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Standard Test Methods for Flash Point by Small Scale Closed Cup Tester[1]

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

INTRODUCTION

The flash point method is generally used for testing a sample at a specific temperature. At a set temperature, the specimen being tested and the air–vapor mixture above it are close to thermal equilibrium. Test methods for other flash point equipment operated at a specific temperature are described in Test Method D 3941.

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 procedures for the determination of the flash point by a small scale closed tester. The procedures may be used to determine the actual flash point temperature of a sample or whether a product will or will not flash at a specified temperature (flash/no flash). When used in conjunction with an electronic thermal flash detector, these test methods are also suitable for flash point tests on fatty acid methyl esters (FAME).

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

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 *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.* Warning statements appear throughout. See also the Material Safety Data Sheets for the product being tested.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 3941 Test Method for Flash Point by the Equilibrium Method With a Closed-Cup Apparatus

D 6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

2.2 *ISO Standards:*[3]

Guide 34 Quality Systems Guidelines for the Production of Reference Materials

Guide 35 Certification of Reference Materials—General and Statistical Principles

EN ISO 3679 Determination of flashpoint with closed cup equilibrium method

EN ISO 3680 Determination of flashpoint with closed cup equilibrium method

[1] These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility.

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This is also a standard(s) of the Energy Institute issued under the fixed designations IP 523 and IP 524 (originally designated IP 303). The final number indicates the year of last revision. These test methods were originally adopted as a joint ASTM-IP standard in 1979.

[2] 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.

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

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

TABLE 1 Calibration of Tester

Material	*p*-xylene[A,B] *(1.4–Dimethylbenzene)* (**Warning**)[C]	n-butanol[A] (Butan-1-ol) (**Warning**)[C]
Specific gravity, 15.6/15.6°C (60/60°F)	0.860 to 0.866	0.810 to 0.812
Boiling range, °C (°F)	2 (4) maximum including 138.35 (281.03)	2 (4) maximum including 117.5 (243.5)
Freezing point, °C (°F)	52.2 (11.23) minimum	–90 (–130) minimum
Flash point, °C (°F) (acceptable range)	25.6 ± 0.5 (78 ± 1)	36.6 ± 0.8 (97.9 ± 1.7)

[A] Available as Flash Point Check Fluid from Special Products Div., Phillips Petroleum Co., Drawer O, Bergen, TX 79007.
[B] Contains less than 500 v ppm of C_6 and lighter hydrocarbons by gas chromatography.
[C] (**Warning**—Handle xylene and n-butanol with care. Avoid inhalation.)

3. Terminology

3.1 *Definitions:*

3.1.1 *equilibrium*—the vapor above the liquid (specimen) and the liquid in a flash point apparatus specimen cup are at the same temperature at the time the ignition source is applied.

3.1.1.1 *Discussion*—This condition may not be fully achieved in practice. Although the temperature pattern is in equilibrium, the temperature is not uniform throughout the specimen cup because of the contrast between the hot liquid test specimen and the cooler lid and shutter.

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

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

3.1.2.2 *Discussion*—Occasionally, particularly near the actual flash point, 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 Test Method

4.1 *Method A—Flash/No Flash Test*—A specimen of a sample is introduced by a syringe into the cup of the selected apparatus that is set and maintained at the specified temperature. After a specific time a test flame is applied and an observation made as to whether or not a flash occurred.

4.2 *Method B—Finite (or Actual) Flash Point*:

4.2.1 A specimen of a sample is introduced into the cup of the selected apparatus that is maintained at the expected flash point. After a specified time a test flame is applied and the observation made whether or not a flash occurred.

4.2.2 The specimen is removed from the cup, the cup cleaned, and the cup temperature adjusted 5°C (9°F) lower or higher depending on whether or not a flash occurred previously. A fresh specimen is introduced and tested. This procedure is repeated until the flash point is established within 5°C (9°F).

4.2.3 The procedure is then repeated at 1°C (2°F) intervals until the flash point is determined to the nearest 1°C (2°F).

