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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 5009.229-2025

**National food safety standard - Determination of acid value
in food**

食品安全国家标准 食品中酸价的测定

Issued on: March 16, 2025

Implemented on: September 16, 2025

**Issued by: National Health Commission of the People's Republic of China;
State Administration for Market Regulation.**

Table of Contents

Foreword	3
1 Scope	4
First method -- Cold solvent indicator titration method	4
2 Principle	4
3 Reagents and materials	4
4 Instruments and equipment	6
5 Analysis steps	6
6 Expression of analysis results	8
7 Precision	9
Second method -- Cold solvent automatic potentiometric titration	9
8 Principle	9
9 Reagents and materials	9
10 Instruments and equipment	10
11 Analysis steps	10
12 Expression of analysis results	12
13 Precision	12
Third method -- Hot ethanol indicator titration method	12
14 Principle	12
15 Reagents and materials	12
16 Instruments and equipment	13
17 Analysis steps	13
18 Expression of analysis results	14
19 Precision	14
Forth method -- Spectrophotometry	14
20 Principle	14
21 Reagents and materials	14
22 Instruments and equipment	15
23 Analysis steps	16
24 Expression of analysis results	19
25 Precision	19
26 Others	19
Annex A Impurity removal and drying and dehydration of oil and fat samples	21
Annex B Schematic diagram of titration end point determination by automatic potentiometric titration method	23

National food safety standard - Determination of acid value in food

1 Scope

This Standard specifies the determination methods for acid value in food.

The first, second and third methods are applicable to the determination of acid value in food [except non-dairy cream, powdered oils and fats, margarine, compound seasonings (mayonnaise, salad dressing, oil-based chili sauce, nut and seed sauce, hot pot base and other semi-solid seasonings)].

The fourth method is applicable to the determination of acid value in non-dairy cream, powdered oils and fats, margarine, compound seasonings (mayonnaise, salad dressing, oil-based chili sauce, nut and seed sauce, hot pot base and other semi-solid seasonings).

First method -- Cold solvent indicator titration method

2 Principle

Based on the principle of acid-base neutralization reaction, potassium hydroxide or sodium hydroxide standard titration solution is used to neutralize the free fatty acids in the sample solution, and the titration end point is determined by the acid-base indicator. The acid value of the sample is calculated based on the consumption of the standard titration solution.

3 Reagents and materials

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

3.1 Reagents

3.1.1 Isopropyl alcohol (C_3H_8O).

3.1.2 Anhydrous ether ($C_4H_{10}O$).

3.1.3 95 % ethanol (C_2H_5OH).

3.1.4 Phenolphthalein ($C_{20}H_{14}O_4$, CAS No.: 77-09-8).

4 Instruments and equipment

4.1 Burette: capacity of 10 mL, minimum graduation value of 0.05 mL.

4.2 Burette: capacity of 25 mL or 50 mL, minimum graduation value of 0.1 mL.

4.3 Balance: the sensitivity is 0.01 g and 0.001 g respectively.

4.4 Food grinder or pounder.

4.5 Laboratory oil press: screw press, non-heating type.

4.6 Porcelain mortar.

4.7 Constant temperature water bath.

4.8 Constant temperature blast drying oven.

4.9 Centrifuge: speed ≥ 8000 r/min.

4.10 Rotary evaporator or equivalent equipment.

4.11 Soxhlet extraction device.

5 Analysis steps

5.1 Preparation of samples

5.1.1 Animal and vegetable oils and fats, edible hydrogenated oils, shortening, cocoa butter substitutes

At room temperature, liquid samples are fully mixed and then directly sampled for testing. For solid (or semi-solid) samples, take a representative sample and place it in a warm water bath or a constant temperature blast drying oven to heat and melt, mix well and then take the oil and fat for testing. If the oil and fat sample is obviously turbid, emulsified, stratified or precipitated, it shall be treated with impurity removal and dehydration according to Annex A.

5.1.2 Vegetable oils, nuts and seeds

Direct pressing method is used for samples susceptible to lipase, such as raw and dried sesame: Take a representative sample, use laboratory oil press to press at room temperature, collect the squeezed material and filter it with filter paper immediately, and then take the oil and fat for determination.

