

Designation: D 93 – 90^{ε1}

Designation: 34/85

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St. Philadelphia, Pa 19103
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An American National Standard
British Standard 2839
American Association State
Highway Transportation Standard
AASHTO No. T73 – 811

Standard Test Methods for Flash Point by Pensky-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 test methods were adopted as a joint ASTM-IP Standard in 1967.

These test methods have 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.

^{ε1} NOTE—Paragraph 13.1 was corrected editorially in October 1991.

INTRODUCTION

This flash point test method depends on definite rates of temperature increases to control the precision of the method. It is considered to be a non-equilibrium method. The rate of heating will not give the precision expected in all cases because of the low thermal conductivity of some materials. To reduce this effect, Test Method D 3941 was issued in which the heating rate is considered to be an equilibrium method. Due to the slower heating rate, the time required to make a determination is considerably longer. If your specification requires Test Method D 93, do not substitute Test Method D 3941 or any other method without obtaining comparative data and agreement from the specifier.

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 of similar viscosities.

NOTE 1—This test method can 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. These regulations are in degrees Fahrenheit.

1.2 Liquids having viscosities less than 5.5 cSt at 40°C (104°F) do not contain suspended solids or do not have a

tendency to form a surface film while under test conditions can be tested in accordance with Test Method D 56.

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

1.4 *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.*

1.5 *This standard does not purport to address all of the safety problems, 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 hazards statements, see Section 7 and Note 5.*

2. Referenced Documents

2.1 ASTM Standards:

D 56 Test Method for Flash Point by Tag Closed Tester³
D 850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials⁴

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.

Current edition approved Oct. 26, 1990. Published December 1990. Originally published as D 93 – 21 T. Last previous edition D 93 – 85.

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, Vol 06.03.



Documents

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- D 1015 Test Method for Freezing Point of High-Purity Hydrocarbons³
- D 1016 Test Method for Purity of Hydrocarbons from Freezing Points³
- D 1078 Test Method for Distillation Range of Volatile Organic Liquids⁴
- D 3941 Test Method for Flash Point by the Equilibrium Method With a Closed Cup Apparatus⁴
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁵
- E 1 Specification for ASTM Thermometers⁶
- E 300 Practice for Sampling Industrial Chemicals⁷

3. Terminology

3.1 Description of a Term Specific to This Standard:

3.1.1 *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.

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

3.1.1.2 *Discussion*—Occasionally, the application of the test flame will cause a blue halo or an enlarged flame. This generally occurs near the actual flash point but in some cases, especially with halogenated hydrocarbons and admixtures, can occur at any temperature. These phenomena are not to be considered true flash points.

4. Summary of Test 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 can indicate gasoline contamination.

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

6. Apparatus

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

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

6.2.2 For tests in which the indicated reading falls within the limits 60 to 140°C (140 to 284°F), inclusive, an ASTM 88C (88F) Vegetable Oil Flash Thermometer having a range of 10 to 200°C (50 to 392°F) and conforming to the requirements of Specification E 1 shall be used. Equally acceptable is IP thermometer IP 101C.

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

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

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

NOTE 3—There are automatic flash point testers available and in use that may be advantageous in that they save testing time and exhibit other factors which may merit their use. In any cases of dispute the flash point as determined manually is considered the referee test.

7. Hazards

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 can 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 8°C (18°F) 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.

8.3 Obtain a sample in accordance with instructions given in Practice D 4057 or E 300.

8.4 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 17°C (30°F) below its expected flash point.

8.5 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

³ Annual Book of ASTM Standards, Vol 05.03.

⁴ Annual Book of ASTM Standards, Vols 05.03 and 14.03.

⁷ Annual Book of ASTM Standards, Vols 06.03 and 15.05.



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cotton. Warming the sample is permitted, but it should not be heated for prolonged periods or above a temperature of 17°C (30°F) below its expected flash point.

NOTE 4—If the sample is suspected of containing volatile contaminants, the treatment described in 8.4 and 8.5 should be omitted.

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 450 mm (18 in.) wide and 600 mm (24 in.) high.

NOTE 5—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 desirable for good results.

