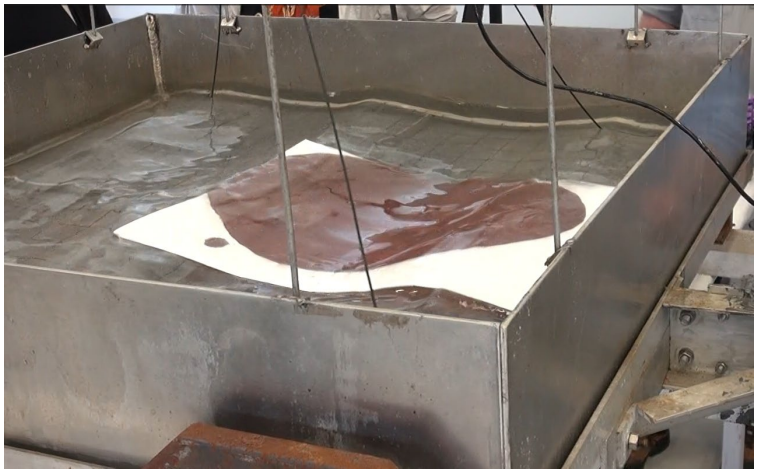
Marine Minerals Administration
Oil Spill Preparedness Division
Field Scale Test Protocol for Type I Sorbents

Final Report

August 2026



(Photo: SL Ross, 2026)

David Cooper and James McCourt

US Department of the Interior
Marine Minerals Administration
Oil Spill Preparedness Division

Field Scale Test Protocol for Type I Sorbents

Final Report

OSRR # 1152

August 2026

Authors:
David Cooper
S.L. Ross Environmental Research Ltd.

and

James McCourt
S.L. Ross Environmental Research Ltd.

Prepared under 140E0124P0027
By
S.L. Ross Environmental Research Ltd.
38 Auriga Drive, Suite #200, Ottawa, ON
K2E 8A5, Canada

US Department of the Interior
Marine Minerals Administration
Oil Spill Preparedness Division

DISCLAIMER

Contracts:

Study concept, oversight, and funding were provided by the US Department of the Interior (DOI), Marine Minerals Administration (MMA), Oil Spill Preparedness Division (OSPD), Sterling, VA, under Contract Number 140E0124P0027. This report has been technically reviewed by MMA, and it has been approved for publication. The views and conclusions contained in this document are those of the authors and should not be interpreted as representing the opinions or policies of the US Government, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

REPORT AVAILABILITY

The PDF file for this report is available through the following sources. Click on the URL and enter the appropriate search term to locate the PDF:

Document Source	Search Term	URL
Marine Minerals Administration (MMA)	Project Number – 1152	https://www.bsee.gov/what-we-do/research/oil-spill-preparedness/oil-spill-response-research
U.S. Department of the Interior Library	Field Scale Test Protocol for Type I Sorbents	https://library.doi.gov/uhtbin/cgisirsi/?ps=8L0mpW5uPV/SIRSI/X/60/495/X
National Technical Reports Library	Field Scale Test Protocol for Type I Sorbents	https://ntrl.ntis.gov/NTRL/

Sources: a) BSEE (2019), b) DOI [2021], c) National Technical Information Service (2021)

CITATION

Cooper D, McCourt J (SL Ross Environmental Research, Ottawa ON). 2026. Field scale test protocol for type 1 sorbents. Sterling (VA): U.S. Department of Interior, Marine Minerals Administration. Report No.: 1152. Contract No. 140E0124P0027. https://doi.org/10.70149/VYFIGG6MZNLU

ABOUT THE COVER

Image by David Cooper of S.L. Ross. Image depicts testing full-size sorbent pad in wave conditions with dyed test liquid for easier visibility.

ACKNOWLEDGEMENTS

The authors acknowledge that assistance from a variety of sources has helped in the enhancement of the new protocol and would specifically like to thank the team at Ohmsett for their support and input into its progress. We would also like to thank Dr. Marie DeLorenzo and Emily Pisarski of NOAA for their input in the development of the new protocol.

MMA OSPD PUBLIC POSTING APPROVAL SHEET

1. **Report Date: 31-08-2026**

2. **REPORT TYPE: Final Report**

3. **START DATE: 23-09-2024**

4. **END DATE: 31-08-2026**

5. **TITLE:**

Field Scale Test Protocol for Type 1 Sorbents

6. **CONTRACT/IAA NUMBER:**

140E0124P0027

7. **OIL SPILL RESPONSE RESEARCH PROJECT NUMBER:**

1152

8. **PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES):**

SL Ross Environmental Research
38 Auriga Drive Suite 200
Ottawa, Ontario
K2E 8A5
Canada

9. **AUTHOR(S):**

David Cooper and James McCourt

10. **DISTRIBTION/AVAILABILITY STATEMENT (INDICATE PUBLIC AVAILABILIY OR DISTRIBTION LIMIATIONS OF THE REPORT: (e.g., PROPRIETARY) Public Availability**

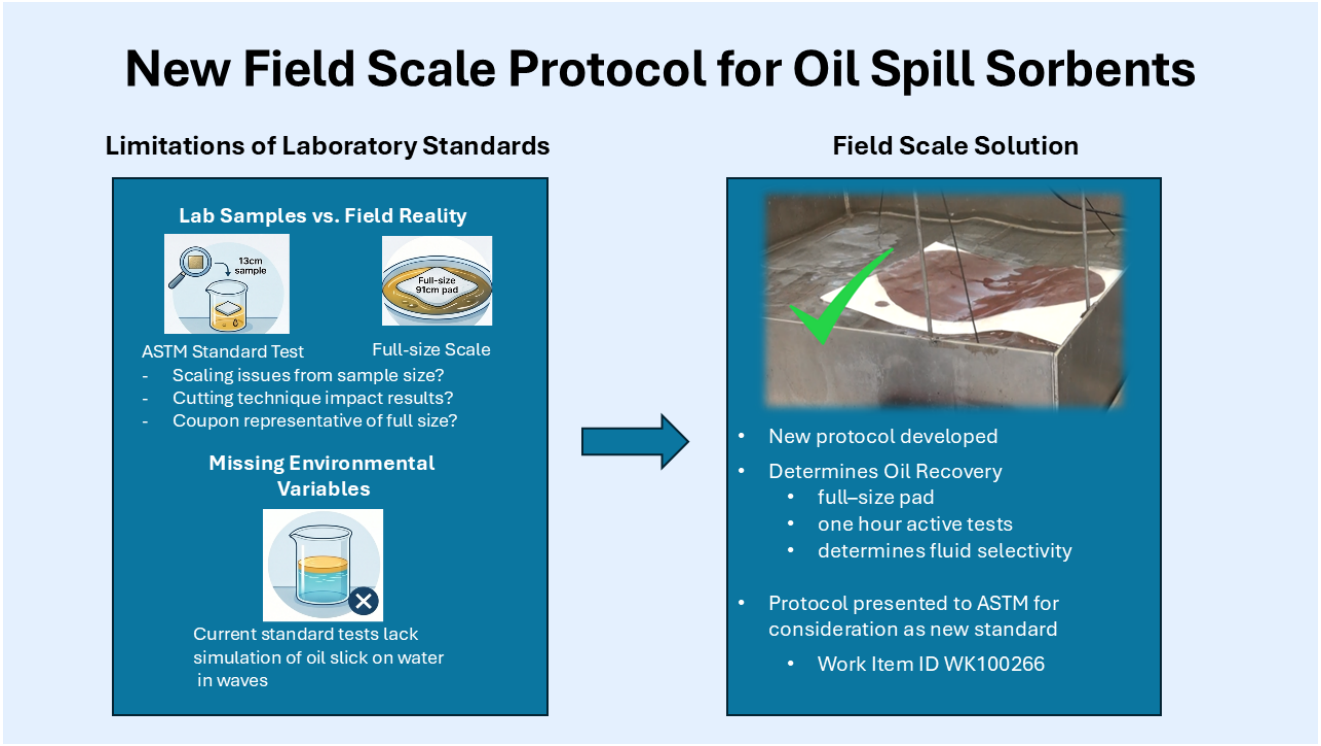
11. **SUPPLEMENTARY NOTES:**

12. **ABSTRACT: (BRIEF, 200 WORD, FACTUAL SUMMARY OF THE MOST SIGNIFICANT INFORMATION)**

The Field Scale Test Protocol for Type I Sorbents project sought to establish a new standard protocol for the testing of absorbent properties of sorbent materials in relevant environments and conditions. This initiative was built upon previously established laboratory protocols used to test sorbent materials. ASTM Protocol F726 (2024) is a current standard test for Type I sorbents, but the protocol has raised concerns of issues of scaling, due to the use of small sorbent sample coupons for testing, rather than full manufactured sorbent sheets. The initial stage of the project focused on a complete evaluation of historic and established protocols related to the testing of sorbents. This literature review allowed for an evaluation of the unique and valuable techniques that could be incorporated into the final protocol. After the initial research stage, a team of experts from a variety of oil spill related organizations were convened to provide input on a draft protocol, done through industry meetings, workshops and private consultation. A demonstration of the draft protocol was conducted at Ohmsett with a team of researchers to observe the protocol in action and identify potential points of error in the current protocol. The demonstration provided researchers with a chance to observe equipment used at Ohmsett to represent dynamic ocean conditions in a laboratory setting, and to observe data collection techniques. The feedback from the Ohmsett demonstration was used to edit the draft protocol. An updated draft protocol was submitted to ASTM International F20.22 subcommittee for review and consideration under Work Item ID WK100266.

13. KEY WORDS/PHRASES: **Sorbent, Crude Oil, ASTM, Ohmsett**	
14. CONTRACTING OFFICER REPRESENTATIVE: **Catherine Robertson**	**15. PHONE NUMBER/EMAIL ADDRESS:** **703-787-1479, catherine_robertson@ios.doi.gov**
16. APPROVER: (Branch Manager) **Karen Stone**	**17. PHONE NUMBER/EMAIL ADDRESS:** **703-787-1810, karen.stone@bsee.gov**

GRAPHICAL ABSTRACT



EXECUTIVE SUMMARY

Sorbents are a response tool that is used to soak up spilled crude oil. They are commonly used in smaller spills (<100L) or as a polishing step for larger spills once conventional response technologies have removed the bulk of the oil. Sorbents used in oil spill response should possess two main characteristics: attract oil (possess oleophilic properties) and repel water (possess hydrophobic properties). This combination helps make them take up oil efficiently while minimizing water uptake. Sorbents can be made from a range of materials, including organic (cotton, peat moss, sawdust), inorganic (perlite, vermiculite, volcanic rock), or synthetic (polyurethane, polypropylene, polypropylene) substances. Common forms include pads and rolls, which are widely used because they are easy to deploy, cover large or targeted areas quickly, and can be removed and replaced as they become saturated. A test standard for small samples (coupons) of sorbent pads currently exists as ASTM F726-17(2024), but there are growing issues with the acceptance of this protocol. One issue is the implication of scaling the results of a test of a small portion of material to a full-size sheet. Minor variances in the fabrication of a sorbent pad or even the method used to cut the coupon can impact the testing results. Additional factors such as the orientation of the sorbent sample when it is retrieved from a test cell and the time allocated for excess oil to drip off will all impact the oil recovery performance. This has led to the desire to develop a new protocol to enable full sorbent sheets to be directly evaluated and have that protocol form the basis of an industry recognized standard so that comparisons of performance results between tests can be made. The goal of this work was to develop a robust protocol that generates reproduceable performance data on a range of sorbent attributes that are important from an oil spill response perspective, including oil recovery capacity and water uptake. Additionally, it should be affordable to perform, using equipment that is commonly available or easy to construct. The protocol should be usable at different test facilities, including Ohmsett. It should also be developed with input from a variety of stakeholders including spill response professionals, researchers, and regulators to allow it to have broad acceptability. To meet the project's objectives, a series of targeted actions were planned and accomplished. These steps included an initial review of existing and historical sorbent test protocols to identify unique or valuable techniques and specific methodologies that may be useful for development. The next step was consultations with industry members at meetings and workshops to solicit input from a wide range of stakeholders to ensure a robust and comprehensive protocol was developed. A demonstration of existing Ohmsett apparatus and in-house protocol was conducted to provide a visual representation of different aspects of testing. This was followed by an analysis of deficiencies and recommendations of alternative testing techniques or apparatus were solicited from stakeholders. Finally, a new draft protocol was generated and has been submitted to ASTM International for review and consideration to become a new standard (under updated Work Item ID WK100266).

Contents

List of Figures

List of Tables

1 Introduction

Sorbents are a common response technology and are used to some degree at most oil spills. They generally work through a combination of two mechanisms:

1. Adsorption (onto surfaces including pores and capillaries – most common phenomena)
2. Absorption (internally, and causes swelling – as defined in ASTM F726-17(2024))

The more generic term "sorbent" is used because both processes may occur depending upon the material of construction and the spilled liquid being picked up. They can be a primary response option for small spills (<100L) that may not warrant the mobilization of larger pieces of response equipment. They are also used in the final stages of clean-up operations of larger spills, particularly when shoreline impacts have occurred. Sorbents are also useful for equipment decontamination at all spills.

Sorbents used for oil spills must have two main characteristics: attract oil (possess oleophilic properties) and repel water (possess hydrophobic properties). Other beneficial properties (Robertson, Fingas, & Solsberg, 1976) may include:

- High sorption capacity – possess a high ratio of weight of oil absorbed or adsorbed to the weight of the substance itself,
- Retentive – retain oil indefinitely,
- Buoyant, when saturated,
- Fast acting
- Self-acting – require little or no mixing energy to sorb oil
- Non-toxic

Sorbents are typically made from a wide range of materials, each with specific advantages and disadvantages. These materials generally include organic materials (such as cotton, peat moss, and sawdust), inorganic materials (such as perlite, vermiculite, and volcanic rock), and synthetic materials (such as polyurethane, polyethylene, and polypropylene) (Robertson, Fingas, & Solsberg, 1976). Sorbents are also available in many formats, which have been categorized by Type:

Type I – Roll, sheet, pad. These products have a length and width much greater than their thickness and typically have sufficient integrity to be handled either saturated or unsaturated.

Type II – Loose. Unconsolidated, particulate material with sufficient form and strength to be handled except with sieves and similar equipment. It is typically applied over a spill and gathered with ancillary equipment for recovery.