4.2.4 If improved accuracy is desired the procedure is repeated at 0.5°C (1°F) intervals until the flash point is determined to the nearest 0.5°C (1°F).

5. Significance and Use

5.1 Flash point measures the response of the specimen of the sample to heat and flame under controlled laboratory conditions. It is only one of a number of properties that 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 and classify them. 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.

5.4 Requires smaller sample (2 to 4 mL) and therefore reduced test time (1 to 2 min).

6. Apparatus

6.1 *Test Cup and Cover Assembly*—The essential dimensions and requirements of the apparatus are shown in Fig. A1.1 and Table A1.1 of Annex A1. The apparatus and accessories are described in detail in Annex A1. The temperature range from –20 to 300°C may require more than one instrument.

7. Hazards

7.1 The operator must exercise and take appropriate safety precautions during the initial application of the test flame to the sample. Samples containing low-flash material can give an abnormally strong flash when the test flame is first applied.

7.2 When using the instruments at elevated temperatures, take care to keep hands away from the cup area, except for the operating handles as temperatures can exceed 40°C (104°F).

8. Sample

8.1 Erroneously high flash points can be obtained when 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 10°C (18°F) below the expected flash point. Do not use specimens from leaky containers for this test.

8.2 Do not store samples in gas–permeable containers since volatile material can diffuse through the walls of the enclosure. Samples in leaky containers are suspect and not a source of valid results.

8.3 A 2 or 4-mL specimen is required for each test. Obtain at least a 50-mL sample from the bulk test site and store in a clean, tightly closed container.

9. Preparation of Apparatus

9.1 Place the tester on a level, stable surface. Unless tests are made in a draft-free area, surround the tester on three sides with a shield for protection. Do not rely on tests made in a laboratory draft hood or near ventilators.

9.2 Read the manufacturer's instructions on the care and servicing of the instrument and for correct operation of controls. Low temperature testing is ambient to 110°C (230°F). High temperature is 100 to 300°C (212 to 572°F).

10. Calibration and Standardization

10.1 Before initial use determine and plot the relationship between the temperature control dial and the thermometer readings:

10.1.1 Turn the temperature control knob (see Note 1) fully counterclockwise ("0" reading). Advance the temperature control knob clockwise until the indicator light is illuminated see Note 2). Advance the knob clockwise to the next numbered line. After the thermometer mercury column ceases to advance, record the dial reading and the temperature. Advance the knob clockwise to the next numbered line. After the thermometer mercury column ceases to advance, record the dial reading and the temperature. Repeat this procedure through the full range of the instrument. Plot the dial readings versus the respective temperatures.

NOTE 1—When the instrument has two temperature control knobs, set the fine control (center, small knob) at its mid-position and allow it to remain there throughout the calibration. The calibration is determined by adjusting the coarse control (larger, out knob) only.

NOTE 2—When testing at low temperatures, it will be found that the indicator light need not illuminate and the temperature need not rise until an upscale temperature control setting is reached.

10.2 Verify the performance of the 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 Method B, Section 12. Procedure of this test method and the observed flash point obtained in 12.6 shall be corrected for barometric pressure (see Section 13). 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).

10.3 Once the performance of the apparatus has been verified, the flash point of secondary working standards (SWS) can be determined along with their control limits. These secondary materials can then be utilized for more frequent performance checks (see Annex A2).

10.4 When the flash point obtained is not within the limits stated in 10.2 or 10.3, 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 (A1.1.1), the action of the shutter, the position of the ignition source (A1.2), and the angle and position of the temperature measuring device. After any adjustment, repeat the test in 10.2 or 10.3 using a fresh test specimen, with special attention to the procedural details prescribed in the test method.

METHOD A—FLASH/NO FLASH TEST

11. Procedure

11.1 Determine the target flash point as follows:

$$\text{(I) Target flash point, °C} = S_c - 0.25(101.3 - A) \quad (1)$$

$$\text{(II) Target flash point, °C} = S_c - 0.03(760 - B) \quad (2)$$

$$\text{(III) Target flash point, °F} = S_f - 0.06(760 - B) \quad (3)$$

where:

S_c = specification, or uncorrected target, flash point, °C,
S_f = specification, or uncorrected target, flash point, °F, and
B = ambient barometric pressure, mm Hg, and
A = ambient barometric pressure, kPa.

