





- 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)
- 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 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 *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 mL 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  $\pm 0.05$  °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  $\pm 0.05$  °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.<sup>3</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.

<sup>3</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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.



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<sup>4</sup> (**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.

## 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 grams per millilitre.)

## 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 **3.2.1** 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 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 mL to 2 mL, of freshly boiled (**Warning**—Handling water at boiling or near

<sup>4</sup> Suitable solvent naphthas are marketed under various designations such as "Petroleum Ether," "Ligroine," or "Precipitation Naphtha."



TABLE 1 Density of Water<sup>A</sup>

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

<sup>A</sup> 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).

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:

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

where:

$T$  = temperature, K, and

$P$  = barometric pressure, kPa.

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:

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

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

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

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

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

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

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 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 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:

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

For relative density:

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

where:

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

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

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

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

## 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.<sup>5</sup> 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 recalibration or 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 calibrating the instrument, be used to confirm testing accuracy.

## 12. Procedure

### 12.1 Manual Injection:

12.1.1 Introduce a small amount, about 1 mL to 2 mL, of sample into the clean, dry sample tube of the instrument using a suitable syringe or alternative, as described in 6.4.

12.1.2 The sample can also be introduced by siphoning. Plug the external TFE-fluorocarbon capillary tube into the entry port of the sample 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 sample tube is properly filled (see Note 1).

12.1.3 Ensure that the sample 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 sample 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 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<sup>6</sup> 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 6.5) 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

<sup>5</sup> ASTM MNL 7, *Manual on Presentation of Data Control Chart Analysis*, Section 3: Control Charts for Individuals, 6th ed., ASTM International, W. Conshohocken, PA.

<sup>6</sup> 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.



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<sup>3</sup> or as relative density. Note that kg/m<sup>3</sup> = 1000 × g/mL.

13.2 *Noncalculating Density Analyzers*—Using the observed  $T$ -value for the sample and the  $T$ -value for water and appropriate instrument constants determined in 10.4.3, calculate the density or relative density using Eq 6 and 7. Carry out all calculations to six significant figures and round the final results to four.

For density:

$$\text{density, g/mL (kg/m}^3\text{) at } t = d_w + K_1(T_s^2 - T_w^2) \quad (6)$$

For relative density:

$$\text{relative density, } t/t = 1 + K_2(T_s^2 - T_w^2) \quad (7)$$

where:

$T_w$  = observed period of oscillation for cell containing water,  
 $T_s$  = observed period of oscillation for cell containing sample,  
 $d_w$  = density of water at test temperature,  
 $K_1$  = instrument constant for density,

$K_2$  = instrument constant for relative density, and  
 $T$  = temperature of test, °C.

13.3 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<sup>3</sup>).

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.

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.

### 15. Precision and Bias<sup>6</sup>

15.1 The precision of the method as obtained by statistical examination of interlaboratory test results at a test temperature of 15 °C is as follows for the following sample types and ranges:

15.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

15.1.1.1 *Density (g/mL) and Relative Density*—See Table 2.

15.1.1.2 *API Gravity*—See Table 3.

15.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

15.1.2.1 *Density (g/mL) and Relative Density*—See Table 4.

15.1.2.2 *API Gravity*—See Table 5.

NOTE 9—The ASTM Interlaboratory Study (ILS) conducted in Fall 1999<sup>6</sup> involved 11 laboratories and 23 samples. The samples included 5 motor gasolines, 1 motor gasoline + ethanol, 4 reformulated gasolines



**TABLE 2 Density (g/mL) and Relative Density (Repeatability)**

Range	Sample Types	Testing Condition	Repeatability
0.71–0.78	Gasoline and RFG	Single Determination (Manual injection)	0.00045
		Average of 2 Determinations (Manual or automated injections)	0.00031
0.80–0.88	Distillates, Basestocks, and Lubricating Oils	Single Determination (Manual or automated injection)	0.00016
		Average of 2 Determinations (Manual or automated injections)	0.00011

**TABLE 3 API Gravity (Repeatability)**

Range	Sample Types	Testing Condition	Repeatability
51–66	Gasoline and RFG	Single Determination (Manual injection)	0.090
		Average of 2 Determinations (Manual or automated injections)	0.063
29–45	Distillates, Basestocks, and Lubricating Oils	Single Determination (Manual or automated injection)	0.032
		Average of 2 Determinations (Manual or automated injections)	0.022

