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Manual of Petroleum Measurement Standards (MPMS), Technical Report 2573

Standard Guide for Sediment and Water Determination in Crude Oil[1]

This standard is issued under the fixed designation D7829; 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 guide covers a summary of the water and sediment determination methods from the API MPMS Chapter 10 for crude oils. The purpose of this guide is to provide a quick reference to these methodologies such that the reader can make the appropriate decision regarding which method to use based on the associated benefits, uses, drawbacks and limitations.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (API *MPMS* Chapter 10.1)

D4006 Test Method for Water in Crude Oil by Distillation (API *MPMS*Chapter 10.2)

D4007 Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure) (API *MPMS* Chapter 10.3)

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products (API *MPMS* Chapter 8.1)

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products (API *MPMS* Chapter 8.2)

D4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration (Withdrawn 2020)[3]

D4807 Test Method for Sediment in Crude Oil by Membrane Filtration

D4928 Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration

D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (API *MPMS* Chapter 8.3)

2.2 *API Standards:*[4]

API *MPMS* Chapter 1 Terms and Definitions Database

API *MPMS* Chapter 8.1 Standard Practice for Manual Sampling of Petroleum and Petroleum Products (ASTM D4057)

API *MPMS* Chapter 8.2 Standard Practice for Automatic Sampling of Petroleum and Petroleum Products (ASTM D4177)

API *MPMS* Chapter 8.3 Standard Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (ASTM D5854)

API *MPMS* Chapter 10.1 Standard Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (ASTM D473)

API *MPMS*Chapter 10.2 Standard Test Method for Water in Crude Oil by Distillation (ASTM D4006)

API *MPMS*Chapter 10.3 Standard Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure) (ASTM D4007)

[1] This guide is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and the API Committee on Petroleum Measurement, and is the direct responsibility of Subcommittee D02.02 /COMQ the joint ASTM-API Committee on Hydrocarbon Measurement for Custody Transfer (Joint ASTM-API). This guide has been approved by the sponsoring committees and accepted by the Cooperating Societies in accordance with established procedures. This practice was issued as a joint ASTM-API standard in 2013.

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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.

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

[4] Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, http://www.api.org.

***A Summary of Changes section appears at the end of this standard**

API *MPMS* Chapter 10.4 Determination of Water and/or Sediment in Crude Oil by the Centrifuge Method (Field Procedure)
API *MPMS* Chapter 10.7 Standard Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration (D4377)
API *MPMS* Chapter 10.8 Standard Test Method for Sediment in Crude Oil by Membrane Filtration (D4807)
API *MPMS* Chapter 10.9 Standard Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration (D4928)

3. Terminology

3.1 *Definitions:*

3.1.1 For terms and definitions used in this guide, refer to Terminology D4175 and API *MPMS* Chapter 1.

4. Significance and Use

4.1 Theoretically, all of the sediment and water determination methods are valid for crude oils containing from 0 % to 100 % by volume sediment and water; the range of application is specified within the scope of each method. The round robins for all methods were conducted on relatively dry oil. All precision and bias statements included in the methods are based upon the round robin data. Analysis becomes more challenging with crude oils containing higher water contents due to the difficulty in obtaining a representative sample, and maintaining the sample quality until analysis begins.

4.2 Currently, Karl Fischer is generally used for dry crude oils containing less than 5 % water. Distillation is most commonly used for dry and wet crude oils and where separate sediment analysis is available or in situations where the sediment result is not significant. The laboratory centrifuge methods allow for determination of total sediment and water in a single analysis. The field centrifuge method is used when access to controlled laboratory conditions are not available.

4.3 In the event of a dispute with regard to sediment and water content, contracting parties may refer to the technical specifications table to determine the most appropriate referee method based upon knowledge of and experience with the crude oil or product stream.

5. Procedure

5.1 To obtain the total sediment and water results when the two are determined by separate methods: First, add the raw, unrounded results together, and then round the sum to the correct significant figure.

5.2 It is critically important that the analytical samples are collected and handled with careful regard to ensure that they are representative of the entire parcel. The crude oil or product stream should be homogenous at the sampling point, and the sample properly mixed prior to analysis. Sub-samples should not be allowed to settle prior to transfer into an analytical device such as a centrifuge tube, distillation apparatus, or extraction thimble. Refer to API MPMS Chapter 8.1 (ASTM D4057) Standard Practice for Manual Sampling of Petroleum and Petroleum Products, API MPMS Chapter 8.2 (ASTM D4177) Standard Practice for Automatic Sampling of Petroleum and Petroleum Products, API MPMS, and Chapter 8.3 (ASTM D5854) Standard Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products for appropriate sample collection and mixing.

5.3 It is imperative that every sample is mixed sufficiently to ensure that it is homogeneous. However, with prolonged mixing the temperature of the sample will increase. This will decrease the representative nature of the sample due to evaporative losses. The mixing system must be evaluated to determine that the mixing time is sufficient without undue temperature increases.

5.4 Sampling error can be a source of bias in analytical methods. This is reflected in the precision and bias statement for each method, but it should be considered when evaluating methods. A larger sample size will increase the probability that a sample is representative of the entire parcel and can minimize the potential bias arising from a less homogenous sample stream. Measuring sample size by weight using an analytical balance is not subject to human error; measuring sample size by volume using analytical glassware is a source of human error.

5.5 When sample size is determined by weight, consider the type of analytical balance used to weigh the sample. Ensure that the resolution is appropriate, having sufficient significant figures, for the analysis.

6. Report

Water Only Determination Methods

6.1 *MPMS Chapter 10.4, Determination of Water and/or Sediment in Crude Oil by the Centrifuge Method (Field Procedure):*

NOTE 1—ASTM Test Methods D96 for Water and Sediment in Crude Oil By Centrifuge (Field Procedure), the technically equivalent ASTM standard was withdrawn without replacement in 2000.

6.1.1 *Range of Application:*

6.1.1.1 There is no approved precision and bias statement for this method.

6.1.1.2 The practical minimum detection limit is 0.025 % v/v water. Lower concentrations of water should be reported as less than 0.025 % v/v or 0 % v/v.

6.1.1.3 This scope of the method indicates that it applies to crude oil, there is no water content specified in the scope.

6.1.2 *Interferences, Biases, and Limitations:*

6.1.2.1 The round robin for this method was conducted for combined sediment and water, not the sediment or water fractions separately. Therefore the precision and bias statements do not apply to analysis of water only.

6.1.2.2 This method will typically quantify light alcohols such as, (but not limited to), methanol and ethanol in the water fraction, biasing the water result high.

6.1.2.3 Centrifuge tubes may be filled directly from a sample tap and analyzed minimizing sample handling. Minimizing sample handling helps to maintain the representative nature of a sample.

6.1.2.4 A 50 mL sample is used for analysis as measured in the centrifuge tube.