

§ 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 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 the apparatus that is maintained at the expected flashpoint. After a specified time a test flame is applied and the observation made whether or not a flash occurred.

(ii) The specimen is removed from the cup, the cup cleaned, and the cup temperature adjusted 5 °C (9 °F), lower or higher depending on whether or not a flash occurred previously. A fresh specimen is introduced and tested. This procedure is repeated until the flashpoint is established within 5 °C (9 °F).

(iii) The procedure is then repeated at 1 °C (2 °F) intervals until the flashpoint is determined to the nearest 1 °C (2 °F).

(iv) If improved accuracy is desired the procedure is repeated at 0.5 °C (1 °F). Test procedures are set forth in detail at §1500.43a(j).

(3) The test procedures will be modified, where necessary, to ensure that the results obtained reflect the hazard of the substance under reasonably foreseeable conditions of use. Thus, for example, the material, if a mixture, will normally be tested as it comes from the container, and/or after a period of evaporation. The period of evaporation for a material which is a mixture will normally be the time required for the mixture to evaporate in an open beaker under ambient conditions to 90 percent of its original volume, or a period of four hours, whichever occurs first. However, this period of evaporation will be changed if the results obtained do not represent the hazard of the substance under reasonably foreseeable conditions of use.

(c) *Definition of flashpoint.* The lowest temperature of the sample, corrected

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

to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of a test flame causes the vapor of the sample to ignite under specified conditions of test. The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the sample. Occasionally, particularly near actual flashpoint, the application of the test flame will cause a halo or an enlarged flame; this is not a flash and should be ignored.

(d) *Test apparatus.* The test apparatus is an equilibrium closed-cup tester with a range up to 100 °C (212 °F). The essential dimensions and requirements are shown in figure 1 and table 3, and are described in §1500.43a(m). Closed-cup flashpoint testers and accessories meeting these requirements are available from commercial suppliers and distributors of laboratory equipment.

(e) *Safety precautions.* The operator must exercise and take appropriate safety precautions during the initial application of the test flame to the sample. Samples containing low-flash material may give an abnormally strong flash when the test flame is first applied.

(f) *Preparation of samples.* (1) Erroneously high flashpoints may be obtained if precautions are not taken to avoid the loss of volatile material. In preliminary tests of materials taken directly from the container, do not open containers unnecessarily and make a transfer unless the sample temperature is at least 10 °C (18 °F) below the expected flashpoint. Do not use samples in leaky containers for this test.

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

(3) A 2-ml specimen is required for each test. If possible, obtain at least a 50-ml sample from the bulk test site and store in a clean, tightly closed container.

(g) *Preparation of apparatus.* (1) Place the tester on a level, stable surface. Unless tests are made in a draft-free area, surround the tester on three sides with a shield for protection. Do not rely on tests made in a laboratory draft hood or near ventilators.

(2) Read the manufacturer's instructions on the care and servicing of the instrument and for correct operation of its controls.

(h) *Calibration and standardization.* (1) Before initial use determine and plot the relationship between the temperature control dial and the thermometer readings at each major (numbered) dial division as follows:

Turn the temperature control knob² fully counterclockwise ("O" reading). Advance the temperature control knob clockwise until the indicator light is illuminated.³ Advance the knob clockwise to the next numbered line. After the thermometer mercury column ceases to advance, record the dial reading and the temperature. Advance the knob clockwise to the next numbered line. After the thermometer mercury column ceases to advance, read the dial reading and the temperature. Repeat this procedure through the full range of the instrument. Plot the dial readings versus the respective temperatures.

(2) Standardize the instrument using a sample of material meeting the specifications in table 1. If the average of two determinations falls within the acceptable limits the instrument is assumed to be operating properly. If the average of the two determinations does not fall within this range, check the manufacturer's operating and maintenance instructions and determine that they are being followed. In particular, be sure that the cup lid assembly makes a vapor-tight seal with the cup, the shutter provides a light-tight seal, and that adequate heat transfer paste surrounds the thermometer bulb and the immersed portion of the barrel.

