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ASTM D4057-22

Standard Practice for Manual Sampling of Petroleum and Petroleum Products

Significance and Use

4.1 Samples of petroleum and petroleum products are obtained for many reasons, including the determination of chemical and physical properties. These properties may be used for: calculating standard volumes; establishing product value; and often safety and regulatory reporting.

4.2 There are inherent limitations when performing any type of sampling, any one of which may affect the representative nature of the sample. As examples, a spot sample provides a sample from only one particular point in the tank, vessel compartment, or pipeline. In the case of running or all-level samples, the sample only represents the column of material from which it was taken.

4.3 Based on the product, and testing to be performed, this practice provides guidance on sampling equipment, container preparation, and manual sampling procedures for petroleum and petroleum products of a liquid, semi-liquid, or solid state, from the storage tanks, flowlines, pipelines, marine vessels, process vessels, drums, cans, tubes, bags, kettles, and open discharge streams into the primary sample container.

Scope

1.1 This practice covers procedures and equipment for manually obtaining samples of liquid petroleum and petroleum products, crude oils, and intermediate products from the sample point into the primary container are described. Procedures are also included for the sampling of free water and other heavy components associated with petroleum and petroleum products.

1.2 This practice also addresses the sampling of semi-liquid or solid-state petroleum products. For the sampling of green petroleum coke, see Practice D8145. For the sampling of calcined petroleum coke, see Practice D6970.

1.3 This practice provides additional specific information about sample container selection, preparation, and sample handling.

1.4 This practice does not cover sampling of electrical insulating oils and hydraulic fluids. If sampling is for the precise determination of volatility, use Practice D5842 (API *MPMS* Chapter 8.4) in conjunction with this practice. For sample mixing and handling, refer to Practice D5854 (API *MPMS* Chapter 8.3).

1.5 The procedures described in this practice may also be applicable in sampling most non-corrosive liquid industrial chemicals provided that all safety precautions specific to these chemicals are followed. Also, refer to Practice E300. The procedures described in this practice are also applicable to sampling liquefied petroleum gases and chemicals. Also refer to Practices D1265 and D3700. The procedure for sampling bituminous materials is described in Practice D140. Practice D4306 provides guidance on sample containers and preparation for sampling aviation fuel.

1.6 Units—The values stated in SI units are to be regarded as the standard. USC units are reflected in parentheses.

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.*

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ASTM D4176-22

Standard Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures)

Significance and Use

5.1 It has long been the practice to include in fuel specifications a requirement that the fuel be *clear and bright and free of visible particulate matter* (see Note 1). However, there has been no standard method for making this determination so that practices have differed. This test method provides standard procedures for the test.

NOTE 1: *Clean and bright* is sometimes used in place of *clear and bright*. The meaning is identical.

5.2 Procedure 1 provides a rapid pass/fail method for contamination in a distillate fuel. Procedure 2 provides a gross numerical rating of haze appearance, primarily as a communication tool. Other test methods, including Test Methods D2276, D2709, and D4860, permit quantitative determinations of contaminants. No relationship has been established between Procedure 2 and these quantitative methods.

5.2.1 Test Method D8148 has established a correlating relationship with Procedure 2 appearance rating numbers by reporting a correlating instrument haze rating (IHR) based upon its spectroscopically determined haze clarity index (HCI). Supporting data can be found in RR:D02-1876.5

5.3 Limited laboratory evaluations of samples that have failed this *clear and bright* test indicate that an experienced tester can detect as little as 40 ppm of free water in the fuel.

Scope

1.1 This test method covers two procedures for estimating the presence of suspended free water and solid particulate contamination in distillate fuels having distillation end points below 400 °C and an ASTM color of 5 or less.

1.1.1 Both procedures can be used as field tests at storage temperatures, or as laboratory tests at controlled temperatures.

1.1.2 Procedure 1 provides a rapid pass/fail method for contamination. Procedure 2 provides a gross numerical rating of haze appearance.

