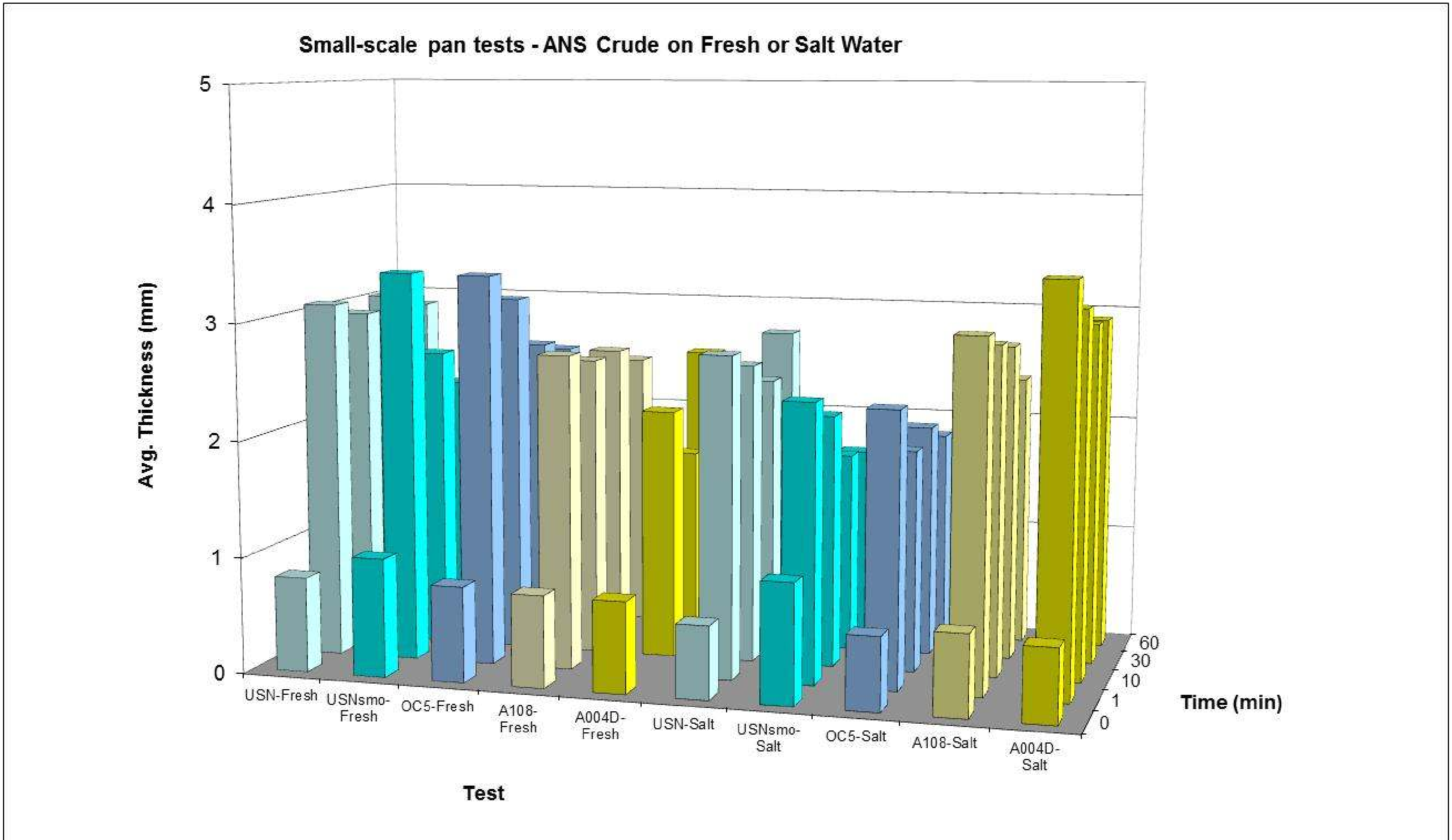

Figure 8. Results of 1-m² pan herding tests using ANS crude.

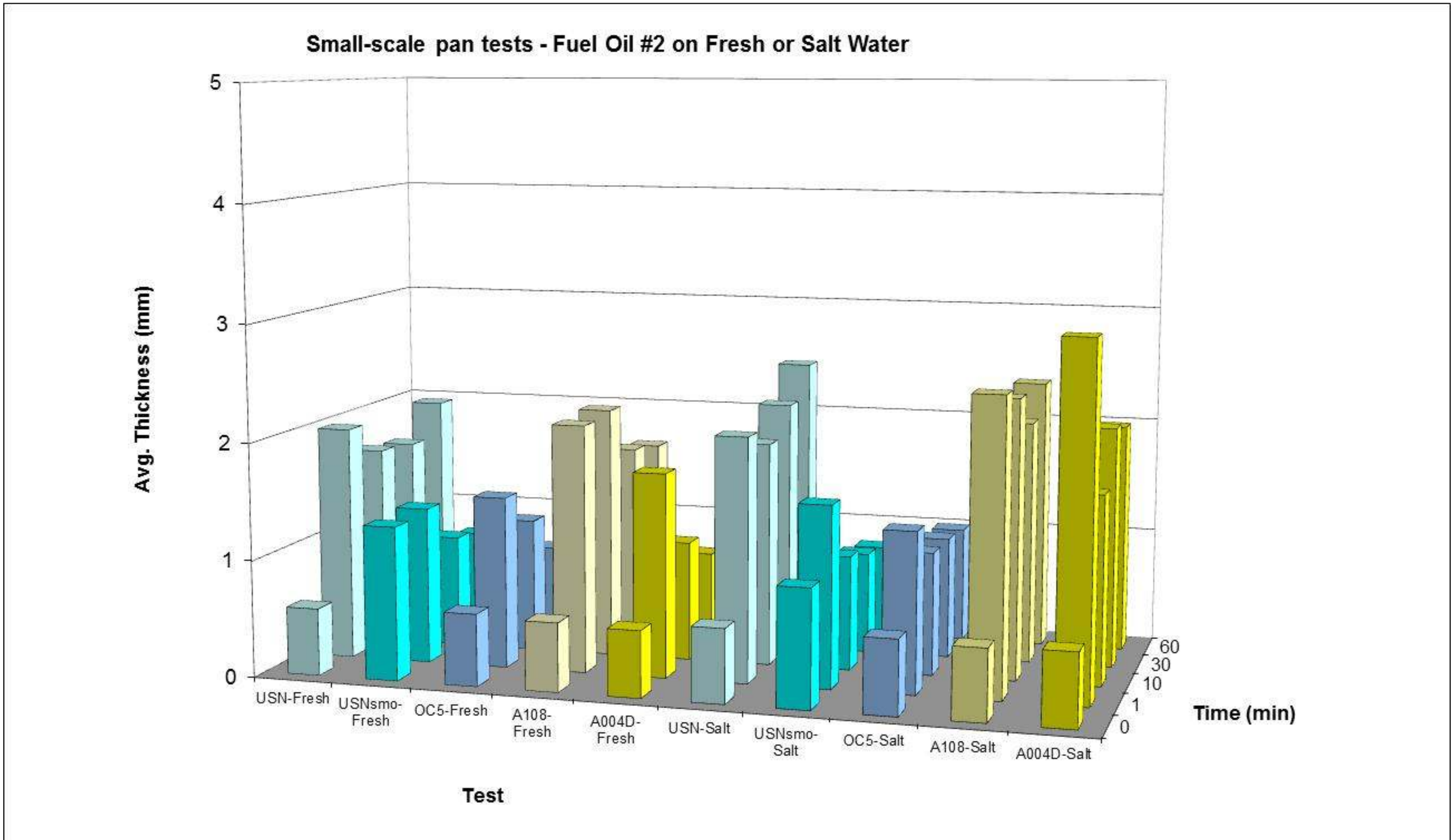

Figure 9. Results of 1-m^2 pan herding tests using Fuel Oil No. 2.

With the Kuparuk crude, the A108 and USN cold-water herder worked the best on fresh water with the OC5 coming third, the USN_{smo} coming fourth and the A004-D the least effective. On salt water, the A108 did a slightly better job initially than the USN cold-water herder but fell below the performance of the USN cold-water herder over the one-hour test. The A004-D came third and the USN_{smo} and OC5 tied for least effective on salt water. In the tests with ANS crude on fresh water the USN cold-water and OC-5 performed best, the A108 came third, the USN_{smo} herder came fourth and the A004-D finished last. On salt water the A004-D performed best, the A108 came second and the USN cold-water herder came third. The USN_{smo} and OC5 tied for least effective.

In the tests with Fuel Oil No. 2 on fresh water the contracted slicks produced by all the herders were much thinner than with the crude oils, except for the A004-D slick on salt water. On fresh water the A108 was best, with the USN cold-water herder a close second the A004-D came third and the OC-5 and USN_{smo} finished last. On salt water the A108 performed best, the A004-D was second-best. The USN cold-water herder was third and the OC-5 and USN_{smo} finished last.

Table 1 summarizes the best herder for each oil/salinity combination. The A108, USN cold-water, OC5 and A004-D herders were selected for additional testing.

Table 1: Summary of best herder at room temperature in 1-m^2 pan tests.

Kupark crude		ANS crude		Fuel Oil No. 2	
Salt	Fresh	Salt	Fresh	Salt	Fresh
A108 & USN	A108 & USN	A004-D & A108	USN & OC5	A108 & A004-D	A108 & USN

5.2.2 DFP Tests

Figure 10 presents the results of the DFP tests with Kuparuk crude for the four herders selected from the 1-m^2 pan tests. Although the USN herder gave the best initial performance, the herded slick thickness for both the USN and OC5 herders declined over the time span of the tests. The A004-D and A108 herders had the best performance in maintaining thick slicks over the rocking period.

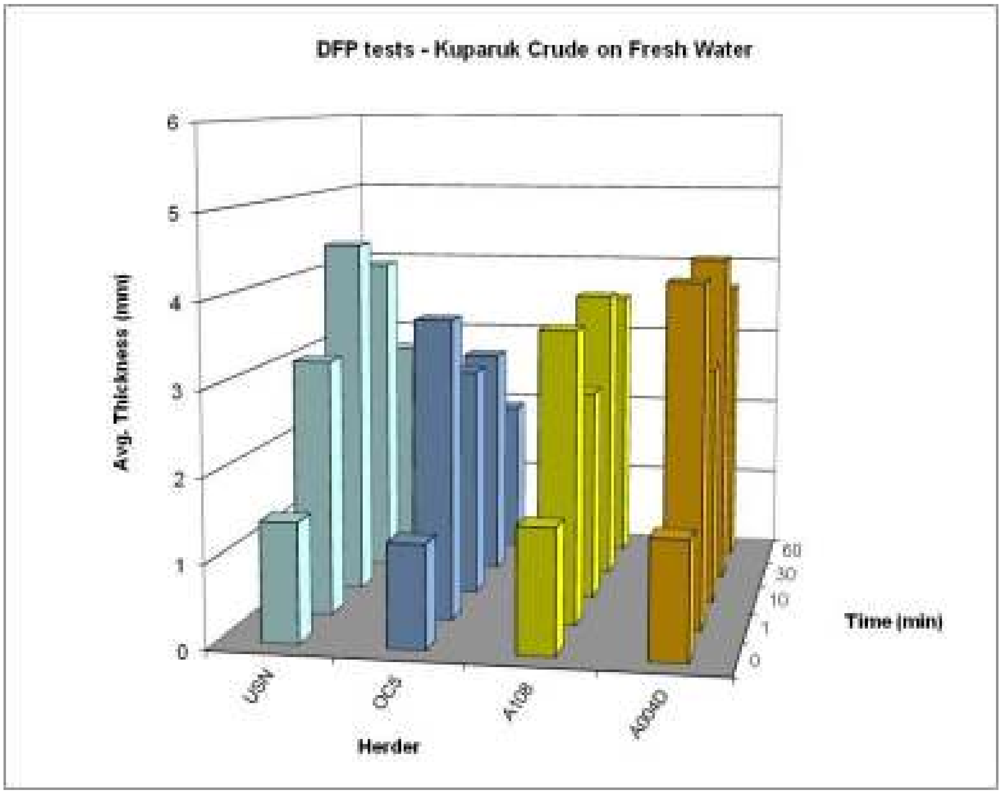

Figure 10. DFP test results with Kuparuk crude.

