Designation: D 2540 – 93

Standard Test Method for Drop-Weight Sensitivity of Liquid Monopropellants[1]

This standard is issued under the fixed designation D 2540; 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 sensitivity of liquid monopropellants to the impact of a drop weight.

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 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.*

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

2. Summary of Test Method

2.1 A small sample of the liquid (0.03 mL) to be tested is enclosed in a cavity (0.06 mL) formed by a steel cup, an elastic ring, and a steel diaphragm (see Fig. 1). A piston rests on the diaphragm and carries a vent hole which is blocked by the steel diaphragm. A weight is dropped onto the piston. A positive result is indicated by puncture of the steel diaphragm accompanied by a loud noise or severe deformation of the diaphragm and evidence that the sample was completely consumed. The sensitivity value for a given sample shall be expressed as the height from which the specified weight is dropped for the probability of explosion to be 50 %.

3. Significance and Use

3.1 In drop-weight testing of liquids, explosions are initiated in a complex compression process involving the degree and rate of pressurization, the thermodynamic gas properties, heat transfer, hydrodynamic properties, etc. At this time, the fundamental significance of the test cannot be exactly defined. The test is considered useful, however, as a rapid and simple means to rate sensitive liquids as to their relative explosive sensitivity. Since it requires only a few grams of sample, it can be an important laboratory tool to determine the handling safety of new materials before substantial quantities are prepared.

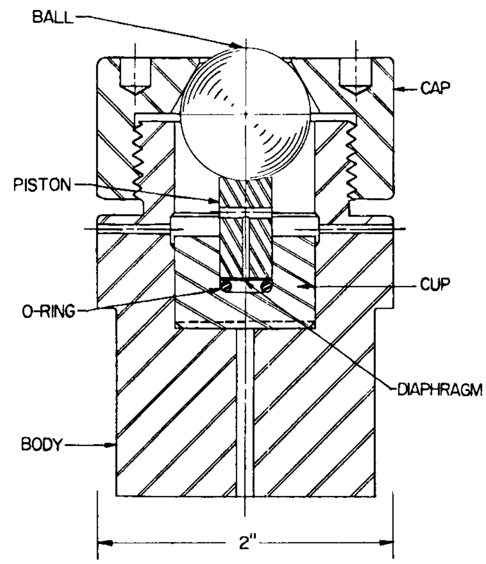

FIG. 1 Sample Cup Assembly

3.2 Tests in which the sample volume is varied (at constant cavity volume of 0.06 mL) show that the degree of filling affects the result. Note that the relationship between sensitivity rating and sample volume is not a characteristic of the test apparatus but is a function specific to each propellant. At 50 % filling (0.03 mL of sample), the dependency of sensitivity on sample volume is moderate so that the error in sample volume measurement has a negligible influence. Tests show that the delivered sample volume is reproducible to ±0.5 % when measured by a fixed-stroke syringe, and 0.03 mL shall be the standard sample volume.

3.3 If the objective justifies the greater effort, the sample volume is varied leading to a plot such as shown in Fig. 2 which represents the relationship between sensitivity rating and sample volume for the specific propellant *n*-propyl nitrate.

[1] This test method is under the jurisdiction of ASTM Committee F-7 on Aerospace Industry Methods and is the direct responsibility of Subcommittee F 07.02on Propellant Technology.

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[2] This method is identical in substance with the JANNAF method, "Drop Weight Test," Test Number 4, Liquid Propellant Test Methods, May 1964, published by the Chemical Propulsion Information Agency, Johns Hopkins University, Applied Physics Laboratory, Johns Hopkins Rd., Laurel, Md. 20810.

