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Standard Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus¹

This standard is issued under the fixed designation D3278; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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

INTRODUCTION

These small scale flash point methods test a sample at a specific temperature, where the specimen being tested and the air-vapor mixture above it are close 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 from that specified.

1. Scope

1.1 These test methods cover procedures for determining whether a material does or does not flash at a specified temperature (flash/no flash Method A) or for determining the lowest finite temperature at which a material does flash (Method B), when using a small scale closed-cup apparatus. The test methods are applicable to paints, enamels, lacquers, varnishes, solvents, and related products having a flash point between $00\text{ }^{\circ}\text{C}$ and $110\text{ }^{\circ}\text{C}$ ($32\text{ }^{\circ}\text{F}$ and $230\text{ }^{\circ}\text{F}$) and viscosity lower than 150 ^{cSt} at $25\text{ }^{\circ}\text{C}$ ($77\text{ }^{\circ}\text{F}$).

NOTE 1—Tests at higher or lower temperatures are possible however the precision has not been determined.

NOTE 2—More viscous materials can be tested in accordance with Annex A4.

NOTE 3—Organic peroxides can be tested in accordance with Annex A5, which describes the applicable safety precautions.

NOTE 4—The U.S. Department of Labor (OSHA, Hazard Communications), the U.S. Department of Transportation (RSPA), and the U.S. Environmental Protection Agency (EPA) have specified Test Methods D3278 as one of several acceptable methods for the determination of flash point of liquids in their regulations.

NOTE 2—More viscous materials may be tested in accordance with Annex A4.

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¹ These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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Note 5—These test methods are similar to International Standards ISO 3679 and ISO 3680.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only after SI units are provided for information only and are not considered standard.

1.3 *This standard should be used to measure and describe the response of materials, products, or assemblies to heat and flame under controlled 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 the test may be used as elements of a fire hazard or a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard or fire risk of a particular end use.* This standard is used to measure and describe the response of materials, products, or assemblies to heat and flame under controlled conditions, but does not by itself incorporate all factors required for fire hazard or fire risk assessment of the materials, products, or assemblies under actual fire conditions.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see 8.1 and H.2.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

- [D56 Test Method for Flash Point by Tag Closed Cup Tester](#)
- [D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester](#)
- [D850D3828 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials Methods for Flash Point by Small Scale Closed Cup Tester](#)
- [D3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings](#)
- [D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance](#)
- [D1015D7236 Test Method for Freezing Points of High-Purity Hydrocarbons Flash Point by Small Scale Closed Cup Tester \(Ramp Method\) \(Withdrawn 2019\)](#)
- [D1078D8174 Test Method for Distillation Range of Volatile Organic Liquids Finite Flash Point Determination of Liquid Wastes by Small-Scale Closed Cup Tester](#)
- [E1137/E1137M Specification for Industrial Platinum Resistance Thermometers](#)

2.2 ISO Standards:³

- [ISO 1513 Paints and varnishes — Examination and preparation of test samples for testing](#)
- [ISO 3679 Paints, varnishes, petroleum and related products—Determination of flash point—Rapid equilibrium—Determination of flash no-flash and flash point—Rapid equilibrium closed cup method](#)
- [ISO 368015528 Paints, varnishes, petroleum and related products—Flash/no flash test—Rapid equilibrium method varnishes and raw materials for paints and varnishes — Sampling](#)
- [ISO 17034 General requirements for the competence of reference material producers](#)
- [ISO 17043 Conformity assessment – General requirements for proficiency testing](#)
- [ISO 60751 Industrial platinum resistance thermometers and platinum temperature sensors](#)
- [Guide 35 Certification of Reference Materials—General and Statistical Principles](#)

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.

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, <http://www.ansi.org>.

3.1.2 *flash point, n—in flash point test methods*, the lowest temperature, corrected to a pressure of 760 mm Hg (101.3 kPa, 1013 mbar), temperature of the test specimen, adjusted to account for variations in atmospheric pressure from 101.3 kPa, at which application of an ignition source causes the vapors of the test specimen to ignite under specified conditions of test.

