Designation: E2931 – 13

Standard Test Method for Limiting Oxygen (Oxidant) Concentration of Combustible Dust Clouds[1]

This standard is issued under the fixed designation E2931; 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 test method is designed to determine the limiting oxygen concentration of a combustible dust dispersed in a mixture of air with an inert/nonflammable gas in a near-spherical closed vessel of 20 L or greater volume.

1.2 Data obtained from this method provide a relative measure of the deflagration characteristics of dust clouds.

1.3 This test method should be used to measure and describe the properties of materials 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 that takes into account all of the factors that are pertinent to an assessment of the fire hazard of a particular end use.

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 *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 precautionary statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke

D3175 Test Method for Volatile Matter in the Analysis Sample of Coal and Coke

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E1226 Test Method for Explosibility of Dust Clouds

E1515 Test Method for Minimum Explosible Concentration of Combustible Dusts

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

2.2 *NFPA Publications:*[3]

NFPA 69 Standard on Explosion Prevention Systems

2.3 *CEN/CENELEC Publications:*[4]

EN 14034–4 Determination of the explosion characteristics of dust clouds—Part 4: Determination of the limiting oxygen concentration LOC of dust clouds

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *dust concentration*—the mass of dust divided by the internal volume of the test chamber.

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

3.1.3 *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.

3.1.3.1 *Discussion*—The Limiting Oxygen (oxidant) Concentration is sometimes also known as *Minimum Oxygen (oxidant) Concentration* or as *Critical Oxygen (oxidant) Concentration*.

3.1.4 $P_{ignition}$—the absolute pressure at the time the ignitor is activated, see Fig. 1.

3.1.5 $\Delta P_{ignitor}$—the pressure rise in the chamber due to the ignitor by itself in air at atmospheric pressure.

3.1.6 $P_{ex,a}$—the maximum explosion pressure (absolute) reached during the course of a single deflagration test.

---

[1] This test method is under the jurisdiction of ASTM Committee E27 on Hazard Potential of Chemicals and is the direct responsibility of Subcommittee E27.05 on Explosibility and Ignitability of Dust Clouds.

Current edition approved June 1, 2013. Published July 2013. DOI: 10.1520/E2931-13.

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

[4] Available from European Committee for Standardization (CEN), Avenue Marnix 17, B-1000, Brussels, Belgium, http://www.cen.eu.

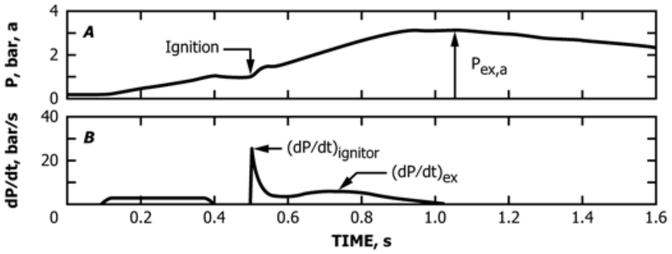

FIG. 1 Typical Data for a Weak Dust Deflagration in a 20-L Chamber

3.1.7 *pressure ratio (PR)*—defined as $PR = (P_{ex,a} - \Delta P_{ignitor}) / P_{ignition}$.

3.1.8 $(dP/dt)_{ex}$—the maximum rate of pressure rise during the course of a deflagration test. The rate of pressure rise can be size-normalized by multiplying by the cube root of the chamber volume, giving $(dP/dt)_{ex} \times V^{1/3}$.

4. Summary of Test Method

4.1 A dust cloud is formed in a closed combustion chamber by dispersion of the material with air and an inert gas (such as nitrogen, carbon dioxide, argon, etc.). The test is normally made at atmospheric pressure and ambient temperature. Proportions of the gaseous components (oxygen and inert gas) are determined by a suitable means.

4.2 Ignition of the mixture is attempted after a specified delay time, and flammability is determined from the pressure rise produced. Fuel, oxygen (oxidant), and inert gas proportions are varied between trials until the following are determined:

4.2.1 *L*—The lowest oxygen (oxidant) concentration for which flame propagation is possible for at least one dust concentration (typically the "worst case" or most flammable fuel concentration range), and

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

5. Significance and Use

5.1 This test method provides a procedure for performing laboratory tests to evaluate relative deflagration parameters of dusts.