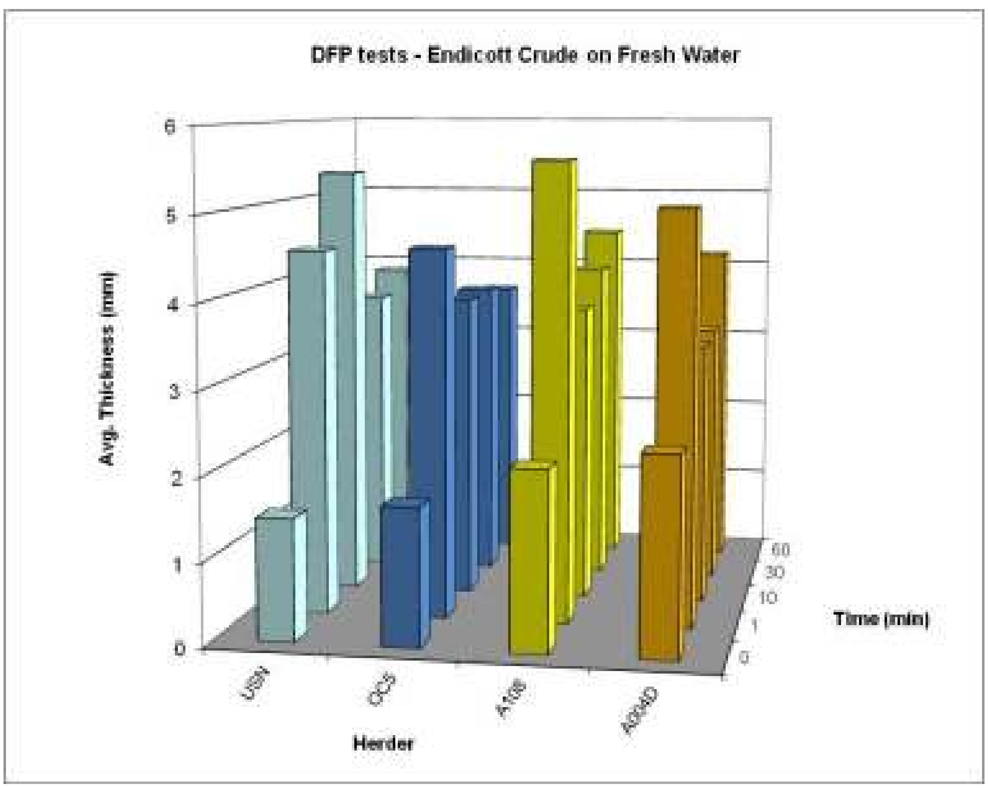

Figure 11. DFP test results with Endicott crude.

For the tests using Endicott crude oil (Figure 11) the best initial and overall performance was with the A108 herder. For both silicone herders, the herded slick thickness declined after reaching its maximum before the rocking started, then slowly increased over the final 50 minutes of rocking. For the USN herder the thickness declined and stayed lower after peaking at the 10-minute mark. The OC5 was the least effective. Based on the DFP tests results, the USN, A108 and A004-D herders were tested in the 10-m^2 pool.

5.2.3 10-m^2 Pool Tests

Figure 12 shows the results for the three herders in the 10-m^2 pool with Kuparuk crude. The A108 herder produced the best initial slick thickness and the herded slick thickness did not decline as much as it did with the A004-D. The thickness of the slick increased with the USN herder up to 30 minutes, and then declined by 60 minutes. The USN herder produced the thickest slicks after 60 minutes. All three herders were tested in the wind/wave tank.

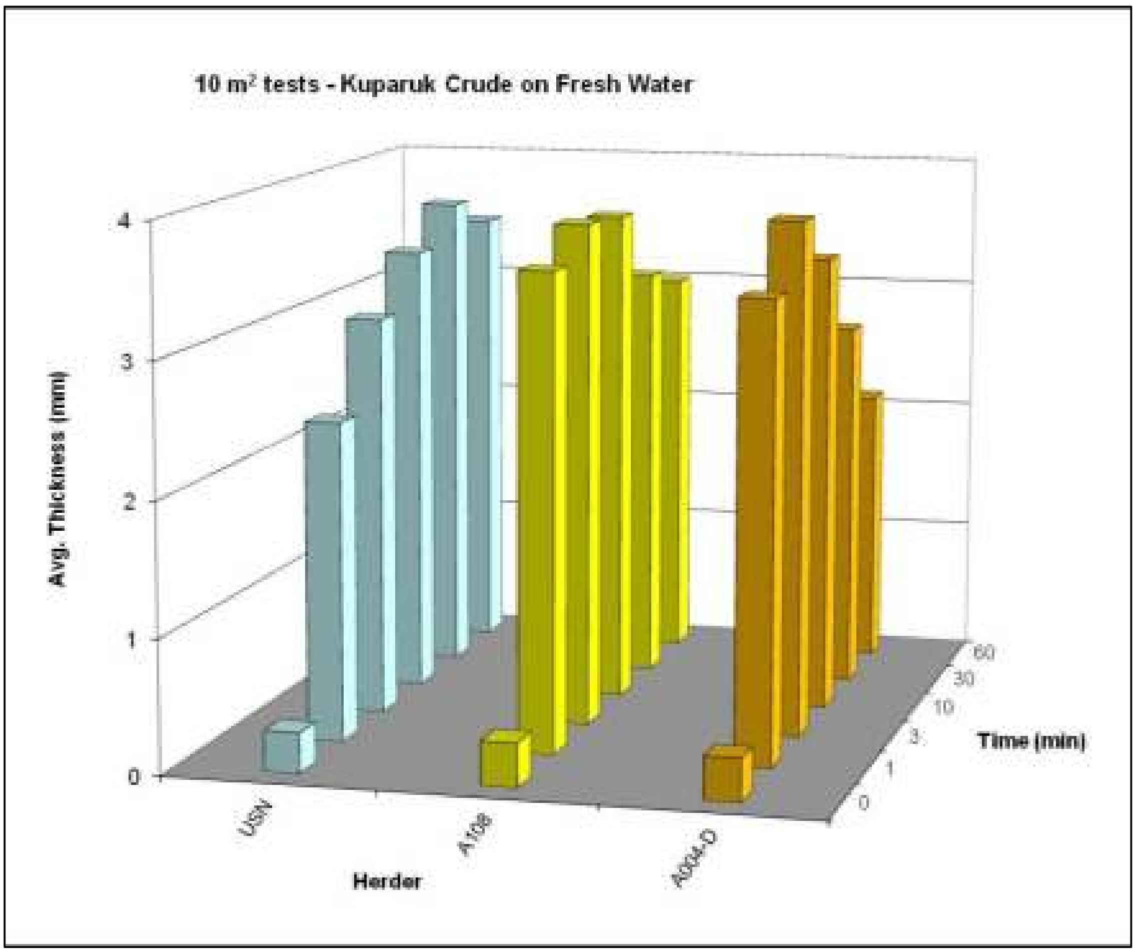

Figure 12. 10-m^2 pool test results.

5.2.4 Wind/Wave Tank Monolayer Persistence Tests

Each herder successfully contained a slick of Kuparuk crude in low, non-breaking waves. As soon as the waves were increased to the point where they began to crest and break, the herded slick broke up into many slicklets, and shortly afterwards, began to spread. It was clear that the breaking waves disrupted the monolayer of herder on the water surface. There was no qualitative difference between the performance of the three herders.

5.2.5 Small-scale Quiescent Burn Tests in the Wind/Wave Tank

Table 2 gives the results of the three burn tests with free-floating fresh ANS crude thickened by herder. Both silicone herders performed as well, or better, than the USN herder in retaining a burning slick. The USN herder has been tested in the field (Sørstrøm *et al.* 2010). Without herder, none of the slicks would be thick enough to ignite (which requires a minimum 1 mm thickness for fresh crude oil – SL Ross 2005).

Table 2: Small scale burn results.

Herder	Oil Weight [g]	Residue Weight [g]	Burn Time [min:sec]	Efficiency [%]
USN	349.2	223.7	1:30	35.9
A108	350.6	219.7	1:52	37.3
A004-D	353.2	176.9	1:33	49.9

6 Ohmsett Experiments on Herder Persistence in Waves

Surfactant film persistence (i.e., how long the monolayer generated by a specific herding agent will last as a function of sea state) and to what degree periodically replenishing the film can counteract this was investigated in an 8-day test program at Ohmsett from May 13 through 20, 2011. The experiments took advantage of the facility's newly upgraded wave making capabilities. Overhead digital video and photographs were taken to qualitatively compare and determine the persistence of three herding agents in calm conditions, a swell and breaking waves. A total of 11 experiments were completed with three herding agents (USN, Silsurf A108 and Silsurf A004-D) in the three wave conditions: 9 tests as per the test protocol plus an additional duplicate test and a control (no herder).

6.1 Experiment Methods

The Ohmsett test plan may be found in Appendix 3.

6.1.1 Preparations

The preparations for the tests included:

- Obtaining 500 mL each of USN herder (65% v/v sorbitan monolaurate [Span 20] and 35% 2-ethyl butanol), Silsurf A108 herder and Silsurf A004-D herder.
- Obtaining a person lift.
- Obtaining one drum of Endicott crude oil.
- Constructing one 15-foot diameter plastic pipe containment ring.
- Erecting the windscreens on either side of the tank (Figure 13). Calm or very-low wind conditions were necessary during testing to allow longer test times.
- Installing booms at either end of the tank (as per a dispersant test).
- Obtaining sorbent sweeps to remove herder and sheen from tank after a test is completed.
- Positioning, checking and calibrating overhead camera(s) for data collection.
- Conducting required safety checks and notifications.
- Conducting several dry run tests to set new wave maker to produce a range of breaking and non-breaking waves and fine tune release and test procedures.

6.1.2 Test Setup, Instrumentation and Procedures

The basic test procedure used for all herder persistence tests was as follows.

1. 15 L (4 gallons) of crude was measured into a 5-gallon plastic pail, weighed and placed on the person lift.
2. 20 mL of herding agent was measured into a syringe, weighed and put on the person lift.
3. Two pre-weighed spray bottles of additional herding agent were placed on the Main Bridge.

Figure 13. Ohmsett tank during herder persistence testing showing wind screens and person lift.

Figure 14. Cleaning water surface in test area with sorbent sweep attached to Main Bridge.

4. The Main Bridge was used to clean the entire length and width of the enclosed test area with a sorbent sweep (Figure 14).
5. The Main Bridge was positioned at the upwind end of the tank within the boomed area.
6. The containment ring was lowered onto the water surface from under the Main Bridge at the up-drift side of the tank and contact with the water around its entire circumference was visually confirmed.
7. Approximately 20 mL of herding agent was carefully placed on the water surface inside the containment ring from the person lift (Figure 15). Since the purpose of these tests was to evaluate the persistence of the herder monolayer, the herder was added first to minimize oil spreading differences between tests.
8. The 15 L (4 gallons) of crude was gently poured along a spill plate onto the water surface inside the containment ring from the person lift.
9. The person lift was moved into position for digital overhead video and photographs.
10. The Main Bridge was positioned over the containment ring in preparation for lifting it out of the water.
11. The wave maker was started (if required) and the plastic containment ring was lifted and secured under the Main Bridge just before the first wave reaches the oil (Figure 17). For swell conditions the wave maker was set at a 20-inch stroke with a frequency of 15 cpm; for breaking waves the stroke was set at 6 inches and the frequency started at 33 cpm, increased to 35 cpm after one minute and to 36 cpm after another minute.
12. The video on the person lift was started. Digital photos of the slick were taken from the man lift as required.
13. The slick was allowed to drift downwind for as long as possible, until it reached a wall or containment boom. Wind speed and direction were recorded at the weather station, but are not representative of the wind speed at the water surface because of the wind screens erected along both sides of the tank.
14. The Main Bridge was moved to keep up with the slick, but not interfere with it.
15. Additional herding agent was added from pre-weighed spray bottles on the Main Bridge as required if the oil began to spread before it reached a wall or boom (Figure 18).
16. The waves were left on until the slick reached a wall or boom in the tank then the wave maker was stopped and the video stopped. The time of additional herder applications, verbal observations of slick behavior and the time for the slick to reach the wall or end of the tank were recorded on the video taken from the person lift.
17. The oil pail, herder syringe and herder spray bottles were reweighed.
18. The water spray from the Main Bridge fire monitors was used to sweep the surface oil remaining on the water surface at the end of the test to a common collection area at one corner of the containment boom.
19. The boom was then raised and the oil pushed under the boom out of the containment area.
20. At the end of each test it was necessary to remove the residual herder from the water surface inside the boomed area. This was accomplished by running a train of breaking waves down the tank for several minutes and using the Main Bridge fire hoses to disperse the herder into the water column.

Figure 15. Placing herder on the water surface inside containment ring

Figure 16. Releasing oil into ring from person lift.

Figure 17. Containment ring lifted up and secured under Main Bridge.

Figure 18. Applying additional herder during test run.

21. Visual observation of the spreading of a small amount (ca. 10 mL) of test oil, randomly placed on the test area's surface, was used to confirm that the herder had been removed from the water surface prior to each test.

6.1.3 Test Matrix

The following parameters were varied in these experiments:

1. Herder type (USN, A108 and A004-D);
2. Wave conditions (calm, swell, occasional breaking waves)

All the tests were conducted with fresh Endicott crude oil. Table 3 gives the matrix for the tests. Testing took take place May 14 through 20, 2011. The control tests were scheduled last to maximize the available weather window for the experiments. Figure 19 shows typical swell waves and Figure 20 shows typical breaking waves.

Table 3. Matrix of Tests at Ohmsett.

Test Number	Wave Condition	Herder
1	Calm	USN
2	Calm	A108
3	Calm	A004-D
4	Breaking	USN
5	Breaking	A108
6	Breaking	A004-D
7	Swell	USN
8	Swell	A108
9	Swell	A004-D
10 (Dup 8)	Swell	A108
11	Swell, then Breaking	None

6.2 Ohmsett Experiment Results

The raw data from the experiments at Ohmsett may be found in Appendix 4. Figure 21 illustrates the typical behaviour of a well-herded slick in swell conditions and Figure 22 shows how breaking waves disrupt the herder monolayer allowing the slick to spread and break up. Figure 23 shows a herded slick in calm conditions.

Table 4 summarizes the data collected from each experiment and Table 5 summarizes the observed slick behavior in each experiment.

Figure 19. Typical swell wave conditions during a herder test.

Figure 20. Typical breaking wave conditions during a herder test.


Figure 21. Herded slick in swell conditions.


Figure 22. Herded slick respreading and breaking up into slicklets in breaking waves.

