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Standard Test Methods for FLASH POINT BY PENSKEY-MARTENS CLOSED TESTER¹

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 (ϵ) indicates an editorial change since the last revision or reapproval.

These methods were adopted as a joint ASTM-IP Standard in 1967.

This method has been adopted for use by government agencies to replace Method 1102 of Federal Test Method Standard No. 791b, and Method 4293 of Federal Test Method Standard No. 141A.

1. Scope

1.1 These test methods cover the determination of the flash point by Pensky-Martens closed-cup tester of fuel oils, lube oils, suspensions of solids, liquids that tend to form a surface film under test conditions, and other liquids.

NOTE 1—This method may be employed for the detection of contamination of lubricating oils by minor amounts of volatile materials.

NOTE 2—The U.S. Department of Transportation (DOT)² and U.S. Department of Labor (OSHA) have established that liquids with a flash point under 100°F (37.8°C) are flammable as determined by methods for those liquids which have a viscosity of 5.8 cSt or more at 100°F (37.8°C) or 9.5 cSt or more at 77°F (25°C), 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 this test.

1.2 Liquids having viscosities less than 5.5 cSt at 104°F (40°C), do not contain suspended solids or do not have a tendency to form a surface film while under test should be tested in accordance with Method D 56.

1.3 *This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame 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 this test 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.

1.4 The values stated in inch-pound units shall be regarded as the standard.

1.5 *This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 ASTM Standards:

D 56 Test Method for Flash Point by Tag Closed Tester³

E 1 Specification for ASTM Thermometer⁴

E 134 Specification for Pensky-Martens Closed Flash Tester⁵

¹ These test methods are under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and are the direct responsibility of Subcommittee D02.08 on Volatility.

In the IP, these methods are under the jurisdiction of the Standardization Committee.

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² For information concerning regulations of U.S. Department of Transportation, see Codes of U. S. Regulations 49 CFR, Chapter I, and of U.S. Department of Labor, see 29 CFR, Chapter XVII. Each of these items is revised annually and may be procured from Superintendent of Documents, Government Printing Office, Washington, DC 20402.

³ Annual Book of ASTM Standards, Vols 05.01 and 06.03.

⁴ Annual Book of ASTM Standards, Vols 05.03 and 14.01.

⁵ Annual Book of ASTM Standards, Vols 05.07 and 14.02.



Documents

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3. Definition

3.1 *flash point*—the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of a test flame causes the vapor of a specimen to ignite under specified conditions of test.

3.1.1 The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the sample.

3.1.2 Occasionally, particularly near the actual flash point, the application of the test flame will cause a blue halo or an enlarged flame; this is not a flash and should be ignored.

4. Summary of Methods

4.1 The sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapor above the sample to ignite.

5. Significance and Use

5.1 Flash point measures tendency of the sample 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 classes.

5.3 Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or nonflammable material. For example, an abnormally low flash point on a sample of kerosine may indicate gasoline contamination.

6. Apparatus

6.1 *Pensky-Martens Closed Flash Tester*, as described in Annex A1.

NOTE 3—There are automatic flash point testers available and in use which may be advantageous in that they save testing time, permit the use of smaller samples, and exhibit other factors which may merit their use. If automatic testers are used, the user must be sure that all of the manufacturer's instructions for calibrating, adjusting, and operating the instrument are fol-

lowed. In any cases of dispute the flash point as determined manually shall be considered the referee test.

6.2 *Thermometers*—Three standard thermometers shall be used with the ASTM Pensky-Martens tester as follows:

6.2.1 For tests in which the indicated reading falls within the limits of 50 to 140°F (10 to 60°C), inclusive, an ASTM 9F(9C) Pensky-Martens low range Thermometer having a range from 20 to 230°F (−5 to +110°C) and conforming to the requirements of Specification E 1 shall be used. Equally acceptable is IP thermometer 15F(15C), with specifications as shown in Annexes A2 and A3.

6.2.2 For tests in which the indicated reading falls within the limits 140 to 284°F (60 to 140°C), inclusive, an ASTM 88F(88C) Vegetable Oil Flash Thermometer having a range of 50 to 392°F (10 to 200°C) and conforming to the requirements of Specification E 1 shall be used.

6.2.3 For the range 140 to 230°F (60 to 110°C) either low or medium range thermometer may be used.

6.2.4 For tests in which the indicated reading falls within 265 to 700°F (130 to 370°C) an ASTM 10F(10C) Pensky-Martens high-range Thermometer having a range from 200 to 700°F (90 to 370°C) and conforming to the requirements of Specification E 1 shall be used. Equally acceptable is IP thermometer 16F(16C), with specifications as shown in Annex A3.

