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Standard Test Method for Laboratory Oil Spill Dispersant Effectiveness Using the Swirling Flask¹

This standard is issued under the fixed designation F2059; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the procedure to determine the effectiveness of oil spill dispersants on various oils in the laboratory. This test method is not applicable to other chemical agents nor to the use of such products or dispersants in open waters.

1.2 This test method covers the use of the swirling flask test apparatus and does not cover other apparatuses nor are the analytical procedures described in this report directly applicable to such procedures.

1.3 The test results obtained using this test method are intended to provide baseline effectiveness values used to compare dispersants and oil types under conditions analogous to those used in the test.

1.4 The test results obtained using this test method are effectiveness values that should be cited as test values derived from this standard test. Dispersant effectiveness values do not directly relate to effectiveness at sea or in other apparatuses. Actual effectiveness at sea is ~~dependent~~dependent on sea energy, oil state, temperature, salinity, actual dispersant dosage, and amount of dispersant that ~~enters~~interacts with the oil.

1.5 The decision to use or not use a dispersant on an oil should not be based solely on this or any other laboratory test method.

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

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~safety, health, and health~~environmental~~ practices and determine the applicability of regulatory limitations prior to use.*

1.8 *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:²

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 F20.13 on Treatment.

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² ~~The normal operating temperature is 20°C (± 1°C). An alternate temperature may be selected based on test requirements.~~ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* on test requirements, volume information, refer to the standard's Document Summary page on the ASTM website.

[F3251 Test Method for Laboratory Oil Spill Dispersant Effectiveness Using the Baffled Flask](#)

3. Terminology

3.1 Definitions:

3.1.1 effectiveness, *n*—the capability of producing a desired result – which in this case is the dispersion of oil into water.

3.1.1.1 Discussion—

Effectiveness is given here as the percent of oil dispersed into the water column as a result of dispersant action and energy.

4. Summary of Test Method

4.1 Dispersant is pre-mixed with oil and placed on water in a test vessel. The test vessel is agitated on a moving table shaker. At the end of the shaking period, a settling period is specified and then a sample of water taken. The oil in the water column is extracted from the water using a dichloromethane solvent and analyzed using gas chromatography.

4.2 The extract is analyzed for oil using a gas chromatograph equipped with a flame ionization detector, (GC-FID). Quantification is by means of the internal standard method. Effectiveness values are derived by comparison with a calibrated set of effectiveness values obtained at the same time and by the same method.

5. Significance and Use

5.1 A standard test is necessary to establish a baseline performance parameter so that dispersants can be compared, a given dispersant can be compared for effectiveness on different oils, and at different oil weathering stages, and batches of dispersant or oils can be checked for effectiveness changes with time or other factors. This test method provides a test at low mixing energy that is useful for discriminating subtle changes in effectiveness between variables when dispersant efficacy is high. A higher energy test alternative is the Baffled Flask (Test Method F3251).

5.2 Dispersant effectiveness varies with oil type, sea energy, oil conditions, salinity, and many other factors. Test results from this test method form a baseline, but are not to be taken as the absolute measure of performance at sea. Actual field effectiveness could be more or less than this value.

5.3 Many dispersant tests have been developed around the world. This test has been developed over many years using findings from world-wide testing to use standardized equipment, test procedures, and to overcome difficulties noted in other test procedures.

6. Interferences and Sources of Error

6.1 Interferences can be caused by contaminants, particularly residual oil or surfactants in solvents, on glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in gas chromatograms. All glassware must be thoroughly cleaned. The cleaning process includes thorough rinsing with dichloromethane to remove the oil, followed by rinsing three times each with tap water, purified water (reverse osmosis), osmosis or similar, and acetone. Once cleaned, precautions must be taken to minimize contact of the glassware with surfactants to prevent undesired interferences.

6.2 Dispersant effectiveness is very susceptible to energy levels. Table top shakers generally start and stop slowly. Shakers that start motion rapidly and stop suddenly impart a high energy to the system and thus cause more dispersion than would be the case with a normal shaker. Furthermore, this variation would not be repeatable. The shaker table used should be observed for rapid movements or stops to ensure that it is usable for these tests. The rotational speed of the shaker should be ~~checked~~ verified with a tachometer ~~every week~~ periodically.

