ASTM INTERNATIONAL

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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 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-shaped, oscillating 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 8 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 6.3 or 6.4, as appropriate for sample type.

1.3 This test method should not be applied to samples so dark in color that the absence of air bubbles in the sample cell cannot 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, such as the "torr" units of pressure in Eq 1. 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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see 7.4, 7.5, and 10.3.

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 Use of the Petroleum Measurement Tables

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

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

D5002 Test Method for Density and Relative Density of Crude Oils by Digital Density Analyzer

D5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04.0D on Physical and Chemical Methods.

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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.

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

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

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

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

4. Summary of Test Method

4.1 A small volume (approximately 1 to 2 mL) of liquid 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, 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 sample 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 the sample temperature as described in 6.2. The instrument shall be capable of meeting the precision requirements described in this test method.

6.2 *Circulating Constant-Temperature Bath,* (optional), capable of maintaining the temperature of the circulating liquid

constant to ±0.05°C in the desired range. Temperature control can be maintained as part of the density analyzer instrument package.

6.3 *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 oscillating tube.

6.4 *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. 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.

6.5 *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 test specimen to the digital density analyzer.

6.6 *Temperature Sensing Device (TSD),* capable of monitoring the observed test temperature to within an accuracy of ± 0.05°C. If a liquid-in-glass thermometer is used as the TSD, it shall be calibrated and graduated to 0.1°C, and have a holder that can be attached to the instrument for setting and observing the test temperature. In calibrating the thermometer, the ice point and bore connections should be estimated to the nearest 0.05°C. For non-mercurial thermometers, the TSD device shall be calibrated at least annually against a certified and traceable standard.

6.7 *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.[3] 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,* redistilled, freshly boiled (**Warning**—Handling water at boiling or near boiling temperature can present a safety hazard. Wear appropriate personal protective equipment.) reagent water for use as a primary calibration standard.

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[3] *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 *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.

7.4 *Cleaning Solvent,* such as petroleum naphtha[4] (**Warning**—Petroleum naphtha is extremely flammable), or other materials that are capable of flushing and removing samples entirely from the sample tube.

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

7.6 *Dry Air,* for drying the oscillator 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 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 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 6.7) 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 6.4, then the test specimen can be delivered directly to the analyzer's sample 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.

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

9. Preparation of Apparatus

9.1 Set up the density analyzer (including the constant temperature bath and related attachments, if necessary) 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 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 sample 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 g/mL.)

10. Calibration of Apparatus

10.1 As a minimum requirement, calibration of the instrument is required when first set up, whenever the test temperature is changed (unless the instrument is capable of performing an adjustment; see 3.1.1.1 Discussion), or as dictated by quality control (QC) sample results (see 11.1).

10.2 When calibration of the instrument is required, it is necessary to calculate the values of the constants *A* and *B* from the periods of oscillation (*T*) observed when the sample cell contains air and redistilled, freshly boiled (**Warning**—Handling water at boiling or near boiling temperature can present a safety hazard. Wear appropriate personal protective equipment.) reagent water. Other calibrating materials such as *n*-nonane, *n*-tridecane, cyclohexane, and *n*-hexadecane (for high temperature applications) can also be used as appropriate, provided the reference materials have density values that are certified and traceable to national standards.

NOTE 4—On certain newer, commercially available instruments, a viscosity correction feature may be available and utilized in density determinations to minimize potential biases. Refer to information in the Section 15 for more specifics.

10.2.1 While monitoring the oscillator period, *T*, flush the sample tube with cleaning solvent, followed with an acetone flush and dry with 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 1 to 2 mL) of redistilled, freshly boiled (**Warning**—Handling water at boiling or near boiling temperature can present a safety hazard. Wear appropriate personal protective equipment.) reagent water into the sample tube using a suitable syringe or alternate, as described in 6.4 and 6.5. The test portion must be homogeneous and free of even the smallest air or gas bubbles. Allow the display to reach a steady reading and record the *T*-value for water.

