



National Standard of the People's Republic of China

GB 5009.239-2016

National food safety standard Determination of acidity in foods

食品安全国家标准

食品中酸度的测定

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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:

——the name of the standard has been revised to “National food safety standard Determination of acidity in foods”;

——the determination methods of acidity in foods in GB 5413.34-2010, GB/T 22427.9-2008 and GB/T 5517-2010 have been combined in this standard.

National food safety standard

Determination of acidity in foods

1 Scope

This standard specifies the method for the determination of acidity in raw milk and milk products, starch and its derived products and grain and its products.

Method I in this standard applies to the determination of acidity in raw milk and milk products, starch and its derived products and grain and its products; Method II in this standard applies to the determination of acidity in milk powder; Method III applies to the determination of acidity in milk and other milk products.

Method I Phenolphthalein Indicator Method

2 Principles

After the treatment of test sample, the phenolphthalein is used as an indicator, it is titrated with 0.1000 mol/L sodium hydroxide standard solution to neutral, and the acidity of the test sample shall be calculated and determined by the consumed volume of sodium hydroxide solution.

3 Reagents and Materials

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

3.1 Reagents

3.1.1 Sodium hydroxide (NaOH).

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

3.1.3 Phenolphthalein.

3.1.4 95% ethanol.

3.1.5 Ether.

3.1.6 Nitrogen: with the purity of 98%.

3.1.7 Chloroform (CHCl_3).

3.2 Reagent preparation

3.2.1 Sodium hydroxide standard solution (0.1000 mol/L)

Weigh 0.75 g of potassium hydrogen phthalate the working chemical which is dried to constant weight in the electric oven at $105\text{ }^\circ\text{C} \sim 110\text{ }^\circ\text{C}$, add 50 mL of carbon dioxide-free water to dissolve, add 2 drops of phenolphthalein indicator solution (10 g/L), titrate the solution to pink with the prepared sodium hydroxide solution, and keep for 30 s. Meanwhile, perform the blank test.

Note: carbon dioxide shall be restricted in washing bottles or drying tube, in order to prevent the concentration of sodium hydroxide from being affected because of absorbing carbon dioxide. Prevent the solution from absorbing carbon dioxide (CO_2) by forming a closed system by connecting the washing bottle containing 10% sodium hydroxide solution with the burette containing sodium hydroxide solution, or by connecting the end of burette containing fresh sodium hydroxide or calcium oxide.

3.2.2 Reference solution

Dissolve 3 g of cobaltous sulfate heptahydrate with water, and dilute to 100 mL.

3.2.3 Phenolphthalein indicator solution

Weigh 0.5 g of phenolphthalein and dissolve in 75 mL of ethanol with the volume fraction of 95%, add 20 mL of water, then dropwise add sodium hydroxide (3.2.1) to pink, and dilute to 100 mL with water.

3.2.4 Neutral ethanol-ether mixture

Take ethanol and ether of same volume, dropwise add 3 drops of phenolphthalein indicator solution after mixing, and dropwise add sodium hydroxide solution (0.1 mol/L) to reddish color.

3.2.5 Distilled and carbon dioxide-free water

Boil the water for 15 min, expel the carbon dioxide, cool down, and seal it.

4 Apparatus and Equipment

4.1 Analytical balance: with the sensitivity of 0.001 g.

4.2 Alkali burette: with the capacity of 10 mL and minimum scale of 0.05 mL.

4.3 Alkali burette: with the capacity of 25 mL and minimum scale of 0.1 mL.

4.4 Water bath.

4.5 Conical flask: 100 mL, 150 mL and 250 mL.

4.6 Ground-glass stoppered conical flask: 250 mL.

4.7 Crusher: with more than 95% of the crushed samples through the CQ16 sieve [equivalent to the aperture of 0.425 mm (mesh)], and grinding chamber generates no heat during grinding the sample.

4.8 Oscillator: reciprocating and with the oscillation frequency of 100 beats/min.

4.9 Medium speed qualitative filter paper.

4.10 Pipette: 10 mL and 20 mL.

4.11 Cylinder: 50 mL and 250 mL.

4.12 Glass funnel and funnel support.

5 Analysis Procedures

5.1 Milk powder

5.1.1 Sample preparation

Transfer all the samples to a clean and dry container about two times the volume of the sample (with a sealing cap), immediately cover the container, rotate and oscillate repeatedly to completely mix the samples. Try to avoid exposing the sample in the air during the operation.

