



## Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester<sup>1</sup>

This standard is issued under the fixed designation D 93; 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 reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

### INTRODUCTION

This flash point test method is a dynamic test method which depends on specified rates of heating to be able to meet the precision of the test method. The rate of heating may not in all cases give the precision quoted in the test method because of the low thermal conductivity of some materials.

Flash point values are a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can therefore only be defined in terms of a standard test method, and no general valid correlation can be guaranteed between results obtained by different test methods, or with test apparatus different from that specified.

### 1. Scope\*

1.1 These test methods cover the determination of the flash point of petroleum products in the temperature range from 40 to 360°C by a manual Pensky-Martens closed-cup apparatus or an automated Pensky-Martens closed-cup apparatus.

NOTE 1—Flash point determination as above 250°C can be performed, however, the precisions have not been determined above this temperature. For residual fuels, precisions have not been determined for flash points above 100°C.

1.2 Procedure A is applicable to distillate fuels (diesel, kerosine, heating oil, turbine fuels), new lubricating oils, and other homogeneous petroleum liquids not included in the scope of Procedure B.

1.3 Procedure B is applicable to residual fuel oils, cutback residua, used lubricating oils, mixtures of petroleum liquids with solids, petroleum liquids that tend to form a surface film under test conditions, or are petroleum liquids of such kinematic viscosity that they are not uniformly heated under the stirring and heating conditions of Procedure A.

1.4 These test methods is applicable for the detection of contamination of relatively nonvolatile or nonflammable materials with volatile or flammable materials.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

NOTE 2—It has been common practice in flash point standards for many decades to alternately use a C-scale or an F-scale thermometer for temperature measurement. Although the scales are close in increments, they are not equivalent. Because the F-scale thermometer used in this procedure is graduated in 5° increments, it is not possible to read it to the 2°C equivalent increment of 3.6°F. Therefore, for the purposes of application of the procedure of the test method for the separate temperature scale thermometers, different increments must be used. In this test method, the following protocol has been adopted: When a temperature is intended to be a converted equivalent, it will appear in parentheses following the SI unit, for example 370°C (698°F). When a temperature is intended to be a rationalized unit for the alternate scale, it will appear after “or,” for example, 2°C or 5°F.

1.6 *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 warning statements, see 6.4, 7.1, 9.3, 9.4, 11.1.2, 11.1.4, 11.1.8, 11.2.2, and 12.1.2.

<sup>1</sup> These test methods are under the joint jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and are the direct responsibility of Subcommittee D02.08 on Volatility. In the IP, these test methods are under the jurisdiction of the Standardization Committee.

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

## 2. Referenced Documents

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

- D 56** Test Method for Flash Point by Tag Closed Cup Tester
- D 4057** Practice for Manual Sampling of Petroleum and Petroleum Products
- D 4177** Practice for Automatic Sampling of Petroleum and Petroleum Products
- E 1** Specification for ASTM Liquid-in-Glass Thermometers
- E 300** Practice for Sampling Industrial Chemicals
- E 502** Test Method for Selection and Use of ASTM Standards for the Determination of Flash Point of Chemicals by Closed Cup Methods

### 2.2 ISO Standards<sup>3</sup>

- Guide 34** Quality Systems Guidelines for the Production of Reference Materials
- Guide 35** Certification of Reference Material—General and Statistical Principles

## 3. Terminology

### 3.1 Definitions:

3.1.1 *dynamic, adj—in petroleum products—in petroleum product flash point test methods*—the condition where the vapor above the test specimen and the test specimen are not in temperature equilibrium at the time that the ignition source is applied.

3.1.1.1 *Discussion*—This is primarily caused by the heating of the test specimen at the constant prescribed rate with the vapor temperature lagging behind the test specimen temperature.

3.1.2 *equilibrium, n—in petroleum products—in petroleum product flash point test methods*—the condition where the vapor above the test specimen and the test specimen are at the same temperature at the time the ignition source is applied.

3.1.2.1 *Discussion*—This condition may not be fully achieved in practice, since the temperature may not be uniform throughout the test specimen, and the test cover and shutter on the apparatus can be cooler.

