

GB

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GB 5413.30-2010

Nation food safety standard

Determination of impurities in milk and milk products

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Forward

This standard cancels and replaces GB/t5413.30-1997 – Determination of Impurities in milk and dried milk.

Appendix A and Appendix B are standard appendix.

Replaced previous published standards:

——GB/T 5413.30-1997。

National food safety standard

Determination of impurities in milk and milk products

1. Scope

This Standard specifies the method for the determination of impurities in milk and milk products.

This Standard applies to the determination of impurities in pasteurized milk, sterilized milk, raw milk, condensed milk and dried milk; however not applies to products containing non-milk proteins, starch-like substance, insoluble colored substance and additives which can affect filtration.

2. Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

3. Principle

Impurities are determined by the amount of visible insoluble substances left on the filter pad after the sample is filtered and washed through the filter pad.

4. Apparatus

4.1 Filter equipment: impurities filter or 2000mL~2500mL filtering flask which can be equipped with filter pad holder.

4.2 Filter pad: $\varnothing 32\text{mm}$, density of $135\text{g}/\text{m}^3$, conform to the requirement described in appendix A. Diameter of filtration pass area is 28.6mm .

4.3 Impurities standard plate.

4.4 Method of making impurities standard plate refers to appendix B.

4.5 Balance. 0.1 g sensitivity.

5. Procedure

For liquid milk, take 500mL sample; for dried milk, weight 62.5g sample (to the nearest of 0.1g) into 8 times water, mix thoroughly and heat to 60°C ; for condensed milk, dissolve 125g sample into 4 times water and heat to 60°C . Filter the sample through the filter pad, vacuum pump can be used to accelerate filtration, and rinse the filter pad with water. Dry the filter pad with drying oven. Impurities of the test sample can be obtained by comparing the filter pad with the impurities standard plate.

The worse class (higher impurities) should be prevailed If the impurities on the filter pad is

between two neighbour-classes of standard plate.

6. Expression of the results

Impurities of the test sample should be expressed by the results of comparison between the filter pad and the impurities standard plate.

7. Precision

Repeat the determination twice if the results of duplicated examination of the same sample are not in accord with each other.

Appendix A

(Standard Appendix)

Determination of impurities using Filter pad

A.1 Materials

A.1.1 Wetting agent: 1% solution.

A.1.2 Vegetable gum solution: Add 0.75g carob bean gum or other appropriate gum into 100 mL water and then mix with stirrer. Eliminate bubbles by vacuum or heat treatment. Add 2 mL 40% formaldehyde solution After boiling and cooling the solution.

For dissolving without stirring, firstly dissolve 0.75 g carob bean gum in a 100 mL volumetric flask with several milliliters of ethanol. Then dilute to the mark with water. Mix thoroughly and treat follow the above instruction.

A.1.3 Sucrose solution: dissolve 750g commercial sugar into 750mL water.

A.1.4 Refined impurities mixture: cattle feces, dirt and charcoal should be dried using a drying oven(100°C) and filtered separately. Component that between 106µm (140 mesh) and 75µm (200 mesh) sieves should be collected and mixed. The procedure is as follow:

Cattle feces and dirt should be filtered no more than 100g and charcoal should be filtered no more than 50 g. Put a 75µm sieve outside the 106µm one and fix the collecting device. Filter 20 g materials once by knocking the cover of the sieves by hand with a speed of 120 times per minute. Filter the fragments that have passed 106µm (140 mesh) sieve using 75µm (200 mesh) sieve for another 5min and collect the components remaining on the second sieve. Mix the materials as thoroughly as possible according to the table A.1:

Table A.1 The proportion of the components remain on the second sieve

Impurities	Content (%)
Cattle feces	66
Dirt	28
Charcoal	6

Wet 2 g of the above mixture with 5 mL wetting agent (A1.1) in a 100 mL volumetric flask. Add 46 mL vegetable gum solution and make the solution near to the mark of the volumetric flask using the sucrose solution (A1.3). Add several drops of ethanol and dilute to the mark with sucrose solution and mix thoroughly.