5.1.2 Determination

Weigh 4 g of the sample (accurate to 0.01 g) in a 250-mL conical flask. Measure 96 mL of water about 20 °C (3.2.5) with cylinder to redissolve the sample, stir, and then stand for 20 min.

Add 2.0 mL of reference solution to a conical flask containing 96 mL of water about 20 °C (3.2.5), rotate gently to mix, and obtain the standard reference color. For determination of several similar products, the reference solution can be used throughout the determination, but the time shall not exceed 2 h.

Add 2.0 mL of phenolphthalein indicator solution to another flask with sample solution, rotate gently to mix. Dropwise add sodium hydroxide solution to the conical flask with a 25 mL alkali burette, rotate the flask during dropwise adding, until the color is similar to that of the reference solution, and does not fade for 5 s, and the entire titration process shall be completed within 45 s. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_1) of sodium hydroxide, accurate to 0.05 mL, and plug into Formula (1) and calculate.

5.1.3 Blank titration

Perform the blank test with 96 mL of water (3.2.5), read the consumed volume (V_0) of sodium hydroxide. The volume of sodium hydroxide consumed by the blank shall not be less than zero, otherwise re-prepare the distilled water and use the distilled water as required.

5.2 Milk and other milk products

5.2.1 Preparation of reference solution

Add 2.0 mL of reference solution to the conical flask with the equal volume of the corresponding solution, rotate gently to mix, and obtain the standard reference color. For determination of several similar products, the reference solution can be used throughout the determination, but the time shall not exceed 2 h.

5.2.2 Pasteurized milk, sterilized milk, raw milk and fermented milk

Weigh 10 g (accurate to 0.001g) of well-mixed sample, place in a 150-mL conical flask, add 20 mL of water freshly boiled and cooled to room temperature, mix, add 2.0 mL of phenolphthalein indicator solution, titrate with sodium hydroxide standard solution after mixing well, rotate the flask during dropwise adding until the color is similar to that of the reference solution, and does not fade for 5 s, and the entire titration process shall be completed within 45 s. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_2) of sodium hydroxide, and plug into Formula (2) and calculate.

5.2.3 Cream

Weigh 10 g (accurate to 0.001 g) of well-mixed sample, place in a 250-mL conical flask, add 30 mL of neutral ethanol-ether mixture, mix well, add 2.0 mL of phenolphthalein indicator solution, titrate with sodium hydroxide standard solution after mixing well, rotate the flask during dropwise adding, until the color is similar to that of the reference solution, and does not fade for 5 s, and the entire titration process shall be completed within 45 s. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_2) of sodium hydroxide, and plug into Formula (2) and calculate.

5.2.4 Condensed milk

Weigh 10 g (accurate to 0.001g) of well-mixed sample, place in a 250-mL conical flask, add 60 mL of water freshly boiled and cooled to room temperature, mix well, add 2.0 mL of phenolphthalein indicator solution, titrate with sodium hydroxide standard solution after mixing well, rotate the flask during dropwise adding, until the color is similar to that of the reference solution, and does not fade for 5 s, and the entire titration operation shall be completed within 45 s. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_2) of sodium hydroxide, and plug into Formula (2) and calculate.

5.2.5 Casein

Weigh 5g (accurate to 0.001 g) of evenly grinded sample in a conical flask, add 50 mL of water (3.2.5), and place at room temperature (18 °C ~20 °C) for 4 h~5 h, or place in a water bath to heat to 45 °C and maintain for 30 min, add another 50 mL of water (3.2.5), filter through a dry filter paper after mixing well. Collect 50 mL of the filtrate in a conical flask, add 2.0 mL of phenolphthalein indicator solution, titrate with sodium hydroxide standard solution after mixing well, rotate the flask during dropwise adding, until the color is similar to that of the reference solution, and does not fade for 5 s, and the entire titration process shall be completed within 45 s. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_3) of sodium hydroxide, and plug into Formula (3) and calculate.

5.2.6 Blank titration

Perform the blank test with equal volume of water (3.2.5), read the consumed volume (V_0) of sodium hydroxide (applicable to section 5.2.2, section 5.2.4 and section 5.2.5). Perform the blank test with 30 mL of neutral ethanol-ether mixture, read the consumed volume (V_0) of sodium hydroxide (applicable to 5.2.3).