Soxhlet extraction method is used for other samples: For samples with shells, peel off the shell first and keep the edible part. Among them, the kernels with green inner

membrane (such as pumpkin seeds, melon seeds, etc.) shall also remove the green inner membrane attached to the surface of the kernels (the method of removing the green inner membrane is to spray the surface of the shelled kernels with laboratory water to moisten, rub off the green inner membrane, and then place the kernels with the green inner membrane removed in a 50 °C constant temperature blast drying oven for 45 min to dry). The sample is fully crushed with a food grinder, pounder or porcelain mortar. If the sample is obviously heated during the crushing process, it shall add an appropriate amount of liquid nitrogen and carry out the crushing in a frozen state. After the sample is prepared, it shall immediately pack the crushed sample with a filter paper tube and place it in a Soxhlet extraction device, add anhydrous ether or petroleum ether from the upper end of the condenser tube of the extraction device to two-thirds of the bottle volume, and heat and extract in a water bath for 4 h. Recover the extract in a flask, place it in a rotary evaporator with a water bath temperature not higher than 40 °C, evaporate the organic solvent under reduced pressure, and use the residue as an oil and fat sample for acid value determination.

If the residue is obviously turbid, emulsified, stratified or precipitated, it shall be treated with impurity removal and dehydration according to Annex A.

5.1.3 Other foods containing fats and oils

Take the edible part of a representative sample (samples with more water content can be drained with gauze first), and use a food grinder, pounder or porcelain mortar to fully crush the sample (so that the free fat in the sample can be fully extracted by petroleum ether). If the sample is obviously heated during the crushing process, it shall add an appropriate amount of liquid nitrogen and carry out the crushing in a frozen state.

Place the crushed sample in a wide-mouth bottle (samples with more water content can be dehydrated by adding an appropriate amount of anhydrous sodium sulfate), add 2 ~ 3 times the volume of petroleum ether, stir and mix, seal and extract for more than 12 h. After stirring, let it stand for a while, filter through a funnel filled with anhydrous sodium sulfate, take the filtrate; in a water bath not higher than 40 °C, use a rotary evaporator to evaporate the petroleum ether under reduced pressure, and use the residue as an oil and fat sample for acid value determination.

If the residue is obviously turbid, emulsified, stratified or precipitated, it shall be treated with impurity removal and dehydration according to Annex A.

5.2 Weighing of samples

According to the estimated acid value of the sample, weigh the oil and fat sample according to the sample weight specified in Table 1 and place it in a 250 mL conical flask.

According to the estimated acid value of the sample, weigh the oil and fat sample according to the sample weight specified in Table 1 and place it in a 200 mL beaker.

11.3 Determination of samples

Add 50 mL ~ 100 mL of cold solvent to the weighed oil and fat sample, then add a polytetrafluoroethylene magnetic stirrer, and place the sample on a magnetic stirrer to stir and dissolve. Then, insert the electrode and burette connected to the automatic potentiometric titrator into the sample solution. Note that the glass bulb of the electrode and the anti-diffusion head of the burette shall be completely immersed below the liquid surface of the sample solution and it shall avoid touching the inner wall of the beaker. Start the automatic potentiometric titrator and titrate with potassium hydroxide or sodium hydroxide standard titration solution.

The reference conditions of the automatic potentiometric titrator are as follows.

- Minimum liquid addition volume: 0.01 mL ~ 0.06 mL.
- Maximum liquid addition volume: 0.1 mL ~ 0.5 mL.
- Signal drift: 20 mV ~ 30 mV.
- Start the automatic monitoring function to automatically plot the corresponding pH-titration volume change curve and the corresponding first-order differential curve in real time, as shown in Annex B.
- End point determination method: The titration end point is the point indicated by the peak of the first-order differential curve caused by the “pH jump” on the “S”-shape pH-titration volume real-time change curve generated when the free fatty acids undergo neutralization reaction (as shown in Figure B.1 in Annex B). After the titration end point, the automatic potentiometric titrator will automatically stop titration, the titration ends, and the volume of the consumed standard titration solution will be automatically displayed. During the titration process, if there are multiple “pH jumps” in the oil and fat sample (such as rice bran oil, etc.), the titration end point is the “pH jump” with the pH at the starting point of the “jump” that is most consistent with or close to the pH range of 7.5 ~ 9.5 (as shown in Figure B.2); if a “direct jump” type pH-titration volume change curve is generated, the titration end point is the peak of the corresponding first-order differential curve (as shown in Figure B.3); if multiple first-order differential peaks are generated on a “pH jump”, the titration end point is the highest peak (as shown in Figure B.4).

