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Standard Practice for Polyurethane Raw Materials: Polyurethane Foam Cup Test¹

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1. Scope*

1.1 This practice covers the determination of cream time (initiation time), top of cup time, free rise time, free rise height, string gel time (pull time), tack free time, settle back, and free rise density of polyurethane foam formulations using a cup foam test.

1.2 Typical definitions, terms, and techniques are described; including procedures for mixing and transferring samples to the foaming container; and data gathering and evaluation. However, agreement between the customer and the testing laboratory for all these items must be obtained prior to use.

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

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.*

NOTE 1—There is no known ISO equivalent to this standard.

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

[D883 Terminology Relating to Plastics](#)

[E456 Terminology Relating to Quality and Statistics](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

[E2935 Practice for Evaluating Equivalence of Two Testing Processes](#)

3. Terminology

3.1 *Definitions—Terminology* Terms used in this practice follows that standard are defined in accordance with Terminology [D883](#), unless otherwise specified. For terms relating to precision and bias and associated issues, the terms used in this standard are defined in accordance with Terminology [E456](#).

¹ This practice is under the jurisdiction of ASTM Committee [D20](#) on Plastics and is the direct responsibility of Subcommittee [D20.22](#) on Cellular Materials - Plastics and Elastomers.

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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.

*A Summary of Changes section appears at the end of this standard

3.2 Definitions of Terms Specific to This Standard:

~~3.2.1 cream time (initiation time)—time, n—the time between the start of mixing and the point at which fine bubbles begin to appear.~~

~~3.2.2 final height, n—height of foam after specified time.~~

~~3.2.3 top of cup free rise density, time—n—the time at which density in kg/m^3 the crown of the rising foam reaches the plane of the top of the of a polyurethane foam prepared in an open cup.~~

~~3.2.4 free rise height, n—height of the foam at free rise time.~~

~~3.2.5 free rise time (end of rise time)—time, n—the time at which the foam stops expanding as observed visually.~~

~~3.2.6 resin blend (formulated polyol), n—complete ingredient formulation without the isocyanate.~~

~~3.2.7 % settle back (% recession, % sigh back, or % sink back), n—percentage decrease from free rise height to final height.~~

~~3.2.8 string gel time (pull time)—time, n—time at which long “strings” of tacky material can be pulled away from the surface of the foam when the surface is touched by the edge of a tongue depressor or similar implement.~~

~~3.2.9 tack free time—time, n—the time at which the surface of the foam can be touched with a gloved finger or tongue depressor without sticking.~~

~~3.2.10 free rise density—top of cup time, n—the density in kg/m^3 of a polyurethane foam prepared in an open the crown of the rising foam reaches the plane of the top of the cup.~~

~~3.2.7 free rise height—height of the foam at free rise time~~

~~3.2.8 final height—height of foam after specified time~~

~~3.2.9 % settle back (% recession, % sigh back, or % sink back)—percentage decrease from free rise height to final height~~

~~3.2.10 resin blend (formulated polyol)—complete ingredient formulation without the isocyanate~~

4. Summary of Practice

4.1 Specific events (cream time, initiation time, top of cup time, free rise time, free rise height, string gel time, and tack free time) from a polyurethane foamed in a cup are measured to verify the resin blend composition or levels of ingredients in formulations used to make polyurethane foams.

4.2 An estimation of the method precision is given. An estimation of bias is not given because there is no suitable reference method.

5. Significance and Use

5.1 General Utility:

5.1.1 It is useful to verify catalyst levels in a resin blend or a polyurethane system.

5.1.2 This practice is suitable for research, quality control, specification testing, and process control.

5.2 Limitations:

5.2.1 Several of the measured parameters are subjective. Therefore, operator-to-operator variability and lab-to-lab variability can be much higher than that of a single operator.

5.2.2 The variability of this practice is dependent on the consistency of mixing of the reactants.

5.2.3 The estimation of precision in this practice is based on typical formulations for rigid and flexible foams. Formulations with faster reaction times will likely have greater variability, particularly cream time (initiation time). Formulations with slower reaction times will likely have greater variability in the measurement of free rise time.

5.2.4 It is possible that low-level (ppm, ppb) ingredient contamination will not be detectable using this practice. Confirmation of such contamination will potentially require large-scale (~~~20 liters~~) (~20 L) tests and is out of the scope of this practice.

