Designation: D2512 – 17

Standard Test Method for Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold and Pass-Fail Techniques)[1]

This standard is issued under the fixed designation D2512; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This method[2,3,4] covers the determination of compatibility and relative sensitivity of materials with liquid oxygen under impact energy using the Army Ballistic Missile Agency (ABMA)-type impact tester. Materials that are impact-sensitive with liquid oxygen are generally also sensitive to reaction by other forms of energy in the presence of oxygen.

1.2 *This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.*

1.3 *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.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[5]

C145 Specification for Solid Load-Bearing Concrete Masonry Units (Withdrawn 1992)[6]

D1193 Specification for Reagent Water

2.2 *Military Standards:*

MIL-D-16791G Detergent, General Purpose (Liquid, Nonionic)[7]

MIL-P-27401C Propellant Pressurizing Agent, Nitrogen[7]

MIL-PRF-25508F Propellant, Oxygen[7]

MIL-T-27602B Trichloroethylene, Oxygen Propellant Compatible[8]

MIL-C-81302D Cleaning Compound, Solvent, Trichlorotrifluorocarbon[7]

2.3 *ASTM Adjuncts:*

Type Impact Tester and Anvil Region Assembly, 38 Drawings

3. Summary of Test Method

3.1 A sample of the test material is placed in a specimen cup, precooled and covered with liquid oxygen, and placed in the cup holder located in the anvil region assembly of the impact tester. A precooled striker pin is then centered in the cup. The plummet is dropped from selected heights onto the pin, which transmits the energy to the test specimen. Observation for any reaction is made and the liquid oxygen impact sensitivity of the test material is noted. Drop tests are continued using a fresh specimen cup and striker pin for each drop, until

[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.

Current edition approved July 1, 2017. Published July 2017. Originally approved in 1966. Last previous edition approved in 2002 as D2512 – 95 (2002)$^{\varepsilon 1}$ which was withdrawn January 2017 and reinstated in July 2017. DOI: 10.1520/D2512-17.

[2] *NASA Handbook 8060. 1B, Ambient LOX Mechanical Impact Screening Test*, September 1981, pp. 4-53 through 4-71. "Oxygen Systems." George C. Marshall Space Flight Center, National Aeronautics and Space Administration. *Specification MSFC 106B*. September 1981.

[3] "Lubrication and Related Research and Test Method Development for Aviation Propulsion Systems." *Technical Report No. 59-726.* Wright Air Development Division, January 1960.

[4] "General Safety Precautions for Missile Liquid Propellants."

[5] 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.

[6] The last approved version of this historical standard is referenced on www.astm.org.

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

[8] Cancelled in 1983. Previously available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://dodssp.daps.dla.mil.

the threshold valve is achieved. A series of drop tests are conducted at an energy level of 98 J (72 ft·lbf) or as specified for the pass-fail tests.

4. Significance and Use

4.1 When this test method is used to measure the threshold impact sensitivity of a material, a relative sensitivity assessment is obtained which permits the ranking of materials.

4.2 This test method may also be used for acceptance-testing materials for use in liquid oxygen systems. Twenty separate samples of the material submerged in liquid oxygen are subjected to 98 J (72 ft·lbf) or as specified. Impact energy delivered through a 12.7-mm (½-in.) diameter contact. More than one indication of sensitivity is cause for immediate rejection. A single explosion, flash, or other indication of sensitivity during the initial series of 20 tests requires that an additional 40 samples be tested without incident to ensure acceptability of the material.

4.3 The threshold values are determined by this test method at ambient pressure. The sensitivity of materials to mechanical impact is known to increase with increasing pressure. Since most liquid oxygen systems operate at pressures above ambient condition, some consideration should be given to increased sensitivity and reactivity of materials at higher pressure when selecting materials for use in pressurized system.

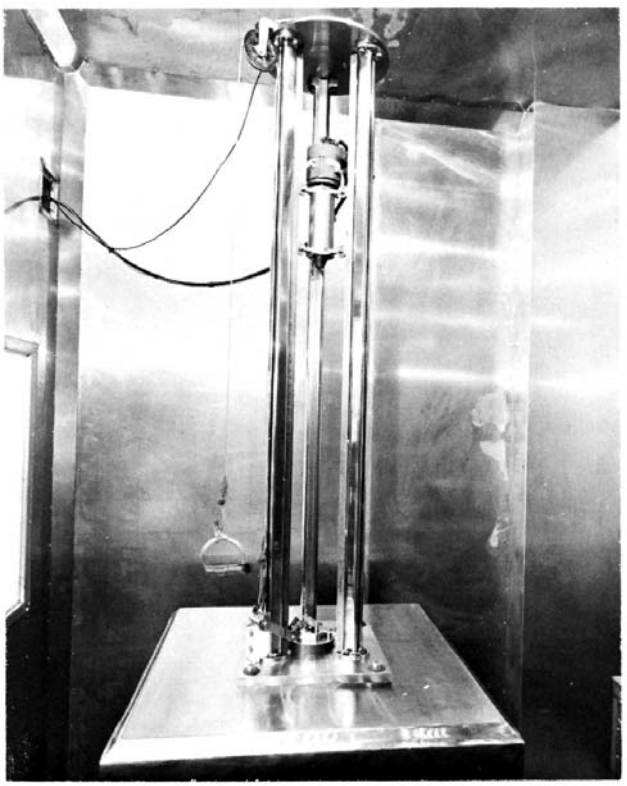


FIG. 1 ABMA-Type Impact Tester

5. Apparatus

5.1 *ABMA-Type Impact Tester*[9] (Fig. 1 , *See* ADJD2512), for determining the sensitivity of materials to liquid oxygen with impact energy. Fig. 2 shows the schematic diagram of the typical power supply. The tester consists of the following parts:

5.1.1 *Three Guide Tracks,* capable of maintaining accurate vertical alignment under repeated shock conditions.

5.1.2 *Plummet,* with a weight of 9.072 ± 0.023 kg (20 ± 0.05 lbs).

5.1.3 *Safety Catch,* operated by a solenoid, and designed to hold the plummet near the base of the magnet. It is used to support the plummet in the event of a power failure.

5.1.4 *Electromagnet,* for supporting or releasing the plummet. The electromagnet is designed to hold 9.072 kg (20 lbs) of weight with a minimum amount of electrical energy.

5.1.5 *Base*—The base of the tester is composed of the following: a rigid 0.61- by 0.61- by 0.61-m (2- by 2- by 2-ft) (min) reinforced concrete block (concrete conforming to Specification C145), a 3.2-mm (⅛-in.) stainless steel sheet, and a 25-mm (1-in.) thick stainless steel base plate. Four stainless steel foundation bolts protruding from the concrete block are used to fasten the plate and sheet to the smooth surface of the concrete block with stainless steel nuts.

NOTE 1—Where not otherwise indicated, stainless steel shall be of the AISI 300 series.

