



Designation: ~~D7042~~—~~21~~ D7042 – 21a

## Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)<sup>1</sup>

This standard is issued under the fixed designation D7042; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 1. Scope\*

1.1 This test method covers and specifies a procedure for the concurrent measurement of both the dynamic viscosity,  $\eta$ , and the density,  $\rho$ , of liquid petroleum products and crude oils, both transparent and opaque. The kinematic viscosity,  $\nu$ , can be obtained by dividing the dynamic viscosity,  $\eta$ , by the density,  $\rho$ , obtained at the same test temperature.

1.2 The result obtained from this test method is dependent upon the behavior of the sample and is intended for application to liquids for which primarily the shear stress and shear rate are proportional (Newtonian flow behavior).

1.3 The precision has only been determined for those materials, viscosity ranges, density ranges, and temperatures as indicated in Section 15 on Precision and Bias. The test method can be applied to a wider range of materials, viscosity, density, and temperature. For materials not listed in Section 15 on Precision and Bias, the precision and bias may not be applicable.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and to determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

[D341 Practice for Viscosity-Temperature Equations and Charts for Liquid Petroleum or Hydrocarbon Products](#)

[D396 Specification for Fuel Oils](#)

[D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids \(and Calculation of Dynamic Viscosity\)](#)

[D975 Specification for Diesel Fuel](#)

[D1655 Specification for Aviation Turbine Fuels](#)

[D2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards](#)

[D2270 Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 °C and 100 °C](#)

<sup>1</sup> 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 D02.07 on Flow Properties.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

- D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
- D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
- D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants
- D6617 Practice for Laboratory Bias Detection Using Single Test Result from Standard Material
- D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material
- D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels
- D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)
- D7566 Specification for Aviation Turbine Fuel Containing Synthesized Hydrocarbons
- D7915 Practice for Application of Generalized Extreme Studentized Deviate (GESD) Technique to Simultaneously Identify Multiple Outliers in a Data Set
- 2.2 *ISO Standards:*<sup>3</sup>
  - ISO 5725 Accuracy (Trueness and Precision) of Measurement Methods and Results
  - ISO 8217 Petroleum products – Fuels (class F)
  - ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories
- 2.3 *Other Documents:*<sup>4</sup>
  - NIST Technical Note 1297 Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results
  - DEF STAN 91-091 Turbine Fuel, Kerosene Type, Jet A-1<sup>5</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *density, n*—mass per unit volume.

3.1.2 *dynamic viscosity [ $\eta$ ], n*—the ratio between the applied shear stress and rate of shear of a liquid at a given temperature.

##### 3.1.2.1 Discussion—

It is sometimes called the coefficient of dynamic viscosity or, simply, viscosity. Thus, dynamic viscosity is a measure of the resistance to flow or to deformation of a liquid under external shear forces.

##### 3.1.2.2 Discussion—

The term dynamic viscosity can also be used in a different context to denote a frequency-dependent quantity in which shear stress and shear rate have a sinusoidal time dependence.

3.1.3 *kinematic viscosity, n*—the ratio of the dynamic viscosity ( $\eta$ ) to the density ( $\rho$ ) of a liquid at a given temperature.

##### 3.1.3.1 Discussion—

For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density ( $\rho$ ). Therefore, the kinematic viscosity ( $\nu$ ) is a measure of the resistance to flow of a liquid under gravity.

3.1.4 *relative density (also called specific gravity (SG)), n*—the ratio of the density of a material at a stated temperature to the density of a reference material (usually water) at a stated temperature.

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *T at 12 mm<sup>2</sup>/s, °C, n*—the temperature at which the material has a kinematic viscosity of 12 mm<sup>2</sup>/s.

##### 3.2.1.1 Discussion—

Term mostly is associated with jet fuel where 12 mm<sup>2</sup>/s is considered a critical viscosity value. The temperature is determined using Practice D341 interpolation or extrapolation calculations from two kinematic viscosity data points. Other critical viscosity data can similarly be determined for other materials as T at (xx) mm<sup>2</sup>/s.

