

Designation: E918 – 19

# Standard Practice for Determining Limits of Flammability of Chemicals at Elevated Temperature and Pressure<sup>1</sup>

This standard is issued under the fixed designation E918; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

#### 1. Scope

1.1 This practice covers the determination of the lower and upper concentration limits of flammability of combustible vapor-oxidant mixtures at temperatures up to 200°C and initial pressures up to as much as 1.38 MPa (200 psia). This practice is limited to mixtures which would have explosion pressures less than 13.79 MPa (2000 psia).

1.2 This practice 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.

1.3 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.

1.4 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:<sup>2</sup>

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

## E2079 Test Methods for Limiting Oxygen (Oxidant) Concentration in Gases and Vapors

2.2 Other Documents:

Bulletin 503 Bureau of Mines, "Limits of Flammability of Gases and Vapors," NTIS AD701575<sup>3</sup>

Bulletin 627 Bureau of Mines, "Flammability Characteristics of Combustible Gases and Vapors," NTIS AD701576<sup>3</sup>

#### 3. Terminology

3.1 Definitions:

3.1.1 lower limit of flammability or lower flammable limit (LFL), *n*—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.

3.1.2 upper limit of flammability or upper flammable limit (UFL), n—the maximum 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.

#### <u>3.2</u> Definitions of Terms Specific to This Standard:

3.2.1 propagation of flames, n—as used in this practice, a combustion reaction that produces at least a 7 % rise of the initial absolute pressure:

$$\frac{P_2}{P_1} \ge 1.07$$

Note 1—This 7 % rise in pressure corresponds to 1 psia (0.007 MPa) per atmosphere of initial pressure.

Note 2—The 7 % pressure criterion may not be appropriate for certain fuel and oxidant mixtures. This is also the case if the test enclosure volume is small, or when the ignition energy is substantially larger than 10 J. It is therefore a prudent practice to perform exploratory tests in the vicinity of limit mixtures to evaluate the validity of the selected pressure rise criterion.<sup>4, 5</sup>

<sup>&</sup>lt;sup>1</sup> This practice 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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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from National Technical Information Service (NTIS), 5301 Shawnee Rd., Alexandria, VA 22312, http://www.ntis.gov.

<sup>&</sup>lt;sup>4</sup> Ural, E. A., and Brandes, E., "Towards A Global Standard For Flammability Determination," *Proceedings of the 42nd Annual Loss Prevention Symposium*, Global Safety Congress, American Institute of Chemical Engineers, New Orleans, LA, Apr. 6–10, 2008.

<sup>&</sup>lt;sup>5</sup> Cashdollar, K. L., Zlochower, I. A., Green, G. M., Thomas, R. A., and Hertzberg, M., "Flammability of Methane, Propane and Hydrogen Gases," *Journal of Loss Prevention in the Process Industry*, Vol 13, 2000, pp. 327–340.

## 4. Summary of Practice

4.1 A mixture of gaseous or vaporized fuel with a gaseous oxidizer is prepared in a steel or other appropriate metal vessel at a controlled temperature and pressure. Proportions of the components are determined by measurement of partial pressures during filling of the vessel. Ignition of the mixture is attempted with a fuse wire, and flammability is deduced from the pressure rise produced. Fuel concentration is varied between trials until the limits of flammability have been determined. Composition of the mixtures which fix the flammable limits are confirmed by appropriate analysis.

#### 5. Significance and Use

5.1 Knowledge of flammable limits at elevated temperatures and pressures is needed for safe and economical operation of some chemical processes. This information may be needed in order to start up a reactor without passing through a flammable range, to operate the reactor safely and economically, or to store or ship the product safely.

5.2 Limits of flammability data obtained in relatively clean vessels must be carefully interpreted and may not always be applicable to industrial conditions. Surface effects due to carbon deposits and other materials can significantly affect limits of flammability, especially in the fuel-rich region. Refer to Bulletin 503 and Bulletin 627.

#### 6. Limitations

6.1 This practice is not applicable to mixtures which undergo spontaneous reaction before ignition is attempted.

6.2 These test methods are limited to mixtures which have maximum deflagration pressures less than the maximum working pressure of the test apparatus.

6.3 These test methods may be used up to the temperature limit of the test system.

6.4 Measurements of flammability are influenced by flamequenching effects of the test vessel walls. Further surface effects due to deposits of carbon or other materials can significantly affect limits of flammability, especially in the fuel-rich region. Refer to Bulletin 503 and Bulletin 627. For certain chemicals (for example, ammonia, halogenated materials, and certain amines) which have large ignitionquenching distances, tests may need to be conducted in vessels larger than that specified below.

#### 7. Apparatus

7.1 Fig. 1 is a schematic diagram of the high initial pressure test apparatus; details and dimensions are presented in Annex A1. The apparatus consists of a metal pressure vessel with a minimum volume of 1 L and a minimum inside diameter of 76 mm (3 in.), an insulated chamber equipped with a source of controlled-temperature inert gas, an ignition device with appropriate power supply, remotely controlled valves, pressure measuring equipment, and a venting system for handling overpressuring.

7.2 In tests where the initial pressure is of the same order of the atmospheric pressure or below, the apparatus described in Test Methods E2079 must be used.

