Designation: G72/G72M – 15

Standard Test Method for Autogenous Ignition Temperature of Liquids and Solids in a High-Pressure Oxygen-Enriched Environment[1]

This standard is issued under the fixed designation G72/G72M; 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 covers the determination of the temperature at which liquids and solids will spontaneously ignite. These materials must ignite without application of spark or flame in a high-pressure oxygen-enriched environment.

1.2 This test method is intended for use at pressures of 2.1 to 20.7 MPa [300 to 3000 psi]. The pressure used in the description of the method is 10.3 MPa [1500 psi], and is intended for applicability to high pressure conditions. The test method, as described, is for liquids or solids with ignition temperature in the range from 60 to 500 °C [140 to 932 °F].

NOTE 1—Test Method G72/G72M normally utilizes samples of approximately 0.20 +/- 0.03-g mass, a starting pressure of 10.3 MPa [1500 psi] and a temperature ramp rate of 5 °C ⁄min. However, Autogenous Ignition Temperatures (AIT) can also be obtained under other test conditions. Testing experience has shown that AIT testing of volatile liquids can be influenced by the sample pre-conditioning and the sample mass. This will be addressed in the standard as Special Case 1 in subsection 8.2.2. Testing experience has also shown that AIT testing of solid or non-volatile liquid materials at low pressures (i.e., < 2.1 MPa) can be significantly influenced by the sample mass and the temperature ramp rate. This will be addressed in the standard as Special Case 2, in subsection 8.2.3. Since the AIT of a material is dependent on the sample mass/configuration and test conditions, any departure from the standard conditions normally used for Test Method G72/G72M testing should be clearly indicated in the test report.

1.3 This test method is for high-pressure pure oxygen. The test method may be used in atmospheres from 0.5 % to 100 % oxygen.

1.4 An apparatus suitable for these requirements is described. This test method could be applied to higher pressures and materials of higher ignition temperature. If more severe requirements or other oxidizers than those described are desired, care must be taken in selecting an alternative safe apparatus capable of withstanding the conditions.

1.5 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

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

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

G93 Practice for Cleaning Methods and Cleanliness Levels for Material and Equipment Used in Oxygen-Enriched Environments

2.2 *Federal Specification:*

BB-O-925 Oxygen, Technical, Gas and Liquid[3]

2.3 *Other Documents:*

MNL 36 Safe Use of Oxygen and Oxygen Systems: Guidelines for Oxygen System Design, Materials, Selection, Operations, Storage, and Transportation[4]

Compressed Gas Association Booklets G-1 and G-4.1[5]

3. Summary of Test Method

3.1 This autogenous ignition temperature test method is designed to expose solid or liquid sample material to increasing temperature in a high-pressure reaction vessel. The reaction

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[1] This test method is under the jurisdiction of ASTM Committee G04 on Compatibility and Sensitivity of Materials in Oxygen Enriched Atmospheres and is the direct responsibility of Subcommittee G04.01 on Test Methods.

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[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 U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

[4] ASTM Manual Series, Available from ASTM International, 100 Barr Harbor Drive, W. Conshohocken, PA 19428.

[5] Available from Compressed Gas Association (CGA), 4221 Walney Rd., 5th Floor, Chantilly, VA 20151-2923, http://www.cganet.com.

vessel (bomb), including a sample holding assembly, is pressurized with the oxygen-enriched environment. The bomb is heated in an electric furnace at a predetermined rate. The temperature of the sample-holding assembly is monitored as a function of time by means of a thermocouple and recording potentiometer.

3.2 The minimum temperature required to cause the sample to ignite spontaneously is determined at any selected system pressure. The point at which spontaneous ignition occurs is denoted by a sudden rise in temperature and the destruction of the sample. The amount of rise in temperature is related to the sample size. A sample size is selected to prevent damage to the equipment caused by exceeding safe system pressure or temperature limits because of the temperature rise.

3.3 The system is pressurized to the desired test pressure at the start of the test. During the test as the temperature is increased, the pressure increases. No effort is made to control the pressure during the test. It is monitored only so that the pressure does not exceed a safe limit for the test equipment.

