# GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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# National food safety standard - Food nutrition enhancer - Manganese carbonate

食品安全国家标准

食品营养强化剂 碳酸锰

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# Table of Contents

# National food safety standard - Food nutrition enhancer - Manganese carbonate

## 1 Scope

This standard applies to the food nutrition enhancer manganese carbonate prepared by chemical synthesis and with manganese sulfate and ammonium bicarbonate as raw materials.

## 2 Molecular formula and relative molecular mass

### 2.1 Molecular formula

$MnCO_3$

### 2.2 Relative molecular mass

114.95 (according to 2018 International Relative Atomic Mass)

## 3 Technical requirements

### 3.1 Sensory requirements

Sensory requirements shall meet the requirements in Table 1.

### 3.2 Physicochemical indexes

The physicochemical indexes shall meet the requirements in Table 2.

# Appendix A

# Testing method

## A.1 Warning

Some of the reagents used in this standard test method are toxic or corrosive, and shall be used with caution and in accordance with relevant regulations. If a toxic or corrosive reagent splashes on the skin, it shall be washed with water immediately; if the hurt is serious, it shall be treated immediately. When volatile acids are used, the operation shall be done in a fume hood.

## A.2 General provision

Unless otherwise specified in this standard, the purity of the reagents used shall be above analytical purity, and the preparation of the standard titration solutions, standard solutions for impurity determination, preparations and products used shall be in accordance with the provisions of GB/T 601, GB/T 602, and GB/T 603; the test water shall meet the requirements of the third-grade water in GB/T 6682. The solution used in the test refers to the aqueous solution when the solvent used for preparation is not indicated.

## A.3 Identification test

### A.3.1 Reagents and solutions

**A.3.1.1** Hydrochloric acid.

**A.3.1.2** Acetic acid.

**A.3.1.3** Nitric acid.

**A.3.1.4** Hydrochloric acid solution: Take 50.0 mL of hydrochloric acid (A.3.1.1), add water to make up to 100 mL, and shake well to obtain the solution.

**A.3.1.5** Nitric acid solution: Take 25.0 mL of nitric acid (A.3.1.3), add water to make up to 100 mL, and shake well to obtain the solution.

**A.3.1.6** Ammonium sulfide.

**A.3.1.7** Sodium bismuthate.

**A.3.1.8** Calcium hydroxide saturated solution: Weigh about 3 g of calcium oxide, and the weight shall be accurate to 0.1 g; put it in a reagent bottle, add 1000 mL of water,

cover the bottle, and shake vigorously; let it stand for clarification, and then take the supernatant for using.

**A.3.2 Instruments and equipment**

Electronic balance: The sense quantity shall be 0.1 mg, 0.01 g, and 0.1 g.

**A.3.3 Identification method**

**A.3.3.1 Manganese identification**

**A.3.3.1.1** Weigh 1 g of the sample, and add hydrochloric acid solution dropwise until the sample is completely dissolved; continue to add hydrochloric acid solution to form 20 mL test sample solution, and then add ammonium sulfide drop by drop to produce an orange-red precipitate. After the mixture stands for 10 min, the acetic acid is added dropwise, and then the precipitate gradually dissolves.

**A.3.3.1.2** Weigh 1 g of the sample, dissolve it in the nitric acid solution, and add a little amount of sodium bismuthate powder to produce the purplish red color.

**A.3.3.2 Carbonate identification**

Weigh 1 g of the sample, add 100 mL of water, add hydrochloric acid solution dropwise to the test solution, and then a colorless and odorless gas releases; introduce it into the saturated solution of calcium hydroxide, the solution becomes white and turbid at first, then continue to introduce the gas and the turbidity becomes clear gradually.

**A.4 Determination of manganese carbonate content (ferrous ammonium sulfate titration method)**

**A.4.1 Method principle**

The sample is put in a phosphoric acid medium, and the perchloric acid is added to oxidize manganese into trivalent; the *N*-Phenylanthranilic acid is used as the indicator, and the ferrous ammonium sulfate standard titration solution is used for titration; the manganese carbonate content can be calculated according to the difference between the consumption of ferrous ammonium sulfate standard titration solution in the titration sample test and blank test.

**A.4.2 Reagents and materials**

**A.4.2.1** Phosphoric acid.

**A.4.2.2** Nitric acid.

**A.4.2.3** Hydrochloric acid.

$c$ --- The concentration of potassium dichromate standard solution, in moles per liter (mol/L);

$V_1$ --- 25.00, the volume of potassium dichromate standard solution pipetted, in milliliters (mL);

$m_1$ --- 114.95, the molar mass of manganese carbonate, in grams per mole (g/mol);

$10^{-3}$ --- The calculation factor;

$V_2$ --- The volume of ferrous ammonium sulfate standard titration solution consumed during titration, in milliliters (mL);

$V_3$ --- The volume of ferrous ammonium sulfate standard titration solution consumed during titration in the blank test, in milliliters (mL).

**A.4.2.9.3** Determination of blank value

Pipette 10.00 mL potassium dichromate standard solution (A.4.2.8) into a 250 mL conical flask, add 40 mL sulfuric acid solution (A.4.2.7), 5 mL phosphoric acid (A.4.2.1), and use the ferrous ammonium sulfate standard titration solution (A.4.2.9) to titrate until the orange-yellow color disappears; add 2 drops of *N*-Phenylanthranilic acid indicator solution (A.4.2.10) dropwise, and continue dripping carefully until the solution just turns green, which is the end point; record the volume $V_4$. Then, pipette 10.00 mL potassium dichromate standard solution (A.4.2.8) into the above-mentioned conical flask, then titrate to the end point with the ferrous ammonium sulfate standard titration solution (A.4.2.9) and record the volume $V_5$. The volume difference ($V_5$-$V_4$) between the consumption of ferrous ammonium sulfate in the two titrations is the blank value $V_3$.

