Designation: D 7129 – 05

Standard Test Method for Determination of Ammonia Trapping in a Grafted Battery Separator[1]

This standard is issued under the fixed designation D 7129; 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 covers the measurement of the ability of a material to capture ammonia.

1.2 This test method is intended primarily for testing grafted polyolefin materials used as a battery separator, although other materials could be tested.

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 determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 123 Terminology Relating to Textiles
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 on other textile terms in this test method, refer to Terminology D 123

4. Summary of Test Method

4.1 A predetermined amount of separator and ammonium hydroxide is added together in a KOH solution. The specimens are conditioned under a controlled temperature for 24 h. After the solution is cooled, the solutions are distilled to determine the amount of ammonia the separator retained.

4.2 Results are calculated as mol/g using the appropriate equations.

5. Significance and Use

5.1 Nickel metal hydride (Ni-MH) cells can exhibit high self-discharge rates. Nitrate impurities in the positive electrode can be reduced to ammonia on the negative electrode. This causes rapid self-discharge. Technical publications have reported a link between the separator ammonia trapping capabilities and the cells charge retention. Grafted polyolefin separators can absorb or trap these impurities—hence, reducing self-discharge rates. The level of ammonia trapping from this method gives an indication of the efficiency of a separator to trap these contaminates, thus helping a battery self discharge (charge retention). A higher value should provide a cell with greater charge retention.

6. Apparatus

6.1 *Flask, Erlenmeyer*, wide mouth, 250 mL capacity with a screw cap.

6.2 *Flask, Erlenmeyer*, wide mouth, 500 mL capacity.

6.3 *Cylinder, Graduated*, Class A, 250 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[3]. If greater accuracy is required for a test result, the graduated trap(s) should be calibrated

6.5 *Pipette, Volumetric*, Class A, 3- mL capacity.

NOTE 3—The tolerance on the accuracy of the graduations in Specification E 438 for this apparatus is ±0.01 mL. If greater accuracy is required for a test result, the graduated trap(s) should be calibrated.

6.6 *Distillation Unit*, Micro or macro Kjeldhal apparatus.

6.7 *Oven*.

7. Reagents

7.1 *Ammonium Hydroxide*, 0.3*M* (NH_4OH), ACS grade.

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

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[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.

Current edition approved March 1, 2005. Published March 2005.

[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.

7.3 *Methyl Red*, ACS grade.

7.4 *Potassium Hydroxide Solution, 8M*—Prepare a sufficient quantity of KOH solution for the testing program immediately at hand as follows.

7.5 To a 1500-cm^3 (mL) beaker add 800 to 850 cm^3 (mL) of distilled water. Slowly add 448 g of potassium hydroxide pellets, certified ACS grade, to the beaker. Allow cooling to room temperature.

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

8. Conditioning

8.1 Determine sampling based on grafting process. Then cut test specimen to a weight of 2.00 ± 0.05 g.

8.1.1 Cut specimen into 1-cm^2 pieces.

8.1.2 Place cut pieces into Erlenmeyer flask as stated in 6.1.

8.2 Measure out 120 mL of KOH solution as stated in 7.3.

8.2.1 Add to the Erlenmeyer flask containing the cut specimen.

8.3 Using the 3 mL volumetric pipette, add 3 ± 0.01 mL of ammonium hydroxide to the Erlenmeyer flask and immediately cap the flask.

8.4 Place the bottle into the oven at 40 °C for 24 ± 0.5 h.

8.5 Condition a control specimen along with the test specimen.

8.6 The control is prepared the same way but without any test specimen placed in the bottle.

9. Procedure

9.1 Allow bottles to cool so that the ammonia returns to liquid phase.

9.2 Precondition the distillation apparatus before distillation as follows:

9.2.1 Distill a 1:1 mixture of ammonia-free water and 50 % NaOH for 5 min just before sample distillation to reduce contamination from atmospheric ammonia. (**Warning**—Follow appropriate safe handling procedures when using acids or bases.)

9.3 Prepare receiving solution as follows:

9.3.1 Add 150 ± 0.1 mL of ammonia-free water to the Erlenmeyer flask as stated in 6.2.

9.3.2 Add 10 ± 0.01 mL of 0.1*M* HCL to the same flask.

9.3.3 Add four drops of methyl red to the flask; this will cause the color to change to red. If the solution does not change to red, check for problems with the procedure.

9.4 Measure out 100 mL of the solution in 8.1 and washings and add to the distillation tube.

9.5 Place tube into the machine.

9.6 Place receiving flask into the machine.

9.7 Begin distillation at a rate of 7.5 mL/min.

9.8 Distillation is complete when receiving solution has captured 200 ± 1 mL of distillate.

9.9 Titrate the receiving flask solution with 0.1*N* potassium hydroxide. The control sample is titrated using 0.1*N* hydrochloric acid.

10. Calculation

10.1 Calculate the non-trapped ammonia as follows:

$$N_2 = ((10 - A) \times 1.25 \times 0.1)/1000 \tag{1}$$

where:

N_2 = amount of non-trapped ammonia and
A = volume of KOH used in titration.

10.1.1 Calculate the control sample as follows:

$$N_1 = ((10 + \mathrm{B}) \times 1.25 \times 0.1)/1000 \tag{2}$$

where:

N_1 = amount of ammonia remaining in the control and
B = volume of KOH used in titration.

10.1.2 Amount of Ammonia Trapped

$$(\mathrm{N}_1 - \mathrm{N}_2)/2 \tag{3}$$

10.1.2.1 Record results in mol/g.

11. Report

11.1 State that the specimens were tested as directed in Test Method D 7129.

11.2 Report the following information:

11.2.1 The amount of ammonia trapped to the nearest 1xE-5

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 values of ammonia trap can only be defined in terms of a test method.

13. Keywords

13.1 ammonia; battery separator; grafted polyolefin, nonwoven