Designation: D 2389 – 83 (Reapproved 1994)

Standard Test Method for Minimum Pressure for Vapor Phase Ignition of Monopropellents[1]

This standard is issued under the fixed designation D 2389; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method[2] covers the determination of the minimum pressure at which a monopropellant ignites in the vapor phase.

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.*

2. Summary of Test Method

2.1 The minimum pressure for vapor-phase ignition is a limiting measure of what is conceived to be a fundamental monopropellant property, the minimum ignition energy. The minimum pressure for vapor-phase ignition is that pressure below which it is impossible to ignite a monopropellant vapor with a fixed quantity of energy applied in a well-defined manner. It is expected that, by employing greater quantities of energy or applying them in somewhat different fashions, ignition may be obtained at lower pressures. However, the quantity obtained using the procedure described in Section 5 gives useful relative values which are for most practical purposes a minimum pressure for vapor-phase ignition. The principal advantage of this test is the small quantity (only a few millilitres) of sample required, the simple apparatus in which the experiment can be performed, and the versatility of the apparatus. (However, it is important to realize the limitations set on interpreting such information.)

3. Significance and Use

3.1 In vapor-air mixtures the minimum spark-ignition energy has been very helpful in evaluating fuels, both for performance and for handling characteristics. The technique reported herein is useful for evaluating a similar characteristic for monopropellants in the vapor phase. For monopropellants that ignite easily at normal pressure, that is, very close to 1 atm, the usual minimum spark-ignition energy techniques can be employed. It has been found, however, that most useful monopropellants will not ignite in the vapor phase at a pressure of 1 atm. At the higher pressures necessary to obtain ignition, experimental difficulties are experienced with electric spark. For example, the high voltages required to jump the spark gap are difficult to handle in this type of system.

3.2 A technique has, therefore, been partially developed to determine the minimum pressure for vapor-phase ignition. This technique involves the electrical fusion of small wires. In practice the experimental evaluation of energy is somewhat difficult; however, a useful quantity, the minimum pressure at which ignition can be obtained with a fixed energy, can be readily determined. The significance of this quantity can be better understood by reference to Fig. 1 where the minimum spark-ignition energy for *n*-pentane-air is plotted as a function of the total pressure. This curve, which is representative of all the air-fuel mixtures studied, is employed because no comparable data are available for monopropellants. The monopropellants already studied behave in the same manner. For acetylene at 100°C and 0.07 J, a minimum pressure of 3.5 atm was obtained in a small 38-mm diameter cylindrical bomb, and 2.2 atm in a larger 76-mm diameter spherical bomb. Instead of obtaining a curve similar to that shown in Fig. 1, which would be desirable, only one point was obtained, namely the pressure at a fixed ignition energy. The temperature is always a variable and must be specified.

4. Apparatus

4.1 Fig. 2 shows a schematic drawing of the apparatus used for determining the ignition pressure limits of vapors by fused wires (Note 1) at any temperature from 25 to 260 °C. The apparatus shall consist of: (*1*) a thermostat-equipped stainless-steel bomb into which the monopropellant vapor is placed, (*2*)

[1] This test method is under the jurisdiction of ASTM Committee F-7 on Aerospace Industry Methods and is the direct responsibility of Subcommittee F07.02 on Propellant Technology.

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[2] This method is identical in substance with the JAANAF method "Minimum Pressure for Vapor Phase Ignition," Test No. 2, Liquid Propellant Test Methods, December 1959, and published by the Chemical Propulsion Information Agency, Johns Hopkins University, Applied Physics Laboratory, Johns Hopkins Rd., Laurel, MD 20707.