Type III – Enclosed sorbent materials, including:

Type IIIa – Pillows and socks. Materials contained by an outer fabric or netting which retains the sorbent material but allows oil to permeate.

Type IIIb – Sorbent booms – contained in or arranged in the form of a long cylinder that can be deployed as a boom. Lack of any skirt below the waterline limits containment abilities.

Type IIIc – Sorbent sweeps – long rolls of sorbent materials attached to a strap or rope along one edge which allows the sorbent material to fan out across the water layer.

Type IV – Agglomeration unit. These include ribbons, strips, pom-poms, and open netting. These sorbents are typically used to remediate spilled oil that is highly viscous (>10,000 cP) (S.L. Ross Environmental Research Limited, 2017)

These specific forms are highlighted in currently accepted testing protocols - with "Type I" sorbents including the most common sheet, pad and mat format. (ASTM International, 2024)

Of course, not all sorbents are created equal, and responders presumably want to select the most effective products based upon performance and cost. Several protocols have been used over the past five decades in published reports to help measure the performance of sorbent products. These protocols have focused on evaluating coupons or small representative samples of larger sorbent sheets. Examples of the core protocols that form the basis of most historical studies include:

1. Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents (including original publication and Update #1 through #4), published by Environment Canada. (Robertson, Fingas, & Solsberg, 1976) (Robertson L. A., 1978) (Robertson L. A., 1983) (Enviroclean, 1985) (S.L. Ross Environmental Research Limited, 1991)
2. Canadian General Standards Board protocol CAN/CGSB-183.2-94. (Canadian General Standards Board, 1994)
3. ASTM F726-2017(2024) Test Method for Sorbent Performance of Adsorbents for use on Crude Oil and Related Spills. (ASTM International, 2024)

These protocols were scaled for laboratory use and evaluated small representative samples (coupons) taken from larger sorbent formats. It can, however, be difficult to extrapolate real-world performance from small-scale tests, and sampling methods can introduce errors. For example, the 13 cm by 13 cm sorbent sample size used in ASTM F0726-17 may not necessarily account for variations in the manufacturing process that would be captured with a larger scale test. Issues such as variability of the material thickness, or even the presence of a manufacturer's branding embedded on the sorbent pad can impact pick-up performance. This can lead to the generation of "coupon" performance data that may not necessarily be representative of the performance of a large full-sized sheet. Another issue that can impact performance is simply the method used to cut a 13 cm by 13 cm sorbent sample from a larger sheet. Using scissors compresses the material and creates a sealed edge on polypropylene pads. Conversely, using a straight guide and an angled utility blade (like an X-Acto knife) cuts the material without

compression. Internal testing at Environment Canada during the 1990s demonstrated that these two methods create different oil-draining profiles when samples are suspended outside the test fluid, and the choice of cutting tool can significantly impact oil retention results. Even the orientation of the sample while draining is important – holding a sorbent sample at one corner will allow excess fluid to drain out the opposite corner of the sample which is greatly reduced when holding a sorbent sample at the middle of one side. These differences can skew the results away from the expected results from a full-sized mat or pad. Relying on inconsistent interpretations or ad-hoc cutting methods introduces uncontrolled variables that compromise data repeatability. This has led some reviewers to comment that small scale testing results from variations of ASTM F726, in particular the method for measuring a sorbent's oil retention, may overstate performance (Bazargan et al. 2015). These issues became a driving force behind the development of an official protocol for the determination of full-sized sorbent performance.

The performance of sorbents as an oil spill response technology has been tested at Ohmsett for many years. BSEE and Ohmsett staff started the development of a Type I test protocol for testing full size or mesoscale samples around 2021. The protocol was designed to focus on testing attributes deemed important to end users, such as:

1. Recoverability of surface oil across a range of viscosities
2. Volume of oil recoverable by the sorbent
3. Water uptake when exposed to oil and water
4. Buoyancy
5. Ability to retrieve the sorbent intact when fully saturated.

The draft BSEE/Ohmsett protocol has been applied in at least two studies in which the following concerns were noted:

- When the draft field-scale protocol was used to recover light dielectric oil, uncertainties arose in the measurement data. These were attributed to differences between the oil types tested and those used during the protocol's development, as well as to equipment variability, observer errors, or the methodologies employed.
- Modifications to the prescribed test method can lead to more favorable results for some products.

Draft test protocols used at Ohmsett have evolved over time which limits direct comparisons of the results, and there remains some inconsistencies over certain aspects of the protocols employed. These issues, combined with the previously described problems associated with the laboratory-scale protocols, formed the basis of the formation of this project, which is tasked with the development of an official, defined, repeatable standard test protocol for evaluating Type I sorbents at field-scale.

2 Objectives and Goals

The goal of this work was to develop a robust protocol that generates reproduceable performance data on a range of sorbent attributes that are important from an oil spill response perspective. These attributes include oil recovery capacity, water uptake, durability, and selectivity (preferential oil pick-up over water when exposed to both). The protocol was created with the following criteria:

I. It should be affordable to perform, using equipment that is commonly available or easy to construct.
II. It should be usable at different test facilities, including Ohmsett.
III. It should be developed with input from a variety of stakeholders, including spill response professionals, researchers, and regulators.
IV. It should generate reproducible data that is based upon actual response methodologies
V. It should be eligible for publication as an industry standard.

3 Approach

To meet the project's objectives, a series of targeted actions were planned and accomplished. These steps included:

i) A review of existing and previously used sorbent test protocols to identify unique or valuable techniques and specific methodologies that could be replicated, as well as identify procedures that would not be appropriate for inclusion.
ii) Consultation with industry members, meetings, workshops to solicit input from a range of stakeholders and ensure a robust and comprehensive protocol was developed.
iii) Demonstration of existing Ohmsett apparatus and in-house protocol to provide a visual representation of different aspects of the protocol and clarify how the testing would occur.
iv) An analysis of deficiencies in the in-house protocol, and recommendations of alternative techniques or apparatus were solicited from stakeholders so that any issues could be identified and addressed in a collaborative environment.
v) Submission of a protocol for ASTM consideration.

4 Review of Historical Sorbent Testing Standards

The existence of historical protocols helps accelerate new protocol development. Identifying previous related protocols and building on their development helps an early consensus on targeted attributes to be reached. Laboratory-scale protocols for testing oil spill sorbents include:

1. Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents (including Update #1 through #4), published by Environment Canada. (historical)

2. Canadian General Standards Board protocol CAN/CGSB-183.2-94. (historical)
3. ASTM F726-17(2024) Standard Test Method for Sorbent Performance of Adsorbents for use on Crude Oil and Related Spills

4.1 Review of Historical Standards

4.1.1 Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents (Robertson, Fingas, & Solsberg, 1976)

Summary
Twenty oil spill sorbents were tested related to their performance and operational criteria (some of the selected sorbents had limited testing performed). Initial measurements of oil pick-up capacities were compared to sorbent prices to calculate oil sorption material costs. The protocols used were then applied in a subsequent evaluation using both an oil-only and oil/water test. Maximum sorbent capacity was correlated and compared with the viscosity of the test oil. Simulated field tests in outdoor tanks were also conducted using crude oil and No. 2 fuel (similar to diesel). Both the oil pick-up capacity and the oil/water ratios were determined (Robertson, Fingas, & Solsberg, 1976) Additional details can be found in Appendix B.

Procedural issues – main protocol
The sample size used for the tests was limited to 2.5 g for particulate sorbents or 3" (~8 cm) diameter circle for mats. Internal testing at Environment Canada subsequently determined that this small size may overstate the performance when compared with full size mats. Additionally, the authors of the study used temperature to vary the viscosity of the test liquids. While this method may work for small differences between the target temperature and room temperature, too much of a gradient creates a gradient within the oil which creates uncertainty with respect to true viscosity. This could be resolved by testing within an environmental chamber or room, but that potentially adds substantial costs to the testing. Testing was performed with two slick thicknesses, a 0.1 mm slick and a 1.0 mm slick. It is difficult to have an oil that has a viscosity greater than a couple hundred cP to spread as thin as 0.1 mm. This creates an artificial condition that a sorbent would not see in an actual spill scenario.

Procedural issues – large scale testing
Testing involved a thin oil layer on water (averaging ~1 mm thickness) and was performed outdoors in a large tank. The lack of movement on the water surface biases the collection of liquid to favor oil pick-up versus water pick-up. Testing results showed lower performance when using higher viscosity crude oil versus the initial testing using No. 2 fuel oil. This test is performed in a limited oil environment which "starves" the sorbent material. While it may approximate conditions one might expect at an actual spill, it negatively impacts the results which will become more variable on a sorbent-by-sorbent basis. Testing with a larger amount of oil would provide more consistent data.

4.1.2 Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: An Update (Robertson L. A., 1978)

Summary

Twenty-one oil spill sorbents were tested at a laboratory scale suing three petroleum products that were weathered to two different states (one-day and seven-days). Diesel, crude oil, and Bunker C (Number 6 fuel oil) were used as test liquids. For this series of tests, test fluid layer thicknesses of 2.5 mm and 0.1 mm were used for diesel, while 5 mm and 0.1mm were used for crude oil, and 5 mm was used for bunker C. Initial capacity of test liquid (fresh sorbent) and maximum capacity of test liquid (maximum of up to 10 reuses) are determined, along with water pick-up. Testing was performed on a 10°C "natural water" (fresh water) layer with a working depth of 10-15 cm (Robertson L. A., 1978). More information can be found in Appendix B.

Procedural issues

In addition to the standard testing described above, enhanced long term testing (48 hours) was performed under quiescent conditions (no waves) which the authors admit provided results that may differ from actual field performance, during which energy is present. Sample size is not explicitly stated, but the procedure used is stated to be similar to the original 1974 Environment Canada funded study which used sample sizes of 3" or 8 cm diameter circles (for Type I mats) that are too small for direct comparisons to be drawn with full size sorbent testing.

4.1.3 Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: Update II (Robertson L. A., 1983)

Summary

Twenty oil sorbents were tested (two were not tested completely) at the laboratory scale in three different petroleum products. Oil used in this study was weathered to two different end points through evaporation. Testing was conducted to determine initial oil pickup, maximum oil pickup, and water pickup by testing on oil/water, oil, and water respectively. Additional testing was performed using a tensile strength tester on a portion of a sorbent mat that had been exposed to oil. (Robertson L. A., 1983). More information can be found in Appendix B.

Procedural issues

This project uses an Instron Tensile Tester (Model 1130) which was an improvement for generating precise data. Data was first collected using samples prior to testing, and again after reaching saturation in a test using pure oil. While this can identify the propensity of a sorbent mat to degrade during use, there is no linkage provided between values obtained and what it might mean for the collection of saturated sorbent mats in the field. In short, this portion of the protocol adds cost to the test but provides very limited (if any) benefits.

Samples were removed from the oil and held in a horizontal orientation during a "drain" phase of the test. This procedure caused an artificial increase in performance when compared to how sorbents are normally retrieved – lifted vertically along an edge. Additionally, the drain timing was 5 minutes which is excessive and not reflective of how sorbents are normally retrieved during an actual spill response.

4.1.4 Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: Update III (Enviroclean, 1985)

Summary

Nineteen commercially available sorbents were assembled for testing with three petroleum products and two hydrocarbon solvents. The petroleum products (diesel, crude oil, Bunker C) were weathered for one and seven days. One inorganic sorbent did not float and was removed from further evaluation. Testing demonstrated linkages between performance and slick thickness. Performance metrics included initial and maximum sorption capacity, water retention, and reusability. Sorbent samples were placed in test cells within a climate-controlled room at "low" (10°C) temperatures (Enviroclean, 1985). More information can be found in Appendix B.

Procedural issues

The use of a cold room to maintain the required test temperature of 10°C would be limiting for many facilities to replicate due to cost. If the test cell temperature is stable and the test fluid viscosity can be established at that stable temperature (and the viscosity targets are attained), there is limited benefit to requiring a cold-room investment.

Reuse of sorbents appears as part of the protocol. Calculations were performed based upon the cost of the initial sorbents ($ on a per mass basis) and taking into account the number of uses that a sorbent sample was able to successfully perform to generate a final cost per mass of oil picked up. The concept of reuse is currently discouraged as it may be prohibited by environmental regulations if the sorbent is not cleaned to pristine conditions prior to reapplication – and the methodologies employed in this protocol (mechanical compression) will fail to achieve that goal.

Sample size is again not explicitly stated, but the procedure used is stated employ the same basic methodology and approach as the original 1974 Environment Canada funded study. That study used sample sizes of 3" or 8 cm diameter circles (for Type I mats) that are too small for direct comparisons to be drawn with full size sorbent testing.

4.1.5 Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: Update IV (S.L. Ross Environmental Research Limited, 1991)

Summary

Sixteen sorbents were tested in three different petroleum products (diesel, crude oil, bunker C) and two hydrocarbon solvents. The diesel and crude oil samples were weathered for one and seven days. Performance metrics include initial and maximum oil sorption capacity, initial water retention, and maximum water retention (on reuse). Testing with the lighter test fluids (diesel, toluene, cyclohexane) was performed using a slick thickness of 2.5 mm and 0.1 mm, while testing with crude oil and bunker C was performed using a slick thickness of 5mm and 0.1 mm. Additionally, a long-term (48 hour) exposure test was also conducted and described as a qualitative test (S.L. Ross Environmental Research Limited, 1991). More information can be found in Appendix B.

Procedural issues
Sample size was not explicitly stated, but the report indicates the procedure was similar to Update III (3" or 8cm diameter circles) which uses samples that are too small for direct comparisons to be drawn with full size sorbent testing.

Agitation was not applied to the test cells but the sorbent samples were turned over once during testing which would artificially inflate the results of tests involving higher viscosity test oils. Additionally, the sorbent samples were drained in a flat orientation for an extended period of five minutes. The report acknowledges that some initial testing was performed with sorbents held in a vertical orientation and simply noted that there was a difference in the results generated.

4.1.6 Laboratory Evaluation of Sorbents (Counterspil Research Inc., 1999)

Summary
Twenty-five sorbent mats (Type I), and seven particulate sorbents (Type II) were tested. The study focused on various structural formats of sorbents for commonly spilled liquids including hydraulic oil, diesel, and glycol. Sorbents with affinities to specific liquids were used in this study, including oil-specific, universal, and chemical spill clean-up. Testing on pure test liquid (oil or water) was performed. A variation of a tensile strength protocol was performed on samples of the Type I sorbents. (Counterspil Research Inc., 1999). See Appendix B for more information.

