GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.227-2023

National food safety standard - Determination of peroxide value in food

食品安全国家标准　食品中过氧化值的测定

Issued on: September 6, 2023 **Implemented on: March 6, 2024**

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

Table of Contents

National food safety standard - Determination of peroxide value in food

1 Scope

This standard specifies the method for the determination of peroxide value in food.

Method A is suitable for the determination of peroxide value in food.

Method B is suitable for the determination of peroxide value in edible animal and vegetable oils, fats, and margarine.

Method A - Indicator titration method

2 Principle

The prepared oil and fat sample is dissolved in chloroform-glacial acetic acid solution, the peroxide reacts with potassium iodide to generate iodine, and the precipitated iodine is titrated with sodium thiosulfate standard titration solution. The amount of peroxide value is expressed as the mass fraction of peroxide equivalent to iodine or the number of millimoles of active oxygen in 1 kg of the sample.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are of analytical grade, and the water is the third-grade water specified in GB/T 6682.

3.1 Reagents

3.1.1 Glacial acetic acid (CH_3COOH).

3.1.2 Trichloromethane ($CHCl_3$).

3.1.3 Potassium iodide (KI).

3.1.4 Petroleum ether: The boiling range is 30 ℃~60 ℃.

Confirmation of petroleum ether: Take 100 mL of petroleum ether in a rotary evaporation bottle, and evaporate to dryness under reduced pressure by using a rotary evaporator in a water bath not higher than 40 °C. Wash the rotary evaporation bottle

several times with 30 mL of chloroform-glacial acetic acid solution, and combine the washing liquid into a 250 mL iodine flask. Accurately add 1.00 mL of saturated potassium iodide solution, plug the bottle cap tightly, and shake gently for 0.5 min. Leave it in a dark place for 3 min, add 1.0 mL of starch indicator, and mix well. If no blue color appears, this petroleum ether can be used for the sample preparation; If a blue color appears after adding 1.0 mL of starch indicator and mixing, the reagent needs to be replaced.

3.1.5 Anhydrous sodium sulfate (Na_2SO_4).

3.1.6 Soluble starch.

3.1.7 Acetone (CH_3COCH_3).

3.1.8 Amylase (CAS number: 9000-92-4): The enzyme activity is ≥2000 U/g.

3.1.9 Papain (CAS number: 9001-73-4): The enzyme activity is ≥6000 U/mg.

3.1.10 Sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$).

3.2 Reagent preparation

3.2.1 Chloroform-glacial acetic acid solution (2+3): Mix trichloromethane and glacial acetic acid in a volume ratio of 2:3.

3.2.2 Starch indicator (10 g/L): Weigh 1 g of soluble starch, add about 5 mL of water to make it into a paste, add 95 mL of boiling water to the paste while stirring, boil for 1 to 2 minutes, and cool. Prepare the indicator fresh just before use.

3.2.3 Saturated solution of potassium iodide: Weigh about 16 g of potassium iodide, add 10 mL of newly boiled and cooled water, shake well, store in a brown bottle, cap the bottle, and store in a dark place for later use. Ensure that there are saturated potassium iodide crystals in the solution. If the blank volume requirement in 5.2 is exceeded, it shall be prepared again.

3.3 Preparation of standard solution

3.3.1 Sodium thiosulfate standard titration solution (0.1 mo1/L): It shall be prepared and calibrated in accordance with the requirements of GB/T 5009.1; or a standard titration solution with national certification and a Reference Material Certificate.

3.3.2 Sodium thiosulfate standard titration solution (0.01 mo1/L): It is prepared from diluting 0.1 mo1/L sodium thiosulfate standard titration solution with newly boiled and cooled water. Prepare the solution fresh just before use.

3.3.3 Sodium thiosulfate standard titration solution (0.002 mo1/L): It is prepared from diluting 0.01 mo1/L sodium thiosulfate standard titration solution with newly boiled

5.1.2.2 Margarine

Place the sample in a closed container, heat it in an electric constant-temperature drying oven at 60 °C to 70 °C until it melts, shake and mix, continue heating until the emulsification breaks down into layers, and filter the oil layer through the rapid qualitative filter paper into a beaker. The filtrate in the beaker is the sample to be tested, and the sample to be tested shall be clarified. Take a sample and measure it immediately while the sample to be tested is in a liquid state.

5.1.2.3 Non-dairy cream

Take a representative sample in a beaker, add about 5 times the sample volume of petroleum ether, and stir for 2 minutes by using an overhead stirrer to mix evenly. While stirring, add anhydrous sodium sulfate of about 1.6 times the mass of the sample, continue stirring and mixing for 5 minutes, remove the beaker, and let the solution stand for 5 minutes to allow the petroleum ether to layer (if emulsification occurs, cover the top of the beaker with a layer of plastic wrap, and place the beaker in a water bath of not higher than 40 °C for 10 minutes to stratify the petroleum ether). Pour out the supernatant, add about 2 times the sample volume of petroleum ether to the beaker, and repeat the above stirring and standing operations; combine the petroleum ethers, filter, and transfer the filtrate into a brown rotary evaporation bottle; in a water bath of not higher than 40 ℃, use a rotary evaporator to evaporate the petroleum ether to dryness under reduced pressure. The residue is the sample to be tested, and the extraction amount is not less than 5 g.

