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Standard Test Method for
Temperature Limit of Flammability of Chemicals[1]

This standard is issued under the fixed designation E1232; 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

The temperature limit of flammability test measures the minimum temperature at which liquid (or solid) chemicals evolve sufficient vapors to form a flammable mixture with air under equilibrium conditions. This temperature is applicable for assessing flammability in large process vessels and similar equipment (Appendix X1 and Appendix X2).

1. Scope

1.1 This test method covers the determination of the minimum temperature at which vapors in equilibrium with a liquid (or solid) chemical will be sufficiently concentrated to form flammable mixtures in air at atmospheric pressure. This test method is written specifically for determination of the temperature limit of flammability of systems using air as the source of oxidant and diluent. It may also be used for other oxidant/diluent combinations, including air plus diluent mixtures; however, no oxidant/diluent combination stronger than air should be used. Also, no unstable chemical capable of explosive decomposition reactions should be tested (see 8.3).

1.2 This test method is designed and written to be run at local ambient pressure and is limited to a maximum initial pressure of 1 atm abs. It may also be used for reduced pressures with the practical lower pressure limit being approximately 13.3 kPa (100 mm Hg). The maximum practical operating temperature of this equipment is approximately 150°C (302°F) (Note A1.2).

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units are provided for information only and are not considered standard.

1.4 This standard 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, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific safety precautions are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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
D3941 Test Method for Flash Point by the Equilibrium Method With a Closed-Cup Apparatus
E220 Test Method for Calibration of Thermocouples By Comparison Techniques
E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples
E502 Test Method for Selection and Use of ASTM Standards for the Determination of Flash Point of Chemicals by Closed Cup Methods
E537 Test Method for The Thermal Stability of Chemicals by Differential Scanning Calorimetry
E681 Test Method for Concentration Limits of Flammability of Chemicals (Vapors and Gases)
E698 Test Method for Arrhenius Kinetic Constants for Thermally Unstable Materials Using Differential Scanning Calorimetry and the Flynn/Wall/Ozawa Method

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[1] This test method is under the 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.

2.2 *ANSI Standard:*[3]

ANSI-MC96.1 Temperature Measurement Thermocouples

2.3 *NFPA Standard:*[4]

NFPA 325 Fire Hazardous Properties Liquids

3. Terminology

3.1 *Definitions:*

3.1.1 *flash point*—the lowest temperature, corrected to a pressure of 101.3 kPa (760 mm Hg, 1013 mbar), at which application of an ignition source causes the vapors of the specimen to ignite under specified conditions of test.

3.1.2 *lower limit of flammability or lower flammable limit, (LFL)*—the minimum concentration of a combustible substance that is capable of propagating a flame through a homogeneous mixture of the combustible and a gaseous oxidizer under the specified conditions of test.

3.1.3 *lower temperature limit of flammability, (LTL)*—the lowest temperature, corrected to a pressure of 101.3 kPa (760 mm Hg, 1013 mbar), at which application of an ignition source causes a homogeneous mixture of a gaseous oxidizer and vapors in equilibrium with a liquid (or solid) specimen to ignite and propagate a flame away from the ignition source under the specified conditions of test.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *propagation of flame*—the upward and outward movement of the flame front from the ignition source to the vessel walls, that is determined by visual observation.

4. Summary of Test Method

4.1 A pool of liquid is stirred in a closed vessel in an air atmosphere. The vapor-air mixture above this liquid is exposed to an ignition source and the upward and outward propagation of flame away from the ignition source is noted by visual observation. Temperature in the test vessel is varied between trials until the minimum temperature at which flame will propagate away from the ignition source is determined.

5. Significance and Use

5.1 The lower temperature limit of flammability is the minimum temperature at which a liquid (or solid) chemical will evolve sufficient vapors to form a flammable mixture with air under equilibrium conditions. Knowledge of this temperature is important in determining guidelines for the safe handling of chemicals, particularly in closed process and storage vessels.

NOTE 1—As a result of physical factors inherent in flash point apparatus and procedures, closed-cup flash point temperatures are not necessarily the minimum temperature at which a chemical will evolve flammable vapors (see Appendix X2 and Appendix X3, taken in part from Test Method E502). The temperature limit of flammability test is designed to supplement limitations inherent in flash point tests (Appendix X2). It yields a result closely approaching the minimum temperature of flammable vapor formation for equilibrium situations in the chemical processing industry such as in closed process and storage vessels.

NOTE 2—As a result of flame quenching effects existing when testing in standard closed-cup flash point apparatus, there are certain chemicals that exhibit no flash point but do evolve vapors that will propagate a flame in vessels of adequate size (X3.2). 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 (6.2).

NOTE 3—The lower temperature limit of flammability (LTL) is only one of several characteristics that should be evaluated to determine the safety of a specific material for a specific application. For example, some materials are found to have an LTL by this test method when, in fact, other characteristics such as minimum ignition energy and heat of combustion should also be considered in an overall flammability evaluation.

5.2 The vapor concentration present at the lower temperature limit of flammability equals the lower flammable limit concentration as measured by Test Method E681 and extrapolated back to the same temperature. (This permits estimation of lower temperature limits of flammability if vapor pressure and concentration limit of flammability data are available (A2.3). A comparison of results of the tests, thus, affords a check on test reliability, the reliability of vapor pressure data, or both.)

