Designation: G74 – 13

Standard Test Method for Ignition Sensitivity of Nonmetallic Materials and Components by Gaseous Fluid Impact[1]

This standard is issued under the fixed designation G74; 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 describes a method to determine the relative sensitivity of nonmetallic materials (including plastics, elastomers, coatings, etc.) and components (including valves, regulators flexible hoses, etc.) to dynamic pressure impacts by gases such as oxygen, air, or blends of gases containing oxygen.

1.2 This test method describes the test apparatus and test procedures employed in the evaluation of materials and components for use in gases under dynamic pressure operating conditions up to gauge pressures of 69 MPa and at elevated temperatures.

1.3 This test method is primarily a test method for ranking of materials and qualifying components for use in gaseous oxygen. The material test method is not necessarily valid for determination of the sensitivity of the materials in an "as-used" configuration since the material sensitivity can be altered because of changes in material configuration, usage, and service conditions/interactions. However, the component testing method outlined herein can be valid for determination of the sensitivity of components under service conditions. The current provisions of this method were based on the testing of components having an inlet diameter (ID bore) less than or equal to 14 mm (see Note 1).

1.4 A 5 mm Gaseous Fluid Impact Sensitivity (GFIS) test system and a 14 mm GFIS test system are described in this standard. The 5 mm GFIS system is utilized for materials and components that are directly attached to a high-pressure source and have minimal volume between the material/component and the pressure source. The 14 mm GFIS system is utilized for materials and components that are attached to a high pressure source through a manifold or other higher volume or larger sized connection. Other sizes than these may be utilized but no attempt has been made to characterize the thermal profiles of other volumes and geometries (see Note 1).

NOTE 1—The energy delivered by this test method is dependent on the gas volume being rapidly compressed at the inlet to the test specimen or test article. Therefore the geometry of the upstream volume (diameter and length) is crucial to the test and crucial to the application of the results to actual service conditions. It is therefore recommended that caution be exercised in applying the results of this testing to rapid pressurization of volumes larger than those standardized by this test method. This energy delivered by this standard is based on the rapid compression of the volume in either a 5 mm ID by 1000 mm long impact tube or a 14 mm ID by 750 mm long impact tube. These two upstream volumes are specified in this standard based on historic application within the industry.

1.5 This test method can be utilized to provide batch-to-batch comparison screening of materials when the data is analyzed according to the methods described herein. Acceptability of any material by this test method may be based on its 50 % reaction pressure or its probability of ignition based on a logistic regression analysis of the data (described herein).

1.6 Many ASTM, CGA, and ISO test standards require ignition testing of materials and components by gaseous fluid impact, also referred to as adiabatic compression testing. This test method provides the test system requirements consistent with the requirements of these other various standards. The pass/fail acceptance criteria may be provided within other standards and users should refer to those standards. Pass/fail guidance is provided in this standard such as that noted in section 4.6. This test method is designed to ensure that consistent gaseous fluid impact tests are conducted in different laboratories.

1.7 The criteria used for the acceptance, retest, and rejection, or any combination thereof of materials and components for any given application shall be determined by the user and are not fixed by this method. However, it is recommended that at a minimum the 95 % confidence interval be established for all test results since ignition by this method is inherently probabilistic and should be treated by appropriate statistical methods.

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

[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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1.9 *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.* For specific precautions see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing

D2463 Test Method for Drop Impact Resistance of Blow-Molded Thermoplastic Containers

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products

D4894 Specification for Polytetrafluoroethylene (PTFE) Granular Molding and Ram Extrusion Materials

G14 Test Method for Impact Resistance of Pipeline Coatings (Falling Weight Test)

G63 Guide for Evaluating Nonmetallic Materials for Oxygen Service

G88 Guide for Designing Systems for Oxygen Service

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

G94 Guide for Evaluating Metals for Oxygen Service

G128 Guide for Control of Hazards and Risks in Oxygen Enriched Systems

G175 Test Method for Evaluating the Ignition Sensitivity and Fault Tolerance of Oxygen Pressure Regulators Used for Medical and Emergency Applications

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

2.2 *Military Standards:*[3]

MIL-STD-1330D Standard Practice for precision Cleaning and Testing of Shipboard Oxygen, Helium, Helium-Oxygen, Nitrogen, and Hydrogen Systems

MIL-STD-1622 Cleaning Shipboard Compressed Air Systems

MIL-D-16791G Detergents, General Purpose (Liquid, Nonionic) (26 Jan 1990)

MIL-O-27210E Amendment 1—Oxygen, Aviator's Breathing, Liquid and Gas

2.3 *CGA Standards:*[4]

CGA V-9 Compressed Gas Association Standard for Compressed Gas Cylinder Valves

2.4 *ISO Standards:*[5]

ISO 291 Plastics—Standard Atmospheres for Conditioning and Testing

ISO 10297 Transportable gas cylinders—Cylinder valves—Specification and type testing

ISO 10524-1 Pressure regulators for use with medical gases—Part 1: Pressure regulators and pressure regulators with flow-metering devices

ISO 10524-2 Pressure regulators for use with medical gases—Part 2: Manifold and line pressure regulators

ISO 10524-3 Pressure regulators for use with medical gases—Part 3: Pressure regulators integrated with cylinder valves

ISO 14113 Gas welding equipment—Rubber and plastics hose and hose assemblies for use with industrial gases up to 450 bar (45 MPa)

ISO 15001 Anesthetic and Respiratory Equipment—Compatibility with Oxygen

ISO 23529 Rubber—General procedures for preparing and conditioning test pieces for physical test methods reference

2.5 *IEST Standards:*[6]

IEST-STD-CC1246D "Product Cleanliness Levels and Contamination Control Program," Clean Rooms, August 2005

3. Summary of Method

3.1 The gaseous impact test system exposes material specimens or components/elements to high-velocity (dynamic) gaseous impact environments. The basic configuration consists of a high-pressure accumulator, a high-speed pressurization (impact) valve, test system pressurization lines, test reaction chamber/fixture (for materials tests), test chamber purge and vent systems, and a valve sequencer/control device for automatic control. Fig. 1 depicts a schematic of a typical 5 mm and 14 mm GFIS test system. Fig. 2a and b depict schematics of the typical reaction chambers used for material screening for this testing. Once a material test sample is installed in the reaction chamber, the assembly is attached to the test article interface. Components to be qualified are attached directly to the test article interface.

3.2 The general test procedure is to prepare the test material or component, record significant pretest data, pressurize the system accumulators to the test pressure, calibrate the pressure rise time, and place the test material in the reaction chamber or install the component on the system interface. The test material or component is then subjected to sequential gaseous impacts by alternately opening and closing the test chamber pressurization (impact) and vent valves. The test data obtained shall include test chamber pressures and temperatures, test chamber pressure rise times, pressurization and vent valve actuation times, test gas temperature and pressure, and cycle-to-cycle sequence times. The test material or component is then

[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 Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://dodssp.daps.dla.mil.

