Designation: G86 – 98a (Reapproved 2011)

Standard Test Method for Determining Ignition Sensitivity of Materials to Mechanical Impact in Ambient Liquid Oxygen and Pressurized Liquid and Gaseous Oxygen Environments[1]

1. Scope

1.1 This test method[2] describes test equipment and techniques to determine the impact sensitivity of materials in oxygen under two different conditions: *(1)* in ambient pressure liquid oxygen (LOX) or *(2)* under pressure-controlled conditions in LOX or gaseous oxygen (GOX). It is applicable to materials for use in LOX or GOX systems at pressures from ambient to 68.9 MPa (0 to 10 000 psig). The test method described herein addresses testing with pure oxygen environments; however, other oxygen-enriched fluids may be substituted throughout this document.

1.2 This test method provides a means for ranking nonmetallic materials as defined in Guide G63 for use in liquid and gaseous oxygen systems and may not be directly applicable to the determination of the sensitivity of the materials in an end-use configuration. This test method may be used to provide batch-to batch acceptance data. This test method may provide a means for evaluating metallic materials in oxygen-enriched atmospheres also; however, Guide G94 should be consulted for preferred testing methods.

1.3 Values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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.* See also Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1193 Specification for Reagent Water

D4080 Specification for Trichloroethylene, Technical and Vapor-Degreasing Grade

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

2.2 *Military Document:*[4]

MIL-D-16791 Detergent, General Purpose (Liquid, Nonionic), Type One

2.3 *American Chemical Society:*[5]

Trichloroethylene, Reagent Grade

2.4 *Compressed Gas Association:*[6]

G-4 Oxygen

G-4.1 Cleaning Equipment for Oxygen Service

G-4.3 Oxygen, Gaseous, Type I B

G-4.3 Oxygen, Liquid, Type II B

G-10.1 Nitrogen, Gaseous, Type I B

G-10.1 Nitrogen, Liquid, Type II B

2.5 *NASA Standard:*[7]

NSS 1740.15 Safety Standard for Oxygen and Oxygen Systems

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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 G04.01 on Test Methods.

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[2] *NASA Handbook 8060.1B, Pressurized Liquid and Gaseous Oxygen Mechanical Impact Test*, Sept. 1981, pp. 4-72.

[3] 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.

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

[5] Available from American Chemical Society (ACS), 1155 Sixteenth Street, NW Washington, DC 20036, http://www.acs.org.

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

[7] Available from National Aeronautics and Space Adminstration (NASA), NASA Headquarters, Suite 1M32, Washington, DC 20546.

2.6 *ASTM Adjuncts:*
ABMA-Type Impact Tester and Anvil[8]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *GOX, n*—gaseous oxygen.

3.1.2 *LOX, n*—liquid oxygen.

3.1.3 *mechanical impact, n*—a blow delivered by a plummet that has been dropped from a preestablished height onto a striker pin in contact with a sample.

3.1.4 *reaction, n*—a chemical change or transformation in the sample initiated by a mechanical impact.

3.1.4.1 *Discussion*—A reaction from ambient pressure, LOX mechanical impact may be determined by an audible report, an electronically or visually detected flash, obvious charring of the sample, cup, or striker pin.

3.1.4.2 *Discussion*—Reactions in pressurized LOX or GOX are typically indicated by an abrupt increase in test sample temperature, chamber pressure, and light levels and may be supplemented by obvious changes in odor, color, or material appearance as a result of thermal decompositions observed during examination after the test.

3.1.5 *pressure threshold, n*—the highest pressure at a given impact energy level for which the passing criteria have been met.

3.1.6 *energy threshold, n*—the highest impact energy level at a given pressure for which the passing criteria have been met.

4. Summary of Test Method

4.1 The mechanical impact test system is designed to expose material samples to mechanical impact in the presence of liquid or gaseous oxygen at pressures from ambient to 68.9 MPa (0 to 10 000 psig). The basic drop tower configuration consists of: an electromagnet, a plummet, plummet guide tracks, plummet hold/release mechanism, base plate, anvil plate, a specimen cup holder, sample cup, and striker pin (see Fig. 1). For tests conducted under pressure-controlled conditions, the anvil plate and specimen cup holder are replaced with a test chamber equipped with a striker pin or striker pin counterloader (see Fig. 2), test chamber purge, pressurization and vent systems (see Fig. 3), and a plummet catcher (see Fig. 4). The general procedure is to prepare the test sample and record significant pretest data.

4.2 *Ambient LOX Impact Test*—The test conditions (pressure and temperature) are the ambient pressure of the test facility and the boiling point of LOX at that pressure. Each sample is placed into a specimen cup (see Fig. 5), precooled in a sample freezing box (Fig. 6), covered with LOX, and placed in the cup holder seater in the anvil assembly of the impact tester. The plummet is dropped from a selected height onto the striker pin, which transmits the energy to the test sample. Observation for any reaction is made and noted. Drop tests are continued using a fresh sample, sample cup, and striker pin for each drop until the threshold level is determined or the test series is completed.

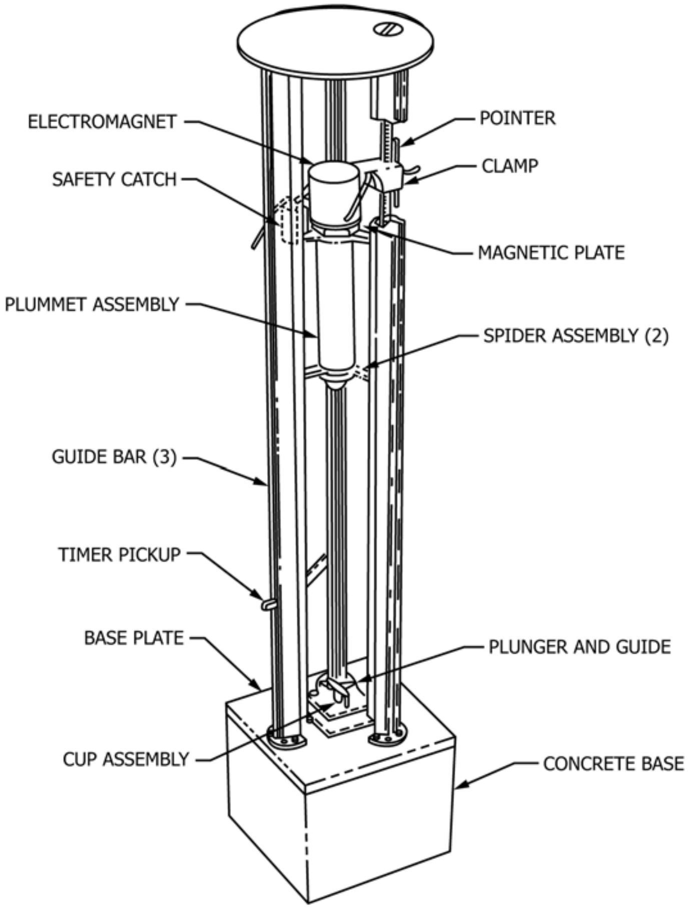

FIG. 1 Oxygen Impact Test Frame

4.3 For materials tested in pressurized LOX or GOX, each sample is placed in the test chamber. The test chamber is filled with liquid or gaseous oxygen, pressurized to the required test pressure, and the striker pin or striker pin counterloader is pressed down against the top of the test sample. The plummet is dropped from a selected height onto the striker pin or striker pin counterloader. Instrumentation devices that monitor the test chamber interior for pressure, temperature, and light emission provide evidence of test sample reaction. The sample is removed from the chamber, and the sample is inspected for other evidence of reaction such as odor or charring. Drop tests are continued using a fresh sample, sample holder, and striker pin or striker pin counterloader for each drop, until the threshold level is determined or the test series is completed. Additional modifications to the above procedure are required when testing is performed at temperatures above ambient.

4.4 This test method may be used to determine the impact sensitivity of a material, batch-to-batch acceptance, or to satisfy other prescribed pass-fail criteria.

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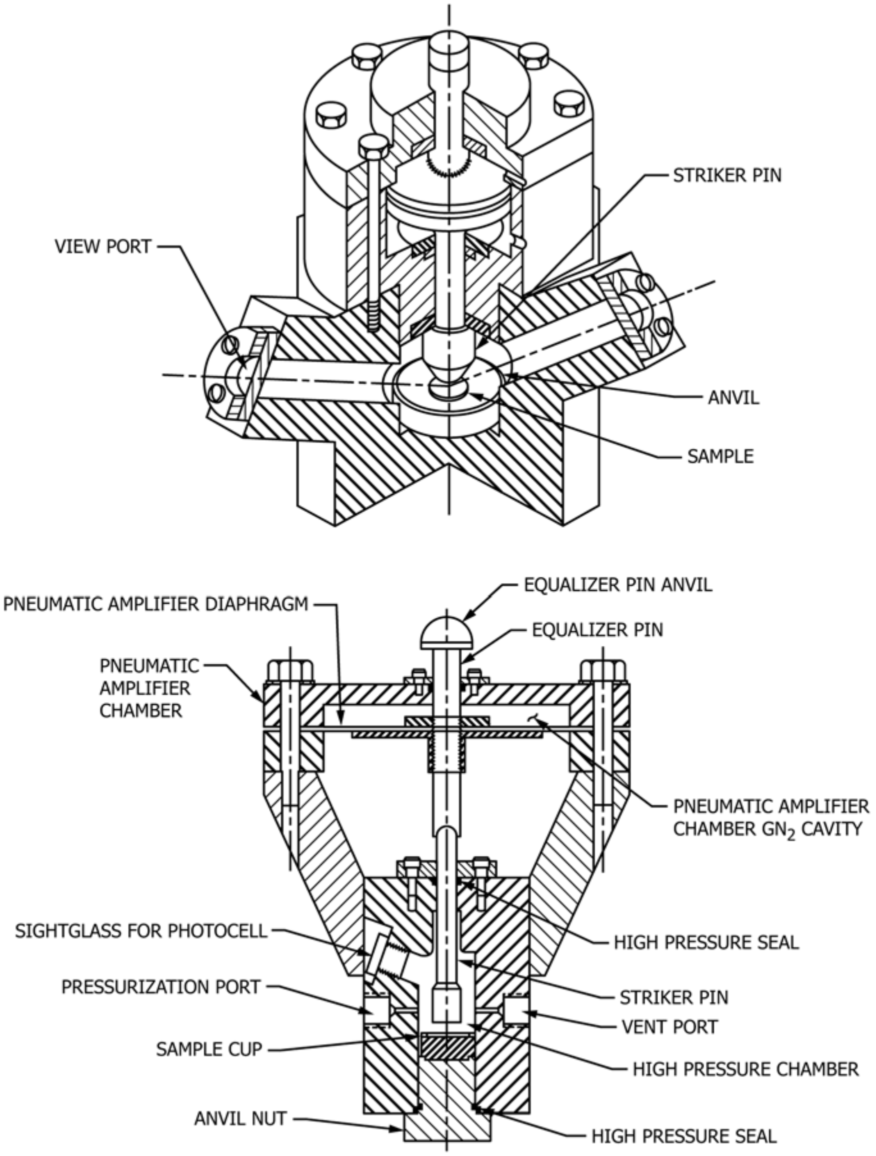
[8] Detailed drawings from the ABMA-Type Impact Tester and Anvil Region Assembly are available at a nominal fee from ASTM International, 100 Barr Harbor Dr., Philadelphia, PA 19428. Request Adjunct ADJD2512.



1	Pneumatic Amplifier Chamber	9	High-Pressure Chamber
2	Equalizer Pin Anvil	10	Sample Cup
3	Equalizer Pin	11	Anvil Nut
4	Pneumatic Amplifier Diaphragm	12	High-Pressure Seal
5	Pneumatic Amplifier Chamber GN$_2$ Cavity	13	Pressurization Port
		14	Vent Port
6 and 8	Striker Pin	15	Sightglass for Photocell
7	High-Pressure Seal		

FIG. 2 Two Types of High-Pressure Test Chambers

5. Significance and Use

5.1 This test method evaluates the relative sensitivity of materials to mechanical impact in ambient pressure liquid oxygen, pressurized liquid oxygen, and pressurized gaseous oxygen.