5.1.6 *Anvil Plate (*Fig. 3*),* made from a 51-mm (2-in.) thick Type 440B heat-treated steel plate, (56 to 58 HRC) that is centered and rests on the base plate. It in turn centers the specimen cup holder.

[9] Detailed drawings for the ABMA-Type Impact Tester and Anvil Region Assembly are available from ASTM International Headquarters. Order Adjunct ADJD2512.

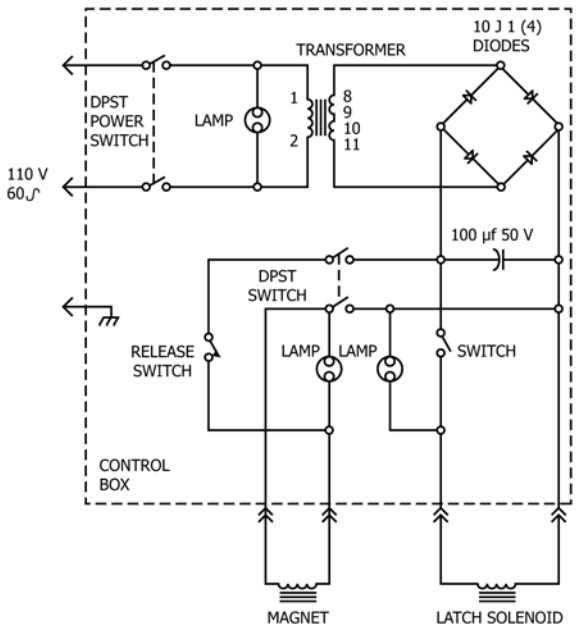


FIG. 2 Schematic Diagram of Power Supply

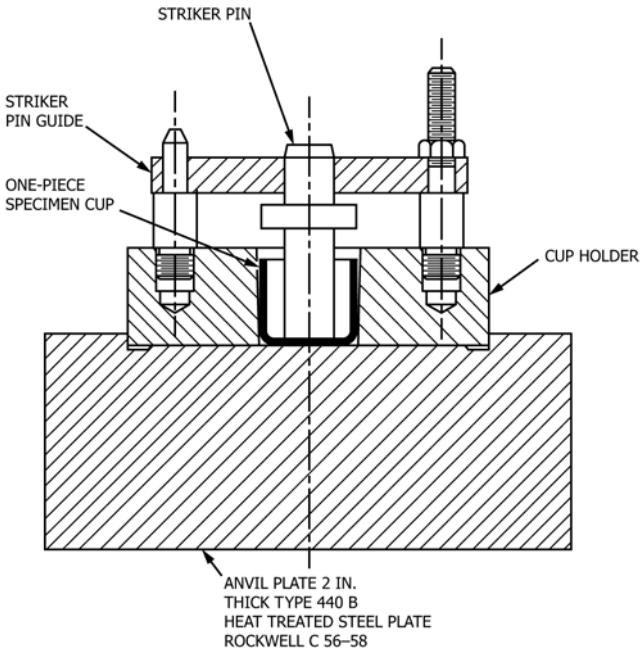
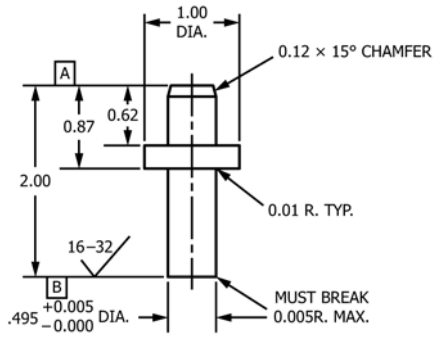


FIG. 3 Anvil Region Assembly

5.1.7 *Striker Pin*—The striker pins shall be machined from AMS 5643D stainless steel, heat condition H-900 (Fig. 4).

5.1.8 *Striker Pin Guide.*

5.1.9 *Specimen Cups*—One- and two-piece specimen cups shall be used. The one-piece specimen cup (Fig. 5) shall be used for liquid and solid test materials. When testing hard samples that are sometimes capable of initiating reactions with the aluminum cup, expendable Type 347 stainless steel disks 17.5 mm (11⁄16 in.) in diameter by 1.6 mm (1⁄16 in.) thick shall be placed in the bottom of the cup. The two-piece cup (Fig. 6) shall be used for testing semisolid materials; a one-piece insert cup (Fig. 7) may also be used. The recess of either of these permits use of a 1.27-mm (0.050-in.) thick sample.



NOTE 1—Break sharp edges approximately 0.015.
NOTE 2—Machine all surfaces 32 rums except as noted.
NOTE 3—Material: stainless steel AMS 5643 D.
NOTE 4—Heat treatment: H-900 to obtain Rc 43 to 44.
NOTE 5—Finish: electropolish after heat treatment.
NOTE 6—Surfaces A and B should be parallel and perpendicular to the center line within 0.001TIR and 16-32 rms along a radius.
All dimensions in inches.

FIG. 4 Striker Pin

5.1.10 *Specimen Cup Holder,* consisting of a 25-mm (1-in.) thick stainless steel block centered on the anvil plate. This holder has two protruding spacers which align the striker pin guide, and in turn the striker pin, with the nose of the plummet, thus ensuring a direct hit by the nose of the plummet on the striker pin in the specimen cup.

5.2 *Test Cell*—The impact tester shall be housed in a test cell containing a concrete floor. Walls shall be constructed of reinforced concrete or metal to provide protection from explosion or fire hazards. The cell shall be provided with a shatterproof observation window, and shall be darkened sufficiently to permit observation of flashes. The operator shall be located in a darkened area. Continuous ventilation shall provide fresh air to the test cell. Construction of the cell shall be directed at providing 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.

5.3 *Freezing Box,* as illustrated in Fig. 8.

5.4 *Auxiliary Equipment*—The auxiliary equipment shall consist of forceps for handling the specimen cups and striker pins, stainless steel spatulas, liquid oxygen handling equipment such as stainless steel Dewar flasks, liquid oxygen protective gloves, lintless laboratory coat, eye protection equipment, and liquid oxygen storage containers. Special handling equipment shall include striker pin holders (Fig. 9), specimen cup trays, covered storage container for specimen cups and striker pins, and a vapor-phase degreaser. The following items are also recommended: microburet, control panel with switches to activate the safety catch and electromagnet, stereomicroscope, micrometer depth gage with leveling blocks, press punch cutter for preparation of plastic specimens, oven, and refrigerator. For checking surface roughness of striker pins and specimen cups, a set of visual roughness comparison standards[10] or a surface roughness measuring instrument shall be required. Timing instrumentation shall be required to measure the drop time of the plummet. A suitable free-fall timing circuit is illustrated in Fig. 10.

6. Reagents and Materials

6.1 *Alkaline Cleaner,* for striker pins and stainless steel inserts, consisting of a solution of 15 g of sodium hydroxide (NaOH), 15 g of trisodium phosphate (Na_3PO_4), and 1 L of distilled or deionized water.

