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.

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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.

*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."

(for high temperature applications) can also be used as appropriate adjustment materials, provided the reference materials have density values that are certified and traceable to national standards.

10.3 Follow the manufacturer's instructions for the proper adjustment of the apparatus. If the apparatus is adjusted using air and reagent water, observe the proper entries of air and water density values.

10.3.1 The density of air varies with pressure and relative humidity (see Table 1). Therefore, it is important that the dewpoint of ambient air is below the adjustment temperature of the instrument as to avoid condensation of water in the U-tube. This can be achieved by flushing ambient air through a desiccant container and into the U-tube.

10.3.2 The density of air varies with ambient pressure as a consequence of site elevation and atmospheric changes in pressure. The air density can be calculated using this formula:

$$\rho_{air} = 0.001293[273.15 / T][P/101.325]\text{g/mL} \quad (2)$$

where:

ρ_{air} = density of air,
T = temperature, K, and
P = site atmospheric pressure at the time of adjustment, kPa.

NOTE 4—P should preferably be determined by direct measurement of the barometric pressure at the site of calibration. If direct measurement is not available, and common sources providing weather data are consulted, the pressure reported is typically corrected to Sea Level, P_{SL}. Therefore, such pressure data must be corrected back to site pressure, P. For correction of P_{SL} to P:

$$P = P_{SL} - [\text{Site Elevation (meters)}/82.3] \quad (3)$$

NOTE 5—In the International Standard Atmosphere, ISA, the pressure drops 1 kPa per 82.3 m of elevation.

10.3.3 The water density values are given in Table 1. Water density values are considered constant with respect to pressure in the range of normally occurring atmospheric pressure.

TABLE 1 Density of Water[A]

NOTE 1—Several metrological entities have issued water density tables and alternative water density data is referenced in publications external to ASTM and this test method. Using water density data from an alternative recognized source does not pose a compliance issue with this test method as the variation in the data typically is limited to the sixth decimal place.

Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL
0.01	0.999844	21.0	0.997996	40.0	0.992216
3.0	0.999967	22.0	0.997773	45.0	0.990213
4.0	0.999975	23.0	0.997541	50.0	0.988035
5.0	0.999967	24.0	0.997299	55.0	0.985693
10.0	0.999703	25.0	0.997048	60.0	0.983196
15.0	0.999103	26.0	0.996786	65.0	0.980551
15.56	0.999016	27.0	0.996516	70.0	0.977765
16.0	0.998946	28.0	0.996236	75.0	0.974843
17.0	0.998778	29.0	0.995947	80.0	0.971790
18.0	0.998599	30.0	0.995650	85.0	0.968611
19.0	0.998408	35.0	0.994033	90.0	0.965310
20.0	0.998207	37.78	0.993046	99.9	0.958421

[A] Densities conforming to the International Temperature Scale 1990 (ITS 90) were extracted from Lemmon, E. W., McLinden, M. O., and Friend, D. G., "Thermophysical Properties of Fluid Systems," *NIST Chemistry WebBook*, NIST Standard Reference Database No. 68, Eds. P.J. Linstrom and W.G. Mallard, National Institute of Standards and Technology, Gaithersburg, MD, http://webbook.nist.gov. (retrieved July 24, 2013).

11. Quality Control Checks

11.1 Confirm the instrument is in statistical control at least once a week when it is in use, by analyzing a quality control (QC) sample that is representative of samples typically analyzed. Analysis of a single QC sample can be sufficient. Analysis of QC sample results can be carried out using control chart techniques.[6] If the QC sample result determined causes the laboratory to be in an out-of-control situation, such as exceeding the laboratory's control limits, instrument adjustment is required. An ample supply of QC sample material should be available for the intended period of use, and must be homogeneous and stable under the anticipated storage conditions. Prior to monitoring the measurement process, the user of the method needs to determine the average and control limits of the QC sample. The QC sample precision should be checked against the method precision to ensure data quality.

11.2 Although not mandatory, it is recommended that periodic analyses of certified density standards (that is, traceable to national standards) that are separate from those that may be used in adjusting the instrument, be used to confirm testing accuracy.

12. Procedure

12.1 *Manual Injection:*

12.1.1 Introduce a volume of about 1 mL to 2 mL, of sample into the clean, dry U-tube of the instrument using a suitable syringe or alternative, as described in 6.3.

12.1.2 The sample can also be introduced by siphoning. Plug the external TFE-fluorocarbon capillary tube into the entry port of the U-tube. Immerse the other end of the capillary in the sample and apply suction to the other port using a syringe or vacuum line until the U-tube is properly filled (see Note 1).

12.1.3 Ensure that the U-tube is properly filled and that no gas bubbles are present. The sample must be homogeneous and free of even the smallest gas bubbles. Check the integrity of the filled sample by using optical or physical methods to verify absence of gas bubbles. If gas bubbles are detected, empty and refill the U-tube, and recheck for gas bubbles.

NOTE 6—If the sample is too dark in color to determine the absence of bubbles with certainty, the density cannot be measured within the stated precision limits of Section 15.

12.1.4 For most instrument models, it is recommended to turn the illumination light off with minimal delay after sample introduction and checking for bubbles because the heat generated can affect the measurement temperature. For some models however, the cell light may be left on without affecting results. Refer to the manufacturer's recommendations concerning whether to leave the illumination light off or on.