5.2 Any change or variation in test sample configuration, thickness, preparation, or cleanliness may cause a significant change in impact sensitivity/reaction threshold.

5.3 Suggested criteria for discontinuing the tests are: *(1)* occurrence of two reactions in a maximum of 60 samples or less tested at the maximum energy level of 98 J (72 ft•lbf) or one reaction in a maximum of 20 samples tested at any other energy level for a material that fails; *(2)* no reactions for 20 samples tested at the 98-J (72-ft•lbf) energy level; or *(3)* a maximum of one reaction in 60 samples tested at the maximum energy level.

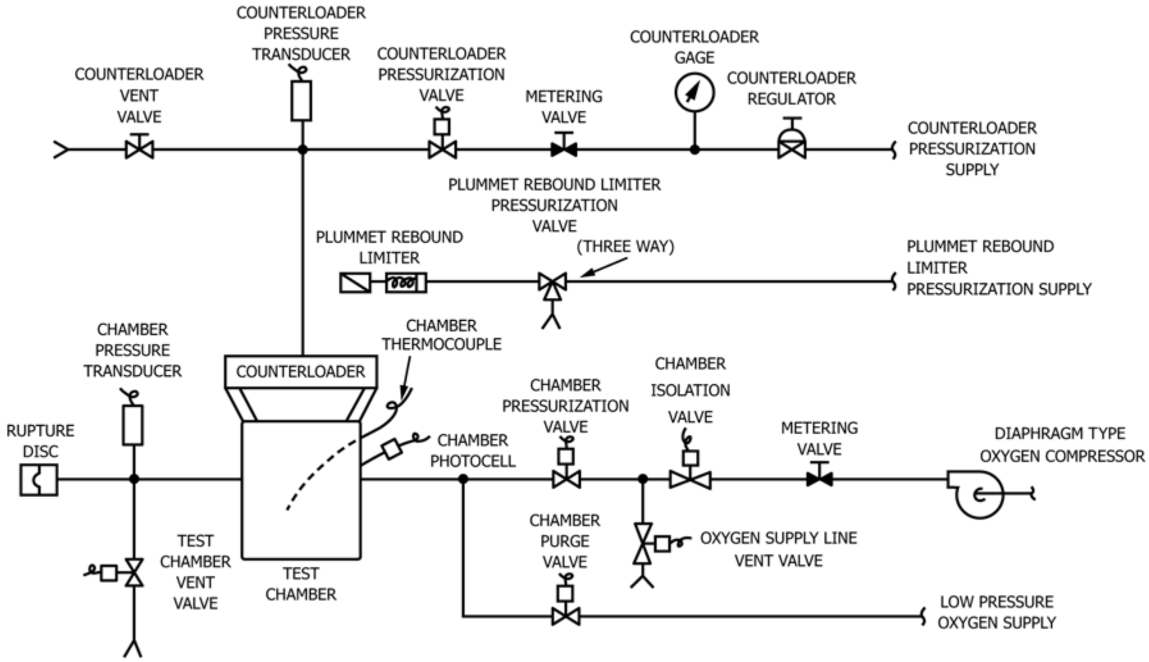


FIG. 3 Typical Pressurization Piping system for a LOX/GOX Pressurized Test System

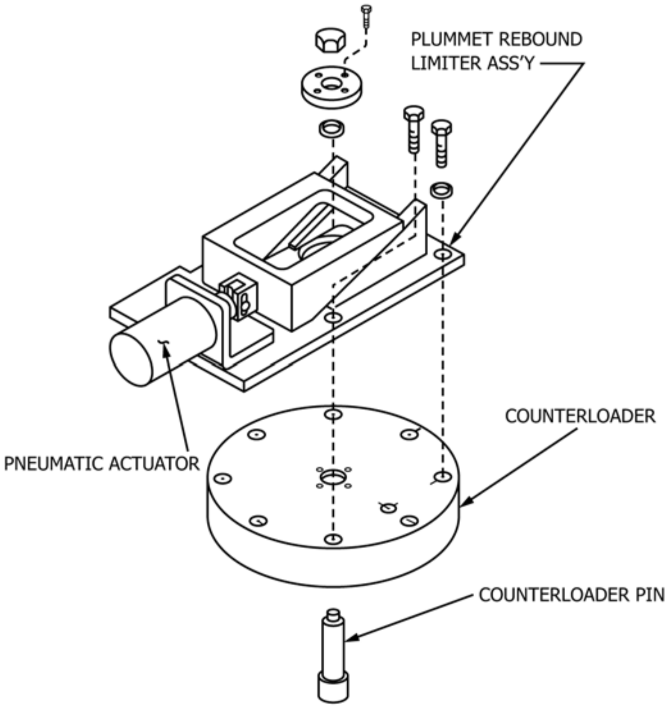
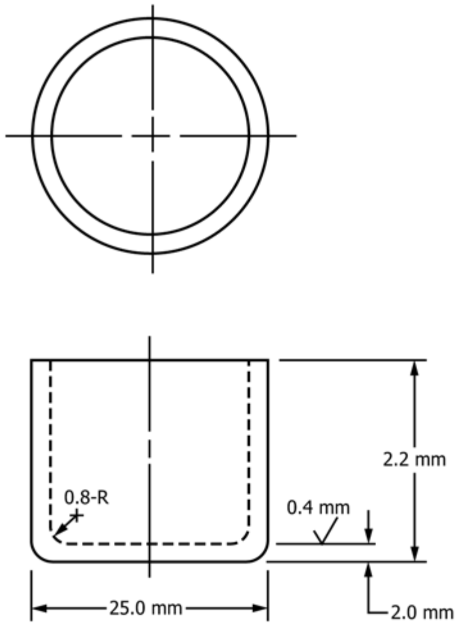


FIG. 4 Typical Plummet Rebound Limiter Assembly

6. Criteria for Acceptance for Ambient LOX and Pressurized LOX and GOX Mechanical Impact Test

6.1 To meet the requirements for acceptability, the material shall show no reaction when being subjected to 20 successive impact tests tested at 98 J (72 ft•lbf) using the equipment described in Section 10.



NOTE 1—Break sharp edges 0.4 mm.

NOTE 2—The cup is formed by deep drawing.

NOTE 3—The thickness and parallelness of the cup bottom shall be controlled to 2.0 mm by coining.

NOTE 4—Material: any 3000 or 5000 series aluminum alloy.

FIG. 5 LOX Impact Tester One-Piece Sample Cup

6.2 The test may be discontinued and the materials considered to have failed if there is one reaction in 20 drops at any energy level less than 98 J (72 ft•lbf).

6.3 A material is acceptable after 60 successive impact tests with not more than one reaction at 98 J (72 ft•lbf). The test may

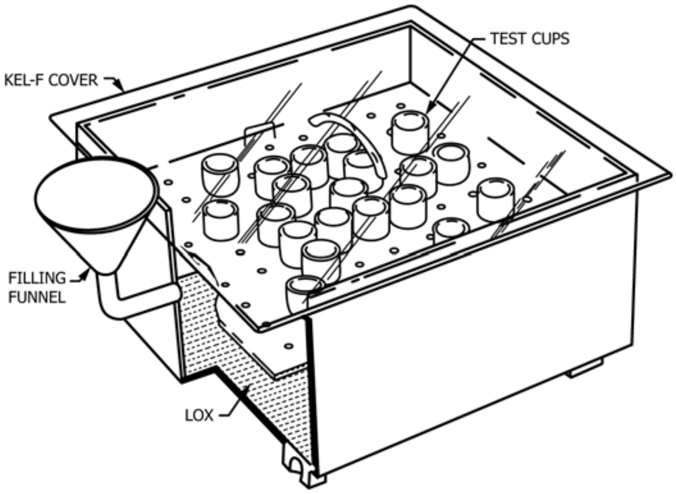


FIG. 6 Typical Sample Freezing Box

be terminated and the material considered to have failed if there are two reactions in 60 tests or less at 98 J (72 ft•lbf).

6.4 The material shall show none of the following reactions during any of the tests.

6.4.1 Audible explosion.

6.4.2 Flash (electronically or visually detected).

6.4.3 Evidence of burning (obvious charring, see Note 1).

6.4.4 Major discoloration (as a result of ignition only rather than other phenomena).

6.4.5 A temperature or pressure spike in elevated temperature tests.

NOTE 1—A burnt odor alone is not considered sufficient proof that a reaction has occurred. If a reaction occurs (including those during bounce of plummet), it shall be reported as evidence of sensitivity. Inclusion of bounce reactions applies to ambient LOX mechanical impact tests only.

6.5 All materials that fail 6.1 criteria and remain candidates for use must be subjected to LOX or GOX mechanical impact energy threshold determinations in the thickness of use.

6.6 The material to be tested must be traceable back to the original manufacturer and to a specific batch or lot numbers, or both.

6.7 The thickness of the sample shall be the worst-case thickness. While the worst-case thickness has been found to vary from material to material, the general trend has been that thinner samples of materials are generally more reactive.

6.8 For the ambient LOX impact test, test conditions (pressure and temperature) are the ambient pressure of the test facility and the boiling point of LOX at that pressure. For the pressurized test, test conditions (pressure and temperature) shall be determined for each test according to the requirements specified by the requester.

6.9 Preparation of the samples for testing involve the following tasks.

6.9.1 Receiving the visually inspecting the material.

6.9.2 Preparing the sample to the specified dimensions.

6.9.3 Cleaning the samples.

6.9.4 Inspecting the samples.

7. Sample Preparation

7.1 The material to be tested must be traceable back to the original manufacturer and to specific batch or lot numbers, or to both. When received, the test material must be accompanied by proper identification, for example, product data sheets, batch or lot numbers identifying the sample, material manufacturer, and appropriate material safety data sheets. The material must be inspected to ensure that it is at the worst-case use thickness and any flaws shall be noted. Preparation of samples for testing involve the following tasks: *(1)* receiving and visually inspecting the material, *(2)* preparing samples to the proper dimensions, *(3)* cleaning the samples, and *(4)* inspecting the samples.

7.1.1 Sufficient material shall be available to permit preparation and testing of 140 separate 17.5-mm (11⁄16-in.) diameter disk samples. Sheet materials up to 6.3-mm (1⁄4-in.) in thickness shall be tested as 17.5 mm (11⁄16-in.) diameter disks in the thickness intended for use (see Table 1).

7.1.2 Materials normally used in thicknesses greater than 6.35 mm (1⁄4 in.) shall be sized and tested as 17.5-mm diameter disks of 6.35- ± 0.13-mm (0.250- ± 0.006-in.) thickness. Failure of samples to meet the requirements of this test method shall be cause for the rejection of the material. Greases, fluids, and other materials, whose thicknesses are directed by conditions of use, shall be tested as 1.27- ± 0.13-mm (0.050- ± 0.005-in.) layers in special test cups. Materials not readily available in sheet form shall be tested in the available configuration. Specimens shall be free of ragged edges, fins, or other irregularities.

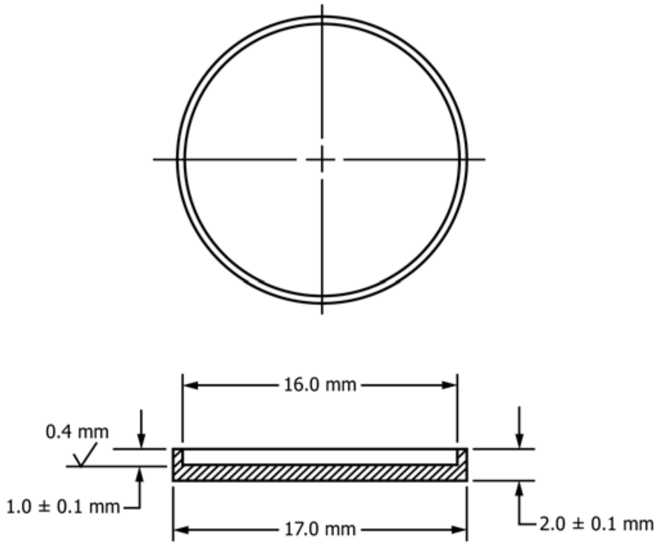
7.2 *Liquid Samples*—Prepare a homogeneous sample. A microburette may be used to transfer the sample into special sample cups 1.27 ± 0.13 mm (0.050 ± 0.005 in.) deep (see Fig. 7). For highly viscous materials, a microsyringe may be used. Determine the volume of the sample required to obtain a sample thickness of 1.27 ± 0.13 mm (0.050 ± 0.005 in.) in the sample cup. This determination is required due to variations in such physical properties as density, surface tension, and volatility from liquid to liquid. A micrometre depth gauge with leveling blocks is suggested for measurement. The work table must be level. Test material should be loaded into the sample cup just before loading the cup into the test chamber (or freezing box, if testing in liquid oxygen).

