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

GB 14756-2010

Replacing GB 14756-1993

**National food safety standards -- Food additives -- Vitamin E
(dl-alpha-acetate tocopherol)
(English Translation)**

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Table of Contents

Foreword

1 Scope

2 Normative references

3 Chemical name, molecular formula, molecular mass and structural formula

3.1 Chemical Name

3.3 Formula

3.4 Relative molecular mass

4 Technical Requirements

Foreword

This standard replaces GB 14756-1993 "Food additive vitamin E (dl-alpha- tocopheryl acetate)." this standard compared with GB 14756-1993, the main changes are as follows.

- Cancel the ferric chloride color reaction bipyridine project identification and the corresponding test methods;
- An increase of infrared absorption spectroscopy to identify the project and the corresponding test methods;
- Modify the determination of vitamin E;
- Modify the determination of heavy metals.

Appendix A of this standard is a normative appendix, Appendix B is an informative annex.

This standard replaces the standards previously issued as follows.

--GB 14756-1993.

National Food Safety Standard Food additives Vitamin E (dl-alpha- tocopherol acetate)

1 Scope

This standard is suitable for use by the trimethylhydroquinone and isophytol as raw material by chemical synthesis of food additives vitamin E (DI-alpha- tocopheryl acetate).

2 Normative references

The following documents contain provisions which, through reference in this standard and become the standard terms. For dated references, only the date of the note Stage version

applies to this standard. For undated references, the latest edition (including any amendments) are applicable to this standard quasi.

3.1 Chemical Name

dl-2,5,7,8- tetramethyl-2- (4,8,12-trimethyl-tridecyl) -6-chroman-3-ol acetate Formula $C_{31}H_{52}O_3$

3.4 Relative molecular mass

472.75 (according to 2007 international relative atomic mass)

4 Technical Requirements

4.1

Sensory requirements. comply with Table 1.

Table 1 Sensory requirements

Project requires test methods Color yellow to yellow or yellow-green, the case of light color gradient depth proper amount of sample is placed in a clean, dry test tube, in natural light the line, observe the color and texture, smell the smell. Odour almost odorless Clear viscous liquid state organization

4.2

Physical indicators. to comply with Table 2.

Table 2. Physical and chemical indicators

Item Index Test Method Vitamin E ($C_{31}H_{52}O_3$), w /% 96.0 ~ 102.0 Appendix A A.4 ≤ acidity test by test A.5 in Appendix A Heavy metals (Pb)/(mg/kg) ≤ 10 Appendix A A.6 Appendix A (Normative) Testing method A.1 Safety Tips Reagents The standard test methods used for toxic or corrosive, according to the relevant provisions of the operation, should be used with caution.

If splashed on the skin should immediately wash with water, severe cases should be treated immediately. When using a volatile acid, to be in the hood.

A.2 General Provisions Unless otherwise indicated in the analysis using only confirm three analytical grade reagents and water GB/T 6682-2008 stipulated.

The test method used in the standard titration solution, impurity measurement standard solution, preparations and products, not specified in other requirements, are According to GB/T 601, GB/T 602, GB/T 603 provisions of the preparation.

A.3 Identification Test A.3.1 nitric acid coloring test A.3.1.1 Reagents and materials

A.3.1.1.1 ethanol.

A.3.1.1.2 nitrate.

A.3.1.1.3 vitamin E reference.

A.3.1.2 analysis step Weigh about 30 mg laboratory samples, after adding 10 mL of anhydrous ethanol dissolved, add 2 mL of nitric acid, shaken, and heated at 75 °C 15 min, Orange-red solution should be obvious.

A.3.2 infrared absorption spectrum test Using potassium bromide smear, according to GB/T 6040 infrared absorption spectrum, infrared spectrum should be measured with reference vitamin E the spectrum.

