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Standard Test Method for Critical Diameter and Detonation Velocity of Liquid Monopropellants¹

This standard is issued under the fixed designation D 2541; 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 evaluation of two properties of a high-energy liquid propellant. In one form, the critical internal diameter is determined in a given type of metal or plastic tubing below which propagation of stable high-velocity detonation will not take place. In the alternative form, which uses more material, detonation rate is concurrently measured. The composite donor of either size may be used in most instances to initiate detonation in experimental trap designs.
- 1.2 This standard does not purport to address all of the safety problems, 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.
- 1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

2. Terminology

- 2.1 Definition:
- 2.1.1 *critical diameter*—the largest diameter that will not detonate when the donor is exploded.

3. Summary of Test Method

3.1 Various diameters of tubing are filled with propellant, and an attempt is made to cause the propellant to detonate by use of a secondary detonating medium (the donor).

4. Significance and Use

4.1 It should be emphasized that the critical diameter, as determined under these conditions, is valid only for these conditions and is not an intrinsic property of the sample. One vital parameter in establishing the critical diameter is that of confinement of the test specimen. The fact that detonation occurs or does not occur in Type 347 stainless steel tube does

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not necessarily imply that the same results would be obtained in an aluminum, copper, glass, etc., tube of similar dimensions. Type 347 stainless steel tube is acceptable for a standard reference test, but for practical application, diameters should be studied in the materials and wall thicknesses proposed for use.

- 4.2 When working with high-energy liquid propellants, serious consideration shall be given to the possibility that a detonation originating in the engine can propagate upstream to the propellant tank and cause a disastrous explosion. Therefore, it is useful to know the minimum diameter of propellant line through which a detonation of the propellant in question can propagate. If it is impracticable to use propellant lines smaller than this minimum, it will be necessary to design and test detonation traps in larger lines. The minimum or critical diameter (often referred to as "failure" diameter), when the conditions are properly defined, can be a useful measure of the shock sensitivity of similar systems. The detonation velocity of the propellant in question is another property of interest.
- 4.3 The three determinations, namely: minimum diameter for propagation, detonation trap requirements, and detonation velocity, have much in common; all presuppose the initiation of a stable detonation in a liquid contained in a tube. The key to the present test method is the use of a donor stage consisting of the material under test. Although a compound initiator comprised of a blasting cap and high-explosive booster is employed, the true donor is a length of the subject material sufficient to assure establishment of a stable detonation characteristic of the test medium ahead of the first test section or measuring station. Questions of wall and boundary discontinuity are thereby eliminated along with the accompanying complications of impedance mismatch and perturbation of the shock front.

5. Apparatus

- 5.1 The liquid under test, depending on what measurement or measurements are to be made, shall be contained in one of the following three assembled units:
- 5.1.1 Assembly No. 1, Critical Diameter Measurement (Fig. 1 (a)):
- 5.1.1.1 Section A, Fig. 1 (a), shall consist of Type 347 stainless steel tubing (1-in. (254-mm) outside diameter by 0.049-in. (1.24-mm) wall thickness by 6-in. (152-mm) length). When filled with test sample, it is considered the "self donor" section.

² This test method is identical in substance with the JANNAF method," Critical Diameter and Detonation Velocity Test," Test Number 8, Liquid Propellant Test Methods, May 1964, published by the Chemical Propulsion Information Agency, Johns Hopkins University, Applied Physics Laboratory, Johns Hopkins Rd., Laurel, MD 20707.

FIG. 1 Diagram of Apparatus

5.1.1.2 Section C, Fig. 1 (a), shall consist of Type 347 stainless steel tubing (30-in. (762-mm) length) of any one of the following sizes:

Outside Diameter,	Wall Thickness,
in. (mm)	in. (mm)
1 (25.4)	0.049 (1.24)
3/4 (19.0)	0.049 (1.24)
5/8 (15.9)	0.035 (0.89)
1/2 (12.7)	0.035 (0.89)
3/8 (9.5)	0.035 (0.89)
1/4 (6.4)	0.035 (0.89)
1/8 (3.2)	0.020 (0.51)

When filled it is considered the "test" section.