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

[5] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

[6] Available from Institute of Environmental Sciences and Technology (IEST), Arlington Place One, 2340 S. Arlington Heights Rd., Suite 100, Arlington Heights, IL 60005-4516, http://www.iest.org.

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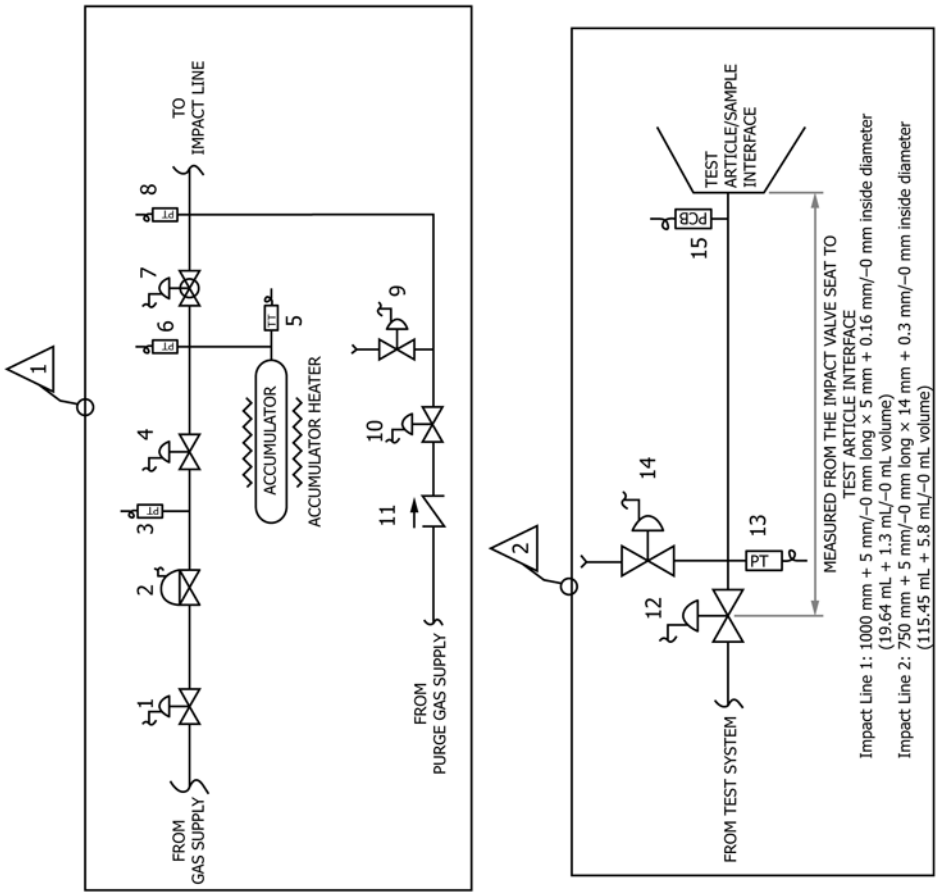

FIG. 1 Gaseous Fluid Impact Test System

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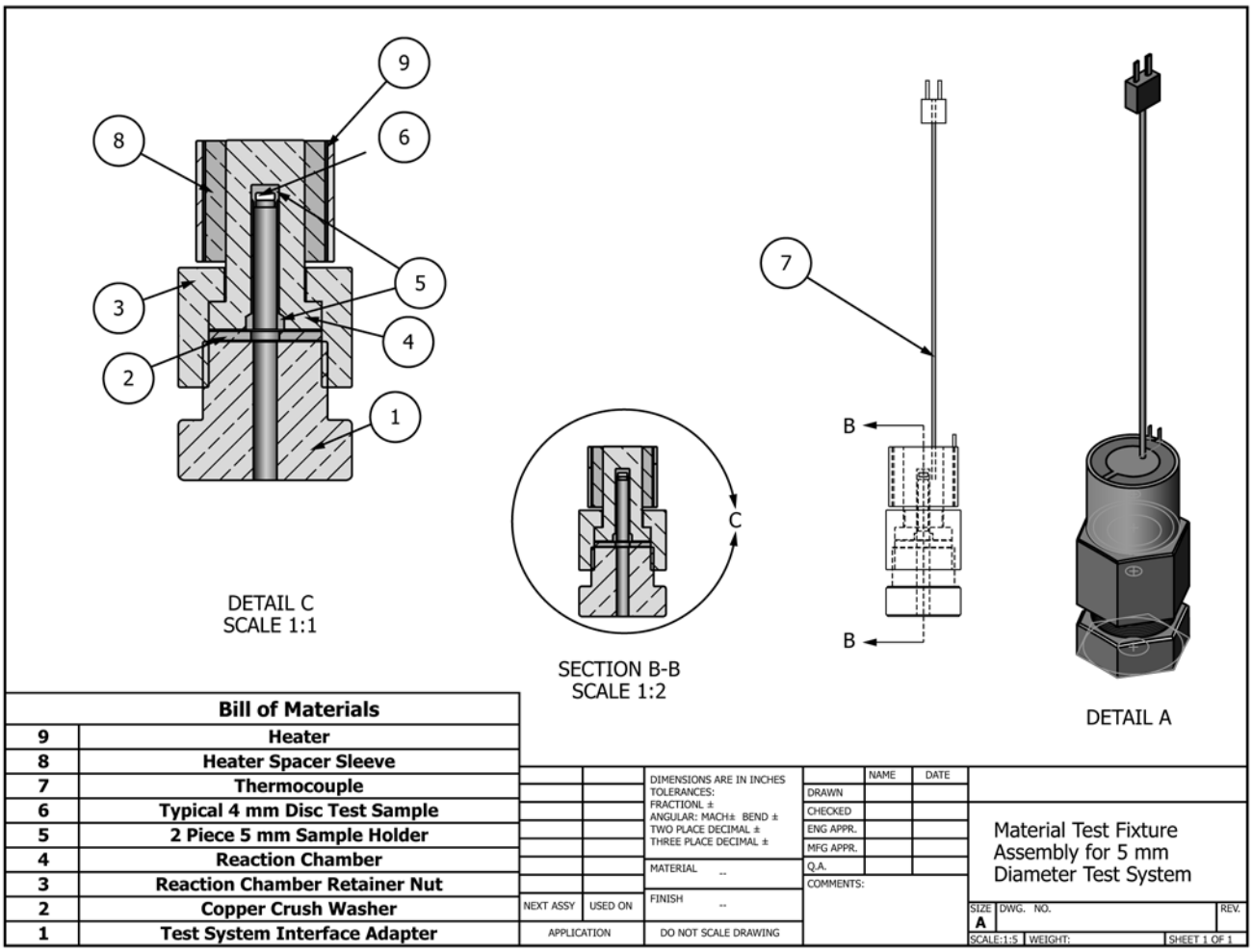

FIG. 2 a Material Test Sample Reaction Chamber Assembly for 5 mm Impact Tube.

removed and examined for any significant changes or evidence of reactions. Pertinent documentation is recorded.

