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Replacing GB/T 9840-2006

Food additive - Vitamin D_3 (powder form)

饲料添加剂 维生素 D_3（微粒）

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

Standardization Administration of PRC.

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Food additive - Vitamin D_3 (powder form)

1 Scope

This standard specifies the requirements, test methods, inspection rules, labeling, packaging, transportation, storage, shelf life of feed additive vitamin D_3 (powder form) products.

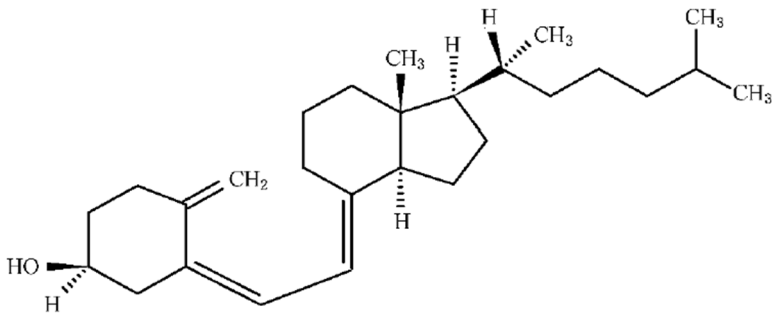
This standard applies to the common feed additive vitamin D_3 (powder form), which is made using the feed additives vitamin D_3 oil as raw materials, supported with a certain amount of antioxidants, adding such auxiliary materials as gelatin, starch; as well as the water-dispersible feed additive vitamin D_3 (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: (3β, 5Z, 7E)-9,10-opened cholesteryl-5,7,10(19)-trien-3-ol

Molecular formula: $C_{27}H_{44}O$

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

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 D_3 content.

4.3.2 Reagents and solutions

4.3.2.1 n-hexane.

4.3.2.2 n-hexane (chromatographically pure).

4.3.2.3 n-pentanol (chromatographically pure).

4.3.2.4 Absolute ethanol.

4.3.2.5 95% ethanol.

4.3.2.6 Anhydrous sodium sulfate.

4.3.2.7 Butylated hydroxytoluene (BHT).

4.3.2.8 Vitamin D_3 standard product: content ≥ 99.0% (1 IU = 0.025 μg).

4.3.2.9 Potassium hydroxide solution: 500 g/L.

Note: Potassium hydroxide solution is a strong corrosive liquid. Operators need to wear protective glasses and gloves, to prevent burns. This solution shall be newly prepared before use.

4.3.2.10 Sodium hydroxide solution: c (NaOH) = 1 mol/L, which is prepared according to GB/T 601.

Note: Same as 4.3.2.9.

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.

4.3.2.15 Hydrochloric acid solution I: c (HCl) = 1 mol/L, which is prepared according to GB/T 601.

4.3.2.16 Hydrochloric acid solution II: c (HCl) = 0.01 mol/L. Pipette 1 mL of

uses 5 mL of water each time. Take out the saponification bottle. Use running water to quickly cool it down. Transfer the saponification solution into a 500 mL separatory funnel [the piston of the separatory funnel is coated with glycerol starch lubricant (4.3.2.13)]. Use water to wash the saponification bottle twice, which uses 5 mL of water each time. Then use n-hexane (4.3.2.1) to wash it twice, which uses 10 mL each time. Combine the washing solution into a 500 mL separatory funnel. Add 60 mL of n-hexane (4.3.2.1) for extraction. Let it stand for stratification. Transfer the water layer into a 250 mL separatory funnel. Then use n-hexane (4.3.2.1) to make extraction for 2 times, 50 mL each time. Discard the water layer. Collect the extract in a 500 mL separatory funnel. First use 80 mL of sodium chloride solution (4.3.2.14) to wash the n-hexane layer. Then use water to wash it several times (50 mL ~ 80 mL of water each time; slowly turn it during washing, to avoid emulsification), until the water layer does not appear red, when meeting the phenolphthalein indicator solution (4.3.2.12). The extract is filtered by a funnel, which is paved with absorbent cotton and anhydrous sodium sulfate (4.3.2.6). The filtrate is placed in a 250 mL brown volumetric flask. Use n-hexane (4.3.2.1) to wash the funnel 3 ~ 5 times. Combine the washing solution into the volumetric flask. Then use n-hexane (4.3.2.1) to dilute it to the mark. Shake well, to obtain the specimen solution.