### 4. Summary of Test Method

4.1 The test specimen is introduced into the measuring cells, which are at a closely controlled and known temperature. The measuring cells consist of a pair of rotating concentric cylinders and an oscillating U-tube. The dynamic viscosity is determined

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

<sup>4</sup> Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

<sup>5</sup> Available from IHS, 15 Inverness Way East, Englewood, CO 80112, <http://www.global.ihs.com>.

from the equilibrium rotational speed of the inner cylinder under the influence of the shear stress of the test specimen and an eddy current brake in conjunction with adjustment data. The density is determined by the oscillation frequency of the U-tube in conjunction with adjustment data. The kinematic viscosity is calculated by dividing the dynamic viscosity by the density.

## 5. Significance and Use

5.1 Many petroleum products, and some non-petroleum materials, are used as lubricants and the correct operation of the equipment depends upon the appropriate viscosity of the liquid being used. In addition, the viscosity of many petroleum fuels is important for the estimation of optimum storage, handling, and operational conditions. Thus, the accurate determination of viscosity is essential to many product specifications.

5.2 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.

5.3 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  $\pm 5^{\circ}\text{C}$ ;  $15^{\circ}\text{C}$ .

## 6. Apparatus

### 6.1 Stabinger Viscometer<sup>6,7</sup>

6.1.1 *Viscosity Measurement*—The Stabinger viscometer uses a rotational coaxial cylinder measuring system. The outer cylinder (tube) is driven by a motor at a constant and known rotational speed. The low-density inner cylinder (rotor) is held in the axis of rotation by the centrifugal forces of the higher density sample and in its longitudinal position by the magnet and the soft iron ring. Consequently, the system works free of bearing friction as found in rotational viscometers. A permanent magnet in the inner cylinder induces eddy currents in the surrounding copper casing. The rotational speed of the inner cylinder establishes itself as the result of the equilibrium between the driving torque of the viscous forces and the retarding eddy current torque. This rotational speed is measured by an electronic system (Hall effect sensor) by counting the frequency of the rotating magnetic field (see Fig. 1 and Fig. 2, No. 2).

6.1.2 *Density Measurement*—The digital density analyzer uses a U-shaped oscillating sample tube and a system for electronic excitation and frequency counting (see Fig. 2, No. 3).

6.1.3 *Temperature Control*—The copper block surrounds both the viscosity and the density measuring cell in a way that both cells are held at the same temperature. A thermoelectric heating and cooling system (see Fig. 2, No. 1) ensures the temperature stability of the copper block within  $\pm 0.005^{\circ}\text{C}$  from the set temperature at the position of the viscosity cell over the whole temperature range. The uncertainty ( $k = 2$ ; 95 % confidence level) of the temperature calibration shall be no more than  $\pm 0.03^{\circ}\text{C}$  over the range from  $15^{\circ}\text{C}$  to  $100^{\circ}\text{C}$ . Outside this range the calibration uncertainty shall be no more than  $\pm 0.05^{\circ}\text{C}$ .

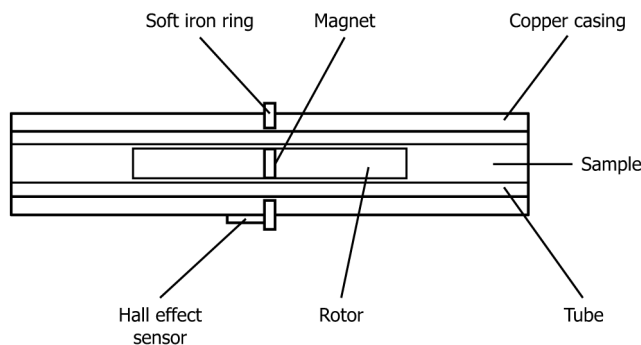


FIG. 1 Viscosity Cell

<sup>6</sup> The Stabinger viscometer is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative to this patented item to the ASTM International headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

<sup>7</sup> The sole source of supply of the apparatus known to the committee at this time is Anton Paar GmbH, Anton-Paar-Str. 20, A-8054 Graz, Austria. If you are aware of alternative suppliers, please provide this information to ASTM International headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

