Designation: D8020 – 15 (Reapproved 2020)

Standard Test Method for Freeze-Thaw Viscosity Stability of Water-Based Inks and Ink Vehicles[1]

This standard is issued under the fixed designation D8020; 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 a procedure for evaluating the effect of freeze-thaw cycling on the properties of water-based inks and ink vehicles.

1.2 This test is based on a similar standard test for coatings; Test Method D2243.

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

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2243 Test Method for Freeze-Thaw Resistance of Water-Borne Coatings

D2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational Viscometer

D4212 Test Method for Viscosity by Dip-Type Viscosity Cups

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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

3. Summary of Test Method

3.1 The waterborne ink or ink vehicle is placed into two 237-mL plastic jars. One jar is stored at room temperature, while the other jar is subjected to cycles of freezing and thawing. After cycling, the ink or ink vehicle is examined for changes in viscosity and uniformity.

4. Significance and Use

4.1 When waterborne inks and ink vehicles are shipped during cold weather, these materials may experience cycles of freezing and thawing. This can damage the material rendering it unusable.

4.2 Cycles of freezing and thawing can cause more damage to waterborne inks or ink vehicles than when the inks or ink vehicles are subjected to steady freezing.

5. Apparatus

5.1 *Test Chamber,* a suitable cabinet, room, or enclosure space large enough to contain the specimens to be tested permitting at least 25 mm (1 in.) of air space between the sides of adjacent jars and capable of being maintained continuously at a temperature of –18°C (0°F).

NOTE 1—Although a variation of the test chamber temperature of ±2°C (3.5°F) is allowed, the test chamber temperature should be maintained as near –18°C (0°F) as practicable and the amount of variation should be recorded and reported.

5.2 *Viscometer,* a dip-type viscosity cup as described in Test Method D4212 or a Brookfield type viscometer as described in Test Methods D2196.

5.3 *Recirculating Water Bath,* capable of maintaining a temperature of 25 ± 1°C (77 ±1.8°F).

5.4 *Cylindrical Container,* with a capacity of 0.5-L (1-pt), 85 mm (3 ⅜ in.) diameter to contain the test specimen during testing.

5.5 *Spatula.*

6. Sampling and Test Specimens

6.1 Ensure that the bulk sample from which the jars are filled is well stirred and uniform, the containers used are clean,

[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.

and the lids are applied promptly to the jars to prevent evaporation losses. Prepare two specimens for testing by filling 0.5-L plastic jars with 400 mL of the ink or ink vehicle. Identify one as the test specimen and the other as the control specimen.

7. Procedure

7.1 Stir the specimen by hand and measure its viscosity in accordance with Test Methods D4212 or D2196.

7.2 Store the control specimen at room temperature.

7.3 Place the test specimen in the chamber maintained at –18°C (0°F) in such a manner that it does not touch the walls or bottom of the chamber and air circulates around it freely. Placing jars on racks that raise them off the bottom of the chamber or upon pieces of insulating board resting on the bottom is suggested. In the case of several test specimens, maintain a minimum of 25 mm (1 in.) of air space between adjacent jars and between jars and the chamber walls.

7.4 Remove the test specimen from the chamber after 17 h. Place it adjacent to the control specimen and allow it to stand for 5 h undisturbed at room temperature, followed by 2 h in a recirculating water bath for a complete freeze-thaw cycle of 24 h.

7.5 Examine both the test and control specimens for any evidence of settling, gelation, and coagulation.

7.6 Stir the test and control ink or ink vehicle by hand using a spatula. Stir carefully so as to avoid air entrainment and foam. Measure the viscosities in accordance with Test Methods D4212 or D2196. Record the temperature at which the viscosity is measured.

7.7 Repeat 7.2 – 7.6 for two additional freeze-thaw cycles, or as many as agreed on between cooperating laboratories or buyer and seller. If any test specimen more than doubles in viscosity compared to the control the test is stopped.

8. Interpretation of Results

8.1 The cycle is considered passed if the viscosity of the test specimen is less than double the viscosity of the control and the test specimen does not exhibit coagulation or gelation. If the viscosity of the test specimen more than doubles, the test is stopped and the specimen fails. If a test specimen passes three cycles, it is considered freeze-thaw stable.