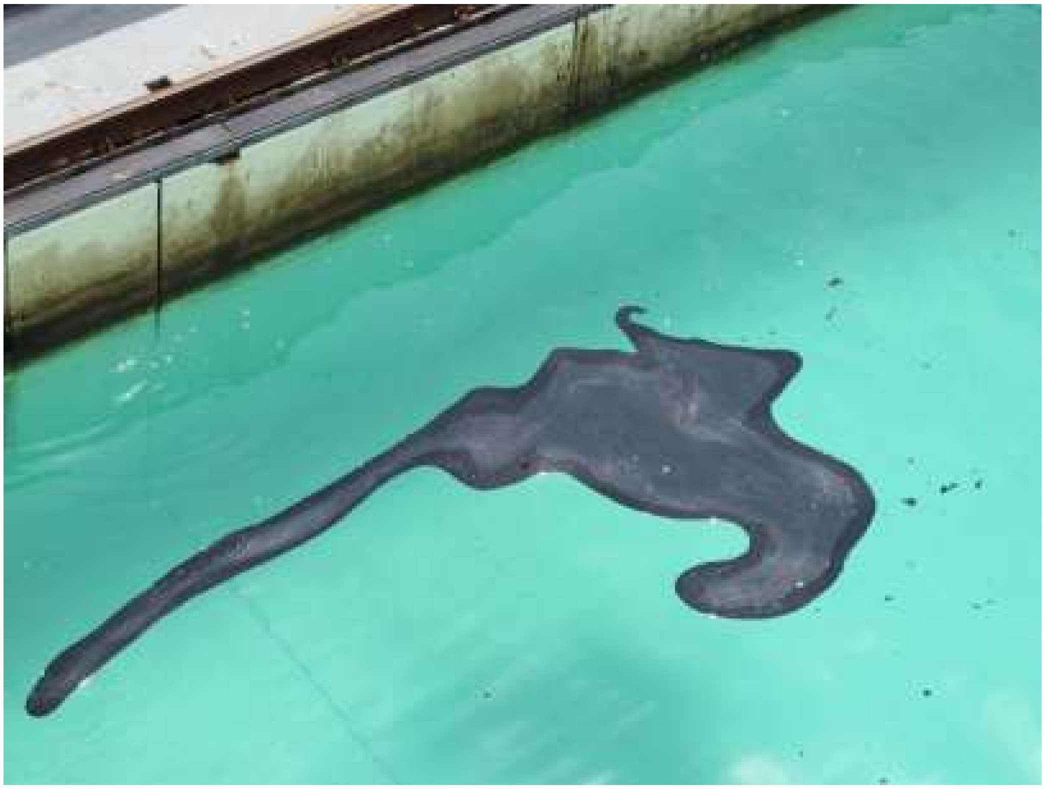

Figure 23. Typical herded slick in calm conditions.

Table 4. Summary of Ohmsett Experiment Data.

			Conditions					Oil						Herder								
Test #	Date	Herder	Waves	Air T (°C)	Water T (°C)	Wind Speed (mph)	Wind Dir (°)	Type	Oil T (°C)	Pail Gross Weight (lb.)	Pail Tare Wt (lb.)	Oil Net Wt (lb)	Oil Volume (L)	Syringe Gross Wt (g)	Syringe Tare Wt (g)	Initial Herder (mL)	Spray #1 Gross Wt (g)	Spray #1 Tare Wt (g)	Spray #2 Gross Wt (g)	Spray #2 Tare Wt (g)	Additional Herder Volume (mL)	Total Herder (mL)
1	15/05/2011	USN	Calm	17.8	19.4	3.8	190	ndico	20	31.19	2.97	28.22	14.4	33.06	14.62	19.0	127.92	110.91	128.71	120.9	25.5	44.5
2	15/05/2011	A108	Calm	17.2	19.4	5.1	143	ndico	20	31.09	2.9	28.19	14.4	35.58	14.59	20.6	139.36	129.98	162.66	155.39	16.3	36.9
3	16/05/2011	A004-D	Calm	12.8	20	21	87	ndico	20	31.22	3	28.22	14.4	35.1	14.59	20.1	112.57	112.59	129	102.28	26.2	46.3
4	16/05/2011	USN	Breaking	13.3	19.4	9.8	75	ndico	19.5	31.22	3.03	28.19	14.4	33.39	14.63	19.3	122.02	82.71	121.88	82.48	80.9	100.2
5	18/05/2011	A108	Breaking	15.4	18.3	7.6	92	ndico	17.5	30.94	3	27.94	14.3	35.6	14.66	20.5	148.27	137.55	139.37	105.47	43.7	64.3
6	18/05/2011	A004-D	Breaking	17.9	18.9	7.9	128	ndico	18	31.08	2.97	28.11	14.4	33.79	14.54	18.9	104.67	92.63	113.14	81.26	43.1	61.9
7	19/05/2011	USN	Swell	17.2	18.9	2.7	72	ndico	17.8	31.18	2.98	28.2	14.4	33.99	14.84	19.7	109.43	71.16	115.81	107.84	47.5	67.2
8	19/05/2011	A108	Swell	16.1	18.9	5.7	83	ndico	17.8	31.16	3.05	28.11	14.4	35.5	14.64	20.5	127.67	111.48	131.37	102.42	44.3	64.7
9	19/05/2011	A004-D	Swell	16.1	19.4	7	82	ndico	18.1	31.95	3	28.95	14.8	34.38	14.71	19.3	97.16	59.86	82.49	71.65	47.2	66.5
0(8DUI	20/05/2011	A108	Swell	15.6	19.4	2.2	303	ndico	18	31.73	2.94	28.79	14.7	35.7	14.59	20.7	111.67	100.72	113.38	80.88	42.6	63.3
11	20/05/2011	None	vell/Breaki	17.8	19.4	3.5	60	ndico	18.4	31.61	2.93	28.68	14.7	0	0	0.0	0	0	0	0	0.0	0.0

Tank surface water salinity = 25.2 ppt @ 18.2°C on 18/05/2011

Table 5. Observed Slick Behavior Summary.

Test No.	Herder	Waves	Slick Persistence [min:sec]	Comments
1	USN	Calm	45:45	Additional herder added to keep slick away from tank wall 1 min 30 sec after ring lifted. Slick moved down tank without touching sides. Slick broken into many slicklets by end of test.
2	A108	Calm	44:27	Additional herder added to keep slick away from tank wall 2 min and 7min 13 sec after ring lifted. Wind shifted 12 to 14 minutes into experiment and slick drift changed by 180°. Slick in three narrow blobs at end.
3	A004-D	Calm	27:43	Windy and raining. Additional herder added to keep slick away from tank wall 1 min 14 sec and 27min 14 sec after ring lifted. Slick in one main blob plus many tiny slicklets.
4	USN	Breaking	0:56	Windy and raining. Slick begins to stretch and break into slicklets when initial waves pass under slick. Additional herder added from both sides to reherd 1 min 15 sec after ring lifted. Slick recoalesces into one long blob with some smaller slicklets. First breaking wave at 7:01. Slick immediately begins to spread and break up into small slicklets. Touches tank wall at 7:57
5	A108	Breaking	1:11	Slick begins to stretch and break into slicklets when initial waves pass under slick. Single breaking wave at 0:23. Additional herder added from all sides to reherd 1 min 13 sec after ring lifted. Slick contracts and recoalesces into several blobs with some smaller slicklets. Continuous breaking waves begin at 4:28.Slick OK after the first few lone breakers, but soon begins to spread and break up into small slicklets. Touches tank wall at 8:20
6	A004-D	Breaking	0:56	Slick begins to stretch and break into slicklets when initial waves pass under slick. Single breaking wave at 0:17. Additional herder added from all sides to reherd 1 min 9 sec after ring lifted. Slick contracts and recoalesces into one blob with some smaller slicklets. Continuous breaking waves begin at 3:25. Herded slick immediately begins to spread and break up into small slicklets. Spread out and dispersing at 4:21. Touches tank end at 10:27.
7	USN	Swell	27:41	Slick begins as large blob with several smaller slicklets. Additional herder added at 1:49. Slick stretches and contracts as waves pass beneath it. Dark band forms around edge of herded slick. Waves help reherd and recoalesce slick. Slick begins to narrow and elongate. One end of slick touches wall at approx. 15:00, but decide to continue test. After approx. 23 minutes long, narrow slick begins to break into streamers. Touches wall 27:41
8	A108	Swell	40:05	Slick begins as large blob with several smaller slicklets. Additional herder added at 0:27. Slick stretches and contracts as waves pass beneath it. Waves help reherd and recoalesce slick. Dark band forms around edge of herded slick. One edge of slick touches wall at approx. 5:00, but decide to continue test. Slick begins to narrow and elongate by 13:00. One end of slick touches wall again at approx. 16:00, but decide to continue test. Touches end at 40:05.
9	A004-D	Swell	18:59	Slick begins as main blob with many smaller slicklets. Additional herder added at 0:20 and 12:09. Slick stretches and contracts as waves pass beneath it. Waves help reherd and recoalesce slick into one blob. Dark band forms around edge of herded slick. One edge of slick touches wall at approx. 15:00, but decide to continue test. Discontinue test at 18:59 as slick remains against wall – still herded.

Test No.	Herder	Waves	Slick Persistence [min:sec]	Comments
10 (Dup 8)	A108	Swell	35:00	Slick begins as large blob. Additional herder added at 0:35, and again several times between 12:00 and 20:00 to keep slick away from wall. Slick stretches and contracts as waves pass beneath it. Dark band forms around edge of herded slick. One edge of slick touches wall at approx. 14:00, but decide to continue test. Slick begins to narrow and elongate. Touches wall again at 35:00.
11	None	Swell, then Breaking	9:54 in swell	Slick begins to spread as soon as ring is lifted. Unfortunately, despite cleaning the tank surface of herder after Test 10, there is considerable oily sheen (as opposed to herder) on the tank surface visible in the video that prevents the control slick from sheening. Slick spreads to a much larger area than same volume of oil when herded. Waves increased to breaking at 9:54, which immediately causes slick to spread, break up into many small slicklets and disperse. Slick area in breaking waves much larger than that of herded slicks in breaking waves.

Based on the visual observations and review of the test video the following observations were made:

- The monolayer of each of the two best herders will survive for more than 45 minutes in a calm sea (the Ohmsett tests were terminated when the slicks reached the end of the tank).
- The presence of breaking or cresting waves rapidly disrupts the herder monolayer and the oil slick resulting in the production of many small slicklets from the herded slick and the respreading of the oil to thin slicks..
- The monolayer survives for considerable periods of time in a swell condition, but the constant stretching and contracting of the herded slick results in elongating the oil slick and slowly breaking the slick into smaller segments.
- The Silsurf A108 herder performed noticeably better than the other two herders in all test conditions.

7 Conclusions and Recommendations

7.1 Conclusions

- In the laboratory herding tests the Silsurf A108, Silsurf A004-D and USN herders performed best.
- The two silicone-based herders retained a small burning crude oil slick and achieved burn efficiencies as good as or better than the USN herder.
- The Silsurf A108 herder performed noticeably better than the Silsurf A004-D and USN herders in all test conditions during the Ohmsett tests.
- The monolayer of each of the three herders will survive for more than 45 minutes in a calm sea.
- The presence of breaking or cresting waves rapidly disrupts the herder monolayer and the oil slick resulting in the production of many small slicklets from the herded slick and the respreading of the oil to thin slicks..
- The monolayer survives for considerable periods of time in a swell condition, but the constant stretching and contracting of the herded slick results in elongating the oil slick and slowly breaking the slick into smaller segments.

7.2 Recommendations

- Work should begin on commercializing the USN and silicone herders, including getting them listed on the EPA NCP Product Schedule and developing suitable application systems.
- The use of the USN and silicone herders to thicken slicks in open water conditions for *in situ* burning should be included in plans for future offshore oil spill response field trials.
- Consideration should be given to how herders could be employed to improve offshore skimming encounter rates in suitable open water conditions.

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Appendix 1 – Laboratory Test Plan

First Draft

Test Plan

Laboratory Spreading Tests to Identify Warm Water Herders

Task 1 of the Project

RESEARCH ON USING OIL HERDING AGENTS FOR RAPID RESPONSE IN SITU BURNING OF OIL SLICKS ON OPEN WATER

BOEMRE CONTRACT NUMBER M11PC00011

to:

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Oil Spill Response Research (OSRR) Program, MS 4021
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March 22, 2011

9 Introduction

Since 2004, the main goal of the research on herders has been to determine their efficacy at enhancing *in situ* burning in ice concentrations too low for natural containment of the oil slick by the ice itself (ice concentrations between <10% and 60%). In the research on herders in drift ice a considerable number of the experiments were performed in open water or trace ice conditions as an integral part of each experiment matrix, certainly enough to know that they will work quite well to thicken slicks for efficient *in situ* burning on quiescent cold seawater.

Herders were studied in the 1970's as an open water oil spill response technique but the goal then was to provide containment for mechanical recovery. In this original application herders were limited to relatively calm conditions because the herder itself dissipated quickly in higher energy sea conditions allowing the slick to re-spread. This dissipation occurred over periods of tens of minutes, which wasn't enough time to allow skimming of the herded slick. *In situ* burning is a process that only requires minutes to initiate and complete. Once ignited, the air being drawn into a large *in situ* oil fire by the combustion process will also contain the burning slick and thicken it further. Thus, there is great potential that *in situ* burning enhanced by chemical herders can be a very effective rapid response technique in ice-free waters – both in the Arctic and in temperate regions. However, there remain two issues requiring further study:

3. Suitability of herders for warmer water conditions; and,
4. Surfactant film persistence in open water sea conditions.

The focus of this test plan is the first issue: the suitability of the best cold-water herders for warmer water conditions (i.e., the original US Navy study identified better surfactant/solvent combinations at room temperature than the "USN" blend that has been selected for icy water conditions). Other herder recipes – e.g., OC-5 - may very well outperform USN at 20°C. Also, the new silicone superwetters identified as the best for cold-water use may not be the best for warmer waters. This portion of the work would be accomplished using various pan experiments in the laboratory.