6.2.5 For the range 265 to 285°F (130 to 140°C) either the medium or high-range thermometer may be used.

7. Safety Precautions

7.1 The operator must exercise and take appropriate safety precautions during the initial application of the test flame, since samples containing low-flash material may give an abnormally strong flash when the test flame is first applied.

8. Sampling

8.1 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily and make a transfer unless the sample temperature is at least the equivalent of 18°F (8°C) below the expected flash point. Do not use samples from leaky containers for these test methods.

8.2 Do not store samples in plastic (polyethylene, polypropylene, etc.) containers, since volatile material may diffuse through the walls of the enclosure.

9. Preparation of Apparatus

9.1 Support the tester on a level, steady table. Unless tests are made in a draft-free room or compartment, it is good practice, but not required, to surround the tester on three sides with a shield, each section of which is about 18 in. (450 mm) wide and 24 in. (600 mm) high. See Annex 14.3.1, 14.3.2, and 14.3.

NOTE 4—Caution—Meticulous attention to all details relating to the flame exposure device, size of test flame, rate of temperature increase, and rate of dipping the flame exposure device into the vapor of the specimen is necessary for good results.

10. Preparation of Sample

10.1 Samples of very viscous materials may be warmed until they are reasonably fluid before they are tested. However, no sample should be heated more than is absolutely necessary. It shall never be heated above a temperature of 30°F (17°C) below its expected flash point.

10.2 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 above a temperature of 30°F (17°C) below its expected flash point.

NOTE 5—If the sample is suspected of containing volatile contaminants, the treatment described in 10.1 and 10.2 should be omitted.

METHOD A—BASIC PROCEDURE

11. Procedure

11.1 Thoroughly clean and dry all parts of the cup and its accessories before starting the test, being sure to remove any solvent which had been used to clean the apparatus. Fill the cup with the sample to be tested to the level indicated by the filling mark. Place the lid on the cup and set the latter in the stove. Be sure to have the locating or locking device properly engaged. Insert the thermometer. Bring the material to be tested and the tester to a temperature of 60 ± 10°F (15 ± 5°C) or 20°F (11°C) lower than the estimated flash point, whichever is lower. Light the test flame and adjust it to 5/32 in. (4 mm) in diameter. Supply the heat at such a rate that the tempera-

ture as indicated by the thermometer increases 9 to 11°F (5 to 6°C)/min. Turn the stirrer 90 to 120 rpm, stirring in a downward direction.

11.2 If the sample is known to have a flash point of 230°F (110°C) or below, apply the test flame when the temperature of the sample is from 30°F (17°C) to 50°F (28°C) below the expected flash point and thereafter at a temperature reading that is a multiple of 2°F (1°C). Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is lowered into the vapor space of the cup in 0.5 s, left in its lowered position for 1 s, and quickly raised to its high position. Do not stir the sample while applying the test flame.

11.3 If the sample is known to have a flash point above 230°F (110°C) apply the test flame in the manner just described at each temperature that is a multiple of 5°F (2°C), beginning at a temperature of 30°F (17°C) to 50°F (28°C) below the expected flash point.

NOTE 6—When testing materials to determine if volatile contaminants are present, it is not necessary to adhere to the temperature limits for initial flame application as stated in 11.2 and 11.3.

11.4 Record as the observed flash point the temperature read on the thermometer at the time the test flame application causes a distinct flash in the interior of the cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash.

METHOD B—DETERMINATION OF FLASH POINT OF SUSPENSIONS OF SOLIDS AND HIGHLY VISCOUS MATERIALS

12. Procedure

12.1 Bring the material to be tested and the tester to a temperature of 60 ± 10°F (15 ± 5°C) or 20°F (11°C) lower than the estimated flash point, whichever is lower. Turn the stirrer 250 ± 10 rpm, stirring in a downward direction. Raise the temperature throughout the duration of the test at a rate of not less than 2 nor more than 3°F (1 to 1.5°C)/min. With the exception of these requirements for rates of stirring and heating, proceed as prescribed in Section 11.

13. Calculation and Report

13.1 Observe and record the ambient barometric pressure (Note 7) at the time of the test. When the pressure differs from 760 mm Hg

(101.3 kPa), correct the flash point at follows:

- (A) Corrected flash point = $C + 0.25 (101.3 - p)$
 (B) Corrected flash point = $F + 0.06 (760 - P)$
 (C) Corrected flash point = $C + 0.033 (760 - P)$

where:

- F = observed flash points, °F,
 C = observed flash point, °C,
 P = ambient barometric pressure, mm Hg,
 and
 p = ambient barometric pressure, kPa.

