



National Standard of the People's Republic of China

GB 5413.30-2016

National Food Safety Standard Determination of Impurities in Dairy and Dairy Products

食品安全国家标准

乳和乳制品杂质度的测定

- Released on 2016-12-23
- Implemented on 2017-06-23
- Issued by NHFPC & CFDA

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Foreword

This standard replaces GB 5413.30-2010 “National food safety standard Determination of impurities in Dairy and Dairy Products”.

Compared with GB 5413.30-2010, the major changes of this standard are as follows:

- The technical requirements for impurities filter pad have been added;
- The inspection procedures in Annex A have been simplified and “determination of impurities loss” has been modified to “determination of impurities residues”;
- The preparation of impurities reference standard plate in the Annex B has been modified to method for preparation of the standard plates for liquid milk and milk powders;
- The impurities components and particle size have been redefined.

National Food Safety Standard

Determination of Impurities in Dairy and Dairy Products

1. Scope

This standard stipulates the method for determination of impurities in dairy and dairy products.

This standard is used to the determination of impurities in raw and fresh milk, pasteurized milk, sterilized milk, condensed milk and milk powder; it does not apply to the dairy and dairy products containing substances affecting filtration and insoluble colored substance.

2. Principles

The samples of raw and fresh milk, liquid milk and reconstituted milk with water are filtered by the impurities filter pad and the limit of impurities are determined according to the comparison of non-white impurities visibly left on the impurities filter pad and the impurities reference standard plate.

3. Reagents and Materials

Unless otherwise specified, all reagents in this method shall refer to analytically pure reagents, and water used shall be Grade 3 water specified in GB/T 6682.

3.1 Impurities filter pad: the white cotton pad with the diameter of 32mm, the mass of $135\text{mg}\pm 15\text{mg}$, the thickness of $0.8\text{mm}\sim 1.0\text{mm}$. It shall comply with the requirements of Annex A. Impurities filter pad shall be inspected according to Annex A.

3.2 Impurities reference standard plate: the method of impurities reference standard plate is shown in Annex B.

4. Apparatus and Equipment

4.1 Balance: with the sensitivity of 0.1g.

4.2 Filter plate: impurities filtering machine or filtering flask. Rapid filtration can be achieved by positive pressure or negative pressure (the filtration time for 1L of water is 10s~15s). After putting in the impurity filter pad, the effective filter diameter is $28.6\text{mm}\pm 0.1\text{mm}$.

5. Analysis Procedures

5.1 Sample solution preparation

5.1.1 For sample of liquid milk, mix absolutely and then take 500mL with measuring cylinder for an immediate determination.

5.1.2 Accurately weigh $62.5\text{g}\pm 0.1\text{g}$ of milk powder sample into a 1000mL beaker, add 500mL of water at $40^{\circ}\text{C}\pm 2^{\circ}\text{C}$, stir absolutely to dissolve and then have an immediate determination.

5.2 Determination

Place the impurities filter pad on the filter device, pour the prepared sample solution to the funnel of filter device (but not from the funnel) and filter. Clean the beaker with water for several times and transfer the washing solution into the funnel and filter. Wash and clean the funnel in several times with wash bottle and filter; after drying, take out the impurities filter pad. Then the sample impurities are obtained by compare in comparison with the impurities standard plate.

6. Expression of Analysis Result

The impurity the filter filtration after filtration was compared with the impurity reference standard plate, that is, the impurity of such sample.

The worse class (higher impurities) should be prevailed if the impurities on the impurities filter pad are between two neighbor-classes; the impurities should be judged to exceed a maximum value if the foreign matter (including fiber and so on) appears.

7. Precision

The same sample shall be determined twice according to the method described in this standard and the results shall be consistent.

Annex A

Inspection of Impurities Filter Pad

A.1 Reagents and Materials

A.1.1 Reagents

A.1.1.1 Anhydrous ethanol (C₂H₅OH)

A.1.1.2 Formaldehyde (HCHO)

A.1.1.3 Carob bean gum: biochemical reagent

A.1.1.4 Sucrose

A.1.2 Reagent preparation

A.1.2.1 Formaldehyde solution (40%): measure 40mL of formaldehyde to a 100mL volumetric flask, dilute it to 100mL with water and filter for later use.

A.1.2.2 Carob bean gum solution: weigh 0.75g±0.01g of carob bean gum into a 250mL beaker, and add 2mL of anhydrous ethanol to make it wet and then add 50mL of water and mix absolutely. Heat slowly to eliminate air bubbles, boil the carob bean gum completely, then cool. Add 2 mL of the filtered 40% formaldehyde solution, mix thoroughly, and transfer to a 100 mL volumetric flask. Dilute with water to the volume.