10 Goal

Experiment in the laboratory with the USN herder formulations, OC-5 and the best silicone herder formulation to find the most effective product for warmer water temperatures.

11 Work Plan

A series of comparative experiments would be conducted with hydrocarbon-based and silicone herders in 1-m^2 pans (Figure 1), a 10-m^2 pan (Figure 2), small pans mounted on a rocking shaker (Figure 3) and the SL Ross tank (Figure 4). Overhead digital photographs would be taken and analyzed by computer to determine the herder effectiveness. Video will also be taken. One or two small-scale (≈ 40 cm diameter) burn experiments would be conducted with the best herders identified for use on warm water to confirm their suitability.

Figure 1. Lined 1-m^2 steel pans used for experiments.

Figure 2. Ice blocks placed in the 10-m^2 frame.

Figure 3. Two-platform rocking shaker.

Figure 4. Free-floating ice in SL Ross tank prior to an experiment.

The following parameters would be varied in the herding experiments:

- Five candidate warm-water herders
    1. The original USN cold water blend (65% Span-20 and 35% 2-ethyl butanol)
    2. Siltech A108
    3. Siltech A004D
    4. OC-5 [the old Exxon Oil Collector]
    5. A Span 80-based herder field tested by the US Navy that was a better warm-water herder than the USN cold water blend (75% Span-80 and 25% 2-ethyl butanol)
- One ambient temperature (≈ 20°C)
- Two water salinities (0 and 35 ‰), [screening tests will be conducted in the 1-m^2 pans to ensure that water salinity does not play a major role in each herder's performance, with the intention that the majority of the experiments would be done with tap water for simplicity]
- Three oil types: two crudes that are fluid at ambient temperatures and a No. 2 fuel oil.

The general procedure for a 1-m^2 pan experiment is:

8. Place 20 L (a depth of 2 cm) of room-temperature water in each 1-m^2 pan (Figure 1) lined with freshly rinsed (with tap water) new plastic film.
9. Take a sample of the water from the surface using a Petri dish and measure the water-air interfacial tension (IFT) using the DuNuoy Ring Tensiometer. If the IFT reading is less than 60, replace the water and film and retry.
10. Carefully pour 1 L of the crude on the water; making sure that it doesn't stick to the bottom of the tray while being poured.
11. Allow the oil to spread to equilibrium and take a digital photograph from overhead for subsequent oil area analysis.
12. Apply prescribed amount (150 µL for USN) of herding agent to open water area with micropipette.
13. Allow the oil to contract and take another digital photograph after one minute, 10 minutes, 30 minutes and 1 hour.
14. Empty water from pans, remove plastic film, dry pans with paper towels.

The general procedure for a rocking shaker tray experiment is:

11. Place 1.115 L of room-temperature water (≈ 2 cm deep) in each of two trays (18 cm wide x 28 cm long) and allow them to equilibrate to the test temperature in the environmental chamber.

12. Take a sample of the water from the surface using a Petri dish and measure the water-air interfacial tension (IFT) using the DuNuoy Ring Tensiometer. If the IFT reading is less than 60, replace the water and retry.
13. Carefully place 50 mL of the test oil on the water, making sure that it doesn't stick to the bottom of the tray while being poured.
14. Allow the oil to spread to equilibrium and take a digital photograph from overhead for subsequent oil area analysis.
15. Apply 5 to 10 µL of herding agent to an open water area with a micropipette (the target dose for the USN herder is 2.5 µL based on a recommended treatment of 50 mg/m^2; however, it is impossible to deliver an accurate dose with the viscous herder which tends to form discrete droplets at the end of the micropipette that would only detach with the higher volumes).
16. Allow the oil to contract and take another digital photograph after one minute.
17. Carefully place the trays on the rocking shaker and start shaker and timer.
18. After 10 minutes and 30 minutes, stop the shaker, gently remove the trays re-photograph the slicks and replace the trays on the shaker.
19. Stop experiment after 1 hour, gently remove the trays re-photograph the slicks.
20. Remove and empty trays, clean with Alconox and hot water and rinse thoroughly with hot water.

The oil and herder combinations to be tested at a scale of 10 m^2 and in the SL Ross wind/wave tank will be selected based on the results of the 1-m^2 and rocking shaker experiments.

The 10-m^2 experiment (Figure 2) is performed in a rectangular, plastic-lined wooden frame (3.05 m x 2.95 m x 9 cm high) lined with a new, rinsed sheet of 1-mil plastic film for most experiments to ensure a clean, uncontaminated surface. Two, white plastic tarpaulins are attached to the bottom of the frame to increase the contrast in the photos between oil and water.

The general procedure for a 10-m^2 pan experiment is:

8. Place 200 L (a depth of ≈ 2 cm) of room-temperature water in the 10-m^2 pool lined with freshly rinsed (with tap water) new plastic film.
9. Take a sample of the water from the surface using a Petri dish and measure the water-air interfacial tension (IFT) using the DuNuoy Ring Tensiometer. If the IFT reading is less than 60, replace the water and film and retry.
10. Carefully pour 2 L of the oil on the water, making sure that it doesn't stick to the bottom of the pool while being poured.
11. Allow the oil to spread to equilibrium and take a digital photograph from the rafters overhead for subsequent oil area analysis.
12. Apply the prescribed amount (1500 µL for USN) of herding agent to open water area with micropipette.
13. Allow the oil to contract and take another digital photograph after one minute, 10 minutes, 30 minutes and 1 hour.

14. Pump water from pool, remove plastic film, and replace film for next experiment.

For the wind/wave tank experiments (Figure 4), two 4' x 8'- sheets of white signboard are laid end to end and weighted down on the bottom of the tank in order to improve the discrimination in the overhead photos and video between oil and water. A test area is created by isolating an area of water surface with floating barriers stretched from one side of the tank to the other. This eases cleaning of the test area water surface between experiments. For the *in situ* burn experiments, metal heat shields are installed along the sides of the tank and the metal fume hood is swung over the burn area. The smoke from the burns is removed with a 200-m^3/min fan, through a 60-cm metal duct that is connected to the fume hood suspended 1 m above the water surface (Figure 5).

Figure 5. Wind/wave tank set up for burn experiments with heat shields and exhaust hood.

The general procedure for a wind/wave tank experiment is:

10. Raise the floating barriers at either end of the test section and thoroughly clean the water surface with sorbent pads to remove any oil or herder traces.

11. Take a sample of the water from the surface using a Petri dish and measure the water-air interfacial tension (IFT) using the DuNuoy Ring Tensiometer. If the IFT reading is less than 60, reclean the test area.
12. Apply prescribed amount (500 µL for USN) of herding agent to open water area with micropipette.
13. Turn on the video.
14. Carefully pour 500 mL of the crude on the water; making sure that it doesn't submerge while being poured.
15. Allow the oil to spread to its herded equilibrium and take a digital photograph from overhead for subsequent oil area analysis.
16. Turn on the wave generator to produce non-breaking waves and turn on the fan to counterbalance the wave drift.
17. At proscribed time intervals (to be determined after an initial dry run) increase the wave generator to produce more energetic waves.
18. Stop wave generator, recover oil and herder with sorbent pads.

A typical *in situ* burn experiment involves the following steps:

1. Raise the floating barriers at either end of the test section and thoroughly clean the water surface with sorbent pads to remove any oil or herder traces.
2. Take a sample of the water from the surface using a Petri dish and measure the water-air interfacial tension (IFT) using the DuNuoy Ring Tensiometer. If the IFT reading is less than 60, reclean the test area.
3. A volume of 400 mL (to form a 40-cm diameter herded slick) of fresh crude oil is measured into a graduated cylinder and weighed.
4. For those experiments involving pre-treatment of the water surface of the tank, 500 µL of the herder is placed on the water surface using a micropipette. Otherwise, the herder is added after the slick had spread to equilibrium.
5. The oil is carefully poured onto the surface of the tank.
6. Ignition is attempted first with a propane soldering torch flame.
7. A stopwatch and video records the following times: initial ignition time, full ignition (100% flame coverage); time to the vigorous (or intense) burn phase; 50% flame coverage; and, extinction.
8. After extinction of the flame, pre-weighed rectangles of sorbent are used to recover the residue from the water surface. After use, each pad is shaken to remove as much water as possible. Then the pads were reweighed to determine the mass of residue. The sorbent pads are then hung up to dry overnight, and reweighed after any water had evaporated. It is assumed that very little of the burn reside evaporates in the same time period.

All the data collected will be processed, analyzed and collated. A summary data report will be written documenting results, conclusions and recommendations arising from this task of the research program.

12 Time Frame

A four-week laboratory experimental program is planned, commencing in late March/early April 2011 in order to be finished in time to plan the experiments at Ohmsett scheduled in mid-May 2011.

13 Quality Control Plan

A quality control plan for the experimental work was approved by the BOEMRE CO and COR. The part of this plan pertaining to the Task 1 laboratory experiments is reproduced below

13.1.1.1.1.1

13.1.1.1.1.2 **Initial Calibration Data.** A check is made to ensure that data is available to show the initial source of calibration data for each piece of instrumentation used in the project. This includes any calibration information necessary to assure that the calibration data is current for the project.

13.1.1.1.1.3

13.1.1.1.1.4 **Pre- and Post-Daily Checks.** These are checks that are performed on the instrumentation (i.e., DuNuoy tensiometer and densitometer calibration checks with pure water) 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 experiments need to be repeated.

Test Checks and Conditions. These checks ensure that the test plan's instructions on how the experiment is to be done are followed and that the records that are to be made during the experiment are completed accurately. Of prime importance for this project is verifying that the water surface in the various experiment apparati is clean and surfactant free prior to starting each individual experiment.

Significant Occurrences/Variations. This part of the quality checks will be concerned with recording any significant occurrences/variations that might occur during the experiments. These will be immediately reported to I. Buist.

Duplicate Experiments. At least 10% of the experiments will be randomly selected to be repeated.

13.2

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 Draft Technical Report and sufficiently described so that they can be used by independent sources to duplicate the results. With respect to written material, all draft material will be reviewed by at least one other SL Ross senior staff professional engineer before submission to MMS.

14 Safety And Environmental Plan

SL Ross staff has been conducting such experiments at the SL Ross facility for more than twenty years without incident. Only minor safety hazards are involved and these are mitigated through the use of proper lab procedures, appropriate PPE, solvent storage systems, fume hoods, fire extinguishers, eyewash stations and emergency showers. The oily waste and spent solvents are disposed of using an approved waste handler in accordance with the regulations of the Province of Ontario.

Appendix 2 – Laboratory Test Data

Test Time

Area

[illegible]

Test - Time	T1AL-00	T1AL-01	T1AL-10	T1AL-30	T1AL-60	T1AR-00	T1AR-01	T1AR-10	T1AR-30	T1AR-60
AirRes	0.481901	0.124803	0.123080	0.159854	0.158092	0.961962	0.112003	0.171911	0.178808	1.89E-41
	3.00E-07	0.003	0.016	0.000	0.040	2.00E-07	0.172	5.00E-07	1.25E-06	2.50E-07
	2.00E-07	0.003	0.003	2.00E-07	2.50E-07	2.00E-07	2.50E-07	2.50E-07	1.25E-06	0.189
	0.467	4.37E-04	2.50E-07	0.134	2.50E-07	2.00E-07	2.50E-07	2.00E-07	5.00E-07	2.00E-07
	2.50E-07	1.66E-04	5.00E-07	2.50E-07	0.151	2.50E-07	7.50E-07	5.00E-07	0.178	2.50E-07
	2.00E-07	0.118	7.50E-07	5.00E-07	2.50E-07	2.50E-07	2.50E-07	1.00E-06	2.50E-07	1.00E-06
		5.00E-07	2.50E-07	5.00E-07	5.38E-07	2.50E-07	5.00E-07	2.50E-07	1.40E-06	1.75E-06
			2.50E-07	5.00E-07	2.50E-07	0.001	2.50E-07	0.171	2.50E-07	1.25E-06
			2.50E-07	5.00E-07	2.50E-07		2.50E-07	1.25E-06		7.50E-07
			5.00E-07	6.97E-04	2.50E-07		2.00E-07	2.50E-07		5.00E-07
			5.00E-07	2.50E-07			2.00E-07	5.40E-07		2.50E-07
			6.40E-04	2.50E-07				2.50E-07		2.00E-07
			2.50E-07					2.50E-07		2.50E-07
			2.50E-07					2.50E-07		
			0.114					2.50E-07		
			7.50E-07					2.50E-07		
			2.50E-07					2.50E-07		
								7.00E-07		
								2.00E-07		
								2.50E-07		
								2.50E-07		
								5.00E-07		
								2.40E-07		
								1.00E-06		
								2.50E-07		
								2.50E-07		
								2.50E-07		

