NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 12456-2021

Replacing GB/T 12456-2008

National food safety standard - Determination of total acid in foods

食品安全国家标准 食品中总酸的测定

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Relationship to previous standards

This document replaces GB/T 12456-2008 in full. It also replaces the total acid determination methods of GB/T 4928-2008, GB/T 5009.39-2003, GB/T 5009.40-2003, GB/T 5009.41-2003, GB/T 10345-2007 and GB/T 21999-2008, which remain in force for their other provisions.

1 Scope

The standard specifies methods for the determination of total acid in fruit and vegetable

products, beverages, alcoholic drinks and condiments.

The first method applies to the determination of total acid in fruit and vegetable products, beverages of the clear and transparent kind, baijiu, rice wine, white grape wine, beer and white vinegar. The second method applies to the determination of total acid in fruit and vegetable products, beverages, alcoholic drinks and condiments. The third method applies to the determination of total acid in fruit and vegetable products, beverages, alcoholic drinks and condiments.

2 First method: principle

On the principle of acid-base neutralisation, the acid in the test solution is titrated with alkali, the end point being fixed with phenolphthalein as indicator. The total acid content of the food is calculated from the amount of alkali consumed.

3 First method: reagents and materials

Unless otherwise stated, all reagents used in the method are of analytical grade and the water is grade 2 water as specified in GB/T 6682.

The reagents are ethanol, 95 %; phenolphthalein; and sodium hydroxide.

Carbon dioxide free water is prepared by boiling water for 15 min to drive off the carbon dioxide, then cooling and sealing it. Phenolphthalein indicator solution at 10 g/L is prepared by weighing 1 g of phenolphthalein, dissolving it in 95 % ethanol and diluting to 100 mL with 95 % ethanol. Sodium hydroxide standard titration solution at 0.1 mol/L is prepared and standardised as required by GB/T 5009.1, or bought as a standard titration solution certified by the state and issued with a certified reference material certificate. Sodium hydroxide standard titration solution at 0.01 mol/L is prepared by transferring 100 mL of the 0.1 mol/L solution with a pipette into a volumetric flask and diluting to 1 000 mL with water; it is prepared immediately before use and restandardised if necessary. Sodium hydroxide standard titration solution at 0.05 mol/L is prepared by transferring 50 mL of the 0.1 mol/L solution with a pipette into a volumetric flask and diluting to 100 mL with water; it is prepared immediately before use and restandardised if necessary.

4 First method: instruments and equipment

An analytical balance with a resolution of 0.01 g and of 0.1 mg; a basic burette of 10 mL capacity with a minimum graduation of 0.05 mL; a basic burette of 25 mL capacity with a minimum graduation of 0.1 mL; a water bath; conical flasks of 100 mL, 150 mL and 250 mL; pipettes of 25 mL, 50 mL and 100 mL; a homogeniser; an ultrasonic generator; a mortar; and a tissue grinder.

5 First method: analytical procedure

5.1 The sample is kept sealed at ambient temperature. Liquid samples free from carbon dioxide are mixed thoroughly and placed in a closed glass container; for samples containing carbon dioxide, at least 200 g of sample, weighed to 0.01 g, is placed in a 500 mL beaker and shaken under reduced pressure for 3 min to 4 min to remove the carbon dioxide from the liquid. For solid samples, at least 200 g of representative sample, weighed to 0.01 g, is placed in a mortar or tissue grinder, an equal quantity of carbon dioxide free water is added, the sample is ground in the mortar or broken up in the grinder and mixed to a slurry and then placed in a closed glass container. For mixed solid and liquid samples, at least 200 g, weighed to 0.01 g, is taken in the solid to liquid ratio of the sample, ground in the mortar or broken up in the grinder, mixed and placed in a closed glass container.

