NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

中华人民共和国国家标准

GB 5413.10-2010

National food safety standard Determination of vitamin K_1 in foods for infants and young children, milk and milk products

食品安全国家标准

婴幼儿食品和乳品中维生素 K_1 的测定

Issued on Mar 26, 2010 **Implemented on Jun 01, 2010**

Issued by the Ministry of Health of P.R of China

Foreword

This present National Standard is identical to AOAC Official Method 999.15, *Vitamin K in Milk and Infant Formulas Liquid Chromatographic Method*.

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

When compared with GB 5413.10 -1997, the following modifications have been carried out in this present National Standard:

— The standard name is modified as 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, the sample should be saponified by NaOH and extracted by normal hexane";

— in this present National Standard, it has been modified as that, after HPLC column, reduction fluorescence method is used for quantitative determination of Vitamin K_1;

— In the apparatus, the original "liquid chromatograph with UV detector" is modified as "HPLC with fluorescence detector".

Annexes A, B, and C of this present National Standard are all informative annexes.

The original editions replaced by this present National Standard include:

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

This present National 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 reference in this text, constitute provisions of this present standard. As for the dated references, all the amendments or revisions after them except the corrigenda are not applicable to this present standard. As for the references that are not dated, their most recent editions are applicable to this present national standard.

3. Principle

The fats and unsaturated fatty acids in test sample are degraded by lipase first; then, for the starch of the samples requiring degradation with starch amylase, after saponification by alkali, Vitamin K_1 is extracted by normal hexane. 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, purity of all reagents used in this present method is analytically pure, and that of water used in the test is the third-graded specified in GB/T 6682.

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

4.2 95% ethyl alcohol.

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, it is filtered through a 0.45 μm membrane.

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