GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 1886.64-2015

National food safety standard – Food additives – Caramel color

食品安全国家标准 食品添加剂 焦糖色

Issued on: November 13, 2015 **Implemented on: May 13, 2016**

Issued by: National Health and Family Planning Commission of the People's Republic of China

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National food safety standard – Food additives – Caramel color

1 Scope

This Standard applies to the food additive caramel color made from sucrose, starch syrup, xylose mother liquor, etc. using plain caramel, caustic sulfite caramel, ammonia caramel or sulfite ammonia caramel.

2 Terms and definitions

2.1 plain caramel color

It is made using carbohydrates as the main raw material, with or without the addition of acid (base), and without the use of ammonia compounds and sulfites.

2.2 caustic sulfite caramel color

It is made using carbohydrates as the main raw material, in the presence of sulfite, with or without the addition of acid (base), and without the use of ammonia compounds.

2.3 ammonia caramel color

It is made using carbohydrates as the main raw material, in the presence of ammonia compounds, with or without the addition of acid (base), and without the use of sulfites.

2.4 sulfite ammonia caramel color

It is made using carbohydrates as the main raw material, in the presence of ammonia compounds and sulfites, with or without the addition of acid (base).

3 Technical requirements

3.1 Sensory requirements

Sensory requirements shall be in accordance with Table 1.

Appendix A

Test methods

A.1 General

Unless otherwise specified in this Standard, the purity of the reagents used shall be analytically pure. The standard titration solutions, standard solutions for impurity determination, preparations and products used shall be prepared in accordance with the provisions of GB/T 601, GB/T 602 and GB/T 603. The test water shall comply with the provisions of Grade 3 water in GB/T 6682. The solutions used in the test shall refer to aqueous solutions unless the solvent used is specified.

A.2 Determination of absorbance $E_{1\ \mathrm{cm}}^{0.1\%}$ (610 nm)

A.2.1 Instruments and apparatuses

Spectrophotometer.

A.2.2 Analysis steps

Weigh about 0.5 g of the sample, accurate to 0.002 g; dissolve it in water; transfer it to a 500 mL volumetric flask; add water to dilute it to the mark; shake it well. If the solution is turbid, centrifuge it to obtain the sample solution. Place this sample solution in a 1 cm colorimetric dish; use water as a blank control; use a spectrophotometer to measure its absorbance at 610 nm (it is recommended to control the absorbance at 0.2 ~ 0.8, otherwise, adjust the sample solution concentration and measure the absorbance again).

A.2.3 Result calculation

Calculate the absorbance $E_{1\ \mathrm{cm}}^{0.1\%}$ (610 nm) according to Formula (A.1):

$$E_{1\ \mathrm{cm}}^{0.1\%}(610\ \mathrm{nm}) = \frac{A}{c \times 1\ 000} \qquad \cdots\cdots\cdots\cdots (\mathrm{A}.1)$$

Where:

A – absorbance of the sample being measured;

c – concentration of the sample solution being measured, in grams per milliliter (g/mL);

1 000 – concentration conversion coefficient.

The test result is based on the arithmetic mean of the parallel determination results. The ratio of the absolute difference between two independent determination results obtained under repeatability conditions and the arithmetic mean shall not exceed 5%.

A – built-in adapter;

B – separating funnel;

C – round bottom flask;

D – gas internal pipe;

E – condenser;

F – connecting ball (see Figure A.2 for the structure of component F);

G – receiver.

Figure A.1 – Diagram of sulfur dioxide determination device

The device in Figure A.1 is used to selectively transfer sulfur dioxide from the sample to the 3% hydrogen peroxide solution in a boiling hydrochloric acid aqueous solution. This device is easier to connect than a conventional device. Since the height of the 3% hydrogen peroxide solution is above the ball tip, the back pressure in the device is difficult to avoid, and component F can reduce the back pressure to the lowest possible level, thereby reducing the possibility of sulfur dioxide loss due to leakage.

Component D in Figure A.1 needs to be equipped with a hose connection. If polyethylene and quartz tubes are used, they shall be pre-boiled before use in this procedure.

