Designation: D4052 – 22

Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter[1]

This standard is issued under the fixed designation D4052; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers the determination of the density, relative density, and API Gravity of petroleum distillates and viscous oils that can be handled in a normal fashion as liquids at the temperature of test, utilizing either manual or automated sample injection equipment. Its application is restricted to liquids with total vapor pressures (see Test Method D5191) typically below 100 kPa and viscosities (see Test Method D445 or D7042) typically below about 15 000 mm^2/s at the temperature of test. The total vapor pressure limitation however can be extended to >100 kPa provided that it is first ascertained that no bubbles form in the U-tube, which can affect the density determination. Some examples of products that may be tested by this procedure include: gasoline and gasoline-oxygenate blends, diesel, jet, basestocks, waxes, and lubricating oils.

1.1.1 Waxes and highly viscous samples were not included in the 1999 interlaboratory study (ILS) sample set that was used to determine the current precision statements of the method, since all samples evaluated at the time were analyzed at a test temperature of 15 °C. Wax and highly viscous samples require a temperature cell operated at elevated temperatures necessary to ensure a liquid test specimen is introduced for analysis. Consult instrument manufacturer instructions for appropriate guidance and precautions when attempting to analyze wax or highly viscous samples. Refer to the Precision and Bias section of the method and Note 9 for more detailed information about the 1999 ILS that was conducted.

1.2 In cases of dispute, the referee method is the one where samples are introduced manually as in 6.2 or 6.3, as appropriate for sample type.

1.3 When testing opaque samples, and when not using equipment that is capable of automatic bubble detection, proper procedure shall be established so that the absence of air bubbles in the U-tube can be established with certainty. For the determination of density in crude oil samples use Test Method D5002.

1.4 The values stated in SI units are regarded as the standard, unless stated otherwise. The accepted units of measure for density are grams per millilitre (g/mL) or kilograms per cubic metre (kg/m^3).

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.* For specific warning statements, see 3.2.1, Section 7, 9.1, 10.2, and Appendix X1.

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]

D287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D1193 Specification for Reagent Water

D1250 Guide for the Use of the Joint API and ASTM Adjunct for Temperature and Pressure Volume Correction Factors for Generalized Crude Oils, Refined Products, and Lubricating Oils: API MPMS Chapter 11.1

D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

[1] 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.

Current edition approved May 1, 2022. Published May 2022. Originally approved in 1981. Last previous edition approved in 2018 as D4052 – 18a. DOI: 10.1520/D4052-22.

[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.

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

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
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
D5191 Test Method for Vapor Pressure of Petroleum Products and Liquid Fuels (Mini Method)
D7042 Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.1.2 *density, n*—mass per unit volume.

3.1.2.1 *Discussion*—The SI unit of density is kg/m^3; the unit of measure g/cm^3 is commonly used in industry.

3.1.3 *relative density, n*—the ratio of the density of a material at a stated temperature to the density of water at a stated temperature.

3.1.3.1 *Discussion*—Relative density is also commonly known as specific gravity. Commonly used stated temperatures are 20 °C⁄20 °C, 15 °C⁄15 °C, 20 °C⁄4 °C and 60 °F⁄60 °F. "Relative density" was historically known as the deprecated term "specific gravity."

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *adjustment, v*—the operation of bringing the instrument to a state of performance suitable for its use, by setting or adjusting the density meter constants.

3.2.1.1 *Discussion*—On some digital density analyzer instruments, an adjustment may be made rather than calibrating the instrument. The adjustment procedure uses air and freshly boiled reagent water (**Warning—**Handling water at boiling or near boiling temperature can present a safety hazard. Wear appropriate personal protective equipment.) as standards to establish the linearity of measurements over a range of operating temperatures.

3.2.2 *API gravity, n*—a special function of relative density 60 °F⁄60 °F, represented by:

$$°\text{API} = \frac{141.5}{\text{relative density}} - 131.5 \quad (1)$$

3.2.2.1 *Discussion*—No statement of reference temperature is required since 60 °F is included in the definition.

3.2.3 *calibration, v*—set of operations that establishes the relationship between the reference density of standards and the corresponding density reading of the instrument.

3.2.4 *sample aliquot, n*—the fraction of the original laboratory sample dedicated for this test.

3.2.4.1 *Discussion*—The sample aliquot is typically residing in syringes, sample vials, beakers, or containers for the purpose of transferring a representative test specimen into the apparatus' U-tube.

3.2.5 *test specimen, n*—the volume of the sample aliquot residing in the U-tube during the measurement cycle.

3.2.5.1 *Discussion*—Sample material residing in filling nozzles, tubing and valve manifolds is not considered "Test Specimen." A test specimen can be measured only once.

4. Summary of Test Method

4.1 A volume of approximately 1 mL to 2 mL of liquid sample is introduced into an oscillating U-tube and the change in oscillating frequency caused by the change in the mass of the U-tube is used in conjunction with calibration data to determine the density, relative density, or API Gravity of the sample. Both manual and automated injection techniques are described.

