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GB 5009.239-2016

National Food Safety Standard – Determination of Acidity in Food

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Foreword

This Standard replaces GB 5413.34-2010, *National Food Safety Standard – Determination of Acidity in Milk and Milk Products*, GB/T 22427.9-2008, *Starch and Derived Products – Determination of Acidity* and GB/T 5517-2010, *Inspection of Grain and Oils – Determination of Acidity in Grain and Produce*.

Compared with GB 5413.34-2010, GB/T 22427.9-2008 and GB/T 5517-2010, the major changes of this Standard are as follows:

- it changes the standard name into “*National Food Safety Standard – Determination of Acidity in Food*”;
- it integrates the methods for the determination of acidity in food specified in GB 5413.34-2010, GB/T 22427.9-2008 and GB/T 5517-2010.

National Food Safety Standard – Determination of Acidity in Food

1 Application Scope

This Standard specifies the methods for the determination of acidity in milk and milk products, starch and derived products, grain and grain products.

The first method of this Standard applies to the determination of acidity in milk and milk products, starch and derived products, grain and grain products; the second method applies to the determination of acidity in milk powder; the third method applies to the determination of acidity in milk and milk products.

Method I Phenolphthalein Indicator Method

2 Principle

Use phenolphthalein as the indicator after the treatment of specimen; use 0.100 0 mol/L sodium hydroxide standard solution to titrate to neutral; use the volume of sodium hydroxide consumed to determine the acidity of specimen by calculation.

3 Reagents and Materials

Unless specified otherwise, all reagents used for this method are analytically pure and the water is of grade 3 as specified in GB/T 6682.

3.1 Reagents

3.1.1 Sodium hydroxide solution (NaOH).

3.1.2 Cobalt sulfate heptahydrate ($\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$).

3.1.3 Phenolphthalein.

3.1.4 95% ethanol.

3.1.5 Diethyl ether.

3.1.6 Nitrogen: purity 98%.

4.6 Ground conical flask with a stopper: 250 mL.

4.7 Pulverizer: it is capable of pulverizing specimen so that more than 95% of specimen passes sieve CQ16 [equivalent to pore diameter 0.425 mm (mesh number 40)]; it will not generate heat in the grinding chamber when pulverizing specimen.

4.8 Oscillator: reciprocating type; oscillation frequency 100 /min.

4.9 Intermediate speed qualitative filter paper.

4.10 Pipette: 10 mL, 20 mL.

4.11 Measuring cylinder: 50 mL, 250 mL.

4.12 Glass funnel and funnel support.

5 Analytical Procedure

5.1 Mild powder

5.1.1 Specimen preparation

Transfer all specimen to a clean, dry container (with a seal lid) which is about 2 times of the volume of specimen; immediately put on the lid; rotate and oscillate repeatedly to mix up specimen. During the operation, efforts shall be taken to avoid specimen from exposure to the air.

5.1.2 Determination

Weigh 4 g of specimen (accurate to 0.01 g) to place into 250 mL conical flask. Use measuring cylinder to take 96 mL of water of about 20°C (3.2.5) to dissolve specimen once again; stir before allowing to stand for 20 min.

Add 2.0 mL of reference solution to a conical flask containing 96 mL of water of about 20°C (3.2.5); rotate it gently to mix up; then obtain standard reference colour. If several similar products are to be determined, the reference solution can be used for the whole determination process, but the time shall not be more than 2 h.

Add 2.0 mL of phenolphthalein indicator to another conical flask containing specimen solution; rotate it gently to mix up. Use 25 mL basic burette to add sodium hydroxide solution to the conical flask; rotate the flask while adding dropwise until the colour is similar to that of reference solution and does not fade away within 5 s. The whole titration process shall be finished within 45 s. During the titration process, blow nitrogen to the conical flask to prevent solution absorbing carbon dioxide from the air. Record the millimetres of sodium hydroxide solution used (V_1), accurate to 0.05 mL, and substitute into Formula (1) for calculation.

5.1.3 Blank titration

millimetres of sodium hydroxide standard titration solution used (V_2), and substitute into Formula (2) for calculation.

5.2.5 Casein

Weigh 5 g (accurate to 0.001 g) of specimen ground and mixed up to place into conical flask; add 50 mL of water (3.2.5); store for 4 h ~ 5 h at room temperature (18°C ~ 20°C) or heat to 45°C in water bath and maintain it at the temperature for 30 min; add another 50 mL of water (3.2.5); filter through dry filter paper after mixing up. Absorb 50 mL of filtrate to place into conical flask; add 2.0 mL of phenolphthalein indicator; use sodium hydroxide standard solution for titration after mixing up; rotate the flask while adding dropwise until the colour is similar to that of reference solution and does not fade away within 5 s. The whole titration process shall be finished within 45 s. During the titration process, blow nitrogen to conical flask to prevent solution absorbing carbon dioxide from the air. Record the millimetres of sodium hydroxide standard titration solution used (V_3), and substitute into Formula (3) for calculation.

5.2.6 Blank titration

Use an equivalent volume of water (3.2.5) to conduct blank test; read the millimetres of sodium hydroxide standard solution consumed (V_0) (applicable to 5.2.2, 5.2.4 and 5.2.5). Use 30 mL of neutral ethanol-diethyl ether mixture to carry out blank test; read the millimetres of sodium hydroxide standard solution consumed (V_0) (applicable to 5.2.3).