4. Significance and Use

4.1 This test standard describes how to evaluate the relative sensitivity of materials and components to dynamic pressure impacts by various gaseous fluid media (can include gas mixtures).

4.2 Changes or variations in test specimen configurations, thickness, preparation, and cleanliness can cause a significant change in their impact ignition sensitivity/reaction. For material tests, the test specimen configuration shall be specified on the test report.

4.3 Changes or variation in the test system configuration from that specified herein may cause a significant change in the severity produced by a dynamic pressure surge of the gaseous media.

4.4 A reaction is indicated by an abrupt increase in test specimen temperature, by obvious changes in odor, color, or material appearance, or a combination thereof, as observed during post-test examinations. Odor alone is not considered positive evidence that a reaction has occurred. When an increase in test specimen temperature is observed, a test specimen reaction must be confirmed by visual inspection. To aid with visual inspection, magnification less than 10× can be used.

4.5 When testing components, the test article must be disassembled and the nonmetallic materials examined for evidence of ignition after completion of the specified pressure surge cycles.

4.6 Ignition or precursors to ignition for any test sample shall be considered a failure and are indicated by burning, material loss, scorching, or melting of a test material detected through direct visual means. Ignition is often indicated by consumption of the non-metallic material under test, whether as an individual material or within a component. Partial ignition can also occur, as shown in Fig. 3a, b, and c, and shall also be considered an ignition (failure) for the purpose of this test standard.

NOTE 2—A representative (exemplar) material or component may be requested by the test laboratory personnel for visual comparison with the post-test condition of the test samples.

4.7 For material testing, the prescribed procedure is conducted on multiple samples until a statistically significant

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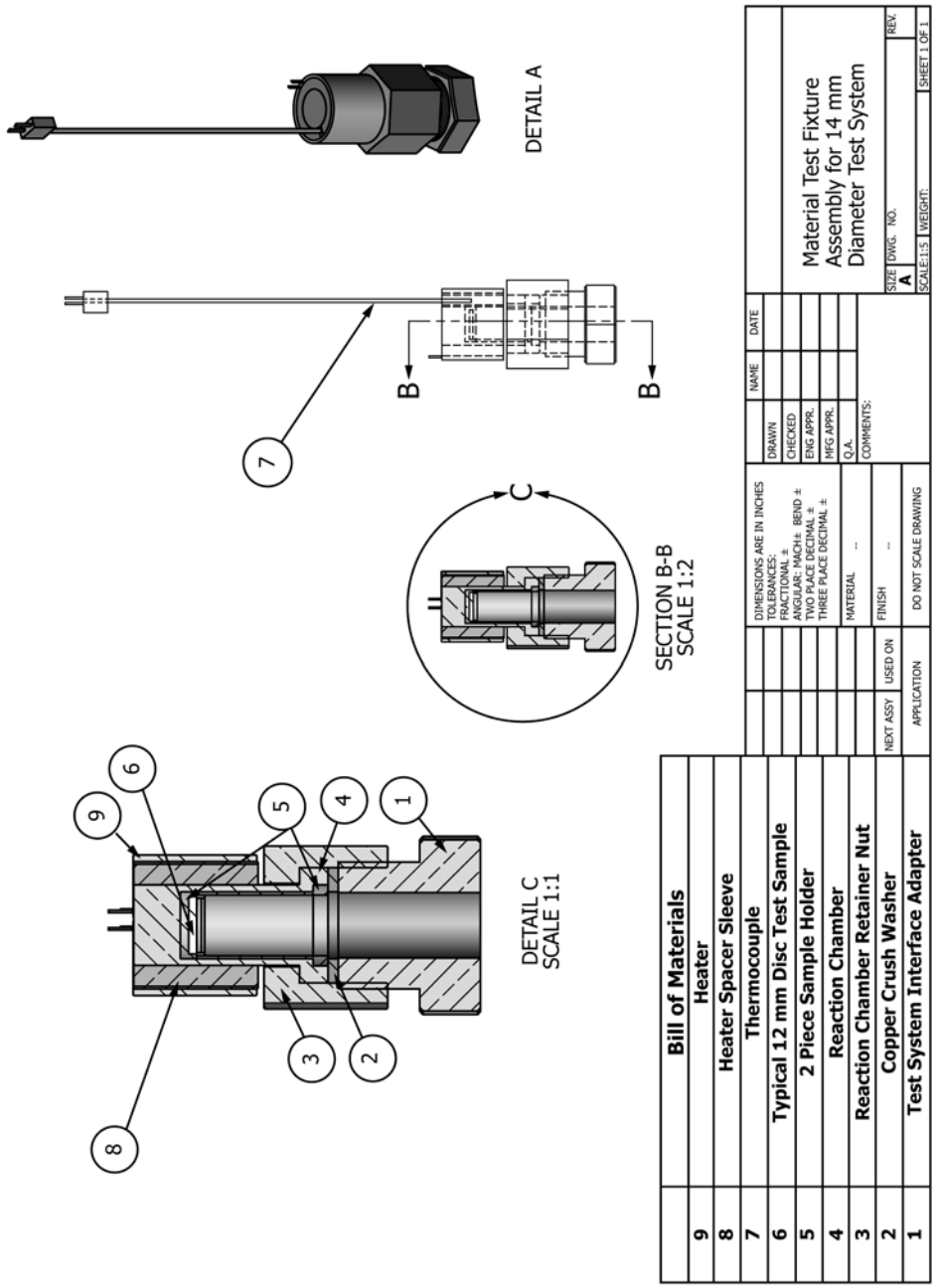

NOTE 1—Detailed drawings for Fig. 2 can be found in Appendix X1.

FIG. 2 b Material Test Sample Reaction Chamber Assembly for 14 mm Impact Tube. *(continued)*

number of ignitions or no-ignitions, or both, are achieved at various test pressures. The data is then analyzed by a procedure that calculates the median failure pressure (i.e., the 50 % reaction pressure) or the functional form of the ignition probability versus pressure by logistic regression analysis. Materials tested in a similar configuration can be ranked against each other by either of these two criteria. The initial test gas temperature may be varied as required depending on the requirements of the test.

4.8 For component testing, a specified number of pressure surge cycles are conducted at a defined test pressure, usually specified by a particular industry test standard. Usually, this pressure is 1.2 times the maximum allowable working pressure of the component. The initial test gas temperature may be varied depending on the requirements of the test; however, most commonly the initial test gas temperature is 60 ± 3 °C.

