

Standard Test Method for Yield Stress of Heterogeneous Propellants by Cone Penetration Method¹

This standard is issued under the fixed designation D 2884; 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 determination of the yield stress of heterogeneous propellants, both of the gel and emulsion types, containing from 0 to 70 % solid additives.

1.2 The values stated in SI units are to be regarded as the standard. In cases where materials, products, or equipment are available in inch-pound units only, SI units are omitted.

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. Referenced Documents

2.1 *ASTM Standards:*

D 2507 Terminology of Rheological Properties of Gelled Rocket Propellants³

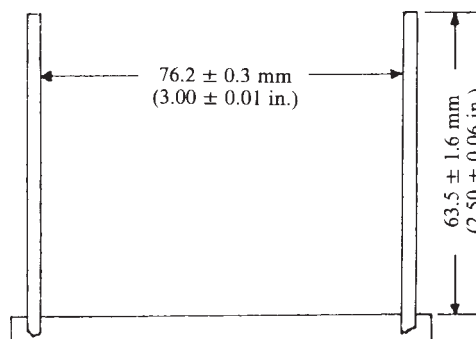
3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *penetration of a propellant, n*—The depth, in tenths of a millimetre that a standard cone penetrates the sample under prescribed conditions of weight, time, and temperature.

3.1.2 *unworked penetration, n*—the penetration at 298 K (77°F) of a sample of the propellant which has received only the minimum disturbance in transfer from the sample can to a grease worker cup or dimensionally equivalent container. This shall be 76.2 ± 0.3 mm (3 ± 0.01 in.) in inside diameter and 63.5 ± 1.6 mm (2.5 ± 0.06 in.) deep, as shown in Fig. 1.

3.1.3 The conversion of penetration to yield stress has not been corrected for the displacement of the sample by the



NOTE 1—This cup is dimensionally equivalent to the grease worker cup.

FIG. 1 Penetrometer Cup

submerged portion of the cone. For this reason cup diameter is critical, and any deviation from 76.2 ± 0.3 mm (3 ± 0.01 in.) must be reported as a nonstandard condition.

3.1.4 *yield stress*—the maximum shear stress that can be applied without causing permanent deformation (see Terminology D 2507).

Specifically in this test method, it is the weight of the 30-g mass cone-test rod assembly in dynes, corrected for buoyancy, divided by the calculated wetted area of the cone (that is, the area of the cone in contact with the propellant after the 5-s drop period).

4. Summary of Test Method

4.1 The penetration is determined at 298 K (77°F) by releasing the cone-test rod assembly from the penetrometer and allowing the assembly to drop for 5 s. The cone will be essentially at rest in less than this time, so that exact timing is not critical.

5. Significance and Use

5.1 The yield stress is a measure of the forces required to initiate and maintain flow from a storage vessel. If all the factors are constant, the propellant with the lower yield stress can be removed more completely from the vessel.

6. Apparatus

6.1 *Penetrometer*, to measure the penetration of the standard cone in the propellant. The cone assembly or the table of the

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.11 on Engineering Sciences of High Performance Fluids and Solids. ASTM Committee F07 on Aerospace and Aircraft maintains a continued interest in this test method and will make use of it in the future.

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² This test method is identical in substance with the JANNAF method, "Heterogeneous Propellant Characterization, Part III, Procedure for Measuring Yield Stress of Heterogeneous Propellants," published by the Chemical Propulsion Information Agency, July 1969, Johns Hopkins University, Applied Physics Laboratory, Johns Hopkins Rd., Laurel, MD 20810.

³ *Annual Book of ASTM Standards*, Vol 15.03.