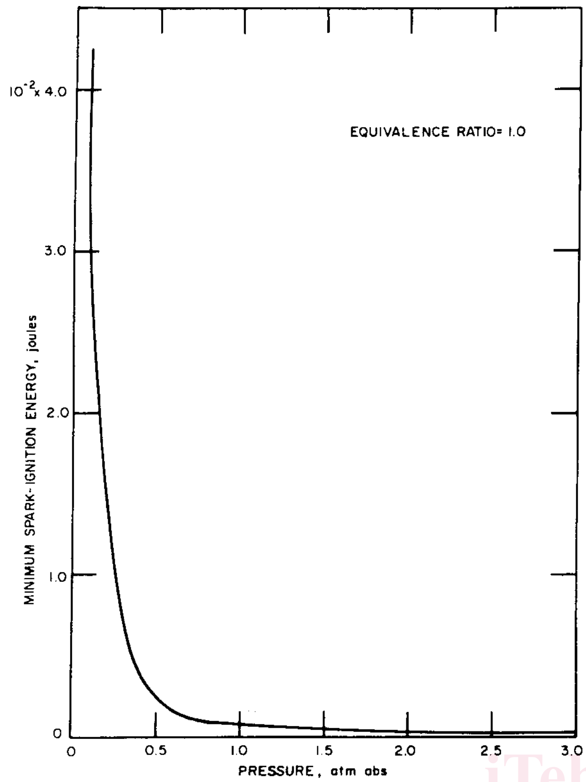


FIG. 1 Effect of Pressure on Minimum Spark-Ignition Energy of Pentane

a means of holding the small wires, (*3*) a means of removing carbonaceous material formed in explosive decomposition (many of the substances which have been evaluated by this technique have been acetylenic compounds which give large quantities of carbonaceous residue), and (*4*) an electric-current supply. In the present apparatus, the wire-fusion time is determined by the voltage applied to the wire and the characteristics of the fuse wire.

NOTE 1—All measurements have been made with Nichrome V wires, 3 mm long. A diameter of 0.0635 mm (2.5 mil) was found to be most satisfactory. Smaller wires (0.024 mm in diameter) gave inconsistent results, and larger wires (0.10 mm in diameter) caused welding of the wire ends to the wire clamps.

4.1.1 *Bomb*[3]—The ignition bomb (Figs. 3 and 4) shall be made of Type 316 stainless steel having an outside diameter of 4⅛ in. (104.8 mm) and an inside diameter of 1½ in. (38.1 mm). The outside height shall be 4 in. (102 mm) and the inside chamber height with liner removed shall be 1⅝in (41.3 mm). Drill a ½-in. (12.7-mm) observation hole into the bottom of the bomb. This hole shall be sealed with a ¼ by 1⅛-in. (6.4 by 28.6-mm) outside diameter borosilicate glass window, polytetrafluoroethylene (PTFE) gaskets, and a brass lock ring. There shall be three ⅛-in. gas inlet valves with PTFE packing. The fourth outlet shall be connected to the pressure transmitter (Fig. 5). The bomb shall be designed with an internal volume of 25 cm^3 (liner in) and 45 cm^3 (liner out) to permit testing small quantities of fuel. The over-all internal volume, including leads and pressure transmitter (Fig. 5 and Fig. 6) shall be 98 cm^3. The stainless steel removable liner also permits sampling and weighing of residual solids left after an explosion. The bomb shall be electrically heated and capable of being regulated from ambient temperature to 260°C by a thermoswitch. In the temperature range from 25 to 260°C, sufficiently high pressures can be obtained with most of the higher-molecular-weight compounds to determine the minimum pressures for vapor-phase ignition. The sample shall be introduced in the gas phase from a conventionally heated manifold for handling organic vapors; fuels with less than adequate vapor pressure shall be introduced as liquids by means of a syringe-type injector through hypodermic tubing, the tubing size being determined by the physical properties of the liquid fuel.

