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National food safety standard - Food additives - Polyethylene glycol

食品安全国家标准 食品添加剂 聚乙二醇

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National food safety standard - Food additives - Polyethylene glycol

1 Scope

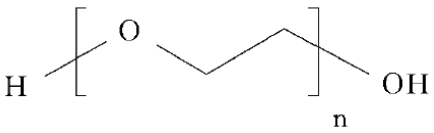
This Standard applies to the food additive polyethylene glycol, which is made by polycondensation of ethylene oxide and water.

2 Chemical name, structural formula, molecular formula and relative molecular mass

2.1 Chemical name

α-hydrogen-ω-hydroxy (oxy-1,2-ethylenediyl) polymer

2.2 Structural formula



2.3 Molecular formula

$H(OCH_2CH_2)_nOH$

2.4 Relative molecular mass

200 ~ 9 500 (according to the international relative atomic mass in 2014)

3 Technical requirements

3.1 Sensory requirements

Sensory requirements shall be in accordance with Table 1.

Table 1 – Sensory requirements

Appendix A

Inspection method

A.1 General provisions

The reagents and water that are used in this Standard, when no other requirements are specified, refer to analytical reagents and grade-III water which is specified in GB/T 6682. The standard solution, the impurity standard solutions, preparations and products, which are used in the test, are all prepared in accordance with the provisions of GB/T 601, GB/T 602, and GB/T 603, when no other requirements are specified. The solution used in the test, if not indicated which solvent is used, refers to aqueous solution.

A.2 Identification test

A.2.1 Reagents and materials

A.2.1.1 Dilute hydrochloric acid: Measure 234 mL of hydrochloric acid; add water to dilute to 1 000 mL.

A.2.1.2 Barium chloride test solution: Weigh 5 g of fine barium chloride powder; add water to dissolve it to 100 mL.

A.2.1.3 10% phosphomolybdic acid.

A.2.1.4 Potassium thiocyanate.

A.2.1.5 Cobalt nitrate.

A.2.1.6 Methyl chloride.

A.2.2 Identification method

A.2.2.1 Weigh 0.05 g of the sample; add 5 mL of dilute hydrochloric acid and 1 mL of barium chloride test solution; shake; filter; add 1 mL of 10% phosphomolybdic acid solution to the filtrate to produce a yellow-green precipitate.

A.2.2.2 Weigh 0.1 g of the sample; put it in a test tube; add 0.1 g of potassium thiocyanate and cobalt nitrate respectively; after mixing, add 5 mL of dichloromethane; the solution is blue.

A.3 Determination of average relative molecular mass

A.3.1 Reagents and materials

A.7.1.6 Ethanol hydrochloric acid titration solution (0.1 mol/L). Slowly add concentrated sulfuric acid to the concentrated hydrochloric acid solution to prepare dry hydrogen chloride gas; pass it into 200 mL of aldehyde-free ethanol solution; stir to promote its dissolution; weigh the mass change of the ethanol solution before and after the injection, which is about 0.73 g. Calibrate before use.

A.7.1.7 Ethanol potassium hydroxide titrant (0.1 mol/L).

A.7.2 Instruments and apparatuses

A.7.2.1 Gas chromatograph: equipped with headspace sampler and hydrogen flame ionization detector (FID).

A.7.2.2 Oscillator.

A.7.3 Reference chromatographic conditions

A.7.3.1 Chromatographic column: capillary column whose polydimethylsiloxane is fixed liquid, or other equivalent chromatographic columns.

A.7.3.2 Carrier gas: nitrogen; the column flow rate is 1.0 mL/min; the split ratio is 10:1.

A.7.3.3 Temperature: keep the initial temperature of 35 °C for 5 min; raise it to 180 °C at a rate of 5 °C/min; then, raise it to 230 °C at a rate of 30 °C/min; keep it for 5 min (it can be adjusted according to specific conditions). The temperature of the inlet is 150 °C; the temperature of the detector is 250 °C. The equilibrium temperature of the headspace bottle is 70 °C; the equilibrium time is 45 min.