7.3 *Leak Check Compounds, Dye, Dye Penetrant, and Emulsifier, Method 1*—Clean, unsealed, sulfuric acid-anodized 6061-T6 aluminum alloy disks (or any other substrate specified

TABLE 1 Recommended Minimum Quantities of Material Required for Testing

Material Form[A]	Minimum Quantities
Sheets	2000-cm^2 (319-in.^{2}) total area by 3.5-mm (1⁄8-in.) maximum thickness
Coatings, inks, and adhesives	120 cm^3 (4 fluid oz.)
Foams	2000-cm^2 (310-in.^{2}) total area by 3.5-mm (1⁄8-in.) maximum thickness
Insulated wires	50 cm (20 in.) in length

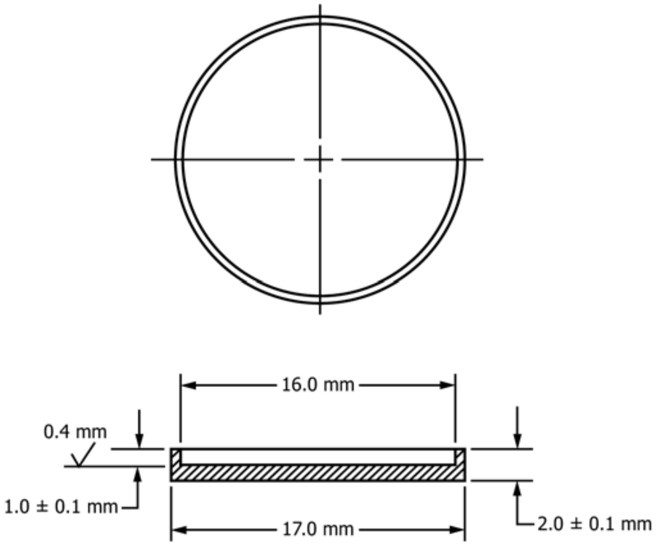
[A] Actual test configurations and material quantities for material forms other than those listed (for example, O-rings and seals) must be established and approved by the responsible procurement or user materials organization.



NOTE 1—Material: any 3000 or 5000 series aluminum alloy.

NOTE 2—Break all sharp edges 0.19 mm.

FIG. 7 LOX Impact Tester Special Insert



NOTE 1—Material: Type 347 stainless steel.

NOTE 2—Break all sharp edges 0.19 mm.

FIG. 8 One-Piece Insert Cup

by the manufacturer or requester), 17.5 mm ($^{11}/_{16}$ in.) in diameter by 1.60 mm (0.063 in.) thick are used as a carrier. Clean the disks before use (see 11.2.2.1). To ascertain the effectiveness of the cleaning procedure, test a minimum of 20 blank disks. After cleaning and blank testing, dip new anodized disks in the test materials for 15 min and drain for 15 min with the disks oriented vertically. Cure the sample as specified, then store the prepared disks in a clean container until required for testing.

7.3.1 Reactions involving materials prepared on aluminum disks have the potential for being extremely severe as a result of ignition of the aluminum disk, which may be initiated by a reaction of the test material. For this reason, Method 2 (7.4) is provided as an alternative procedure for preparing this type of material. In conjunction with Method 2, the user or test agency may elect to use sample cups of the same material on which the dye, dye penetrant, or emulsifier is used in actual service.

7.4 *Preparation of Dye, Dye Penetrant, Leak Check Compounds, and Emulsifier, Method 2*—Clean sample cups 1.27 ± 0.13 mm (0.050 ± 0.005 in.) deep or Type 316 stainless steel disks (see Fig. 8) of the same dimensions specified for the aluminum disks in 10.1.1.5 are used as a carrier (unless other base metal is specified). Before use, the sample cups or disks are cleaned as directed in 11.2.5, and the effectiveness of the cleaning procedure is verified in 11.4. The test material is applied to the inside bottom of the sample cup or to one side of the stainless steel disks in a thickness and a manner simulating actual use, then dried or cured as it would be in actual use. (Alternative method: dip and drain as directed in 7.8.) The resultant thickness is measured and recorded on the test data sheet. Store the prepared test samples in a clean container until required for testing.

7.5 *Preparation of Greases and Semisolids*—Press a sufficient amount of sample material (a slight excess) into a special sample cup 1.27 ± 0.13 mm (0.050 ± 0.005 in.) deep (see Fig. 7) with a cleaned, stainless steel spatula to form a uniform sample free of bubbles and void areas. Scrape the excess sample level to the rim of the sample cup until a smooth surface is obtained. It is necessary to fill the sample cup uniformly. Store the prepared sample cups in a clean container until required for testing.

7.6 *Preparation of Solids*—Cut and prepare samples of solid material to a diameter of 17.5 mm ($^{11}/_{16}$ in.). Sheet material not available in 1.52- ± 0.13-mm (0.060- ± 0.005-in.) thickness is tested in the thickness intended for use when that thickness is not more than 6.35 mm (0.250 in.). Materials normally used in a thickness greater than 6.35 mm (0.250 in.) are cut to provide disks of 1.52- ± 0.13-mm (0.060- ± 0.005-in.) thickness. The samples should be cleaned by the same method that will be used in the material application. Alternatively, the samples may be cleaned by rinsing with an oxygen-compatible solvent that is compatible with the test material, then detergent rinsed, distilled-water rinsed, and dried using filtered (25-µm absolute or smaller filter rating) dry air or inert gas, unless otherwise specified. If the sample material cannot be wetted with any cleaning solution without altering the test sample, the samples shall be blown clean using filtered (25-µm absolute or smaller filter rating) dry air or inert gas.

7.7 *Preparation of Solder (Solid or Flux-Core Type)*—Prepare solder samples as follows: melt the solder (solid or flux-core type) at a temperature not higher than 25°C above the melting point of the solder in a mold to form an ingot. Roll the ingot to form a flat sheet 0.51 ± 0.13 (0.020 ± 0.005 in.) thick. Punch disks of 17.5-mm ($^{11}/_{16}$-in.) diameter from the sheet. Clean the disks by detergent washing, water rinsing, drying, and vapor degreasing in an appropriate solvent. Store the prepared samples in a clean container until required for testing.

7.8 *Preparation of Coatings, Paints, Adhesives, and Potting Compounds*—Materials of this type are prepared as follows.

7.8.1 Coating materials, such as paints, dry film lubricants, and conformal coatings, shall be applied to 17.5-mm ($^{11}/_{16}$-in.)

diameter by 1.6-mm (0.063-in.) thick 316 or 347 stainless steel disks in the same manner and to the same thickness intended for use. After the samples have dried, they shall be placed in the regular sample cups for ambient pressure testing and used as prepared in the pressurized impact tester.

7.8.2 Elastomeric coatings and adhesives shall be applied as a coating to 316 or 347 stainless steel disks using Method 1 or Method 2 described below and cured according to applicable instructions.

7.8.2.1 *Method 1 (Single-Dip Coat)*—Dip coat inserts to specified thickness and place on clean aluminum foil or on PTFE to air dry. The coated inserts shall be removed from the foil and turned over after 30 min to allow both sides to dry. The specimens shall be cured as specified before testing. The coating thickness shall be checked on at least four samples and recorded.

7.8.2.2 *Method 2 (Brush Coat)*—Material shall be applied to inserts using a single brush stroke with a soft nonshedding brush, in single brush coats of finished coating as specified. Each specimen shall be visually examined for contamination (especially bristles from the brush) following application of each coat. The coated specimens shall be air dried for a minimum of 24 h following application of the final coat before testing.

7.9 *O-Rings*—Each size from each batch of O-rings or O-ring materials or both shall be sampled and tested as follows unless it can be demonstrated that test results on different sizes and batches are comparable. To clean O-rings before testing, rinse with tap water, rinse in nonionic detergent solution, rinse in DI water, drain for a minimum of 10 min, and dry using a gaseous nitrogen purge.

7.9.1 *Extruded O-Rings*—140 sample disks 17.5-mm (11⁄16-in.) diameter by the thickness of the O-rings shall be cut from a strip after the chopping operation. The disks shall be similarly processed and deflashed with the same equipment used for the O-rings. The disks shall be cleaned as specified for the material and its use.

7.9.2 *Molded O-Rings*—140 sample disks 17.5-mm (11⁄16-in.) diameter by the thickness of the O-rings, and which have been similarly processed and deflashed, shall be furnished.

7.9.3 *O-Rings From Standard Stock or Where Above Procedures Are Impractical*—O-rings 1.27-cm (1⁄2-in.) outside diameter or less shall be sampled and tested as a complete O-ring. O-rings larger than 1.27-cm (1⁄2-in.) outside diameter shall be tested as one segment (approximately 1.90 cm (3⁄4-in.) long). To clean O-rings before testing, rinse with tap water, wash in nonionic detergent solution, rinse in DI water, drain for a minimum of 10 min, and dry using a gaseous nitrogen purge. If a sample is not impacted during testing, it shall be placed in a new cup and precooled before retesting. As an alternative, sufficient samples may be prepared to account for the normal impact misses.

7.10 *Heat Shrink Tubing*—Heat shrunk tubing shall be preshrunk before testing in accordance with the manufacturer's instructions.

7.11 *Nonmetallic, Solid, Metallic, and Solvent-Resistant Samples*—If received in a certified clean condition, test in the as-received condition. Otherwise, clean the sample before testing by rinsing with tap water, then washing in nonionic detergent solution, then finally rinsing in DI water. Drain for a minimum of 10 min and dry using a gaseous nitrogen purge.

8. Reagents and Materials

8.1 *Alkaline Cleaner,* for test chambers, striker pins, sample cups, and sample holder units, 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.

8.2 *Deionized Water,* conforming to Specification D1193, Type IV.

8.3 *Detergent,* a noncorrosive cleaner that is liquid oxygen compatible in the concentration used, conforming to MIL-D-16791.

8.4 *Gaseous Oxygen,* conforming to CGA G–4.3 Type I B. Oxygen of higher purity may be used if desired (see Note 2).

8.5 *Liquid Oxygen,* conforming to CGA G–4.3, Type II B.

NOTE 2—Gaseous mixtures of the appropriate oxygen concentration and certified analysis may be purchased commercially.

8.6 *Gaseous Nitrogen,* CGA G–10.1, Type IB.

8.7 *Liquid Nitrogen,* CGA G–10.1, Type IIB.

8.8 *Trichloroethylene,* ACS reagent grade or Specification D4080.

9. Safety Precautions

9.1 *LOX*

9.1.1 When testing is to be performed in liquid oxygen, normal safety precautions applicable to the handling and use of liquid oxygen must be used.

9.1.2 The hazards associated with handling oxygen are very serious. Contact with the skin can cause frostbite. Contact of liquid oxygen with hydrocarbons or other fuels constitutes a fire or explosion hazard because such mixtures can be sensitive to shock, impact, or vibration.

9.1.3 Personnel working with liquid oxygen must be familiar with its characteristics. Approved goggles or face shields, fire-retardant protective clothing, gloves, and boots must be worn during handling or transfer. Such operations should be performed by no less than two persons, as a minimum. Extreme caution should be exercised in preventing contact with oils or other combustible materials. All tools must be degreased before use. Precautions should be taken to prevent accumulation of moisture in lines, valves, traps, and so forth to avert freezing and plugging which would cause subsequent pressure ruptures. Care should also be taken to prevent undesired entrapment of liquid oxygen in unvented sections of any system.

