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Poly-(Tetra Methylene Ether Glycol) (PTMEG) for Industrial Use

工业用聚四亚甲基醚二醇（PTMEG）

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Poly-(Tetra Methylene Ether Glycol) (PTMEG) for Industrial Use

1 Scope

This Document specifies the classification and naming, technical requirements, test methods, inspection rules, marking and accompanying documents, packaging, transportation and storage of poly-(tetra methylene ether glycol) (PTMEG) for industrial use.

This Document applies to industrial PTMEG produced by polymerization of tetrahydrofuran monomer.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 601 Chemical Reagent – Preparations of Reference Titration Solutions

GB/T 603 Chemical Reagent – Preparations of Reagent Solutions for Use in Test Methods

GB/T 3143 Color Determination Method of Liquid Chemicals (Hezen Unit – Platinum – Cobalt Scale)

GB/T 4472-2011 Determination of density and relative density for chemical products

GB/T 6283 Chemical products – Determination of water Karl Fischer method (general method)

GB/T 6678 General Principles for Sampling Chemical Products

GB/T 6680 General rules for sampling liquid chemical products

GB/T 6682 Water for analytical laboratory use – Specification and test methods

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

6.7.2.3 Analytical balance, with graduation value of 0.01g.

6.7.3 Reagents

6.7.3.1 pH standard buffer solution: pH=4.00.

6.7.3.2 pH standard buffer solution: pH=6.86.

6.7.3.3 Lithium chloride, guaranteed reagent.

6.7.3.4 Electrode replenishment solution: Prepare a saturated solution of lithium chloride in ethanol; and dilute the saturated solution with water in a ratio of 2+1 (volume ratio).

6.7.3.5 Electrode soaking solution: Prepare a saturated solution of lithium chloride in ethanol; and dilute the saturated solution with water in a ratio of 1+1 (volume ratio).

6.7.3.6 Standard hydrochloric acid titration solution, *c*(HCl) is approximately 0.005mol/L: dilute by standard hydrochloric acid titration solution with *c*(HCl) approximately 0.1mol/L before use.

6.7.3.7 Standard sodium hydroxide solution: *c*(NaOH) is about 0.1mol/L.

6.7.3.8 Methanol solvent: Take 60mL of distilled water into a 2000mL volumetric flask. Add 0.8mL standard sodium hydroxide solution; dilute to the mark with methanol; transfer to a glass bottle; and stir for 4h~6h.

6.7.4 Determination procedures

6.7.4.1 Instrument settings

Set the control parameters according to the operating instructions of the automatic potentiometric titrator; and set the end point determination method to pH=5.3.

6.7.4.2 Determination

Take 10g of the specimen (accurate to 0.01g) into a 100mL glass beaker; add 50.00mL of methanol solvent; stir until completely dissolved; perform potentiometric titration with standard hydrochloric acid titration solution; record the standard titration solution volume (V_1) required when the titration reaches pH=5.3. At the same time, measure the blank solution volume (V_{10}).

6.7.5 Calculation of the result

The acid value (by consumption of KOH) of PTMEG (AN) is calculated according to Formula (1):

$$AN = \frac{(V_{10} - V_1) \times c_1 \times M}{m_1} \qquad \cdots\cdots(1)$$

3.06460 - the negative logarithm of the value K of PTMEG in the Mark-Houwink equation.

Calculation results of the molar mass ratio are rounded to two digits after the decimal point.

6.10 Stabilizer (BHT) content

6.10.1 Method summary

Prepare a standard solution based on the features with characteristic absorption of BHT at 278nm; and establish a standard curve between concentration and absorbance.

6.10.2 Instruments

6.10.2.1 Volumetric analytical instruments and vessels for general laboratory use.

6.10.2.2 UV-visible spectrophotometer with scanning function.

6.10.2.3 Analytical balance with graduation value of 0.1mg, 0.01g.

6.10.3 Reagents

6.10.3.1 Methanol.

6.10.3.2 standard BHT solution, 1mg/mL: Weigh 0.1g of BHT (accurate to 0.0002g) in a 100mL volumetric flask; fully dissolve it with methanol; and dilute to the mark.