9. Report

9.1 Report the following information:

9.1.1 The condition of the ink or ink vehicle in the jar in accordance with 7.5 after each cycle.

9.1.2 The change in viscosity between the test and control inks or ink vehicles in accordance with 7.6.

9.1.3 The number of cycles passed by the test specimen.

10. Precision and Bias[3]

10.1 The precision of this test method is based on an interlaboratory study of ASTM D8020, Test Method for Freeze-Thaw Viscosity Stability of Water-Based Inks and Ink Vehicles, conducted in 2013. A total of six laboratories tested five different samples, using two methods. Every "test result" represents an individual determination, and one participant was asked to report duplicate test results. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D01-1180.

10.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

10.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

10.1.1.2 Repeatability limits are listed in Tables 1-8.

10.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus 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 20.

10.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1180. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Brookfield Viscosity (cps)
Initial

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{X}$	s_r	S_R	r	R
Control 1	316.050	5.485	44.682	15.358	125.109
Control 2	340.633	6.928	57.275	19.399	160.370
Control 3	321.300	6.351	43.168	17.782	120.871
Control 4	318.133	6.351	41.722	17.782	116.822
Control 5	316.383	2.021	51.938	5.658	145.426

[A] The average of the laboratories' calculated averages, when duplicates were reported.

TABLE 2 Brookfield Viscosity (cps)
After 1 Cycle

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{X}$	s_r	S_R	r	R
Control 1	299.033	9.815	37.637	27.482	105.384
Test 1	283.883	6.640	41.566	18.591	116.384
Control 2	315.867	6.351	31.108	17.782	87.103
Test 2	338.717	3.753	37.497	10.508	104.992
Control 3	311.050	6.640	36.289	18.591	101.609
Test 3	342.633	4.619	35.549	12.933	99.538
Control 4	301.783	10.104	32.286	28.290	90.400
Test 4	352.550	3.753	35.062	10.508	98.175
Control 5	301.283	1.443	30.219	4.041	84.613
Test 5	419.717	2.598	74.624	7.275	208.947

[A] The average of the laboratories' calculated averages, when duplicates were reported.

TABLE 3 Brookfield Viscosity (cps)
After 2 Cycles

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{X}$	s_r	S_R	r	R
Control 1	306.633	2.598	46.984	7.275	131.555
Test 1	305.633	0.000	49.440	0.000	138.433
Control 2	320.550	1.443	40.122	4.041	112.343
Test 2	365.217	3.753	42.159	10.508	118.044
Control 3	312.717	0.866	38.818	2.425	108.691
Test 3	397.633	1.732	69.054	4.850	193.351
Control 4	305.050	3.753	37.384	10.508	104.676
Test 4	411.550	1.443	69.273	4.041	193.964
Control 5	312.133	2.887	38.025	8.083	106.470
Test 5	551.533	14.722	128.407	41.223	359.541

[A] The average of the laboratories' calculated averages, when duplicates were reported.

TABLE 4 Brookfield Viscosity (cps)
After 3 cycles

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{X}$	s_r	S_R	r	R
Control 1	310.967	2.309	54.256	6.466	151.916
Test 1	316.550	2.021	50.095	5.658	140.267
Control 2	328.383	5.485	46.462	15.358	130.092
Test 2	383.717	7.794	48.622	21.824	136.140
Control 3	319.800	1.155	49.092	3.233	137.458
Test 3	426.217	3.753	60.506	10.508	169.415
Control 4	314.633	0.577	47.073	1.617	131.805
Test 4	467.300	9.815	79.299	27.482	222.037
Control 5	319.967	1.732	46.271	4.850	129.559
Test 5	615.633	14.434	110.186	40.415	308.520

[A] The average of the laboratories' calculated averages, when duplicates were reported.

10.1.2.2 Reproducibility limits are listed in Tables 1-8.

10.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

10.1.4 Any judgment in accordance with statements 10.1.1 and 10.1.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of materials tested and laboratories reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. The repeatability limit and the reproducibility limit should be considered as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.