NOTE 7—The barometric pressure used in this calculation must be the ambient pressure for the laboratory at the time of test. Many aneroid barometers, such as those used at weather stations and airports, are precorrected to give sea level readings. These must not be used.

13.2 Record the corrected flash point to the nearest 1°F (or 0.5°C).

13.3 Report the recorded flash point as the Pensky-Martens Closed Cup Flash Point ASTM D 93 – IP 34, of the sample tested.

14. Precision and Bias

14.1 *Method A*—The following criteria should be used for judging the acceptability of results (95 % probability):

14.1.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Material	Flash Point Range, °F (°C)	Repeatability, °F (°C)
Suspensions of solids	95 to 110	4
	(35 to 43)	(2)
All others	220 (104) and under	4
		(2)
	Above 220 (104)	10
		(5.5)

14.1.2 *Reproducibility*—The results submitted by each of two laboratories should be considered suspect if the two results differ by more than the following amounts:

Material	Flash Point Range, °F (°C)	Reproducibility, °F (°C)
Suspensions of solids	95 to 110	6
	(35 to 43)	(3.5)
All others	220 (104) and under	6
		(3.5)
	Above 220 (104)	15
		(8.5)

14.2 *Method B*—The following criteria should be used for judging the acceptability of results (95 % confidence) obtained on viscous or heavily pigmented (paint or varnish) materials, or both,

which tend to form a surface film:

14.2.1 *Repeatability*—The average of two tests by the same operator on the same day compared to the average of two tests on another day should be considered suspect if they differ by more than 9°F (5°C).

14.2.2 *Reproducibility*—The average of two tests by an operator on the same day compared to the average of two tests by another operator (or another laboratory) on any one day should be considered suspect if they differ by more than 18°F (10.0°C).

14.2.3 The definition of repeatability and reproducibility given here represents different parameters of the variance of the test methods; those given in 14.1 are derived from standards of ASTM Committee D-2 and the Institute of Petroleum, while those in 14.2 are from ASTM Committee D-1.

14.3 Calibration of Tester

14.3.1 Determine the flash point of the *p*-xylene,⁶ following the directions in Sections 9 through 11. When the tester is operating properly, a value of $81 \pm 2^\circ\text{F}$ ($27.2 \pm 1.1^\circ\text{C}$) will be obtained.

14.3.2 If the flash point obtained on *p*-xylene is not within the limits stated in A4.1, check the condition and operation of the apparatus to ensure conformity with the details listed in Annex A1, especially with regard to the tightness of the lid (A1.1.2.1), the action of the shutter, and the position of the test flame (A1.1.2.2). After adjustment, if necessary, repeat the test. *P*-xylene having a flash point of $81 \pm 2^\circ\text{F}$ ($27.2 \pm 1.1^\circ\text{C}$) is not a suitable reference in the high-temperature range of the Pensky-Martens Closed Tester which may be as high as 700°F (370°C).

14.3.3 *p*-Xylene shall conform to the following requirements: Specific gravity 15.56/15.56°C...0.860 min, 0.866 max. Boiling range...2°C from start to dry, point when tested in accordance with Method D 850, or Method D 1078. The range shall include the boiling point of pure *p*-xylene which is 138.35°C (281.03°F). Purity...95 % min (freezing point of 11.23°C), min calculated in accordance with Method D 1016, from the experimentally determined freezing point, measured by Method D 1015.

14.4 *Bias*—A bias statement is being developed for these test methods.

⁶ Satisfactory *p*-xylene may be obtained as Flash Point Check Fluid from the Special Products Div., Chemical Dept., Phillips Petroleum Co., Drawer O, Borger, TX 79007.

ANNEXES

(Mandatory Information)

A1. APPARATUS SPECIFICATIONS

A1.1 A typical assembly of the apparatus, gas heated, is shown in Fig. A1.1. The apparatus shall consist of a test cup, cover, and stove conforming to the following requirements:

A1.1.1 *Cup*—The cup shall be of brass, or other nonrusting metal of equivalent heat conductivity, and shall conform to the dimensional requirements in Fig. A1.2. The flange shall be equipped with devices for locating the position of the cup in the stove. A handle attached to the flange of the cup is a desirable accessory. The handle shall not be so heavy as to tip over the empty cup.