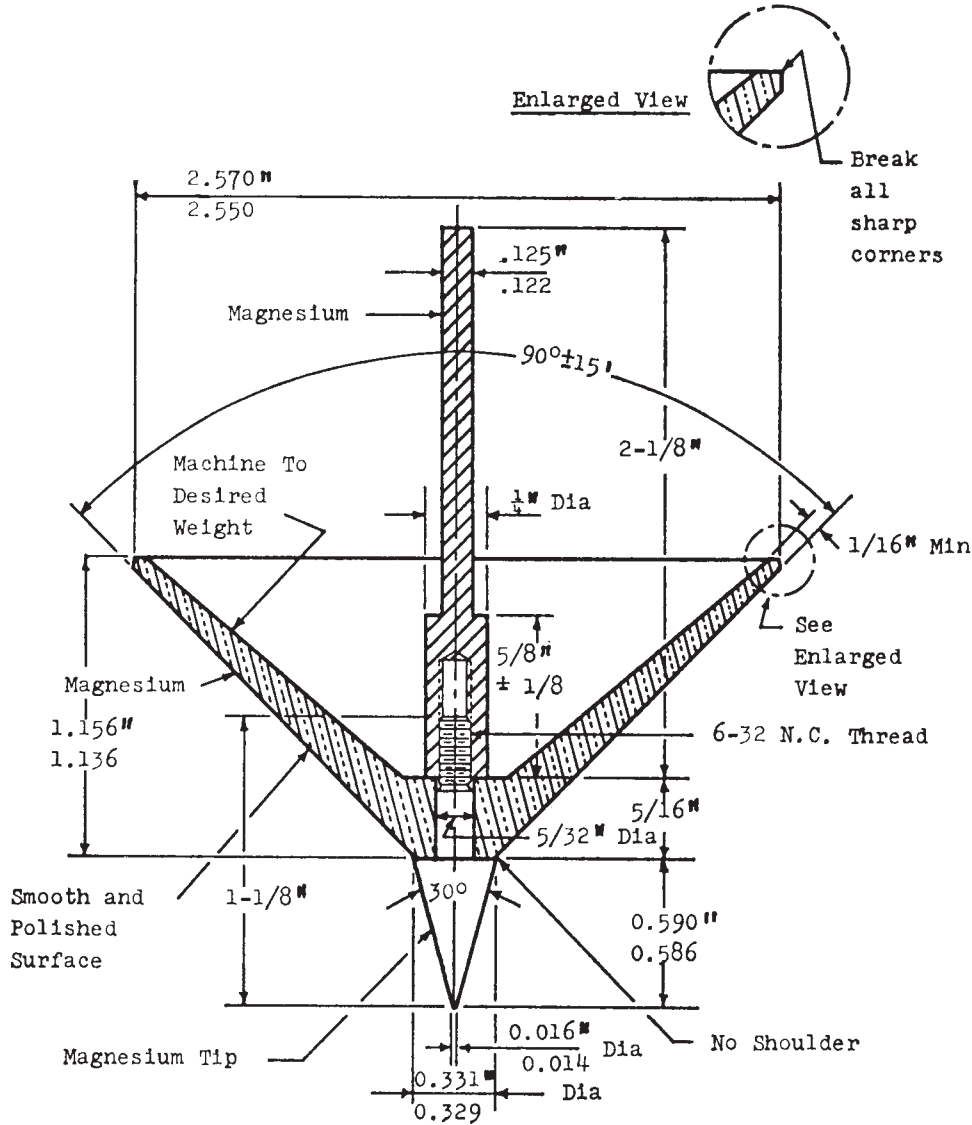
penetrometer shall be adjustable to enable accurate placement of the cone on the level surface of the propellant while maintaining a zero reading on the indicator. The cone should fall, when released, without appreciable friction for at least 42.0 mm but not more than 60.0 mm so the cone will not hit the bottom of the container. The instrument shall be provided with leveling screws to maintain the cone shaft in a vertical position and a spirit level to determine the attitude of the instrument.

6.1.1 A 15-g test rod shall be substituted for the regular 47.5-g rod in accordance with the manufacturer's instructions.

6.2 Cone, of the dimensions shown in Fig. 2.

NOTE 1—Some cones have correct dimensions and weight (15.0 g) but react with hydrazine gels. The user should plan to have one made to order.

6.3 Grease Worker Cup, or equivalent container. The cup shall be made of material known to be compatible with all



NOTE 1—Tolerances on all fractional dimensions shall be $\frac{1}{16}$ in.

NOTE 2—The total mass of the cone shall be 15.00 ± 0.05 g and the total mass of its movable attachments shall be 15.00 ± 0.05 g.

NOTE 3—Table of Metric Equivalents

in.	mm	in.	mm	in.	mm	in.	mm
0.014	0.356	$\frac{1}{8}$	3.18	0.331	8.407	1.136	28.854
0.016	0.406	$\frac{5}{32}$	3.98	0.586	14.884	1.156	29.362
$\frac{1}{16}$	1.59	$\frac{1}{4}$	6.35	0.590	14.986	$2\frac{1}{8}$	53.98
0.122	3.10	$\frac{5}{16}$	7.94	$\frac{5}{8}$	15.88	2.550	64.77
0.125	3.18	0.329	8.357	$1\frac{1}{8}$	28.575	2.570	65.28

FIG. 2 Magnesium Penetrometer Cone

propellants which are to be tested in it. Construction may be two-piece as shown in Fig. 1, using a silver solder or braze which meets the compatibility requirement to seal the joint. Several users have had cups machined in one piece from bar stock, or from a rough casting.