6. Apparatus

6.1 *Stirrer for Mixing:*

6.1.1 Stirring motor,

6.1.2 Propeller or other type of mixing apparatus, and

6.1.3 Stainless steel shaft.

6.2 *Cups*

6.3 *Knife*—Cutting length of the blade must be large enough to cleanly cut through the risen foam at the top of the cup.

6.4 *Thermometers*—Accurate to $\pm 0.5^{\circ}\text{C}$; $\pm 0.5^{\circ}\text{C}$.

6.5 *Stopwatch*—Capable of measuring to 0.1 s.

6.6 *Balance*—Capacity of 100 g and capable of weighing to the nearest 0.1 g.

6.7 *Tachometer*—Capable of measuring to the nearest rpm.

6.8 *Ruler*—Capable of measuring to the nearest 1 mm.

7. Test Conditions

7.1 Since isocyanates react with moisture, keep laboratory humidity low, preferably around 50 % relative humidity.

7.2 Some of the parameters are influenced by atmospheric pressure. The quantitative influence is not known at this time. If results are to be compared between laboratories located at significantly different elevations above sea level, it is advisable to measure the barometric pressure and develop a suitable, empirical correction factor. (**Warning**—Diisocyanates are eye, skin, and respiratory irritants at concentrations above the occupational exposure limit (TLV or PEL). Diisocyanates can cause skin and respiratory sensitization (asthma) in some people. Once sensitized, it is essential to limit further exposure to diisocyanates. Use a combination of engineering controls and personal protective equipment, including respiratory, skin and eye protection, to prevent over-exposure to diisocyanates. Consult the product suppliers' Safety Data Sheet (SDS) for more detailed information about potential health effects and other specific safety and handling instructions for the ~~product~~product.)

8. Procedure

8.1 Check the stirrer speed with a tachometer and rotation direction in air with no load and turn the stirrer off after checking.

8.2 Weigh an empty cup and record the weight.

8.3 Weigh reactants into the cup in accordance with previously established order of addition.

NOTE 2—Ensure that the temperature of the reactants is as specified prior to use.

NOTE 3—This cup is then nested into another cup to prevent spilling chemicals in case the propeller cuts the first cup. Some practitioners use plastic cups with thicker walls and omit the second cup.

NOTE 4—If users elect to mix in one cup and pour into a second cup, results may vary.

8.4 Immerse the stirrer blade completely to a starting position in contact with the bottom of the cup and retract it slightly.

8.5 Simultaneously, turn on the mixer switch, and start the stopwatch.

NOTE 5—In some cases, it is better to ramp up the mixer speed. Such modifications need to be established prior to testing.

8.6 Mix the materials for a specified time then, remove the cup from the spinning mixing blade.

NOTE 6—Care must be taken to remove the cup slowly so that splashing of the reaction mixture does not occur. If appropriate, stop the mixer at the end of the specified time and then remove the cup.

8.7 Place the cup in a fume hood and record the times as defined in Section 3.

8.8 Clean the stirrer blade thoroughly after each test.

8.9 After a specified time, cut off the crown of the foam as close to the top edge of the paper cup as possible.

8.10 Record the weight of the cup plus remaining foam to the nearest 0.1 g.

9. Calculation

9.1 Calculate the Free Rise Density and express as kg/m^3 .

9.1.1 An example of how Free Rise Density can be determined is as follows:

9.1.1.1 Calculate the weight of the foam in the cup as follows:

$$W_{(foam)} = [W_{(cup + cut\ foam)} - W_{(empty\ cup)}]$$

~~where:~~

~~where:~~

~~$\frac{W_{(foam)}}{W_{(cup + cut\ foam)}}$ = weight in grams of the remaining foam in the cup~~

~~$\frac{W_{(cup + cut\ foam)}}{W_{(empty\ cup)}}$ = weight in grams of the cup plus remaining foam~~

~~$\frac{W_{(empty\ cup)}}{W_{(empty\ cup)}}$ = weight in grams of the empty cup~~

~~$\frac{W_{(foam)}}{W_{(cup + cut\ foam)}}$ = weight in grams of the remaining foam in the cup,~~

~~$\frac{W_{(cup + cut\ foam)}}{W_{(empty\ cup)}}$ = weight in grams of the cup plus remaining foam, and~~

