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Standard Test Methods for Limiting Oxygen (Oxidant) Concentration in Gases and Vapors¹

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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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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.*

1.6 *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.*

1.7 *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.*

¹ These test methods are under the jurisdiction of ASTM Committee E27 on Hazard Potential of Chemicals and are the direct responsibility of Subcommittee E27.04 on Flammability and Ignitability of Chemicals.

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2. Referenced Documents

2.1 *ASTM Standards*:²

E1445 Terminology Relating to Hazard Potential of Chemicals

2.2 *CGA Publication*:³

CGA P-23 Standard for Categorizing Gas Mixtures Containing Flammable and Nonflammable Components, 2015

2.3 *ISO Publication*:⁴

ISO 10156 Gases and Gas Mixtures — Determination of Fire Potential and Oxidizing Ability for the Selection of Cylinder Valve Outlets, 2010

2.4 *NFPA Publications*:⁵

NFPA 69 Standard on Explosion Prevention Systems
NFPA 86 Standard for Ovens and Furnaces

3. Terminology

3.1 *Definitions*—See also Terminology E1445.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *flammable, n*—capable of propagating a flame.

3.2.2 *ignition, n*—the initiation of combustion.

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

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

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

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.

³ Available from Compressed Gas Association (CGA), 14501 George Carter Way, Suite 103, Chantilly, VA 20151, <http://www.cganet.com>.

⁴ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

⁵ Available from National Fire Protection Association (NFPA), 1 Batterymarch Park, Quincy, MA 02169-7471, <http://www.nfpa.org>.

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 (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.

NOTE 1—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 (1, 2).⁶

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.

5.2 Examples of LOC data applications can be found in references (3-5).

NOTE 2—The LOC values reported in references (6-8), and relied upon by a number of modern safety standards (such as NFPA 69 and NFPA 86) were obtained mostly in a 5-cm diameter flammability tube. This diameter may be too small to mitigate the flame quenching influence impeding accurate determination of the LOC of most fuels. The 4-L minimum volume specified in Section 7 would correspond to a diameter of at least 20 cm. As a result, some LOC values determined using these test methods are approximately 1.5 vol % lower than the previous values measured in the flammability tube, and are more appropriate for use in fire and explosion hazard assessment studies.

5.3 Much of the previous literature LOC data (6-8) were measured in the flammability tube.

5.4 Accepted LOC values (when nitrogen is the inert gas) determined for the five reference gases using these test methods in 20-L and 120-L test enclosures have been reported in Zlochower (9), and are summarized below:

Hydrogen—4.6 % in 120-L, 4.7 % in 20-L

Carbon Monoxide—5.1 % in 120-L

Methane—11.1 % in 120-L, 10.7 % in 20-L

Ethylene—8.5 % in 120-L, 8.6 % in 20-L

Propane—10.7 % in 120-L, 10.5 % in 20-L

⁶ The boldface numbers in parentheses refer to the list of references at the end of this standard.

NOTE 3—For carbon monoxide, results are sensitive to the humidity of the test mixture in the enclosure. Presence of a small concentration of water vapor facilitates combustion and promotes flame propagation by supplying the hydrogen (H) and hydroxyl (OH) free radicals for the chain branching reactions. For conservative results, provisions are made to humidify the test air to near saturation.

5.5 These test methods are often used to determine the LFL (lower flammability limit) and UFL (upper flammability limit) of gases and vapors initially at or near atmospheric pressure. Accepted LFL and UFL values determined for the five reference gases using these test methods have been reported in Zlochower (9).

5.6 These test methods are also used to determine the maximum content of flammable gas which, when mixed with specified inert gas, is not flammable in air (ISO 10156, CGA P-23).

5.7 A minimum purity of 99 % is recommended for the standard reference gases used for the commissioning (qualification) of the test apparatus and for the periodic verification of data quality.

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 Bulletin 503 (6) and Bulletin 627 (7). 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 4—A survey of practitioners of these test methods indicates that test vessels in the size range of 4 to 120 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 5—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 6—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 7—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.