Procedural issues
The tensile strength "durability" test involved only one sample. A minimum of 3 should be used for statistical purposes. The report also fails to assign a linkage between the results produced (Breaking Weight) and how that might impact the performance of a sorbent pad during application and retrieval during an actual spill. The test samples for capacity testing were cut but there is no information on how they were cut (scissors, knife), other than indicating they were cut from a template to fit into a weigh tray. Additionally, there is no information on how the samples were held after retrieval from the test cell, while they were dripping - prior to weighing. The cutting and handling can impact the retention results.

4.1.7 Temporary Ohmsett Protocol (Coolbaugh & McKinney, 2021)

Summary
A new test apparatus was designed and built over multiple testing phases, beginning in 2018, to accommodate typical sorbent mats at their full size (up to 0.92m x 0.92m). Multiple performance metrics were determined including maximum oil capacity, oil adsorbed at a prescribed time interval, and water update. Secondary performance metrics were also identified (Coolbaugh & McKinney, 2021). See Appendix B for more information.

Procedural issues
Multiple tests occurred but some issues with variability in the results were identified. The quantity of oil added to a test cell is not a defined variable – it relies on performing preliminary testing with ASTM F726 to determine a saturation mass, then a calculated percentage is used for

full scale testing. This aspect limits comparisons that can be made when sorbents are presented with different quantities of oil.

The field retrieval aspect of the protocol requires manual penetration of the sorbent pad by a single pitchfork prong – which is performed manually and may lead to inconsistent tearing of the sorbent mat and lead to variabilities in ultimate tear strength measurements.

5 Formation of Working Group

A working group of five volunteer members was formed to help with the development of a new draft protocol. Participant organizations included BSEE, Ohmsett (ARA) staff members, researchers from NOAA, and researchers from S.L. Ross. Some expressed an interest in being kept informed as to progress of the protocol development and volunteered to provide input once the protocol was officially presented to the ASTM F20.22 subcommittee for review and consideration.

A second group consisting of the ASTM F20.22 subcommittee on Mitigation reviewed an early draft protocol at the 2024 ASTM F20 committee meeting and supplied initial inputs. This initial contact was done to solicit input from a wider range of stakeholders, and to gain acceptance once the protocol was updated and enhanced.

A meeting with the core Working Group was convened at Ohmsett in March of 2026 to review the historical protocol and identify deficiencies that could be resolved for incorporation into the new draft. It provided an opportunity to witness the complexity of attempting to perform full-scale testing and demonstrate how minor adjustments (example: clearing the retrieval rack of spurious water droplets) can impact overall results.

5.1 Review meeting at Ohmsett

The agenda included demonstrations of small-scale testing (ASTM F726) in the laboratory with test coupons, along with demonstrations of large-scale testing that took place using full sorbent mats. Specific aspects of the protocol reviewed included sorbent application, sorbent retrieval, oil quantity, oil/water layer thickness, and field retrieval (pitchfork prong) test.

Figure 1: Review of ASTM F726 Protocol During Meeting at Ohmsett
Laboratory-Scale protocol review discussion at the Chemistry Laboratory - Ohmsett (Photo: ARA, 2026.)

Figure 2: Demonstration of Large-Scale Testing at Ohmsett
Review of large-scale testing procedures helps identify areas of concern requiring modification (Photo: ARA ,2026.)

Discussion included topics such as sensitivity of the measurements, repeatability, reproducibility and closeness to matching actual spill recovery methodologies. Concepts such as the need to have water movement (dynamic conditions) when testing water pick-up were reviewed. Another aspect, specifically the field retrieval (pitchfork prong) test was demonstrated and immediately highlighted difficulties in piercing some of the sorbent sheets. This suggested that alternate procedures or equipment might be better at achieving the same goals of determining if a fully saturated sorbent would be able to support its own weight when being retrieved without tearing. An alternative (heavy duty battery clamps) was suggested, subsequently tested, and demonstrated to support the equivalent fully saturated weight of sorbent pads. This solution has now been incorporated into the new draft protocol.

6 Proposed New Protocol

Based upon a review of previous laboratory scale testing and the current draft BSEE/Ohmsett protocol for full scale sorbent testing at Ohmsett and comments received from stakeholders a new draft protocol was developed and is presented in an ASTM format.

Draft Standard Test Method for Full Scale Performance of Adsorbents for use on Crude Oil and Related Spills[1]

1. Scope

1.1 This test method measures the performance of sorbents in removing crude oils, emulsified oils and other floating immiscible liquids from the surface of water.

1.2 Testing is done in a controlled environment at larger scales than other laboratory tests, using full sheets of sorbents or pillows, or large sections of socks.

1.3 The measurement values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 This standard does not purport to address all safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in 8.3.1.

1.5 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards

[1] This test method is under the jurisdiction of ASTM Committee F20 on Hazardous Substances and Oil Spill Response and is the direct responsibility of Subcommittee F20.22 on Mitigation Actions.

F726-Standard Test Method for Sorbent Performance of Adsorbents for use on Crude Oil and Related Spills.

3. Terminology

3.1 General Terminology:

3.1.1 *gellant* – a material such as a colloidal network or other aggregate network that pervades and holds a liquid in a highly viscous fragile structure. Many gels may rapidly liquify with added heat or ionic/polar addition. These materials are soluble/flowable in excess liquid.

3.1.2 *sorbent* - an insoluble material or mixture of materials used to recover liquids through the mechanisms of absorption or adsorption, or both.

3.1.3 *thickener* – a material (usually of high molecular weight) that is soluble in excess liquid. These materials go from dry to gummy (viscoelastic) to flowable and then soluble. The final viscosity depends on the liquid to solid ratio.

3.1.4 *Universal sorbent* - an insoluble material or mixture of materials that will sorb both hydrophobic and hydrophilic liquid spills.

3.2 Definitions

3.2.1 *absorbent* - a material that picks up and retains a liquid distributed throughout its molecular structure causing the solid to swell (50 % or more). The absorbent is at least 70 % insoluble in excess fluid.

3.2.2 *adsorbent* - an insoluble material that is coated by a liquid on its surface including pores and capillaries without the solid swelling more than 50 % in excess liquid.

3.3 Definitions of Terms Specific to This Standard: This test method does not apply to belt, rope, or weir type skimming devices.

3.3.2 *oil* - a substantially water immiscible organic liquid that will float on water (density less than 1 g/mL), typically with surface tension less than 40 mN/m.

3.3.3 *Type I adsorbent* (roll, film, sheet, pad, mat, blanket, web)—a material with length and width much greater than thickness and which has both linear form and strength sufficient to be handled either saturated or unsaturated.

3.3.4 *Type II adsorbent (loose)*—an unconsolidated, particulate material without sufficient form and strength to be handled except with scoops and similar equipment.

3.3.5 *Type III adsorbent (enclosed)*:

3.3.5.1 *IIIa, pillows*—adsorbent material contained by an outer fabric or netting that is permeable to oil, but with openings small enough to substantially retain the sorbent material within the fabric or netting.

3.3.5.2 *IIIb, adsorbent booms*—similar to pillows, but the lengthwise dimension substantially exceeds other dimensions and with strength members running parallel with length. Booms may include connections for coupling sections together.

3.3.6 *Type IV-agglomeration unit*—an assemblage of strands, open netting, or other physical forms giving an open structure that minimally impedes the intrusion into itself of high viscosity oils. Normally for use with viscous oils, typically above 10 000 cP viscosity. Said oils are then held in this structure permitting the composite oil/structure to be handled (e.g., pompoms).

3.3.7 *reuse*—extracting adsorbed liquids from an adsorbent through rolls or other compression techniques permitting the adsorbent to be used again; There may be regulations against introducing oil-containing material to waterways that restrict reuse of sorbents (e.g., the U.S. Clean Water Actor other legal restrictions.

4. Summary of Test Method

4.1 A sorbent sheet, pillow, or section of sock or other format, is lowered onto a known amount of oil floating on water in an apparatus that is then agitated to simulate wave motion. The test measures oil and water sorption by weight, and tear strength.

5. Significance and Use[2]

5.1 This test method is to be used as a basis for comparison of full-size sorbents in a consistent manner.

6. Apparatus

6.1 Test Cell – The dimensions of the test cell shall be 1.00m ± 0.05m x 1.00m ± 0.05m with walls that are at least 0.20m high. The test cell shall be mounted on a track with a drive mechanism capable speed control of 60 cycles per minute and a horizontal amplitude (stroke distance) of 2.5 cm.

6.2 Load Cell or Balance – A 0-10kg load cell (to accommodate the sample plus hardware to hold the sorbent) with an accuracy of 0.02% or better, or a table-top balance with a similar capacity to accommodate a weighing pan plus the sorbent with a resolution of 0.1g may be used to weigh fresh and saturated sorbents.

6.3 Retention clamps – Clamps to hold the sorbent while it is being retrieved from the test cell. Recommended clamps attach over an area of 25 mm x 20 mm and are placed within 5 cm of a corner along a short edge of a sorbent sheet with a spreader bar to prevent the sheet corners from folding in during retrieval. Heavy duty battery clamps or similar equipment can be used.

6.4 Roller Extraction (Manual Laundry Wringer) – manually operated device used to squeeze fluid out of a saturated sorbent sheet so that a water content determination can be made.

7. Conditioning

[2] Ensure that material compatibilities exist between the sorbent and the hazardous substance which may be sorbed.

7.1 Condition all sorbent test specimens: 20°C ± 4°C and 70% ±20% relative humidity for not less than 24 hours prior to testing. Condition specimens in a fully exposed state with no coverings or wrapping that would hinder the ambient equilibration process

7.2 If temperature or humidity conditions other than those indicated above are expected to be important, then conditioning and testing should be carried out at the temperature and humidity level of interest.

8. Testing fluids

8.1 Testing involves the use of oils with a range of viscosities and densities as indicated below.

Table 1 Test Oil Properties

Oil Type	Viscosity Range	Density Range	Example
Light	1 to 10 cP	0.820 to 0.870 g/mL	Diesel fuel, mineral oil
Medium	200 to 400 cP	0.860 to 0.970 g/mL	Crude oil, canola oil, mineral oil
Heavy	1500 to 2500 cP	0.930 to 1.000 g/mL	Bunker C or residual fuel, mineral oil
Weathered	8000 to 10 000 cP	0.930 to 1.000 g/mL	Emulsified crude oil, mineral oil

8.2 The test is normally performed using salt water, although freshwater can be used if warranted. The type of water must be noted in the testing report.

9. Test Procedures

9.2 Full Scale Dynamic Water Pick-up Test

This procedure is designed to test for water take-up and to determine oleophilic properties of a full-scale sorbent under dynamic conditions. This test is normally performed at 23°C ± 4°C, unless another temperature is desired. Operating at a higher or lower temperature must be documented in the results report.

9.2.1 A test sample (full sorbent sheet) is first weighed and then placed in the approximately 1 m^2 tray filled to a depth of 5cm of fresh water (record the temperature) that is agitated at 60 cycles per minute and a horizontal amplitude of 2.5 cm for a duration of 60 minutes.

9.2.2 After this period, stop the tray movement and allow to rest for 2 minutes. Observations of the condition of the sorbent and the water are recorded. Any sorbents that do not remain floating at the surface of the water are considered to have failed this test.

9.2.3 Pick the sorbent up out of the water along a short edge using two clamps and allow it to drain for 30 s.

9.2.4 Weigh the sorbent using load cell in line with the lifting clamps or use a large weighing pan and balance.

9.2.5 Calculate the water pick-up ratio from the weight measurements. Salt water may be used in lieu of fresh water but must be noted in the test results. Testing is performed in triplicate.

9.3 Calculations:

9.3.1 Using the data obtained in 9.2, calculate the water sorbency as the ratio of the weight of the water picked up versus the weight of the dry sorbent as follows:

Water sorbency = $(S_w - S_o) / S_o$

Where:

S_o = initial dry sorbent weight

S_w = net water sorbed

Example:

Initial sample weight $S_o = 45.3$

Weight after water test $S_w = 58.4$

Water sorbency $=(S_w - S_o)/ S_o = (58.4-45.3)/45.3 = 0.29$ water pick-up ratio

9.4 Oil capacity

9.4.1 Fill the test cell to a depth of 5 cm with water. Then place a pre-weighed test sample (full sorbent sheet) in the test cell and start agitation at an operating frequency of 60 cycles per minute and a horizontal amplitude of 2.5 cm for a duration of 15 minutes. At the end of the initial 15 minutes stop the movement of the tray and allowed it to settle for a period of 2 minutes.

9.4.2 Pick the sorbent up along a short edge using the two-clamp lifting assembly and lift the sorbent out of the tray and allow it to drain for a 30 s drain period. The sorbent is then weighed either using an attached strain gauge or by transferring the sorbent to a large weighing tray and determining its weight.

9.4.3 Add five litres of oil to the top of the water layer in the test cell (resulting in an average oil layer thickness of 5 mm - assuming complete coverage), and the return the sorbent sample to the test cell and allow to sorb oil for 15 minutes under calm conditions. The test cell movement system is then engaged at an operating frequency of 60 cycles per minute and a horizontal amplitude of 2.5 cm for a duration of 30 minutes. Stop the tray movement at the end of the half hour portion of the test and allowed it to settle for a period of 2 minutes. Record observations pertaining to the condition of the sorbent and the water.

9.4.4 Pick the sorbent up along a short edge using the two-clamp lifting assembly and allow it to drain for a specified period (30 s drain period for light, medium, and heavy oil; 2 minutes for weathered oil). If the sorbent material tears and cannot support its weight, then it must be noted as a failure to maintain integrity during collection.

9.4.5 The sorbent is then weighed either using an attached strain gauge or by transferring the sorbent to a large weighing tray and determining its weight. The fluid pick-up ratio is calculated from the weight measurements. Testing is performed in triplicate. The water content of the recovered fluid can be estimated by extracting fluid from the sorbent by wringing it using a manual roller extractor or similar device

9.4.6 Fluid Extraction - The roller to be used should have rollers of sufficient length to accommodate the unfolded sorbent sample and a diameter from 3 cm to 10 cm. Force is applied at the rate of 4 kg/cm of nip while the adsorbent travels through the extractor at a velocity between 5 cm/s and 10 cm/s.