5.1.2.4 Powder oil products

Weigh a representative sample into a brown iodine flask, and add 0.02 g of papain and 0.02 g of amylase for every 1 g of the sample. Add 2 times the sample volume of water, mix well, and cap the bottle. Place the iodine flask in a 50 °C constant-temperature water bath oscillator, oscillate 60 times/min to 100 times/min for 30 minutes, take it out, and cool it. Add acetone of the same volume as water and mix. Add 3 times the sample volume of petroleum ether and shake to extract for 1 minute; transfer it to a separatory funnel, let it stand for 30 minutes to separate layers, and discard the lower layer. If emulsification occurs, the layers can be separated by a high-speed refrigerated centrifuge (5000 r/min, 15 °C, 3 min), and then the organic phase can be transferred to a separatory funnel. Add water of the same volume as petroleum ether to wash the organic phase, discard the lower layer, and transfer the upper organic phase to a funnel containing anhydrous sodium sulfate for filtration. Transfer the filtrate into a brown rotary evaporation bottle, and use a rotary evaporator to evaporate the petroleum ether to dryness under reduced pressure in a water bath of not higher than 40 °C. The residue is the sample to be tested, and the extraction amount is not less than 5 g.

NOTE: For wall material samples containing only protein, only papain can be added; for wall material samples containing only carbohydrates, only amylase can be added.

5.1.3 Plant foods and their products (made by frying, puffing, baking, preparing, stir-frying, steaming, and other processing techniques) and animal food products (made by quick freezing, drying, pickling, frying, and other processing techniques)

Take out the edible part of the representative sample from all the samples taken, and remove the oil-free part. Quick-frozen prepared meat samples containing more moisture can be prepared after draining the water with gauze. Crush and mix the sample thoroughly, place it in a wide-mouth bottle, add 2 to 3 times the sample volume of petroleum ether, shake well, mix thoroughly, seal, and let it stand and extract for more than 12 hours; ultrasonic treatment for 5 to 10 minutes if necessary. Filter through a funnel containing anhydrous sodium sulfate, take the filtrate, and use a rotary evaporator to evaporate the petroleum ether to dryness under reduced pressure in a water bath of not higher than 40 °C. The residue is the sample to be tested, and the extraction amount is not less than 5 g.

5.2 Sample determination

Sample determination shall be avoided under direct sunlight. Weigh 2 g~3 g of the prepared sample (accurate to 0.001 g), place it in a 250 mL iodine flask, add 30 mL of chloroform-glacial acetic acid solution, and shake the sample gently until it is completely dissolved. Accurately add 1.00 mL of saturated potassium iodide solution, plug the bottle cap tightly, shake gently for 0.5 min, and place in a dark place for 3 min. Take out and add 100 mL of water, shake well and immediately use sodium thiosulfate standard titration solution (when the estimated peroxide value is 0.15 g/100 g and below, use 0.002 mol/L standard titration solution; when the estimated peroxide value is greater than 0.15 g/100 g, use 0.01 mol/L standard titration solution) to titrate the precipitated iodine. When the solution turns to light yellow, add 1 mL of starch indicator, continue titrating, and shake vigorously until the blue color of the solution disappears, that is the end point. A blank test is carried out at the same time. The volume V_0 of sodium thiosulfate standard titration solution consumed in the blank test shall not exceed 0.1 mL.

6 Presentation of analysis results

6.1 When the peroxide value is expressed as the mass fraction of peroxide equivalent to iodine, it is calculated according to formula (1).

$$X_1 = \frac{(V - V_0) \times c \times 0.126\ 9}{m} \times 100 \qquad \cdots\cdots (1)$$

where:

X_1 -- peroxide value, the unit is grams per hundred grams (g/100 g);

The preparation is the same as 3.3.

10 Instruments and equipment

10.1 Balance: The sensitivities are 0.01 g, 0.001 g, and 0.0001 g, respectively.

10.2 Electric constant-temperature drying oven.

10.3 Potentiometric titrator: The accuracy is ±2 mV.

10.4 Magnetic stirrer.

NOTE: All vessels used in this method must not contain reducing or oxidizing substances. The ground glass surface must not be coated with oil.

11 Analysis steps

11.1 Sample preparation

The preparation is the same as 5.1.1 and 5.1.2.2.

11.2 Sample determination

Weigh 5 g of the prepared sample (accurate to 0.001 g) and place it into the titration cup of the potentiometric titrator, add 50 mL of isooctane-glacial acetic acid solution, and shake gently to completely dissolve the sample. If the solubility of the sample is poor (such as stearin or animal fat), first add 20 mL of isooctane to the titration cup, shake gently to dissolve the sample, then add 30 mL of glacial acetic acid, and mix well.

Accurately add 1.00 mL of saturated potassium iodide solution to the titration cup, start the magnetic stirrer, and react at a suitable stirring speed for 60 s±1 s. Immediately add 30 mL~100 mL water to the titration cup, insert the electrode and titration head, set the titration parameters, run the titration program, perform the titration, and observe the titration curve and potential changes. The added amount of sodium thiosulfate standard titration solution is generally controlled at 0.05 mL/drop~0.2 mL/drop. After reaching the titration end point, record the volume of standard solution consumed at the titration end point. After the titration of a sample is completed, the stirrer or stirring magnet, titration head, and electrode need to be immersed in isooctane to clean the oils and fats on the surface.

A blank test is performed at the same time. Carry out titration and observe the titration curve and potential changes. The added amount of sodium thiosulfate standard titration solution is generally controlled at 0.005 mL/drop. After reaching the titration end point, record the volume V_0 of the standard solution consumed at the titration end point. The

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------