11.2 The barometric pressure used in this calculation shall 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 shall not be used.

11.3 Select the proper instrument, as recommended by the manufacturer, for the target flash point (see 8.2).

11.3.1 Inspect the inside of the sample cup, lid, and shutter mechanism for cleanliness. Use an absorbent paper tissue to wipe clean, when necessary. Put cover in place and lock securely. The filling orifice can be conveniently cleaned with a pipe cleaner.

11.4 *For Target Temperature Above Ambient*—Switch the instrument on and turn the coarse temperature control knob fully clockwise (full on) causing the indicator light to illuminate (see Note 3). When the thermometer indicates a temperature about 3°C (5°F) below the target (or specification) temperature, reduce the heat input to the sample cup by turning the coarse temperature control knob counterclockwise to the desired control point (see 11.1). When the indicator light slowly cycles on and off read the temperature on the thermometer. When necessary, adjust the fine (center) temperature control knob to obtain the desired test (target) temperature. When the test temperature is reached and the indicator lamp slowly cycles on and off, prepare to introduce the specimen of the sample.

NOTE 3—The target temperature can be attained by originally turning the coarse temperature control knob to the proper setting (see 9.1) for the temperature desired rather than to the maximum setting (full on). The elapsed time to reach the temperature will be greater, except for maximum temperature; however, less attention will be required during the intervening period.

11.5 Charge the syringe with a 2-mL portion of the sample to be tested; transfer the syringe to the filling orifice, taking care not to lose any sample; discharge the test portion into the cup by fully depressing the syringe plunger; remove the syringe. When testing B100 Fatty Acid Methyl Esters (FAME), use a 2-mL sample and a 1-min test time, otherwise, for all other sample types for testing above 100°C, conduct this portion of the procedure by charging the sample cup with the required total of 4 mL of sample and setting a test time of 2 min. Alternatively, use the 5-mL syringe preset to deliver 4 mL, and charge all of the specimens at one time. Refer to A1.5.

11.6 Set the timer by rotating its knob clockwise to its stop. Light a match or other source of flame. Slowly open the gas control valve and light the pilot and test flames. Adjust the test flame with the pinch valve to conform to the size of the 4-mm (5⁄32-in.) gage.

11.6.1 After the time signal indicates the portion is at test temperature, apply the test flame by slowly and uniformly

opening the shutter and closing it completely over a period of approximate 2½ s (**Warning**—When the target or specification temperature is not less than 5°C (40°F), crushed ice and water can be used as a charging (cooling) fluid. If below 5°C (40°F) a suitable charging (cooling) fluid is solid carbon dioxide (dry ice) and acetone. (**Warning**—Acetone is extremely flammable. Dry ice shall not contact the eyes or skin.). If the refrigerant-charged cooling module is unavailable, refer to the manufacturer's instruction manual for alternative methods of cooling.) Watch closely for a flash at the cup openings.

11.6.1.1 Never apply the test flame to the portion more than once. A fresh specimen of the sample must be used for each test.

11.6.2 The specimen is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the specimen (see 3.1.2).

11.7 Record the test result as flash (or no flash) and the test temperature. It is also recommended the instrument used and the appropriate ASTM or IP standard number be recorded.

11.8 Turn off the pilot and test flames using the gas control valve. Remove the sample and clean the instrument. It may be necessary to allow the cup temperature to fall to a safe level before cleaning.