6.10.4 Drawing of working curve

6.10.4.1 Add 1.0mL, 2.0mL, 3.0mL, 4.0mL and 5.0mL of standard BHT solution to the 100mL volumetric flask respectively, and dilute to the mark by methanol.

6.10.4.2 Use methanol as a blank and measure at the wavelength of 278nm using a 10mm cuvette.

6.10.4.3 Draw the standard curve with BHT (mg/100mL) in 100mL standard solution as the abscissa and the corresponding absorbance as the ordinate.

6.10.5 Determination of sample

Weigh 10g of the sample (accurate to 0.01g), add it to a 100mL volumetric flask; dilute to the mark with methanol; and shake well. Use methanol as a blank, use a 10mm cuvette to perform spectral scanning; perform data processing on the spectrum; and calculate the corrected absorbance at 278nm.

6.10.6 Calculation of result

Except for the molar mass ratio, all other items are exit-factory inspection items, and the exit-factory inspection items shall be inspected batch by batch.

7.2 Batching rules

Products are inspected in batches, and the mixing volume in the same storage tank is considered a batch, and the batch size does not exceed 3000t.

7.3 Sampling

Carry out nitrogen replacement and protection sampling according to the provisions in GB/T 6678 and GB/T 6680. The sample size shall be no less than 0.5kg. The sample shall be mixed and divided into two clean, dry, and sealed glass bottles or plastic bottles. Attach a label to indicate the product name, manufacturer, batch number, batch size, sampling date, sampling location, and sampler. One bottle is used for testing, and the other bottle is kept for future reference.

7.4 Judgment rules

The inspection results are judged according to the rounding value comparison method specified in GB/T 8170 to determine whether they meet the requirements in Clause 5. If an index does not meet the technical requirements in Clause 5, double the amounts of samples shall be taken from the storage tank or packaging unit for reinspection. Even if one index does not meet the technical requirements in Clause 5, the whole batch of products shall be judged to be unqualified.

8 Marking and Accompanying Documents

8.1 Marking

PTMEG products shall be accompanied by product marks, including: product name, product standard number, manufacturer name and address, net content, production batch number, certificate of conformity, etc.

8.2 Accompanying files

Each batch of products shall be accompanied by safety technical instructions and quality inspection reports, including: name of the manufacturer, product name, technical requirements, net content, batch number, or production date, etc., and shall be stamped with a special quality inspection seal and the signature (seal) of the inspector.

9 Packaging, Transportation and Storage

9.1 Packaging

Appendix A

(Normative)

Determination of Average Molar Mass and Hydroxyl Value

A.1 Method overview

The hydroxyl group at the end of PTMEG reacts with excessive p-toluenesulfonyl isocyanate (TSI) to generate acidic carbamate. Excessive TSI reacts with water to form sulfamide; and the acidic carbamate is directly titrated potentiometrically with tetrabutylammonium hydroxide (TBAH) in a low-water medium.

A.2 Instruments

A.2.1 Volumetric analytical instruments and vessels for general laboratory use.

A.2.2 Automatic potentiometric titrator: equipped with a 20mL burette and a pH aqueous phase composite electrode. The volume resolution of the burette is less than or equal to 1/20000.

A.2.3 Analytical balance with graduation value of 0.1mg.

A.3 Reagents

A.3.1 Acetonitrile: moisture shall not exceed 300mg/kg.

A.3.2 Absolute ethanol.

A.3.3 Isopropyl alcohol.

A.3.4 Standard pH buffer solution: pH=4.00.

A.3.5 Standard pH buffer solution: pH=6.86.

A.3.6 Lithium chloride, guaranteed reagent.

A.3.7 Electrode replenishment solution: Prepare a saturated solution of lithium chloride in ethanol; and dilute the saturated solution with water at a ratio of 2+1 (volume ratio).

