Designation: D5002 – 16

Standard Test Method for
Density and Relative Density of Crude Oils by Digital Density Analyzer[1]

1. Scope*

1.1 This test method covers the determination of the density or relative density of crude oils that can be handled in a normal fashion as liquids at test temperatures between 15 °C and 35 °C utilizing either manual or automated sample injection equipment. This test method applies to crude oils with high vapor pressures provided appropriate precautions are taken to prevent vapor loss during transfer of the sample to the density analyzer.

1.2 This test method was evaluated in round robin testing using crude oils in the 0.75 g/mL to 0.95 g/mL range. Lighter crude oil can require special handling to prevent vapor losses. Heavier crudes can require measurements at higher temperatures to eliminate air bubbles in the sample.

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

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in 7.4, 7.5, and 7.6.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer (Withdrawn 1993)[3]
D1193 Specification for Reagent Water
D1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer
D1250 Guide for Use of the Petroleum Measurement Tables
D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration

3. Terminology

3.1 *Definitions:*

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

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

4. Summary of Test Method

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

5.2 The density or relative density of crude oils is used for the conversion of measured volumes to volumes at the standard temperatures of 15 °C or 60 °F and for the conversion of crude mass measurements into volume units.

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

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

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[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.

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

6. Apparatus

6.1 *Digital Density Analyzer*—A digital analyzer consisting of a U-shaped, oscillating sample tube and a system for electronic excitation, frequency counting, and display. The analyzer must accommodate the accurate measurement of the sample temperature during measurement or must control the sample temperature as described in 6.2 and 6.6. The instrument shall be capable of meeting the precision requirements described in Test Method D4052.

6.2 *Circulating Constant-Temperature Bath,* capable of maintaining the temperature of the circulating liquid constant to ±0.05 °C in the desired range. Temperature control can be maintained as part of the density analyzer instrument package.

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

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

6.5 *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 *Thermometer,* calibrated and graduated to 0.1 °C, and a thermometer holder that can be attached to the instrument for setting and observing the test temperature. In calibrating the thermometer, the ice point and bore corrections should be estimated to the nearest 0.05 °C. 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 significant figure.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[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 better.

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

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

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

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

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

8. Sampling, Test Specimens, and Test Units

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

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

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

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

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

9. Preparation of Apparatus

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

10. Calibration of Apparatus

10.1 Calibrate the instrument when first setting up and whenever the test temperature is changed. Thereafter, conduct

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

calibration checks at least weekly during routine operation or more frequently as may be dictated by the nature of the crude oils being measured (see 10.3).

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

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

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

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

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

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

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

where:

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

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

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

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

$$A = [T_w^{\,2} - T_a^{\,2}]/[d_w - d_a] \tag{2}$$

$$B = T_a^{\,2} - (A \times d_a) \tag{3}$$

where:

T_w = observed period of oscillation for cell containing water, μs,
T_a = observed period of oscillation for cell containing air, μs,
d_w = density of water at test temperature, g/mL, and
d_a = density of air at test temperature, g/mL.

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

TABLE 1 Density of Water[A] (in vacuo)

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).

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

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

10.2.11 To calibrate the instrument to determine 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 – 10.2.9, but substitute 1.000 for d_w in performing the calculations described in 10.2.8.

10.3 Since some crude oils can be difficult to remove from the sample tube, frequent calibration checks are recommended. These checks and any subsequent adjustments to Constants A and B can be made if required, without repeating the calculation procedure.

NOTE 2—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, as described below, it is good practice to clean the tube with warm chromic acid solution (**Warning—**Causes severe burns. A recognized carcinogen.) whenever a major adjustment is required. Chromic acid solution is the most effective cleaning agent; however, surfactant-type cleaning fluids have also been used successfully.

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 *T*-value or 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 reagent water into the sample tube as described in 10.2.3 and allowing the display to reach a steady reading. (See Warning note in 7.3.) If the instrument has been calibrated to display the density, adjust the reading to the correct value for water at the test temperature (see 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 3—In applying this periodic calibration procedure, it has been found that more than one value each for A and B, differing in the fourth decimal place, will yield the correct 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, that must be used to calculate the density or relative density from the observed data. Use the procedure in 10.4.1, 10.4.2, and 10.4.3 in this case.

10.4.1 Flush and dry the sample tube as described in 10.2.1 and allow the air to reach equilibrium at the test temperature and the readout to display a steady value. Record the *T*-value for air.

10.4.2 Introduce a small volume, 1 mL to 2 mL, of freshly-boiled reagent water described in 10.2.3, allow the display to reach a steady reading and record the *T* -value for water. (See Warning note in 7.3.)

10.4.3 Using the observed *T*-values and the reference values for water and air (10.2.5 and 10.2.6), calculate the instrument constant, *K*, using the following equations:

for density:

$$K_1 = \left[d_w - d_a\right]/\left[T_w^2 - T_a^2\right] \tag{4}$$

for relative density:

$$K_2 = \left[1.0000 - d_a\right]/\left[T_w^2 - T_a^2\right] \tag{5}$$

where:

T_w = observed period of oscillation for cell containing water, µs,
T_a = observed period of oscillation for cell containing air, µs,
d_w = density of water at test temperature, g/mL, and
d_a = density of air at test temperature, g/mL.

11. Procedure

11.1 Introduce about 1 mL to 2 mL of crude oil into the clean, dry, sample tube of the instrument using a suitable syringe. Leave the syringe in place and plug the exit port.

11.1.1 Ensure that the sample tube is properly filled and that no gas bubbles are present. The sample shall 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.

