

national standards of People's Republic of China

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National Food Safety Standard **Food nutrition enhancer manganese citrate**

(Draft for comments)

20 ××-××-×× released

20 ××-××-×× implementation

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National Food Safety Standard

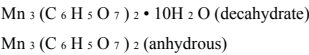
Food nutrition enhancer manganese citrate

1 Scope

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

2 Molecular formula, structural formula and relative molecular mass

2.1 Molecular formula



2.2 Structural formula (anhydrous)

2.3 Relative molecular mass

723.17 (decahydrate) (according to 2018 international relative atomic mass)

543.02 (anhydrous) (according to 2018 international relative atomic mass)

3 Technical requirements

3.1 Sensory requirements

Sensory requirements should meet the requirements of Table 1.

Table 1 Sensory requirements

project	Claim	Testing method
Color	Light pink or powder white	Take an appropriate amount of sample and place it in a clean, dry white porcelain dish,
status	Fine-grained solid	Observe its color and condition under natural light

3.2 Physical and chemical indicators

Should meet the requirements of Table 2.

Table 2 Physical and chemical indicators

project	Finger Mark	Testing method
Manganese citrate content (based on $\text{Mn}_3(\text{C}_6\text{H}_5\text{O}_7)_2$, dry basis), w / %	96.5 ~ 104.8	A.4 in Appendix A
Loss on drying, w / %	Decahydrate	23.0 ~ 26.0
	Anhydrous	≤ 2.0

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Arsenic (As) / (mg / kg)	≤	3.0	Appendix A A.6
Lead (Pb) / (mg / kg)	≤	2.0	Appendix A.7
Sulfate (as SO ₄), w / %	≤	0.02	Appendix A A.8

II

Appendix A

Testing method

A.1 Safety tips

Some of the reagents used in this standard test method are toxic or corrosive. Use caution and follow the relevant regulations when using. If splashing
It should be rinsed with water immediately on the skin and treated immediately in severe cases. When using volatile acids, it should be carried out in a fume hood.

A.2 General provisions

The reagents and water used in this standard refer to analytically pure reagents and tertiary water specified in GB / T 6682 and other
On the test water. The standard titration solution used in the test, the standard solution for the determination of impurities, preparations and products, unless other requirements are noted,
All are prepared in accordance with GB / T 601, GB / T 602, GB / T 603. When the solution used in the test does not indicate which solvent is used for preparation, it refers to
Water solution.

A.3 Identification test

A.3.1 Reagents and materials

A.3.1.1 Hydrochloric acid solution: 1 mol / L. Measure 8.4 mL of hydrochloric acid (36.5% ~ 38.0%), dissolve in water and dilute to 100 mL.

A.3.1.2 Ammonium sulfide solution: analytically pure, content $\geq 8\%$. The amount of burning residue of this test solution shall not be greater than 0.05%, in case of magnesium sulfate test solution. The calcium test solution must not be cloudy. If there is obvious sulfur precipitation in this test solution, the solution cannot be used. This solution should be placed in a dark brown vial Medium, full, stored in a cool, dark place.

A.3.1.3 Glacial acetic acid.

A.3.1.4 Pyridine.

A.3.1.5 Acetic anhydride.

A.3.2 Instruments and equipment

Analytical balance: the sensitivity is 0.1 g and the sensitivity is 1 mg.

A.3.3 Identification method

A.3.3.1 Identification of manganese ions

A.3.3.1.1 Sample solution

Weigh about 1 g (accurate to 0.1 g) of the sample and dissolve it in 20 mL of 1 mol / L hydrochloric acid.

A.3.3.1.2 Method summary

When the manganese salt solution meets the ammonium sulfide test solution, it produces a light orange precipitate, which is soluble in glacial acetic acid.

A.3.3.1.3 Operation steps

Ammonium sulfide solution was added dropwise to the 20 mL test sample solution, resulting in a light orange precipitate. After standing for 10 minutes, add glacial acetic acid dropwise, The precipitate is soluble.

A.3.3.2 Identification of citrate

A.3.3.2.1 Method summary

Citrate produces carmine in pyridine / acetic anhydride.

