



# Standard Test Methods for Flash Point by Small Scale Closed Cup Tester<sup>1</sup>

This standard is issued under the fixed designation D3828; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

## INTRODUCTION

These small scale flash point methods are generally used for testing a sample at a specific temperature where the specimen being tested and the air–vapor mixture above it are close to thermal equilibrium. Test Method **D3941** covers other flash point equipment operated at a specific temperature.

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 flash point tests, within the range of –30 to 300°C, of petroleum products and biodiesel liquid fuels, using a small scale closed cup tester. The procedures may be used to determine, whether a product will or will not flash at a specified temperature (flash/no flash Method A) or the flash point of a sample (Method B). When used in conjunction with an electronic thermal flash detector, these test methods are also suitable for flash point tests on biodiesels such as 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 *Warning*—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA’s website (<http://www.epa.gov/mercury/faq.htm>) for additional information. Users should be aware that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

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 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:<sup>2</sup>

**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

**D4177** Practice for Automatic Sampling of Petroleum and Petroleum Products

**D6299** Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

**E300** Practice for Sampling Industrial Chemicals

#### 2.2 ISO Standards:<sup>3</sup>

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

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

**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 Flash Point—Rapid Equilibrium Closed Cup Method

**EN ISO 3680** Determination of Flash/No Flash—Rapid Equilibrium Closed Cup Method

2.3 *Energy Institute Standards*:<sup>4</sup>

**IP 523** Determination of flash point — Rapid equilibrium closed cup method

**IP 524** Determination of flash/no flash — Rapid equilibrium closed cup method

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *equilibrium, n—in flash point test methods*, the condition where the vapor above the test specimen, and the test specimen 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, since the temperature may not be uniform throughout the test specimen, and the test cover and shutter on the apparatus can be cooler or warmer.

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

### 4. Summary of Test Methods

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

4.2 *Method B—Finite (or Actual) Flash Point*—This method essentially repeats Method A a number of times, and by changing the test temperature and test specimen a number of times, determines the flash point.

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

4.2.2 The test specimen is removed from the test cup; the test cup and cover are cleaned, and the test temperature adjusted 5°C (9°F) lower or higher depending on whether or not a flash occurred previously. A fresh test 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).

#### 4.3 Test Time and Specimen Volume:

4.3.1 For all products, except biodiesel; for test temperatures up to and including 100°C (212°F), the test time is 1 minute and the specimen volume is 2 mL.

4.3.2 For all products, except biodiesel; for test temperatures over 100°C (212°F), the test time is 2 minute and the specimen volume is 4 mL.

4.3.3 For biodiesel; for all test temperatures, the test time is 1 minute and the specimen volume is 2 mL.

### 5. Significance and Use

5.1 Flash point measures the response of the test specimen to heat and ignition source 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 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 These test methods use a smaller sample (2 to 4 mL) and a shorter test time (1 to 2 min) than traditional test methods.

5.5 Method A, **IP 524** and **EN ISO 3680** are similar methods for flash no-flash tests. Method B, **IP 523** and **EN ISO 3679** are similar methods for flash point determination.

### 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 is from –30 to 300°C. Some versions of the apparatus may not cover the full temperature range.

6.2 *Barometer*, accurate to 0.5 kPa. Barometers that have been pre-corrected for use at weather stations or airports are not suitable.

6.3 *Draft Shield*—A shield located at the back and on two sides of the instrument, for use in circumstances where protection from drafts does not exist.

### 7. Reagents and Materials

7.1 *Cleaning Solvent*—Use only non-corrosive solvents capable of cleaning the test cup and cover. Two commonly used solvents are toluene and acetone. (**Warning**—Toluene, acetone and many other solvents are flammable and a health hazard. Dispose of solvents and waste material in accordance with local regulations.)

7.2 *Butane, Propane and Natural Gas*—For use as a pilot and ignition source (not required if an electric ignitor is used). (**Warning**—Butane, propane and natural gases are flammable and a health hazard.)

### 8. Sampling

8.1 Obtain at least a 50 mL sample from a bulk test site in accordance with Practices **D4057**, **D4177**, **E300** or other comparable sampling practices.

<sup>4</sup> Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., <http://www.energyinst.org.uk>.