Connect the entire device according to the requirements of Figure A.1. Except for the connection between the separating funnel and the flask, the sealing surfaces of all other connectors shall be coated with a thin layer of piston lubricant. All connectors shall be clamped tightly to ensure sealing during analysis. The volume of the separating funnel B shall be greater than or equal to 100 mL. It must be equipped with a built-in adapter A with a hose connector to ensure that a certain pressure is maintained above the internal solution. (It is not recommended to use a constant pressure titration funnel because the condensed water may contain sulfur dioxide and adhere to the inner wall of the funnel or the wall of the tube). The round bottom flask C, with a volume of 1 000 mL, is provided with 3 24/40 mm conical interfaces. The gas internal pipe D shall be of sufficient length to ensure that the introduced nitrogen can reach 2.5 cm from the bottom of the flask. The jacket length of the condenser E shall be 300 mm. The connecting ball F is a custom-made glass part according to the requirements of Figure A.2, with the same size as the 50 mL measuring barrel. The 3% hydrogen peroxide solution is placed in a receiver G, which has an inner diameter of 2.5 cm and a length of 18 cm.

connection is completed, the nitrogen flow through the 3% hydrogen peroxide solution shall be restored immediately, and the connection shall be checked to ensure it is sealed.

The rubber ball above the separating funnel is equipped with a valve to ensure that there is sufficient pressure above the hydrochloric acid solution. Open the separating funnel valve to allow the hydrochloric acid solution to flow into the flask. Continue to ensure that there is sufficient pressure above the solution. If necessary, the valve can be temporarily closed to replenish the pressure. To prevent sulfur dioxide from escaping into the separating funnel, the valve shall be closed before the last few milliliters flow out of the separating funnel.

Connect the power supply for heating, and control the heating rate so that the reflux liquid is 80 ~ 90 drops per minute. After distillation for 105 minutes, cool the contents in the 1 000 mL flask at the above reflux rate. Transfer the contents in receiver G.

A.4.4.4 Titration

Add 3 drops of methyl red indicator solution and use standard titration solution to titrate the above solution to the yellow end point; ensure that it does not fade within 20 seconds.

A.4.5 Result calculation

Calculate the mass fraction w_2 of sulfur dioxide (as SO_2) according to Formula (A.3):

$$w_2 = \frac{M \times V \times c \times 0.1}{2 \times m \times 1\ 000 \times A_{610}} \times 100\% \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (\text{A.3})$$

Where:

M – molar mass of sulfur dioxide, in grams per mole (g/mol), [M (SO_2) = 64.06];

V – volume of the sodium hydroxide standard titration solution consumed in the titration, in milliliters (mL);

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

0.1 – conversion coefficient;

2 – conversion coefficient;

m – mass of the sample added to the 1 000 mL flask, in grams (g);

1 000 – conversion coefficient;

A_{610} – absorbance of the sample at 610 nm.

A.5.5.1 Preparation of 4-methylimidazole standard stock solution

Accurately weigh 50 mg of 4-methylimidazole standard; place it in a 50 mL volumetric flask; use acetone to dilute to the mark; mix well; refrigerate for later use.

A.5.5.2 Preparation of 4-methylimidazole standard solution

Take 1.0 mL, 1.5 mL, 2.0 mL, 2.5 mL, 3.0 mL, 3.5 mL, 4.0 mL and 5.0 mL of 4-methylimidazole standard stock solution; respectively place them in 10 mL volumetric flasks; use acetone to dilute to the mark; mix well; prepare 4-methylimidazole standard solutions with concentrations of 100 μg/mL, 150 μg/mL, 200 μg/mL, 250 μg/mL, 300 μg/mL, 350 μg/mL, 400 μg/mL and 500 μg/mL, respectively. Keep refrigerated and use within 1 month.

A.5.5.3 Preparation of sample solution

Weigh 10.0 g of the sample; place it in a 250 mL beaker; add 5 mL of sodium hydroxide solution; shake well. Add 20.0 g of diatomaceous earth to the beaker; use a stainless-steel spatula to stir thoroughly until a semi-dry, uniform mixture is formed.