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

TABLE 1 Density of Water[A]

Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL
0.0	0.999840	21.0	0.997991	40.0	0.992212
3.0	0.999964	22.0	0.997769	45.0	0.990208
4.0	0.999972	23.0	0.997537	50.0	0.988030
5.0	0.999964	24.0	0.997295	55.0	0.985688
10.0	0.999699	25.0	0.997043	60.0	0.983191
15.0	0.999099	26.0	0.996782	65.0	0.980546
15.56	0.999012	27.0	0.996511	70.0	0.977759
16.0	0.998943	28.0	0.996231	75.0	0.974837
17.0	0.998774	29.0	0.995943	80.0	0.971785
18.0	0.998595	30.0	0.995645	85.0	0.968606
19.0	0.998404	35.0	0.994029	90.0	0.965305
20.0	0.998203	37.78	0.993042	100	0.958345

[A] Densities conforming to the International Temperature Scale 1990 (ITS 90) were extracted from Appendix G, *Standard Methods for Analysis of Petroleum and Related Products 1991*, Institute of Petroleum, London.

$$d_a, \text{g/mL} = 0.001293[273.15/T][P/760] \qquad (1)$$

where:

T = temperature, K, and
P = barometric pressure, torr.

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

10.2.6 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:

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

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

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.

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

10.2.7 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. Alternatively, if the instrument is equipped to do so, let it make the appropriate corrections in the calibration or adjustment constants as part of the built in calibration or adjustment procedure.

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

10.2.9 To calibrate the instrument to display relative density, that is, the density of the sample at a given temperature referred to the density of water at the same temperature, follow 10.2.1-10.2.7, but substitute 1.000 for d_w in performing the calculations described in 10.2.6.

10.3 On some density meter analyzers, weekly calibration adjustments to constants A and B can be made if required, without repeating the calculation procedure. The need for a change in calibration is generally attributable to deposits in the sample tube that are not removed by the routine flushing procedure. Although this condition can be compensated for by

adjusting A and B, it is good practice to clean the tube with a strong oxidizing acid (**Warning**—Causes severe burns) or surfactant cleaning fluids whenever a major adjustment is required.

10.3.1 Flush and dry the sample tube as described in 10.2.1 and allow the display to reach a steady reading. If the display does not exhibit the correct density for air at the temperature of test, repeat the cleaning procedure or adjust the value of constant B commencing with the last decimal place until the correct density is displayed.

10.3.2 If adjustment to constant B was necessary in 10.3.1 then continue the recalibration by introducing redistilled, freshly boiled (**Warning**—Handling water at boiling or near boiling temperature can present a safety hazard. Wear appropriate personal protective equipment.) reagent water into the sample tube as described in 10.2.3 and allow the display to reach a steady reading. If the instrument has been calibrated to display the density, adjust the reading to the correct value for water at the test temperature (Table 1) by changing the value of constant A, commencing with the last decimal place. If the instrument has been calibrated to display the relative density, adjust the reading to the value 1.0000.

NOTE 5—If performing a weekly calibration adjustment, it can be found that more than one value each for A and B, differing in the fourth decimal place, will yield the correct density reading for the density of air and water. The setting chosen would then be dependent upon whether it was approached from a higher or lower value. The setting selected by this method could have the effect of altering the fourth place of the reading obtained for a sample.

10.4 Some analyzer models are designed to display the measured period of oscillation only (T-values) and their calibration requires the determination of an instrument constant K, which must be used to calculate the density or relative density from the observed data.

10.4.1 Flush and dry the sample tube as described in 10.2.1 and allow the display to reach a steady reading. Record the T-value for air.

10.4.2 Introduce redistilled, freshly boiled (**Warning**—Handling water at boiling or near boiling temperature can present a safety hazard. Wear appropriate personal protective equipment.) reagent water into the sample tube as described in 10.2.3, allow the display to reach a steady reading and record the T-value for water.

10.4.3 Using the observed T-values and the reference values for water and air (10.2.4 and 10.2.5), calculate the instrument constant K using the following equations:

For density:

$$K_1 = [d_w - d_a]/[T_w^2 - T_a^2] \qquad (4)$$

For relative density:

$$K_2 = [1.0000 - d_a]/[T_w^2 - T_a^2] \qquad (5)$$

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.