



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

GB 1903.6-2015

**National food safety standard -- Food Nutrition Enhancer --
Vitamin E, Calcium succinate**

食品安全国家标准 食品营养强化剂 维生素E琥珀酸钙

(English Translation)

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1 Scope

This standard applies to vitamin E (d-alpha- tocopherol) and succinic anhydride for esterification, food and nutrition and then with calcium to form strong Vitamin E succinate calcium agent.

2 Molecular formula, relative molecular mass and structural formula

Formula 2.1

C₆₆H₁₀₆CaO₁₀

2.3 Relative molecular mass

1099.64 (according to 2011 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 Color white to pale yellow Status granular powder, unbonded the appropriate amount of sample is placed in a clean, dry white porcelain dish, under natural light, observed Color and 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 succinate calcium content (dry basis), w /% 96.0 ~ 102.0

Appendix A A.4 Alkalinity comply with Appendix A A.5 Absorbance, $E_{1\text{ m}}(286\text{nm})$ 36.0 ~ 40.0 A.6 in Appendix A Loss on drying, $w/\% \leq 2.0$ Appendix A A.7 α -tocopherol, $w/\% \leq 0.5$ Appendix A A.8 Chloride (Cl dollars), $w/\% \leq 0.212$ A.9 in Appendix A Specific rotation, $[\alpha]_{\text{D}20}^{\circ\text{C}}/[(^{\circ}) \cdot \text{dm}^2 \cdot \text{kg}^{-1}] \geq 24.0$ Appendix A A.10 Arsenic (As)/(mg/kg) ≤ 2.0 GB 5009.76 Lead (Pb)/(mg/kg) ≤ 2.0 GB 5009.12 Appendix A Testing method A.1 Safety Tips Reagents The standard test methods used for toxic or corrosive, care should be taken. As splashed on the skin should stand that is rinsed with water, severe cases should be treated immediately. When using a volatile acid, it should be carried out in a fume hood.

A.2 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, the provisions of GB/T 603 preparation, test water should be consistent with GB/T 6682-2008 in Three water regulations. Solution was used in the tests did not indicate what is formulated with solvent, it refers to an aqueous solution.

A.3 Identification Test A.3.1 nitric acid color reaction A.3.1.1 Reagents and materials

A.3.1.1.1 acetic acid.

A.3.1.1.2 ethanol.

A.3.1.1.3 nitrate.

A.3.1.2 analysis step Weigh the sample 0.05g, add 1mL of acetic acid dissolved, add ethanol 9mL when mixed with 2mL nitric acid, 75 °C water bath 15min. Light red to orange solution was presented.

A.3.2 qualitative calcium reaction A.3.2.1 Reagents and materials A.3.2.1.1 chloroform.

A.3.2.1.2 hydrochloric acid. 36% to 38%.

A.3.2.1.3 ammonia. 25% to 28%.

A.3.2.1.4 ammonium carbonate.

A.3.2.1.5 ammonium oxalate ($\text{C}_2\text{H}_8\text{N}_2\text{O}_4 \cdot \text{H}_2\text{O}$).

A.3.2.1.6 potassium chromate.

A.3.2.1.7 acetic acid.

A.3.2.1.8 10% aqueous ammonia. amount of 400mL ammonia (A.3.2.1.3) diluted with water to 1000mL.

A.3.2.1.9 ammonium carbonate solution. Weigh 20g of ammonium carbonate, plus 20mL10% dissolved ammonia, diluted with water to 100mL.

A.3.2.1.10 ammonium oxalate solution. Weigh 3.5g of ammonium oxalate, dissolved in water and diluted to 100mL.

A.3.2.1.11 Potassium chromate solution. Weigh 10g potassium chromate, dissolved in water and diluted to 100mL.

A.3.2.2 analysis step A.3.2.2.1 Weigh the sample 5g, chloroform was added to dissolve 30mL, plus 10mL hydrochloric acid, mixed with shaking and allowed to stand 10min. Water points Meet 5mL, 10% aqueous ammonia and, as the test solution and the solution of calcium a qualitative response.