9.4.7 The fluid is collected in a graduated container and allowed to settle and separate overnight. Heating the container in a water bath at 50°C for an hour can help destabilize any weak emulsion and accelerate the separation process, as can the addition of 3 drops of Alcopol (aka Drimax). The volumetric difference in the recovered fluid phases or the difference in heights of a graduated cylinder can provide an estimate of the water percentage of the recovered fluid. It is noted that any water collected during the initial portion of the test may be displaced during the remaining 45 minutes of the test. The fluid and oil pick-up ratios are calculated from the weight and volume (graduated cylinder) measurements. Testing is performed in triplicate.

9.5 Calculation:

9.5.1 Using the data obtained in 8.4, calculate the initial water sorbency as percentage of water sorbed to dry sorbent weight as follows:

Initial Water sorbency = $(S_{w15} - S_o)/ S_o$

Where:

S_o = initial dry sorbent weight

S_{w15} = net water sorbed in the first 15 minutes

Example:

Initial sample weight So = 44.7g

Weight after water test S_{w15} = 56.2

Initial Water sorbency = $(S_{w15} - S_o)/ S_o$ = (56.2-44.7)/44.7 = 25.7% water pick-up

9.5.2 Using the data obtained in 8.4, calculate the final fluid sorbency as percentage of total fluid sorbed to dry sorbent weight as follows:

Fluid sorbency = $(S_F - S_o)/ S_o$

Where:

S_o = initial dry sorbent weight

S_F = weight of sorbent at end of test

Example:

Initial sample weight S_o = 44.7g

Weight at end of test S_F = 483.2

Initial Fluid sorbency = $(S_F - S_o)/ S_o$ = (483.2-44.7)/44.7 = 9.8 fluid pick-up ratio.

9.5.3 Using the data obtained in 8.4, calculate the final water content of the recovered fluid as a percentage.

Water content = $H_W /(H_F)$

Where:

H_W = height of water layer in graduated cylinder

H_F = overall height of fluid graduated cylinder (including water and oil layers)

Example:

Overall fluid height in graduated cylinder H_F = 150mm

Height of water layer in graduated cylinder H_W = 9mm

Water content of collected fluid = H_W / H_F = 9/150 = 6.0%

10. Keywords

10.1 absorbent, adsorbent, sorbent.

7 Conclusions and Recommendations

Historical protocols that form the basis of most modern protocols were reviewed, analyzed, and summarized. This helped to identify portions of protocols that have proven to successfully generate reproduceable data. It also helped identify problematic areas that should either be rejected or subjected to additional study. This further analysis helped determine why the generated data is either not reproducible within target limits or inconsistent with practical oil spill clean-up procedures.

A working group of five participants was successfully created with representatives from target organizations including BSEE, current Ohmsett staff, industry, and researchers who helped provide input into the drafting of a new protocol. Additionally, a second group consisting of the ASTM F20.22 subcommittee on Mitigation reviewed an early version of the draft and supplied

initial inputs. Soliciting input from diverse stakeholders helps develop a robust protocol and helps gain protocol acceptance from the standards organization and response community.

A new protocol was successfully developed that allows the performance evaluation of full-size sorbent mats. The protocol precisely defines the apparatus, conditioning for the sorbent mat samples and the testing fluid parameters suggested for use. During testing, the protocol accounts for water pick-up under dynamic conditions, oil capacity, and preferential oil displacement which are the main performance criteria. Additional areas address sinking and mat integrity.

The draft protocol has been submitted to the ASTM International subcommittee for review and consideration as an official standard under updated Work Item ID WK100266. Once accepted, it will be maintained by the subcommittee responsible, F20 on Hazardous Substances and Oil Spill Response.

8 References

ASTM International. (2024). *F716-24 Standard Test Methods for Sorbent Performance of Absorbnets for Use on Chemical and Light Hydrocarbon Spills.* West Conshohocken, PA: ASTM International.

ASTM International. (2024). *F726-17(2024) Standard Test Method for Forbent Performance of Adsorbents for use on Crude Oil and Related Spills.* West Conshohocken, PA: ASTM International.

Bazargan, A., Tan, J., & and McKay, G. (2015). *Standardization of Oil Sorbent Perfomance Testing.* West Conshohocken, PA: ASTM International.

Canadian General Standards Board. (1994). *CAN/CGSB-183.2-94 Method for Testing Sorbents.* Ottawa, ON: Canadian General Standards Board.

Coolbaugh, G., & McKinney, K. (2021). Applicastion of the Field Scale Test Protocol for Type I Sorbents Recovering Oil on Water`. *Proceedings of thee Forty-third AMOP Technical Seminar* (pp. 112-131). Ottawa: Environment and Climate Change Canada.

Counterspil Research Inc. (1999). *Laboratory Evaluation of Sorbents.* Ottawa, ON: Environment Canada.

Enviroclean. (1985). *Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: Update III.* Ottawa: Environment Canada.

Robertson, L. A. (1978). *Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: An Update.* Ottawa: Environment Canada.

Robertson, L. A. (1983). *Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: Update II.* Ottawa: Environment Canada.

Robertson, L. A., Fingas, M. F., & Solsberg, L. B. (1976). *Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents.* Ottawa: Environment Canada.

S.L. Ross Environmental Research Limited. (1991). *Selection Criteria and Laboratory Evaluation of Oilspill Sorbents Update IV.* Ottawa: Environment Canada.

S.L. Ross Environmental Research Limited. (2017). World Catalog of Oil Spill Response Products. In *World Catalog of Oil Spill Response Products* (p. 447). Ottawa.

Appendices

Appendix A: Technical Summary

REPORT TITLE: Field Scale Test Protocol for Type 1 Sorbents

CONTRACT NUMBER(S): 140E0124P0027

FISCAL YEARS(S) OF PROJECT FUNDING: FY2025 - FY2026

CUMULATIVE PROJECT COST: $149,700.00

COMPLETION DATE OF REPORT: 31Aug 2026

MMA/BSEE COR(S): Steve Buschang, Kevin Cabaniss

MMA/BSEE CO(S): Catherine Robertson

PROJECT MANAGER(S): David Cooper

AFFILIATION OF PROJECT MANAGER: SL Ross Environmental Research LTD

ADDRESS: 38 Auriga Dr. Suite #200, Ottawa, ON K2E 8A5, Canada

PRINCIPAL INVESTIGATOR(S)*: David Cooper and James McCourt

KEY WORDS: Adsorbent Absorbent Sorbent Evaluation Protocol Oil Spill ASTM Ohmsett

* The affiliation of the Principal Investigators(s) may be different than that listed for Project Manager(s).

Appendix B – Historical Protocols

Appendix B.1 Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents.

Details compiled from *Robertson, L. A., Fingas, M. F., & Solsberg, L. B. (1976). Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents. Ottawa: Environment Canada.*

Summary

A total of twenty oil spill sorbents were studied related to their performance and operational criteria (some of the sorbents selected had limited testing performed). Initial testing involved the determination of oil pick-up capacity of several oil sorbents using a Canadian western crude oil Capacities were correlated to sorbent prices to determine oil sorption material costs. The protocols used were then applied in a subsequent evaluation using both an oil-only and oil/water test. Maximum sorbent capacity was correlated and compared with the viscosity of the test oil. Simulated field tests in outdoor tanks were also conducted using crude oil and No. 2 fuel (similar to diesel). Both the oil pick-up capacity and the oil/water ratios were determined.

Sorbents used in the study

Inorganic Sorbents

Absorbent 1012 BioSorb	Saneringsull Vermiculite

Organic Sorbents

Conwed (modified organic) Peat Moss Petro-pak Petropeat	Slikwik Sorb-Oil Straw

Polymeric Sorbents

Capillardiamin Grab (Petro-Grab) Graboil Oil Snare	3M Oleogon Polyurethane Typar

Mixed Sorbents (organic/inorganic)

Cel-U-Con Fibreperl	Sorbent C

Performance Characteristics

According to the report, a sorbent should ideally be:

1. Oleophilic – have a greater attraction for oil than water,
2. Hydrophobic – repel water
3. High sorption capacity – possess a high ratio of weight of oil absorbed or adsorbed to the weight of the substance itself,
4. Retentive – retain oil indefinitely,
5. Buoyant, when saturated,
6. Fast acting
7. Self acting – require little or no mixing energy to sorb oil
8. Non-toxic, and
9. Reuseable – will not deteriorate upon reapplication in a water environment (if/when allowed to do so)

Influences on Sorption Performance

The report acknowledges limitations to testing in controlled laboratory environments, and specifically identifies factors that will affect the results of testing including:

1. Water and air temperature
2. Thickness of the oil slick
3. Physical state of the oil (i.e. emulsification, aging (weathering), etc.)
4. Presence of debris
5. Mode of sorbents selected (i.e. chips, mats, sweeps, etc.)

There are three main performance criteria, as defined below:

Sorption capacity, defined as the amount of oil that a particular material (sorbent) is capable of picking up, usually given as grams of recovered oil per gram of sorbent,

Maximum sorption capacity, defined as the maximum pick-up capacity of oil by a sorbent testing on oil only, expressed as gram of recovered oil per gram of sorbent, and;

The Oil/Water Ratio, defined as the weight of oil versus the weight of water in the liquid recovered by the sorbent.

As a conclusion to the principal findings, the polymeric sorbents (as opposed to those based upon organic materials such as peat moss, or straw) exhibited high maximum sorption capacities and high oil/water ratios.

General recommendations for further research included the following:

1. Assess the performance of sorbents in actual field situations under various weather and water conditions;
2. Assess the feasibility of using sorbents for shoreline protection and restoration;
3. Develop methods, techniques and equipment to apply and harvest sorbents;
4. Develop techniques and equipment for the reuse and disposal of sorbents;

5. Assess the effects of sorbents on the biosphere considering the biodegradability and toxicity of residual sorbent (contaminated with oil); and
6. Evaluate the performance of sorbents recovering other petroleum products, (other than crude oil and No. 2 fuel oil) as well as several chemicals.

Sorbent Types

Sorbents classified by their material origins, were divided into three classes: natural products, modified natural products, and synthetic products. A further distinction can be made by differentiating between organic and inorganic sorbents. Some sorbents may also be mixtures of the above.

Laboratory Evaluation of Sorbents

It is acknowledged that the procedure developed by the Environmental Emergency Branch's Centre of Spill Technology should serve as a guide and actual field use involving varying weather conditions, sea state, water conditions and oil characteristics would affect the sorption results.

Main Protocol

Materials and Apparatus

Carver Laboratory Press (0-100 psi, Model "C") Haake Temperature Control, Model Kt33 Normal Laboratory Apparatus	Wester Crude Oil (high sulphur content) Various Sorbents

Oil was placed in a constant temperature bath and brought to a stable temperature (10°C). A sample of sorbent (2.5 g of particulate or 3" diameter circle of pad – depending on the format) was weighed, placed on the oil, and left for one hour. The sorbent was drained for 5 minutes and then weighed. The press was then employed to remove oil from the sorbent (maximum pressure 40 psi). The sorbent was then reweighed and the weight of the oil determined. To ascertain the reusability, this procedure was repeated until the sorbent disintegrated or the recovered oil fell below 5 g oil/g sorbent (up to a maximum of 10 repetitions).

Additional Testing by Imperial Oil Research

The procedure was repeated and enhanced to include oil films on water, and to study the effect of viscosity on sorbent capacity.

Additional Protocols – using two slick thicknesses on water (Part II) and changing viscosity (Part III)

1. (Part II) A layer of oil (0.1 mm slick and 1.0 mm slick) was used.

2. (Part III) Studied the impacts of changing viscosity on oil pick-up capacity (temperature of seven-day crude oil was varied between 7°C and 37°C to obtain viscosities from 10 to 1200 cP).

Simulated Field Evaluations of Sorbent (Large Scale Testing)

Apparatus

A tank with a diameter of 4.57m (15') and a height of 1.37m (4.5') was used for oil-on-water testing. A heavy polyethylene liner was placed inside the tank.

A screen, 0.61m x 0.61m with 1 x 3 cm openings was placed in a rigid frame and suspended from a scaffold by a rope and pulley mechanism. This screen was used to retrieve and drain sorbent mats/sheets.

A press was constructed to support a 0.61 x 0.61 m iron grating on a 0.61m x 3.05m wooden frame structure. A lever arm was hinged at one end to apply the squeezing force needed to the sorbent.

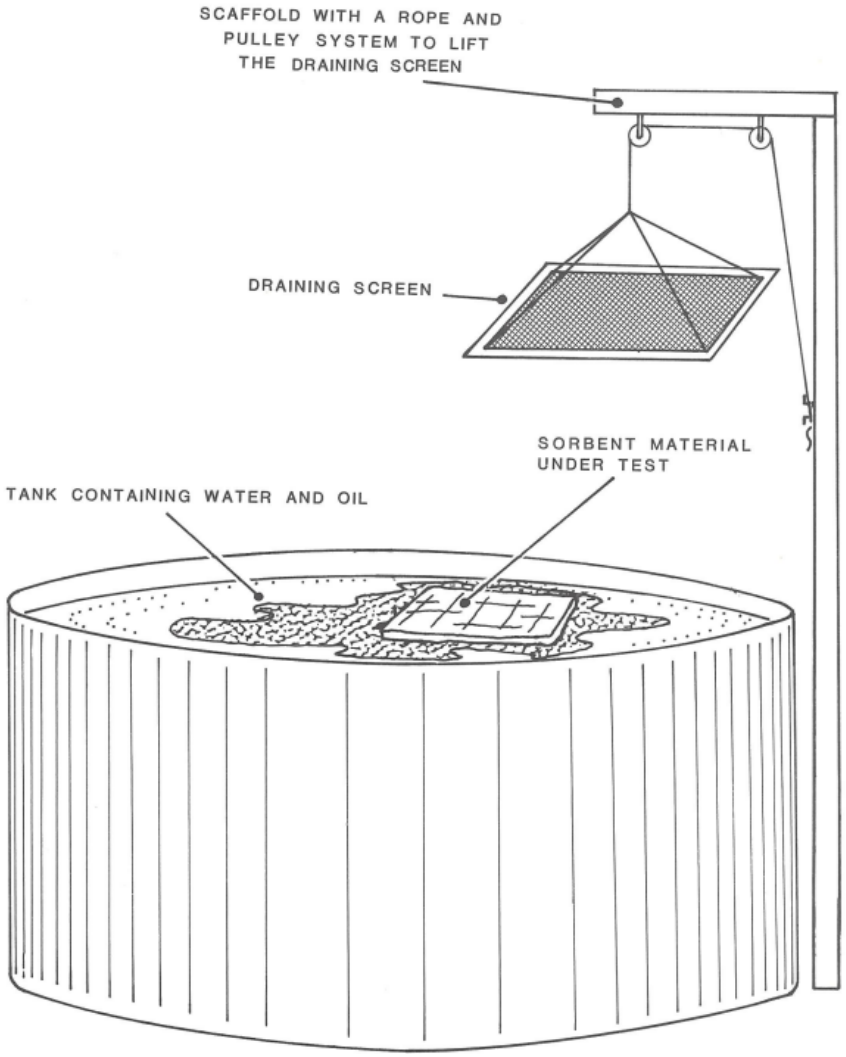


Figure 1 Selection Criteria Report 1976, Schematic Diagram of the Test Equipment

For testing in pure oil, a framed wooden box having dimensions of 1.22m x 1.22m x 0.30m was constructed and lined with 0.15mm thick polyethylene sheet as a liner.

