Designation: G 72 – 01

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 G 72; 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 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 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). The method, as described, is for liquids or solids with ignition temperature in the range from 60 to 425°C (140 to 800°F).

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

1.4 An apparatus suitable for these requirements is described. This 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 *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:*

D 1193 Specification for Reagent Water[2]

G 93 Practice for Cleaning Methods and Cleanliness Levels for Material and Equipment Used in Oxygen-Enriched Environments[3]

2.2 *Federal Specification:*[4]

BB-O-925 Oxygen, Technical, Gas and Liquid

2.3 *Other Documents:*

MNL 36 Safe Use of Oxygen and Oxygen Systems: Guidelines for Oxygen System Design, Materilas, Selection, Operations, Storage, and Transportation[5]

Compressed Gas Association Booklets G-1 and G-4.1[2,4]

3. Summary of 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 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

---

[1] This method is under the jurisdiction of ASTM Committee G4 on Compatibility and Sensitivity of Materials in Oxygen Enriched Atmospheres and is the direct responsibility of Subcommittee G04.01 on Test Methods.

Current edition approved Sept. 10, 2001. Published January 2002. Originally published as G 72–82. Last previous edition G 72–82(1996)[e1].

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from Compressed Gas Assn., 500 Fifth Ave., New York, NY 10110.

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

several materials in the standard apparatus is generally in conformity with field experience.

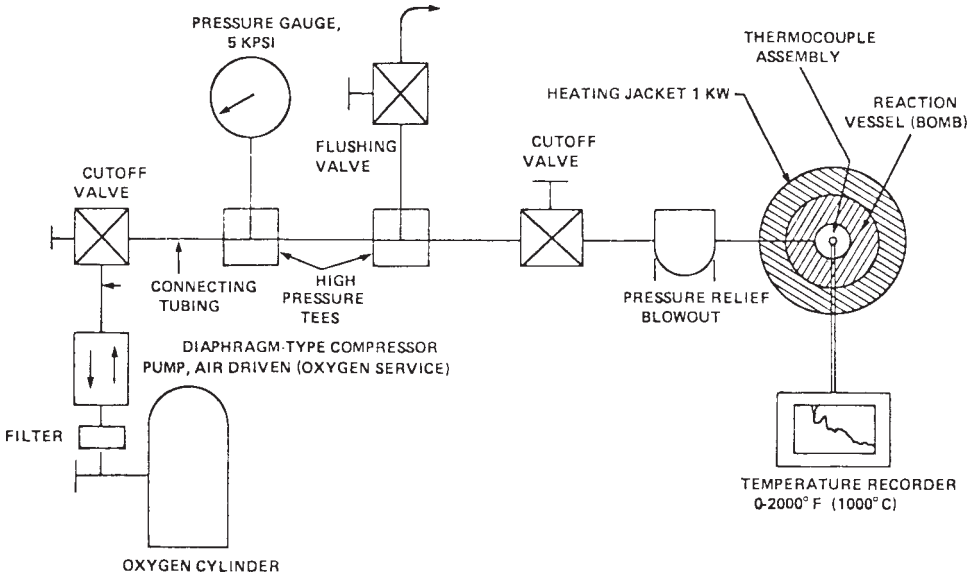

FIG. 1 AIT Equipment Assembly

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 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.

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]

5.10 *Reaction Vessel (Bomb)*—A suitable reaction vessel for the method is cylindrical, approximately 65 mm (2⁹⁄16 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

[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.

[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.

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 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.

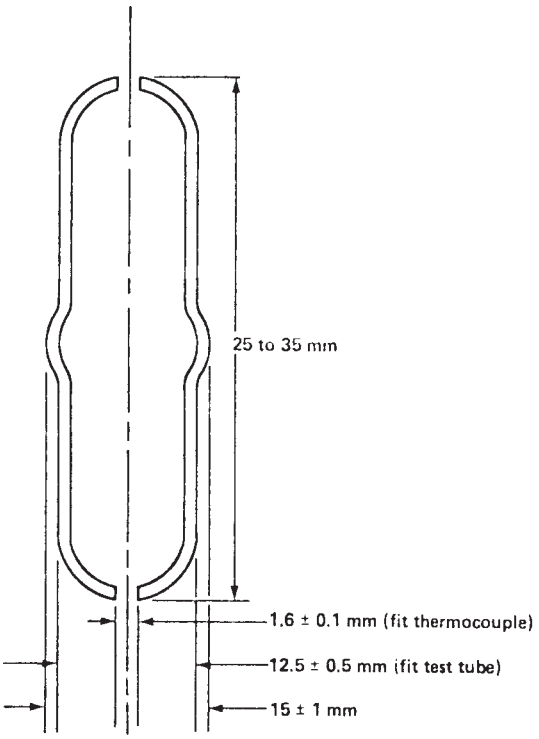

FIG. 3 Inner Reaction Vessel Stopper

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 D 1193, 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.