4. Significance and Use

4.1 Most organic liquids and solids will ignite in a pressurized oxidizing gas atmosphere if heated to a sufficiently high temperature and pressure. This procedure provides a numerical value for the temperature at the onset of ignition under carefully controlled conditions. Means for extrapolation from this idealized situation to the description, appraisal, or regulation of fire and explosion hazards in specific field situations, are not established. Ranking of the ignition temperatures of several materials in the standard apparatus is generally in conformity with field experience.

4.2 The temperature at which material will ignite spontaneously (AIT) will vary greatly with the geometry of the test system and the rate of heating. To achieve good interlaboratory agreement of ignition temperatures, it is necessary to use equipment of approximately the dimensions described in the test method. It is also necessary to follow the described procedure as closely as possible.

4.3 The decomposition and oxidation of some fully fluorinated materials releases so little energy that there is no clear-cut indication of ignition. Nor will there be a clear indication of ignition if a sample volatilizes, distilling to another part of the reaction vessel, before reaching ignition temperature.

5. Apparatus

5.1 Suitable components shall be assembled so that the specified reaction vessel (bomb), including sample-holding assembly, can be charged with oxygen and heated. The assembly shall provide a means of recording time and temperature at which ignition occurs. A suitable assembly is illustrated in Fig. 1.

5.2 *Cylinder Oxygen,* conforming to Federal Specification BB-O-925, Type I or oxygen of 99.5 % minimum purity. Oxygen of higher purity may be used if desired.

5.3 *Line Filter,* sintered stainless steel, 5-µm porosity, maximum pressure 206.8 MPa [30 000 psi], for 6.35-mm [¼-in.] high-pressure tubing with a 3.18-mm [⅛-in.] port.[6]

5.4 *Compressor Pumps,* diaphragm-type, air-driven.[7]

5.5 *Valves,* 6.35 mm [¼ in.], 206.8 MPa [30 000 psi] working pressure, nonrotating stem valves.[8]

5.6 *Pressure Gage,* 20.7 MPa [3000 psi], 216 mm [8½ in.].[9] Heise 2 or equivalent has been found satisfactory.

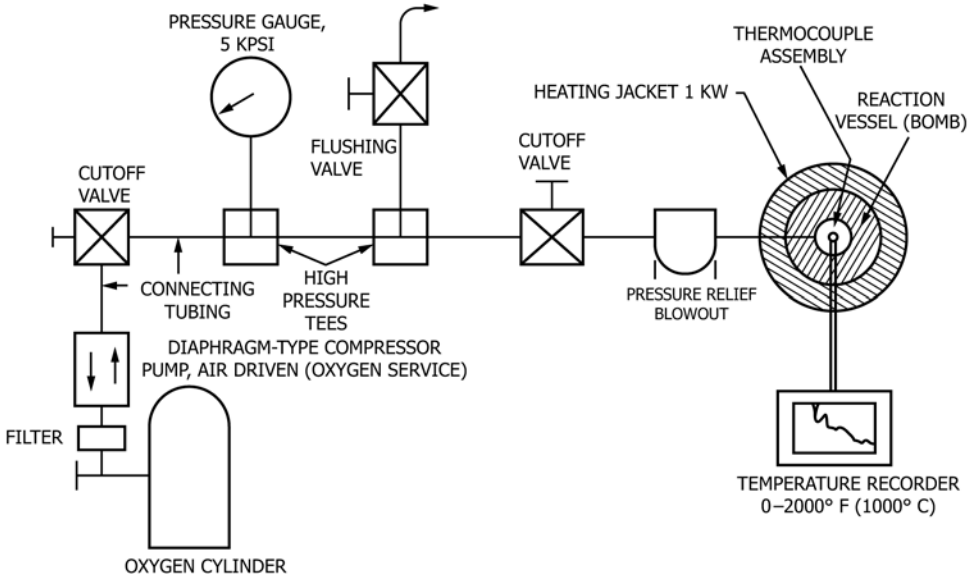

FIG. 1 AIT Equipment Assembly

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[6] Catalog No. 49-14405 available from Superpressure, Inc., Silver Spring, Md. 20910 or equivalent has been found satisfactory.

