

Designation: D3941 – 14

# Standard Test Method for Flash Point by the Equilibrium Method With a Closed-Cup Apparatus<sup>1</sup>

This standard is issued under the fixed designation D3941; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

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

#### INTRODUCTION

Test Methods **D56** and **D93** 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 D56 and D93 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 0 to  $110^{\circ}$ C (32 to  $230^{\circ}$ F).

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

1.4 Warning—Mercury has been designated by many regulatory 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 Safety Data Sheet (SDS) for details and EPA's website, http://www.epa.gov/ mercury/faq.htm, for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

1.5 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.

1.6 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:<sup>2</sup>
- D56 Test Method for Flash Point by Tag Closed Cup Tester D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
- E1 Specification for ASTM Liquid-in-Glass Thermometers
- E1137 Specification for Industrial Platinum Resistance Thermometers
- E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids
- E2877 Guide for Digital Contact Thermometers

<sup>&</sup>lt;sup>1</sup> 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 D01.21 on Chemical Analysis of Paints and Paint Materials.

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<sup>&</sup>lt;sup>2</sup> 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.

# 2.2 ISO Standard:

**ISO 1523** Paints, varnishes, petroleum, and related products—Determination of flash point—Closed cup equilibrium method<sup>3</sup>

## 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  $0.5^{\circ}$ C ( $1.0^{\circ}$ F) in not less than 1.5 min so that the difference in temperature between the specimen in the cup and bath never exceeds  $2.0^{\circ}$ C ( $3.5^{\circ}$ F). Flash determinations are made at intervals of not less than 1.5 min.

#### 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 D3941, 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 D56 (Tag) and D93 (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  $160^{\circ}$ 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 D56 and D93. Alternative temperature measuring devices are permitted providing they have a similar response time and meet the respective performance and immersion depth specifications of thermometers listed in Specification E1. See Specifications E1137 and E2251, and Guide E2877 when selecting alternative temperature measuring devices. 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 E1.

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 460 mm (18 in.) wide and 610 mm (24 in.) 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 airtight 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  $10^{\circ}C$  ( $20^{\circ}F$ ) 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.

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