After the titration of each sample ends, the electrode, burette and stirrer shall be rinsed with solvent first and then with water before the next sample can be measured. A blank test shall be performed at the same time.

15.2.1 Phenolphthalein indicator (10 g/L): weigh 1 g of phenolphthalein, dissolve in 95 % ethanol and dilute to 100 mL.

15.2.2 Thymolphthalein indicator (20 g/L): weigh 2 g of thymolphthalein, dissolve in 95 % ethanol and dilute to 100 mL.

15.2.3 Alkali blue 6B indicator (20 g/L): weigh 2 g of alkali blue 6B, dissolve in 95 % ethanol and dilute to 100 mL.

15.3 Preparation of standard solutions

Same as 3.3.

16 Instruments and equipment

Same as Clause 4.

17 Analysis steps

17.1 Preparation of samples

Same as 5.1.

17.2 Weighing of samples

Same as 5.2.

17.3 Determination of samples

Take another 250 mL conical flask, add 50 mL ~ 100 mL of 95 % ethanol, then add 0.5 mL ~ 1 mL of phenolphthalein indicator, shake well and place it in a water bath to heat until it is slightly boiling. Take out the conical flask, and while the temperature of the ethanol solution is still above 70 °C, titrate it with potassium hydroxide or sodium hydroxide standard titration solution until it turns slightly red and does not fade for 15 s. Pour the solution into the conical flask containing the sample while it is still hot, then place it in a water bath to heat until it is slightly boiling, and gently shake the sample until it is completely melted. Take out the conical flask, and while the temperature of the solution is still above 70 °C, titrate it with potassium hydroxide or sodium hydroxide standard titration solution until it turns slightly red and does not fade for 15 s, which is the titration end point. This method does not require a blank test, and $V_0 = 0$ mL.

NOTE: When the color of the oil and fat sample affects the determination of end point, thymolphthalein indicator or alkali blue 6B indicator can be used instead of phenolphthalein indicator. When using thymolphthalein indicator, the solution color turns blue, which is the titration end point; when using alkali blue 6B indicator, the solution color fades from blue to slightly red,

21.2 Preparation of reagents

21.2.1 Copper acetate solution (50 g/L): weigh 25 g of (accurate to 0.001 g) of copper acetate monohydrate, add 450 mL of water and stir until completely dissolved, adjust the solution pH to 6.10 ~ 6.20 with pyridine, and finally dilute to 500 mL with water.

21.2.2 Cyclohexane-isopropyl alcohol solution (98 + 2): mix cyclohexane and isopropyl alcohol in a volume ratio of 98 : 2.

21.2.3 Petroleum ether-methyl tert-butyl ether solution (1 + 3): mix petroleum ether and methyl tert-butyl ether in a volume ratio of 1 : 3.

21.3 Standards

Oleic acid standard ($C_{18}H_{34}O_2$, CAS No.: 112-80-1): purity $\geq 98\%$, certified by the state and awarded with a standard material certificate.

21.4 Preparation of standard solutions

21.4.1 Oleic acid standard stock solution (10 mg/mL): accurately weigh 1 g (accurate to 0.0001 g) of oleic acid standard, dissolve with an appropriate amount of cyclohexane, transfer to a 100 mL brown volumetric flask, and dilute to the mark with cyclohexane. Store at $2\text{ }^{\circ}\text{C} \sim 8\text{ }^{\circ}\text{C}$ away from light, the shelf life is 3 months.

21.4.2 Oleic acid standard series working solutions: take different volumes of oleic acid standard stock solution, dilute with cyclohexane to oleic acid standard working solutions with mass concentrations of 0.0 mg/mL, 0.1 mg/mL, 0.5 mg/mL, 1.0 mg/mL, 3.0 mg/mL and 5.0 mg/mL. Prepare it before use.

21.5 Materials

21.5.1 10 mL stoppered graduated test tube.

21.5.2 15 mL centrifuge tube.