8.2 Store samples in clean, tightly sealed containers at normal room temperature (20 to 25°C) or colder.

8.3 Do not store samples for an extended period of time in gas permeable containers, such as those made of plastic, because volatile material can 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. (**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.

NOTE 1—Results of flash point tests can be affected if the sample volume falls below 50 % of the container’s capacity.

8.7 If sufficiently fluid, mix samples by gently shaking by hand prior to removal of the test specimen, taking care to minimize the loss of volatile components. If the sample is too viscous at ambient temperature, gently warm the sample in its container to a temperature not warmer than 10°C below the test temperature, such that the sample can be mixed by gentle shaking.

8.8 If the sample cannot be made sufficiently fluid to be introduced into the test cup through the orifice by heating in accordance with 8.7, transfer the test specimen with a solids dispenser or spatula while the cover is open. The specimen size can be the mass equivalent of the required volume and the specimen should be spread over the bottom of the test cup as evenly as possible. Precision has not been determined for solid samples.

## 9. Preparation of Apparatus

9.1 Place the apparatus on a level, stable surface. Unless tests are made in a draft-free area, surround the tester on three sides with a draft shield (see 6.3) for protection. Do not rely on tests made in a laboratory draft hood unless the extracted air and vapors can be withdrawn without causing air currents over the test cup during the ignition source application period.

9.2 Read the manufacturer’s instructions on the care and servicing of the instrument and for the correct operation of any controls.

9.3 Prepare the apparatus for operation in accordance with the manufacturers’ instructions for calibrating, checking and operating the equipment, especially the operation of the ignition source. (**Warning**—An incorrectly set test flame size or setting of an electric ignitor can significantly affect the test result.)

9.4 Clean the test cup, cover and its accessories with an appropriate solvent (7.1) to remove any traces of gum or

residue from the previous test. Wipe dry with absorbent paper. A stream of dry clean air may be used to remove the last traces of solvent used. A pipe cleaner may be used to clean the filler orifice.

9.5 Measure and record the barometric pressure (6.2) before commencing a test.

9.6 Use an electronic thermal flash detector for flash point tests on biodiesels such as fatty acid methyl esters (FAME) (see A1.7).

9.7 For sub-ambient test temperatures see Annex A4, unless the apparatus has integral test cup cooling facilities.

## 10. Verification of Apparatus

10.1 Verify that the temperature measuring device is in accordance with A1.2.2.

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 and the observed flash point obtained in 12.1.6 and 12.1.6.1 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 cover (A1.2.1), the action of the shutter, the size or intensity of the ignition source, the position of the ignition source (A1.3), the operation of the flash detector (if fitted) and correct reading 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 For specification purposes carry out a flash/no flash test at a temperature (actual) that allows for a correction due to the ambient atmospheric pressure at the time of the test. Use the following equations to convert a specification flash point test requirement to the actual test temperature required for the test and then round to the nearest 0.5°C (1°F).

$$\text{Actual test temperature, } ^\circ\text{C} = S_c - 0.25(101.3 - A) \quad (1)$$

$$\text{Actual test temperature, } ^\circ\text{C} = S_c - 0.03(760 - B) \quad (2)$$

$$\text{Actual test temperature, } ^\circ\text{F} = S_f - 0.06(760 - B) \quad (3)$$

where:

$S_c$  = specification, or uncorrected target test temperature, °C,

$S_f$  = specification, or uncorrected target test temperature, °F,

$B$  = ambient barometric pressure, mm Hg, and

$A$  = ambient barometric pressure, kPa.

11.2 Inspect the test cup and cover for cleanliness and correct operation, especially with regard to tightness of the cover (A1.2.1), the action of the shutter, the size or intensity of the ignition source and the position of the ignition source (A1.3). Clean if necessary (9.3). Put the cover in place and close securely.

11.3 Follow the manufacturer's instructions to set the test temperature and the test time, and select the test specimen volume in accordance with Table 1.

11.4 When the test cup is at the test temperature, fill the appropriate syringe (A1.5, A1.6) with the sample to be tested; transfer the syringe to the filling orifice, taking care not to lose any sample; discharge the test specimen into the test cup by fully depressing the syringe plunger; remove the syringe.