(i) *Test Method A—for determining Flash/No Flash.* (1) Determine the target flashpoint as follows:

²If the instrument has two temperature control knobs, set the fine control (center, small knob) at its mid-position and allow it to remain there throughout the calibration. The calibration is determined by adjusting the coarse control (large, outer knob) only.

³When using the tester, it will be found that the indicator light may not illuminate and the temperature may not rise until a temperature control dial setting between one and two is reached.

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- (i) Target flashpoint, °C = $S_c - 0.25$
(101.3—A)
- (ii) Target flashpoint, °C = $S_c - 0.03$
(760—B)
- (iii) Target flashpoint, °F = $S_f - 0.06$
(760—B)

where:

S_c = specification, or uncorrected target, flashpoint, °C,

S_f = specification, or uncorrected target, flashpoint, °F,

B = ambient barometric pressure, mm Hg,⁴ and

A = ambient barometer pressure, kPa.⁴

(2) Inspect the inside of the sample cup, lid, and shutter mechanism for cleanliness and freedom from contamination. Use an absorbent paper tissue to wipe clean, if necessary. Put cover in place and lock securely. The filing orifice may be conveniently cleaned with a pipe cleaner.

(3) Set the instrument at the target temperature.

(i) For target temperature below ambient. The instrument power switch is to be in the off position. Fill the refrigerant-charged cooling block with a suitable material.⁵ Raise the lid and shutter assembly, and position the base of the block in the sample cup, being careful not to injure or mar the cup. When the thermometer reads approximately 6 to 10 °C (10 to 20 °F) below the target temperature, remove the cooling block and quickly dry the cup with a paper tissue to remove any moisture. Immediately close the lid and shutter assembly and secure. Prepare to introduce the sample using the syringe, both of which have been precooled to a temperature 5 to 10 °C (10 to 20 °F) below the target temperature.

⁴The barometric pressure used in this calculation must be the ambient pressure for the laboratory at the time of test. Many aneroid barometers, such as those used at weather stations and airports, are precorrected to give sea-level readings; these must not be used.

⁵If the target or specification temperature is not less than 5 °C (40 °F) crushed ice and water may be used as charging (cooling) fluid. If below 5 °C (40 °F), a suitable charging (cooling) fluid is solid carbon dioxide (dry ice) and acetone. If the refrigerant charged cooling module is unavailable, refer to the manufacturer's instruction manual for alternative methods of cooling.

(A) Caution: Do not cool the sample block below -38 °C, the freezing point of mercury.

(B) Caution: Acetone is extremely flammable. Keep away from heat, sparks, and flames and keep container closed when not actually pouring acetone. Use only in a well-ventilated area. Avoid inhalation and contact with the eyes or skin. Use cloth or leather gloves, goggles or safety shield, and keep dry ice in a canvas bag, especially when cracking.

(ii) For target temperature above ambient. Switch the instrument on and turn the coarse temperature control knob fully clockwise (full on) causing the indicator light to illuminate.⁶ When the thermometer indicates a temperature about 3 °C (5 °F) below the target (or specification) temperature, reduce the heat input to the sample cup by turning the coarse temperature control knob counter-clockwise to the desired control point (see §1500.43a(i)(1)). When the indicator light slowly cycles on and off read the temperature on the thermometer. If necessary, adjust the fine (center) temperature control knob to obtain the desired test (target) temperature. When the test temperature is reached and the indicator lamp slowly cycles on and off, prepare to introduce the sample.

(4) Charge the syringe with a 2-ml specimen of the sample⁷ to be tested; transfer the syringe to the filling orifice, taking care not to lose any sample; discharge the test specimen into the cup by fully depressing the syringe plunger, remove the syringe.

(5)(i) Set the timer⁸ by rotating its knob clockwise to its stop. Open the

⁶The target temperature may be attained by originally turning the coarse temperature control knob to the proper setting (see §1500.43a(h)(1) for the temperature desired rather than the maximum setting (full on). The elapsed time to reach the temperature will be greater, except for maximum temperature. However, less attention will be required during the intervening period.