4.1.2 *Bomb Head and Liner*—The bomb head (Fig. 4) shall be made of Type 316 stainless steel with an outside diameter of 1⅞ in. (47.6 mm) at the top and a depth of 15⁄16in. (23.8 mm) not including the wire holder posts. The wire holders (Note 2) shall be made of Type 316 stainless steel tubing, the outer tube having a ⅛-in. (3.2-mm) outside diameter, the inner tube, a 3⁄32-in. (2.4-mm) outside diameter. The outer tube shall be slotted to permit movement of the inner tube by means of a small stainless steel guide rod, which is driven through the inner tube. A stainless steel piano wire spring shall provide the necessary tension to hold the fuse wire. Since good electrical contact is essential, it is advisable to have the tension on the spring as great as possible without breaking the fuse wire. This shall be determined by screwing an extra long spring on the holder post and backing off until it no longer breaks the fuse wire. The positive wire terminal shall be connected to the external electrical circuit by means of an insulating terminal which is insulated further by PTFE gaskets. The hypodermic tubing shall be brought through the bomb head and sealed with a PTFE gasket by means of a brass packing nut. The bomb liner (or cup) and the bomb head shall be threaded for easy attachment. The bomb head shall be provided with a handle to facilitate removing from the bomb (Fig. 7). A brass locking ring, equipped with lugs, shall seal the bomb head to the bomb. A 1⁄16-in. (1.6-mm) PTFE gasket shall be used as the sealing agent. A wrench shall be used to secure the bomb head to the bomb.

NOTE 2—The wire holders shall be spring-loaded, and the wire threaded through small holes in each post. The spring holds a plunger firmly against the wire so that good electrical contact is made. It becomes necessary when carbonaceous products are formed to clean the holes and the plunger face each time a new wire is threaded.

4.1.3 *Spool, Head, and Cup Holders*—The wire spool holder, head holder, and cup holder (Fig. 8) are not necessary, but they are convenient tools for speeding up the operation. The spool holder shall consist simply of a ½-in. (12.7-mm) aluminum rod on which the spool can freely roll. The fuse wire shall be threaded through two pieces of foam rubber mounted on a ½-in. (12.7-mm) rod to keep the wire from unwinding. The thermostated cup holder (Fig. 8) shall retain the cup for easy assembly and maintain the cup at the operating temperature. The head holder shall provide a means for keeping the

[3] Detailed drawings of the ignition bomb are available from the American Institute of Aeronautics and Astronautics, 1290 Avenue of the Americas, New York, N.Y. 10019. Request Supplement I of American Rocket Society Recommended Test No. 2.

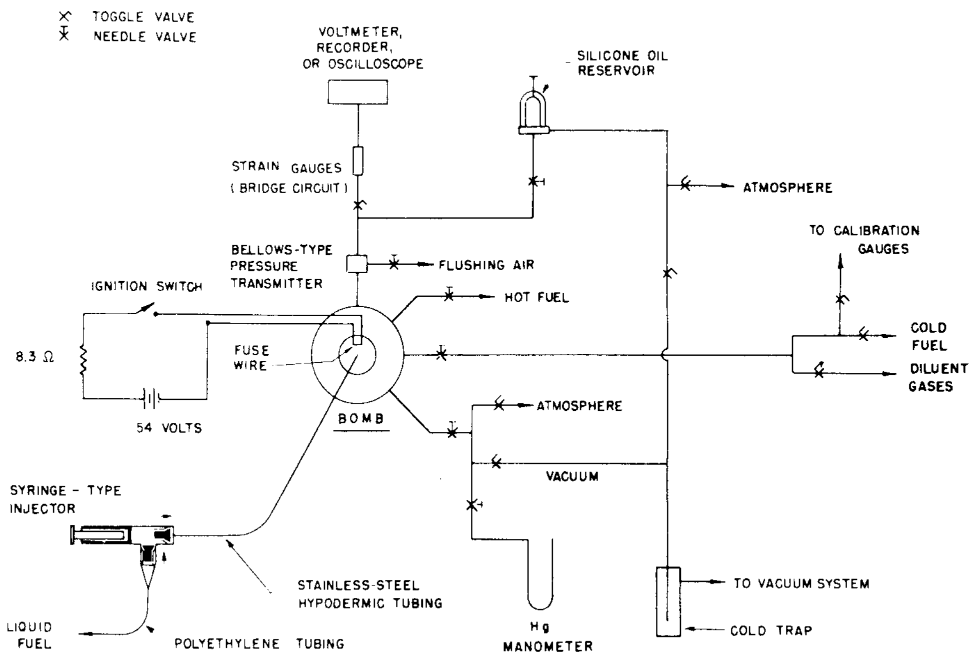


FIG. 2 Fuse-Wire-Ignition Apparatus

head at the operating temperature and shall hold the head for easy threading of the wire.

