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NATIONAL STANDARD
OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 5009.39-2003
Replacing GB/T 5009.39-1996

Method for analysis of hygienic standard of soybean sauce

酱油卫生标准的分析方法

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Foreword

This standard replaces GB/T 5009.39-1996 " *Method for analysis of hygienic standard of soybean sauce*".

Compared with GB/T 5009.39-1996, the major changes of this standard are as follows:

- According to GB/T 20001.4-2001 "*Rules for drafting standards - Part 4: Methods for chemical analysis*" of the original structure has been revised standards;
- ADD Amino Nitrogen Colorimetric Method as the second method.

This standard was proposed and managed by Ministry of Health of the People's Republic of China.

Drafting organizations of this standard: Beijing Municipal Health and Epidemic Prevention Station, Handan City Health and Epidemic Prevention Station, and Tangshan City Health and Epidemic Prevention Station.

This standard was first-time released in 1985, and the first-revision in 1996. This is the second revision.

Method for analysis of hygienic standard of soybean sauce

1 Scope

This standard specifies the analysis method of health indicators of soybean sauce.

This standard applies to the analysis of health indicators of soybean sauce which is brewed or prepared from the raw materials such as grain and its by-products soybean meal and wheat bran.

The detection limit of the 2nd method - Amino Nitrogen Method in this standard, is 0.070 μg/mL, the linear range is 0-10 μg/mL.

2 Reference standards

The following documents contain provisions which, through reference in this Standard, become provisions of this Standard. For dated references, their subsequent amendments (excluding corrections) or revisions do not apply to this Standard. However, the parties who enter into agreement based on this Standard are encouraged to investigate whether the latest versions of these documents are applicable. For undated reference documents, the latest versions apply to this Standard.

GB/T 5009.2 Determination of relative density of foods
GB/T 5009.11 Determination of total arsenic and abio-arsenic in foods
GB/T 5009.12 Determination of lead in foods
GB/T 5009.22 Determination of aflatoxin B1 in foods
GB/T 5009.29 Determination of sorbic acid and benzioc acid in foods

3 Sensory inspection

3.1 Take 2 mL of sample in 25 mL with-stopper colorimetric tube. Add water to the mark. Shake and observe the color and clarity. It shall be no-dirty, and no-sediment.

3.2 Take 30 mL of sample in 50 mL beaker. It shall be no-mold, no mildew and floating film.

3.3 After stirring the sample in beaker with a glass rod, its taste shall be without smell such as sour, bitter, or astringent.

4.2.1.5 Result calculation

Amino nitrogen content of the sample shall be calculated according to equation (1).

$$X = \frac{(V_1 - V_2) \times c \times 0.014}{5 \times V_3/100} \times 100 \quad \cdots\cdots\cdots\cdots (1)$$

In which:

X – the amino nitrogen content of the sample, in units of grams per hundred milliliters (g/100 mL);

V_1 – the volume of consumed sodium hydroxide standard titration solution after formaldehyde is added into the test sample dilution, in units of milliliters (mL);

V_2 – the volume of consumed sodium hydroxide standard titration solution after formaldehyde is added into the reagent blank test, in units of milliliters (mL);

V_3 - the taken amount of sample dilution, in units of milliliters (ml);

c - the concentration of sodium hydroxide standard titration solution , in units of moles per liter (mol/L);

0.014 – the mass of nitrogen corresponding to 1.00 mL of sodium hydroxide standard titration solution [c(NaOH) = 1.000 mol/L], in units of grams (g).

Calculation result shall retain 2 significant figures.

4.2.1.6 Precision

Under repeatability conditions, the absolute difference obtained from 2 independent test results shall not exceed 10% of the arithmetic mean.

4.2.2 Second method - Colorimetry

4.2.2.1 Principle

In pH 4.8 sodium acetate-acetic acid buffer solution, the amino nitrogen is reacted with acetyl acetone and formaldehyde to form a yellow 3,5-diacetyl-2,6-dimethyl-1,4-dihydropyridine amino acid derivatives. Measure the absorbance at wavelength of 400 nm. Compare quantitatively with the standard series.

4.2.2.2 Reagents

4.2.2.2.1 Acetic acid solution (1 mol/L): Take 5.8 mL of glacial acetic acid. Add water to dilute to 100 mL.

4.2.2.2.2 Sodium acetate solution (1 mol/L): Take 41 g of anhydrous sodium acetate or 68 g of sodium acetate ($CH_3COONa \cdot 3H_2O$). Add water to dissolve. And dilute to 500 mL.

standard regression equation. Calculate the sample content.