A.4 Determination of vitamin E A.4.1 Method summary by gas chromatography, under the selected operating conditions, the non-polar silica capillary columns by contacting the sample with separation of impurities with hydrogen Flame ionization detector, with the internal standard method to calculate the vitamin E content in the sample.

A.4.2 Reagents and materials A.4.2.1 hexane. chromatographically pure.

A.4.2.2 palmitic acid cetyl alcohol esters.

A.4.2.3 vitamin E reference.

A.4.3 Instruments and Equipment Gas Chromatograph.

A.4.4 Chromatography conditions Recommended chromatographic columns and typical operating conditions are shown in Table A.1, vitamin E (dl-alpha- tocopheryl acetate) Determination Typical Gas See Appendix B in the chromatogram Figure B.1, relative retention time of the components in Appendix B, Table B.1. Others can achieve the same degree of separation Columns and chromatographic conditions can be used.

Table A.1 column chromatography and typical operating conditions

Column length 30 m, column diameter 0.53 mm or 0.32 mm, fixative is 100% methyl polysiloxane Column temperature 280 °C Detector flame ionization detector, a temperature of 300 °C Inlet temperature of 290 °C; split injection, split ratio of 1.20; 1 µL injection volume Nitrogen carrier gas flow rate of 5 mL/min A.4.5 Analysis step A.4.5.1 Preparation of internal standard solution Weigh palmitic acid ester of cetyl alcohol amount, add n-hexane was dissolved, diluted per 1 mL solution containing 3 mg cetyl alcohol, palmitic acid esters, shake Even as the internal standard solution.

A.4.5.2 Preparation of the reference solution Weigh about 30 mg of vitamin E reference, accurate to 0.000 1 g, set 10 mL brown volumetric flask, add internal standard solution to dissolve and Diluted to the mark, shake, as the reference solution.

A.4.5.3 Preparation of sample solution Weigh about 30 mg laboratory samples, accurate to 0.000 1 g, set 10 mL brown volumetric flask, add internal standard solution and diluted to the mark, shake, as a sample solution.

A.4.5.4 system suitability test with methyl silicone as the stationary phase of silica capillary column (30 m x 0.53 mm), column temperature of 280 °C, number of theoretical plates for a living Vitamin E shall be not less than 3000, and the separation of vitamin E and the internal standard peak should be greater than 3.

Determination A.4.5.5 Content 1.0μL weighed sample solution into the gas chromatograph, record the chromatograms, another reference solution, the same method. According to the internal standard peak Calculate the area of content in the sample C₃₁H₅₂O₃.

A.4.6 Calculation Results Vitamin E (C₃₁H₅₂O₃) content mass fraction w₁, expressed in%, according to the formula (A.1), (A.2) Calculated.

$mAf_w\%$ (A.1)

twenty one

$mAf \times$

Where.

f - relative correction factor of vitamin E;

A₁ - Numerical control solution of the standard substance peak area;

A₂ - the value of vitamin E in the reference solution peak area;

A₃ - the value of vitamin E in the sample solution peak area;

A₄ - Numerical solution of the sample peak area of the standard;

m₁ - the quality of internal control solution in standard material values, in milligrams (mg);

m₂ - mass value of vitamin E in the reference solution, in milligrams (mg);

m₃ - mass of the sample solution in the sample values in milligrams (mg);

m₄ - numerical solution of the internal standard sample mass, in milligrams (mg).

Take two parallel determination results of the arithmetic average of the measurement results, the results of two parallel determination of the absolute difference is not more than 1.5%.

A.5 Determination of acidity A.5.1 Reagents and materials A.5.1.1 ethanol.

A.5.1.2 ether.

A.5.1.3 sodium hydroxide standard titration solution. c (NaOH) = 0.1 mol/L.

A.5.1.4 phenolphthalein indicator solution. 10 g/L ethanol solution.

A.5.2 Analysis step

Amount of ethanol and 15 mL, respectively, ether, set conical flask, add 0.5 mL of