BRIEFING

⟨915⟩ Measurement of Structural Strength of Semisolids by Penetrometry. This proposed chapter describes empirical methods of measuring the structural strength or consistency of a semisolid raw material or dosage form with a penetrometer. In addition, this chapter outlines a method for performing the gravity-driven penetrometry measurements of semisolids, as well as an alternate method for using computer-controlled instruments to perform penetrometry measurements to achieve results that are comparable to the traditional gravity-driven method.

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Add the following:

▪⟨915⟩ MEASUREMENT OF STRUCTURAL STRENGTH OF SEMISOLIDS BY PENETROMETRY

INTRODUCTION

This chapter describes empirical methods of measuring the structural strength, or consistency, of a semisolid raw material or dosage form with a penetrometer. Viscosity represents the proportionality of the shear stress to the shear rate for a Newtonian fluid, whereas consistency is the term for this proportionality for semisolids that exhibit non-Newtonian viscoelastic rheological behavior. Because the term “consistency” may be confused with uniformity or homogeneity the term “structural strength” is now the preferred term. In the remainder of this chapter we will use the term structural strength to refer to this property of semisolids. One component of the structural strength of a semisolid is its hardness (yield stress). Penetrometry is one method for quantifying the hardness (yield stress) of semisolid materials.

Penetrometry allows a metal cone with standardized dimensions and weight to penetrate into a semisolid until the buoyancy of the cone and the yield stress of the semisolid exactly balance the gravity-applied force, driving the penetrating object into the semisolid. This observed yield stress of the semisolid is a measure of the semisolid hardness. The yield stress (hardness) of the semisolid will be inversely proportional to the penetration depth of the cone.

This chapter outlines the method for performing the gravity-driven penetrometry measurements of semisolids. Although the results of the penetrometry measurements may be used to calculate the yield stress of the semisolid (see *Measurement of Hardness of Semisolids* ⟨1912⟩ for more information), the results of the penetrometry experiment are reported as the observed penetration depth in tenths of millimeters (dmm). This chapter also outlines an alternate method for using computer-controlled instruments to perform penetrometry measurements to achieve results that are comparable to the traditional gravity-driven method.

APPARATUS

The apparatus consists of a penetrometer made up of a stand and a penetrating object. A suitable stand is shown in *Figure 1*, and a suitable penetrating object (cone) is shown in *Figure 2*.

