

ICS 67.040
CCS X 09



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 1903.18-2016

**National Food Safety Standard -- Food Nutrition Enhancer --
Calcium Citrate-malate**

食品安全国家标准 食品营养强化剂 柠檬酸苹果酸钙

(English Translation)

Issued on: December 23, 2016

Implemented on: June 23, 2017

**Issued by: National Health and Family Planning Commission of the
People's Republic of China, State Food and Drug Administration**

Table of Contents

- 1 Scope
- 2 Chemical name, molecular formula, structural formula
 - 2.1 Chemical name
 - 2.2 Molecular formula
 - 2.3 Structural formula
- 3 Technical requirements
 - 3.1 Sensory requirements
 - 3.2 Physical and chemical indicators

1 Scope

This standard applies to calcium carbonate and other calcium sources, citric acid, malic acid as raw materials, according to a certain proportion of mixed reaction after the neutralization, precipitation, separation, Filtration and drying process and the production of food nutrition enhancer citric acid malate calcium.

2.1 Chemical name

Calcium citrate Calcium citrate

2.2 Molecular formula

$\text{Ca}_x (\text{C}_6\text{H}_5\text{O}_7)_y (\text{C}_4\text{H}_4\text{O}_5)_z \cdot n\text{H}_2\text{O}$

2.3 Structural formula

Taking $\text{Ca}_6 (\text{C}_6\text{H}_5\text{O}_7)_2 (\text{C}_4\text{H}_4\text{O}_5)_3 \cdot 5\text{H}_2\text{O}$ as an example, the structure is as follows.

$\text{Ca}_6 (\text{C}_6\text{H}_5\text{O}_7)_2 (\text{C}_4\text{H}_4\text{O}_5)_3 \cdot 5\text{H}_2\text{O}$

3.1 Sensory requirements

Sensory requirements shall comply with the requirements of Table 1.

Table 1 sensory requirements

The project requires a test method Color white State powder crystals Take the appropriate amount of sample in a clean, dry white porcelain dish Under the light, observe the color and state

3.2 Physical and chemical indicators

Physical and chemical indicators should be consistent with the provisions of Table 2.

Table 2 Physical and chemical indicators

Item Index Test Method Citrate calcium malate content (Ca, dry basis), w /% 20 ~ 26
 Appendix A A.4 pH (100g/L) 5 ~ 8 Appendix A A.5 Loss on drying, w /% ≤ 10 Appendix A
 A.6 Hydrochloric acid insoluble matter, w /% ≤ 0.2 Appendix A A.7 Solubility, w /% ≥ 80
 Appendix A, A.8 Fluoride (in terms of F)/(mg/kg) ≤ 50 GB/T 5009.18 Heavy metals
 (Pb)/(mg/kg) ≤ 10 GB 5009.74 Lead (Pb)/(mg/kg) ≤ 2.0 GB 5009.12 Total arsenic (in
 As)/(mg/kg) ≤ 2.0 GB 5009.11 Appendix A Testing method A.1 Warning Some of the
 reagents used in this standard test procedure are corrosive and require careful operation
 such as splashing into the skin.

Rinse, severe treatment immediately. The use of drugs, should be strictly in accordance
 with the relevant provisions of management; use should avoid inhalation or contact with the
 skin, must should be carried out in a fume hood. Exposed parts of the wound can not touch.

A.2 General provisions the reagents and water used in this standard test method, when not
 specified in other requirements, refer to the analytical reagent and the third grade specified
 in GB/T 6682 water. Standard solution used in the test, the standard solution of impurities,
 preparations and products, in the absence of other requirements, according to GB/T 601,
 GB/T 602, GB/T 603. The solution used in the test refers to the aqueous solution when it is
 not specified in the formulation of the solvent.

A.3 Identification test A.3.1 Reagents and materials A.3.1.1 Ammonia solution. (2 3, volume
 ratio).

A.3.1.2 p-Aminobenzenesulfonic acid. 10 g/L.

A.3.1.3 Sodium hydroxide solution. 40 g/L.

A.3.1.4 sodium nitrite solution..200 g/L.

A.3.2 Malate Root Identification Weigh the sample 0.5g, accurate to 0.01g. Placed in 50mL
 test tube, add water 10mL dissolved. Neutralized with ammonia solution to neutral, plus 1
 mL of p-aminobenzenesulfonic acid solution and heated in a boiling water bath for 5 min.
 Add sodium nitrite solution 5mL, and then placed in a water bath for 3min After adding
 sodium hydroxide solution 5mL, the test solution should be immediately color.