6. Interferences

6.1 This test method is not applicable to materials that undergo chemical changes when mixed with air. Examples include, but are not limited to, oxidation and polymerization.

6.2 Measured temperature limits are influenced by flame quenching effects of the test vessel walls. The test vessel employed in this test method is of sufficient size to eliminate these effects for most materials. For certain amines, halogenated materials, etc., that have large ignition-quenching distances, tests should be conducted in vessels with larger diameters than the one listed in this test method (A1.1). Quenching effects become increasingly significant as the test pressure decreases.

6.3 Measured temperature limits of flammability of chemicals can be greatly influenced, as are flash points, by the presence of various impurities or known mixture components. Small quantities of volatile flammable impurities can reduce temperature limit values, and volatile inert diluents can raise temperature limit values or produce complete inerting. (See 8.2.3 and Annex A3 for a discussion of mixture testing.)

7. Apparatus

7.1 Fig. 1 is a schematic diagram of the apparatus; details and dimensions are presented in Annex A1. The apparatus consists of the following:

7.1.1 *Glass Test Vessel,*

7.1.2 *Insulated Chamber,* equipped with a source of controlled-temperature air,

7.1.3 *Ignition Device,* with an appropriate power supply, and

7.1.4 *Magnetic Stirrer and Cover,* equipped with the necessary operating connections and components.

8. Hazards

8.1 *Tests should not be conducted in this apparatus with gaseous oxidants stronger than air* since explosive violence increases as oxidizer strength increases. Do not use oxygen, nitrous oxide, nitrogen dioxide, chlorine, etc. in this glass apparatus.

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[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] Available from National Fire Protection Association (NFPA), 1 Batterymarch Park, Quincy, MA 02169-7471, http://www.nfpa.org.

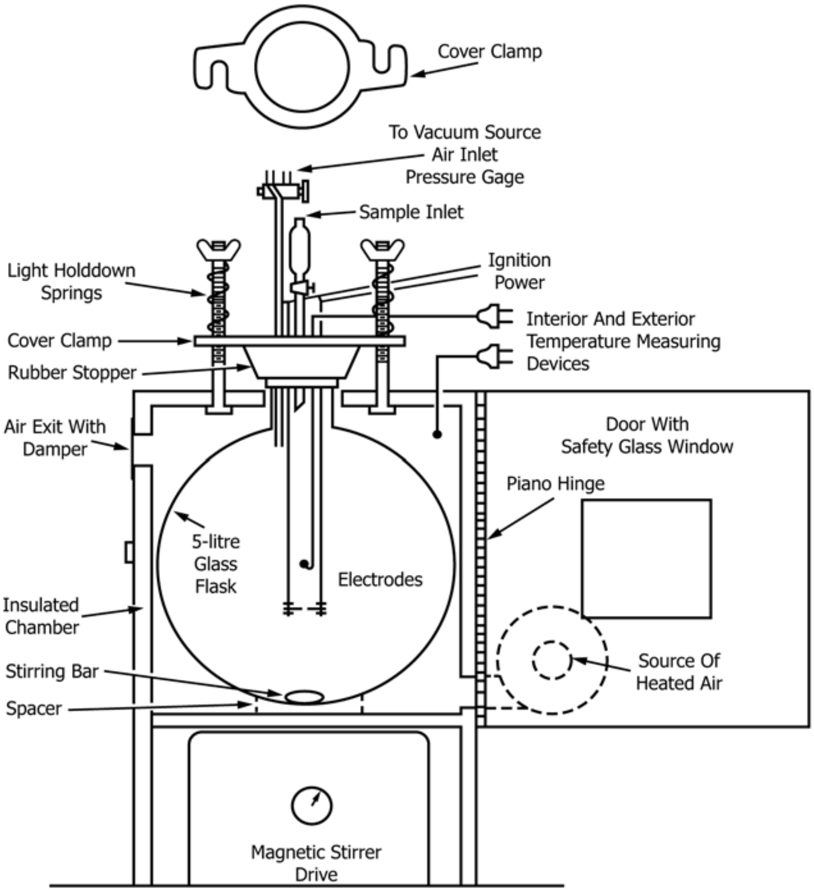

FIG. 1 Schematic Diagram of Test Apparatus

8.2 *Adequate shielding must be provided* to prevent injury in the event of equipment rupture, due to both implosions and explosions. A metal enclosure such as that recommended in A1.2 is one method suitable for this purpose.

8.2.1 Implosion of the test vessel at high vacuum levels is possible and, therefore, all evacuations must be made with the required shielding to protect against flying fragments.

8.2.2 Energetic explosions may be produced if tests are made at temperatures above the LTL. The determination of the LTL should always be initiated at a temperature below the estimated LTL, and successive ignition trials made at intervals of not more than a 2°C temperature increase. Methods for estimating initial test temperatures, discussed in Annex A2, should be employed to ensure that initial trials are conducted at temperatures less than the LTL (Note 4). The glass test vessel, equipped with a lightly held or loose cover, vents most explosions adequately. Nevertheless, shielding is required to protect against any possibility of test vessel rupture.

