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Standard Test Method for Viscoelastic Properties of Paste Ink Vehicle Using an Oscillatory Rheometer[1]

This standard is issued under the fixed designation D7271; 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 covers the procedure for determining the viscoelastic properties of printing ink vehicles by measuring the G', G", and tan delta using a controlled strain cone and plate oscillatory rheometer.

1.2 This test method provides the flexibility of using several different types of rheometers to determine viscoelastic properties in ink vehicles.

1.3 This test method is not intended for systems that are volatile at procedure temperatures as evaporation may occur effectively changing the percent solids before testing is finished and significantly altering the rheology.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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]

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:* [3]

3.1.1 *frequency sweep test, n*—most rheometers have programs specific for their instrument.

3.1.1.1 *Discussion*—The user provides a specified geometry, frequency range, strain % or oscillatory stress and temperature of the test. This test will produce the data required for this method.

3.1.2 *G', n*—the elastic (storage) modulus obtained from an oscillatory test represents the energy stored during each frequency cycle, where the stress is divided by the corresponding linear elastic strain.

3.1.3 *G", n*—the viscous (loss) modulus obtained from an oscillatory test represents the amount of energy lost during each frequency cycle or the imaginary part of the complex modulus (for shear).

3.1.4 *geometry, n*—the cone used in the test.

3.1.5 *shear strain, n*—relative deformation in shear; term often abbreviated to shear.

3.1.6 *shear stress, n*—the component of stress parallel to (tangential to) the area considered.

3.1.7 *strain, n*—the measurement of deformation relative to a reference configuration.

3.1.8 *tan delta*(δ), *n*—the ratio of G" (viscous modulus) to G' (elastic modulus).

3.1.9 *viscoelasticity, n*—the phenomena exhibited by a liquid when energy is applied and once the force is released, the liquid recovers towards its original state by means of stored energy.

4. Summary of Test Method

4.1 Apply the ink vehicle to the plate of a rheometer.

4.2 Select the geometry (cone) and set to the required gap to the plate.

4.3 Remove the excess vehicle with the ink spatula.

4.4 Set the required temperature, strain or stress and frequency range for the frequency sweep. (Most rheometers have a test sequence that can be pre-prepared.)

4.5 Allow five minutes for temperature calibration and the vehicle to relax to its lowest energy state.

NOTE 1—Do not run a pre-shear sequence as this may affect results.

4.6 Start the frequency sweep test.

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.37 on Ink Vehicles

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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.

[3] Many of the definitions came wholly or in part from "An Introduction to Rheology," H.A. Barnes, J.F. Hutton, and K. Walters, Elesevier, 1989.

5. Significance and Use

5.1 This test method has found acceptance in the lithographic ink industry in predicting rheological behavior of a vehicle under press conditions caused by extrusion, shear-thinning rollers and dot gain recovery.

5.2 This test method is restricted within the torque limitations and strain resolution of the rheometer used.

5.3 Results may not be reproducible if the vehicle is not homogenous.

6. Apparatus

6.1 *Oscillatory Rheometer,* capable of over 400 Newtons of force, running at the required frequency range, and controlling strain in the 5 to 10 % range, as well as computer and software capable of running this test.

6.2 *Cone,* preferably two cm and 2° (if the viscosity of the vehicle is too high for this geometry, other geometries may be used by agreement between customer and supplier).

6.3 *Spatula,* non-abrasive to administer vehicle to the instrument.

7. Reagents and Materials

7.1 Cleaning solvent.

7.2 Lint free rags or tissue.

8. Calibration and Standardization

8.1 Calibrate the instrument using the procedure recommended by the manufacturer.

9. Conditioning

9.1 Since paste ink vehicles are shear thinning, care is needed to ensure the test sample has not been sheared by stirring or kneading prior to testing.

9.2 Allow vehicle to sit a minimum of five minutes if the vehicle has experienced shear forces before testing.

10. Procedure

10.1 After the instrument has been calibrated and cleaned, separate the geometry to administer approximately 1 g of vehicle (or sufficient amount to completely fill geometry gap) to the center of the bottom plate.

NOTE 2—Do not leave air bubbles in the vehicle. If air-bubbles exist or sample does not fill entire geometry gap, either re-administer the sample or carefully try to eliminate them with the spatula.

10.2 Set the cone and plate to the required gap. Many of the rheometers will do this automatically.

10.3 Using the spatula, carefully trim around cone any excess vehicle that may have been pushed from under the cone.

10.4 Allow the vehicle to equilibrate to 25°C (or required temperature) for five minutes. (This is the test temperature that should be programmed into the instrument prior to starting.)

NOTE 3—This method suggests that no pre-shear sequence is run. This changes the rheology of the sample and though the pre-shear sequence may be reproducible, the resulting data is tampered by the pre-shear.

10.5 Open the Frequency Sweep Test on the computer attached to the instrument.

10.6 The frequency range should be 0.1 to 1.0 Hz in logarithmic ramp mode. Set the number of readings to 20 in a decade.

10.7 The strain should be 5 to 10 %. (If this instrument is a controlled-strain rheometer, set the strain at 5 %.)

10.8 Start the test as per instructions for the operation procedure of the rheometer.

10.9 The results will be over the entire frequency range tested. The only results of interest for this method will be at 1.0 Hz.

10.10 Record the G', G", and tan delta at 1.0 Hz.

10.11 Clean the instrument.

11. Report

11.1 Report test data along with temperature, geometry size, and frequency.

12. Precision and Bias

12.1 *Precision*—An interlaboratory study of the rheological data of two varnishes, one resin solution and one lithographic printing ink were run by 13 rheometers in eight laboratories.

12.1.1 The precision estimates here are based on one analyst in each laboratory performing duplicate determinations on each of the samples.

12.1.2 Practice E691 was used in developing these precision statements.

12.2 *Repeatability (Within-Laboratory)*—The 95 % repeatability of each sample is recorded in Table 1. The average deviation from average is 2.76 %.

12.3 *Reproducibility (Multilaboratory)*—The 95 % reproducibility of each sample is recorded in Table 2. The average deviation from average is 4.66 %.

12.4 *Bias*—The procedure in this test method has no bias because there is no standard calibration fluid for this test method.

13. Keywords

13.1 frequency sweep; G'; G"; oscillatory rheometer; printing ink vehicles; rheology; tan delta; viscosity

TABLE 1 Repeatability

	Average	Repeatability	% from Average
Varnish A			
G'	6933.5	106.2	1.53
G"	15548.5	326.0	2.10
tan delta	2.2	0.0	0.0
Varnish B			
G'	11677.3	279.5	2.39
G"	20191.5	347.1	1.72
tan delta	1.7	0.0	0.0
Resin Solution			
G'	2617.1	147.1	5.62
G"	6541.6	285.2	4.36
tan delta	2.5	0.2	8.0
Ink			
G'	3215.0	93.9	2.92
G"	5372.6	241.5	4.50
tan delta	1.7	0.0	0.0

TABLE 2 Reproducibility

	Average	Reproducibility	% from Average
Varnish A			
G'	6933.5	372.4	5.37
G"	15548.5	877.1	5.64
tan delta	2.2	0.0	0.0
Varnish B			
G'	11677.3	581.1	4.98
G"	20191.5	856.8	4.24
tan delta	1.7	0.0	0.0
Resin Solution			
G'	2617.1	233.5	8.92
G"	6541.6	433.7	6.62
tan delta	2.5	0.2	8.0
Ink			
G'	3215.0	211.3	6.57
G"	5372.6	300.1	5.59
tan delta	1.7	0.0	0.0