



Designation: **D5002—13** **D5002 – 15**

## Standard Test Method for Density and Relative Density of Crude Oils by Digital Density Analyzer<sup>1</sup>

This standard is issued under the fixed designation D5002; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope\*

1.1 This test method covers the determination of the density or relative density of crude oils that can be handled in a normal fashion as liquids at test temperatures between  $\pm 15\text{ }^{\circ}\text{C}$  and  $35\text{ }^{\circ}\text{C}$ .<sup>2</sup> This test method applies to crude oils with high vapor pressures provided appropriate precautions are taken to prevent vapor loss during transfer of the sample to the density analyzer.

1.2 This test method was evaluated in round robin testing using crude oils in the  $0.750.75\text{ g/mL}$  to  $0.950.95\text{ g/mL}$  range. Lighter crude oil can require special handling to prevent vapor losses. Heavier crudes can require measurements at higher temperatures to eliminate air bubbles in the sample.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. The accepted units of measurement of density are grams per millilitre and kilograms per cubic metre.

1.4 *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.* Specific warning statements are given in 7.4, 7.5, and 7.6.

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer (Withdrawn 1993)<sup>3</sup>

D1193 Specification for Reagent Water

D1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer

D1250 Guide for Use of the Petroleum Measurement Tables

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration

### 3. Terminology

3.1 *Definitions:*

3.1.1 *density*—mass per unit volume at a specified temperature.

3.1.2 *relative density*—the ratio of the density of a material at a stated temperature to the density of water at a stated temperature.

### 4. Summary of Test Method

4.1 Approximately 1 mL to 2 mL of crude oil sample is introduced into an oscillating sample tube and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0D on Physical and Chemical Methods.

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<sup>2</sup> 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.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

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

## 5. Significance and Use

5.1 Density is a fundamental physical property that can be used in conjunction with other properties to characterize the quality of crude oils.

5.2 The density or relative density of crude oils is used for the conversion of measured volumes to volumes at the standard temperatures of  $\pm 5^{\circ}\text{C}$   $15^{\circ}\text{C}$  or  $60^{\circ}\text{F}$   $60^{\circ}\text{F}$  and for the conversion of crude mass measurements into volume units.

5.3 The application of the density result obtained from this test method, for fiscal or custody transfer accounting calculations, can require measurements of the water and sediment contents obtained on similar specimens of the crude oil parcel.

## 6. Apparatus

6.1 *Digital Density Analyzer*—A digital analyzer consisting of a U-shaped, oscillating sample tube and a system for electronic excitation, frequency counting, and display. The analyzer must accommodate the accurate measurement of the sample temperature during measurement or must control the sample temperature as described in 6.2 and 6.5. The instrument shall be capable of meeting the precision requirements described in Test Method D4052.

6.2 *Circulating Constant-Temperature Bath*, capable of maintaining the temperature of the circulating liquid constant to  $\pm 0.05^{\circ}\text{C}$   $\pm 0.05^{\circ}\text{C}$  in the desired range. Temperature control can be maintained as part of the density analyzer instrument package.

6.3 *Syringes*, at least  $2\text{ mL}$   $2\text{ mL}$  in volume with a tip or an adapter tip that will fit the inlet of the density analyzer.

6.4 *Flow-Through or Pressure Adapter*, for use as an alternative means of introducing the sample into the density meter.

6.5 *Thermometer*, calibrated and graduated to  $0.1^{\circ}\text{C}$   $0.1^{\circ}\text{C}$ , and a thermometer holder that can be attached to the instrument for setting and observing the test temperature. In calibrating the thermometer, the ice point and bore corrections should be estimated to the nearest  $0.05^{\circ}\text{C}$   $0.05^{\circ}\text{C}$ . Precise setting and control of the test temperature in the sample tube is extremely important. An error of  $0.1^{\circ}\text{C}$   $0.1^{\circ}\text{C}$  can result in a change in density of one in the fourth significant figure.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D1193; or better.

7.3 *Water*, ~~redistilled, freshly boiled reagent water~~ *reagent water, freshly boiled, to remove dissolved gasses*, for use as a primary calibration standard. (**Warning**—Handling water at boiling or near boiling temperature can present a safety hazard. Wear appropriate personal protective equipment.)

7.4 *Acetone*, for flushing and drying the sample tube. (**Warning**—Extremely flammable.)

7.5 *Petroleum Naphtha*, for flushing viscous petroleum samples from the sample tube. (**Warning**—Extremely flammable.)

NOTE 1—Suitable solvent naphthas are marketed under various designations such as “petroleum ether,” “ligroine,” or “precipitation naphtha.”

7.6 *n-Nonane, n-tridecane or cyclohexane*, 99 % purity or better, or similar pure material for which the density is known precisely from literature references or by direct determination in accordance with Test Method D941 or D1217. (**Warning**—Extremely flammable.)