Test - Time	T2AL-00	T2AL-01	T2AL-10	T2AL-30	T2AL-60	T2AR-00	T2AR-01	T2AR-10	T2AR-30	T2AR-60
AirRes	0.469811	0.148092	0.182801	0.222006	0.231982	0.469875	0.289456	0.229756	0.289221	0.362229
	0.49	2.50E-07	5.00E-07	0.222	0.231	2.00E-07	0.289	0.229	0.289	0.362
	7.00E-07	0.168	0.182	2.50E-07	5.00E-07	2.50E-07	2.50E-07	2.00E-07	2.50E-07	5.00E-07
	2.00E-07	2.50E-07		5.00E-07	1.00E-05	0.406	1.00E-06	5.00E-07	2.50E-07	2.28E-04
	2.50E-07	5.00E-07		2.50E-07	5.00E-07	7.99E-04	2.50E-07	2.50E-07	2.28E-04	
	2.50E-07	2.50E-07		5.00E-07		7.40E-05	2.00E-07	2.50E-07		
	5.00E-07	5.00E-07		2.50E-07			1.72E-04	2.50E-07		
	2.50E-07			5.00E-07			1.62E-04	2.50E-07		
	5.00E-07			1.25E-06			7.20E-05	2.50E-07		
	5.00E-07			2.50E-07			1.00E-04	5.00E-07		
	2.50E-07			5.00E-07			2.50E-07	1.10E-04		
	7.50E-07			5.00E-07			2.50E-07	2.50E-07		
	5.00E-07			2.50E-07			2.00E-07	2.50E-07		
	1.25E-06			2.50E-07			2.50E-07	2.50E-07		
	7.50E-07						2.50E-07			
	1.25E-06						2.50E-07			
	2.25E-06									
	2.50E-07									
	2.50E-07									

Test - Time	T3AL-00	T3AL-01	T3AL-10	T3AL-30	T3AL-60	T3AR-00	T3AR-01	T3AR-10	T3AR-30	T3AR-60
AirRes	0.389113	0.372081	0.529808	0.580081	0.615812	0.496008	0.321426	0.58700	0.574082	0.627849
	0.374	0.368	0.52	0.58	0.616	0.496	0.327	0.034	0.574	0.627
	2.50E-07	2.00E-07	5.00E-07	2.00E-07	2.00E-07	2.50E-07	2.00E-07	2.00E-07	2.50E-07	2.50E-07
	2.50E-07	2.00E-07	5.00E-07	2.00E-07	2.00E-07	2.50E-07	2.00E-07	2.00E-07	2.00E-07	2.43E-04
	2.50E-07	2.50E-07	7.00E-07	2.50E-07	2.50E-07	2.50E-07	2.50E-07	3.50E-07	2.50E-07	4.00E-04
	2.50E-07	2.50E-07	2.50E-07		2.00E-07	2.50E-07	2.30E-04	2.50E-07	2.50E-07	5.00E-07
	5.00E-07	1.00E-06	2.50E-07		5.00E-07	2.50E-07	5.61E-04	2.50E-07	5.00E-07	2.50E-07
	2.50E-07	5.00E-07	2.50E-07		2.00E-07	2.50E-07	2.00E-07	7.50E-07	2.00E-07	2.00E-07
	2.50E-07	2.50E-07	2.50E-07		2.00E-07	2.50E-07	0.32E-04	7.00E-07	2.00E-07	5.00E-07
	0.005	2.50E-07	5.00E-07		2.50E-07	2.50E-07	0.006	2.50E-07		1.00E-06
	5.00E-07	2.50E-07	2.50E-07		1.00E-06	2.50E-07	0.977	1.79E-04		2.50E-07
	2.50E-07	2.50E-07	2.00E-07		5.00E-07	2.50E-07	2.50E-07	2.50E-07		7.50E-07
	0.001	2.50E-07	2.50E-07		2.00E-07	2.50E-07	2.50E-07	2.50E-07		5.00E-07
	2.50E-07	2.50E-07	2.50E-07		1.25E-06	1.00E-06	0.000	4.31E-04		2.50E-07
	2.50E-07	2.50E-07	2.50E-07		2.50E-07	2.50E-07	5.00E-07	4.37E-04		2.50E-07
	2.50E-07	2.50E-07	2.50E-07		2.50E-07	5.00E-07	0.002	0.002		2.50E-07
	2.50E-07	2.50E-07	2.50E-07		5.00E-07	2.50E-07	5.00E-07	2.50E-07		2.50E-07
	2.50E-07	2.00E-07	2.00E-07		2.00E-07	2.50E-07	2.00E-07	5.00E-07		2.50E-07
	2.50E-07	2.50E-07	2.50E-07		2.50E-07	2.50E-07	4.00E-07	2.50E-07		2.50E-07
	1.23E-04	0.984			2.00E-07	5.00E-07				2.50E-07
	2.28E-05				2.00E-07	2.50E-07				2.50E-07
	2.50E-07				2.50E-07	2.50E-07				
	7.10E-07				2.00E-07	2.50E-07				
	1.50E-06				2.00E-07	2.50E-07				
	5.00E-07				2.00E-07	2.50E-07				
					5.00E-07	2.50E-07				
					2.50E-07	2.50E-07				
					2.50E-07	2.50E-07				
					2.00E-07					
					1.00E-06					
					5.00E-07					

[illegible]

Sum of Area(?)

Test(?)	Time(?)	[illegible]	Total Area (in2)(?)	Avg. Thickness (mm)(?)	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

Appendix 3 – Ohmsett Test Plan

First Draft

Test Plan for

Task Order No. 493

Task 2 – Ohmsett Testing of Herder Persistence in Waves of RESEARCH ON USING OIL HERDING AGENTS FOR RAPID RESPONSE *IN SITU* BURNING OF OIL SLICKS ON OPEN WATER

Sponsors: U.S. DEPARTMENT OF THE INTERIOR
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Time Frame: MAY 2011

May 5, 2011

Test Plan for

Task 2 – Ohmsett Testing of Herder Persistence in Waves
of
RESEARCH ON USING OIL HERDING AGENTS FOR RAPID RESPONSE *IN SITU* BURNING OF OIL SLICKS ON OPEN WATER

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Sponsors: US DEPARTMENT OF THE INTERIOR
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1. **INTRODUCTION**

Surfactant film persistence (i.e., how long the monolayer will last as a function of sea state) and to what degree periodically replenishing the film can counteract this will be investigated in a 7-day test program at Ohmsett, taking advantage of the facility's newly upgraded wave making capabilities.

1.1 Background

The use of specific chemical surface-active agents, sometimes called oil herders or oil collecting agents, to clear and contain oil slicks on an open water surface is well known (Garrett and Barger, 1972; Rijkwaterstaat, 1974; Pope *et al.,* 1985; MSRC, 1995). These agents have the ability to spread rapidly over a water surface into a monomolecular layer, as a result of their high spreading coefficients, or spreading pressures. The best herding agents have spreading pressures in the mid-40 mN/m range, whereas most crude oils have spreading pressures in the 10 to 20-mN/m range. Consequently, small quantities of these surfactants (about 5 L per kilometre or 50 mg/m^2) will quickly clear thin films of oil from large areas of water surface, contracting the oil into thicker slicks.

Herders sprayed onto the water surrounding an oil slick result in formation of a monolayer of surfactants on the water surface. These surfactants reduce the surface tension of the surrounding water significantly (from 73 mN/m to 25-30 mN/m). When the surfactant monolayer reaches the edge of a thin oil slick it changes the balance of interfacial forces acting on the slick edge and causes the oil/water and oil/air interfacial tensions to contract the oil into thicker layers. Herders do not require a boundary to "push against" and work in open (boundary free) water. A conceptual drawing of the herding process in pack ice is shown in Figure 1. Although commercialized in the 1970s herders ultimately were not used offshore because they only worked in very calm conditions: physical containment booms were still needed to hold or divert slicks in wind speeds above 2 m/s and breaking waves disrupted the herder layer.

1.1.1 Past Research on Herders to Thicken Oil for Burning in Drift Ice

The key to effective *in situ* burning is thick oil slicks. Concentrated pack ice can enable *in situ* burning by keeping slicks thick. In loose pack ice conditions oil spills can rapidly spread to become too thin to ignite. Fire booms can collect and keep slicks thick in open water; however, field deployment tests of booms and skimmers in pack ice conditions in the Alaskan Beaufort Sea highlighted the severe limitations of booms in even trace concentrations of drift ice (Bronson *et al.,* 2002). If slicks could be thickened to the 2- to 5-mm range in drift ice (less than 6 to 7 tenths coverage), even with no possibility of physical booming, effective burns could be carried out (SL Ross 2003). For application in loose pack ice, the intention is to herd freely-drifting oil slicks to a burnable thickness, then ignite them with a Helitorch. The herders will work in conjunction with the limited containment provided by the ice to allow a longer window of opportunity for burning.

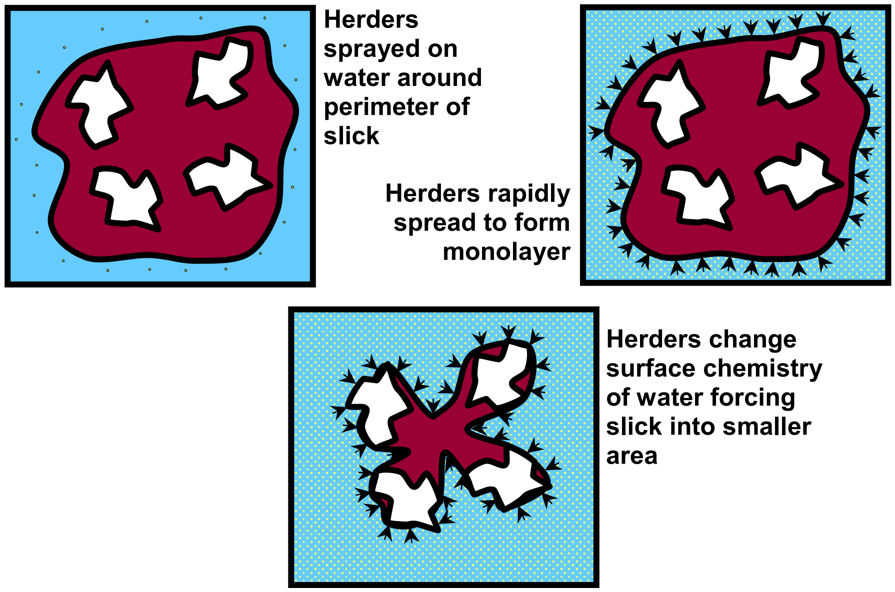

Figure 1. Conceptual drawing depicting the herding process in drift ice.

A comprehensive, multi-year, multi-partner research program to study the use of chemical herding agents to thicken oil slicks in order to ignite and burn the oil *in situ* in loose pack ice has been underway since 2004. The program has included:

9. A very small scale (1 m^2) preliminary assessment of a shoreline-cleaning agent with oil herding properties was carried out to assess its ability to herd oil on cold water and among ice (SL Ross 2004).
10. Small-scale experiments to explore the relative effectiveness of three oil-herding agents in simulated ice conditions; larger scale (10 m^2) quiescent pan experiments to explore scaling effects; small-scale (2 to 6 m^2) wind/wave tank testing to investigate wind and wave effects on herding efficiency; and, small ignition and burn tests (SL Ross 2005).
11. Experiments at the scale of 100 m^2 in the indoor Ice Engineering Research Facility Test Basin at the US Army Cold Regions Research and Engineering Laboratory (CRREL) in November 2005 (SL Ross 2005).
12. Experiments at the scale of 1000 m^2 at Ohmsett in artificial pack ice in February 2006 (SL Ross 2005).
13. A series of 20 burn experiments at the scale of 30 m^2 with herders and crude oil in a specially prepared test basin containing broken sea ice in November 2006 at the Fire Training Grounds in Prudhoe Bay, AK (SL Ross 2005).
14. Field tests of a herder in pack ice in the Barents Sea in 2008 (Buist *et al*. 2010a)
15. Studies on better herding surfactants (Buist *et al*. 2010b).
16. Research on improving other marine spill countermeasures with herders (Buist *et al*. 2010c).

The U.S. Navy cold-water herder formulation (65% Span-20 and 35% 2-ethyl butanol) used in the earlier experiments proved effective in significantly contracting fluid crude and refined oil slicks in brash and slush ice concentrations of up to 70% ice coverage. Slick thicknesses in excess of 3 mm, the minimum required for ignition of weathered oil *in situ*, were routinely achieved. The presence of frazil ice restricted the spreading of the oil and the effectiveness of the herder. Short, choppy waves in the test ice caused a herded slick to break up into small slicklets, although this may be an artifact of the relatively small volumes of oil used in the experiments. Longer, non-breaking waves, simulating a swell in pack ice, did not appear to cause a herded slick to break up, and in fact may have assisted the process by promoting spreading of the herder over water to the slick's edge.

Application of the herder to the water prior to the oil being spilled resulted in thicker slicks than post-spill application. This approach might be used in the event of a chronic spill event in pack ice conditions, such as a blowout or a pipeline leak.

Otherwise unignitable crude oil slicks that were contracted by the USN herder could be ignited and burned *in situ* in both brash and slush ice conditions at air temperatures as low as –17°C. Measured oil removal efficiencies for herded slicks averaged 50% for 7.5-L slicks and 70% for 15-L slicks. The efficiencies measured for the herded slicks were only slightly less than the theoretical maximums achievable for equivalent-sized, mechanically contained slicks on open water. The type of ice (brash or slush) did not significantly affect the burn efficiency.

When ignited, the herded slicks did spread slightly, but once the flames began to die down, the residue was re-herded by the agent remaining on the water surrounding the slick. Generally, it was not possible to reignite re-herded residue. Steeper, cresting waves detracted from the burn efficiency while longer, non-breaking waves did not. The oil removal rate for the slicks was in the range expected for equivalent-sized, mechanically contained slicks on open water.

