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Standard Practice for Polyurethane Raw Materials: Gel Tests for Polyurethane Non-Foam Formulations¹

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1. Scope

1.1 This practice covers procedures for determining the gel times of polyurethane non-foam formulations using commercially available gel test equipment.

1.2 Definitions, terms, and techniques are described along with procedures for calculating sample weights.

1.3 The values stated in SI units are to be regarded as 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

3. Terminology

3.1 *Definitions*—For general definitions of terms used in this practice see Terminology **D883**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *gel point*—the extent of polymerization at which the upper limit, as defined by the procedure being run, is reached.

¹ This test method 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.

3.2.2 *gel time*—the time from the initiation of the reaction to the gel point.

3.2.3 *resin blend (formulated polyol)*—complete ingredient formulation without the isocyanate component.

3.2.4 *index*—the ratio of the equivalents of the isocyanate component to the equivalents of the resin blend of a polyurethane formulation.

4. Summary of Practice

4.1 The gel time of a polyurethane non-foam formulation is determined by measuring the time required for the viscosity of the polymerizing system to increase to a set level using a gel meter. It is recommended that the torque of the gel meter be verified with a gauge certified to NIST standards.

5. Significance and Use

5.1 *General Utility:*

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

5.1.2 It is useful to define and verify the reactivity of non-foam polyurethane formulations.

5.2 *Limitations:*

5.2.1 Operator-to-operator variability and lab-to-lab variability can be significant.

5.2.2 The variability of this practice is dependent on the equipment used to measure the gel time. It is recommended that the testing laboratory and the client agree on the equipment and the conditions to be used that include the following:

5.2.2.1 Gel Tester and gel point criteria,

5.2.2.2 Speed/rpm of the mixer,

5.2.2.3 Type and shape of the mix blades,

5.2.2.4 Size and type (for example, shape, lined or unlined) of container for mixing the components and for measuring the gel time, and

5.2.2.5 The volume (or height) of material to be placed in the container for measuring the gel time and the depth of the measuring wire or spindle of the gel tester from the bottom of the container.

5.2.3 Users of this practice shall develop their own precision data to determine if these procedures meet their requirements.

5.2.4 It is possible that low-levels (ppm, ppb) of contaminants will not be detected using this practice.

6. Sampling

6.1 Since organic isocyanates react with atmospheric moisture, take special precautions in sampling. Usual sampling methods, even when conducted rapidly, can cause contamination of the sample with insoluble urea. Therefore, blanket the sample with dry air or nitrogen at all times. (**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.)

7. Test Conditions

7.1 Isocyanate samples shall remain sealed against moisture until immediately before testing.

8. Equipment and Reagents

8.1 *Commercial gel meter* or other equipment capable of determining a large increase in viscosity that occurs in a polymerizing urethane system at or near the gel point.

8.2 *Balance*, capable of weighing to 0.1 mg.

8.3 *Containers* appropriate for the particular gel test being run.

8.4 *Stopwatch or electronic timer*.

8.5 *Heating device* as described in the specific procedure.

8.6 *Methyl ethyl ketone (MEK)* or other solvent appropriate for cleaning the equipment after use.

8.7 *Motorized mixer with blades* (or spatula for manual mixing).

8.8 *Two component non-foam polyurethane formulation* consisting of a resin blend as needed to obtain the desired gel time and an isocyanate component.

NOTE 2—Additional equipment may be listed in specific procedures in the Appendixes.

9. General Procedure

9.1 A general procedure is outlined below with examples of specific steps provided in the Appendixes.

9.1.1 Charge the container used in the gel test with a carefully weighed amount of the resin blend and the isocyanate being reacted. A 1.05 index is typical for gel tests but can be adjusted for a specific test or formulation.

9.1.2 Immediately start the timer or stopwatch and mix the components thoroughly (the timer is to be started at the beginning of the mix, that is, when the reaction is initiated).

9.1.3 Introduce the container with the reacting system to the gel meter and monitor the reaction. Because some formulations solidify, consider using disposable stirring paddles or probes.

9.1.4 Stop the timer when the reaction reaches the gel point. Many commercial units will shut off automatically at the defined gel point.

9.1.5 Record the gel time.

10. Sample Calculations

10.1 Calculate the equivalent weight, EW_p , of the resin blend as follows:

$$EW_p = \frac{100}{\frac{(OH + Ac)100}{56100} + \frac{\%W}{9}} \quad (1)$$

where:

OH = the hydroxyl number of the resin blend in milligrams of KOH per gram of sample,

Ac = the acid number of the resin blend in milligrams of KOH per gram of sample,

$\%W$ = the percent water in the resin blend,

56100 = the equivalent weight of KOH in milligrams of KOH per equivalent, and

9 = the equivalent weight of water in grams per equivalent.