**A.4.2.10** *N*-Phenylanthranilic acid indicator solution: 0.2 g/L. Weigh 0.2 g of *N*-Phenylanthranilic acid and dissolve it in a small amount of water; add 0.2 g of sodium carbonate, heat at low temperature to dissolve it, add water to the volume of 1000 mL, and mix well.

**A.4.3 Analysis steps**

**A.4.3.1 Blank test**

Carry out a blank test together with the sample; carry out the titration according to A.4.2.9.3, but do not add sulfuric acid and phosphoric acid; record the volume $V_6$.

**A.4.3.2 Determination**

Weigh 0.20 g of the sample (the weight shall be accurate to 0.0001 g), place it in a 250 mL conical flask, wet the sample with a small amount of water, and shake it carefully

The nitric acid is added to dissolve the sample; the silver nitrate solution is added to the acidic medium, and then the chloride ions and silver ions will form a white silver chloride suspension, which is compared with the standard turbidimetric solution treated at the same time and in the same way.

**A.5.2 Reagents and solutions**

**A.5.2.1** 30% hydrogen peroxide solution.

**A.5.2.2** Nitric acid.

**A.5.2.3** Nitric acid solution: 1+3 (volume ratio); take 1 part of nitric acid (A.5.2.2), add 3 parts of the same volume of water, and shake well to obtain the solution.

**A.5.2.4** Silver nitrate solution: 17 g/L.

Weigh 17.0 g of silver nitrate, put it in a 250 mL beaker, and add an appropriate amount of water to dissolve it; transfer the solution into a 1000 mL brown volumetric flask, dilute to the mark with water, and mix well.

**A.5.2.5** Chloride standard solution: 1 mL of the solution shall contain 0.01 mg of chlorine (Cl), and it shall be prepared according to the requirements of GB/T 9729.

**A.5.3 Analysis steps**

Weigh 1.00 g of the sample, and the weight shall be accurate to 0.01 g; place it in a beaker, moisten it with a small amount of water, and add the nitric acid solution (A.5.2.3) dropwise until the sample is dissolved; add 1 drop of hydrogen peroxide (A.5.2.1) to make the dark color fade, transfer the solution to a 100 mL volumetric flask, dilute to the mark, and shake well (filter if necessary). Pipette 10 mL of test solution into a 25 mL colorimetric tube, add 1 mL of nitric acid solution (A.5.2.3), 1 mL of silver nitrate solution (A.5.2.4), dilute to the mark with water, shake well, and let it stand for 2 min; then, compare it with the standard turbidimetric solution under the black background.

Standard turbidimetric solution: Pipette 1 mL of chloride standard solution, put it in a 25 mL colorimetric tube, and add water to 10 mL; the subsequent steps starting from “add 1 mL of nitric acid...” are the same and simultaneous to the treatment to the sample solution.

The turbidity of the sample solution shall not be deeper than that of the standard turbidimetric solution, that is, the chloride content in the sample shall not be more than 0.01%.

**A.6 Determination of sulfate content (visual turbidimetry)**

**A.6.1 Method principle**

The hydrochloric acid solution is added to dissolve the sample. In an acidic medium, sulfate ions and barium ions form insoluble barium sulfate. When the content of sulfate ions is low, barium sulfate is suspended within a certain period of time, and the solution becomes turbid. The sulfate ethanol solution is added and used as the crystal seed liquid, which can make the particle size appropriate and uniform, thus it is used for the visual turbidimetric determination of sulfate.

**A.6.2 Reagents and materials**

**A.6.2.1** 30% hydrogen peroxide.

**A.6.2.2** Hydrochloric acid.

**A.6.2.3** Hydrochloric acid solution: 1+1 (volume ratio); take 1 part of hydrochloric acid (A.6.2.2), add 1 part of the same volume of water, and shake well to obtain the solution.

**A.6.2.4** Sulfate ethanol solution: 0.148 g/L.

Weigh 0.148 g of anhydrous sodium sulfate dried at 105 °C~110 °C to constant mass, dissolve it in 95% ethanol solution, and dilute the solution to 1000 mL with 95% ethanol solution.

**A.6.2.5** Barium chloride solution: 250 g/L.

**A.6.2.6** Sulfate standard solution: 1 mL solution shall contain 0.10 mg of sulfate ($SO_4$).

**A.6.3 Analysis steps**

Weigh 1.00 g of the test sample, and the weight shall be accurate to 0.01 g; put it in a 100 mL beaker, add 10 mL water, and add the hydrochloric acid solution (A.6.2.3) dropwise until the sample is completely dissolved; then, add 30% hydrogen peroxide (A.6.2.1) to make the dark color fade. Heat it to boil for 2 min, cool to room temperature, transfer all to a 100 mL volumetric flask, dilute to the mark with water, and shake well. Use a pipette to transfer 2.0 mL of the test solution into a 50 mL beaker, add water to 25 mL, and add 0.5 mL of hydrochloric acid solution (A.6.2.3).

Standard turbidimetric solution: Pipette 1.0 mL sulfate standard solution (A.6.2.6) into a beaker, add water to 25 mL, and add 0.5 mL hydrochloric acid solution (A.6.2.3).

Add 0.25 mL sulfate ethanol solution (A.6.2.4) and 1 mL barium chloride solution (A.6.2.5) into two colorimetric tubes respectively, and let them stand for 1 min accurately; pour the sample solution and standard solution in beakers into a colorimetric tube separately, shake well, and let them stand for 10 min.

The turbidity of the sample solution shall not be deeper than that of the standard turbidimetric solution, that is, the sulfate content in the sample shall not be more than

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