Designation: D 3941 – 90 (Reapproved 2001)

Standard Test Method for Flash Point by the Equilibrium Method With a Closed-Cup Apparatus[1]

This standard is issued under the fixed designation D 3941; 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 (ε) 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

Test Methods D 56 and D 93 describe procedures using the Tag Closed Tester and the Pensky-Martens Tester, respectively. Both test methods depend on a definite rate of temperature increase to control the precision of the test method. The rate of heating may not in all cases give the accuracy expected because of low thermal conductivity of certain materials. To reduce this effect, ISO/TC 35, Paints and Varnishes, and ISO/TC 28, Petroleum Products and Lubricants, have issued ISO 1523 in which the heating rate is considerably slower. This test method is similar to ISO 1523, but uses standard ASTM cups, style, and format. Due to the slower heating rate, the time required to make a determination of a flash point is considerably longer than for Test Methods D 56 and D 93 but the accuracy is improved.

1. Scope

1.1 This test method covers the determination of the flash point of liquids in which the specimen and the air/vapor mixture above it are approximately in temperature equilibrium.

1.2 This test method is limited to a temperature range from 32 to 230°F (0 to 110°C).

1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard should be used to measure and describe the properties of material, 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 56 Test Method for Flash Point by Tag Closed Tester[2]

D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester[3]

E 1 Specification for ASTM Thermometers[4]

2.2 *ISO Standard:*

ISO 1523 Paints, varnishes, petroleum, and related products—Determination of flash point—Closed cup equilibrium method[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *flash point, n*—the lowest temperature corrected to a pressure of 760 mm Hg (101.3 kPa) (1013 millibars) at which application of an ignition source causes the vapor of the specimen to ignite under specified conditions of test.

4. Summary of Test Method

4.1 A specimen is heated in a closed cup of standard design in a suitable liquid bath at the rate of 1.0°F (0.5°C) in not less than 1.5 min so that the difference in temperature between the specimen in the cup and bath never exceeds 3.5°F (2.0°C). Flash determinations are made at intervals of not less than 1.5 min.

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.22 on Health and Safety.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

[4] Available from American National Standards Institute, 13th Floor, 11 W. 42nd St., New York, NY 10036.

5. Significance and Use

5.1 This test method, using a slow rate of heating, provides a uniform temperature throughout the specimen. The slow rate of heating is necessary because of the low thermal conductivity of some liquids such as paints, resins, and related products, and also because of the poor heat transfer by convection in high-viscosity products. Since the specimen is being heated at a reduced rate, the longer time interval between each determination is necessary to reestablish after each flash test the saturation concentration of vapor in the air space above the specimen.

NOTE 1—ISO 1523 is used in United Nations Recommendations for Transportation of Dangerous Goods and in the International Civil Aviation Organization (ICAO) regulations and for similar regulations in the International Maritime Dangerous Goods (IMDG) code. Test Method D 3941, which is similar to ISO 1523, is used in the United States Department of Transportation (USDOT) regulations. The ICAO and IMDG codes are used for transshipment of hazardous materials through the United States to other countries.

6. Apparatus

6.1 *Test Cups*, equipped with their lid as specified in Test Methods D 56 (Tag) and D 93 (Pensky-Martens). Remove the test cup assembly (including lid and specimen thermometer) from the Tag and Pensky-Martens apparatus to permit either to be used in a separate water bath.

6.1.1 If a stirrer is fitted to the test cup used, it shall operate during the heating period but must be stopped during the flashing determination. If the stirrer originally fitted to the test cup is removed, the aperture in the cover shall be securely plugged before starting the test.

6.2 *Liquid Bath*—Any suitable liquid bath capable of being adjusted to the required temperature and of adequate heat capacity to meet the requirements of the test. A bath fitted with a stirrer and an adjustable thermostat is convenient (see 4.1).

NOTE 2—The bath should be fitted with a cover, especially when the specified temperature is above 160°F. At about this temperature the inerting effect of the water vapor may prevent or delay the flashing of the liquid under test.

6.3 *Thermometers*—Standard thermometers as specified in Test Methods D 56 and D 93. One thermometer shall be used to measure the temperature of the water bath and one shall be used for measuring the temperature of the specimen in the cup.

NOTE 3—The thermometers for the water bath should be mounted at the correct level of immersion for which they are specified in Specification E 1.

6.4 *Support*, for holding the test cup in the liquid bath so that the cover and upper edge are horizontal and the cup is immersed in direct contact with the liquid in such a position that the level of the specimen in the cup is the same as that of the liquid in the bath.

6.5 *Shield*—A three sided shield, open in front, each side 18 in. (460 mm) wide and 24 in. (610 mm) high, is recommended.

7. Reagents and Materials

7.1 *Water or Mixture of Water and Ethylene Glycol (1 + 1)* may be used as a bath liquid.

8. Preparation of Sample

8.1 Obtain a representative sample of the product under test and keep in an air-tight container.

8.2 Because of the possibility of loss of volatile constituents, the sample should receive only the minimum treatment to ensure uniformity. After removing the specimen, immediately close the sample container tightly to ensure that no volatile flammable components escape from the container (otherwise a new sample may be necessary if further testing is required).

