

and only those animals without eye defects or irritation shall be used. The animal is held firmly but gently until quiet. The test material is placed in one eye of each animal by gently pulling the lower lid away from the eyeball to form a cup into which the test substance is dropped. The lids are then gently held together for one second and the animal is released. The other eye, remaining untreated, serves as a control. For testing liquids, 0.1 milliliter is used. For solids or pastes, 100 milligrams of the test substance is used, except that for substances in flake, granule, powder, or other particulate form the amount that has a volume of 0.1 milliliter (after compacting as much as possible without crushing or altering the individual particles, such as by tapping the measuring container) shall be used whenever this volume weighs less than 100 milligrams. In such a case, the weight of the 0.1 milliliter test dose should be recorded. The eyes are not washed following instillation of test material except as noted below.

(2) The eyes are examined and the grade of ocular reaction is recorded at 24, 48, and 72 hours. Reading of reactions is facilitated by use of a binocular loupe, hand slit-lamp, or other expert means. After the recording of observations at 24 hours, any or all eyes may be further examined after applying fluorescein. For this optional test, one drop of fluorescein sodium ophthalmic solution U.S.P. or equivalent is dropped directly on the cornea. After flushing out the excess fluorescein with sodium chloride solution U.S.P. or equivalent, injured areas of the cornea appear yellow; this is best visualized in a darkened room under ultraviolet illumination. Any or all eyes may be washed with sodium chloride solution U.S.P. or equivalent after the 24-hour reading.

(b)(1) An animal shall be considered as exhibiting a positive reaction if the test substance produces at any of the readings ulceration of the cornea (other than a fine stippling), or opacity of the cornea (other than a slight dulling of the normal luster), or inflammation of the iris (other than a slight deepening of the folds (or rugae) or a slight circumcorneal injection of the blood vessels), or if such substance

produces in the conjunctivae (excluding the cornea and iris) an obvious swelling with partial eversion of the lids or a diffuse crimson-red with individual vessels not easily discernible.

(2) The test shall be considered positive if four or more of the animals in the test group exhibit a positive reaction. If only one animal exhibits a positive reaction, the test shall be regarded as negative. If two or three animals a positive reaction, the test is repeated using a different group of six animals. The second test shall be considered positive if three or more of the animals exhibit a positive reaction. If only one or two animals in the second test exhibit a positive reaction, the test shall be repeated with a different group of six animals. Should a third test be needed, the substance will be regarded as an irritant if any animal exhibits a positive response.

(c) To assist testing laboratories and other interested persons in interpreting the results obtained when a substance is tested in accordance with the method described in paragraph (a) of this section, an "Illustrated Guide for Grading Eye Irritation by Hazardous Substances" will be sold by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.¹ The guide will contain color plates depicting responses of varying intensity to specific test solutions. The grade of response and the substance used to produce the response will be indicated.

[38 FR 27012, Sept. 27, 1973; 38 FR 30105, Nov. 1, 1973; 62 FR 46667, Sept. 4, 1997]

§ 1500.43 Method of test for flashpoint of volatile flammable materials by Tagliabue open-cup apparatus.

SCOPE

1. (a) This method describes a test procedure for the determination of open-cup flashpoints of volatile flammable materials having flashpoints below 175 °F.

(b) This method, when applied to paints and resin solutions which tend to skin over

¹The Illustrated Guide is out of print and, as of January 1, 1981, no longer available. However, information about the test method, and black and white photocopies may be obtained by writing to the Directorate for Epidemiology and Health Sciences, CPSC, Washington, D.C. 20207, (301) 504-0957.

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or which are very viscous, gives less reproducible results than when applied to solvents.

OUTLINE OF METHOD

2. The sample is placed in the cup of a Tag Open Tester, and heated at a slow but constant rate. A small test flame is passed at a uniform rate across the cup at specified intervals. The flashpoint is taken as the lowest temperature at which application of the test flame causes the vapor at the surface of the liquid to flash, that is, ignite but not continue to burn.