8.2.3 The testing of materials that are reactive with the metal parts of the apparatus can effect results, and may cause energetic explosions. For example, acids and alkaline materials can generate hydrogen gas. When testing such materials, variable results due to the generation of hydrogen may be detected by varying the holding time of several trials at a specific temperature. If corrosion occurs, materials of construction should be changed to corrosion resistant types.

8.2.4 Testing should be carried out in a manner that prevents accidental activation of the ignition source at incorrect stages of the procedure.

8.3 Tests should not be conducted on peroxides, monopropellants, or other thermally unstable materials that might undergo explosive gas or liquid phase decomposition reactions. For example, some monomers may undergo energetic vapor phase polymerization reactions. For information on evaluating the thermal stability of proposed test materials, see DS-51A, and Test Methods E537 and E698.

8.4 Tests should be conducted in a fume hood or other ventilated area to prevent exposure of personnel to toxic chemicals or combustion products.

8.5 Precautions must be taken to ensure that the high voltage spark ignition source is always adequately insulated from other electrical circuits and metal parts of the apparatus, fume hood, etc. to prevent electrical hazards to personnel and instrumentation. Careful attention to electrical insulation integrity plus the use of disconnection procedures are required to achieve a satisfactory protection against electrical hazards.

9. Calibration

9.1 System temperature and pressure and barometric pressure measuring devices must be calibrated against adequate standards. For information on calibration of thermocouples, see

Test Method E220, Specification E230, and ANSI-MC96.1. The pressure sensing devices should be calibrated against a traceable standard such as a primary standard piston gage, commonly called a dead weight gage.

10. Procedures

10.1 *Lower Temperature Limit of Flammability Test:*

10.1.1 Assemble the equipment, as shown in Fig. 1, within an appropriate fume hood or other ventilated area and secure the door of the metal enclosure. Clean and dry the test vessel and all components. Evacuate the system and flush with air, or other specified test gas, sufficiently to ensure removal of residual volatile materials that may be present as a result of cleaning or prior tests.

10.1.2 Based on methods given in Annex A2, adjust the flask to the desired test temperature below the anticipated lower temperature limit of flammability.

NOTE 4—A prudent operator will use a wide safety factor in choosing initial test temperatures (6.3). This may necessitate a few additional trials but will provide increased safety for the operation.

10.1.3 It may be necessary to separately heat, insulate, or heat and insulate cover components and lines, to prevent vapor condensation at cool sites within the vapor space. The liquid, mist, or both, that may otherwise be formed can cause erroneous results.

10.1.4 Make certain that all safety precautions have been taken.

10.2 *Sample Introduction of Liquids:*

10.2.1 Introduce 50 cm^3 of liquid to the flask using a separatory funnel or other inlet device.

NOTE 5—The 50 cm^3 of liquid provide substantially more than theoretically required. Smaller sample sizes are adequate for pure chemicals and larger sample sizes may be required for mixtures (Annex A3).

10.2.2 Turn on the stirrer at a speed of approximately 400 rpm.

10.2.3 Close the hood door. (Cover hold-down devices should be loose).

10.2.4 Stir for at least 5 min after attainment of thermal equilibrium. Slower stirrer speeds, longer mix times, or both, may be required for viscous materials. Observe results obtained with different mixing times and speeds, at constant temperature, as a check to ensure that complete mixing and thermal equilibrium are being achieved without generation of mist. If a visible mist is generated, decrease stirring speed until it is eliminated.

NOTE 6—If mixing is inadequate, vapor concentrations can vary throughout the flask, and inconsistent results will be obtained. Some regions may contain insufficient fuel to propagate a flame at temperatures above the true equilibrium flammable limit temperature.

10.2.5 Turn off the stirrer.

10.2.6 Record the test temperature and system pressure (usually barometric pressure unless system is being operated at sub-ambient pressure).

10.2.7 Disconnect instrumentation lines as required and connect the ignition wires.

10.2.8 Check for liquid condensation or mist in the vapor regions of the flask. Heat, insulate, or both, to prevent condensation and then repeat the test (10.3).

NOTE 7—Although this test method is intended to be applied to vapor situations only, it is theoretically possible to generate mist in some situations. Any mist tends to give a more conservative (lower) temperature limit.

10.2.9 Darken the viewing area. Activate the ignition source. Observe for ignition and flame propagation away from the ignition source. At each test temperature record any occurrence of flame propagation.

NOTE 8—It is recommended that the ignition source not be activated until 30 s after the stirrer is turned off to allow the mixture to become quiescent. However, to prevent stratification activate the ignition source within 60 s.

NOTE 9—At concentrations just outside the flammable range a small cap of flame will be visible above the arc position. Absence of a flame cap may be an indication of insufficient ignition energy. The onset of spherical, upward, and partial outward flame propagation signifies a limit or near-limit temperature. It is suggested that detailed observations of flame behavior be recorded on all trials. Include such notes as flame cap, no flame cap, upward and outward propagation, downward propagation, etc. These observations can serve as a guide to narrowing the region of uncertainty between go and no-go trials.

10.2.10 Flush the test vessel sufficiently with air, or other specified test gas, to remove possible decomposition, combustion products, or both.