3.1.3 *flash point, n—in petroleum products*, the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of an ignition source causes the vapors of a specimen of the sample to ignite under specified conditions of test.

## 4. Summary of Test Method

4.1 A brass test cup of specified dimensions, filled to the inside mark with test specimen and fitted with a cover of specified dimensions, is heated and the specimen stirred at specified rates, by either of two defined procedures (A or B). An ignition source is directed into the test cup at regular intervals with simultaneous interruption of the stirring, until a flash is detected (see 11.1.8). The flash point is reported as defined in 3.1.3.

## 5. Significance and Use

5.1 The flash point temperature is one measure of the tendency of the test specimen to form a flammable mixture with air under controlled laboratory conditions. It is only one of a number of properties which must be considered in assessing the overall flammability hazard of a material.

5.2 Flash point is used in shipping and safety regulations to define *flammable* and *combustible* materials. One should consult the particular regulation involved for precise definitions of these classifications.

NOTE 3—The U.S. Department of Transportation (DOT)<sup>4</sup> and U.S. Department of Labor (OSHA) have established that liquids with a flash point under 37.8°C (100°F) are flammable, as determined by these test methods, for those liquids which have a kinematic viscosity of 5.8 mm<sup>2</sup>/s (cSt) or more at 37.8°C or 9.5 mm<sup>2</sup>/s (cSt) or more at 25°C (77°F), or that contain suspended solids, or have a tendency to form a surface film while under test. Other classification flash points have been established by these departments for liquids using these test methods.

5.3 These test methods should be used to measure and describe the properties of materials, products, or assemblies in response to heat and an ignition source under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of these test methods may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

5.4 These test methods provides the only closed cup flash point test procedures for temperatures up to 370°C (698°F).

## 6. Apparatus

6.1 *Pensky-Martens Closed Cup Apparatus (manual)*—This apparatus consists of the test cup, test cover and shutter, stirring device, heating source, ignition source device, air bath, and top plate described in detail in Annex A1. The assembled manual apparatus, test cup, test cup cover, and test cup assembly are illustrated in Figs. A1.1-A1.4, respectively. Dimensions are listed respectively.

6.2 *Pensky-Martens Closed Cup Apparatus (automated)*—This apparatus is an automated flash point instrument that is capable of performing the test in accordance with Section 11 (Procedure A) and Section 12 (Procedure B) of these test methods. The apparatus shall use the test cup, test cover and shutter, stirring device, heating source, and ignition source device described in detail in Annex A1.

6.3 *Temperature Measuring Device*—Thermometer having a range as shown as follows and conforming to the requirements prescribed in Specification E 1 or in Annex A3, or an electronic temperature measuring device, such as resistance thermometers or thermocouples. The device shall exhibit the same temperature response as the mercury thermometers.

<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.

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

<sup>4</sup> For information on U.S. Department of Transportation regulations, see Codes of U.S. Regulations 49 CFR Chapter 1 and the U.S. Department of Labor, see 29 CFR Chapter XVII. Each of these items is revised annually and may be procured from the Superintendent of Documents, Government Printing Office, Washington, DC 20402.

Temperature Range	Thermometer Number	
	ASTM	IP
-5 to +110°C (20 to 230°F)	9C (9F)	15C
+10 to 200°C (50 to 392°F)	88C (88F)	101C
+90 to 370°C (200 to 700°F)	10C (10F)	16C

6.4 *Ignition Source*—Natural gas flame, bottled gas flame, and electric ignitors (hot wire) have been found acceptable for use as the ignition source. The gas flame device described in detailed in Fig. A1.4 requires the use of the pilot flame described in A1.1.2.3. The electric ignitors shall be of the hot-wire type and shall position the heated section of the ignitor in the aperture of the test cover in the same manner as the gas flame device. (**Warning**—Gas pressure supplied to the apparatus should not be allowed to exceed 3 kPa (12 in.) of water pressure.)

6.5 *Barometer*—With accuracy of  $\pm 0.5$  kPa.

NOTE 4—The barometric pressure used in this calculation is the ambient pressure for the laboratory at the time of the test. Many aneroid barometers, such as those used at weather stations and airports, are precorrected to give sea level readings and would not give the correct reading for this test.

