Designation: E502 – 21a

Standard Test Method for Selection and Use of ASTM Standards for the Determination of Flash Point of Chemicals by Closed Cup Methods[1]

This standard is issued under the fixed designation E502; 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.

1. Scope

1.1 This test method covers the determination of the flash point of liquid and solid chemical compounds flashing from below −10 to 370°C (16 to 700°F). The procedures and apparatus in Test Methods D56, D93, D3278, D3828, and D3941 are to be used. Modification to these procedures are specified for tests on solids and viscous liquids. The significance of the results obtained is discussed along with possible sources of error and factors that might cause interference.

1.2 Suggestions for adapting this procedure to mixtures of chemicals are included (see Appendix X2).

1.3 This test method 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 or assemblies under actual fire conditions. However, results of this test method may be used as elements of a fire risk assessment that take into account all of the factors that are pertinent to an assessment of the fire hazard of a particular end use.

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

1.5 **Warning—**Mercury has been designated by the United States Environmental Protection Agency (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, into your state may be prohibited by state law.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* See also Section 8.

1.7 *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

D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D270 Methods of Sampling Petroleum and Petroleum Products (Withdrawn 1984)[3]

D1310 Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus

D3278 Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus

D3828 Test Methods for Flash Point by Small Scale Closed Cup Tester

D3934 Test Method for Flash/No Flash Test—Equilibrium Method by a Closed-Cup Apparatus

D3941 Test Method for Flash Point by the Equilibrium Method With a Closed-Cup Apparatus

E681 Test Method for Concentration Limits of Flammability of Chemicals (Vapors and Gases)

E1232 Test Method for Temperature Limit of Flammability of Chemicals

[1] This test method is under jurisdiction of ASTM Committee E27 on Hazard Potential of Chemicals and is the direct responsibility of Subcommittee E27.04 on Flammability and Ignitability of Chemicals.

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[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] The last approved version of this historical standard is referenced on www.astm.org.

3. Terminology

3.1 *Definitions:*

3.1.1 *flash point, n—in flash point test methods*, the lowest 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 sample to ignite under specified conditions of test.

4. Summary of Test Method

4.1 The specimen is placed in a closed cup and, in the small scale method, equilibrated at a test temperature; in the Pensky-Martens method, heated at a controlled rate with stirring; and in the tag method, also heated at a controlled rate but without stirring. A small flame is directed into the vapor space of each cup at specified intervals, with simultaneous interruption of stirring in the Pensky-Martens method, to determine whether a flash occurs or not. In Test Method D3941, the specimen is heated at a slower rate than in the other controlled heating methods, maintaining a small temperature differential between bath and specimen.

5. Significance and Use

5.1 The flash point measures the response of the sample to heat and flame 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 As a result of physical factors inherent in the apparatus and procedure, the closed cup flash point does not necessarily represent the minimum temperature at which a material can evolve flammable vapors, and the absence of a flash point does not guarantee nonflammability (see Appendix X1 and Appendix X2).

5.3 Flash point is used in shipping and safety regulations to define flammable and combustible materials. Test Methods D56, D93, and D3278 are specified as test methods for determining the flash point of these materials.

5.4 If the process or handling conditions dictate the usage of a flammable material at temperatures ranging upward from 5 to 10°C below the closed-cup flash point, then a flammable vapor might be present above the liquid. In such cases, it may be more appropriate to use the temperature limit of flammability (as determined by Test Method E1232) instead of flash point.

5.5 For single component samples, small-scale methods involving equilibrium procedures and only one flame pass per specimen are preferred.

5.6 For mixtures containing small concentrations of volatile components, special procedures are needed to minimize the loss of volatiles, with consequent elevation of the flash point, while the sample is being heated. (See X2.5.)

5.7 In cases where errors caused by loss of volatiles, downwards flame direction and quenching are unacceptable, the "lower temperature limit of flammability" can be determined instead using Test Method E1232. The temperature limit of flammability test chamber is sufficiently large to overcome flame quenching effects in most cases of practical importance, thus, usually indicating the presence of vapor-phase flammability if it does exist.

6. Interferences

6.1 Incorrect flash points can be obtained when testing chemicals corrosive to the materials of construction of the cup. (For example, certain amines and acid chlorides react with the standard aluminum small scale cup causing erroneously low flash points, perhaps due to hydrogen formation.) Cups employing alternative materials of construction, electroplating or plastic coating can provide corrosion resistance. Results in non-standard cups, particularly in non-equilibrium tests, may differ slightly from those obtained in this test method.