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

1.3 *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.*

1.4 *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.*

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ASTM D7464-20

Standard Practice for Manual Sampling of Liquid Fuels, Associated Materials and Fuel System Components for Microbiological Testing

Significance and Use

5.1 Representative samples of fuel products and associated substances are required for the determination of microbial contamination in fuels and fuel systems in order to accurately assess the biodeterioration risk posed to the fuel, fuel-system components or both. Uncontrolled microbial contamination can affect fuel specification properties adversely.6 As discussed in Guide D6469, microbes can cause a variety of operational problems, including filter plugging and microbially influenced corrosion (MIC), the latter of which causes valve failure, tank and pipeline failure.

5.2 These practices for microbiological sampling decrease the risk of contaminating samples with extraneous microbes, thereby increasing the probability that the original microbial population in the sample does not change significantly between the time of sampling and the time of testing.

5.3 The objective of sampling for microbiological testing is to obtain representative samples that are likely to reflect the degree and nature of microbial contamination in the system from which the samples are collected. Manual 477 addresses the rationale for and design of microbial contamination programs. Recognizing that microbiological contamination is not distributed uniformly throughout fuel systems, both the number and types of samples collected will normally be different from the samples collected per Practice D4057 in order to determine whether product meets specifications.

5.4 The physical, chemical and microbiological property tests to be performed on a sample will dictate the sampling procedures, the sample quantity required, and many of the sample handling requirements.

5.5 Fuel systems are not normally designed to facilitate optimal microbiological sampling. Consequently, the selection of sampling device and sample source reflect compromises between accessibility and suitability for meeting the sample collection objective.

5.6 The guidance provided in Practice D4057 generally applies to this practice as well. Consequently, this practice will address only those procedures that apply uniquely to microbiological sampling.

Scope

1.1 This practice covers aspects of sample device preparation and sample handling that prevent samples from becoming contaminated with microorganisms not originally contained within the sample.

1.2 This practice also covers sample handling considerations that reflect the perishability of samples collected for microbiological testing.

1.3 This practice supplements Practice D4057 by providing guidance specific to the manual sampling of fuels when samples are to be tested for microbial contamination.

1.4 The values stated in SI units are to be regarded as 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.*

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ASTM D7687-23

Standard Test Method for Measurement of Cellular Adenosine Triphosphate in Fuel and Fuel-associated Water With Sample Concentration by Filtration

Significance and Use

5.1 This test method measures the concentration of cellular-ATP present in the sample. ATP is a constituent of all living cells, including bacteria and fungi. Consequently, the presence of cellular-ATP is an indicator of total metabolically active microbial contamination in fuels. ATP is not associated with matter of non-biological origin.

5.2 This test method is similar to Test Method E2694 except for the volumes sampled.

5.3 This test method differs from Test Method D4012 in that it utilizes filtration and wash steps designed to eliminate interferences that have historically rendered ATP testing unusable with complex organic fluids such as fuel and fuel-associated water.

5.4 This test method differs from Test Method D7463 in several regards:

5.4.1 Test Method D7463 reports relative light units (RLU). Consistent with Test Methods D4012 and E2694, this test method reports ATP concentration.

5.4.2 This test method detects only cellular-ATP and it can be used to detect cellular-ATP in fuels and fuel stocks from which small quantities of water do not separate readily (for example, ethanol blended gasoline containing ≥5 % v/v ethanol). Test Method D7463 cannot be used to recover ATP from fuels from which small quantities of water do not separate readily (for example, ethanol blended gasoline containing ≥5 % v/v ethanol).

5.4.3 This test method measures cellular-ATP in a single measurement (as pg ATP/mL). Test Method D7463 detects total ATP (as RLU) and extra-cellular ATP (as RLU) using two separate analyses and permits computation of cellular-ATP (as RLU) as the difference between total and extracellular ATP.

5.4.4 Test Method D7463 suggests a nominal 500 mL fuel sample volume. This test method suggests a nominal 20 mL fuel sample.