9.1.4 Direct physical contact with LOX, cold vapor, or cold equipment can cause serious tissue damage. Medical assistance should be obtained as soon as possible for any cold injury. Proper immediate bystander response be as follows:

(1) If it is safe to do so, remove the patient from the source of the cold.

(2) In the event of limb-size cryogenic exposure, appropriate response may include an attempt to warm the affected

area rapidly with moist heat from a shower, eyewash, or warm water bath, not exceeding 39°C (102°F).

(3) Massive full-body cryogenic exposures present significant additional concerns, but removal of the victim from the exposure atmosphere and keeping the victim's airway open are important. Loosely wrapping the victim in a blanket until the arrival of the ambulance team is also advised.

(4) Some important don'ts: don't remove frozen gloves, shoes, or clothing; slaveageable skin may be pulled off inadvertently. Don't massage the affected part; don't expose the part to temperatures higher than 45°C (112°F), such as heat or fire; this superimposes a burn and further tissue damage; don't apply, ice, snow, or ointments.

9.1.5 Safety shower and other protective equipment should be inspected periodically to ensure that they are operational when needed. Personnel handling liquid oxygen must ensure that oxygen vapors do not remain absorbed in their clothing before smoking or approaching any source of ignition. Desorbtion of oxygen may be accomplished by remaining in a well-ventilated area for 30 min after exiting the test area.

9.2 *GOX*

9.2.1 This is a hazardous test. Normal safety precautions applicable to the operation and maintenance of high-pressure gas systems must be followed when working with the test system.

9.2.2 Complete isolation of personnel from the test apparatus is required whenever the test chamber contains a test sample and is pressurized above atmospheric pressure with oxygen. Violent reactions between test materials and high-pressure oxygen must be expected at all times. Test chamber component failure caused by violent test sample reaction has produced shrapnel, flying objects, dense smoke, and high-pressure gas jets and flames inside the test cell. Test cell design and layout, test procedures, personnel access controls, and emergency shutdown procedures must be designed with this type of failure expected any time the test system contains oxygen.

9.2.3 Complete isolation is assured by locating the test apparatus in an enclosure and behind an barricade. The operator is stationed in a control room on the other side of the barricade. Visual observation of the test shall be accomplished by means such as a reinforced window, periscope, mirrors, or closed-circuit television.

9.2.4 Equipment used in a 69-MPa (10 000-psig) oxygen system must be properly designed and rated for oxygen service. Proper design of high-pressure oxygen systems includes designing for minimum internal volumes, thereby limiting the magnitude of catastrophic reactions that may occur while testing materials. Components such as valves, regulators, gages, filters, and the like, must be fabricated from materials that have a proven record of suitability for high-pressure oxygen service. Examples of such materials are Monel 400, Inconel 600, nickel, and selected stainless steels (see Note 3).

NOTE 3—Where not otherwise indicated, stainless steel shall be of the AISI 300 series.

9.2.5 High-pressure oxygen systems require the utmost cleanliness. Therefore, components should be designed to facilitate disassembly, thorough cleaning, and reassembly without compromise of cleanliness level. Screening tests performed on nonmetallic materials have shown that the impact sensitivity of these materials can vary from batch to batch. Since nonmetallic materials are usually the most easily ignited components in a high-pressure oxygen system, nonmetallic items to be used in this test apparatus, such as seats, seals, and gaskets, should be chosen from the best (that is, least sensitive) available batch of material.

9.2.6 The test chamber shall be isolated from the oxygen source by a double-block-and-bleed valve arrangement consisting of two block valves in series with a vent valve between them. Each block valve shall be locked closed and the vent valve locked open whenever personnel are working in test cell. By ensuring two-valve isolation and continuous venting, the chance of exposing personnel to high-pressure oxygen as a result of inadvertent valve actuation or leakage during sample changeout is minimized (see Fig. 9).

9.2.7 When testing is to be performed at elevated temperature, normal safety precautions applicable to the operation and maintenance of electrical systems must be followed.

9.2.8 The sample heater, heater wiring, and control system must be designed for continuous usage. Adequate precautions must be taken to eliminate the potential for electrical shock. The heater circuit shall be equipped with a safety switch and warning lights in the immediate vicinity of the tester to permit personnel working on the test chamber to assess the condition of the heater circuit. A local method of opening the circuit must be provided in the test chamber area which is in series with the control area heater control switch. A typical instrumentation control system is shown in Fig. 3.

9.2.9 When performing tests at elevated temperatures, personnel must wear heat-resistant gloves for handling hot components or allow the heated components to cool completely before handling them.

9.3 *Trichloroethylene*

9.3.1 **Warning—**Harmful if inhaled. High concentrations may cause unconsciousness or death. Contact may cause skin irritation and dermatitis. Avoid prolonged or repeated breathing of vapor or spray mist. Use only with adequate ventilation. Eye irritation and dizziness are indications of overexposure. Do not take internally. Swallowing may cause injury, illness, or death. Avoid prolonged or repeated contact with skin. Do not get in eyes. Do not allow to contact hot surfaces, since toxic products can be formed.

9.4 *Oxygen*

9.4.1 **Warning—**Oxygen vigorously accelerates combustion. Keep oil and grease away. Do not use oil or grease on regulators, gages, or control equipment, except as suggested by Guide G63. Guides G63, G88, and G94 should be used in the selection of materials used in test systems. 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

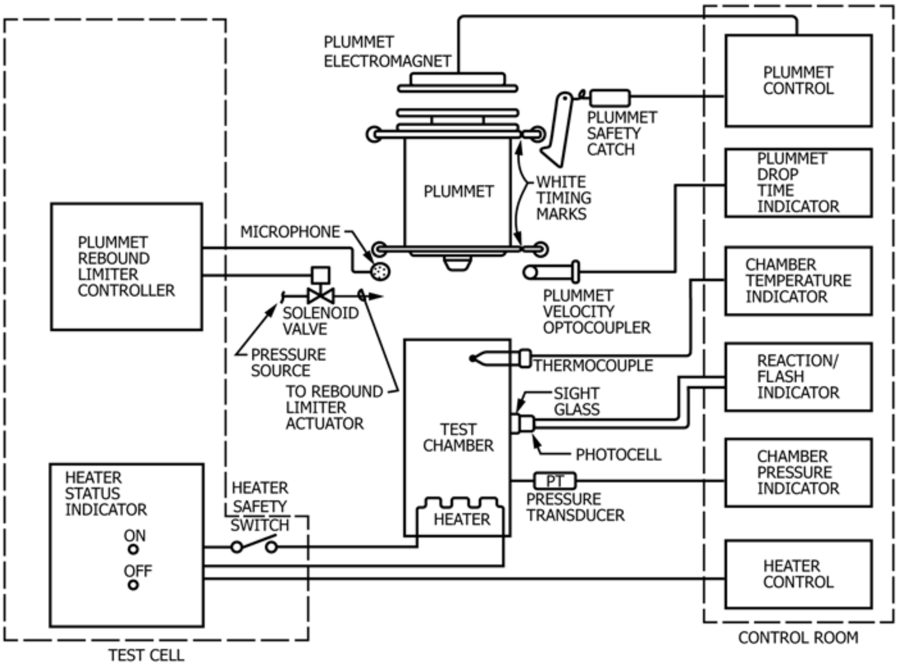


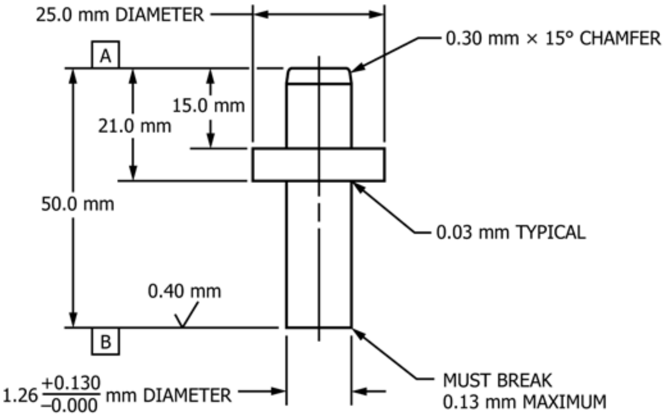
FIG. 9 Typical Instrumentation/Control Diagram for Pressurized LOX/GOX System

valve. All equipment and containers used must be suitable and recommended for oxygen service. Never attempt to transfer oxygen from the cylinder in which it is received to any other cylinder. Do not mix gases in cylinders. Do not drop the cylinder. Make sure cylinders are maintained upright and secured at all times. Keep cylinder valve closed and capped 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.

9.4.2 See Compressed Gas Association Pamphlets G-4, "Oxygen" and G-4.1, "Cleaning Equipment for Oxygen Service" for details of safe practice in the use of oxygen. Practice G93 may be consulted for cleaning practices also.

10. Test Apparatus

10.1 The impact tester for the ambient LOX impact test shall have a rugged structural frame capable of maintaining accurate vertical alignment under repeated shock conditions (see Fig. 1), a mechanism for dropping a plummet which weighs 9.072 ± 0.023 kg (20 ± 0.05 lb) (see Note 4) through a distance of 1.10 ± 0.005 m (43.3± 0.2 in.), which will transmit to the test sample an approximate impact energy of 98 J (72 ft•lbf), a striker pin (see Fig. 10) 1.27 cm (½ in.) in diameter and 5.08 cm (2 in.) long, and a one- or two-piece sample cup (see Fig. 5 and Fig. 11) approximately 2.22-cm (⅞-in.) inside diameter by approximately 2.22-cm (⅞-in.) inside depth made from 0.16-cm (1⁄16-in.) thick aluminum alloy. The initial alignment and subsequent operation of the impact tester shall be such that the plummet falls uniformly under essentially friction-free conditions. This shall be verified by suitable means on each drop to ensure that ±3 % of the theoretical drop time is attained. Measurement shall be made as close to the striker pin as possible. See Section 12 for the calibration of the impact tester.



NOTE 1—Break sharp edges.

NOTE 2—Machine all surfaces to 0.40 mm or smoother.

NOTE 3—Material: stainless steel AMS 5643D.

NOTE 4—Heat treatment: obtain HRC 40–45.

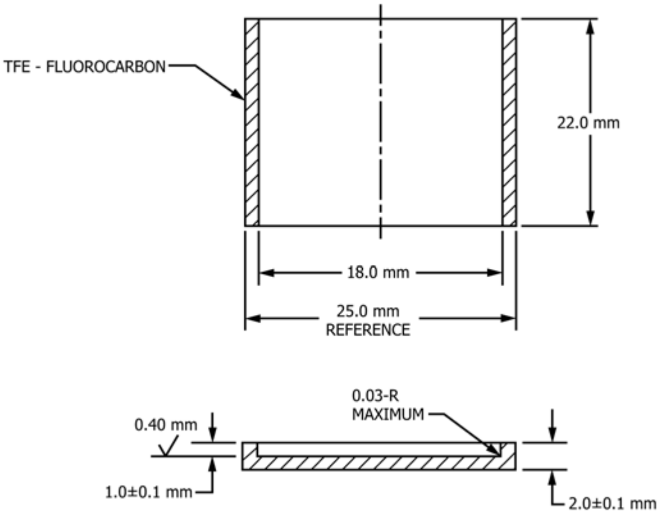
NOTE 5—Finish: electropolish after heat treatment.

NOTE 6—Surfaces A and B should be parallel and perpendicular to the center line.

FIG. 10 LOX Impact Striker Pin

NOTE 4—The weight times the drop height specified is not duplicated for the purposes of this test by combinations other than 9.072 ± 0.023 kg (20± 0.05 lb) through a distance of 1.10 ± 0.005 m (43.3 + 0.2 in.). For example, doubling the mass of the plummet and halving the drop height would not duplicate the specified requirement. Drop height shall be measured from the nose of the plummet to the top of the striker pin with the cup and stainless steel disk in position.

10.1.1 The tester also consists of the following parts:



NOTE 1—Break sharp edges 0.19 mm.

NOTE 2—Surfaces marked shall be parallel within 0.05 mm.

NOTE 3—Material: aluminum alloy 3000 or 5000 series.