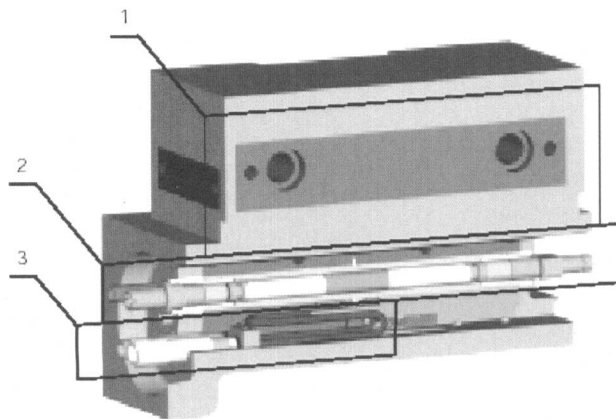


FIG. 2 Cell Block

6.1.4 The thermal equilibration time depends on the heat capacity and conductivity of the liquid and on the difference between injection temperature and test temperature. Adequate temperature equilibration of the test specimen is automatically determined when successive viscosity values are constant within  $\pm 0.07\%$  over 1 min and successive density values are constant within  $\pm 0.00003 \text{ g/cm}^3$  over 60 s.

NOTE 1—The Stabinger Viscometer, manufactured by Anton Paar GmbH, fulfills the stated requirements when operated in the most precise mode of operation.

6.2 *Syringes*, commercially available, at least 5 mL in volume, with a Luer tip. All construction materials for syringes shall be fully compatible with all sample liquids and cleaning agents, which contact them.

6.3 *Flow-Through or Pressure Adapter*, for use as an alternative means of introducing the test specimen into the measuring cells either by pressure or by suction, provided that sufficient care and control is used to avoid any bubble formation in the test specimen. All construction materials for adaptors shall be fully compatible with all sample liquids and cleaning agents, which contact them.

6.4 *Hot Filling Adapter*, for use with manual syringe filling for the purpose of preventing the precipitation of waxy components dissolved in sample and lowering sample viscosity for easier sample introduction and cleaning routines.

6.5 *Autosampler*, 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 into the measuring cells. The autosampler shall transfer the test specimen from the sample vial to the measuring cells of the apparatus without interfering with the integrity of the test specimen. The autosampler shall be able to mimic the procedure for sample handling as set forth in 11.1 and 11.2. The autosampler may have heating capability as a means to prevent the precipitation of waxy components dissolved in the sample and lower the viscosity of the sample for filling the measuring cells.

6.6 *Screen*, with an aperture of 75  $\mu\text{m}$ , to remove particles from the sample.

6.7 *Magnet*, strong enough to remove iron filings ferromagnetic materials from the sample. Magnetic stirring rods are suitable.

6.8 *Ultrasonic Bath, Unheated (optional)*, with an operating frequency between 25 kHz to 60 kHz and a typical power output of  $\leq 100 \text{ W}$ , of suitable dimensions to hold container(s) placed inside of bath, for use in effectively dissipating and removing air or gas bubbles that can be entrained in viscous sample types prior to analysis. It is permissible to use ultrasonic baths with operating frequencies and power outputs outside this range, however it is the responsibility of the laboratory to conduct a data comparison study to confirm that results determined with and without the use of such ultrasonic baths does not materially impact results.

## 7. Reagents and Materials

7.1 *Sample Solvent*, completely miscible with the sample.

7.1.1 For most samples, a volatile petroleum spirit or naphtha is suitable. If the solvent dries up without residues in an applicable time frame, the use of a separate drying solvent is not required.

7.1.2 For residual fuels, a prewash with an aromatic solvent such as toluene or xylene may be necessary to remove asphaltic material.

7.2 *Drying Solvent*, a volatile solvent miscible with the sample solvent (see 7.1).

7.2.1 Highly concentrated ethanol (96 % or ~~higher~~ higher), n-hexane or n-heptane is suitable.

7.3 *Dry Air or Nitrogen*, for blowing the measuring cells.

7.3.1 If the measuring cell temperature is below or near the dew point temperature of the ambient air, the use of an appropriate desiccator is required.

## 8. Sampling, Test Specimens, and Test Units

### 8.1 *General Considerations and Guidelines:*

8.1.1 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.

8.1.2 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 6.8) without the heater turned on (if so equipped), has been found effective in dissipating bubbles typically within 1 min.

8.1.3 *Particles*—For samples that are likely to contain particles (for example, used oils or crude oils) pass the sample through a 75 µm screen to remove the particles. For the removal of ~~iron filings~~ ferromagnetic materials the use of a magnet is appropriate. Waxy samples must be heated to dissolve the wax crystals prior to filtration and a preheated filter shall be used.

