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Standard Test Methods for Specific Gravity and Density of Halogenated Organic Solvents and Their Admixtures[1]

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This standard has been approved for use by agencies of the Department of Defense.

[1] These test methods are under the jurisdiction of ASTM Committee D26 on Halogenated Organic Solvents and Fire Extinguishing Agents and are the direct responsibility of Subcommittee D26.04 on Test Methods.

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1. Scope

1.1 These test methods cover the determination of the specific gravity of halogenated organic solvents and solvent admixtures. They define suitable apparatus and procedures and furnish details underlying the interpretation of test data and the selection of numerical limits for agreement among interested persons and agencies.

1.2 T~~wo~~hree methods are covered as follows:

1.2.1 *Method A*, specific gravity by means of a hydrometer.

1.2.2 *Method B*, specific gravity and density by means of a pycnometer.

NOTE 1—In referee problems, Method B may be used.

1.2.3 *Method C*, specific gravity and density by means of an electronic densitometer.

1.3 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 1 Specification for ASTM Thermometers[2]

E 100 Specification for ASTM Hydrometers[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *specific gravity*—the ratio of the mass in air of a given volume of the material at a stated temperature to the mass in air of an equal volume of distilled water at a stated temperature.

3.1.1.1 *Discussion*—When the temperature of the material and of the water are the same, the specific gravity of the material is expressed as follows:

$$\text{Specific gravity } x/x°\text{C, example } 25/25°\text{C} \quad (1)$$

When the temperature of the material and of the water are not the same, the specific gravity of the material is expressed as follows:

$$\text{Specific gravity } x/y°\text{C, example } 20/4°\text{C} \quad (2)$$

Note that when the density of water is expressed as 4°C, the specific gravity at the stated temperature is the same as density at the stated temperature. For example, SG 20/4°C = density at 20°C.

When using an electronic densitometer to determine specific gravity, the temperature of the material to be tested and the water reference will be the same. Examples 25/25°C, 20/20°C.

3.1.2 *density*—the mass of a given material per unit volume.

3.1.2.1 *Discussion*—Density for chlorinated solvents is normally stated in grams per cubic centimetre. Pounds per gallon is also commonly used.

[2] *Annual Book of ASTM Standards*, Vol 14.03.

4. Significance and Use

4.1 The density or specific gravity of a pure chlorinated solvent at a given temperature is constant. Density or specific gravity can be used in identification of materials, the assay of binary mixtures, and as an indication of purity of a given solvent.

5. Test Temperatures

5.1 ASTM specifications normally state the temperatures for specific gravity of halogenated organic solvents at 25/25°C. 20/20°C and 60/60°F are other commonly used temperatures.

METHOD A—SPECIFIC GRAVITY BY MEANS OF A HYDROMETER

6. Apparatus

6.1 *Hydrometer*—The hydrometers to be used shall be those specified in Specification E 100, as follows:

Nominal Specific Gravity Range	ASTM Hydrometer No.
0.900 to 0.950	107H
0.950 to 1.000	108H
1.000 to 1.050	125H
1.050 to 1.100	126H
1.100 to 1.150	127H
1.150 to 1.200	128H
1.200 to 1.250	129H
1.250 to 1.300	130H
1.300 to 1.350	131H
1.350 to 1.400	132H
1.400 to 1.450	133H
1.450 to 1.500	134H
1.500 to 1.550	135H
1.550 to 1.600	136H
1.600 to 1.650	137H

6.2 *Hydrometer Cylinder*—The vessel in which the sample for the gravity test is confined shall be made of clear glass and shall be cylindrical in shape. For convenience in pouring, it may have a lip on the rim. The inside diameter shall be at least 25.0 mm (1.0 in.) greater than the outside diameter of the hydrometer used in it. The height of the cylinder shall be such that the length of the column of sample it contains is greater by at least 25.0 mm than the portion of the hydrometer that is immersed beneath the surface of the sample after a state of equilibrium has been reached.

6.3 *Thermometer*— An ASTM Gravity Thermometer having a range from − 20 to + 102°C and conforming to the requirements for Thermometer 12C as prescribed in Specification E 1.

6.4 *Water Bath*, capable of maintaining ~~a~~ the test temperature ~~of 25.0~~ ± 0.5°C during the test.

7. Procedure

7.1 Cool the sample in the original container to about ~~24°C.~~ 1°C below the test temperature. Rinse each piece of equipment with a portion of the sample. Pour the sample into the clean hydrometer cylinder without splashing, so as to avoid formation of air bubbles. Remove any air bubbles adhering to the surface by touching them with a piece of clean filter paper. Select a location that is free of air currents. Place the cylinder vertically in the water bath and let the temperature of the sample reach ~~25.0 ± 0.5°C~~ the test temperature as follows: Stir the contents of the cylinder, being careful to avoid formation of air bubbles. When the temperature of the sample is ~~24.5°C,~~ 0.5°C below the test temperature, slowly and carefully lower the hydrometer into the sample to a level two smallest scale divisions below that at which it will float, and then release the hydrometer. After it has come to rest and floats freely away from the walls of the cylinder, read the gravity as the point at which the surface of the sample apparently cuts the hydrometer scale.