11.5 Start the test timer; light the pilot light and adjust the test flame (if used) to conform to the 4 mm ( $5/32$  in.) gauge.

11.6 When the end of the test time is indicated, apply the ignition source by slowly and uniformly opening the shutter and closing it completely over a period of  $2\frac{1}{2}$  s. Watch closely for a flash at the test cup openings.

11.6.1 The test specimen is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the test specimen (see 3.1.2). Occasionally, particularly near the actual flash point, application of the ignition source can cause a blue halo or an enlarged flame; this is not a flash and should be ignored.

11.7 Record the test result as flash (or no flash) and the test temperature.

11.8 Turn off the pilot and test flames (if used). Remove the test specimen and clean the test cup and cover. It may be necessary to allow the test cup temperature to fall to a safe level before cleaning.

## METHOD B—FLASH POINT DETERMINATION

### 12. Procedure

12.1 This procedure repeats the procedures of Method A (11.3 through 11.8) a number of times. Each test uses a fresh test specimen and a different temperature. The change of temperature allows the determination of two temperatures  $1^\circ\text{C}$  ( $2^\circ\text{F}$ ) or  $0.5^\circ\text{C}$  ( $1^\circ\text{F}$ ) apart for which the lower temperature did not result in a flash while the higher temperature resulted in a flash (the flash point).

12.1.1 Select the expected flash point of the sample as the initial test temperature and follow the procedure 11.3 to 11.8.

12.1.2 If a flash is observed repeat the procedure given in 11.3 to 11.8 testing a fresh specimen at a temperature  $5^\circ\text{C}$  ( $9^\circ\text{F}$ ) lower each time until no flash is observed.

**TABLE 1 Test Conditions**

Sample	Test Temperatures	Specimen Volume	Test Time
All except biodiesel	$\leq 100^\circ\text{C}$ ( $212^\circ\text{F}$ )	2 mL	1 min
All except biodiesel	$> 100^\circ\text{C}$ ( $212^\circ\text{F}$ )	4 mL	2 min
Biodiesel	$\leq 300^\circ\text{C}$ ( $572^\circ\text{F}$ )	2 mL	1 min

12.1.2.1 Proceed to 12.1.4.

12.1.3 When no flash was observed repeat the procedure given in 11.3 to 11.8 testing a fresh specimen at a temperature  $5^\circ\text{C}$  ( $9^\circ\text{F}$ ) higher each time until a flash is observed.

12.1.4 Having established a flash within two temperatures  $5^\circ\text{C}$  ( $9^\circ\text{F}$ ) apart, repeat the procedure at  $1^\circ\text{C}$  ( $2^\circ\text{F}$ ) intervals from the lower of the two temperatures until a flash is observed.

12.1.5 Record the temperature of the test when this flash occurs as the flash point, allowing for any known thermometer correction.

12.1.6 The flash point determined in 12.1.4 will be to the nearest  $1^\circ\text{C}$  ( $2^\circ\text{F}$ ).

12.1.6.1 If improved accuracy is desired (that is, to the nearest  $0.5^\circ\text{C}$  ( $1^\circ\text{F}$ )) test a fresh test specimen at a temperature  $0.5^\circ\text{C}$  ( $1^\circ\text{F}$ ) below that at which the flash was observed in 12.1.4. If no flash is observed, the temperature recorded in 12.1.4 is the flash point to the nearest  $0.5^\circ\text{C}$  ( $1^\circ\text{F}$ ). If a flash is observed at the lower temperature (12.1.6), record this latter temperature as the flash point.

12.1.7 Turn off the pilot and test flames, if used. When the cup temperature falls to a safe level, remove the test specimen and clean the cup and cover.

### 13. Calculation

13.1 *Method A*—No further calculations are required as the effects of barometric pressure were corrected for in Eq 1-3 in 11.1.