Insert fine glass wool at the bottom of a 300 mm × 22 mm (inner diameter) chromatographic column with a polytetrafluoroethylene stopper; transfer the mixture quantitatively to the chromatographic column. The column layer is about 150 mm high. Place a little glass wool on the top of the column. Use dichloromethane to wash the sample beaker; open the stopper; pour the washing solution into the column. When dichloromethane flows through the chromatographic column to the stopper, close the stopper and let dichloromethane stay in the column layer for 5 minutes. Open the stopper again and let dichloromethane pass through the column at a flow rate of 5 mL/min. Collect 200 mL of eluent in a 300 mL round-bottom flask; place it on a rotary evaporator; maintain the water bath temperature at 35 °C and the pressure at 350 mm ~ 390 mm Hg to evaporate most of the solvent in the eluent and concentrate it to about 1 mL. During the concentration process, pay attention to the flask to prevent boiling. Use a pipette to quantitatively transfer the concentrate to a 5 mL volumetric flask; use acetone to wash the flask several times (about 0.7 mL each time); transfer all the washings to a 5 mL volumetric flask; use acetone to dilute to the mark; mix well; set aside.

A.5.5.4 Determination

Adjust the gas chromatograph to the operating conditions shown in Table A.1. After the instrument is stable, perform chromatographic analysis on the 4-methylimidazole standard solution and sample solution of different concentrations; quantify by the external standard method.

A.6 Determination of total sulfur (as S)

A.6.1 Reagents and materials

A.6.1.1 Magnesium oxide.

A.6.1.2 Magnesium nitrate [$Mg(NO_3)_2 \cdot 6H_2O$].

A.6.1.3 Sucrose.

A.6.1.4 Nitric acid.

A.6.1.5 Hydrochloric acid solution: 1+1.

A.6.1.6 Barium chloride solution: mass fraction 10%.

A.6.2 Analysis steps

Select the largest porcelain crucible that matches the high-temperature furnace (to prevent splashing of the reaction); add 1 g ~ 3 g of magnesium oxide or an equivalent amount of magnesium nitrate [$Mg(NO_3)_2 \cdot 6H_2O$] (6.4 g ~ 19.2 g), 1 g of sucrose, and 50 mL of nitric acid. Weigh an appropriate amount of sample (5 g, if total sulfur content ≤ 2.5%; 1 g, if total sulfur content > 2.5%), accurate to 0.01 g; add it to the porcelain crucible. Evaporate in a steam bath to a paste; then, carbonize on an electric furnace until smokeless; then, put it in a high-temperature furnace; heat to 525 °C; maintain the temperature for 4 h ~ 5 h; cool. Dissolve the sample in 100 mL of water; use hydrochloric acid solution to neutralize it to pH7; add 2 mL of hydrochloric acid solution; filter the solution into a beaker; heat to boiling; slowly add 20 mL of barium chloride solution to the hot solution while stirring; boil for 5 min; leave it overnight. Filter it through the ash-free filter paper; transfer all the precipitate to the filter paper; use hot water to thoroughly wash the filter paper and precipitate. Then, put the filter paper and precipitate into a tongs pan that has been weighed in a high-temperature furnace at 800 °C in advance; keep it in an oven at 105 °C for 1 hour; take it out. Use an electric furnace to heat slowly until it is carbonized. Then, ash it in a high-temperature furnace at 800 °C for 1 hour; cool it; and weigh it. Perform a blank test in the same way.

A.6.3 Result calculation

Calculate the mass fraction w_3 of total sulfur (as S) according to Formula (A.4):

$$w_3 = \frac{(m_S - m_B) \times 0.137 \times 0.1}{m \times A_{610}} \times 100\% \quad \cdots\cdots\cdots\cdots (A.4)$$

Where:

m_S – residual mass of barium sulfate after burning, in grams (g);

m_B – residual mass obtained from the blank test after burning, in grams (g);

0.137 – coefficient for converting barium sulfate to sulfur;

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