4. Summary of Test Methods

4.1 *Method A—Flash/No Flash Test*—By means of a syringe, 2 mL of the sample is introduced through a leakproof entry. A 2 mL test specimen is introduced, by a syringe (6.5 port), into the tightly closed small seal closed-cup test cup of the selected apparatus or directly into the cup that has been brought to the required test temperature. As a flash/no flash test, the expected flash point temperature may be a specification or other operating requirement. After 1 min, a test flame is applied inside the cup and note is taken that is set and maintained at the specified test temperature. After one minute, an ignition source is applied and a determination made as to whether or not the test specimen flashes. A fresh specimen must be used if a repeat test is necessary: a flash occurred.

4.2 *Method B—Finite (or Actual) Flash Point*—For a finite flash point measurement, the temperature is sequentially increased through the anticipated range, the test flame being applied at 5°C (9°F) intervals until a flash is observed. A true determination is then made using a fresh specimen, starting the test at the temperature of the last interval before the flash point of the material and making tests at increasing 0.5°C (1°F) intervals. 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 iteratively.

4.2.1 A 2 mL test specimen is introduced into the test cup of the selected apparatus that is maintained at the expected flash point. After one minute, 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 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.

4.2.3 The procedure is then repeated at 0.5 °C intervals until the flash point is determined to the nearest 0.5 °C.

4.2.4 A second determination is made in accordance with 4.2.3 and the average of the two determinations recorded as the flash point.

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 by governmental regulatory agencies to define flammable and combustible materials and to classify them. Consult the particular regulation involved for precise definitions of these classes.

5.3 Flash point is one of the properties used to define the flammability of a liquid. It is used to classify liquids according to their flammability by governmental regulatory agencies. It may also be used to determine the presence of can indicate the possible presence of highly volatile and flammable impurities or contaminants in a given liquid, such as the presence of residual solvents in solvent-refined drying oils. See Test Methods D56 and D93 for alternate flash point testing.

5.4 These equilibrium flash point test methods use a smaller specimen (2 mL) and a shorter test time (1 min) than traditional non-equilibrium test methods such as Test Method D56 and Test Methods D93.

5.5 Test Methods D3828, Test Method D8174, and ISO 3679 are similar test methods and use the same apparatus.

6. Apparatus

6.1 *Test Cup and Cover Assembly* Assembly: =

6.1.1 The essential dimensions and requirements of the apparatus are shown in Fig. A1.1 and described in Annex A1.

6.1.2 The temperature range is from 0 °C to 110 °C.

6.1.2.1 Versions of the apparatus can cover a wider temperature range.

6.1.3 Some apparatuses can automate temperature control down to sub-ambient temperatures, timing and dipping, including hot wire ignition, automatically detecting a valid flash point and measuring barometric pressure.

~~6.2 *Thermometers; Barometer*, low, medium, high temperature small scale. Test to determine that the scale error does not exceed 0.25°C (0.5°F). The use of a magnifying lens assists in making temperature observations 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.~~

~~6.4 *Syringe (optional)*—General purpose for use with very viscous samples, to dispense 2 mL to 4 mL specimens (see [Annex A4](#)).~~

~~6.5 *Glass Syringe*, 22 mL ± 0.1 mL 0.1 mL capacity at 25°C (77°F), 25 °C (77 °F), to provide a means of taking a uniform specimen. Check the capacity by discharging water into a weighing bottle and weighing. Adjust plunger if necessary. A disposable syringe of equal precision may be used.~~

~~6.4 *Aluminum Cooling Block*⁸(describing in [Annex A2](#)), that fits snugly within the test cup used for rapid cooling of the sample cup.~~

~~6.5 *Barometer*.~~

7. Reagents and Materials

~~7.1 *p-Xylene, Cleaning Solvent*—reference standard. Use only non-corrosive solvents capable of cleaning the test cup and lid assembly. 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 *n-Butanol*,¹⁰ reference standard.~~

~~7.2 *Cooling Mixture Butane, Propane, and Natural Gas*—of ice and water or solid Optional for use as a pilot and ignition source. (**Warning**—~~C~~Butane, propane,⁷ (dry ice) and acetone and natural gases are flammable and a health hazard.)~~

~~7.3 *Liquefied Petroleum Gas. Verification Fluids*—See [Annex A2](#).~~

~~7.5 *Heat Transfer Paste*.¹¹~~

8. Sampling

8.1 Obtain at least a 25 mL sample from a bulk test site in accordance with Practice [D3925](#), ISO 1513, or ISO 15528, or a comparable sampling practice.