The volume of sodium hydroxide consumed by the blank shall not be less than zero, otherwise re-prepare the distilled water and use the distilled water or neutral ethanol-ether mixture as required.

5.3 Starch and its derived products

5.3.1 Sample pretreatment

Samples shall be completely mixed well.

5.3.2 Sample weighing

Take 10 g (accurate to 0.1 g) of sample, transfer to a 250-mL conical flask, add 100 mL of water, shake and mix well.

5.3.3 Titration

Add 2.0 mL of reference solution to a conical flask containing 100 mL of water about 20 °C, rotate gently to mix, and obtain the standard reference color. For determination of several similar products, the reference solution can be used throughout the determination, but the time shall not exceed 2 h.

Add 2 drops~3 drops of phenolphthalein indicator solution to a flask containing sample, rotate gently to mix, titrate with sodium hydroxide standard solution, rotate the flask during dropwise adding, until the color is similar to that of the reference solution, and does not fade for 5 s, and the entire titration process shall be completed within 45 s. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_4) of sodium hydroxide, and plug into Formula (4) and calculate.

5.3.4 Blank titration

Perform the blank test with 100 mL of water (3.2.5), read the consumed volume (V_0) of sodium hydroxide.

The volume of sodium hydroxide consumed by the blank shall not be less than zero, otherwise re-prepare the distilled water and use the distilled water as required.

5.4 Grain and its products

5.4.1 Sample preparation

Take 80 g~100 g of well-mixed samples, crush with a crusher, the required grinding fineness is of more than 95% through the CQ16 sieve [equivalent to the aperture of 0.425 mm (mesh)], completely mix all the sieving samples crushed, put into a ground-glass bottle, and the prepared sample shall be determined immediately.

5.4.2 Determination

Weigh 15 g of the test sample (5.4.1), place in a 250-mL ground-glass stoppered conical flask, add 150 mL of water (3.2.5) (V_{51}) (add a small amount of water in the sample to mix into paste, and then add all), drop in 5 drops of chloroform, shake well after covering the stopper, place and extract at room temperature for 2 h, shake once for every 15 min (or oscillate in the oscillator for 70 min), stand for several minutes after extraction and filter with moderate speed qualitative filter paper, pipette 10 mL (V_{52}) of filtrate with a pipette, inject into a 100-mL conical flask, add 20 mL of water (3.2.5) and 3 drops of phenolphthalein indicator solution, titrate with sodium hydroxide standard solution after mixing well, rotate the flask during dropwise adding, until the color is similar to that of the reference solution, and does not fade for 5 s, and the entire titration operation shall be completed within 45 s. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_5) of sodium hydroxide, and plug into Formula (5) and calculate.

5.4.3 Blank titration

Perform the blank test with 30 mL of water (3.2.5), read the consumed volume (V_0) of sodium hydroxide.

Note: perform in a well-ventilated draught cupboard during operation, for the trichloromethane is toxic.

6 Expression of Analysis Results

The acidity value of test sample in milk powder, expressed as ($^{\circ}$ T), shall be calculated in accordance with Formula (1):

$$X_1 = \frac{c_1 \times (V_1 - V_0) \times 12}{m_1 \times (1 - \omega) \times 0.1} \dots\dots\dots (1)$$

Where,

X_1 —the acidity of test sample, (°T) [expressed as the volume of 0.1 mol/L sodium hydroxide consumed by 100 g of reconstituted milk containing 12% dry matter, mL/100 g];

c_1 —the concentration of sodium hydroxide standard solution, mol/L;

V_1 —the volume of sodium hydroxide standard solution consumed by titration, mL;

V_0 —the volume of sodium hydroxide standard solution consumed by blank test, mL;

12—12 g milk powder is equivalent to 100 mL of reconstituted milk (9 g for skim milk powder, 7 g for skim whey powder);

m_1 —the mass of weighed sample, g;

ω —the mass fraction of moisture in test sample, g/100 g;

$1-\omega$ —the mass fraction of milk powder in test sample, g/100 g;

0.1— the molar concentration of sodium hydroxide defined by acidity theory, mol/L.

The arithmetical mean value of the two independent determination results obtained under the repeatability conditions shall be taken as the test result, and the result shall keep three significant figures.

