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Standard Guide for Preparing Weathered Samples of Oil Using a Rotary Evaporator[1]

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1. Scope

1.1 This guide summarizes methods to produce representative residual oil following evaporative weathering using a rotary evaporator.

1.1.1 The results of this guide can provide weathered oil samples for further study and characterization.

1.2 This guide covers general procedures for artificial weathering of oil by using rotary evaporation devices and does not cover all possible procedures that may be applicable to this topic.

1.3 The accuracy of this 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 time a sample is taken.

1.4 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.5 *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.*

1.6 *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]

F3337 Guide for Taking Property and Behavior Measurements on Weathered Fractions of Oil

3. Summary of Guide

3.1 Oil is evaporatively weathered to at least three stages with the first stage being fresh oil, the second being intermediate, and the final stage being extensive. Extensive weathering is reasonably maximum weathering, for example, ten days equivalent or with little change in properties occurring in the final substages of weathering. For this guide, accelerated weathering is applied for 48 h.

3.1.1 An exception is some residual fuel oils that have limited volatile content and do not have evaporative mass loss greater than 5 % over the 48 h period of the procedure. For these products, an intermediate weathered sample may not be informative.

3.2 Subsamples of the oil are taken at each weathering stage and further analysis can be performed on the samples.

4. Significance and Use

4.1 A standard procedure is necessary to prepare samples of oils or petroleum products at different oil weathering stages with compositional distribution that is representative of weathered spilled oil.

4.2 This procedure uses standardized equipment and test procedures.

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

5. Interferences and Sources of Error

5.1 Large proportions of inorganic substances in the oil sample, such as water and sediment that are produced with the oil at source, can distort the evaporation profile of the bulk hydrocarbons. Water and sediment are typically removed during crude petroleum processing; however, if the oil sample is sourced upstream of this step, then it is necessary to decant the water before weathering to obtain an accurate value for hydrocarbon mass loss. The mass percentage of sediment shall then be determined separately to adjust the initial mass of hydrocarbon to enable the correct percentage mass loss for the sample to be determined.

[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] 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.

5.2 Interferences can be caused by contaminants, particularly residual oil or surfactants on labware, and other sample-handling supplies and apparatus that lead to nonrepresentative oil samples. All glassware shall 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 shall be taken to minimize contact of the labware with contaminants to prevent interferences.

5.3 Temperature is a factor in the evaporation rate, so it is important that the oil is below 80 °C or the selected test temperature before starting the air flow to begin evaporation.

5.4 The handling of the samples is important. Contaminants that are introduced onto the vessel or handling equipment will affect the weight determination.

5.5 Oil sources, especially crude oil sources, vary greatly with production time and conditions. Oil samples shall 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.

6. Handling and Storing Oil

6.1 *Crude or Raw Oil Sample Storage and Preparation*—The bulk oil as received is mechanically mixed until fully homogenized before obtaining a working sample. Working samples are transferred to high-density polyethylene or glass bottles with leak-resistant screw closures with minimized head space and stored in a temperature-controlled room at 5 °C to reduce the potential for weathering. The sample is then shaken or tumbled mechanically before use.

7. Weathering the Oil

7.1 *Overview*—Evaporative weathering can significantly alter the composition and properties of oil because of the loss of volatile and semi-volatile components. The loss of mass is tracked by weighing the remaining bulk oil and reported as a weight percent of the initial sample to represent the degree of weathering caused by evaporation. It is recommended that at least three weathered samples of the oil be prepared. The final point is taken at a time or exposure that would represent a high-weathering stage. The temperature of exposure should be below 85 °C to avoid formation of oxygenated or pyrolytic compounds. Evaporative weathering can be accomplished using many methods. This method is by rotary evaporation. Oil is evaporated in a rotary evaporator maintained at 80 °C. A given weight of oil is placed into the rotary evaporator and the unit is operated at 80 °C for up to 48 h while a vacuum pump is used to draw air through the system. Periodically, the contents are weighed to determine the percentage mass loss of the weathered sample. Samples are taken as desired and their weathering percentage noted.[3,4]

7.2 *Equipment*

7.2.1 *Rotary Evaporator*—A 10 L rotary evaporator is typically used (rotary evaporator with a 10 L flask, an integral water bath, a circulating bath, and a vacuum pump).

7.2.2 *Balance*—A balance capable of measuring up to 20 kg with an accuracy of ±0.1 g is required.

7.2.3 *Collar*—A collar to hold the round rotary evaporator vessel is required when not mounted on the rotary evaporator such as during weight measurements.

7.3 *Rotary Evaporator Weathering*

7.3.1 *Temperature Adjustment*—The water bath on the rotary evaporator is brought to a temperature of 80 °C.

7.3.2 *Weighing and Filling*—The empty rotary flask is weighed, approximately 2 L of oil is added, and the flask is reweighed.

7.3.3 *Operation of the Rotary Evaporator*—The flask is mounted onto the water bath and partially immersed in the water bath and spun at 135 rpm. A constant flow of air at 13 L⁄min is maintained through the flask by the vacuum pump.

7.3.4 *Measurements and Samples*—At set intervals, the sample flask is removed and weighed to track the progress of the evaporation. Measurements are initially taken more frequently while the rate of evaporation is highest, then less frequently as evaporative losses slow. Periodically, approximately 1 g of sample may be taken for chemical analysis.

7.3.5 *Duration of Weathering*—The rotary evaporator is operated for 48 h to represent a relatively extensive degree of weathering of the oil. At this time, the rotary evaporator is stopped, the flask removed, and weighed. The contents are removed, bottled, and marked at the weathering percentage calculated for the full extent of weathering.

7.3.6 *Interruptions*—The procedure may be stopped, for example, at the end of the workday. The flask is removed, covered, and placed into a refrigerator at 5 °C. Upon resumption of weathering, the flask is mounted on the rotary evaporator and rotated in the heated water bath without air flow until the oil returns to 80 °C. Once up to temperature, the air flow is then re-engaged and timing to the next measurement interval resumed.

8. Property Measurements on the Oil Samples

8.1 Each of the unique weathering fraction samples should be characterized by oil property measurements appropriate for the intended application. Procedures for the recommended set of properties measurements to characterize the oil are given in Guide F3337.

9. Calculation and Reporting

9.1 The values of weathering percentages are reported for each sample taken. The accuracy is a function of the balance uncertainty and any external contaminants that add mass back to the vessel and support collar. Accuracy is expected to be kept within 0.1 % for any given measurement. The fraction mass-loss is calculated as:

$$\%\text{weathering} = (\text{mi} - \text{mf}) / (\text{mi} - \text{me}) \times 100\ \% \qquad (1)$$

[3] Fieldhouse, B., Hollebone, B. P., Singh, N. R., Tong, T. S., and Mullin, J., "Artificial weathering of oils by rotary evaporator," in *Proceedings of the 33rd AMOP Technical Seminar on Environmental Contamination and Response, Environment Canada*, Ottawa, ON, Vol 1, 2010, pp. 159-180.

[4] "Summary methods for the analysis of physical properties, chemical composition, and behavior of petroleum products," *Emergencies Science and Technology Section (ESTS), Environment and Climate Change Canada*, 2019.