21.5.3 50 mL centrifuge tube.

21.5.4 0.22 μm or 0.45 μm nylon filter membrane.

21.5.5 Solid phase extraction column (silica gel) or equivalent purification column: 6 mL, 500 mg.

22 Instruments and equipment

22.1 Balance: the sensitivity is 0.001g and 0.0001g respectively.

22.2 Spectrophotometer: equipped with 1 cm cuvette, the maximum detection wavelength is not less than 800 nm.

temperature not higher than 40 °C, evaporate the organic solvent under reduced pressure, and use the residue as an oil and fat sample for acid value determination.

If the residue is obviously turbid, emulsified, stratified or precipitated, it shall be treated with impurity removal and dehydration according to Annex A.

23.1.3 Compound seasonings (mayonnaise, salad dressing, oil-based chili sauce, nut and seed sauce, hot pot base and other semi-solid seasonings)

Take the edible part of a representative sample (samples with high water content can be drained with gauze first), and use a food grinder, pounder or porcelain mortar to fully crush the sample (so that the free fat in the sample can be fully extracted by petroleum ether). If the sample is obviously heated during the crushing process, add an appropriate amount of liquid nitrogen and carry out the crushing in a frozen state.

Put the crushed sample in a wide-mouth bottle (samples with high water content can be dehydrated by adding an appropriate amount of anhydrous sodium sulfate first), add 2 ~ 3 times the volume of petroleum ether, stir and mix, seal and let it stand for more than 12 h. After stirring, let it stand for a while, filter through a funnel filled with anhydrous sodium sulfate, take the filtrate; in a water bath not higher than 40 °C, use a rotary evaporator to evaporate the petroleum ether under reduced pressure, and use the residue as an oil and fat sample for acid value determination.

If the residue is obviously turbid, emulsified, stratified or precipitated, it shall be treated with impurity removal and dehydration according to Annex A.

23.2 Purification of oil and fat samples

In order to eliminate the interference of possible stearyl lactylate emulsifiers, the sample shall be purified according to the following steps.

Rinse and soak the solid phase extraction column with 10 mL of cyclohexane-isopropyl alcohol solution (98 + 2) and connect it to the solid phase extraction device for use.

Weigh 1 g (accurate to 0.001 g) of oil and fat sample into a 15 mL centrifuge tube, add 4 mL of cyclohexane-isopropyl alcohol solution (98 + 2) to dissolve (if the oil and fat sample solidifies, it shall be placed in a warm water bath to fully dissolve, and then cooled to room temperature), transfer all the sample solution to the solid phase extraction column, turn on the vacuum pump and adjust the flow rate, pass through the column at a rate of 1 drop/s ~ 2 drops/s, collect the effluent, and then rinse the centrifuge tube with 6 mL of cyclohexane-isopropyl alcohol solution (98 + 2) and pass through the column, combine the effluent, transfer to a flask, use a rotary evaporator at 60 °C to evaporate the organic solvent under reduced pressure, the residue is used as the purified oil and fat sample, and is treated according to 23.3.

23.3 Preparation of oil and fat sample solutions

23.3.1 Preparation of liquid oil and fat sample solutions

Weigh 1 g (accurate to 0.001 g) of liquid oil and fat sample into a 10 mL stoppered graduated test tube, dissolve it with cyclohexane and dilute to 5 mL, then transfer all of it to a 50 mL centrifuge tube, add 3 mL ~ 5 mL of water, cover and seal, and fully vortex and mix it on a vortex mixer for 40 s ~ 60 s, let stand to allow it to separate into layers (or use a centrifuge to make it to separate into layers), take the upper layer solution and determine it according to the steps in 23.4.2.

23.3.2 Preparation of solid (semi-solid) oil and fat sample solutions

Place the solid (or semi-solid) oil and fat sample in a warm water bath or a constant temperature blast drying oven to heat and melt, mix well and weigh 1 g (accurate to 0.001 g) of oil and fat sample into a 10 mL stoppered graduated test tube, dissolve it with cyclohexane and dilute to 10 mL, then transfer all of it to a 50 mL centrifuge tube, add 3 mL ~ 5 mL of water, cover and seal, fully vortex and mix on a vortex mixer for 40 s ~ 60 s, let stand to allow it to separate into layers (or use a centrifuge to make it to separate into layers), take the upper layer solution and determine it according to the steps in 23.4.2.

