Designation: D7409 – 15

Standard Test Method for Carboxyl End Group Content of Polyethylene Terephthalate (PET) Yarns[1]

This standard is issued under the fixed designation D7409; 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 procedure is based significantly on the GRI GG7 test procedure, Carboxyl End Group Content of Polyethylene Terephthalate (PET) Yarns.

1.2 This test method is used to determine the concentration, in mmol/Kg, of carboxyl end groups (CEG) found in poly(ethylene terephthalate) (PET) yarns by titration.

1.3 This test is applicable to geogrid yarns that are made from PET resin.

NOTE 1—This test is also applicable to high strength geotextile yarns that are made from PET and are used in reinforcement applications.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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]

D123 Terminology Relating to Textiles

3. Terminology

3.1 *Definitions:*

3.1.1 *automatic titration, n*—a titration method in which a machine is used that automatically dispenses small volumes of the titrant and monitors the pH or the electrochemical potential of the solution.

3.1.2 *Carboxyl End Groups (CEG), n*—non-reacted carboxylic acid groups of terephthalic acid located at either end of a polymer chain.

3.1.3 *colorimetric titration, n*—a titration method that monitors the change in color of the solution as the titration proceeds. A chemical indicator sensitive to pH is added to create the color change.

3.1.4 *fibers, n*—a generic term for any one of the various types of material that form the basic structure of a geogrid (or geotextile), which are characterized by having a length at least 100 times its diameter.

3.1.5 *filaments, n*—a continuous fiber of extremely long length.

3.1.6 *potentiometric titration, n*—a titration method that monitors the change in electrochemical potential, expressed as mV, of the solution as the titration proceeds.

3.1.7 *titration, n*—titration is a standard laboratory method of quantitative chemical analysis which can be used to determine the concentration of a known reactant. Typically, the reaction is a neutralization reaction between an acid and a base.

3.1.8 *yarns, n*—a generic term for a continuous strand of fibers, filaments, or material in a form suitable for knitting, weaving, or otherwise intertwining to form a textile related geogrid (or geotextile).

4. Summary of Test Method

4.1 This method defines a specific procedure for the determination of carboxyl end groups (CEG) of PET by a titrimetric method.

4.2 PET yarns are dissolved in o-cresol at 80°C. The solution is diluted with dichloro methane, and titrated against KOH dissolved in methanol, by either a potentiometric or colorimetric method. The amount of potassium hydroxide required to complete the titration with the PET solution is measured and used to calculate to the concentration of CEGs.

5. Significance and Use

5.1 This test method can be used for manufacturing quality control or manufacturing quality assurance purposes to determine the CEG concentration of PET yarns used in either geogrids or geotextiles.

[1] This test method is under the jurisdiction of ASTM Committee D35 on Geosynthetics and is the direct responsibility of Subcommittee D35.02 on Endurance Properties.

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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 The CEG content of the PET yarns may have an influence on the properties of the geosynthetic, such as its hydrolysis resistance. The lower the value, the higher the hydrolysis resistance of the yarns.

5.3 This test does not set the limiting (maximum) value for various engineering applications. Such a specification is a decision of the design engineer, owner, and/or regulator.

6. Apparatus

6.1 An *automatic titrator* is the preferred equipment for this test method. However, acceptable results can be achieved manually with the use of a micro burette, with 10 ml capacity and accurate to 0.02 ml.

6.2 A *potentiometric titration* assembly is required for a potentiometric titration. The assembly consists of an automatic titrator fitted with a three electrode system together with a buret assembly, magnetic stir bar, magnetic stir plate, and glass beaker.

6.3 A temperature bath or hot plate that can maintain a temperature of 100°C.

7. Reagents

7.1 In all cases, reagent grade chemicals must be used.

7.2 *Ortho-Cresol (>99 %)*—The solvent should be dispensed with an automatic dispensing system to prevent air or water from entering the bottle. Alternatively, the solvent can be distilled prior to use. A different solvent may be used provided the user first demonstrates equivalence to test results measured using Ortho-Cresol.

7.3 *Dichloro Methane (>99.5 %)*—The solvent shall be from 1L bottles, opened within one week. Alternatively, the solvent can be distilled prior to use. A different solvent may be used provided the user first demonstrates equivalence to test results measured using Ortho-Cresol.

7.4 *Potassium Hydroxide*—0.005M potassium hydroxide in methanol.

8. Safety

8.1 *O-Creosol*—It is both toxic and combustible. It may be absorbed through the skin. Avoid inhalation and skin contact. Wear the appropriate safety equipment required for handling at all times. If contact is made with the skin wash the affected area with soap and water for at least 15 to 20 min. In case of eye contact, flush the eyes with water for 20 min and get medical attention immediately.

