Designation: D8174 – 18

Standard Test Method for Finite Flash Point Determination of Liquid Wastes by Small-Scale Closed Cup Tester[1]

This standard is issued under the fixed designation D8174; 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

Flash point values are a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can, therefore, only be defined in terms of a standard test method, and no general valid correlation can be guaranteed between results obtained by different test methods or with different test apparatus.

This test method, and Test Method D8175, are used to determine the flash point of liquid wastes.

This procedure is primarily derived from Method B of Test Methods D3828 (EN ISO 3679 and IP 523) and is informally known in federal and other regulations as the Setaflash method.

1. Scope

1.1 This test method covers the procedure for a flash point test, within the range of –20 to 70 °C, of liquid wastes using a small-scale closed cup tester.

NOTE 1—Some apparatus are not designed for subambient temperature tests, so the testing range would be between 20 °C and 70 °C.

NOTE 2—This test method is not applicable for liquid waste that forms a surface film (see Test Method D8175 for Finite Flash Point Determination of Wastes by Pensky-Martens Closed Cup Tester).

1.2 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.3 This standard measures the ignitability properties of liquid wastes (which may be any discarded material), which may include secondary materials, off-specification products, and materials containing free liquids recovered during emergency response actions. Results from this test method may be used as part of a fire risk assessment of the material, but it is the responsibility of the user to perform any additional characterization needed for determination of storage, transport, treatment, or disposal per current regulations.

1.4 *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.* Warning statements appear throughout. See applicable Safety Data Sheets (SDS) for information about certified reference materials (CRMs) or secondary working standards (SWSs) that may be used in this test method. SDS may also be useful if some components of the waste sample are known.

1.5 *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]

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

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D7236 Test Method for Flash Point by Small Scale Closed Cup Tester (Ramp Method)

D8175 Test Method For Finite Flash Point Determination of Liquid Wastes by Pensky-Martens Closed Cup Tester

E502 Test Method for Selection and Use of ASTM Standards for the Determination of Flash Point of Chemicals by Closed Cup Methods

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

Current edition approved March 15, 2018. Published March 2018. Originally approved in 2018. DOI: 10.1520/D8174-18.

[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.

2.2 *ISO Standards:*[3]

ISO 17034 General Requirements for the Competence of Reference Material Producers

ISO Guide 35 Reference Materials—General and Statistical Principles for Certification

EN ISO 3679 Determination of Flash No-Flash and Flash Point—Rapid Equilibrium Closed Cup Method

ISO 60751 Industrial Platinum Resistance Thermometers and Platinum Temperature Sensors

2.3 *Energy Institute Standards:*[4]

IP 523 Determination of Flash Point—Rapid Equilibrium Closed Cup Method

IP 534 Determination of Flash Point—Small Scale Closed Cup Ramp Method

3. Terminology

3.1 *Definitions:*

3.1.1 *ambient barometric pressure, n—in waste flash point test methods,* the atmospheric pressure in the immediate surroundings where the flash point apparatus is located.

3.1.2 *ambient temperature, n—in waste flash point test methods,* the temperature in the immediate surroundings where the flash point apparatus is located.

3.1.3 *equilibrium, n—in waste flash point test methods,* the condition in which the vapor above the subsample and the subsample are at the same temperature at the time the ignition source is applied.

3.1.3.1 *Discussion*—This condition may not be fully achieved in practice since the temperature may not be uniform throughout the subsample, and the test cover and shutter on the apparatus can be cooler or warmer.

3.1.4 *flash point, n—in waste flash point test methods,* the lowest temperature of the subsample, adjusted to account for the variation in atmospheric pressure from 101.3 kPa, at which the application of an ignition source causes the vapors of the subsample to ignite under the specified conditions of the test.

3.1.5 *subambient temperature, n—in waste flash point test methods,* a lower temperature than the immediate surroundings where the flash point apparatus is located.

4. Summary of Test Method

4.1 A subsample is introduced into the test cup of the apparatus that is set and maintained at the expected flash point temperature. After a specified time, an ignition source is applied and a determination made as to whether or not a flash occurred. This procedure is repeated by changing the test temperature and subsample a number of times to determine the finite flash point.