A1.1.2 *Cover*:

A1.1.2.1 *Cover Proper*—The cover shown in Fig. A1.3 shall be of brass (A1.1.1) and shall have a rim projecting downward almost to the flange of the cup. The rim shall fit the outside of the cup with a clearance not exceeding 0.014 in. (0.36 mm) on the diameter. There shall be a locating or locking device, or both, engaging with a corresponding device on the cup. The four openings in the cover, *A*, *B*, *C*, and *D*, are shown in Fig. A1.3. The upper edge of the cup shall be in close contact with the inner face of the cover throughout its circumference.

A1.1.2.2 *Shutter*—The cover shall be equipped with a brass (Section 3) shutter (Fig. A1.4), approximately $\frac{3}{32}$ in. (2.4 mm) thick, operating on the plane of the upper surface of the cover. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal center of the cover between two stops, so placed, that when in one extreme position, the openings *A*, *B*, and *C* in the cover are completely closed, and when in the other extreme position, these openings are completely opened. The mechanism operating the shutter should be of the spring type and constructed so that when at rest the shutter shall exactly close the three openings. When operated to the other extreme, the three cover openings, shall be exactly open and the tip of the exposure tube shall be fully depressed.

A1.1.2.3 *Flame-Exposure Device*—The flame-exposure device (Fig. A1.4) shall have a tip with an opening 0.027 to 0.031 in. (0.69 to 0.79 mm) in diameter. This tip shall be made preferably of stainless steel, although it may be fabricated of other suitable metals. The flame-exposure device shall be equipped with an operating mechanism which, when the shutter is in the "open" position, depresses the tip so that the center of the orifice is between the planes of the under and upper surfaces of the cover proper at a point on a radius passing through the center of the larger opening *A* (Fig. A1.3).

A1.1.2.4 *Pilot Flame*—A pilot flame shall be provided for automatic relighting of the exposure flame. A bead $\frac{3}{32}$ in. (4 mm) in diameter may be mounted on the cover so that the size of the test flame can be regulated by comparison. The tip of the pilot flame shall have an opening the same size as the tip of the flame exposure device (0.027 to 0.031 in. (0.69 to 0.79 mm) in diameter).

A1.1.2.5 *Stirring Device*—The cover shall be equipped with a stirring device (Fig. A1.4) mounted in the center of the cover and carrying two 2-bladed metal propellers. In Fig. A1.4 lower propeller is designated by the letters *L*, *M*, and *N*. This propeller shall measure approximately 38 mm from tip to tip, with each of its two blades 8 mm in width with a pitch of 45°. The upper propeller is designated by the letters *A*, *C*, and *G*. This propeller measures approximately 19 mm, tip to tip, each of its two blades is also 8 mm in width with a pitch of 45°. Both propellers are located on the stirrer shaft in such a manner that, when viewed from the bottom of the stirrer, the blades of one propeller are at 0 and 180° while the blades of the other propeller are at 90 and 270°. A stirrer shaft may be coupled to the motor by a flexible shaft or a suitable arrangement of pulleys.

A1.1.2.6 *Stove*—Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. The stove shall consist of an air bath and a top plate on which the flange of the cup rests.

A1.1.2.7 *Air Bath*—The air bath shall have a cylindrical interior and shall conform to the dimensional requirements in Fig. A1.1. The air bath may be either a flame or electrically heated metal casting (Note A1.1), or an electric-resistance element (Note A1.2). In either case, the air bath must be suitable for use at the temperatures to which it will be subjected without deformation.

NOTE A1.1—If the heating element is a flame or an electric heater, it shall be so designed and used that the temperatures of the bottom and the walls are approximately the same. In order that the air bath internal surfaces should be at a uniform temperature, it should not be less than $\frac{1}{4}$ in. (6.4 mm) in thickness unless the heating element is designed to give equal heat flux densities over all the wall and bottom surfaces.

NOTE A1.2—If the heater is of the electric resistance type, it shall be constructed so that all parts of the interior surface are heated uniformly. The wall and bottom of the air bath shall not be less than $\frac{1}{4}$ in. (6.4 mm) in thickness unless the resistance heating elements are distributed over at least 80 % of the wall and all the bottom of the air bath. A heater having such a distribution of the heating elements positioned at least $\frac{3}{32}$ in. (4.0 mm) away from the internal surface of the heating unit may be used in conjunction with a minimum thickness of $\frac{1}{16}$ in. (1.58 mm) for the wall and bottom of the air bath.

A1.1.2.8 *Top Plate*—The top plate shall be of metal, and shall be mounted with an air gap between it and the air bath. It may be attached to the air bath by means of three screws and spacing bushings. The bushings should be of proper thickness to define an air gap of $\frac{1}{16}$ in. (4.8 mm), and they shall be not more than $\frac{3}{8}$ in. (9.5 mm) in diameter.