6.2 *Alkaline Cleaner,* for cups; a nonetch-type solution such as Enthone NE[11] or equivalent shall be used.

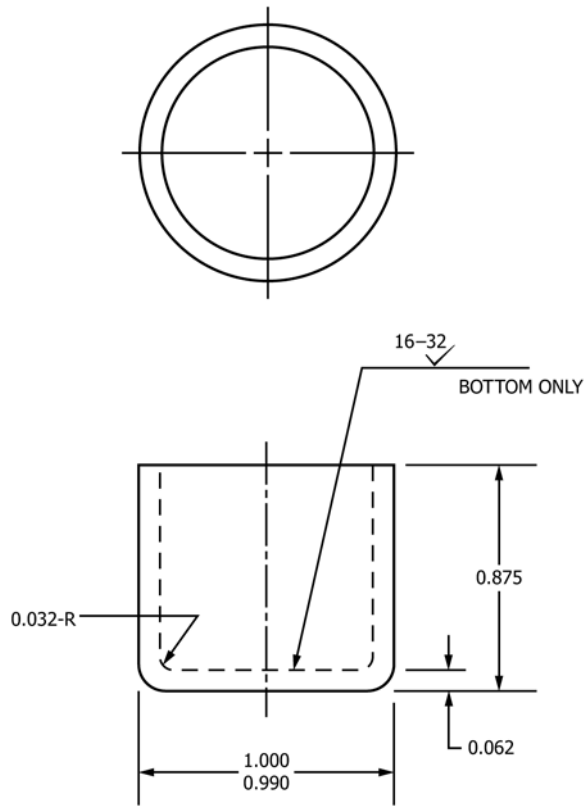
6.3 *Aqua Regia*—Mix 18 parts of concentrated HNO_3 (sp gr 1.42) with 82 parts of concentrated hydrochloric acid (HCl, sp gr 1.19) by volume.

6.4 *Deionized Water,* conforming to Specification D1193.

6.5 *Detergent, General-Purpose (Liquid, Nonionic),* conforming to MIL-D-16791G.

[10] American National Standard B46.1-1962. Surface Texture standards may be used.

[11] Available from Enthone, Inc., a division of American Smelting and Refining Co., Box 1900, New Haven, CT 06508.



NOTE 1—Break sharp edges 0.015.
NOTE 2—The cup is formed by deep drawing.
NOTE 3—The thickness and parallelness of the cup bottom shall be controllled to 0.0610 to 0.0630 by coining.
NOTE 4—Materal: aluminum alloy QQ-A-318 (5052) temper H32.
All dimensions in inches.

FIG. 5 One-Piece Specimen Cup

6.6 *Hydrofluoric Acid* (48.0 to 51.0 %)—Reagent grade concentrated hydrofluoric acid (HF).

6.7 *Liquid Nitrogen,* conforming to MIL-P-27401C. (**Warning—**Contact with the skin can cause frostbites resembling burns.)

6.8 *Gaseous Nitrogen,* conforming to MIL-P-27401C. (**Warning—**Compressed gas under high pressure. Always use a pressure regulator. Release regulator tension before opening cylinder.)

6.9 *Liquid Oxygen,* conforming to MIL-PRF-25508F. (**Warning—**Oxygen vigorously accelerates combustion. Contact with skin can cause frostbite resembling burns.)

6.10 *Nitric Acid* (relative density 1.42)—Reagent grade nitric acid (HNO_3).

6.11 *Trichloroethylene,* conforming to MIL-T-27602. (**Warning—**Harmful if inhaled. High concentrations may cause unconsciousness or death. Contact may cause skin irritation and dermatitis.)

NOTE 2—The use of trichloroethylene is banned in California by the California Air Pollution Board.

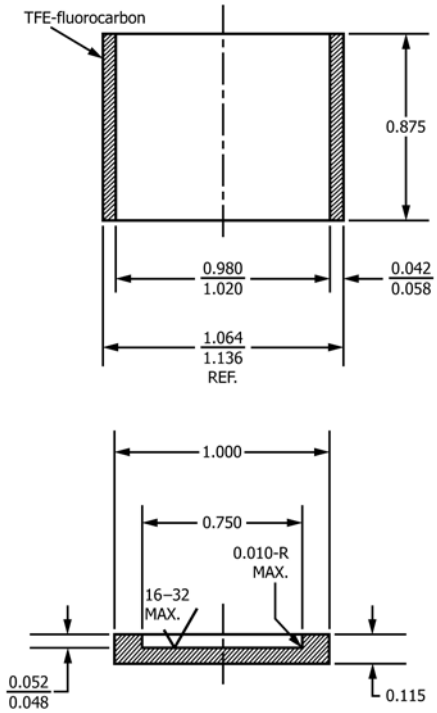
6.12 **Trichlorotrifluoroethane**, conforming to MIL-C-81302D Type I. (**Warning—**Harmful if inhaled.)

7. Safety Precautions

7.1 The hazards involved with liquid oxygen are very serious. Contact with the skin can cause frostbites resembling burns. Contact with hydrocarbons or other fuels causes an explosion hazard, as such mixtures are usually shock, impact, and vibration-sensitive.

7.2 The first-aid procedure for liquid oxygen contact is to flush the affected area with water. This treatment should be followed by medical attention. A safety shower must be available in the immediate area.

7.3 The following safety rules must be observed: personnel working with liquid oxygen must be familiar with its nature and characteristics. Approved goggles or face shields, protective clothing, gloves, and boots must be worn during handling or transfer. Such operations shall be performed by not less than two persons as a group. Extreme caution shall be exercised in preventing contact with oils or other combustible materials. All tools must be degreased before use. Precautions shall be taken to prevent accumulation of moisture in lines, valves, traps, and so forth to avert freezing and plugging with subsequent pressure ruptures. Care shall also be taken to prevent entrapment of liquid oxygen in unvented sections of any system.

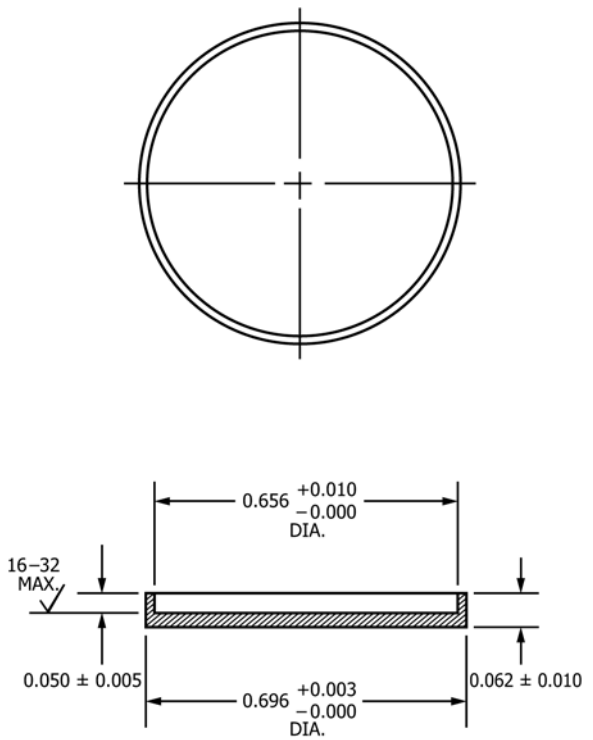


NOTE 1—Break sharp edges 0.015.

