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# Standard Test Methods for Measurement of Yield Stress of Paints, Inks and Related Liquid Materials[1]

This standard is issued under the fixed designation D7836; 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 These test methods cover three approaches for determining yield stress values of paints, inks and related liquid materials using rotational viscometers. The first method uses a rotational viscometer with coaxial cylinder, cone/plate, or plate/plate geometry. The second method uses a rheometer operating in controlled stress mode with similar geometries. The third method uses a viscometer with a vane spindle.

1.2 A non-rotational technique, the falling needle viscometer (FNV), also can be used to measure yield stress values in paints, inks and related materials. See Test Methods D5478, Test Method D, Yield Stress Determination for details.

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:*[2]

D3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings

D5478 Test Methods for Viscosity of Materials by a Falling Needle Viscometer

## 3. Terminology

3.1 *vane spindle, n*—spindle in which several (4 to 6) rectangular vanes are attached to the shaft giving the appearance of a cross or star when viewed from the end.

3.1.1 *Discussion*—A vane spindle can be immersed in a specimen without destroying the shear-sensitive structure.

3.2 *yield stress, n*—the critical stress at which a material goes from being a deformable solid to showing fluid-like behavior.

3.2.1 *Discussion*—Examples of such fluids include many paints and pigment pastes and certain food materials such as ketchup.

## 4. Summary of Test Methods

4.1 *Test Method A* uses a viscometer with coaxial cylinder, cone/plate, or plate/plate geometry running a several different low rotational speeds. The materials is sheared at each speed and a shear stress value is measured. By plotting shear stress versus shear rate, a dynamic yield stress value is determined by extrapolating the data curve to zero shear rate. "Dynamic" indicates that the material has been allowed to flow and that the yield stress value is mathematically calculated by using a best-fit line through the measured data points.

4.2 *Test Method B* uses a controlled stress rheometer to determine a yield stress value. This can be done more readily with cone/plate or plate/plate geometry, but can also be accomplished with coaxial cylinder geometry. The rheometer applies a stress ramp to the material, starting at zero and increasing to a preset stress value above the yield stress of the material. As the torque applied to the spindle increases, the spindle will start to move when the yield stress in the material is exceeded. The stress reading at the onset of spindle rotation is the yield stress value for the material.

4.3 *Test Method C* uses a rotational viscometer or rheometer with a vane spindle immersed in the material. The vane spindle is rotated slowly at a fixed speed and the torque value is recorded continuously. The yield stress value is determined when the torque value reaches a maximum.

## 5. Significance and Use

5.1 The yield stress of a material is a measure of the amount of force required to initiate movement of that material in a pipe, through a pump, or from nozzle. The yield stress also characterizes the ability of the material to maintain particles in suspension. Along with viscosity measurements, yield stress measurements have been useful in establishing root causes of flow problems such as excessive orange peel and sagging and in explaining resistance to such problems. After a coating has

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

been applied, flow and leveling tends to be inversely related to yield stress and sag resistance tends to be directly related to yield stress. The ability of an automotive basecoat to keep aluminum and/or mica flakes oriented has been related to yield stress (direct relationship).

## 6. Apparatus

6.1 *Multi-speed Rotational Viscometer*—with coaxial (concentric) cylinder (either built-in or as an attachment), cone/plate, or plate/plate geometry or with a vane spindle.

6.2 *Controlled Stress Rheometer*—with cone/plate or plate/plate geometry.

## 7. Sampling

7.1 Take a representative sample of the product to be tested in accordance with Practice D3925. Minimal disturbance to the sample is important since the yield stress property is indicative of the material's physical structure when at rest.

## 8. Preparation of Specimen

8.1 Fill the coaxial cylinder with the proper amount of material. This is dependent on the spindle and chamber combination in use. Alternatively, for cone/plate or plate/plate place the proper amount of material on the plate.

8.2 Allow material to equilibrate to temperature if required.

8.3 For testing with vane spindle, insert the vane directly into the material. Allow the material to equilibrate to temperature beforehand if required.

8.4 The sample being tested experiences some handling prior to the start of the test. Therefore, there may be some adverse impact on the specimen structure that could affect the test results. Keeping to a specific step by step procedure for handling of specimens is very important in order to achieve repeatable results.

## 9. Procedure

9.1 *Test Method A* determines the yield stress of a material mathematically using data from a rotational viscometer. Yield stress may be determined at several discrete temperatures if necessary. Allow the specimen to equilibrate to the desired temperature before proceeding with the test. Coaxial cylinder, cone/plate, or plate/plate geometry may be used to measure shear stress versus shear rate. A yield stress value is determined by extrapolating the data for the measured shear stress values to zero shear rate. A best fit line applied to the accepted mathematical procedure for determining the stress value at which the material yields.

9.2 *Test Method B* determines yield stress based on a controlled stress ramp being applied to a material. Yield stress values may be determined at several discrete temperatures if necessary. Cone/plate or plate/plate geometry is typically used because the specimen undergoes minimal disturbance when it is placed on the plate. When using coaxial cylinder geometry, the spindle is immersed causing disturbance to the material structure; therefore, time must be allowed for the specimen to reestablish its structure before proceeding with the test. The smaller sample size required for cone/plate or plate/plate geometry permits rapid determination of the yield stress value because temperature equilibration of the specimen is quickly established. The stress ramp applied to the material goes from zero to a preselected maximum value which exceeds the yield stress of the material. The rate at which the stress ramp is applied may have an effect on the measured yield stress value. The torque applied to the spindle gradually increases until rotation of the spindle commences. Once the spindle starts to rotate, the torque value at that instant is converted into a yield stress value.

9.3 *Test Method C* uses a viscometer or rheometer with a vane spindle to measure yield stress. Yield stress values may be determined at several discrete temperatures if necessary by conditioning the material appropriately. The advantage of this method is that the material can be tested in the container in which it is packaged, such as a can of paint or ink. The vane spindle is immersed directly into the material with minimal disturbance to the sample. The spindle is rotated at a slow fixed rotational speed. Torque is measured continuously until a maximum value is achieved. The yield stress is calculated based on the maximum torque value and the geometry of the vane spindle used. The choice of low rotational speed may affect the measured yield stress value. A faster speed may result in a higher yield stress value.

## 10. Report

10.1 Report the following information:

10.1.1 Reference to this test method and the viscometer or rheometer model and the spindle geometry,

10.1.2 Type and identification of the product under test,

10.1.3 Test method used,

10.1.4 The control parameters for the instrument such as spindle speed (shear rate) or spindle stress (shear stress),

10.1.5 If Test Method C was used, report the rate at which the stress ramp was applied,

10.1.6 The measured yield stress value, and

10.1.7 Temperature of the specimen.

## 11. Precision and Bias

11.1 *Precision*—At this time, no precision data is available. Interlaboratory testing for the purpose of developing a precision statement will be attempted. Repeatability (using fresh specimens from a given sample) will be possible to establish, but meaningful Reproducibility is not likely to result from such testing. This is because yield stress values of materials vary so much depending on the handling, storage and shear history of the sample. Because of this, few if any comparisons of yield stress for a given material are ever made between laboratories.

11.2 *Bias*—Since there is no accepted reference material suitable for determining the bias values for the procedures in this standard, bias has no and will not be determined.

## 12. Keywords

12.1 coaxial cylinder geometry; cone/plate geometry; controlled stress rheometer; rotational speed; rotational viscometer; shear rate; shear stress; vane spindle geometry; yield stress