5. Significance and Use

5.1 Density is a fundamental physical property that can be used in conjunction with other properties to characterize both the light and heavy fractions of petroleum and petroleum products.

5.2 Determination of the density or relative density of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperature of 15 °C.

6. Apparatus

6.1 *Digital Density Analyzer*—A digital analyzer consisting of a U-shaped, oscillating tube, U-tube, and a system for electronic excitation, frequency counting, and display. The analyzer shall accommodate the accurate measurement of the sample temperature during measurement or shall control and keep the sample temperature constant to ±0.05 °C. The instrument shall be capable of meeting the precision requirements described in this test method.

6.2 *Syringes,* for use primarily in manual injections, at least 2 mL in volume with a tip or an adapter tip that will fit the opening of the U-tube.

6.3 *Flow-Through or Pressure Adapter,* for use as an alternative means of introducing the sample into the density analyzer either by a pump, by pressure, or by vacuum.

NOTE 1—It is highly recommended that a vacuum not be applied to samples prone to light-end loss, as it can easily lead to the formation of bubbles in the U-tube. It is recommended to fabricate a special cap or stopper for sample containers so that air, such as from a squeeze pump, is used to displace a test specimen to the U-tube measuring cell by the flow-through method.

6.4 *Autosampler,* required for use in automated injection analyses. The autosampler shall be designed to ensure the integrity of the test specimen prior to and during the analysis and be equipped to transfer a representative portion of sample aliquot to the digital density analyzer.

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

6.5 *Ultrasonic Bath, Unheated,* (optional), of suitable dimensions to hold container(s) placed inside of bath, for use in effectively dissipating and removing air or gas bubbles that may be entrained in viscous sample types prior to analysis.

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.[4] 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 higher.

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

7.4 *Cleaning Solvent,* such as petroleum naphtha[5] or solvent known to be miscible with the sample material without causing corrosion of the sample cell. Consult manufacturer's information for details. (**Warning—**Petroleum naphtha is extremely flammable.)

7.5 *Acetone,* or other highly volatile solvent for flushing and drying the U-tube. (**Warning—**Extremely flammable.)

7.6 *Dry Air,* for drying the U-tube.

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 capacity to mix the sample and 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*—A portion or volume of sample aliquot obtained from the laboratory sample and delivered to the density analyzer U-tube. The test specimen is obtained as follows:

8.3.1 Mix the sample if required to homogenize, taking care to avoid the introduction of air bubbles. 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 volatile material from certain sample types (for example, gasoline samples), so mixing in closed, pressurized containers or at least 10 °C below ambient temperature is required for such sample types where loss of volatile material is a potential concern. For some sample types, such as viscous lube oils that are prone to having entrained air or gas bubbles present in the sample, the use of an ultrasonic bath (see 6.5) without the heater turned on (if so equipped), has been found effective in dissipating bubbles typically within 10 min.

NOTE 2—When mixing samples with volatile components, consider the sample properties in relation to both ambient temperature and pressure.

8.3.2 For manual injections, draw the test specimen from a properly mixed laboratory sample using an appropriate syringe. If the proper density analyzer attachments and connecting tubes are used, as described in 6.3, then the test specimen can be delivered directly to the analyzer's U-tube from the mixing container. For automated injections, it is necessary to first transfer a portion of sample by appropriate means from a properly mixed laboratory sample to the autosampler vials, and take the necessary steps to ensure the integrity of the test specimen prior to and during the analysis. Sample vials for the autosampler shall be sealed immediately after filling up to 80 % ± 5 % and shall be kept closed until the auto sampler transfers the test specimen into the measuring cell. For highly volatile samples, cool the sample prior to measurement. Follow the manufacturer's instructions.

NOTE 3—Overfilled sample vials can result in cross-contamination between sample vials.

9. Preparation of Apparatus

9.1 Set up the density analyzer following the manufacturer's instructions. Set the internal temperature control so that the desired test temperature is established and maintained in the U-tube of the analyzer. Verify the instrument's calibration at the same temperature at which the density or relative density of the sample is to be measured or perform an adjustment (see 3.2.1–Discussion) in preparation of analyzing samples. (**Warning—**Precise setting and control of the test temperature in the U-tube is extremely important. An error of 0.1 °C can result in a change in density of one in the fourth decimal when measuring in units of grams per millilitre.)

10. Verification and Adjustment

10.1 As a minimum requirement, calibration verification of the instrument is required when first set up and whenever the test temperature is changed. Whenever the apparatus fails a calibration verification without discernible cause, the apparatus must be adjusted. See 3.2.1.

10.2 The adjustment routine for digital density meters involves using a minimum of two reference media. Typically, this will be air and freshly boiled reagent water under atmospheric conditions. (**Warning—**Handling water at boiling or near boiling temperature can present a safety hazard. Wear appropriate personal protective equipment.) Other materials such as *n*-nonane, *n*-tridecane, cyclohexane, and *n*-hexadecane

[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar 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.

[5] Suitable solvent naphthas are marketed under various designations such as "Petroleum Ether," "Ligroine," or "Precipitation Naphtha."