

Designation: D56 - 21a D56 - 22

Standard Test Method for Flash Point by Tag Closed Cup Tester¹

This standard is issued under the fixed designation D56; 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.

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

INTRODUCTION

This dynamic flash point test method employs a prescribed rate of temperature rise for the material under test. The rate of heating may not in all cases give the precision quoted in the test method because of the low thermal conductivity of certain materials. To improve the prediction of flammability, Test Method D3941, which utilizes a slower heating rate, was developed. Test Method D3941 provides conditions closer to equilibrium where the vapor above the liquid and the liquid are at about the same temperature. If a specification requires Test Method D56, do not change to Test Method D3941 or other test method without permission from the specifier.

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.

Document Preview

1. Scope*

- 1.1 This test method covers the determination of the flash point, by Tag manual and automated closed testers, of liquids with a viscosity below 5.5 mm²/s (cSt) at 40 °C (104 °F), or below 9.5 mm²/s (cSt) at 25 °C (77 °F), and a flash point below 93 °C (200 °F).
- 1.1.1 Two sets of test conditions are used within this test method: low temperature (LT) test conditions for expected flash points $< 60 \,^{\circ}$ C, and high temperature (HT) test conditions for expected flash points of $\ge 60 \,^{\circ}$ C.
- 1.1.2 For the closed-cup flash point of liquids with the following properties: a viscosity of 5.5 mm²/s (cSt) or more at 40 °C (104 °F); a viscosity of 9.5 mm²/s (cSt) or more at 25 °C (77 °F); a flash point of 93 °C (200 °F) or higher; a tendency to form a surface film under test conditions; or containing suspended solids, Test Method D93 can be used.
- 1.1.3 For cut-back asphalts refer to Test Methods D1310 and D3143.

Note 1—The U.S. Department of Transportation (RSTA)² 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 this test method for those liquids that have a viscosity less than 5.5 mm²/s (cSt) at 40 °C (104 °F) or 9.5 mm²/s (cSt) or less at 25 °C (77 °F), or do not contain suspended solids or do not have a tendency to form a surface film while under test. Other flash

¹ 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 D02.08 on Volatility.

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² For information on United States Department of Transportation regulations, see Codes of United States Regulation 49 CFR Chapter 1 and for information on United States Department of Labor regulations, see Code of United States Regulation 29 CFR Chapter XVII. Each of these items are revised annually and may be procured from the Superintendent of Documents, Government Printing Office, Washington, DC 20402.



point classifications have been established by these departments for liquids using this test method.

- 1.2 This test method can be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and cannot 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 method can be used as elements of fire risk assessment that takes into account all of the factors that are pertinent to an assessment of the fire hazard of a particular end use.
- 1.3 Related standards are Test Methods D93, D1310, D3828, D3278, and D3941.
- 1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.
- 1.5 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use Caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.
- 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. For specific warning statements see 6.5, 7.1, 9.3, 11.1.4, and refer to Safety Data Sheets.
- 1.7 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

iTeh Standards

2.1 ASTM Standards:³

D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D1310 Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus

D3143 Test Method for Flash Point of Cutback Asphalt with Tag Open-Cup Apparatus

D3278 Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus

D3828 Test Methods for Flash Point by Small Scale Closed Cup Tester

D3941 Test Method for Flash Point by the Equilibrium Method With a Closed-Cup Apparatus

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

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

E1 Specification for ASTM Liquid-in-Glass Thermometers

E502 Test Method for Selection and Use of ASTM Standards for the Determination of Flash Point of Chemicals by Closed Cup Methods

2.2 Federal Test Method Standards:⁴

Method 1101, Federal Test Method Standard No. 791b

Method 4291, Federal Test Method Standard No. 141A

2.3 ISO Standards:⁵

ISO 17034 General requirements for the competence of reference material producers

ISO Guide 35 Reference materials—Guidance for characterization and assessment of homogeneity and stability

3. Terminology

- 3.1 Definitions:
- 3.1.1 *flash point, n—in flash point test methods*, the lowest temperature of the test specimen, adjusted to account for variations in atmospheric pressure from 101.3 kPa, at which application of an ignition source causes the vapors of the test specimen to ignite under specified conditions of test.