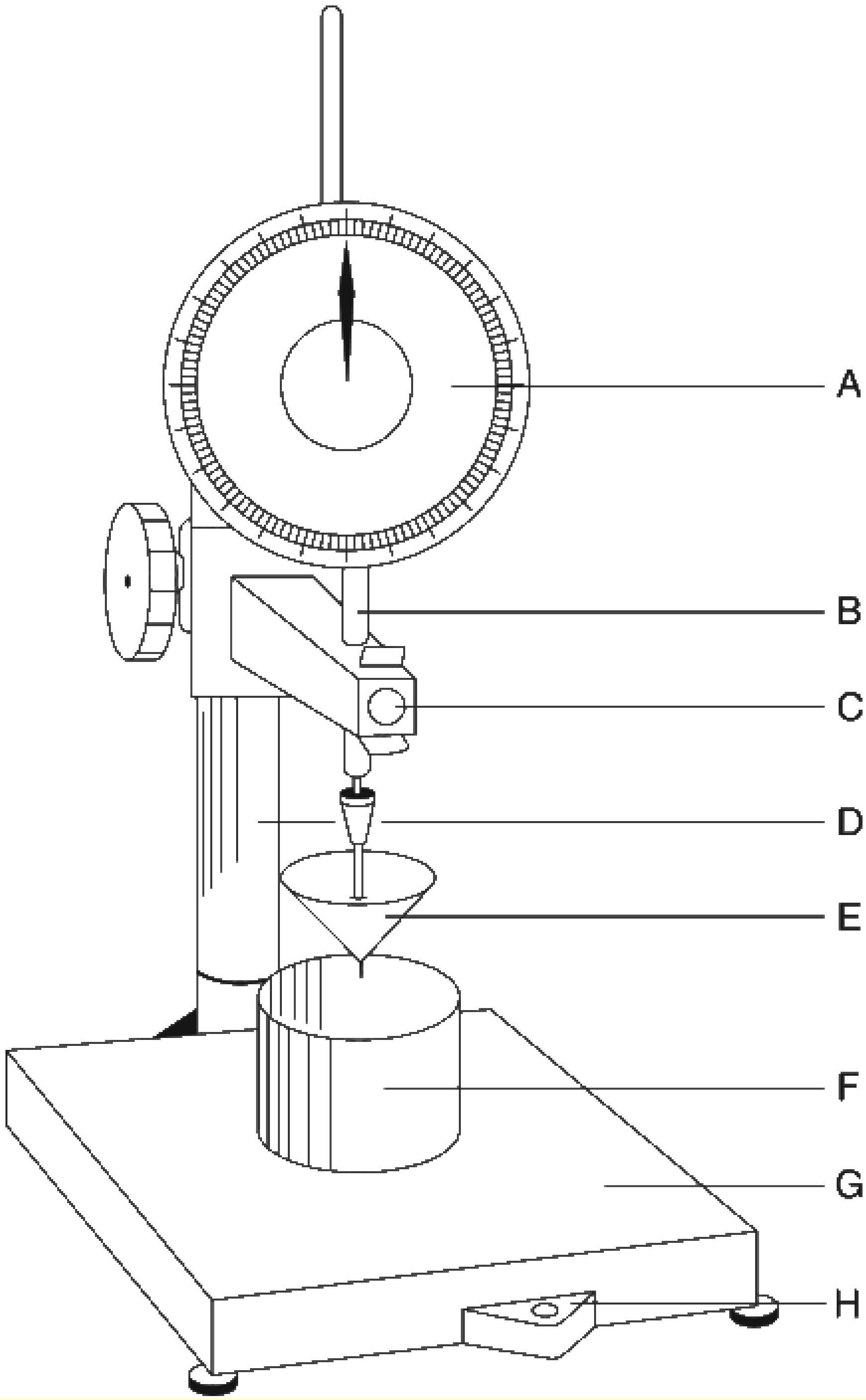

Figure 1. Penetrometer apparatus (gravity-driven). *A*: Scale showing the depth of penetration, graduated in dmm. *B*: Vertical shaft to maintain and guide the penetrating object. *C*: Device to retain and to release the penetrating object. *D*: Device to ensure that the penetrating object is vertical and that the base is horizontal. *E*: Penetrating object made of a suitable material, has a smooth surface, and is characterized by its shape, size, and mass (see Figure 2). *F*: Sample container. *G*: Horizontal base. *H*: Control for the horizontal base.

PROCEDURE

Sample Preparation

With samples having penetrations <200 dmm, three tests (and sometimes more) may be made in one container by proper spacing. To prevent one test from being affected by the disturbed area of a previous test, the tip of the cone must not be placed nearer the edge of a previous test than the penetration distance of the sample. Some harder samples tend to form a marked depression in the center upon solidifying; such samples should not be tested in this depression because the results obtained may be different from those obtained in off-center positions on the level surface.

With samples having penetrations >200 dmm, only one test may be made in a container by placing the cone tip near the center of the container.

Fill the required number of containers to within 6 mm (1/4 inch) of their rims. Allow the filled containers to cool in a location free from drafts and at a temperature controlled to 25 ± 2° for 16–18 h, then cover and place the samples in the water bath for 2 h to bring the temperature to 25 ± 0.5° before testing.