In the spring of 2008 a field trial was carried out in pack ice east of Svalbard involving the release of 630 L of fresh crude onto water in a large lead. The free-drifting oil was allowed to spread for 15 minutes until it was far too thin to ignite, and then USN herder was applied around the slick periphery. The slick contracted and thickened for 20 minutes at which time the upwind end was ignited. A 9-minute burn ensued that consumed an estimated 90% of the oil (Sørstrøm *et al.* 2010).

In an attempt to further improve the herder technique, in 2008 and 2009, a series of laboratory tests and ice-basin tests were carried out to compare the efficacy of several silicone-based surfactants (superwetters) as potential oil slick herding agents to the results obtained with the USN (Buist *et al*. 2010b). Silicone surfactants are known to be more effective than hydrocarbon-based surfactants in other applications.

The best silicone-based herder of the three tested significantly outperformed the USN herder in most tests with similar conditions. These tests only evaluated the herding capability of the silicone herders – the slicks weren't ignited. Recent experiments in the lab confirmed the ability of the silicone herders to hold a burning slick as well as the USN herder does.

1.1.2 Toxicity Issues

Concern has been expressed regarding the potential toxicity risk of using herding agents in broken ice. These agents should not cause harm to the marine environment because they are of low toxicity and extremely small quantities are used. Although the leading chemical herders are apparently no longer produced, a Nalco product designed as a shoreline cleaner (Corexit

EC9580) exhibits slick herding abilities and is commercially available. The toxicity data in the US EPA's National Contingency Plan indicates that EC9580 is only about half as toxic as approved chemical dispersants and much less toxic than the oil itself. EC9580, and the main surface-active ingredients of successful herders are not soluble in water (they are dispersible) and are not intended to enter the water column, only to float on the surface. When used as directed, the products are applied at very low application rates (5×10^{-2} g/m^2 = 0.05 gal/acre) compared with dispersants (5 gallons/acre = 5 g/m^2) and, if dispersed, would produce concentrations in the water column far below levels of concern (dispersing a 5×10^{-2} g/m^2 layer of herder into the top metre of the water column would produce a concentration of only 0.05 ppm). In addition, studies indicate that silicone surfactants that enter the environment quickly degrade to silicate (sand), water, and carbon dioxide.

1.2 Objective and Goal

The objective of this research is to evaluate the feasibility of using herders to enable *in situ* burning as a rapid-response technique in open water.

More specifically, the goal of the work described here is to conduct experiments at Ohmsett to determine the persistence of the herder monolayer in realistic waves.

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, Bureau of Ocean Energy Management, Regulation, and Enforcement 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, this testing is on weekdays and weekends , and begins at 0700.**

Bureau of Ocean Energy Management, Regulation, and Enforcement (BOEMRE):

- Funds the operation of Ohmsett
- Reviews and approves the Work Order Proposal
- Provides the Work Order to MAR, Inc.
- Funds and administers the participation CRREL in Task 3
- Funds and administers the participation SL Ross in Task 3
- Reviews and approves the Final Report

SL Ross Environmental Research

- Prepares the Test Plan with MAR input
- Designs the experiments
- Provides the herding agent
- Assists with the equipment assembly and operation
- Directs the testing
- Takes overhead digital photos for area analysis

- Analyses the data
- Writes the final report

MAR, Inc:

- Prepares the Test Plan with SL Ross
- Erects the wind screen on either side of the tank
- Provides and operates man lift
- Prepares test fluids and confirms suitability
- Collects test data including oil distribution volumes, initial oil properties, recovered oil volumes and overhead digital video and photography
- Collects background data including oil/water temperatures, and wave data
- Photographs and videotapes the trials
- Cleans and demobilizes the test equipment after the experiments have been completed
- 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
Program Manager Bill Schmidt	Control Tower	Oversight
Test Engineer/Director Paul Meyer	Test Basin	Overall supervision of testing
QA Engineer TBD	Roving	Monitors fluid sampling, data collection and test parameter accuracy.
Chemical Technician Susan Cunneff	Oil Analysis Lab	Handles and analyzes fluid samples.
H&S Specialist TBD	Roving	Monitors personnel safety.
Fluid Transfer Technician TBD	Main Tank Deck	Operates oil transfer system, Operates fill and off-loading pumps
Video Technician TBD	Roving	Operates hand-held video and digital still camera
Rigger/Oil Transfer Technicians TBD	Roving	Deploy boom, transfer oil, collect oil, clean and demobilize equipment
SL Ross Sr. Engineer Ian Buist	Roving	Design and direct tests. Apply herding agent. Take overhead video and still photographs Provide advice on test

2. TEST PROCEDURES

2.1 Preparation

The preparations for the tests include:

- SL Ross supplying 500 mL each of USN herder (65% v/v Sorbitan Monolaurate [Span 20] and 35% 2-ethyl butanol), Silsurf A108 herder and Silsurf A004-D herder
- MAR providing a man lift
- MAR supplying one drum of Endicott crude oil
- MAR supplying one 15-foot diameter plastic pipe containment ring
- Erecting windscreen on either side of the tank
- Installing booms at either end of the tank (as per a dispersant test).
- Obtaining sorbent sweeps to remove herder and sheen from tank after a test is completed
- Positioning, checking and calibrating overhead camera(s) for data collection
- Conducting required safety checks and notifications.
- Conducting several dry run tests to set new wave maker to produce a range of breaking and non-breaking waves and fine tune release and test procedures

2.2 Test Set-up, Instrumentation and Procedures

The amounts of herder used for each test are very small (only about 20 mL). Even if all the herder from one test were dispersed into the water at Ohmsett, it would amount to a concentration of only 0.002 ppm. Visual observation of the spreading of a small amount (ca. 10 mL) of test oil inside several small (1 m^2 area) floating plastic circles, randomly placed on the test area's surface, can be used to confirm that the herder has been removed from the water surface prior to each test.

With the installation of a totally new wave maker system, it will be necessary to spend time on Day 1 determining what setting for the new wave maker produce waves suitable for the tests. A range of breaking and non-breaking waves is desired to determine the survival time of the monolayer of herder in a variety of sea states.

The basic test procedure used for all herder/dispersant effectiveness tests will be as follows.

4. 15 L (4 gallons) of crude is measured into a 5-gallon plastic pail, weighed and placed on the man lift.
5. 20 mL of herding agent is measured into a syringe, weighed and placed on the man lift.

6. Two pre-weighed spray bottles of additional herding agent are placed on the Main Bridge.
7. The Main Bridge is used to clean the entire length and width of the test area with a sorbent sweep.
8. The Main Bridge is positioned at the upwind end of the tank within the boomed area.
9. The containment ring is lowered onto the water surface from under the Main Bridge at the up-drift side of the tank and contact with the water around its entire circumference is visually confirmed.
10. Approximately 20 mL of herding agent is carefully placed on the water surface inside the containment ring from the man lift.
11. The 15 L (4 gallons) of crude is gently poured onto the water surface inside the containment ring from the man lift.
12. The man lift is moved into position for digital overhead video and photographs.
13. The Main Bridge is positioned over the containment ring in preparation for lifting it out of the water.
14. The wave maker is started and the plastic containment ring is lifted and secured under the Main Bridge just before the first wave reaches the oil.
15. The video on the man lift is started. Digital photos of the slick are taken from the man lift as required.
16. The slick is allowed to drift downwind for as long as possible, until it reaches a wall or containment boom.
17. The Main Bridge is moved to keep up with the slick, but not interfere with it.
18. Additional herding agent is added from pre-weighed spray bottles on the Main Bridge as required if the oil begins to spread before it reaches a wall or boom.
19. The waves are left on until the slick reaches a wall or boom in the tank then the wave maker is stopped and the video stopped.
20. The oil pail, herder syringe and herder spray bottles are reweighed.
21. The water spray from the Main Bridge fire monitors is used to sweep the surface oil remaining on the water surface at the end of the test to a common collection area at one corner of the containment boom.
22. The boom is then raised and the oil pushed under the boom out of the containment area.
23. At the end of each test it will be necessary to remove the residual herder from the water surface inside the boomed area. This would be accomplished by running a train of breaking waves down the tank for several minutes and using the Main Bridge fire hoses to disperse the herder and dispersant into the water column.

2.3 Test Matrix and Schedule

15.1.1.1.1.1.1 Test Matrix Variables

3. Herder type (USN and two silicone-based herders);

4. Wave conditions (calm, swell, occasional breaking waves [as per dispersant tests])

Calm or very-low wind conditions are necessary during testing to allow longer test times. A total of nine tests are planned over a seven-day test period. Table 2 gives the proposed matrix for the tests. Testing is to take place May 14 through 20, 2011.

Table 2. Preliminary Matrix of Tests

May	Day	Test	Wave Condition	Herder
14	1	Setup/calibrate wave maker		
15	2	1	Calm	USN
		2	Calm	A108
16	3	3	Calm	A004-D
		4	Breaking	USN
17	4	5	Breaking	A108
		6	Breaking	A004-D
18	5	7	Swell	USN
		8	Swell	A108
19, 20	6,7	9	Swell	A004-D
		Duplicates/Wind Delays	Oseberg Weathered	TBD*

* To Be Determined

At the conclusion of each test any herder remaining on the water surface would be dispersed into the tank with breaking waves and the fire monitors on the Main Bridge. Just prior to each test, the test area would be swept with sorbent to remove any surfactant that has resurfaced.

Final tank clean up would involve sweeping the length of the tank with boom, polishing several times with sorbent sweeps and running breaking waves and fire hoses to disperse any remaining herder or sheen from the surface.

3. DELIVERABLES

3.1 Test Data

Original data logs, computer generated data files, video, digital images 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 test logs.

- Digital and film photographs and digital video.
- Ohmsett laboratory analyses.

3.2 Video Documentation

High-resolution, digital videos 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 or DVD format as deliverable items for SL Ross. Logs will accompany the videos specifying test number, date, time and location on the videotape. Photos, digital and 35 mm, will also be duplicated as deliverables. All original video and photographic 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

Liquid Hydrocarbons:

- Crude oil (MSDS in Appendix – not yet)

Other Products/Chemicals:

- USN herder (65% Sorbitan Monolaurate and 35% 2-ethyl butanol – MSDSs attached)
- Silsurf A108 (MSDS attached)
- Silsurf A004-D (MSDS attached)

According to available product safety information, respiratory protection is not needed, as:

- the evaporation rate of the Endicott crude oil is moderate, resulting in the off-gassing of little VOC, if any;
- sorbitan monolaurate has a low vapor pressure at room temperature and is not identified as a particular inhalation hazard
- 2-ethyl butanol may be harmful if inhaled, but has a low vapor pressure at room temperature and only small amounts will be used in each experiment (about 4.2 mL per test)

All personnel involved in testing will be informed of associated health hazards, as well as the proper personal protective measures required to eliminate exposure to the oil and chemicals, 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 person involved in testing.

4.3 Generic Job Safety Analysis

The following table lists basic or generic tasks necessary for the “Herders to Improve Operational Efficiency of Dispersant Operations” Tests at Ohmsett. Hazards associated with the tasks are listed with preventive measures to be followed by affected personnel.

Table 3. Task Hazard Prevention

TASK	HAZARDS	PREVENTION/CONTROL
1) Materials handling, general set-up	a) Lifting material(s) (muscle strains, back injuries) b) Forklift operations (objects striking) c) Jib crane(s) operations (objects striking) d) Mobile crane (contractor personnel, objects striking) e) Hand/power tools (muscle strains, pinch points, electrocution)	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) Follow acceptable safe practices for operators. c) Do not stand under raised loads. Do not exceed capacity of jib crane. Use one signal man. d) Only qualified crane operator and signal man will control lift operations. Do not stand under raised loads. 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 or bridge (falls) b) Cable handling (pinch points) c) Positioning bridges (objects striking) d) Positioning boom equipment. Mobile crane operations (objects striking)	a) Personnel on work boat MUST wear PFD's. Evenly distribute weight and do not overload. Life preservers are in place as needed. b) Wear hand protection during rigging. c) Have appropriate lines of continual communication. 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) Addition of Herding Agent	a) Exposure to herder formulation b) Working on bridges c) Deployment and general operations (testing)	a) 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) Sorbent boom sweeping.	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.
9) Cleanup of equipment	a) Disassembly of rigging from work boat/ bridges (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 a 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 Ohmsett.

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
- Insulated coveralls (Temperatures will be 0°C to –15°C)
- Warm hat
- Oil resistant gloves (neoprene, nitrile)
- Eye protection (safety glasses, goggles)
- Safety shoes
- Personal flotation devices (for workboat operations) mandatory
- Life rings
- Splash suits, for tank 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 – 732 - 615 - 2100
- Riverview Medical Center 9 – 732 - 741 - 2700
- Bayshore Hospital 9 – 732 - 739 - 5900
- Poison Control Center 9 - 1 - (800) 962-1253

5. Ohmsett Testing of Herder Persistence in Waves Quality Assurance

5.1 Introduction

Ohmsett Testing of Herder Persistence in Waves Test Quality is the active application of The Ohmsett "General Quality Procedures and Documentation Plan Manual" and the "Ohmsett Testing of Herder Persistence in Waves Test Quality Checklist."

The Quality Checklist has a list of those items in the Ohmsett Testing of Herder Persistence in Waves 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

Ohmsett Testing of Herder Persistence in Waves Test Quality Checklist is implemented as follows:

Ohmsett Testing of Herder Persistence in Waves 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 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 ensure 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 ensure 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.