7.2 When the ~~temperature~~ sample is ~~25.0°C,~~ at the test temperature, make this observation by placing the eye slightly below the level of the liquid and slowly raise the eye until the surface of the sample first seen as a distorted ellipse seems to become a straight line cutting the hydrometer scale. Determine the temperature of the sample just before and also, for referee tests, just after reading the hydrometer.

METHOD B—SPECIFIC GRAVITY OR DENSITY BY MEANS OF A PYCNOMETER

8. Apparatus

8.1 *Pycnometer*, 25-mL capacity with a ground-glass stopper having a capillary opening, a chamber to provide for expansion up to room temperature, and a cap to prevent evaporation.

8.2 *Water Bath*, capable of maintaining ~~a~~ the temperature ~~of 25.0 ± 0.5°C, or other required temperature,~~ 0.5°C during the test.

8.3 *Thermometer*— An ASTM Low Softening Point Thermometer having a range from − 2 to + 80°C and conforming to the requirements for Thermometer 15C as prescribed in Specification E 1.

8.4 *Analytical Balance*, having a sensitivity of ±0.1 mg.

9. Procedure—Specific Gravity

9.1 Clean the pycnometer by filling it with a saturated solution of chromic acid in concentrated sulfuric acid ~~(H_2SO_4, sp gr 1.84),~~ allowing it to stand for a few hours, emptying, and rinsing well with distilled or deionized water.

9.2 Fill the pycnometer with freshly boiled distilled or deionized water that has been cooled to ~~22 to 24°C.~~ 2 or 3°C below the test temperature. Place it in the water bath maintained at ~~25.0 ± 0.5°C~~ the test temperature until the pycnometer and its contents are at a constant ~~volume at 25°C.~~

~~9.2 After~~ volume.

9.3 After immersion in the bath for at least 30 min, adjust the level of liquid to the proper point on the pycnometer, put the stopper in place, remove from the bath, wipe dry, and weigh. ~~The drying and weighing procedure should be carried out as rapidly as possible to avoid changing the temperature from 25.0°C.~~ Care should be taken to avoid touching the pycnometer with bare hands due to weight changes that will occur from picking up moisture and oils. ~~Empty~~ Record the ~~pycnometer, rinse successively with alcohol and the solvent being tested, remove the vapor of the solvent, immerse in the bath, and bring to 25.0°C as was done before. After immersion at 25.0°C for at least 30 min, put the stopper in place, remove from the bath, wipe dry, and weigh. Subtract the~~ weight ~~of the empty pycnometer from the weight when filled with water in order to get the weight of the contained water at 25.0°C in air. Call this difference~~ as ~~*W*~~ W_1.

~~9.3 Cool~~

9.4 Empty the ~~sample to 22 to 24°C, fill~~ pycnometer, rinse successively with alcohol or acetone, remove the ~~pycnometer~~ vapor of the solvent by purging with clean, dry air or nitrogen, immerse in the bath, and bring to ~~25.0°C~~ the test temperature as was done before. After immersion at ~~25.0°C~~ the test temperature for at least 30 min, ~~adjust the liquid level,~~ put the stopper in place, remove from the bath, wipe dry, and weigh. ~~Subtract~~ Record the weight ~~of the empty pycnometer from the weight when filled with sample in order to obtain the weight of the contained sample at 25.0°C. Call this difference~~ as SW_2.

9.5 Subtract the weight of the empty pychometer from the weight when filled with water in order to get the weight of the contained water at the test temperature in air. The difference

$W = W_1 - W_2$.

9.6 Cool the sample to 2 or 3°C below the test temperature, fill the pycnometer with it, immerse in the bath, and bring to the test temperature as was done before. After immersion at the test temperature for at least 30 min, adjust the liquid level, put the stopper in place, remove from the bath, wipe dry, and weigh. Record the temperature as S_1

9.7 Subtract the weight of the empty pycnometer from the weight when filled with the sample in order to obtain the weight of the contained sample. The difference $S = S_1 - W_2$.