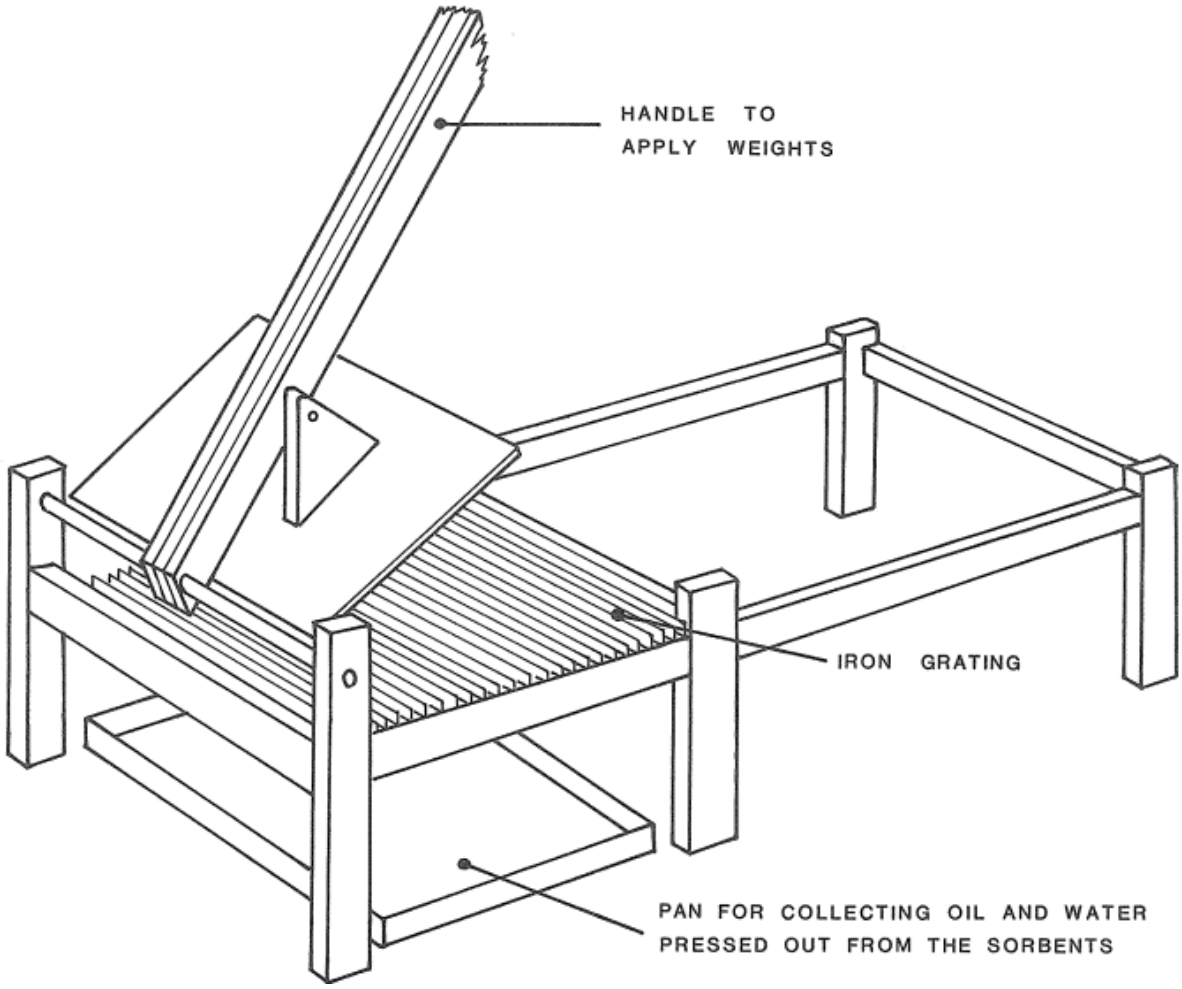


Figure 2 Selection Criteria Report 1976, Diagram of Apparatus for Squeezing Out Liquids

Procedure Summary

Testing was conducted outdoors under a wide range of climactic conditions. For oil on water testing, the tank was filled with water drawn from Lake Ontario to a depth of 1.22m (4'). Approximately 13.6kg of oil (~15-17L) of oil was poured onto the surface to produce a slick of 1.0mm thickness.

Mat or Sheet type sorbents were used as supplied; however loose sorbents were placed in a polypropylene bag with a 10x12 coarse weave. Overall dimensions of the bag measured 0.61m x 0.61 m x 0.05m (2' x 2' x 2"). It was acknowledged that this packaging format could affect the contact of the sorbent material with the oil and impact the results.

Three samples of each sorbent material were placed on the oil at staggered time intervals with a one-hour residence time on the tank surface. Samples were occasionally moved away from the perimeter of the tank if they happen to have been driven there by the wind. At the end of the test time, the samples were lifted out of the water and placed on the draining screen for 5 minutes. The test sample was then placed on the draining board where the oil was pressed out of the

sorbent. Extracted liquid was collected and measured via a graduated cylinder. A small subsample was removed for further analysis.

Water picked up by the sorbent was determined by the ASTM D95-70 distillation method. A portion of the soaked sorbent was collected at the end of the test and placed in a sealed container. A weighed amount of material was placed in a distillation apparatus to determine the volume of water entrained.

Similar procedures were followed to determine the maximum sorption capacity pure oil. Sorbent samples were placed on an oil layer for a period of one hour without agitation. Testing was conducted in triplicate.

Appendix B.2 Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: An Update

Details compiled from *Robertson, L. A. (1978). Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: An Update. Ottawa: Environment Canada.*

Summary

Twenty-one oil spill sorbents were tested at the laboratory scale via immersion in three different petroleum products that had been artificially aged for one and seven days. Synthetic sorbents exhibited the highest initial and maximum results. Testing demonstrated a reduction in sorbent performance as the test oil layer decreased in thickness from 2.5mm (diesel) and 5mm (crude oil) to 0.1mm along with a concurrent increase in water pick-up.

Sorbents used in the study

Inorganic Sorbents

Zorbite	

Organic Sorbents

Conwed (modified organic) Peat Moss Rubbermaid Black (ground rubber)	Slickwik Straw

Synthetic Sorbents (polymeric)

Conwed Durable Pads Graboil Imbiber Beads Leomat Oil Snare Qwik-Wick Spill Control FPD Spill Control PEP	Tafmat 3M Sheet 3M Fibre Winkler Foam – 50-PS-PU Winkler Foam – 50-R-PS Winkler Foam – 80-R-PS

Mixed Sorbents (Organic/Inorganic)

Sorbent C	

Study Parameters

Three quantitative aspects are highlighted:

Initial capacity - the amount of oil that a particular sorbent is capable of picking up on its initial exposure to the oil/water testing system, expressed as grams of oil per gram of sorbent

Maximum capacity - the maximum amount of oil recovered by a sorbent either on its initial exposure to the oil/water test system or on subsequent exposures, expressed as grams of oil per gram of sorbent

Water pickup – the weight of water picked up by a sorbent during testing, expressed as grams of water per gram of sorbent

Main Protocol

The protocol used was similar to the original protocol from the 1976 report with slight modifications and expanded to include additional test oils.

Materials and Apparatus

Carber Laboratory Press (0-100 psig Model "C") Haake Temperature Control Unit, Model KT33 Normal Laboratory Apparatus	Test Oil 1: Diesel Test Oil 2: Crude (high sulphur) Test Oil 3: Bunker C Natural Water (lake water) Various sorbents

Test oils were aged by continuous mixing in the presence of air – volatiles were vented via fume hood. Oils were aged for periods of 24 hours (1 day) and 168 hours (7 days).

A water bath using "natural water" with a depth of 10-15cm was maintained at 10°C. A layer of test oil (diesel – 2.5mm thick; crude and Bunker C - 5mm thick) was used during the tests. Additional testing was performed with a thin layer (0.1mm) using the 1-day weathered diesel and crude oils.

A sample of the sorbent was weighed and placed on oil/water layer for 1 hour without mechanical agitation. The test sample was then drained for 5 minutes and weighed. Fluid was then removed from the sorbent using the press (operated at a maximum pressure of 2.82 kg/cm^2) and the sorbent is reweighed.

The recovered liquid is subjected to water content analysis (ASTM D95 or centrifuging). Reusability was determined by repeating the test procedure until the sorbent disintegrated or the recovered oil fell below 50 percent of the initial capacity (up to a maximum of 10 repetitions). Oil was replenished after each test to maintain a constant oil layer thickness. Each sorbent was tested in triplicate.

Calculations:

$$\textit{Initial Capacity} = \frac{(\text{weight of oil, water and sorbent after initial exposure}) - (\text{weight of water recovered}) - (\text{initial sorbent weight})}{\text{Initial weight of sorbent}}$$

$$\textit{Maximum Capacity} = \frac{\text{(maximum weight of oil, water and sorbent} - \text{(weight of water recovered)} - \text{(initial sorbent weight)}}{\text{Initial weight of sorbent}}$$

$$\textit{Water Pickup} = \frac{\text{(weight of water recovered)}}{\text{(initial weight of sorbent)}}$$

For "reuse" calculations, the weight of the sorbent plus oil remaining after pressing was subtracted, rather than the initial dry weight of the sorbent.

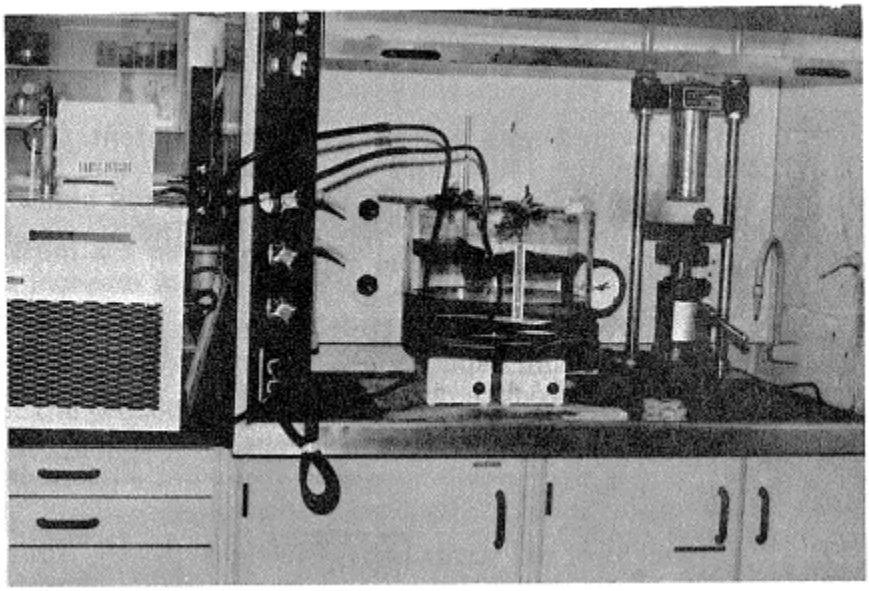


Figure 1 Selection Criteria Update Report 1978, Laboratory Setup for Sorbent Evaluation

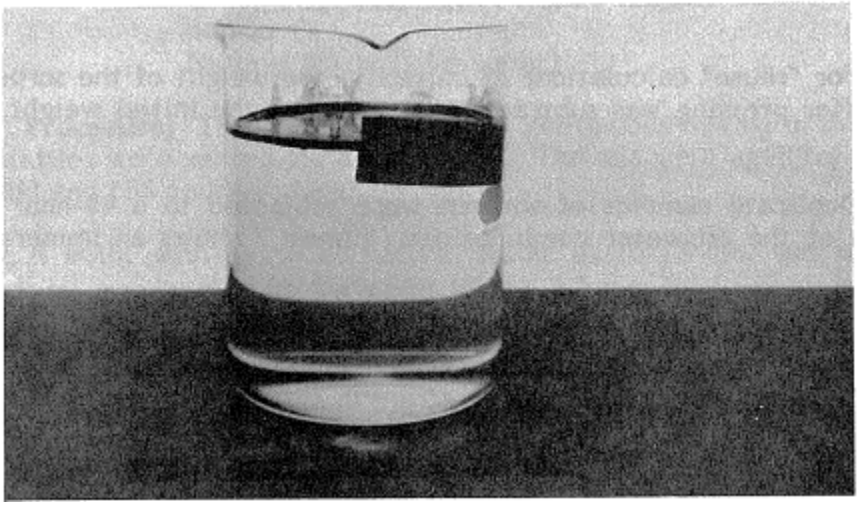


Figure 2 Selection Criteria Update Report 1978, Immersion Testing

Immersion testing for a period of 48 hours was conducted to determine if the sorbent performance would degrade if left for extended periods (material cohesiveness). After 48-hour immersion testing, parameters such as percentage of sorbent still afloat, physical characteristics and integrity of sorbent, strength compared to new sorbent, and any apparent chemical reactions were noted. The report summarizes the results of each individual sorbent with photos of the products and summarizes the individual results.

