Designation: E918 – 83 (Reapproved 2011)

Standard Practice for Determining Limits of Flammability of Chemicals at Elevated Temperature and Pressure[1]

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 (ε) 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 may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

2.2 *Other Documents:*

Bulletin 503 Bureau of Mines, "Limits of Flammability of Gases and Vapors," NTIS AD701575

Bulletin 627 Bureau of Mines, "Flammability Characteristics of Combustible Gases and Vapors," NTIS AD701576

3. Terminology

3.1 *Definitions:*

3.1.1 *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.

3.1.2 *upper limit of flammability or upper flammable limit (UFL)*—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.

3.2 *Definitions of Terms Specific to This Standard:*

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

$$\frac{P_2}{P_1} \geqq 1.07.$$

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

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

[1] 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.

Current edition approved Aug. 1, 2011. Published August 2011. Originally approved in 1983. Last previous edition approved in 2005 as E918 – 83 (2005). DOI: 10.1520/E0918-83R11.

[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.

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 Measured limits of flammability are influenced by flame-quenching effects of the test vessel walls. The vessel described in this practice is suitable for use with most mixtures at elevated temperatures and pressures. For certain amines, halogenated materials etc., which have large ignition-quenching distances, tests may need to be conducted in larger diameter vessels.

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 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.

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.

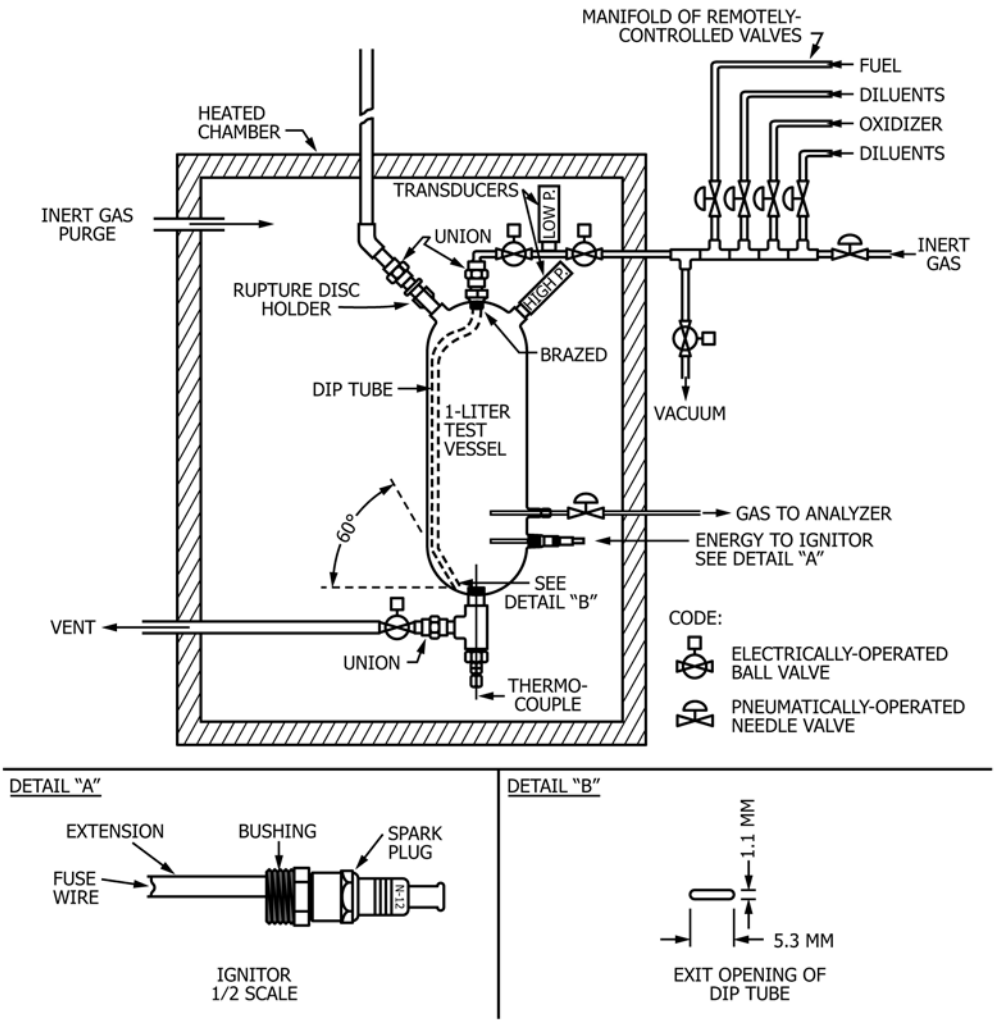

FIG. 1 Schematic Diagram of Test Apparatus

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 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.

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 2—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 3—Where the vessel configuration will permit, an internal mixing device may be used.