6.3 The Erlenmeyer flasks used in this test are tapered and the energy level varies with the amount of fill.

6.4 The output is highly sensitive to the volume of oil, water, and extractant delivered. All pipets and dispensers should be calibrated frequently and verified daily.

6.5 The use of positive displacement pipets is mandatory for all controlled volumes of microlitre quantities. Use of volume displacement pipets will result in erroneous results due to the viscosity of the dispersants and oils, the variable viscosity of the oils to be tested (some semi-solid), and the density of dichloromethane.

6.6 The order of addition of the dispersant and oil has effects on the accuracy of results, as the dispersant may interact with the vessel walls if added first, thereby reducing the quantity available in the premix. It is therefore important to add oil to the vessel first, and add the dispersant directly to the oil. A second addition of oil is suggested simply because it is easier to control a large volume of oil than a minute volume of dispersant when attempting to achieve a specific ratio of 25±1:1.25 (dispersant:oil).

6.7 Following surfactant addition, vigorous mixing is required to thoroughly homogenize the sample. Sharp, manual strokes are suggested for light oils, while very heavy oils may require stirring with a clean glass rod or spatula.

6.8 There are indications that the results for some premixed dispersant/oil combinations change over time. It is necessary to take precautions against this potential source of variation. ~~The~~ As a precaution it is highly recommended that the testing should be concluded ~~conducted~~ as soon as possible after the premix is prepared, practical following preparation of the premix, generally within a few ~~hours.~~ hours, and not stored for use at a later time. Results from samples stored for periods as long as a week should not be considered reliable.

6.9 Since the performance of the dispersant is affected by salinity, the salt water should be thoroughly mixed, stored in airtight containers, and checked with a salinity meter prior to use. Salinity should be 3.33.3 % ± 1%.0.1 %.

6.10 Temperature is a factor in dispersion, so it is important that all components (salt water, pre-mix, and temperature controlled chamber) are stable at 20°C±20 °C (or the chosen operating temperature) before starting.

NOTE 1—The normal operating temperature is 20 °C (± 1 °C). An alternate temperature may be selected based on test requirements.

6.11 Extreme care should be taken when applying the oil to the surface so that mixing does not occur. The oil should gently glide across the water to form a slick. If the oil streams out into the water, the agitation can disperse the oil, increasing the amount of oil dispersed and erroneously raising the final dispersion result.

6.12 Dispersion effectiveness generally declines with increased evaporation of oil components. Care should be taken to avoid unnecessary exposure of the oil and dispersant/oil premix before and during the test procedure to protect sample integrity and minimize variability related to oil weathering. Special care should be taken to minimize the time to apply the dispersant-oil premix to all six vessels in a test run.

<https://standards.iteh.ai/catalog/standards/sist/60349146-63dd-4890-86ad-6b03ee4da1a2/astm-f2059-21>

6.13 A slick may form at the water surface in the spout of the swirling flask during mixing and settling. It is important this oil does not enter the water sampled for analysis. Therefore it is important to drain the contents of the spout (about 3 mL) prior to sampling, and ensure any adhering droplets do not enter the sample.

6.14 The procedure is time critical for the elements of mixing, settling, and sampling. Care should be taken to adhere to the times indicated in the procedure for both the mixing and settling element, as variations in energy input, and especially time allowed for droplet creaming, can impact results. Since the water samples cannot be sampled simultaneously, this step must be performed with as much careful haste as possible, to reduce the difference in settling times experienced by the samples in the test run.

6.15 Analysis of the gas chromatograph-detectable Total Petroleum Hydrocarbon (TPH) content is subject to variability in GC operation and repeatability. Therefore, it is imperative that a rigorous quality assurance program is in place to ensure the GC is functioning properly and valid results are obtained.

6.16 Care should be taken to determine the baseline in a valid and repeatable manner for both samples and calibration sets.

6.17 The accuracy and repeatability of the test can be verified and compared using standard oil and dispersant samples

7. Apparatus

7.1 *Modified 120-mL Erlenmeyer Flask*, used as the test vessel. A side spout is added to a standard Erlenmeyer flask to enable sampling from the water column with minimal disturbance of resurfaced oil. This vessel is illustrated in **Fig. 1**.

