



Standard Test Method for Flash Point by Small Scale Closed Cup Tester (Ramp Method)^{1, 2}

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INTRODUCTION

This flash point test method is a dynamic test method and depends on a definite rate of temperature increase to control 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 certain materials. The use of an equilibrium method such as Test Methods **D 3828**, Method B improves the prediction of flammability for such materials, as the vapors above the test specimen and the test specimen are closer to thermal equilibrium.

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 than that specified.

1. Scope

1.1 This test method covers the determination of the flash point of aviation turbine fuel, diesel fuel, kerosine and related products in the temperature range of 40 to 135°C by a small scale closed cup apparatus.

1.2 This test method is only applicable to homogeneous materials that are liquid at or near ambient temperature and at temperatures required to perform the test.

1.3 This test method is not applicable to liquids contaminated by traces of highly volatile materials.

1.4 This test method is a dynamic method and depends on a definite rate of temperature increase. It is one of many flash point methods available, and every flash point test method, including this one, is an empirical one.

1.5 If the user's specification requires a defined flash point method, neither this test nor any other method should be substituted for the prescribed method without obtaining comparative data and an agreement from the specifier.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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 hazard statements, see Section 7 and the Material Safety Data Sheet for the product being tested.

2. Referenced Documents

2.1 ASTM Standards:³

D 3828 Test Methods for Flash Point by Small Scale 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

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

D 6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

E 300 Practice for Sampling Industrial Chemicals

2.2 ISO Standards:⁴

ISO Guide 34 Quality Systems Guidelines for the Production of Reference Materials

¹ This test method is 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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² This test method is being jointly developed and harmonized with the Energy Institute.

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

ISO Guide 35 Certification of Reference Material—General and Statistical Principles

3. Terminology

3.1 Definitions:

3.1.1 *flash point, n*—lowest temperature corrected to a pressure of 101.3 kPa, at which application of a test flame causes the vapors of a test specimen of the sample to ignite momentarily under the specified conditions of the test.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *dynamic, adj*—the condition where the vapor above the test specimen, and the test cup, are not in temperature equilibrium at the time the test flame is applied.

4. Summary of Test Method

4.1 A 2 ± 0.1 mL test specimen is introduced into the test cup that is then heated automatically at a constant rate of $2 \pm 0.5^\circ\text{C}/\text{min}$. A gas test flame is directed through an opening shutter, in the test cup lid, at specified temperature intervals until a flash is detected by the automatic flash detector. The flash is reported as defined in 3.1.1.

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 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. Consult the particular regulation involved for precise definitions of these classifications.

5.3 This test method can be used to measure and describe the properties of materials in response to heat and a test flame under controlled laboratory conditions and shall not be used to describe or appraise the fire hazard or fire risk of materials under actual fire conditions. However, results of this test method may be used as elements of a 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.

6. Apparatus

6.1 *Flash Point Apparatus*—The apparatus consists of an electrically heated test cup that is controlled automatically to give a temperature increase of $2.0 \pm 0.5^\circ\text{C}/\text{min}$, a lid and shutter assembly, a pilot and test flame, an automatic flash detector and a temperature display that displays and holds the test temperature when a flash is detected. The key parts and dimensions are described in **Annex A1** and illustrated in **Fig. A1.1**.

6.2 *Syringe*, 2 mL, adjusted to deliver 2.00 ± 0.1 mL.

6.3 *Barometer*, accurate to 0.5 kPa. Barometers pre-corrected to give sea level readings, such as those used at weather stations and airports, shall not be used.

NOTE 1—Automatic barometric correction in accordance with 12.2 may be installed in the apparatus.

6.4 *Cooling Bath or Freezer*, for cooling the samples, if required, and capable of cooling the sample to at least 10°C

below the expected flash point. If a freezer is used, it shall be of explosion-protected design.

6.5 *Draft Shield*, a shield fitted at the back and on two sides of the instrument, for use in circumstances where natural protection from drafts does not exist.

NOTE 2—A shield 350-mm high, 480-mm wide, and 240-mm deep, is suitable.

7. Reagents and Materials

7.1 *Cleaning Solvent*—Use only noncorrosive solvents capable of cleaning the test cup and lid assembly. Two 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.)

7.2 *Butane or Natural Gas*—For use as the pilot and test flame. (**Warning**—Butane and natural gases are flammable and a health hazard.)

8. Sampling

8.1 Obtain at least a 25 mL sample from a bulk test site in accordance with Practices **D 4057**, **D 4177**, **E 300** or other comparable sampling practices.

8.2 Store samples in clean, tightly sealed containers at normal room temperature (20 to 25°C) or colder. Ensure that the container is 85 to 95 % full.

8.3 Do not store samples for an extended period of time in gas permeable containers, such as plastic, because volatile material may diffuse through the walls of the container. Samples in leaky containers are suspect and not a source of valid results.

8.4 Erroneously high flash points can be obtained when precautions are not taken to avoid loss of volatile materials. Do not open containers unnecessarily. Do not make a transfer unless the sample temperature is at least 10°C below the expected flash point. Where possible perform the flash point as the first test on the sample.

8.5 Samples containing dissolved or free water may be dehydrated with calcium chloride. Warming the sample is permitted, but do not heat the sample above a temperature of 10°C below its expected flash point. (**Warning**—If the sample is expected of containing volatile components, the treatment as described in 8.5 should be omitted.)