10. Calibration

10.1 Determine the flash point of p-xylene⁸ (Warning—See Note 6.), following the directions in Sections 9 through 11. When the tester is operating properly, a value of $27.2 \pm 1.1^\circ\text{C}$ ($81 \pm 2^\circ\text{F}$) will be obtained.

NOTE 6: Warning—P-xylene is extremely flammable. Harmful if inhaled. Keep away from heat, sparks and open flame. Keep container closed. Use with adequate ventilation. Avoid buildup of vapors and eliminate all sources of ignition, especially non-explosion proof electrical apparatus and heaters. Avoid breathing vapor or spray mist. Avoid prolonged or repeated contact with skin.

10.2 If the flash point obtained on p-xylene is not within the limits stated in 14.1.2, 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 position of the test flame (A1.1.2.2). After adjustment, if necessary, repeat the test. P-xylene having a flash point of $27.2 \pm 1.1^\circ\text{C}$ ($81 \pm 2^\circ\text{F}$) is not a suitable reference in the high temperature range of the Pensky-Martens Closed Tester which can be as high as 370°C (700°F).

10.3 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 Test Method D 850 or 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 Test Method D 1016, from the experimentally determined freezing point, measured by Test Method D 1015.

PROCEDURE A—DETERMINATION OF FLASH POINT OF ORDINARY LIQUIDS

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 $15 \pm 5^\circ\text{C}$ ($60 \pm 10^\circ\text{F}$) or 11°C (20°F) lower than the estimated flash point, whichever is lower. Light the test flame and adjust it to 4 mm ($\frac{1}{2}$ in.) in diameter. Supply the heat at such a rate that the temperature as indicated by the thermometer increases 5 to 6°C (9 to 11°F)/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 110°C (230°F) or below, apply the test flame when the temperature of the sample is from 17°C (30°F) to 28°C (50°F) below the expected flash point and thereafter at a temperature reading that is a multiple of 1°C (2°F). 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 110°C (230°F) apply the test flame in the manner just described at each temperature that is a multiple of 2°C (5°F), beginning at a temperature of 17°C (30°F) to 28°C (50°F) below the expected flash point.

NOTE 7—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.

PROCEDURE 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 $15 \pm 5^\circ\text{C}$ ($60 \pm 10^\circ\text{F}$) or 11°C (20°F) 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 1 nor more than 1.5°C (2 to 3°F)/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 8) at the time of the test. When the pressure differs from 101.3 kPa (760 mm Hg), correct the flash point as follows:

$$\text{Corrected flash point} = C + 0.25 (101.3 - p) \quad (1)$$

$$\text{Corrected flash point} = F + 0.06 (760 - P) \quad (2)$$

$$\text{Corrected flash point} = C + 0.033 (760 - P) \quad (3)$$

where:

C = observed flash point, °C,

F = observed flash points, °F,

P = ambient barometric pressure, mm Hg and

⁸ 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.

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p = ambient barometric pressure, kPa.

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

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

NOTE 8—The barometric pressure used in this calculation is 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 and would not give the correct reading for this test.

14. Precision and Bias

14.1 *Precision*—The precision of Test Procedure A as determined by the statistical examination of the interlaboratory test results is as follows:

14.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the following values in 1 case in 20.

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

14.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical material would, in the long run, exceed the following values only in 1 case in 20.

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

14.1.3 *Bias*—The procedure of this test method has no bias because flash point can be defined only in terms of a test method.

14.1.4 The precision data is not known to have been developed in accordance with Research Report D-2-1007.

14.2 *Precision*—The precision of Test Procedure B as determined by the statistical examination of the interlaboratory test results is as follows:

14.2.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the following value in 1 case in 20:

Repeatability 5°C (9°F)

14.2.2 *Reproducibility*—The difference between 2 single and independent results obtained by different operators working in different laboratories on identical material would, in the long run, exceed the following value only in 1 case in 20.

Reproducibility 10°C (18°F)

14.2.3 The procedure of this test method has no bias because flash point can be defined only in terms of a test method.

14.2.4 The precision data is not known to have been developed in accordance with Research Report D-2-1007.

14.3 The definition of repeatability and reproducibility given here represents different components 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 of 14.2 are from ASTM Committee D-2.

15. Keywords

15.1 flash point; flammability; Pensky-Martens closed cup