

ICS 65.120
CCS B 46



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

GB 9840-2017

Replacing GB/T 9840-2006

Feed additive - Vitamin D3 (powder form)

饲料添加剂 维生素D3（微粒）

Issued on: October 14, 2017

Implemented on: May 1, 2018

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine;
Standardization Administration of the PRC**

Table of Contents

1	Scope
2	Normative references
3	Requirements
4	Test method
4.2	Identification
4.2.1	Reagents and solutions
4.3.2	Reagents and solutions
4.3.2.11	L-ascorbic acid solution. Weigh
4.3.3	Instruments and equipment
4.3.4.1	Preparation of standard solution
4.3.4.4	Determination of pre-vitamin D3 correction factor Pipette
4.6	Heavy metals
4.6.1	Reagents and solutions
4.6.2	Analytical procedures
4.7	Total arsenic
4.7.1	Reagents and solutions
4.7.2	Analytical procedures
4.7.2.1	Preparation of specimen arsenic spot Weigh
5	Inspection rules
6	Labeling, packaging, transportation, storage
7	Shelf life

Foreword

Chapter 1, Chapter 3, Chapter 5 of this standard are mandatory; the rest are recommended. This standard was drafted in accordance with the rules given in GB/T 1.1-2009. This standard replaces GB/T 9840-2006 "Feed additive - Vitamin D3 beadlets". Compared with GB/T 9840-2006, the main differences on the technical content of this standard are as follows: - In this standard, ADD the relevant technical requirements for the water- dispersed products of feed additive vitamin D3 (powder form). - The English name of the original standard is "Feed additive - Vitamin D3 beadlets". In this standard, CHANGE the name into "Food additive - Vitamin D3 (powder form)". - In the original standard, it is stated that "This standard applies to... as a vitamin feed additive". In this standard,

CHANGE it into that "This standard applies to the common feed additive vitamin D3 (powder form), which is made using the feed additives vitamin D3 oil as raw materials, supported with a certain amount of antioxidants, adding such auxiliary materials as gelatin, starch; as well as the water-dispersible feed additive vitamin D3 (powder form), which is made by adding such auxiliary materials as maltodextrin and emulsifiers. The auxiliary materials shall comply with the provisions of the "Catalogue of feed ingredients", "Catalogue of feed additives", "Feed hygienic standards". - CHANGE the chemical name "9,10-opened cholesteryl-5,7,10(19)-triene- 3 β -ol" in the original standard, into "(3 β ,5Z,7E)-9, 10-opened cholesteryl- 5,7,10(19)-trien-3-ol" in this standard. - CHANGE the range of the content in the technical indicators in the labeling amount from "90.0% ~ 120.0%" in the original standard, into "90.0% ~ 110.0%" in this standard. - In the technical indicators of this standard, ADD the limit requirements for the hygienic indicators, such as heavy metals, total arsenic, chromium. - In the determination method of the vitamin D3 content, CHANGE the detection wavelength from 265 nm to 254 nm. MODIFY the determination method of the vitamin D3 correction factor. ADD the applicability of the system. CHANGE concentration of the standard solution from 10 μ g/mL to 50 μ g/mL. CHANGE the sample weighing amount from

0.2 g ~

0.5 g to about 1 g (accurate to

0.1 mg). In the second method, ADD the "constant volume of n-hexane extract". - In the original standard, it is stated that "the shelf life is not less than 12 months". In this standard, CHANGE it into that "the shelf life of ordinary products in the original packaging is 24 months; the shelf life of water- dispersible products is 12 months". - ADD appendix A (informative). This standard was proposed by AND shall be under the jurisdiction of the National Feed Industry Standardization Technical Committee (SAC/TC 76). Drafting organizations of this standard. Zhejiang Veterinary Drug and Feed Supervision Institute, Zhejiang Garden Biotech Co., Ltd., Zhejiang Pharmaceutical Co., Ltd. The main drafters of this standard. Shi Xingfen, Zhu Congying, Lu Weijun, Zhang Caiju, Jiang Hongjun, Chen Yong, Qian Guoping, Yao Yuanyuan, Huang Juan, Zhou Zhiqiang, Mei Na, Qiu Chengjun, Zhou Fengchao. This standard replaces the standard previously issued as follows: - GB/T 9840-1988, GB/T 9840-2006. Food additive - Vitamin D3 (powder form)