APPARATUS

3. The Tag open-cup tester is illustrated in Fig. 1. It consists of the following parts, which must conform to the dimensions shown, and have the additional characteristics as noted:

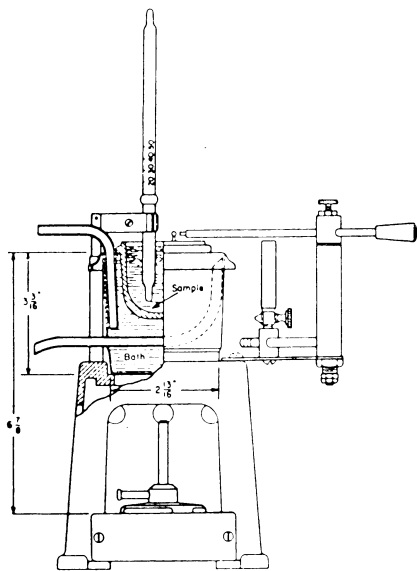


FIGURE 1—Tag open-cup flash tester

(a) *Copper bath*, preferably equipped with a constant level overflow so placed as to maintain the bath liquid level 1/8-inch below the rim of the glass cup.

(b) *Thermometer holder*. Support firmly with ringstand and clamp.

(c) *Thermometer*. For flashpoints above 40 °F., use the ASTM Tag Closed Tester Thermometer, range of +20 to +230 °F., in 1 °F. divisions, and conforming to thermometer 9F. of ASTM Standard E 1. For flashpoints from 20 °F. to 40 °F., use ASTM Tag Closed Tester, Low Range, Thermometer 57F. For flashpoints below 20 °F., use ASTM Thermometer 33F. The original Tag Open-Cup

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(Paper Scale) Thermometer will be a permissible alternate until January 1, 1962. It is calibrated to -20 °F.

(d) *Glass test cup*. Glass test cup (Fig. 2), of molded clear glass, annealed, heat-resistant, and free from surface defects.

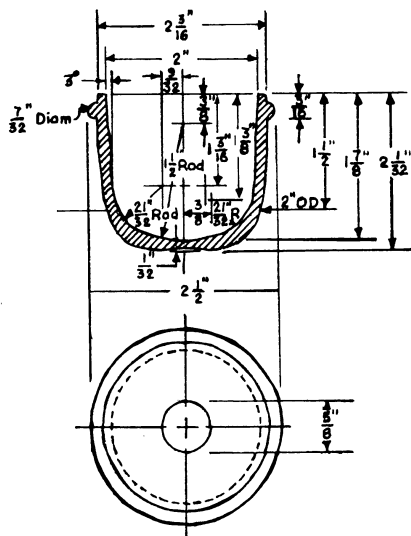


FIGURE 2—Glass test cup

(e) *Leveling device*. Leveling device or guide, for proper adjustment of the liquid level in the cup (Fig. 3). This shall be made of No. 18-gage polished aluminum, with a projection for adjusting the liquid level when the sample is added to exactly 1/8-inch below the level of the edge or rim of the cup.

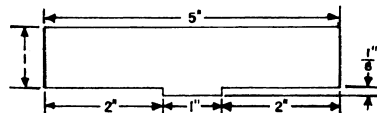


FIGURE 3—Leveling device for adjusting liquid level in test cup

(f) "Micro," or small gas burner of suitable dimensions for heating the bath. A screw clamp may be used to help regulate the gas. A small electric heater may be used.

(g) *Ignition taper*, which is a small straight, blow-pipe type gas burner. The test flame torch prescribed in the method of test for flash and fire points by Cleveland Open Cup (ASTM designation: D 92) is satisfactory.

(h) Alternative methods for maintaining the ignition taper in a fixed horizontal plane above the liquid may be used, as follows:

(1) *Guide wire*, 3/32-inch in diameter and 3 1/2 inches in length, with a right-angle bend 1/2-inch from each end. This wire is placed snugly in holes drilled in the rim of the bath, so

that the guide wire is $\frac{3}{8}$ -inch from the center of the cup and resting on the rim of the cup.

(2) Swivel-type taper holder, such as is used in ASTM METHOD D 92. The height and position of the taper are fixed by adjusting the holder on a suitable ringstand support adjacent to the flash cup.