5.2 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, shut down or operate a process while avoiding the creation of flammable dust-gas atmospheres therein, or to pneumatically transport materials safely. NFPA 69 provides guidance for the practical use of LOC data, including the appropriate safety margin to use.

5.3 Since the LOC as measured by this method may vary with the energy of the ignitor and the propagation criteria, the LOC should be considered a relative rather than absolute measurement.

5.4 If too weak an ignition source is used, the measured LOC would be higher than the "true" value and would not be sufficiently conservative. This is an ignitability limit rather than a flammability limit, and the test could be described as "underdriven." Ideally, the ignition energy is increased until the measured LOC is independent of ignition energy (that is, the "true" value). However, at some point the ignition energy may become too strong for the size of the test chamber, and the system becomes "overdriven." When the ignitor flame becomes too large relative to the chamber volume, a test could appear to result in an explosion, while it is actually just dust burning in the ignitor flame with no real propagation beyond the ignitor (**1-3**).[5] This LOC value would be overly conservative.

5.5 The recommended ignition source for measuring the LOC of dusts in 20-L chambers is a 2500-J pyrotechnic ignitor.[6] This ignitor contains 0.6 g of a powder mixture of 40 % zirconium, 30 % barium nitrate, and 30 % barium peroxide. Measuring the LOC at several ignition energies will provide information on the possible overdriving of the system to evaluate the effect of possible overdriving in a 20-L chamber, comparison tests may also be made in a larger chamber such as a 1-m^3 chamber (**1-3**).

5.6 The values obtained by this testing technique are specific to the sample tested (particularly the particle size distribution) and the method used and are not to be considered intrinsic material constants.

NOTE 1—Much of the previously published LOC data (**4**). were obtained using a spark ignition source in a 1.2-L Hartmann chamber and may not be sufficiently conservative. The European method of LOC determination EN 14034–4 uses two 1000-J pyrotechnic igniters in the 20-L chamber.

6. Interferences/Limitations

6.1 Unburned dust or combustion products remaining in the chamber or disperser from a previous test may affect results. The chamber and disperser should both be cleaned thoroughly before each test is made.

6.2 Carbon dioxide should not be used as the diluent gas when determining the LOC for most metal dusts because it may react with the metal. Some diluent gases might react with the material to be tested. For example, it is known that carbon dioxide will react with some metals such as aluminum, magnesium, titanium and zirconium.

6.3 This test method is limited to mixtures which have maximum deflagration pressures less than the maximum working pressure of the test apparatus.

6.4 This test method may be used up to the temperature limit of the test system. Note that the ignitors may melt at some elevated temperatures if the test is not performed quickly enough.

---

[5] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[6] The sole source of supply of the pyrotechnic ignitors known to the committee at this time is Cesana Corp., P.O. Box 182, Verona, NY 13478, or Fr. Sobbe, GmbH, Beylingstrasse 59, Postfach 140128, D-4600 Dortmund-Derne, Germany. 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. Apparatus

7.1 The equipment consists of a closed steel combustion chamber with an internal volume of at least 20 L, spherical or cylindrical (with a length to diameter ratio between 1.3:1 and 0.7:1) in shape.

7.2 The vessel should be designed and fabricated in accordance with the ASME Boiler and Pressure Vessel Code, Section VIII. A maximum allowable working pressure (MAWP) of at least 15 bar is recommended.

7.3 A dispersion reservoir contains the pressurized gas used to disperse the dust.

7.4 The apparatus must be capable of generating a well-dispersed dust cloud of the material.

7.5 Optical dust probes, such as those described in Refs. (**5**) and (**6**), may be used to monitor the uniformity of the dust dispersion.

7.6 The pressure transducer and recording equipment must have a combined response rate that is greater than the maximum measured rate of pressure rise.

7.7 An example of a chamber and specific procedures that have been found suitable are shown in Appendix X1. Additional information and details about suitable chambers may be found in Appendix X1 of Test Method E1226 (Siwek chamber) and Appendix X1 of Test Method E1515 (additional information on Bureau of Mines chamber).

7.8 An analyzer or sensor may be used to verify the oxidizer content of the final pre-ignition gas mixture of a blank test (that is, no ignitor or dust present).

