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National food safety standard
Determination of acidity in milk and milk
products

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Forward

Method 1 in this standard specifies two methods. Reference method is equivalent adoption of International Dairy Federation standard IDF 86: 1978 – Dried milk - Determination of Titratable Acidity (Reference method); routine method is equivalent adoption of International Dairy Federation standard IDF 81: 1981 – Dried milk - Determination of Titratable Acidity (Routine method); reference method is the referee method.

This standard cancels and replaces acidity determination in GB/T 5009.46-2003 - Method of analysis of hygienic standard of milk and milk products, acidity determination in GB/T 5416-85 – Analytical method for butter, milk freshness test in GB/T 5409-85 – analytical method for milk and GB/T 5431.28-1997 – Dried milk – Determination of Titratable Acidity.

Replaced previous published standards:

——GB/T 5409-85;

——GB/T 5413.28-1997;

——GB/T5416-85;

——GB 5425-85;

——GB 5009.46-1985, GB/T 5009.46-1996, GB/T 5009.46-2003.

National food safety standard

Determination of acidity in milk and milk products

1 Scope

This standard specifies the method for the determination of the titratable acidity of dried milk, pasteurized milk, sterilized milk, raw milk, fermented milk, condensed milk, butter and casein.

Method 1 in this standard applies to the determination of the titratable acidity of dried milk; and method 2 applies to the determination of the titratable acidity of pasteurized milk, sterilized milk, raw milk, fermented milk, condensed milk, butter and casein.

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.

Method 1 Determination of titratable acidity of dried milk

Reference method

3 Principle

titrated with a 0.1mol/L sodium hydroxide solution to the pH of 8.3. The titratable acidity of the sample could be calculated according to the volume of 0.1mol/L sodium hydroxide solution required to titrate 100 mL reconstituted milk (12% dry matter) to the pH of 8.3.

4 Reagents and materials

Unless otherwise specified, all reagents in this method shall be of analytical quality. Water shall be 3th grade water according to GB/T6682.

4.1 Sodium hydroxide standard volumetric solution: 0.1000mol/L.

4.2 Nitrogen.

5 Apparatus

5.1 Balance: 1 mg sensitivity.

5.2 Burette: graduated to 0.1 mL and with an accuracy of 0.05 mL.

5.3 pH meter, with a glass electrode and a suitable reference electrode.

5.4 Magnetic stirrer.

6 Procedure

6.1 Preparation of the test sample

Transfer the sample to a clean, dry container (provided with an air-tight lid) of a capacity about twice the volume of the sample. Close the container immediately and thoroughly mix the contents by repeatedly shaking and inverting the container. During these operations, exposure of the sample to the atmosphere should be avoided as far as possible, to minimize absorption of water.

6.2 Determination

6.2.1 Weigh 4 ± 0.01 g of the test sample into the conical flask.

6.2.2 Reconstitute the test sample with 96 mL water at about 20°C (using measuring cylinder), agitate thoroughly and allow standing for about 20 min.

6.2.3 Titrate the contents of the conical flask by adding the sodium hydroxide solution from the burette until the pH has reached 8.3. During the titration, the solution should be stirred using the magnetic stirrer, and absorption of carbon dioxide from the air should be avoided by flushing the conical flask with nitrogen. The titration should be completed within 1 min. Record the volume, in millilitres, of sodium hydroxide solution used, to the nearest 0.05 ml. Calculate using formula (1).

Routine method

7 Principle

Titrate 100 mL reconstituted milk (12% dry matter) with a 0.1 mol/L sodium hydroxide solution till the solution has been turned to pink using phenolphthalein as indicator and cobaltous sulphate as a reference colour solution. The titratable acidity could be calculated according to the volume of 0.1 mol/L sodium hydroxide solution that has been used.

8 Reagents and materials

Unless otherwise specified, all reagents in this method shall be of analytical quality. Water shall be 3th grade water according to GB/T6682.

8.1 Sodium hydroxide standard volumetric solution: same as 4.1.

8.2 Reference solution. Dissolve 3 g cobaltous sulphate heptahydrate ($\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$) in distilled water and make up to 100 mL.

8.3 Phenolphthalein indicator solution: Dissolve 0.5 g phenolphthalein in 75 mL 95% (v/v) ethyl alcohol and add 20 mL water. Add sodium hydroxide solution (3.2.1) till a pink colour appears. Finally make the solution up to 100 ml.

9 Apparatus

9.1 Balance: 1 mg sensitivity.

9.2 Burette: graduated to 0.1 mL and with an accuracy of 0.05 mL.

10 Procedure

10.1 Preparation of the test sample is the same as 6.1

10.2 Determination

10.2.1 Measurement and dissolving of the sample are the same as 6.2.1 and 6.2.2.

10.2.2 Add 2.0 mL of the reference colour solution (8.2) to one of the conical flasks to obtain a colour standard, mix by slight swirling. If a series of determinations of similar samples is to be carried out, this colour standard can be used throughout. The colour standard, however, should not be used for more than 2 hours.

10.2.3 Add 2.0 mL of the phenolphthalein indicator solution (8.3) to the second conical flask, mix slight swirling. Titrate the content of the second conical flask by adding the sodium hydroxide solution (8.1), while swirling until a colour similar to the colour standard persists for about 5 sec. The titration time should not exceed 45 sec. Record the number, in milliliters, of sodium hydroxide solution used to the nearest 0.05 ml. Calculate using formula (1).