- 5.1.1.3 Section A and Section C is connected by means of a stopper of rubber or other suitable material compatible with the propellant under test. The top of Section C is flush with the top of the stopper.
- 5.1.1.4 The downstream end of Section C is closed by crimping, plugging, or clamping, the latter being shown in Fig. 1 (a) and (c). A pinch clamp over vinyl tubing shall be used in freeing the container, especially one of small diameter, of entrapped air during the filling operation.
- 5.1.2 Assembly No. 2, Detonation Velocity Measurement (Fig. 1 (b)):
- 5.1.2.1 Section D or "test" section, Fig. 1 (b), shall consist of Type 347 stainless steel tubing (1-in. (25.4-mm) outside diameter by 0.049-in. (1.24-mm) wall thickness by 11-in. (279-mm) length). Two timing stations of either ionization wires or T-1 targets (Note 1), 100 mm apart, and located at approximately 5 and 9-in. (127 and 229-mm) levels from the booster end, shall be used for the rate measurements. The probes inserted in the container can be sealed with epoxy

cement or passed through neoprene sleeves, provided either is compatible with the test liquid.

- (a) T-1 targets are pressure-shorting switches encased in a copper tube $\frac{1}{4}$ in. (6.4 mm) in diameter by 1 in. (25.4 mm) long. These switches are inserted through holes in the side of the container. (The same item in an aluminum case bears the designation T-2 target.)
- 5.1.2.2 The downstream end of Section D is closed by crimping or plugging.
- 5.1.2.3 A longer container and more distance between stations, or a greater number of stations is required if greater accuracy in rate measurement is required.
- 5.1.2.4 If the test sample is limited, smaller diameters can be used.
- 5.1.3 Assembly No. 3, Combination Critical Diameter and Detonation Velocity Measurement (Fig. 1(c)):
- 5.1.3.1 Section B, or "self donor" section, Fig. 1(c) (see 5.1.2.1).
 - 5.1.3.2 Section C, or "test" section, Fig. 1(*c*) (see 5.1.1.2).
 - 5.1.3.3 Section C, connection to Section B (see 5.1.1.3).
 - 5.1.3.4 Section C, closure at bottom (see 5.1.1.4).
- 5.1.3.5 Additional timing stations can be positioned along the length of Section C if rates are desired in small-diameter tubes
- 5.1.3.6 The apparatus as described is suitable for determining critical diameters up to 1 in. (25.4 mm) (the donor itself acts as the 1-in. section), but if the minimum diameter for propagation is greater than 1 in., a larger donor shall be used. This donor should be $1\frac{1}{2}$ or 2 in. (38.1 or 50.8 mm) in diameter, as necessary, but otherwise of the same length and

wall thickness (0.049 in.) (1.24 mm) as the standard donor. The diameter of the high-explosive booster and detonator holder shall be scaled up to match, and the constant L/D of 2 shall be maintained. For instance, if the donor is 2 in. in diameter, the booster will be at least 2 in. in diameter by 4 in. (102 mm) long.

- 5.1.4 Assembly No. 4, Trap Testing—In testing detonation traps, the trap to be tested is attached to either Assembly No. 1 or 3 in place of the small-size tubing being tested for critical diameter (Section C). Certain configurations can require filling with liquid before assembly with the donor section. In this event, the precautions under Section 6 shall be observed.
- 5.1.5 *Booster*—The booster charge shall consist of a cylindrical pentolite pellet (or equivalent high oxidizers), nominally $2\frac{1}{2}$ in. (64 mm) long by 1 in. (25.4 mm) in diameter, weighing 51 ± 0.3 g with a density of 1.65 ± 0.01 g/cm³, and containing an axial cavity $\frac{1}{4}$ in. (6.4 mm) in diameter by $\frac{1}{2}$ in. (12.7 mm) deep for insertion of the electric detonator.
- 5.1.5.1 **Warning**—Pentolite is not considered to be a particularly sensitive explosive, but handle with due respect. Careless or rough handling can be fatal. Remembered, too, that practically all high explosives are quite toxic. Handle them with particular care to avoid spreading the material by contact of the hands with other parts of the body. Wash hands with soap and water frequently. Working garments shall be free from dust-collecting features such as trouser-cuffs, and laundered frequently.
- 5.1.6 *Detonator*—Detonation in the booster pellet shall be initiated by an electric blasting cap which fits snugly into the hole in the booster. The cap used with the pentolite booster shall be a No. 8 commercial cap.
- 5.1.6.1 **Warning**—Electric blasting caps contain primary explosives, which are easily initiated by relatively mild physical shock. Consequently, every precaution shall be taken by those who work with them, with particular emphasis on gentle handling and protection from electrostatic charges. Accumulation of static charges by personnel shall be prevented by use of

all-cotton clothing and special conductive shoes.