8. Safety Precautions

8.1 Prior to handling a dust, the toxicity of the sample and its combustion products must be considered. This information is generally obtained from the manufacturer or supplier. Appropriate safety precautions must be taken if the material has toxic or irritating characteristics. Tests using this apparatus should be conducted in a ventilated hood or other area having adequate ventilation.

8.2 Before initiating a test, a physical check of all gaskets and fittings should be made to prevent leakage.

8.3 If chemical ignitors are used as an ignition source, safety in handling and use is a primary consideration. Premature ignition by electrostatic discharge or other means must be considered a possibility. When handling these ignitors, eye protection must be worn at all times. A grounded, conductive tabletop is recommended for preparation. Federal, state, and local regulations for the procurement, use, and storage of chemical ignitors must be followed.

8.4 All testing should initially be conducted with small quantities of sample to prevent overpressurization due to high energy material.

8.5 Explosive, highly reactive, or easily decomposed materials should not be tested unless they have been characterized by prior testing. Procedures such as the use of barricades, hoods, and personal protective equipment should be used as judgment indicates.

8.6 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.

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

8.8 Where oxidizers stronger than air are used, the potential safety consequences of enhanced reactivity must be addressed.

8.9 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. Sampling, Test Specimens, and Test Units

9.1 It is not practical to specify a single method of sampling dust for test purposes because the character of the material and its available form affect selection of the sampling procedure. Generally accepted sampling procedures should be used as described in the Manual on Test Sieving Methods, MNL 32.[7]

9.2 Tests may be run on an as-received sample. However, due to the possible accumulation of fines at some location in a processing system, it is recommended that the test sample be at least 95 % minus 200 mesh (75 µm).

9.3 To achieve this particle fineness (≥95 % minus 200 mesh (75 µm)), the sample may be ground or pulverized or it may be sieved.

NOTE 2—The operator should consider the thermal stability and the friction and impact sensitivity of the dust during any grinding or pulverizing operation. In sieving the material, the operator must verify that there is no selective separation of components in a dust that is not a pure substance.

9.4 Dust samples that are much finer than 200 mesh (75 µm) may have even lower LOC values.

NOTE 3—It may be desirable in some cases to conduct dust deflagration tests on materials as sampled from a process because process dust streams may contain a wide range of particle sizes or have a well-defined specific moisture content, materials consisting of a mixture of chemicals may be selectively separated on sieves and certain fibrous materials which may not pass through a relatively coarse screen may produce dust deflagrations. When a material is tested in the as-received state, it should be recognized that the test results may not represent the most severe dust deflagration possible. Any process change resulting in a higher fraction of fines than normal or drier product than normal may increase the explosion severity and decrease the LOC value.

9.5 The moisture content of the test sample should not exceed 5 % in order to avoid test results of a given dust being noticeably influenced.

---

[7] Manual on Test Sieving Methods, MNL 32, is available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

NOTE 4—There is no single method for determining the moisture content or for drying a sample. ASTM lists many methods for moisture determination in the Annual Book of ASTM Standards. Sample drying is equally complex due to the presence of volatiles, lack of or varying porosity, and sensitivity of the sample to heat. Therefore, each must be dried in a manner that will not modify or destroy the integrity of the sample. Hygroscopic materials must be desiccated. (Test Methods D3173 and D3175 may be used to measure moisture and volatility, respectively.)

10. Calibration and Standardization

10.1 Because a number of factors (uniformity of dispersion, ignition energy, etc.) can affect the test results, any test vessel design other than that listed in the appendix or that given in EN 14034–4 must be standardized using dust samples whose limiting oxygen values are known. A minimum of five dust samples over a range of LOC values is required. The LOC for each dust must agree to within the repeatability and reproducibility limits stated in Section 14. The comparison must be made using the same dust, ignitor energy, and chamber volume.