A.3.3.2.2 Operation method

Add 5-10 mg (accurate to 1 mg) of the sample to 15 mL of pyridine, then add 5 mL of acetic anhydride, shake gently to produce rouge red.

A.4 Determination of manganese citrate ($\text{Mn}_3(\text{C}_6\text{H}_5\text{O}_7)_2$) content

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A.4.1 Method summary

In ammonia-ammonium chloride buffer solution with pH 10, using chrome black T as indicator, titrate with EDTA standard solution For the sample solution, calculate the content of manganese citrate in terms of $\text{Mn}_3(\text{C}_6\text{H}_5\text{O}_7)_2$ according to the amount of standard titration solution of disodium ethylenediaminetetraacetic

A.4.2 Reagents and materials

A.4.2.1 Hydrochloric acid.

A.4.2.2 Standard titration solution of disodium ethylenediaminetetraacetic acid: $c(\text{Na}_2\text{EDTA}) = 0.05 \text{ mol / L}$.

A.4.2.3 Sodium hydroxide solution: 1.0 mol / L.

A.4.2.4 Ammonia-ammonium chloride buffer solution: pH \approx 10. Weigh 67.5 g of ammonium chloride, dissolve in appropriate amount of water, add 570 mL of 28% ammonia water, dilute with water Release to 1000 mL.

A.4.2.5 Chrome black T test solution: Dissolve 200 mg of chrome black T and 2 g of hydroxylamine hydrochloride ($\text{NH}_2\text{OH} \cdot \text{HCl}$) in an appropriate amount of methanol and dilute to 50 mL, then filtered. Store the solution in a dark container and use within two weeks.

A.4.3 Instruments and equipment

Balance: 1 mg.

A.4.4 Analysis steps

Weigh about 0.35 g sample (accurate to 0.001 g), place it in a 250 mL beaker, add 100 mL water and 1 mL hydrochloric acid, at the same time Keep stirring, heating on the hot plate at about 75 °C ~ 80 °C, so that the sample is completely dissolved. Add 25 mL 0.05 mol / L ethylenediaminetetraacetic acid Disodium, if necessary, adjust the pH to 10.0 \pm 0.2 with 1 mol / L NaOH. Add 10 mL of ammonia-ammonium chloride buffer solution and about 8 drops of chrome black T indicator liquid. Use 0.05 mol / L ethylenediaminetetraacetic acid disodium titration solution to continue titration until the pure blue is the end point, and the blue color remains at least 3

minute. Carry out a blank test at the same time.

A.4.5 Calculation of results

The mass fraction w_1 of manganese citrate ($\text{Mn}_3(\text{C}_6\text{H}_5\text{O}_7)_2$) content (calculated on a dry basis) is calculated according to formula (A.1):

$$w_1 = \frac{(V - V_0) \times c \times M}{m_1 \times (1 - w_2) \times 1000} \times 100\% \quad \text{..... (A.1)}$$

In the formula:

V — The volume of EDTA titration solution consumed by the titration sample solution, the unit is milliliter (mL)

V_0 — the volume of EDTA titrant consumed in the titration of the blank solution of the sample, in milliliters (mL);

c — Accurate value of EDTA standard titration solution concentration, the unit is mole per liter (mol / L);

M — the molar mass of manganese citrate in grams per mole (g / mol), [$\text{M}(\text{Mn}_3(\text{C}_6\text{H}_5\text{O}_7)_2) = 543.02$];

m_1 — The mass of the sample in grams (g);

w_2 — Quality score of dry weight loss of actual test sample, %;

3 — Conversion factor;

1000 — conversion factor.

The experimental results are based on the arithmetic mean of the parallel determination results. Two independent determination results obtained under repeatability conditions. The absolute difference is not more than 0.2%.

A.5 Loss on drying

A.5.1 Instruments and equipment

A.5.1.1 Balance: the sense is 0.1 mg.

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A.5.1.2 Oven.

A.5.1.3 Vacuum drying oven.