10.2.11 Raise the vessel temperature in steps no greater than 2°C to find the minimum temperature, T_1, that gives flame propagation and the maximum temperature, T_2, below T_1, that does not give flame propagation. (The difference between T_1 and T_2 is a measure of the variability of the procedure for the material being studied (Note 14.))

10.2.12 Conduct several preliminary trials on a given liquid charge. It is necessary to remove the vessel for periodic cleaning and recharging with liquid.

10.2.13 Each final trial should be in a clean vessel using a fresh sample.

NOTE 10—Ignition failures and inconsistent performance are occasionally encountered when, for example, electrically highly conductive or insulating materials, or materials having a very high ignition energy, are tested using the spark ignition source. Activate the spark ignition source in air to determine whether the equipment or material under test is causing performance problems. Limits for materials causing inconsistent spark performance should be determined using a fuse wire ignition source. Fuse wire ignition should also be used to confirm results if temperature limits are conducted at reduced pressure.

10.2.14 Record the values of the test temperatures, T_1 and T_2, and the test pressure (barometric pressure in most situations) in the vessel.

10.3 *Sample Introduction of a Solid:*

10.3.1 As with liquids, place 50 cm^3 of the solid in the flask.

NOTE 11—This technique is only suitable for powdered or small crystalline solids.

10.3.2 Add chemicals having melting points above room temperature to the test vessel as solids. If the chemical melts at the test temperature, the procedure is identical to that given in 10.5.

10.3.3 An occasional solid will sublime sufficiently to have a temperature limit of flammability while still solid. These materials are tested by the same techniques as liquids. However, some difficulty can be encountered with stirring. Employ reduced stirring speeds and longer holding times for attainment of equilibrium.

11. Calculation

11.1 Calculate ltl, the uncorrected temperature limit of flammability, using Eq 1. Correct this limit to LTL at standard atmospheric pressure, 101.3 kPa (760 mm Hg), using either Eq 2 or Eq 3.

$$\mathrm{ltl} = 1/2\left(T_1 + T_2\right) \tag{1}$$

$$LTL = \mathrm{ltl} + 0.25\left(101.3 - p\right) \tag{2}$$

where:

p = absolute initial pressure in the vessel in kPa.

$$LTL = \mathrm{ltl} + 0.03\left(760 - P\right) \tag{3}$$

where:

P = absolute initial pressure in the vessel in mm Hg.

All temperatures are in degrees Celsius.

NOTE 12—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.

NOTE 13—The barometric correction in Eq 2 and Eq 3 is an approximation based on a material of average lower flammable limit, having a vapor pressure of average slope. Theoretically, a separate barometric adjustment would be required for each material; however, the approximation in Eq 2 and Eq 3 is adequate for most cases. For non-standard materials, for temperature limit measurements made at high altitudes (Denver, for example), or for data being used to evaluate hazards at high altitudes, corrections might better be based on the actual vapor pressure data of the material in question.

12. Report

12.1 The report shall include the following:

12.1.1 Temperature limit, LTL, to the nearest 1°C (2°F); report T_1, T_2, and the test pressure,

12.1.2 Ignition source used,

12.1.3 Date,

12.1.4 Purity of the material, if known, and any special sample preparation,

12.1.5 Type and concentration of oxidant and diluent if other than air,

12.1.6 Deviations made from the procedure as written in this method, for example, vessel size or ignition source, and

12.1.7 For those samples tested that do not exhibit the presence of sufficient vapors to form flammable mixtures with air, the report shall state either *no flame propagation to boiling* or *no flame propagation in tests from __°C to __°C by ASTM Test Method E1232.*

13. Precision and Bias

13.1 An interlaboratory study of the repeatability and reproducibility of this test method has not been carried out. However, a single laboratory repeatability study is available. Duplicate or triplicate test were performed over a 12 year period by different operators to determine the LTL of 14 substances and UTL of 13 substances. The maximum LTL deviation has been found to be bounded by the following formulae:

$$Abs\left[Max\ Dev\ (C)\right] = 2.2\ 10^{-5} * \left[LTL\ (deg.\ R)\right]^2$$

NOTE 14—Generally a clear-cut change from no flame propagation to flame propagation occurs over a 3°C (5°F), or less, temperature range for lower limit tests at 38°C (100°F) and over a 6°C (10°F), or less, temperature range for tests at 93°C (200°F).

13.2 An analysis of a subset (LTL of 7 substances and UTL of 4 substances) of the data covering limit temperatures in the range from 42°F to 359°F indicates that there is no discernible bias between the test results and the theoretical predictions using the vapor pressure data and limit concentration.

13.3 A report including the data and analysis is available from ASTM Headquarters.[5]

ANNEXES

(Mandatory Information)

A1. DIMENSIONS AND SPECIFICATIONS OF APPARATUS (Fig. 1)

A1.1 *Test Vessel*—The test vessel shall be a borosilicate glass boiling flask, short-ring neck, 5000 cm^3 capacity, approximately 222 mm (8¾ in.) in diameter and 305 mm (12 in.) in height.

A1.2 *Insulated Chamber*—The dimensions shall be as follows:

Inside, 279 by 279 by 305 mm (11 by 11 by 12 in.) high,

Height, 483 mm (19 in.), adjust to accommodate stirrer unit,

Rear panel, >200 by 200-mm vent area,

Top hole, 70.0 mm (2¾ in.) diameter,

Air inlet hole, to fit air supply unit, and

Air exit hole, to accommodate a simple slide damper.