A.1.2.3 Sucrose solution: weigh 750g±0.1g of sucrose into a 1000mL beaker, add 750mL of water to dissolve completely and then filter for later use.

A.1.3 Materials

Impurities: dry the dirt on the ground with the constant temperature drying oven (100°C±1°C), use the standard sieve to collect the dirt components with the particle size of 75µm~106µm and then dry to the constant weight.

A.2 Apparatus and Equipment

A.2.1 Balance: with the sensitivity of 0.1g and 0.1mg

A.2.2 Standard sieve

A.2.3 Desiccator: with effective desiccant

A.2.4 Constant temperature drying oven: with the precision of ±1°C

A.2.5 Filter plate: the same as 4.2

A.3 Inspection Procedures

A.3.1 Preparation of impurity solution: weighing 2.00g±0.001g of impurities, add it into a 250mL beaker and then make it wet with 5mL of anhydrous ethanol. Add 46mL of carob bean gum solution, 40mL of sucrose solution, mix absolutely and then transfer to a 100mL volumetric flask and add sucrose solution to dilute to the volume and mix absolutely. Transfer 10mL (corresponding to 200mg of impurities) into a 1000mL volumetric flask, dilute it to the volume and mix absolutely.

A.3.2 Place the impurities filter pad into constant temperature drying oven (100°C ±1°C) and dry it to the constant weight and record the mass *N*₁.

A.3.3 Place the impurities filter pad on the filter plate, transfer 60mL (corresponding to 12mg of impurities) of impurity solution accurately that has been mixed absolutely, filter and clean the pipette with water and filter the washing solution together; clean the filter pad with 200mL of water at 40°C±2°C in several times and after filter to dryness, take down the impurities filter pad and dry it to the constant weight at constant temperature drying oven (100°C±1°C) and record the mass *N*₂.

A.4 Evaluation

A.4.1 $M = N_2 - N_1$, *M* shall be not less than 10mg. Cut down the surface layer of impurities filter pad with a sharp blade and check the rest part; no impurities shall appear.

A.4.2 10 pads in every 1000 pads shall be inspected; for less than 1000 pads, it shall be calculated as 1000 pads.

Annex B

Preparation of the impurities reference standard plate

B.1 Reagents and Materials

B.1.1 Reagents

B.1.1.1 Arabic gum: biochemical reagent

B.1.1.2 Sucrose

B.1.1.3 Cow feces and scorched particles: collect the cow feces and scorched particles respectively, smash and then dry at constant temperature drying oven ($100^{\circ}\text{C}\pm 1^{\circ}\text{C}$).

B.1.2 Reagent preparation

B.1.2.1 Arabic gum solution (0.75%): weighing 1.875g of Arabic gum into a 100mL beaker, add 20mL of water, heat to dissolve and then cool down. Transfer to a 250mL volumetric flask with water and dilute to the volume and filter.

B.1.2.2 Sucrose solution (50%): weighing 1000g of sucrose into a 1000mL beaker, add 500mL of water to dissolve and then transfer to a 2000mL volumetric flask with water and dilute to the volume and filter.

B.1.3 Material preparation

B.1.3.1 Cow feces

B.1.3.1.1 A: use the standard sieve to collect the cow feces with the particle size of $0.150\text{mm}\sim 0.200\text{mm}$, ready for use.

B.1.3.1.2 B: use the standard sieve to collect the cow feces with the particle size of $0.125\text{mm}\sim 0.150\text{mm}$, ready for use.

B.1.3.1.3 C: use the standard sieve to collect the cow feces with the particle size of $0.106\text{mm}\sim 0.125\text{mm}$, ready for use.

B.1.3.2 Scorched particles:

B.1.3.2.1 D: use the standard sieve to collect the scorched particles with the particle size of $0.300\text{mm}\sim 0.450\text{mm}$, ready for use.

B.1.3.2.2 E: use the standard sieve to collect the scorched particles with the particle size of $0.200\text{mm}\sim 0.300\text{mm}$, ready for use.

B.1.3.2.3 F: use the standard sieve to collect the scorched particles with the particle size of $0.150\text{mm}\sim 0.200\text{mm}$, ready for use.

B.2 Apparatus and Equipment

B.2.1 Balance: with the sensitivity of 0.1g and 0.1mg

B.2.2 Standard sieve

B.2.3 Filter plate: the same as 4.2

B.3 Procedures for making the reference standard impurity plate of liquid milk

B.3.1 Preparation of impurity reference standard solution of liquid milk

B.3.1.1 Weigh 500.0mg of cow feces A, B and C into three 100mL beakers respectively. Adding 2mL of water and 23mL of Arabic gum solution, mix absolutely and then transfer to 500mL volumetric flasks with sucrose solution and dilute to the volume and mix absolutely until the impurities are uniformly distributed; then the cow feces impurities reference standard solution a_0 , b_0 and c_0 with the concentration of 1.0mg/mL are obtained.