NOTE 2—Surfaces marked shall be parallel within 0.002 TIR.

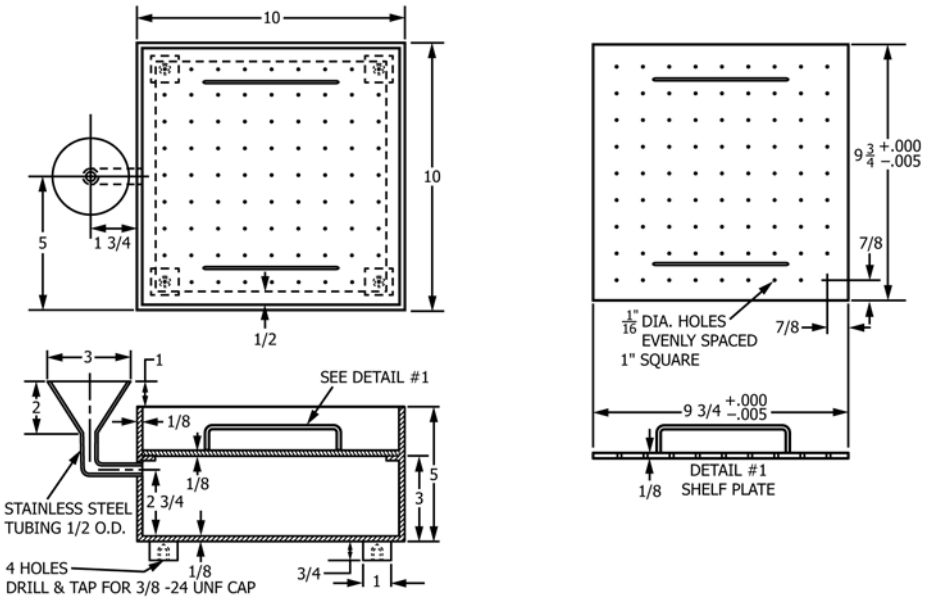
NOTE 3—Material: aluminum alloy QQ-A-318 (5052), temper H-32. All dimensions in inches.

FIG. 6 Two-Piece Specimen Cup



NOTE 1—Material: aluminum alloy QQ-A-318 (5052), temper H-32. All dimensions in inches.

FIG. 7 One-Piece Insert Cup



NOTE 1—Weld all joints.

NOTE 2—The top cover shall be made of clear PCTFE plastic 0.32 thick.

NOTE 3—Material: stainless steel AISI 300 series.

All dimensions in inches.

FIG. 8 Freezing Box

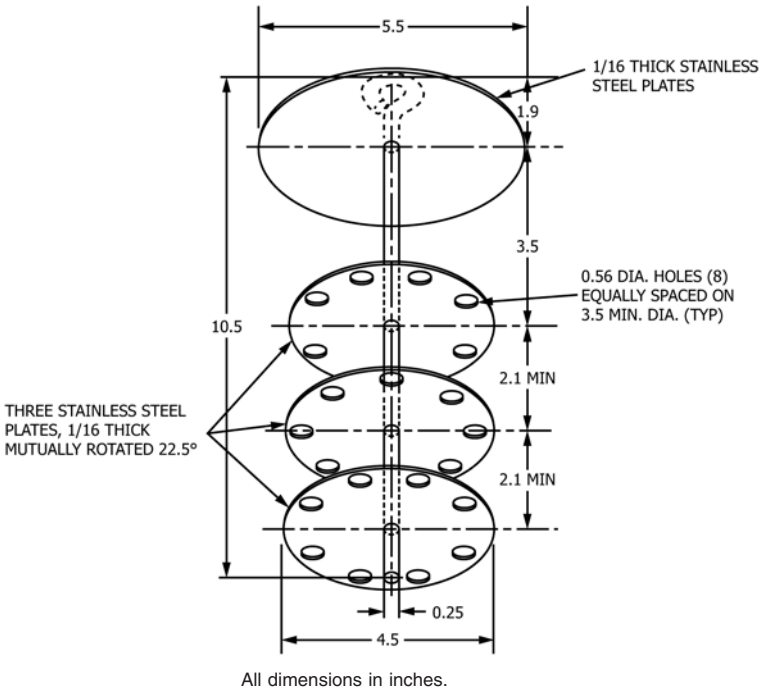


FIG. 9 Striker Pin Holder

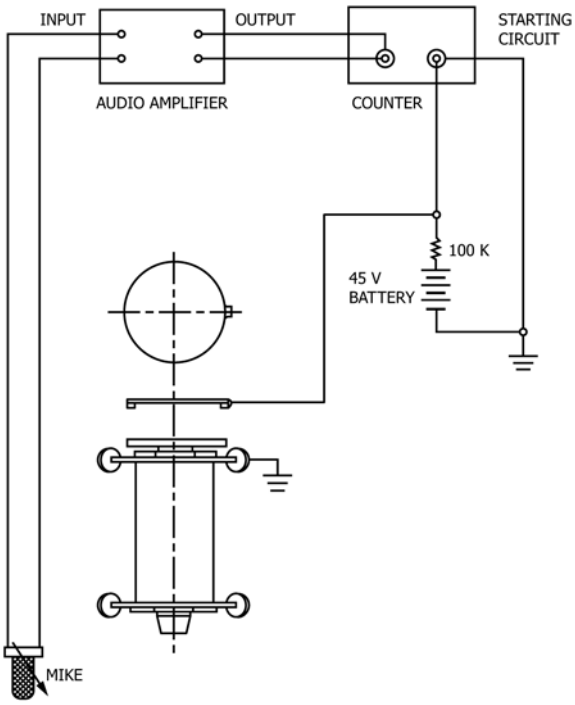


FIG. 10 Free-Fall Timing Circuit

7.4 Safety shower and other protective equipment shall be inspected periodically and before each handling of liquid oxygen. Personnel leaving the working or storage area shall take steps to make sure that no oxygen remains absorbed in clothing before smoking or approaching any source of ignition.

7.5 The threshold limit value, that is, the time-weighed average concentration of trichloroethylene believed safe for continuous exposure during a normal 8-h workday, has been established by the American Conference of Governmental Industrial Hygienists at 100 ppm. Operations using trichloroethylene should always be conducted in a well-ventilated area. The comparable figure for trichlorotrifluorethane is 1000 ppm, and normal ventilation is usually adequate. When a ventilation system is used, an effort should be made to have the natural air

currents in the vicinity assist rather than oppose the mechanical ventilation. For cleaning by vapor degreasing, the vapor level shall be controlled by heat input and cooling coils, which establish a vapor "ceiling." Such vapor degreasing units should always be installed in a location that is free from draft conditions and should be ventilated by horizontal slot exhausts.