FIG. 11 LOX Impact Tester Two-Piece Cup

10.1.1.1 *Electromagnet*—The electromagnet (if used) shall be designed with a sufficient safety factor to hold over 9.08 kg (20 lb) of weight with a minimum energizing wattage. Mechanical suspension/release devices shall be designed with a sufficient safety factor to hold a 9.08-kg (20-lb) plummet positively.

10.1.1.2 *Safety catch*—The solenoid-operated safety catch shall be designed to hold the plummet near the base of the magnet in the event of a power failure.

10.1.1.3 *Base plate*—The base shall be constructed from 2.54-cm (1-in.) minimum thick stainless steel plate and shall rest solidly and level on a base of reinforced concrete. It is recommended that grout be applied to prevent any spilled LOX from being trapped under the steel plate. A minimum of four stainless steel foundation bolts shall be used to anchor the plate to the concrete.

10.1.1.4 *Anvil plate and sample cup holder* (see Fig. 12)—A stainless steel, Type 440B, or equivalent, heat-treated anvil plate 12.7 by 12.7 by 5.1 cm (5 by 5 by 2 in.) thick, shall be bolted to the base plate in the center of the machine. This plate shall center the specimen cup holder and provide the base plate with protection from denting upon impact. A 12.7- by 12.7-cm (5- by 5-in.) stainles steel specimen cup holder, 2.54 cm (1 in.) thick, shall be bolted on top of the anvil plate. The specimen cup holder shall have a slightly tapered hole into which the test cup can be placed.

10.1.1.5 *Sample cups*—One-piece sample cups (see Fig. 5) shall be made of any dead soft 3000 or 5000 series aluminum alloy. A special insert cup made of any 3000 or 5000 series aluminum alloy (see Fig. 7) with an inside depth of 0.127 ± 0.013 cm (0.050 + 0.005 in.) shall also be used when testing semisolid materials. These special insert cups shall be placed inside the one-piece specimen cups.

10.1.1.6 *Striker pins*—The striker pins shall be made from 17-4 PH stainless steel, or equivalent, with hardness in accordance with the applicable detail drawings (see Fig. 10). A sufficient number of pins shall be provided for testing and discard.

10.1.1.7 *Auxiliary equipment*—The auxiliary equipment shall consist of stainless steel forceps for handling the specimen cups and striker pins, stainless steel spatulas, and LOX handling eqipment (for example, stainless steel dewar flasks, fireproof lintless laboratory coats, safety goggles, gloves, and LOX storage containers). Additional handling equipment shall include a grease insert cup holder (see Fig. 8), a sample freezing box (see Fig. 6), striker pin baskets, specimen cup trays, microburettes, syringes, a control panel for the operator to activate the safety catch and electromagnet, and timing instrumentation to measure the drop time of the plummet or its velocity just before impact.

10.1.1.8 *Timer*—A universal counter and timer shall be used to measure drop time. The overall drop time shall be measured and recorded for each drop to ensure that the rated accuracy of the equipment is maintained. A typical timing circuit is shown in Fig. 13.

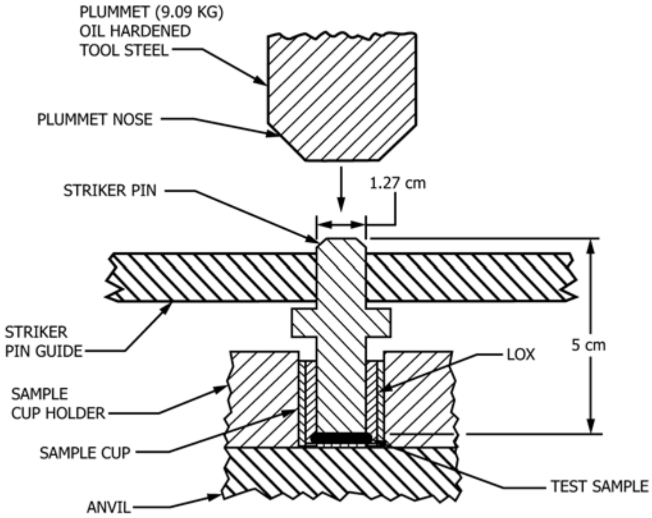


FIG. 12 LOX Impact Tester Sample Cup, Sample Cup Holder, Striker Pin, and Anvil Configuration

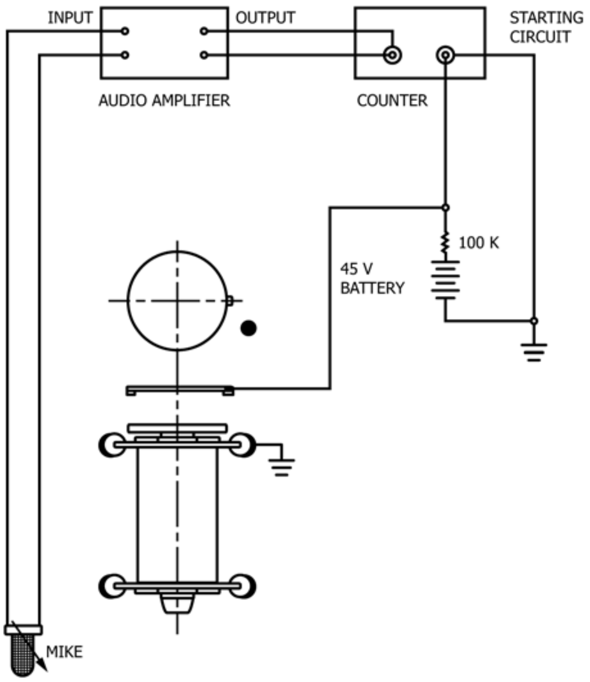


FIG. 13 Typical Free-Fall Timing Circuit

10.1.1.9 *Test cell*—The impact tester shall be housed in a test cell with a concrete floor. Walls shall be constructed of reinforced concrete or metal to provide protection from explosion or fire hazards. The cell shall contain a shatterproof glass observation window. It shall be darkened sufficiently for observation of flashes. Continous ventilation shall provide fresh air to the test cell. Construction of the test cell shall be directed at providing a facilitiy that can be economically maintained at a high level of good housekeeping.

10.2 The mechanical impact test system used for determining the sensitivity of materials to mechanical impact in pressurized LOX or GOX is similar to the ABMA-type tester[8] as described in 10.1 except that the guide tracks have been extended approximately 0.45 m (18 in.) to accommodate the pressurizable test chamber and plummet rebound limiter.

10.2.1 The tester also consists of the following parts:

10.2.1.1 At least two different types of test chambers have been designed and fabricated for the pressurized LOX and GOX tests. Of the two types of chamber designs (see Fig. 2) that have been evaluated, both designs produce equivalent rankings of materials.[9] This test chamber, which contains the test sample and desired test environment during test, has the following features.

10.2.1.2 *Striker pin*—The striker pin has a sample contact diameter of 12.7 mm (0.500 in.) and a sample contact surface finish of 0.406 µm (16 µin.) or smoother, machined from 17-4PH stainless steel (HRC 40-45) or equivalent. A typical striker pin is depicted in Fig. 10.

10.2.1.3 *Sample holder*—The sample holder is for supporting the sample during test, as well as holding liquid oxygen around the test sample when testing in liquid oxygen. For tests requiring elevated sample temperatures, the sample holder or test chamber is equipped with an electrical resistance heater. A typical sample holder is depicted in Fig. 12.

10.2.1.4 *Thermocouple*—The thermocouple is for monitoring test sample temperature.

10.2.1.5 *Pressure transducer*—The pressure transducer shall have a minimum range from ambient to 69 MPa (0 to 10 000 psig) for monitoring test chamber internal pressure.

10.2.1.6 *Photocell*—The photocell is for detecting light emission in the wavelength of 550 to 650 nm produced by material reaction during test, with attendant detector circuitry.

10.2.1.7 *Sample cup*—The sample cup is one piece for liquid or other nonsolid test materials (see Fig. 14). No cup is required for solid materials; solid material samples are placed directly on top of the sample holder plate and retained under the striker pin by a centering ring.

10.2.1.8 *Counterloader*—The counterloader positions the striker pin in contact with the top surface of the test sample when the Type 2 test cell is pressurized.

10.2.1.9 *Plummet catcher*—The plummet catcher limits the plummet to one impact on the striker pin per test (see Fig. 4).

10.2.1.10 *Test cell*—The test cell is constructed of noncombustible material (such as concrete or metal) with suffiicient strength to provide protection from explosion or fire hazards; it houses the impact tester. Continous ventilation shall provide fresh air to the test cell. The cell shall provide a facility that can be maintained economically at a high level of good housekeeping. The test cell shall be cleaned periodically to ensure cleanliness of sample and equipment.

10.2.1.11 *Piping system*—The piping system is used for purging, pressurizing, and venting the test chamber; pressurizing and venting the counterloader; and activating the plummet catcher. The piping system shall be designed to permit test chamber purge, pressurization, and venting and counterloader operation without the presence of personnel in the test cell. The chamber shall be purged and pressurized through one line and vented through a separate line to minimize the chances of contaminant migration into the pressurization line, which could influence subsequent tests. A typical piping system from this test is shown in Fig. 9.

10.2.1.12 *Control area*—The control area isolates test personnel from the test cell during impact tests. This control area shall be provided with the necessary control and instrumentation features to perform test chamber purge, pressurization and venting operations (and sample heating when testing at elevated temperatures), counterloader operation, electromagnet release, and monitoring of the test chamber instrumentation during test. A means of measuring and recording the plummet drop time shall also be provided. A typical instrumentation and control system is illustrated in Fig. 13.

10.2.1.13 *Freezing box*—The freezing box is for pretest conditioning of samples to be tested in liquid oxygen, as illustrated in Fig. 6.

10.2.1.14 *Auxiliary equipment*—Auxiliary equipment consists of clean, powder-free gloves for handling the sample cups,

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[9] NASA Technical Publication 74106, "LOX/GOX Mechanical Impact Tester Assessment."