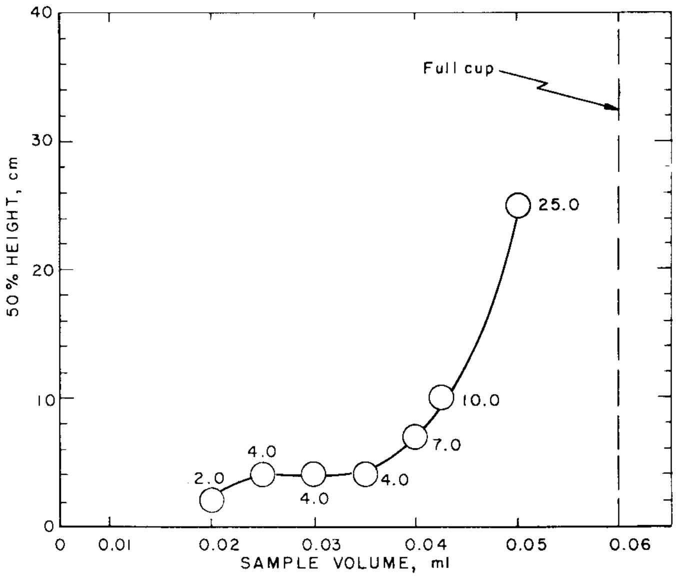

FIG. 2 Impact Sensitivity, H_{50}, of NPN versus Sample Volume at 70°F (21.1°C) Using a 2-kg Weight

4. Apparatus

4.1 *Sample Cup Assembly*—The sample cup assembly is shown in Fig. 1, and an exploded view in Fig. 3. The assembly shall consist of the following parts:

Part No.	Name
1	body
2	cup
3	O-ring (expendable)
4	diaphragm (expendable)
5	piston
6	ball
7	cap

Since the sample cup assembly is the critical part of the drop-weight tester, detailed dimensions of its components are given in Fig. 4.

4.2 *Weight*—The weight shall be one integral assembly, weighing 2 kg ± 1 g (Fig. 5). The weight shall be held suspended by an electromagnet. The electromagnet shall itself be held in the first version of the drop-weight tester by a stud at the top which fits in the recess formerly designed to hold and release the weight. The release shall be tied down to hold the magnet firmly in place. The present magnet plus weight shall be of such a length that the scale on the right hand post will read the correct drop height.

4.2.1 In using this weight, constant vigilance shall be maintained to see that the weight tip does not become excessively worn or damaged. If excessive wear is indicated the apparatus should be rechecked on a standard sample. Damaged weights should be discarded.

4.3 *Drop-Weight Assembly* (Fig. 6), consisting of a base plate with four leveling screws; column; two guide rods (one graduated); body retainer; release mechanism, adjustable to retain magnet; and top plate.

4.4 *Tools*, consisting of a torque wrench, 0 to 1.7-N·m (0 to 15-lb·in.) torque wrench adaptor to fit cap (part 7 of Fig. 4); hypodermic syringe of fixed stroke; O-ring seating tool; brass pick; and spanner wrench.

4.5 *Expandables*, such as O-rings: either AN 6227B-5 or 6.07± 0.13-mm (0.239 ± 0.005-in.) inside diameter and 1.78-mm (0.070-in.) cross section width, made from MIL-P-5516 elastomer, or both; and diaphragms of Type 302 stainless steel 0.41 ± 0.013 mm (0.016 ± 0.0005 in.) thick, 9.22 mm (0.363 in.) in diameter.

5. Safety Precautions

5.1 A positive safety latch shall be provided to prevent injury resulting from the premature fall of the weight. It is realized that this test might be employed for the evaluation of ultra-high-energy materials. This fact, combined with the possibility of faulty fabrication of components, could result in the production of shrapnel. It is therefore recommended that the apparatus be shielded (Fig. 7).

5.2 If the test apparatus is to be employed for the evaluation of toxic materials, or if toxic products may be formed from the decomposition of the sample, necessary steps shall be taken to prevent the buildup of dangerous concentrations of these materials.