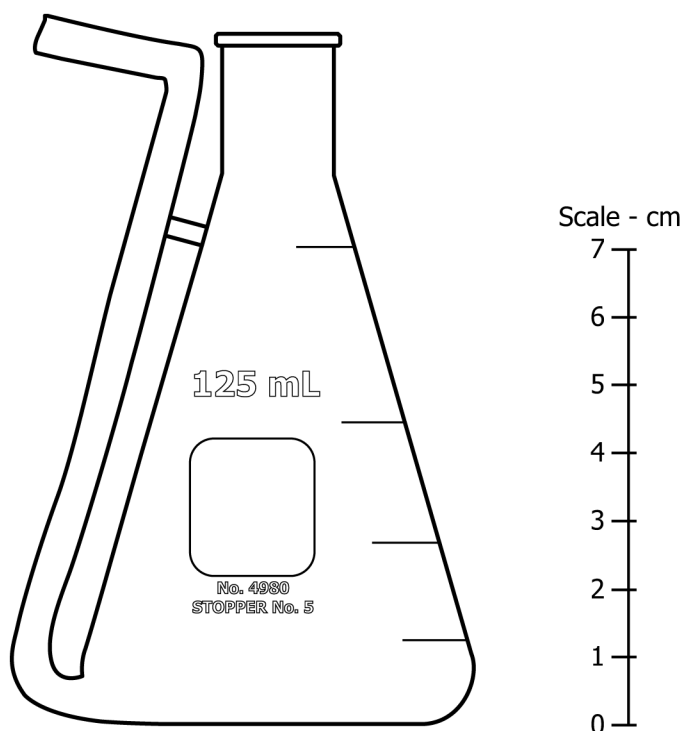


FIG. 1 Flask with Side Spout

7.2 *Moving-Table Shaker*, with an orbital motion of 1 in. (25.4 mm) and fitted with flask holders. Ideally such shakers should be constructed inside temperature-controlled chambers. If such an enclosed chamber is not used, the measurement must be conducted inside temperature-controlled rooms.

7.3 *Gas Chromatograph (GC)*, equipped with a flame ionization detector is used for analysis. The column is a fused silica column.

7.4 The following is a list of other necessary supplies. Suppliers of suitable units are footnoted. Equivalent supplies are acceptable in every case. Quantities of supplies are given to conduct a full set of six samples and calibration set:

- 7.4.1 *Eighteen Crimp Style Vials*, with aluminum/PTFE (polytetrafluoroethylene) seals, ± 2 12 mm by 32 mm,
- 7.4.2 *Twelve Erlenmeyer Flasks*, ± 25 mL–125 mL Glass, modified with the addition of a drain spout attached to base,³
- 7.4.3 *Six Graduated Mixing Cylinders and Stoppers*, 25 mL glass,
- 7.4.4 *Six Separatory Funnels and Stoppers*, glass, 125 mL,
- 7.4.5 *Six Graduated Mixing Cylinders and Stoppers*, glass, 100 mL,
- 7.4.6 *Six Separatory Funnels and Stoppers*, glass, 250 mL,
- 7.4.7 *Six Graduated Cylinders*, glass, 50 mL,
- 7.4.8 *Six Dispenser or Glass Graduated Cylinders*, 55 mL to 25 mL,
- 7.4.9 *Positive Displacement Pipet*, ± 0 10 μ L to 100 μ L,

³ The sole source of supply of the apparatus known to the committee at this time is Pro Science, Inc., 770 Birchmount Road, Unit 25, Scarborough, Ontario M1K5H3. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

- 7.4.10 *Positive Displacement Pipet*, variable volume, 1 mL,
- 7.4.11 *Two Digital Timers*,
- 7.4.12 *Dispenser or Graduated Cylinders*, 20 mL to 100 mL, and
- 7.4.13 *One Plastic Carboy*, 20 L.