## 7. Reagents and Materials

7.1 *Cleaning Solvents*—Use suitable solvent capable of cleaning out the specimen from the test cup and drying the test cup and cover. Some commonly used solvents are toluene and acetone. (**Warning**—Toluene, acetone, and many solvents are flammable and a health hazard. Dispose of solvents and waste material in accordance with local regulations.)

## 8. Sampling

8.1 Obtain a sample in accordance with instructions given in Practices D 4057, D 4177, or E 300.

8.2 At least 75 mL of sample is required for each test. Refer to Practice D 4057. When obtaining a sample of residual fuel oil, the sample container shall be from 85 to 95 % full. For other types of samples, the size of the container shall be chosen such that the container is not more than 85 % full or less than 50 % full prior to any sample aliquot being taken.

8.3 Successive test specimens can be taken from the same sample container. Repeat tests have been shown to be within the precisions of the method when the second specimen is taken with the sample container at least 50 % filled. The results of flash point determinations can be affected if the sample volume is less than 50 % of sample container capacity.

8.4 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily, to prevent loss of volatile material or possible introduction of moisture, or both. Avoid storage of samples at temperatures in excess of 35°C or 95°F. Samples for storage shall be capped tightly with inner seals. Do not make a transfer unless the sample temperature is at least the equivalent of 18°C or 32°F below the expected flash point.

8.5 Do not store samples in gas-permeable containers, since volatile material may diffuse through the walls of the enclosure. Samples in leaky containers are suspect and not a source of valid results.

8.6 Samples of very viscous materials shall be heated in their containers, with lid/cap slightly loosened to avoid buildup

of dangerous pressure, at the lowest temperature adequate to liquefy any solids, not exceeding 28°C or 50°F below the expected flash point, for 30 min. If the sample is then not completely liquefied, extend the heating period for additional 30 min periods as necessary. Then gently agitate the sample to provide mixing, such as orbiting the container horizontally, before transferring to the specimen cup. No sample shall be heated and transferred unless its temperatures is more than 18°C or 32°F below its expected flash point. When the sample has been heated above this temperature, allow the sample to cool until its temperature is at least 18°C or 32°F below the expected flash point before transferring.

NOTE 5—Volatile vapors can escape during heating when the sample container is not properly sealed.

NOTE 6—Some viscous samples may not completely liquefy even after prolonged periods of heating. Care should be exercised when increasing the heating temperature to avoid unnecessary loss of volatile vapors, or heating the sample too close to the flash point.

8.7 Samples containing dissolved or free water may be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Warming the sample is permitted, but it shall not be heated for prolonged periods or greater than a temperature of 18°C 32°F below its expected flash point.

NOTE 7—If the sample is suspected of containing volatile contaminants, the treatment described in 8.6 and 8.7 should be omitted.

## 9. Preparation of Apparatus

9.1 Support the manual or automated apparatus on a level steady surface, such as a table.

9.2 Tests are to be performed in a draft-free room or compartment. Tests made in a laboratory hood or in any location where drafts occur are not reliable.

NOTE 8—A shield, of the approximate dimensions 460 mm (18 in.) square and 610 mm (24 in.) high, or other suitable dimensions, and having an open front is recommended to prevent drafts from disturbing the vapors above the test cup.

NOTE 9—With some samples whose vapors or products of pyrolysis are objectionable, it is permissible to place the apparatus along with a draft shield in a ventilation hood, the draft of which is adjustable so that vapors can be withdrawn without causing air currents over the test cup during the ignition source application period.

9.3 Prepare the manual apparatus or the automated apparatus for operation in accordance with the manufacturer's instructions for calibrating, checking, and operating the equipment. (**Warning**—Gas pressure should not be allowed to exceed 3 kPa (12 in.) of water pressure.)

9.4 Thoroughly clean and dry all parts of the test cup and its accessories before starting the test, to ensure the removal of any solvent which had been used to clean the apparatus. Use suitable solvent capable of removing all of the specimen from the test cup and drying the test cup and cover. Some commonly used solvents are toluene and acetone. (**Warning**—Toluene, acetone, and many solvents are flammable. Health hazard. Dispose of solvents and waste material in accordance with local regulations.)