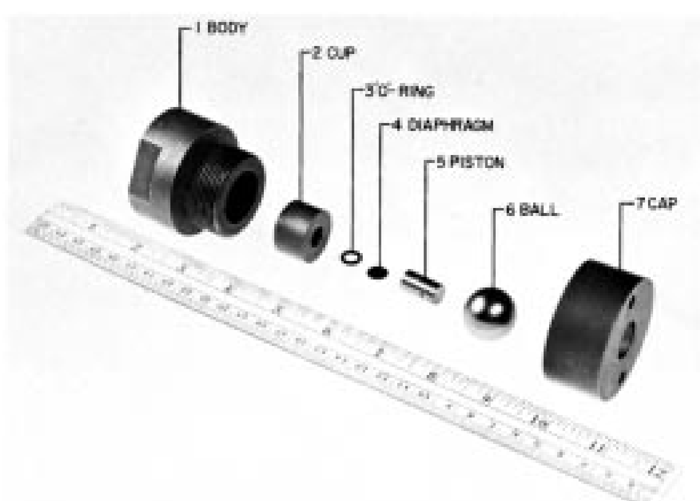

FIG. 3 Sample Cup Assembly, Exploded View

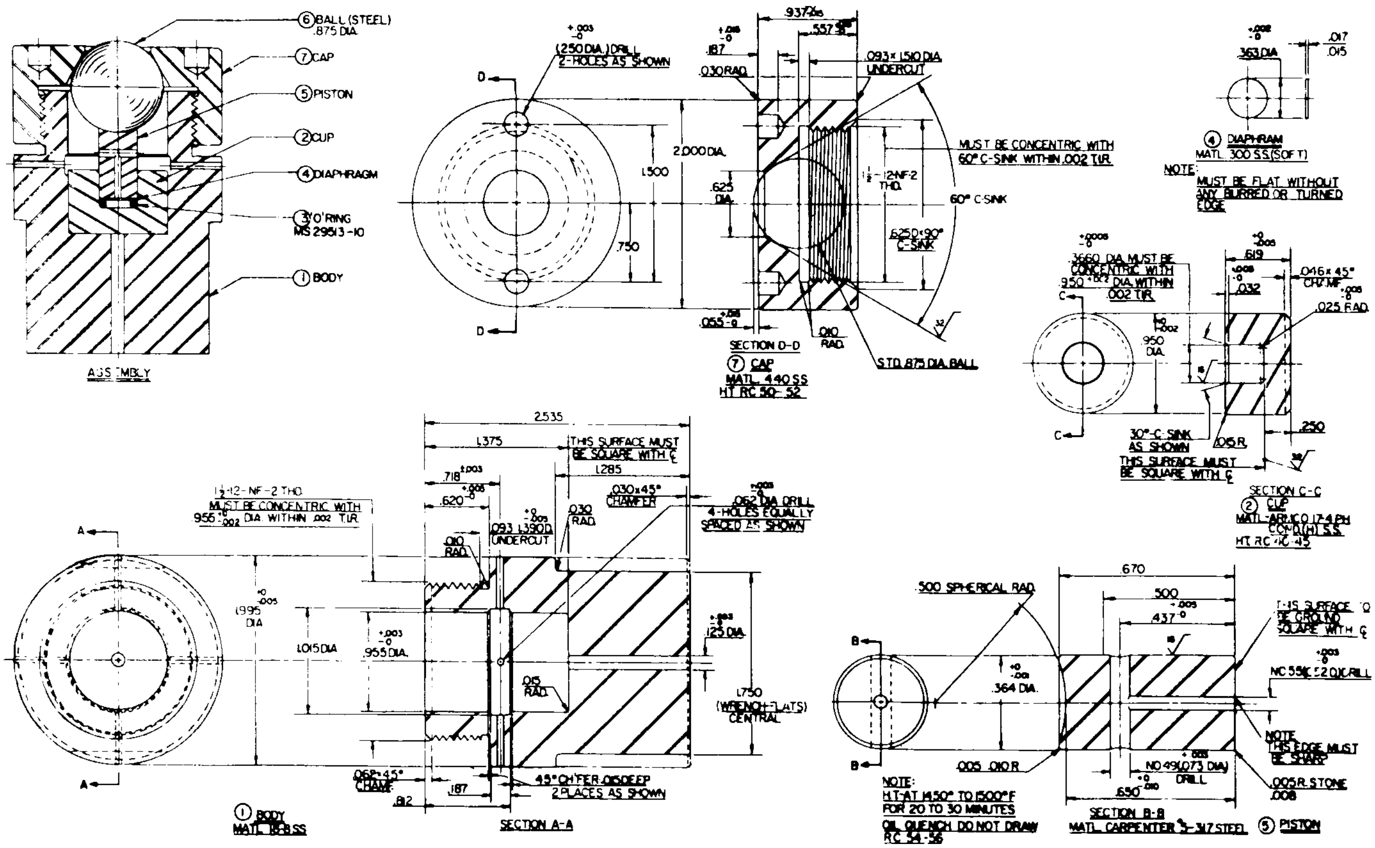

FIG. 4 Sample Cup Assembly, Detailed View

6. Preparation of Apparatus

6.1 Experience has shown that an appreciable difference in the behavior of the apparatus can result from the manner in which it is mounted. Therefore, the following conditions shall be met:

6.1.1 The machine shall be mounted on and firmly attached to a solid concrete foundation, preferably anchored to the foundation of the building.

6.1.2 The machine shall be perfectly plumb with guides lubricated to minimize friction during the fall of the weight.

6.2 The drop-weight sensitivity of sensitive liquids is, or course, dependent on the purity of the sample. The magnitude of this dependency will vary with the material. If attempts are being made to reproduce data obtained by other investigations, care shall be taken to obtain samples having identical analyses. Particular care shall be taken to keep the samples dry, as moisture may have an adverse effect.

7. Procedure

7.1 Results of this test have been found to be temperature-dependent. It is therefore very important to provide means to thermostat the sample cups, pistons, body of the assembly, and the liquid to be tested unless the whole equipment is kept in a constant-temperature room. Make all tests at 21.1 ± 1.1°C (70 ± 2°F).