10.2 Calculate the equivalents of the resin blend, EQ_p , as follows:

$$EQ_p = X_p / EW_p \quad (2)$$

where:

X_p = the amount of resin blend to be reacted with the isocyanate component in grams

10.3 Calculate the equivalents, EQ_i , of the isocyanate component needed to prepare the batch at an index of 1.05 as follows:

$$EQ_i = 1.05 \times EQ_p \quad (3)$$

where:

1.05 = the index of the batch

10.4 Calculate the equivalent weight, EW_i , of the isocyanate component as follows:

$$EW_i = \frac{42.02 \times 100}{\%NCO} \quad (4)$$

where:

$\%NCO$ = the percent by weight of NCO groups present in the sample and

42.02 = grams NCO per equivalent of NCO.

10.5 Calculate the weight, W_i , of isocyanate in grams needed to prepare the batch as follows:

$$W_i = EW_i \times EQ_i \quad (5)$$

11. Report

11.1 Report the gel time and the procedure used.

12. Keywords

12.1 gel time; isocyanate; polyol; polyurethane; raw material; resin blend

APPENDIXES
X1. EXAMPLES OF GEL TIME TEST PROCEDURES FOR POLYURETHANE NON-FOAM FORMULATIONS
(Nonmandatory Information)

X1.1 The information in these nonmandatory appendixes is given to provide the reader with examples of test conditions as defined by individual companies. Other acceptable test conditions are possible.

X1.2 Specific suppliers are provided below as examples. Other suppliers are available, and users of this practice are encouraged to identify suppliers and parts that meet their specific needs.

X1.3 Test Procedure A is used to determine the gel time of

a polyester polyol with an isocyanate component using a Sunshine Gel Meter.

X1.4 Test Procedure B is used to determine the gel time of an isocyanate component with a resin blend using a Gardco Gel Tester.

X1.5 Test Procedure C is used to determine the gel time of a two-component elastomer system using a Shyodu Gel Timer and includes information on verifying the torque of the Shyodu Gel Timer with a desktop IMADA DTX-15B Torque Tester.

X2. TEST PROCEDURE A: GEL TIME OF A POLYESTER POLYOL WITH AN ISOCYANATE COMPONENT USING A SUNSHINE GEL METER
X2.1 Scope

X2.1.1 This procedure determines the reactivity of a polyester polyol by measuring the gel time of a non-foam polyurethane formulation using a Sunshine Gel Meter.

X2.2 Summary of Practice

X2.2.1 A polyester polyol is reacted with an isocyanate and the reactivity is determined by measuring the time required for the viscosity of the polymerizing system to increase to a set level using a gel meter.

X2.3 Equipment and Chemicals

- X2.3.1 *Gel meter*, Sunshine Scientific Instruments.
- X2.3.2 *Balance*, analytical, 0.01-g readability.
- X2.3.3 *Oven*, circulating air, 110°C.
- X2.3.4 *Baths*, oil, circulating high temperature.
- X2.3.5 *Stopwatch*.
- X2.3.6 *Methyl ethyl ketone (MEK)*.
- X2.3.7 *Polymeric MDI reagent*.
- X2.3.8 *Oil*, high temperature silicone bath.
- X2.3.9 *Test tubes*, 18-mm by 150-mm.
- X2.3.10 *Jar*, glass, 237-mL (8-oz).
- X2.3.11 *Spatula*.

X2.4 Procedure

X2.4.1 Weigh 50 g of polyester polyol into a 237-mL (8-oz) jar. Cover and place the jar in the 110°C oven for one hour.

X2.4.2 Set the oil bath to the desired temperature (example: For a polyester polyol with hydroxyl number of 35 mgKOH/g, set oil bath temperature to 130°C). Set up the gel meter as described in the operation manual.

X2.4.3 Weigh the appropriate amount of isocyanate to the nearest 0.1 g. (See X2.5.) Remove the jar of polyester polyol from the circulating air oven and rapidly add the isocyanate.

X2.4.4 Start the stopwatch as soon as the isocyanate is added.

X2.4.5 Immediately mix with a spatula for 1.5 min and fill an 18-mm by 150-mm test tube to a height of 9.5 cm from the bottom of the tube.

X2.4.6 Place the tube in the gel meter bath and install the spindle so that it remains 2.5 cm off the bottom of the tube.

X2.4.7 Start the gel meter and stop the stopwatch at the same time. Record the time, *SW*, on the stopwatch to the nearest second.

X2.4.8 When the gel meter stops and the buzzer sounds, record the gel meter time, *G*, to the nearest second.

X2.4.9 Immediately remove the spindle from the unit and clean it with MEK.