Note: if the acidity is expressed as the content of lactic acid, the content of lactic acid of the sample (g/100 g)= $T \times 0.009$. T is the titration acidity of the sample (0.009 is the conversion coefficient of lactic acid, namely, 1 mL of 0.1 mol/L sodium hydroxide standard solution is equivalent to 0.009 g of lactic acid.)

The acidity value of test sample in pasteurized milk, sterilized milk, raw milk, fermented milk, cream and condensed milk, expressed as (°T), shall be calculated in accordance with Formula (2):

$$X_2 = \frac{c_2 \times (V_2 - V_0) \times 100}{m_2 \times 0.1} \dots\dots\dots (2)$$

Where,

X_2 —the acidity of test sample, (°T) [expressed as the volume of 0.1 mol/L sodium hydroxide consumed by 100 g of the sample, mL/100 g];

c_2 —the molar concentration of sodium hydroxide standard solution, mol/L;

V_2 —the volume of sodium hydroxide standard solution consumed by titration, mL;

V_0 —the volume of sodium hydroxide standard solution consumed by blank test, mL;

100—100 g of test sample;

m_2 —the mass of test sample, g;

0.1— the molar concentration of sodium hydroxide defined by acidity theory, mol/L.

The arithmetical mean value of the two independent determination results obtained under the repeatability conditions shall be taken as the test result, and the result shall keep three significant figures.

The acidity value of test sample in casein, expressed as (°T), shall be calculated in accordance with Formula (3):

$$X_3 = \frac{c_3 \times (V_3 - V_0) \times 100 \times 2}{m_3 \times 0.1} \dots\dots\dots (3)$$

Where,

X_3 —the acidity of test sample, (°T) [expressed as the volume of 0.1 mol/L sodium hydroxide consumed by 100 g of the sample, mL/100 g];

c_3 —the molar concentration of sodium hydroxide standard solution, mol/L;

V_3 —the volume of sodium hydroxide standard solution consumed by titration, mL;

V_0 —the volume of sodium hydroxide standard solution consumed by blank test, mL;

100—100 g of test sample;

2—the dilution ratio of the test sample;

m_3 —the mass of test sample, g;

0.1—the molar concentration of sodium hydroxide defined by acidity theory, mol/L.

The arithmetical mean value of the two independent determination results obtained under the repeatability conditions shall be taken as the test result, and the result shall keep three significant figures.

The acidity value of test sample in starch and its derived products, expressed as (°T), shall be 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 —the acidity of test sample, (°T) [expressed as the volume of 0.1 mol/L sodium hydroxide consumed by 10 g of the sample, mL/10 g];

c_4 —the molar concentration of sodium hydroxide standard solution, mol/L;

V_4 —the volume of sodium hydroxide standard solution consumed by titration, mL;

V_0 —the volume of sodium hydroxide standard solution consumed by blank test, mL;

10—10 g of test sample;

m_4 —the mass of test sample, g;

0.1000—the molar concentration of sodium hydroxide defined by acidity theory, mol/L.

The arithmetical mean value of the two independent determination results obtained under the repeatability conditions shall be taken as the test result, and the result shall keep three significant figures.

The acidity value of test sample in grain and its products, expressed as (°T), shall be calculated in accordance with Formula (5):

$$X_5 = (V_5 - V_0) \times \frac{V_{51}}{V_{52}} \times \frac{c_5}{0.1000} \times \frac{10}{m_5} \dots\dots\dots (5)$$

Where,

X_5 —the acidity of test sample, (°T) [expressed as the volume of 0.1 mol/L sodium hydroxide consumed by 10 g of the sample, mL/10 g];

V_5 —the volume of sodium hydroxide standard solution consumed by titration, mL;

V_0 —the volume of sodium hydroxide standard solution consumed by blank test, mL;

V_{51} —the volume of water for extraction of test sample, mL;

V_{52} —the volume of test sample filtrate for titration, mL;

c_5 —the molar concentration of sodium hydroxide standard solution, mol/L;

0.1000—the molar concentration of sodium hydroxide defined by acidity theory, mol/L;

10—10 g of test sample;

m_5 —the mass of test sample, g.

The arithmetical mean value of the two independent determination results obtained under the repeatability conditions shall be taken as the test result, and the result shall keep three significant figures.

7 Precision

The absolute difference between the two independent determination results obtained under the

repeatability conditions shall not exceed 10% of their arithmetical mean value.