8. Preparation of Apparatus

8.1 *General*—The impact tester, its accessories, and the test cell shall be maintained in clean condition to ensure reproducibility of results and to meet the requirements of blank testing as described in 8.5. The guide tracks, plummet, anvil plate, striker pin guide, specimen 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 by using cheesecloth soaked with fresh trichloroethylene or trichlorotrifluoroethane. In addition, the anvil plate, specimen cup holder, striker pin guide, and plummet nose shall be cleaned with fresh trichloroethylene or trichlorotrifluoroethane at least after every tenth impact during a test series. After completion of testing for the day, the impact tester handling equipment (Dewar flasks and forceps), and sample preparation equipment, shall be rinsed with fresh trichloroethylene or trichlorotrifluoroethane.

8.2 *Striker Pins and Insert Disks*—Before each test, the striker pin shall be checked for dimensional conformance to drawing and then examined to ensure freedom from scratches, nicks, metallic slivers, and other imperfections. The striker pins shall be cleaned, adhering to a specified cleaning sequence. Fig. 9 shows a suitable striker pin holder which holds as many as 24 striker pins. By using such a tool during the entire handling of the pins, the striking surfaces of the pins are protected from possible damage as a result of handling. The pins shall be placed in the striker pin holder, rinsed in liquid trichloroethylene or trichlorotrifluoroethane, and vapor-degreased for 5 min. This sequence shall be followed with an air dry. The striker pins shall be immersed in an alkaline cleaner (see 6.1) for a minimum of 15 min at 20 to 35°C. This procedure shall be followed by a rinse in running tap water and then a rinse in distilled or deionized water. The striker pins shall then be dried in an oven at 125 to 150°C until free of water. They shall be removed from the oven and stored in a suitable covered container. The stainless steel insert disks shall be handled in an analogous manner through the same cleaning steps.

8.3 *Specimen Cups*—The one-piece cup, lower part of the two-piece specimen cup, and one-piece insert (Fig. 5, Fig. 6, and Fig. 7, respectively) are made of aluminum alloy. They shall be cleaned as follows: vapor degrease in trichloroethylene in an inverted position for 15 min. Rinse with tap water. Soak for 20 min in hot alkaline cleaner (see 6.2). Rinse thoroughly with deionized water. Immerse for 15 min at ambient temperature in a mixture of the following: 0.5 volume % of concentrated HF (48 %), 5.0 volume % of concentrated HNO_3 (relative density 1.42), and 94.5 volume % of deionized water. Rinse with deionized water. Desmut by immersing at 20 to 35°C in a 50 volume % solution of concentrated HNO_3 (relative density 1.42) in deionized water for 5 min or until smut is removed. Rinse thoroughly with deionized water. Dry in an oven at 125 to 150°C until free of water. The cups shall not be used until at least 72 h after cleaning.

8.4 *Sleeve for Two-Piece Specimen Cup*—The cup includes a sleeve made of polytetrafluoroethylene (PTFE). This sleeve shall be cleaned as follows: soak in aqua regia for a minimum of 10 min. Rinse with tap water; rinse with detergent; rinse with distilled or deionized water, drain for a minimum of 10 min, and rinse with trichloroethylene or trichlorotrifluoroethane. Dry in an oven at 125 to 150°C until free from water. The sleeves may be used as soon as they are cooled.

8.5 A liquid oxygen cleanliness check (blank) shall be performed with at least 20 cleaned specimen cups and striker pins selected at random from every batch cleaned. The blank cups filled with liquid oxygen, the striker pins, and the cup holder shall be precooled as specified in 10.10. These blank tests shall be performed from a height of 1.1 m (43.3 in.) or the drop height of the test. If no reaction occurs, the specimen cups, striker pins, and test facility may be considered clean. If a reaction occurs, the source of the reaction must be determined and corrected, and the check procedure repeated.

9. Calibration of Tester

9.1 It is necessary to ascertain the alignment of the vertical guide tracks and the friction of the plummet assembly. This is accomplished by timing the plummet fall from a given height. For this test, the free-fall timing is required to be within 3 % of theoretical for the prevailing gravitational field.

9.2 The approximate plummet rebound height shall be determined visually using the one-piece cup filled with liquid oxygen or nitrogen and noting the rebound height of the top plate of the plummet. The approximate plummet rebound heights shall be determined for each drop height specified in Table 1.

10. Procedure

10.1 *Preparation of Liquid Samples as Supplied*—Prepare a homogenous sample. A microburet may be used to transfer the specimen into the specimen cups. For 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 specimen cup before freezing. (This determination is required due to variations from liquid to liquid in physical properties such as density, surface tension, and volatility.) A micrometer depth gage with leveling blocks is suggested for measurement. The work table must be level.

10.2 *Preparation of Liquid Samples, Concentrated*—Concentrate the liquid specimens of organic cleaning solvents

TABLE 1 Drop Height Schedule for Approximate Threshold Value Determination

	mm (in.)
First height	1100 (43.3)
Second height	838 (33)
Third height	610 (24)
Fourth height	406 (15)
Fifth height	152 (6)

before their addition to the one-piece specimen cup if acceptance is based on a nonvolatile residue insensitivity requirement. Concentrate the liquid sample to 2 % of its original volume by evaporating the sample in a large round-bottom flask heated in a constant-temperature water bath, at a temperature no greater than 5°C below the boiling point of the sample. Pass air over the surface of the sample at a rapid rate. A full description of the apparatus and procedure for the sample preparation is given in the Annex A1, Method A. Add the 2 % concentrated sample to the one-piece specimen cup until a thickness of 1.27 ± 0.13 mm (0.050 ± 0.005 in.) is obtained.

10.3 *Preparation of Dye, Dye Penetrant, and Emulsifier*—Clean, unsealed, sulfuric acid-anodized 6061 T 6 aluminum alloy disks, 17.5 mm (11⁄16 in.) in diameter by 1.60 mm (0.063 in.) thick are used as a carrier. Before use, vapor-degrease the disks in trichloroethylene or trichlorotrifluoroethane. To ascertain the effectiveness of the cleaning procedure, test a minimum of 20 blank disks. After cleaning and blank testing, dip the anodized disks in the test dye, dye penetrant, or emulsifier for 15 min and drain for 15 min at 90° horizontal in a test rack as shown in Fig. 11. Then place the disks on the stainless steel inserts which are located in the one-piece specimen cup.

10.4 *Preparation of Liquid Sample Residues*—Place 5 mL of the concentrated sample (obtained as is described in 10.2) in the one-piece cups and heat in an oven at 5°C below the boiling point. Blow filtered air over the samples and remove the vapors by vacuum. A detailed description of the apparatus and the procedure is presented in Annex A1, Method B.