8.2 Store samples in clean, tightly sealed containers, suitable for the type of liquid being sampled, at normal room temperature (for example 20 °C to 25 °C (68 °F to 77 °F)) 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 The specimen size for each test is 2 mL. Obtain at least a 25-mL sample from the bulk source and store in a nearly full, tightly closed, clean glass container or in other container suitable for the type of liquid being sampled. (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. ~~Warning—Erroneously high flash points may be obtained if precautions are not taken to avoid loss of volatile material. Do not open sample containers unnecessarily and do not transfer the specimen to the cup unless the temperature of the specimen is at least 10°C (20°F) below the expected flash point. Discard samples in leaky containers.) (on~~ **Warning—Do not store samples in plastic bottles (polyethylene, polypropylene, etc.), as volatile material may diffuse through the walls.)** the sample.

8.5 Decant any free water in the sample container.

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.

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.

NOTE 5—Results of flash point tests can be affected if the sample volume falls below 50 % of the container's capacity.

8.8 If the sample is highly viscous and not sufficiently fluid to be drawn into the 2 mL syringe and be introduced into the test cup through the filler orifice, follow the procedure given in [Annex A4](#).

9. Preparation of Apparatus

9.1 ~~Prior to initial use or after removal of the thermometer, insert the thermometer into its pocket (see~~ Place the apparatus on a level, stable surface. Unless tests are made in a draft-free area, surround the tester on three sides [Fig. A1.1](#)) with a good heat transfer draft shield (see [6.3](#) ~~paste~~) 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 To help in making the necessary settings during a test, before the initial use determine the relationship between the temperature control dial and thermometer readings at intervals not over 5°C (9°F) throughout the scale range of the heater.

9.3 Place the tester in a subdued light and in a position not exposed to disturbing drafts. Provide a black-coated shield, if necessary.

9.2 Read the manufacturer's operating and maintenance instructions on the care and servicing of the tester. ~~Observe the specific suggestions regarding the operation of the various controls.~~ 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. See [Appendix X1](#) for detailed cleaning instructions.

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

9.6 An optional electronic thermal flash detector may be used for all product types. It is essential where the ignition of the vapors of the test specimen is not visible or when the apparatus is automated.

9.7 Check the accuracy of the tester by duplicate determination of the flash point of the ~~For sub-ambient test temperatures see~~ p-xylene reference standard ([Annex A3](#)). The mean of the results should be $27.2 \pm 0.8^{\circ}\text{C}$ ($81 \pm 1.5^{\circ}\text{F}$). An additional reference standard, ~~, unless the apparatus has integral test~~ n-butanol ([Annex A3](#)), may be used to check the accuracy at approximately the temperature specified by U.S. regulatory agencies in defining flammable liquids. The mean of duplicates should be $36.7 \pm 0.8^{\circ}\text{C}$ ($98 \pm 1.5^{\circ}\text{F}$). If not, remove the thermometer and observe whether sufficient heat transfer paste surrounds the thermometer to provide good heat transfer from the cup to the thermometer. cup cooling facilities.

10. Verification of Apparatus

10.1 Verify that the temperature measuring device is in accordance with [A1.2.2](#) and [Annex A6](#).

10.2 Verify the performance of the apparatus at least once per year by determining the flash point of a certified reference material (CRM): suitable materials are listed in [Annex A2](#). Use a CRM that 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 detected flash point obtained in [12.1.11](#) and [12.1.12](#) shall be corrected for barometric pressure (see Section [13](#)). The mean of the two determined flash points shall be within the limits stated in [Table A2.1](#) for the identified CRM or within the limits calculated for an unlisted CRM (see [A2.1.1](#)).

10.3 Once the performance of the apparatus has been verified, the flash point of secondary working standards (SWS) 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.

TEST METHOD A—FLASH/NO FLASH

10. Procedure—Ambient to 110°C (230°F)

10.1 Inspect the inside of the test cup, lid, and shutter mechanism for cleanliness and freedom from contamination. Use an absorbent tissue to wipe clean, if necessary. Lock the cover lid tightly in place.