⁷For target or expected temperatures below ambient, both syringe and sample must be precooled to cup temperature (see §1500.43a(i)(3)(i)) before the specimen is taken.

⁸For target temperatures below ambient, do not set the timer. Adjust the test flame

gas control valve and light the pilot and test flames. Adjust the test flame with the pinch valve to conform to the size of the 4-mm (5/32-in.) gage.

(ii) After the time signal indicates the specimen is at test temperature⁸, apply the test flame by slowly and uniformly opening the shutter and closing it completely over a period of approximately 2½ s.⁹ Watch closely for a flash at the cup openings.

(iii) The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the sample (see § 1500.43a(c)).

(6) Record the test results as “flash” or “no flash” and the test temperature.

(7) Turn off the pilot and test flames using the gas control valve. Remove the sample and clean the instrument. It may be necessary to allow the cup temperature to decline to a safe level before cleaning.

(j) *Test Method B—for determining Finite or Actual Flashpoint.* (1) Inspect the inside of the sample cup, lid, and shutter mechanism for cleanliness and freedom from contamination. Use an absorbent paper tissue to wipe clean, if necessary. Put cover in place and lock securely. The filling orifice may be conveniently cleaned with a pipe cleaner.

(2) For expected flashpoints below ambient. (i) The instrument power switch is to be in off position. Fill the refrigerant-charged cooling block with a suitable material.⁵ Raise the lid and shutter assembly, and position the base of the block in the sample cup, being careful not to injure or mar the cup. When the thermometer reaches a temperature 5 to 10 °C (10 to 20 °F) below the expected flashpoint, remove the cooling block and quickly dry the cup with a paper tissue to remove any moisture. Immediately close the lid and shutter assembly and secure. Prepare to introduce the sample using the syringe, both of which have been precooled to a temperature 5 to 10 °C

(10 to 20 °F) below the expected temperature (See § 1500.43a(j)(5)).

(ii) Caution: Do not cool the sample block below −38 °C, the freezing point of mercury.

(3) For tests where the expected flashpoint is above ambient. Turn the coarse temperature control knob fully clockwise (full on) causing the indicator light to illuminate. When the thermometer reaches a temperature 3 °C (5 °F) below the estimated flashpoint, turn the coarse temperature knob counter-clockwise to the dial reading representing the estimated flashpoint temperature as shown on the calibration curve (See § 1500.43a(h)(1)). When the indicator light slowly cycles on and off, read the temperature on the thermometer. If necessary, adjust the fine temperature control knob to obtain the exact desired temperature.

(4)(i) Charge the syringe⁷ with a 2 ml specimen of the sample⁷ to be tested; transfer the syringe to the filling orifice, taking care not to lose any sample; discharge the test specimen into the cup by fully depressing the syringe plunger; remove the syringe.

(ii) Set the timer¹⁰ by rotating its knob clockwise to its stop. Open the gas control valve and ignite the pilot and test flames. Adjust the test flame with the pinch valve to conform to the size of the 4-mm (5/32-in.) gage.

(iii) After the audible time signal indicates the specimen is at test temperature,¹⁰ apply the test flame by slowly and uniformly opening the shutter and then closing it completely over a period of approximately 2½ s. Watch closely for a flash at the cup opening.

(iv) The sample is deemed to have flashed only if a large flame appears and instantaneously propagates itself over the surface of the sample. (See § 1500.43a(c).)

(v) Turn off the pilot and test flames using the gas control valve. When the cup temperature declines to a safe

and allow the temperature to rise under ambient conditions until the target temperature is reached. Immediately apply the test flame as detailed.

⁹Never apply the test flame to the specimen more than once. Fresh portions of the sample must be used for each test.

¹⁰For expected flashpoint below ambient, do not set the timing device. Adjust the test flame. Allow the temperature to rise under ambient conditions until the temperature reaches 5 °C (9 °F) below the expected flashpoint. Immediately apply the test flame.

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level, remove the sample and clean the instrument.