5.5 This test method can be used with all fuels specified in Specifications D396, D975, D1655, D2069, D2880, D3699, D6751, and D7467 and other fuels with nominal viscosities ≤75 cSt at 20° ± 2°.

5.6 The ATP test provides rapid test results that reflect the total bioburden in the sample. It thereby reduces the delay between test initiation and data capture, from the 36 h to 48 h (or longer) required for culturable colonies to become visible, to approximately 5 min.

5.7 Although ATP data generally covary with culture data in fuel and fuel-associated water, different factors affect ATP concentration than those that affect culturability.

5.7.1 Culturability is affected primarily by the ability of captured microbes to proliferate on the growth medium provided, under specific growth conditions. Consequently, a proportion of the active or inactive microbial population present in a sample may be viable but not detected by any one culture test.4

5.7.2 ATP concentration is affected by: the microbial species present, the physiological states of those species, and the total bioburden (see Appendix X1).

5.7.2.1 One example of the species effect is that the amount of ATP per cell is substantially greater for active fungal cells than bacteria.

5.7.2.2 Within a species, cells that are more metabolically active will have more ATP per cell than dormant cells, such as fungal spores. Because fungal spores are more hydrophobic than active fungal material (mycelium), spores may be the only indicator of fungal proliferation when fuel samples are taken from some fuel systems, but they will not be detected by a test for ATP.

5.7.2.3 The greater the total bioburden, the greater the ATP concentration in a sample.

5.7.3 The possibility exists that the rinse step (11.15) may not eliminate all chemical substances that can interfere with the bioluminescence reaction (11.37).

5.7.3.1 The presence of any such interferences can be evaluated by performing a standard addition test series or dilution series as described in Appendix X4. The precision statement in Section 13 will not apply.

5.8 As explained in Test Method D7978, there are inherent difficulties in assessing precision of microbiological procedures for fuels on account of the inherent variability of the determinant and various determinable and indeterminable sources of inaccuracy (see Guide D7847).

5.8.1 The precision of any microbiological analytical method will generally be considerably less than that of methods widely used in the petroleum industry for analysis of physical and chemical properties of fuels.

Scope

1.1 This test method covers a protocol for capturing, extracting and quantifying the cellular adenosine triphosphate (cellular-ATP) content associated with microorganisms found in fuels and fuel-associated water.

1.2 The ATP is measured using a bioluminescence enzyme assay, whereby light is generated in amounts proportional to the concentration of cellular-ATP in the samples. The light is produced and measured quantitatively as relative light units (RLU) which are converted by comparison with an ATP standard, computation to pg ATP/mL and optional further transformation to Log_{10}[pg ATP/mL].

1.3 This test method is equally suitable for use as a laboratory or portable method.

1.4 This test method is limited to fuels with a nominal viscosity ≤75 cSt at test temperature.

1.5 This test method detects ATP concentrations in the range of 5.0 pg ATP/mL (=0.699 log_{10}[pg ATP/mL]) to 100 000 pg ATP/mL (=5.000 log_{10}[pg ATP/mL]) for 20 mL samples of fuel and 20 pg ATP/mL (=1.301 log_{10}[pg ATP/mL]) to 400 000 pg ATP/mL (=5.602 log_{10}[pg ATP/mL]) for 5 mL samples of fuel-associated water.

NOTE 1: These ranges were calculated with the formula for calculating sample ATP in pg/mL provided in 12.1 based on the minimum recommended RLU for a 1 ng/mL ATP standard when using the reagents specified in Section 7 and the luminometer specified in 6.4 and corrected with a reagent-method blank as determined in Appendix X5.

1.6 Providing interferences can be overcome, bioluminescence is a reliable and proven method for qualifying and quantifying ATP. This test method does not differentiate between ATP from different sources, for example: from different types of microorganisms, such as bacteria and fungi.

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

1.8 *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.*

1.9 *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.*

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