Appendix B.3 Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: Update II

Details compiled from *Robertson, L. A. (1983). Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: Update II. Ottawa: Environment Canada.*

Summary

A four-part laboratory scale sorbent testing procedure was developed to evaluate upwards of twenty sorbent products. The sorbents were tested in three different petroleum products that had been artificially aged for one and seven days.

Sorbents used in the study

Inorganic Sorbents – none used in the study

Organic Sorbents

Bedex Conwed (modified organic) Conwed Rug	Peat Moss Slikwik

Synthetic Sorbents (polymeric)

Conwed Durable Pads Conwed Viscous Oil Sorbent Graboil Imbiber Beads Leomat Neo Attack Ace	Oil Snare Quickwick Reichhold Foam Spill Control FPD Spill Control PEP 3M Grabbers 3M Sheet

Mixed Sorbents (Organic/Inorganic)

Fiberperl	Solid Solvent

Study Parameters

Three initial quantitative aspects are identified:

Initial Oil Pickup or IOP – the amount of oil picked up by a sorbent on its initial exposure to a pure oil or an oil/water test system, expressed as grams of oil per gram of sorbent.

Maximum Oil Pickup or MOP – the maximum amount of oil recovered by a sorbent either on initial or subsequent exposure to a pure oil or an oil/water test system, expressed as grams of oil per gram of sorbent

Water pickup – the amount of water recovered by a sorbent on exposure to an oil/water test system, expressed as grams of water per gram of sorbent.
One additional parameter was identified

Change in tensile strength – the percentage change in strength of a product as measured by the identified Instron "Tensile Tester".

Main Protocol

The evaluation was broken down into four main tests:

1. Initial tests involving pure oil (oil only) exposure and determining oil recoveries, capability of reuse and changes in tensile strength. This is considered baseline information, not necessarily indicative of field performance.
2. The second test exposes each sorbent to a 1.0mm and a 0.1mm layer of test oil on water. Some indications of expected recovery in field situations may be obtained (when dealing with similar slick thicknesses). Changes in tensile strength could relate to mode of retrieval of sorbents.
3. The third test is the 48-hour immersion test, with shaking. The impacts of long-term exposure to agitation in the presence of oil and water are measured.
4. The fourth test is to determine oil/water preference by evaluating the oleophilicity after exposure to water. Parameters measured include oil pickup, water pickup, and percent of product floating at the end of the test.

Materials and Apparatus

Instron Tensile Tester (Model 1130) Eberbach Tabletop Shaker Normal Laboratory Apparatus	Test Oil 1: Deisel Test Oil 2: Crude Test Oil 3: Bunker C Natural Water (lake water) Various sorbents

Procedures

Pure Oil

Oil bath was maintained at a temperature of 10°C. A sample of the sorbent was weighed, placed on the oil, and left for a period of 15 minutes without mechanical agitation. The sample was then retrieved and drained in a horizontal orientation for 5 minutes and weighed. If not completely saturated, it was returned to the oil bath and saturated with the aid of mechanical agitation. The saturated sorbent was subsequently drained for 5 minutes as before and reweighed.

The sorbent was then pressed at 294.2 kPa for 30 seconds and weighed again. It was then returned to the oil, and the procedure was repeated up to 10 times (maximum) or until the weight of the recovered oil fell below 50% of the IOP.

Tensile tests were performed on a second set of samples after they had been fully saturated. Each sorbent was tested in triplicate.

The calculations used were:

$$\text{IOP} = \frac{(\text{initial weight of sorbent + oil}) - (\text{initial sorbent weight})}{(\text{initial weight of sorbent})}$$

$$\text{MOP} = \frac{(\text{maximum weight of sorbent + oil}) - (\text{initial sorbent weight})}{(\text{initial weight of sorbent})}$$

$$\text{Oil Recovered} = \frac{(\text{weight of sorbent + oil}) - (\text{initial sorbent weight})}{(\text{initial weight of sorbent})}$$

$$\%\ \text{Change in Tensile Strength} = \frac{(\text{initial tensile strength}) - (\text{saturated tensile strength})}{(\text{initial tensile strength})}$$

Oil Layer on Water

A bath with 10 to 15 cm of natural water maintained at 10°C was layered with oil to produce a 1mm thick layer. The use of viscous oil (Bunker C) resulted in discrete oil slicks and target layer thickness could not be achieved. Additional thin slick (0.1mm) testing was performed with 1-day aged diesel.

A sorbent sample was weighed, placed in the oil/water test system at 10°C and left for a period of 15 minutes without mechanical agitation. The sample was then drained in a horizontal position for 5 minutes then weighed. Oil was extracted using a press for 30 seconds at 294.2 kPa. The liquid recovered was subjected to water content determination using either ASTM D95-70 or by centrifuging. This procedure was repeated (up to 10 times) to determine reusability of the sorbent. Testing stopped if the sorbent broke apart (disintegrated) or if the oil recovery fell below 50 percent of the IOP. Oil was replenished after each test to maintain a constant oil layer thickness. Tensile tests were performed on a duplicate set of samples.

Calculations for these tests were:

$$\text{IOP} = \frac{(\text{weight of sorbent after initial exposure}) - (\text{weight of water picked up}) - (\text{initial weight of sorbent})}{(\text{initial weight of sorbent})}$$

$$\text{MOP} = \frac{(\text{maximum weight of sorbent + liquid}) - (\text{weight of water picked up}) - (\text{initial weight of sorbent})}{(\text{initial weight of sorbent})}$$

$$\text{Oil Recovered} = \frac{(\text{weight of sorbent + liquid}) - (\text{weight of sorbent after previous pressing})}{(\text{initial weight of sorbent})}$$

$$\text{Water Picked Up} = \frac{(\text{weight of water in recovered oil}) \times (\text{weight of liquid picked up})}{(\text{initial weight of sorbent}) \quad (\text{weight of liquid recovered})}$$

$$\%\ \text{Change in Tensile Strength} = \frac{(\text{initial tensile strength}) - (\text{saturated tensile strength})}{(\text{initial tensile strength})} \times 100$$

48-Hour Immersion Test

500mL of water plus sufficient oil to produce a 1mm layer of oil was added to a 1L wide-mouth bottle. Oil was added when the bottle was placed on its side. A pre-weighed sample of sorbent was then added to the bottle, which was placed on the shaker table. The shaker table was adjusted to oscillate at 60 cycles/min over an amplitude of 4cm for 48 hours. At the end of the test, all floating sorbent was removed and weighed. The water content of any liquid sorbed was determined using ASTM D95-70 distillation method. Tensile testing was performed on duplicate sets of samples. Any sorbent not floating at the end of the test was removed, cleaned (using suitable solvent), dried, and weighed to determine the percentage of sorbent remaining floating. Sorbent testing was performed in triplicate with the average values being used to calculate MOP, water pickup, % sorbent floating, and % change in tensile strength.

The calculations were as follows:

$$\text{MOP} = \frac{(\text{weight of sorbent + liquid}) - (\text{weight of water picked up}) - (\text{initial weight of sorbent})}{(\text{initial weight of sorbent})}$$

$$\text{Water Pick Up} = \frac{(\text{weight of water picked up})}{(\text{initial weight of sorbent})}$$

$$\%\ \text{Sorbent Floating} = \frac{(\text{initial weight of sorbent}) - (\text{dry weight of sorbent not floating})}{(\text{initial weight of sorbent})} \times 100$$

$$\%\ \text{Change in Tensile Strength} = \frac{(\text{initial tensile strength}) - (\text{tensile strength after sorbent test})}{(\text{initial tensile strength})} \times 100$$

Oil/Water Preference

A pre-weighed sorbent sample is added to a 1L bottle (wide mouth) containing 500 mL of water. The bottle is placed on its side on a shaker table adjusted to oscillate at 60 cycles/min over an amplitude of 4cm. The bottle was shaken for 30 minutes and observations noted. The bottle was then removed, and a 5mL aliquot of the test oil was added by pipette (the tip of the pipette was placed just below the surface of the water). The jar was returned to the shaker table for a further 30 minutes of shaking. The oil additions were repeated until a film of oil was observed on the water surface. The volume of oil added was noted and the sorbent material was removed from the jar, allowed to drain for 5 minutes, and then weighed. As previously stated, water content was determined using ASTM D95-70. Any non-floating sorbent was removed, cleaned and dried then weighed to determine the percentage of sorbent still floating. Samples were tested in triplicate, with average results used to calculate MOP, water pickup, and % sorbent floating

The calculations were as follows:

$$\text{MOP} = \frac{(\text{weight of sorbent + liquid}) - (\text{weight of water picked up}) - (\text{initial weight of sorbent})}{(\text{initial weight of sorbent})}$$

$$\text{Water Pick Up} = \frac{(\text{weight of water picked up})}{(\text{initial weight of sorbent})}$$

$$\text{\% Sorbent Floating} = \frac{(\text{initial weight of sorbent}) - (\text{dry weight of sorbent not floating})}{(\text{initial weight of sorbent})} \times 100$$

Appendix B.4 Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: Update III

Details compiled from *Enviroclean. (1985). Selection Criteria and Laboratory Evaluation of Oil Spill Sorbents: Update III. Ottawa: Environment Canada.*

Summary

Nineteen commercially available oil spill sorbents were tested using three petroleum products and two hydrocarbon solvents. The petroleum products were aged for one and seven days. The sorbents were evaluated based upon initial and maximum capacity, water pickup and the number of reuses in each test liquid.

Testing demonstrated higher capacities and greater reuse potential for the synthetic sorbents versus the organic materials in this series of evaluations. Decreasing the oil layer thickness decreases the sorbent capacity and usually increases the water pickup.

Sorbents used in the study

Inorganic Sorbents

Muck-up*	

*Did not float, was not tested further.

Organic Sorbents

Conwed Sorbent Blanket (modified organic) Oclansorb (modified peat moss) Seaclean	Verdyol Zugol

Synthetic Sorbents

AB & B Pads AB & B Oil Swab Alsorb Conwed Petro Mesh Conwed D-Sorbent Pads Graboil Hazorb	Quick Wick S.C.C.-FPD S.C.C.-PEP S.C.C.-SPD S.P.C. Pads 3M Type 156 Pads

Study Parameters

Three quantitative aspects are indicated below:

Initial Capacity (g test liquid/g sorbent): the amount of oil (test liquid) picked up by a sorbent on initial exposure to a test liquid.

Maximum Capacity (g test liquid/g sorbent): the maximum amount of oil (test liquid) picked up by a sorbent on either the initial exposure to a test liquid or in subsequent reuse tests.

Water Pickup (g water/g sorbent): The weight of water picked up by a sorbent during a test.

Main Protocol

The procedure was similar to the previous tests (Update II) with the main modification being the use of a cold room to maintain the required test temperature of 10°C.

Materials and Apparatus

Carver Laboratory Press (Model C) Normal Laboratory Apparatus and Glassware	Test Oil 1: Diesel Test Oil 2: Crude Oil (high sulphur) Test Oil 3: Bunker "C" Test Solvent 1: toluene (technical) Test Solvent 2: cyclohexane (technical) Tap Water (dechlorinated with sodium thiosulphate)

Procedure

Test liquids (oils) were weathered by continuous stirring in a fume hood for the required period of one day and seven days. Test cells containing water at a depth of approximately 20cm were maintained at 10°C. Test liquids were layered on top at their prescribed thickness. Pre-weighed sorbent samples are placed on the test liquid and left standing for one hour without agitation. The sorbent samples are removed and allowed to drain for several minutes (the actual time is not indicated, but possibly 5 minutes to match previous testing). The sorbed fluid was pressed out of the sample (maximum pressure 40 psig) and the sample was reweighed.

Water content of the recovered fluid was determined by centrifuging. To determine reusability, each sorbent sample was repeatedly tested until it disintegrated (broke apart) or until the test liquid recovered dropped below 50 percent of the initial capacity or until a maximum of 10 repetitions. Test liquids were replenished after each test to maintain a constant starting layer thickness. The sorbents were tested in triplicate with the average of the three samples being used for each parameter calculated.

Calculations

$$\textit{Initial Capacity} = \frac{\text{(weight of test liquid, water and sorbent after initial exposure)} - \text{(weight of water recovered)} - \text{(initial sorbent weight)}}{\text{Initial weight of sorbent}}$$

$$\textit{Maximum Capacity} = \frac{(\text{maximum weight of test liquid, water and sorbent}) - (\text{weight of water recovered}) - (\text{weight of sorbent used})}{\text{Initial weight of sorbent}}$$

$$\textit{Water Pickup} = \frac{(\text{weight of water recovered})}{(\text{initial weight of sorbent})}$$

Two samples of each sorbent were also subjected to 48-hour immersion testing in each test liquid. Observations such as the amount of sample still floating, and the integrity of the sample were made.

Appendix B.5 Selection Criteria and Laboratory Evaluation of Oil spill Sorbents: Update IV

Details compiled from *S.L. Ross Environmental Research Limited. (1991). Selection Criteria and Laboratory Evaluation of Oilspill Sorbents Update IV. Ottawa: Environment Canada.*

Summary

Sixteen commercially available sorbents were tested in three different petroleum products and two hydrocarbon solvents. The petroleum products were aged 1 and 7 days. Sorbents were evaluated in terms of initial and maximum capacities, water pickup, and reuse potential. Synthetic sorbents generally exhibited the highest capacities and potential for reuse than the organic sorbents tested. Decreasing the test liquid layer thickness led to lower sorbent capacities and higher water pickup ratios to be measured.

Sorbents used in this study

Inorganic Sorbents

Clay	

Organic sorbents

Alfob	Oclansorb
CCD Wood Chips	Sawdust
Cork	Wool

Synthetic

Alsorb II	Hazorb
Eco Oil Sorbent	Matasorb
E100 (Ergon)	Pig Skimmer
Foam “X”	Slikwik (Anderson S100)
Graboil	

Study Parameters

This study uses a similar methodology and approach that was used in previous studies. Sorbent samples were evaluated for their ability to sorb test liquids from a test liquid/water interface. A long-term exposure test was also conducted.

There are four quantitative aspects identified:

Initial Capacity (g test liquid/g sorbent): amount of test liquid a sorbent picked up upon initial exposure to the test liquid-water system.

Maximum Capacity (g test liquid/g sorbent): the maximum amount of test liquid picked up by a sorbent on either the initial exposure to the hydrocarbon – water system or on subsequent reuse tests.