10. Calculation

10.1 Calculate the specific gravity ~~at 25/25°C~~ (in air) as follows:

~~Specific gravity 25/25°C = S/W~~ (3)

Specific gravity $T/T = S/W$ (3)

where:

T = test temperature

10.2 Material specifications often specify different temperatures at which specific gravity shall be measured. In order to convert to any selected temperature, the coefficient of cubical expansion for the material being tested must be used. In addition, the absolute density of water at the desired temperature is taken. The absolute densities of water at different temperatures are tabulated in various handbooks. In converting to any desired temperature basis, the following equation is used:

~~Specific gravity T_3/T_4°C = specific gravity T_1/T_2 / $[1 + k(T_3 - T_1)] \frac{(d_{H_2O} \text{ at } T_4}{d_{H_2O} \text{ at } T_2}$~~ (4)

Specific gravity T_3/T_4°C = specific gravity T_1/T_2 / $[1 + k(T_3 - T_1)] \frac{(d_{H_2O} \text{ at } T_4}{d_{H_2O} \text{ at } T_2}$ (4)

where:

T_1/T_2 = original temperature conditions,

T_3/T_4 = new temperature conditions,

k = coefficient of cubical expansion (0.00117 at 0 to 40°C for trichloroethylene, 0.00102 at 0 to 25°C for perchloroethylene, 0.00125 at 0 to 30°C for 1,1,1-trichloroethane, 0.00137 at 0 to 40°C for methylene chloride, and 0.000927 at 0 to 30°C for fluorocarbon-113), and

d_{H_2O} = absolute density of water at the specified temperature.

10.2.1 *Example*—Assume a specific gravity of 1.4550 at 25/25°C for trichloroethylene. This is to be converted to 15/4°C and 20/20°C.

Specific gravity 15/4°C
= 1.4550/[1 + 0.00117(15− 25)] (0.999973/0.997044)
= 1.4550/[0.9883] 1.00293
= 1.4679

where 0.999973/0.997044 = ratio of the absolute density of water at 4°C compared to the absolute density of water at 25°C.

Specific gravity 20/20°C
= 1.4550/[1 + 0.00117 (20−25)] (0.998203/0.997044)
= 1.4550/[0.99415] 1.00116
= 1.4619

where 0.998203/0.997044 = ratio of the absolute density of water at 20°C compared to the absolute density of water at 25°C.

NOTE 2—Figures for absolute density of water are taken from the *Handbook of Chemistry and Physics*, Forty-ninth edition (1968–1969), published by The Chemical Rubber Co.

11. ~~Precision and Bias~~

~~11.1 When Test Method B is employed, different laboratories using different instruments should be able~~Procedure—Density

11.1 Clean the pycnometer by filling it with a saturated solution of chromic acid in concentrated sulfuric acid, allowing it to ~~obtain results that differ~~ stand for a few hours, emptying, and rinsing well with distilled water. Remove the water from the ~~means~~ pycnometer by ~~not more than 0.0002.~~

~~11.2 The limits of precision~~ rinsing it with alcohol or acetone, and ~~bias of any method~~ blow the vapor out with clean, dry air or nitrogen.

11.2 Place the empty pycnometer in the water bath and bring it to the test temperature. Allow the pycnometer to remain at the test temperature for ~~determining specific gravity depend upon~~ 30 min.

11.3 Remove the pycnometer from the water bath, wipe it dry, put the stopper in place, and weigh the pycnometer. Record the weight as *P*. Do not touch the pycnometer with bare hands, as moisture and oils from the hands can affect the weight.

11.4 Fill the pycnometer with the sample that ~~is given~~ has been cooled to ~~details of calibration and technique. Consideration,~~ 2 or 3°C below the test temperature. Place it in ~~general, must be given to~~ the ~~problems of keeping a large volume of liquid (sometimes unstirred)~~ water bath maintained at the test temperature ±0.5°C until the pycnometer and its contents are at a constant ~~temperature, providing~~ volume.

11.5 After immersion in the bath for at least 30 min, adjust the ~~effects~~ level of ~~humidity or static electricity during weighing of pycnometers,~~ liquid to the proper point on the pycnometer, put the stopper in place, remove the pycnometer from the bath, wipe it dry, and ~~weighing relatively large loads.~~

~~METHOD C—SPECIFIC GRAVITY BY MEANS OF AN ELECTRONIC DENSITOMETER~~ weigh it. Record the weight as *S*.

12. Calculation

12.1 Calculate the density by the formula:

$$d = \frac{S - P}{V} \tag{5}$$

where
P = is the weight of pycnometer,
S = is the weight of the filled pycnometer, and
V = is the volume (25 mL in this case) of the pycnometer.

13. Precision and Bias

13.1 When Test Method B is employed, different laboratories using different instruments should be able to obtain results that differ from the means by not more than 0.0002.

13.2 The limits of precision and bias of any method for determining specific gravity depend upon the attention that is given to details of calibration and technique. Consideration, in general, must be given to the problems of keeping a large volume of liquid (sometimes unstirred) at a constant temperature, providing for the effects of humidity or static electricity during weighing of pycnometers, and weighing relatively large loads.