7. Apparatus

7.1 *Tag Closed-Cup Tester,* including thermometers, shall be as shown in Test Methods D56 and D3941.

7.2 *Penksy-Martens Closed-Cup Tester,* including thermometers, shall be as shown in Test Methods D93.

7.3 *Small Scale Closed Tester,* including thermometers, shall be as shown in Test Methods D3278 or D3828.

NOTE 1—Some automatic flash point testers may save testing time and permit the use of small samples. If automatic testers are used, the user must be certain that all instructions for calibration and operation are followed to ensure that the results are equivalent to those obtained on the ASTM standard equipment. For regulation purposes or in cases of dispute, the flash point as determined on the manual tester shall be the accepted value.

NOTE 2—ASTM thermometers 33C or 33F may be used in the tag tester instead of those specified in Test Method D56 when conducting tests at temperatures below –10°C (14°F). Slight stem corrections may be necessary and care should be taken to avoid freezing the mercury in the thermometer by cooling below –40°C (–40°F).

7.4 *Shield,* as described in Test Method D3941 or D1310.

8. Hazards

8.1 *Toxicity of Chemical and Combustion Products:*

8.1.1 Isolate or control operations on toxic or corrosive materials to prevent exposure to any personnel.

8.1.2 Since flash point tests are conducted in still air, the use of forced circulation for removal of toxic or nuisance fumes or combustion products is restricted. However, a laboratory fume hood equipped with an exhaust damper that can be completely closed provides an ideal location for maintaining draft-free conditions and provides the ability to readily exhaust dangerous vapors and combustion products when necessary.

8.1.3 Use respiratory and splash protective devices as appropriate with toxic or corrosive materials. In most cases, approved cartridge respirators are adequate respiratory protection for the concentrations normally encountered in flash-point testing. Certain toxic or unusual materials, however, may require an air-supplied respirator and extreme cases may require complete protective coverage such as an air-supplied plastic suit. (Two examples of the latter type of material are dimethyl sulfate and pure mercaptans.) Tests on these highly toxic or obnoxious materials may also be conducted in completely isolated, closed systems, such as glove boxes. In this case, procedures should ensure an uncontaminated air system in the box, and should prevent a buildup of vapors from the material under test.

8.2 *Dry Ice Use:*

8.2.1 Exercise care in the use of dry ice for sample and apparatus cooling. Avoid contact with dry ice to prevent frostbite. Glass bottles or vials of chemicals should not be placed directly in dry ice or dry ice baths because of the possibility of breakage due to thermal shock.

8.3 *Tests of Explosives and Propellants:*

8.3.1 Flash tests should not be conducted on potential or known explosive or propellant materials without complete prior knowledge that burning will not result in propagation to an explosive decomposition. Properly barricaded or remotely operated automatic testers should be used if precise flash points are needed.

8.4 *Pyrophoric Materials:*

8.4.1 Flash point apparatus is not applicable for the evaluation of pyrophoric materials and should not be used for this purpose.

9. Preparation of Sample

9.1 Obtain samples representative of the batch under test. Methods D270 can be used as a reference on sampling techniques. With mixtures and with samples containing impurities, take care to avoid the loss of volatile components during sampling and handling for testing. When heating viscous or solid materials for ease of pouring, samples must be held at temperatures below, or as close as possible to, those specified in the various test methods. Discard samples from leaking or contaminated containers. Samples that are hygroscopic should not be exposed to moisture or moist air.

9.2 Samples should not be stored in plastic (polyethylene, polypropylene, etc.) bottles, since volatile materials may diffuse through the walls of the bottle.

10. Preparation of Apparatus

10.1 Support the appropriate flash-point tester on a level, steady work surface in a draft-free location. If a draft-free location is not available, use a shield surrounding the tester on three sides. The shield should be approximately 460 mm (18 in.) wide and 610 mm (24 in.) high.

NOTE 3—An area capable of being partially darkened is advantageous since it aids in the detection of the relatively nonluminous flames sometimes encountered in flash-point testing.

NOTE 4—Test Method D1310 gives a design for a draft shield suitable for standard flash-point testers.

11. Calibration

11.1 Check the condition and operation of the tag, Pensky-Martens, and small-scale testers as specified in Test Methods D56, D93, D3278, or D3828, respectively.

12. Procedure

12.1 Follow the procedures outlined in Test Methods D56 or D3941 (tag closed cup), D3278 or D3828 (small-scale closed cup), and D93 (Pensky-Martens closed cup), as is necessary. Certain explanatory notes and procedure modifications not contained in the individual methods are given below. Occasionally, particularly near the temperature of the actual flash point, the application of the test flame will cause a halo or test flame enlargement that should be ignored. In some cases this test flame enlargement will not lead to a flash point on an increase in temperature.