Water Pickup (g water/g sorbent): the quantity of water picked up by a sorbent during a test. Maximum Water Pickup (g water/g sorbent): the maximum amount of water picked up either on initial exposure to the hydrocarbon-water system or on subsequent reuse tests.

Materials and Apparatus

Normal Laboratory Apparatus and Glassware	Test Oil 1: Diesel Test Oil 2: Crude Oil (high sulphur) Test Oil 3: Bunker "C" Test Solvent 1: toluene (technical) Test Solvent 2: cyclohexane (technical) Tap Water

Procedure

Test oils were aged for periods of one and seven days, except diesel which was only weathered one day (expected to be similar to 7-day evaporative weathering). Toluene and cyclohexane were used as received, with no weathering. Two baths containing multiple test cells were maintained at 10°C. Test liquids were layered on water and pre-weighed sorbent samples were placed in the test cells and left for 30 minutes. A cover over the baths was used to minimize evaporation during the tests. The samples were not subjected to agitation; however, the samples were turned over once. At the end of the sorption test the samples were drained in a horizontal orientation for 5 minutes and re-weighed. The water content of the recovered fluid was determined by liquid extraction using toluene. Volumes were measured using a graduated cylinder.

Thin-film tests were performed using a 0.1mm layer of test liquid on water. Reuse experiments used a slick thickness of 2.5mm for lighter test fluids (toluene, cyclohexane, and diesel) and 5.0mm for heavier test fluids (crude oil, Bunker C). To determine reusability, each sorbent sample was repeatedly tested until it disintegrated (broke apart) or until the test liquid recovered fell below 50% of the initial capacity (to a maximum of ten reuses for cyclohexane and toluene, and five reuses for diesel, crude and Bunker C). The test liquids were replenished after each trial to maintain the starting layer thickness.

Oil and water were recovered from the sorbents using a hydraulic press and various plates which were altered to suit each test fluid. Additionally, the plate was heated when processing Bunker C laden sorbents to facilitate recovery. Tests were performed in triplicate with the average used for each parameter calculated.

A separate qualitative was performed exposing two samples of each sorbent to each test liquid for a period of 48 hours under quiescent conditions. Observations were made during and at the end of the test, defining parameters such as the amount of the sample still afloat, integrity of the sample, and any apparent changes in the physical characteristics of the sample.

Calculations

$$\textit{Initial Capacity} = \frac{\text{(weight of test liquid, water and sorbent after initial exposure)} - \text{(weight of water recovered)} - \text{(initial sorbent weight)}}{\text{Initial weight of sorbent}}$$

$$\textit{Maximum Capacity} = \frac{\text{(maximum weight of test liquid, water and sorbent} - \text{(weight of water recovered)} - \text{(weight of sorbent used)}}{\text{Initial weight of sorbent}}$$

$$\textit{Water Pickup} = \frac{\text{(weight of water recovered)}}{\text{(initial weight of sorbent)}}$$

Appendix B.6 Laboratory Evaluation of Sorbents

Details compiled from *Counterspil Research Inc. (1999). Laboratory Evaluation of Sorbents. Ottawa, ON: Environment Canada.*

Summary

A sorbent testing suite was performed in March 1999. It focused on testing various structural formats of sorbents for commonly spilled liquids including hydraulic oil, diesel, and glycol. Mat type sorbents as well as particulates were evaluated. (Counterspil Research Inc., 1999)

Sorbents used in the study

Various types/formats of sorbent pads were collected from manufacturers and suppliers including

Up to 3 thicknesses or weights Melt-blown Sonic-bonded	Air-laid Outer sheet included and/or laminated.

Sorbent pads (19 oil-only, 5 universal, and 1 chemical) were obtained from different companies including:

Ergon Spilltech Matasorb SPC	Devcon New Pig Gem Products Cansorb

Particulate products (Type II sorbents) were also evaluated including:

Leaksorb Fasorb Absorbent W Lite-Dri	Environ-Dri Absorbent 7640 Cansorb

Laboratory Set-up

Normal Laboratory Apparatus Balance with 1200.0 g capacity Graduated cylinders Disposable beakers Disposable weighing trays Plastic trays (30cm x 28cm x 6 cm) Metal tongs Scissors	Hydraulic oil (Nuto H32) in 25 L container Diesel Glycol (MotoMaster antifreeze) Tensile Apparatus (see below)

Procedures Summary

Sorbent samples (Type I) were cut (method is not specified) from a sorbent pad (in triplicate) and weighed. Samples were placed on a layer of pure test liquid until saturated (no explicit time given). Each sample was retrieved and held above the tray for 20 seconds before being weighed. This was repeated for each test fluid. A fourth sample of each sorbent was immersed in water for approximately 3 hours, shaken, and weighed before and after each test to determine water pick-up.
Durability testing – for each oil-only sorbent pad, a single sample (2.5cm strip) was placed in the test apparatus (see below). Water was added into a 2L container until the sorbent completely tore. The final water/container weight was recorded.

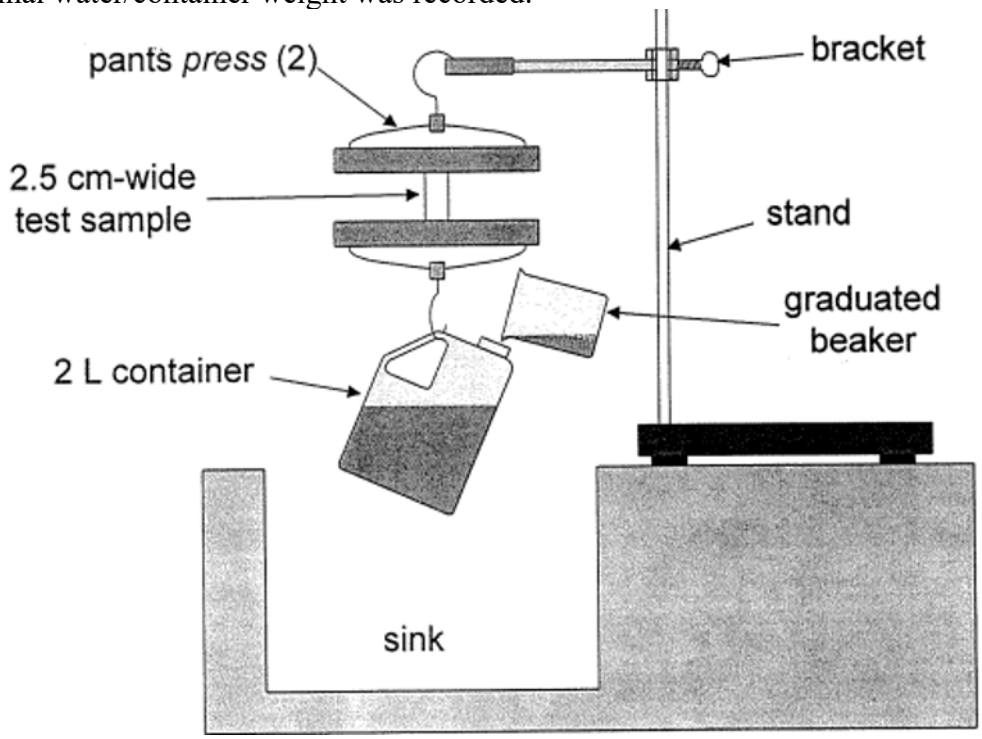


Figure A-1 Lab Evaluation of Sorbents 1999, Tensile Test Apparatus

Appendix C Temporary BSEE/Ohmsett Protocol

Details compiled from *Coolbaugh, G., & McKinney, K. (2021). Application of the Field Scale Test Protocol for Type I Sorbents Recovering Oil on Water`. Proceedings of thee Forty-third AMOP Technical Seminar (pp. 112-131). Ottawa: Environment and Climate Change Canada.*

Test apparatus was redesigned and fabricated over multiple testing phases since 2018 to accommodate typical sorbent pads at their full size. Data resulting from testing performed with a selection of various sorbent materials revealed significant performance variances, as well as highlighted unique behaviors and characteristics.

C.1 Performance Characteristics of Interest

A number of performance characteristics are described within the protocol and are identified below:

- *Maximum Oil Capacity* – volumetric measurement of oil adsorbed by a sorbent where neither contact with additional oil nor a longer exposure time would result in increasing the amount sorbed.
- *Oil Adsorbed* - A volumetric measurement of oil sorbed by a sorbent when deployed onto an oil slick floating on water, taken at prescribed time intervals.
- *Water Update* - A volumetric measurement of water adsorbed by a sorbent when deployed onto an oil slick floating on water, taken at prescribed time intervals.
- Additional secondary performance characteristics were also identified:
- *Oil Viscosity Range* - Consideration given towards acceptable oil recovery by the sorbent over a range of viscosities.
- *Buoyancy* - The ability of the sorbent to remain afloat indicated typically by a portion of the sorbent having freeboard.
- *Field Retrieval* - A measure of sorbent strength indicating the ability to retrieve it from a spill when fully saturated.
- *Manual Oil Recovery* - A volumetric measurement of oil manually recovered from a sorbent by way of a wringer roller following adsorption test. Note: this performance characteristic was not initially considered during the test protocol development but was added for the testing during August of 2019.

C.2 Test Apparatus

Two test cells were designed to accommodate typical commercially available sorbent sizes (up to 0.92m x 0.92m)

Each test apparatus was comprised of:

1. Fluid tray: the tray to contain test fluids was designed and fabricated using 0.48 cm (3/16 in) aluminum sheet material 102 cm (40 in) square x 20 cm (8 in) deep. Each tray had a bottom mounted 3.2 cm (1¼ in) drainpipe to facilitate draining and cleaning. Guide rods

along the side walls of each tray allowed for a controlled removal of a sorbent sample from the test fluid by way of a hoist.

2. Sorbent Support Rack: The sorbent support rack was designed to support the sorbent while it was lowered into the test fluid and to extract the sorbent from the test fluid after exposure. Sorbents hung in a vertical orientation demonstrated the effects of gravity as fluid moved down through the sorbent and drained out the bottom corner, potentially leading to artificially low fluid volume retention when testing full or meso-scale samples. When supported horizontally, the sorbent reached a point of no dripping in a relatively short time. To minimize the effects of gravity and to better facilitate a reproducible lift method, the use of a horizontal support rack was adopted. The support rack was designed to be lightweight and horizontally supportive of the sorbent while minimizing contact with the sorbent. Each support rack was fabricated using 1.9 cm (3/4 in) x 0.32 cm (1/8 in) aluminum angle topped with a thin gauge wire mesh.

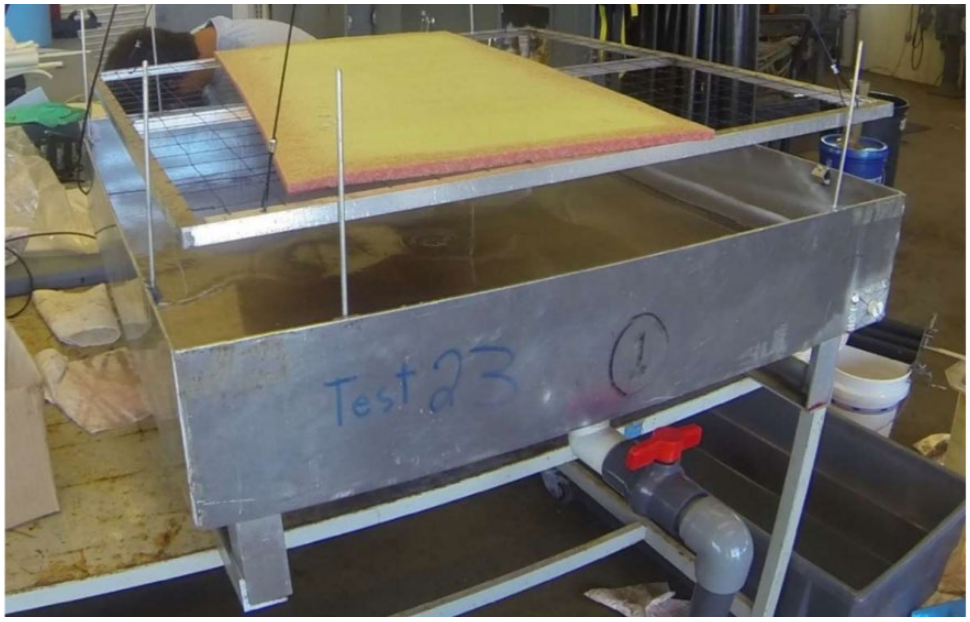


Figure 1 Application for Field Scale Test Protocol for Type I Sorbents 2021, Test cell with support rack

3. Load Cell: The weight measurement device, key to obtaining accurate test data, was selected for its stated measurement accuracy within the desired weight range. A 0-45 kg S-Beam load cell with an accuracy of 0.02 % (0.01 kg) with an analog to digital converter was used in conjunction with a continuous data logging application.

Figure 2 Application for Field Scale Test Protocol for Type I Sorbents 2021, Load cell (lbs)

4. Eccentric Drive: A variable speed motor was used to move the fluid tray back and forth to create surface energy. A right-angle gear reduction allowed for a controlled speed of 30-80 cycles per minute. The fluid tray supported on the linkage was able to freely move respective to the motor. The drive roller with an eccentricity of 1.3 cm (½ in) provided a 2.5 cm (1 in) overall stroke distance. During testing, speed was maintained at 60 cycles per minute. This setup attained surface waves approximately 1.3 cm (½ in) high with a period of 15.2 cm (6 in).

Figure 3 Application for Field Scale Test Protocol for Type I Sorbents 2021, Variable Speed Motor drive unit

C.3 Test Fluid Selection

Test fluids aligning to the viscosity ranges defined in ASTM F726-17(2024) are selected

Oil Type	*Viscosity Range*	*Density Range*	*Example*
Light	1 to 10 cP	0.820 to 0.870 g/cm^3	Diesel fuel, mineral oil
Medium	200 to 400 cP	0.860 to 0.970 g/cm^3	Crude oil, canola oil, mineral oil
Heavy	1500 to 2500 cP	0.930 to 1.000 g/cm^3	Bunker C or residual fuel, mineral oil
Weathered	8000 to 10 000 cP	0.930 to 1.000 g/cm^3	Emulsified crude oil, mineral oil

Figure 4 Oil types from ASTM F726-17

C.4 Detailed Steps

Support Rack

The use of a support rack introduces possible errors in measurements as the rack itself may be wetted by the oil test liquids (and/or water) and artificially increase the resultant measurements. The effects of fluid adhering to the sorbent support rack are addressed by the following steps:

1. Obtain sorbent sample pre-test weight (typically measured on a dry support rack)
2. Immerse the support rack without sorbent sample into the test fluid for approximately two minutes
3. Raise the rack from the test fluid while recording load cell readings and obtain a "wet rack" tare weight once a stable weight is reached
4. Place sorbent sample on support rack and document net weight as initial weight. This procedure provides repeatable results and allows for continued minimization of external disruption or interference during the testing process.