10.5 *Preparation of Semisolids*—Press a sufficient amount of sample material (a slight excess) in the specimen cup (Fig. 6 or 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 edge of the specimen cup until a smooth surface is obtained. It is necessary to fill the depression of the specimen cup uniformly. Assemble the two-piece specimen cup by slipping the PTFE sleeve onto the filled specimen cup. Place the special insert cup inside the one-piece specimen cup.

10.6 *Preparation of Solids*—Cut and prepare samples of solid material as follows: cut the sample to a diameter of 17.5 to 19.1 mm (0.69 to 0.75 in.). Measure the thickness of the specimen with a micrometer and record it. Test sheet material not available in 1.27-mm (0.050-in.) thickness in the thickness intended for use when that thickness is not more than 6.35 mm (0.250 in.). Cut materials normally used in greater thickness to give disks of 1.27 ± 0.13 mm (0.050 ± 0.005 in.) and 6.35 ± 0.13 mm (0.250 ± 0.005 in.) and test at both thicknesses. The specimens may be cleaned by rinsing with a solvent insensitive to liquid oxygen and compatible with the test material at test condition, detergent rinsed (see 6.5), distilled-water rinsed, and dried using a gaseous nitrogen purge, unless otherwise specified. Place the solid material specimens in the one-piece specimen cup. Test solid materials that may not be prepared in the above manner, such as O-rings, and so forth, in the available configuration until such time the test results of the sheet stock and the available configuration give comparable results. Handle powders by spreading a layer of the material as uniformly as possible over the bottom of the one-piece specimen cup to a thickness of 0.050 in. The use of the one-piece insert cup is optional. Precool the sample and cup, but do not add the liquid oxygen to the cup until the striker pin is in place on the top of the sample within the anvil region assembly (Fig. 3).

10.7 *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 to 0.64 mm (20 to 25 mils) thick. Punch disks of 17.5 mm (11⁄16 in.) in diameter from the sheet. Clean the disks by detergent washing, water rinsing, drying, and vapor degreasing in trichloroethylene or trichlorotrifluoroethane. Test the sample by placing it on a stainless steel insert which is located on the bottom of a one-piece cup.

10.8 *Preparation of Coating*—Prepare dry film lubricant and paint specimens for testing as follows: apply the coatings to the stainless steel disks (5.1.9), unless other base metal is specified, in the same manner and to the same thickness recommended. Upon completion of the curing process of the coating, place the disk inside the one-piece specimen cup.

10.9 *Precooling Procedure*—Use the freezing box shown in Fig. 8 taking steps to ensure that it is level. Place approximately 40 specimen cups over the holes in the retainer plate.

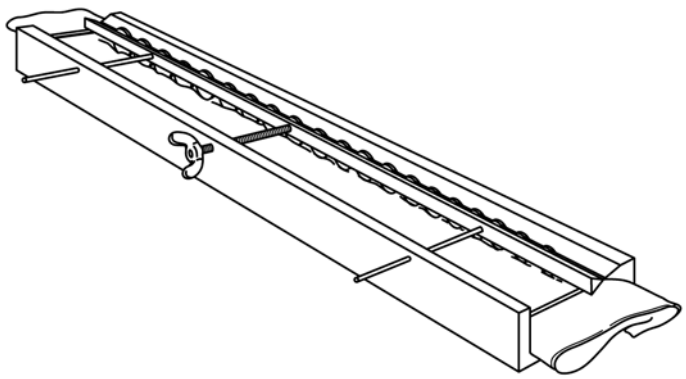


FIG. 11 Test Rack for Dye Penetrant

Using an automatic discharge on a 25-L Dewar flask, pressurize the flask to 17.24 kPa gage (2.5 psig) with the same gas that is to be used in the liquid phase for cooling. Either oxygen or nitrogen may be used for this purpose. Maintaining the pressure at 2.5 psig, open the discharge valve approximately half way, based on experience with the specific unit to be used, and start to fill the freezing box. 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. After this step, make a careful inspection. Reject any specimens showing voids or holes. Check to see that sample material has not separated from the bottom of the cup. Cracking of samples shall not be cause for rejection. Storage at this stage of the procedure shall not exceed 4 h. Check the thickness of four liquid samples after they are frozen and record.

10.10 *Drop Test*—Adjust the magnet located on the fourth rail of the impact tester to the height to be used. Immerse the cleaned striker pins that are stored in the holder in a Dewar flask containing liquid oxygen or nitrogen. Cool the specimen cup holder and the anvil region by pouring liquid oxygen or liquid nitrogen from a transfer container. Place the precooled specimen cup containing liquid oxygen in the specimen cup holder. Place a chilled striker pin in the specimen cup and secure the guide plate in place. Top the specimen with liquid oxygen to assure that there is an excess quantity. The operator shall leave the test cell and close the test cell door. All illumination in the cell and observation area shall be eliminated. Release the safety catch by a switch located on the electrical control panel. Release the plummet. Conduct four blank tests at each test drop height giving an equivalent to one blank for every five test drops.

10.11 *Determination of Approximate Threshold Value*—The approximate threshold value shall be obtained as follows: Perform 20 successive drop tests at a test height of 1.1 m (43.3 in.). If a reaction occurs, continue the 20 drop tests at height levels as indicated in the drop height schedule in Table 1. The first height at which no reaction is obtained in 20 drops is the approximate threshold value. When testing samples that are sometimes capable of initiating false reactions with the aluminum cups, use stainless steel disks as false bottoms in the cups. If there is no reaction at 43.3 in., adjust the height level to a maximum height of 1.2 m (48 in.).

10.12 *Determination of Definitive Threshold Value*—Determine the definitive threshold value by 20 drop tests at height levels of 76-mm (3-in.) increments, starting at a height 152-mm (6 in.) above the approximate threshold value. Perform the drop tests until two series of 20 drops are conducted at consecutive levels without a reaction. The definitive threshold is the potential energy level for the higher of the two highest adjacent heights at which no reaction was obtained in 20 drops, and below which level no reaction occurred.

10.13 *Pass-Fail Test*—The following steps shall be accomplished with sufficient care and dispatch so that the specimen cup will be full of liquid oxygen at all times before and during impact.

10.13.1 Adjust the magnet to 1.1 m (43.3 in.) or as specified.

10.13.2 Use clean tongs to set the precooled specimen cups into the specimen cup holder.

10.13.3 Make visual check to assure that the frozen sample has not separated from the cup bottom. Discard samples that have separated.

10.13.4 Place the striker pin in the cup, and hold in position by the striker pin guide.

10.13.5 Add liquid oxygen to the specimen cup to ensure that the specimen cup is full.

10.13.6 Cover the exposed container of the liquid oxygen.

10.13.7 Close the test cell door. Darken the cell light and set the timer.

10.13.8 Release the safety catch and the plummet by means of the control panel located outside of the test cell near the observation window.