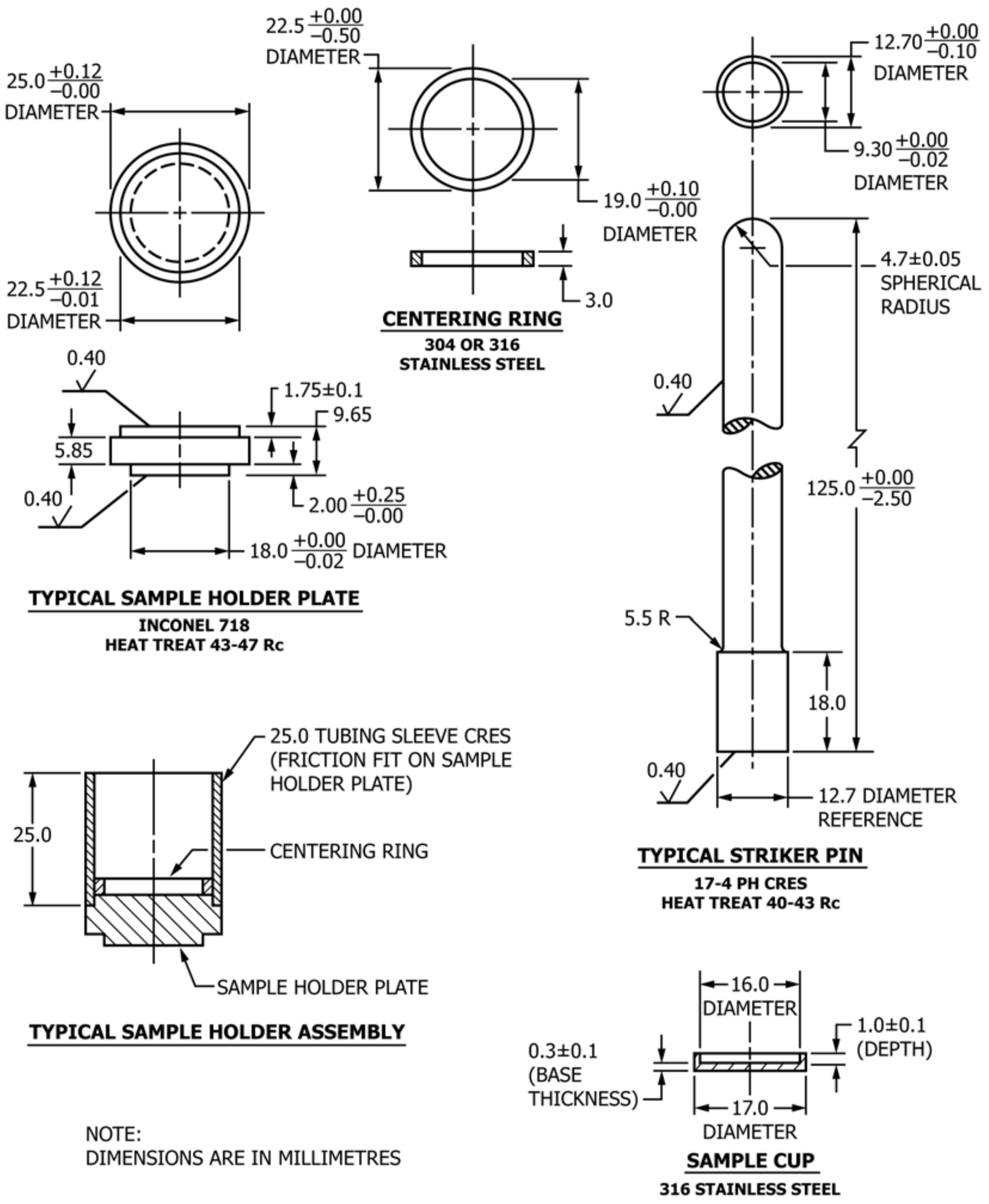

FIG. 14 Typical Test Chamber Internal Components

striker pins, sample cup trays, and covered storage containers for sample cups and striker pins. For tests involving liquid oxygen, liquid oxygen handling equipment, such as stainless steel Dewar flasks, liquid oxygen storage containers, clean cloth or insulating-type protective gloves, clean fire-retardant laboratory coat or coveralls, and eye protection equipment, are also required. When performing tests at elevated temperature, a means of energizing and controlling the sample heater is required. The following items are also required: equipment for preparing test samples; a set of visual roughness comparison standards or a surface roughness measurement instrument for checking surface roughness of striker pins, sample cups, and sample holder plates; and necessary equipment and materials for cleaning test chambers, striker pins, sample cups, and sample holder units.

11. Preparation of Equipment for Testing

11.1 The impact tester, its accessories, and the test cell shall be maintained in clean condition to ensure reproducibility of results and meet the requirements of blank testing as described in 11.4.

11.2 *Ambient LOX Impact Tester*

11.2.1 *General*—The guide tracks, plummet, anvil plate, striker pin guide, sample cup holder, and base plate of the impact tester shall be cleaned thoroughly at the start of each test and between tests of different materials using lint-free cloths and a suitable cleaner such as trichloroethylene or equivalent. As needed, the striker pin guide, plummet, and guide tracks of the impact tester shall be wiped with a lint-free cloth wet with trichloroethylene or equivalent. After completion of testing for the day, the impact tester shall be thoroughly cleaned. Cleanliness shall be maintained throughout any series of impact tests to minimize erroneous results.

11.2.2 *Striker Pins and Insert Disks*—Striker pins shall be used only once and then cleaned. Before each test, the striker pins shall be checked for dimensional conformance to the applicable drawing and then examined to ensure freedom from scratches, nicks, metallic slivers, and other imperfections on the impacting surface of the pins. When required, the pins shall be remachined to remove scratches and pits. All striker pins shall be cleaned, dried, and stored in a suitable container to maintain cleanliness.

11.2.2.1 The striker pins and insert disks shall be rinsed in trichloroethylene, or equivalent, air dried, immersed in a hot alkaline cleaner for a minimum of 15 min, rinsed with running tap water, rinsed with DI water, and dried in a convection oven at 125 to 150°C. They shall be removed from the oven and stored in a clean, dry container. An alternate cleaning method to a visible clean level may be used per Practice G93 recommended procedures.

11.2.2.2 *Remachined striker pins*—The length of remachined striker pins for the ambient LOX impact test shall be 5.080 plus 0.025 or minus 0.254 cm (2.000 plus 0.010 or minus 0.100 in.). Striker pins shall be remachined if the striking area exhibits scratches or pits or both. It may be necessary to remachine pins that are bent or flattened during impact tests. Badly bent and remachined pins, in which the diameter near the striking area is greater than 1.283 cm (0.505 in.) or is less than 1.257 cm (0.495 in.), shall be discarded.

11.2.3 *Stainless Steel Inserts*—Type 347 stainless steel or equivalent inserts (see Fig. 8), 1.75-cm (11⁄16-in.) diameter, shall be cleaned in accordance with the method specified for striker pins. Inserts shall be used only once and then discarded.

11.2.4 *Specimen Cups*—The one-piece cup, lower part of the two-piece specimen cup, and one-piece insert are made of an aluminum alloy. These parts shall be cleaned by rinsing with trichloroethylene or equivalent, rinsing in tap water, soaking for 20 min in a hot alkaline detergent, and rinsing thoroughly in DI water. This is followed by a 15-min immersion in a solution of the following mixture at room temperature: 0.5 volume percent of concentrated (48 %) HF, 5.0 volume percent of concentrated HNO_3 (relative density of 1.42), and 94.5 volume percent of DI water. Rinse in DI water and desmut by immersing the parts for 5 min or until the smut is removed in a 50 volume percent solution of concentrated HNO_3 (relative density of 1.42). Rinse with DI water and dry in an oven at 125 to 150°C until dry. The cups shall not be used until at least 72 h after cleaning. An alternate cleaning method to a visibly clean level may be used per Practice G93 recommended procedures.

11.2.5 *Sleeve for the Two-Piece Specimen Cup*—The cup includes a sleeve made of stainless steel. Clean this sleeve as follows: soak in aqua regia for a minimum of 10 min, rinse with tap water, rinse in nonionic detergent solution, rinse in DI water, drain for a minimum of 10 min, rinse with trichloroethylene or equivalent, and dry in an oven at 125 to 150°C. The sleeves may be used as soon as they cool.

11.3 *Pressurized LOX/GOX Impact Tester*

11.3.1 Thoroughly clean the guide tracks, plummet, and chamber region at the start of each day using lint-free wipes and a suitable cleaner, such as trichloroethylene or equivalent. Clean the sample preparation equipment as required to prevent cross-contamination of test materials.

11.3.2 *Striker Pins and Sample Holder Units*—Before each test, check the striker pins and sample holder units for dimensional and finish conformance to drawing requirements. The striker pins shall be fabricated from 17-4 PH stainless steel (HRC 40 - 45) or equivalent. The striker pins and sample holder units shall be rinsed in trichloroethylene, or equivalent, air dried, immersed in a hot alkaline cleaner for a minimum of 15 min, rinsed with running tap water, then rinsed again with DI water, and dried in a convection oven at 125 to 150°C. They shall be removed from the oven and stored in a clean, dry, covered container. An alternate cleaning method to a visibly clean level may be used per Practice G93 recommended procedures.

11.3.3 *Sample Cups*—Clean the sample cups in the same manner as striker pins and sample holder units.

11.3.4 *Test Chamber*—Initially, clean the test chamber in the same manner as the striker pins and sample holder units. Thereafter, clean the test chamber interior after any test in which a reaction is observed or suspected and after completion of the testing of each material. As needed, perform solvent cleaning per Practice G93 recommended procedures to a visibly clean level. Cleaning may be performed with the chamber in place. After cleaning, blow-dry the chamber with filtered (25-µm absolute or smaller filter rating) dry air or gaseous nitrogen. After inital cleaning, maintain a visibly clean test chamber interior.

11.3.5 *Test System Piping and Components*—Clean test system pressurization piping and components that provide gas to the test chamber to the same level as the test chamber before initial use. Thereafter, maintain cleanliness of the test system piping and components to the level that will provide clean oxygen to the test chamber.

11.4 *Blank Check*—The cleaning procedures specified herein will provide an adequate level of cleanliness for oxygen service. Other cleaning agents and procedures may be substituted if they provide a level of cleanliness equal to or better than the level obtained using the agents and procedure specified above. Check the effectiveness of any cleaning procedure used to clean striker pins, sample holder units, and sample cups by testing at the 98-J (72-ft•lbf) level with a series of 20 striker pins and sample holder units (and sample cups, if applicable) cleaned in accordance with the procedure. In addition to these blank tests, perform four blank tests at random during any 8-h period of testing. Additional blank tests may be made at the discretion of the operator. The striker pins and sample cups shall be handled by forceps or tongs and kept out of contact with the operator's hands or other sources of contamination.

11.5 *Cleaning Procedures for Auxiliary Equipment*

11.5.1 *Test Cell*—The interior surfaces of the test cell shall be maintained in a sufficiently clean condition to preclude biasing of the test data which may occur as a result of introduction of local contamination onto the test material or apparatus.

11.5.2 *Stainless Steel Ware*—Stainless steel ware, such as striker pin baskets, forceps, tongs, spatulas, and inserts, shall be cleaned in the same manner as the striker pins. Once integrated into the handling procedure, a thorough rinse of the stainless steel ware with trichloroethylene or equivalent should be the only cleaning necessary.

11.5.3 *Glassware Check*—Any glassware, such as microburettes, beakers, or syringes, shall be cleaned in the same manner as the striker pins, dried, and stored in suitable containers to maintain cleanliness.

12. Calibration of Tester

12.1 Verify that the vertical guide tracks are properly aligned and the plummet assembly will encounter minimal friction so that essentially free-fall conditions are obtained (see Note 5). This is accomplished by timing the plummet fall from a given height. For this test, the free-fall time shall be within ±3 % of the theoretical drop time calculated by using the prevailing test site gravitational acceleration in the calculation.

NOTE 5—The ideal conditions for plummet free fall are obtained when the guide rails are aligned perfectly vertical, the center of gravity of the plummet lies exactly on the longitudinal axis of the plummet, the plummet base and magnet face are perfectly flat and perpendicular to the longitudinal axis of the plummet, the magnetic field collapses uniformly when the magnet is deenergized, and the plummet bearings have sufficient clearance to avoid contact with the guide rails during the downward plunge, yet keep the plummet restrained within the rails during the rebound. The theoretical drop time of the plummet may be calculated by the equation $t = \sqrt{\frac{2h}{g}}$, where t is the theoretical free fall time, h is the drop height, and g is gravitational constant for the latitude of the test site.

12.1.1 Establish the energy delivered by the tester to a calibration sample before the tester is placed into service and at least once per year thereafter. Evaluate the effect of any basic design changes to the tester by this calibration technique before continuing use of the tester.

12.1.2 Establish a calibration curve for each tester initially by determining the penetration depth (d) at each level given in Table 2 and plotting the square of this value versus energy level. Perform subsequent verification at the 14-, 41-, 69-, and 98-J (10-, 30-, 50-, and 72-ft•lbf) energy levels.

12.1.3 The calibration test specimen shall be 304 stainless steel disk annealed to a hardness of 150 ± 15 HB, 17.48 ± 0.254 mm (0.688 ± 0.010 in.) in diameter, and 8.890 ± 0.254 mm (0.350± 0.010 in.) thick, with a surface of 0.41 µm (16 µin.) or better, cleaned as directed in 11.2.2.1.

12.2 *Calibration of Ambient Pressure Mechanical Impact LOX Testers*

12.2.1 This procedure uses liquid nitrogen as the fluid medium.

12.2.2 The basic ambient pressure mechanical impact tester (described in 10.1) shall be used with appropriate modifications as follows.

TABLE 2 Drop Height Schedule for Energy Threshold Value Determination Using a 9.07-kg (20-lb) Plummet

Energy		Drop Height	
J	Ft•lbf	Metres	Inches
98	72	1.1	43.3
88	65	0.99	39.0
81	60	0.91	36.0
75	55	0.84	33.0
69	50	0.76	30.0
61	45	0.69	27.0
54	40	0.61	24.0
48	35	0.53	21.0
41	30	0.46	18.0
34	25	0.38	15.0
27	20	0.31	12.0
20	15	0.23	9.0
14	10	0.15	6.0

12.2.3 The standard striker pin shall be used with the tip remachined into a 12.7- ± 0.127-mm (0.500- ± 0.005-in.) hemisphere having an 0.2-µm (8-µin.) or smoother surface finish (see Fig. 14).