NOTE 4—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.

10.12 Install another spark plug fitted with a fuse wire.

NOTE 5—By having the spark plug positioned in front of a socket wrench-sized hole in the wall of the heated chamber, the plug can be changed without appreciably cooling off the chamber. Use a deep socket wrench which fits the bushing, not the spark plug.

10.13 Vary fuel concentration (percent of the total vapor pressure) as required to find the minimum concentration, L_1, that gives flame propagation and the maximum concentration, L_2, below L_1, that does not give flame propagation. Flame propagation by this method is defined as a pressure ratio

$$\frac{P_2}{P_1}$$

of 1.07 or more. Record values for L_1 and L_2 measured by pressure during filling of the test vessel and by analysis of the mixtures. Repeat the analysis and test on composition L_2 to confirm its non-flammability.

10.14 Commence upper limit tests in the nonflammable region at a concentration greater than the anticipated U_2. (See 10.16.) Fuel concentration at the upper limit may be substantially greater at elevated temperature and pressure than it is at atmospheric conditions.

10.15 Remove any carbon deposits that may be left in the test vessel after a fuel-rich explosion.

NOTE 6—These deposits are likely to affect subsequent results. To remove the carbon insert through the spark plug hole an L-shaped piece of 6.4-mm (¼-in.) metal tubing attached by a flexible hose to an inert gas supply. Blow inert gas vigorously around inside the test vessel until no more carbon is displaced. Finish cleaning out the carbon by igniting, as if it were the next sample, a fuel-lean mixture such as 6 % methane in air. After trying this burn-out procedure, remove the spark plug, insert a light bulb through the spark plug hole, and inspect the vessel for cleanliness. Repeat the lean mixture explosion if necessary to clean the vessel. Mechanical means may also be used to remove unwanted carbon.

10.16 Record the values for the highest fuel concentration, U_1, that will propagate a flame and the lowest concentration, U_2, above U_1, that will not propagate a flame. Make duplicate tests on U_2.

METHOD B—FUEL WHICH MUST BE HEATED TO REACH ITS LIMITS OF FLAMMABILITY

11. Procedure

11.1 Set up the equipment as described in Method A except for the introduction of fuel. A small cylinder of liquid fuel may be placed in the heated chamber with the test vessel or heated separately.

NOTE 7—The cylinder of liquid fuel must be fitted with a pressure-relief device which discharges outside the heated chamber and outside any other structure which would confine the material. Fuels which might undergo hazardous reaction in the heated cylinder must not be tested by this procedure. If any uncertainty exists as to whether the sample may react in the heated chamber, thermal stability testing should be performed.

11.2 Fit the fuel cylinder with a remotely controlled needle valve. If the fuel will evaporate without fractionation, place the valve in contact with the vapor phase in the cylinder. Otherwise, place the needle valve in contact with the liquid phase even though this makes control of the flow more difficult. Connect the needle valve to the section of fill line holding the low range pressure transducer. This allows the ball valve shown at the right side of this pressure transducer to be used to keep fuel from condensing outside the heated chamber.

11.3 After the vessel is evacuated, add the fuel component first. With the desired amount of fuel pressure in the test vessel, proceed with the test starting with the last sentence of 10.2.

12. Calculations

12.1 Calculate the lower limit of flammability (LFL) and upper limit of flammability (UFL) from the values recorded in 10.13 and 10.16.

$$LFL = ½\ (L_1 + L_2) \quad (1)$$

$$UFL = ½\ (U_1 + U_2) \quad (2)$$

NOTE 8—Limit concentrations are usually reported in terms of volume percent. It is sometimes appropriate to report limits in terms of weight per unit volume at the test conditions, particularly with multicomponent fuels or materials exhibiting a high degree of nonideal vapor phase behavior. Report, for example, *x* mg/l at *y* MPa and *z*°C.

ANNEX

(Mandatory Information)

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

A1.1 Test Vessel[3]

A1.1.1 The test vessel shall be a metal vessel having a volume no less than 1 L and a minimum inside diameter of 7.6 mm (3 in.). After fabrication, the vessel shall be satisfactorily tested to 20.68 MPa (3000 psi). Fig. 1 shows apparatus with three ½-in. NPT female pipe connections added to accommodate the ignitor, rupture disc holder, and pressure transducer and with a ¼-in. NPT female pipe connection for the sampling tube.

A1.2 Rupture Disk Holder[4]

A1.3 Ignitor

A1.3.1 A holder for the fuse wire used as ignition source can be made by modifying a spark plug which has a continuous metal contact to the center terminal. A Champion N-12Y spark plug is suitable. Fit the spark plug with a bushing made by reaming out a ¼ by ½-in. NPT bushing and rethreading it inside to fit the 14-mm spark plug. Using steel rod of about 3-mm diameter, extend the spark plug terminals so that they will reach the vertical axis of the test vessel. For each test attach a piece of No. 40 (0.076 mm diameter) tinned copper wire at the extended spark plug terminals (Note A1.1) to leave about 10-mm length of wire between terminals. Supply energy to the ignitor from an isolating transformer rated 500VA at 115V.