7.1.1 Clean and dry all components of the body assembly. It is good practice to wash the metal parts in acetone and blow out both the exhaust hole and the cup with clean dry air. Wipe the cup clean with a tissue or soft cloth. After positive tests, check the exhaust hole and ports of the piston to be sure they are clear of the blown out section of the diaphragm. Note the condition of the pistons and cups. Replace cracked, pitted, or worn components.

NOTE 1—For temperature uniformity and speed of operation, it is recommended that a separate cup and piston be used for each test in a series. This also ensures that possible cup damage will not affect the results of subsequent tests in the series.

7.1.2 Set the height by loosening the locking handle that binds the release mechanism to the support column, and sliding the mechanism until the height indicator is properly aligned with the graduated guide rod. Tighten the locking handle and set the safety latch.

7.1.3 Place an AN 6227B-5 O-ring in the bottom of the cup, and with the brass O-ring seating tool force it down until it is firmly seated.

7.1.4 Fill the syringe with liquid and sweep out all entrapped air; wipe the point of the needle free of liquid; lower the point to the bottom of the cavity; carefully inject 0.03 mL into the cavity.

7.1.5 Slide a diaphragm across the top surface of the cup so that it drops flat onto the O-ring. Place the piston in the cup.

7.1.6 Place the cup in the body.

7.1.6.1 Jouncing or tilting of the cup prior to insertion in the machine shall be avoided particularly for low-viscosity liquids.

7.1.7 Place the ball on the top of the piston. Screw the cap to the body and tighten with the torque wrench to 0.8 N·m (7 lb·in.) holding the body in a vise.