Method II pH Meter Method

8 Principles

The volume of 0.1000 mol/L sodium hydroxide is consumed for neutralizing the solution to pH value of 8.30, and the acidity is determined by calculation.

9 Reagents and Materials

Unless otherwise specified, all the reagents used in this method are analytically pure reagents, and the water is the Grade 3 water specified in GB/T 6682

9.1 Sodium hydroxide standard solution: the same as section 3.2.1.

9.2 Nitrogen: with the purity of 98%.

9.3 Distilled and carbon dioxide-free water: the same as section 3.2.5.

10 Apparatus and Equipment

10.1 Analytical balance: with the sensitivity of 0.001 g.

10.2 Alkali burette: with scale division of 0.1 mL, and accurate to 0.05 mL. Or use the automatic burette to meet the same requirements.

10.3 PH meter: with a glass electrode and a proper reference electrode.

10.4 Magnetic stirrer.

10.5 High speed mixer, such as homogenizer.

10.6 Thermostat water bath.

11 Analysis Procedures

11.1 Preparation of test sample

Transfer all the samples to a clean and dry container about two times the volume of the sample (with a sealing cap), immediately cover the container, rotate and oscillate repeatedly to completely mix the samples. Try to avoid exposing the sample in the air during the operation.

11.2 Determination

Weigh 4 g of the sample (accurate to 0.01 g) in a 250-mL conical flask. Measure 96 mL of water about 20 °C (9.3) with cylinder to re-dissolve the sample, stir, and then stand for 20 min.

Dropwise add sodium hydroxide standard solution (9.1) to the conical flask with a burette, until the pH value is stable at 8.30 ± 0.01 for 4 s~5 s. During the titration, use the magnetic stirrer to stir all the time, blow the nitrogen (9.2) into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. The entire titration shall be completed within 1 min. Record the consumed volume (V_6) of sodium hydroxide, accurate to 0.05 mL, and plug into Formula (6) and calculate.

11.3 Blank titration

Perform the blank test with 100 mL of distilled water (9.3), read the consumed volume (V_0) of sodium hydroxide.

Note: the volume of sodium hydroxide consumed by the blank shall not be less than zero, otherwise re-prepare the distilled water and use the distilled water as required.

12 Expression of Analysis Results

The acidity value of test sample in milk powder, expressed in ($^{\circ}$ T), shall be calculated in accordance with Formula (6):

$$X_6 = \frac{c_6 \times (V_6 - V_0) \times 12}{m_6 \times (1 - \omega) \times 0.1} \dots\dots\dots (6)$$

Where,

X_6 —the acidity of test sample, (°T);

c_6 —the concentration of sodium hydroxide standard solution, mol/L;

V_6 —the volume of sodium hydroxide standard solution consumed by titration, mL;

V_0 —the volume of sodium hydroxide standard solution consumed by blank test, mL;

12—12 g milk powder is equivalent to 100 mL of reconstituted milk (9 g for skim milk powder, 7 g for skim whey powder);

m_6 —the mass of weighed sample, g;

ω —the mass fraction of moisture in test sample, g/100 g;

$1-\omega$ —the mass fraction of test sample, g/100 g;

0.1— the molar concentration of sodium hydroxide defined by acidity theory definition for acidity of , mol/L.

The arithmetical mean value of the two independent determination results obtained under the repeatability conditions shall be taken as the test result, and the result shall keep three significant figures.

Note: If the acidity is expressed as the content of lactic acid, the content of lactic acid of the sample (g/100 g)= $T \times 0.009$. T is the titration acidity of the sample (0.009 is the conversion coefficient of lactic acid, namely, 1 mL of 0.1 mol/L sodium hydroxide standard solution is equivalent to 0.009 g of lactic acid.)

13 Precision

The absolute difference between the two independent determination results obtained under the repeatability conditions shall not exceed 10% of their arithmetical mean value.

Method III Potentiometric Titrator Method

14 Principles

The volume of 0.1000 mol/L sodium hydroxide is consumed for neutralizing 100 g of test sample to pH value of 8.30, and the acidity is determined by calculation.