B.3.1.2 Pipette 100mL of cow feces impurities reference standard solution a_0 , b_0 and c_0 into 500mL volumetric flasks respectively, dilute to the volume with sucrose solution and then cow feces impurities reference standard intermediate solution a_1 , b_1 and c_1 with the concentration of 0.2mg/mL are obtained.

B.3.1.3 Pipette 10mL of cow feces impurities reference standard intermediate solution a_1 , b_1 and c_1 into 100mL

volumetric flasks. Respectively, dilute to the volume with sucrose solution and then cow feces impurities reference standard working solution a_2 , b_2 and c_2 with the concentration of 0.02mg/mL are obtained.

B.3.2 Preparation of reference standard impurity plate of liquid milk

B.3.2.1 Take 100mL of sucrose solution, filter on the filter plate pre-placed the impurities filter pad, clean the filter pad with 100mL of water at $40^{\circ}\text{C}\pm 2^{\circ}\text{C}$ in several times, and dry in the air; This impurity plate is the impurity reference standard plate A_1 (with the impurity relative content of 0mg/kg in liquid milk).

B.3.2.2 Pipette 6.25mL of cow feces impurities reference standard working solution c_2 into a 100mL volumetric flask accurately, dilute to the volume with the sucrose solution, mix absolutely and then filter on the filter plate pre-placed the impurities filter pad, clean the volumetric flask with water and filter the washing solution together. Clean the filter pad with 100mL of water at $40^{\circ}\text{C}\pm 2^{\circ}\text{C}$ in several times, and dry in the air; This impurity plate is the impurity reference standard plate A_2 (with the impurity relative content of 2mg/8L in liquid milk).

B.3.2.3 Pipette 12.5mL of cow feces impurities reference standard working solution b_2 into a 100mL volumetric flask accurately, dilute to the volume with the sucrose solution, mix absolutely and then filter on the filter plate pre-placed the impurities filter pad, wash the volumetric flask with water and filter the washing solution together. Clean the filter pad with 100mL of water at $40^{\circ}\text{C}\pm 2^{\circ}\text{C}$ in several times, and dry in the air; This impurity plate is the impurity reference standard plate A_3 (with the impurity relative content of 4mg/8L in liquid milk).

B.3.2.4 Pipette 18.75mL of cow feces impurities reference standard working solution a_2 into a 100mL volumetric flask accurately, dilute to the volume with the sucrose solution, mix absolutely and then filter on the filter plate pre-placed the impurities filter pad, wash the volumetric flask with water and filter the washing solution together. Clean the filter pad with 100mL of water at $40^{\circ}\text{C}\pm 2^{\circ}\text{C}$ in several times, and dry in the air; This impurity plate is the impurity reference standard plate A_4 (with the impurity relative content of 6mg/8L in liquid milk).

B.3.3 Take 500mL of liquid milk as the sample weight; refer to Figure B.1 to prepare the liquid milk impurity reference standard board according to Table B.1.

Table B.1 Comparison table for impurity reference standard plates of liquid milk

Reference standard plate No.	A_1	A_2	A_3	A_4
Impurities solution concentration/(mg/mL)	0	0.02	0.02	0.02
Volume of impurities solution taken/mL	0	6.25	12.5	18.75
Impurities absolute content/(mg/500mL)	0	0.125	0.250	0.375
Impurities relative content/(mg/8L)	0	2	4	6

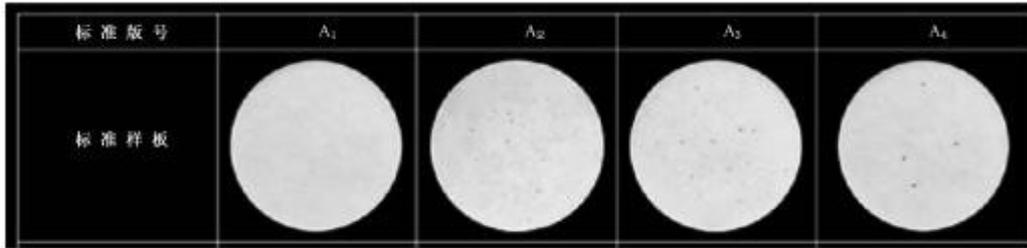


Figure B.1 Impurity reference standard plates of liquid milk

B.4 Procedures for making the impurity reference standard plate of milk powder

B.4.1 Preparation of impurity reference standard solution of milk powder

B.4.1.1 Weigh 500.0mg of scorched particle D, E and F into three 100mL beakers respectively. Add 2mL of water and 23mL of Arabic gum solution, mix absolutely and then transfer to 500mL volumetric flasks with sucrose solution and dilute to the volume and mix absolutely until the impurities are uniformly distributed; then the scorched particle impurities reference standard solution d_0 , e_0 and f_0 with the concentration of 1.0mg/mL are obtained.