12.2.4 No sample cups shall be used.

12.2.5 Use a plummet catcher.

12.3 *Calibration of Pressurized Mechanical Impact Tester*

12.3.1 Verify that the plummet catcher will limit the plummet to one impact on the counterloader pin per test.

12.3.2 Calibrate the test chamber pressure versus counterloader pressure for each increment of test pressure. Using gaseous nitrogen, pressurize the test chamber to the test pressure, then increase the counterloader pressure until the striker pin moves into positive contact with the test sample. Affix a force gauge to the counterloader pin. Adjust counterloader pressure until the downward counterload force on the test specimen is 220 ± 20 N (50 ± 5 lbf). Verify the calibration a minimum of five times for each test pressure. Rate of force application is important to preclude damage to the test sample; therefore, do not pressurize the counterloader in less than 1 s.

12.3.3 Perform the calibration with the test chamber stabilized at atmospheric pressure and a temperature of 20 ± 5.5°C (70 ± 10°F). The user may select other calibration conditions, if desired, to evaluate further or compare tester performance.

12.3.4 Calibration is performed using the tester in the standard configuration, except the standard striker pin is replaced with one that has the tip machined into a 12.7- ± 0.127-mm (0.500- ± 0.005-in.) diameter hemisphere having an 0.2-µm (8-µin.) or smoother surface finish (see Fig. 15).

12.3.5 After verifying that the tester ancillary equipment is in current calibration and working properly, load the prepared calibration sample into the test chamber in the same manner as a test sample.

12.3.6 Perform a standard drop test on a total of five calibration specimens at each approrpriate energy level. Examine the striker pin after each impact and replace if deformed. Deformation can be expected to occur at the higher energy levels.

12.4 *Calculation of the Penetration Function*

12.4.1 Measure the diameter of the indentation in each of the calibration specimens to the nearest 0.013 mm (0.0005 in.) by averaging two readings taken 90° apart.

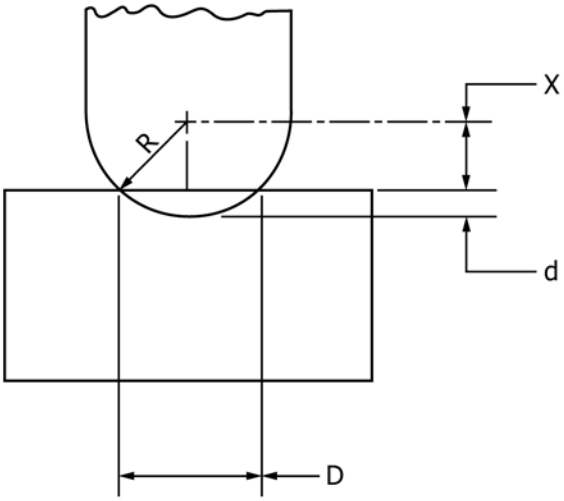

FIG. 15 Determination of the Penetration Depth from the Measured Diameter

12.4.2 The penetration depth (*d*) is determined from the measured diameter of the indentation (see Fig. 15) by the following equation:

$$d = R - \sqrt{R^2 - 0.25D^2} \tag{1}$$

where:

R = striker radius (6.35 mm):
D = dent diameter, (mm); and
d = penetration depth, mm.

12.4.3 Plot the penetration function (d^2) versus energy level using a linear least-squares curve fit forced through the origin. Determine the correlation coefficient for this line. The calibration and vertification curves must have a slope greater than 0.0100 mm^2 per J and must have a correlation coefficient greater than 0.985 when the curve is forced through the origin.[10]

13. Ambient Pressure LOX Mechanical Impact and Screening Test

13.1 The energy loss as a result of friction shall be controlled by measuring and recording the drop time of each drop of any given height. The observed plummet drop time shall not deviate more than 3 % from the theoretical drop time.

13.2 The effectiveness of the cleaning procedure shall be checked by testing blank cups. The cups shall be prepared as specified in 13.3.3.

13.3 *Precooling Procedure*

13.3.1 Precooling shall be accomplished by lowering the temperature of the material to the boiling point of LOX at atmospheric pressure.

13.3.2 Stainless steel Dewar flasks shall be precooled by slowly pouring LOX into the flask until the LOX ceases to boil. When cooled, the flask shall be filled with LOX and covered with clean aluminum foil or a stainless steel cover. Since glass Dewar flasks are easily broken, they should not be used.

13.3.3 Clean, blank specimen cups shall be precooled by slowly pouring LOX into a sample freezing box until the LOX covers and fills the cups. To minimize the cracking of samples in specimen cups during freezing, the cups containing the samples shall be precooled slowly by using the freezing box shown in Fig. 6. First, level the box before use. Then, place approximately 40 specimen cups over the holes in the retainer plate. Using an automatic delivery tube with a 25-L Dewar flask, pressurize the Dewar flask to approximately 120 kPa (2.5 psig) with oxygen. While maintaining the pressure at approximately 120 kPa (2.5 psig), open the discharge valve approximately halfway and start to fill the freezing box. As an alternate, a nonpressurized hand Dewar may be used. The time required to fill the cups and the freezing box 3.2 mm (⅛ in.) from the top shall be 35 to 40 min. After this step, a careful inspection shall be made. Any sample that separates from the bottom of the cup and floats shall be forcibly submerged in LOX during conditioning. Any sample that shatters as a result of thermal shock shall be discarded. The samples shall be transferred to a suitable (precooled) container for storage until tested. Additions of LOX shall be made, as necessary, while tests are in progress to ensure that each sample cup is completely filled at time of testing.

13.3.4 Cleaned striker pins shall be precooled by placing pins in a Dewar flask and adding liquid nitrogen or liquid oxygen to the flask until the pins are covered.

13.3.5 The specimen cup holder and anvil plate shall be precooled by adding liquid nitrogen or liquid oxygen to the stainless steel moat surrounding the specimen cup holder and base plate. The moat should be filled with liquid nitrogen or liquid oxygen to approximately 12.7 mm (½ in.) below the top of the specimen cup holder. Time should be allowed to bring the base plate and anvil to equilibrium temperature before starting the test.

13.4 *Test Procedure*—Adjust the magnet located on the impact tester to the proper drop height (see Table 2). Using clean tongs, set the precooled specimen cups into the specimen cup holder. Visually check to assure that the frozen sample is fully submerged in LOX. Samples that float shall be restrained under the LOX surface by the striker pin. Place the striker pin in the cup and hold it in place by the striker pin guide. Add the LOX to the specimen cup to ensure that the specimen cup is full. Cover the exposed container of LOX. The operator shall leave the test cell, close the test cell door, and extinguish all illumination in the test cell and observation area. Release the plummet safety catch, then release the plummet. Observe visually or electronically and record the results of the impact reaction on the data sheet. Verify that the drop time of the plummet was within specified tolerance.

13.5 *Test Logic*—Table 2 lists a set of recommended drop heights to be used for this test method and the potential energy for each height represented. Initiate testing at the drop height of 1.1 m (43.3 in.) unless otherwise specified. If no reactions occur, perform 20 test drops. If a reaction occurs, test until 2 reactions occur in 60 tests drops. If no more than 1 reaction occurs in 60 test drops, testing may be considered complete. If

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[10] *Lox/Gox Mechanical Impact Tester Assessment*, J. W. Bransford, C. J. Bryan, S. L. Stohler, NASA Technical Memorandum TM-74106, Kennedy Space Center, FL, Feb. 1980 .

2 reactions occur in 60 test drops, decrease the drop height until no reactions occur in 20 test drops. The energy threshold is the highest impact energy level for which the passing critieria has been met (see Section 5.3).

13.6 Failure of any sample to meet the requirements of this specification shall be cause for rejection of the lot represented. Rejected materials shall not be submitted for approval without full details being furnished concerning the previous rejection and measures taken to correct the defects. If not more than one specimen of the original test sample fails to meet the test requirements specified herein, the manufacturer may request a retest. If all test specimens of the retest meet the requirements specified herein, the material shall be considered compatible for use in LOX systems.

13.7 *Data Collection*—Collect and record the following data. Determine whether or not a positive reaction was obtained for each drop and report as (1) no reaction, (2) evidence of light emission, (3) obvious discoloration or char, (4) odor (however, a burnt odor alone is not considered sufficient evidence that a reaction has occurred), and (5) material deformation as a result of reaction (such as, melting of striker pins, sample cups, and the like). Determine and record the drop height of the plummet. The data sheet shall also include sample size and number of blank tests performed. Fig. 16 provides a sample data sheet.

14. Pressurized LOX/GOX Mechanical Impact and Screening Test

14.1 The energy loss as a result of friction shall be controlled by measuring and recording the drop time of each drop of any given height. The observed plummet drop time shall not deviate more than 3 % from the theoretical drop time.

14.2 The effectiveness of the cleaning procedure shall be checked by testing blank cups. The cups shall be prepared as specified in 13.3.3.

14.3 *Precooling Procedure*—If the material is to be tested in liquid oxygen, the samples must be preconditioned in liquid oxygen. Use a freezing box such as shown in Fig. 6, taking steps to ensure that it is level. Place the sample holder units, each containing a test sample, over the holes on top of the retainer plate. Fill the freezing box and cups with liquid oxygen. The time required to fill the cups and box to 3.2 mm (⅛ in.) from the top shall be between 35 and 40 min. Submerge the test samples in the liquid oxygen during the precooling process. Cracking of samples shall not be cause for rejection; however, samples that shatter shall be discarded. Additions of liquid oxygen shall be made, as necessary, while testing is in progress to ensure that each sample remains submerged and each sample holder unit is completely filled when it is loaded into the test chamber. Precool cleaned striker pins by the same procedure described in 13.3.4.

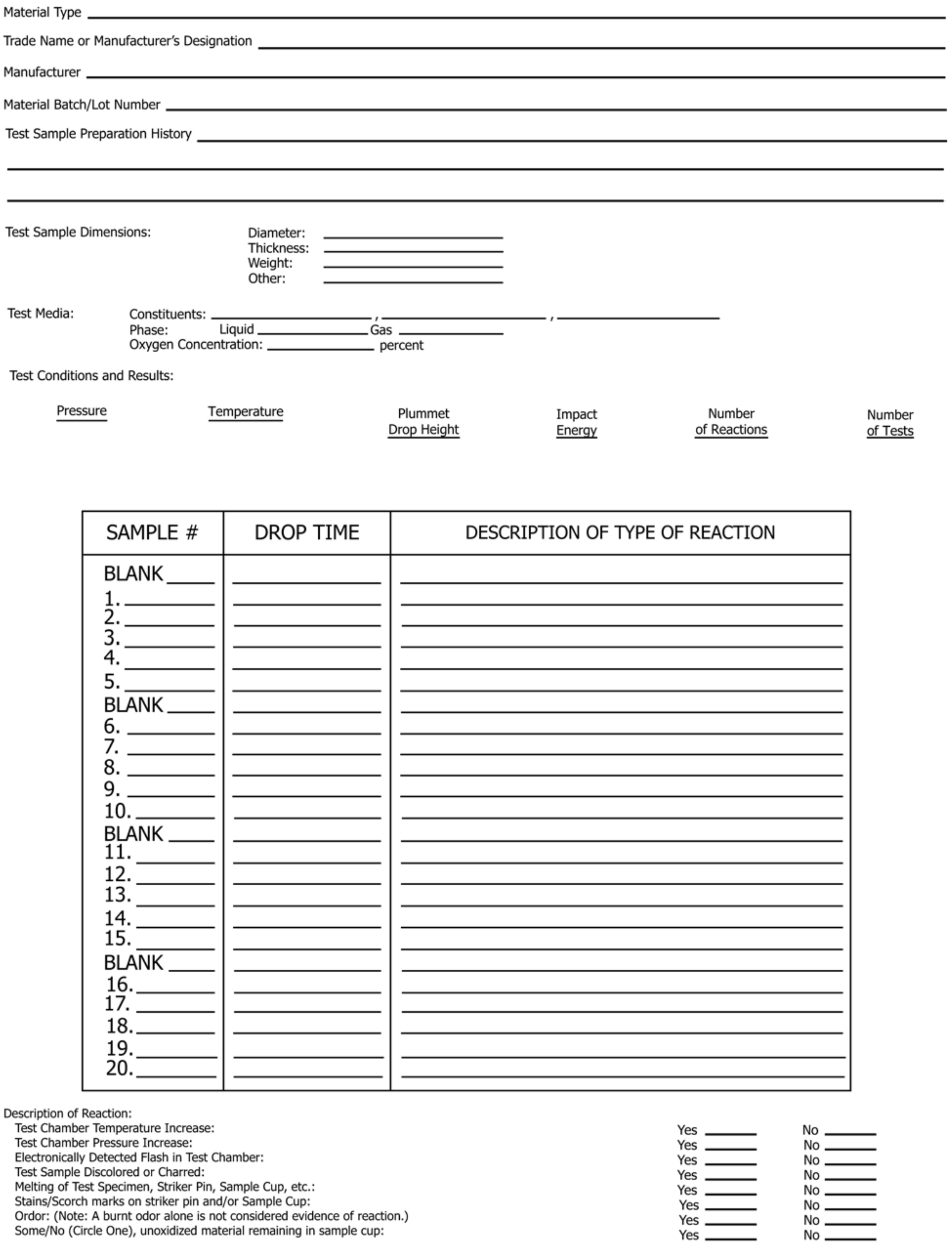
14.4 *Drop Test Using Liquid Oxygen*—Verify that the instrumentation and the test chamber, counterloader (Type 2 test chamber only), and plummet-catcher pressure systems are activated and ready to support testing. Adjust the magnet of the impact tester to the drop height to be used. Adjust the supply-pressure regulator to provide the pressure that will be required to press the striker pin positively against the top surface of the test sample after test chamber pressurization. Verify that the precooled sample holder unit containing the test sample is completely filled with liquid oxygen.