4.2 If the expected flash point temperature is not known, then a screening procedure may be used whereby the temperature is increased while keeping the same subsample and the ignition source applied at intervals to establish an estimate of the flash point. An example of this procedure is given in Appendix X1.

5. Significance and Use

5.1 This procedure is intended to be used to evaluate the ignitability of liquid wastes.

5.2 Flash point measures the response of the subsample to heat and an ignition source under controlled laboratory conditions. It is only one of a number of properties that shall be considered in assessing the overall flammability hazard of a liquid waste material.

5.3 Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or nonflammable material.

5.4 This test method uses a small sample volume (2 mL) and short test time (1 min).

6. Interferences

6.1 Metals such as aluminum react with corrosive wastes to give off hydrogen gas, which can cause a false positive flash. For corrosive wastes, a cup with a stainless steel insert shall be used.

6.2 *Liquid Waste That Forms a Surface Film Under Test Conditions*—For these types of waste, Test Method D8175 shall be used. However, if the waste forms a surface film and is also corrosive, the small-scale tester with a stainless steel cup shall be used.

NOTE 3—If a regulatory decision point is being assessed for a waste that forms a surface film, a flash in the small-scale tester should be considered a maximum flash point and may not satisfy the regulatory assessment.

6.3 *Halogenated Constituents:*

6.3.1 The presence of some halogenated constituents in the waste may cause the flash to appear green instead of blue.

6.3.2 Some halogenated compounds can flash and some halogenated compounds only burn. The burning of a halogenated constituent within the waste should not be confused with a flash.

6.4 Because of the nature of the waste itself, the flash point results of the waste can be inconsistent (greater than reproducibility). An example would include volatile droplets suspended in a gel. Additional testing may be required to determine the lowest detectable flash point.

7. Apparatus

7.1 *Flash Point Tester*—The essential dimensions and requirements of the apparatus are shown in Fig. A1.1 and Table A1.1 of Annex A1. The apparatus and accessories are described in detail in Annex A1. The temperature range is from –20 to 70 °C. Some versions of the apparatus do not cover the full temperature range.

NOTE 4—It is possible that the lowest starting temperature of the apparatus is significantly higher than the actual detectable flash point of the sample. The conditions may exceed the upper explosive limit and result in a false negative.

[3] Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

[4] Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., http://www.energyinst.org.

7.2 *Barometer*—It should have an accuracy of 0.5 kPa or better. Barometers that have been pre-corrected for use at weather stations or airports are not suitable.

7.3 *Draft Shield*—A shield that is located at the back and on two sides of the instrument, for use in circumstances in which protection from drafts does not exist.

7.4 *Cooling Device*—A device used to lower the testing temperature of the sample cup. This can be integral to or independent from the flash point apparatus. See A1.2.3 and Annex A3.

8. Reagents and Materials

8.1 *Cleaning Solvent*—Use a solvent suitable for cleaning out the subsample from the test cup. Two commonly used solvents are toluene and acetone. **Warning—**Toluene, acetone, and many other solvents are flammable and a health hazard.

8.2 *Butane, Propane, and Natural Gas*—These are for use as a pilot and ignition source (not required if an electric ignitor is used). **Warning—**Butane, propane, and natural gases are flammable and a health hazard.

8.3 *Heat Transfer Paste*—This is used to make direct contact between the cup and temperature measuring device.

8.4 *Reference Materials (CRMs)*—See Annex A2.

9. Sampling

9.1 Sampling should address the intended use of the analysis, such as representative subsampling from the source waste material. When possible, obtain at least a 50-mL sample from the source waste material for single-phase wastes. If multiple-phase waste is suspected, obtain a greater volume of sample to ensure adequate phase volumes for testing. A 2-mL subsample will be used for each step in a finite flash point determination.

9.2 Samples that have multiple phases (layers) shall be phase separated into single phases. Each separate liquid phase is tested.

