



# Standard Test Methods for Limiting Oxygen (Oxidant) Concentration in Gases and Vapors<sup>1</sup>

This standard is issued under the fixed designation E 2079; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover the determination of the limiting oxygen (oxidant) concentration of mixtures of oxygen (oxidant) and inert gases with flammable gases and vapors at a specified initial pressure and initial temperature.

1.2 These test methods may also be used to determine the limiting concentration of oxidizers other than oxygen.

1.3 Differentiation among the different combustion regimes (such as the hot flames, cool flames and exothermic reactions) is beyond the scope of these test methods.

1.4 *These test methods 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.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

E 681 Test Method for Limits of Flammability of Chemicals<sup>2</sup>

E 918 Practice for Determining the Limits of Flammability of Chemicals at Elevated Temperature and Pressure<sup>2</sup>

### 2.2 NFPA Publication

NFPA 69, Standard on Explosion Prevention Systems<sup>3</sup>

### 2.3 NTIS Publications

Coward, H.F. and Jones, G.W., Bulletin 503, Bureau of

Mines, "Limits of Flammability of Gases and Vapors," NTIS AD701575, 1952<sup>4</sup>

Zabetakis, M.G., Bulletin 627, Bureau of Mines, "Flammability Characteristics of Combustible Gases and Vapors," NTIS AD701576, 1965<sup>4</sup>

Kuchta, J.M., Bulletin 680, Bureau of Mines, "Investigation of Fire and Explosion Accidents in the Chemical, Mining, and Fuel-Related Industries - A Manual," NTIS PB87113940, 1985<sup>4,5,6</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 *flammable*—capable of propagating a flame.

3.1.2 *ignition*—the initiation of combustion.

3.1.3 *limit of flammability*—the boundary in composition space dividing flammable and nonflammable regions.

3.1.4 *limiting oxygen (oxidant) concentration (LOC) of a fuel-oxidant-inert system*—the oxygen (oxidant) concentration at the limit of flammability for the worst case (most flammable) fuel concentration.

3.1.4.1 *Discussion*—Limiting oxygen (oxidant) concentration is also known as *minimum oxygen (oxidant) concentration* or as *critical oxygen (oxidant) concentration*.

## 4. Summary of Test Method

4.1 A mixture containing one or more flammable components (fuel), oxygen (oxidant) and inert gas(es) (such as nitrogen, carbon dioxide, argon, etc.) is prepared in a suitable test vessel at a controlled initial temperature and made to the specified initial pressure. Proportions of the components are determined by a suitable means. Ignition of the mixture is attempted and flammability is determined from the pressure rise produced. The criterion for flammability is a pressure rise of  $\geq 7\%$  above the initial absolute test pressure. Fuel, oxygen

<sup>4</sup> National Technical Information Service, 5285 Port Royal Road, Springfield VA 22161.

<sup>5</sup> Subramaniam, T.K. and Cangelosi, J.N., "Predict Safe Oxygen in Combustible Gases," *Chem. Eng. Prog.*, Dec. 1989, pp. 108-113.

<sup>6</sup> Britton, L.G., "Flammability Hazards of Lower Aliphatic Aldehydes at Elevated Pressure and Temperature," *Process Safety Progress*, Vol 17, No. 2, 1998, pp. 138-148.

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee E-27 on Hazard Potential of Chemicals and are the direct responsibility of Subcommittee E27.04 on Flammability and Ignitability of Chemicals.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>3</sup> National Fire Protection Association, P.O. Box 9101, Quincy MA 02269-9101.

(oxidant), and inert gas proportions are varied between trials until:

4.1.1 *L*—The lowest oxygen (oxidant) concentration for which flame propagation is possible for at least one combination of fuel and inert gas (the “worst case” or most flammable fuel concentration range), and

4.1.2 *H*—The highest oxygen (oxidant) concentration for which flame propagation is not possible for the same worst case fuel concentration range, are identified.

## 5. Significance and Use

5.1 Knowledge of the limiting oxygen (oxidant) concentration is needed for safe operation of some chemical processes. This information may be needed in order to start up or operate a reactor while avoiding the creation of flammable gas compositions therein, or to store or ship materials safely. NFPA 69 provides guidance for the practical use of LOC data, including the appropriate safety margin to use.

## 6. Limitations

6.1 These test methods are 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 flame-quenching 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 Bureau of Mines Bulletins 503 and 627. For certain chemicals (for example, ammonia, halogenated materials, and certain amines) which have large ignition-quenching distances, tests may need to be conducted in vessels larger than that specified below.