8. Reagents

8.1 *Reagents*—Water purified by reverse osmosis or equivalent means is used for the test water. Dichloromethane is distilled-in-glass grade. Fine granular sodium chloride ~~or table salt, non-iodized,~~ (or table salt, non-iodized) or reagent-grade sea salt mix (for example, SigmaAldrich or equivalent), is used for making the salt water. The chemical dispersant is used as supplied by the manufacturer. Oil is used as received.

9. Procedure

9.1 *Crude Oil and Dispersant Sample Collection Storage and Storage—Preparation*—The bulk oil is mechanically mixed prior to obtaining a working sample. Working samples are stored in ~~high-density polyethylene bottles with polypropylene screw closures,~~ cleaned, airtight containers. The working sample is mechanically shaken for 30 min at $20^{\circ}\text{C}\pm 2^{\circ}\text{C}$ (or the chosen operating temperature) prior to removing a sub-sample for testing. When not in use, all samples should be stored in a ~~temperature-controlled~~ temperature-controlled room at $5^{\circ}\text{C}\pm 5^{\circ}\text{C}$. The dispersant is manually shaken, vigorously, prior to sampling.

9.2 *Dispersant Sample Storage and Preparation*—When not in use, samples should be stored at room temperature in opaque or amber containers. Prior to use, the sample is equilibrated to the temperature of the test. The dispersant is then manually shaken, vigorously, prior to sampling.

9.3 *Premix Sample Preparation*—A small amount of oil (approximately 1.0 mL) is weighed into a ~~5-mL~~ 5 mL amber vial with PTFE-lined cap. Approximately 100 mg of dispersant is added to the oil. ~~Oil~~ Additional oil is added until a nominal 1:25 ratio (by weight) of dispersant to oil is achieved. This is added to achieve the 1:25 ratio within 1% by weight, achieved (tolerance between 1:24.9 to 1:25.1). The sample is well mixed by manual shaking or stirring (see also 6.6).

9.4 *Salt-Water Preparation*—Granular salt is weighed and added to water from reverse osmosis (RO) filtration to obtain a 3.3 % (w/v) solution. The water temperature is stabilized to $20^{\circ}\text{C}\pm 2^{\circ}\text{C}$ (or the chosen operating temperature) before use.

9.5 *Swirling Flask Preparation*—The ~~120-mL~~ 120 mL of salt water is placed into a ~~125-mL~~ 125 mL modified Erlenmeyer flask. The flask is inserted into the flask holders on the oscillating table of the shaker. A ~~100- μL~~ 100 μL volume of pre-mix solution is carefully applied onto the surface of the water using a positive displacement pipet. The tip of the pipet is placed at the water surface and the dispersant/oil mixture gently expelled. Extreme care should be taken when applying the oil to the surface such that mixing does not occur. The oil should gently glide across the water to form a slick. If the oil streams out into the water, the agitation can disperse the oil, increasing the amount of oil dispersed and erroneously raising the final dispersion result. Herding of the oil and some creeping of the mixture up the vessel wall is normal but can be minimized.

9.6 *Shaking of Swirling Flasks*—The flask and contents are mechanically mixed on the shaker in a temperature controlled chamber at $20^{\circ}\text{C}\pm 2^{\circ}\text{C}$ (or the chosen operating temperature), immediately after applying the ~~oil~~ oil/dispersant mixture to the surface of the water. A rotation speed of 150 r/min and a mixing time of 20 min are used to agitate the samples followed by a ~~10-min~~ 10 min settling period. The flasks should be removed from the table-mounted holders prior to the settling period to limit the agitation between settling and sampling.

9.7 *Sample Collection*—After the settling time is complete, 3 mL of oil-in-water phase from the spout of the flask are drained and disposed of to remove any oil residing in the spout and to obtain a representative sample. A ~~30-mL~~ 30 mL aliquot of the dispersed oil in water sample is collected in a graduated cylinder and transferred to a ~~125-mL~~ 125 mL separatory funnel. The oil is extracted three times with 5 mL of the dichloromethane solvent, shaken vigorously for at least 1 min, and the extract collected in a ~~25-mL~~ 25 mL graduated mixing cylinder. ~~The final extraction volume is adjusted to 15 mL.~~ Care is taken to ensure that water is not taken along with the solvent. The final extraction volume is adjusted to 15 mL, and then a small amount of activated sodium