



Designation: D3828-07a



Designations: 523/05 and 524/05 Designation: D 3828 - 09

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

This standard is issued under the fixed designation D 3828; 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

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 D3941.

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 D 3941 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 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).

1.1 These test methods cover procedures for flash point tests, within the range of  $-30$  to  $300^{\circ}\text{C}$ , 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 *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.* 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.

<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.

Current edition approved July 15, 2007. Published August 2007. Originally approved in 1979. Last previous edition approved in 2007 as D3828-07.

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.

Current edition approved Sept. 1, 2009. Published September 2009. Originally approved in 1979. Last previous edition approved in 2007 as D 3828-07a.

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

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>

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

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

E 300 Practice for Sampling Industrial Chemicals

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

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 3679 Determination of Flash Point—Rapid Equilibrium Closed Cup Method

~~EN ISO 3680 Determination of flashpoint with closed cup equilibrium method~~ 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—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. ~~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.~~ 8-09

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.

—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—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. ~~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 of a sample 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 a test flame an ignition source is applied and an observational determination made as to whether or not a flash occurred.

<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>.

<sup>4</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1214.

<sup>4</sup> Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., <http://www.energyinst.org.uk>.

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

4.2.1A 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.

4.2.2The 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). —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 of the sample to heat and flame-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. 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).

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 from –20 to 300°C may require more than one instrument. 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. Hazards

7.1The 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.

7.2When 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). *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. Sample

8.1Erroneously 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. Sampling~~

~~8.1 Obtain at least a 50 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.~~

~~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 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).~~

~~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. 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 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. Procedure of this test method and the observed flash point obtained in 12.6 shall be corrected for barometric pressure (see Section 13) 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 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, 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 Determine the target flash point as follows:

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).

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

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

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

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

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

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

$$- 0.06760 - B \quad \text{ards.iteh.ai/catalog/standards/sist/2380262e-b225-4453-93fd-7eaf65fe3b0a/astm-d3828-09}$$

where:

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

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

$B$  = 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

11.2 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.

11.6 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

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 2½ 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):

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

**TABLE 1 Test Calondibration of Tester**

| MSateriample  | Test Temperatures   | Specimen Volume                       | Test Time |
|---|---|---------------------------------------|-----------|
| p-xyl ene <sup>A,B</sup> (1,4-Dimethyl benzene) ( <b>Warning</b> ) <sup>C</sup> | n-butanol <sup>A</sup> (Butan-1-ol) ( <b>Warning</b> ) <sup>C</sup> |                                       |           |
| All except biodiesel  | ≤ 1-ol) ( <b>Warning</b> ) <sup>C</sup>                             |                                       |           |
| Specific gravity, 15.6/15.6°C (60/60°F)   | 0.860 to 0.866  | 0.810 to 0.812                        |           |
| Specific gravity, 15.6/15.600°C (212°F)   | 2 mL  | 1 min                                 |           |
| Boiling range, °C (°F)  | 2 (4) maximum including 138.35 (281.03)                             | 2 (4) maximum including 117.5 (243.5) |           |
| Boiling range, °C (°F)  | All except biodiesel  | > 117.5 (243.5)                       |           |
| Freezing point, °C (°F)   | 52.2 (11.23) minimum  | -90 (-130) minimum                    |           |
| Freezing point, 00°C (212°F)  | 4 mL  | 2 min                                 |           |
| Flash point, °C (°F) (acceptable range)   | 25.6 ± 0.5 (78 ± 1)   | 36.6 ± 0.8 (97.9 ± 2 mL               | 1.7)      |
| Biodiesel   | ≤ 300°C (572°F)   | 2 mL                                  | 1 min     |

<sup>A</sup>Available as Flash Point Check Fluid from Special Products Div., Phillips Petroleum Co., Drawer O, Bergen, TX 79007.

<sup>B</sup>Contains less than 500 v ppm of C<sub>6</sub> and lighter hydrocarbons by gas chromatography.

<sup>C</sup>(**Warning**—Handle xylene and n-butanol with care. Avoid inhalation.)