5.1.7 Rate-Measurement Apparatus—A 10-MHz counter, or an oscilloscope (with suitable camera attachment) with a 5-µs/cm sweep frequency, can be used to measure the time of propagation between the stations (Note 1). The oscilloscope has an advantage in that the trace can give some evidence as to the cause of malfunctions when they occur.

Note 1—It can be desirable to use more than two stations or probes, thus obtaining replicate rate measurements. A circuit diagram for single-oscilloscope rate measurements is given in Fig. 2.

- 5.1.7.1 Time-Interval or Counter-Chronograph Apparatus—The instrument shall be a 10-MHz counter-chronograph (0.1 μ s time base) with a resolution of 0.1 μ s in the range from 0.3 μ s to 1 s. The unit shall have an input sensitivity of 0.2 V rms. The input impedance shall be 1 M Ω , direct or a-c coupled, trigger slopes either positive or negative. Step attenuators shall provide trigger voltage adjustment having a range of ± 1 , ± 10 , and ± 100 V.
- 5.1.7.2 Counter-Chronograph Input Circuitry—Counter-chronographs currently in use require input voltage pulses with relatively fast rise times and moderate amplitudes. Both of these conditions can be met with the simple R-C circuit described in two forms in Figs. 3 and 4. Since most counter-chronographs permit polarity and slope selection of the triggering pulses, it is convenient and frequently desirable to provide maximum pulse isolation by using opposite polarities for "start" and "stop" triggering pulses from adjacent probes. The circuits shown schematically in Figs. 3 and 4 were designed to provide output pulses of opposite polarity when the inputs are "shorted" through ionization probes or T-1 targets. With the supply voltage polarities as shown, the output pulse at J_3 is negative when J_1 is shorted, while the output pulse at J_4 is positive when J_2 is shorted.
- 5.1.7.3 Oscillograph Circuitry—The circuit for the oscillograph is shown in Fig. 5 and the circuit for the power supply is

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All resistors \pm 10 percent, 1 W R_1-2000~\Omega R_2-50~\Omega R_3-1~M\Omega C_1-3000~pf, <math display="inline">\pm 10 percent, 600 V, dc (C_1 may be changed to lengthen or shorten the pulse width) C_2-0.05\mu F, \pm 20 percent, 600 V, dc D-10.05\mu F, \pm 20 percent, 600
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FIG. 3 Bacis R-C Pulse-Forming Circuit

FIG. 4 Practical 2-Channel R-C Pulse-Forming Circuit Producing a Positive Pulse in One Channel and a Negative Pulse in the Other Channel

shown in Fig. 6. With this apparatus, it is necessary to synchronize the circuit, and for this a twisted wire (No. 32 B & S gage (0.202-mm) enameled copper wire is satisfactory) shall be inserted between the pentolite donor and the acceptor.

5.1.8 Firing Chamber—It is necessary to provide protection from high-velocity fragments and some means of recovering the remains, if any, of the acceptor tube. In some instances it is also desirable to reduce noise from the shot. One solution consists in using an all-steel chamber in the shape of a simple maze (Fig. 7). Less elaborate structures have been developed at other laboratories and function satisfactorily. Another chamber is illustrated in Fig. 8. The reinforced concrete wall is employed to protect personnel who conduct the test from a distance of 200 ft (61 m). This type of enclosure is only acceptable where three sides of the test site are unoccupied for a distance of several hundred feet since it is possible that some fragments may travel this distance. It is recommended that the side apron of the metal shield be lined with a layer of high strength steel since this area sustains the most severe damage. Additional liners can be welded on at the site as needed. Fig. 9 illustrates another possible" test shelter."