5. Apparatus

5.1 A typical gaseous impact test system used for determining the sensitivity of materials to gaseous fluid impact is schematically depicted in Fig. 1. The major test system components are described as follows:

5.1.1 The accumulator provides gaseous storage and is precharged to the desired test pressure (potential energy source).

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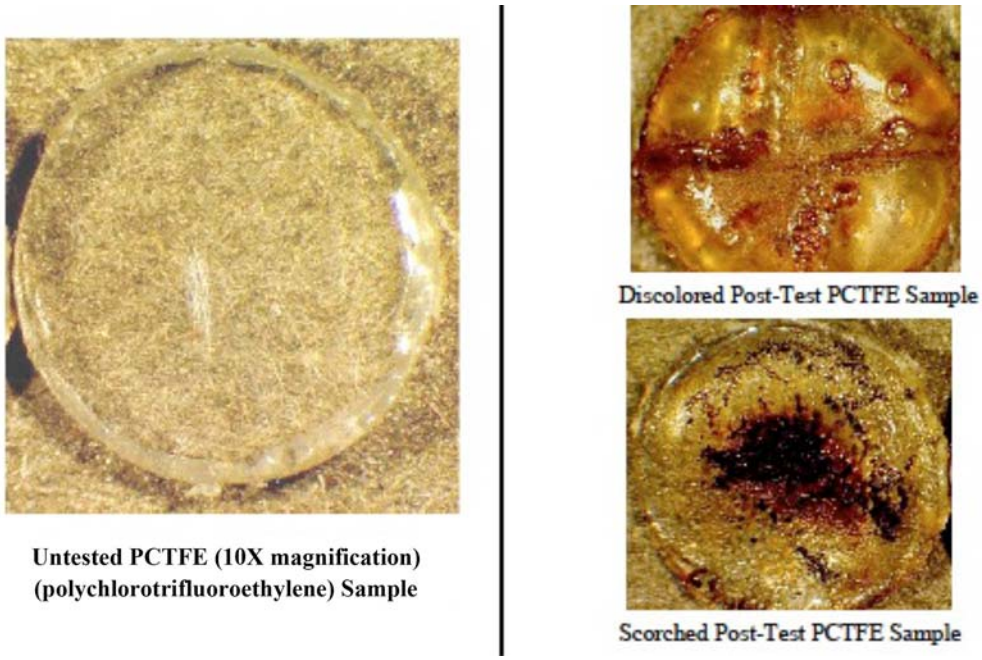
Untested PCTFE (10X magnification) (polychlorotrifluoroethylene) Sample
Discolored Post-Test PCTFE Sample
Scorched Post-Test PCTFE Sample

FIG. 3 a Untested PCTFE (10X Magnification) (Polychlorotrifluoroethylene) Sample.

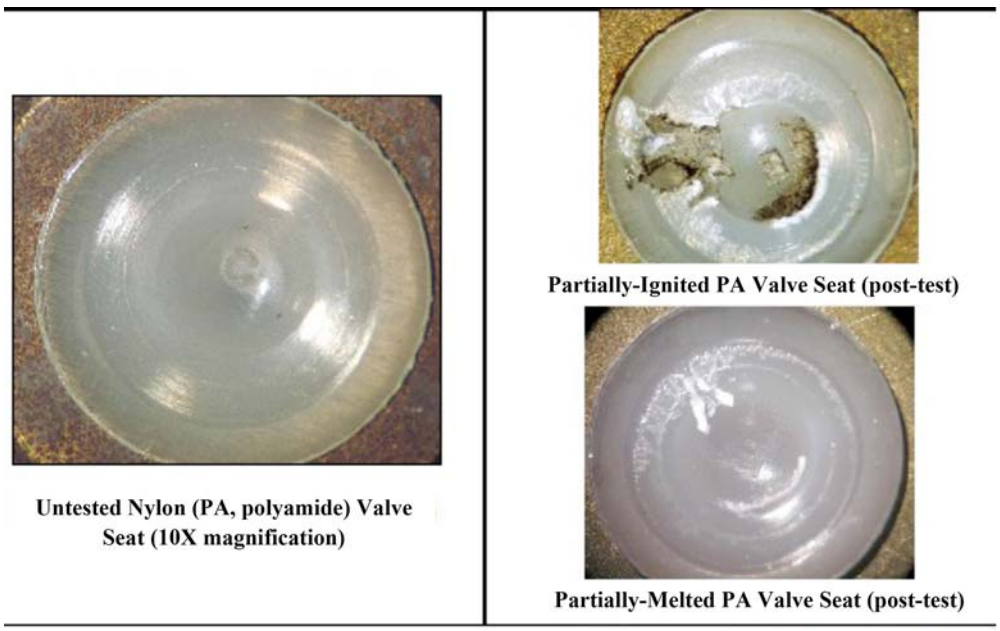
Untested Nylon (PA, polyamide) Valve Seat (10X magnification)
Partially-Ignited PA Valve Seat (post-test)
Partially-Melted PA Valve Seat (post-test)

FIG. 3 b Untested Nylon (PA, polyamide) Valve Seat (10X magnification) *(continued)*

The capacity requirement is dependent on the test chamber volume and line size and the number of impacts required per test sequence. It is sized to limit static head loss to less than 3% of initial pressure during any single cycle or series of test cycles. The accumulator is also heated such that the test gas is initially at the required gas temperature, usually 60 ± 3 °C, measured inside the accumulator. The accumulator may be recharged between test cycles by an appropriate compressor as long as the gas temperature does not exceed the required starting gas temperature. The test pressure is established for either a material or component test by the initial pressure within the accumulator.

5.1.2 The pressurization rate shall be established based on the time difference between 10 % and 90 % of the first pressure peak on the rising pressure profile, as indicated in Fig. 4. The 10 % to 90 % pressurization rate shall be within 15 to 20 ms (see 9.2.7 and Note 6).

5.1.3 The high-speed pressurization (impact) valve shall be of a suitable design to achieve the pressurization rate specified in 5.1.2 and satisfy the test severity requirements specified in the precision and bias section of this standard. Experience indicates that more repeatable results are achieved when the immediate outlet of the high-speed valve is equipped with an orifice to control the pressurization rate for tests on the 3 mm

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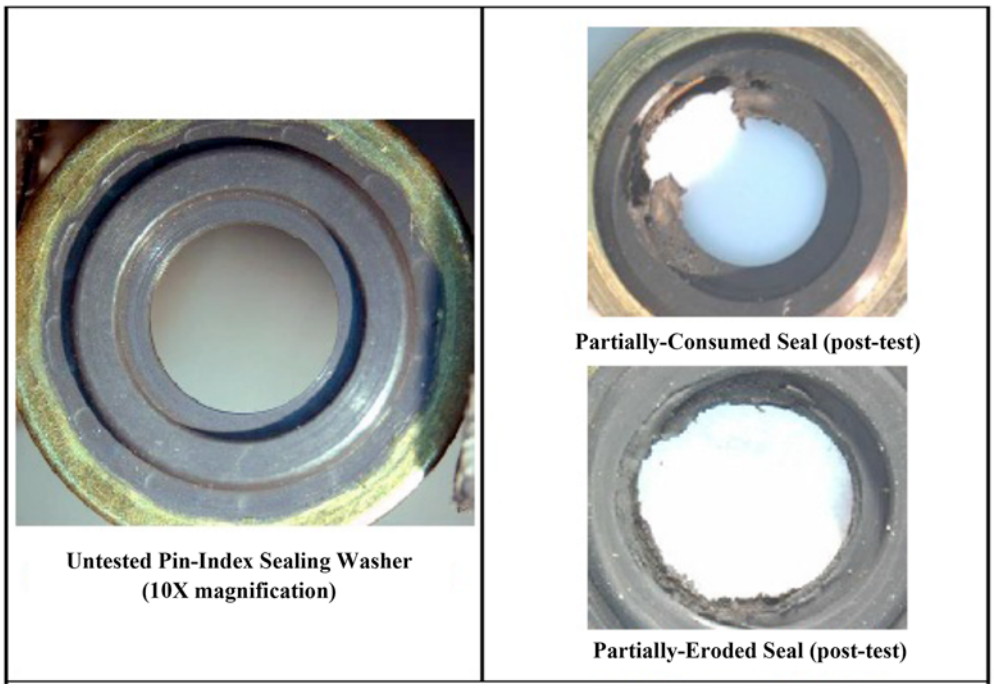

