



Designation: F3251 – 21

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

This standard is issued under the fixed designation F3251; 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 reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

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 Baffled 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 on sea energy, oil state, temperature, salinity, actual dispersant dosage, and amount of dispersant that 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, health, and 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 standard-*

ization 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

F2059 Test Method for Laboratory Oil Spill Dispersant Effectiveness Using the Swirling Flask

2.2 *EPA Standard:*³

SW-846 Method 8270D, Revision 4 Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

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.

3.1.2 *trypsinizing, n*—the process of cell dissociation using trypsin, an enzyme which breaks down proteins, to dissociate adherent cells from the vessel in which they are being cultured.

3.1.2.1 *Discussion*—This test method uses only the vessel designed for that process (with the addition of a withdrawal tube).

4. Summary of Test Method

4.1 The 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

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.

Current edition approved Sept. 1, 2021. Published September 2021. Originally approved in 2017. Last previous edition approved in 2017 as F3251–17. DOI: 10.1520/F3251-21

² 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* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>; <https://www.epa.gov/sites/production/files/2015-12/documents/8270d.pdf>

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 second test at higher mixing energy in addition to the Swirling Flask (Test Method **F2059**).

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 at high mixing energy, 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 in recent years and provides higher mixing energies compared to other laboratory scale tests.

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 rinsing with dichloromethane to remove the oil, followed by rinsing three times each with tap water, purified water (reverse 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 verified with a tachometer periodically.

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

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

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 1:25(dispersant:oil).

6.7 Following dispersant addition, vigorous mixing is required to 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. As a precaution it is highly recommended that the testing should be conducted as soon as practical following preparation of the premix, generally within a few 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.

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 (or the chosen operating temperature) before starting.

6.11 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.

6.13 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 rise, can impact results. Since the water samples cannot be sampled simultaneously, this step must be performed as quickly as possible, to reduce the difference in settling times experienced by the samples in the test run.

6.14 Analysis of the gas chromatograph-detectable Total Petroleum Hydrocarbon (TPH) content is subject to variability