Prepare the test samples using one of the following procedures:

1. Carefully and completely fill three containers without forming air bubbles. Level, if necessary, to obtain a flat surface. Store the samples at 25 ± 0.5° for 24 h, unless otherwise prescribed.
2. Store three samples at 25 ± 0.5° for 24 h. Apply a suitable shear to the samples for 5 min. Carefully and completely fill three containers, without forming air bubbles, and level if necessary to obtain a flat surface.
3. Liquify (i.e., heat to above the melting point) three samples, and carefully and completely fill three containers without forming air bubbles. Store the samples at 25 ± 0.5° for 24 h, unless otherwise prescribed.

Determination of Penetration

This method requires the use of a penetrating object that complies with the dimensions of *Figure 2*. For gravity-driven penetration measurements, the penetrating object should have a total effective mass of 150 g (including the weight of the penetrating object and additional masses firmly attached during the penetration experiment).

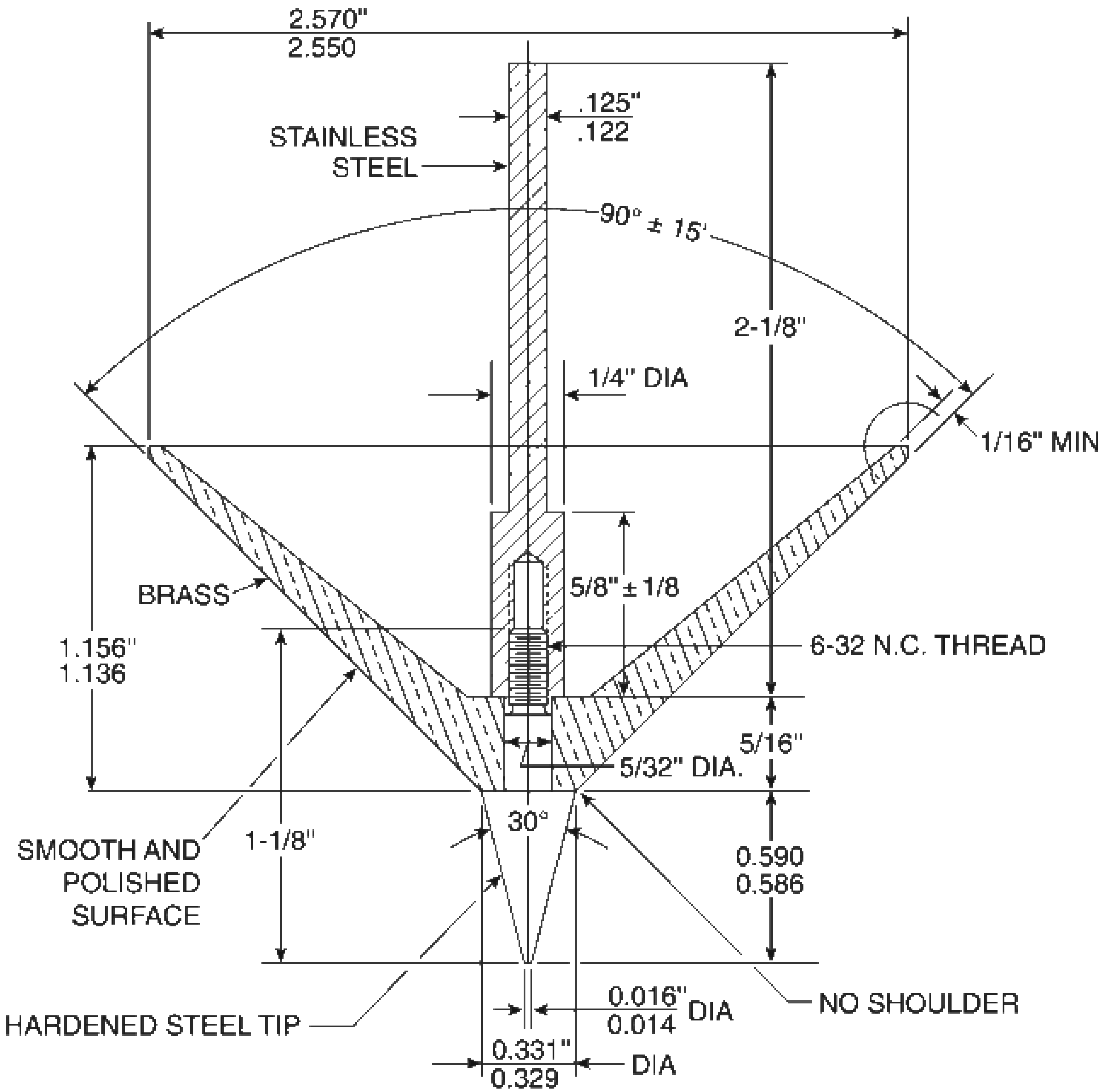

Figure 2. Penetration cone dimensions. DIA, diameter; N.C., National Coarse.

Method I (gravity-driven measurement of structural strength)

1. Place the test sample on the base of the penetrometer and verify that its surface is perpendicular to the vertical axis of the penetrating object.
2. Bring the temperature of the penetrating object to 25 ± 0.5° and then adjust its position such that its tip just touches the surface of the

sample. [NOTE—Watching the shadow of the tip is an aid in accurate setting.]

3. Release the penetrating object and allow it to descend, without restraint, for 5 s.
4. Clamp the penetrating object and measure the depth of penetration.
5. Repeat the test with the two remaining containers.

Modern, computer-controlled instruments are able to control the speed of the penetrating object and may produce more precise results. To produce results using these instruments, which are comparable to the traditional (gravity-driven) penetrometry method, the following procedure should be followed.

Method II (constant-speed measurement of structural strength)

1. Place the test sample on the base of the penetrometer and verify that its surface is perpendicular to the vertical axis of the penetrating object.
2. Bring the temperature of the penetrating object to 25 ± 0.5° and then adjust its position such that its tip just touches the surface of the sample. [NOTE—Watching the shadow of the tip is an aid to accurate setting.]