[7] Catalog No. 46-14035 available from Superpressure, Inc., Silver Spring, Md. 20910 or equivalent has been found satisfactory.

[8] Catalog No. 44-13121 available from Superpressure, Inc., Silver Spring, Md. 20910 or equivalent has been found satisfactory.

[9] Model C available from Heise Bourdon Tube Co., Newton, Conn. 06740 or equivalent has been found satisfactory.

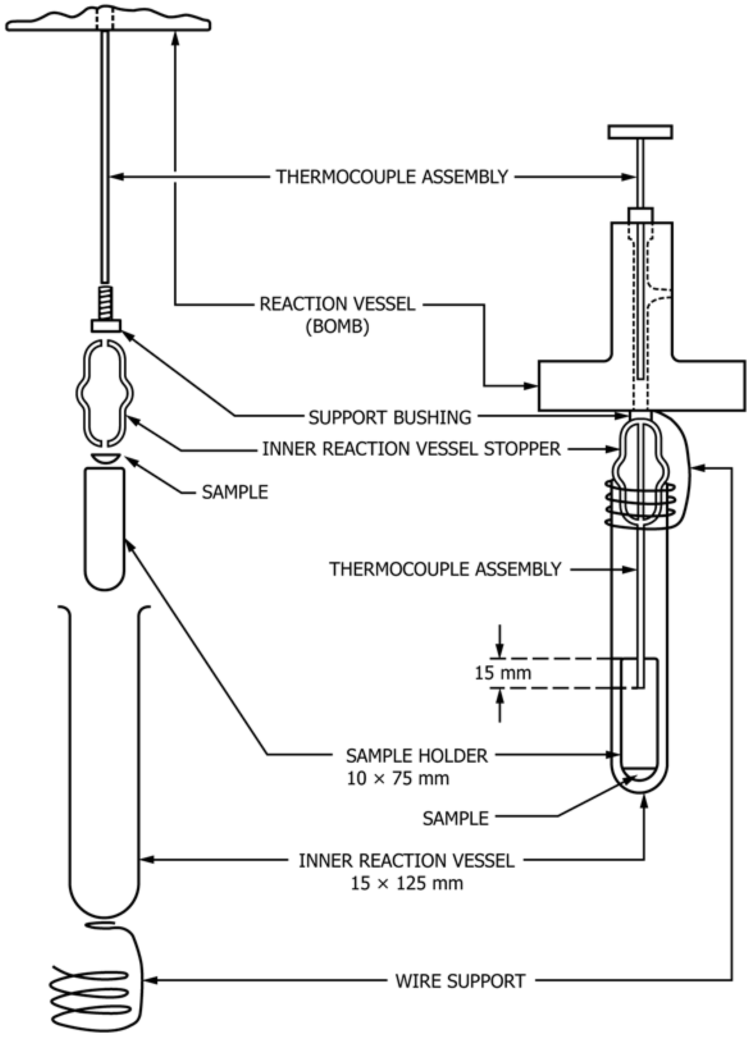

FIG. 2 Sample Holding Assembly

5.7 *Connecting Tubing,* Type 316 stainless steel, 6.35 mm [¼ in.], 448.1 MPa [65 000 psi] pressure rating at 37.8 °C [100 °F].[10]

5.8 *High-Pressure Tees,* Type 316 stainless steel with gland nuts and sleeves of Type 416 stainless steel, 6.35 mm [¼ in.] high-pressure. Superpressure, Inc., Catalog No. 45-14311.[11] All connection fittings shall be of cold-drawn Type 316 stainless steel, 413.7 MPa [60 000 psi] maximum pressure, tubing size 6.35 mm [¼ in.] high-pressure and 14.3-mm [9⁄16-in.] insertion depth.[12]

5.9 *Pressure-Relief Blowout with Rupture Discs,* pressure-relief blow-out assembly, Type 316 stainless steel, 6.35 mm [¼ in.], angle type[13] with 48.3 MPa [7000 psi] at 22.2 °C [72 °F] rupture disks.[14]

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[10] Catalog No. 45-11021 available from Superpressure, Inc., Silver Spring, Md. 20910 or equivalent has been found satisfactory.