(5)(i) If a flash was observed in § 1500.43a(j)(4)(iii) repeat the procedure given in § 1500.43a(j)(2) or (3), and in § 1500.43a(j)(4), testing a new specimen at a temperature 5 °C (9 °F) below that at which the flash was observed.

(ii) If necessary, repeat the procedure in § 1500.43a(j)(5)(i), lowering the temperature 5 °C (9 °F) each time, until no flash is observed.⁹

(iii) Proceed to § 1500.43a(j)(7).

(6)(i) If no flash was observed in § 1500.43a(j)(4)(iii) repeat the procedure given in § 1500.43a(j)(2) or (3), and in § 1500.43a(j)(4), testing a fresh specimen at a temperature 5 °C (9 °F) above that at which the specimen was tested in § 1500.43a(j)(4)(iii).

(ii) If necessary repeat the procedure in § 1500.43a(j)(6)(i), above, raising the temperature 5 °C (9 °F) each time until a flash is observed.⁹

(7) Having established a flash within two temperatures 5 °C (9 °F) apart, repeat the procedure at 1 °C (2 °F) intervals from the lower of the two temperatures until a flash is observed.⁹ Record the temperature of the test when this flash occurs as the flashpoint, allowing for any known thermometer correction. Record the barometric pressure.⁴

(8) The flashpoint determined in § 1500.43a(j)(7) will be to the nearest 1 °C (2 °F). If improved accuracy is desired (that is, to the nearest 0.5 °C (1 °F)), test a fresh specimen at a temperature 0.5 °C (1 °F) below that at which the flash was observed in § 1500.43a(j)(7). If no flash is observed, the temperature recorded in § 1500.43a(j)(7), is the flashpoint to the nearest 0.5 °C (1 °F). If a flash is observed at the lower temperature, record this latter temperature as the flashpoint.

(9) Turn off the pilot and test flames using the gas control valve. When the cup temperature declines to a safe level, remove the sample and clean the instrument.

(k) *Calculations.* If it is desired to correct the observed finite flashpoint for the effect of barometric pressure, proceed as follows: Observe and record the ambient barometric pressure⁴ at the time of the test. If the pressure differs

from 101.3 kPa (760 mm Hg), correct the flashpoint as follows:

(1) Corrected flashpoint (°C)=C+0.25 (101.3–A)

(2) Corrected flashpoint (°F)=F+0.06 (760–B)

(3) Corrected flashpoint (°C)=C+0.03 (760–B)

Where: F=Observed flashpoint, °F,

C=observed flashpoint, °C,

B=ambient barometric pressure, mm Hg; and

A=ambient barometric pressure, kPa.

(1) *Precision.* The precision of the method as determined by statistical examination of interlaboratory results is as follows:

(1) *Repeatability.* The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the values shown in table 2 only in 1 case in 20.

(2) *Reproducibility.* The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the values shown in table 2 only in 1 case in 20.

(m) *Flash Test Apparatus.* (1)(i) Unit consisting of an aluminum alloy or nonrusting metal block of suitable conductivity with a cylindrical depression, or sample cup, over which is fitted a cover. A thermometer is embedded in the block.

(ii) The cover is fitted with an opening slide and a device capable of inserting an ignition flame (diameter 4 ± 0.5 mm) into the well when the slide device shall intersect the plane of the underside of the cover. The cover is also provided with an orifice extending into the sample well for insertion of the test sample and also a suitable clamping device for securing the cover tightly to the metal block. The three openings in the cover shall be within the diameter of the sample well. When the slide is in the open position, the two openings in the slide shall coincide exactly with the two corresponding openings in the cover.

(iii) Electrical heaters are attached to the bottom of the cup in a manner

that provides efficient transfer of heat. An electronic heat control is required to hold the equilibrium temperature, in a draft-free area, within 0.1 °C (0.2 °F) for the low-temperature tester. A visual indicator lamp shows when energy is or is not being applied. Energy may be supplied from 120 or 240 V, 50 or 60 Hz main service.