10.2 Representative LOC data (**2**) in %O_2 for seven dusts are listed in Table 1. The precision of the data in Table 1 is ±0.5 % oxygen. For the 20-L chamber data, the ignitor was near the wall and pointed horizontally across the chamber. For the 1-m^3 chamber data, there were two 5-kJ ignitors, pointed down at 90º apart. The gilsonite (an asphaltic material mined in Utah) has ~91 % minus 200 mesh (75 µm), a mass median diameter of ~28 m, and 82–84 % volatility. The Pittsburgh seam bituminous coal has a volatility of ~36 %. The minus 40 mesh (425 µm) Pittsburgh coal has ~24 % minus 200 mesh (75 µm) and a mass median diameter of ~160 m. The pulverized Pittsburgh coal has ~80 % minus 200 mesh (75 µm) and a mass median diameter of ~48 m. The extra fine Pittsburgh coal has 98 % minus 200 mesh (75 µm) and a mass median diameter of 11–12 m. The pulverized Pocahontas seam bituminous coal has ~77 % minus 200 mesh (75 µm), a mass median diameter of ~50 m, and ~17 % volatility. RoRo93 was distributed worldwide by A. Kühner (Basel, Switzerland) as a round robin test material in 1993. It is a 2,2,6,6tetramethylpiperidine derivative (light stabilizer). The aluminum was Alcoa 123 atomized powder with 99 % minus 200 mesh (75 µm).

10.3 In addition to the initial calibration and standardization procedure, at least one reference dust should be retested periodically to verify that the dispersion and other characteristics of the chamber and oxidizer/inert gas mixture preparation have not changed.

TABLE 1 Representative LOC Data (2) in %O_2 for Seven Dusts

Dust	NOISH-PRL 20-L Chamber^A			Fike 1-m^3 Chamber^B
	1 000 J	2 500 J	5 000 J	10 000 J
Gilsonite	. . .	10½ %	8½ %	11½ %
Pittsburgh coal, –40 mesh	. . .	12½ %	. . .	. . .
Pittsburgh coal, pulverized	13½ %	11 %	9½ %	13½ %
Pittsburgh coal, extra fine	. . .	10½ %	8½ %	. . .
Pocahontas coal, pulverized	. . .	13½ %	. . .	. . .
RoRo93	. . .	10½ %	8½ %	10½ %
Aluminum	. . .	9½ %	8½ %	9½ %

^{A} 20-L Chamber at Pittsburgh, PA. See Appendix X1 and Refs. (**1**), (**2**), and (**7**).
^{B} 1-m^3 Chamber at Blue Springs, MO. See Refs. Refs. (**1**) and (**2**)

11. Procedure

11.1 These general procedures are applicable for all suitable chambers. The detailed procedures specific to a particular chamber are listed in the appendix or manufacturer's manual. These procedures for dusts are similar to the procedures for gases and vapors in Test Method E2079.

11.2 Inspect equipment to be sure it is thoroughly clean and in good operational condition.

11.3 Place a weighed amount of dust in or on the disperser according to detailed instructions in the appendixes.

11.4 Place ignition source in the chamber. The recommended ignition source for measuring the LOC in 20 L chambers is a 2500 J pyrotechnic ignitor (see 5.4 and 5.5).

11.5 Seal and evacuate the chamber.

11.6 *Pre-Mixed Test Method*—Use preformulated oxidant mixtures (such as oxygen and nitrogen mixtures of known concentrations in gas cylinders) to fill the dispersion reservoir and to partially fill the test chamber (after first evacuating both). The dispersion gas reservoir pressure and dispersion time are set so that, after the dispersion gas has entered the test chamber, the desired chamber pressure (normally ~1 bar absolute) will be reached prior to initiation of the deflagration test. The oxygen concentration for the test is the oxygen concentration in the preformulated cylinder mixture since the oxygen concentrations are the same in the dispersion reservoir and main chamber. It may be prudent to occasionally verify the oxidizer concentration of the post dispersion gas mixture on a blank test run (that is, no fuel or ignition source present in the chamber).

11.7 *Test Method with Multiple Volumes of Different Oxidant Concentrations*—There are four gas volumes in the test system that may be of different oxygen concentrations. The first volume is the residual air volume in the dispersion reservoir. The second is the pressurized gas mixture that is added to the dispersion reservoir. Note that if the dispersion reservoir is evacuated prior to filling, these two volumes will have the same oxygen concentration and can be considered as one volume. An alternative to evacuation of the reservoir is to pressurize the reservoir with the cylinder gas and bleed the pressure back to atmospheric several times until the residual air in the reservoir is negligible. The third volume is the residual air volume in the main test chamber, if the test chamber is not pre-evacuated and then partially filled with the gas mixture before the test. The fourth volume is the gas mixture injected into the main chamber from the dispersion reservoir (comprised of the first and second volumes). The volume, pressure, and oxygen concentration of each must be considered when determining the final oxygen concentration in the test chamber at the time of ignition. It may be prudent to occasionally verify the oxidizer concentration of the post dispersion gas mixture on a blank test run (that is, no fuel or ignition source present in the chamber).