13.1.1 If the effects of barometric pressure were not corrected before the flash/no flash test, Eq 4-6 shall be used to calculate the corrected test temperature.

$$\text{Corrected (specification) test temperature } (^\circ\text{C}) = C + 0.25(101.3 - A) \quad (4)$$

$$\text{Corrected (specification) test temperature } (^\circ\text{F}) = F + 0.06(760 - B) \quad (5)$$

$$\text{Corrected (specification) test temperature } (^\circ\text{C}) = C + 0.03(760 - B) \quad (6)$$

where:

$C$  = actual test temperature,  $^\circ\text{C}$ ,

$F$  = actual test temperature,  $^\circ\text{F}$ ,

$A$  = ambient barometric pressure, kPa, and

$B$  = ambient barometric pressure, mm Hg.

13.2 *Method B*—If the ambient barometric pressure (9.5) differs from 101.3 kPa (760 mm Hg) correct the flash point as follows:

$$\text{Corrected flash point } (^\circ\text{C}) = C + 0.25(101.3 - A) \quad (7)$$

$$\text{Corrected flash point } (^\circ\text{F}) = F + 0.06(760 - B) \quad (8)$$

$$\text{Corrected flash point } (^\circ\text{C}) = C + 0.03(760 - B) \quad (9)$$

where:

$C$  = observed flash point,  $^\circ\text{C}$ ,

$F$  = observed flash point,  $^\circ\text{F}$ ,

$A$  = ambient barometric pressure, kPa, and

$B$  = ambient barometric pressure, mm Hg.

## 14. Report

14.1 Method A (flash/no flash), report flash (or no flash) at the specification (corrected) temperature (report temperature) rounded to the nearest 0.5°C (1°F), and that Method A was used.

14.2 Method B (flash point determination), report the corrected flash point rounded to the nearest 0.5°C (1°F), and that Method B was used.

14.3 Report the identification of the material tested, the test date and any deviation, by agreement or not, from the procedures specified.

## 15. Precision and Bias <sup>5</sup>

15.1 *Precision*—The precision of this test method as determined by statistical examination of interlaboratory results is as follows. See **Annex A3** for provisional precision of fatty acid methyl esters (FAME).

**NOTE 2**—The precision or relative bias has not yet been determined when using electric ignitors. However supporting data, covering the 22 to 110°C range, has been made available.<sup>6</sup> A full interlaboratory study is being planned in conjunction with the Energy Institute to reestablish the precision of this test method, and to establish the degree of agreement between gas and electric ignitors.

15.1.1 *Repeatability*—The difference between the two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material,

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1214.

<sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1670.

would, in the long run, in the normal and correct operation of this test method, exceed the values shown in **Table 2** only in 1 case in 20.

Range	Repeatability
20 to 70°C	0.5°C
above 70°C	0.022 $M^{0.9}$ °C
where: $M$ = mean of two results	

15.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, would, in the long run, in the normal and correct operation of this test method, exceed the values shown in **Table 2** only in 1 case in 20.

Range	Reproducibility
20 to 70°C	0.03 ( $M + 29$ ) °C
above 70°C	0.083 $M^{0.9}$ °C
where: $M$ = mean of the two results	

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

## 16. Keywords

16.1 combustible; fire risk; flammable; flash point; volatile

**TABLE 2 Calculated Repeatability and Reproducibility**

Temperature, °C	Repeatability, °C	Reproducibility, °C
20	0.5	1.5
70	0.5	3.0
93	1.3	4.9
149	2.0	7.5
204	2.6	10.0
260	3.3	12.4

## ANNEXES

### (Mandatory Information)

#### A1. FLASH TEST APPARATUS

A1.1 *Flash Test Apparatus*, see **Fig. A1.1** and **Table A1.1**.

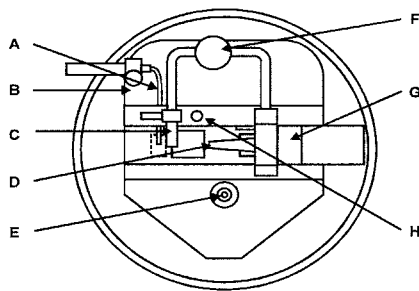
A1.2 *Test Cup*, consisting of an aluminum alloy or nonrusting metal block of suitable conductivity with a cylindrical depression over which is fitted a cover. A temperature measuring device is inserted in the block.

