Designation: D 3934 – 90 (Reapproved 2001)

Standard Test Method for Flash/No Flash Test—Equilibrium Method by a Closed-Cup Apparatus[1]

This standard is issued under the fixed designation D 3934; 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.

INTRODUCTION

ASTM Flash Point Test Methods D 56, D 93, D 3278, and D 3941 are specified by government departments and agencies for determining whether liquids are flammable or combustible. These classifications are used as the basis for regulating the handling and shipping of liquids.

ISO/TC 35, Paints and Varnishes, and ISO/TC 28, Petroleum and Related Products, have issued ISO 1516 as a common standard, applicable to paints, varnishes, petroleum, and related products. This method is similar to ISO 1516 but uses standard ASTM cups and style and format. Test Methods D 3278 and D 3828 operate on the equilibrium principle by using the Setaflash tester that has a temperature-control device.

This test method does not determine the finite flash point but whether or not flashing occurs at a single specified temperature. The latter determination is made more accurate by ensuring that the test is carried out only when the material under test and the air/vapor mixture above it are in approximate equilibrium at the specified temperature.

1. Scope

1.1 This test method covers the determination of whether a liquid complies with the closed-cup flash point requirements in government regulations, or in specifications, or as agreed between the purchaser and the seller.

1.2 This test method is limited to a temperature range between 32 and 230°F (0 and 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 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.*

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[2]

D 3278 Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus[3]

D 3828 Test Methods for Flash Point by Small Scale Closed Tester[4]

D 3941 Test Method for Flash Point by the Equilibrium Method with a Closed-Cup Apparatus[3]

E 1 Specification for ASTM Thermometers[5]

2.2 *ISO Standard:*

ISO 1516 Paints, varnishes, petroleum, and related products—Flash/no-flash test—Closed cup equilibrium method[6]

3. Terminology

3.1 *Definitions:*

3.1.1 *flash point, n*—the lowest temperature corrected to a

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

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 05.02.

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

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

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 in a closed cup of standard design is heated in a suitable liquid bath. After the specimen has been maintained under equilibrium conditions for at least 10 min at the lowest allowable flash point temperature (within a permitted tolerance) of the specification or regulation, it is determined whether the specimen does or does not flash.

5. Significance and Use

5.1 This test method ensures that before the flash test is run the air/vapor space above the specimen has attained a saturation concentration of vapor by using standard closed cups under equilibrium conditions. The saturation concentration of the vapors will be attained at some temperature between that of the liquid and the cooler apparatus lid. However, this temperature will be close to the temperature of the specimen after it has been maintained at the specified temperature for at least a 10-min period. This test method does not provide for the determination of the actual flash point but only whether a specimen does or does not flash at a specified temperature.

6. Apparatus

6.1 *Test Cups*, 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.

NOTE 1—If a stirrer is fitted to the test cup, it shall be operated during the heating period but must be stopped during the flashing determination. If a 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*, capable of being adjusted to the required specification 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 9.2).

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 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 in the bath 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 water bath.

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

7. Reagents and Materials

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

8. Preparation of Sample

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

8.2 Because of the possibility of loss of volatiles, the sample shall receive only the minimum treatment to assure uniformity. After removal of 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 flash point.

8.4 Discard samples in leaky containers.

8.5 Do not store samples in plastic (polyethylene, polypropylene, etc.), 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 with the shield for protection from drafts. Tests made in a laboratory draft hood or near ventilation are not reliable.

9.2 Adjust the temperature of the bath to, and maintain it within 1.0°F (0.5°C) of the specified test temperature, correcting this temperature for any difference from standard barometric pressure by raising the test temperature for a higher pressure or lowering it for a lower pressure.

9.3 Carefully clean and dry the test cup, the cover, and the cup thermometer and bring them to at least 4°F (2°C) below the minimum specified test temperature.

10. Procedure

10.1 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 specified test temperature, corrected for barometric pressure (Note 4). Remove bubbles on the surface of the specimen. Wipe the inside of the cover with a clean cloth or absorbent tissue paper.

NOTE 4—Correct the temperature at which the test is to be performed in accordance with the appropriate equation:

$$°F = S - 0.06\,(760 - P) \quad (1)$$

$$°C = T - 0.03\,(760 - P)$$

$$°F = S - 0.42\,(101.3 - B)$$

$$°C = T - 0.23\,(101.3 - B)$$

where:

°F, °C	=	test temperature when the barometric pressure differs from 760 mm of Hg (101.3 kPa),
S*(T)*	=	specified flash point, °F (°C), and
P*(B)*	=	actual barometric pressure, mm of Hg (kPa).

10.2 Immediately after filling the 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

water and with the surface of the specimen at the same level as the liquid in the bath.

10.3 Light the flame of the ignition device and adjust it to the size of a bead of diameter 5⁄32in. (4 mm).

10.4 Adjust the temperature of the specimen to within 1.0°F (0.5°C) of the minimum corrected test temperature and hold at this temperature for 10 min. Apply the test flame by opening the slide, inserting and removing the nozzle of the ignition device, 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.

NOTE 5—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 material is only deemed to have flashed if a comparatively large blue 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 is much lower than the test temperature. In such circumstances, if further classification is desired, test a fresh specimen at the temperature limit for the next lower flash point classification.

10.5 Repeat the test using a fresh specimen.

11. Report

11.1 Report the following:

11.1.1 Whether the product did, or did not, flash at the specified flash point, and

11.1.2 The cup used.

12. Precision and Bias

12.1 *Precision*:

12.1.1 While the precision of this test method has not been determined, the precision of a similar definitive test described in Test Method D 3941 can be used as an indication of the precision of Test Method D 3934. This test method also can be compared to results in the flash, no-flash procedures in Test Methods D 3278 and D 3828 that are equilibrium methods.

12.1.2 The precision of flash point determinations is improved by the use of equilibrium methods. While vapors in this method will reach an equilibrium at some temperature between that of the liquid and that of the cooler apparatus cover, the temperature of the liquid under conditions of Test Method D 3934 will more closely approach true equilibrium between the liquid under test and the vapor–air mixture above it than in other methods run at finite heating rates.

12.2 *Bias*:

12.2.1 The procedure in this test method has no bias because the value of whether the material did or did not flash can be defined only in terms of a test method.

13. Keywords

13.1 closed cup; flash/no flash; flash point; Pensky-Martens; Tag