11.8 Actuate the timing circuit to conduct the test.

NOTE 5—The dust sample is automatically dispersed through a dispersion system into the chamber. The deflagration is then initiated when a defined ignition delay time has elapsed. This effective ignition delay time t_d, is the length of time between the first pressure rise due to dust dispersion and the moment normal pressure has been reached in the chamber and ignition is activated (see Fig. 1). The length of this time defines the degree of turbulence and in some cases the concentration of the dust dispersed in the chamber at the moment of ignition.

11.9 The pressure time curve is recorded on a suitable piece of equipment, such as a high speed chart recorder, a storage oscilloscope, or a computer based data acquisition system. The explosion data, $P_{ignition}$, $P_{ex,a}$, and dP/dt_{ex}, can be obtained according to Fig. 1 and Fig. 2.

11.10 After the test, open a valve to vent pressure from the chamber. Open the chamber, remove residue, and thoroughly clean the chamber and dispersion system.

11.11 It is recommended that an initial concentration of ~500 g/m^3 be tested. Alternately, the concentration that corresponded to the maximum pressure or rate of pressure, or both, rise according to Test Method E1226 could be used as a starting concentration.

11.12 The criterion for ignition/explosion is $PR=(P_{ex,a} - \Delta P_{ignitor})/P_{ignition} \geq 2$. The tests identified as ignitions or "go's" have explosion pressures greater than the criterion and the nonignitions or "no go's" have pressures less than the criterion.

NOTE 6—The European method of LOC determination EN 14034–4 uses an ignition/explosion criterion of $P_{ex,a} - P_{ignition} \geq 0.3$ bar in the 1-m^3 chamber using two 5-kJ ignitors as an ignition source and $P_{ex,a} - P_{ignition} \geq 0.5$ bar in the 20-L chamber using two 1-kJ ignitors as an ignition source. As a result direct comparisons of ASTM LOC determinations and European CEN/CENELEC LOC determinations may not be possible.

11.13 The explosibility data from individual tests may be plotted versus dust concentration as shown in Fig. 3. The dust concentration must be varied over a wide enough range so that the "worst case" or most flammable concentration is found.

11.14 Using some search algorithm, the dust concentration and the oxygen (oxidant) and inert gas are varied to identify:

11.14.1 *L*—The lowest oxygen (oxidant) concentration for which flame propagation is possible for at least one dust concentration (typically the "worst case" or most flammable fuel concentration), and

11.14.2 *H*—The highest oxygen (oxidant) concentration for which flame propagation is not possible for the same "worst case" dust concentration.

11.14.3 At the LOC, the span between *L* and *H* should be no more than 1.0 vol% oxygen. At least six "no go's" over a range of at least three dust concentrations are necessary to confirm the *H* value.

11.15 An alternative to the criterion in 11.12 can be used if the test is conducted at a starting pressure at ignition of 1 bar. This alternative criterion is a pressure rise:

$$(P_{ex,a} - P_{ignition} - \Delta P_{ignitor}) \geq 1 \text{ bar, gauge} \tag{1}$$

12. Calculations

12.1 The dust concentration is the mass of dust divided by the volume of the test chamber.

12.2 Pressures and rates of pressure rise are determined from pressure-time records. Fig. 1 and Fig. 2 are typical records from which these values are obtained. $P_{ignition}$ is the absolute pressure in the chamber measured at a time just before activation of the ignitor. The value of $P_{ex,a}$ for a test at a given concentration is the highest deflagration pressure (absolute) as shown in Fig. 1(*A*) and (*B*). The value of dP/dt_{ex} for a given test is the maximum slope of the pressure trace Fig. 2 (*A*) or the highest value on the rate of pressure rise trace Fig. 2 (*A*).

12.3 The pressure ratio is:

$$PR = (P_{ex,a} - \Delta P_{ignitor})/P_{ignition} \tag{2}$$

12.4 The ignitions and nonignitions may be plotted as a function of dust concentration and oxygen percent as shown in Fig. 3.

