Designation: ~~D6437 – 05 (Reapproved 2016)$^{\varepsilon 1}$~~ D6437 – 22

Standard Test Method for Polyurethane Raw Materials: Alkalinity in Low-Alkalinity Polyols (Determination of CPR Values of Polyols)[1]

This standard is issued under the fixed designation D6437; 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.

~~ε^1 NOTE—Reapproved with editorial changes in April 2016.~~

1. ~~Scope~~ Scope*

1.1 This test method covers measuring alkalinity in low-alkalinity (<0.002 meq/g basicity) polyols. This alkalinity is often expressed as CPR (controlled polymerization rate) of polyether polyols. This test method is not applicable to amine-based polyols.

1.2 The values stated in SI units are to be regarded as ~~the~~ standard.

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

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

1.4 *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

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

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:*

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[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.

Current edition approved ~~April 1, 2016~~March 15, 2022. Published ~~April 2016~~March 2022. Originally approved in 1999. Last previous edition approved in ~~2010~~2016 as D6437 - 05 ~~(2010)~~(2016)$^{\varepsilon 1}$. DOI: ~~10.1520/D6437-05R16E01.~~10.1520/D6437-22.

[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] The last approved version of this historical standard is referenced on www.astm.org.

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

3.1.1 ~~The terminology~~Terms used in this ~~test method is~~standard are defined in accordance with Terminology D883~~the standard terminology defined in~~ , 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 ~~D883~~E456.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *CPR*—controlled polymerization rate is expressed as basicity in milliequivalents per 30 kg of sample (meq/30 kg).

4. Summary of Test Method

4.1 This test method is a potentiometric titration for sample basicity in methanol solvent. This test method uses a relatively large amount of sample and the titration is performed with dilute acid solution to determine trace quantities of basicity.

5. Significance and Use

5.1 This test method is suitable for quality control, as a specification test and for research. The urethane reaction between polyols and isocyanates to form polyurethane polymers is known to be sensitive to the presence of basic substances. This is particularly important in the preparation of polyurethane prepolymers which contain isocyanate groups that are known to react in the presence of trace amounts of basic substances. Since many polyether polyols are often made with strongly basic catalysts, it is important to have an analytical method capable of detecting small quantities of residual basic substances. This test method is capable of detecting ppm levels of base (as KOH).[4]

6. Apparatus

6.1 *Potentiometric Automatic Titrator,* capable of detecting multiple titration end points.

6.2 *Autotitrator Buret, 5 mL (see Note 2).*

6.3 *Buret or Dosing Device,* capable of dosing 50 mL.

6.4 *pH Glass Electrode and Reference Electrode or a Combination Glass Electrode.*

6.5 *Analytical Balance,* capable of weighing to the nearest 0.01 g.

NOTE 2—A 1-mL titrator buret can be used if available. Due to the low volumes of titrant typically required (0 to 0.5 mL), larger burets will give less precise results.

7. Reagents and Materials

7.1 *HCl Aqueous, 0.01 N*—Standardize to detect changes of 0.0001 *N*.

7.2 *Methanol,* reagent grade

8. Procedure

8.1 Set up the autotitrator to find multiple end points with a maximum volume of 5 mL.

8.2 Place 50 ± 0.1 mL of methanol in a 100-mL titration cup and titrate a blank using 0.01 *N* aqueous HCl.

8.3 Weigh 30 ± 1.00 g of sample into a titration cup. Add 50 ± 0.1 mL of methanol, stir to mix well, and titrate with 0.01 *N* aqueous HCl. There will be as many as three end points (breaks) in these titrations. Use the volume to the last end point for calculation.

[4] H.G. Scholten, J.G. Schuhman, R.E. TenHoor, *Journal of Chemical Engineering Data*, 5, 1960, p. 396.

NOTE 3—If the viscosity of the sample appears too high, use 100 mL of methanol to dissolve the sample.

9. Calculation

9.1 Calculate the CPR as meq/30 kg of the polyol as follows:

~~$$CPR = (V_{sam} - V_{blk}) \cdot N \cdot 30\,000/W \quad (1)$$~~

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where:

V_{sam} = volume of titrant to the last break in sample titration, mL
V_{blk} = volume of titrant to the last break in blank titration, mL
N = normality of HCl,
30 000 = conversion factor for 30-kg sample, and
W = weight of sample, g.

9.2 Calculate alkalinity as micromoles per gram of sample as follows:

~~$$\mu mol/g = (V_{sam} - V_{blk}) \cdot N \cdot 1000/W \quad (2)$$~~

$$\mu mol/g = (V_{sam} - V_{blk}) \cdot N \cdot 1{,}000/W \quad (2)$$

where:

1000 = conversion from millimole to μmole.

9.3 Calculate alkalinity as micrograms KOH per gram of sample (ppm KOH) as follows:

~~$$ppm\ KOH = (V_{sam} - V_{blk}) \cdot N \cdot 56.1/W \quad (3)$$~~

$$ppm\ KOH = (V_{sam} - V_{blk}) \cdot N \cdot 56{,}100/W \quad (3)$$

where:

~~56.1 = equivalent weight of KOH in mg/meq.~~

56,100 = equivalent weight of KOH in mg/meq (56.1) times conversion factor from milligrams to micrograms.

10. Precision and Bias[5]

10.1 *Precision*—Table 1 is based on a round robin conducted in 1997 in accordance with Practice E691 with the data analysis in accordance with Practice E180, involving five samples tested by seven laboratories. Each test result was the average of two individual determinations. Each laboratory made duplicate determinations on each material on each of two days. (**Warning—**The following explanations of *r* and *R* (10.1.1 – 10.1.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 must not be rigorously applied to the acceptance or rejection of material, as those

TABLE 1 Round-Robin CPR Data in Accordance with Practice E180

	Values in CPR Units					
	Average	S_r[A]	S_R[B]	r[C]	R[D]	n[E]
Terathane 1000	0.34	0.07	0.20	0.20	0.56	7
Voranol 4702	0.45	0.06	0.11	0.18	0.32	6
Voranol 2120	0.63	0.04	0.12	0.12	0.33	5
ARCOL E-656	0.60	0.06	0.16	0.16	0.46	7
Multranol 7057	1.24	0.11	0.36	0.30	1.0	6
Pooled data	. . .	0.07	0.21	0.20	0.59	. . .

[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.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1210.