4.3.4.2.2 The second method (ultrasonic extraction method)

Weigh about 1 g of the specimen (accurate to 0.1 mg, equivalent to 5.0×10^5 IU of vitamin D_3). Add 10 mL of hydrochloric acid solution I (4.3.2.15), in a 250 mL brown volumetric flask. Ultrasonically extract it in a water bath, at 50 °C, for 5 min. Cool it down. Add absolute ethanol (4.3.2.4) to about 80% of the volumetric flask. Ultrasound for 5 minutes, at room temperature. Cool it down. Use absolute ethanol (4.3.2.4) to make its volume reach to the mark.

Add 17 mL of hydrochloric acid solution II (4.3.2.16) and 30 mL of n-hexane (4.3.2.1) in a 250 mL separatory funnel. Add 25.00 mL of the above specimen solution. Shake for 5 min. Discard the water layer. Pour the n-hexane layer into a 50 mL brown volumetric flask. Use a small amount of n-hexane (4.3.2.1) to rinse the separatory funnel twice. Merge it into the volumetric flask. Then use n-hexane (4.3.2.1) to make its volume reach to the mark. Shake well, to obtain the specimen solution.

4.3.4.3 Determination

4.3.4.3.1 Reference chromatographic conditions

The reference chromatographic conditions are as follows:

- Chromatographic column: Silica gel, column length 250 mm, inner diameter 4.6 mm, particle size 5 μm or equivalent;

4.6.1.4 Lead standard solution: 1000 μg/mL.

4.6.1.5 Ammonia solution (10%): It is prepared according to GB/T 603.

4.6.1.6 Hydrochloric acid solution III: Take 63 mL of hydrochloric acid. Add an appropriate amount of water, to make it reach to 100 mL. Shake well.

4.6.1.7 Hydrochloric acid solution IV: Take 18 mL of hydrochloric acid. Add an appropriate amount of water to make it reach to 100 mL. Shake well.

4.6.1.8 Sodium hydroxide solution: 40 g/L.

Note: Sodium hydroxide solution is a strong corrosive liquid. Operators need to wear protective glasses and gloves, to prevent burns.

4.6.1.9 Thioacetamide solution: Take 4 g of thioacetamide. Add water to dissolve it to 100 mL. Store it in the refrigerator. Before use, take 1.0 mL, as well as 5.0 mL of the mixed solution [composed of 15 mL of sodium hydroxide solution (4.6.1.8), 5.0 mL of water, 20 mL of glycerol]. Heat it on a water bath for 20 s. Mix well. Cool it down. Use it immediately.

4.6.1.10 Acetate buffer (pH 3.5): Take 25 g of ammonium acetate. Add 25 mL of water to dissolve it. Add 38 mL of hydrochloric acid solution III (4.6.1.6). Use hydrochloric acid solution IV (4.6.1.7) or ammonia solution (4.6.1.5), to make accurate adjustment, to make the pH reach to 3.5 (indicated by the potentiometer). Use water to dilute it to 100 mL. Shake well.

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

4.6.1.12 Preparation of lead standard working solution: Take 2.00 mL of lead standard solution (4.6.1.4). Put it in a 200 mL measuring flask. Use water to dilute to the mark. Shake well (each milliliter is equivalent to 10 μg of Pb).

4.6.2 Analytical procedures

4.6.2.1 Preparation of specimen solution

Weigh 1 g of the specimen (accurate to 10 mg). Place it in a porcelain crucible. Slowly burn it, until it is completely carbonized. Let it cool naturally. Add 0.5 mL ~ 1 mL of sulfuric acid (4.6.1.1) to make it wet. Heat it at low temperature, until the sulfuric acid vapor is exhausted. Burn it at 550 °C, to completely ash it. Let it cool naturally. Add 0.5 mL of nitric acid (4.6.1.2). Evaporate to dryness, until the nitrogen oxide vapor is exhausted. Let it cool naturally. Add 2.0 mL of hydrochloric acid (4.6.1.3). Put it in a water bath. Evaporate to dryness. Add 15 mL of water. Add dropwise ammonia solution (4.6.1.5), until the p-phenolphthalein indicator solution (4.6.1.11) is slightly red. Then add 2.0 mL of

