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GB

NATIONAL STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

GB 5413.10-2010

National food safety standard

Determination of vitamin K_1 in foods for infants and young children, milk and milk products

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Foreword

This Standard equivalently adopts AOAC 999.15 *Vitamin K in Milk and Infant Formulas Liquid Chromatographic Method*.

This Standard replaces GB 5413.10-1997 *Milk powder and formula foods for infant and young children - Determination of Vitamin K_1 content.*

Compared with GB 5413.10-1997, the main changes in this Standard are as follows:

— The standard name is modified to *Determination of vitamin K_1 in foods for infants and young children, milk and milk products.*

— The treatment of sample is modified as: After enzyme hydrolysis, use NaOH to saponify; and use normal hexane to extract;

— The determination is changed to: Use high performance liquid chromatography column and then reduction fluorescence method to quantitatively determine vitamin K_1;

— In the instruments, the “High pressure liquid chromatography with UV detector” is modified to “High pressure liquid chromatography with fluorescence detector”.

Annexes A, B, and C of this Standard are informative.

The previous editions replaced by this Standard are:

— GB/T 5413-1985, and GB/T 5413.10-1997.

National food safety standard

Determination of vitamin K_1 in foods for infants and young children, milk and milk products

1 Scope

This Standard specifies the method for determination of Vitamin K_1 in foods for infants and young children, milk and milk products.

This Standard is applicable to the determination of Vitamin K_1 in foods for infants and young children, milk and milk products.

2 Normative references

The following standards contain provisions which, through the reference in this text, constitute provisions of this Standard. For the dated references, all the amendments or revisions after them, except the corrigenda, are not applicable to this Standard. For the references that are not dated, their most recent editions are applicable to this Standard.

3 Principle

Use lipase to degrade the fats and unsaturated fatty acids in test sample. For the sample containing starch, it needs to use starch amylase to degrade the starch in sample; after saponification by alkali, use normal hexane to extract Vitamin K_1. After separation by liquid chromatography, post-column reduction of Vitamin K_1 is carried out. The fluorescence detector is used for detection; and external standard method is used for quantification.

4 Reagents and materials

Unless otherwise specified, all reagents used in this method are analytically pure. Water is the Grade 1 specified in GB/T 6682.

4.1 Sodium hydroxide solution (10 mol/L): Prepare before use.

4.2 95% ethanol.

4.3 Saturated sodium chloride solution.

4.4 Normal hexane: Chromatographically pure.

4.5 Anhydrous sodium sulfate.

4.6 Methanol: Chromatographically pure.

4.7 Dichloromethane: Chromatographically pure.

4.8 Glacial acetic acid.

4.9 Zinc chloride.

4.10 Anhydrous sodium acetate.

4.11 Mobile phase: 900 mL of methanol (4.6), 100 mL of dichloromethane (4.7), 0.3 mL of glacial acetic acid (4.8), 1.5 g of zinc chloride (4.9), and 0.5 g of anhydrous sodium acetate (4.10); after dissolution, use 0.45 μm membrane to filter.

4.12 Amylase: Enzyme activity ≥ 1.5 U/mg.

4.13 Lipase: Enzyme activity ≥ 700 U/mg.

4.14 Zinc powder: Particle size: 50μm ~ 70 μm.

4.15 Standard solution of Vitamin K_1: The calibration method of standard solution concentration is shown in Annex A.

4.15.1 Standard stock solution of Vitamin K_1 (2 mg/mL): Weigh 0.05 g of standard substance of Vitamin K_1 (accurate to 0.1 mg). Put into a 25 ml volumetric flask. Use normal hexane to dissolve and fix the volume.

4.15.2 Standard intermediate solution of Vitamin K (20 μg/ml). Take 1 mL of standard stock solution (4.15.1). Add normal hexane to fix the volume to 100 mL.

5 Instruments and apparatuses

5.1 High-pressure liquid chromatograph: Equipped with fluorescence detector.

5.2 Balance: Sensitivity is 0.1 mg.

5.3 Separating funnel: 250 ml.

5.4 Rotatory evaporator.

5.5 Constant-temperature air bath shaker.

5.6 Centrifuge: Rotary speed ≥ 3000 rpm.

5.7 Nitrogen evaporator.

6 Analytical procedure

6.1 Pretreatment of sample

6.1.1 Starch-contained sample

Weigh about 2.5 g of solid sample that has been mixed well or 10 g of liquid sample (accurate to 0.1 mg). Put into a conical flask. Add 0.5 g of amylase (4.12). Dissolve it with 30 mL of warm water.

6.1.2 Starch-free sample

Annex B

(Informative)

Filling method of zinc reduction column

Fill the zinc powder (4.14) densely into the reduction column (4.6 mm × 50 mm, stainless steel material). During the filling, the zinc powder shall be filled in small amount and multiple times; gently slap it while filling; so as to fill in the zinc powder densely.

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