The volume of sodium hydroxide consumed by blank shall not be less than 0, or else, distilled water or neutral ethanol-diethyl ether mixture as required shall be prepared and used once again.

5.3 Starch and derived products

5.3.1 Specimen pretreatment

Specimen shall be mixed up.

5.3.2 Specimen weighing

Weigh 10 g (accurate to 0.1 g) of specimen to transfer to 250 mL conical flask; add 100 mL of water; vibrate for mixing up.

5.3.3 Titration

Add 2.0 mL of reference solution to a conical flask containing 100 mL of water of about 20°C; rotate it gently to mix up; then obtain standard reference colour. If several similar products are to be determined, the reference solution can be used for the whole determination process, but the time shall not be more than 2 h.

Add 2 ~ 3 drops of phenolphthalein indicator to another conical flask containing specimen solution; use sodium hydroxide standard solution for titration after mixing up;

m_3 – mass of specimen, expressed in g;

0.1 – molar concentration of sodium hydroxide for the theoretical definition of acidity, expressed in mol/L.

The result is expressed by the arithmetic mean value of the results obtained from two independent determinations under repeatable conditions, which is rounded off to the third decimal place.

The acidity of the specimen of starch and derived products is expressed in °T and calculated in accordance with Formula (4):

$$X_4 = \frac{c_4 \times (V_4 - V_0) \times 10}{m_4 \times 0.1000} \dots\dots\dots (4)$$

where,

X_4 – acidity of specimen, expressed in °T [calculated by the millimetres of 0.1 mol/L sodium hydroxide solution consumed by 10 g of specimen, expressed in mL/10 g];

c_4 – molar concentration of sodium hydroxide standard solution, expressed in mol/L;

V_4 – volume of sodium hydroxide standard solution consumed during titration, expressed in mL;

V_0 – volume of sodium hydroxide standard solution consumed during blank test, expressed in mL;

10 – 10 g of specimen;

m_4 – mass of specimen, expressed in g;

0.100 0 – molar concentration of sodium hydroxide for the theoretical definition of acidity, expressed in mol/L.

The result is expressed by the arithmetic mean value of the results obtained from two independent determinations under repeatable conditions, which is rounded off to the third decimal place.

The acidity of the specimen of grain and grain products is expressed in °T and calculated in accordance with Formula (5):

$$X_5 = (V_5 - V_0) \times \frac{V_{31}}{V_{32}} \times \frac{c_5}{0.1000} \times \frac{10}{m_5} \dots\dots\dots (5)$$

where,

X_5 – acidity of specimen, expressed in °T [calculated by the millimetres of 0.1 mol/L sodium hydroxide solution consumed by 10 g of specimen, expressed in mL/10 g];

13 Accuracy

The absolute difference between the results obtained from two independent determination results under repeatable conditions shall not exceed 10% of the arithmetic mean value.

Method III Potentiometric Titrator Method

14 Principle

The acidity is determined through calculation based on the volume of 0.100 0 mol/L sodium hydroxide for the neutralization of 100 g of specimen to pH 8.30.

15 Reagents and Materials

Unless specified otherwise, all reagents used for this method are analytically pure and the water is of grade 3 as specified in GB/T 6682.

15.1 Sodium hydroxide standard solution: as in 3.2.1.

15.2 Nitrogen: purity 98%.

15.3 Neutral ethanol-diethyl ether mixture: as in 3.2.4.

15.4 Distilled water free of carbon dioxide: as in 3.2.5.

16 Apparatus

16.1 Analytical balance: sensitivity 0.001 g.

16.2 Potentiometric titrator.

16.3 Basic burette: scale division 0.1 mL.

16.4 Water bath.

17 Analytical Procedure

17.1 Pasteurized milk, sterilized milk, raw milk and fermented milk

Take 10 g (accurate to 0.001 g) of specimen mixed up to place into 150 mL conical flask; add 20 mL of water newly boiled and cooled to room temperature; mix up; use

18 Expression of Analytical Results

The acidity of the specimen of pasteurized milk, sterilized milk, raw milk, fermented milk, cream and condensed milk is expressed in °T and calculated in accordance with Formula (7):

$$X_7 = \frac{c_7 \times (V_7 - V_0) \times 100}{m_7 \times 0.1} \quad \text{..... (7)}$$

where,

X_7 – acidity of specimen, expressed in °T;

c_7 – molar concentration of sodium hydroxide standard solution, expressed in mol/L;

V_7 – volume of sodium hydroxide standard solution consumed during titration, expressed in mL;

V_0 – volume of sodium hydroxide standard solution consumed during blank test, expressed in mL;

100 – 100 g of specimen;

m_7 – mass of specimen, expressed in g;

0.1 – molar concentration of sodium hydroxide for the theoretical definition of acidity, expressed in mol/L.

The result is expressed by the arithmetic mean value of the results obtained from two independent determinations under repeatable conditions, which is rounded off to the third decimal place.

The acidity of the specimen of casein is expressed in °T and calculated in accordance with Formula (8):

$$X_8 = \frac{c_8 \times (V_8 - V_0) \times 100 \times 2}{m_8 \times 0.1} \quad \text{..... (8)}$$

where,

X_8 – acidity of specimen, expressed in °T;

c_8 – molar concentration of sodium hydroxide standard solution, expressed in mol/L;

V_8 – volume of sodium hydroxide standard solution consumed during titration, expressed in mL;

V_0 – volume of sodium hydroxide standard solution consumed during blank test, expressed in mL;

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