NOTE 1—For the purpose of this standard, test samples that visually appear in these conditions, or similar, are considered to be representative of ignition.

FIG. 3 c Untested Pin-Index Sealing Washer (10X magnification) *(continued)*

FIG. 3 Photographs Representing Partial Reactions Including Scorching, Discoloration, Melting and Material Loss or Material Consumption. For the purpose of this standard, test samples that visually appear in these conditions, or similar, are considered to be representative of ignition.

test system (Fig. 1). The 14 mm test system is not usually equipped with an orifice.

NOTE 3—Typical orifices are designed with a sharp-edge profile and vary in inside diameter based on the flow dynamics of the upstream impact valve. However, typical sizes for the 5 mm ID impact tube usually range between 2 mm and 4 mm, depending on the valve used.

5.1.4 The inside diameter and the length of the pressurization line to the test chamber are critical to this test method. The lines and fittings between the outlet of the high-speed (impact) valve and the test material or test article interface fitting shall maintain a constant diameter and length according to Fig. 1 and the tolerances specified. The fittings that accommodate the vent valve and pressure transducers shall not restrict flow and shall not affect the pressure rise in the impact tube.

5.1.5 The connecting tube shall comply with the geometric requirements of 5.1.4 and be fabricated of a copper-nickel alloy material, such as Monel 400™, Monel K-500™, or equivalent, to ensure that the heat transfer characteristics are the same in the connecting tube from one laboratory to another. Heat transfer losses will change if different material types are used for this connecting tube.

5.1.6 The fluid lines between the accumulator and high-speed (impact) valve shall be sized to minimize flow losses and enable pressurization of test materials or components in accordance with 5.1.2. These fluid lines shall also be sized to preclude a pressure drop upstream of the high-speed (impact) valve during the pressure surge. It is recommended that this line contain an isolation valve to provide a safety factor for system operation. The isolation valve and interconnecting lines shall have a flow factor at least equal to the pressurization (impact) valve. The isolation valve shall be located upstream of the pressurization (impact) valve or shall not restrict the flow to the test material or test article.

5.1.7 The test system vent valve shall be sized to allow the test chamber pressure to decay to atmospheric pressure between impacts so that the required 3 s minimum time at ambient pressure is achieved between successive pressure surge cycles, as shown in Fig. 4. This ambient pressure hold time between cycles is intended to allow the test material or component to cool between successive pressure surge cycles.

NOTE 4—A given gaseous impact test system screens materials and components based on at least four basic parameters: the test article pressurization rate, the accumulator pressure, the fluid dynamics in the system, and the heat transfer in the connecting tube. Variations in the test article pressurization rate of different test systems at a given test (accumulator) pressure are believed to have the greatest influence and therefore must comply with 5.1.2 and 5.1.3. The surface area to volume ratio of the impact tube and its heat transfer characteristics are believed to have the next greatest influence due to heat transfer effects and must therefore comply with 5.1.4 – 5.1.7. The driving pressure and temperature of the gas in the accumulator are believed to have the next greatest influence and therefore must be maintained according to 5.1.1. If these requirements are maintained, the data produced by the test system should enable the screening and ranking of materials and components consistently with other test systems. For example, a properly functioning test system should rank most batches of chloroprene rubbers below most batches of vinylidene fluoride hexafluoropropylene elastomers, which should rank below most batches of polytetrafluoroethylene polymers. This ranking cannot, however, be considered absolute due to material batch differences imposed by contamination, differences in types and amounts of mold release agents, differences in cures, new formulations, etc.

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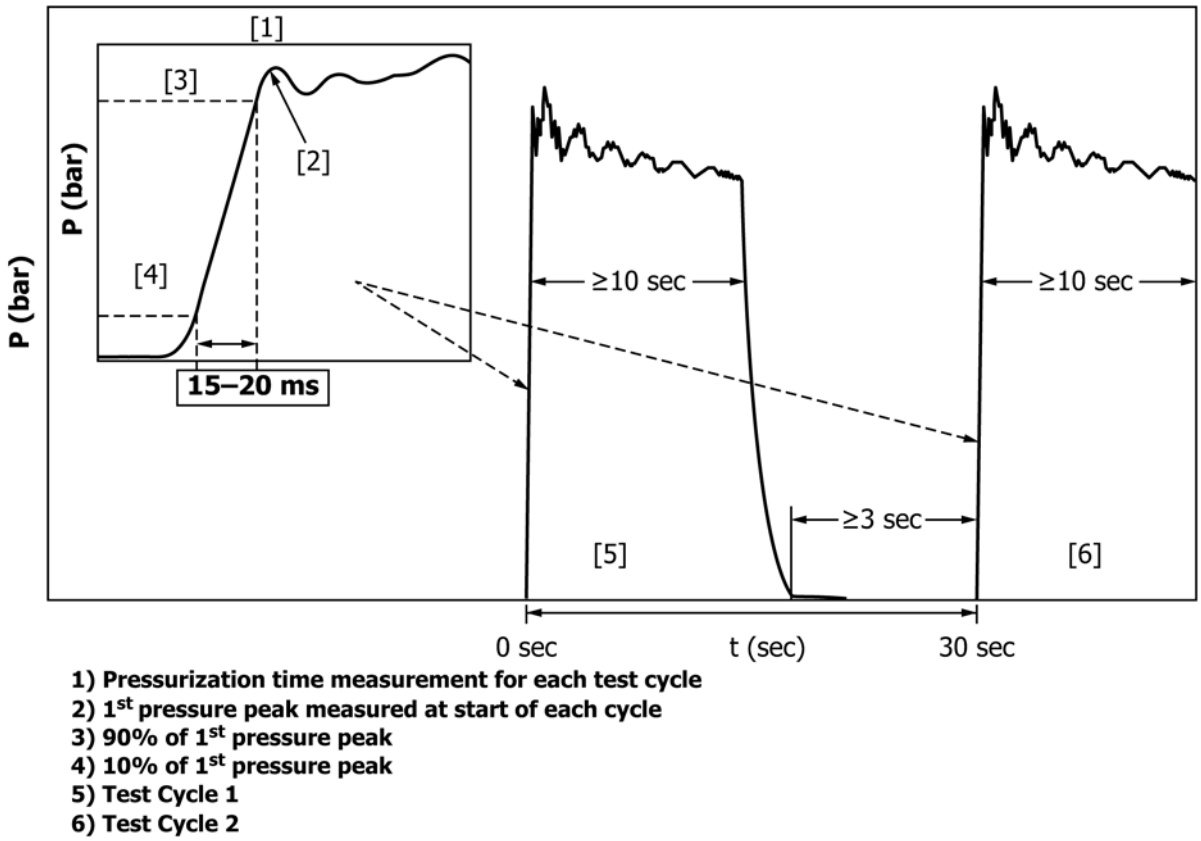