A1.2.1 The cover comprises a lid fitted with an opening shutter and a device capable of inserting an ignition source into the test cup when the shutter is open. When inserted, the nozzle of the ignition source shall intersect the plane of the underside of the cover. The cover is also provided with an orifice extending into the sample well for insertion of the test specimen and also a suitable clamping device for securing the cover tightly to the metal block. The three openings in the cover shall be within the diameter of the sample well. When the shutter is in the open position, the two openings in the shutter shall coincide with the two corresponding openings in the cover.

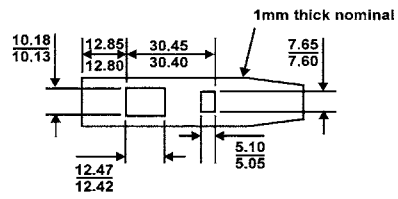
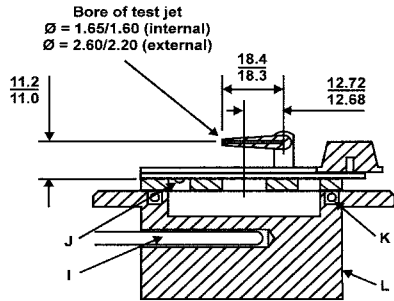
A1.2.2 Electrical heaters are attached to the test cup in a manner that provides efficient transfer of heat. The heater control shall be capable of controlling the test cup temperature, as measured by the temperature measuring device and in a draft-free area, to within  $\pm 0.5^\circ\text{C}$  ( $\pm 1.0^\circ\text{F}$ ) for test temperatures up to 100°C, and  $\pm 2.0^\circ\text{C}$  ( $\pm 4.0^\circ\text{F}$ ) for test temperatures above 100°C during the test. Cooling of the test cup may use Peltier effect devices, an external cryostat or a cooling insert (see **Annex A4**).

A1.3 *Test Flame and Pilot Flame*, for dipping into the test cup to test for a flash, and a pilot flame, to maintain the test flame, are required. When inserted, the nozzle of the ignition source shall intersect the plane of the underside of the cover. These flames may be fueled from an external butane or propane supply<sup>7</sup> or from a self-contained or attached tank of butane or

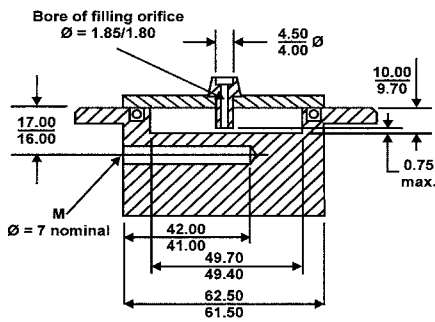
<sup>7</sup> External fuel adapters are available from instrument sources.



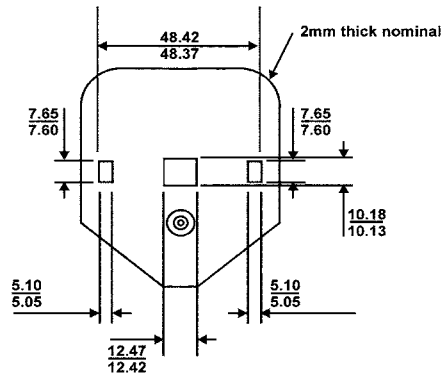
- Key**
- A Flash detector (optional)
  - B Lid
  - C Pilot flame jet
  - D Test flame nozzle
  - E Filling orifice
  - F Test flame gas control screw
  - G Shutter
  - H Test flame gage
  - I Thermometer
  - J Locating notch for flash detector
  - K Lid sealing O-ring
  - L Test cup block
  - M Thermometer well



SHUTTER



TEST CUP AND COVER



LID

Dimensions in mm

NOTE—All dimensions are in millimetres.  
**FIG. A1.1 Test Cup and Cover Assembly**