(2)(i) Test flame and pilot flame-regulatable test flame, for dipping into the sample cup to try for flash, and a pilot flame, to maintain the test flame, are required. These flames may be fueled by piped gas service. A gage ring 4mm (5/32 in.) in diameter, engraved on the lid near the test flame, is required to ensure uniformity in the size of the test flame.

(ii) Caution: Never recharge the self-contained gas tank at elevated temperature, or with the pilot or test flames lighted, nor in the vicinity of other flames.

(iii) Audible Signal is required. The audible signal is given after 1 min in the case of the low-temperature tester.

(iv) Syringe. 2ml capacity, equipped with a needle suitable for use with the apparatus, adjusted to deliver 2.00±0.05 ml.

(3) Essential dimensions of the test apparatus are set forth in table 3.

(n) *Testing high-viscosity liquids.* (1) High-viscosity materials may be added to the cup by the following procedure:

(i) Back load a 5 or 10-ml syringe with the sample to be tested and extrude 2 ml into the cup. Spread the specimen as evenly as possible over the bottom of the cup.

(ii) If the sample cannot be loaded into a syringe and extruded, other means of adding the sample to the cup may be used such as a spoon. Add approximately 2 ml of material to the spoon and then push the material from the spoon into the cup.

(iii) If the test specimen does not close the sampling port in the cup, seal the cup externally by suitable means.

(2) Using the appropriate procedure, either Method A in §1500.43a(i) or Method B in §1500.43a(j), determine the flashpoint of the specimen which has been added to the tester in accordance with §1500.43a(n)(i), except that the time specified is increased from 1 to 5

minutes for samples at or above ambient temperature.

TABLE 1—CALIBRATION OF TESTER

Material	p-xylene ^A (Caution). ^B
Specific gravity, 15.6/15.6 °C (60/60 °F)	0.850 to 0.866.
Boiling range	2 °C maximum including 138.35 °C (281.03 °F).
Freezing point	11.23 °C (52.2 °F) minimum.
Flashpoint °C (acceptable range)	25.6±0.5 (78±1 °F).

^A Available as Flash Point Check Fluid (p-xylene) from Special Products Div., Phillips Petroleum Co., Drawer 'O,' Borger, Texas 79007.

^B Caution: Handle xylene with care. Avoid inhalation; use only in a well-ventilated area. Avoid prolonged or repeated contact with skin. Keep away from flames and heat, except as necessary for the actual flash point determination.

TABLE 2—REPEATABILITY AND REPRODUCIBILITY

Temperature, °C (°F)	Repeatability, °C (°F)	Reproducibility, °C (°F)
20(68)	0.5(0.9)	1.4(2.6)
70(158)	0.5(0.9)	2.9(5.3)
93(200)	1.3(2.3)	4.9(8.8)
150(300)	2.0(3.6)	7.5(13.5)
200(400)	2.6(4.7)	9.9(17.9)
260(500)	3.3(5.9)	12.4(22.3)

TABLE 3—ESSENTIAL DIMENSIONS OF FLASH TEST APPARATUS ^{A,B}

Sample Block	
Block diameter	61.5–62.5
Sample well diameter	49.40–49.70
Sample well depth	9.70–10.00
Top of block to center of thermometer hole	16.00–17.00
Diameter of thermometer hole (approx.)	7.0004
Cover	
Large opening length	12.42–12.47
Large opening width	10.13–10.18
Small opening length	5.05–5.10
Small opening width	7.60–7.65
Distance between extreme edges of small openings	48.37–48.32
Filling orifice diameter	4.00–4.50
Bore or filler tube	1.80–1.85
Maximum distance of filler tube from base of well with cover closed (max.)	0.75
Slide	
Large opening length	12.42–12.47
Large opening width	10.13–10.18
Small opening length	5.05–5.10
Small opening width	7.60–7.65
Near edge of large opening to end of slide	12.80–12.85
Extremes of large and small openings	30.40–30.45
Jet	
Length of jet	18.30–18.40
External diameter at end of jet	2.20–2.60
Bore of jet	1.60–1.65
Height of jet center above top surface of cover	11.00–11.20
Jet pivot to center of block with cover closed	12.68–12.72

^A The O-seal or gasket which provides a seal when the cover is shut, should be made of a heat-resistant material capable of withstanding temperatures up to 150 °C for the low-range apparatus.