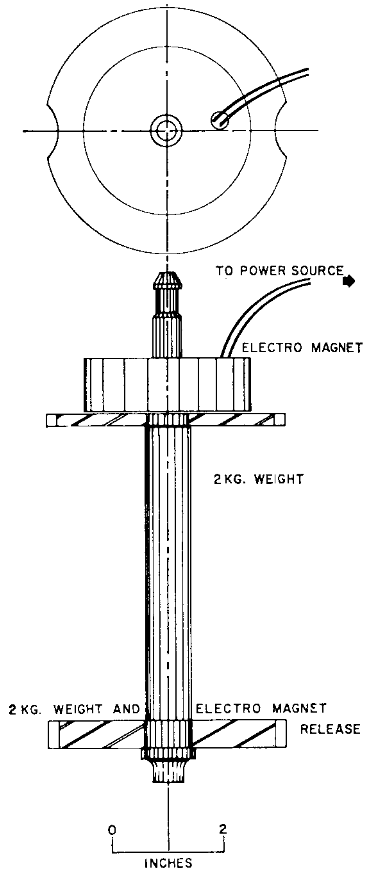

FIG. 5 Two-Kilogram Weight and Electromagnet Release

FIG. 6 Drop-Weight Tester

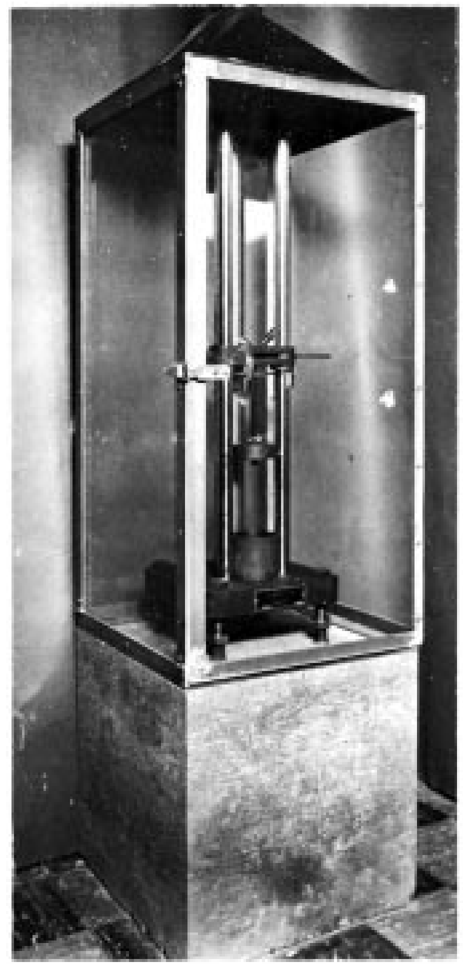

FIG. 7 Drop-Weight Tester in a Protective Enclosure

7.1.7.1 Care must be taken to ensure that the small pellets punched out of the diaphragm in positive tests do not get lodged under the cup in the holder, under the holder on the stand, or caught in the threads. If the operator notices some slight blockage or stiffness due to dirt or a metal chip fouling the threads, halt the test and clean the threads until they work freely. If it becomes necessary to remove burrs which may have developed on the threads, a fine lapping compound can be worked into the threads, but it shall be removed and the threads thoroughly cleaned before testing is continued. Use a thread lubricant, but take great care not to overlubricate.

7.1.8 Place the body assembly into the retainer on stand. Release the safety latch. Release the weight by de-energizing the electromagnet. (See Fig. 8 for an illustration of the first-bounce catching pin.)

7.1.9 Record the test result. A typical data sheet is shown in Fig. 9.

7.1.10 Take the body assembly from the stand, remove the cap by hand, or if jammed, by spanner wrench. Jamming will often result from internal pressure after a combustion that did not puncture the diaphragm. Remove the cup, lift the piston out, and work the diaphragm free with a brass pick. Discard the diaphragm and O-ring after each test (including negative tests).