11 Calculation and expression of results

Titrate acidity of the test sample is expressed by (° T) and calculated according to formula (1):

$$X_1 = \frac{c_1 \times V_1 \times 12}{m_1 \times (1 - w) \times 0.1} \dots\dots\dots (1)$$

Where

X₁— Titrate acidity of the test sample, ° T

c₁ — the concentration of sodium hydroxide standard volumetric solution, mol/L,

V₁ — the number, in milliliters, of sodium hydroxide solution used during titration, mL,

m₁ — the weight of the test sample, g,

w — mass fraction of water in the test sample, %,

12 — 12 g dried milk is equal to 100 mL reconstituted milk (dried skim milk is 9 and dried skim whey is 7).

0.1 —the molar concentration of sodium hydroxide solution in theoretical definition of acidity, mol/L

The arithmetic mean of two results of duplicate determination is used to be the final result. Express the result to three decimal place.

Note: When express acidity of the test sample as lactic acid content, the lactic acid content of the test sample (g/100g) = T X 0.009. Where T is the titrate acidity of the test sample (0.009 is the conversion parameter of lactic acid, i.e. 1 mL 0.1mol/L sodium

hydroxide standard solution correspond to 0.009 g lactic acid.)

12 Precision

The absolute difference between results of duplicate determination should not exceed 1.0 °T.

Method 2 Determination of titratable acidity of milk and other milk products

13 Principle

100g of the test sample is titrated with sodium hydroxide standard solution (0.1000 mol/L) to the end point using phenolphthalein as indicator. The titratable acidity of the test sample could be calculated according to the amount of sodium hydroxide solution that has been used.

14 Reagents and materials

Unless otherwise specified, all reagents in this method shall be of analytical quality. Water shall be 3rd grade water according to GB/T6682.

14.1 Neutral ethanol-ether mixture: mix ethanol and ether with same volume. After adding several drops of phenolphthalein, titrate with sodium hydroxide solution (4 g/L) until a faint pink colour appears.

14.2 Sodium hydroxide standard volumetric solution: same as 4.1.

14.3 Phenolphthalein indicator solution: same as 8.3.

15 Apparatus

15.1 Balance: 1 mg sensitivity.

15.2 Potentiometric titrator.

15.3 Burette: graduated to 0.1 mL

15.4 Water bath,

16 Procedure

16.1 Pasteurized milk, sterilized milk, raw milk and fermented milk

Weight 10 g (to the nearest 0.001g) of homogenized test sample into a 150 mL conical flask. Add 20 mL ambient water that has been just boiled, mix well and titrate by sodium hydroxide standard solution (14.2) with potentiometric titrator until the pH has reached 8.3; or add 2.0 mL of phenolphthalein indicator solution (14.3) into the dissolved sample solution, mix well and titrate by sodium hydroxide standard solution (14.2) until a faint colour persists for 30 sec. Record the volume, in millilitres, of sodium hydroxide solution used. Calculate using formula (2).

16.2 Butter

Weight 10 g (to the nearest 0.001g) of homogenized test sample. Add 30 mL neutral ethanol-ether mixture (8.1), mix well and test according to 16.1 'titrate by sodium hydroxide standard solution with potentiometric titrator until the pH reaches 8.3'.

16.3 Casein

Weight 5 g (to the nearest 0.001 g) of ground sample into a conical flask, add 50 mL water and keep at room temperature (18°C ~20°C) for 4-5 h; or heat to keep at 45°C in the water bath for 30 min. Add 50mL more water, mix well and filter through dry filter paper. Take 50 mL filtrate into another conical flask and titrate by sodium hydroxide standard solution (14.2) with potentiometric titrator until the pH reaches 8.3; or add 2.0 mL phenolphthalein indicator solution (14.3) into the 50 mL filtrate, mix well and titrate by sodium hydroxide standard solution (14.2) until a faint colour persists for 30 sec. Record the volume, in millilitres, of sodium hydroxide solution used. Calculate using formula (3).

16.4 Condensed milk

Weight 10 g (to the nearest 0.001g) of homogenized test sample into a 250 mL conical flask. Add 60 mL ambient water that has been just boiled, mix well and test according to 16.1 'titrate by sodium hydroxide standard solution with potentiometric titrator until the pH reaches 8.3'.
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17 Calculation and expression of results

Titrate acidity of the test sample is expressed by (° T) and calculated according to formula (2):

$$X_2 = \frac{c_2 \times V_2 \times 100}{m_2 \times 0.1} \dots\dots\dots (2)$$

Where

X₂— Titrate acidity of the test sample, ° T

c₂ — the molar concentration of sodium hydroxide standard volumetric solution, mol/L,

V₂ — the number, in milliliters, of sodium hydroxide solution used during titration, mL,

m₂ — the weight of the test sample, g,

0.1 — the molar concentration of sodium hydroxide solution in theoretical definition of acidity, mol/L.

The arithmetic mean of two results of duplicate determination is used to be the final result. Express the result to three decimal place.

$$X_3 = \frac{C_3 \times V_3 \times 100 \times 2}{M_3 \times 0.1} \dots\dots\dots (3)$$

Where

X₃— Titrate acidity of the test sample, ° T

C₃ — the molar concentration of sodium hydroxide standard volumetric solution, mol/L,

V_3 – the number, in milliliters, of sodium hydroxide solution used during titration, mL,

M_3 – the weight of the test sample, g,

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

2 – dilution multiple of the test sample,,

The arithmetic mean of two results of duplicate determination is used to be the final result. Express the result to three decimal place.

18 Precision

The absolute difference between results of duplicate determination should not exceed 1 ° T.