9.3 Store samples in clean, tightly sealed containers at normal room temperature (20 to 25 °C) or colder. Avoid freezing the sample. If the sample is frozen, allow the sample to warm such that it is a liquid. Avoid storage of samples at temperatures in excess of 20 °C. Do not store samples for an extended period of time in gas-permeable containers, such as some types of plastic, because volatile material can diffuse through the walls of the container. Samples in leaky containers are suspect, as volatiles can be lost and may not provide valid results.

NOTE 5—If a regulatory decision point is being assessed for a waste in a leaky container, the lack of a flash point or a flash point exceeding the regulatory decision point may not satisfy the regulatory assessment.

9.4 Erroneously high flash points can be obtained when precautions are not taken to avoid loss of volatile materials. Do not open containers unnecessarily.

10. Preparation of Apparatus

10.1 Place the apparatus on a level, stable surface. Unless tests are made in a draft-free area, surround the tester on three sides with a draft shield (see 7.3) for protection. Do not rely on tests made in a laboratory ventilation hood unless the extracted air and vapors can be withdrawn without causing air currents over the test cup during the ignition source application period.

10.2 Read the manufacturer's instructions on the care and servicing of the instrument and for the correct operation of any controls.

10.3 Prepare the apparatus for operation in accordance with the manufacturer's instructions for calibrating, checking, and operating the equipment, especially the operation of the ignition source. **Warning—**An incorrectly set test flame size or incorrect setting for an electric ignitor can significantly affect the test result.

10.4 Clean the test cup, cover, and its accessories with an appropriate solvent (8.1) to remove any traces of gum or residue from the previous test. Wipe dry with absorbent paper. A stream of dry, clean air may be used to remove the last traces of solvent used. A pipe cleaner may be used to clean the filler orifice. Dispose of solvents and waste material in accordance with local regulations.

10.5 If not automatically recorded by the instrument, measure and record the ambient barometric pressure at the time of each test.

10.6 An electronic thermal flash detector (A1.6) may be used in lieu of a visual observation of the flash. In cases of dispute, the visual observation shall be used.

10.7 For subambient test temperatures (typically –20 to 20 °C), see Annex A3, unless the apparatus has integral test cup cooling.

11. Verification of Apparatus

11.1 Verify and correct, if necessary, the readings on the temperature measuring device (A1.8) at least every twelve months according to the manufacturer's instructions, and that the temperature measuring device is in accordance with A1.2.3 and Annex A4.

11.2 Verify the correct operation of the apparatus and associated components (barometer, temperature measuring device, cooling device, and so forth) before initial use. Follow the manufacturer's recommendations.

11.3 Verify the performance of the apparatus before initial use and at least once per year by determining the flash point of a CRM. Examples of suitable liquids, and their approximate flash points, are listed in Annex A2. Use a CRM that has a flash point that is reasonably close to the expected temperature range of the samples to be tested. The flash point of the reference material shall be tested in accordance with Sections 12 and 13. The flash point obtained shall be within the limits stated in Table A2.1 for the identified CRM or within the limits calculated for an unlisted CRM (Annex A2).

11.4 Once the performance of the apparatus has been verified, the flash point of secondary working standards (SWSs) can be determined along with their control limits. These secondary materials can then be used for more frequent performance checks (Annex A2).

11.5 When the flash point result obtained from 11.3 or 11.4 is not within accepted limits, check the condition and operation of the apparatus to ensure conformity with the details listed in Annex A1. Check the tightness of the cover (A1.2.2); the action of the shutter; the size, intensity, and position of the ignition source (A1.3); and the operation of the flash detector (if fitted). Check for an adequate amount of heat transfer paste around the temperature measuring device and the reading of the temperature measuring device. Additionally, ensure that the barometric pressure measurements are accurate, that there is adequate shielding of the apparatus from drafts, and that calculations are accurate. After any adjustment, repeat the test in 11.3 or 11.4 using a fresh subsample.

12. Procedure

12.1 Inspect the cup and cover for cleanliness and correct operation, especially with regard to tightness of the cover (A1.2.2), the action of the shutter, and the size, intensity, and position of the ignition source (A1.3). Clean and adjust if necessary (10.4). Put the cover in place and close securely.