A.7.4 Analysis steps

A.7.4.1 Preparation of ethylene oxide standard stock solution

Measure 300 μL of ethylene oxide (equivalent to 0.25 g of ethylene oxide); put it in a 100 mL measuring flask that contains 50 mL of processed polyethylene glycol 400 (rotate and evaporate at 60 °C, 1.5 kPa ~ 2.5 kPa, for 6 h to remove volatile components); add the same solvent to dilute to the mark; shake well, as a standard stock solution of ethylene oxide. Weigh 1 g (accurate to 0.000 1 g) of cold ethylene oxide standard stock solution; place it in a 50 mL volumetric flask that contains 40 mL of processed polyethylene glycol 400; add the same solvent to dilute to the mark. Accurately weigh 10 g; place it in a 50 mL measuring flask that contains 30 mL of water; add water to dilute to the mark. Measure 10 mL; place in a 50 mL measuring flask; add water to dilute to the mark; shake well, as an ethylene oxide standard solution (about 2 μg/mL).

Where:

m_i – mass of the added to-be-tested component i of known content, in grams (g);

A_{is} – chromatographic peak area of component i after adding the standard solution;

A_i – chromatographic peak area of component i in the test product;

m – mass of the test product, in grams (g).

A.8 Determination of the total amount of ethylene glycol and diethylene glycol

A.8.1 Determination of the total amount of ethylene glycol and diethylene glycol in polyethylene glycol samples of an average relative molecular mass less than 450

A.8.1.1 Reagents and materials

A.8.1.1.1 Ethylene glycol (chromatographic pure).

A.8.1.1.2 Diethylene glycol (chromatographic pure).

A.8.1.2 Instruments and apparatuses

A.8.1.2.1 Gas chromatograph: it is equipped with hydrogen flame ionization detector (FID).

A.8.1.2.2 Oscillator.

A.8.1.3 Reference chromatographic conditions

A.8.1.3.1 Chromatographic column: the stainless steel-packed 3 mm × 1.5 m chromatographic column is packed with untreated diatomaceous earth that bears 12% sorbitol, or other equivalent chromatographic columns.

A.8.1.3.2 Carrier gas: nitrogen or other suitable inert gas.

A.8.1.3.3 Temperature: The column temperature is kept at 160 °C; the inlet temperature is kept at 260 °C; the detector temperature is kept at 280 °C.

A.8.1.4 Analysis steps

A.8.1.4.1 Standard solution preparation: Take 50 mg of ethylene glycol and diethylene glycol respectively; place in a 100 mL volumetric flask; add water to dilute to the mark; shake well, as a control solution.

A.8.2.1.4 Diethylene glycol (chromatographic grade).

A.8.2.1.5 Diphenyl ether.

A.8.2.2 Instruments and apparatuses

A.8.2.2.1 Distilling flask.

A.8.2.2.2 Spectrophotometry.

A.8.2.3 Analysis steps

A.8.2.3.1 Preparation of standard solution: Place 62.5 mg of diethylene glycol in a 25 mL volumetric flask; use a newly distilled acetonitrile and water mixed solution to dissolve the diethylene glycol and dilute to the mark; mix well.

A.8.2.3.2 Preparation of test solution: In a 250 mL distillation flask, dissolve 50.0 g of polyethylene glycol in 75 mL of diphenyl ether. If there are crystals, preheat the solution to dissolve the crystals. Slowly distill under the pressure of 0.133 kPa ~ 0.266 kPa; collect the distillate in a 100 mL receiving container with 1 mL graduation, until 25 mL of distillate is collected. Add 20.0 mL of water to the distillate; vigorously shake; then, stand for stratification; ice bath the mixture to solidify the diphenyl ether and separate the solid and liquid phases. Filter and separate to obtain the aqueous layer; collect the filtrate. Use 5.0 mL of ice water to wash the diphenyl; filtrate the cleaning mixture and collect. Mix the filtrate and cleaning mixture into a 25 mL volumetric flask; add water to dilute to the mark; shake well; if necessary, warm the solution to room temperature. In a 125 mL conical flask with a glass stopper, mix the solution with 25.0 mL of freshly distilled acetonitrile.

A.8.2.3.3 Determination

Pipette 10.0 mL of standard solution and test solution to a 50 mL flask that contains 15.0 mL of ceric ammonium nitrate solution; mix well. Within 2 min ~ 5 min, use a 1 cm cuvette to simultaneously measure the absorbance of the two solutions at the maximum absorption wavelength of 450 nm. The blank control is a mixed solution that contains 15.0 mL of ceric ammonium nitrate solution and 10.0 mL of freshly distilled acetonitrile and water in equal volumes. The absorbance of the test product shall not exceed the standard solution, that is, the total content of ethylene glycol and diethylene glycol shall not exceed 0.25%.

A.9 Determination of ignition residue

A.9.1 Reagents and materials

Sulfuric acid

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