



Voluntary Report - Voluntary - Public Distribution

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# **Report Name:** China Notifies Draft National Food Safety Standard for the Food Nutritional Fortifier Manganese Carbonate - SPS 1156

Country: China - Peoples Republic of

Post: Beijing

Report Category: FAIRS Subject Report

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#### **Report Highlights:**

On May 12, 2020, China notified the National Food Safety Standard for the Food Nutritional Fortifier Manganese Carbonate (Draft for Comments) to the WTO SPS Committee as G/SPS/N/CHN/1156. The comment deadline is July 11, 2020. Comments can be sent to China's SPS Enquiry Point at sps@customs.gov.cn. This standard is applicable to manganese carbonate, a food nutritional fortifier prepared by chemical synthesis using manganese sulfate and ammonium bicarbonate as raw materials. China has not announced a proposed date of entry into force of the standard. This report contains an unofficial translation of the draft standard.

THIS REPORT CONTAINS ASSESSMENTS OF COMMODITY AND TRADE ISSUES MADE BY USDA STAFF AND NOT NECESSARILY STATEMENTS OF OFFICIAL U.S. GOVERNMENT POLICY

#### **General Information**

#### **BEGIN TRANSLATION**

### National Food Safety Standard Food Nutritional Fortifier - Manganese Carbonate

#### 1. Scope

This standard applies to manganese carbonate, a food nutritional fortifier prepared by chemical synthesis using manganese sulfate and ammonium bicarbonate as raw materials.

#### 2. Molecular formula, relative molecular mass

#### 2.1 Molecular formula

MnCO<sub>3</sub>

#### 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 provisions of Table 1.

#### **Table 1 Sensory Requirements**

Item	Requirements	Testing Method		
Color	Light red to light brown.	Take proper amount of sample and place it in a clean, dry		
Status	Powder	white porcelain dish. Observe its color and condition under natural light.		

#### **3.2 Physical and Chemical Indexes**

Physical and chemical indexes shall conform to provisions of Table 2.

#### **Table 2 Physical and Chemical Indexes**

Items			Testing Method
Manganese carbonate content (calculated in MnCO3), w $/\% \geq$		90.0	A.4 in appendix A
Chloride (in Cl), w /%	$\leq$	0.01	A.5 in appendix A
Sulfate (in SO4), w /%	$\leq$	0.5	A.6 in appendix A
Total arsenic (in As)/(mg/kg)	$\leq$	3.0	GB 5009.11 or GB 5009.76
Lead (Pb)/(mg/kg)	$\leq$	2.0	GB 5009.12 or GB 5009.75

#### **Appendix A**

#### **Testing Method**

#### A.1 Operation tips

Some of the reagents used in testing method are toxic or corrosive. Please use caution and follow relevant regulations when using them. Please immediately wash the reagent splashed on skin with water; serious cases should be treated immediately. When using volatile acids, the tests shall be performed in a fume hood.

#### A.2 General provisions

Unless otherwise specified, the purity of the reagents used should be above analytical purity. The standard titration solution, the standard solution for impurity determination, the preparations and products should be prepared in accordance with GB/T 601, GB/T 602, GB/T 603. Water used in experiments shall comply with the provisions of tertiary water in GB/T 6682. When the solvent in the test is not specified, it refers to aqueous solution.

#### A.3 Identification test

#### A.3.1 Reagents and solutions

A.3.1.1Hydrochloric acid.

A.3.1.2Acetic acid.

A.3.1.3Nitric acid.

A.3.1.4Hydrochloric acid solution: Take 50.0 mL of hydrochloric acid (A.3.1.1), dilute with water to 100 mL, and shake well.

A.3.1.5Nitric acid solution: Take 25.0 mL of nitric acid (A.3.1.3), dilute with water to 100 mL, and shake well.

A.3.1.6Ammonium sulfide.

A.3.1.7Sodium bismuthate.

A.3.1.8Saturated solution of calcium hydroxide: Weigh about 3 g of calcium oxide with an accuracy to 0.1 g, put it in the reagent bottle, and add 1000 mL of water; cover the stopper, shake vigorously, and set aside to let the liquid settle; take the clear liquid on the top of the reagent bottle for test.

