Designation: D 7131 – 05

Standard Test Method for Determination of Ion Exchange Capacity (IEC) in Grafted Battery Separator[1]

This standard is issued under the fixed designation D 7131; 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 test method evaluates an important characteristic of polyolefin or other fibrous nonwoven sheet material intended for use in alkaline battery separator applications. The hydrophilic properties of the material are enhanced by grafting a functional group onto the polyolefin, and this test method is the primary test method to determine the treatment level, that is, level of monomer grafted to the base polymer. This test method can also be used for film or membranes

1.2 This test method is intended primarily for testing copolymer-grafted polyolefin materials used as battery separators, but could be used for any grafted material. These separators have radiation-initiated grafting of acrylic acid monomer (for example) onto a polyolefin base-web material to generate hydrophilic sites on the material. This process is a very good method for surface modification of polymer materials, and is used to make separators hydrophilic. Grafting can be accomplished by irradiation on common polymers such as polyethylene, polypropylene and fluoropolymers with various forms of energy, such as UV, gamma rays, electron beams (EB) or X-rays..

1.3 The values stated in SI units are to be regarded as the 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 to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 123 Terminology Relating to Textiles

D 1711 Terminology Relating to Electrical Insulation

E 438 Specification for Glasses in Laboratory Apparatus

E 1272 Specification for Laboratory Glass Graduated Cylinders

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of textile terms, refer to Terminology D 123.

3.1.2 For definitions of electrical terms, refer to Terminology D 1711.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *grafted battery separator, n*—a sheet material whose fiber surface has been grafted to add a functional group to the base polymer of the fiber by some form of radiation, whereas these grafted sites are hydrophilic, thus increasing the sheet material's hydrophilic properties.

3.2.2 *ion-exchange capacity, n*—the number of ionic sites on the separator fiber surface that can participate in the exchange process. The exchange capacity is expressed in milliequivalents per gram.

4. Summary of Test Method

4.1 A predetermined amount of separator in its acid form is added to a bottle with a potassium hydroxide (KOH) solution. The specimens are conditioned under a controlled temperature for 2 h. After being allowed to cool, the amount of grafted monomer functionality of the grafted surface in the sample can be determined by titration.

4.2 Results are calculated as milliequivalents per gram (meq/g) using the appropriate equations.

4.3 Sampling must be determined based on experience with the separators uniformity and consistency of the grafting process.

5. Significance and Use

5.1 Nickel-metal hydride (Ni-MH) cells have a tendency to exhibit high rates of self-discharge that may be caused by contamination within a battery cell. The contamination source has been shown to originate from electrode impurities. Grafted separators can trap and hold these impurities, thus reducing self-discharge rates and enhancing battery quality. This test

[1] This test method is under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and is the direct responsibility of Subcommittee D09.19 on Dielectric Sheet and Roll Products.

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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.

method determines an ionic exchange capacity that can be used to quantify the level of grafting of a separator.

5.2 This method is useful for research, quality control, and material specifications.

6. Apparatus

6.1 *Beaker*, 600 mL.

6.2 *Bottles, Glass or HDPE*, 125-mL capacity with screw cap.

6.3 *Cylinder, Graduated*, Class A, 100- mL capacity.

NOTE 1—The tolerance on the accuracy of the graduations in Specification E 1272 for this apparatus is ±1.4 mL. If greater accuracy is required for a test result, the graduated trap(s) should be calibrated.

6.4 *Pipette, Volumetric*, Class A, 10- mL capacity.

NOTE 2—The tolerance on the accuracy of the graduations in Specification E 438 for this apparatus is ±0.02 mL. If greater accuracy is required for a test result, the graduated trap(s) should be calibrated.

6.5 *Oven*.

7. Reagents and Materials

7.1 *Hydrochloric Acid*, 0.1*M* (HCL), ACS grade.

7.2 *Hydrochloric Acid*, 1.0*M* (HCL), ACS grade.

7.3 *Methyl Red*, ACS grade.

7.4 *Potassium Hydroxide*, 0.1*M* (KOH), ACS grade. (**Warning**—Follow appropriate safe handling procedures when using acids or bases.)

7.5 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[3]

8. Conditioning

8.1 Cut test specimen to a weight of 0.50 ± 0.05 g. Convert the specimen into acid form by immersion in 1.0*M* HCl at 60 ± 3 ºC for at least 1 h.

8.2 Wash the specimen with water so the pH, using litmus paper, of the specimen is between a pH of 6 to 7.

8.3 Dry the specimen to a constant weight (W_1), recording the weight.

8.4 For each specimen, add 90 ± 0.5 mL of distilled water to each bottle as described in 6.2.

8.5 For each specimen, add 10 ± 0.05 mL of 0.1*M* KOH using the volumetric pipette (as described in 6.4) to the same bottles as in 8.4.

8.6 Add the dried specimen to the conditioned bottles.

8.7 Run a control, using the same instruction as stated in 8.4 and 8.5 but without the test specimen.

8.8 Bottles are then stored together at 60 ºC for 2 to 2.5 h.

9. Procedure

9.1 Allow bottles to cool back to room temperature.

9.2 The contents of each bottle are then transferred to a glass beaker as stated in 6.1

9.3 A further 100 mL of washings are added to the beakers from each bottle.

9.4 The amount of KOH remaining in each bottle is determined through titration.

10. Calculation or Interpretation of Results

10.1 Calculate the IEC in meq/g as follows:

$$IEC = (t_2 - t_1)/(10W_1) \quad (1)$$

Where:

T_1 = the titration value of 0.1*M* HCl from the bottle with the sample,

T_2 = the titration value of 0.1*M* HCl from the bottle without the sample, and

W_1 = the weight of the dried sample in acid form as stated in 8.3.

11. Report

11.1 State that the specimens were tested as directed in Test Method D 7131.

11.2 Report the following information:

11.2.1 The calculated IEC value (10.1) to the nearest 0.0000X meg/g.

12. Precision and Bias

12.1 *Precision*—The precision of this test method is to be established on or before 2008.

12.2 *Bias*—The procedure in this test method has no bias because the value of IEC can be defined only in terms of a test method.

13. Keywords

13.1 battery separator; grafted polyolefin material; ion exchange capacity

[3] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.