1 Scope

China's mandatory standard for vitamin D3 in powder form used as a feed additive - the supplement that governs calcium and phosphorus metabolism, and whose absence produces rickets in growing animals and, in laying hens, thin shells and eventually no shells at all. Animals kept indoors cannot make their own vitamin D from sunlight, which is why in modern production the supplement is not an improvement but a precondition. It specifies the requirements, the test methods, the inspection rules and the labelling, packaging,

transport, storage and shelf life of feed additive vitamin D3 in powder form. The words powder form in the title carry the substance of the standard. Vitamin D3 itself is an oil and one of the least stable of the vitamins: it is destroyed by oxygen, by heat, by light, by trace metals and by the acidity and moisture it meets in a premix, and it cannot survive in a feed mill as such. So it is supplied as a formulated product, the active substance dispersed in a matrix with an antioxidant and spray-dried or coated into beadlets, and the standard therefore has to specify not just how much vitamin is present but how well it is protected. The requirements accordingly cover the assay expressed in international units, the appearance and the particle size, the loss on drying, the flowability, and - most importantly - the stability, both on storage and through the conditions of pelleting, together with the limits on the heavy metals and on the residues of the synthesis. The packaging, which must exclude air and light, and the stated shelf life are part of that same requirement. The feed hygiene limits of GB 13078 apply throughout. Issued on 14 October 2017 and in force since 1 May 2018, it replaces GB/T 9840-2006.

This standard specifies the requirements, test methods, inspection rules, labeling, packaging, transportation, storage, shelf life of feed additive vitamin D3 (powder form) products. This standard applies to the common feed additive vitamin D3 (powder form), which is made using the feed additives vitamin D3 oil as raw materials, supported with a certain amount of antioxidants, adding such auxiliary materials as gelatin, starch; as well as the water-dispersible feed additive vitamin D3 (powder form), which is made by adding such auxiliary materials as maltodextrin and emulsifiers. The auxiliary materials shall comply with the provisions of the "Catalogue of feed ingredients", "Catalogue of feed additives", "Feed hygienic standards". Chemical name. (3beta, 5Z, 7E)-9,10-opened cholesteryl-5,7,10(19)-trien-3-ol Molecular formula. C₂₇H₄₄O Relative molecular mass. 384.64 (2007 international relative atomic mass) Chemical Structure.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 601 Chemical reagent - Preparations of standard volumetric solutions

GB/T 603 Chemical reagent - Preparations of reagent solution for use in testing method

GB/T 6435 Determination of moisture in feedstuffs

GB/T 6682 Water for analytical laboratory use - Specification and test methods

3 Requirements

3.1 Product specifications 3.1.1 500000 IU/g.

3.2 Appearance and traits This product is beige to yellow-brown particles, which has fluidity. It is easy to degrade, when exposed to heat, light or moisture, resulting in a decrease in content

3.3 Technical indicators The technical indicators shall meet the requirements of Table 1.

4 Test method

Except for special instructions, the reagents used are all analytically pure. The water, which is used in chromatographic and spectroscopic analysis, shall meet the requirements for first-grade water in GB/T 6682.

4.1 Sensory inspection Take an appropriate amount of sample. Place it in a clean, dry white porcelain dish.

4.2.1 Reagents and solutions

4.2.1.1 Trichloromethane.

4.2.1.2 Acetic anhydride.

4.2.2 Identification test Weigh about 100 mg of specimen. Add 10 mL of chloroform (4.2.1.1). Grind for a few minutes. Filter it.

4.3 Vitamin D3 content Warning. The detection process shall be operated in the dark!

4.3.1 Principle Vitamin D3 in the specimen is saponified and refluxed (the first method) or ultrasonically extracted (the second method). After extraction with n-hexane, it is injected into the chromatographic column, separated by mobile phase elution, measured at 254 nm. The external standard method is used to calculate the vitamin D3 content.

4.3.2 Reagents and solutions

4.3.2.8 Vitamin D3 standard product. content $\geq 99.0\%$ (1 IU = 0.025 μg).

4.3.2.9 Potassium hydroxide solution. 500 g/L.

4.3.2.11 L-ascorbic acid solution. Weigh

3.5 g of L-ascorbic acid. Dissolve it in 20 mL of sodium hydroxide solution (4.3.2.10). This solution shall be newly prepared before use.

4.3.2.12 Phenolphthalein indicator solution. It is prepared according to GB/T 603.

4.3.2.13 Glycerol starch lubricant. Weigh 22 g of glycerol. Add 9 g of soluble starch. Heat to 140 °C. Keep it for 30 min. Stir constantly. Let it cool naturally.

4.3.2.14 Sodium chloride solution. 100 g/L.