10.13.9 Observe and document the drop time of the plummet for each drop and the results of any reaction.

10.14 *Data Collection*—The following data shall be collected and recorded: It shall be determined whether or not a positive reaction was obtained. The description shall be reported as (*1*) none, (*2*) flash, (*3*) audible report, (*4*) sustained burning, (*5*) obvious char, and (*6*) material deformation as a result of a reaction (cups and pins). The drop height of the plummet shall be determined and recorded. The drop time shall also be determined after every drop and recorded. The data sheet should also include sample size and number of blank cups tested. Fig. 12 illustrates a typical data sheet used for recording the test data.

11. Report

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

11.1.1 Type of material and composition (if known),

11.1.2 Sample size, thickness or volume,

11.1.3 Test type:

11.1.3.1 Definitive threshold value, or

11.1.3.2 Pass or fail, including description of reaction if applicable

11.1.4 Any other pertinent remarks.

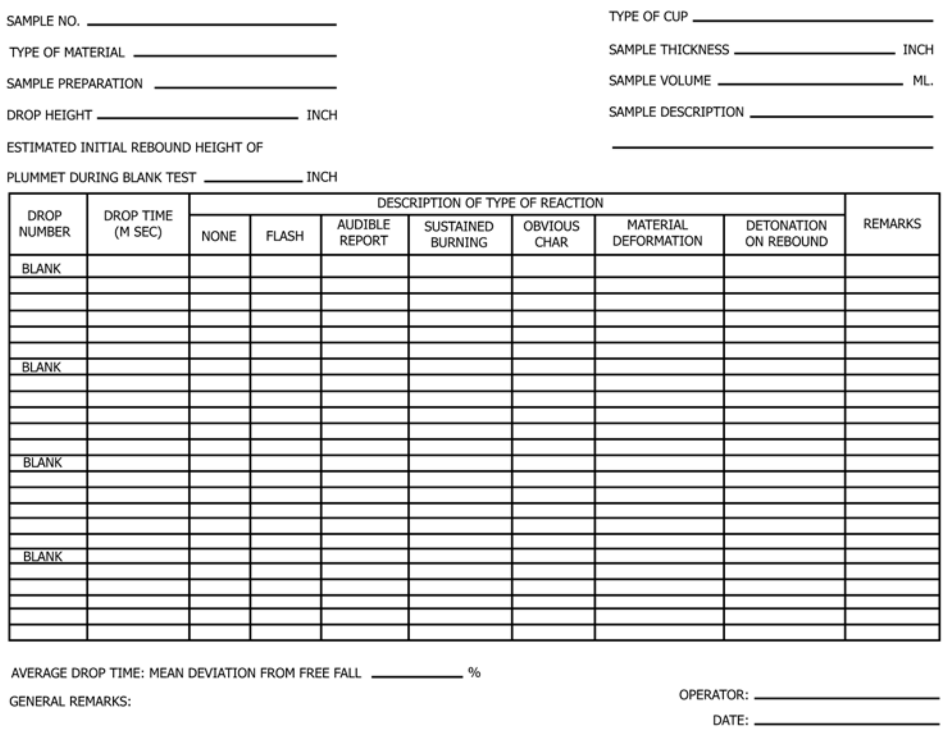
12. Precision and Bias

12.1 A statement of precision, based on limited data, is expressed in Table 2. Two results should be considered suspect if they differ by more than the amounts stated.

12.2 No bias statement is possible as no standard materials are available that are traceable to the National Bureau of Standards.

13. Keywords

13.1 compatibility of materials; impact sensitivity threshold; liquid oxygen; pass-fail techniques


SAMPLE NO. ____
TYPE OF MATERIAL ____
SAMPLE PREPARATION ____
DROP HEIGHT ____ INCH
ESTIMATED INITIAL REBOUND HEIGHT OF PLUMMET DURING BLANK TEST ____ INCH
TYPE OF CUP ____
SAMPLE THICKNESS ____ INCH
SAMPLE VOLUME ____ ML.
SAMPLE DESCRIPTION ____

DROP NUMBER	DROP TIME (M SEC)	DESCRIPTION OF TYPE OF REACTION							REMARKS
		NONE	FLASH	AUDIBLE REPORT	SUSTAINED BURNING	OBVIOUS CHAR	MATERIAL DEFORMATION	DETONATION ON REBOUND	
BLANK									
BLANK									
BLANK									
BLANK									

AVERAGE DROP TIME: MEAN DEVIATION FROM FREE FALL ____ %
GENERAL REMARKS:
OPERATOR: ____
DATE: ____

FIG. 12 ASTM Liquid Oxygen Impact Sensitivity Test Method Data Sheet

TABLE 2 Precision Data

Threshold Value Drop Height, mm (in.)	Repeatability, One Operator and Apparatus	Reproducibility, Different Operators and Apparatus
610 (24)	17.0	40.3
406 (15)	10.6	25.2
152 (6)	4.3	10.1

ANNEX

(Mandatory Information)

A1. PREPARATION OF LIQUID SAMPLE CONCENTRATES AND RESIDUES FOR LIQUID OXYGEN SENSITIVITY TEST METHOD FOR DETERMINING THRESHOLD VALUE OR PASS-FAIL RESULTS

METHOD A

A1.1 Summary of Method

A1.1.1 The liquid sample is evaporated without agitation in a clean closed system at a temperature no greater than 5°C below the boiling point of the sample. The sample is concentrated by evaporating it in a large round-bottom flask placed in a constant-temperature water bath. Air is passed over the surface of the sample at a rapid rate.

A1.2. Apparatus (Fig. A1.1)

A1.2.1 *Water Bath*—A cylindrical borosilicate glass jar (400 mm in diameter by 300 mm high), filled with water within 63 mm from the top.

A1.2.2 *Heaters,* for heating the water bath, and also as follows:

A1.2.2.1 *Immersion Heater,* 1000 W,

A1.2.2.2 *Fixed Immersion Heaters,* 300 and 285 W, and

A1.2.2.3 *Intermittent Heater,* 500 W.

A1.2.3 *Temperature Regulation System,* consisting of a 500-W heater operating through an intermittent thermoregulator relay and a microset thermoregulator.

A1.2.4 *Air Supply*—The air regulated to 15 psig (103 kPa[2] gage) maximum shall be passed through an air purifier and flow equalizer, a drying tube filled with glass wool-cotton-glass wool, and a preheater coil to be placed in the water bath. The preheater coil shall consist of 17 to 20 turns of 6.3-mm clean copper tubing, and the coil shall be about 75 mm in diameter.