**TABLE 4 Density (g/mL) and Relative Density (Reproducibility)**

Range	Sample Types	Testing Condition	Reproducibility
0.71–0.78	Gasoline and RFG	Single Determination (Manual injection)	0.00190–0.0344 (D–0.75)
		Average of 2 Determinations (Manual or automated injections)	0.00195–0.0315 (D–0.75)
0.80–0.88	Distillates, Basestocks, and Lubricating Oils	Single Determination (Manual or automated injection)	0.00052
		Average of 2 Determinations (Manual or automated injections)	0.00050

where: D = density or relative density value obtained

**TABLE 5 API Gravity (Reproducibility)**

Range	Sample Types	Testing Condition	Reproducibility
51–66	Gasoline and RFG	Single Determination (Manual injection)	0.60 + 0.040 (G–60)
		Average of 2 Determinations (Manual or automated injections)	0.60 + 0.037 (G–60)
29–45	Distillates, Basestocks and Lubricating Oils	Single Determination (Manual or automated injection)	0.133
		Average of 2 Determinations (Manual or automated injections)	0.128

where: G = API Gravity value obtained

(RFG), 2 lube oils, 1 basestock oil, 4 Jet-A fuels, 1 JP8 fuel, and 5 diesel fuels. The ILS protocol required laboratories to perform a total of four measurements per sample, which included both manual and automated injections, with the first two determinations representing the first analysis set and the second two determinations representing the second analysis set. This protocol was used to evaluate if a precision effect existed between taking the first determination from each analysis set (that is, single determination) versus taking the average of the two determinations from each analysis set, as well as determining if there was a precision effect between manual and automated injections. The repeatability and reproducibility statements determined by the statistician reflect the corresponding precision values associated with testing conditions identified. It should be noted that the precision statements determined for the gasoline and RFG sample types in the 1999 ILS were poorer than expected relative to the precision statements determined for the distillates, basestocks, and lubricating oils as well as relative to the previously published method version precision statements (which were based on the analysis of just three pure compounds (benzene, toluene, and xylene) analyzed by 3 laboratories in duplicate). The poorer than expected precision statements for the gasoline and RFG sample types are likely due to the volatile nature

of these materials, even though the ILS participants were provided instructions for how to properly handle these materials to minimize or prevent sample losses. The subcommittee responsible for the method is planning to conduct a subsequent ILS for gasoline and RFG sample types with additional scrutiny paid to sample handling issues as a means to try and improve the precision statements for these materials.

NOTE 10—Four (4) of the samples analyzed in the ILS indicated in **Note 9** were ASTM Interlaboratory Crosscheck samples (that is, Gasoline–MG9808 and MG9812, RFG–RFG9807, and RFG9809). The robust mean of the total vapor pressures reported by Test Method **D5191** for these samples ranged from a low of 7.77 psi (MG9808) to a high of 13.40 psi (RFG9809). This corresponds to an approximate total vapor pressure range of 54 to 92 kPa.

**15.2 Bias**—After suggestions of its existence from literature,<sup>7</sup> a study has been performed which has confirmed

<sup>7</sup> Fitzgerald, H. and D., “An Assessment of Laboratory Density Meters,” *Petroleum Review*, November 1992, pp. 544–549.



the presence of a bias between known density values for reference materials and from values determined according to this test method on the same reference materials. The matrix for this bias study comprised 15 participants, each analyzing four reference oils with certified density values, established by the Netherlands Meet Instituut (NMI), by pycnometry, covering densities in the range of 747 kg/m<sup>3</sup> to 927 kg/m<sup>3</sup> at 20 °C, with viscosities between 1 mPa.s and 5000 mPa.s (also at 20 °C). This study is documented in ASTM Research Report RR:D02-1387.<sup>8</sup> Method users should, therefore, be aware that

results obtained by this test method can be biased by as much as 0.6 kg/m<sup>3</sup> (0.0006 g/mL).

NOTE 11—Newer instrumentation exists which purports to correct density results due to the influence of viscosity. However, the responsible ASTM Technical Committee has not yet performed a new interlaboratory study to confirm the elimination of the bias described in ASTM Research Report RR:D02-1387.<sup>8</sup>

## 16. Keywords

16.1 API Gravity; density; digital density analyzer; petroleum distillates; petroleum products; relative density

<sup>8</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1387. Contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org).

## SUMMARY OF CHANGES

Subcommittee D02.04 has identified the location of selected changes to this standard since the last issue (D4052 – 16) that may impact the use of this standard. (Approved May 1, 2018.)

- |  |                                     |
|--|-------------------------------------|
| <p>(1) Revised subsection <b>12.2</b>, deleting former subsections 12.2.2, 12.2.3, and 12.2.4; subsequent subsections were renumbered accordingly.</p> | <p>(2) Added new <b>Note 7</b>.</p> |
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