NOTE 1—Two pressurization cycles showing the 10 % to 90 % rise time profile for each cycle illustrated in the detail to the left.

NOTE 2—The baseline pressure is sea level ambient pressure (1 atm). For a given final pressure, initial baseline pressures below 1 atm (sub-ambient, other than vacuum) will increase the final temperature of the compressed gas since this increases the pressure ratio. Further, initial baseline pressures above 1 atm will decrease the final temperature since this decreases the pressure ratio. Therefore, in this standard, the "Test Pressure" is defined herein as the starting pressure in the test system accumulators and 1 atm is required as the initial test article pressure. Users are also cautioned that dynamic overshoot may cause a momentary pressure higher than the design/test pressure desired. Efforts to minimize dynamic overshoot, such as the incorporation of a suitably sized orifice, shall be exercised.

FIG. 4 Example Pressure Surge Cycles and Pressure Rise Rate Illustration

5.1.8 For material testing, the reaction chamber subassembly is configured to hold and position the test sample. Details of typical reaction chambers for the 5 mm and 14 mm systems are shown in Fig. 2 a and b. The reaction chamber contains a thermocouple to monitor the test sample temperature and to detect an ignition. The reaction chamber is configured with a sample cup to facilitate installation of a test sample and a heating collar to allow for material screening at elevated temperatures (if desired). Other requisites include the ability to readily install and remove the test specimen.

5.2 The test specimen instrumentation and control requirements include the following equipment:

5.2.1 An automatic, remote valve sequencer which controls the opening and closing of the test chamber pressurization (impact) and vent valves during the test so that each impact/vent cycle will be completed in identical, prescribed time periods. It is preset to perform a prescribed number of impact/vent cycles typically at 30 s intervals as shown in Fig. 4.

5.2.2 Test material or component instrumentation and data requirements include test fluid and test article temperatures, system static pressure, system chamber pressure, pressurization (impact) rate or pressure rise time and valve actuation/timing. All instrumentation and controls should have appropriate response times.

6. Reagents and Materials

6.1 *Alkaline Cleaner,* as required for test chambers, plumbing, and specimen substrates, such as sodium hydroxide (NaOH) or trisodium phosphate (Na_3PO_4) diluted with an appropriate amount of distilled or deionized water.

6.2 *Deionized or Distilled Water,* for test material or system component-part rinsing.

6.3 *Detergent*—A noncorrosive, oxygen-compatible cleaner in the concentration used, conforming to MIL-D-16791G.

6.4 *Gaseous Oxygen,* conforming to MIL-O-27210E, Amendment 1, Federal Specification BB-O-925, Type 1, or oxygen of 99.5 % purity or better. Oxygen of higher purity may be used if desired. The oxygen purity used in this test shall be controlled to an accuracy level at least as high as the oxygen concentration of the intended gas service. The oxygen purity shall be recorded on the test data sheet.

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6.5 *Gases* used to dilute oxygen for testing in atmospheres other than pure oxygen shall have a purity at least equal to that specified for the material service condition or oxygen component under test. Some research indicates that ultra-high purity oxygen may influence the reactivity of some materials.

7. Safety Precautions

7.1 This is a hazardous test. The test area shall be capable of withstanding short-term energetic fires, pressure releases, and shrapnel ejections from the effects of test system or test article reactions with high-pressure oxygen or oxidizing gas mixtures.

7.2 It is recommended that an appropriate pressure isolation valve be installed in the line between the accumulator and the pressurization (impact) valve. This valve may be either manually or remotely operated, but if present, must provide for personnel protection during test article loading and unloading operations.

7.3 **Caution**—Approved eye protection shall be worn in the test area at all times. Other protective equipment such as gloves and ear protection shall be required if the system vent is adjacent to the test system.

7.4 No personnel shall be permitted in the test cell when remotely controlled valves are operated or when testing is in progress.

7.5 The housekeeping and maintenance characteristics of the test area shall be considered for both safety and cleanliness aspects.

7.6 See "Safe Use of Oxygen and Oxygen Systems: Handbook for Design, Operation, and Maintenance" (MNL 36), "Guide for Control of Hazards and Risks in Oxygen Enriched Systems" (Guide G128), "Guide for Designing Systems for Oxygen Service" (Guide G88), "Guide for Evaluating Non-Metallic Materials for Oxygen Service (Guide G63), and "Guide for Evaluating Metals for Oxygen Service" (Guide G94) for details of safe practices related to the use of oxygen.

7.7 It must be understood that this standard does not provide for all safety requirements that may be deemed mandatory by local, regional, and national regulations. Users of this standard shall comply with all such regulations.

8. Test Specimens

8.1 *Component Tests*—Components (i.e., valves, regulators, etc.) shall be tested in their "as-received" condition or as specified by the test requirements. The initial cleanliness of the component is crucial to the outcome of the test. Therefore, handling of the component after its arrival at the test laboratory shall maintain the "as-received" cleanliness without potential contamination before test. If required, an interface fitting of minimum volume, cleaned for oxygen service, may be installed between the system's test article interface and the component to allow for the tests to be conducted.

8.2 *Nonmetallic Material Tests:*

8.2.1 Nonmetallic material tests may be conducted on either the 5-mm system or 14-mm system depending on the requirements of the user or the applicable industry standard. The material test report, however, shall specify which system was used for the material tests.

8.2.2 Since nonmetallic materials can vary significantly in their geometry, use configuration, and viscosity, no specific material preparation requirement is specified herein; however, materials shall be tested in their end-use condition. Physical properties such as ignition of nonmetallic materials are influenced by temperature and relative humidity in a manner that materially affects test results. In order to make reliable comparisons between different materials and between different laboratories, it is necessary to standardize the humidity conditions, as well as the temperature, to which specimens of these materials are subjected prior to and during testing.

8.2.2.1 *Plastic Materials*—Preparation and conditioning of plastic test specimens shall be accomplished according to Practice D618, ISO 291 or equivalent.