[11] Catalog No. 45-14311 available from Superpressure, Inc., Silver Spring, Md. 20910 or equivalent has been found satisfactory.

[12] Catalog No. 45-11311 available from Superpressure, Inc., Silver Spring, Md. 20910 or equivalent has been found satisfactory.

[13] Catalog No. 45-19123 available from Superpressure, Inc., Silver Spring, Md. 20910 or equivalent has been found satisfactory.

[14] Catalog No. 45-19210 available from Superpressure, Inc., Silver Spring, Md. 20910 or equivalent has been found satisfactory.

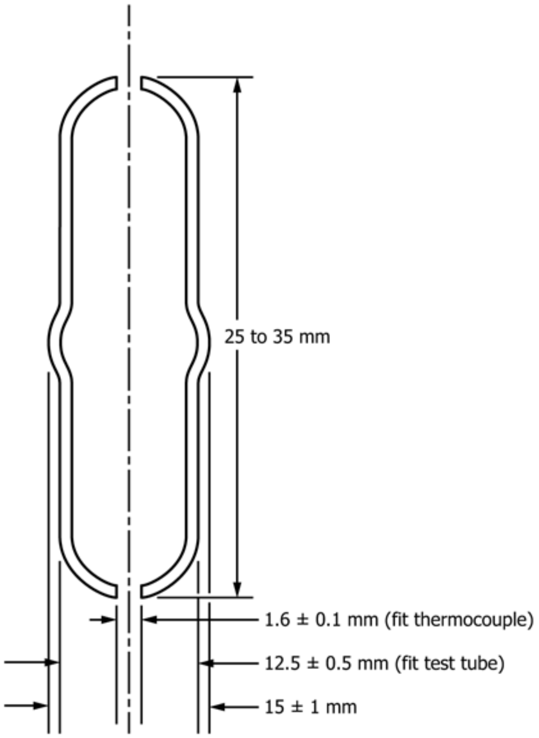

FIG. 3 Inner Reaction Vessel Stopper

5.10 *Reaction Vessel (Bomb)*—A suitable reaction vessel for the test method is cylindrical, approximately 65 mm [$2\frac{9}{16}$ in.] in outside diameter and 298 mm [11¾ in.] long and weighs 9.75 kg [21½ lb]. The vessel is bored from a solid forging of AISI 316SS [8¼ in.] depth, with a volume equal to approximately 110 mL. The maximum working pressure at 427 °C [800 °F] is 82.7 MPa [12 000 psi].[15]

5.11 *Thermocouple Assembly*—A Chromel-Alumel thermocouple with suitable high-pressure fittings for the reaction vessel with a 203-mm [8-in.] thermocouple to extend into the reaction chamber.[16]

5.12 *Heating Jacket*—A 230-V, 1000-W single-phase heating jacket designed to fit the reaction vessel should be used.[17]

5.13 *Recorder*, 0 to 1000 °C [0 to 2000 °F]—A strip chart recording pyrometer in the temperature range for the test method should be used.[18] The scale must be such that a sudden change of 20 °C [36 °F] or more in temperature in the reaction vessel is clearly indicated.

5.14 *Inner Reaction Vessel*—A borosilicate glass test tube 15 by 125 mm.[19]

5.15 *Sample Holder*—A borosilicate glass culture tube 10 by 75 mm.[20]

5.16 *Wire Support,* fashioned from Chromel A, No. 21 AWG wire.[10] Several turns of wire are wound on a mandrel of sufficient size so that the resulting spring-like structure fits the inner reaction vessel snugly. A loop of wire is bent to hold the vessel at the proper height, positioning the thermocouple assembly in the mouth of the sample holder (Fig. 2).

