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

GB/T 5413.23-1997

Replacing GB/T 5413.23-1997

**National food safety standard -- Determination of iodine in
foods for infants and young children, milk and milk products
(English Translation)**

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Table of Contents

Foreword
1 Scope
2 Normative references
4 Reagents and materials
5 Apparatus
6 Step, Analysis
6.1 Sample Processing
6.2 Preparation of sample measurement solution
6.3 Preparation of standard iodine solution Determination
6.4 Determination
7 Expression analysis
8 Precision
9 Other

Foreword

This standard replaces GB/T 5413.23-1997 "infant formula and milk powder Determination of Iodine" this standard compared with GB/T 5413.23-1997, main changes are as follows.

- To fill the column quartz capillary column;
- Increase the detection limit.

Appendix A of this standard is an informative annex.

This standard replaces the standards previously issued as follows.

--GB 5413-1985, GB/T 5413.23-1997.

National Food Safety Standard Determination of infant foods and dairy iodine

1 Scope

This standard specifies the method for determination of infant foods and dairy iodine.

This standard applies to the determination of infant foods and dairy iodine.

2 Normative references

The standard file referenced in the application of this standard is essential. For dated references, only the edition date of the note Apply to this standard. For undated references, the latest edition (including any amendments) applies to this standard.

Principle 3

Iodine in the sample under conditions of sulfuric acid and iodine derivatives generate butanone and butanone reacted by gas chromatography with electron capture detector inspection Testing, external standard.

4 Reagents and materials

Unless otherwise specified, the reagents used in this method are analytically pure water GB/T 6682 provided a water.

4.1

Peak's (Taka-Diastase) amylase. enzyme activity ≥ 1.5 U/mg.

4.2

potassium iodide (KI) or potassium iodate (KIO₃). pure class distinctions.

4.3

butanone (C₄H₈O). chromatography.

4.4

sulfuric acid (H₂SO₄). pure class distinctions.

4.5

n-hexane (C₆H₁₄).

4.6

anhydrous sodium sulfate (Na₂SO₄).

4.7

Hydrogen peroxide (3.5%). lessons 11.7 mL volume fraction of 30% hydrogen peroxide diluted to 100 mL.

4.8

potassium ferrocyanide solution (109 g/L). Weigh 109 g of potassium ferrocyanide, water volume to 1000 mL volumetric flask.

4.9

zinc acetate solution (219 g/L). Weigh 219 g of zinc acetate, water volume to 1000 mL volumetric flask.

4.10

iodine standard solution.

4.10.1

iodine standard stock solution (1.0 mg/mL). Weigh 131 mg of potassium iodide (4.2) (accurate to 0.1 mg) or 168.5 mg potassium iodate (4.2) (accurate to 0.1 mg), dissolved in water and dilute to 100 mL, 5 °C ± 1 °C refrigerated within a week effective.

4.10.2

iodine standard working solution (1.0 µg/mL). draw 10 mL standard stock solution of iodine, water volume to 100 mL and mix, then draw 1.0 mL, 100 mL water to the mix, prepared before use.

5 Apparatus

5.1

Balance. a sense of the amount of 0.1 mg.

5.2

Gas chromatography with electron capture detector.

6.1.1 Starch-free sample

Weigh mixed solid sample 5 g, liquid sample 20 g (accurate to 0.0001 g) in 150 mL Erlenmeyer flask, with a solid sample 25 mL of hot water about 40 °C dissolved.

6.1.2 Sample containing starch

Weigh mixed solid sample 5 g, liquid sample 20 g (accurate to 0.0001 g), in 150 mL flask was added 0.2 g Peak's amylase (4.1), solid sample with 25 mL of hot water about 40 °C fully dissolved, placed in 50 °C ~ 60 °C incubator hydrolysis 30 min, Remove to cool.

6.2 Preparation of sample measurement solution

6.2.1

precipitate. the processed sample solution transferred to 100 mL volumetric flask, add 5 mL potassium ferrocyanide solution (4.8) and 5 mL zinc acetate solution (4.9), the water volume to the mark still shaken after 10 min. After the filter paper to absorb the filtrate 10 mL 100 mL separating funnel, add 10 mL of water.

6.2.2

Derivatives and extraction. separating funnel was added to 0.7 mL of sulfuric acid (4.4), 0.5 mL butanone (4.3), 2.0 mL of hydrogen peroxide (4.7), Mix well, kept at room temperature for 20 min was added 20 mL of n-hexane (4.5) oscillation was extracted 2 min. After resting stratification, the aqueous phase shifted Another separatory funnel, and then a second extraction. The combined organic phase was washed with water two or three times.

Dehydrated over anhydrous sodium sulfate and filtered shift Into 50mL volumetric flask with normal hexane, this is the sample measurement solution.

6.3 Preparation of standard iodine solution Determination

Pipette 1.0 mL, 2.0 mL, 4.0 mL, 8.0 mL, 12.0 mL iodine standard working solution (4.10.2), equivalent to 1.0 µg, 2.0 µg, 4.0 µg, 8.0 µg, 12.0 µg of iodine, other analytical step with 6.2.

6.4.1 Reference chromatographic conditions

Column. filler is 5% cyanopropyl - methyl silicone capillary column (column length 30 m, an inner diameter of 0.25 mm, film thickness 0.25 µm) or equivalent performance of the column.

Inlet temperature. 260 °C.

ECD detector temperature. 300 °C.

Split ratio. 1. 1.

Injection volume. 1.0 µL.

Temperature program shown in Table 1.

Table 1 temperature program

Heating rate (/ min °C) Temperature (°C) Duration (min)

6.4.2 Standard curve

Determination of the iodine standard solution (6.3) were injected into a gas chromatograph (chromatogram see Appendix A) to give a peak area standard assay solution (Or peak height). In the standard assay solution peak area (or peak height) for the vertical axis, to the quality of (6.3) standard working solution of iodine in iodine as the horizontal Standard curve.

6.4.3 Determination of sample solution

Determination of the sample solution (6.2) is injected into the gas chromatograph to obtain the peak area (or peak height), to obtain a sample from the standard curve of iodine-containing the amount (µg).