8.3 Do not open containers unnecessarily. Do not make transfers unless the sample temperature is at least 20°F (10°C) below the expected flash point, except for materials that are too viscous to be handled at that temperature. In these cases, transfer the specimens at the lowest possible temperature at which the material can be accurately measured into the cup.

8.4 Discard samples in leaky containers.

8.5 Do not store samples in plastic (polyethylene, polypropylene, etc.) bottles, since volatile material may diffuse through the walls of the bottles.

9. Preparation of Apparatus

9.1 Support the cup and liquid bath on a level steady table. Unless tests are made in a draft-free room or compartment, surround the tester on three sides by the shield for protection from drafts. Tests made in a laboratory draft hood or near ventilators are not reliable.

9.2 Adjust the temperature of the bath to 10°F (5°C) below the approximate flash point determined by Section 10.

9.3 Carefully clean and dry the test cup, the cover, and the cup thermometer, and bring them to approximately the same temperature as the bath liquid.

10. Procedure

10.1 *Preliminary Test*—Determine the approximate flash point of the material by one or more preliminary tests. See 10.2 through 10.7.

10.2 Fill the test cup with the appropriate amount of specimen for the cup being used at a temperature that is at least 20°F (10°C) below the approximate flash point (10.1). (See 8.3 for viscous materials.) Remove any bubbles on the surface of the specimen. Wipe the inside of the cover with a clean cloth or absorbent tissue paper.

10.3 Immediately after filling the test cup, place the cover in position and support the cup in the bath so that the cover is horizontal and the cup is immersed in direct contact with the bath liquid and with the surface of the specimen at the same level as or below that of the liquid in the bath. Confirm that the bath is at a temperature 10°F (5°C) below the approximate flash point.

10.4 Light the flame of the ignition device and adjust it to the size of a bead of diameter 5⁄32in. (4.0 mm).

10.5 As soon as the specimen has attained the same temperature as the bath (that is, the starting temperature of the definitive tests), perform a flash point test by opening the slide, inserting and removing the test flame, and closing the slide again, over a period of 2.5 ± 0.5 s. While the test flame is inserted, observe whether there is a flash.

10.6 If ignition occurs (Note 4), the initial temperature selected was too high. Repeat the complete procedure from

10.2 with a fresh specimen at a temperature about 10°F (5°C) lower.

NOTE 4—When the vapor mixture under test is near the flash-point temperature, application of the test flame may give rise to a halo; however, the product is only deemed to have flashed if a comparatively large flame appears and propagates itself over the surface of the liquid. If a large blue flame does not appear as a flash, but instead a continuous luminous flame burns in the orifice when the slide is opened and the ignition flame introduced, then the flash point of the product is much lower than the test temperature.

10.7 If no ignition occurs (Note 4), heat the bath at a rate such that the difference in temperature between the bath and the specimen never exceeds 3.5°F (2°C). When the specimen has increased in temperature by 1°F (0.5°C) (that is, after not less than 1.5 min nor more than 5 min), repeat the ignition test, and if no ignition occurs, repeat the procedure until a temperature is reached at which ignition occurs. Read to the nearest 1°F (0.5°C) the temperature indicated by the cup thermometer.

10.8 Make two determinations repeating the procedure from 10.2, using the flash point temperature determined in 10.7 as the initial temperature. Correct each temperature reading for any known thermometer correction and record the result as the flash point temperature at the barometric pressure prevailing during the test. Record also the barometric pressure in millimetres of mercury or kilopascals. Correct each flash point reading when the barometric pressure differs from 760 mm Hg (101.3 kPa). (see 11.1). Determine the mean of the corrected results of the two determinations.

NOTE 5—As volatile components are liable to be present in the products being tested, the total duration of the test should not exceed 1 h.

11. Calculation of Correction for Barometric Pressure

11.1 When the barometric pressure differs from 760 mm Hg (101.3 kPa) calculate the corrected flash point temperature *T* by means of the following equation:

$$\begin{aligned} T &= F + 0.06\,(760 - P) \\ &= C + 0.03\,(760 - P) \\ &= F + 0.42\,(101.3 - B) \\ &= C + 0.23\,(101.3 - B) \end{aligned} \quad (1)$$

where:
F (C) = observed flash point, °F (°C) and
P (B) = barometric pressure, mm of Hg (kPa).

12. Report

12.1 Report the flash point (the mean of two results) to the nearest 1°F (0.5°C) and the cup used in the test.

13. Precision and Bias

13.1 *Precision*—ISO reported the following:

13.1.1 *Repeatability*—Two results, each the mean of two determinations, obtained by the same operator using the same apparatus should be considered suspect if they differ by more than 3.5°F (2°C).

13.1.2 *Reproducibility*—Two results, each the mean of two determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 5°F (3°C).

13.2 *Bias*—The procedure for measuring flash point in this test method has no bias because the value of the flash point can be defined only in terms of a test method.

14. Keywords

14.1 closed cup; equilibrium method; flash point; Pensky-Martens; Tag