12.5 The values of $\Delta P_{ignitor}$ for the ignition source by itself must be established in the apparatus.

12.6 Calculate the limiting oxygen (oxidant) concentration from the values of L and H determined above as follows:

$$\text{LOC} = (\text{L} + \text{H})/2 \tag{3}$$

12.7 The LOC value should be rounded down to the nearest 0.5 % oxygen, unless the steps in oxygen concentration are fine enough to allow greater precision.

13. Report

13.1 The report should include the following:

13.1.1 Complete identification of the materials tested (including type of dust, source, code numbers, forms, and previous history);

13.1.2 Particle size distribution of the sample as received and as tested;

13.1.3 Moisture or volatile content, or both, of the as-received and as-tested material, if applicable;

13.1.4 Test pressure and temperature, that is, pressure and temperature at time of ignition;

13.1.5 Type and energy of the ignition source;

13.1.6 Description of the test vessel (including volume);

13.1.7 Descriptions of the measurement and data collection system, calibration, and the test procedure may be included;

13.1.8 Method used to determine the test mixture oxidant concentration, for example, cylinder gas mixtures of known

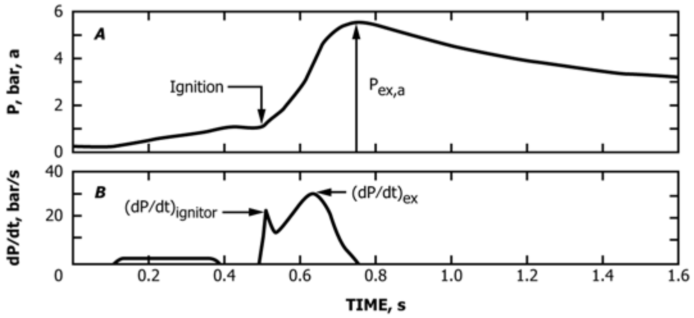

FIG. 2 Typical Data for a Moderate Dust Deflagration in a 20-L Chamber

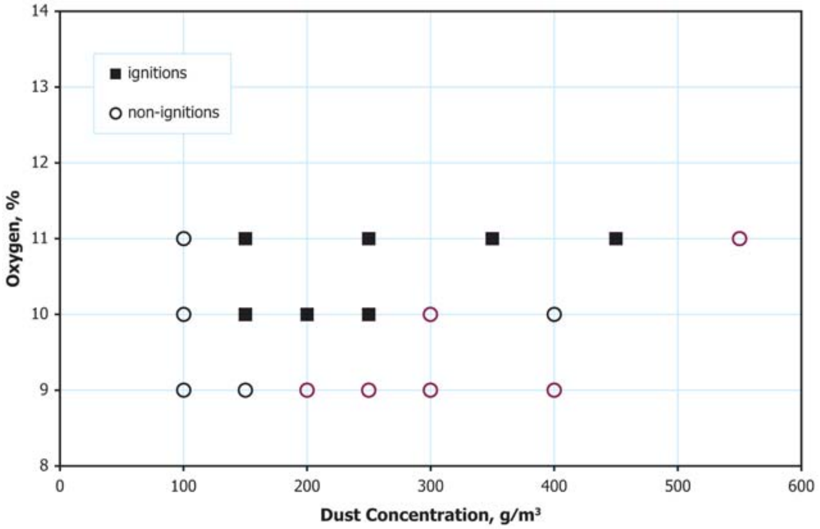

FIG. 3 Typical Test Data Used to Determine the Limiting Oxygen Concentration (LOC) for a Dust

oxygen content, partial pressure method using ideal gas approximation, gas analysis, gas chromatography, etc.;

13.1.9 Any deviations from this standard method;

13.1.10 Table of data (including, for each test, initial temperature and pressure, dust concentration, oxygen concentration, maximum pressure after actuation of ignitor, and classification of result as a *Go* or *No-Go*) may be included;

13.1.11 Graphical presentation of data should be included (see Fig. 3 as example); and

13.1.12 LOC value, plus determined L and H values.

14. Precision and Bias

14.1 The precision of this test method is based on an interlaboratory study conducted in 2012. Ten laboratories participated in the study, testing a single type of food grade starch. Each analyst was asked to report three test results for this study. Practice E691 was followed for the study design; the details are given in ASTM Research Report No. E27-1007.[8]

14.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same paint, obtained by the same operator using the same equipment on the same day in the same laboratory.