X2.5 Calculations

X2.5.1 Calculate the equivalent weight, EW_{PE} , of the polyester polyol as follows (see Note X2.1):

$$EW_{PE} = \frac{56.1 \times 1000}{OH} \quad (X2.1)$$

where:

- EW_{PE} = the equivalent weight of the polyester polyol sample,
- 56.1 = the equivalent weight of KOH in milligrams per milliequivalents,
- 1000 = the factor for converting milliequivalents to equivalents, and
- OH* = the hydroxyl number of the polyester polyol sample in milligrams of KOH per gram of sample as determined by the appropriate specification method.

NOTE X2.1—Equation in X2.5.1 assumes negligible contribution from

the acid number and water content of the polyester polyol. The user of this practice must validate this assumption for their particular system

X2.5.2 Calculate the equivalents, E_{PE} , in 50 g of the polyester polyol as follows:

$$E_{PE} = \frac{50}{EW_{PE}} \quad (X2.2)$$

where:

50 = the amount of the polyester polyol used in the reactivity test in grams

X2.5.3 Calculate the equivalent weight, EW_I , of the appropriate isocyanate as follows:

$$EW_I = \frac{42.02 \times 100}{\%NCO} \quad (X2.3)$$

where:

42.02 = the equivalent weight of an NCO group in grams per equivalent,

100 = the factor for converting % NCO to grams of NCO, and

%NCO = the %NCO of the isocyanate as determined by the appropriate specification method.

X2.5.4 Calculate the weight, W_I , of isocyanate to be used in the reactivity test as follows:

$$W_I = E_{PE} \times EW_I \quad (X2.4)$$

where:

W_I = the weight of the isocyanate in grams

X2.5.5 Calculate the total reaction time, T , as follows:

$$T = G + SW \quad (X2.5)$$

where:

G = the time from the gel meter in seconds and

SW = the time from the stopwatch in seconds.

X2.6 Report

X2.6.1 Report gel time result, T , to the nearest one second.

X2.7 Precision

X2.7.1 The precision of this test method is not known at this time because inter-laboratory data is not available.

X3. TEST PROCEDURE B: GEL TIME OF AN ISOCYANATE COMPONENT WITH A RESIN BLEND USING A GARDCO GEL TESTER

X3.1 Scope

X3.1.1 This procedure is used to characterize the reactivity of Isocyanate X with a resin blend.

X3.2 Summary of Practice

X3.2.1 A sample of Isocyanate X is reacted with a resin blend that consists of polytetrahydrofuran diol and 1,4- butanediol producing a product that is similar to one that is manufactured by end users of Isocyanate X. Although the final polyurethane product has a moderately low average molecular weight, it can be classified as a thermoplastic. As the reaction proceeds, the viscosity of the reaction mixture increases and this increase is monitored with a gel meter. The time required for the mixture to reach the point where the wire stirrer makes only one revolution in 60 seconds is reported as the gel time.

X3.3 Equipment and Chemicals

X3.3.1 *Gardco Hot Pot Gel Timer*, Fast Cure (Paul N. Gardner, Inc.).

X3.3.2 *Oven, forced air*, temperature control to $\pm 0.2^\circ\text{C}$.

X3.3.3 *Oven, convection*.

X3.3.4 *Balance, analytical*, 0.1-mg readability.

X3.3.5 *Balance, top pan*, 1-kg capacity.

X3.3.6 *Cup*, aluminum, for Gardco gel timer.

X3.3.7 *Stirrer*, wire.

X3.3.8 *Stopwatch*, 1-s readability.

X3.3.9 *Polytetrahydrofuran diol*, nominal OH No. 112 mgKOH/g.

X3.3.10 *1,4-Butanediol*, anhydrous.

X3.3.11 *Xylene*, raw material grade.

X3.3.12 *Dibutyltin dilaurate catalyst*.

X3.3.13 *Syringe*, glass, 50- μL .

X3.3.14 *Bottle, screw-capped*, 30-mL (1-oz).

X3.3.15 *Bottle, screw-capped*, 120- mL (4-oz)

X3.3.16 *Pail*, 2-L (0.5-gal).

X3.3.17 *Mixer*, for 2-L (0.5-gal) pail.

X3.3.18 *Nitrogen gas*.

X3.3.19 *Polyol blend* (approximately 17.77 to 1.0 weight ratio of polytetrahydrofuran to 1,4 -butanediol).

X3.3.20 *Catalyst solution* (approximately 4.53 w/w % dibutyltin dilaurate catalyst in xylene solution).

X3.4 Instrument Conditions

X3.4.1 Gel meter conditions, 90°C .

X3.4.2 Oven, forced air, 60°C .

X3.4.3 Oven, convection, 90°C .

X3.5 Procedure

X3.5.1 Prepare the polyol blend. The steps to make a 500-g batch are given below as an example.

X3.5.1.1 Place the polytetrahydrofuran diol in a $40\text{--}50^\circ\text{C}$ oven until it is completely melted.