3. Set the instrument penetration rate to a constant speed in the range of 1–20 mm/s.
4. Either set the instrument to stop penetration when the applied force reaches 1471 mN, or set the instrument to record the applied force versus distance for the duration of the experiment.
5. Zero (tare) the force of the instrument prior to initiating the penetration experiment.
6. Record the penetration result as the depth (either actual or interpolated) where the applied force was ≥1471 mN.
7. Repeat the test with the two remaining containers.

EXPRESSION OF THE RESULTS

Report the mean and standard deviation of at least three replicate measurements in units of penetration depth. The penetration depth is expressed in dmm. If any of the individual results differ from the mean by >3%, repeat the test and report the mean and standard deviation of six replicate measurements in units of penetration depth.

GLOSSARY

Note that the following definitions are provided to clarify the use of these terms in the context of this chapter. These definitions are not intended to supersede or contradict definitions found elsewhere in *USP–NF*.

Hardness: Hardness is a term used synonymously with yield stress—a harder semisolid also exhibits a larger apparent yield stress. In penetrometry, hardness has been more specifically defined as $H = C \times W/p^n$, where C is a constant dependent on the cone geometry, W is the weight of the penetrating cone, p is the depth of penetration, and n is an exponent. When the exponent, n, is 2, hardness has the same units as yield stress (Pa). See ⟨1912⟩ for more information.

Penetrometer: An instrument that measures the structural strength of semisolid materials by measuring the depth to which a specified cone or needle under a given force falls into the material.

Structural strength (consistency)**:** This term "consistency" is sometimes used synonymously with viscosity, albeit incorrectly. Viscosity represents the proportionality of the shear stress to the shear rate for a Newtonian fluid, whereas consistency is the term for this proportionality for semisolids that exhibit non-Newtonian viscoelastic rheological behavior. Because the term consistency may be confused with uniformity or homogeneity the term "structural strength" is now the preferred term. See ⟨1912⟩ for more information.

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