5.17 *Support Bushing,* fitting into the reaction vessel cover and supporting the entire sample-holding assembly.[21]

5.18 *Inner Reaction Vessel Stopper,* fashioned from 12.5-mm borosilicate glass tubing to fit in the inner reaction vessel. It must also conform to the dimensions in Fig. 3.

6. Materials

6.1 *Nitric Acid*—Consisting of 5 % by volume of Analytical Reagent grade nitric acid and deionized water.

6.2 *Alkaline Cleaner*—Consisting of a solution of 15 g of sodium hydroxide (NaOH), 15 g of trisodium phosphate (Na_3PO_4), and 1 L of distilled or deionized water.

6.3 *Deionized or Distilled Water,* conforming to Specification D1193, Type IV.

6.4 *Oxygen,* conforming to Federal Specification BB-0-925, Type I or oxygen of 99.5 % purity. Oxygen of higher purity may be used if desired.

7. Safety Precautions

7.1 *Nitric Acid:*

Warning! Harmful by inhalation, skin contact, and if swallowed.

Although not combustible, is a powerful oxidizing agent, which may cause combustible materials to ignite.

Wear appropriate NIOSH-approved respirator, chemical resistant gloves (Butyl rubber), safety goggles.

7.2 *Sodium Hydroxide:*

Warning! Harmful by inhalation, skin contact, and if swallowed.

Use adequate ventilation.

Wear face shield, lab coat, rubber apron.

Store away from strong acids

7.3 *Oxygen:*

Warning! *Oxygen vigorously accelerates combustion.*

Keep oil and grease away. Do not use oil or grease on regulators, gages, or control equipment.

Use only with equipment conditioned for oxygen service by carefully cleaning to remove oil, grease, and other combustibles.

Keep combustibles away from oxygen and eliminate ignition sources.

Keep surfaces clean to prevent ignition or explosion, or both, on contact with oxygen.

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[15] Type B Reaction Vessel Catalog No. 41-12555, available from Superpressure, Inc., Silver Spring, Md. 20910 or equivalent will meet these requirements.

[16] Thermocouple Assembly Catalog No. 45-17620 available from Superpressure, Inc. or equivalent can be used.

[17] Heating Jacket, Catalog No. 43-12113 available from Superpressure, Inc., or equivalent can be used.

[18] Strip chart recorders available from Honeywell, Inc., 2701 4th Ave., Minneapolis, Minn. 55408 or equivalent can be used.

[19] Catalog No. 9800, available from Corning Glass Works, Houghton Park, Corning, NY 14830 or equivalent can be used.

[20] Catalog No. 9820 available from Corning Glass Works, Houghton Park, Corning, NY 14830 or equivalent has been found satisfactory.

[21] Catalog No. 15-21AF1HM4-T available from High Pressure Equipment Co., 1222 Linden Ave., Erie, PA. 16505 or equivalent has been found satisfactory.

Always use a pressure regulator. Release regulator tension before opening cylinder valve.

All equipment and containers used must be suitable and recommended for oxygen service.

Never attempt to transfer oxygen from cylinder in which it is received to any other cylinder. Do not mix gases in cylinders.

Do not drop cylinder. Make sure cylinder is secure at all times.

Keep cylinder closed when not in use.

Stand away from outlet when opening cylinder valve.

For technical use only. Do not use for inhalation purposes.

Keep cylinder out of sun and away from heat.

Keep cylinder from corrosive environment.

Do not use cylinder without label

Do not use dented or damaged cylinders.

7.3.1 See Compressed Gas Association booklets G-4 and G-4.1 for details of safe practice in the use of oxygen.

8. Procedure

8.1 Clean all components of the system as follows:

8.1.1 Soak glass parts in chromic acid cleaning solution, rinse in distilled water, and dry.

8.1.2 Clean stainless steel components by immersing in an alkaline cleaner (see 6.2) for a minimum of 15 min at 20 to 35 °C. Follow the immersion with a thorough rinse in running tap water, followed by a thorough rinse in distilled or deionized water. Perform a water break test during the rinsing step to verify that organic material has been removed. Blow dry with clean, dry, oil-free nitrogen to remove the excess water, place in an oven at 52 to 66 °C until free of water. Components may be cleaned using any process that will produce a cleanliness level at least as good as the level provided by the above process. Follow Practice G93 or ASTM Manual Series MNL 36 recommended procedures.