## 7. Apparatus

7.1 The test vessel must have a volume of at least 4 L.

NOTE 1—A survey of practitioners of this method indicates that test vessels in the size range of 4 to 35 L are used.

7.2 Test vessels must be nearly spherical. The maximum aspect ratio of the test vessel (the ratio of largest to smallest internal dimension) must be smaller than or equal to two.

7.3 Test vessel may be equipped with a means of mechanical agitation to ensure uniform mixing of components before an ignition attempt.

7.4 If tests are to be conducted at an elevated temperature, the test vessel may be heated using a heating jacket, heating mantle or placed inside a heated chamber. The heating system must be capable of controlling the gas temperature inside the test vessel to within  $\pm 3^\circ\text{C}$  both temporally and spatially. An appropriate device such as a thermocouple should be used to monitor the gas temperature within the test vessel.

7.5 Ignition point must be positioned near the center of the vessel and away from any surfaces or obstacles inside the test vessel.

7.6 One design of an acceptable test vessel is described in Appendix X1.

7.7 The maximum allowable working pressure (MAWP) of the test vessel at the maximum test temperature must exceed the maximum expected deflagration pressure.

### 7.8 Pressure Transducers:

7.8.1 *Low-Range Transducer*—A low-range pressure transducer may be used for the purpose of making partial pressure additions of gases and vapors to the test vessel. The transducer and its signal conditioning/amplifying electronics should have an accuracy, precision and repeatability sufficient to accurately resolve the required changes in the gas partial pressure for the component used in lowest concentration. The transducer should be protected from deflagration pressures by means of an isolation valve. A pressure gage may be used if an error analysis is performed to demonstrate that the internal volume of the pressure gage and piping will not significantly affect the test mixture.

7.8.2 *High-Range Transducer*—This transducer has the purpose of measuring the pressure rise on ignition of the gas mixture. It should have sufficient range to withstand the highest pressure it is expected to experience while also having sufficient accuracy and resolution to measure small pressure rises of the order of 7 % of the initial absolute test pressure.

7.8.3 The pressure transducer and recording equipment must have adequate time resolution to capture the maximum rate of pressure rise developed by the combustion event.

7.8.4 Calibration of the pressure transducer and data acquisition system must be verified over the range of pressures at which the system is expected to operate.

7.9 *Ignition Source*—Several possible means of ignition may be used which include those described below. The means of ignition used must be described in the test report.

7.9.1 *Fuse Wire*—A fuse wire igniter can be constructed, for example from a piece of No. 40 (0.076 mm diameter) copper, nichrome or platinum wire fastened to power supply terminals in such manner as to leave a filament of wire between the terminals approximately 10 mm long. A 500 VA/115 V isolating transformer, or a properly sized discrete discharge capacitor circuit will serve as an adequate igniter energy supply.

7.9.2 *Carbon Spark*—Four 2 mm diameter graphite rods wrapped by the leads coming from an electrical pulse generator. The two electrical leads are separated by a 6 to 10 mm distance. The resulting discrete spark is in the form of a surface discharge over the graphite rods.

7.9.3 *Continuous Electric Arc*—An electric arc igniter may consist of a pair of electrodes (steel or graphite) spaced approximately 6 mm apart across which a 30 mA arc of typically less than 1 s duration can be supplied from a 115/15 000 volt transformer (so-called luminous tube transformer).

7.9.4 *Discrete Electric Spark*—An electric spark igniter may consist of a pair of electrodes (steel or graphite) spaced approximately 6 mm apart across which a short duration spark (lasting for typically 1 ms or less) is caused to occur upon a single discharge of a capacitor. The electrical energy stored on or discharged from the capacitor, or both, should be measured and reported. The energy dissipated in the spark gap may also be measured by appropriate means. Use of at least 10 Joules of

nominal (stored) spark energy is recommended.

NOTE 2—Electric arcs and sparks listed in 7.9.3 and 7.9.4 may fail to discharge when testing fuels with high dielectric strength and during tests conducted at a high initial pressure.

**7.9.5 Chemical Igniter**—Some materials (such as chloro-fluoro-carbons) require a higher ignition energy than that can be provided by the electrical means described above. In that case, tests with chemical igniters (for example, electric matches, electrically activated kitchen match heads, or Sobbe igniters) may be necessary to determine the true limiting oxidant concentration (or the flammability limit) as opposed to an “ignitability limit.” If tests are conducted in a sufficiently large vessel, electric matches or Sobbe igniters may be used. However, it should be kept in mind that these igniters produce significantly larger and sometimes multiple ignition kernels than the electrical ignition sources. Chemical igniters are likely to overdrive combustion events in small test vessels, and in that case, measured LOC values are expected to be lower than the actual LOC values. If a chemical igniter is used, the pressure rise from the igniter, by itself, must be determined. During a test, there is also an additional pressure generated by the combustion of the fuel gas within the igniter flame, even though there is no propagation. One way to partially correct for these igniter effects is to use a more stringent ignition criterion than the standard 7 % pressure rise. Appropriate ignition criterion may be determined from a series of baseline tests conducted on actual fuel-oxidant-diluent mixtures chosen near the non-flammable vicinity of the composition H defined in Section 4.