8.1.4 *Test Specimen*—A portion or volume of sample obtained from the laboratory sample and delivered to the measuring cells. The test specimen is obtained as follows:

8.1.4.1 Mix the sample, if required, to homogenize. Mixing at room temperature in an open container can result in the loss of volatile material; mixing in closed, pressurized containers, or at sub-ambient temperatures is recommended.

8.1.4.2 Draw the test specimen from a properly mixed laboratory sample using an appropriate syringe. Alternatively, if the proper attachments and connecting tubes are used, the test specimen may be delivered directly to the measuring cells using a flow through or pressure adapter (see 6.3) or autosampler (see 6.5) from the mixing container. For waxy or other samples with a high pour point, before drawing the test specimen, heat the laboratory sample to the desired test temperature, which has to be high enough to dissolve the wax crystals.

### 8.2 *Instructions for Residual Fuel Oils:*

8.2.1 (**Warning**—Exercise care as vigorous boil-over can occur when opaque liquids which contain high levels of water are heated to high temperatures. Wear appropriate personal protective equipment for handling hot materials.) Place the required number of disposable syringes or samples vials to be used for the batch analysis into a sample preheat apparatus (such as an oven, bath, or heating block) held between 60 °C and 65 °C. If using manual syringe filling, a hot filling adapter must be installed on the apparatus and the injection adapters must also be pre-warmed along with the syringes.

8.2.2 Place the first batch of residual fuel samples to be analyzed for the day in their original containers in a sample preheat apparatus that is between 60 °C and 65 °C for 1 h. Ensure the cap on each container is tightly closed. For samples of a very waxy nature or oils of high kinematic viscosity, it may be necessary to increase the heating temperature above 60 °C to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking.

8.2.3 Vigorously stir each sample for approximately 20 s with a glass or steel rod of sufficient length to reach the bottom of the container.

8.2.4 Remove the stirring rod and inspect for sludge or wax adhering to the rod. If there is sludge or wax adhering to the rod, continue stirring until the sample is ~~homogene~~homogeneous.

8.2.5 Recap each container tightly and shake vigorously for 1 min. Then loosen the cap, retighten to finger tight, then back off  $\frac{3}{4}$  to a full turn and place back into the sample preheat apparatus.

8.2.6 Upon completion of 8.2.5 for all samples in the batch, increase the sample pre-heat apparatus temperature to between 100 °C and 105 °C and heat for 30 min.

8.2.7 Remove each container from the sample pre-heat apparatus, close tightly, and shake vigorously for 60 s.

8.2.8 If a heated auto sampler is used, follow instructions in 8.2.8.1 below. If a sample handler is used, follow instructions in 8.2.8.2 below.

8.2.8.1 Ensure the sample vial magazine is held at a temperature between 60 °C and 80 °C. Load each sample into its own preheated sample vial from 8.2.1, insert the vials into the sample vial magazine, and wait for 10 min to 15 min before commencing measurement.

8.2.8.2 Load a preheated sample vial from 8.2.1 with the first sample to be tested in the batch and place the sample vial into the sampler handler that is held between 60 °C and 80 °C. Loosen the cap of the other containers, retighten to finger tight, then back off  $\frac{3}{4}$  to a full turn and place the containers back into the sample preheat apparatus that is reset to hold temperature between 60 °C and 80 °C.

8.2.9 Complete the measurements according to 11.1 or 11.3. Analysis of all samples in the batch must be completed within 1 h from completion of 8.2.7.

## 9. Calibration and Verification

9.1 Use only a calibrated apparatus as described in 6.1. The calibration shall be checked periodically using certified reference standards as described in 9.2 and 9.3. The recommended interval for viscosity and density calibration is once a month, for temperature control once a year. For the calibration procedure follow the instructions of the manufacturer of the apparatus.

9.2 *Certified Viscosity and Density Reference Standards*—These are for use as confirmatory checks on the procedure in the laboratory. Certified viscosity and density reference standards shall be certified by a laboratory, which has shown to meet the requirements of ISO/IEC 17025 or a corresponding national standard by independent assessment. Viscosity standards shall be traceable to master viscometer procedures described in Test Method D2162. Density standards shall have a certified uncertainty of the density values of 0.0001 g/cm<sup>3</sup>. The uncertainty of the reference standards shall be stated for each certified value (k = 2; 95 % confidence level). See ISO 5725 or NIST Technical Note 1297.

