



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 1886.72-2016

**National Food Safety Standard -- Food Additives --
Polyoxyethylene polyoxypropylene amine ether**

食品安全国家标准 食品添加剂 聚氧乙烯聚氧丙烯胺醚

(English Translation)

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1 Scope

This standard applies to ethylene oxide, propylene oxide, low molecular weight amines (such as triisopropanolamine) as the main raw material, potassium hydroxide as a catalyst, Polymerization of food additives polyoxyethylene polyoxypropylamine ether.

2.1 Chemical name

Polyoxyethylene polyoxypropylamine ether

2.2 Molecular formula

$N [(CHCH_3CH_2O)_m (C_2H_4O)_n]_3$

2.3 Structural formula

CH₃

$(CH-CH_2O)_m (CH_2-CH_2O)_n H (CHCH_3-CH_2O)_m (CH_2-CH_2O)_n H (CH-CH_2O)_m (CH_2-CH_2O)_n H CH_3$

2.4 Relative molecular mass

3000 ~ 4200 (according to the 2007 international relative atomic mass)

3.1 Sensory requirements

Sensory requirements shall comply with the requirements of Table 1.

Table 1 sensory requirements

The project requires a test method Colorless or yellowish color State oily liquid Odor slightly aromatic smell Take the appropriate sample in a clean, dry beaker, under natural light, Observe its color and state, smell its smell

3.2 Physical and chemical indicators

Physical and chemical indicators should be consistent with the provisions of Table 2.

Table 2 Physical and chemical indicators

Item Index Test Method Hydroxyl value (in KOH)/(mg/g) 40.0 to 56.0 Appendix A, A.3 Acid value (in KOH)/(mg/g) ≤ 0.50 Appendix A A.4 Cloud Point/ $^{\circ}\text{C}$ ≤ 40.0 Appendix A A.5 Lead (Pb)/(mg/kg) ≤ 2.0 GB 5009.12 Total arsenic (in As)/(mg/kg) ≤ 1.0 GB 5009.11 Appendix A Testing method A.1 Safety Tips Some of the reagents used in this test method are toxic or corrosive and should be handled with care. Such as splashing on the skin should be immediately water Rinse, severe should be treated immediately.

A.2 General provisions in addition to the provisions of this standard, the purity of the reagents used should be above the analytical level, the use of standard titration solution, impurity determination standard solution, Preparations and products should be prepared according to GB/T 601, GB/T 602 and GB/T 603, test water should be consistent with GB/T 6682 in the three Water regulations. The solution used in the test refers to the aqueous solution when it is not specified in the formulation of the solvent.

A.3 Determination of hydroxyl value (in KOH) A.3.1 Methodological Summary Under the reflux conditions, the hydroxyl groups of the sample are esterified with phthalic anhydride dissolved in pyridine. With phenolphthalein as an indicator, with hydrogen Potassium Oxide Standard titration solution Neutralize the acid produced by the esterification reaction and the excess phthalic anhydride produced by hydrolysis of the phthalic acid produced by titration the difference between the standard solution of potassium hydroxide and the amount of potassium hydroxide used in the sample solution and the blank solution to calculate the hydroxyl value.

A.3.2 Reagents and materials A.3.2.1 Pyridine. Boiling point at 114°C to 116°C .

A.3.2.2 Potassium hydroxide standard titration solution. $c(\text{KOH}) = 0.5 \text{ mol/L}$.

A.3.2.3 phenolphthalein indicator. Weigh 1 g of phenolphthalein in 100 mL of pyridine.

A.3.2.4 Phthalic anhydride. Purity of not less than 99.5%.

A.3.2.5 Phthalic anhydride pyridine solution (acylation reagent).

Preparation. $140\text{g} \pm 1\text{g}$ phthalic anhydride, placed in 2L brown glass bottle. Add 1L of pyridine and shake it vigorously until it is completely dissolved solution. If the method is determined by the method specified in GB/T 3143, the color can not be used if the color is more than 200 black units.

Concentration of the test. take 25.0mL acylation reagent in 250mL Erlenmeyer flask, add phenolphthalein indicator 4 drops to 5 drops, with potassium hydroxide labeled Titration solution titration to the end, the consumption of potassium hydroxide standard titration solution should be 83mL ~ 87mL.

