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

GB 14755-2010

Replacing GB/T 14755-1993

**National food safety standards -- Food additives -- Vitamin D2
(lysergic ergocalciferol)
(English Translation)**

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Foreword

This standard replaces GB 14755-1993 "Food additive vitamin D2".

This standard compared with GB 14755-1993, the main changes are as follows.

- Added Infrared Spectroscopy;
- Determination of vitamin D2 system suitability test of the sample to the vitamin D2 vitamin D3, the content was measured by HPLC the method of the internal standard method to the external standard method;
- Digitonin test checks ergosterol amended as thin layer chromatography;
- An increase of reducing substance indicators and test methods;
- An increase of heavy metal indicator and test methods;
- Increased arsenic indicators and test methods.

The Standard Annexes A and B are normative appendix Appendix C is informative appendix.

This standard replaces the standards previously issued as follows.

--GB 14755-1993

National Food Safety Standard Food additive vitamin D2 (ergocalciferol)

1 Scope

This standard applies to ergosterol for raw materials of food additive vitamin D2.

2 Normative references

The standard file referenced in the application of this standard is essential. For cited documents with dates, only the date of Version applies to this standard. For undated references, the latest edition (including any amendments) applies to this standard.

3.1 Chemical Name

9,10 open loop ergot 5,7,10 (19), 22-tetraene--3beta- alcohol Formula 3.2 C₂₈H₄₄O

3.3 Formula

CH₂

CHCH

H₃C

CH₃

CHCH

CH₃

CH (CH₃)₂

3.4 Relative molecular mass

396.66 (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 Colorless or white color 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 odorless Organization Status needle crystal or crystalline powder

4.2

Physical indicators. to comply with Table 2.

Table 2. Physical and chemical indicators

Item Index Test Method Vitamin D2 (C₂₈H₄₄O), /% 98.0 ~ 103.0 Appendix A A.4 Ergosterol, w /% ≤ 0.2 A.5 in Appendix A Specific rotation α_D^{20} (20 °C, D)/[(°) · dm² · kg⁻¹] 102.0 ~ 107.0 A.6 in Appendix A Mass absorption coefficient α (265nm)/(L/cm · g) 46 ~ 49 in Appendix A A.7 Reducing substance (tetrazolium color test), w /% ≤ 0.002 Appendix A A.8 Arsenic (As)/(mg/kg) ≤ 2 Appendix A A.9 Heavy metals (Pb)/(mg/kg) ≤ 20 Appendix A A.10 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 the Splashed on the skin should immediately wash with water, severe cases should be treated immediately. When using a volatile acid, to be carried out in a fume hood.

A.2 General Provisions the reagents used in this standard, unless otherwise noted, only in the analysis confirmed analytically pure reagents and GB/T 6682-2008 in Regulation Given three water.

Test methods used in the determination of impurities standard solution, preparations and products, did not indicate when the other requirements, according to GB/T 602, GB/T 603 provisions of the preparation.

A.3 Identification Test A.3.1 acetic anhydride concentrated sulfuric acid coloring test A.3.1.1 Reagents and materials A.3.1.1.1 chloroform.

A.3.1.1.2 acetic anhydride.

A.3.1.1.3 sulfuric acid.

A.3.1.2 analysis step Laboratory samples taken from about 0.5 mg, after adding 5 mL chloroform dissolved, add 0.3 mL of acetic anhydride and 0.1 mL of sulfuric acid, shaking, previews yellow, Gradient red to purple immediately, and finally turns green.

A.3.2 IR test

Using potassium bromide tablet method according to GB/T 6040 test, IR spectra laboratory samples should be consistent (for the control of According to Atlas, see Appendix B).

A.4 Determination of vitamin D2 A.4.1 Method summary by high performance liquid chromatography, under the selected operating conditions, the sample was separated by column chromatography with UV absorbance detector, with external standard method to calculate the content of the sample of vitamin D2.

A.4.2 Reagents and materials A.4.2.1 pentanol. chromatography.

A.4.2.2 hexane. chromatographically pure.

A.4.2.3 isooctane. chromatography.

A.4.2.4 vitamin D2 reference.

A.4.2.5 vitamin D3 reference.

A.4.3 Instruments and Equipment High performance liquid chromatography (HPLC).

A.4.4 Chromatography conditions Recommended chromatographic columns and typical operating conditions are shown in Table A.1, vitamin D3 HPLC system suitability test diagram see Fig.

C.1, the relative retention time of the components in Table C.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 250mm, column diameter 4.6mm, silica gel column the mobile phase n-hexane - n-amyl alcohol (10 003) A flow rate of 2 mL/min Detector wavelength 254 nm
A.4.5 Analysis step A.4.5.1 Preparation of vitamin D3 reference system suitability test stock solution Weigh about 25 mg of vitamin D3 reference, accurate to 0.000 1 g, brown set 100 mL volumetric flask, add 80 mL iso-octane, Sonication solubilization 1min, avoid heated to complete dissolution, coupled with iso-octane to the mark, shake nitrogen Mesa, dark, below 0 °C save.

A.4.5.2 laboratory sample preparation solution it weighs about 25 mg laboratory samples, accurate to 0.000 1 g, brown set 100 mL volumetric flask, add 80 mL iso-octane, with Sonication solubilization 1min, avoid heated to complete dissolution, coupled with iso-octane to the mark. Measure 5 mL $\pm$ 0.05 mL above Solution, and placing 10 mL brown volumetric flask, add hexane to the mark.

A.4.5.3 Preparation of standard solutions of Vitamin D2 Weigh about 25 mg of vitamin D2 reference, accurate to 0.000 1 g, brown set 100 mL volumetric flask, add 80 mL iso-octane, Sonication solubilization 1min, avoid heated to complete dissolution, coupled with iso-octane to the mark, amount of the above solution 5 mL $\pm$ 0.05 mL, Set 10 mL brown volumetric flask, add hexane to the mark.

A.4.5.4 system suitability test Silica gel as a filler column, hexane - n-amyl alcohol (10 003) as the mobile phase, detection wavelength 254 nm, flow rate of about 2 mL/min. Amount of vitamin D3 reference substance stock solution 5 mL, set stoppered glass container, after nitrogen Mesa, set 90 °C water bath 1h, Remove rapid cooling, n-hexane 5 mL $\pm$ 0.05 mL, shake, set stoppered 1 cm quartz absorption cell in two main wavelengths of 254 nm and a 365 nm UV lamp, a quartz absorption cell diagonal 45 °, and from the tube 5 cm ~ 6 cm, irradiation 5min, the solution containing Provitamin D3, trans-vitamin D3, vitamin D3 and speed sterol D3. Of this solution into the liquid chromatograph to measure the peak of vitamin D3 Value, has injection five times the relative standard deviation should not exceed 2.0%, provitamin D3 (vitamin D3 than the retention time of 0.5) and Trans-vitamin D3 (vitamin D3 than the retention time of approximately 0.6) and vitamin D3 and D3-speed sterols (vitamin D3 retention ratio Time is about 1.1), a peak separation should be greater than 1.0.