9.3 *Thermometer*—For calibration and adjustment of the temperature control, a digital thermometer with a probe diameter of 6.25 mm and a maximal length of 80 mm shall be used. For smaller probes the use of an adapter is suitable. The uncertainty (k = 2; 95 % confidence level) of this thermometer must be no more than  $\pm 0.01$  °C and has to be certified by a laboratory which has shown to meet the requirements of ISO/IEC 17025 or a corresponding national standard by independent assessment. A suitable thermometer is available from the manufacturer of the apparatus.

9.4 *Acceptable Tolerance*—If the determined values of a calibration check measurement do not agree within the acceptable tolerance band of the certified values, as calculated from Annex A1, recheck each step in the procedure, including the special cleaning procedure from 12.2, to locate the source of error.

NOTE 2—Values exceeding the acceptable tolerance are generally attributable to deposits in the measuring cells that are not removed by the routine flushing procedure.

## 10. Adjustment

10.1 An adjustment has to be carried out when repeated calibration check measurements do not agree with the Acceptable Tolerance Band as stated in 9.4 and the error cannot be located elsewhere. For the adjustment procedure follow the instructions of the manufacturer of the apparatus.

10.2 For an adjustment, use only certified viscosity and density reference standards that fulfill the requirements as stated in 9.2. The reference standards have to be within the viscosity, density, and temperature range specified by the manufacturer of the apparatus.

10.3 After an adjustment procedure a calibration check measurement shall be performed.

## 11. Procedure

11.1 Standard procedure (rinsing and drying)

11.1.1 Set the internal temperature control to the desired measuring temperature.

11.1.2 Set the determinability limits and temperature stability criteria to the values stated in Table 1 for the specific product.

11.1.3 Make sure that the measuring cells are clean and dry as described in 12.1.

11.1.4 Load a minimum of ~~3 mL~~ 5 mL of the test specimen to the ~~syringe~~ syringe; if enough sample is available it is recommended to fill the entire syringe. Pour at least 2 mL of the test specimen into the measuring cells. Leave the syringe in the inlet opening and start the ~~measurement~~ motor and wait for about 10 s, then stop the motor again. Inject an additional 1 mL, leave the syringe in the inlet opening, and start the measurement. Alternatively, the instrument may be configured to perform pre-wetting automatically prior to starting the measurement. Follow manufacture's instructions. Wait for the instrument to indicate that the determination is valid and record the values.

11.1.5 Inject a further 1 mL without taking off the syringe and repeat the measurement.

11.1.6 If the deviation between two consecutive determinations exceeds the determinability limits as stated in Table 1 for this product, repeat step 11.1.5 until the deviation is within these limits. Discard all previously determined values and report the values of the last determination as the result.

11.1.6.1 For products not listed in the precision section, it is the responsibility of the user of this standard to establish reasonable determinability limits by a series of tests.

11.1.6.2 If the syringe is empty before obtaining a valid determination, rinse and dry the measuring cells as described in 12.1 and repeat step 11.1.4. If it is not possible to obtain a valid result within an applicable number of repetitions, report the robust mean value and the standard deviation ( $k = 2$ ; 95 % confidence level) together with a remark indicating that the determinability exceeded the limits stated in 15.2.1 for this product.

11.1.7 Remove the test specimen immediately, rinse and dry the measuring cells as described in 12.1.

**TABLE 1 Determinability Limits and Temperature Stability Criteria**

NOTE 1—X is the final determination.

	RDV (Determinability, dynamic viscos- ity)	RDD (Determinability, density)	Temperature Stability	Viscosity Stability	Density Stability	Time	Repeats	Equilibra- tion Time
For ALL materials at ALL temperatures unless specifically listed below	0.10 % 0.001 X	0.0002 g/cm <sup>3</sup>	±0.005 °C	±0.07 %	0.00003 g/cm <sup>3</sup>	60 s	3	0 s
Residual Fuel Oils at 50 °C and 100 °C	0.35 % 0.0035 X	0.0003 g/cm <sup>3</sup>	±0.010 °C	±0.10 %	0.00005 g/cm <sup>3</sup>	40 s	3	30 s
Jet fuel at -20 °C and -40 °C, Scanning procedure	0.0011889 X	0.000113 g/cm <sup>3</sup>	±0.005 °C	±0.07 %	0.0001 g/cm <sup>3</sup>	60 s	0	0 s