A.5.2 Analysis steps

Weigh the sample from 1.0 to 2.0 g (accurate to 0.0001 g), and refer to GB 5009.3 Moisture Determination Second Method Vacuum Drying operating. The pressure and temperature of the vacuum drying oven under reduced pressure: the pressure does not exceed 20 mm Hg, the drying temperature is 135 °C, when 16 hours.

The drying weight of the sample is calculated according to formula (A.2):

$$X = \frac{m_1 - m_2}{m_1 - m_3} \times 100\% \quad \text{..... (A.2)}$$

In the formula:

m_1 — The weight of the weighing bottle and sample, the unit is gram (g);

m_2 — The weight of the weighing bottle and sample after drying, the unit is gram (g);

m_3 — The weight of the weighing bottle in grams (g);

Refer to GB 5009.3 moisture determination for the number of significant digits.

A.6 Determination of arsenic

A.6.1 Reagents and materials

Refer to GB 5009.76.

A.6.2 Instruments and equipment

Refer to GB 5009.76.

A.6.3 Operation steps

The preparation method of the sample solution is as follows: Weigh an appropriate amount of sample (accurate to 0.001g) according to GB 5009.76, add about 10 mL of hydrochloric acid. The hot plate is heated 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

Refer to the first method, the third method or the fourth method of GB 5009.12, or the first method or the second method of GB 5009.75.

A.7.2 Instruments and equipment

Refer to the first method, the third method or the fourth method of GB 5009.12, or the first method or the second method of GB 5009.75.

A.7.3 Operation steps

The sample pretreatment is carried out in accordance with 5.2 sample pretreatment in GB 5009.12-2017. The sample digestion only uses nitric acid. Other operations Refer to the first method, the third method or the fourth method of GB 5009.12, or the first method or the second method of GB 5009.75.

A.8 Determination of sulfate

A.8.1 Method summary

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In an acidic medium, the sulfate ions and barium ions in the sample form a barium sulfate precipitate, which is the same as the sulfate standard Comparison of quasi-solutions, limited experiment.

A.8.2 Reagents and materials

A.8.2.1 Hydrochloric acid.

A.8.2.2 Hydrochloric acid solution: 1 + 1 (V + V).

A.8.2.3 Hydrogen peroxide.

A.8.2.4 Ethanol (95%).

A.8.2.5 Potassium sulfate.

A.8.2.6 Potassium sulfate ethanol solution (0.2 g / L): Weigh 0.2 g potassium sulfate, dissolve in 700 mL water, and use ethanol (95%) Dilute to 1000 mL.

A.8.2.7 Standard solution of sulfate (SO_4^{2-}): 0.1 mg / mL. Weigh 0.181 g of potassium sulfate, add water to dissolve, and then move in In a 1000 mL volumetric flask, dilute to the mark.

A.8.2.8 Barium chloride solution: 250 g / L.

A.8.3 Analysis steps

A.8.3.1 Sample solution: Weigh 2.0 g sample (accurate to 0.01 g), place it in a 100 mL beaker, add 10 mL of water, add dropwise Hydrochloric acid until the sample is dissolved, add 1 drop of hydrogen peroxide to fade the dark color, heat and boil for 2 min, cool to room temperature, transfer to 100 mL In a volumetric flask, dilute to the mark with water and shake well. Transfer 25.0 mL of the test solution with a pipette and place it in a 25 mL colorimetric tube, add 0.5 mL Hydrochloric acid solution.

A.8.3.2 Seed solution: Mix 0.25 mL of potassium sulfate ethanol solution with 1 mL of barium chloride solution, and place it accurately for 1 minute.

A.8.3.3 Standard solution: accurately take 1.0 mL of sulfate standard solution in a 25 mL colorimetric tube, add water to the mark, and then add 0.5 mL hydrochloric acid solution.

A.8.3.4 Add 1.25 mL of seed solution to the sample solution and the standard solution colorimetric tube, shake well, and leave for 5 minutes. On a blue background, observe axially. The turbidity of the sample solution shall not be deeper than the standard turbidity solution, the index is qualified.