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

4.7.1.11 Preparation of arsenic standard working solution: Take 5.00 mL of arsenic standard solution (4.7.1.4). Put it in a 100 mL measuring flask. Use water to dilute to the mark. Shake well. Then take another 2.00 mL of the solution. Put it in a 100 mL measuring flask. Use water to dilute to the mark. Shake well (each milliliter is equivalent to 1 μg of As).

4.7.1.12 Mercury bromide test paper: It is prepared according to GB/T 603. It is stored in a brown mouth-ground bottle.

4.7.1.13 Lead acetate cotton: Take absorbent cotton. Immerse it in a mixed solution of lead acetate solution (4.7.1.9) and water, at equal volume. After soaking it, drain the excess solution. Make it loose. After drying at below 100 °C, store it in a glass bottle with ground stopper, for later use.

4.7.2 Analytical procedures

4.7.2.1 Preparation of specimen arsenic spot

Weigh 1.0 g of specimen (accurate to 10 mg) in a porcelain crucible. Add 10 mL of magnesium nitrate solution (4.7.1.5) and 1 g of magnesium oxide (4.7.1.2). Mix well. Soak for 4 h. Evaporate dryness on low temperature or water bath. Slowly burn with a small fire, until it is fully carbonized. Let it cool naturally. Burn it at 550 °C to completely ash it. Let it cool naturally. Add 2 mL of water to moisten the ash. Add 1 drop of phenolphthalein indicator solution (4.7.1.10). If it is red, add dropwise hydrochloric acid solution (4.7.1.6), until the red color fades. Transfer it to a conical flask. Use 21 mL of water to wash the porcelain crucible, for several times. Combine the washing solution into the conical flask. Then add 5 mL of hydrochloric acid (4.7.1.1), 5 mL of potassium iodide solution (4.7.1.7), 5 drops of acidic stannous chloride solution (4.7.1.8). After standing at room temperature for 10 minutes, add 2 g of arsenic-free zinc (4.7.1.3). Immediately seal the air-way tube, where there is mercury bromide test paper (4.7.1.12) on the top end plane AND loaded with lead acetate cotton (4.7.1.13), into the conical flask. Place the conical flask in a water bath of 25 °C ~ 40 °C, to react for 45 min. Take out the mercury bromide test paper, to obtain it.

4.7.2.2 Preparation of standard arsenic spot

Take another reagent for preparing the specimen arsenic spot. Put it in a porcelain crucible. Treat it in the same way as the specimen. Transfer it into a conical flask. Add 5 mL of hydrochloric acid (4.7.1.1) and 21 mL of water. Then add 2.00 mL of arsenic standard working solution (4.7.1.11). Operate in the same way from "Potassium iodide solution" under "Preparation of specimen arsenic spot" (4.7.2.1).

c) When production is resumed after suspension for more than three months;

d) When there is a big difference between the exit-factory inspection result and the last type inspection result.

5.5 Judgment rules

When an indicator of the inspection result does not meet the requirements of this standard, it shall be re-sampled from twice the volume of the packaging unit, for re-inspection. If there is still an indicator that does not meet the requirements of this standard in the re-inspection result, the entire batch of products is judged as unqualified.

6 Labeling, packaging, transportation, storage

6.1 Label

It shall meet the requirements of GB 10648.

6.2 Packaging

Use airtight, light-proof packaging. The packaging materials shall be non-toxic and harmless. It shall meet the requirements of the corresponding standards.

6.3 Transportation

This product shall be protected from light, moisture, high temperature, during transportation. The packaging shall not be damaged. It shall not be mixed with toxic and harmful substances.

6.4 Storage

This product shall be stored in a ventilated, dry and dark place below 25 °C. It is forbidden to store it, together with toxic and harmful substances.

7 Shelf life

Under the specified packaging and storage conditions, the shelf life of ordinary products in the original packaging is 24 months; the shelf life of water-dispersible products is 12 months.

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