15 Reagents and Materials

Unless otherwise specified, all the reagents used in this method are analytically pure reagents, and the water is the Grade 3 water specified in GB/T 6682

15.1 Sodium hydroxide standard solution: the same as section 3.2.1.

15.2 Nitrogen: with the purity of 98%.

15.3 Neutral ethanol-ether mixture: the same as section 3.2.4.

15.4 Distilled and carbon dioxide-free water: the same as section 3.2.5.

16 Apparatus and Equipment

16.1 Analytical balance: with the sensitivity of 0.001 g.

16.2 Potentiometric titrator.

16.3 Alkali burette: with scale division of 0.1 mL.

16.4 Water bath.

17 Analysis Procedures

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

Weigh 10 g (accurate to 0.001 g) of well-mixed sample, place in a 150-mL conical flask, add 20 mL of water freshly boiled and cooled to room temperature, mix well, and potentiometric titrate with sodium hydroxide standard solution to the pH value of 8.3, which is the end point. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_7) of sodium hydroxide, and plug into Formula (7) and calculate.

17.2 Cream

Weigh 10 g (accurate to 0.001 g) of well-mixed sample, place in a 250-mL conical flask, add 30 mL of neutral ethanol-ether mixture, mix well, and potentiometric titrate with sodium hydroxide standard solution to the pH value of 8.3, which is the end point. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_7) of sodium hydroxide, and plug into Formula (7) and calculate.

17.3 Condensed milk

Weigh 10 g (accurate to 0.001g) of well-mixed sample, place in a 250-mL conical flask, add 60 mL of water freshly boiled and cooled to room temperature, mix well, and potentiometric titrate with sodium hydroxide standard solution to the pH value of 8.3, which is the end point. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_7) of sodium hydroxide, and plug into Formula (7) and calculate.

17.4 Casein

Weigh 5 g (accurate to 0.001 g) of evenly grinded sample in a conical flask, add 50 mL of water (15.4), and place at room temperature (18 °C~20 °C) for 4 h~5 h, or place in a water bath heated to 45 °C and maintain for 30 min, add another 50 mL of water (15.4), filter through a dry filter paper after mixing well. Pipette 50 mL of the filtrate in a conical flask, and potentiometric titrate with sodium hydroxide standard solution to the pH value of 8.3, which is the end point. During the titration, blow the nitrogen into the conical flask to prevent the solution from absorbing the carbon dioxide in the air. Record the consumed volume (V_8) of sodium hydroxide, and plug into Formula (8) and calculate.

17.5 Blank titration

Perform the blank test with corresponding volume of water (15.4), read the consumed volume (V_0) of sodium hydroxide (apply to section 17.1, section 17.3 and section 17.4). Perform the blank test with 30 mL of neutral ethanol-ether mixture, read the consumed volume (V_0) of sodium hydroxide (apply to section 17.2).

Note: the volume of sodium hydroxide consumed by the blank shall not be less than zero, otherwise the distilled water shall be reprepared and the distilled water or neutral ethanol-ether mixture as required shall be used.

18 Expression of Analysis Results

The acidity value of test sample in pasteurized milk, sterilized milk, raw milk, fermented milk, cream and condensed milk, expressed as ($^{\circ}\text{T}$), shall be calculated in accordance with Formula (7):

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

Where,

X_7 —the acidity of test sample, ($^{\circ}\text{T}$);

c_7 —the molar concentration of sodium hydroxide standard solution, mol/L;

V_7 —the volume of sodium hydroxide standard solution consumed by titration, mL;

V_0 —the volume of sodium hydroxide standard solution consumed by blank test, mL;

100—100 g of test sample;

m_7 —the mass of test sample, g;

0.1— the molar concentration of sodium hydroxide defined by acidity theory, mol/L.

The arithmetical mean value of the two independent determination results obtained under the repeatability conditions shall be taken as the test result, and the result shall keep three significant figures. The acidity value of test sample in casein, expressed as (°T), shall be calculated in accordance with Formula (8):

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

Where,

X_8 — the acidity of test sample, (°T);

c_8 — the molar concentration of sodium hydroxide standard solution, mol/L;

V_8 — the volume of sodium hydroxide standard solution consumed by titration, mL;

V_0 — the volume of sodium hydroxide standard solution consumed by blank test, mL;

100— 100 g of test sample;

2— the dilution ratio of the test sample;

m_8 — the mass of test sample, g;

0.1— the molar concentration of sodium hydroxide defined by acidity theory, mol/L.

The arithmetical mean value of the two independent determination results obtained under the repeatability conditions shall be taken as the test result, and the result shall keep three significant figures.

19 Precision

The absolute difference between the two independent determination results obtained under the repeatability conditions shall not exceed 10% of their arithmetical mean value.