12.1.5 After the instrument displays a steady reading to four significant figures for density, relative density, or API Gravity, and five for *T*-values, indicating that temperature equilibrium has been reached, record the density, relative density, API

[6] ASTM MNL 7, *Manual on Presentation of Data Control Chart Analysis*, Section 3: Control Charts for Individuals, 6th ed., ASTM International, W. Conshohocken, PA.

Gravity or *T*-values, or both, as appropriate. For instruments that can print out results from the display, the print out can be used to meet the recording requirements.

12.1.6 Based on the 1999 ILS[7] testing protocols (see Note 9), precision determinations involving a single manual injection, as well as taking the average of two manual injections, was evaluated. See the Precision and Bias section for more details. If the laboratory performs a single manual determination, the density, relative density, or API Gravity values recorded in 12.1.5, or a combination thereof, is to be used for reporting purposes. In most cases, a single manual injection determination is sufficient for sample analysis.

12.1.6.1 If the laboratory decides to perform a second manual injection determination for a given sample, repeat 12.1.1 – 12.1.5. If the two determinations do not differ by more than 0.0002 g⁄mL for density or 0.0002 for relative density, average the two determinations, otherwise, discard both determinations and repeat the analysis using two new test specimens until the acceptance criteria identified above is satisfied. With respect to API Gravity determinations of gasoline or RFG samples, if the two determinations do not differ by more than 0.05° API Gravity units, average the two determinations, otherwise, discard both determinations and repeat the analysis using two new test specimens until the acceptance criteria is satisfied. With respect to API Gravity determinations of distillates, basestocks, and lubricating oils, follow the protocol in the prior sentence except use 0.03° API Gravity units as the acceptance criteria. Averaged results meeting the criteria identified in this section is to be used for reporting purposes.

12.2 *Automated Injection:*

12.2.1 The use of an autosampler (see 6.4) is required when analyzing samples by automated injection. Follow manufacturer's instructions for ensuring the integrity of the test specimen prior to analysis, as well as transferring a representative test specimen into the instrument for analysis.

12.2.1.1 If the laboratory decides to perform a second automated injection determination for a given distillate, basestock, or lubricating oil (see Note 9 and the Precision and Bias section), use the same acceptance criteria in 12.1.6.1 for allowed differences between each determination for density (0.0002 g⁄mL), relative density (0.0002) and API Gravity units (0.03°) as for manually injected samples. Averaged results meeting the necessary acceptance criteria are to be used for reporting purposes.

NOTE 7—Density meters equipped with autosamplers allow for unattended operation, and erroneous results rooted in filling problems may pass unnoticed. Performing duplicate determinations may uncover sample filling issues regardless of the sample material being tested.

12.2.2 Opaque samples may be tested using either manual injection method described in 12.1 or using an autosampler as described in 12.2. When testing opaque samples, proper procedure shall be established by which the absence of air bubbles in the measuring cell can be confirmed with certainty. Such procedures include multiple determinations and evaluation of closeness of determinations, utilizing optical or physical methods for air bubble detection, or both.

12.2.2.1 This test method does not offer precision and bias values for opaque samples and because sample properties vary greatly, determinability criteria is not offered, but shall be established by a series of tests determinations on representative sample material. Perform a minimum of three consecutive determinations of different test specimens from the same syringe or sample vial without removing the syringe from the apparatus and without any rinsing or cleaning between determinations. Average the two determinations with the highest density or lowest API number and report per 12.2.3.

12.2.3 Record the density, relative density, or API Gravity results, or a combination thereof, determined by the analyses as appropriate, such as by using the instrument print out of results to meet the recording requirements.

13. Calculation

13.1 *Calculating Density Analyzers*—The recorded or printed value is the final result when a single determination is conducted (or taking the average of two determinations as the final result as identified in Section 12), expressed either as density in g/mL, kg/m^3 or as relative density. Note that kg/m^3 = 1000 × g/mL.

13.2 If it is necessary to convert a result obtained using the density meter to API Gravity, or a density or relative density at another temperature, Guide D1250 can be used only if the glass expansion factor has been excluded.

NOTE 8—Some digital density analyzers are equipped to automatically calculate and report sample results in API gravity units (see Test Method D287 or D1298), based on the density or relative density results, or a combination thereof, determined by this test method and using the appropriate conversion equation derived from Guide D1250 (which excludes the glass expansion factor), for the specific sample type being analyzed.

14. Report

14.1 In reporting density, state the test temperature and the units (for example: density at 20 °C = 0.8765 g⁄mL or 876.5 kg⁄m^3).

14.2 In reporting relative density, state both the test temperature and the reference temperature, but no units (for example: relative density, 20/20 °C = 0.xxxx).

14.3 Report the final result for density or relative density to four significant figures and reference this test method.

14.4 If reporting sample results in API gravity units (see Note 8), report the final results to the nearest 0.1° API.

14.5 In some cases, it may be necessary to report if results were based on a single determination or an average of two determinations, as well as if results were determined by manual injection or automated injection to meet specific requirements, such as data recipients wanting to know such information in order to understand how to interpret results relative to the published method precision values. In such cases, it is permissible to report this information along with the test results.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1734. Contact ASTM Customer Service at service@astm.org.