^B When in position, the thermometer bulb should be surrounded with heat-conducting thermoplastic compound, such as a paste comprised of zinc oxide and mineral oil.

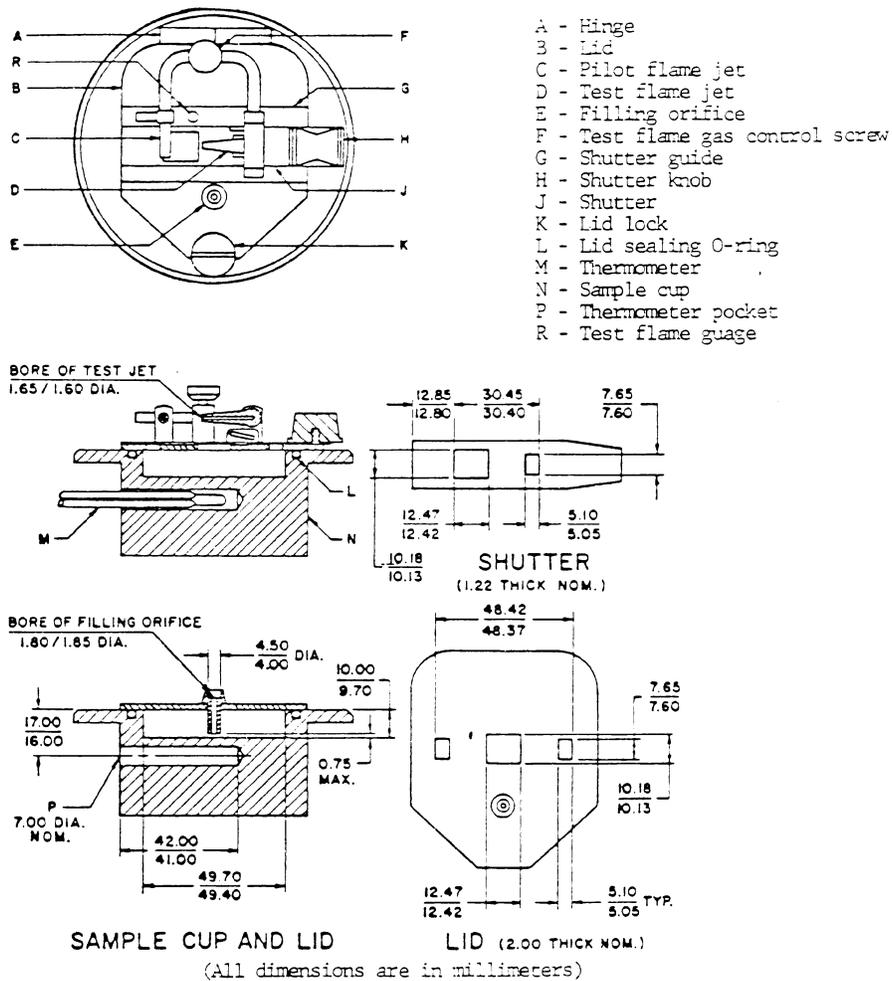


FIGURE 1 - Closed-cup tester

[51 FR 28539, Aug. 8, 1986]

§ 1500.44 Method for determining extremely flammable and flammable solids.

- (a) *Preparation of samples*—(1) *Granules, powders, and pastes.* Pack the sample into a flat, rectangular metal boat with inner dimensions 6 inches long × 1 inch wide × one-fourth inch deep.
- (2) *Rigid and pliable solids.* Measure the dimensions of the sample and sup-

port it by means of metal ringstands, clamps, rings, or other suitable devices as needed, so that the major axis is oriented horizontally and the maximum surface is freely exposed to the atmosphere.

(b) *Procedure.* Place the prepared sample in a draft-free area that can be ventilated and cleared after each test. The temperature of the sample at the