5.7 Significant Occurrences/Variations

This part of the Ohmsett Testing of Herder Persistence in Waves 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 Test Director.

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 Section 3.

6. SCHEDULE

The following schedule is planned for the Ohmsett Testing of Herder Persistence in Waves Tests.

DATE	EVENT
May 5, 2011	Submit Draft Test Plan
May 14 to 20, 2011.	Tests at Ohmsett
May 31, 2011	Deliver Raw and Processed Data, Observations and Photo Video Documentation to SL Ross
December 31, 2011	Submission of Draft Final Report

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Appendix A – MSDS Sheets

Material Safety Data Sheet

2-ETHYL-1-BUTANOL, 98%

Section 1 - Chemical Product and Company Identification

MSDS Name:	2-ETHYL-1-BUTANOL, 98%
Catalog Numbers:	**11817-0000, 11817-1000**
Synonyms:	2-Ethylbutyl alcohol
Company Identification:	**Acros Organics BVBA** Janssen Pharmaceuticalaan 3a 2440 Geel, Belgium
Company Identification: (USA)	**Acros Organics** **One Reagent Lane** **Fair Lawn, NJ 07410**
For information in the US, call:	**800-ACROS-01**
For information in Europe, call:	**+32 14 57 52 11**
Emergency Number, Europe:	**+32 14 57 52 99**
Emergency Number US:	**201-796-7100**
CHEMTREC Phone Number, US:	**800-424-9300**
CHEMTREC Phone Number, Europe:	**703-527-3887**

Section 2 - Composition, Information on Ingredients

CAS#	Chemical Name:	%	EINECS#
97-95-0	2-ETHYL-1-BUTANOL	98%	202-621-4

Hazard Symbols: XN

Risk Phrases: 21/22

Section 3 - Hazards Identification

EMERGENCY OVERVIEW

Harmful in contact with skin and if swallowed.

Potential Health Effects

Eye:	May cause eye irritation.
Skin:	May cause skin irritation. Harmful if absorbed through the skin.
Ingestion:	Harmful if swallowed. May cause irritation of the digestive tract.
Inhalation:	May cause respiratory tract irritation. May be harmful if inhaled.
Chronic:	Not available.

Section 4 - First Aid Measures

Eyes:	Flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical aid immediately.
Skin:	Get medical aid. Flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Remove contaminated clothing and shoes.
Ingestion:	If victim is conscious and alert, give 2-4 cupfuls of milk or water. Get medical aid immediately. Do NOT induce vomiting. If conscious and alert, rinse mouth and drink 2-4 cupfuls of milk or water.

Inhalation: Get medical aid immediately. Remove from exposure and move to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.

Notes to Physician:

Section 5 - Fire Fighting Measures

General Information: As in any fire, wear a self-contained breathing apparatus in pressure-**demand, MSHA/NIOSH (approved or equivalent), and** full protective gear. Flammable liquid and vapor.

Extinguishing Media: In case of fire, use water, dry chemical, chemical foam, or alcohol-resistant foam. Use agent most appropriate to extinguish fire.

Section 6 - Accidental Release Measures

General Information: Use proper personal protective equipment as indicated in Section 8.

Spills/Leaks: Absorb spill with inert material (e.g. vermiculite, sand or earth), then place in suitable container. Clean up spills immediately, observing precautions in the Protective Equipment section. Remove all sources of ignition. Use a spark-**proof tool.**

Section 7 - Handling and Storage

Handling: **Use spark**-proof tools and explosion proof equipment. Empty containers retain product residue, (liquid and/or vapor), and can be **dangerous. Keep away from heat, sparks and flame.**

Storage: Keep away from heat, sparks, and flame. Keep away from sources of ignition.

Section 8 - Exposure Controls, Personal Protection

Engineering Controls:

Use adequate general or local explosion-proof ventilation to keep airborne levels to acceptable levels.

Exposure Limits

CAS# 97-95-0:

Personal Protective Equipment

Eyes: Wear chemical splash goggles.

Skin: **Wear appropriate protective gloves to prevent skin exposure.**

Clothing: Wear appropriate protective clothing to minimize contact with skin.

Respirators: **A respiratory protection program that meets OSHA's 29 CFR 1910.134 and ANSI Z88.2 requirements or European Standard EN 149 must be followed whenever workplace conditions warrant a respirator's use. Wear a NIOSH/MSHA or European Standard EN** 149 approved full-facepiece airline respirator in the positive pressure mode with emergency escape provisions.

Section 9 - Physical and Chemical Properties

Physical State: Clear liquid

Color: colorless - light yellow

Odor: None reported.

pH: Not available.

Vapor Pressure: 1.7 hPa @ 20 C

Viscosity: **7.6 MPA 20.00 deg C**

Boiling Point: **146 deg C @ 760.00mm Hg (294.80°F)**

Freezing/Melting Point: **0 deg C (32.00°F)**

Autoignition Temperature: **315 deg C (599.00 deg F)**

Flash Point: **57 deg C (134.60 deg F)**

Explosion Limits: Lower: Not available.

Explosion Limits: Upper: Not available.

Decomposition Temperature:

Solubility in water: 10 g/l (20°C)

Specific Gravity/Density: **.8300g/cm3**

Molecular Formula: $C_6H_{14}O$

Molecular Weight: 102.18

Section 10 - Stability and Reactivity

Chemical Stability: **Stable under normal temperatures and pressures.**

Conditions to Avoid:	Incompatible materials, ignition sources.
Incompatibilities with Other Materials	Strong oxidizing agents, strong acids.
Hazardous Decomposition Products	Carbon monoxide, irritating and toxic fumes and gases, carbon dioxide.
Hazardous Polymerization	**Has not been reported.**

Section 11 - Toxicological Information

RTECS#:	CAS# 97-95-0: EL3850000
LD50/LC50:	**CAS# 97-95-0: Oral, rabbit: LD50 = 1200 mg/kg;** **Oral, rat: LD50 = 1850 mg/kg;** **Skin, rabbit: LD50 = 1260 uL/kg;**
Carcinogenicity:	2-ETHYL-1-BUTANOL - **Not listed as a carcinogen by ACGIH, IARC, or NTP.**
Other:	See actual entry in RTECS for complete information.

Section 12 - Ecological Information

Not available.

Section 13 - Disposal Considerations

Dispose of in a manner consistent with federal, state, and local regulations.

Section 14 - Transport Information

	IATA	IMO	RID/ADR
Shipping Name:	2-ETHYLBUTANOL	2-ETHYLBUTANOL	2-ETHYLBUTANOL
Hazard Class:	3	3	3
UN Number:	2275	2275	2275
Packing Group:	III	III	III

Section 15 - Regulatory Information

European/International Regulations

European Labeling in Accordance with EC Directives

Hazard Symbols: XN

Risk Phrases:

R 21/22 Harmful in contact with skin and if swallowed.

Safety Phrases:

WGK (Water Danger/Protection)

CAS# 97-95-0: 1

Canada

CAS# 97-95-0 is listed on Canada's DSL List

US Federal

TSCA

CAS# 97-95-0 is listed on the TSCA Inventory.

Section 16 - Other Information

MSDS Creation Date: 7/16/1996

Revision #0 Date Original.

The information above is believed to be accurate and represents the best information currently available to us. However, we make no warranty of merchantibility or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall Fisher liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential, or exemplary damages howsoever arising, even if Fisher has been advised of the possibility of such damages.

BASF Corporation

Material Safety Data Sheet

Original Date: 03/09/1998
Revision Date: 09/13/2002

BASF CORPORATION
PERFORMANCE CHEMICALS
3000 CONTINENTAL DRIVE NORTH
MOUNT OLIVE, NJ 07828
(800) 832-HELP

EMERGENCY TELEPHONE: (800) 424-9300 CHEMTREC
(800) 832-HELP (BASF Hotline)
BOTH NUMBERS ARE AVAILABLE DAYS, NIGHTS, WEEKENDS, & HOLIDAYS.

SECTION 1 - PRODUCT INFORMATION

S-MAZ® 20 M1 SORBITAN MONOLAURATE
Product ID: NCS 558695
Common Chemical Name:
SORBITAN MONOLAURATE
Synonyms:
NONE
Molecular Formula:

Chemical Family: Not Applicable
Molecular Wt.: NOT APPLICABLE

SECTION 2 - INGREDIENTS

Chemical Name:	CAS	Amount
SORBITAN, MONODODECANOATE	1338-39-2	~ 100.0 %
PEL/TLV NOT ESTABLISHED		
Vitamine E Alcohol	10191-41-0	~ 100.0 PPM
PEL/TLV NOT ESTABLISHED		

SECTION 3 - PHYSICAL PROPERTIES

Color: Dark Brown
Form/Appearance: Liquid
Odor: Ester
Odor Intensity: Mild

	Typical	Low/High	U.O.M.			
Specific Gravity:	1.05			@	25	DEG C

pH: NOT AVAILABLE

	Typical	Low/High	Deg.	@	Pressure
Boiling Pt:	> 300		F	1	ATMOSPHERES

Freezing Pt: NOT AVAILABLE
Decomp. Tmp: NOT AVAILABLE
Solubility in Water Description: Dispersible
Vapor Pressure: < 1 MM HG X 25 DEG. C XX
Vapor Density (Air = 1): >1
Volatile by Vol. %: < 1

SECTION 4 - FIRE AND EXPLOSION DATA

	Typical	Low/High	Deg.	Method
Flash Point:	> 200		F	PENSKY-MARTENS CLOSED C
Autoignition:	NOT AVAILABLE			

Extinguishing Media:
Use water, dry extinguishing media, carbon dioxide (CO_2) or foam.

Fire Fighting Procedures:
Firefighters should be equipped with self-contained breathing apparatus and turn out gear.

Unusual Hazards:
There are no known unusual fire or explosion hazards.

SECTION 5 - HEALTH EFFECTS

Routes of entry for solids and liquids include eye and skin contact, ingestion and inhalation. Routes of entry for gases include inhalation and eye contact. Skin contact may be a route of entry for liquified gases.

Acute Overexposure Effects:
Contact with the eyes and skin may result in irritation. Inhalation may result in respiratory irritation. Ingestion may result in gastric disturbances.

Chronic Overexposure Effects:
There are no known chronic effects associated with this material.

First Aid Procedures - Skin:
Wash affected areas with soap and water. Remove and launder contaminated clothing before reuse. If irritation develops, get medical attention.

First Aid Procedures - Eyes:
Immediately rinse eyes with running water for 15 minutes. If irritation develops, get medical attention.

First Aid Procedures - Ingestion:
If swallowed, dilute with water and immediately induce vomiting. Never give fluids or induce vomiting if the victim is unconscious or having convulsions. Get immediate medical attention.

First Aid Procedures - Inhalation:
Move to fresh air. Aid in breathing, if necessary, and get immediate medical attention.

First Aid Procedures - Notes to Physicians:
None known.

First Aid Procedures - Aggravated Medical Conditions:
No data is available which addresses medical conditions that are generally recognized as being aggravated by exposure to this product. Please refer to the effects of overexposure section for effects (if any) observed in animals.

First Aid Procedures - Special Precautions:
None

SECTION 6 - REACTIVITY DATA

Stability Data:
Stable

SECTION 6 - REACTIVITY DATA (cont)

Incompatability:
Strong oxidizers.
Conditions/Hazards to Avoid:
No data available.
Hazardous Decomposition/Polymerization:
Hazardous Decomposition Products: No Data Available.
Corrosive Properties:
Not corrosive.
Oxidizer Properties:
Not an oxidizer
Other Reactivity Data:
None known.

SECTION 7 - PERSONAL PROTECTION

Clothing:
Gloves, coveralls, apron, boots as necessary to minimize contact.
Eyes:
Chemical goggles; also wear a face shield if splashing hazard exists.
Respiration:
Approved organic vapor mist respirator as necessary.
Ventilation:
Use local exhaust to control vapors/mists.
Explosion Proofing:
None required.

SECTION 8 - SPILL-LEAK/ENVIRONMENTAL

General:
Spills should be contained, solidifed, and placed in suitable containers for disposal in a licensed facility. This material is not regulated by RCRA or CERCLA ("Superfund"). Wear appropriate respiratory protection and protective clothing and provide adequate ventilation during clean-up.
Waste Disposal:
Incinerate in a licensed facility. Do not discharge into waterways or sewer systems.
Container Disposal:
Dispose of in a licensed facility. Recommend crushing or other means to prevent unauthorized reuse.

SECTION 9 - STORAGE AND HANDLING

General:
Keep containers closed.
Store in well ventilated areas below 120 F.

SECTION 10 - REGULATORY INFORMATION

TSCA Inventory Status
Listed on Inventory: YES

RCRA Haz. Waste No .: NA
CERCLA: NO Reportable Qty.: (If YES)

SECTION 10 - REGULATORY INFORMATION (cont)

State Regulatory Information: (By Component) NJ/PA/MA RTK

CAS: 1338-39-2 NO
NAME: SORBITAN, MONODODECANOATE
CAS: 10191-41-0 NO
NAME: Vitamine E Alcohol

Hazard Ratings:

	Health:	Fire:	Reactivity:	Special:
HMIS	1	1	0	NA
NFPA	1	1	0	NA

This product is not hazardous according to the OSHA Hazard Communication Standard.