³ 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.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

⁵ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.



3.1.1.1 Discussion—

The specimen is deemed to have flashed when a flame appears and instantaneously propagates itself over the entire surface of the fluid.

3.1.1.2 Discussion—

When the ignition source is a test flame, the application of the test flame may cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash and should be ignored.

- 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *dynamic* (*non-equilibrium*)—in this type of flash point apparatus, the condition of the vapor above the specimen and the specimen are not at the same temperature at the time that the ignition source is applied.

3.2.1.1 Discussion—

This is primarily caused by the heating of the specimen at the constant prescribed rate with the vapor temperature lagging behind the specimen temperature. The resultant flash point temperature is generally within the reproducibility of the test method.

3.2.2 *equilibrium*—in that type of flash point apparatus or test method, the vapor above the specimen and the specimen are at the same temperature at the time the ignition source is applied.

3.2.2.1 Discussion—

This condition may not be fully achieved in practice, since the temperature is not uniform throughout the specimen and the test cover and shutter are generally cooler.

4. Summary of Test Method

4.1 The specimen is placed in the cup of the tester and, with the lid closed, heated at a slow constant rate. An ignition source is directed into the cup at regular intervals. The flash point is taken as the lowest temperature at which application of the ignition source causes the vapor above the specimen to ignite.

5. Significance and Use

(https://standards.iteh.ai)

5.1 Flash point measures the tendency of the specimen to form a flammable mixture with air under controlled laboratory conditions. It is only one of a number of properties that shall be considered in assessing the overall flammability hazard of a material.

ASTM D56 22

- 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 kerosene can indicate gasoline contamination.

6. Apparatus

- 6.1 Tag Closed Tester (Manual)—The apparatus is shown in Fig. 1 and described in detail in Annex A1.
- 6.2 Tag Closed Tester (Automated)—This apparatus is an automated flash point instrument that is capable of performing the test in accordance with Section 11. The dimensions for the test cup and test cover are shown in Fig. A1.1 and Fig. A1.2.
- 6.3 Shield—A shield 460 mm (18 in.) square and 610 mm (24 in.) high, open in front, is recommended.
- 6.4 *Temperature Measuring Device*—A liquid-in-glass thermometer, as prescribed in Table 1, or an electronic temperature measuring device such as a resistance device or thermocouple. The device shall exhibit the same temperature response as the liquid-in-glass thermometer.
- Note 2—Whenever thermometers complying with ASTM requirements are not available, thermometers complying with the requirements for The Institute of Petroleum thermometer IP 15C PM-Low can be used.
- 6.5 Ignition Source—Natural gas flame, bottled gas flame, and electric ignitors (hot wire) have been found acceptable for use as

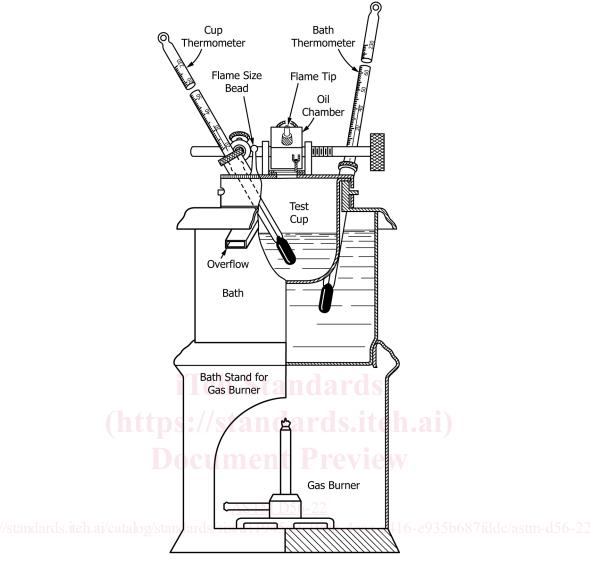


FIG. 1 Tag Closed Flash Tester (Manual)