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.

[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.

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.

process. Follow Practice G 93 or ASTM Manual Series MNL 36 recommended procedures.

8.2 Weigh out a 0.20 ± 0.03-g sample, either in liquid or solid form, into the sample holder.

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

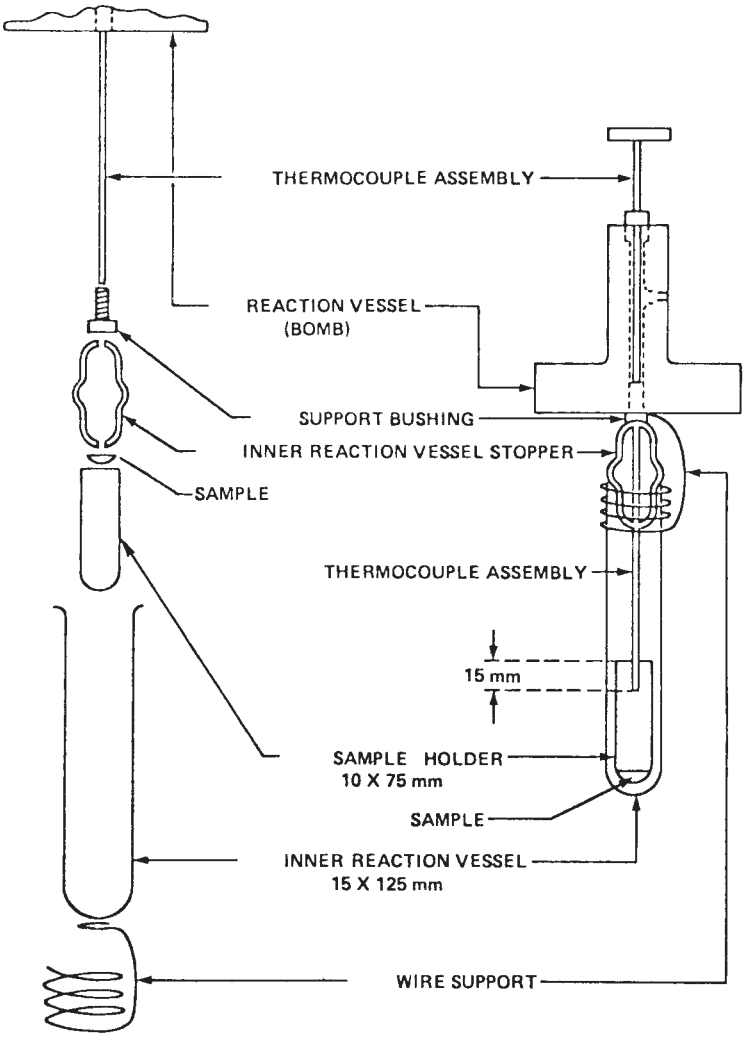

FIG. 2 Sample Holding Assembly

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

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.

8.7 Ignition of the sample is indicated by a rapid temperature rise of at least 20°C (36°F). 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 1—Avoid breathing or physical contact with the decomposition products, which may be toxic, that are vented as pressure is released.

8.8 If no ignition occurs up to the maximum safe operating temperature of the reaction vessel, which is 425°C (800°F), stop the heating and release the pressure as above.

9. Report

9.1 The report shall include the following:

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 425°C (800°F), report the ignition temperature as greater than 425°C (800°F).

10. Precision and Accuracy

10.1 *Precision*—The method is repeatable to ±10°C (±18°F). Cooperative data are not available to permit a complete statement of precision of the method at this time. Such data will be obtained.

10.2 *Accuracy*—The accuracy of this method cannot be reported at this time since there are no absolute standards for autogenous ignition at high pressure.

11. Keywords

11.1 autogenous ignition temperature; ignition temperature; oxygen enriched environment