



National standard of People's Republic of China

GB 5413.23—2010

National Food Safety Standard
Determination of iodine in foods for infants and young
children, raw milk and dairy products

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Preface

This standard replaces GB/T 5413.23-1997, "Measurement of iodine in formulated foods and milk powder for infants and young children"

The main changes in this standard compared with GB 5413.23-1997 are as follows:

- Chromatographic column filling is changed into quartz capillary column
- Add the detection limit

The annex A of this standard is an informative annex

Versions of previous standards substituted by this standard are:

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

National Food Safety Standard

Measurement of idoine in formulated foods and milk powder for infants and young children

1 Scope

This standard regulate the determination method of idoine in foods and dairy products for infants and young children.

This standard applies to the determination of idoine in foods and dairy products for infants and young children.

2 Normative cited documents

The reference cited in this standard is necessary. For the cited documents which are labeled with date, all their subsequent modification sheets or modified versions are not applicable for this standard. For the cited documents which are not labeled with date, their latest versions are applicable for this standard.

3. Principles

The iodine in the sample can react with butanone and produce the derivates of butanone and iodine, and they are subjected to gas chromatography analysis and electron capture detector measurement and quantitation with the external standard method.

4. Reagents and materials

Unless specified, the reagents used by this method are all analytical pure reagents, and the water is the grade two water specified by GB/T6682.

4.1 Taka-Diastase amylase: the enzymatic activity is not lower than 1.5 U/mg.

4.2 Potassium iodide or potassium iodate: GR

4.3 Butanone: chromatography grade.

4.4 Concentrated sulfuric acid: GR.

4.5 N-hexane

4.6 Sodium sulphate anhydrous

4.7 Hydrogen peroxide (3.5%): pipette 11.7 ml 30% (v/v) and diluted to 100 ml

4.8 potassium ferrocyanide solution (109g/L): dissolve 109g potassium ferrocyanide and make up to 1000 ml.

4.9 zinc acetate (219g/L): dissolve 219 g zinc acetate and make up to 1000 ml

4.10 Idoine standard solution

4.10.1 Idoine standard stock solution (1.0 mg/mL)

Dissolve 131 mg potassium iodide (4.2) (weigh nearest to 0.1 mg) or 168.5mg potassium iodate (4.2) (weigh nearest to 0.1 mg) then make up to 100 ml. Store at $5\pm 1^{\circ}\text{C}$. Can store 1 week

4.10.2 Idoine standard working solution (1.0 $\mu\text{g/mL}$):.

Into 100 ml volumetric flask, pipette 10 mL standard stock solution make up to the mark, well mixe. Pipette 1.0 mL of the prepared solution into 100 mL volumetric flask and

make up to mark. Mix well. This solution should be fresh prepared each time

5. Instruments and equipments

5.1 balance: the nearest to 0.1 mg.

5.2 Gas chromatography (electron capture detector).

6. Analytical procedures

6.1 Sample preparation

6.1.1 The sample does not contain starch

Into 5 g 150 mL conical flasks, weigh 5g homogenated solid sample or 20g liquid sample (the nearest to 0.0001g) , solid sample should dissolved in 25 ml 40°C water.

6.1.2 The sample containing starch

Into 5 g 150 mL conical flasks, weigh 5g homogenated solid sample or 20g liquid sample (the nearest to 0.0001g) , add 0.2 g Taka-Diastase amylase (4.1), solid sample should dissolved in 25 ml 40°C water, stand for 30°C in oven of 50-60°C, cool it.

6.2 The preparation of the measurement solution of the sample

6.2.1 Precipitation: the above mentioned processed sample solution is transferred to a 100 mL volumetric flask, add 5 mL potassium ferrocyanide (4.8) and 5 mL zinc acetate (4.9) , make up to the mark with water, completely mix, stand for 10 minutes. After filtration with filter paper, pipette 10 mL filtrate into a 100 mL separating funnel , add 100 mL water

6.2.2 Derivation and extraction: Add 0.7 mL concentrated sulphuric acid (4.4), 0.5 mL butanone (4.3) and 2.0 mL hydrogen peroxide(4.7) to funnel, completely mix, stand for 20 min under room temperature. Add 20 mL N-hexane (4.5) and shake for extraction for 2 min. Stand for separation. Transfer water layer to another separating funnel for the second extraction. Combine two times organic phases and rinse with water for two or three times. Pass through anhydrous sodium sulphate to remove water, transfer to a 50 mL volumetric then make up to the mark with N-hexane. This is the measurement solution of the sample.

6.3 The preparation of the iodine standard measurement solution

Pipette 1.0, 2.0, 4.0, 8.0, and 12.0 mL iodine standard working solution (4.10.2), which is equivalent to 1.0, 2.0, 4.0, 8.0, and 12.0ug of iodide, and other analytical procedures are all the same as those in 6.2.

6.4 Measurement

6.4.1 The chromatographic conditions for reference

The chromatographic column: capillary column, column packing is 5% cyanopropyl methyl polysilicon, (the length 30 m, the inner diameter 0.25 mm, the thickness of the membrane 0.25 µm); or the chromatographic column with the same performance.

Sample injection Temperature: 250 □

Temperature of the ECD detector: 300 □

The split ratio: 1:1

The injection size: 2.0 µL.

The programmed temperature increase is shown in Table 1:

Table 1 The programmed temperature increase

Temperature increasing rate (□/min)	Target temperature (□)	Duration (min)
	50	9
30	220	3

6.4.2 The preparation of the standard curve

The iodine standard measurement solution (6.3) is injected into the gas chromatography (chromatography chart reference in annex A) and the peak area (or the peak height) for the standard measurement solution can be obtained. The standard curve is plotted by using the peak area (or the peak height) for the standard measurement solution as the Y-axis and the mass of the iodine standard (µg) as the X-axis.

6.4.3 The measurement of the sample solution

The sample measurement solution (6.2) is injected into the gas chromatography and the peak area (or the peak height) for the standard measurement solution can be obtained. The content of iodine in the sample (µg) can be found in the standard curve.

7. The calculation and representation of the results

Calculate the content of iodine in the sample according to formula (1):

$$X = \frac{C_s}{m_i} \times 100 \dots\dots\dots (1)$$

In this formula:

X – content of iodide in the sample, ug/100g

Cs — the content of iodine in the sample (µg) that is found in the standard curve.

m1—the mass of the sample (g).

Report the result by mean of two independent measurement results to 0.1

8. The precision

The absolute difference between the two repeated measurements should be not more than 5% of the mean.