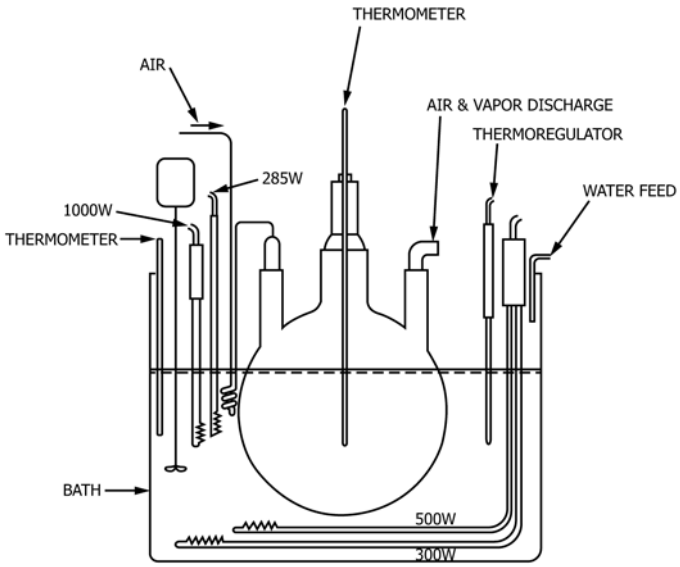


FIG. A1.1 Apparatus for Concentrating Liquid Samples by Method A

The air supply shall be connected to the evaporation flask by means of a glass adapter (Fig. A1.2). This device permits splitting the air flow to the top of the flask and above the surface of the liquid. As the evaporation progresses and the level of the liquid becomes lower, the adapter shall be replaced by one with a longer delivery tube. The flow rate of the air shall be regulated at 25 to 30 L/min.

A1.2.5 *Sample Container*—The sample shall be evaporated and concentrated in a 5-L, three-neck, round-bottom borosilicate glass flask with center neck standard taper 45/50 and side neck standard taper 24/40 joints. The flask shall be marked at the 120- and 140-mL levels, respectively.

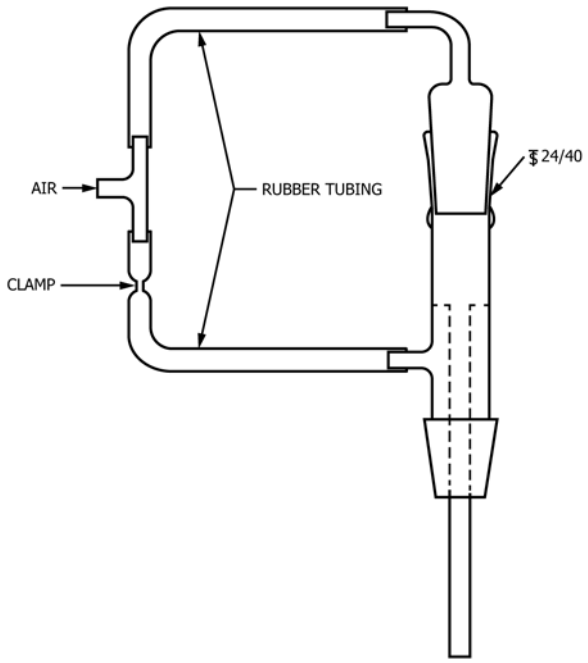


FIG. A1.2 Glass Adapter

A1.2.6 *Condenser*—The vapors shall be partially condensed in a wide-bore water-cooled condenser connected to a 3-L receiver. The effluent of the receiver flask shall be directed to a hood. The water outlet from the condenser serves to provide makeup water for that lost by evaporation from the water bath. The flow rate shall be regulated to just exceed this loss. A constant-leveling device prevents overflow.

A1.2.7 *Stirrer,* heavy-duty, of variable speed.

A1.2.8 *Thermometers,* having 0.1°C divisions.

A1.3. Procedure

A1.3.1 Rinse a clean, dry, 500-mL polyethylene squeeze-type wash bottle three times with the test sample. Add about 300 mL of the test sample to this wash bottle. Rinse a 5-L graduate and a 5-L, 3-neck flask with three 50-mL portions of the test sample. Add3 L of the test sample (using the prerinsed graduate) to the 5-L flask.

A1.3.2 Place the flask in the water bath and connect the air supply. Place the thermometer in the bath and connect the condenser.

A1.3.3 Start the stirrer. Connect the immersion heaters, thermoregulator, and relay to a 110-V ac power supply. Control the temperature of the water by adjusting the thermoregulator.

A1.3.4 When the level of the liquid has dropped sufficiently, replace the adapter (Fig. A1.2) with one having a longer delivery tube. When the level of the liquid drops to 120 mL, recharge the flask with an additional 3 L of test sample. Continue the evaporation until 120 mL of test sample concentrate remain.

METHOD B

A1.4 Summary of Method

A1.4.1 The test sample concentrate derived from Method A is placed in a one-piece cup and heated in an oven at 5°C below the boiling point of the test sample. Filtered air if blown over the samples, and the vapor is removed by vacuum.

A1.5 Apparatus (Fig. A1.3)

A1.5.1 *Oven*—A small laboratory oven capable of regulating temperature to ±0.5°C.

A1.5.2 *Air Supply*—See A1.2.4.

A1.5.3 *Vacuum*—A water aspirator is adequate and a trap is required between the aspirator and the oven.

A1.6. Procedure

A1.6.1 Add 5-mL portions of the concentrated test sample obtained from Method A to the one-piece specimen cups.

A1.6.2 Place the cups containing the concentrated sample in the oven which has been preheated to 5°C below the boiling point of the test sample.

A1.6.3 Turn on the air supply and allow the water aspirator to operate. Position the funnel over the one-piece cups and set the air line under the funnel (Fig. A1.3).

A1.6.4 Allow the system to operate undisturbed for 2 h. When all traces of liquid sample have disappeared, remove the

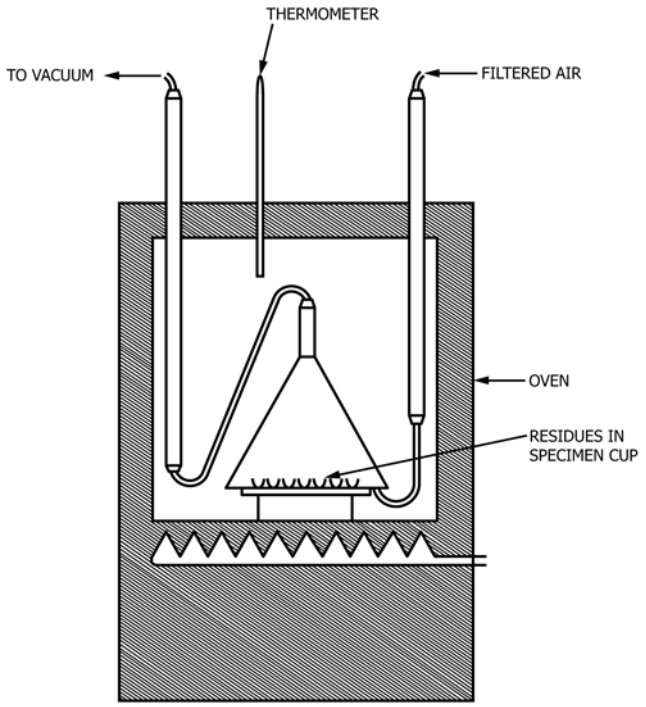


FIG. A1.3 Apparatus for Preparing Liquid Sample Residues by Method B

one-piece cups from the oven and store in a glass vessel.