(i) Draft shield, consisting of two rectangular sheets of noncombustible material, 24 inches \times 28 inches, are fastened together along the 28-inch side, preferably by hinges. A triangular sheet, 24 inches \times 24 inches \times 34 inches is fastened by hinges to one of the lateral sheets (to form a top when shield is open). The interior of the draft shield shall be painted a flat black.

PROCEDURE

4. (a) Place the tester on a solid table free of vibration, in a location free of perceptible draft, and in a dim light.

(b) Run water, brine, or water-glycol solution into the bath to a predetermined level, which will fill the bath to $\frac{1}{8}$ -inch below the top when the cup is in place. An overflow is permissible for water-level control.

(c) Firmly support the thermometer vertically halfway between the center and edge of the cup on a diameter at right angles to the guide wire, or on a diameter passing through the center of the cup and the pivot of the taper. Place so that the bottom of the bulb is $\frac{1}{4}$ -inch from the inner bottom surface of the cup. If the old Tagliabue thermometer is used, immerse to well cover the mercury bulb, but not the wide body of the thermometer.

(d) Fill the glass cup with the sample liquid to a depth just $\frac{1}{8}$ -inch below the edge, as determined by the leveling device.

(e) Place the guide wire or swivel device in position, and set the draft shield around the tester so that the sides from right angles with each other and the tester is well toward the back of the shield.

(f) If a guide wire is used, the taper, when passed, should rest lightly on the wire, with the end of the jet burner just clear of the edge of the guide wire. If the swivel-type holder is used, the horizontal and vertical positions to the jet are so adjusted that the jet passes on the circumference of a circle, having a radius of at least 6 inches, across the center of the cup at right angles to the diameter passing through the thermometer, and in a plane $\frac{1}{8}$ -inch above the upper edge of the cup. The taper should be kept in the "off" position, at one end or the other of the swing, except when the flame is applied.

(g) Light the ignition flame and adjust it to form a flame of spherical form matching in size the $\frac{5}{32}$ -inch sphere on the apparatus.

(h) Adjust heater source under bath so that the temperature of the sample increases at a rate of 2 ± 0.5 °F. per minute. With viscous materials this rate of heating cannot always be obtained.

INITIAL TEST

5. Determine an approximate flashpoint by passing the taper flame across the sample at intervals of 2 °F. Each pass must be in one direction only. The time required to pass the ignition flame across the surface of the sample should be 1 second. Remove bubbles from the surface of the sample liquid before starting a determination. Meticulous attention to all details relating to the taper, size of taper flame, and rate of passing the taper is necessary for good results. When determining the flashpoint of viscous liquids and those liquids that tend to form a film of polymer, etc., on the surface, the surface film should be disturbed mechanically each time before the taper flame is passed.

RECORDED TESTS

6. Repeat the procedure by cooling a fresh portion of the sample, the glass cup, the bath solution, and the thermometer at least 20 °F. below the approximate flashpoint. Resume heating, and pass the taper flame across the sample at two intervals of 2 °F. until the flashpoint occurs.

REPORTING DATA

7. The average of not less than three recorded tests, other than the initial test, shall be used in determining the flashpoint and flammability of the substance.

STANDARDIZATION

8. (a) Make determinations in triplicate on the flashpoint of standard paraxylene and of standard isopropyl alcohol which meet the following specifications:

(i) *Specifications for p-xylene, flashpoint check grade. p-xylene shall conform to the following requirements;*

Specific gravity: 15.56 °C./15.56 °C., 0.860 minimum, 0.866 maximum

Boiling range: 2 °C. maximum from start to dry point when tested in accordance with the method of test for distillation of industrial aromatic hydrocarbons (ASTM designation: D 850), or the method of test for distillation range of lacquer solvents and diluents (ASTM) designation D 1078). The range shall include the boiling point of pure *P*-xylene, which is 138.35 °C. (281.03 °F.).