4.1.4 *Pressure-Measuring System*—The pressure-measuring system shall include a bellows-type pressure transmitter (Fig. 5 and Fig. 6) used to isolate the pressure gages from the temperature of the bomb, a means for flushing the transmitter, four pressure transducer strain gages covering the range from 0 to 1000 psia (0 to 6.9 MN/m^2 absolute), a voltmeter, a silicone oil reservoir, approximately ½ pt (235 cm^3) of silicone oil, a regulation transformer, and the necessary resistors to provide regulation of the input to the gages. The control circuit diagram is shown in Fig. 9. Fig. 2 shows the position of each component in the pressure-measuring system. It is imperative that all points in the hydraulic system be below the oil reservoir so that there will be no air bubbles trapped in the lines. The system must be evacuated before filling with oil in order to give consistent pressure measurements. Frequent calibration of the strain gages is necessary because it is suspected that the silicone oil jells after prolonged exposure to the high temperatures in the bellows assembly, thus causing an expansion in the hydraulic system.

4.1.5 *Liquid-Fuel Injector*—The liquid-fuel-injection system shall consist of a continuous pipetting outfit with interchangeable syringes of ½, 1, and 2.0-cm^3 capacities. The rubber check valves have been replaced with similar valves of stainless steel using 1⁄16-in. (1.6-mm) O-rings (compatible with propellant) for sealing. (The rubber valves proved to be unsatisfactory because of swelling and cracking and because of their inability to withstand the high pressures generated in the bomb and those necessary to inject the liquid fuels.) The rubber tubing that is supplied with the pipetting outfit has been replaced with polyethylene tubing for compatibility. The syringe injector shall be connected to the bomb with stainless steel hypodermic tubing, the size of the tubing being determined by the physical properties of the liquid fuel under test. Many fuels tend to polymerize in the tube at the point of contact with the bomb.

4.1.5.1 Rather than attempt to clean the tip of the injector tube, it is more expedient to pull the tube through and break off the end after first nicking it with a file. The tubing shall be made longer than the dimensions of the bomb dictate. It is always advisable to check the tubing before reassembling the bomb head to make sure that the tubing is unrestricted. If the flow is partially restricted, one or two shots will plug the tubing again. Another trouble spot is the valve seating spring on the injector. The tension on this spring must be slightly greater than that supplied in the pipetting outfit in order to seal the bomb during evacuation. With fuels that have low boiling points (less than 75°C) it is imperative that the injector be placed far enough away from the bomb assembly so that heat from the bomb does not vaporize the fuel in the injector assembly. Bubbles in the syringe, valve, or tubing between the fuel reservoir and injector will cause the injector to become inoperative.

4.1.6 *Ignition System*—The ignition system shall consist of a 0 to 10-Ω, ten-turn potentiometer-rheostat, a heavy-duty electrical switch, nine 6-V batteries connected to a rotary tap switch to enable one to obtain the desired voltage, the necessary electrical leads, and the wire holders. The positive terminal shall be connected by means of a banana plug soldered to the fusinite terminal. The ground terminal shall be connected to the bomb head handle by means of a heavy-duty battery clip.

4.1.7 *Vacuum System*—A glass cold trap (dry ice and acetone bath) shall be located in the vacuum line leading to the vacuum pump. A mercury manometer shall be used to check evacuation of the system, to check the system for leaks, and to calibrate the pressure gages.