10.2 Switch the heater on, if not already at stand-by. To rapidly approach the specification flash temperature of the material under test, turn the heater dial fully clockwise ([Note 6](#)) causing the heater signal (red) light to glow. When the thermometer indicates a temperature of about 3°C (5°F) below the specification or target flash-point temperature, reduce the heat input to the test cup by slowly turning the heater control dial counter clockwise until the signal light goes out ([Note 7](#)).

[NOTE 6](#)—When a desired temperature is dialed on the controller, the elapsed time to reach this temperature may be greater than if the controller is turned “full on,” but less attention is required.

[NOTE 7](#)—The test cup temperature is stable when the signal light slowly cycles on and off.

10.3 Determine the barometric pressure to determine the corrected specification temperature at that barometric pressure (see [15.1](#)).

10.4 After the test-cup temperature has stabilized at the specification or target flash point, charge the syringe with the sample being tested and insert the tip of the syringe into the filling orifice ([Fig. A1.1](#)), taking care not to lose any material. Discharge the specimen into the test cup by completely depressing the syringe plunger, then remove the syringe. If the material has a viscosity greater than 45 SUS at 38°C (100°F) or equivalent of 9.5 cSt at 25°C (77°F), raise the lid and discharge the contents of the syringe directly into the cup. Immediately close the lid tightly.

10.5 Set the 1-min timing device. In the meantime, open the gas control valve and light the pilot and test flames. Adjust the test flame size with the pinch valve to match the size of the $\frac{5}{32}$ -in. (4-mm) diameter flame gage.

10.6 After 1 min has elapsed, observe the temperature. If at the specification temperature (accounting for the differences of the barometer reading from 760 mm), apply the test flame by slowly and uniformly opening the slide fully and closing completely over a period of approximately 2½ s ([Note 8](#)), watching for a flash ([Note 9](#)) while the flame is inserted.

[NOTE 8](#)—When inserted, the nozzle of the ignition device should intersect the plane of the underside of the cover (see [A1.1](#)).

[NOTE 9](#)—The material is considered to have flashed only if a comparatively large blue flame appears and propagates itself over the surface of the liquid. Occasionally, particularly near the actual flash-point temperature, application of the test flame may give rise to a halo; this effect should be ignored.

10.7 Turn off the test and pilot flame. Clean the apparatus in preparation for the next test.

11. Procedure—0°C (32°F) to Ambient

11.1 If the specification or target flash point is at or below ambient temperature, cool the sample to 5 to 10°C (10 to 20°F) below that point by some convenient means.

11.2 Cool the tester to approximately the temperature of the sample by inserting the cooling block (Fig. A2.1) filled with a cooling mixture into the sample well. Dry the cup with a paper tissue to remove any collected moisture prior to adding the specimen using a precooled syringe. (**Warning**—Be careful in handling the cooling mixture and cooling block; wear gloves and goggles. Mixtures such as dry ice and acetone can produce severe frost bite.) (**Warning**—Be careful when inserting the cooling block into the tester cup to prevent damage to the cup.)

11.3 Introduce the specimen as in 10.4. Allow the temperature to rise under ambient conditions or increase the temperature of the cup by rotating the heater controller clockwise slowly until the specification temperature adjusted for barometric pressure is reached. Determine whether the material flashes as in 10.5 and 10.6.

11.4 Turn off the test and pilot flames. Clean the apparatus.

TEST METHOD B—FINITE FLASH POINT

11. Procedure—Ambient to 110°C (230°F) Procedure

11.1 *Preliminary or Trial Test:* 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.

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

$$\text{Actual test temperature (°C)} = S_c - 0.033(760 - B) \quad (2)$$

where: <https://standards.iteh.ai/catalog/standards/sist/275511bd-fbe4-41df-8b45-3a902a9731aa/astm-d3278-21>

S_c ≡ Specification, or uncorrected target test temperature, °C,

A ≡ Ambient barometric pressure, kPa, and

B ≡ Ambient barometric pressure, mm Hg.

Warning—Eq 1 and Eq 2 are only to be used when testing at a prescribed specification or target temperature is required.