A.3.2.2.2 take platinum wire, with hydrochloric acid wet, dipped in the above test solution, colorless alcohol lamp burning in the flame, the flame that was brick red.

A.3.2.2.3 test solution 1mL taken in a test tube, adding ammonium carbonate solution, a white precipitate.

A.3.2.2.4 take the test solution 1mL in a test tube, add ammonium oxalate solution, a white precipitate; plus acid insoluble precipitate, plus hydrochloric acid Shen Precipitate dissolved.

A.3.2.2.5 take the test solution 1mL in a test tube, add 10 drops of potassium chromate solution, the heating does not produce precipitation.

A.3.3 IR

Weigh pre-dried (drying method, see A.7) after sample 0.08g, was added 0.2mL of carbon tetrachloride was dissolved in the solution by Test GB/T 6040 liquid film method, and standard spectra comparison. IR spectrum shown in Figure B.1.

A.4 Determination of calcium content of vitamin E succinate A.4.1 Method summary
Vitamin E succinate in the role of calcium acetate, methyl vitamin E succinate, and then by high-performance liquid chromatography, external standard method Calculation of vitamin E succinate and calcium content.

A.4.2 Reagents and materials A.4.2.1 ethanol.

A.4.2.2 acetic acid.

A.4.2.3 methanol.

A.4.2.4 vitamin E succinate standards (TocopherolacidSuccinate, CAS. 4345-03-3).

A.4.2.5 alpha- tocopherol standards (AlphaTocopherol, CAS. 10191-41-0).

A.4.2.6 acetic acid solution. 15.

A.4.2.7 ethanol - acetic acid. 9 parts ethanol and 1 part of acetic acid solution (A.4.2.6) mixture.

A.4.3 Instruments and Equipment High performance liquid chromatograph. with UV detection, or other equivalent detectors.

A.4.4 reference chromatographic conditions A.4.4.1 Column. C18 column, 4.6mm x 250mm, particle size 5 μ m.

A.4.4.2 Mobile phase. methanol. water. acetic acid = 97.2.1.

A.4.4.3 Column temperature. 30 °C.

A.4.4.4 flow rate. 2.2mL/min.

A.4.4.5 detection wavelength. 284nm.

A.4.4.6 Injection volume. 20 μ L.

A.4.5 Analysis step

A.4.5.1 Preparation of standard solutions Weigh vitamin E succinate standard 50mg (accurate to 0.0001g), with ethanol - acetic acid dissolved solution (A.4.2.7) and a constant volume to 50mL, shake, as the standard solution. The standard solution containing 1mg per milliliter of vitamin E succinate.

A.4.5.2 Preparation of sample solution Weigh pre-dried (drying methods, see A.7) samples 50mg (accurate to 0.0001g), with ethanol - acetic acid dissolved solution (A.4.2.7) and set the volume to 50mL, shake, as the sample solution.

A.4.5.3 system suitability test Weigh vitamin E succinate standards 50mg and alpha-tocopherol standard 50mg, with ethanol - acetic acid dissolved solution (A.4.2.7) and set the volume to 50mL, shake, as a mixed standard solution. The solution contains 1mg of vitamin E succinate and 1mg per milliliter alpha- tocopherol standard. A.4.4 using chromatographic conditions, multiple injections of mixed standard solution chromatography. Vitamin E succinate Esters and alpha- tocopherol separation should not be less than 2.0. Take a mixed solution of sample 5 consecutive times, the relative standard vitamin E succinate peak area Deviation of not more than 0.8%.

A.4.5.4 Determination A.4.4 under chromatographic conditions, respectively standard solution and sample solution chromatography. According to the standard solution and the sample solution chromatogram FIG vitamin E succinate peak area, according to equation (A.1) calculates the sample of vitamin E succinate and calcium content.

A.4.6 Calculation Results Vitamin E succinate calcium content mass fraction w₁, according to equation (A.1) Calculated.

1099.6

1061.6 x 100%

Where.

Ms --- Quality standard solution of vitamin E succinate, in milligrams (mg);

Mt --- the quality of the sample, in milligrams (mg);