8.6 Cool or adjust the temperature of the sample and its container to at least 10°C below the expected flash point before opening to remove the test specimen. If an aliquot of the original sample is to be stored prior to testing, ensure that the container is filled to between 85 and 95 % of its capacity. Gently mix the subsample to ensure uniformity while minimizing the possible loss of volatile components and light ends.

NOTE 3—Results of flash point tests may be affected if the sample volume falls below 85 % of the container's capacity.

9. Preparation of Apparatus

9.1 Support the apparatus (**Annex A1**) on a level and steady surface in a draft-free position (see Notes 4 and 5).

NOTE 4—A draft shield (6.5) is recommended to be used when natural protection from drafts does not exist.

NOTE 5—When testing materials that may produce toxic vapors, the apparatus may be located in a fume hood with an individual control of air flow, adjusted such that the toxic vapors can be withdrawn without causing air currents around the test cup during the test.

9.2 Clean the test cup, cover, and its accessories with an appropriate solvent (7.1) to remove traces of gum or residue from the previous test.

NOTE 6—A stream of clean dry air may be used to remove the last traces of solvent used.

NOTE 7—The filler orifice may be cleaned with a pipe cleaner.

10. Verification of Apparatus

10.1 Verify that the temperature measuring device is in accordance with A1.7.

10.2 Verify the performance of the apparatus at least once per year by determining the flash point of a Certified Reference Material (CRM) that has a certified flash point reasonably close to the expected range of the samples to be tested. Examples of materials that may be used to create CRMs are listed in Appendix X1. The flash point shall be determined using the procedures stated in Section 11. Appendix X1 indicates typical limits in Table X1.1 for a typical CRM and shows how to calculate limits for an unlisted CRM.

10.3 Once the performance of the apparatus has been verified using a CRM, the flash point of Secondary Working Standards (SWS) can be determined along with their control limits. These SWS can then be utilized for more frequent performance checks. (See Appendix X1.)

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 conformance to the details listed in Annex A1, especially with regard to the action of the shutter, the position and size of the test flame, the heating rate and the temperature sensor readings. 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.

11. Procedure

11.1 Set the test cup temperature to $15 \pm 5^\circ\text{C}$ below the expected flash point temperature.

11.2 When the test cup is at the required temperature, charge a clean and dry syringe (6.2), adjusted to a temperature at least 10°C below the expected flash point, with a 2 mL test specimen. Close the sample container immediately after withdrawal of the test specimen, to minimize any loss of volatile components.

11.3 Carefully transfer the syringe to the filler orifice and discharge the test specimen into the test cup by fully depressing the syringe plunger. Remove the syringe.

11.4 Ignite the pilot and test flames. Adjust the test flame to conform to the size of the 4 mm gauge ring. Reset the flash detector (A1.9). Start the test.

NOTE 8—The ignition of the test flame and control of the gas supply may be automated on some instruments.

11.5 The test cup is heated automatically at such a rate that the temperature, as indicated by the temperature sensor, increases by 1.5 to $2.5^\circ\text{C}/\text{min}$. The test flame is dipped into the test cup, as the shutter is slowly and uniformly opened and

closed over a 2 to 3-s period, at 1°C intervals for temperatures up to and including 100°C and thereafter every 2°C for temperatures over 100°C .

11.5.1 Observe the first two dips of the flame for continuous burning in the orifice. If a continuous luminous flame burns in the orifice when the slide is opened and the test flame is introduced, then the flash point lies considerably below the test temperature. In such cases, discontinue the test and repeat the test with a fresh test specimen using a lower expected flash point.

11.5.2 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 is ignored by the flash detector.

11.6 Record the displayed and held temperature of the temperature sensor when the application of the test flame causes a flash to be automatically detected.

11.7 Record the ambient barometric pressure using a barometer (6.3) in the vicinity of the apparatus at the time of the test.

NOTE 9—It is not considered necessary to correct the barometric pressure reading to 0°C , although some barometers are designed to make this correction automatically.

11.8 When the temperature at which the flash is observed is less than 10°C or greater than 30°C from the start temperature of the test, the result is not valid. Repeat the test using a fresh test specimen adjusting the temperature of the first application of the test flame until a valid determination is made.

12. Calculation

12.1 *Barometric Pressure Conversion*—If the barometric pressure reading is measured in a unit other than kilopascals, convert to kilopascals using one of the following equations:

$$\text{Reading in hPa} \times 0.1 = \text{kPa} \quad (1)$$

$$\text{Reading in mbar} \times 0.1 = \text{kPa} \quad (2)$$

$$\text{Reading in mmHg} \times 0.1333 = \text{kPa} \quad (3)$$

12.2 *Correction of Detected Flash Point to Standard Atmospheric Pressure*—Calculate the flash point corrected to a standard atmospheric pressure of 101.3 kPa, T_c , using the following equation:

$$T_c = T_D + 0.25 (101.3 - p) \quad (4)$$

where:

T_D = the detected flash point temperature at ambient barometric pressure, expressed in degrees Celsius, and
 p = the ambient barometric pressure, expressed in kilopascals.

NOTE 10—This equation is strictly correct only within the barometric pressure range from 98.0 to 104.7 kPa.

13. Report

13.1 Report the source, type and identification of the material tested, plus the dates sampled and tested.

13.2 Refer to this test method and report any deviation, by agreement or otherwise, from the procedure specified.

13.3 Report the corrected flash point of the test specimen rounded to the nearest 0.5°C in accordance with Test Method D 7236.