

ICS 67.020
CCS C 54



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

GB 29942-2013

Food additive -- Vitamin E (dl-tocopherol)

食品添加剂 维生素E(dl- α -生育酚)

(English Translation)

Issued on: November 29, 2013

Implemented on: June 1, 2014

**Issued by: National Health and Family Planning Commission of the
People's Republic of China**

Table of Contents

- 1 Scope
- 2 Chemical name, molecular formula, molecular mass and structural formula
 - 2.1 Chemical Name
 - 2.3 Formula
 - 2.4 Relative molecular mass
- 3 Technical requirements
 - 3.1 Sensory requirements
 - 3.2 Physical and Chemical Indicators

1 Scope

This standard applies to different plant alcohol trimethyl hydroquinone as the main raw material, chemical synthesis of food additives, vitamin E (dl-alpha-Tocopherol).

2.1 Chemical Name

dl-2,5,7,8- tetramethyl-2- (4,8,12-trimethyl - tridecyl) -6-chromanol ol Formula 2.2
C₂₉H₅₀O₂

2.3 Formula

OCH₃

CH₃

CH₃ CH₃ H HCH₃ CH₃ CH₃ CH₃

2.4 Relative molecular mass

430.71 (according to 2007 international relative atomic mass)

3.1 Sensory requirements

Sensory requirements shall comply with the requirements of Table 1.

Table 1 Sensory requirements

Project requires test methods Pale yellow to amber color, exposure to air or light color becomes deep porcelain drip proper amount of sample was placed on the board, in natural light the line, observe its color and clear viscous liquid state status

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 Vitamin E (dl-alpha- tocopherol) content, w /% 96.0 ~ 102.0
 Appendix A A.3 Refractive index 20Dn 1.503 ~ 1.507 GB/T 614 Absorbance) (nm292 m1E
 71 ~ 76 in Appendix A A.4 Residue on ignition, w /% ≤ 0.1 GB/T 9741 by acidity test A.5 in
 Appendix A Lead (Pb)/(mg/kg) ≤ 2 GB 5009.12 Appendix A Testing method A.1 General
 Provisions Unless otherwise specified in this standard, the use of analytical reagent purity
 should be more than the standard titration solution, standard solution for measuring
 impurities, Preparations and products, should be GB/T 601, GB/T 602, GB/T 603
 requirements preparation, test water should be consistent with GB/T 6682-2008 in three
 Water provisions. Solution was used in the tests did not indicate what is formulated with
 solvent, it refers to an aqueous solution.

A.2 Identification Test A.2.1 solubility Insoluble in water, soluble in ethanol, miscible in ether.

A.2.2 Specific rotation test A.2.2.1 Reagents and materials Chloroform. chromatography.

A.2.2.2 instruments and equipment Polarimeter.

A.2.2.3 analysis step Weigh the sample 5.0 g, add chloroform to dissolve and dilute to 50 mL, the sample solution is placed in this polarimeter in determination, specific analysis step Step according to the provisions GB/T 613 will be. The measured specific rotation alphas (20 °C, D) should be $0 (^{\circ}) \cdot \text{dm}^2 \cdot \text{kg}^{-1} \pm 0.05 (^{\circ}) \cdot \text{dm}^2 \cdot \text{kg}^{-1}$.

A.2.3 nitric acid coloring test A.2.3.1 Reagents and materials A.2.3.1.1 ethanol.

A.2.3.1.2 nitrate.

A.2.3.2 analysis step Weigh a sample of about 10 mg, dissolved add 10 mL of anhydrous ethanol, add 2 mL of nitric acid, shaken, and heated at 75 °C water bath for 15 min, the solution it should exhibit bright red to orange.

The maximum absorption wavelength A.2.4 Test A.2.4.1 instruments and equipment UV spectrophotometer.

A.2.4.2 analysis step Weigh the sample 0.01 g, add ethanol dissolved and diluted to 200 mL, shake, placed in the scanning UV spectrophotometer at $292 \text{ nm} \pm 2 \text{ nm}$ at the maximum absorption peak due.

A.3 Determination of Vitamin E (dl-alpha- tocopherol) Content A.3.1 Method summary by gas chromatography, under the selected operating conditions, the non-polar silica capillary columns by the sample and separation of impurities with hydrogen flame Ionization detector and quantified using internal standard, the sample is calculated vitamin E (dl-alpha- tocopherol) content.