#### A.3.2 Identification method

A.3.2.1 Manganese identification

A.3.2.1.1 Weigh 1g of sample, add hydrochloric acid solution dropwise until the sample is completely dissolved, and then continue to add hydrochloric acid solution to produce 20mL test sample solution; add ammonium sulfide dropwise to produce an orange-red precipitate, and after rest for 10 minutes, add glacial acetic acid dropwise to dissolve the precipitate gradually.

A.3.2.1.2 Weigh 1g sample, dissolve in nitric acid solution, and add a small amount of sodium bismuthate powder to make it purple.

#### A.3.2.2Carbonate identification

Weigh 1g sample, add 100mL water, and add hydrochloric acid solution dropwise into the test solution to produce colorless and odorless gas; pass the gas into the saturated solution of calcium hydroxide, the solution will become a white turbid liquid, and it will become clear when the gas is continuously passed into.

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

#### A.4.1 Principle

Perchloric acid is added to the sample in phosphoric acid medium to oxidize the manganese to trivalent. With N-phenylanthranilic acid as indicator, titrate with standard ferrous ammonium sulfate titration solution, the content of manganese carbonate is calculated according to the difference of consumption of standard titration solution of ammonium ferrous sulfate by the titration sample and in the blank test.

#### A.4.2 Reagents and materials

A.4.2.1Phosphoric acid.

A.4.2.2Nitric acid.

A.4.2.3Hydrochloric acid.

A.4.2.4Hydrochloric acid.

A.4.2.5Sulfuric acid: 1 + 1 (V + V).

A.4.2.6Sulfuric acid: 5 +95 (V + V).

A.4.2.7Sulfuric acid: 1 + 4 (V + V).

A.4.2.8Standard solution of potassium dichromate[  $c(\frac{1}{6}K_2Cr_2O_7)=0.040\ 00\ mol/L$ ]:Weigh 1.9615g standard potassium dichromate (dry in advance at 150°C for 2h and cool in a desiccator) to room temperature), place in a 250mL beaker, and add an appropriate amount of water to dissolve; transfer to a 1000mL volumetric flask, dilute to the mark with water, and mix well.

A.4.2.9Standard titration solution of ferrous ammonium sulfate[ $c(NH_4)_2$  Fe(SO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O $\approx$ 0.04 mol/L], calibrate before each use.

A.4.2.9.1 Preparation

Weigh 15.68g ferrous ammonium sulfate and dissolve in 1000mL sulfuric acid (A.4.2.6) and mix well.

A.4.2.9.2 Calibration

Pipette 25.00mL standard solution of potassium dichromate into a 250mL Erlenmeyer flask, add 40mL sulfuric acid (A.4.2.7), 5mL phosphoric acid, and titrate with standard ferrous ammonium sulfate titration solution until the orange color disappears; add 2 drops of N-benzoanthranilic acid indicator,

continue to titrate carefully until the solution just turns green, when it is the end point.

Calculate the titer T1 of standard ferrous ammonium sulfate titration solution to manganese carbonate according to formula (A.1), with the unit of grams per milliliter (g/mL);

In which:

 $T_{I}$ . Calculate the titer of standard ferrous ammonium sulfate titration solution to manganese carbonate, with the unit of grams per milliliter (g/mL);

C - The concentration of standard solution of potassium dichromate, with the unit of mole per liter (mol/L);

 $V_{1}$  \_ 25.00, volume of standard solution of potassium dichromate transferred, with the unit of milliliter (mL);

 $m_1$ . 114.95, molar mass of manganese carbonate, with the unit of grams per mole (g/mol);

 $V_2$ . Consumption volume of standard titration solution of ammonium ferrous sulfate in titration, with the unit of milliliter (mL);

 $V_3$  . Consumption volume of standard titration solution of ammonium ferrous sulfate in blank titration, with the unit of milliliter (mL);

 $10^{-3}$  - Calculation factor.