TABLE 1 Thermometers

For tests	Below 4 °C (40 °F)	At 4 °C to 49 °C	Above 49 °C
		(40 °F to 120 °F)	(120 °F)
Use ASTM Thermometer ^A	57C or (57F)	9C or (9F) 57C or (57F)	9C or (9F)

^A Complete specifications for these thermometers are given in Specification E1.

the ignition source. The gas flame device is described in A1.1.3.3 and Table A1.1. 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.)

6.6 *Cooling System (Optional)*—Samples with low flash point can require a source of cooling for the heating area (see 11.2.1 and 11.3.1).

6.7 Barometer, with accuracy of 0.5 kPa

Note 3—The barometric pressure used in Section 12 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 pre-corrected 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 Erroneously high flash points can be obtained when precautions are not taken to avoid the loss of volatile material. Containers should not be opened unnecessarily to prevent loss of volatile material and possible introduction of moisture. Transfers should not be made unless the sample temperature is at least 10 °C (18 °F) below the expected flash point. When possible, flash point shall be the first test performed on a sample and the sample must be stored at low temperature.
- 8.2 Do not store samples in gas-permeable containers since volatile materials may diffuse through the walls of the enclosure. Samples in leaky containers are suspect and not a source of valid results.
- 8.3 At least 50 mL of sample is required for each test. Refer to sampling information in Practice D4057.

9. Preparation of Apparatus

- 9.1 Support the 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 4—A shield (6.3), having an open front is recommended to prevent drafts from disturbing the vapors above the test cup.

Note 5—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 When using a liquid bath (A1.1.4), for expected flash points < 13 °C (55 °F) or \geq 60 °C (140 °F), use as a bath liquid a 1 + 1 mixture of water and ethylene glycol (Warning—Ethylene Glycol—Poison. Harmful or fatal if swallowed. Vapor harmful. Avoid contact with skin.) For expected flash points \geq 13 °C (55 °F) and < 60 °C (140 °F), either water or a water-glycol mixture can be used as bath liquid. The temperature of the liquid in the bath shall be at least 10 °C (18 °F) below the expected flash point at the time of introduction of the specimen into the test cup. Do not cool bath liquid by direct contact with dry ice (solid carbon dioxide).

Note 6—For flash points < 0 °C (32 °F) the formation of ice on the slide and ignitor dipping mechanism can be minimized by the use of a high vacuum silicone lubricant.

- 9.4 Prepare the manual apparatus or the automated apparatus for operation in accordance with the manufacturer's instructions for calibrating, checking, and operating the equipment.
- 9.5 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 (7.1) capable of removing all of the specimen from the test cup and drying the test cup and cover.

10. Verification of Apparatus

10.1 Verify the performance of apparatus at least once per year by determining the flash point of a certified reference material (CRM), such as those listed in Annex A2, which is reasonably close to the expected temperature range of the samples to be tested. The material shall be tested according to the procedure of this test method and the observed flash point obtained in 11.2 or 11.3 shall be corrected for barometric pressure (see Section 12). The flash point obtained shall be within the limits stated in Table A2.1 for the identified CRM or within the limits calculated for an unlisted CRM (see Annex A2).



- 10.2 Once the performance of the apparatus has been verified, the flash point of secondary working standards (SWSs) can be determined along with their control limits. These secondary materials can then be utilized for more frequent performance checks (see Annex A2).
- 10.3 When the flash point obtained is not within the limits stated in 10.1 or 10.2, check the condition and operation of the apparatus to ensure conformity with the details listed in Annex A1, especially with regard to tightness of the lid (see A1.1.3), the action of the shutter, the position of the ignition source (see A1.1.3.3), and the angle and position of the temperature measuring device (see A1.1.3.4). After any adjustment, repeat the test in 10.1 using a fresh test specimen, with special attention to the procedural details prescribed in the test method.