14.1.1.1 Repeatability limits are listed in Table 2.

14.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "R" value for that material; "R" is the interval representing the critical difference between two test results for the same paint, obtained by different operators using different equipment in different laboratories.

14.1.2.1 Reproducibility limits are listed in Table 2.

14.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

14.1.4 Any judgment in accordance with statements 14.1.1 and 14.1.2 would have an approximate 95 % probability of being correct.

14.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

14.3 The precision statement was determined through statistical examination of 26 results, from a total of ten laboratories, on a single food grade starch.

15. Keywords

15.1 inerting; limiting oxygen concentration; dust explosions

TABLE 2 Limiting Oxygen Concentration

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	s_R	r	R
Food Grade Starch	12.267	0.447	0.745	1.252	2.085

[A] The average of the laboratories' calculated averages.

---

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E27-1007. Contact ASTM Customer Service at service@astm.org.

APPENDIX

(Nonmandatory Information)

X1. NIOSH-PRL[9] 20-L CHAMBER

X1.1 General Description

X1.1.1 Fig. X1.1 and Fig. X1.2 are vertical and horizontal cross section drawings of the 20-L explosibility test chamber at the Pittsburgh Research Laboratory (PRL) of the National Institute for Occupational Safety and Health (NIOSH). Further details on the chamber and its operation may be found in Refs. (**1**), (**2**), and (**7**). Additional machine drawings of the chamber may be found in Appendix X1 of Test Method E1515.

X1.1.2 The chamber is made of type 304 stainless steel and has a pressure rating of 21 bar. It has a volume of 20 L and a wall thickness of 13 mm. The hinged top is secured with six 19-mm-diameter steel bolts which are not shown on the drawings.

X1.1.3 One or two optical dust probes (**5 and 6**) are normally used to monitor the uniformity of the dust dispersion. The optical probes measure the transmission over a 38 or 95 mm path length through the dust cloud. Thin jets of air keep the windows of the probes clean.

X1.1.4 The absolute pressure is measured with a strain gauge pressure transducer.

X1.1.5 The data from the various instruments are collected by a high speed personal computer based data acquisition system. It can sample data from 16 channels at a maximum rate of 10 kHz if all channels are used or at faster rates if fewer channels are used.

X1.1.6 The dispersion air comes from a 16-L reservoir which is connected by tubing to the chamber. A solenoid valve and a swing check valve are in the air line.

X1.1.7 The standard ignitors are electrically activated, pyrotechnic ignitors with energies of 1000, 2500, or 5000 J. These ignitors contain a mixture of 40 % zirconium, 30 % barium nitrate, and 30 % barium peroxide.

X1.2 Test Procedures

X1.2.1 Calculate and weigh the amount of dust necessary to give the desired dust cloud concentration in the 20-L chamber. Place the dust in the dispersion nozzle or on top of the nozzle if there is too much dust to fit in the nozzle or if the dust particles are too large for the holes in the nozzle.

X1.2.2 Place the ignitor in the ignitor holder which is at a height of 17 cm and is 7 cm from the center axis. When initiated, the ignitor flame will shoot across the center of the chamber toward the opposite wall.

X1.2.3 After the dust and ignitor have been placed in the chamber, secure the top of the chamber with the six bolts.

X1.2.4 Use gas cylinders filled with various mixtures of oxygen and nitrogen for tests at different oxygen concentrations. Confirm the oxygen concentration of each cylinder by appropriate means.

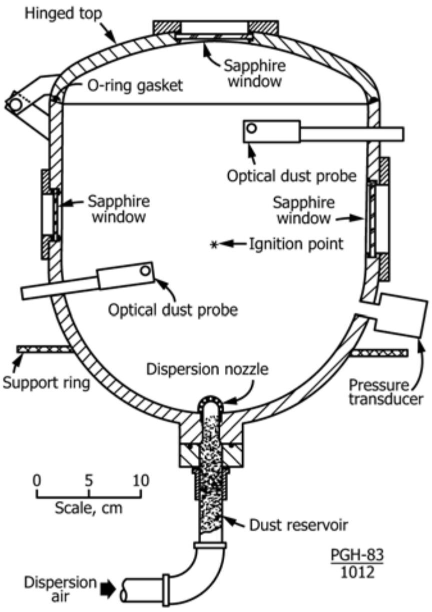

FIG. X1.1 Vertical Cross-Section of NIOSH-PRL 20-L Chamber

---

[9] The Pittsburgh Research Laboratory was part of the U.S. Bureau of Mines before transferring to the National Institute for Occupational Safety and Health (NIOSH) in October 1996.