A.3.3 Qualitative identification of infrared spectroscopy Potassium bromide compression
 method, in accordance with the provisions of GB/T 6040 test, the infrared spectrum of the
 sample should be consistent with the control spectrum. Correct the spectrum is shown in
 Appendix B.

A.4 Determination of calcium citrate content (Ca, dry basis) A.4.1 Methodological Summary

in the test solution, a small amount of magnesium ions as the auxiliary indicator, that is, magnesium ions and chromium black T generated a stable complex indicates the color change, Titration with ethylenediamine tetraacetic acid disodium (EDTA) standard titration solution, according to ethylenediamine tetraacetic acid disodium (EDTA) standard titration solution consumption Calculate the calcium content.

A.4.2 Reagents and materials A.4.2.1 hydrochloric acid solution (14, volume ratio).

A.4.2.2 Ammonia-ammonium chloride buffer solution (pH ~ 10).

A.4.2.3 zinc oxide reference reagent (purity not less than 99.9%).

A.4.2.4 Magnesium Sulfate Solution (120 g/L).

A.4.2.5 Chromium Black T (5 g/L).

A.4.2.6 Disodium ethylenediamine tetraacetate (EDTA) standard titration solution. c (EDTA) ~0.05mol/L.

Ethylenediamine tetraacetic acid disodium standard titration solution preparation. Weigh 20g EDTA, dissolved in water, diluted with water to 1000mL, shake.

Calibration. accurately weighed 0.15g by 800 °C ± 50 °C in the high temperature furnace to the constant weight of the zinc oxide reference reagent (A.4.2.3), with less the amount of water wetting, add 2mL hydrochloric acid solution (20%) dissolved, add 100mL water, with ammonia solution (10%) to adjust the solution pH to 7 ~ 8, plus 10 mL ammonia-ammonium chloride buffer solution (pH ~ 10) and 5 drops of chromium black T solution, titrated with a solution of EDTA solution to the solution by purple for pure blue. At the same time as a blank test.

EDTA standard titration solution concentration, the unit is mol per liter (mol/L), according to formula (A.1) calculation.

c (EDTA) =

$m \times 1000$

$(V_1 - V) \times M$

Where.

m - the mass of the zinc oxide reference reagent in grams (g);

V₁ - consumption of EDTA standard solution in milliliters (mL);

V - blank consumption of EDTA standard solution in milliliters (mL);

M - the molar mass of zinc oxide in grams per mole (g/mol) [M (ZnO) = 81.39 g/mol];

1000 --- conversion factor.

A.4.2.7 Ammonia solution (10%).

A.4.3 Analysis steps 2 drops of magnesium sulfate solution, add ammonia - ammonium chloride buffer 15mL, plus chromium black T indicator solution 2 drops, with EDTA standard solution titration to Pure blue; another dry sample after about 0.35g, accurate to 0.0001g, add water 30mL to dissolve, add triethanolamine 5 drops, and with the above titration of magnesium sulfate solution, and then EDTA standard solution titration, to the color from purple to pure blue is the end point. with Do blank test.

A.4.4 Calculation of results Calcium citrate content (Ca, dry basis) of the mass fraction w_1 , calculated according to formula (A.2).

$$(V-V_0) \times c \times M$$

$$1000 \times m \times$$

$$100\% \text{ (A.2)}$$

Where.

V --- sample consumption EDTA standard solution titration volume, in milliliters (mL);

V_0 --- blank test consumption EDTA standard solution titration volume, in milliliters (mL);

c - the concentration of EDTA standard solution in moles per liter (mol/L);

M - the molar mass of calcium in grams per mole (g/mol) [$M(\text{Ca}) = 40.1 \text{ g/mol}$];

m --- sample quality, in grams (g);

1000 --- conversion factor.

The results of the test are based on the arithmetic mean of the parallel measurement results (1 decimal place is reserved). Two independent tests obtained under repetitive conditions the absolute difference of the result is not more than 5% of the arithmetic mean.

A.5 Determination of pH Take 10.0g sample, add water 100mL stirring dissolved, to be clarified, and then according to the provisions of GB/T 9724 measured sample solution pH.

A.6 Determination of dry weight loss A.6.1 Reagents and materials A.6.1.1 weighing bottle. diameter 40mm x 25mm.

A.6.1.2 Analyze the balance.

A.6.1.3 Dryers.

A.6.1.4 Oven.

A.6.2 Analysis steps Weigh the sample 1g ~ 2g, accurate to 0.0001g, placed in the 150 °C ± 5 °C drying oven to the already constant weight of the weighing bottle in. Such as the sample for the larger crystal or block, should be quickly crushed to the diameter of 2mm