6.4 *Water Bath*, capable of regulation to 298 ± 0.5 K ($77 \pm 1^\circ$ F). For unworked penetrations, means must be provided for protecting the propellant surface from atmosphere moisture.

NOTE 2—A constant-temperature room or an air bath may be used instead of the water bath. A special atmosphere may be required for some propellant samples.

6.5 *Spatula*, corrosion-resistant, square-ended, having a stiff blade 30 mm wide and at least 150 mm long.

7. Procedure for Unworked Penetration

7.1 *Test Specimen*—Sufficient propellant test specimen (at least 450 g) to overfill the cup of the standard grease worker shall be required. If the anticipated penetration is above 200, at least three times the amount needed to fill the cup shall be required.

7.2 *Preparing Test Specimen for Measurement*—Place the empty assembled grease worker or suitable container of equal inside dimensions and an appropriate amount of the sample in a sealed container in the water bath maintained at 298 K (77° F) for sufficient time to bring the temperature of the sample to 298 ± 0.5 K ($77 \pm 1^\circ$ F). Transfer from the container a portion of the sample, preferably in one lump, to overfill the cup of the grease worker or container of equal inside dimensions. Make this transfer in such a manner that the sample will be worked as little as possible. Jar the container to drive out trapped air and pack the sample with the spatula, with as little manipulation as possible to obtain a capful without air pockets. Scrape off the excess sample extending above the rim by moving the blade of the spatula, held inclined toward the direction of motion at an angle of 45° across the rim of the cup. Do not perform any further leveling or smoothing of the surface throughout the determination of unworked penetration, and determine the measurement immediately.

7.3 *Cleaning Cone and Shaft*—Clean the penetrometer cone carefully before each test. Bending of the test rod can be avoided by holding it securely in its raised position while cleaning. Do not permit grease, oil, or propellant on the test rod, as they can cause drag on the assembly. Do not rotate the cone, as this may cause wear on the aluminum test rod.

7.4 Penetration Measurement:

7.4.1 Place the cup on the penetrometer table, making certain that it cannot teeter. Set the mechanisms to hold the cone in the zero position, and adjust the apparatus carefully so that the tip of the cone just touches the surface at the center of the test sample. Watching the shadow of the cone tip is an aid to accurate setting. Release the test rod rapidly, and allow it to drop for 5.0 ± 0.5 s. The clutch jaws must not drag on the shaft. Gently depress the depth gage rod until stopped by the lower stop, and read the penetration from the indicator.

7.4.2 If the sample has a penetration over 200, center the cone carefully in the container; this sample can then be used for only one test. If the sample has a penetration of 200 or less,

perform three tests in a single container, spacing these tests on three radii 120° apart and midway between the center and side of the container so that the cone will neither strike the side of the container nor impinge on the disturbed area made in a previous test.

7.5 *Additional Testing*—Make a total of three tests on the sample (either in three containers or in one, as described in 7.4), unless the first reading is greater than 420.

8. Report

8.1 *Ordinary Samples*—The three test results shall be averaged and used to calculate the yield stress, S_y , as follows:

$$S_y = \frac{981(30.0 - (1.047 \times 10^{-6})(P - 107.4)^3 d - 0.198d)}{4.44 \times 10^{-4}(P - 107.4)^2 + 1.260} \quad (1)$$

where:

P = penetration, tenths of a mm,

d = density, g/cm^3 , and

S_y = yield stress, dynes/cm^2 (Note 5).

S_y^p = $S_y/10$ = yield stress, Pa.

8.2 *Excessively Soft Samples*—If the penetration exceeds 420, the propellant will overflow into the cone. This invalidates the use of the equation, and the sample shall be reported as too soft.

8.2.1 If the material from which the cone is made is other than magnesium, this fact shall be stated in the report along with sufficient information to permit identification of the material used. It is known that slippage along the cone surface can take place with surfaces which are highly lipophilic, such as polyethylene, PTFE (polytetrafluoroethylene), and so forth. Such cones should be used only when the purpose of the test is to determine an apparent yield stress in relation to a specific structural material or lining.

9. Precision and Bias

9.1 The precision of the method as determined by statistical examination of interlaboratory results is as follows:

9.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

40 %


9.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

75 %

9.2 *Bias*—No general statement is made on the bias for this test method since the results cannot be compared with an accepted reference material.

10. Keywords

10.1 cone penetration; heterogeneous propellant; propellants; yield strength

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