6. Hazards

6.1 Because of the fairly large quantities of explosives involved in propagation tests, tests cannot be performed in the laboratory, but shall be carried out at a suitable firing site. Before attempting to employ the test, those lacking experience should be thoroughly educated in the safe handling of explosives. Special safety precautions are recommended wherever hazards exist that are peculiar to the materials or procedures of the test. No attempt has been made to treat the general aspects of safety in explosives handling, since the literature ((1) through (7))³ amply cover this subject. State and local regulations concerning transportation, storage, and use of explosives should be consulted and *followed*.

- 6.2 Before each shot, the firing circuit shall be tested for continuity with a blasting galvanometer. The shot can be conveniently fired from the remote control point by means of a portable blasting machine. The firing line shall consist of 16-gage (1.29-mm) or heavier duplex copper conductor cable.
- 6.3 It is recommended that the firing line and all instrument lines have a positive disconnect at the firing position. The safest practice is to provide an *ungrounded* shunt block for each of the lines, best located in a box with a hinged cover and equipped with a lock. Routine inspection of all lines that are subject to physical damage by fragments or abrasion due to blasting shall be made and the lines replaced rather than repaired by splicing and taping. The shunts are removed and the connections made in the instrument and firing lines after the blast area is cleared and secured just prior to firing the shot.

7. Preparation of Apparatus

7.1 Since the density of liquids varies with temperature, and detonation velocity varies with density, it will be necessary, when determining detonation velocity, to measure and control the temperature.

Note 2—For example, the velocity of nitromethane varies about 3.7 m/s- $^{\circ}$ C over the range from -20 to 70 $^{\circ}$ C.

7.2 In the determination of critical diameter, temperature will affect the result since the shock sensitivity generally increases with temperature. Tests should therefore be made at 21° C within a tolerance of $\pm 5^{\circ}$ C. Temperature control can be provided by means of a jacket of insulated electrical heating tape around the sample container(s) in conjunction with a thermocouple(s). The heating tape can be fabricated tape $\frac{3}{16}$ by 0.003-in. (4.8 by 0.08-mm) Nichrome ribbon and $\frac{1}{4}$ -in. (6.4-mm) glass fiber sleeving.

8. Procedure

8.1 The first operation in setting up a shot consists of assembling the necessary components for Assembly No. 1, No. 2, No. 3, or No. 4, depending on which measurement is to be made. This assembly is best carried out at a table or bench in a charge preparation area near the firing chamber. The container shall then be suspended by a wire in the firing chamber, and when applicable the electrical connections to the target probes made. Warning—These probes can operate with relatively high potential. It is possible that an electrical fault can cause a premature initiation. Therefore, the target probe circuit shall be constructed with the same safety precautions used in the firing circuit. Pour the liquid under test at the desired temperature slowly and carefully into the container. In filling small diameter tubes, entrapped air shall be removed by opening the pinch clamp at the downstream end. Close the clamp when bubbles are no longer seen coming out in the liquid stream.

8.2 The liquid level shall be as high as possible without risking overflow. Then cover the container with a film of polyethylene (2 to 5 mils (0.05 to 0.13 mm) in thickness) or other plastic compatible with the test sample, to separate it from the pentolite booster and detonator. The finished assembly shall be tested at this time for leaks.

8.3 Prepare the cap by carefully removing it from its cardboard packing tube and straighten the attached 12-ft

³ The boldface numbers in parentheses refer to the list of references appended to this test method.

FIG. 5 Two-Channel Pulse Generator for Propagation Rate Measurements

(3.66-m) leads. If these leads are not long enough for eventual connection to the firing circuit at a safe distance (preferably out of line-of-sight) from the firing chamber, extension leads shall be used. (Electric blasting caps can be ordered with various lead lengths.) By means of the blasting galvanometer, check the extension leads for circuit continuity to make sure that they are shorted out (connected together) at the point where connection will be made to the firing circuit. After carefully inserting the cap into a length of heavy steel pipe (12 in. (305 mm) long by $1\frac{1}{2}$ in. (38.1 mm) in inside diameter by $2\frac{1}{4}$ in. (57.2 mm) in outside diameter, preferably located behind a shield or around the corner from the operator), make a similar continuity test to ensure that the wiring within the cap is not

defective. Then connect the cap leads to the extension leads by tight twisting; take care to make sure that the two splices cannot short out the cap by making contact with each other or with the ground. If no extension leads are used, short the cap leads by twisting together (usually the cap is received this way from the vendor). Then insert the detonator into the booster and place the booster-detonator assembly on top of the cap. Hold the whole assembly in place at the top by a single wrap of masking tape. Eliminate any air gap or bubble between the liquid level and the booster.