5.2 For liquid samples, 25 g of sample is weighed to 0.01 g, or 25.0 mL is transferred with a pipette, into a 250 mL volumetric flask, which is made up to the mark with carbon dioxide free water and shaken; the solution is filtered through rapid filter paper and the filtrate collected for the determination. For other samples, 25 g of sample is weighed to 0.01 g into a 150 mL conical flask fitted with a condenser, about 50 mL of carbon dioxide free water at 80 °C is added and mixed in, and the flask is boiled in a boiling water bath for 30 min, being shaken two or three times so that all the organic acids in the sample dissolve; it is then removed, cooled to room temperature, made up to 250 mL with carbon dioxide free water and filtered through rapid filter paper, the filtrate being collected for the determination.

5.3 According to the likely total acid content of the sample, 25 mL, 50 mL or 100 mL of the test solution is transferred with a pipette into a 250 mL conical flask, 2 to 4 drops of phenolphthalein indicator solution at 10 g/L are added and the solution is titrated with sodium hydroxide standard titration solution at 0.1 mol/L, or, for samples such as baijiu with a total acid content not greater than 4 g/kg, at 0.01 mol/L or 0.05 mol/L, until a faint pink colour persists for 30 s. The volume of sodium hydroxide standard titration solution consumed is recorded. A blank test is carried out as in 5.3.1 with the same volume of carbon dioxide free water in place of the test solution and the volume of sodium hydroxide standard titration solution consumed is recorded.

6 First method: calculation of results

The total acid content is calculated by Formula (1). The symbols are: the total acid content of the sample, in grams per kilogram or grams per litre; the concentration of the sodium hydroxide standard titration solution, in moles per litre; the volume of sodium hydroxide standard titration solution consumed in titrating the test solution, in millilitres; the volume consumed in the blank test, in millilitres; the conversion factor of the acid, which is 0.067 for malic acid, 0.060 for acetic acid, 0.075 for tartaric acid, 0.064 for citric acid, 0.070 for citric acid monohydrate, 0.090 for lactic acid, 0.036 for hydrochloric acid, 0.049 for sulfuric acid

and 0.049 for phosphoric acid; the dilution factor of the test solution; the mass of the sample in grams or the volume of sample taken in millilitres; and the conversion factor 1 000.

The result is expressed as the arithmetic mean of two independent determinations obtained under repeatability conditions and is retained to two decimal places.

7 First method: precision

The absolute difference between two independent determinations obtained under repeatability conditions shall not exceed 10 % of their arithmetic mean.

8 Second method: principle

On the principle of acid-base neutralisation, the acid in the test solution is titrated with sodium hydroxide standard titration solution and the end point is taken as the point at which the sample solution is neutralised to pH 8.2, or, where the acid is phosphoric acid, to pH 8.7 to 8.8. The total acid content of the food is calculated from the amount of alkali consumed.

9 Second method: reagents and materials

Unless otherwise stated, all reagents used in the method are of analytical grade and the water is grade 2 water as specified in GB/T 6682.

The reagents are sodium hydroxide, dipotassium hydrogen phosphate and potassium dihydrogen phosphate. Sodium hydroxide standard titration solution at 0.1 mol/L is as in 3.2. pH 8.0 buffer solution is prepared by taking 5.59 g of dipotassium hydrogen phosphate and 0.41 g of potassium dihydrogen phosphate and making up to 1 000 mL with water.

10 Second method: instruments and equipment

An analytical balance with a resolution of 0.01 g and of 0.1 mg; a pH meter with an accuracy of plus or minus 0.1 pH; a magnetic stirrer; a water bath; beakers of 100 mL, 150 mL and 250 mL; and pipettes of 25 mL, 50 mL and 100 mL.

11 Second method: analytical procedure

11.1 The sample is prepared as in 5.1 and the test solution as in 5.2.

11.3 According to the likely total acid content of the sample, 25 mL, 50 mL or 100 mL of the test solution is transferred with a pipette into a 150 mL beaker. The pH meter is switched on and, once stable, calibrated according to its calibration procedure or with the pH 8.0 buffer solution. The beaker containing the test solution is placed on the magnetic stirrer and the electrode immersed in the solution. The pH reading switch is pressed, the stirrer started and the solution titrated rapidly with sodium hydroxide standard titration solution at 0.1 mol/L, or, for samples such as baijiu with a total acid content not greater than 4 g/kg, at