12.2 For liquids with a viscosity less than 5.8×10^{-6} m^2/s (5.8 cSt) at 38°C (100°F), or 9.5×10^{-6} m^2/s (9.5 cSt) at 25°C (77°F), observe the following:

NOTE 5—The first viscosity threshold point is stated either as "5.8×10^{-6} m^2/s (5.8 cSt) at 100°F (38°C)", or as "5.5×10^{-6} m^2/s (5.5 cSt) at 40°C (104°F), in different flash point test standards. The choice is indicative of only the unit system preferred by individual test standards. In practice the two forms are considered equivalent.

12.2.1 If the flash point is below 93°C (200°F), use the small-scale (Test Method D3278 or D3828) or tag (Test Method D56) apparatus and procedures.

12.2.2 If the flash point is 93°C (200°F) or above, use the small-scale (Test Method D3828) or Pensky-Martens (Test Methods D93) apparatus and procedures.

NOTE 6—The electric heaters on some tag testers may be of insufficient capacity to maintain the specified heating rates when operating in the upper ranges of this test method. Heat input can be increased slightly by using a variable transformer to increase the voltage slightly on the heaters. Insulation can be applied to the exterior of the bath to reduce heat losses.

NOTE 7—With low temperature operation in the small scale methods, equilibrium may be difficult to maintain due to heating by natural convection. It, therefore, will be necessary to cool the cup and sample below the anticipated flash point before specimen introduction (see Test Methods D3278).

NOTE 8—In the tag method (Test Method D56), natural warming rates sometimes exceed 1°C (2°F)/min. These rates can be reduced by insulating the outside of the bath container. A laboratory refrigerated circulator may be used. One advantage of this system is that circulation of the refrigerant bath with the system gradually warming up can serve as a control on heating rate.

NOTE 9—With low-temperature operation in the tag and small scale methods, difficulties can be created by the formation of frost on the surface of the tester. If precise flash points are needed in the temperature range where frost conditions are encountered, tests can be conducted in a dry box or a room of very low humidity. When ice formation on the lid and cover parts cannot be avoided, the results will be unreliable. Sticking of the slide due to ice formation can be minimized by carefully lubricating the slide with a high vacuum silicone lubricant. Portions of the cover and slide in the vicinity of the pilot flame and openings should be wiped free of frost just prior to the initial flame insertion at 5°C (10°F) below the flash point.

12.3 For liquids with a viscosity equal to or greater than 5.8×10^{-6} m^2/s (5.8 cSt) at 38°C (100°F), or 9.5×10^{-6} m^2/s (9.5 cSt) at 25°C (77°F) and less than 15×10^{-3} m^2/s (150 St) at 25°C (77°F), and a flash point below 110°C (230°F), the following procedure applies:

12.3.1 Use of Pensky-Martens (Test Methods D93) or the small scale (Test Method D3278 or D3828) apparatus and procedure.

12.4 For liquids with a viscosity equal to or greater than 5.8×10^{-6} m^2/s (5.8 cSt) at 38°C (100°F) or 9.5×10^{-6} m^2/s (9.5 cSt) at 25°C (77°F) and a flash point of 93°C (200°F) or above, the following procedure applies:

12.4.1 Use the Pensky-Martens (Test Methods D93) and the small scale (Test Method D3828) apparatus and procedure.

NOTE 10—Testing time may be reduced by initially heating samples at higher rates than those specified in the test procedures, provided that the

specified heating rates are maintained in the temperature range in the vicinity of the flash point. This is permissible provided that, during the fast heat-up period, the highest temperature of the material (next to the cup wall) never exceeds a temperature 11°C (20°F) below the flash point for the Pensky-Martens method. Use extreme care when using fast heat-up in the Pensky-Martens method since there are no provisions for bath temperature measurement.

12.5 For liquids with a viscosity equal to or greater than 15×10^{-3} m^2/s (150 St at 25°C) (77°F) and solid materials that flash while solid (Note 12), the following procedures apply:

12.5.1 Use the small scale Test Methods D3278 or Test Method D3828 with the following modification:

12.5.1.1 Determine the flash point in the small scale unit using a holding time of 6 min at the test temperature instead of 1 or 2 min normally employed.

12.5.1.2 Methods for loading the sample cup with highly viscous liquids or solids are given in Test Methods D3278. Solid materials can be loaded with a spoon.