11.1.2 Allow the sample to equilibrate to the test temperature before proceeding to evaluate the test sample for the presence of unseen air or gas bubbles.

11.1.3 For dark crude oil samples the observation of air or gas bubbles in the sample tube is very difficult. The presence of bubbles can often be detected, however, by observing the fluctuations of the digital display of the *T*-value or density value. Air or gas bubbles cause large random variations in the third and fourth significant figures for density reading and fifth and sixth significant figures for *T* readings. When bubbles are absent and the sample is at equilibrium with the test temperature, the displayed values are stable, do not drift, and show only small variations of the order of ±1 to 2 units in the last significant figure. If stable values are not observed after a few minutes, then repeat the injection of a new sample into the tube.

NOTE 4—When viscous liquids are being measured, a stable reading can be achieved even when air or gas bubbles are present. Careful injection of fresh sample will often eliminate bubbles. Since bubbles contribute to lower density readings, an observed increase in the density of the liquid after injection of fresh sample tends to suggest that bubbles were previously present.

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

11.1.5 Flush and dry the sample tube as described in 10.2.1 and check the calibration as described in 10.3.1 prior to introducing another sample.

11.2 *Automated Injection:*

11.2.1 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 for transferring a representative test specimen into the instrument for analysis.

11.2.2 When using an autosampler for samples expected or known to contain high quantities of volatile components, use two separate test specimens per sample, in order that errors due to potential sample handling of volatile materials and potential gas bubble formation may be detected and the system performance monitored. For all other samples, a single determination using an autosampler is sufficient.

11.2.2.1 If the lab decides to perform a second automated injection determination for a given sample, the differences between each determination should not exceed a determinability criterion determined by a series of tests on a representative crude sample and which assures that the repeatability performance of 14.1.1, Table 2 is met. Averaged results meeting the necessary acceptance criteria are to be used for reporting purposes.

11.2.2.2 If the two determinations fall outside this acceptance criteria, both determinations are to be discarded and 11.2.2 shall be repeated until the acceptance criteria identified in the previous sentence is satisfied. In cases where the acceptance criteria is not initially satisfied, the lab may need to investigate and take corrective actions before proceeding with subsequent analyses.

11.3 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.

12. Calculation

12.1 *Calculating Density Analyzers*—The recorded value is the final result, expressed either as density in g/mL, kg/m^3 or as relative density. Note that kg/m^3 = 1000 × g/mL.

12.2 *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 Eq 7. Carry out all calculations to six significant figures and round the final results to four. Note that kg/m^3 = 1000 × g/mL.

for density:

$$\text{density, g/mL at } t = d_w + K_1\left(T_s^{\,2} - T_w^{\,2}\right) \tag{6}$$

for relative density:

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

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.

12.3 If it is necessary to convert a result obtained using the density analyzer to a density or relative density at another temperature, Guide D1250 can be used only if the table values have not been corrected for the glass expansion factor.

13. Report

13.1 In reporting density, give the test temperature and the units, (for example: density at 20 °C = 0.8765 g/mL or 876.5 kg/m^3 (in vacuo)).

13.2 In reporting relative density, give both the test temperature and the reference temperature, but no units, (for example: relative density, 15/15 °C = x.xxxx).

13.3 Report the final result to four significant figures.

14. Precision and Bias[5,6]

14.1 The precision of this test method as obtained by statistical examination of interlaboratory test results at test temperatures of 15 °C and 20 °C is as follows:

TABLE 2 Precision Values

Density	Repeatability	Reproducibility
0.70	0.0007	0.0029
0.75	0.0008	0.0031
0.80	0.0008	0.0033
0.85	0.0009	0.0035
0.90	0.0009	0.0037
0.95	0.0010	0.0039

14.1.1 *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 this test method, exceed the following value only in 1 case in 20 (see Table 2):

range	repeatability
0.75 to 0.95	0.00105*X*

where:

X = sample mean.

14.1.2 *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 1 case in 20 (see Table 2):

range	reproducibility
0.75 to 0.95	0.00412*X*

where:

X = sample mean.

14.2 *Bias*—After suggestions of its existence from literature,[7] a study has been performed which has confirmed 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 was comprised of 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^3 to 927 kg/m^3 at 20 °C, with viscosities between 1 mPa.s and 5000 mPa.s (also at 20 °C). Users should, therefore, be aware that results obtained by this test method can be biased by as much as 0.6 kg/m^3 (0.0006 g/mL).[8]

15. Keywords

15.1 crude oils; density; digital density analyzer; relative density

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1257.

[6] Biased results for high viscosity samples (>ca. 100 mPa-s dynamic viscosity) has been reported in the literature. For additional information, consult the *Journal of Physical Chemistry*, Vol 84, 1980, pp. 158–162 and the *Journal of the Chemical Society Faraday Translation* , Vol 86(1), 1990, pp. 145–149.

[7] Fitzgerald, H. and D., "An Assessment of Laboratory Density Meters," *Petroleum Review*, November 1992, pp. 544–549.

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1387.

SUMMARY OF CHANGES

Subcommittee D02.04 has identified the location of selected changes to this standard since the last issue (D5002 – 15) that may impact the use of this standard. (Approved Jan. 1, 2016.)

(1) Revised subsections 1.1, 4.1, and 8.3.2.
(2) Added new subsection 6.5.
(3) Renumbered subsection 11.1 and its subsections; added new subsections 11.2 and 11.3.

Subcommittee D02.04 has identified the location of selected changes to this standard since the last issue (D5002 – 13) that may impact the use of this standard. (Approved Dec. 15, 2015.)

(1) Revised Table 1, density of water.
(2) Replaced "redistilled water" with "reagent water" throughout.

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