8.2.2.2 *Rubber Materials*—Preparation and conditioning of rubber test specimen from sheet and products shall be accomplished according to Practice D3182, Practice D3183, ISO 23529 or equivalent.

8.2.3 The samples may be prepared in disc geometries or into divided segments (multiple pieces). The material test sample configuration, mass and preparation procedure shall be recorded in the test report. Several preparation options are provided below.

8.2.4 The following material preparation steps are provided as options for preparation of test material specimens for test, as desired by the user. The material test sample configuration and mass shall be recorded in the test report.

8.2.4.1 Test the nonmetallic material samples in a thickness of 1.5 ± 0.13 mm (standard thickness), or in the end-use thickness if less than 1.40 mm. If specimens are tested in a thickness other than 1.5 ± 0.13 mm, the deviation shall be recorded in the test report. The samples should be representative of the as-used condition where possible. The as-used condition may be either the installed condition, or where preferable, the condition that exists at any time in the service life.

8.2.4.2 The nonmetallic test materials shall be prepared to a diameter that fits loosely in the sample cup and shall geometrically be disk-shaped. The standard diameter range for the 5-mm system shall be 3.75 to 4.0 mm to fit loosely. The standard diameter range for the 14-mm system shall be 11.5 to 12.0 mm to fit loosely. Once an appropriate punch or equivalent preparation method is selected, all samples should be prepared to the same nominal diameter (± 0.1 mm nominal).

8.2.4.3 An alternate preparation method is to subdivide each nonmetallic test sample into 4 to 8 nominally equal pieces by subdividing the initially prepared sample after step 8.2.4.2 in order to ensure that samples are loosely held in the sample cup.

8.2.4.4 Apply coatings and paint in end-use thickness onto a brass or 316 stainless steel (or other suitable metal) substrate. The substrate surface should be clean for oxygen use and prepared according to the coating manufacturer's recommended procedures. Prepare applied material in accordance with the manufacturer's recommendations. Record the final coating thickness (or mass), application steps, and preparation procedure for test reporting purposes.

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8.2.4.5 Prepare specimens of O-rings as subdivided samples in their as-used diameter and approximately the same surface-area-to-volume ratio as for subdivided samples prepared from sheet or rod stock.

8.2.4.6 Apply greases or semisolid materials onto a woven inert disc (such as fiberglass) or equivalent that has been prepared to a thickness consistent with 8.2.4.1. Greases and semisolid materials may also be applied as an approximately consistent surface layer covering the bottom of the lower sample cup. Record the mg/m^2 (or mass equivalent) surface coverage.

8.2.4.7 Irregular nonmetallic materials should be prepared as subdivided samples of approximate mass or surface area-to-volume ratio as for samples prepared from sheet or rod stock.

8.2.5 Maintain specimen cleanliness at all times. Prepare and handle the specimens with new, visibly clean, vinyl surgical gloves or equivalent. Do not touch the materials or samples with bare hands during or after the cleaning process. Do not expose gloves to reactive solvents and then handle the material test specimen.

NOTE 5—The 50% reaction pressure of single diameter disks, subdivided samples, greases, and irregular shape specimen may not be the same. Round-robin testing to evaluate configuration variables is yet to be performed. Therefore it is recommended that only similarly-prepared materials be ranked against each other.

8.3 Document the prepared test specimen configurations for inclusion in the test report.

8.4 Test specimen cleaning procedures are as follows:

8.4.1 Clean solid specimens, coatings, paints, and O-rings by soaking with agitation in a mild aqueous detergent compatible with the test material and rinsing with distilled or deionized water or a solvent that is inert with the test material and will not absorb into the material or leach additives from the material, consistent with the recommendations in Practice G93 or MNL 36. Wash with detergent, then rinse with distilled or deionized water and dry using a filtered (25-µm absolute or smaller filter rating) inert gas or air purge. If the specimen cannot be wetted with any cleaning solution, blow the specimens clean using filtered (25-µm absolute or smaller filter rating) inert gas or air.

8.4.2 Clean brass or Type 316 stainless steel substrates on which coating, paint, or grease samples are to be applied by immersing the substrates in an alkaline cleaner (see 6.1) for a minimum of 15 min at elevated temperature (refer to Practice G93). Follow immersion with a thorough rinse in running tap water, followed by a thorough rinse(s) in distilled or deionized water. Perform a water break test during the rinsing step to verify that organic material has been removed from the surface. Blow dry the substrate with a clean, dry, oil-free nitrogen to remove the excess water, place the substrate in an oven at elevated temperature (typically greater than 50° C) until free of water. Remove from the oven and store in a clean covered container until ready for use. Substrates may be cleaned using any process that will produce a cleanliness level at least as good as the level provided by the above process or achieve a level 100A as specified by Pracitce G93 or MNL 36.

8.5 It may be desired to evaluate the reactivity of materials already in use. In this case, specimens shall be prepared in the appropriate configuration (see 8.2), but cleaning in accordance with 8.4 may be omitted to permit full evaluation of the use condition.

9. Procedure

9.1 Preparation of the test system shall include the following. These precautions are required to ensure test results independent of the effects of extraneous materials.

9.1.1 Initially, clean all component parts of the test system that are exposed to the test media or test specimen, or both, to a level equivalent with the requirements of the test media. Follow Practice G93 or MNL 36 recommended procedures. Reference to MIL-STD-1330D or IEST-STD-CC1246 may also be helpful.

9.1.2 Pressurization cycles shall be conducted on an impact tube (see Fig. 1) instrumented with a fast-response pressure transducer to ensure that the pressure rise rate is consistent with 5.1.2 and Fig. 4 (see also Note 6).

9.1.3 For material tests, install a clean new sample cup and test sample for each test pressure or each replication test. The test samples, prepared according to the instructions in Section 8, should be positioned in the sample cup and subjected to gaseous fluid impact according to Section 9.2.

9.2 The gaseous impact test for materials and components shall include the following sequence and procedures:

9.2.1 Install the test sample cup and sample in the reaction chamber (Fig. 2a or b). Take care not to contaminate the chamber hardware or test specimen. Install the test fixture assembly on the test article interface adaptor.

9.2.2 Pressurize the accumulator with the desired gas or gas mixture to the required test pressure.

9.2.3 Clear the test cell area of personnel, then purge the test chamber and specimen with low-pressure test media sufficiently to ensure that ambient air is completely purged from the test chamber and associated tubing.

9.2.4 Ensure that all data acquisition equipment is operating properly.

9.2.5 Open the impact line vent valve until the chamber pressure transducer indicates atmospheric pressure (see note on Fig. 4); then close the impact line vent valve.

9.2.6 Ensure the high-speed (impact) valve is closed and open the impact isolation valve.

9.2.7 The following operations shall be controlled with an automatic valve sequencer to minimize valve timing/sequence changes. A typical gaseous impact test cycle is shown in Fig. 4.

9.2.7.1 Verify that the instrumentation systems are operational; then open the impact line vent valve. After the preset duration (which allows the chamber pressure to decay to atmospheric), close the impact line vent valve.