7.1.11 Clean as described in 7.1.1.

8. Calculation

8.1 Calculate the H_{50} value as follows:

$$H_{50} = H_0 + \Delta[(\Sigma_{in}/\Sigma_n) \pm 1/2] \tag{1}$$

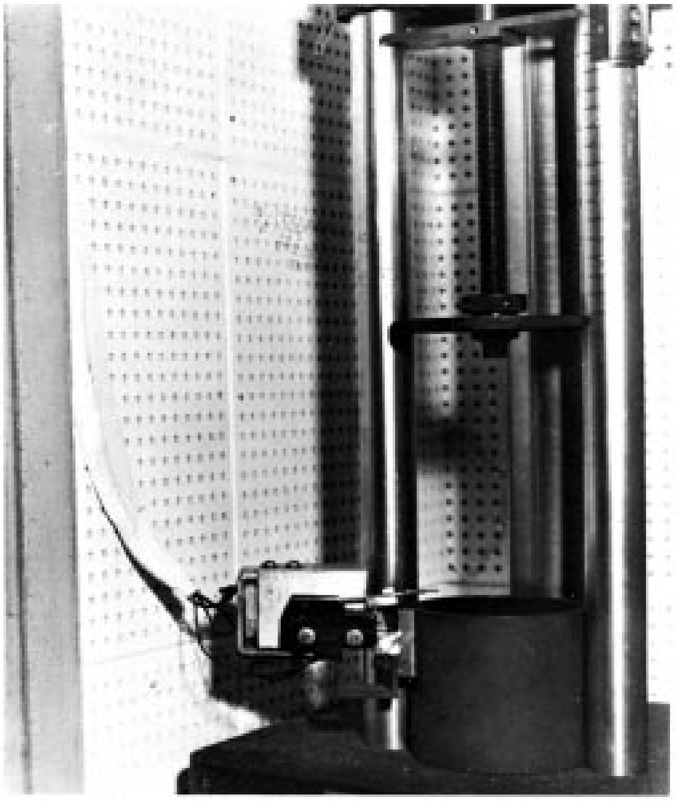

FIG. 8 Close-Up of Drop-Weight Tester Showing a First-Bounce Catching Pin

where:

Δ = interval between the trials (2 for the sample given),
H_o = height corresponding to the lowest level on which the less frquent occurs (3 for the sample given), cm.

When positive trials are used for calculating the H_{50}, the minus sign is applicable. When negative trials are used the plus sign is applicable.

9. Interpretation of Results

9.1 In some trials the sample will be found to have been completely burned, but the diaphragm dimpled rather than perforated, and gas pressure may be observed in the cavity. If this reaction occurs, count the result as positive. Deformation of the diaphragm can take place with large heights without ignition of the sample. Examination of the sample holder will allow such negative results to be distinguished from positive because the cup will be wet with the sample, and no gas pressure will be observed. Note all instances of this type in the Remarks column of the data sheet (Fig. 9).

9.2 In some cases it has been observed that ignition of the sample does not occur upon the initial impact, but does occur following a second or third impact. Since the height associated with subsequent impacts cannot be reproduced precisely, and since the rating of the sensitive liquid is based on the initial height of the weight, ignitions produced by more than one impact shall not be counted as positive. A first-bounce catching pin may be employed if differentiation cannot be made between ignitions resulting from the first or subsequent impacts. (See Fig. 8 for an illustration of the first-bounce catching pin.)

9.3 If ten successive trials at the maximum height (50 cm) are negative, discontinue and report the result as negative. If the first trial at a 50 cm height is positive, conduct a second test at 25 cm. If this second test is negative, testing shall be continued by varying the height between 25 and 50 cm. Enter the data as shown in Fig. 9. Bracketing of the 50 percent point shall be carried out by increasing the height after a negative result and decreasing it after a positive result with the gap continually narrowed by reducing the change in height by half with each trial. When a change in sign (positive or negative) is obtained with a change in height of 1 cm, a minimum of 20 trials should be conducted increasing the height by 1 cm when a negative test occurs and decreasing it by 1 cm when a positive test occurs.

9.4 If a trial at 2 cm is positive, repeat the trial for a minimum of 6 times. If any negative results occur, the bracketing procedure described in 9.3 should be employed. If six positive results occur within 10 trials at the minimum height, discontinue testing and report the 50 % point as less than 1 cm.

9.4.1 The 50 % points for nitroglycerin, ethyl nitrate, and diethyleneglycol dinitrate are all approximately 1 cm. Any materials yielding positive results for heights at or below this value are considered to be sensitive explosives. Handle with extreme caution.

9.5 In this drop weight test, as in other sensitivity tests, the critical stimulus needed for ignition may vary from trial to trial. The height that yields a 50 % probability of ignition, H_{50}, shall be determined on several samples of the same material instead of by direct and repeated measurements on a single sample and shall be used as a measure of sensitivity. The test procedure used is based on an established statistical method (known as the" up-and-down" method) for obtaining the H_{50} with a minimum number of trials.