12.2 Set the cup temperature to the expected flash point for the sample, following the manufacturer's instructions.

12.2.1 If the expected flash point of the sample is not known, make an estimate of the expected flash point. An example of a suitable procedure to estimate the expected flash point can be found in Appendix X1.

12.2.2 When the cup is at the test temperature, fill the syringe with the sample to be tested; discharge the subsample into the cup through the filling port by fully depressing the syringe plunger. If necessary, the subsample may be added directly into the cup, and then ensure the cover is locked tightly in place. If the subsample is too viscous to get into a syringe, a scoop may be used to transfer approximately 2 mL to the cup. The subsample size may be the mass equivalent of the required volume. Ensure the subsample is evenly spread within the cup.

12.2.3 Start the timer for a 1-min thermal equilibrium period; if applicable, light the pilot light and adjust the flame to conform to the 4-mm gauge.

12.2.4 At the end of the 1-min thermal equilibrium period, apply the ignition source by slowly and uniformly opening the shutter and closing it completely over a period of 2½ s. Watch closely for a flash in a test cup opening if an automatic flash detector is not used.

12.2.5 The subsample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the subsample. Occasionally, particularly near the actual flash point, the application of the ignition source can cause a blue halo or an enlarged flame; this is not a flash and should be ignored.

12.2.6 If a flash is detected, repeat the procedure given in 12.2.2 – 12.2.5, testing a subsample at a temperature 5 °C lower each time until no flash is detected, then proceed to 12.2.8.

12.2.7 If no flash was detected, repeat the procedure given in 12.2.2 – 12.2.5, testing a fresh specimen at a temperature 5 °C higher each time until a flash is detected. If no flash is detected up to 70 °C, stop the test and report as no flash.

12.2.8 Having established a flash within two temperatures 5 °C apart, repeat the procedure at 1 °C intervals from the lower of the two temperatures until a flash is detected.

12.2.9 Record the temperature of the test when this flash occurs as the detected flash point, allowing for any known thermometer correction.

12.2.10 The flash point determined in 12.2.8 will be to the nearest 1 °C.

12.2.10.1 If improved accuracy is desired (that is, to the nearest 0.5 °C), test a fresh subsample at a temperature 0.5 °C below that at which the flash was detected in 12.2.8. If no flash is detected, the temperature recorded in 12.2.8 is the flash point to the nearest 0.5 °C. If a flash is detected at the lower temperature (12.2.8), record this lower temperature as the flash point.

12.2.11 Record whether there was a flash or no flash, the test temperature, and the ambient barometric pressure.

12.2.12 If applicable, turn off the pilot and test flame. Remove the subsample and clean the cup and cover. **Warning—**Allow the cup temperature to fall to a safe level before cleaning.

12.2.13 Repeat the test with one or more subsamples at the detected flash point temperature.

12.2.13.1 Correct each result for barometric pressure, and average the corrected results for the sample.

12.2.13.2 Two detected results obtained within the repeatability precision in Section 15 are acceptable. If results between two or more tests are inconsistent, then use the lower temperature in place of the mean corrected flash point temperature.

13. Calculation

13.1 *Pressure Correction Calculation*—If the ambient barometric pressure (10.5) differs from 101.3 kPa, correct the detected flash point using Eq 1 as follows:

$$\text{Corrected Flash Point} = C + 0.25(101.3 - A) \quad (1)$$

where:

C = detected flash point, °C, and
A = ambient barometric pressure, kPa.

13.2 *Average Corrected Finite Flash Point Result Calculation*—Average two or more corrected results for the final reported flash point of a sample:

$$\text{Average Corrected Finite FP Result: } \bar{x} = \frac{1}{n}(x_1 + \ldots + x_n) \quad (2)$$

where:

n = number of subsample tests,
$\bar{x}$ = average finite flash point result, and
x_n = corrected subsample flash point result.

NOTE 6—Supplemental information for non-SI units can be found in Appendix X4.