4.1.8 *Temperature Controls*—There shall be five separate electrical heating units (Nichrome resistance wire), each

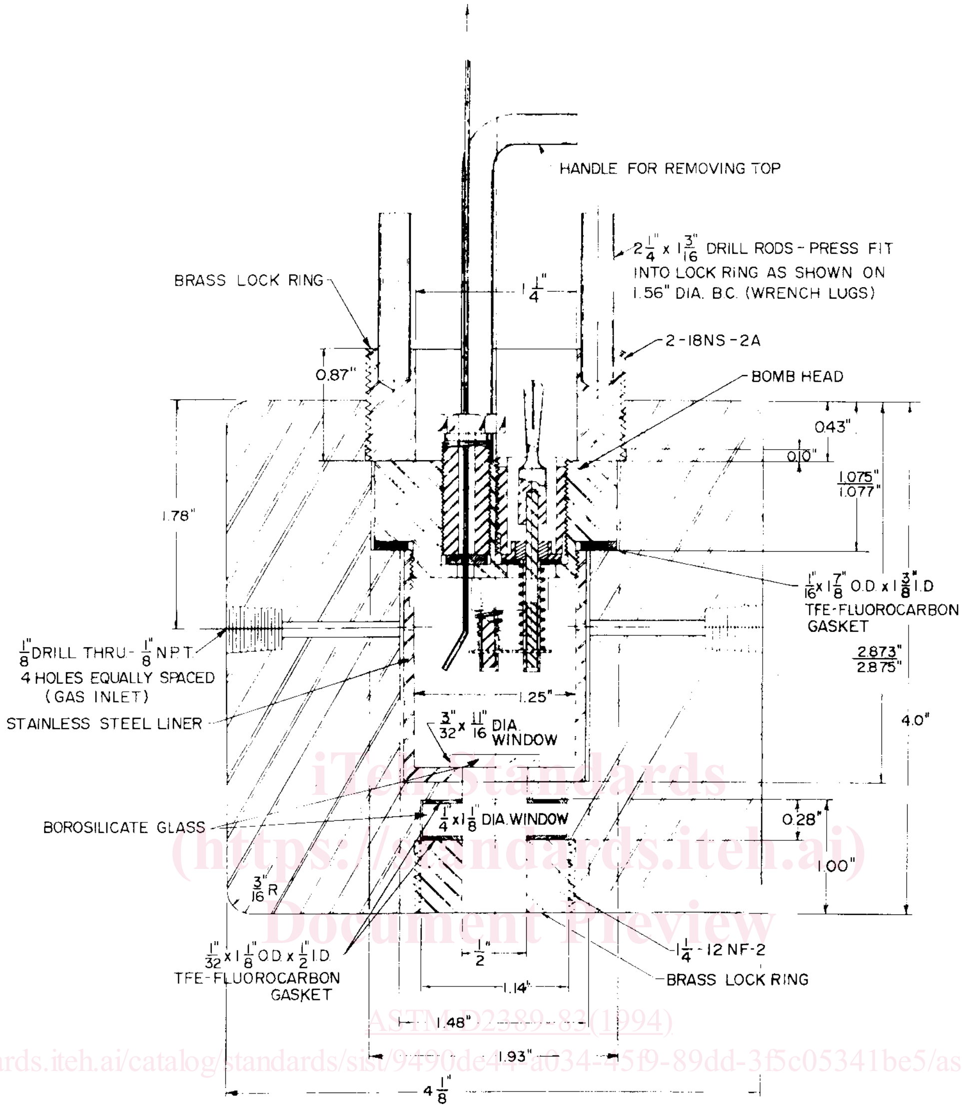


FIG. 3 Stainless Steel Ignition Bomb

equipped with a thermoswitch, a variable transformer (7½ A) to enable gradual heating (thus avoiding heater burnout) a pilot light and fuse assembly, and an on-off switch. The heaters shall be located on the ignition bomb, the valves and pressure transmitter, the hot fuel line, the heat and cup holders, and the hot oil bath. The ignition bomb heater shall be a 500-W, 110-V heater. It shall consist of 24 ft (7.31 m) of No. 22 B & S gage (0.644-mm) resistance wire. Each heating unit shall be equipped with one or more iron-consultant thermocouples. A microammeter shall be used to indicate temperatures. This unit shall be calibrated periodically. A 1⁄32-in. (0.8-mm)-sheet copper jacket shall cover the exterior surface of the bomb for better heat distribution. The copper jacket shall be covered with a layer of glass insulating tape and the Nichrome wire wound over it. The heater wire shall then be secured with high-temperature cement. A layer of suitable insulating cement shall cover the entire assembly to a depth of approximately ¾ in. (19 mm).