A.3.8 Electrode soaking solution: Prepare a saturated solution of lithium chloride in ethanol; and dilute the saturated solution with water at a ratio of 1+1 (volume ratio).

A.3.9 Standard KHP titration solution, $c(C_8H_5KO_4)$ = 0.1000mol/L, prepared according to GB/T 601.

A.3.10 Stabilizer (BHT).

A.3.11 1,4-butanediol (BDO).

A.3.12 Tetrahydrofuran (THF) solvent, moisture not exceeding 300mg/kg:

Weigh 0.2g (accurate to 0.01g) of BDO and BHT each; add to 5000mL of THF; mix well and set aside.

A.3.13 Standard TBAH titration solution, $c(C_{16}H_{37}NO)$ is about 0.1mol/L:

a) Preparation: Take 128mL of distilled water and 49mL of TBAH (55%) into a 1000mL volumetric flask. After cooling, dilute to the mark with isopropyl alcohol and shake well;

b) Calibration: Pipette 10.00mL of standard KHP titration solution into a 100mL tall beaker; add water to about 60mL; titrate the standard KHP titration solution with TBAH solution; determine the end point by potentiometric titration. Repeat the titration four times; and take the arithmetic average as the concentration of the standard TBAH titration solution;

c) Calculation: standard TBAH titration solution is calculated according to Formula (A.1), accurate to four digits after the decimal point.

$$c_2 = \frac{c_3 \times V_3}{V_2} \qquad \text{……(A.1)}$$

Where:

c_2 - concentration of standard TBAH titration solution, in mol/L;

c_3 - concentration of standard KHP titration solution, in mol/L;

V_3 - the volume of pipetted standard KHP titration solution, in mL;

V_2 - the volume of the consumed TBAH solution, in mL.

A.3.14 TSI solution, 6.2%: Weigh 25g (accurate to 0.01g) of TSI (96%); dissolve it in acetonitrile; transfer it to a 500mL volumetric flask; dilute it to the mark with acetonitrile; and shake well.

A.4 Test procedures

A.4.1 Instrument settings: Set the control parameters according to the automatic potentiometric titrator operating instructions.

A.4.2 Measure the sample: Weigh 0.5g~1.0g (accurate to 0.0002g, adjust the weighing according to the product type) of specimen into a 100mL tall glass beaker; add 50mL of THF solvent, 10mL of TSI solution; and stir for 10 min. Add 2 mL of distilled water and stir for 5 min. Use standard TBAH titration solution to titrate to the end point, and the instrument will record two end points V_4 and V_5.

Appendix B

(Normative)

Determination of Carbonyl Ratio

B.1 Method overview

Determine the sum of the absorption of the carbonyl band in the infrared region and the absorption of the composite band of the ether compound; and calculate the carbonyl ratio (*C*/*R*).

B.2 Instruments

B.2.1 Volumetric analytical instruments and vessels for general laboratory use.

B.2.2 Fourier transform infrared spectrometer: The sensitivity and stability of the whole machine shall comply with the relevant regulations in GB/T 21186.

B.2.3 Stopwatch with resolution of 0.1s.

B.2.4 Analytical balance with graduation value of 0.1mg and 0.1g.

B.3 Reagents

B.3.1 Adipic acid, standard substance.

B.3.2 Nitrogen with volume fraction of 99.999%.

B.3.3 PTMEG1000, the carbonyl ratio is less than 0.1.

B.3.4 Standard adipic acid stock solution:

Weigh 1.5g (accurate to 0.0002g) of adipic acid in a 2000mL glass bottle; weigh 1498.5g (accurate to 0.1g) of PTMEG1000 in the above bottle; pass nitrogen into the above glass bottle at a lower flow rate; and put in an oven at 50℃±5℃ and keep constant temperature for 16h.

B.3.5 Potassium bromide, spectrally pure.

B.4 Test procedures

B.4.1 Preparation of infrared spectrum samples

Use the liquid film method to prepare infrared spectrum samples. The preparation shall be carried out according to the instrument operating instructions to avoid the generation of bubbles. The finished potassium bromide salt tablets can also be purchased.

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