SECTION 11 - TRANSPORTATION INFORMATION

DOT Proper Shipping Name:
N/A
DOT Technical Name:
N/A
DOT Primary Hazard Class:
N/A
DOT Secondary Hazard Class:
N/A
DOT Label Required:
N/A
DOT Placard Required:
N/A
DOT Poison Constituent:
N/A
BASF Commodity Codes: NA NA UN/NA Code: E/R Guide: N/A
Bill of Lading Description:
NOT REGULATED BY THE DEPARTMENT OF TRANSPORTATION

"IMPORTANT: WHILE THE DESCRIPTIONS, DESIGNS, DATA AND INFORMATION CONTAINED HEREIN ARE PRESENTED IN GOOD FAITH AND BELIEVED TO BE ACCURATE, IT IS PROVIDED FOR YOUR GUIDANCE ONLY. BECAUSE MANY FACTORS MAY AFFECT PROCESSING OR APPLICATION/USE, WE RECOMMEND THAT YOU MAKE TESTS TO DETERMINE THE SUITABILITY OF A PRODUCT FOR YOUR PARTICULAR PURPOSE PRIOR TO USE. NO WARRANTIES OF ANY KIND, EITHER EXPRESSED OR IMPLIED, INCLUDING WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE, ARE MADE REGARDING PRODUCTS DESCRIBED OR DESIGNS, DATA OR INFORMATION SET FORTH, OR THAT THE PRODUCTS, DESIGNS, DATA OR INFORMATION MAY BE USED WITHOUT INFRINGING THE INTELLECTUAL PROPERTY RIGHTS OF OTHERS. IN NO CASE SHALL THE DESCRIPTIONS, INFORMATION, DATA OR DESIGNS PROVIDED BE CONSIDERED A PART OF OUR TERMS AND CONDITIONS OF SALE. FURTHER, YOU EXPRESSLY UNDERSTAND AND AGREE THAT THE DESCRIPTIONS, DESIGNS, DATA, AND INFORMATION FURNISHED BY BASF HEREUNDER ARE GIVEN GRATIS AND BASF ASSUMES NO OBLIGATION OR LIABILITY FOR THE DESCRIPTION, DESIGNS, DATA AND INFORMATION GIVEN OR RESULTS OBTAINED, ALL SUCH BEING GIVEN AND ACCEPTED AT YOUR RISK".

END OF DATA SHEET

SILTECH CORP.

Material Safety Data Sheet

Silsurf A004-D
MSDS NR: 2001.4

Revised: June 14, 2007

SECTION 1. CHEMICAL PRODUCT/COMPANY IDENTIFICATION

Material Identification: Silsurf A004-D
Chemical Name: Polydimethylsiloxane Copolymer
CTFA Name: None

Company Identification: Siltech Corp.
225 Wicksteed Avenue
Toronto, Ont.
M4H 1G5
(416) 424-4567

SECTION 2. Composition/Information On Hazardous Ingredients

CAS NR	Wt %	Components	Exposure Limits
None			

THIS MATERIAL IS FOR INDUSTRIAL USE ONLY, NOT FOR FOOD, MEDICAL OR DRUG USE.

SECTION 3. HAZARDS IDENTIFICATION

There are no potential health effects expected from handling this material. Good manufacturing practices are always recommended when handling any chemical.
There are no significant laboratory data to suggest any hazard to humans.

Eyes: Direct contact may cause temporary redness and discomfort.
Skin: No significant irritation expected from a single short-term irritation.
Inhalation: No significant effects expected from a single short-term exposure.
Oral: Low ingestion hazard in normal use.

Repeated exposure effects:
Skin: Repeated or prolonged exposure may cause irritation.
Inhalation: No known applicable information.
Oral: No known applicable information.

Comments: None.

SECTION 4. FIRST AID MEASURES

Eyes: Immediately flush with water for 15 minutes. Obtain medical attention if irritation occurs.
Skin: Remove contaminated clothing and wash with soap and water. No first aid should be needed.
Inhalation: Short-term harmful health effects are not expected from vapour generated at ambient temperatures. If first aid is required move victim to fresh air.
Oral: No first aid should be needed. If discomfort occurs, obtain medical attention.

Comments: Treat symptomatically.

SECTION 5. FIRE FIGHTING MEASURES

Flash Point : > 100 C by Pensky Martens closed cup.
Autoignition Temperature: Not determined.
Flammability Limits in Air: Not determined.
Extinguishing Media: Carbon dioxide ($C0_2$) water spray. Dry chemical foam water can be used to cool fire exposed containers.
Fire Fighting Procedures: Self-contained breathing apparatus and protective clothing should be worn in fighting fires involving chemicals.
Hazardous Decomposition Products: Silicon dioxide. Carbon oxides and traces of incompletely burned carbon compounds.

SILTECH CORP.

Material Safety Data Sheet

Silsurf A108
MSDS NR: 2004

Revised: April 01, 2007

SECTION 1. CHEMICAL PRODUCT/COMPANY IDENTIFICATION

Material Identification: Silsurf A108
Chemical Name: Polydimethylsiloxane Copolymer
INCI Name: PEG-8 Dimethicone

Company Identification: Siltech Corp.
225 Wicksteed Avenue
Toronto, Ont.
M4H 1G5
(416)424-4567

SECTION 2. Composition/Information On Hazardous Ingredients

CAS NR	Wt %	Components	Exposure Limits
None			

THIS MATERIAL IS FOR INDUSTRIAL USE ONLY, NOT FOR FOOD, MEDICAL OR DRUG USE.

SECTION 3. HAZARDS IDENTIFICATION

There are no potential health effects expected from handling this material. Good manufacturing practices are always recommended when handling any chemical.
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Inhalation: No significant effects expected from a single short-term exposure.
Oral: Low ingestion hazard in normal use.

Repeated exposure effects:
Skin: Repeated or prolonged exposure may cause irritation.
Inhalation: No known applicable information.
Oral: No known applicable information.

Comments: None.

SECTION 4. FIRST AID MEASURES

Eyes: Immediately flush with water for 15 minutes. Obtain medical attention if irritation occurs.
Skin: Remove contaminated clothing and wash with soap and water. No first aid should be needed.
Inhalation: Short term harmful health effects are not expected from vapour generated at ambient temperatures. If first aid is required move victim to fresh air.
Oral: No first aid should be needed. If discomfort occurs, obtain medical attention.

Comments: Treat symptomatically.

SECTION 5. FIRE FIGHTING MEASURES

Flash Point: > 100 C by Pensky Martens closed cup.
Autoignition Temperature: Not determined.
Flammability Limits in Air: Not determined.
Extinguishing Media: Carbon dioxide ($C0_2$) water spray. Dry chemical foam water can be used to cool fire exposed containers.
Fire Fighting Procedures: Self-contained breathing apparatus and protective clothing should be worn in fighting fires involving chemicals.
Hazardous Decomposition Products: Silicon dioxide. Carbon oxides and traces of incompletely burned carbon compounds.

SECTION 6. ACCIDENTAL RELEASE MEASURES

Containment/Clean up: Sections 13 and 15 of this MSDS provide information regarding certain Federal and local requirements. Collect for disposal. Clean up remaining materials from spill with suitable absorbent. For large spills provide diking or other appropriate containment to keep material from spreading. If diked material can be pumped, store recovered material in appropriate container. Clean area as appropriate since some silicone material, even in small quantities, may present a slip hazard. Final cleaning may require steam, solvents or detergents. Observe all personal protection equipment recommendations described in Sections 5 and 8 of this MSDS. Observe all Federal, Provincial and local regulations that may apply to the clean up of this material.

SECTION 7. HANDLING AND STORAGE

Handling (Personnel): Safety Glasses and PVC Gloves.
Storage: Keep container tightly closed. Product is "non-hazardous".

SECTION 8. EXPOSURE CONTROLS AND PERSONAL PROTECTION

Engineering Controls: Local Exhaust: None should be needed.
General Ventilation: Recommended.

Personal Protective Equipment for Routine Handling
Eyes: Use proper protection - safety glasses as a minimum.
Skin: Washing at mealtime and end of shift is adequate.
Suitable Gloves: No special protection is needed.
Inhalation: No respiratory protection should be needed.
Suitable Respirator: None should be needed.

Personal Protective Equipment for Spills
Eyes: Use proper protection - safety glasses as a minimum.
Skin: Washing at mealtime and end of shift is adequate.
Inhalation/Suitable Respirator: No respiratory protection should be needed.
Precautionary Measures: Avoid eye contact.

Note: These precautions are for room temperature handling. Use at elevated temperatures or aerosol spray applications may require added precautions.

SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

Physical form:	Clear Liquid	**Viscosity:**	Not determined
Colour:	4 Max	**Melting Point:**	Not applicable
Odour:	Mild	**Boiling point:**	>100 C @ 760 mmHg
Specific Gravity @ 25C:	1.02 gm/ml	**Vapour Pressure @ 25C:**	Not determined
Solubility in Water:	Soluble	**pH:**	Not determined

Section 10. Stability and Reactivity

Chemical Stability: Stable
Hazardous Polymerization: Will not occur.
Conditions to Avoid: None known.
Materials to Avoid: None known.

Section 11. Toxicological Information

Optional Section - Complete information not yet available.

Section 12. Ecological Information

Optional Section - Complete information not yet available.

Section 13. Disposal Considerations

Landfill and/or incineration where permitted under Federal, Provincial or local laws. Observe all local, Provincial, and Federal waste management regulations.

Section 14. Transportation Information

Shipping Name: Not applicable
Technical Name: Not applicable
Primary Class: Not applicable
Subsidiary Risk: Not assessed
Product Identification Number: Not applicable
Packing Group: Not applicable

Section 15. Regulatory Information

This product has been classified in accordance with the hazard criteria of the CPR, and this MSDS contains all the information required by the CPR.

WHMIS Classification: This product is not subject to WHMIS regulations

Section 16. Other Information

The data in this MSDS relates only to the specific material designated herein and does not relate to use in combination with any other material or in any process. This data is offered in good faith as typical values and not as product specifications. No warranty, either expressed or implied, is hereby made. The recommended industrial hygiene and safe handling procedures are believed to be generally applicable. However, each user should review these recommendations in the specific context of the intended use and determine whether they are appropriate.

Responsibility for MSDS:	David Coelho
Address:	Siltech Corp. 225 Wicksteed Avenue Toronto, Ontario, M4H 1G5
Telephone:	(416) 424-4567

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Appendix 4 – Ohmsett Test Data

Ohmsett Test Data

Test #	Date	Herder	Conditions: Waves	Air T (°C)	Water T (°C)	Wind Speed (mph)	Wind Dir (°)	Oil: Type	Oil T (°C)	Pail Gross Weight (lb)	Pail Tare Wt (lb)	Oil Net Wt (lb)	Oil Volume (L)
1	15/05/2011	USN	Calm	17.8	15.4	3.8	190	Endicott	20	31.19	2.97	28.22	14.4
2	15/05/2011	A108	Calm	17.3	15.4	5.1	143	Endicott	16	31.06	2.9	28.15	14.4
3	15/05/2011	A004-D	Calm	12.8	20	21	87	Endicott	20	31.22	3	28.22	14.4
4	16/05/2011	USN	Breaking	13.3	15.4	9.8	75	Endicott	16.5	31.22	3.03	28.19	14.4
5	18/05/2011	A108	Breaking	15.4	18.3	7.5	92	Endicott	17.5	30.94	3	27.94	14.3
6	18/05/2011	A004-D	Breaking	17.9	18.9	7.3	128	Endicott	18	31.08	2.97	28.11	14.4
7	19/05/2011	USN	Swell	17.2	18.9	2.7	72	Endicott	17.8	31.18	2.98	28.2	14.4
8	19/05/2011	A108	Swell	16.1	18.9	5.7	83	Endicott	17.8	31.16	3.05	28.11	14.4
9	19/05/2011	A004-D	Swell	16.1	15.4	7	82	Endicott	18.1	31.95	3	28.95	14.8
10(REP)	20/05/2011	A108	Swell	15.6	15.4	2.2	103	Endicott	18	31.75	2.94	28.79	14.7
11	20/05/2011	None	Swell/Breaking	17.8	15.4	3.5	86	Endicott	18.4	31.41	2.59	28.68	14.7

Test #	Herder: Syringe Gross Wt (g)	Syringe Tare Wt (g)	Initial Herder (mL)	Spray #1 Gross Wt (g)	Spray #1 Tare Wt (g)	Spray #2 Gross Wt (g)	Spray #2 Tare Wt (g)	Additional Herder Volume (mL)	Total Herder (mL)
1	15.06	14.62	15.0	137.92	130.91	136.71	130.9	29.5	44.5
2	15.50	14.59	20.6	159.36	129.58	163.66	155.79	16.3	36.9
3	15.1	14.59	20.1	113.57	111.59	129	102.38	26.2	46.3
4	15.39	14.66	19.1	122.02	82.71	123.86	82.46	80.9	100.1
5	15.6	14.56	20.5	148.27	137.55	159.37	105.47	41.7	64.3
6	15.79	14.54	18.9	104.67	92.63	113.34	81.39	43.1	63.9
7	15.59	14.64	18.7	109.43	71.36	135.81	107.84	47.5	67.1
8	15.5	14.64	20.5	117.67	111.48	113.37	102.42	44.3	64.7
9	14.38	14.71	19.3	97.36	59.06	82.49	71.65	47.2	66.5
10(REP)	15.7	14.59	20.7	111.67	100.72	111.88	88.88	42.6	63.3
11	0	0	0.0	0	0	0	0	0.0	0.0

Tank surface water salinity = 25.1 ppt @ 16.2°C on 18/05/2011

Additional raw test data may be found in the report submitted by MAR, Inc. on the tests entitled "Task Order 493 – Ohmsett Testing of Herder Persistence in Waves, May 14 – 20, 2011"