4.1.9 *Valve Manifold and Tubing*—The valve manifold shall consist of ten toggle and eleven needle valves. All valves shall be panel-mounted except four needle valves which are mounted adjacent to the bomb for control and heating purposes. All tubing employed in the system shall be ¼-in. (6.4-mm). Type 316 stainless steel seamless tubing. Approximately 30 ft (9 m) of tubing was used in constructing the system described herein.

5. Procedure

5.1 Determine the minimum pressure for vapor-phase ignition by adjusting the thermoswitches (in all parts of the system in contact with fuel vapors) to the setting necessary to maintain the desired temperature. Thread the fuse wire into the wire holder and secure the bomb head by means of the lock ring. When liquid fuels are being evaluated, connect the hypodermic tubing to the fuel injector. When the bomb assembly and all leads have come to equilibrium temperature, evacuate the bomb. Flush the bomb and leads and evacuate several times with the fuel to be tested, and then evacuate again. Fill the bomb with vapor to a pressure approximating the estimated minimum pressure for vapor-phase ignition, and attempt the

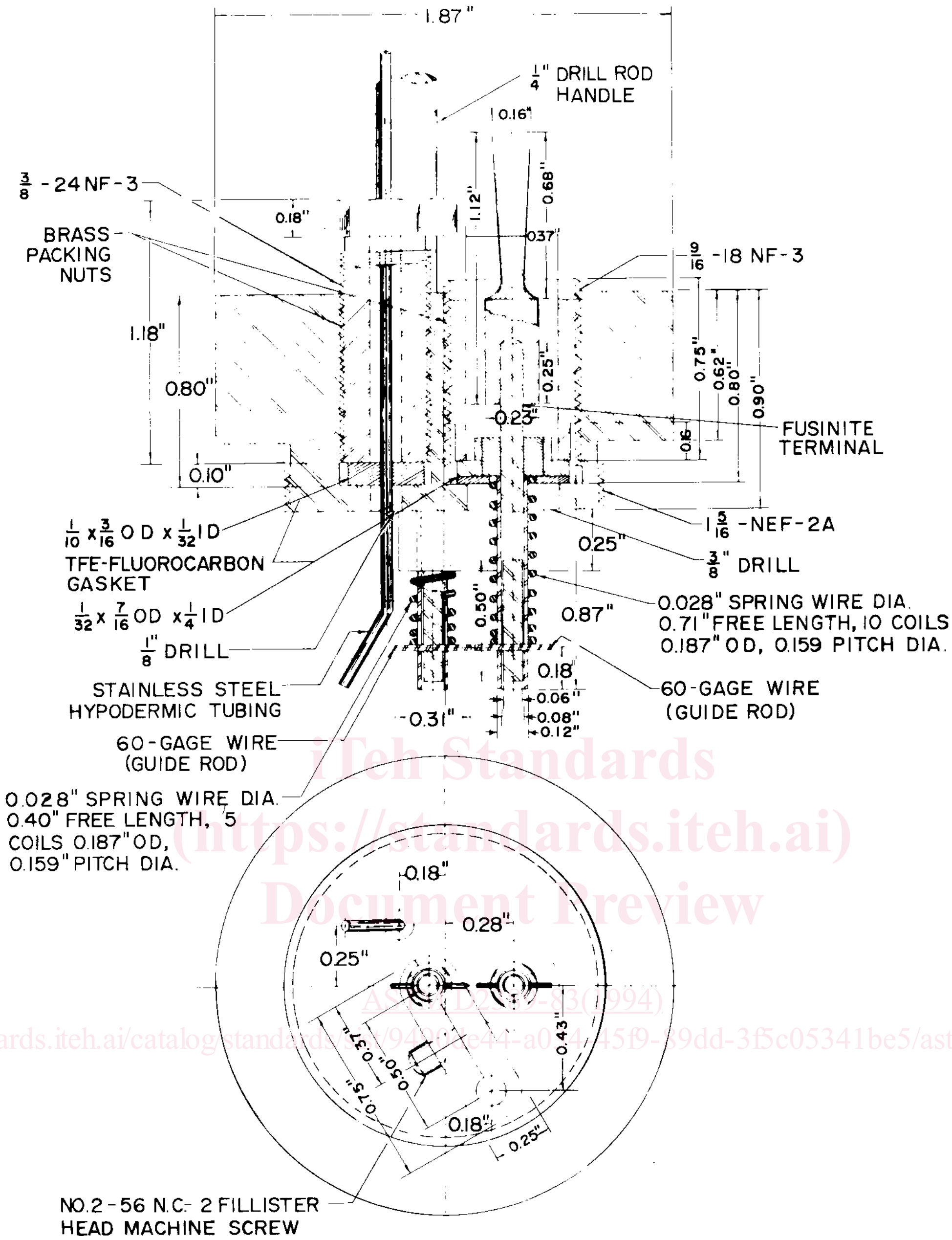
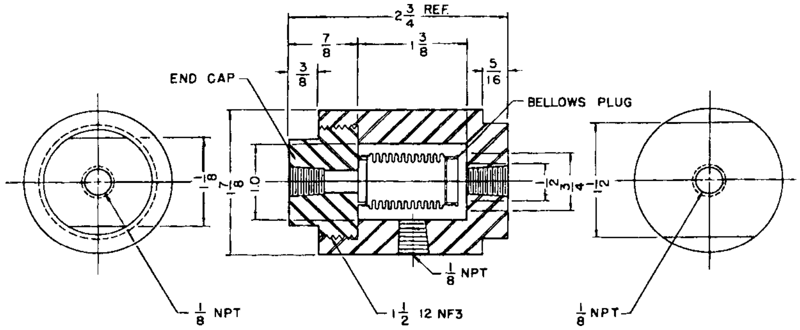