NOTE 3—Igniters dissipating large quantities of energy (especially chemical igniters) are capable of producing a finite pressure rise in the smaller test vessels, even in the absence of flammable test mixtures. The pressure rise due to igniter must be quantified before the LOC testing, and must be subtracted from the peak pressure rise measured at each test (see 10.1.11). If the pressure rise due to igniter is a non-negligible fraction of the absolute pressure of the test mixture, the accompanying compressive heating of the test mixture must be considered.

NOTE 4—Some igniters may not be capable of dissipating all or any of their rated energy at the extremes of pressure and temperature. If there is any doubt, the reliability of the igniter function must be demonstrated at the test conditions.

## 8. Safety Precautions

8.1 Adequate shielding must be provided to prevent injury in the event of equipment rupture. The apparatus should be set up so that the operator is isolated 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. The test vessel may be fitted with a rupture disk vented to a safe location.

8.2 In the selection of the safe location for the vessel discharge, whether it is through discharge piping or through a rupture disk, full consideration should be given to the safety of the personnel, environment and property. The impact of both unburnt and burnt test mixture venting must be considered, and

necessary protection and mitigation measures must be implemented.

8.3 If the fuel can inadvertently be vented inside the heated chamber or inside the enclosed area, the heated chamber should be fitted with an inert gas purge or the area should be adequately ventilated to prevent buildup of a flammable mixture in the large space.

8.4 It is recommended that LOC evaluations be performed at atmospheric pressure prior to conducting evaluations at elevated initial pressure. This measure should provide baseline data which will help to avoid unexpectedly energetic explosions at high initial pressure.

8.5 Where the LOC is expected to exceed 21 %, testing should begin at 21 % oxygen and the oxygen concentration should be increased in small increments.

NOTE 5—The maximum deflagration pressure that can be developed during the test should be estimated by a suitable means, before testing.

8.6 Test matrix must be planned carefully to avoid testing of detonable mixtures.

8.7 Compressed gas cylinders should be secured by means appropriate to the size of cylinder. Gas cylinder valves should be closed when not in use. Gas cylinders should be fitted with pressure regulators of the correct pressure range and type suited for use with the gas contained therein. Regulator delivery pressure should be set to the lowest value required for efficient gas transfer. The use of check valves in gas supply lines is recommended. All connections in gas transfer lines should be checked for tightness.

## 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 test system components and check for leak points.

9.3 Verify that the test system is at the required operating temperature and check for leaks.

## 10. Procedure

10.1 Two different test methods are used depending on the state of the fuel at room temperature:

### TEST METHOD A—WHERE SAMPLE COMPONENTS ARE GASES AT ROOM TEMPERATURE

10.1.1 Connect the gas supply lines to the manifold of metering valves and flush the lines.

10.1.2 Verify that the test vessel has been thoroughly purged of gases from prior tests.

10.1.3 Evacuate the test vessel and manifold, as required. By use of the valves, add to the test vessel the component most appropriately added first; usually, this is the component to be used in the smallest amount. Record the partial pressure of this component using the low range-transducer.

10.1.4 If the internal volume of the manifold or any piping system connected to the test vessel is appreciable compared to the test vessel volume, purging, evacuation or other measures must be implemented to ensure the accuracy of the test mixture.

10.1.5 Add the second component up to the desired pressure, as measured by the transducer. Repeat the procedure to introduce other 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. Where the vessel configuration will permit, an internal mixing device may be used.

10.1.6 Allow the test gas mixture to equilibrate in temperature and pressure after the addition of each component.

NOTE 6—If the pressure and temperature do not achieve a steady state value after a component is added, this may indicate reaction prior to ignition. Reaction of oxygen (oxidant) will probably cause a pressure rise.

10.1.7 Close the valve between the test vessel and the low-range pressure transducer in order to protect this transducer from deflagration pressure.

10.1.8 Ensure uniform mixing of the components before attempting ignition. The mixing may be achieved by an internal fan, recirculation, or rapid addition of the major component which is added last. Mixture uniformity may be confirmed by gas analysis.