7.3 In tests where the initial pressure is of the same order of the atmospheric pressure or below, the test vessel must have a volume of at least 4 L.

#### 8. Safety Precautions

8.1 Adequate shielding must be provided to prevent injury in the event of equipment rupture. The apparatus is set up so that the operator is isolated by a blast-proof wall from the test vessel while the vessel contains a charge of reactants, including the time while the vessel is being filled. The test apparatus should be equipped with interlocks so that the ignition source cannot be activated unless the operator has taken necessary steps to protect personnel and equipment. Activation of the ignition source should be possible only from a position shielded from the test vessel.

8.2 The test vessel shall be fitted with a rupture disk vented outside any enclosed area. Fuel may inadvertently be vented inside the heated chamber or inside the enclosed area, so the heated chamber should be fitted with an inert gas purge and the area should be adequately ventilated to prevent buildup of an explosive mixture in the large space.

8.3 Undesirably energetic explosions may be produced if tests are made at high initial pressures with mixtures well within the flammable range. Very strong oxidizers greatly increase explosion severity and also greatly increase the fuel-rich limit. To help in avoiding testing highly energetic mixtures, limits of flammability should first be determined at atmospheric pressure. These limits are covered in Test Method E681. With this knowledge, the operator should proceed in cautious steps of initial pressure increase to work at higher pressures and temperatures.

#### 9. Preparation of Apparatus

9.1 Clean and dry the test vessel and other gas-handling equipment. Make sure that no oil, grease, or other combustible is left inside the parts.

9.2 Assemble the equipment as shown in Fig. 1. Purge the vessel with inert gas and then evacuate the system.

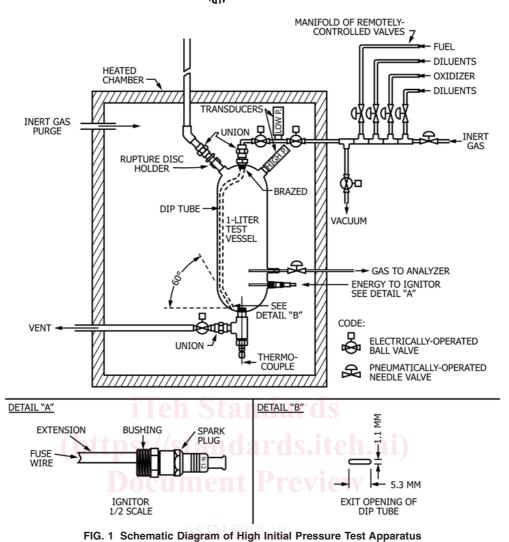
9.3 Set the zero and gain on the pressure transducers so that their output represents true pressure after the test vessel is at the working temperature.

#### METHOD A SAMPLE COMPONENTS WHICH HAVE ADEQUATE VAPOR PRESSURE AT ROOM TEMPERATURE

# **10. Procedure**

10.1 Attach pressure regulators to the supply cylinders of gases to be used in the tests. Connect the regulators to the manifold of remotely-controlled metering valves.

10.2 Flush each line from the supply cylinder to the metering valve. Evacuate the test vessel and manifold. By use of the remotely controlled valves, add to the test vessel the component most appropriately added first; usually, this is the smallest component. Close the ball valve next to the test vessel and evacuate or purge the manifold. E918 – 19



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10.3 Add the second component up to the desired pressure, as measured by the transducer. Repeat the clearing of the manifold and add components until the desired partial pressure of each component has been added to the test vessel. Obtain mixing of gas in the test vessel by adding the largest component last and at high velocity.

NOTE 3—Both fast addition of the last component and restricting the tip of the dip tube are necessary to achieve homogeneity. One way to add gas at high velocity with low risk of overshooting is to make use of a quick-opening dump valve on the pneumatic actuator system for the metering valve. The last component should be added in less than 15 s.

Note 4—Where the vessel configuration will permit, an internal mixing device may be used.

NOTE 5—If the pressure and temperature do not hold steady after a component is added this may indicate reaction prior to ignition. Reaction of a halogen will probably cause a pressure drop. Reaction of oxygen will probably cause a pressure rise.

10.4 Close the remotely controlled valve between the test vessel and the low-range pressure transducer in order to protect this transducer from explosion pressure.

10.5 Allow the test gas mixture to equilibrate to test conditions.

10.6 Early in the test series, use an appropriate method such as gas chromatography to confirm composition of gas mixtures made ready for explosion test. Make any changes in technique necessary to ensure homogeneous mixture. These mixtures may not have the composition expected, due to nonideal gas behavior. Errors will vary with the order of mixing, temperature, pressure, and the particular materials. Also, the greater the dead volume in tubing etc., not involved in mixing with the charge in the cylinder, the greater will be the difference from expected composition. If the composition is wrong make adjustments in partial pressure to get desired composition.

10.7 Record the temperature and pressure of the test gas.

10.8 Activate the pressure recording equipment.

10.9 Attempt ignition of the gas mixture by applying 115 V across the fuse wire.

10.10 Record the maximum pressure.

10.11 Vent the test vessel through the exhaust valve. Purge the vessel with inert gas from the manifold.