11. Procedure

11.1 General:

- 11.1.1 Low Temperature (LT) Test Conditions—When the expected flash point of the sample is < 60 °C (140 °F), the heat shall be applied and adjusted during the test so that the temperature of the test specimen rises at a rate of 1 °C (2 °F)/min \pm 6 s. When the temperature of the specimen in the test cup reaches 5 °C (9 °F) below its expected flash point, the ignition source shall be applied and then repeated after each 0.5 °C (1 °F) rise in temperature of the specimen.
- 11.1.2 High Temperature (HT) Test Conditions—When the expected flash point of the sample is \geq 60 °C (140 °F), the heat shall be applied and adjusted during the test so that the temperature of the specimen rises at a rate of 3 °C (5 °F)/min \pm 6 s. When the temperature of the specimen in the test cup reached 5 °C (9 °F) below its expected flash point, the ignition source shall be applied and then repeated after each 1 °C (2 °F) rise in temperature of the specimen.
- 11.1.3 When using LT test conditions, a corrected flash point < 60.0 °C is considered to meet the conditions and does not require a retest. A corrected flash point ≥ 60.0 °C shall be retested under HT test conditions.
- 11.1.4 When using HT test conditions, a corrected flash point of < 60.0 °C shall be retested under LT test conditions.

Note 7—In practice heating rates are not achieved immediately after application of the heat due to the thermal inertia of the apparatus.

11.1.5 (Warning—For certain mixtures containing halogenated hydrocarbons, such as, methylene chloride or trichloroethylene, no distinct flash, as defined, is observed. Instead a significant enlargement of the test flame (not halo effect) and change in color of the test flame from blue to yellowish-orange occurs. Continued heating and testing of these samples above ambient temperature can result in significant burning of vapors outside the test cup, and can be a potential fire hazard. See Appendix X1 and Appendix X2 for more information.)

11.2 Manual Apparatus:

- 11.2.1 Fill the liquid bath (see A1.1.4) with bath liquid in accordance with 9.3 or use a suitable external cooling system (6.6). The bath liquid shall be at an initial temperature necessary for the heating area to be at least 10 °C (18 °F) below the expected flash point, in accordance with the specimen temperature requirements shown below. Using a graduated cylinder and taking care to avoid wetting the cup above the final liquid level, measure $50 \text{ mL} \pm 0.5 \text{ mL} \pm 0.5 \text{ mL}$ of the sample into the cup, both the sample and graduated cylinder being precooled, when necessary, so that the specimen temperature at the time of measurement will be 27 °C ± 5 °C (80 °F ± 9 °F) or at least 10 °C (18 °F) below the expected flash point, whichever is lower. It is essential that the sample temperature be maintained at least 10 °C (18 °F) below the expected flash point during the transfers from the sample container to the cylinder and from the cylinder to the test cup. Destroy air bubbles on the surface of the specimens by use of knife point or other suitable device. Wipe the inside of the cover with a clean cloth or absorbent tissue paper; then attach the cover, with the temperature measuring device in place, to the bath collar.
- 11.2.2 Light the test flame, when used, adjusting it to the size of the small bead on the cover. Commence the heating and test in accordance with 11.1.1 or 11.1.2 as appropriate. When the temperature of the specimen in the test cup reaches 5 °C (9 °F) below the expected flash point, operate the mechanism on the cover in such a manner as to introduce (dip) the ignition source into the vapor space of the cup, and immediately bring it up again. The time consumed for the full operation should be 1 s, allowing equal time periods for the introduction and return. Avoid any hesitation in the operation of depressing and raising the ignition source.



If a flash is observed on the initial operation of the dipping mechanism, discontinue the test and discard the result. In this case, repeat the test from 11.2.1 using a fresh specimen using an expected flash point 10 °C (18 °F) below the previous expected flash point value.