14. Report

14.1 Report the corrected finite flash point average rounded to the nearest 1 or 0.5 °C.

14.1.1 If required, convert the flash point to degrees Fahrenheit using Eq 3:

$$\text{Corrected Flash Point, in Fahrenheit} = ((\text{Corrected Flash Point, in Celcius}) \times 1.8) + 32 \quad (3)$$

14.2 Report the test method used, the identification of the material tested, the test date, and any deviation, by agreement or not, from the procedure specified in Section 12.

14.3 Because of the nature of some waste materials, it may be difficult to determine two or more detected/corrected flash points within repeatability. If the waste flashes over a range of temperatures, the lowest flash point shall be reported (see 12.2.13.2). Additionally, if adequate for the use of the data, a "less than" result may be reported.

15. Precision and Bias

15.1 *Precision*—It is not possible to specify the precision of the procedure in this test method for measuring flash point because, by its nature, every sample of liquid waste is compositionally unique and therefore it is not meaningful to provide a precision statement. Limited work to demonstrate repeatability precision for some surrogate liquid wastes is shown in Appendix X5.

15.2 *Bias*—The procedure in this test method has no bias because flash point can be defined only in terms of this test method.

16. Keywords

16.1 combustible; fire risk; flammable; flash point; ignitability; liquid wastes; volatile

ANNEXES

(Mandatory Information)

A1. FLASH TEST APPARATUS

A1.1 Flash Point Test Apparatus

A1.1.1 See Figs. A1.1 and A1.2[5] and Table A1.1.

A1.2 Test Cup

A1.2.1 Consisting of an aluminum or non-rusting metal block of suitable conductivity with a cylindrical depression over which a cover is fitted. A temperature measuring device is inserted in the block. A stainless steel cup insert may be fitted into the metal block to afford protection from corrosive wastes; in this case, the final dimensions, including the stainless steel cup insert, shall comply with Fig. A1.1.

A1.2.2 The cover is comprised of a lid fitted with an opening shutter and a device capable of inserting an ignition source into the test cup when the shutter is open. The cover is also provided with an orifice extending into the sample well for insertion of the subsample, 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 shutter is in the open position, the two openings in the shutter shall coincide with the two corresponding openings in the cover.

A1.2.3 Electrical heaters are attached to the test cup in a manner that provides efficient transfer of heat. The heater control shall be capable of controlling the test cup temperature, as measured by the temperature measuring device and in a draft-free area, to within ±0.5 °C for test temperatures up to 70 °C during the test. Cooling of the test cup may use Peltier effect devices, an external cryostat, or a cooling insert (see Annex A3).

A1.3 Ignition Source

A1.3.1 For dipping into the test cup to test for a flash.

