Designation: F 2059 – 00

Standard Test Method for
Laboratory Oil Spill Dispersant Effectiveness Using The Swirling Flask[1]

This standard is issued under the fixed designation F 2059; 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 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 effectiveness values that should be cited as test values derived from this standard test. Effectiveness values do not directly relate to effectiveness at sea or in other apparatuses. Actual effectiveness at sea is dependant on sea energy, oil state, temperature, salinity, actual dispersant dosage, and amount of dispersant that enters the oil.

1.4 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.

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

1.6 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 Dispersant is pre-mixed with oil, 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 pentane/dichloromethane mixture and analyzed using gas chromatography.

2.2 The extract is analyzed for oil using a gas chromatograph equipped with a flame ionization detector, (GC-FID). Quantification is by means of comparison to an internal standard. Effectiveness values are derived by calibration at fixed effectiveness values.

3. Significance and Use

3.1 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.

3.2 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.

3.3 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.

4. Interferences and Sources of Error

4.1 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), and acetone. Once cleaned, precautions must be taken to minimize contact of the glassware with surfactants to prevent undesired interferences.

4.2 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

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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 with a tachometer every week.

4.3 The Erlenmeyer flasks used in this test are tapered and the energy level varies with the amount of fill. The dispenser used to fill the vessels with water should be frequently checked by weighing the amount of water delivered.

4.4 The output is highly sensitive to the volume of oil, water, and extractant delivered. All pipets and dispensers should be calibrated on a weekly basis using water and a balance with an accuracy consistent with the weights being measured.

4.5 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.

4.6 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. The 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.

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

4.8 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 testing should be concluded as soon as possible after the premix is prepared, generally within a few hours. Results from samples stored for periods as long as a week should not be considered reliable.

4.9 Since the performance of the dispersant is affected by salinity, thorough mixing of the salt water is required. Care should also be observed to avoid evaporation from open containers of salt water. Over a period of days and weeks, the loss of water can significantly increase the salinity. An airtight closure is recommended to maintain salinity levels at 3.3 %.

4.10 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 before starting.

4.11 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.

4.12 Water in the spout attached to the swirling flask may contain more or less oil than the water in the flask itself. Therefore, it is important to drain the contents of the spout (about 3 mL) before sampling. Oil sometimes migrates into the

spout and may form a plug. This is especially true for heavy oils. It is important that the plug does not enter the sample.

4.13 The performance of the test can be verified and compared using standard oil and dispersant samples.

5. Apparatus

5.1 *Modified 120-mL Erlenmeyer Flask*, used as the test vessel. A side spout is added to enable taking the water sample with minimal disturbance of resurfaced oil. These are illustrated in Fig. 1.

5.2 *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 environmentally-controlled chambers, thereby increasing temperature control. If such an enclosed chamber is not used, the measurement must be conducted inside temperature-controlled rooms.

5.3 *Gas Chromatograph*, equipped with a flame ionization detector is used for analysis. The column is a fused silica column.

5.4 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:

5.4.1 *Fifteen Crimp Style Vials,* with aluminum/TFE-fluorocarbon seals, 12 by 32 mm,

5.4.2 *Twelve Erlenmeyer Flasks, 125 mL Glass,* modified with the addition of a drain spout attached to base,[2]

5.4.3 *Six Graduated Mixing Cylinders and Stoppers,* 25 mL glass,

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[2] Available from Pro Science, Inc., 770 Birchmount Road, Unit 25, Scarborough, Ontario M1K5H3.
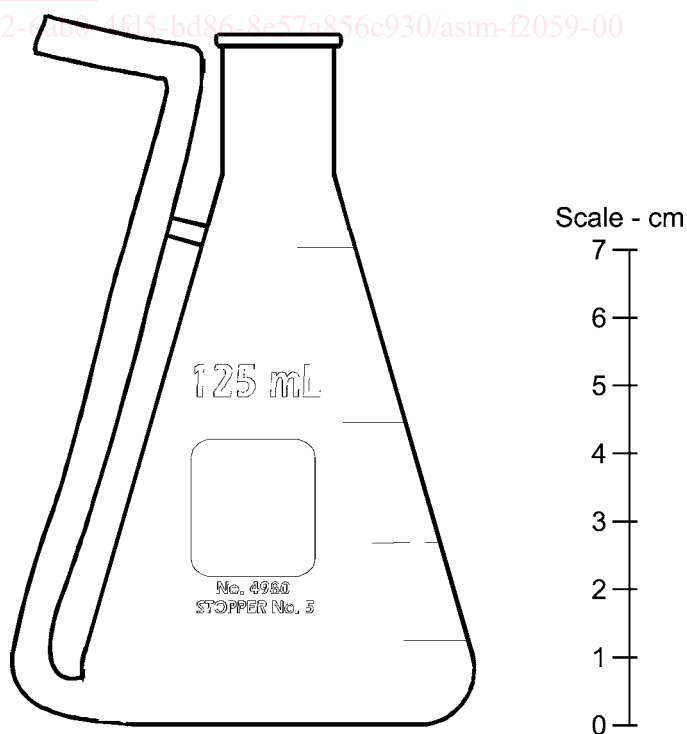

125 mL

No. 4980
STOPPER No. 5

Scale - cm
7
6
5
4
3
2
1
0

FIG. 1 Flask with Side Spout