B.4.1.2 Pipette 100mL of scorched particle impurities reference standard solution d_0 , e_0 and f_0 into 500mL volumetric flasks respectively, dilute to the volume with sucrose solution and then scorched particle impurities reference standard working solution d_1 , e_1 and f_1 with the concentration of 0.2mg/mL are obtained.

B.4.2 Preparation of reference standard impurity plate of milk powder

B.4.2.1 Pipette 2.5mL of scorched particle impurities reference standard working solution f_1 into a 100mL volumetric flask accurately, dilute to the volume with the sucrose solution, mix absolutely and then filter on the filter plate pre-placed the impurities filter pad, wash the volumetric flask with water and filter the washing solution together. Clean the filter pad with 100mL of water at $40^{\circ}\text{C}\pm 2^{\circ}\text{C}$ in several times, and dry in the air; This impurity plate is the impurity reference standard plate B₁ (with the impurity relative content of 8mg/kg in milk powder).

B.4.2.2 Pipette 3.75mL of scorched particle impurities reference standard working solution e_1 into a 100mL volumetric flask accurately, dilute to the volume with the sucrose solution, mix absolutely and then filter on the filter plate pre-placed the impurities filter pad, wash the volumetric flask with water and filter the washing solution together. Clean the filter pad with 100mL of water at $40^{\circ}\text{C}\pm 2^{\circ}\text{C}$ in several times, and dry in the air; This impurity plate is the impurity reference standard plate B₂ (with the impurity relative content of 12mg/kg in milk powder).

B.4.2.3 Pipette 5mL of scorched particle impurities reference standard working solution d_1 into a 100mL volumetric flask accurately, dilute to the volume with the sucrose solution, mix absolutely and then filter on the filter plate pre-placed the impurities filter pad, wash the volumetric flask with water and filter the washing solution together. Clean the filter pad with 100mL of water at $40^{\circ}\text{C}\pm 2^{\circ}\text{C}$ in several times, and dry in the air; This impurity plate is the impurity reference standard plate B₃ (with the impurity relative content of 16mg/kg in milk powder).

B.4.2.4 Pipette 3.75mL of scorched particle impurities reference standard working solution d_1 and 2.5mL of scorched particle impurities reference standard working solution e_1 into a 100mL volumetric flask accurately, dilute to the volume with the sucrose solution, mix absolutely and then filter on the filter plate pre-placed the impurities filter pad, clean the volumetric flask with water and filter the washing solution together. Clean the filter pad with 100mL of water at $40^{\circ}\text{C}\pm 2^{\circ}\text{C}$ in several times, and dry in the air; This impurity plate is the impurity reference standard plate B₄ (with the impurity relative content of 20mg/kg in milk powder).

B.4.3 Take 62.5g of milk powder as the sample weight; refer to Figure B.2 for the impurity reference standard plates

of milk powder prepared in accordance with Table B.2 Comparison table for impurity reference standard plates of milk powder.

Table B.2 Comparison table for impurity reference standard plates of milk powder

Reference standard plate No.	B ₁	B ₂	B ₃	B ₄
Impurities solution concentration/(mg/mL)	0.2	0.2	0.2	0.2
Volume of impurities solution taken/mL	2.5	3.75	5.0	6.25
Impurities absolute content/(mg/62.5g)	0.500	0.750	1.000	1.250
Impurities relative content/(mg/kg)	8	12	16	20

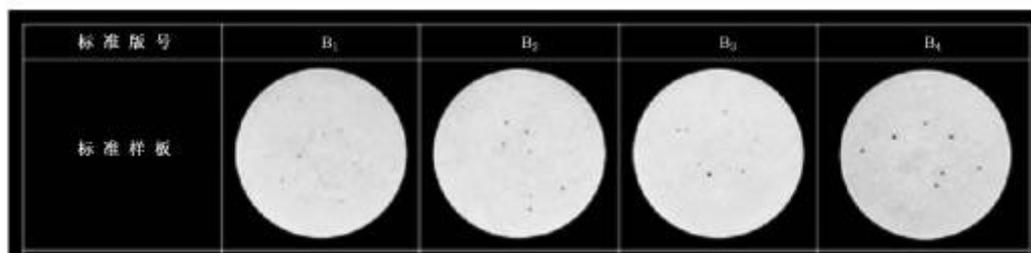


Figure B.2 Impurity reference standard plates of milk powder