A.3.3 Instruments and equipment A.3.3.1 Analysis of balance. accurate to 1mg.

A.3.3.2 Basic Burette. 50 mL.

A.3.3.3 Milling Flat Bottles. 250 mL, with a tapered glass interface.

A.3.3.4 Condenser tube. with a conical grinding glass connector, with the flask with.

A.3.3.5 Pipettes. 25 mL.

A.3.3.6 Electromagnetic stirrer.

A.3.3.7 Oil bath equipment.

A.3.4 Analysis steps A.3.4.1 Acylation Weigh the sample 7g (accurate to 0.001g) in a dry flask and add 25.0mL of phthalic anhydride pyridine solution. Will be preceded by pyrrole the eluted condensate tube is connected to the flask and swung to shake the sample in the flask. The flask was poured into an oil bath at $115^{\circ}\text{C} \pm 2^{\circ}\text{C}$ 1h, and then cooled to room temperature.

A.3.4.2 Hydrolysis and titration Rinse the condenser with pyridine, remove the flask and rinse the glass joint with water. The stirrer was placed in the flask and the flask was placed in electromagnetism Stir the mixer and turn on the stirrer. With the basic burette accurately add 50.0mL potassium hydroxide standard titration solution, add 4 drops to 5 drops of phenolphthalein Indicator, titration with potassium hydroxide titration solution to the solution was pink, and to maintain the fade for 15s is the end point.

A.3.4.3 Blank test

At the same time as the test, without the sample, with the solvent for two blank test.

The difference between the titration solution volume of the potassium hydroxide standard for the blank test and the sample is 10 mL to 15 mL.

If the difference between the two volume is greater than 15mL, indicating that the sample volume is too much (hydroxyl value than the estimated value), should reduce the amount of sample, if the difference between the two Less than 10mL, indicating that the sample volume is too small (hydroxyl value is smaller than the estimated value), should increase the amount of sample.

A.3.5 Calculation of results Hydroxyl value (in KOH) mass fraction w_1 , in milligrams per gram (mg/g), calculated according to formula (A.1).

$(V_0 - V) \times c \times M$

Where.

V_0 --- volume of potassium hydroxide standard titration solution consumed in blank solution in milliliters (mL);

V - titration of the sample solution consumed by the potassium hydroxide standard titration solution in milliliters (mL);

c - concentration of potassium hydroxide standard titration solution in moles per liter (mol/L);

M - the molar mass of potassium hydroxide in grams per mole (g/mol), [$M(\text{KOH}) = 56.10$];

m --- the quality of the sample, in grams (g).

The results of the test are based on the arithmetic mean of the parallel measurement results (leave a decimal). Two independent tests obtained under repetitive conditions the absolute difference between the results is not more than 1.5mg/g.

A.4 Determination of acid value (in KOH) A.4.1 Methodological Summary the phenol solution of the sample was titrated with the standard solution of potassium hydroxide using phenolphthalein as indicator, and the solution was titrated according to the standard of consumption of potassium hydroxide Volume calculated by acid value.

A.4.2 Reagents and materials A.4.2.1 Potassium hydroxide standard titration solution. $c(\text{KOH}) = 0.1 \text{ mol/L}$.

A.4.2.2 phenolphthalein indicator. Weigh 1 g of phenolphthalein in 100 mL of 95% ethanol solution.

A.4.2.3 Neutral Ethanol Solution. (95 5).

Remove the carbon dioxide. slowly refluxing the ethanol solution for 5 min to remove the carbon dioxide. Cooled to room temperature, add phenolphthalein indicator 4 drops ~ 5 drops, neutralized with potassium hydroxide standard solution until the solution just turned pink.

A.4.3 Instruments and equipment A.4.3.1 Balance. accurate to 1mg.

A.4.3.2 Conical Bottle. Capacity 250mL.

A.4.3.3 Basic burette. capacity 25mL.

A.4.4 Analysis steps Weigh the sample 10g (accurate to 0.001g) in a conical flask, add 100mL ethanol solution, shake the cone to completely dissolve the sample, Add 4 drops to 5 drops of phenolphthalein indicator, with potassium hydroxide standard titration solution titrated to the solution was pink, and maintain 15s does not fade is the end point. Record the amount of potassium hydroxide standard solution.

A.4.5 Calculation of results the mass fraction w_2 in grams (in KOH), in milligrams per gram