8.4 Open the firing-circuit terminal box (locked safety box "A," Fig. 7), adjacent to the firing chamber. Check the circuit leading to the control point for continuity, and disconnect the

FIG. 6 Power Supply for 2-Channel Pulse Generator

FIG. 7 Firing Site

extension leads (or cap leads, if no extension leads are used) from each other, and connect them to the respective firing-circuit terminals. Then at the control point at the remote end of

the firing circuit, unlock the terminal box (locked safety box "B," Fig. 7), energize the velocity probe circuit, if used, and connect the blasting machine to the terminals there. After

FIG. 8 Firing Site (Alternative)

FIG. 9 Firing Site (Alternative)

sounding whatever warning device is used (siren, horn, buzzer, etc.), fire the shot by operation of the blasting machine.

8.5 Provision shall be made for adequate ventilation of the firing chamber, for the gases present after a shot are usually

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highly toxic. When such gases have dissipated, the firing chamber may be entered (or opened) for recovery of the remains and preparation of the next shot.

9. Interpretation of Results

- 9.1 In every case, the length of tubing containing the donor section, that is, the forerunning section, should be completely destroyed and reduced to fine fragments.
- 9.2 In the critical-diameter tests (Assembly No. 1 or 3), the tubing under test will be completely fragmented its entire length if it is greater than critical diameter or fragmented for only a short distance if it is less than critical diameter.
- Note 3—The critical diameter is reported as lying between the incremental diameters experimentally employed.
- 9.3 In detonation trap tests (Assembly No. 4) the tubing will be similarly disintegrated for a length corresponding to the persistence of high-velocity detonation. Bursting or splitting of the tube, although having possible significance as regards safety of a particular system, is not a criterion of stable high-velocity detonation.
- 9.4 A sufficient number of trials should be made to establish whether reproducibility has been obtained.

10. Report

10.1 Detonation velocities are calculated directly from measured time lapses over the distance between stations (Assembly

- No. 2 or 3). They should be 1000 m/s or more. Generally, high-velocity detonations are characterized by measured rates between 3000 and 8500 m/s.
- 10.2 In reporting the results, the number of shots should be stated and, where possible, some mathematical expression of variation should be given, such as average deviation or standard deviation.
- 10.3 Any deviations from the recommended procedures should be reported with the test results.

11. Precision and Bias

11.1 Due to the complex nature of this test method and the expensive equipment involved in the initial setup of the apparatus, there is not a sufficient number of volunteers to permit a cooperative laboratory program for determining the precision and bias of this test method. If the necessary volunteers can be obtained a program will be undertaken at a later date.

12. Keywords

12.1 critical diameter; detonation velocity; liquid monopropellants; monopropellants; propellants

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REFERENCES

- (1) "Blaster's Handbook," 14th ed., 1958, Explosives Dept., E. I. duPont de Nemours and Co., Wilmington, Del. 19898.
- (2) Ordinance Safety Manual (ORD M7-224), Ordinance Corps, Dept. of the Army, 1951, as revised.
- (3) "Stray Currents in Electric Blasting" (*Data Sheet D-MIN.* 2), National Safety Council, 425 N. Michigan Ave., Chicago, Ill. 60611 (1950).
- (4) "Blasting from Electric Power Circuits" (Data Sheet D-MIN. 10), National Safety Council, 425 N. Michigan Ave., Chicago, Ill. 60611 (1950).
- (5) Sax, N. I., "Dangerous Properties of Industrial Materials," Reinhold Publishing Corp., New York, N. Y., 1968.
- (6) "Motor Carriers' Explosives and Dangerous Articles Tariff No. 10" (I.C.C. Regulations for Transportation of Explosives and Other Dangerous Articles by Motor, Rail and Water), American Trucking Association, Inc., November 1958, as amended.
- (7) "Liquid Propellant Handling, Storage, and Transportation," Chemical Rocket/Propellant Hazards, JANNAF Hazards Working Group, CPIA Publication No. 194, Chemical Propulsion Information Agency, Johns Hopkins University, Applied Physics Laboratory, Silver Spring, Md., Vol III, May 1970.

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