Designation: D 5931 – 96

An American National Standard

Standard Test Method for Density and Relative Density of Engine Coolant Concentrates and Aqueous Engine Coolants by Digital Density Meter[1]

1. Scope

1.1 This test method covers the determination of the density or relative density of engine coolant concentrates and aqueous engine coolants.

1.2 This test method should not be applied to samples so dark in color that the absence of air bubbles in the sample cell cannot be established with certainty.

1.3 The accepted units of measure for density are grams per milliliter or kilograms per cubic meter.

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.* For specific hazard statements, see Note 1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter[3]

3. Terminology

3.1 *Definitions:*

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

4. Summary of Test Method

4.1 A small volume 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 of the sample.

5. Significance and Use

5.1 Density is a fundamental physical property that can be used in conjunction with other properties to characterize engine coolant concentrates and aqueous engine coolants.

5.2 Determination of the density or relative density of these products is necessary for the conversion of measured volumes to volumes at the standard temperature of 20°C (68°F).

6. Apparatus

6.1 *Digital Density 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. The instrument must meet the precision requirements described in this test method.

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

6.3 *Syringes*, 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 or by vacuum.

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

7. Reagents and Materials

7.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, 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

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[1] This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants and is the direct responsibility of Subcommittee D15.03 on Physical Properties.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[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 *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.

permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water mean reagent water as defined by Type II of Specification D 1193.

7.3 *Water*, redistilled, freshly boiled and cooled reagent water for use as a primary calibration standard.

7.4 *Acetone*, for flushing and drying the sample tube.

NOTE 1—**Warning:** Acetone is extremely flammable.

7.5 *Dry Air*, for drying the oscillator tube.

8. Preparation of Apparatus

8.1 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.

NOTE 2—Precise setting and control of the test temperature in the sample tube is extremely important. An error of 1.0°C can result in a change in density of one in the third decimal place.

9. Calibration of Apparatus

9.1 Calibrate the instrument when first set up and whenever the test temperature is changed. Thereafter, conduct calibration checks at weekly intervals during routine operation.

9.2 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 air and redistilled, freshly boiled and cooled reagent water.

9.2.1 While monitoring the oscillator period, (*T*), flush the sample tube with distilled water followed by an acetone flush and drying 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.

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

9.2.3 Introduce a small volume of redistilled, freshly boiled and cooled reagent water into the sample tube opening using a suitable syringe. The test portion must be homogeneous and free of even the smallest air or gas bubbles. The sample tube does not have to be completely full as long as the liquid meniscus is beyond the suspension point. Allow the display to reach a steady reading and record the *T*-value for water.

9.2.4 Record the density of air at the temperature and atmospheric pressure of the test. Calculate the density of air at the temperature of test using the following equation:

$$d_a\text{, g/m}L = 0.001293[273.15/T][P/760] \tag{1}$$

where:

T = temperature, degrees Kelvin, K, and

P = barometric pressure, torr.

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

9.2.6 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:

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

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

where:

T_w = observed period of oscillation for cell containing water,

T_a = observed period of oscillation for cell containing air,

d_w = density of water at test temperature, °C, and

d_a = density of air at test temperature,° C.

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

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

9.2.8 Check the calibration and adjust if needed by performing the routine calibration check described in 9.3.

9.2.9 To calibrate the instrument to display relative density, that is, the density of the sample at a given temperature relative to the density of water at the same temperature, follow 9.2.1-9.2.7, but substitute 1.000 for d_w in performing the calculations described in 9.2.6.

9.3 Weekly calibration adjustments to constants *A* and *B* can be made if required without repeating the calculation procedure.

NOTE 3—The need for a change in calibration generally is 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 sequentially with water, petroleum ether, and acetone.

9.3.1 Flush and dry the sample tube as described in 9.2.1 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.

9.3.2 If adjustment to constant *B* was necessary in 9.3.1, continue the recalibration by introducing redistilled, freshly

TABLE 1 Density of Water[A]

Temperature, ° C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL
0	0.99987	21	0.99802	40	0.99224
3	0.99999	22	0.99780	45	0.99025
4	1.00000	23	0.99756	50	0.98807
5	0.99999	24	0.99732	55	0.98573
10	0.99973	25	0.99707	60	0.98324
15	0.99913	26	0.99681	65	0.98059
15.56	0.99904	27	0.99654	70	0.97781
16	0.99897	28	0.99626	75	0.97489
17	0.99880	29	0.99597	80	0.97183
18	0.99862	30	0.99567	85	0.96865
19	0.99843	35	0.99406	90	0.96534
20	0.99823	37.78	0.99307	100	0.95838

[A] From Test Method D 4052.

boiled and cooled reagent water into the sample tube as described in 9.2.3 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 (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 1.000.

NOTE 4—In applying this weekly calibration procedure, it can be found that more than one value 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 then would be dependent upon whether it is approached from a higher or lower value. The setting selected by this test method could have the effect of altering the fourth place of the reading obtained for a sample.

9.4 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.

9.4.1 Flush and dry the sample tube as described in 9.2.1 and allow the display to reach a steady reading. Record the *T*-value for air.

9.4.2 Introduce redistilled, freshly boiled and cooled reagent water into the sample tube as described in 9.2.3, and allow the display to reach a steady reading and record the *T*-value for water.

9.4.3 Using the observed *T*-values and the reference values for water and air (9.2.4 and 9.2.5), calculate the instrument constant *K* using the following equations:

For density:

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

For relative density:

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

where:

T_w = observed period of oscillation for cell containing water,
T_a = observed period of oscillation for cell containing air,
d_w = density of water at test temperature, °C, and
d_a = density of air at test temperature,° C.

10. Procedure

10.1 Mix the sample, if required, to be completely homogeneous. If suspended solids are present in the sample, filter through a 0.5 µm filter. Introduce a small amount (about 0.7 mL) of sample into the clean, dry sample tube of the instrument using a suitable syringe.

10.2 Turn on the illumination light and examine the sample tube carefully. Make sure that no bubbles are trapped in the tube and that it is filled to just beyond the suspension point. The sample must be homogeneous and free of even the smallest bubbles.

NOTE 5—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 13.

10.3 Turn the illumination light off immediately after sample introduction because the heat generated can affect the measurement temperature.

10.4 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. Calculation

11.1 *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.

11.2 *Noncalculating Density Analyzers*—Using the observed *T*-value for the sample and the *T*-value for water and appropriate instrument constants determined in 9.4.3, 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 five.

For density:

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

For relative density:

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

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. Report

12.1 In reporting density, give the test temperature and the units, for example, density at 20°C = 1.0360 g/mL or 1036 kg/m^3.

12.2 In reporting relative density, give both the test temperature and the reference temperature but no units, for example, relative density at 20/20°C = 1.0361.

12.3 Report the final results to the five significant figures.

13. Precision and Bias [5]

13.1 The precision of this test method as obtained by statistical examination of interlaboratory test results at test temperature of 20°C is as follows:

13.1.1 *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, be expected to be as follows:

Range	Repeatability	Standard Deviation

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[5] Supporting data are available from ASTM Headquarters. Request RR:D15-1011.

1.0360–1.1140 g/mL	0.0002	0.0008

13.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test materials would, in the long run, in the normal and correct operation of this test method, be expected to be as follows:

Range	Reproducibility	Standard Deviation
1.0360–1.1140 g/mL	0.0020	0.0008

13.2 *Bias*—This test method is an absolute measure of physical property so no bias is expected.

14. Keywords

14.1 aqueous engine coolants; density; digital density analyzer; engine coolant concentrates; relative density

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