Pour the above solution into a 250 mL beaker or a bottle with screw closure, a small amount of air is allowed to be taken into the solution, and agitate at 200~300r/min using small mechanical stirrer till the impurities has been visibly distributed evenly under bright illumination. The small particles should be prevented from depositing around the whirlpool at the bottom. Pipette 10mL (approximately 200 mg impurities mixture) above solution into a 1000 mL volumetric flask and dilute to the mark with water.

A.2 Apparatus

A.2.1 Balance: 0.1mg sensitivity.

A.2.2 Desiccator: with effective desiccant.

A.2.3 Drying oven

A.2.4 Filter equipment

A.2.5 Filter pad.

A.2.6 Filter paper: Diameter 7cm or 9cm.

A.3 Procedure

A.3.1 Put a piece of filter paper (A2.6) in a Buchner funnel and rinse with ~200 mL water. Dry the filter paper in 100°C drying oven till constant weigh.

A.3.2 Filter 60 mL of the refined impurities mixture (A 1.4) through filter pad (A 2.5) installed in the filter equipment after thoroughly mixing. Stir the 60mL refined impurity mixture (A1.4) thoroughly. This equals 12mg impurities.

Collect the filtrate with a clean conical flask and transfer to a beaker. Rinse the flask twice with water and transfer to the beaker.

A.3.3 Filter the filtrate again using Buchner funnel with filter paper (A3.1) which has been rinsed, dried and weighed. Rinse the beaker and the filter paper thoroughly with water and dry the filter paper in 100°C oven till constant weight.

A.3.4 Determine at least two filter pads.

A.4 Evaluation

The average amount of impurities that passed through three or more than three filter pad should not exceed 2.8mg. Impurities should not appear beneath the standard pad which is made from refined impurities mixture.

Appendix B:

(Standard Appendix)

How to make the impurities standard plate:

B.1 Materials

Filter scorched particles, dirt, cow feces and charcoals through appropriate sieves and dry in drying oven (100°C). Mix them according to the following ratio:

Scorched particles: 40%, in which

10% can pass through 20 mesh sieve but cannot pass through 40 mesh sieve

30% can pass through 40 mesh sieve but cannot pass through 60 mesh sieve

Earth: 30%, Can pass through 40 mesh sieve

Cow feces: 20%, in which

2% can pass through 20 mesh sieve but cannot pass through 40 mesh sieve

8% can pass through 40 mesh sieve but cannot pass through 60 mesh sieve

10% can pass through 60 mesh sieve but cannot pass through 80 mesh sieve

Charcoals: 10% in which

4% can pass through 20 mesh sieve but cannot pass through 40 mesh sieve

6% can pass through 40 mesh sieve but cannot pass through 60 mesh sieve

B.2 Procedure

B.2.1 Mix the above impurities thoroughly (total weight: ~50 g) and weight 1.000g into a 500mL volumetric flask. Add 2 mL distilled water and 23 mL filtered 0.75% (V/V) gum arabic solution. Dilute with filtered 50% sugar solution to the mark and mix. The impurities concentration of this solution is 2mg/mL.

B.2.2 10mL of 2mg/mL impurities solution is diluted with filtered 500g/L sugar solution to 100mL. The impurities concentration of this solution is 0.2mg/mL.

B.2.3 10mL of 0.2mg/mL impurities solution is diluted with filtered 500g/L sugar solution to 100mL. The impurities concentration of this solution is 0.02mg/mL.

B.2.4 Sample 500mL milk or 62.5g dried milk, make the standard impurities plate according to table B1:

Code of standard plate	Relative mass concentration of the impurities, milk mg/L; dried milk mg/kg		absolute content of impurities (mg)	volume of the impurities mixture solution (mL)
	500mL milk	62.5g dried milk		
1	0.25	2	0.125	6.25
2	0.75	6	0.375	18.75
3	1.50	12	0.750	3.75
4	3.0	16	1.000	5.00