10.1.9 Test mixtures prepared using the partial pressure method may not have the composition expected, due to non-ideal gas behavior. The composition error may 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 test vessel, the greater will be the difference from expected composition. Early in the test series an appropriate method such as gas analysis may be used to confirm composition of gas mixtures made ready for the test. Alternatively, an appropriate non-ideal gas mixture model may be used to quantify the magnitude of the concentration errors. This information may be used to correct the test mixture compositions and to set error bounds on the reported data.

10.1.10 Carbon deposits are likely to affect test results. The test vessel should be inspected periodically to determine whether carbon deposits may have formed on the vessel walls. Such deposits should be removed by suitable means which may include mechanical cleaning, fuel-lean deflagrations, or other cleansing techniques.

10.1.11 Record the initial temperature and pressure (absolute) of the test mixture in the test chamber.

10.1.12 Activate the ignition source.

10.1.13 Record the maximum pressure achieved. Subtract from the maximum pressure the initial pressure and the pressure contribution due to action of the ignition source alone. The result is the net pressure rise due to combustion. Record the result as a *Go* (ignition and subsequent flame propagation) if the net pressure rise is greater than or equal to 7 % of the initial test pressure. Otherwise classify the result as a *No-Go* (*non-ignition*).

10.1.14 Vent the test vessel through the exhaust valve. Purge the vessel with inert gas.

NOTE 7—The venting should be to a safe location since the vented gases may form a flammable mixture when mixed with air. See 8.2.

10.1.15 *Inspect the Igniter*—If it is a one-time device (for example, fuse wire, or a chemical igniter), verify visually that it has functioned in the intended manner. If it is a multiple use

device (for example, an electric spark) verify that it still conforms to the igniter specifications.

10.1.16 Vary fuel-oxygen (oxidant)-inert concentrations (partial pressures) as required to find the limiting oxygen (oxidant) concentration for all possible combinations of the specified fuel and inert components. The most flammable (worst case) fuel-inert concentration combination is likely to vary with oxygen (oxidant) concentration. A number of search algorithms can be devised and used to find the LOC. The following procedures have been used successfully:

10.1.16.1 Calculate or estimate the stoichiometric fuel concentration for the oxidizing gas (air or other oxidant being used). Appendix X2 provides a calculation procedure for a generalized fuel and air. Use this fuel concentration in the first series of tests. Conduct first test near an estimated value of the LOC. A composition with an oxygen (oxidant) volume fraction of 10 to 12 % may serve as a practical starting point in many cases.

NOTE 8—In order to keep the number of tests at a minimum, different LOC search algorithms may be used for different fuels. The stoichiometric fuel concentration is proportional to the oxygen (oxidant) concentration as the oxygen concentration is reduced to reach the LOC. The most reactive (worst case) mixture usually falls on the rich side of the stoichiometric concentration. Therefore, keeping the fuel concentration, rather than the mixture stoichiometry, constant for the first test series may provide a more efficient convergence to LOC in higher hydrocarbons. On the other hand, following the stoichiometric line may be more efficient for lower hydrocarbons. If the test procedure is followed correctly, the LOC result will be independent of the initial search algorithm used.

10.1.16.2 Calculate the partial pressures of fuel, oxygen (oxidant), and inert required to both achieve the desired fuel concentration and to establish the desired test oxygen (oxidant) concentration.

10.1.16.3 Vary the oxygen (oxidant) concentration until a preliminary value of the minimum oxygen concentration that supports combustion is found. Then vary the fuel to find the range of concentrations that are flammable. Then conduct tests at the next lower oxygen concentration to determine if any mixtures are ignitable.

10.1.16.4 Repeat this procedure until the lowest oxygen (oxidant) concentration which supports combustion has been determined for any combination of components, that is, the “worst case”.

10.1.17 Using some search algorithm such as those in 10.1.14, the proportions of fuel, oxygen (oxidant) (air), and inert gas are varied to identify:

10.1.17.1 *L*—The lowest oxygen (oxidant) concentration for which flame propagation is possible for at least one combination of fuel and inert gas (the “worst case” or most flammable fuel concentration range), and

10.1.17.2 *H*—the highest oxygen (oxidant) concentration for which flame propagation is not possible for the same worst case fuel concentration range.

10.1.17.3 At the LOC, the span between *L* and *H* should be no more than 1.0 vol %.

NOTE 9—This definition will result in the fuel - inert gas mixture compositions for *H* and *L* coinciding with each other.

10.1.18 An aid to selecting new fuel concentration values is a graphical plot (see Appendix X3) of the *L* and *H* values on a