~~$\frac{W_{(empty\ cup)}}{W_{(empty\ cup)}}$ = weight in grams of the empty cup.~~

9.1.1.2 Calculate the Free Rise Density as follows:

$$\text{Free Rise Density } (\text{kg/m}^3) = g/L = W_{(foam)} / V_{(cup)}$$

~~where:~~

~~where:~~

~~$\frac{W_{(foam)}}{W_{(foam)}}$ = weight in grams of the remaining foam in the cup~~

$V_{(cup)}$ = the volume of the cup in litres

$\frac{W_{(foam)}}{V_{(cup)}}$ \equiv weight in grams of the remaining foam in the cup, and
 \equiv the volume of the cup in litres.

9.1.2 An example of how to calculate % Settle Back as follows:

$$\% \text{ Settle Back} = (H_{rise} - H_{final}) \times 100 \% / H_{rise}$$

where:

where:

H_{rise} = free rise height of foam in millimeters

H_{final} = final foam height in millimeters

H_{rise} \equiv free rise height of foam in millimeters, and

H_{final} \equiv final foam height in millimeters.

10. Report

10.1 Report all time results as previously agreed, typically to the nearest whole second.

10.2 Report Free Rise Density as previously agreed, generally, Free Rise Density is reported to two decimal places.

10.3 Report % Settle Back as previously agreed, generally to the nearest percent.

11. Precision and Bias

11.1 *Precision*—Attempts to develop a precision and bias statement for this practice have not been successful. For this reason, only estimates of data on precision and bias can be given. Because this practice does not contain an acceptable numerical precision and bias statement, it shall not be used as a referee test method in case of dispute. Anyone wishing to participate in the development of precision and bias data is to contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

12. Keywords

12.1 cream time; cup test; foam testing; formulated polyol; free rise density; free rise height; free rise time; initiation time; isocyanate; polyurethane; pull time; raw material; resin blend; settle back; sigh back; sink back; string gel time; tack free time; top of cup time

APPENDIX

(Nonmandatory Information)

X1. EXAMPLE OF CUP FOAM TEST CONDITIONS FOR POLYURETHANE FOAM FORMULATIONS

X1.1 Scope

X1.1.1 Information provided in this non-mandatory appendix is given to provide the reader with a more-specific example of test conditions as defined by one company. Other acceptable test conditions are possible.

X1.2 Referenced Documents

X1.2.1 Referenced documents for this appendix are as listed in ~~Section 2.1.~~

X1.3 Terminology

X1.3.1 Terminology for this appendix are as defined in Section 3.

X1.4 Apparatus

NOTE X1.1—Users of this practice are encouraged to identify suppliers and parts that meet their specific needs for this practice.

X1.4.1 *Stirring station*—Standard ServoDyne electronic mixer system, electronic digital timer, available from Cole Parmer Instrument Company, 625 East Bunker Ct., Vernon Hills, IL 60061, United States, <http://www.coleparmer.com/>.

X1.4.1.1 *Three Blade Propeller*—3-in. diameter by $\frac{5}{16}$ -in. diameter bore.

X1.4.1.2 *316 Stainless Steel Shaft*—12-in. long by $\frac{5}{16}$ -in. diameter, cut to 7-in. length.

X1.4.2 *Paper Cups*—63 ~~oz.~~oz unwaxed paper buckets.

X1.4.3 *Electric Knife*—Cutting length of the blade must be greater than the upper inner diameter of the cup.

X1.4.4 *Thermometers*—Accurate to $\pm 0.5^{\circ}\text{C}$; $\pm 0.5^{\circ}\text{C}$.

<https://standards.iteh.ai/catalog/standards/astm/fbfab423-8549-4389-a6be-cba1b5aa97fe/astm-d7487-24>
X1.4.5 *Beakers*—Polypropylene, 50-mL and 250-mL volumes.

X1.4.6 *Stop Watch or timer*—Preferably with greater than five memories.

X1.4.7 *Tongue Depressors*— $\frac{3}{4}$ in. by 6 in.

X1.4.8 *Balance*—Capacity of 100 g and capable of weighing to the nearest 0.1 g.

X1.5 Reagents

X1.5.1 *Methylene Chloride or Acetone*: ~~Reagent~~—Reagent grade.

X1.6 Procedure