



Designation: D 2389 – 83 (Reapproved 1994)

Standard Test Method for Minimum Pressure for Vapor Phase Ignition of Monopropellants¹

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 method² 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)

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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.

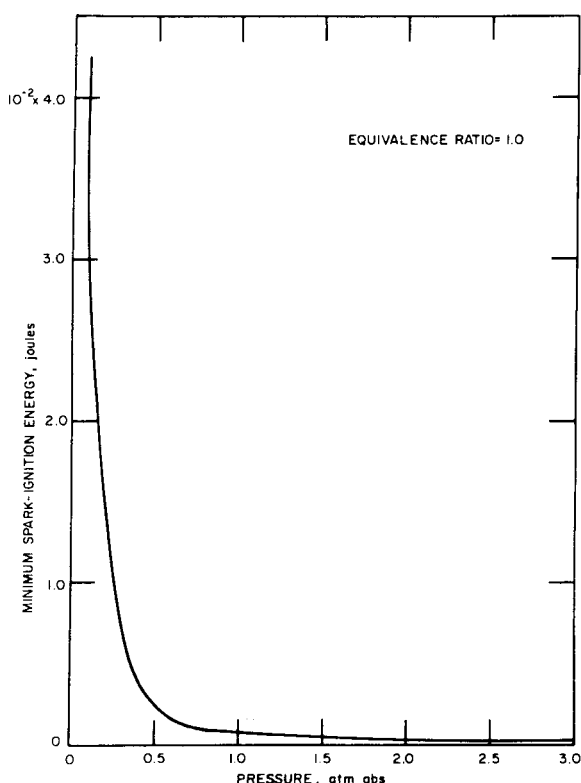


FIG. 1 Effect of Pressure on Minimum Spark-Ignition Energy of Pentane

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 4½ 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 1½ in. (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 1½-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

cm³ (liner in) and 45 cm³ (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 cm³. 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 thermostwitch. 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 1⅝ in. (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 ⅜-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 ¼-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.

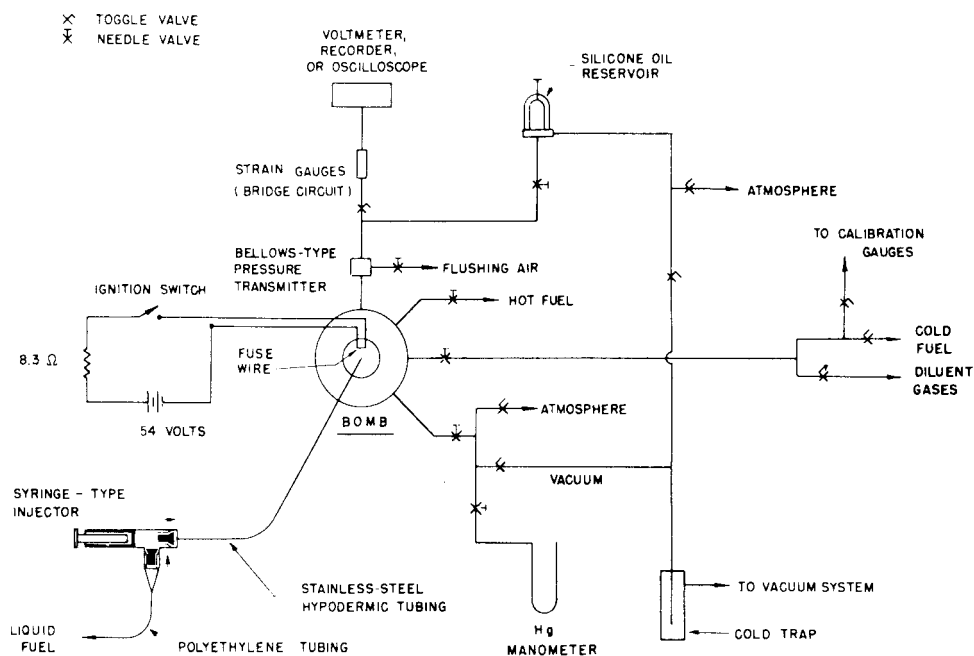


FIG. 2 Fuse-Wire-Ignition Apparatus

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/m² absolute), a voltmeter, a silicone oil reservoir, approximately ½ pt (235 cm³) 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-cm³ capacities. The rubber check valves have been replaced with similar valves of stainless steel using ¼-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