METHOD C—SPECIFIC GRAVITY AND DENSITY BY MEANS OF AN ELECTRONIC DENSITOMETER

14. Apparatus

12~~4~~.1 *Electronic Densitometer*, capable of measuring and displaying results to the fourth decimal place. These analyzers consist of a U-shaped, oscillating sample tube and a system for electronic excitation, frequency counting, and display. The density of the

sample changes the mass of the tube, which changes the frequency of oscillation. The instrument must have means of maintaining the temperature of the sample tube to ±0.05°C in the desired range.

~~12.1.1 Examples include but are not limited to: Kyoto Electronics Manufacturing Model DA-300 and Anton Paar DM-411.~~

~~12.2~~

14.2 *Syringe* or other device suitable for introduction of a sample into the densitometer. Refer to the manufacturer's instructions.

12~~4~~.3 *Thermometer*, calibrated and graduated to 0.1°C for observing and setting the temperature of measurement. The densitometer may display sample temperature, in which case the thermometer is not used.

13~~5~~. Calibration

13~~5~~.1 The densitometer must be calibrated for use at a given temperature. Follow the manufacturer's instructions for calibration. A densitometer is generally calibrated by setting the instrument's output (display) at the density of dry air (0.0012 at 60°F, 20°C, and 25°C) when the measuring cell is empty, and then similarly setting the output at the density or specific gravity (1.0000) of water at the set temperature when the cell is full of degassed, deionized water (see 12~~5~~.2 or 12~~5~~.3).

13~~5~~.2 *Calibration for Specific Gravity* —Fill the measuring cell with degassed, deionized water and allow the cell and water to come to the set temperature as shown by a constant output reading. This will take approximately ~~two minutes.~~ 2 min. Following the manufacturer's instructions, set the output to 1.0000. The instrument is now calibrated for specific gravity.

13~~5~~.3 *Calibration for Density*—Fill the measuring cell with ~~degassing,~~ degassed, deionized water and allow the cell and water to come to the set temperature as shown by a constant output reading. This will take approximately ~~two minutes.~~ 2 min. Following the manufacturer's instructions, set the output to the density of water at the set temperature of the densitometer.

13~~5~~.4 *Absolute density of water*:

13~~5~~.4.1 *Selected values*:

Temperature,° C	Temperature, °F	Density, g/cm^3
4	...	0.9999123
~~...~~	~~15.55 (60°F)~~	~~0.9990123~~
15.56	(60°F)	0.9990123
20	...	0.9913203
25	...	0.9912044

13~~5~~.4.2 Refer to reference books such as *CRC Handbook of Chemistry and Physics* or *Lang's Handbook of Chemistry* for values not listed in 13~~5~~.4.1.

13~~5~~.4.3 The user should calibrate the instrument at set-up, when the temperature is changed, and on a regular basis at the operating conditions. The frequency of calibration may vary with different instruments and operating conditions. The user should check the calibration frequently (daily or weekly) until there is enough data to determine what the calibration frequency for that instrument should be.

14~~6~~. Procedure

14~~6~~.1 Clean and dry the cell before each use. Rinse the cell with acetone, ethanol, or other suitable solvent. Dry the cell by passing dry air through the cell until the output is constant (0.0012 at 15 to 25°C).

14~~6~~.2 Fill the measuring cell with the sample using a syringe or other suitable device. Allow the cell and sample to equilibrate with the set temperature as indicated by a constant output (approximately ~~two minutes).~~

~~14.3 Record~~ 2 min).

16.3 Record the output as specific gravity or density at the specified temperature.

14~~6~~.4 Clean and dry in accordance with 13~~6~~.1.

15~~7~~. Precision and Bias

15~~7~~.1 *Repeatability (Single Analyst)* —When Method C is employed, the standard deviation of results (each the average of triplicates) obtained by the same analyst on different days, has been estimated to be 0.00006 absolute at 12 degrees of freedom. The 95 % limit for the difference between two such averages is 0.00016 absolute. The electronic densitometers generally display to the fourth decimal place, so the above values are rounded to 0.0001 and 0.0002, respectively.

15~~7~~.2 *Reproducibility (Multilaboratory)* —When Method C is employed, the standard deviation of results (each the average of triplicates) obtained by analysts in different laboratories, has been estimated to be 0.00027 absolute at 5 degrees of freedom. The 95 % limit for the difference between two such averages is 0.00076 absolute. The electronic densitometers generally display to the fourth decimal place, so the above values are rounded to 0.0003 and 0.0008, respectively.

15~~7~~.3 The bias of this test method was not determined, as standards of known density were not available.

16~~8~~. Keywords

16~~8~~.1 chlorinated solvent; densitometer; hydrometer; pycnometer