11.9 *For Target Temperature Below Ambient*:

11.9.1 The instrument power switch is to be in the off position. Fill the refrigerant-charged cooling block with a suitable material (**Warning—**When the target or specification temperature is not less than 5°C (40°F), crushed ice and water can be used as a charging (cooling) fluid. If below 5°C (40°F) a suitable charging (cooling) fluid is solid carbon dioxide (dry ice) and acetone (**Warning**—Acetone is extremely flammable. Dry ice shall not contact the eyes or skin.). If the refrigerant-charged cooling module is unavailable, refer to the manufacturer's instruction manual for alternative methods of cooling.). Raise the lid and shutter assembly, and position the base of the block in the sample cup, being careful not to damage or mar the cup. When the thermometer reads approximately 10°C (18°F) (**Warning**—Do not cool the sample block below −38°C (−36°F), the freezing point of mercury.) below the target temperature, remove the cooling block and quickly dry the cup and underside of lid and shutter with a paper tissue to remove any moisture. Immediately close the lid and shutter assembly and secure. Prepare to introduce the portion of the sample using the syringe, both of which have been precooled to a temperature 10 to 20°F (5 to 10°C) below the target temperature.

11.9.2 Follow the procedure in 11.5-11.8.

11.9.3 For target temperatures below ambient do not set the timer. Adjust the test flame and allow the temperature to rise under ambient conditions until the target temperature is reached. Immediately apply the test flame as detailed. To reduce time for running the test, as the temperature nears ambient, increase the temperature of the cup by rotating the tester controller clockwise slowly until the target temperature is reached.

11.9.4 Continue as directed in 11.6.2-11.8.

METHOD B—FINITE OR ACTUAL FLASH POINT DETERMINATION

12. Procedure

12.1 Select the proper instrument, as recommended by the manufacturer, for the expected flash point (see 9.2).

12.1.1 Inspect the inside of the specimen cup, lid, and shutter mechanism for cleanliness. Use an absorbent paper tissue to wipe clean, if necessary. Put cover in place and lock securely. The filling orifice can be conveniently cleaned with a pipe cleaner.

12.2 *For Tests Where the Expected Flash Point is Above Ambient*—Turn the coarse temperature control knob fully clockwise (full on), causing the indicator light to illuminate. When the thermometer reaches a temperature 3°C (5°F) below the estimated flash point, turn the coarse temperature knob counterclockwise to the dial reading representing the estimated flash point temperature as shown on the calibration curve (see 9.1). When the indicator light slowly cycles on and off, read the temperature on the thermometer. If necessary, adjust the fine temperatures control knob to obtain the exact desired temperature.

12.3 Charge the syringe with a 2 mL portion of the sample to be tested; transfer the syringe to the filling orifice, taking care not to lose any specimen; discharge the test specimen into the cup by fully depressing the syringe plunger; remove the syringe. When testing B100 Fatty Acid Methyl Esters (FAME), use a 2-mL sample and a 1-min test time, otherwise for all other sample types for testing above 100°C, conduct this portion of the procedure by charging the sample cup with the required total of 4 mL of sample and setting a test time of 2 min. Alternatively, use the 5-mL syringe preset to deliver 4 mL and charge all of the specimen at one time. Refer to A1.5.

12.3.1 Set the timer by rotating its knob clockwise to its stop. Light the match or other source of flame. Slowly open the gas control valve and ignite the pilot and test flames. Adjust the test flame with the pinch valve to conform to the size of the 4-mm (5⁄32-in.) gage.

12.3.2 After the audible time signal indicates the specimen is at test temperature, apply the test flame by slowly and uniformly opening the shutter and then closing it completely over a period of approximately 2½ s. Watch closely for a flash at the cup opening.

12.3.2.1 The specimen is deemed to have flashed only when a large flame appears and instantaneously propagates itself over the surface of the specimen (see 13.1.1).

12.3.3 Turn off the pilot and test flames using the gas control valve. When the cup temperature falls to a safe level, remove the specimen and clean the instrument.

12.4 If a flash is observed in 12.3.2 repeat the procedure given in 12.2 and 12.3 testing a new specimen at a temperature 5°C (9°F) below that at which the flash is observed.

12.4.1 If necessary, repeat 12.4 lowering the temperature 5°C (9°F) each time, until no flash is observed (**Warning**—When the target or specification temperature is not less than 5°C (40°F), crushed ice and water can be used as a charging (cooling) fluid. If below 5°C (40°F) a suitable charging (cooling) fluid is solid carbon dioxide (dry ice) and acetone (**Warning**—Acetone is extremely flammable. Dry ice shall not