A1.2.1 *Materials*—Sheet metal of at least 16 gage covered with insulation. Generally a portion of the metal bottom must be partially removed and replaced with nonmagnetic material to permit operation of the magnetic stirrer. The rear panel

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E27-1004. Contact ASTM Customer Service at service@astm.org.

should be equipped with a vent (>200 by 200 mm) providing explosion relief at low over-pressures: <6.9 kPa (1 psi). A lightly held panel of insulating board may be used.

A1.2.2 *Door, hinged and latched*—fitted with a 102 to 127 mm (4 to 5 in.) square safe viewing window made of polycarbonate and at least 12.7 mm (½ in.) thickness, or equivalent.

A1.2.3 *Bolts*—top-fitted with 2, ¼-20 bolts on 127 mm (5 in.) centers to secure test vessel cover.

A1.2.4 *Spacer*—A cylindrical spacer constructed of perforated, light-gage metal is placed under the test vessel. It is sized so as to position the top of the neck of the test vessel just above the top of the test chamber. This permits air circulation and facilitates insertion and removal of the test vessel.

NOTE A1.1—If heavy construction is employed for the front, top, and side walls of the chamber, and if the rear and bottom panels of the chamber are of lightweight materials, explosion venting will be to the rear, away from the operator, in the event of vessel rupture.

A1.2.5 *Alternatives*—Other thermostated chambers or ovens and heating means may be employed if they permit temperature control and proper test manipulation and observation with adequate safety.

A1.3 *Heater*—Heated air is supplied from a blower, at the rate of approximately 0.38 m^3/min (13.5 ft^3/min), feeding air through a variable electric heater of approximately 2400 W. Commercial blowers, heaters, and manual or automatic controls and combination, thereof, are available.

A1.4 *Ignition Device:*

A1.4.1 *Electrode rods*—3.175 to 4.76 mm (⅛ to 3⁄16 in.) diameter stainless steel, 317.5 mm (12½ in.) long. The upper ends are threaded for connection to a high-voltage source and the lower ends are threaded for attachment of spark gap points, or fuse wire, or both. Electrode rods are spaced at least 32 mm (1¼ in.) apart. The spark gap points are suspended approximately 70.3 mm (2¾ in.) above the bottom of the flask. Other materials of construction may be used as needed.

A1.4.2 *Spark gap*—having 6.4 mm (¼ in.) electrode spacing. Gap electrode extensions may be fabricated of platinum or tungsten wire held in wire connector lugs.

A1.4.3 *Fuse wire*—A 19 mm (¾ in.) loop of 40-gage copper wire attached to threaded electrode rods in place of spark gaps.

A1.4.4 *Power*—approximately 30 mA at 15 kV, supplied by the secondary of a 120-V, 60 Hz luminous tube transformer, or by an equivalent device. Power for the fuse wire is 120 V, 60 Hz.

A1.4.5 *Timer*—to limit spark duration to 0.2 to 0.4 s. Commercial interval timers are available.

A1.5 *Stirring Devices:*

A1.5.1 *Stirring bar*—63.5 mm (2½ in.) egg-shaped, plastic-coated, magnet bar.

A1.5.2 *Drive*—Laboratory magnetic stirrer capable of functioning through the bottom of the test chamber and vessel.

A1.6 *Test Vessel Cover*—The cover can be constructed of a Number 14 rubber stopper with necessary holes for electrodes, sample inlet device, air inlet, and evacuation connection and temperature-measuring device (see Fig. 1). It is important to note that the stopper rests on top and not inside the neck of the flask in order to facilitate venting.

NOTE A1.2—It is possible to operate at temperatures greater than 150°C (302°F) and to obtain more positive vacuum sealing through the use of specially constructed metal covers. High temperature O-ring seals for the flask top and inlet separatory funnel, and ceramic feedthroughs for the spark ignition source may be employed.

A1.7 *Cover Retainer*—This device (see Fig. 1) held in place with wing nuts, light springs, and¼-20 bolts can improve vacuum tightness of the test vessel when used to clamp down on the vessel cover.

A1.8 *Temperature Measurement:*

A1.8.1 Thermocouple, thermistor, resistance thermometer or other device with an accuracy of ±0.5°C may be used.

NOTE A1.3—Certain bare wire thermocouples may cause catalytic oxidation of test vapors, as evidenced by a persistent high-temperature excursion of the thermocouple junction. If this occurs, other thermocouple materials should be employed.

A1.8.2 A temperature measuring device outside the test vessel but in the heating chamber or inlet air stream can aid in controlling test temperature.

A1.8.3 Measurements of temperature uniformity within the test vessel should be conducted and recorded at a series of temperatures on the initial setup of an apparatus of this type. This can reveal the presence of potential cool sites (10.3) or general nonuniformities in heating.

A1.9 *Pressure Measurement:*

A1.9.1 *Atmospheric Pressure*—A barometer reading actual pressure at the test site accurate to 0.067 kPa (0.5 mm Hg) is adequate.

