**National Standard of the People's Republic of China**

GB 5413.6 – 2010

# National food safety standard

# Determination of insoluble dietary fiber in foods for infants and young children, milk and milk products

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# Foreword

This standard Replacing GB/T 5413.6 - 1997 Determination of Insoluble Dietary Fiber in Formula Foods and Milk Powder for Infants and Young Children.

The versions replaced by this standard are:

- GB12394 - 90, GB/T 5413.6- 1997.

# National food safety standard

# Determination of insoluble dietary fiber in foods for infants and young children, milk and milk products

## 1 Scope

This standard specifies the method for determination of insoluble dietary fiber in foods for infants and young children, milk and milk products.

This standard applies to determination of insoluble dietary fiber in foods for infants and young children, milk and milk products.

## 2 Normative References

The normative documents referenced in the text are indispensable to the application of this standard. For dated references, only the edition bearing such date applies to this standard. For undated references, the lastest edition of the normative document referred to (including all the amendments) applies.

## 3 Principles

The sugar, starch, protein and pectin in the test sample are dissolved and removed using neutral detergent; the residue can't be dissolved is insoluble dietary fiber, it mainly consists of cellulose, hemicellulose, lignin, cutin and silicon dioxide, and also insoluble ash.

## 4 Reagents and materials

4.1 Anhydrous sodium sulfite.

4.2 Petroleum benzine: boiling range 30 - 60℃.

4.3 Acetone.

4.4 Toluene.

4.5 EDTA disodium salt

4.6 Sodium tetraborate (containing $10H_2O$)

4.7 Lauryl sulphuric acid sodium

4.8 Ethylene glycol monoethyl ether

4.9 Anhydrous dibasic sodium phosphate

4.10 Phosphoric acid

4.11 Dibasic sodium phosphate

4.12 α - amylase

4.13 Neutral detergent solution: place 18.61g EDTA disodium salt and 6.81g sodium tetraborate (containing $10H_2O$) to a beaker, add about 150mL water, heat to dissolve; dissolve 30.00g lauryl sulphuric acid sodium (chemical pure) and 10mL ethylene glycol monoethyl ether (chemical pure) in about 650mL hot water, combine the above-mentioned two solutions; dissolve 4.56g anhydrous dibasic sodium phosphate into 150mL hot water, incorporate it into the above-mentioned solution, adjust pH of the above-mentioned mixed solution with phosphoric acid to 6.9 - 7.1, and then add water to 1000mL.

4.14 Buffer phosphate: It is mixed from 38.7mL 0.1mol/L dibasic sodium phosphate and 61.3mL 0.1mol/L sodium dihydrogen phosphate, with a pH value of 7.0±0.2.

4.15 2.5% α - amylase solution: weigh 2.5g α – amylase and dissolve with 100mL buffer phosphate, centrifuge and filtrate, store the filtered enzyme solution for later use.

4.16 Heat resisting glass cotton (it can resist 130℃, glass cotton which resists to heat and is not easy to be broke) is used.

## 5 Apparatus

5.1 Balances: with a reciprocal sensibility of 0.1g

5.2 Drying oven: 110 - 130℃.

5.3 Thermostat oven: 37±2℃.

5.4 Fibrometer system.

5.5 If there is no fibrometer system, the following components can be used:

a) Hot plate: with a temperature control device.

b) Tall beaker without spout: 600mL.

c) Crucible type acid-resisting glass filter: with a volume of 60mL, and pore size of 40 - 60μm.

d) Reflux condensation device.

e) Decompress filter device: it consists of a suction flask, a filter bed and a water pump.

f) pH meter: with accuracy 0.01.

## 6 Analytical procedures

6.1 Weigh 0.5g - 1.00g solid test sample or 8.0 g liquid test sample (accurate to 0.1 mg) to a tall beaker without spout; if the content of fat in the test sample exceeds 10%, remove fat first, for instance, extract 1.00g test sample with 30℃ - 60℃petroleum benzene (4.2) for 3 times, each time with 10mL.

6.2 Add 100mL neutral detergent solution (4.13), and then add 0.5g anhydrous sodium sulfite (4.1).

6.3 Heat it to boil in 5 - 10 min with an electric stove, transfer to a hot plate and maintain small boil

condition for 1h.

6.4 Spread out 1 - 3g glass cotton on a heat resisting glass filter, transfer it to a drying oven, dry at 110℃ for 4h, take it out and then place into a desiccator to cool to room temperature, and weigh it; the mass acquired is m1 (accurate to 0.0001 g).

6.5 Pour the boiled test sample into the filter while it is still hot; treat it with suction filtration with a water pump. Wash the beaker and filter with 500mL (90 - 100)℃ hot water in several fractions, filter it to dry. Wash away the fluid and foam at the inferior part of the filter, and put onto the rubber stopper.

6.6 Add enzyme solution (4.15) to the filter, cover the liquid surface with fiber, add several drops of toluene (4.4) with a thin needle from which air has been squeezed, cover with glass and then incubate in a 37℃ thermostat oven overnight.

6.7 Take out the filter, remove the stopper at the bottom, suction to remove enzyme solution, wash away residual enzyme solution with 300mL hot water in fractions, test whether there is residual starch with iodine solution; if there is residual starch, add enzyme to catalyze hydrolysis; if starch is completely removed, wash it for further 2 times with acetone (4.3).

6.8 Place the filter to a drying oven and dry at 110℃ for 4h, take it out and put into a desiccator, cool to room temperature and then weigh the mass; the mass acquired is $m_2$ (accurate to 0.0001 g).

## 7 Expression of results

Calculate the content of insoluble dietary fiber in the test sample according to formula (1):

$$X = \frac{m_2 - m_1}{m} \times 100 \quad \text{.....(1)}$$

Where,

*X* - The content of insoluble dietary fiber in the test sample, g/100 g;

$m_1$ -the mass of glass cotton added into the filter, g;

$m_2$ - -the mass of glass cotton added into the filter and the fiber in the test sample, g;

*m* - Mass of the test sample, g.

The results of two independent determinations acquired under repeatability conditions are expressed with arithmetic mean; three significant figures should be retained in the results.

## 8 Precision

The absolute error of the results of two independent determinations acquired under repeatability conditions shouldn’t exceed 10% of the arithmetic mean.