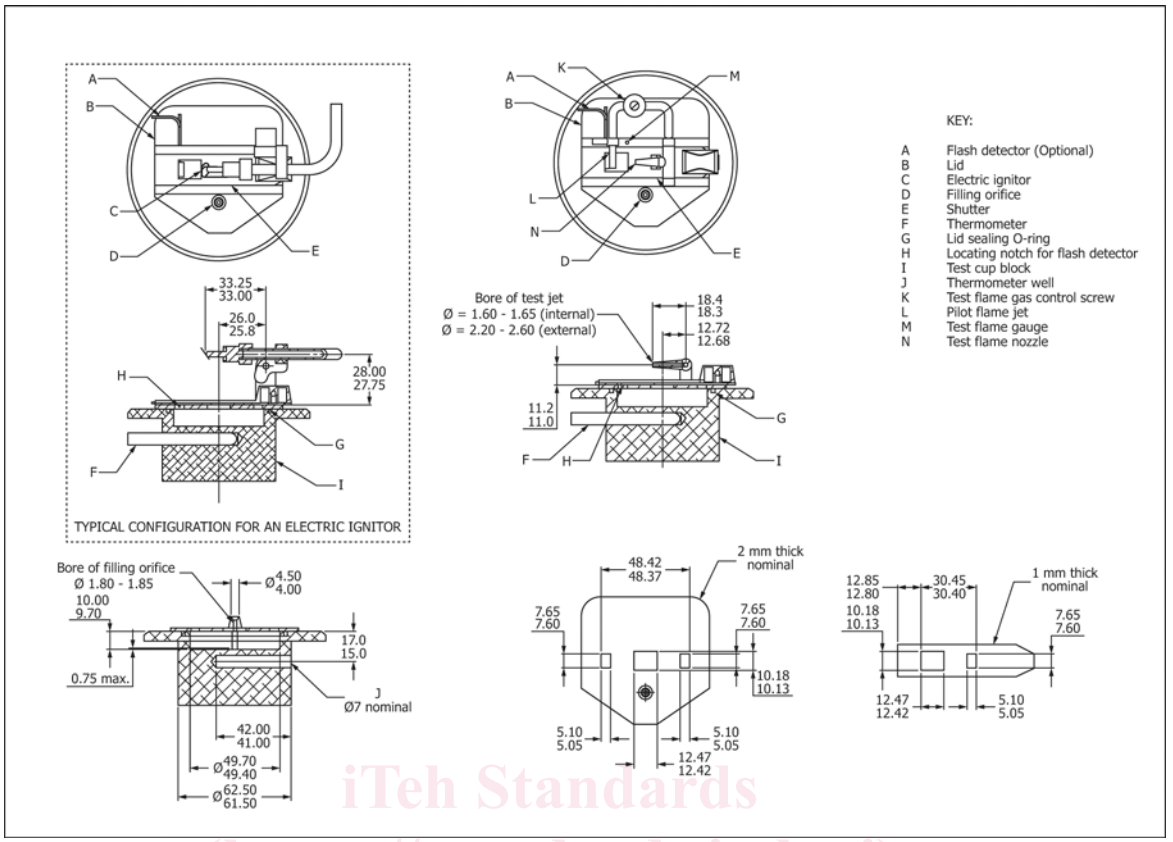
A1.3.2 *Test Flame and Pilot*—A test flame with a suitable mechanism for dipping into the test cup to test for a flash, and a pilot to maintain the test flame, are both required for this ignition source. When inserted, the nozzle of the ignition source shall intersect the plane of the underside of the cover. The flame may be fueled from an external butane or propane supply[6] or a self-contained or attached tank of butane or propane. A gauge ring 4 mm in diameter, engraved on the cover near the test flame, aids uniformity in the size of the test flame. **Warning—**Never recharge or change the self-contained gas tank at elevated temperatures, or with the pilot or test flames lighted, nor in the vicinity of other flames.

A1.3.3 *Electronic Ignitor*—An electric ignitor with a suitable mechanism for dipping into the test cup to test for a flash and a screen (A1.9). The electric ignitor shall be of the electric resistance (hot wire) type and shall position the heated section horizontally and intersect the underside of the cover. Follow the manufacturer's instructions for ensuring the correct operation of the ignitor. In the event of a dispute, the gas ignition source is the referee.

A1.4 **Audible Signal**—Indicates when to dip the ignition source into the cup.

[5] The instrument shown, and any likeness to commercial products, are for informational purposes only. The U.S. Environmental Protection Agency (EPA) does not endorse any particular product for this purpose.

[6] External fuel adapters are available from instrument sources.



NOTE 1—All dimensions are in millimetres.

FIG. A1.1 Test Cup and Cover Assembly

FIG. A1.2 Typical Flash Point Test Apparatus

A1.5 **Syringe**—Equipped with a nozzle suitable for use with the apparatus, adjusted to deliver 2.00 ± 0.05 mL.

A1.6 **Flash Detector (Optional)**—A low-mass thermocouple device for the detection of the flash point. A flash is indicated if a temperature rise of 6.0 °C is detected within 100 ms.

A1.7 **Timing Device**—An electronic timer.

A1.8 **Temperature Measuring Device**—An electronic temperature measuring device or a liquid-in-glass thermometer with an accuracy of better than 0.5 up to 70.0 °C (Annex A4).

A1.9 **Electric Ignitor Screen**—A metal screen to optically screen the ignitor from the operator. Only required when an electric ignitor is used.