4.2.2.5 Result calculation

Amino nitrogen content of the sample shall be calculated according to formula (2).

$$X = \frac{c}{V_1 \times \frac{V_2}{50} \times 1\,000 \times 1\,000} \times 100 \qquad \cdots\cdots\cdots\cdots\cdots\cdots (2)$$

In which:

X – the amino nitrogen content of the sample, in units of grams per hundred milliliters (g/100 mL);

c - the nitrogen mass in test sample solution, in units of micrograms (μg);

V_1 - sample volume, in units of milliliters (mL);

V_2 - volume of test sample solution, in units of milliliters (mL).

4.2.2.6 Precision

Under repeatability conditions, the absolute difference obtained from 2 independent test results shall not exceed 10% of the arithmetic mean.

4.3 Salt (counted in NaCl)

4.3.1 Principle

Use silver nitrate standard solution to titrate sodium chloride in the sample. Generate silver chloride to precipitate. After all silver chloride is precipitated, the extra titrated silver nitrate and potassium chromate indicator generate silver chromate, until the solution shows orange, that is the end. From the consumption of silver nitrate standard titration solution, calculate the sodium chloride content.

4.3.2 Reagents

4.3.2.1 Silver nitrate standard titration solution [c($AgNO_3$) = 0.100 mol/L].

4.3.2.2 Potassium chromate solution (50 g/L): Weigh 5 g of potassium chromate. Use a small amount of water to dissolve. Dilute to 100 mL.

4.3.3 Instruments

10 ml microburette.

4.3.4 Analysis steps

Draw 2.0 mL of sample dilution in a 150 mL-200 mL conical flask. Add 100 mL of water and 1 mL of potassium chromate solution (50 g/L). Mix well. Use silver nitrate standard

4.8 Benzoic acid, sorbic acid

Operate according to GB/T 5009.29.

4.9 Ammonium salt (semi-micro Kjeldahl method)

4.9.1 Principle

Sample is heated in an alkaline solution by distillation. Ammonia is evaporated. It is absorbed by boric acid solution. Then use hydrochloric acid standard solution to titrate and calculate the content.

4.9.2 Reagents

4.9.2.1 Magnesium oxide.

4.9.2.2 Boric acid solution (20 g/L).

4.9.2.3 Hydrochloric acid standard titration solution [c(HCl) = 0.100 mol/L].

4.9.2.4 Mixed indicator solution: 1 portion of methyl red-ethanol solution (2 g/L) and 5 portions of bromocresol green-ethanol solution (2 g/L). Mix well when using.

4.9.3 Analysis steps

Draw 2 mL of sample. Place in a 500 mL distillation flask. Add about 150 mL of water and about 1 g of magnesium oxide. Connect the distillation device. The connection bent tube at lower end of condensation tube is inserted below the liquid surface of receiving bottle. In the receiving bottle, it contains 10 mL of boric acid solution (20 g/L) and 2-3 drops of mixed indicator solution. Heat for distillation. Steam for about 30 min, counted from the boiling. Rinse the bent tube with a little water. Use hydrochloric acid standard solution (0.100 mol/L) to titrate to the end. Take the same amount of water, magnesium oxide, and boric acid solution to conduct reagent blank test according the same method.

4.9.4 Result calculation

Ammonium salt content of the sample (counted in ammonia) shall be calculated according to formula (5).

$$X = \frac{(V_1 - V_2) \times c \times 0.017}{V_3} \times 100 \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (5)$$

In which:

X – the ammonium salt content (counted in ammonia), in units of grams per one hundred milliliters (g/100 mL);

V_1 - the volume of hydrochloric acid standard titration solution consumed in test sample dilution, in units of milliliters (mL);

V_2 - the volume of hydrochloric acid standard titration solution consumed in reagent blank, in units of milliliters (mL);

c - the actual concentration of hydrochloric acid standard titration solution, in units of moles per liter (mol/L);

0.017 - the ammonium salt (counted in ammonia) mass corresponding to 1.00 ml of hydrochloric acid standard solution [c(HCl)=1.000 mol/L], units in grams (g);

V_3 - the sample volume, in units of milliliters (mL).

The result shall retain 2 significant figures.

4.9.5 Precision

Under repeatability conditions, the absolute difference obtained from 2 independent test results shall not exceed 10% of the arithmetic mean.

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