14.4.1 Alternative methods of providing a liquid oxygen environment around the test sample after the test sample is loaded into the test chamber are to flow liquid oxygen into the test chamber after the test chamber is sealed or to flow gaseous oxygen into the chamber after the tester is sealed and chilled to LOX temperature and allow the oxygen to condense.

14.5 Load the sample into the test chamber, using noncontaminating tongs, taking care not to spill any liquid oxygen from the sample holder unit. Seal the chamber, then verify the plummet catcher is set in the ready position. Remove all personnel from the test cell. Purge the test chamber with oxygen, pressurize the test chamber to the desired test pressure ±3 %, and then actuate the counterloader pressure control valve if a Type 2 test chamber is used. Release the plummet safety catch, then release the plummet. Observe the test chamber instrumentation for evidence of a reaction. Verify that the plummet drop time was within specification and that the plummet catcher precluded more than one impact of the plummet on the striker pin. When test chamber conditions appear to be stabilized after impact, vent the test chamber to atmospheric pressure. Allow the test chamber vent valve to remain open. Enter the test cell and raise and latch the plummet to the magnet safety catch. Vent the counterloader (if used) and the plummet catcher and allow them to return to their neutral positions. Remove the sample holder unit from the test chamber and perform an inspection of the test sample and test chamber interior.

14.5.1 The previous steps must be performed as rapidly as possible to minimize the loss of liquid oxygen from the sample holder unit before dropping the plummet.

14.6 *Drop Test Using Gaseous Oxygen at Ambient Temperature*—Verify that the instrumentation and the pressure systems of the test chamber, counterloader (if used), and plummet catcher pressure systems are activated and ready to support testing. Adjust the magnet of the impact tester to the drop height to be used. If a Type 2 test chamber is being used, adjust the supply pressure regulator of the counterloader to provide the pressure required to restrain the striker pin positively against the top surface of the test sample after test chamber pressurization. Load the sample holder unit containing the test sample into the test chamber. Seal the chamber, then verify that the plummet catcher is set in the ready position. Remove all personnel from the test cell. Purge the test chamber with gaseous oxygen to remove the air. A 1-min purge from a 446-kPa (50-psig) system is adequate for this purpose. Pressurize the test chamber to the desired test pressure ±3 %, then actuate the counterloader pressure control valve if a Type 2 test chamber is being used. Release the plummet safety catch, then release the plummet. Observe the test chamber instrumentation for evidence of a reaction. Verify that the plummet drop time was within specification. When test chamber conditions appear to be stabilized after impact, vent the test chamber to atmospheric pressure. Allow the test chamber vent valve to remain open. Enter the test cell and raise and latch the plummet to the



Material Type ____________________

Trade Name or Manufacturer's Designation ____________________

Manufacturer ____________________

Material Batch/Lot Number ____________________

Test Sample Preparation History ____________________

____

____

Test Sample Dimensions:
Diameter: __________
Thickness: __________
Weight: __________
Other: __________

Test Media:
Constituents: __________ , __________ , __________
Phase: Liquid __________ Gas __________
Oxygen Concentration: __________ percent

Test Conditions and Results:

Pressure	Temperature	Plummet Drop Height	Impact Energy	Number of Reactions	Number of Tests

SAMPLE #	DROP TIME	DESCRIPTION OF TYPE OF REACTION
BLANK _____	_____	_____
1. _____	_____	_____
2. _____	_____	_____
3. _____	_____	_____
4. _____	_____	_____
5. _____	_____	_____
BLANK _____	_____	_____
6. _____	_____	_____
7. _____	_____	_____
8. _____	_____	_____
9. _____	_____	_____
10. _____	_____	_____
BLANK _____	_____	_____
11. _____	_____	_____
12. _____	_____	_____
13. _____	_____	_____
14. _____	_____	_____
15. _____	_____	_____
BLANK _____	_____	_____
16. _____	_____	_____
17. _____	_____	_____
18. _____	_____	_____
19. _____	_____	_____
20. _____	_____	_____

Description of Reaction:

Test Chamber Temperature Increase:	Yes _____	No _____
Test Chamber Pressure Increase:	Yes _____	No _____
Electronically Detected Flash in Test Chamber:	Yes _____	No _____
Test Sample Discolored or Charred:	Yes _____	No _____
Melting of Test Specimen, Striker Pin, Sample Cup, etc.:	Yes _____	No _____
Stains/Scorch marks on striker pin and/or Sample Cup:	Yes _____	No _____
Ordor: (Note: A burnt odor alone is not considered evidence of reaction.)	Yes _____	No _____
Some/No (Circle One), unoxidized material remaining in sample cup:	Yes _____	No _____

FIG. 16 Sample Data Sheet

magnet safety catch. Vent the counterloader (if used) and the plummet catcher and allow them to return to their neutral positions. Remove the sample holder unit from the test chamber and perform and inspection of the test sample and test chamber interior.

14.7 *Drop Test Using Gaseous Oxygen at Elevated Temperature*—Verify that the instrumentations and the test chamber, counterloader, and plummet catcher pressure systems are activated and ready to support testing. Adjust the magnet of the impact tester to the drop height to be used. If a Type 2 test chamber is being used, adjust the counterloader supply-pressure regulator to provide the pressure required to restrain the striker pin positively against the top surface of the test sample after test chamber pressurization (empirically determined valves of 220- ± 20-N (50- ± 5-lbf) force are currently used). Load the sample holder unit containing the test sample into the test chamber. Verify that the test chamber thermocouple is positioned so that it will contact the top surface of the sample outside the area in contact with the striker pin. Seal the chambers and connect the heater circuit leads to the heater terminals. Verify that the plummet catcher is set in the ready position. Pressurize the test chamber to the desired test pressure ±3 %, then, if a Type 2 test chamber is being used, actuate the counterloader pressure control valve. Do not pressurize the counterloader in less than 1 s. Select the heater safety switch to the ON position. Remove all personnel from the test cell. Purge the test chamber with gaseous oxygen to remove the air. A 1-min purge from 445-kPa (50-psig) system is adequate for this purpose. Energize the heater and bring the test sample to the desired test temperature (see Note 6).

NOTE 6—The effects of heating on the chamber pressure should be considered when pressurizing the chamber so that overpressurization does not occur. It is undesirable to vent the test chamber after heating is initiated due to the possible loss of volatile material which may have been offgassed from the test material and the possible effects of this losss on the indicated reactivity of the material.

14.7.1 When the desired test conditions of pressure and temperature are attained, turn the heater off. Release the plummet safety catch, then release the plummet. Observe the test chamber instrumentation for evidence of a reaction. Verify that the plummet drop time was within specification. When test chamber conditions appear to be stabilized after impact, vent the test chamber to atmospheric pressure. Allow the test chamber vent valve to remain open. Enter the test cell, and turn the heater safety switch to the OFF position. Raise and latch the plummet to the magnet safety catch. Vent the counterloader (if used) and the plummet catcher and allow them to return to their neutral position. Carefully remove the sample holder unit from the test chamber and perform an inspection of the test sample and test chamber interior.

14.8 *Test Logic*—Table 2 lists a set of recommended drop heights to be used for this test method and the potential energy each height represents. The capabilities of the equipment used in this test method permit the screening of materials by either pressure (14.8.1) or impact energy (14.8.2).

14.8.1 *Pressure Threshold Screening Method*—This screening method varies the test chamber pressure and keeps the impact energy constant. Prepare a minimum of 80 samples. Perform tests at a drop height of 1.1 m (43.3 in.) unless otherwise specified. Vary the pressure in increments/decrements of 3.45 MPa (500 psi) unless otherwise specified. Initiate testing at a pressure level estimated to produce reactions. If the material passes (no reactions in 20 drop tests or no more than 1 reaction in 60 drop tests), increase the pressure. If the material fails, decrease the pressure until the material passes. For tests at a drop height less than 1.1 m (43.3 in), follow the same logic with the exception that the criteria for failure is one reaction obtained in a maximum of 20 samples tested.

14.8.2 *Energy Threshold Screening Method*—This screening method varies the impact energy and keeps the chamber pressure constant. Prepare a minimum of 80 samples. Perform tests at a series of drop heights such as those listed in Table 2. Initiate testing at a drop height of 1.1 m (43.3 in.) unless otherwise specified. If not reactions are observed, perform 20 tests. If reactions are observed, test until 2 reactions are obtained in 60 tests. If no more than 1 reaction is obtained in 60 tests, testing can be considered complete. If two reactions are observed in 60 tests, decrease the drop heighht until no reactions are observed in 20 tests.

14.9 *Data Collection*—Collect and record the following data. Determine whether or not a positive reaction was obtained for each drop and report as (1) no reaction, (2) test chamber temperature increase, (3) test chamber pressure increase, (4) evidence of light emission in the test chamber, (5) obvious discoloration or char, (6) odor (however, a burnt odor alone is not considered sufficient evidence that a reaction has occurred), and (7) material deformation reaction (such as, melting of striker pins, sample cups, and the like). Determine and record the drop height of the plummet and the test chamber predrop pressure and temperature. The data sheet shall also include sample size and number of blank tests performed. Fig. 16 provides a sample data sheet.

15. Report

15.1 In reporting the test results, the following data shall be included:

15.1.1 Type of material, generic name, trade name (if any), manufacturer, composition, and batch/lot number (if known);

15.1.2 Material preparation and cure information (if known);

15.1.3 Sample dimensions and weight (average) of four samples randomly selected;

15.1.4 Threshold level (impact energy);

15.1.5 Test pressure;

15.1.6 Test temperature;

15.1.7 Test thickness,

15.1.8 Description of reaction; and

15.1.9 Any other pertinent remarks.

16. Precision and Bias

16.1 *Precision*—An interlaboratory study of the threshold value of ambient pressure LOX impact testers and pressurized LOX/GOX impact testers was conducted. Two results should be suspect if they differ by more than 7.3 J within one laboratory and by more than 14.4 J between laboratories.

16.2 *Accuracy*—No accuracy statement is possible since no standard reference materials are available for this test method that are traceable to NIST.

17. Keywords

17.1 compatibility; ignition; impact test; mechanical impact; oxygen; pressurized oxygen; liquid oxygen; gaseous oxygen

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