Designation: D7487 – 13

Standard Practice for Polyurethane Raw Materials: Polyurethane Foam Cup Test[1]

This standard is issued under the fixed designation D7487; 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 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 and health practices and determine the applicability of regulatory limitations prior to use.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

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

3. Terminology

3.1 *Definitions*—Terminology used in this practice follows that defined in Terminology D883.

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.2 *top of cup time*—the time at which the crown of the rising foam reaches the plane of the top of the cup.

3.2.3 *free rise time (end of rise time)*—the time at which the foam stops expanding as observed visually.

3.2.4 *string gel time (pull time)*—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.

3.2.5 *tack free time*—the time at which the surface of the foam can be touched with a gloved finger or tongue depressor without sticking.

3.2.6 *free rise density*—the density in kg/m^3 of a polyurethane foam prepared in an open 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:*

[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.

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 litres) 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 ±0.5°C.

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

6.6 *Balance*—capacity of 100 g and capable of weighing to 0.1 g.

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

6.8 *Ruler*—capable of measuring to 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—**Many diisocyanates are known or suspected sensitizers. Over-exposure to diisocyanates can lead to adverse health effects which may include the development of occupational asthma and other respiratory, skin, and eye effects. Engineering controls, or personal protective equipment, or both, including respiratory, skin, and eye protection, are to be used when there is a potential for over-exposure to diisocyanates. The product suppliers' Safety Data Sheet (SDS) provides more detailed information about potential adverse health effects and other important safety and handling information. Always follow the specific instructions provided on the SDS.)

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:

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

where:

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

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

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

9.1.1.2 Calculate the Free Rise Density as follows:

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

where:

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

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

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

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

where:

H_{rise} = free rise height of foam in millimetres
H_{final} = final foam height in millimetres

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—Specific suppliers and part numbers are provide below as examples and are based upon one company's experience. Other suppliers are available. 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 system (catalog #J-4440-00), electronic digital time (115 volt AC; catalog #J-8683-50), available from Cole Parmer Instrument Co., Int'l. Sales Div., 7425 N. Oak Park Avenue, Niles, IL 60714.

X1.4.1.1 *Three Blade Propeller*—3-in. diameter by 5⁄16-in. diameter bore (catalog #J-4553-68), available from Cole Parmer Instrument Co.

X1.4.1.2 *Stainless Steel Shaft*—12-in. long by 5⁄16-in. diameter, cut to 7-in. length (catalog #J-4553-50), available from Cole Parmer Instrument Co.

X1.4.2 *Paper Cups*—1 L unwaxed, *Dixie* cup No. 2195, available from Dixie/Marathon.

X1.4.3 *Electric Knife*—Black & Decker Slim Grip EK100 Type 1, available from Black & Decker Inc., Shelton, CT 06484. Cutting length of the blade must be greater than the upper inner diameter of the cup.

X1.4.4 *Thermometers*—accurate to ±0.5°C, available from Fisher Scientific, 711 Forbes Avenue, Pittsburgh, PA 15219.

X1.4.5 *Tripour Beakers*—polypropylene, 50-mL and 250-mL volumes, available from Fisher Scientific.

X1.4.6 *Stop Watch*—6 memory Accusplit 760XM, available from M. Ducommun Co., Inc., 58 Main Street, Warwick, NY 10990.

X1.4.7 *Tongue Depressors*—¾ in. by 6 in., *Puritan* brand, available from Fisher Scientific.

X1.4.8 *Balance*—capacity of 100 gm and capable of weighing to 0.1 gm.

X1.5 Reagents

X1.5.1 *Methylene Chloride or Acetone*: reagent grade, available from Aldrich Chemical Co., Inc., 1001 W. St. Paul Avenue, PO Box 355, Milwaukee, WI.

X1.6 Procedure

X1.6.1 Check the stirrer speed with a tachometer and rotation direction in air with no load. Set the stirrer speed at 3000 rpm and the rotation such that the flow of air is downward. Turn the stirrer off.

X1.6.2 Weigh a cup and record the weight. Nest this cup into another cup.

X1.6.3 Wet-tare a 50-mL Tripour beaker with isocyanate. (Fill the empty Tripour beaker to the weight of 23.0 g for a rigid foam or 16.0 g for a flexible foam. Quickly pour the isocyanate into a waste container and drain the contents for three seconds. Set the Tripour beaker upright and tare the wet beaker.) This procedure approximately compensates for the amount of isocyanate that will not drain from the beaker when the isocyanate sample is transferred to the tared cup containing the polyol.

X1.6.4 Add the required amount of isocyanate, 23.0 g for a rigid foam or 16.0 g for a flexible foam to the wet-tared beaker and record the weight to the nearest 0.1 g.

X1.6.5 Add the required amount of resin blend, 20 g for a rigid foam or 35 g for a flexible foam, to the nested cups and record the weight to the nearest 0.1 g. Start mixing within one minute of adding the resin blend to the cup.

X1.6.6 Pour the isocyanate for three seconds into the paper cups containing the resin blend.

X1.6.7 Immerse the stirrer blade completely to a starting position near the bottom of the cup.

X1.6.8 Simultaneously, turn on the mixer switch, and start the stopwatch. Mix the materials for five seconds. At the end of five seconds, remove the cup from the spinning mixing blade.

X1.6.9 Place the cup in a fume hood and observe. Record the characteristic times as defined in Section 3.

X1.6.10 Clean the stirrer blade thoroughly after each test, with methylene chloride at 300 rpm.

X1.6.11 Approximately 15 minutes after the start of mixing, use an electric knife to cut off the crown of the foam as close to the top edge of the paper cup as possible.