9.2.7.2 Open the high-speed (impact) valve to pressurize the test sample/article (see Note 6).

NOTE 6—It is critical that the rise time from 10 % to 90 % of the first peak be between 15 and 20 ms, as shown in Fig. 4. The pressurization rate requirement shall be confirmed with one of the following configurations: *(1)* Configuration 1 – This configuration verifies the rise time with the material reaction chamber or test article attached at the test article interface (preferred); or, *(2)* Configuration 2 – This configuration verifies

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the rise time with the impact line plugged at the test article interface (i.e., impact line dead-ended without a test article attached). Configuration 1 is the preferred methodology since it eliminates the influence of the test specimen on the pressurization rise time. However, some industry test standards require the use of Configuration 2 for determination of the rise time and may be used where required. The configuration utilized shall be specified in the test report along with representative rise time data to document the typical pressurization profile and pressure rise dynamics.

9.2.7.3 Close the high-speed (impact) valve after a preset duration, usually 10 s, or when a reaction is observed.

9.2.7.4 Monitor instrumentation for indication of ignition.

9.2.7.5 For MATERIAL testing:

(1) All tests on a material start at a pressure estimated by experience to approximate the pressure at which 50 % of the samples should ignite (50 % reaction pressure). If uncertain, it is recommended that testing start at approximately 10 MPa.

(2) For every sample, repeat 9.2.7.1 – 9.2.7.4 until 5 test cycles (impacts) at the same pressure have been performed in 30-s intervals or until ignition of the sample occurs as indicated by a sudden/abrupt increase of the reaction chamber temperature. Whether or not an indication of ignition was observed during the test, the sample condition shall be evaluated by post-test visual inspection of the sample.

(3) If ignition occurs, the test pressure shall be reduced by approximately 1 MPa for the next sample (other test pressure decrements may be used as required). If ignition does not occur, the test pressure shall be increased by 1 MPa for the next sample (again, other test pressure increments may be used as required). This process of decreasing or increasing the test pressure for each successive sample shall continue until a change in the ignition/no-ignition result occurs. The successive increase/decrease of pressure with no-ignition/ignition of a sample shall continue for 20 to 40 samples, or until a statistically significant 50 % reaction pressure can be calculated.

(4) If a logistic regression analysis is required, then additional samples shall be performed at pressure levels above and below the 50 % reaction pressure. Usually, it is recommended that 5 to 6 pressure levels in addition to the 50 % reaction pressure level be selected and at least 5-10 samples be completed at each pressure level. The highest pressure tested should ideally result in ignitions of all of the samples. The lowest pressure level tested should ideally result in no ignitions of the test samples. The other pressure levels typically result in mixed results (ignitions and no-ignitions). See Figure 10 as an example of following the procedure described above.

(5) Record all ignition and no-ignition data on a permanent record such as the data sheet shown in Figure 10, as required.

(6) Close the impact isolation valve.

(7) Ensure that the test chamber pressure and temperature conditions are stabilized and indicate that the test chamber may be vented. Vent the test system and allow the chamber to vent to ambient pressure. Close and isolate the system valves for safe access to the system.

(8) Remove the reaction chamber and sample cup and examine for evidence of reaction. Record all observations on a permanent record form such as the data sheet shown in Figure 10.

NOTE 7—The procedure above requires that each test specimen be subjected to five successive impacts in 30-s intervals at a given pressure. The material may be ranked on the basis of the 50 % reaction pressure (Section 9.2.7.5*(3)*) or by a logistic regression analysis (Section 9.2.7.5*(4)*). Section 9.2.7.5*(3)* represents a Bruceton Up-Down methodology (see Test Method D2463) to allow estimation of the 50 % reaction pressure with a minimum number of samples. An example of a Bruceton Up-Down procedure is shown in Fig. 5. The data analysis is discussed in Section 10. The Up-Down method consists of testing at specific pressures and then raising or lowering the pressure based on the result of the previous test. Initially, larger test pressure increments/decrements are used to narrow down the level at which the 50 % pressure will occur. However, after mixed results (ignitions/no-ignitions) are achieved on a few pressure levels and the 50% pressure is bracketed, a single consistent pressure increment/decrement should be used for the remainder of the samples. It is noteworthy that some materials will ignite with relative ease at very low pressures resulting in difficulty in performing the analysis suggested above. Other materials may not ignite with high frequency even at very high pressures, also complicating the data analysis. However, for most common materials, the procedures specified above will provide a consistent ranking.

9.2.7.6 For COMPONENT tests:

(1) Install a component on the test article interface (see Fig. 1).

(2) Repeat 9.2.7.1 – 9.2.7.4 until the required number of test cycles (impacts) have been performed on a single component or ignition occurs, according to the test configurations and number of cycles required by an industry qualification test standard such as CGA V-9, ISO 10297, ISO 10524-1, ISO 10524-2, ISO 10524-3, ISO 14113, ISO 15001, Test Method G175, or other relevant standard.

(3) Close the impact isolation valve.

(4) Ensure that the test chamber pressure and temperature conditions are stabilized and indicate that the test chamber may be vented. Vent the test system and allow the component to come to ambient pressure. Close and isolate system valves for safe access to the system.

9.2.7.7 For COMPONENT tests, after the desired number of test cycles are completed, remove the component and examine for evidence of ignition or ignition precursors (see Fig. 3a, b, and c). The component must be disassembled to visually assess the condition of the nonmetallic materials (seats and seals). Record all observations on a permanent record form such as the data sheet shown in Figure 10.

9.2.8 Repeat the procedure of Section 9 at the test pressures for the required number of test samples until the test series is completed.

10. Data Analysis

10.1 For MATERIAL tests, two methods of data analysis may be utilized. The first calculates the median ignition pressure also known as the 50 % reaction pressure. The second method calculates the ignition probability based on the entire population of results. The first method requires that at least 20 to 40 samples be tested; however, the standard deviation of the results shall be calculated to determine the scatter in the data if this method of data analysis is used. The second method requires that 40 to 100 samples be tested in order to predict the probability of ignition with high confidence in the

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MPa	bar	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25
10	100																									
10.5	105																									
11	110																									
11.5	115																							P		
12	120																									
12.5	125	P						P															F		P	
13	130																									
13.5	135		P				F		P		P		P						P		P	F				P
14	140																									
14.5	145			P		F				F		F		P				F		F						
15	150																									
15.5	155				F										P		F									
16	160																									
16.5	165															F										

MPa	bar	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50
10	100																									
10.5	105													P												
11	110																									
11.5	115												F		P		P				P					
12	120																									
12.5	125							P				F				F		P		F		P				
13	130																									
13.5	135						F		P		F								F				P			
14	140																									
14.5	145	P		P		F				F														P		
15	150																									
15.5	155		F		F																				P	
16	160																									
16.5	165																									F

FIG. 5 Typical Material Test Sequence of Results Using Bruceton Up/Down Procedure for 5-mm ID Test System

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