GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5413.30-2016

National Food Safety Standard -

Determination of Impurities in Milk and Milk Products

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China Food and Drug Administration.

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Foreword

This Standard replaces GB 5413.30-2010, *National Food Safety Standard – Determination of Impurities in Milk and Milk Products.*

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

-- the technical requirements for impurities filter plate are added;

-- the testing procedures in Annex A are simplified and the measurement of impurities loss is modified into the measurement of impurities residuals in the Annex;

-- the preparation of impurities reference standard plate is modified into the methods for the preparation of two standard plates for liquid milk and milk powder;

-- the constituents and particle size of impurities are redefined.

National Food Safety Standard - Determination of Impurities in Milk and Milk Products

1 Application 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 fresh and raw milk, pasteurized milk, sterilized milk, condensed milk and milk powder; it does not apply to the determination of impurities in milk and milk products added with substances which decrease the efficiency of filter and insoluble coloured matters.

2 Principle

The specimen of fresh and raw milk, liquid milk, water reconstituted milk powder is filtered through impurities filter plate; the limit of impurities in the specimen is determined by comparing the visible non-white impurities remaining on the impurities filter plate with the impurities reference standard plate.

3 Reagents and Materials

Unless stated otherwise, the reagents used in this Method are analytically pure and the water is grade-3 water as specified in GB/T 6682.

3.1 Impurities filter plate: white cotton plate of diameter 32 mm, mass 135 mg ± 15 mg and thickness 0.8 mm ~ 1.0 mm, which shall be as specified in Annex A. Impurities filter plate shall be tested in accordance with Annex A.

3.2 Impurities reference standard plate: see Annex B for the method for the preparation of impurities reference standard plate.

4 Apparatus

4.1 Balance: sensitivity 0.1 g.

4.2 Filter equipment: impurities filter machine or suction flask, which is capable of achieving rapid filter by positive pressure or negative pressure (the filter time of each litre of water is 10 s ~ 15 s). The effective filter diameter after mounting impurities filter plate is 28.6 mm ± 0.1 mm.

B.1.3.2.3 F: use a standard screen to collect coke powder of particle size 0.150 mm ~ 0.200 mm as standby.

B.2 Apparatus

B.2.1 Balance: sensitivities 0.1 g and 0.1 mg.

B.2.2 Standard screen.

B.2.3 Filter equipment: as in 4.2.

B.3 Procedures for preparing liquid milk reference standard impurities plate

B.3.1 Preparation of liquid milk impurities reference standard solution

B.3.1.1 Weigh accurately 500.0 mg of cow dung to place into three 100-mL beakers, namely A, B and C. Add 2 mL of water, add 23 mL of Arabic gum solution, use sucrose solution to transfer to a 500-mL volumetric flask after mixing up and add dropwise to the scale, fully mix up until the impurities distributes uniformly, and obtain the cow dung impurities reference standard solutions of concentration 1.0 mg/mL, namely a_0, b_0 and c_0.

B.3.1.2 Absorb respectively 100 mL of cow dung impurities reference standard solutions a_0, b_0 and c_0 to pour into 500-mL volumetric flasks, use sucrose solution to dilute and add dropwise to the scale, and obtain the cow dung impurities reference standard intermediate solutions of concentration 0.2 mg/mL, namely a_1, b_1 and c_1.

B.3.1.3 Absorb respectively 10 mL of cow dung impurities reference standard intermediate solutions a_1, b_1 and c_1 to pour into 100-mL volumetric flasks, use sucrose solution to dilute and add dropwise to the scale, and obtain the cow dung impurities reference standard working solutions of concentration 0.02 mg/mL, namely a_2, b_2 and c_2.

B.3.2 Preparation of liquid milk reference standard impurities plate

B.3.2.1 Measure 100 mL of sucrose solution, filter on the filter equipment where the impurities filter plate is mounted, use 100 mL of water of 40°C ± 2°C to wash the filter plate for several times and dry by airing. Then the impurities plate is the impurities reference standard plate A_1 for the impurities' relative content in the liquid milk 0 mg/kg.

B.3.2.2 Absorb accurately 6.25 mL of cow dung impurities reference standard working solution c_2 to pour into a 100-mL volumetric flask, use sucrose solution to dilute and add dropwise to the scale, filter on the filter equipment where the impurities filter plate is mounted after mixing up, use water to wash the volumetric flask, and filter the washings together. Then use 100 mL of water of 40°C ± 2°C to wash the filter plate for several times, and dry by airing. Then the impurities plate is the impurities reference standard plate A_2 for the impurities' relative content in the liquid milk 2 mg/8 L.

B.3.2.3 Absorb accurately 12.5 mL cow dung impurities reference standard working

mg/mL, namely d_0, e_0 and f_0.

B.4.1.2 Absorb 100 mL of the coke powder impurities reference standard solutions d_0, e_0 and f_0 to pour into 500-mL volumetric flasks respectively, use sucrose solution to dilute and add dropwise to the scale and obtain the coke powder reference standard working solutions of concentration 0.2 mg/mL, namely d_1, e_1 and f_1.

B.4.2 Preparation of milk powder reference standard impurities plates

B.4.2.1 Absorb accurately 2.5 mL of the coke powder impurities reference standard working solution f_1 to pour into a 100-mL volumetric flask, use sucrose solution to dilute and add dropwise to the scale, filter on the filter equipment where the impurities filter plate is mounted after mixing up, use water to wash the volumetric flask, and filter the washings together. Then use 100 mL of water of 40°C ± 2°C to wash the filter plate for several times, and dry by airing. The impurities plate is the impurities reference standard plate B_1 for the impurities' relative content in milk powder 8 mg/kg.

B.4.2.2 Absorb accurately 3.75 mL of the coke powder impurities reference standard working solution e_1 to pour into a 100-mL volumetric flask, use sucrose solution to dilute and add dropwise to the scale, filter on the filter equipment where the impurities filter plate is mounted after mixing up, use water to wash the volumetric flask, and filter the washings together. Then use 100 mL of water of 40°C ± 2°C to wash the filter plate for several times, and dry by airing. The impurities plate is the impurities reference standard plate B_2 for the impurities' relative content in milk powder 12 mg/kg.

B.4.2.3 Absorb accurately 5 mL of the coke powder impurities reference standard working solution d_1 to pour into a 100-mL volumetric flask, use sucrose solution to dilute and add dropwise to the scale, filter on the filter equipment where the impurities filter plate is mounted after mixing up, use water to wash the volumetric flask, and filter the washings together. Then use 100 mL of water of 40°C ± 2°C to wash the filter plate for several times, and dry by airing. The impurities plate is the impurities reference standard plate B_3 for the impurities' relative content in milk powder 16 mg/kg.

B.4.2.4 Absorb accurately 3.75 mL of the coke powder impurities reference standard working solution d_1 and 2.5 mL of the coke powder impurities reference standard working solution e_1 to pour into a 100-mL volumetric flask, use sucrose solution to dilute and add dropwise to the scale, filter on the filter equipment where the impurities filter plate is mounted after mixing up, use water to wash the volumetric flask, and filter the washings together. Then use 100 mL of water of 40°C ± 2°C to wash the filter plate for several times, and dry by airing. The impurities plate is the impurities reference standard plate B_4 for the impurities' relative content in milk powder 20 mg/kg.

B.4.3 Use 62.5 g of milk powder as the sampling quantity and see Figure B.2 for the milk powder impurities reference standard plates prepared in accordance with Table B.2 – Correlation of Milk Powder Impurities Reference Standard Plates.

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