NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.84-2016

National food safety standard - Determination of vitamin B1 in foods

Issued on: September 6, 2023 **Implemented on: September 6, 2024**

Issued by: National Health and Family Planning Commission

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Foreword

This standard replaces GB/T 5009.84-2003 "Determination of thiamine (vitamin B1) in foods", GB 5413.11-2010 "National food safety standard - Determination of vitamin B1 in foods for infants and young children, milk and milk products", GB/T 7628- 2008 "Determination of vitamin B1 in cereals", GB/T 9695.27-2008 "Meat and meat products - Determination of vitamin B1 content".

As compared with GB/T 5009.84-2003, the main changes of this standard are as follows.

- CHANGE the standard name into "National food safety standard - Determination of Vitamin B1 in foods";

- ADD the high performance liquid chromatography as the first method, USE the fluorescence spectrophotometry as the second method;

- MODIFY the expression of detection limit, ADD the quantitative limits of method;

- ADD the qualitative identification method of chloride ion in the artificial zeolite pretreatment;

- ADD the color change characteristics of the solution when using the bromocresol green as the indicator;

- DELETE the Figure 1 (reaction bottle) and Figure 2 (base salt exchange tube) structure;

- ADD the weighing amount of artificial zeolite when it is expressed in wet weight.

National food safety standard -

Determination of vitamin B1 in foods

1 Scope

This standard specifies the method for determining the vitamin B1 in foods by the high performance liquid chromatography and fluorescence spectrophotometry.

This standard applies to the determination of vitamin B1 content in food.

Method 1.High performance liquid chromatography

2 Principles

Samples are hydrolyzed, neutralized, then rehydrated in dilute hydrochloric acid medium at constant temperature. The hydrolyzate is derivatized by alkaline potassium ferricyanide solution, extracted with n-butanol, separated by C18 reversed-phase column, detected by high performance liquid chromatography-fluorescence detector, quantified by external standard method.

3 Reagents and materials

Unless otherwise indicated, the reagents used in this method are of analytical pure, the water is level I water as specified in GB/T 6682.

3.2.1 Potassium ferricyanide solution (20 g/L). WEIGH 2 g of potassium ferricyanide, USE water to dissolve it, MAKE its volume reach to 100 mL, SHAKE it uniformly. PREPARE it before use.

3.2.6 Sodium acetate solution (0.05 mol/L). WEIGH 6.80 g of sodium acetate, ADD 900 mL of water to dissolve it, USE glacial acetic acid to adjust the pH between 4.0 to 5.0, ADD water to make the volume reach to 1000 mL. USE

3.2.7 Sodium acetate solution (2.0 mol/L). WEIGH 27.2 g of sodium acetate, USE water to dissolve it, MAKE its volume reach to 100 mL, SHAKE it uniformly.

3.3 Standard substances

Vitamin B1 standard substance. thiamine hydrochloride (C12H17ClN4OS - HCl)), CAS. 67-03-8, purity >= 99.0%.

5.1.1 Liquid or solid powder samples. Immediately after the sample is well- mixed, MAKE determination in time or PRESERVE it in refrigerator.

5.1.3 Other solid samples with lower moisture content. for example, the grain having a moisture content at about 15%, TAKE about 100 g of sample, USE crusher to crush the sample to obtain the power of constant uniformity, MAKE determination in time or PRESERVE it in refrigerator.

5.2.1 Test solution extraction

WEIGH 3 g ~ 5 g (accurate to 0.01 g) of solid sample or 10 g ~ 20 g of liquid sample, PLACE it into a 100 mL conical flask (with a soft stopper), ADD 60 mL of 0.1 mol/L hydrochloric acid solution, SHAKE it uniformly, PLUG the soft stopper, PLACE it in the autoclave at 121 °C for 30 min. After finishing hydrolysis and cooling it to below 40 °C, TAKE it out, SHAKE it gently for several times; U

5.3.1 Column. C18 reversed-phase column (particle size 5 μm, 250 mm x 4.6 mm) or equivalent.

5.4 Standard curve production

INJECT the standard series working solution derivative into the high performance liquid chromatograph to determine the corresponding peak area of vitamin B1, USE the standard working solution concentration (μg/mL) as the abscissa, the peak area as the vertical axis, to draw the standard curve.

5.5 Determination of the specimen solution

In accordance with the chromatographic conditions of clause 5.3, INJECT the specimen derivative solution into a high performance liquid chromatograph to obtain the peak area of vitamin B1, CALCULATE the concentration of vitamin B1 in the solution to be determined in accordance with the standard curve.

6 Expression of analytical results

Sample vitamin B1 (by thiamine) content is calculated in accordance with the formula (1).

7 Precision

The absolute difference between two independent determinations obtained under repeatability conditions shall not exceed 10% of the arithmetic mean.

8 Others

When the amount of weighing sample is 10.0 g, in accordance with the constant volume of this standard method, the detection limit of vitamin B1 in food is 0.03 mg/100 g, the limit of quantitation is 0.10 mg/100 g.

9 Principles

Thiamin is oxidized to thiopyrimidine in alkaline potassium ferricyanide solution, under the irradiation of ultraviolet light, the thiopyrimidine fluoresces. Under the given conditions where there is no interference from other fluorescent substance, the intensity of this fluorescence is proportional to the amount of thiopyrimidine amount, i.e.,