Designation: D7149 – 05 (Reapproved 2016)

Standard Practice for Determining the Freeze Thaw Stability of Adhesives[1]

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1. Scope

1.1 This practice determines the ability of an adhesive to withstand repeated freezing and thawing cycles at specific temperatures without any significant change in physical properties.

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

1.3 *This practice 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 practice to establish appropriate safety and health practices and determine the applicability of regulatory limitation prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D907 Terminology of Adhesives

D1084 Test Methods for Viscosity of Adhesives

D1293 Test Methods for pH of Water

D2556 Test Method for Apparent Viscosity of Adhesives Having Shear-Rate-Dependent Flow Properties Using Rotational Viscometry

3. Terminology

3.1 *Definitions:*

3.1.1 Many of the terms utilized in this practice are defined in Terminology D907.

4. Significance and Use

4.1 This practice will provide a procedure for determining the stability of an adhesive to withstand five repeated cycles, or as otherwise specified, of freezing and thawing at a specific temperature.

4.2 This practice will allow the user to determine whether or not the adhesive can be appropriately applied after cycling of freezing and thawing.

4.3 This practice will identify any significant changes in the manufactured viscosity of the product after five cycles, or as otherwise specified, of freezing and thawing at a specific temperature.

4.4 This practice will help determine if the pH of adhesive has changed significantly due to the freezing and thawing cycles.

5. Apparatus

5.1 A 1.0 L (1 qt) container, or larger, with lid. Other container sizes may be applicable for some adhesives.

5.2 Freezer capable of maintaining -18 ± -2°C (0.0 ± -5°F) to -7 ± -2°C (20 ± -5°F).

5.3 Brookfield RVT Viscometer 1-spindle TC at 5 – 20 rpms, Heliopath stand.

NOTE 1—Other viscometer models, their speed and spindle size, may be utilized as long as they comply to Test Methods D1084 or D2556 and are recommended by the manufacturer for such use.

5.4 pH meter.

5.5 Tongue depressor or similar.

6. Conditioning

6.1 Condition the adhesive to be tested 24 h at 23 ± -2°C (73 ± -4°F) with a relative humidity if 50 ± -5 % prior to testing.

7. Procedure

7.1 Fill the container with adhesive to be tested.

7.2 Check the viscosity of the adhesive after it is transferred into the container, as defined in Test Methods D1084 (Method B) or Test Method D2556.

7.3 Check the pH of the adhesive after it is transferred into the container, as defined in Test Method D1293.

7.4 Close the container with lid and place it into the freezer at the specified temperature to be tested for a period of not less than 16 h.

7.5 Remove container from freezer after the appropriate time limit and allow to thaw until the adhesive reaches room

[1] This practice is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.10 on Working Properties.

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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.

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temperature as defined in 6.1. It is recommended that a constant temperature oven with a temperature set at 24 ± -2°C (75 ± -4°F) be used for the thawing procedure. This constitutes one freeze / thaw cycle. Remove container lid by appropriate means; if the adhesive forms a surface film remove with the tongue depressor without stirring contents.

7.6 Insert clean tongue depressor about one inch into the adhesive and remove slowly.

7.7 If the adhesive "strings" from the tongue depressor or appears gelled, the sample has failed the test. Report which particular cycle displayed a change in the physical properties or that had failed.

7.8 If adhesive appears to be of normal consistency, measure viscosity and pH and record.

7.9 Repeat steps 7.3 – 7.8 for a total of five cycles or until a change in physical properties or failure is noted.

8. Reporting

8.1 Report the following information:

8.1.1 Type of adhesive.

8.1.2 Temperature at which product was frozen and tested.

8.1.3 Initial viscosity and pH of adhesive.

8.1.4 Viscosity and pH of the individual freeze thaw cycles or if applicable after which cycle product was no longer classified as usable.

9. Precision and Bias

9.1 The precision and bias of this practice for measuring viscosity is essentially as specified in Test Method D1084. Other results produced from this practice are non-quantitative.

10. Keywords

10.1 freeze; stability; thaw

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