**TABLE A1.1 Essential Dimensions of Flash Test Apparatus<sup>A,B</sup>**

	mm
<i>Specimen Block:</i>	
Block diameter	61.5–62.5
Specimen well diameter	49.40–49.70
Specimen well depth	9.70–10.00
Top of block to center of thermometer hole	16.00–17.00
Diameter of thermometer hole	7.00 approx
<i>Cover:</i>	
Large opening length	12.42–12.47
Large opening width	10.13–10.18
Small opening length	5.05–5.10
Small opening width	7.60–7.65
Distance between extreme edges of small openings	48.37–48.42
Filling orifice diameter	4.00–4.50
Bore of filler tube	1.80–1.85
Maximum distance of filler tube from base of well with cover closed	0.75 max
<i>Shutter:</i>	
Large opening length	12.42–12.47
Large opening width	10.13–10.18
Small opening length	5.05–5.10
Small opening width	7.60–7.65
Near edge of large opening to end of shutter	12.80–12.85
Extremes of large and small openings	30.40–30.45
<i>Jet:</i>	
Length of jet	18.30–18.40
External diameter of end of jet	2.20–2.60
Bore of jet	1.60–1.65
Height of jet center above top surface of cover	11.00–11.20
Jet pivot to center of block with cover closed	12.68–12.72

<sup>A</sup> The O-ring seal or gasket, which provides a seal when the cover is shut, must be made of a heat-resistant material capable of withstanding the test temperatures and the materials being tested.

<sup>B</sup> When in position the temperature measuring device must be in good thermal contact with the test cup block.

propane. (**Warning**—Never recharge or change the self-contained gas tank at elevated temperatures, or with the pilot or test flames lighted, nor in the vicinity of other flames.) A gauge ring 4 mm ( $5/32$  in.) in diameter, engraved on the cover near the

test flame, aids uniformity in the size of the test flame. An electric ignitor together with a suitable dipping mechanism and cover (A1.10) is also permitted, however the precision and degree of agreement with the gas flame ignitor have not yet been determined.<sup>6</sup> The electric ignitor shall be of the electric resistance (hot wire) type and shall position the heated section horizontally and intersect the underside of the cover. Follow the manufacturer's instructions for ensuring the correct operation of the ignitor. In the event of a dispute the gas ignition source is the referee.

A1.4 *Audible Signal*, indicates for the operator to dip the ignition source into the cup.

A1.5 *Syringe*, equipped with a nozzle suitable for use with the apparatus, adjusted to deliver  $2.00 \pm 0.05$  mL. It is permissible to use this syringe twice to enable a 4 mL specimen to be used.

A1.6 *Syringe*, equipped with a nozzle suitable for use with the apparatus, adjusted to deliver  $4.0 \pm 0.1$  mL.

A1.7 *Flash Detector (optional)*, a low mass thermocouple device for the detection of the flash point. A flash is indicated if a temperature rise of 6.0°C (10.0°F) is detected within 100 ms.

A1.8 *Timing Device*—An electronic timer.

A1.9 *Temperature Measuring Device*—A liquid in glass thermometer or an electronic temperature measuring device with an accuracy of better than 0.5°C (1°F) up to 100.0°C, and 2.0°C (4°F) at higher temperatures respectively.

A1.10 *Electric Ignitor Screen*—A metal screen to optically screen the ignitor from the operator. Only required when an electric ignitor is used.

## A2. VERIFICATION OF APPARATUS PERFORMANCE

A2.1 *Certified Reference Material (CRM)*—CRM is a stable, pure (99+ mole % purity) hydrocarbon or other stable petroleum product with a method-specific flash point established by a method-specific interlaboratory study following ASTM research report guidelines<sup>8</sup> or ISO Guides 34 and 35.

A2.1.1 Typical values of the flash point corrected for barometric pressure for some reference materials and their typical limits are given in Table A2.1 (see Note A2.2). Suppliers of CRMs will provide certificates stating the method-specific flash point for each material of the current production batch. Calculation of the limits for these other CRMs can be determined from the reproducibility value of this test method, reduced by interlaboratory effect and then multiplied by 0.7. (See research report value.<sup>5</sup>)

<sup>8</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1007.