FIG. 4 Diagram of Bomb Head

ignition by fusion of the wire (see Fig. 10). Previously set the voltage and resistance in the igniting circuit to give the desired energy and fusion time (Note 3). If ignition is not obtained, increase the pressure until ignition occurs. In order to ascertain the point accurately, always carry out several additional shots at slightly higher and lower pressures. A judicious choice of test pressures reduces the number of tests. Record the output from the strain gages on a voltmeter (a recording potentiometer or oscilloscope shall be used when the dynamic pressure in the reaction is measured); the input shall be supplied as indicated in Fig. 9.

NOTE 3—An energy value of approximately 0.07 J and 5 ms fusion time can be obtained with 54 V dc supply voltage (nine 6-V "A" batteries) and a fixed resistance of 8.3 Ω. The oscilloscope shown in Fig. 10 is used to record the energy-time relationships involved in fusing the wire. Energy and time have been varied extensively; however, a fixed energy and fusion time are sufficient for purposes of screening monopropellants.

5.2 At the completion of each minimum pressure determination, verify the exact value of pressure by calibrating the pressure transmitter with nitrogen pressure in the bomb and using Bourdon tube gages that have been standardized by means of a dead-weight tester. Also check these gages periodically with a Heise test gage. Use a vacuum cleaner and stiff brush to remove any excess carbon from the bomb. Clean the pressure transmitter by flushing it with a high-pressure air jet. Admit fuels that have vapor pressures above 1.0 atm to the bomb through the "hot fuel" inlet. Contain the fuel in a small stainless-steel cylinder. Raise a hot oil bath so that the fuel tank is immersed in the oil until the required vapor pressure is obtained in the bomb. Lower the oil bath immediately to avoid



NOTE 1—Transmitter is all stainless steel, electrically welded. Use ½ by ¾-in. (12.7 by 19.1-mm) stainless steel bellows with 12 convolutions, one-ply.