11.1.1 Follow steps 10.1–10.5, omitting the barometric reading and using an estimated finite flash point instead of specification flash-point temperature; the manufacturer's instructions to set the test temperature and the test time.

11.1.2 When the test cup is at the test temperature, fill the syringe (6.5) with the sample to be tested; transfer the syringe to the filling orifice, taking care not to lose any sample; discharge the 2 mL test specimen into the test cup by fully depressing the syringe plunger; remove the syringe.

11.1.2.1 If the sample is too viscous to be syringed through the orifice, see Annex A4.

11.1.3 Start the 60 s test timer; light the pilot light and adjust the test flame (if used) to conform to the 4 mm (5/32 in.) gauge.

11.1.4 After 1 min has elapsed, observe the temperature. Apply the test flame. When the end of the test time is indicated, apply the ignition source by slowly and uniformly opening the slide fully shutter and closing it completely over a period of 22 s½ s (to 3 s. Note 8), watching Watch closely for a flash (at Note 9) while the flame is inserted; the test cup openings.

11.1.5 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.

11.1.6 Record the test result as flash (or no flash), allowing for any known thermometer correction and the test temperature.

11.1.7 Turn off the pilot and test flames (if used). Remove the test specimen and clean the test cup and cover (see [Appendix X1](#)). It may be necessary to allow the test cup temperature to fall to a safe level before cleaning.

12.2 If a flash is observed, proceed as in 12.3. If no flash is observed, proceed as in 12.4.

12.3 Using a temperature 5°C (9°F) lower than the temperature observed in 12.1.2, repeat 12.1 (Note 4). If a flash is still observed, repeat at 5°C lower intervals until no flash is observed.

Note 10—Never make a repeat test on the same specimen. Always take a fresh portion for each test.

12.3.1 After establishing the approximate flash point, repeat 12.1 with a new specimen, but stabilizing the test temperature at which no flash occurred previously. Observe if a flash occurs at this temperature. If not, increase the temperature by making a small adjustment to the temperature controller so that an increase of 0.5°C (1°F) occurs within 1 min. Test for a flash at each 0.5°C interval, recording the temperature at which the flash actually occurs. Record the barometric pressure. Clean the tester.

12.3.1.1 Repeat 12.3.1 with a new specimen. Calculate the corrected mean temperature in accordance with 15.2. Turn off pilot and test flames and clean the tester.

12.4 Using a test temperature 5°C (9°F) higher than the temperature observed in 12.2, repeat 12.1 (Note 10). If no flash is observed, repeat at 5°C higher intervals until a flash is observed.

12.4.1 After establishing the approximate flash point (12.4) with new specimens, make two determinations of the flash point in accordance with 12.3.1 and calculate the corrected mean temperature in accordance with 15.2.

13. Procedure—0°C (32°F) to Ambient Temperature

13.1 *Preliminary or Trial Test:*

13.1.1 Cool the sample to 3 to 5°C (5 to 10°F) below the expected flash point.

13.1.2 Cool the tester to approximately the temperature of the sample by inserting the cooling block filled with a cooling medium into the sample well. (**Warning**—See 11.2.)

13.1.3 Introduce the specimen using a precooled syringe as in 10.4. Set the 1-min timing device. After 1 min observe the temperature, then apply the test flame by slowly and uniformly opening the slide fully and closing completely over a period of 2½ s, watching for a flash (Note 9) while the flame is inserted. Record the temperature.

13.2 If a flash is observed, proceed as in 13.3. If no flash is observed, proceed as in 13.4.

13.3 Take a new specimen and recool the sample cup to 5°C (9°F) below the previous temperature (13.1.3). After 1 min, check for a flash as in 13.1.3. If the material flashes, repeat at 5°C lower intervals until no flash is observed.

13.3.1 After establishing the approximate flash point, repeat 13.1.1 and 13.1.3 with a new specimen but stabilizing the test temperature at which no flash occurred previously. Observe if a flash occurs at this temperature. If not, increase the temperature by making a small adjustment to the temperature controller so that an increase of 0.5°C (1°F) occurs within 1 min. Test for a flash at each 0.5°C interval, recording the temperature at which the flash actually occurs. Record the barometric pressure.