Sorbent Maximum Oil Capacity Test Method in Oil Only Environment

Based on testing with a wide variety of sorbents, a time frame of 15 minutes was chosen for a sorbent's first exposure to oil, after which the sorbent support rack with sorbent was raised, allowed to drip while weight measurements were collected, and then redeployed onto the slick. The sorbent was then allowed to adsorb for 45 minutes after which it was raised, and the same process followed for dripping and collecting weight measurements. For the third increment until the end of the test, the time for oil exposure was one hour. This process was repeated until the sorbent did not adsorb additional oil within the increment. This required that the sorbent be exposed to the oil for a minimum of two increments and a minimum of one hour.

The test steps are listed below:

1. Perform load cell calibration
2. Measure sorbent dimensions
3. Obtain and record pre-test net weight of the rack and sorbent sample (initial weight)
4. Place sorbent sample onto the support rack and lower rack, allowing sorbent to float freely on oil surface
5. Start timer and video documentation

6. After 15 minutes begin load cell data collection and raise support rack with sorbent.
7. Monitor change in gross weight and observe for oil dripping to tapering off to point of no dripping. Calculate the weight-of-oil to weight-of-sorbent ratio (oil/sorbent ratio).
8. Lower sorbent back into fluid. After 45 minutes begin load cell data collection and raise support rack with sorbent. Repeat step 7. If the second measurement for oil/sorbent ratio repeats, that serves as confirmation the sorbent has reached its maximum capacity. If the measurement does not repeat, then the process is repeated using one-hour intervals until the oil/sorbent ratio repeats.

Weight measurements were taken continuously once the sorbent was raised from the oil and allowed to drip. Initial measured gross weight of the sorbent and rack was erroneously high due to free oil dripping from the support rack and sorbent and diminished until it reached a point of little change over time. This data can be plotted as a drip curve, either as net oil weight versus time, or as an oil/sorbent ratio versus time. Measurements obtained were later used to determine the actual weight or volume of oil adsorbed. Important to note is how much the oil/sorbent ratio changes over drip time. Reporting of oil/sorbent ratio should include additional information about drip time or should be presented as a drip curve.

Sorbent Water Uptake Test in Oil on Water Environment

This portion of the protocol is to quantify water sorbed when the sorbent is exposed to a relatively thin oil slick on water. The approach was designed to expose test sorbents to a volume of oil less than the predetermined maximum capacity, thereby ensuring contact with both oil and water. This condition allowed for the potential of a sorbent to adsorb water, possibly by way of wicking action or inferior hydrophobic properties. Once values were obtained for the weight of oil and water adsorbed, the amount of each fluid adsorbed could be represented as a ratio, hence Oil to Water Ratio.

One half of the maximum oil capacity determined by the Maximum Oil Capacity test method described above was determined to provide a reasonable oil volume while leaving sorbent capacity for water uptake. After deploying a half-max volume of oil onto the water surface the sorbent was lowered onto the fluid surface for a prescribed one-hour residence time, removed, and allowed to drip until the point of no dripping while weight measurements were recorded. The fluid remaining in the tray was then decanted of free water and the remaining oil was recovered and measured. Recorded values for the oil volume initially dispensed and the volume recovered allowed the volume remaining in the sorbent to be calculated.

The specific test steps are listed below:

1. Place 2.5 cm of water into the tray
2. Place the appropriate volume of oil on the water surface (half of the sorbent's maximum capacity)
3. Obtain and record pre-test net weight of the rack and sorbent sample (initial weight)
4. Place the sorbent on the sorbent support rack and lower it into the fluid for one hour
5. Raise the sorbent support rack and allow it to drain to the point of no drip while recording weight

6. Swing the sorbent and sorbent support rack away from the fluid tray and dispose of the sorbent (to prevent any fluid from dripping back into the fluid tray during sorbent removal)
7. Decant free water from the fluid tray
8. Collect the oil and remaining water into graduated cylinders and allow it to separate
9. Calculate weight of oil recovered from tray by converting volume to weight using specific gravity
10. Subtract recovered oil weight from initial weight of oil dispensed to obtain weight of oil removed by sorbent
11. Subtract weight of oil in sorbent and sorbent tare weight from post-test net sorbent and fluid weight to obtain weight of water in sorbent

Through testing it became apparent that applying a sorbent to a quiescent surface of oil and water did not promote water uptake. To further enhance the method and provide conditions more closely mimicking field use, comparative tests were performed with the addition of surface energy. The test parameters and method were replicated in each case, with the only change being the introduction of energy. In each case, the amount of water retained in the sorbents increased significantly for the tests, including energy. The test protocol thus currently includes the use of energy for this test method.

Sorbent Water Uptake Test in Oil on Water Environment "Update"

Significant changes have been introduced to the protocol. Changes included the amount of oil initially added to the test tray, sorbent exposure times, and drip times. For these tests, the volume of oil used was determined based on ASTM F726-17 testing rather than sorbent maximum capacity tests. F726-17 testing produced maximum oil capacity which was scaled relative to the sorbent sample size. The relative amount of oil was then added on water in the fluid tray to create a surface slick. The sorbent was applied to the water surface, allowed to adsorb incrementally for a total time of 60 minutes, and weighed after a 30 second drip time.

The revised protocol used the following steps:

1. Perform load cell calibration
2. Measure sorbent dimensions
3. Obtain and record pre-test net weight of the rack and sorbent sample
4. Place sorbent sample onto rack and lower until sample floats freely on the fluid surface
5. Begin timer and video or time-lapse documentation
6. After 15 minutes, load cell data collection and withdraw support rack and sorbent for 30 seconds before re-immersing sample in test fluid
7. After 30 minutes, load cell data collection and withdraw support rack and sorbent for 30 seconds before re-immersing sample in test fluid
8. After 60 minutes begin load cell data collection and withdraw support rack and sorbent for 30 seconds concluding submersion
9. Swing the sorbent and sorbent support rack away from the fluid tray and dispose of the sorbent (to prevent any fluid from dripping back into the fluid tray during sorbent removal)

10. Decant free water from the fluid tray
11. Collect the oil and remaining water into graduated cylinders and allow it to separate
12. Calculate weight of oil recovered from tray by converting volume to weight using specific gravity
13. Subtract recovered oil weight from initial weight of oil dispensed to obtain weight of oil removed by sorbent
14. Subtract weight of oil in sorbent and sorbent tare weight from post-test net sorbent and fluid weight to obtain weight of water in sorbent

One advantage of this revised test method is that it allows the ability to test without first conducting the Sorbent Maximum Oil Capacity Test Method in Oil Only Environment protocol; however, it does require that portions of ASTM F726-17 be performed to determine the restricted oil volume to use.

Field Retrieval

The ability to retrieve a fully saturated sorbent intact was determined to be an important function from an operational perspective and a protocol was devised to measure the load at which a sorbent pad might fail during typical field retrieval. Discussions with responders identified an ordinary pitchfork as a commonly used tool to retrieve sorbents. For this reason, the retrieval test was designed to impose the most stringent yet practical approach of retrieving fully saturated sorbents by employing a single typical pitchfork prong formed into a hook. The hook was suspended on the load cell.

The following steps formed this protocol:

1. A sorbent that had undergone maximum oil adsorption was placed on the fluid tray and lowered into test oil.
2. The sorbent was allowed five minutes to re-saturate.
3. The sorbent was attached to the pitchfork prong by puncturing it 15.2 cm (6 in) inboard shortest edge.
4. With load cell operating, the sorbent was raised from the oil bath.
5. The sorbent passed if it remained stable on the prong and failed if it tore from the prong.
6. Sorbents that passed were further subjected to a downward force until failure was reached and the force was recorded.

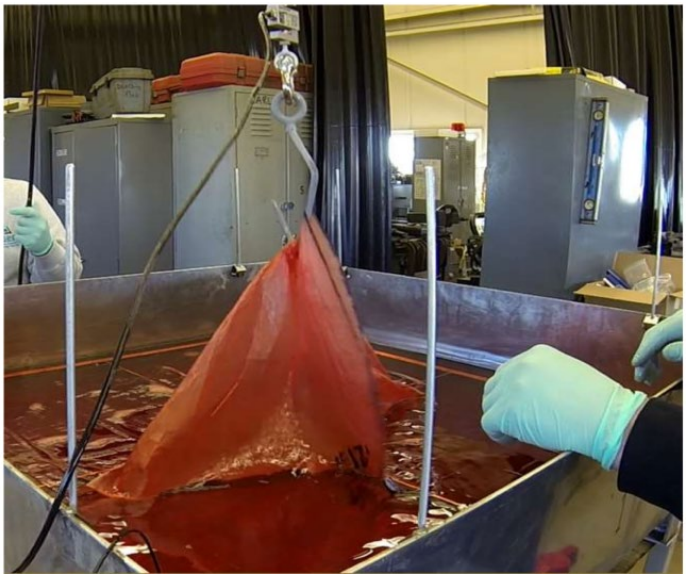


Figure 5 Application for Field Scale Test Protocol for Type I Sorbents 2021, Single-ply sorbent retrieval test

Manual Oil Recovery

A manual roller system was employed to evenly apply pressure to saturated sorbent samples to recover most of the oil sorbed during the saturation test. Performing a mass balance on the sorbent sheet can allow the oil remaining in the sorbent to be determined.

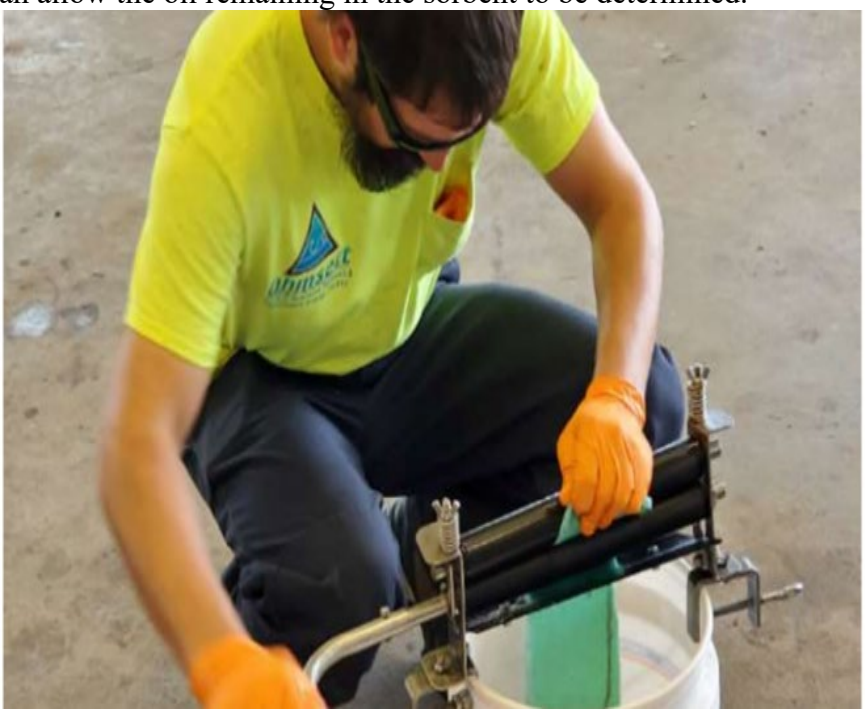


Figure 6 Application for Field Scale Test Protocol for Type I Sorbents 2021, Manual Roller System

Appendix D: Abbreviations and Acronyms

[Short Form]	[Long Form]
BSEE	Bureau of Safety and Environmental Enforcement
CO	Contracting Officer.
COR	Contracting Officer's Representative
CSE	Council of Science Editors
DM	Departmental Manual
DOI	Department of the Interior
MMA	Marine Mineral Administration (formerly BSEE)
NRS	National Response System
NRTL	National Technical Reports Library
OSPD	Oil Spill Preparedness Division
OSRR	Oil Spill Response Research
ppi	Pixel Per Inch
PV	Preparedness Verification
Template	Oil Spill Preparedness Division Report Template

Department of the Interior (DOI)
The Department of the Interior protects and manages the Nation's natural resources and cultural heritage; provides scientific and other information about those resources; and honors the Nation's trust responsibilities or special commitments to American Indians, Alaska Natives, and affiliated island communities.

Marine Minerals Administration (MMA)
The mission of the Bureau of Safety and Environmental Enforcement works to promote safety, protect the environment, and conserve resources offshore through vigorous regulatory oversight and enforcement.

MMA Oil Spill Preparedness Program
MMA administers a robust Oil Spill Preparedness Program through its Oil Spill Preparedness Division (OSPD) to ensure owners and operators of offshore facilities are ready to mitigate and respond to substantial threats of actual oil spills that may result from their activities. The Program draws its mandate and purpose from the Federal Water Pollution Control Act of October 18, 1972, as amended, and the Oil Pollution Act of 1990 (October 18, 1991). It is framed by the regulations in 30 CFR Part 254 – *Oil Spill Response Requirements for Facilities Located Seaward of the Coastline*, and 40 CFR Part 300 – *National Oil and Hazardous Substances Pollution Contingency Plan*. Acknowledging these authorities and their associated responsibilities, MMA established the program with three primary and interdependent roles:

- Preparedness Verification,
- Oil Spill Response Research, and
- Management of Ohmsett - the National Oil Spill Response Research and Renewable Energy Test Facility.

The research conducted for this Program aims to improve oil spill response and preparedness by advancing the state of the science and the technologies needed for these emergencies. The research supports the Bureau's needs while ensuring the highest level of scientific integrity by adhering to MMA's peer review protocols. The proposal, selection, research, review, collaboration, production, and dissemination of OSPD's technical reports and studies follows the appropriate requirements and guidance such as the Federal Acquisition Regulation and the Department of Interior's policies on scientific and scholarly conduct.