A1.9.2 *Other Pressure Measurement, (Needed for tests at pressures below one atmosphere)*—Any pressure-measuring system accurate to 0.067 kPa (0.5 mm Hg) in the range from 0.067 to 101.3 kPa (0.5 to 760 mm Hg) absolute and capable of being operated at temperatures greater than the condensation temperature of the materials under test is adequate for this unit. Vapor volume in the pressure sensing device outside the test vessel itself should be held to a minimum since all components must be above the condensation temperature of the materials being tested. Electrical heating tapes may be employed for heating components to the desired temperature.

A2. ESTIMATION OF INITIAL TEST TEMPERATURE OF FLAMMABILITY STUDIES

A2.1 It is important that temperature limit of flammability tests be conducted to avoid trials in the concentration range (8.2.2) that may produce energetic explosions. Lower limit tests should commence at temperatures below the lower temperature limit of flammability.

A2.2 Closed-cup flash points measured by Test Methods D3278, D3828, or D3941 approximate the lower temperature limit of flammability, and thus can be used to estimate initial test temperatures.

A2.2.1 Since impurities, as well as known low percentage components, can have a significant influence on closed-cup flash point and temperature limit of flammability, it is important that the closed-cup flash point be determined for the actual sample under study.

A2.2.2 Based on previous experience with this test method, the following are suggested guidelines for using flash point to determine starting temperatures for temperature limit studies:

A2.2.2.1 With materials having measured closed-cup flash points (Test Methods D3278, D3828, or D3941) on the sample under study below 38°C (100°F), commence lower temperature limit tests at least 8°C (15°F) below the flash point.

A2.2.2.2 With materials having measured closed-cup flash points Test Methods D3278, D3838, or D3941 in the 38 to 93°C (100 to 200°F) range, commence lower temperature limit testing at least 14°C (25°F) below the flash point.

A2.2.2.3 With materials having measured closed-cup flash points Test Methods D3278, D3828 or D3941 above 93°C (200°F), starting temperatures for lower limit studies should be from 22 to 44°C (40 to 80°F) below the closed-cup flash point.

A2.2.3 With those few materials having no closed-cup flash point (Appendixes X2.2, X3.2, X3.3), but that are, for various reasons, being tested for flammability in the temperature limit of flammability apparatus, methods of estimating temperature limit of flammability may be employed. However, wide safety factors should be used in initial trials.

NOTE A2.1—Because of the influence of impurities, flash point tests should be run on a sample falling into the class of materials covered in A2.2.1.

NOTE A2.2—Because of sample variability and the influence of impurities, it is strongly recommended that flash points estimated by the various techniques which have appeared in the literature not be used, without experimental verification, as a basis for selecting starting temperatures for lower temperature limit of flammability tests.

A2.3 An estimated lower temperature limit of flammability can be made if vapor pressure and lower concentration limit of flammability data are available.

A2.3.1 Lower concentration limits of flammability can be determined by Test Method E681, obtained from the literature or estimated using techniques summarized in Test Method E681.

NOTE A2.3—Impurities greatly affect the normal relationships between concentration limit of flammability, vapor pressure, and temperature limit of flammability. Thus, unless samples are pure, well characterized, or both, it is strongly recommended that the flash point-temperature limit relationships given in Annex A2.2.2, be used in estimating temperature limit of flammability, in preference to the concentration limit-vapor pressure relationships discussed in Annex A2.3.

A2.3.2 The estimated lower temperature limit of flammability (LTL_e) is that temperature at which the vapor pressure, P, results in a vapor concentration equal to that present at the measured lower concentration limit of flammability (LFL_m). The following employs terminology similar to that from Test Method E681: LTL_e is the temperature, obtained from vapor pressure data, at which:

$$P(LTL_e) = (LFL_m/100) \times P_o \tag{A2.1}$$

where:

$P(LTL_e)$ = vapor pressure at the estimated lower temperature limit,

P_o = test pressure, generally 1 atmosphere, (101.3 kPa or 760 mm Hg), and

LFL_m = measured lower flammable limit in volume percent.

NOTE A2.4—Since lower concentration limits of flammability are generally determined at temperatures above the saturation temperature of the vapors (in order to avoid condensation during testing) a slight correction to LFL_m may be required in the calculation in A2.3.2.

NOTE A2.5—Certain chemicals (organic acids for example), exhibit a high degree of non-ideal vapor phase behavior, being highly associated in the vapor phase. In order to properly interpret the vapor pressure/volume percent/weight percent/temperature relationships, a knowledge of the degree of non-ideality (association and molecular weight) is necessary. (Concentration limits of flammability for these materials are generally expressed in terms of weight per unit volume. See Test Method E681).

A2.3.3 Lower temperature limit of flammability testing should commence at a temperature below the estimated lower temperature limit obtained in A2.3.2.

A3. TESTING OF MIXTURES

A3.1 The apparatus and procedures of this test method can be used to determine the lower temperature limit of flammability of chemical mixtures.

NOTE A3.1—Mixtures of conventional flammable solvents with certain halogenated hydrocarbons, or with water, can exhibit the type of behavior discussed in Appendix X3.2. Mixtures having no flash point can exhibit flammability in the temperature limit apparatus. Temperature differences between temperature limit and flash point can be high (A2.2.2) and other non-standard behavior can be observed (X2.6, X3.2, X3.3).