13.3.1.1 Repeat 13.3.1 with a new specimen. Calculate the corrected mean in accordance with 15.2. Turn off pilot and test flames and clean the tester.

13.4 Using a test temperature 5°C (9°F) higher than the temperature observed in 13.1.3, repeat 13.1.3 (Note 10). If no flash is observed, repeat at 5°C higher intervals until a flash is observed.

13.4.1 After establishing the approximate flash point (13.4), make the determinations of the flash point in accordance with 13.3 and calculate the corrected mean in accordance with 15.2.

TEST METHOD B—FINITE FLASH POINT DETERMINATION

14. Clean-up of Apparatus and Preparation for Next Test

14.1 Unlock the lid assembly of the tester and raise to the hinge stop. Soak up liquid with an absorbent paper tissue and wipe dry. Clean the underside of the lid and filling orifice. A pipe cleaner may be of assistance in cleaning the orifice.

14.2 If the material is a viscous liquid or contains dispersed solids, after soaking up most of the specimen add a small amount of a solvent suitable for the sample to the cup. Then soak up the solvent and wipe clean the interior surfaces of the cup with an absorbent tissue paper.

Note 11—If necessary to remove residual high-boiling solvent residues, moisten tissue with acetone and wipe dry.

Note 12—If any further cleaning is necessary, remove the lid and shutter assembly. Disconnect the silicone rubber hose and slide the lid assembly to the right to remove. If warm, handle carefully.

14.3 After the cup has been cleaned, its temperature may be rapidly increased to some stand-by value by turning the temperature control dial to an appropriate point.

Note 13—It is convenient to hold the test cup at some stand-by temperature (depending on planned usage) to conserve time in bringing the cup within the test temperature range. The cup temperature may be quickly lowered by inserting the aluminum cooling block filled with an appropriate cooling mixture into the cup.

14.4 The syringe is easily cleaned by filling it several times with acetone or other compatible solvent, discharging the solvent each time, and allowing the syringe to air dry with the plunger removed. Replace the plunger, and pump several times to displace any solvent vapor with air.

12. Procedure

12.1 This procedure repeats the procedure of Method A a number of times. Each test uses a fresh test specimen and a different temperature. The changes of temperature allow the determination of two temperatures 5 °C apart and then two temperatures 0.5 °C apart for which the final 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.

12.1.1.1 If the expected flash point of the sample is unknown, Appendix X2 may be used to obtain an approximate flash point.

12.1.2 Follow the manufacturer's instructions to set the test temperature and the one minute test time.

12.1.3 When the test cup is at the test temperature, fill the syringe (6.5) with the sample to be tested; transfer the syringe to the filling orifice, taking care not to lose any sample; discharge the 2 mL test specimen into the test cup by fully depressing the syringe plunger; remove the syringe.

12.1.3.1 If the sample is too viscous to be syringed through the orifice, see Annex A4.

12.1.4 Start the 60 s test timer; light the pilot light and adjust the test flame (if used) to conform to the 4 mm (5/32 in.) gauge.

12.1.5 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 s to 3 s. Watch closely for a flash at the test cup openings.

12.1.6 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.

12.1.7 Note the test result as flash (or no flash) and the test temperature.

12.1.8 If a flash is detected, repeat the procedure given in 12.1.3 to 12.1.7 testing a fresh specimen at a temperature 5 °C lower each time until no flash is detected.

12.1.8.1 Proceed to 12.1.10.

12.1.9 When no flash was detected, repeat the procedure given in 12.1.3 to 12.1.7 testing a fresh specimen at a temperature 5 °C higher each time until a flash is detected.

12.1.10 Having established a flash within two temperatures 5 °C apart, repeat the procedure testing a fresh specimen at 0.5 °C intervals from the lower of the two temperatures until a flash is detected.

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

12.1.12 Repeat 12.1.10 and 12.1.11 to obtain and record a second detected flash point for the sample.

12.1.13 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 (see Appendix XI).

15. Correction for Barometric Pressure

15.1 Determine the corrected specification flash point to be used in Test Method A by the following equations:

$$F = S - 0.06(760 - P)$$

$$C = T - 0.03(760 - P)$$

$$F = S - 0.42(101.3 - B)$$

$$C = T - 0.23(101.3 - B)$$

where:

$F(C)$ = flash point to be observed to obtain the specification flash point at standard pressure, °C (°F),

$S(T)$ = specification flash point, °C (°F), and

$P(B)$ = ambient barometric pressure, mm Hg (kPa).