A.3.2 Reagents and materials A.3.2.1 hexane. chromatographically pure.

A.3.2.2 palmitic acid cetyl alcohol esters.

A.3.2.3 Vitamin E (dl-alpha- tocopherol) standard.

A.3.2.4 Vitamin E (dl- alpha- tocopheryl acetate) standard.

A.3.3 Instruments and Equipment Gas chromatograph with flame ionization detector.

A.3.4 reference chromatographic conditions A.3.4.1 Column. length 30 m, column diameter 0.25 mm, stationary phase polydimethylsiloxane, a thickness of 0.25 μm .

A.3.4.2 Column temperature. 280 $^{\circ}\text{C}$.

A.3.4.3 Detector. flame ionization detector, temperature 290 $^{\circ}\text{C}$.

A.3.4.4 Inlet. temperature 290 $^{\circ}\text{C}$; split injection, split ratio 1.30; injection volume was 1 μL .

A.3.4.5 carrier gas. nitrogen, flow rate of 1 mL/min.

A.3.4.6 A.3.4.1 ~ A.3.4.5 recommended chromatographic column and typical operating conditions, dl-alpha- tocopherol determination typical GC

Figure in Appendix B. Other to achieve equivalent separation columns and chromatographic conditions can be used.

A.3.5 Analysis step

A.3.5.1 Preparation of internal standard solution Weighed amount of palmitic acid ester of cetyl alcohol (internal standard), adding hexane to dissolve, dilute, shake, as the internal standard solution. This internal standard solution per ml L containing 3 mg palmitic acid cetyl alcohol esters.

A.3.5.2 Preparation of standard solutions it weighs about 30 mg of vitamin E (dl-alpha- tocopherol) standard, accurate to 0.000 1 g, placed in 10 mL brown volumetric flask, add internal standard solution Was dissolved and diluted to the mark, shake, as the standard solution.

A.3.5.3 Preparation of sample solution Weigh about 30 mg samples, accurate to 0.000 1 g, placed in 10 mL brown volumetric flask, add internal standard solution to dissolve and dilute to volume, shake Even as the sample solution.

A.3.5.4 system suitability test Weigh the same amount of vitamin E (dl-alpha- tocopherol) standard and vitamin E (dl-alpha- tocopheryl acetate) standard, add n-hexane solution Solution, formulated as per milliliter of n-hexane containing respectively 1 mg of vitamin E (dl-alpha- tocopherol) standard and 1 mg of vitamin E (dl-alpha- tocopherol acetate) A mixed solution standard. In A.3.4 reference chromatographic conditions, multiple injections, the mixed solution was subjected to chromatographic analysis, measurement resolution R, R should not less than 3.

A.3.5.5 Instrument calibration in A.3.4 reference chromatographic conditions, take A.3.5.2 standard solution consecutive injections until three consecutive needle relative response factor f is a constant (relative Deviation within 2%). According to the standard retention time qualitative measurement of vitamin E (dl- α -tocopherol) and the internal standard peak area, the phase the corresponding values were recorded as AS and AP. According to formula (A.1) to calculate the relative response factor f .

A.3.5.6 Determination of content Amount of 1.0 μL sample solution is injected into the gas chromatograph, record the chromatograms, and measure the vitamin E (dl- α -tocopherol) and the peak area of internal standard, the corresponding values recorded as A2 and A1. According to formula (A.2) calculates the sample of vitamin E (dl- α -tocopherol) content.

A.3.5.7 Calculation Results A.3.5.7.1 relative response factor Relative response factor f according to formula (A.1) Calculated.

$Af \times = (A.1)$

Where.

AS-- standard solution chromatogram of vitamin E (dl- α -tocopherol), a peak area value;

Peak area in the chromatogram of the standard solution AP-- standard material;

MP-- quality standards within the standard material solution, in milligrams (mg);

Quality mS-- standard solution of vitamin E (dl- α -tocopherol), in units of milligrams (mg).

A.3.5.7.2 content

Vitamin E (dl- α -tocopherol) content of the mass fraction w_1 according to formula (A.2) Calculated.

twenty one

$1 \times \times \times = m_A$

$m_w (A.2)$

Where.

Quality in the specimen solution m_1 -- standard material, in milligrams (mg);

f -- relative response factor;

A2-- chromatogram of the sample solution of vitamin E (dl- α -tocopherol), a peak area value;

Peak area in the chromatogram of the sample solution A1-- standard material;