9.6 When a minimum of 20 trials has been completed, as described in Section 7, the 50 % point can be determined by the following analytical procedure: the points shall be marked on the computation sheet. A typical computation sheet is shown in Fig. 10 using (+) signs to indicate positive results and (0) signs to indicate negatives. The total of positive and negative results at each level shall then be indicated. For purposes of computing the 50 % probability designated H_{50}, only the positive or negative trials (whichever are present in smaller number) shall be used. The trials to be used shall then be tabulated. Under the column headed *H*, enter the levels in centimeters at which trials were conducted starting from the lowest in the first row. Under the column headed *i*, enter the number of the levels in sequence starting from zero in the first row. Under the column headed *n*, enter the number of trials at each level. Under the column *in*, enter the product of the *i* column and the *n* column for that level. Add the figures in the column headed *n* and enter the total as Σ_n at the bottom of the column. Add the column headed *in* and enter the total asΣ_{in} at the bottom.

10. Report

10.1 Report the H_{50} value for the propellant tested along with the properties of this test method.

DROP-WEIGHT TESTER
DATA SHEET

Sample: *n* Propyl nitrate

Sample Volume: 0.03 mL

Ambient Temperature: 71°F

Sheet No.:

Date:

Operator:

Test No.	Height, cm	Weight, kg	*E*, kg·cm	Test Results		Remarks
				Positive	Negative	
1	6	2	12		0	
2	7	2	14		0	
3	8	2	16	+		
4	7	2	14	+		
5	6	2	12		0	
6	7	2	14	+		
7	6	2	12	+		
8	5	2	10		0	
9	6	2	12		0	
10	7	2	14	+		
11	6	2	12	+		
12	5	2	10		0	
13	6	2	12	+		
14	5	2	10	+		
15	4	2	8	+		
16	3	2	6		0	
17	4	2	8	+		
18	3	2	6		0	
19	4	2	8		0	
20	5	2	10	+		
21	4	2	8	+		
22	3	2	6		0	
23	4	2	8		0	
24	5	2	10	+		
25	4	2	8		0	
26	5	2	10	+		
27	4	2	8	+		
28	3	2	6		0	
29	4	2	8		0	
30	5	2	10		0	
31	6	2	12	+		
32	5	2	10	+		
			Total	17	15	

FIG. 9 Typical Data Sheet

11. Precision and Bias

11.1 *Precision*:

11.1.1 Repeatability experience with the apparatus has demonstrated that the standard deviation will be about 1 cm.

11.1.2 Any new operator may wish to reassure his technique by testing a liquid whose drop-weight rating has been well established. *n*-Propyl nitrate (NPN) can be used for this purpose. Typical results with NPN are shown in Fig. 9 and Fig. 10. The material as received was 99.9 % pure.

11.1.3 Data over the range 0.7 to 7 cm indicate that the results submitted by each of two laboratories should be considered suspect if they differ by more than 1.4 times their mean.

11.2 *Bias*—The procedure in this test method has no bias because the value of the drop-weight sensitivity is defined only in terms of this test method.

12. Keywords

12.1 drop-weight sensitivity; explosive sensitivity; impact test; monopropellant; liquid; propellant; liquid

DROP-WEIGHT TESTER
COMPUTATION SHEET

Sample: *n* Propyl nitrate
Operator:
Test Series No.:
Temperature: 71°F
Data Sheet No.:
Water Calibration:
Date:

H, cm	Number of Trials																									Total		Percent Positive
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	+	–	
6	+																		+							2	6	100
5		+						+				+		+				0		+						5	1	83
4			+		+		0		+		0		0		+		0									4	4	50
3				0		0				0						0										0	4	0
																					Total					11	9	

Sensitivity Number (H_{50}):

$H_{50} = H_0 + \Delta(\Sigma_{in}/\Sigma_n \pm 1/2)$[a] { + when negative trials are considered; – when positive trials are considered }

$H_o = 3\Delta = 1$

Considering negative trails:

H	*i*	*n*	*in*
3	0	4	0
4	1	4	4
5	2	1	2
6	3	0	0

$\Sigma_n = 9$ $\Sigma_{in} = 6$

$H_{50} = 3 + 1\ (6/6 + 1/2)$
$H_{50} = 3 + 1\ (1.2)$
$H_{50} = 4.2$

[a] If the number of negative and positive trials is unequal, consider smaller number.

FIG. 10 Typical Computation Sheet

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