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Standard Guide for Taking Property and Behavior Measurements on Weathered Fractions of Oil¹

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1. Scope

1.1 This guide summarizes methods to fractionate oil by evaporative weathering and then measure the properties and behavior of the weathered oil. The results of this guide can provide oil behavior data for input into oil spill models and response method selection.

1.2 This guide covers general procedures for oil weathering and behavior and does not cover all possible procedures which may be applicable to this topic.

1.3 The results obtained using this guide are intended to provide baseline data for the behavior of oil and petroleum products when spilled and input to oil spill models.

1.4 The results obtained using this guide can be used directly to predict certain facets of oil spill behavior or as input to oil spill models.

1.5 The accuracy of the guide depends very much on the representative nature of the oil sample used. Certain oils can have different properties depending on their chemical contents at the moment a sample is taken.

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 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.*

¹ This guide is under the jurisdiction of ASTM Committee F20 on Hazardous Substances and Oil Spill Response and is the direct responsibility of Subcommittee F20.16 on Surveillance and Tracking.

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2. Referenced Documents

2.1 ASTM Standards:²

- D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
- D97 Test Method for Pour Point of Petroleum Products
- D971 Test Method for Interfacial Tension of Insulating Liquids Against Water by the Ring Method
- D1310 Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus
- D1824 Test Method for Apparent Viscosity of Plastics and Organosols at Low Shear Rates
- D4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy Dispersive X-ray Fluorescence Spectrometry
- D4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration (Withdrawn 2020)³
- D5002 Test Method for Density, Relative Density, and API Gravity of Crude Oils by Digital Density Analyzer
- D5853 Test Method for Pour Point of Crude Oils
- D5949 Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)
- D6352 Test Method for Boiling Range Distribution of Petroleum Distillates in Boiling Range from 174 °C to 700 °C by Gas Chromatography
- D6450 Test Method for Flash Point by Continuously Closed Cup (CCCFP) Tester
- D6560 Test Method for Determination of Asphaltenes (Heptane Insolubles) in Crude Petroleum and Petroleum Products
- D7042 Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)
- D7094 Test Method for Flash Point by Modified Continuously Closed Cup (MCCCFP) Tester

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 The last approved version of this historical standard is referenced on www.astm.org.

D7169 Test Method for Boiling Point Distribution of Samples with Residues Such as Crude Oils and Atmospheric and Vacuum Residues by High Temperature Gas Chromatography

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

F3045 Test Method for Evaluation of the Type and Viscoelastic Stability of Water-in-oil Mixtures Formed from Crude Oil and Petroleum Products Mixed with Water

F3251 Test Method for Laboratory Oil Spill Dispersant Effectiveness Using the Baffled Flask

F3633 Guide for Measuring the Adhesion of Crude Oils and Fuel Oils

F3634 Guide for Preparing Weathered Samples of Oil Using a Rotary Evaporator

3. Terminology

3.1 *adhesion*—the tendency of dissimilar particles or surfaces to cling to one another. In this case oil to surfaces such as oleophilic skimmer surfaces.

3.2 *emulsion*—a type of colloid, specifically, a dispersion of small droplets of one liquid in another.

3.3 *interfacial tension*—the elastic tendency of a fluid surface which makes it acquire the least surface area possible.

3.4 *simulated distillation (SIM DIS)*—measurement of the boiling temperatures of fractions of oil using chromatography alone.

3.5 *stability index*—an index describing the stability of an emulsion. In this guide, it is calculated using data derived from rheological measurements.

3.6 *water-in-oil emulsion*—an emulsion consisting of a continuous phase of oil containing a dispersed phase of water.

4. Summary of Guide

4.1 Oil is evaporatively weathered to at least three (3) stages, with the first stage being fresh oil and the second being intermediate and with the final stage being extensive. Extensive weathering is reasonably maximum weathering for example, ten (10) days equivalent or with little change in properties occurring in the final sub-stages of weathering.

4.2 Sub-samples of the oil are taken at each weathering stage and desired properties and behaviors (such as density, viscosity, emulsion formation, etc.) are measured.

5. Significance and Use

5.1 A standard procedure is necessary to establish property changes for spilled oils or petroleum products at different oil weathering stages.

5.2 This procedure employs standardized equipment, and test procedures.

5.3 This procedure should be performed at the stages of weathering corresponding to the spill conditions of interest.

6. Interferences and Sources of Error

6.1 Interferences can be caused by contaminants, particularly residual oil or surfactants on labware, and other sample

handling supplies and apparatus that lead to irregular results. All glassware must be thoroughly cleaned. The cleaning process includes rinsing with dichloromethane to remove the oil, followed by rinsing three times once each with tap water, purified water, and acetone. Once cleaned, precautions must be taken to minimize contact of the labware with contaminants to prevent interferences.

6.2 The specified fill volumes of the test vessels must be carefully observed as the accuracy varies with the amount of fill.

6.3 Temperature is a factor in all measurements, so it is important that all components (test water, instruments, and temperature-controlled chamber) are stable at 15 °C or the selected test temperature, before starting.

6.4 The handling of the samples is important. Care must be taken to take a representative sample. Water should be avoided when sampling.

6.5 Oils sources, especially crude oil sources, vary much with production time and conditions. Oil samples must be treated as unique and are not necessarily representative of the source. Depending on the actual conditions under which this oil was sampled, different values for properties are measured.

7. Handling and Storing Oil

7.1 *Crude or Raw Oil Sample Storage and Preparation*—the bulk oil as received is mechanically mixed for 2 to 4 h prior to obtaining a working sample. Working samples are stored in high-density polyethylene bottles (especially made and coated for oil samples) with polypropylene screw closures. The working sample is mechanically shaken for 30 min at 15 °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 room at 5 °C. Prior to use, the sample is brought to the temperature of the test for at least 1 to 2 h. The sample is then shaken mechanically prior to use. The head space of air should be minimized in all containers used for shaking.

8. Weathering the Oil

8.1 *Overview*—Evaporative weathering changes the properties and behavior of oil to a large degree. The data are taken as weight and reported as weight percent (lost through evaporation). It is recommended that at least three weathered stage samples of the oil be prepared. The final point is taken at a time or exposure which would represent a high weathering stage. The temperature of exposure should be below 85 °C to avoid formation of oxygenated or pyrolytic compounds. Some oils are subject to photo-oxidation which may affect how they are weathered. Repeatable evaporative weathering can be accomplished using many methods, the most commonly accepted are summarized below (1).⁴

8.1.1 *Weathering by Pan Evaporation*—Oil is weathered by pan evaporation until the weight loss is minimal. This typically occurs with light oils at times between 10 to 14 days, but less with heavier oils (1).

⁴ The boldface numbers in parentheses refer to a list of references at the end of this standard.