## 8. Sampling, Test Specimens, and Test Units

8.1 Sampling is defined as all the steps required to obtain an aliquot of the contents of any pipe, tank or other system, and to place the sample into the laboratory test container. The laboratory test container and sample volume shall be of sufficient dimensions to allow mixing as described in 8.3.1. Mixing is required to obtain a homogeneous sample for analysis.

8.2 *Laboratory Sample*—Use only representative samples obtained as specified in Practices D4057 or D4177 for this test method.

8.3 *Test Specimen*—The aliquot of sample obtained from the laboratory sample and delivered to the density analyzer sample tube. The test specimen is obtained as follows:

8.3.1 Mix the sample of crude oil to homogenize any sediment and water present. The mixing may be accomplished as described in Practice D4177 or Test Method D4377. Mixing at room temperature in an open container can result in the loss of light ends, so mixing in closed, pressurized containers or at sub-ambient temperatures is recommended.

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.3.2 Draw the test specimen from a properly mixed laboratory sample using an appropriate syringe. Alternatively, if the proper density analyzer attachments and connecting tubes are used then the test specimen can be delivered directly to the analyzer's sample tube from the mixing container.

## 9. Preparation of Apparatus

9.1 Set up the density analyzer and constant temperature bath following the manufacturer's instructions. Adjust the bath or internal temperature control so that the desired test temperature is established and maintained in the sample compartment of the analyzer. Calibrate the instrument at the same temperature at which the density of the sample is to be measured.

## 10. Calibration of Apparatus

10.1 Calibrate the instrument when first setting up and whenever the test temperature is changed. Thereafter, conduct calibration checks at least weekly during routine operation or more frequently as may be dictated by the nature of the crude oils being measured (see 10.3).

10.2 Initial calibration, or calibration after a change in test temperature, necessitates calculation of the values of the Constants A and B from the periods of oscillation, ( $T$ ), observed when the sample cell contains certified reference liquids such as air and ~~re-distilled~~ freshly boiled reagent water. (See Warning note in 7.3.) Other calibrating materials such as  $n$ -nonane,  $n$ -tridecane, cyclohexane, and  $n$ -hexadecane (for high temperature applications) can also be used as appropriate.

10.2.1 While monitoring the oscillator period,  $T$ , flush the sample tube with petroleum naphtha, followed with an acetone flush and dry with dry air. Continue drying until the display exhibits a steady reading. In cases where saline components can be deposited in the cell, flush with distilled water followed by acetone and dry air. Contaminated or humid air can affect the calibration. When these conditions exist in the laboratory, pass the air used for calibration through a suitable purification and drying train. In addition, the inlet and outlet ports for the U-tube must be plugged during measurement of the calibration air to prevent ingress of moist air.

10.2.2 Allow the dry air in the U-tube to come to thermal equilibrium with the test temperature and record the  $T$ -value for air.

10.2.3 Introduce a small ~~volume (about volume, about 1 mL to 2 mL)~~ 2 mL, of ~~re-distilled~~ freshly boiled reagent water into the sample tube using a suitable syringe. (See Warning note in 7.3.) The water must be free of even the smallest air or gas bubbles. The sample tube shall be completely full. Allow the water to reach thermal equilibrium at the test temperature and record the  $T$ -value for water and the test temperature.

10.2.4 Alternatively introduce one of the hydrocarbon calibration standards and measure the  $T$ -value as in 10.2.3.

10.2.5 Calculate the density of air at the temperature of test using the following equation:

$$d_a = 0.001293[273/T][P/760] \text{ g/mL} \quad (1)$$

$$d_a = 0.001293[273.15/T][P/101.325] \text{ g/mL} \quad (1)$$

where:

$T$  = temperature, K, and

$P$  = barometric pressure, torr.

$P$  = barometric pressure, kPa.

10.2.6 Determine the density of water at the temperature of test by reference to **Table 1**.

10.2.7 Alternatively record the density at the test temperature for the hydrocarbon calibrant used in 10.2.4 as obtained from an appropriate reference source or from direct determination (see 7.6).

10.2.8 Using the observed  $T$ -values and the reference values for water and air, calculate the values of the Constants A and B using the following equations:

$$A = [T_w^2 - T_a^2][d_w - d_a] \quad (2)$$

$$B = T_a^2 - (A \times d_a) \quad (3)$$

where:

$T_w$  = observed period of oscillation for cell containing water,

$T_a$  = observed period of oscillation for cell containing air,

$d_w$  = density of water at test temperature, and

$d_a$  = ~~density of air at test temperature.~~

$T_w$  = observed period of oscillation for cell containing water,  $\mu$ s,

$T_a$  = observed period of oscillation for cell containing air,  $\mu$ s,

$d_w$  = density of water at test temperature, g/mL, and

$d_a$  = density of air at test temperature, g/mL.

Alternatively, use the  $T$  and  $d$  values for the other reference liquid if one is used.

10.2.9 If the instrument is equipped to calculate density from the Constants A and B and the observed  $T$ -value from the sample, then enter the constants in the instrument memory in accordance with the manufacturer's instructions.

10.2.10 Check the calibration and adjust if needed by performing the routine calibration check described in 10.3.