X1.6.12 Record the weight of the cup plus remaining foam to the nearest 0.1 g.

X1.7 Calculations

X1.7.1 Calculations for this appendix are as defined in Section 9.

X1.8 Precision and Bias

X1.8.1 A limited round robin was performed with four laboratories within a single company. Tables X1.1-X1.6 are based on this round robin which was conducted in 2004 and are in general accordance with Practice E691, involving two materials tested by two operators in each of four laboratories. Each laboratory made duplicate determinations on each material on each of two days. (**Warning—**The following explanations of *r* and *R* (X1.8.1.1 and 6.4) are only intended to present a meaningful way of considering the approximate precision of this practice. The data in Tables 1-6 must not be rigorously applied to the acceptance or rejection of material, as those data are specific to the limited round robin and will not be representative of other lots, conditions, materials, and laboratories. Users of this practice must apply the principles outlined in Practice E691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of X1.8.1.1 and 6.4 would then be valid for such data.)

TABLE X1.1 Estimated Precision Cream Time (Initiation Time)

Initiation time in seconds						
Material	Average	S_r[A]	S_R[B]	r[C]	R[D]	n[E]
rigid resin blend	6.8	0.85	1.60	2.34	4.48	4
flexible resin blend	11.4	0.93	2.57	2.60	7.20	4

[A] S_r = within-laboratory standard deviation of the replicates.
[B] S_R = between-laboratory standard deviation of the averages.
[C] r = within-laboratory repeatability limit = 2.8*S_r.
[D] R = between-laboratory reproducibility limit = 2.8*S_R.
[E] n = number of laboratories contributing valid data for this material.

TABLE X1.2 Estimated Precision for Top of Cup Time (TOC)

Top of Cup Time in seconds						
Material	Average	S_r[A]	S_R[B]	r[C]	R[D]	n[E]
rigid resin blend	21.9	1.57	10.7	4.38	29.9	3
flexible resin blend	46.4	3.24	5.13	9.07	14.4	3

[A] S_r = within-laboratory standard deviation of the replicates.
[B] S_R = between-laboratory standard deviation of the averages.
[C] r = within-laboratory repeatability limit = 2.8*S_r.
[D] R = between-laboratory reproducibility limit = 2.8*S_R.
[E] n = number of laboratories contributing valid data for this material.

TABLE X1.3 Estimated Precision for String Gel Time (Pull Time)

String Gel Time in seconds						
Material	Average	S_r[A]	S_R[B]	r[C]	R[D]	n[E]
rigid resin blend	32.7	4.51	4.65	12.6	13.0	3
flexible resin blend	71.9	1.44	9.87	4.02	27.6	3

[A] S_r = within-laboratory standard deviation of the replicates.
[B] S_R = between-laboratory standard deviation of the averages.
[C] r = within-laboratory repeatability limit = 2.8*S_r.
[D] R = between-laboratory reproducibility limit = 2.8*S_R.
[E] n = number of laboratories contributing valid data for this material.

TABLE X1.4 Estimated Precision for Tack Free Time

Tack Free Time in seconds						
Material	Average	S_r[A]	S_R[B]	r[C]	R[D]	n[E]
rigid resin blend	47.6	7.50	13.3	21.0	37.2	4
flexible resin blend	113	18.1	20.0	50.5	56.1	3

[A] S_r = within-laboratory standard deviation of the replicates.
[B] S_R = between-laboratory standard deviation of the averages.
[C] r = within-laboratory repeatability limit = 2.8*S_r.
[D] R = between-laboratory reproducibility limit = 2.8*S_R.
[E] n = number of laboratories contributing valid data for this material.

TABLE X1.5 Estimated Precision for Free Rise Density (FRD)

Material	Average	S_r[A]	S_R[B]	r[C]	R[D]	n[E]
rigid resin blend	25.6	1.75	2.66	4.91	7.46	3
flexible resin blend	83.0	3.39	3.39	9.49	9.49	3

[A] S_r = within-laboratory standard deviation of the replicates.
[B] S_R = between-laboratory standard deviation of the averages.
[C] r = within-laboratory repeatability limit = 2.8*S_r.
[D] R = between-laboratory reproducibility limit = 2.8*S_R.
[E] n = number of laboratories contributing valid data for this material.

X1.8.1.1 *Repeatability, r*—(Comparing two results for the same material, obtained by the same operator using the same equipment.) It is estimated that the two results will be judged not equivalent if they differ by more than the *r* value for that material.

X1.8.1.2 *Reproducibility, R*—(Comparing two results for the same material, obtained by different operators using different equipment in different laboratories on different days.) It is estimated that the two results will be judged not equivalent if they differ by more than the *R* value for that material.

X1.8.1.3 Any judgment in accordance with X1.8.1.1 and 6.4 has an approximate 95 % (0.95) probability of being correct.

X1.8.2 *Bias*—The bias of this practice has not yet been determined.

TABLE X1.6 Estimated Precision for Free Rise Time

Free Rise Density in kg/m^3						
Material	Average	S_r[A]	S_R[B]	r[C]	R[D]	n[E]
rigid resin blend	62.1	6.89	10.7	19.3	30.0	4
flexible resin blend	123	10.9	18.4	30.6	51.5	4

[A] S_r = within-laboratory standard deviation of the replicates.
[B] S_R = between-laboratory standard deviation of the averages.
[C] r = within-laboratory repeatability limit = 2.8*S_r.
[D] R = between-laboratory reproducibility limit = 2.8*S_R.
[E] n = number of laboratories contributing valid data for this material.