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

GB 29943-2013

Food additive -- Retinyl palmitate (palmitate vitamin A)

食品添加剂 棕榈酸视黄酯(棕榈酸维生素A)

(English Translation)

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National Food Safety Standard Food Additives retinyl palmitate (retinyl palmitate A)

1 Scope

This standard applies to acetic acid vitamin A (or beta- ionone) and palmitic acid as the main raw material, processed prepared food additive Palm Acid retinyl esters (vitamin A palmitate or vitamin A palmitate).

2 chemical name, molecular formula, molecular mass and structural formula

2.1 Chemical Name

3,7-dimethyl-9- (2,6,6-trimethyl-1-cyclohexen-1-yl) -2,4,6,8-nonatetraenoic en-1-palmitate
Formula 2.2 C₃₆H₆₀O₂ 2.3 formula 2.4 relative molecular mass 525.09 (according to 2007 international relative atomic mass)

3 Technical requirements

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 yellow-brown color, take appropriate sample is placed in white porcelain drip plate in natural light the line was observed (curable at low temperatures) and its color STATUS oily liquid

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 Retinyl palmitate (vitamin A palmitate) content of a/(IU/g) $\geq 5 \times 10^5$ Appendix A A.4 Acid value (in dollars KOH)/(mg/g) ≤ 2.0 A.5 in Appendix A Peroxide value/(meq/kg) ≤ 10.0 GB/T 5538 Lead (Pb)/(mg/kg) ≤ 2 GB 5009.12 Note. The

commercialization of retinyl palmitate (retinyl palmitate A) the product shall comply with the standards of retinyl palmitate (palmitic acid, vitamin A) as a raw material, may be added. Comply with the relevant food quality specifications of edible vegetable oil, starch, dextrin, sucrose, antioxidants, anti-caking agents, thickeners and other accessories made of.

a vitamin A to vitamin A alcohol or equivalent international units (IU) represents, 1μg vitamin A alcohol equivalents = 3.33 IU. Retinyl palmitate (retinyl palmitate A) the actual amount of the labeled amount of 95.0% to 110.0%.

Appendix A

Testing method

A.1 operating tips

Samples should be stored in a closed, dark and does not exceed 25 °C condition. Sample solution should be formulated as soon as possible, it is recommended to use within 2 h. Sample as the crystallization can be mixed at 65 °C condition, but the heating time not too long. Determination process, should be minimal exposure to light and air Oxygen and other oxidants, low-actinic glassware.

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 the preparation of GB/T 603, the 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.3 Identification Test A.3.1 Reagents and materials A.3.1.1 retinyl palmitate (retinyl palmitate A) reference.

A.3.1.2 cyclohexane solution. 1 g per liter of cyclohexane containing dibutylhydroxytoluene.

A.3.1.3 eluent. cyclohexane-diethyl ether (41).

A.3.1.4 chromatography on silica gel plates.

A.3.2 Analysis step

A.3.2.1 Preparation of sample solution Weighed amount of sample with cyclohexane solution formulated as per ml containing about 3.3 IU palmitic acid sample solution retinyl ester (retinyl palmitate A) of.

A.3.2.2 control solution preparation Weighed amount of retinyl palmitate (retinyl palmitate A) reference with cyclohexane solution formulated as per ml containing about 3.3 IU retinyl palmitate Huang ester (retinyl palmitate A) of the control solution.

A.3.2.3 TLC

Were weighed sample solution and control solution 3 μ L, after spotting chromatography on silica gel plates were filled with developing solvent into the chromatography tank, exhibition Open to the solvent front from the origin Chaoguo 15 cm, remove the silica gel plate, drying, UV light (at a wavelength of 254 nm) was observed. Display sample solution the main spots should be consistent with the position of the main spots of the control solution is displayed.

A.4 Determination of retinyl palmitate (retinyl palmitate A) Content A.4.1 Reagents and materials A.4.1.1 pentane.

A.4.1.2 isopropanol. chromatography.

A.4.1.3 Methanol. chromatographically pure.

A.4.1.4 Water. GB/T 6682-2008 level in water.

A.4.1.5 isopropanol solution. isopropanol containing 1 g per liter of dibutyl hydroxy toluene.

A.4.1.6 tetrabutyl ammonium hydroxide solution. 0.1 mol/L, isopropanol as solvent formulation.