- 11.2.2.1 Exercise care when using a test flame, if the flame is extinguished it cannot ignite the specimen and the gas entering the vapor space can influence the result. When the flame is prematurely extinguished the test shall be discontinued and any result discarded.
- 11.2.3 When the application of the ignition source causes a distinct flash in the interior of the cup, as defined in 3.1.1, observe and record the temperature of the specimen as the observed flash point. Do not confuse the true flash with the bluish halo that sometimes surrounds the ignition source during applications immediately preceding the actual flash.
- 11.2.4 Discontinue the test and remove the source of heat. Lift the lid and wipe the temperature measuring device bulb. Remove the test cup, empty, and wipe dry.
- 11.2.5 If, at any time between the first introduction of the ignition source and the observation of the flash point, the rise in temperature of the specimen is not within the specified rate, discontinue the test, discard the result and repeat the test, adjusting the source of heat to secure the proper rate of temperature rise, or using a modified "expected flash point," or both, as required.
- 11.2.6 Never make a repeat test on the same specimen of sample; always take fresh specimen of sample for each test.
- 11.3 Automated Apparatus:
- 11.3.1 Adjust the external cooling system, if required, to a temperature necessary to cool the heating area 10 °C below the expected flash point.
- 11.3.2 Place the test cup in position in the instrument.
- 11.3.3 Enter the Expected Flash Point; this will allow the heating area to be set to the required minimum starting temperature.

Note 8—To avoid an abnormal heating rate when the specimen is at a low temperature, it is recommended to precool the test cup and cover. This may be accomplished by placing the assembly into position in the instrument while it is cooling to 10 °C (18 °F) below the programmed Expected Flash Point.

Note 9—Flash Point results determined in an "unknown Expected Flash Point mode" should be considered approximate. This value can be used as the Expected Flash Point when a fresh specimen is tested in the standard mode of operation.

11.3.4 Using a graduated cylinder and taking care to avoid wetting the cup above the final liquid level, measure 50 mL \pm 0.5 mL of the sample into the cup, both the sample and the graduated cylinder being precooled, when necessary, so that the specimen temperature at the time of the measurement is 27 °C ± 5 °C (80 °F ± 9 °F) or at least 10 °C (18 °F) below the expected flash point, whichever is lower. It is essential that the sample temperature be maintained at least 10 °C (18 °F) below the expected flash point during the transfers from the sample container to the cylinder and from the cylinder to the test cup. Destroy air bubbles on the surface of the specimen by use of knife point or other suitable device. Wipe the inside of the cover with a clean cloth or absorbent tissue paper; then attach the cover, with the temperature measuring device in place, to the bath collar. Connect the shutter and ignition source activator, if so equipped, into the lid housing. When using a gas test flame, light the pilot flame and adjust the test flame to 4 mm (5/32 in.) in diameter. If the instrument is equipped with an electrical ignition device, adjust according to the manufacturer's instructions. Test the ignition source dipping action, if so equipped, and observe if the apparatus functions correctly. Set up the heating and test in accordance with 11.1.1 or 11.1.2 as appropriate. Press the start key. When the temperature of the specimen in the test cup reaches 5 °C (9 °F) below the expected flash point, the ignition source is introduced (dipped) into the vapor space of the cup, and immediately brought up again. The time consumed for the full operation is 1 s, allowing equal time periods for the introduction and return. If a flash is observed on the initial operation of the dipping mechanism, discontinue the test and discard the result. In this case, repeat the test from 11.3.1 using a fresh specimen using an expected flash point 10 °C (18 °F) below the previous expected flash point value.

Note 10—Care should be taken when cleaning and positioning the lid assembly so as not to damage or dislocate the flash detection system or temperature measuring device. See manufacturer's instructions for proper care and maintenance.

11.3.5 The apparatus shall automatically perform the test procedure as described in 11.2. When the flash point is detected, the