12.6 For equilibrium flash point method using the tag closed cup tester, the following applies:

12.6.1 For liquids with a viscosity equal to or greater than 5.8×10^{-6} m^2/s (5.8 cSt at 38°C (100°F) or 9.5×10^{-6} m^2/s (9.5 cSt) at 25°C (77°F) and a flash point below 93°C (200°F), Test Method D3941 may be used using the tag closed cup.

12.6.2 Test Method D3941 may also be used for the high viscosity liquids and solids covered in 12.6 (Note 12). Observe the temperature differences between bath and sample specified in Test Method D3941. (The tag tester is very inefficient for testing these materials since large sample quantities and very long testing times are required.)

NOTE 11—With highly viscous materials it may be advantageous to fill the tag closed cup vessel directly to a 50-mL line on the sample cup rather than to use the graduated cylinder specified in Test Method D56. Significant quantities of a viscous material may adhere to the walls of the graduated cylinder when the transfer is made. Certain manufacturers supply tag cups with a 50-mL line inscribed on the inner cup wall.

NOTE 12—The small scale procedure is the preferred method for solid materials.

NOTE 13—The intent of the low heat rate specified in Test Method D3941 is to ensure that misleading results are not obtained because of the poor heat transfer characteristic of viscous materials. The test thermometer should closely reflect the highest temperature to which the specimen throughout the cup is being subjected. If a small temperature difference between the temperature of the bath and the specimen is not maintained, warm materials next to the cup walls will evolve vapors resulting in a positive flash test while the test thermometer registers the temperature of the cooler materials near the center of the cup.

12.7 Follow the procedures outlined in Test Methods D56, D3278, D93, D3828, and D3941 pertaining to recording of flash point, discarding of results, number of samples to be run, etc.

13. Corrections for Barometric Pressure

13.1 Observe and record the ambient barometric pressure at the time and place of the test.

NOTE 14—The barometric pressure used in this calculation is the ambient pressure for the laboratory at the time of the test. Many aneroid barometers, such as those used at weather stations and airports, are precorrected to give sea level readings and would not give the correct reading for this test.

13.2 If the pressure differs from 760 mm Hg (101.3 kPa), correct the flash point as follows:

$$\text{Corrected flash point (°C)} = C + 0.25\,(101.3 - A) \quad (1)$$

$$\text{Corrected flash point (°F)} = F + 0.06\,(760 - B)$$

$$\text{Corrected flash point (°C)} = C + 0.033\,(760 - B)$$

where:

F = observed flash point, °F,
C = observed flash point, °C,
B = ambient barometric pressure, mm Hg, and
A = ambient barometric pressure, kPa.

NOTE 15—The above barometric correction is an approximation based on a material of average lower explosive limit having a vapor pressure curve of average slope. Theoretically, a separate barometric adjustment would be required for each material; however, the above approximation is adequate for most cases. For "non-standard" materials, for flash point measurements made at high altitudes (Denver, CO, for example) or for data being used to evaluate hazards at high altitudes, corrections might better be based on the actual vapor pressure and explosive limit data of the material in question.

13.3 Round the final average-corrected flash point downward to the nearest whole number.

14. Report

14.1 The report shall specify the following:

14.1.1 Flash point rounded downward to the nearest 1°C (or 1°F),

14.1.2 Test method used,

14.1.3 Date,

14.1.4 Purity of the material if known (commercial grade, reagent grade, chemically pure, etc.), and

14.1.5 Special preparation of sample (for example, the degree and method of evaporation if a mixture is so treated).

14.2 A report shall be issued for those samples tested that do not flash. This latter report shall state either "no flash to boiling at __°C (°F)" or "no flash to __°C (°F)."

15. Precision and Bias[4]

15.1 The following criteria shall be used for judging the acceptability of results as shown in the respective test methods.

15.1.1 The precision and bias of these methods of measuring flash point are as specified in Test Methods D56, D92, D93, D3278, D3828, D3934, and D3941.

15.1.2 *Repeatability*—Duplicate results by the same operator shall not be considered suspect unless they differ by more than the amount in Table 1.

15.1.3 *Reproducibility*—The results submitted by each of two laboratories shall not be considered suspect unless they differ by more than the amount in Table 2.

[4] Supporting data obtained from an interlaboratory test on chemicals, of insufficient magnitude to establish limits of precision, have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E27-1000. These data indicate that, for chemicals, precision limits are within those of Test Methods D56, D93, D3278, and D3828. Contact ASTM Customer Service at service@astm.org.