NOTE A1.1—Problems with the fuse wire not making good electrical contact with the extended terminals can be eliminated by soldering. Apply a coating of silver solder near the extended ends of the spark plug terminals. The fuse wire can then be easily attached with lead-tin solder.

[3] Hoke Part No. 8HD1000 has been used satisfactorily as a test vessel after modifications by making more openings in the cylinder and welding pipe fittings over these openings; available without modification from Hoke, Inc., One Tenakill Park, Cresskill, NJ 07626. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[4] A Fike Assembly No. 1/2-30SB is suitable; available from Fike Metal Products Corp., 704 South 10th Street, Blue Springs, MO 64015. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

A1.4 Needle Valves[5, 6]

A1.5 Block Valves[7]

A1.5.1 The electric actuators are mounted outside the heated chamber and the ball valves inside the heated chamber.

A1.6 Heated Chamber

A1.6.1 The heated chamber can be made from a steel electrical box 610 by 760 by 410 mm (24 by 30 by 16 in. deep).[8] Insulate the inside of the box with a 50-mm (2-in.) thickness of ceramic fiber blanket.[9] Provide a blow-out vent by cutting out a section of the steel wall leaving only insulation over this wall area. Cut slits to weaken the edges of this blow-out section of insulation. Fit the chamber with a 0.047 m^3/s (100 ft^3/min) blower recirculating air over a 3 kW heater. Modify the blower if necessary so that the blower motor can be placed outside the heated area.

A1.7 Pressure Transducers[10]

A1.7.1 The test vessel should be connected to two pressure transducers capable of continuous operation at 200°C. The low range transducer should be capable of measuring the sample components to an accuracy of 1 % relative to that component. The high range transducer should have suffi-cient range not to be damaged by the explosion pressure and be sensitive enough to detect a 7 % pressure rise.

A1.8 Thermocouple and Connector

A1.8.1 Measure temperature inside the test vessel using a Type-K thermocouple inside a 1.6-mm (1⁄16 in.) diameter metal sheath.[11]

A1.9 Tubing

A1.9.1 Tubing-Type 316 stainless steel 6.4 mm (0.25 in.) outside diameter with 0.89-mm (0.035-in.) wall thickness is suitable for lines from the gas cylinders to the test vessel.

A1.10 Connections

A1.10.1 Compression stainless steel fittings are suitable for attaching the tubing to valves, pipe fittings, etc.[12] Silver solder may be used to attach the dip tube to the ¼ by ½-in. NPT bushing which supports the tube. Pipe unions may be used (as illustrated at the top of the vessel in Fig. 1) to facilitate removal of the vessel. All fittings shall be capable of holding 20.68 MPa (3000 psi) pressure.

---

[5] The Research Control Valve Serial 55541 with 0 size trim, is satisfactory for remotely controlling flow to the test vessel; available from Precision Products Division, Badger Meter, Inc., Tulsa, OK 74115. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[6] To get rapid response of the pneumatically operated Research Control valves, a Humphrey Products Tyna-Myte solenoid-operated valve can be used to quickly turn on or dump the actuating gas; available from Humphrey Products, Kilgore at Sprinkle, P. O. Box 2008, Kalamazoo, MI 49003. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[7] Hills-McCanna ball valves code ¼-in. F602-S6-R-S6 with Hills-McCanna Model 3B3 electric actuators can be used to isolate the test vessel; available from Hills-McCanna Company, 400 Maple Ave., Carpenterville, IL 60110. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[8] This box is available as a NEMA Type 12 Enclosure, Catalog Number A-302416LP, from Hoffman Engineering Co., Division of Federal Cartridge Corp., 9th and Tyler St., Anoka, MN 55303. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[9] Kaowool insulation, available from Babcock and Wilcox, Refractories Div., P. O. Box 923, Old Savannah Rd, Augusta, GA 30903, has been found suitable. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend. Kaowool and all related terms are trademarked Babcock and Wilcox.

[10] Sensotec T-series transducers have been found suitable for this purpose; available from Sensotec, Inc., 1200 Chesapeake Ave., Columbus, OH 43212. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[11] The thermocouple and connector can be obtained from Omega Engineering, Inc., P. O. Box 4047, Stamford, CT 06907. Their catalog designation for the thermocouple is TJ36-CA1N-116G-12 and for the connector is OST-CHAL-MF. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[12] Swagelok brand compression fittings are available from Crawford Fitting Co., 29500 Solon Road, Solon, OH 44139. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.