Purity: 95 percent minimum, calculated in accordance with the method of test for determination of purity from freezing points of high-purity compounds (ASTM designation: D 1016), from the experimentally determined freezing point, measured by the method of test for measurement of freezing points of high-purity compounds for evaluation of purity (ASTM designation: D 1015).

(ii) *Specifications for isopropanol, flash point check grade. Isopropanol shall conform to the following requirements:*

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Specific gravity: 0.8175 to 0.8185 at 20 °C./20 °C. as determined by means of a calibrated pycnometer.

Distillation range: Shall entirely distill within a 1.0 °C. range which shall include the temperature 80.4 °C. as determined by ASTM method D 1078.

Average these values for each compound. If the difference between the values for these two compounds is less than 15 °F. (8.5 °C.) or more than 27 °F. (16 °C.), repeat the determinations or obtain fresh standards.

(b) Calculate a correction factor as follows:

$$X = 92 - A$$

$$Y = 71 - B$$

$$\text{Correction} = (X + Y) / 2.$$

Where:

A=Observed flash of *p*-xylene, and

B=Observed flash of isopropyl alcohol.

Apply this correction of all determinations. Half units in correction shall be discarded.

PRECISION

9. (a) For hydrocarbon solvents having flashpoints between 60 °F. and 110 °F., repeatability is ± 2 °F. and the reproducibility is ± 5 °F.

(b) If results from two tests differ by more than 10 °F., they shall be considered uncertain and should be checked. This calibration procedure provided in this method will cancel out the effect of barometric pressure if calibration and tests are run at the same pressure. Data supporting the precision are given appendix III of the 1956 Report of Committee D-1 on Paint, Varnish, Lacquers and Related Products, Proceedings, Am. Soc. Testing Mats., Vol. 56 (1956).

NOTE: The test apparatus and procedure described in §1500.43 may be used by manufacturers and labelers of products subject to the Federal Hazardous Substances Act to determine flashpoint temperatures of those products under the conditions set forth in §1500.3(c)(6)(iv), as amended.

[51 FR 28537, Aug. 8, 1986]

§ 1500.43a Method of test for flashpoint of volatile flammable materials.

(a) *Scope.* (1) This method describes the test procedure which the Commission will use for the determination of the flashpoint of volatile flammable materials, using a Setaflash¹ low-range closed tester, or an apparatus producing equivalent results. The method described in this section is essentially a Setaflash equilibrium procedure

¹Setaflash is a registered trademark of Stanhope-Seta Limited, Surrey, England.

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which closely parallels the test method designated ASTM D 3828-81, "Standard Test Methods for Flash Point by Setaflash Closed Tester," published by the American Society for Testing and Materials (ASTM), 1916 Race Street, Philadelphia, Pennsylvania 19103. Manufacturers and labelers of products subject to labeling and other requirements under the Federal Hazardous Substances Act may use other apparatus and/or test methods which produce equivalent results.

(2) At the option of the user, the procedures described in this section may be used to determine the actual flashpoint temperature of a sample or to determine whether a product will or will not flash at a specified temperature (flash/no flash).

(3) If the substance to be tested has a viscosity greater than 150 Stokes at 77 °F (25 °C), see paragraph (n) of this section for modifications to the testing procedure.

(4) If the Commission has reason to believe on the basis of reliable experience or other relevant information or data that the flammability hazard of a substance is greater or less than its flammability classification based on flashpoint temperature determined in accordance with this §1500.43a and that the substance should be reclassified, the Commission will initiate a rule-making proceeding for reclassification of the substance. Product manufacturers and labelers may use reliable experience or other relevant information or data in addition to the flashpoint temperature of a substance as a basis for compliance with any applicable requirements of the Federal Hazardous Substances Act in the absence of a rule issued by the Commission to reclassify the substance.

(b) *Summary of test methods.* (1) Method A—Flash/No Flash Test. A specified volume of sample is introduced by a syringe into the cup of the apparatus that is set and maintained at the specified temperature. After a specific time a test flame is applied and an observation made as to whether or not a flash occurred. Test procedures are set forth in detail in §1500.43a(i).

(2) Method B—Finite (or Actual) Flashpoint. (i) A specified volume of sample is introduced into the cup of