23.4 Determination

23.4.1 Plotting of standard curve

Take six 50 mL centrifuge tubes, respectively add 5 mL of oleic acid standard working solution with mass concentrations of 0.0 mg/mL, 0.1 mg/mL, 0.5 mg/mL, 1.0 mg/mL, 3.0 mg/mL and 5.0 mg/mL, add 2 mL of cupric acetate solution to each tube, cover and place them on a vortex mixer for 30 s, then let stand to allow the solutions to separate into layers, take the upper layer clear liquid and filter through a filter membrane, use a 1 cm cuvette, take the oleic acid standard working solution with a mass concentration of 0.0 mg/mL as a reference, measure the absorbance at a wavelength of 710 nm, and plot a standard curve.

23.4.2 Determination of samples

Take 4 mL ~ 5 mL of oil and fat sample solution (23.3) in a 50 mL centrifuge tube, add 2 mL of copper acetate solution, cover and place it on a vortex mixer to vortex and mix for 30 s, then let stand to allow the solution to separate into layers (if emulsification occurs, use a centrifuge to make the solution to separate into layers), take the upper layer clear liquid and filter through a filter membrane, use a 1 cm colorimetric dish, take the oleic acid standard working solution with a mass concentration of 0.0 mg/mL as a reference, measure the absorbance at a wavelength of 710 nm, and obtain the total free fatty acid concentration (in terms of oleic acid) in the sample solution from the standard curve, which is converted to the acid value of the sample.

If the color of the oil and fat sample solution is darker, take an equal amount of oil and fat sample for a blank test (except that the copper acetate solution is not added, the rest

Annex A

Impurity removal and drying and dehydration of oil and fat samples

A.1 Impurity removal of oil and fat samples

If the oil and fat sample is obviously turbid, emulsified, stratified or precipitated, it shall be treated with impurity removal. First, place the oil and fat sample in a 50 °C water bath or a constant temperature drying oven, heat the oil and fat to 50 °C and shake it thoroughly to melt possible oil and fat crystals. If the oil and fat sample becomes clear and has no precipitation at this time, it can be used as a sample. Otherwise, the insoluble impurities shall be filtered out with filter paper while it is hot, and the filtrate is be taken as a sample. The filtration process shall be completed as soon as possible.

If the impurity content in the oil and fat sample is high and the particles are small and difficult to filter clean, the oil and fat sample can be centrifuged at 8000 r/min for 10 min ~ 20 min to remove impurities.

For samples with a freezing point higher than 50 °C or containing oil and fat components with a freezing point higher than 50 °C, place the oil and fat in a water bath or constant temperature drying oven about 10 °C higher than its freezing point, heat the oil and fat and shake it thoroughly to melt oil and fat crystals. If filtration is required, place the oil and fat in a constant temperature drying oven about 10 °C higher than its freezing point, filter the insoluble impurities with filter paper, and take the filtered clear liquid oil and fat as a sample. The filtration process shall be completed as soon as possible.

A.2 Drying and dehydration of oil and fat samples

If the oil and fat sample contains water and still cannot become clear after treatment in A.1, it shall be dried and dehydrated. For oil and fat samples without crystallization or coagulation, and oil and fat samples without crystallization or coagulation after treatment in A.1 and cooling to room temperature, add 1 g ~ 2 g of anhydrous sodium sulfate per 10 g of oil and fat sample, stir and mix thoroughly, then filter with filter paper, and take the clear liquid oil and fat after filtration as a sample.

If the water content in the oil and fat sample is high, centrifuge the oil and fat sample at 8000 r/min for 10 min ~ 20 min, and after it separates into layers, take the upper layer of oil and fat sample and dehydrate it by adsorption with anhydrous sodium sulfate.

For oil and fat samples that have crystallization or solidification at room temperature, and oil and fat samples that have obvious crystallization or solidification after treatment in A.1 and cooling to room temperature, it can completely dissolve the oil and fat sample with an appropriate amount of petroleum ether in a water bath at 40 °C ~ 55 °C, and then add an appropriate amount of anhydrous sodium sulfate, stir and mix

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