NOTE A3.2—Since the flammability behavior of mixtures may be hard to predict and since certain mixtures will burn in this apparatus and not in a flash point tester (Appendix X3.2), it is important that all safety precautions (Section 8) be observed, and that a conservative approach to test starting temperature (Note 4) be used.

A3.2 Details of sample preparation are beyond the scope of this test method but certain basic points should be noted as follows:

A3.2.1 Samples should be representative of the process or product under investigation. (For example, studies of the flammability characteristics of an evaporating mixed solvent system would require samples taken at various degrees of evaporation under conditions duplicating or simulating the actual process.)

A3.2.2 Sample sizes should be sufficient for completion of all required flash point, temperature limit, analysis, and other tests.

A3.2.3 Samples should be completely sealed in containers that will preclude loss of volatile components between sampling and testing and reaction with the container material. In many instances plastic containers will be inadequate to prevent loss of trace components; glass and metal containers are recommended. (Loss of trace amounts of highly volatile flammable and non-flammable components can have a major effect on vapor phase flammability characteristics of a mixture (Appendix X2.8)).

A3.3 Procedure for testing of mixtures parallel those given in Section 10. However, some modifications to those procedures are given as follows:

A3.3.1 Determine by Test Methods D3278, D3828, or D3941 the closed-cup flash point of a specimen of the sample to be tested for temperature limit of flammability.

A3.3.1.1 If the sample has no closed-cup flash point, but is felt to have properties of the type discussed in Appendix X3.2 and X3.3, an open-cup flash point test should be determined.

A3.3.1.2 If no open-cup flash point is obtained, and temperature limit testing is to be pursued, test starting temperatures must be conservatively selected (Note 4).

A3.3.2 Based on the closed-cup flash point Test Methods D3278, D3828, and D3941, adjust the flask to the desired test temperature based on methods given in Annex A2. If starting temperature is to be based on an open-cup flash point value, double the temperature differences given in A2.2.2.

A3.3.3 Place a specimen of liquid in the flask appropriate for the composition being tested.

NOTE A3.3—A specimen size, larger than normal (10.5.1), is required to ensure a sufficient quantity of all components for the attainment of vapor-liquid equilibrium prior to test. Smaller specimens will be adequate for 50-50 mixtures, for example, and larger specimen sizes may be necessary with mixtures having trace amounts of components of interest. (Calculations based on known compositions can reveal specimen sizes theoretically required.)

A3.3.4 Stir as specified in 10.5.2 and 10.5.4, observing requirements for attainment of equilibrium.

A3.3.4.1 Observe for inconsistent results (Note 6). With mixtures, loss of one component after a trial can alter results and it may become necessary to change samples (on preliminary trials) more frequently than is required (10.5.12) for neat materials.

A3.3.5 All final trials (10.5.13) should be on a fresh specimen in a clean vessel.

NOTE A3.4—If ignition problems are encountered, a fuse wire ignition source may be required (Note 9).

A3.3.6 Data reported for mixtures should include as much information as possible on the composition and identity of the sample. Slight differences in composition can have a major effect, Note A2.1 and Note A2.2).

A3.4 Estimation of starting temperature for limit of flammability testing of mixtures.

A3.4.1 In the interest of safety it is recommended that starting temperatures for lower temperature limit of flammability testing of mixtures be based on actual flash point measurement and the use of A2.2 and A3.3.2.

A3.4.2 The starting temperature should be well outside the flammable range. Proper safety considerations should be observed (Section 8), and careful observations of results (Note 6 and Note 8), should continue as testing proceeds.

A3.4.3 Methods of estimating (other than use of an actual flash point) given in Annex A2 can be used; however, necessary information is not generally available.

A3.4.4 Care must be exercised in the use of estimated flash points for temperature limit testing of mixtures. There are many mixtures (non-ideal solutions) having flash points below the flash point of any component.

A3.4.5 Unless detailed data are available it is difficult to estimate temperature limits of flammability of mixtures.

APPENDIXES

(Nonmandatory Information)

X1. APPLICABILITY OF THE TEMPERATURE LIMIT OF FLAMMABILITY TEST

X1.1 This test method was developed to provide a means of assessing the lowest temperature at which a chemical evolves vapor in sufficient concentration to be flammable in air.

X1.2 This test method yields a result applicable for assessing potential flammability hazards in chemical process and storage vessels.

X1.3 This test method is not considered to be appropriate as a replacement for conventional flash point testing for regulatory purposes. flash point tests, properly conducted and interpreted, have proven to be adequate for regulatory purposes through years of experience.

X2. RATIONALE OF THE TEMPERATURE LIMIT OF FLAMMABILITY TEST

X2.1 Conventional flash point methodology may not yield results sufficiently precise for use in chemical process hazard evaluation. See Note 1 and Appendix X3.

X2.2 In addition, conventional flash point methods may yield a negative result for materials that indeed do evolve flammable vapors in air (Appendix X2.2). The concepts of temperature limit of flammability[6] and vapor phase flammability of materials having no flash point[7] have been known for some time.

X2.3 Potential deficiencies associated with conventional flash point methodology are discussed in Appendix X3.