A.4.1.7 all-trans vitamin A acetate standard. Press A.4.3.1 ultraviolet spectrophotometry standard content of vitamin A acetate. In test Given process, with all-trans vitamin A acetate as a standard sample, the sample solution should be in the maximum absorption peak near 326 nm, and the absorbance ratio Value A_{λ}/A_{326} comply with the requirements of Table A.1.

A.4.2 Instruments and Equipment A.4.2.1 UV spectrophotometer.

A.4.2.2 high performance liquid chromatography with UV detector (detection wavelength 326 nm).

A.4.3 Analysis step

A.4.3.1 UV spectrophotometry A.4.3.1.1 Method summary Vitamin A molecule contains 5 conjugated double bonds, in the vicinity of a wavelength of 326 nm has a maximum absorption peak position of the maximum absorption peak with the solvent does not with vary, which can be used to determine the content.

A.4.3.1.2 Preparation of sample solution Weigh the sample 25 mg ~ 100 mg, to the nearest 0.001 g, 5 mL of pentane was added, and diluted with isopropanol formulated as per ml 10 IU ~ 15 IU Sample solution retinyl palmitate (vitamin A palmitate) or vitamin A acetate.

A.4.3.1.3 Determination Take appropriate sample solution by UV spectrophotometry validation sample solution maximum absorption peak is in the vicinity of 326 nm. If the sample solution the maximum absorption peak near 326 nm, the sample solution was measured at a wavelength of 300 nm, 326 nm, 350 nm and the absorbance at 370 nm, and the count Operators A_{λ} absorbance at different wavelengths and absorbance at 326

nm A326 ratio A_{λ}/A_{326} .

If the absorbance ratio of different wavelengths in line with the requirements of Table A.1, press A.4.4 result of the calculation formulas (A.1) in the sample is calculated palmitic acid Retinyl esters (vitamin A palmitate) or vitamin A acetate content.

If the maximum absorption of the sample solution is not in the vicinity of 326 nm, or have one or more absorbance ratio does not meet the requirements of Table A.1, the A.4.3.2 shall liquid chromatography was used.

Table A.1 absorbance ratio of different wavelengths

Absorbance ratio index $A_{300}/A_{326} \leq 0.60$ $A_{350}/A_{326} \leq 0.54$ $A_{370}/A_{326} \leq 0.14$ A.4.3.2 liquid chromatography A.4.3.2.1 Method summary Retinyl palmitate (vitamin A palmitate) at tetrabutyl ammonium hydroxide, hydrolysis of vitamin A alcohol, a reverse phase column on elution with mobile phase, UV detector is measured and calculated retinyl palmitate (palmitic acid, vitamin A) content of the external standard method.

A.4.3.2.2 reference chromatographic conditions A.4.3.2.2.1 Column. C18 HPLC column, 125 mm x 4 mm, particle size 5 μm , or equivalent column.

A.4.3.2.2.2 Mobile phase. methanol. water = 95 (volume ratio).

A.4.3.2.2.3 flow rate. 1 mL/min.

A.4.3.2.2.4 detection wavelength. 325 nm.

A.4.3.2.2.5 Injection volume. 10 μL .

A.4.3.2.2.6 retention times. retention time of vitamin A alcohol is about 3 min.

A.4.3.2.3 Preparation of sample solution it weighs about 0.1 g samples, accurate to 0.0001 g, placed in 100mL flask, quickly dissolved in 5 mL of pentane. Was added 40 mL of tetrabutylammonium hydrogen Oxide hydroxide solution. Shaken gently, so that the mixture at 60 °C ~ 65 °C hydrolysis reaction is 10 min (water bath sonication may be employed). Cooling to room temperature, Diluted with isopropyl alcohol solution to 100 mL, and mix gently rocking to prevent air bubbles to obtain a sample stock solution. Pipette appropriate amount of sample stock solution with isopropanol Diluted formulated as containing about 100 IU per milliliter of sample solution palmitic acid retinyl ester (retinyl palmitate A) of.

A.4.3.2.4 Preparation of standard solution Weighed amount of all-trans vitamin A acetate standard (called sample containing about 1×10^5 IU all-trans acid vitamin A), accurate to 0.000 1 g, Placed 100 mL volumetric flask with 5 mL pentane quickly dissolved. Was added 40 mL of tetrabutyl ammonium hydroxide solution. Jiggle mix, let the mixture at 60 °C Hydrolysis reaction at ~ 65 °C 10 min (water bath sonication may be employed). Cooled to room temperature, diluted with isopropyl alcohol solution to 100 mL, and mix gently rocking Prevent bubbles, get the standard stock solution.