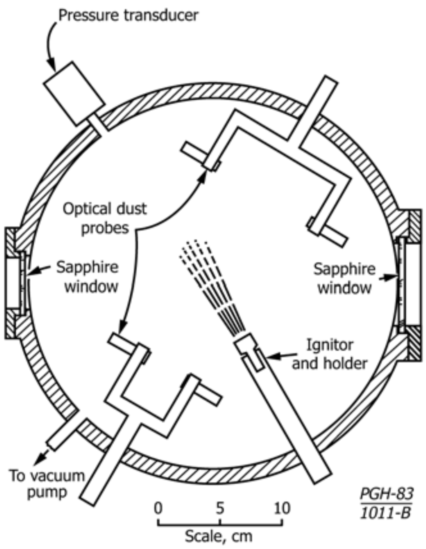

FIG. X1.2 Horizontal Cross-Section of NIOSH-PRL 20-L Chamber

X1.2.5 Evacuate the dispersion reservoir and fill with the gas mixture from the cylinder. An alternative method is to pressurize the reservoir with the cylinder gas and bleed the pressure back to atmospheric several times until the residual air in the reservoir is negligible.

X1.2.6 Totally evacuate the 20-L chamber and then back-fill with the cylinder gas mixture to 0.14 bar absolute.

X1.2.7 Start the automatic timing sequence. A short blast of gas (0.3 s duration at 8–9 bar from the reservoir) disperses the dust and returns the chamber pressure to about 1 bar absolute. There is an additional 0.1 s delay after dispersion before the ignitor is activated. Therefore, the total delay time is 0.4 s. The personal computer automatically collects the data and displays them after the test.

X1.2.8 After the test, the chamber is vented, opened, and thoroughly cleaned.

X1.3 Calculations

X1.3.1 The standard propagation criterion is $PR \geq 2.0$ and $(dP/dt)V^{1/3} \geq 1.5$ bar-m/s. The second part of the criterion is added to require that there be some real propagation of the dust flame and not just a pressure rise due to dust burning within the ignitor flame. This additional criterion for the PRL 20-L chamber partially corrects for the possible overdriving (see 5.4 and 5.5) of the 20-L system by strong ignitors (**1 and 2**).

X1.3.2 If a low *dP/dt* is obtained (Fig. 1 (*A*)), a weak deflagration may have occurred. Under these conditions, it is important that the *dP/dt* measurement is not taken from the ignition source but from the dust-air mixture itself.

REFERENCES

(1) Cashdollar, K. L., and Chatrathi, K., "Minimum Explosible Dust Concentrations Measured in 20-L and 1-m3 Chambers," *Combustion Science and Technology*, Vol 87, 1993, pp. 157–171.

(2) Going, J. E., Chatrathi, K., and Cashdollar, K. L., "Flammability Limit Measurements for Dusts in 20-L and 1-m^3 Vessels," *Journal of Loss Prevention in the Process Industries*, Vol 13, 2000, pp. 209–219.

(3) Siwek, R., "Reliable Determination of the Safety Characteristics in 20 L Apparatus," Paper in Flammable Dust Explosion conference proceedings, November 2–4, 1988, St. Louis, MO, IBC USA, Holliston, MA, 1988, pp. 529–573.

(4) Nagy, J., Dorsett, H. G. Jr., and Jacobson, M., *Preventing Ignition of Dust Dispersions by Inerting*, RI 6543, U.S. Bureau of Mines, 1964, 29 pp.

(5) Cashdollar, K. L., Liebman, I., Conti, R. S., *Three Bureau of Mines Dust Probes*, RI 8542, U.S. Bureau of Mines, 1981, 26 pp.

(6) Conti, R. S., Cashdollar, K. L., and Liebman, I., "Improved Optical Dust Probe for Monitoring Dust Explosions," *Review of Scientific Instruments*, Vol 53, 1982, pp. 311–313.

(7) Cashdollar, K. L., and Hertzberg, M., "20-L Explosibility Test Chamber for Dusts and Gases," Review of Scientific Instruments, Vol 56, 1985, pp. 596–602.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/