8.2 *Potassium Hydroxide*—It is toxic and flammable. Avoid skin contact and breathing vapors. If ingested, could cause pain in the nose, mouth and throat. Could induce vomiting, diarrhea, dizziness, hypotension, collapse, coma, or death. In case of eye or skin contact, wash with water for 15 to 20 min and seek medical attention immediately.

8.3 *Methanol*—It is moderately toxic and flammable. Avoid inhalation and skin contact. Methanol may be absorbed through the skin. Wear the appropriate safety clothing that is required for the chemical laboratory when handling. If contact is made, wash the affected area with water for at least 15 or 20 min, get medical treatment immediately. Keep solvent away from sparks and flames.

8.4 *Dichloro Methane*—It is toxic and flammable. Avoid breathing vapors and skin contact. If contact is made, wash the affected area with water for 15 to 20 min. Get medical attention immediately.

9. Sample Preparation

9.1 Collect a representative sample of yarns (3 to 4 g) from the longitudinal direction of the geogrid (or geotextile) to perform three (3) replicate CEG determinations.

NOTE 2—The coating on geogrid yarns must be completely removed. The recommended solvent for removal of PVC coatings is methyl ethyl ketone. Other coatings may require different solvents.

NOTE 3—If the test is being performed on high strength geotextiles, collect a representative sample from the warp direction fibers, filaments or yarns to perform three (3) replicate CEG determinations.

9.2 The fibers, filaments or yarns are cut into small pieces, 2 to 5 mm in length.

9.3 The fibers, filaments or yarns should be washed in ethyl ether to remove sizing agents, and subsequently dried overnight at 50°C.

10. Preparation of KOH/Methanol Solution

10.1 Dissolve approximately 0.2 g KOH (3 pellets) in 1L of methanol. After dissolution is complete, titrate with Potassium Hydrogen Phthalate (KHP) to determine the molarity of the KOH solution.

10.2 Measure 0.01 to 0.02 g of KHP crystals into a 150 ml beaker. Record mass of KHP to ±0.0001 g. Dissolve KHP in approximately 50 ml of DI water.

10.3 Titrate the KHP with the KOH/methanol solution until an endpoint of pH 7 is reached. Record the volume required to reach the endpoint.

10.4 Repeat steps 10.2 and 10.3 until three measurements have been obtained.

10.5 Calculate the molarity of the KOH/methanol solution using the following formula:

$$[OH] = (w/204.23)\ x\ (1/v) \quad (1)$$

where:

w = weight of KHP (g), and
v = volume to titrate to pH7 (L).

11. Procedure

11.1 Approximately 0.25 g of PET yarn is weighed to an accuracy of ±0.0001 g and placed into a 125 ml erlenmeyer flask. Then, 15 ml ± 0.1 ml of orthocresol solvent and a magnetic stir bar are added.

11.2 The flask is covered with a watch glass and placed in a hot source. The temperature of the solvent should be 80°C (±5°C). The mixture of solvent and yarn should be maintained at this temperature and stirred until the yarn is completely dissolved.

NOTE 4—If the yarn is not completely dissolved in 30 min, continue

heating, but inspect every 5 min. The total heating time should not be greater 45 min.

11.3 Remove the flask from the hot source as soon as the PET yarn is completely dissolved and quench by carefully adding 60 ml of dichloromethane to the orthocreosol solution. Stir for an additional 5 min.

11.4 Titrate the PET solution with KOH/methanol solution using an autotitrator.

11.4.1 If colorimetric titration is used, 2 drops of 1 % bromophenol blue indicator in methanol should be added to the PET solution and titrated to a violet endpoint.

11.5 The test procedure is performed on three samples and a blank. The blank shall be treated identically to the samples.

12. Calculation

12.1 Calculation to Determinate the CEG Content.

12.2 CEG content is expressed in the units of mmol/Kg.

12.3 The calculation is as follows:

$$CEG = [(Vs - Vb)*(1000)*(M)]/w \quad (2)$$

where:

Vs (ml) = volume of KOH solution to titrate the sample,
Vb (ml) = volume of KOH solution to titrate the blank,
M (mol/l) = molarity of KOH/methanol solution, and
w (g) = weight of PET sample.

13. Report

13.1 Report the following information:

13.1.1 Identify the product and location from which the test sample was taken from the product.

13.1.2 Report the individual CEG values, the average value and the coefficient of variation.

14. Keywords

14.1 carboxyl end group content; CEG; geogrid yarns; geotextile yarns; PET; polyethylene terephthalate