NOTE 14—The barometric pressure used in this calculation must 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 must not be used.

15.2 When the barometric pressure in Test Method B differs from 760 mm Hg, correct the flash point temperature, A , by means of the following equations:

$$A = F + 0.06(760 - P)$$

$$= C + 0.03(760 - P)$$

$$= F + 0.42(101.3 - B)$$

$$= C + 0.23(101.3 - B)$$

where:

$F(C)$ = observed flash point, °C (°F), and

$P(B)$ = ambient barometric pressure, mm Hg (kPa).

13. Calculations

13.1 Method A—No further calculations are required if the effects of barometric pressure were corrected for in Eq 1 or Eq 2 in 11.1.

13.1.1 If the effects of barometric pressure were not corrected before the flash/no flash test, Eq 3 or Eq 4 shall be used to calculate the corrected test temperature.

$$\text{Corrected test temperature} (^{\circ}\text{C}) = C + 0.25(101.3 - A) \quad (3)$$

$$\text{Corrected test temperature} (^{\circ}\text{C}) = C + 0.033(760 - B) \quad (4)$$

where:

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

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 temperatures of the two recorded detected flash points as follows:

$$\text{Corrected temperature} (^{\circ}\text{C}) = C + 0.25(101.3 - A) \quad (5)$$

$$\text{Corrected temperature} (^{\circ}\text{C}) = C + 0.033(760 - B) \quad (6)$$

where:

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

A = Ambient barometric pressure, kPa, and

B = Ambient barometric pressure, mm Hg.

NOTE 6—The above formula has been proven for barometric pressures down to 82.0 kPa⁴ and is strictly correct only up to 104.7 kPa.

14. Report

14.1 Method A (flash/no flash), When using the flash/no flash method, report whether or not the sample flashed at the required flash point and that Test report flash or no flash, and the specification temperature used, rounded to the nearest 0.5 $^{\circ}\text{C}$, and that Method A was used.

14.2 Method B (Flash point determination), If an actual flash point was determined, report the mean of duplicate determinations the two corrected flash points to the nearest 0.5 $^{\circ}\text{C}$ (1 $^{\circ}\text{F}$), 0.5 $^{\circ}\text{C}$ as the flash point, provided the difference between the values does not exceed 1 $^{\circ}\text{C}$ (2 $^{\circ}\text{F}$) and that Test Method B was used. is not >1 $^{\circ}\text{C}$.

14.2.1 If the difference is greater, then report this deviation and use the lowest value of the duplicate results or repeat the determinations.

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 Bias⁵

15.1 Precision—No statement is made about either the precision or bias. The precision of Test Method A-Flash/No-Flash, since the result merely states whether there is conformance to the criteria for success specified in the procedure. B was developed from an ILS in 1972. Five laboratories made two determinations on two different days on four solvents, three resins, and two paints, covering the flash point range of 6 $^{\circ}\text{C}$ (43 $^{\circ}\text{F}$) to 84 $^{\circ}\text{C}$ (183 $^{\circ}\text{F}$). A gas flame was used as the ignition source.

15.1.1 No statement is made about either the precision or bias of Test Method A flash/no flash, since the result merely states whether there is conformance to the criteria for success specified in the procedure.

15.1.2 Repeatability—The difference between two independent results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short

³ Closed-cup flash point testers and their accessories meeting the requirements of the unit shown in Fig. A1.1 are available from ERDCO Engineering Corp., 721 Custer Ave., Evanston, IL 60202 or Stanhope-Seta Ltd., Park Close Englefield Green, Engham, Surrey, TW20 0XD, England.

⁴ Thermometers Supporting data have been filed at the Energy Institute, 61 New Cavendish Street, London W1G 7AR. UK <http://www.energyinst.org> and may be obtained from the suppliers of the Small Scale Tester by requesting Research Report IP 523.

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1000. Contact ASTM Customer Service at service@astm.org. These data are also reported in the *Journal of Paint Technology*, Vol 45, No. 581, p. 44.