**TABLE A2.1 D3828 Typical Flash Point Values and Typical Limits for CRM**

Hydrocarbon	Purity (mole %)	Flash Point (°C)	Limits (°C)
<i>n</i> -decane	99+	49.7	$\pm 1.6$
<i>n</i> -undecane	99+	65.9	$\pm 1.9$

NOTE A2.1—Supporting data for the interlaboratory study to generate the flash point in Table A2.1 can be found in the research report.<sup>9</sup>

NOTE A2.2—Materials, purities, flash point values and limits stated in Table A2.1 were developed in an ASTM interlaboratory program to determine suitability of use for verification fluids in flash point test methods. Other materials, purities, flash point values, and limits can be suitable when produced according to the practices of ASTM D02-1007<sup>8</sup> or ISO Guides 34 and 35. Certificates of performance of such materials

<sup>9</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: S15-1010.

should be consulted before use, as the flash point value will vary dependent on the composition of each CRM batch.

A2.2 *Secondary Working Standard (SWS)*—SWS is a stable, pure (99+ mole % purity) hydrocarbon, or other petroleum product whose composition is known to remain appreciably stable.

A2.2.1 Establish the mean flash point and the statistical control limits ( $3\sigma$ ) for the SWS using standard statistical techniques (see Practice [D6299](#)).

### A3. PRECISION FOR FATTY ACID METHYL ESTERS (FAME)

#### A3.1 Background

A3.1.1 A pan European round robin was conducted using test method [EN ISO 3679](#) in 2001 following a robustness trial. The round robin involved ten laboratories that tested ten blind duplicate samples (twenty containers for each participant). Two FAME types were used: rapeseed and sunflower seed. Each sample type was contaminated with 0, 0.05, 0.1, 0.2, and 0.5 % methanol.

#### A3.2 Equivalence

A3.2.1 [EN ISO 3679](#) (IP 523) and the associated flash no-flash method [EN ISO 3680](#) (IP 524) are technically equivalent to Test Methods D3828 and use the same procedure, apparatus, and precision.

#### A3.3 Precision and Bias<sup>10</sup>

A3.3.1 *Precision*—The precision of this test method, as determined by statistical examination of interlaboratory results is as follows:

A3.3.1.1 *Repeatability*—The difference between the two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of this test method, exceed the values shown in [Table 2](#) only in one case in twenty.

Repeatability = 1.9°C

A3.3.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, would, in the long run, in the normal and correct operation of this test method, exceed the values shown in [Table 2](#) only in one case in twenty.

Reproducibility = 15°C

A3.3.2 *Bias*—The procedure in this test method has no bias, because flash point can be defined only in terms of a test method.

<sup>10</sup> Supporting data available from CEN as CEN/TR 15160. European Committee for Standardization (CEN), 36 Rue de Stassart, B1050, Brussels, Belgium.

### A4. SUB-AMBIENT TESTING FOR MANUAL APPARATUS WITHOUT INTEGRAL TEST CUP COOLING FACILITIES

A4.1 *Apparatus Using a Liquid in Glass Thermometer*—Switch off the power to the test cup heater.

A4.1.1 If the manual apparatus uses an electronic thermometer follow the apparatus manufacturer's instructions for setting the required sub-ambient test temperature.

A4.2 Fill the refrigerant-charged cooling block with a suitable material. 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 cover, and position the base of the block in the sample cup, being careful not to damage or mar the test cup. When the thermometer reads approximately 10°C (18°F) below the target

temperature, remove the cooling block and quickly dry the test cup and underside of the cover with a paper tissue to remove any moisture. Immediately close the cover and secure. (**Warning**—If a mercury in glass thermometer is being used, do not cool the sample block below -38°C (-36°F), the freezing point of mercury.) Prepare to introduce the test specimen into the test cup using the syringe, both of which have been precooled to a temperature at least 10°C (18°F) below the required test temperature.

A4.3 Adjust the test flame and allow the test cup temperature to rise under ambient conditions until the required test temperature is reached. Immediately apply the test flame. Watch closely for a flash at the cover openings.

A4.4 To determine the flash point of any materials carry out repeat tests, with a new specimen each time, in accordance with the steps given in [12.1](#).



**SUMMARY OF CHANGES**

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D3828–07a) that may impact the use of this standard.

- |   |  |
|---|--|
| (1) General editorial changes.  | (4) Updating of the procedure.                                     |
| (2) Changes to clarify the sample size and test time throughout the method. | (5) Allowing the use of non-mercury temperature measuring devices. |
| (3) Changes to cover the introduction of electric ignition sources.         | (6) Updated <b>Fig. A1.1</b> .                                     |

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