X2.4 Various test configurations designed to overcome certain inherent scientific deficiencies of conventional flash point methodology were considered. The spherical glass test equipment of Test Method E681, concentration limit of flammability, was considered to be adequate for the evaluation of most chemicals in air at atmospheric pressure.

X2.4.1 Flame propagation is in an upward and outward direction, vessel size is sufficient to eliminate most flame quenching effects and thermal equilibrium is achieved to a high degree.

X2.5 Temperature limit of flammability results obtained by this method are consistent with vapor pressure and concentration limit of flammability data. This provides a built in check of results on pure materials and leads to a high degree of confidence in the results obtained for mixtures.

X2.6 Materials having no flash point, yet yielding a positive result in this test method, most often do not burn in an open pool configuration when subjected to an ignition source. Failure to *pool* burn can occur with materials having a low heat of combustion or other characteristics of *slow* burning.

X2.6.1 These materials yielding a positive result on this test, do make a net heat contribution when oxidized in air. Thus, these materials will contribute energy when exposed in a fire.

X2.6.2 When mixed with conventional flammable solvents these materials generally act as flammable diluents in both vapor phase and pool burning configurations.

X2.7 Materials having no flash point but exhibiting a temperature limit of flammability possess a degree of flammability which must be adequately considered in each application.

X2.8 Liquid mixtures containing flammable and nonflammable components can exhibit flash point and temperature limit of flammability behavior ranging from complete flammability to complete inerting.

X2.8.1 Any spillage, chemical processing, evaporation, drying, or other operation can completely alter initial flammability characteristics. Loss of volatile, nonflammable components may result in a flammable residue, and the complete opposite can also be observed.

X2.8.2 Proper evaluation of these mixtures requires testing of original material and all degrees of evaporation which might be expected under normal process and use conditions. Compositions and temperatures to be encountered in abnormal processes, use, and spill conditions must also be considered.

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[6] See U.S. Bureau of Mines Bulletin 627, 1965. Available from U.S. Bureau of Mines, Cochrans Mill Rd., P.O. Box 18070, Pittsburgh, PA 15236.

[7] See U.S. Bureau of Mines RI 6766, 1966. Available from U.S. Bureau of Mines, Cochrans Mill Rd., P.O. Box 18070, Pittsburgh, PA 15236.

TABLE X3.1 A Comparison of LTL and Flash Point Values for Selected Chemicals

Material	LTL (°C)	Flash Pint (°C)[A]
isobutyl isobutyrate	35[B]	38
acetic acid	37[B]	40
propanoic acid	48[B]	52
diethylene glycol dimethyl ether	52[B]	70
methyl benzonate	73[B]	83
1.2–dimethoxybenzene	82[B]	87
ethyl iodo acetate	83[B]	77
N-ethyl-3–methyl benzeneamine	83[B]	89
4–methyl methylbenzoate	90[B]	90
sulfur	247[C]	207
phthalic anhydride	140[B]	152
dimethhyl sulfoxide	84[B]	89
2.6–diethyl aniline	80[B]	123
nicotine	79[B]	101
bicyclohexyl	74[B]	92

[A] Determined in accordance with Test Method E1232. (See Research Report RR:E27-1004.)

[B] Reported by Zabetakis in US Bureau of Mines Bulletin 627.[6]

[C] Flash point values shown above has been compiled in July 2007 from available sources such as Material Safety Data Sheets or NFPA 325. Flash points can depend on the test method used in their determination.

X3. COMMENTARY ON THE FLASH POINT TEST

INTRODUCTION

This section is taken mainly from Test Method E502. This has been revised and updated where appropriate, for this issue.

X3.1 For the following reasons, flash point may not represent the minimum temperature at which a material can evolve flammable vapors:

X3.1.1 flash point tests are often run at a finite heating rate, and therefore, vapor concentrations may not be representative of equilibrium conditions. Newer equilibrium and most Setaflash methods overcome this rate deficiency.

X3.1.2 flash point testing employs downward and horizontal propagation of flame. Flame propagation in these directions generally requires slightly higher vapor concentrations than is required for upward flame propagation.

X3.1.3 In the flash point test the flame is introduced at a finite distance above the liquid surface. Since the vapors are more dense than air, the vapor concentration is often higher at the liquid surface than at the flame position.

X3.1.4 Covers in flash point testers are not generally heated and may be cooler than the measured bulk temperature. This may result in condensation back to a temperature slightly below the measured temperature.

NOTE X3.1—The relative and absolute magnitudes of the effects discussed in X3.1 depend on the material and the test method measured flash points, relative to the temperature limit of flammability. The *offset* increases with increasing flash point temperature (A2.2.2). Sample comparisons are provided in Table X3.1.

X3.2 There are instances where the absence of a flash point does not ensure freedom from flammability. Included in this category are materials that require large diameters for flame propagation, such as trichloroethylene and certain acetic acid-water mixtures. These materials will not propagate a flame in a conventional flash point tester, but their vapors may be flammable and may burn when ignited in a vessel of adequate size.

X3.3 Some materials having very dense vapors, a narrow range of flammability, or the requirement for being somewhat superheated to burn will not exhibit a conventional flash point but can form flammable vapor-air mixtures if heating and mixing are optimum and temperatures are raised.

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