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NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 1903.57-2022

National food safety standard - Food nutritional fortification substance - Manganese(II) citrate

Issued on: June 30, 2022 **Implemented on: December 30, 2022**

Issued by: NHC; SAMR

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1 Scope

This standard applies to the food nutrition enhancer manganese citrate prepared by a chemical reaction and with citric acid, manganese sulfate, and manganese carbonate as the main raw materials.

2 Chemical name, molecular formula, structural formula, and

relative molecular mass

2.1 Chemical name

Manganese citrate

2.2 Molecular formula

Mn3(C6H5O7)2 - 10H2O (decahydrate)

Mn3(C6H5O7)2 (anhydrous substance)

2.4 Relative molecular mass

723.17 (decahydrate) (according to 2018 International Relative Atomic Mass)

543.02 (anhydrous substance) (according to 2018 International Relative Atomic Mass)

Appendix A Testing method

A.1 Warning Some of the reagents used in the test method of this standard are toxic or

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corrosive, and they must 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 a volatile acid is used, the operation shall be done in a fume hood.

A.2 General regulation The reagents and water used in this standard refer to the analytical reagents and the third grade and above test water stipulated in GB/T 6682 unless other requirements are specified. The standard titration solution, standard solution for impurity determination, preparations, and products used in the test shall be prepared according to the provisions of GB/T 601, GB/T 602, and GB/T 603 unless other requirements are specified. 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 materials A.3.1.1 Glacial acetic acid.

A.3.1.2 Pyridine.

A.3.1.3 Acetic anhydride.

A.3.1.4 Ammonium sulfide solution: The content shall be >=8%. The amount of residue on ignition of this test solution shall not exceed 0.05%, and shall not be turbid when it encounters magnesium sulfate test solution or calcium chloride test solution. If there is an obvious sulfur precipitate in this test solution, the solution cannot be used. This solution shall be held in a dark brown vial; the vial shall be filled and stored in a cool, dark place.

A.3.1.5 Hydrochloric acid solution: 1 mol/L. Take 8.4 mL hydrochloric acid (36.5%~38.0%), dissolve it in water, and dilute the solution to 100 mL.

A.3.2 Instruments and equipment Analytical balance: The sense quantity shall be 0.1 g and 1 mg.

A.3.3 Identification method A.3.3.1 Identification of manganese ions.

A.3.3.1.1 Method summary When the manganese salt solution meets the ammonium sulfide test solution, a light orange precipitate will be produced, which is soluble in glacial acetic acid.

A.3.3.1.2 Sample solution Weigh about 1 g of the sample (the weight shall be accurate to 0.1 g) and dissolve it in

20 mL of 1 mol/L hydrochloric acid solution.

A.3.3.1.3 Operation steps Add ammonium sulfide solution dropwise to 20 mL of the sample solution to form a light orange precipitate. After standing for 10 minutes, add glacial acetic acid dropwise, and then the precipitate can be dissolved.

A.3.3.2 Identification of citrate A.3.3.2.1 Method summary Citrate produces a carmine color in pyridine-acetic anhydride.

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A.3.3.2.2 Operation method Add 5 mg~10 mg of the sample (the weight shall be accurate to 1 mg) into 15 mL of pyridine, then add 5 mL of acetic anhydride, and shake gently to produce a carmine color.

A.4 Determination of the manganese citrate [Mn3(C6H5O7)2] content A.4.1 Method summary In an ammonia-ammonium chloride buffer solution with a pH of 10, the eriochrome black T is used as an indicator, and the sample solution is titrated with the disodium EDTA standard solution; the content of manganese citrate [by Mn3(C6H5O7)2] can be calculated according to the usage amount of the disodium EDTA standard titration solution.

A.4.2 Reagents and materials A.4.2.1 Hydrochloric acid.

moles per liter (mol/L);

M --- The molar mass of manganese citrate, in grams per mole (g/mol), [M(Mn3(C6H5O7)2=543.02];

m1 --- The mass of the sample, in grams (g);

w2 --- The mass fraction of the weight loss on drying of the sample, %;

3 --- The conversion factor;

1000 --- The conversion factor.

The arithmetic mean of the parallel determination results shall be taken as the test result.

The absolute difference between two independent determination results obtained under repeatability conditions shall be not more than 0.2%.

A.5 Loss on drying A.5.1 Instruments and equipment A.5.1.1 Balance: The sense quantity shall be 0.1 mg.

A.5.1.2 Vacuum drying oven.

A.5.2 Analysis steps Weigh 1.0 g~2.0 g of the sample (the weight shall be accurate to 0.0001 g), and carry out the analysis according to the second method "Vacuum Drying Method" of GB 5009.3. The pressure and temperature in the vacuum drying oven used for the vacuum drying method shall be as follows: the pressure does not exceed 20 mm Hg, the drying temperature is 135 °C, and the time is 16 hours.

The loss on drying of the sample is calculated according to the formula (A.2).

where:

m2 --- The mass of the weighing bottle and the sample, in grams (g);

m3 --- The mass of the weighing bottle and the sample after drying, in grams (g);

m4 --- The mass of the weighing bottle, in grams (g).

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When the loss on drying is >=1 g/100 g, the calculation result retains three significant figures; when the loss on drying is < 1 g/100 g, the calculation result retains two significant figures.

A.6 Determination of arsenic A.6.1 Reagents and materials They shall be prepared according to GB 5009.76.

A.6.2 Instruments and equipment They shall be prepared according to GB 5009.76.

A.6.3 Operation steps The preparation method of the sample solution is as follows: Weigh an appropriate amount of the sample according to GB 5009.76 (the weight shall be accurate to 0.001 g), add about 10 mL of hydrochloric acid, and heat on an electric heating plate until the sample is completely dissolved.

Other operations are carried out according to GB 5009.76.

A.7 Determination of lead A.7.1 Reagents and materials They shall be prepared according to the first method, the third method or the fourth method of GB 5009.12, or according to GB 5009.75.

A.7.2 Instruments and equipment They shall be prepared according to the first method, the third method or the fourth method of GB 5009.12, or according to GB 5009.75.

A.7.3 Operation steps Sample pretreatment shall be carried out according to "5.2 Sample pretreatment" in GB 5009.12-2017, and only nitric acid is used for sample digestion. Other operations shall be carried out according to the first method, the third method or the fourth method of

GB 5009.12-2017, or according to GB 5009.75.

A.8 Determination of sulfate A.8.1 Method summary In an acidic medium, the sulfate ion and barium ion in the sample form barium sulfate precipitate, which is compared with the standard solution of sulfate treated in the same method for a limit test.

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