8.2 Weigh out a sample into the sample holder.

8.2.1 Standard samples of solid or liquid sample weight should be 0.20 +/- 0.03 g. Samples for volatile liquids or low pressure tests are addressed in Special Cases 1 and 2, as follows.

8.2.2 *Special Case 1*—For volatile liquids such as cleaning solvents, a larger sample weight up to 1.00 +/- 0.10 g may be required to obtain a valid AIT result. It is good practice to pre-chill volatile liquids with boiling points near or below room temperature using an ice bath to prevent excessive loss of solvent prior to test. It is recommended a final weight be taken immediately before test to verify quantity present.

NOTE 2—A lab may choose to incrementally approach the sample size of 1g, evaluating pressure spikes and system safety limits as sample size increments are increased.

NOTE 3—A non-ignition at maximum temperature when testing at lower pressures (<1000 psi) may indicate an insufficient oxidizer-to-fuel ratio. When testing at lower pressures, if obtaining a non-ignition at maximum temperature, it is recommended that testing be performed at higher pressures until an AIT is obtained. If suspected, testing at the standard 1500 psia or higher and increased sample mass (suggested 1.0 g) is recommended to confirm an unreactive material.

8.2.3 *Special Case 2*—For testing at low pressures, less than 2.1 MPa [< 300 psig] a high heating rate may be required to successfully develop an AIT for the material. For low pressure testing, higher heating rates of up to 110 °C/min may be used. Higher heating rates have been successfully used for sample masses ranging from 20 mg to 500 mg. Weigh out a sample with a +/- 15% mass tolerance, either in liquid (non-volatile liquid) or solid form, into the sample holder. Specify whether the sample is prepared whole or divided into pieces. If the samples are prepared in a divided form, provide the number of pieces and maintain consistency between tests to ensure that the surface area-to-volume ratio between samples is maintained.

NOTE 4—When testing at low pressures, two significantly different sample masses should be tested so that a minimum AIT can be determined, and ensure that sample mass influences are minimized. Sample masses of 20 and 200 mg have been used with good repeatability as long as the temperature ramp rate utilized is between 100 and 110 °C/min.

NOTE 5—Testing experience has shown that a higher surface area-to-volume ratio (i.e., divided sample) can produce a lower AIT (Swindells, I., et.al; STP 986).

8.3 Assemble equipment as shown in Fig. 1 and Fig. 2, and as directed by the reaction vessel manufacturer.

8.4 Flush the system twice with oxygen, meeting the requirements of 5.1, by pressurizing the system to 5.0 MPa [725 psi] and releasing the pressure.

8.5 Fill the reaction vessel with the oxygen specified in 7.3 to a pressure of 11.5 MPa [1650 psi] and allow to stand at room temperature for 15 min. The pressure will drop approximately 0.5 MPa [45 psi] while the gas cools, but should remain nearly constant thereafter. A steady pressure drop indicates a system leak which must be corrected before proceeding. After assuring the absence of leaks, adjust the pressure to 10.3 MPa [1500 psi].

8.6 Start the reaction vessel heating jacket and the recorder. Heat the reaction vessel at a rate of 5 ± 1 °C [9 ± 1 °F]/min. This rate of heating should be maintained from 60 to 260 °C [140 to 500 °F]. Above 250°C [500 °F], difficulty may be encountered maintaining this heating rate, but it must be maintained above 3 °C [5 °F]/min.

8.6.1 For Special Case 2 testing (see 8.2.3), set the heating rate as desired and ensure the temperature ramp rate does not vary by more than a +/- 20%.

NOTE 6—Testing experience has shown that higher temperature ramp rates can produce lower AITs (Swindells, I., et.al; STP 986).

