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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 5413.38-2016

National Food Safety Standard - Determination of Freezing Point in Raw Milk in Food

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National Food Safety Standard -

Determination of Freezing Point in Raw Milk in Food

1 Scope

This Standard specifies the method of determining freezing point in raw milk with thermistor freezing point meter.

This Standard is applicable to the determination of freezing point in raw milk.

2 Principle

Cool down raw milk sample to an appropriate temperature; when the test sample cools down to $-3\text{ }^{\circ}\text{C}$, instantaneously release heat to crystalize the sample. Wait till the sample temperature reaches a balanced state, and temperature rise $\leq 0.5\text{ m }^{\circ}\text{C}$ within 20 s, the instant temperature shall be considered as the freezing point of the sample.

3 Reagents and Materials

Unless otherwise indicated, the reagents adopted under this method are of analytical purity or above. The water is second-grade water as specified in GB/T 6682.

3.1 Reagents

3.1.1 Ethylene glycol ($\text{C}_2\text{H}_6\text{O}_2$).

3.1.2 Sodium chloride (NaCl).

3.2 Preparation of Reagents

3.2.1 Sodium chloride (NaCl): grind sodium chloride, then, place it in a drying oven; start drying at $130\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for over 24 h. Cool it down in the dryer to room temperature.

3.2.2 Coolant: measure-take 330 mL of ethylene glycol (3.1.1) and place it in 1,000 mL volumetric flask; add water to the constant volume and shake it well; its volume fraction is 33%.

3.3 Sodium Chloride Standard Solution

3.3.1 Standard solution A: weigh-take 6.731 g of sodium chloride (3.2.1), then, dissolve it in $1,000\text{ g} \pm 0.1\text{ g}$ of water. Divide standard solution and store it in polyethylene plastic

of corresponding instrument. During normal stirring, metal stirring rod shall not touch glass sensor or sample tube wall.

c) Crystallization device

When the test sample reaches $-3.0\text{ }^{\circ}\text{C}$, initiate the mechanical vibration device for crystallization. During crystallization, increase the amplitude of the metal stirring rod within $1\text{ s} \sim 2\text{ s}$, and touch the tube wall of the sample.

4.3 Drying oven: temperature can be controlled at $130\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

4.4 Sample tube: borosilicate glass, length: $50.5\text{ mm} \pm 0.2\text{ mm}$, external diameter: $16.0\text{ mm} \pm 0.2\text{ mm}$, internal diameter: $13.7\text{ mm} \pm 0.3\text{ mm}$.

4.5 Weighing bottle.

4.6 Volumetric bottle: 1,000 mL, comply with Grade-A requirements in GB/T 12806-2011.

4.7 Dryer: with silicone hygrometer inside.

4.8 Pipette: $1\text{ mL} \sim 5\text{ mL}$.

4.9 Polyethylene bottle: capacity: $\leq 250\text{ mL}$.

5 Analytical Procedures

5.1 Sample Preparation

Test sample shall be preserved in the refrigerator at $0\text{ }^{\circ}\text{C} \sim 6\text{ }^{\circ}\text{C}$; complete the determination within 48 h. Before determination, the sample shall be placed till it reaches the room temperature. The temperature of the test sample shall be maintained consistent with the temperature of sodium chloride standard solution during the determination.

5.2 Instrument Pre-cooling

Initiate thermistor freezing point meter (4.2), wait till the sensor probe of thermistor freezing point meter rises, then, open the cold trap cover; add corresponding volume of coolant (3.2.2) as prescribed by the manufacturer; close the cover, initiate pre-cooling of the freezing point meter. After 30 min of pre-cooling, start to determine the freezing point.

5.3 Calibration

5.3.1 Principle

Before calibration, prepare sodium chloride standard solution (with different freezing

in turn; in initiated cold trap, insert a sample tube that contains calibration solution C. When repeatedly measured value is at calibration value ($-600 \text{ m } ^\circ\text{C} \pm 2 \text{ m } ^\circ\text{C}$), complete the calibration.

5.3.3 Quality control calibration

Before each determination, quality control calibration shall be adopted. In successive determination of milk sample, quality control calibration shall be conducted at least every hour on the freezing point meter. If the difference between the arithmetic mean value of two measurements and sodium chloride standard solution ($-512 \text{ m } ^\circ\text{C}$) is $> 2 \text{ m } ^\circ\text{C}$, re-conduct instrument calibration (5.3.2).

5.4 Sample Determination

5.4.1 Slightly shake the test sample (5.1), avoid the inlet of air, which will generate bubbles. Transfer-take 2.5 mL of sample to a dry and clean sample tube; place the sample tube in the measuring hole of previously calibrated thermistor freezing point meter (4.2). Initiate the freezing point meter and cool down the sample. When the temperature reaches $-3.0 \text{ } ^\circ\text{C} \pm 0.1 \text{ } ^\circ\text{C}$, the sample starts to freeze; when the temperature reaches a balance (temperature rise $\leq 0.5 \text{ m } ^\circ\text{C}$ within 20 s), the freezing point meter stops measurement, and the sensor probe rises. The displayed temperature is the freezing point of the sample. After determination is completed, guarantee that the probe and metal stirring rod are clean and dry.

5.4.2 If the sample starts to freeze before the temperature reaches $-3.0 \text{ } ^\circ\text{C} \pm 0.1 \text{ } ^\circ\text{C}$, re-take a sample for determination (5.4.1). If the sample still freezes too early, heat up the remaining sample at $40 \text{ } ^\circ\text{C} \pm 2 \text{ } ^\circ\text{C}$ for 5 min to melt crystallized fat, then, repeat the steps of sample determination (5.4.1).

5.4.3 After the determination is completed, remove the sample tube; use water to rinse the temperature sensor and metal stirring rod, then, wipe them clean.

5.4.4 Record the determined freezing point of the sample.

6 Expression of Analysis Results

Take the average value (unit: $\text{m } ^\circ\text{C}$) of two determination results as the measured freezing point of raw milk sample; the result shall retain three significant figures.

7 Precision

The absolute difference between the two independent determination results obtained under repeatability conditions shall not exceed $4 \text{ m } ^\circ\text{C}$.

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