FIG. 5 Pressure Transmitter

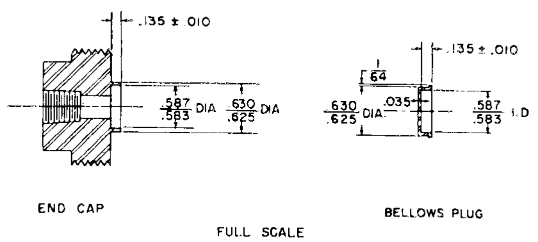


FIG. 6 Pressure Transmitter Details

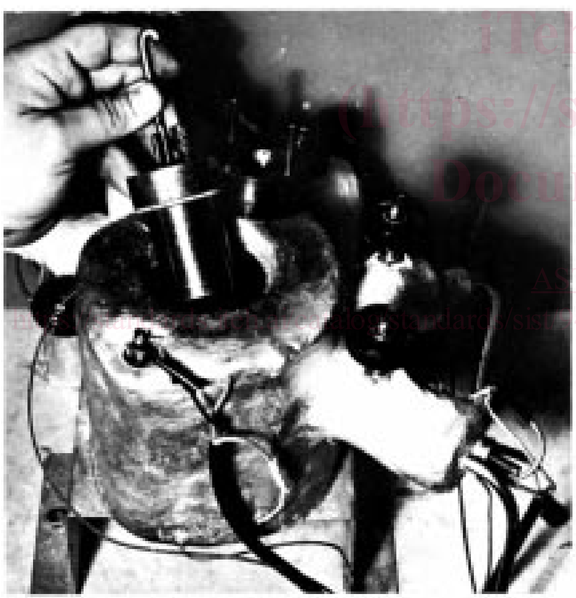


FIG. 7 Illustration of Bomb Head Showing Handle

possible decomposition in the fuel tank.

6. Interpretation of Test Results

NOTE 4—At the present state of development and understanding, the results cannot be interpreted with complete clarity. Large variation in bomb size appears to have an effect (Table 1). The data shown were, obtained in an ignition bomb, at 15 to 17°C, similar to that described in Section 4. Initial conditions were; however, very different. The ignition source in these experiments was a 10-mm length of 0.5-mm-diameter platinum wire with an ignition current sufficient to cause an "instantaneous fusion" of the wire (*H* = approximately 15 J).

6.1 The interpretation of data is complicated by the unusual nature or shape of the curves obtained. If one plots the fuse-wire ignition energy as a function of the pressure, loops are sometimes obtained in the curve which are dependent upon the fusion time. Because of the nature of the electrical system, an increase in energy (applied voltage) decreases the fusion time. As the fusion time increases, heat loss from the wire due to thermoconductivity and convection effects can be expected to become significant. The unusual shape of the ignition energy-pressure curves (Figs. 11 and 12) can probably be explained by a detailed consideration of such effects. It is apparent that before ignition by fusing wires is as well understood as the minimum spark ignition energy process, a considerable amount of basic work will be required. Nevertheless, the process is useful for evaluating monopropellants.

6.2 For comparison of monopropellants, it is necessary to fix arbitrarily some of the experimental conditions. Thus, the fusion time and ignition energy are held constant (Note 5) (approximately 5 ms and 0.07 J), and the minimum ignition pressure determined. The results might thus be looked upon as giving the lower pressure limit at a given energy level and time for the wire to fuse. Data obtained in this bomb and in a larger bomb, although showing different sizes for the loops, gave equivalent relative results in comparing the different fuels. Some typical data are reproduced in Table 2. This table includes not only the value normally considered for the minimum ignition pressure, that is, the value at $t = 5$ ms and $H = 0.07$ J, but also the actual minimum pressure taken from curves such as Figs. 11 and 12. The fusion time and actual energy are indicated. Most work has been attempted at a temperature of 100°C; however, this is frequently impossible as with *n*-propyl nitrate because of the lack of sufficient vapor pressure at 100°C to obtain the minimum pressure.

NOTE 5—In an experiment the approximate fusion time and energy are fixed by setting the applied voltage and resistance. Ignition trials are then made at different pressures while the critical pressures between ignition and nonignition are observed. Occurrence of ignition is indicated by the flash observed through the borosilicate-glass window in the bottom of the bomb by means of a mirror, the bomb being mounted several inches above the table top.

7. Precision and Bias

7.1 Committee F-7 has no immediate plan at this time to develop precision and bias statements for this test method.