8.7 Ignition of the sample is indicated by a rapid temperature rise of at least 20°C [36°F] or pressure rise, or both. When ignition is complete, but not less than 3 min after it starts, turn off the heater and stop the recorder. Release reaction vessel pressure into a suitable exhaust system.

NOTE 7—The pressure at ignition can often spike with trailing oscillations potentially due to a combustion pressure wave(s) traveling and reflecting in the test system. The oscillations have been shown to occur at a set frequency based on the system configuration and volume. This set frequency can be used with a second order band-pass filter to process the pressure data post-test and help identify the ignition point. This data processing can be useful for testing material that exhibits a low heat of combustion or for test samples with a low sample mass (or both), both of which can produce minimal temperature increases and can be difficult to distinguish from the temperature ramp rate, especially when using higher temperature ramp rates.

8.8 If no ignition occurs up to the maximum safe operating temperature of the reaction vessel, stop the heating and release the pressure as above.

8.9 Perform testing on a minimum of 3 samples to obtain an average AIT.

9. Report

9.1 The report shall include the following for each test:

9.1.1 Test atmosphere composition,

9.1.2 Sample weight,

9.1.3 Ignition temperature,

9.1.4 Temperature rise on ignition, and

9.1.5 The system's initial and final gas pressure.

9.2 Also report the pressure rise on ignition and residue appearance in the sample holder.

9.3 Report any volatility or low heat release problems.

9.4 If no ignition occurs up to 500 °C [932 °F], report the ignition temperature as greater than 500 °C [932 °F].

9.5 Report the average AIT for each identical set of test conditions (i.e., pressure, temperature ramp rate, sample mass, and configuration).

10. Precision and Bias

10.1 *Precision*—The precision of this test method is based on an interlaboratory study of ASTM G72 Standard Test Method for Autogenous Ignition Temperature of Liquids and Solids in a High-Pressure Oxygen-Enriched Environment, conducted in 2009. Very few laboratories in the world are capable of performing this test due to the hazards of high-pressure gaseous oxygen; therefore, all five laboratories worldwide known to run this test methodology participated in the round robin. Each of the five labs reported five replicate test results for five different materials. Every "test result" reported represents an individual determination. Except for the use of only five laboratories, Practice E691 was followed for the design and analysis of the data; the details are given in an ASTM Research Report.[22]

10.1.1 *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 material, obtained by the same operator using the same equipment on the same day in the same laboratory.

10.1.1.1 Repeatability limits are listed in Table 1.

10.1.2 *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 material, obtained by different operators using different equipment in different laboratories.

10.1.2.1 Reproducibility limits are listed in Table 1.

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

10.1.4 Any judgment in accordance with statements 10.1.1 and 2.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the repeatability limit and the reproducibility limit as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.

10.2 *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.

10.3 The precision statement was determined through statistical examination of 125 results, from five laboratories, on five materials. These five materials were described as the following:

Material 1: Buna S
Material 2: EPDM
Material 3: HDPE
Material 4: Viton A
Material 5: Zytel 42

10.3.1 Based on the results of the interlaboratory study the repeatability for tests conducted within the individual laboratories ranged from 3.6 to 10.6 ºC depending on the material being tested. The reproducibility between the laboratories participating in the study ranged between 13.4 to 33.8 ºC depending on the material being tested.

NOTE 8—To judge the equivalency of two test results at another laboratory, it is recommended to choose the material closest in characteristics to the test material.

TABLE 1

Material	Average 1 (°C)[A]	Repeatability Standard	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	x	s_r	s_R	r	R
Buna S	162	1.3	4.8	3.6	13.4
EPDM	203	1.9	6.5	5.4	18.2
HDPE	193	2.6	7.6	7.3	21.2
Viton A	306	3.8	12.1	10.6	33.8
Zytel 42	207	3.0	8.5	8.3	23.8

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

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[22] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:G0004-1000.

11. Keywords

11.1 autogenous ignition temperature; ignition temperature; oxygen enriched environment

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