A.4.2.9.3 Determination of blank value

Pipette 10.00mL potassium dichromate standard solution into a 250mL Erlenmeyer flask, add 40mL sulfuric acid (A.4.2.7) and 5mL phosphoric acid, and titrate with the standard titration solution of ammonium ferrous sulfate until the orange color disappears. Add 2 drops of N-benzoanthranilic acid indicator, continue to titrate carefully until the solution just turns green as the end point, and record the volume V4. Transfer 10.00mL potassium dichromate standard solution to the above conical flask, titrate with the standard titration solution of ammonium ferrous sulfate to the end point and record the volume V5. The difference between the volumes of ammonium ferrous sulfate of the two times of titrations respectively (namely, V5-V4) is the blank value V3.

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

#### A.4.3 Analysis steps

#### A.4.3.1 Analysis frequency

Repeat the analysis for the same sample at least twice.

#### A.4.3.2Sample volume

Weigh 0.20g of dry sample (accurate to 0.0001 g).

#### A.4.3.3Blank test

Carry out a blank test with the same batch of sample, and the titration shall be according to

A.4.2.9.3; No sulfuric acid and phosphoric acid will be added, and thus V6 is recorded.

#### A.4.3.4Determination

Weigh 0.20g (accurate to 0.0001g) of the sample, place in a 250mL Erlenmeyer flask, and moisten the sample with a small amount of water; shake carefully to disperse, add 5mL hydrochloric acid, and shake well; add 20mL phosphoric acid, and shake well. Then heat to dissolve until it is slightly boiling, and add 5mL of nitric acid while it is hot; shake while adding, shake the conical flask well after completion of addition, and heat until it smokes slightly (liquid surface is calm); remove, add 2mL Perchloric acid, and shake while adding; heat until the liquid level of the solution is calm to complete the oxidation of the divalent manganese, and remove.

Cool the above solution to 70°C, add 50mL of water, and shake while adding. After the addition is complete, shake well to dissolve the salts and cool to room temperature with running water. Titrate with a standard titration solution of ammonium ferrous sulfate until it is light red, and add 2 drops of N-benzoanthranilic acid indicator solution; continue to titrate until it is bright yellow, which means the end point, and record the volume V7.

#### A.4.4 Calculation of results

Calculate the manganese carbonate content (mass fraction) w in the sample according to formula (A.2), in%:

$$w = \frac{(V_7 - V_6) \times T_1}{m_2} \times 100 \quad (A.2)$$

In which:

 $T_1$ —the titer of standard ferrous ammonium sulfate titration solution to manganese carbonate, with the unit of grams per milliliter (g/mL);

 $V_7$ —the volume of standard titration solution of ammonium ferrous sulfate consumed in titration, with the unit of milliliter (mL);

 $V_6$ —the volume of standard titration solution of ammonium ferrous sulfate consumed in blank titration, with the unit of milliliter (mL);

 $m_2$ —the amount of sample, with the unit of gram (g); 100—calculation factor.

The arithmetic mean of the parallel determination results is taken as the measurement result, and the absolute difference between the two parallel determination results shall be not more than 0.30%.

#### A.5 Determination of chloride content (visual turbidimetry)

#### A.5.1 Method summary

Add nitric acid to dissolve the sample, and add silver nitrate solution to the acidic medium. Compare the white silver chloride suspension produced from the ions of chloride and silver with the standard turbidity solution produced similarly at the same time.

A.5.2 Reagents and solutions

A.5.2.130% hydrogen peroxide solution.

A.5.2.2Nitric acid.

A.5.2.3Nitric acid solution: 1 + 3 (V + V).

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

A.5.2.5Standard chloride solution: 1mL solution contains 0.01 mg of chlorine (C1-), which is prepared according to the requirements of GB/T 9729-2007.

#### A.5.3 Analysis steps

Weigh 1.00g sample with an accuracy to 0.01g, place in a beaker, and moisten with a small amount of water; add nitric acid solution dropwise until the sample dissolves, add 1 drop of hydrogen peroxide to fade the dark color, and transfer to a 100mL volumetric flask; Dilute to the mark and shake well (filter if necessary). Pipette 10mL test solution into a 25mL colorimetric tube, add 1mL nitric acid solution and 1mL silver nitrate solution, and dilute to the mark with water; shake well, set aside for 2 min, and compare with standard turbidity solution on black background.

Standard turbidity solution: Pipette 1mL standard chloride solution, place it in a 25mL colorimetric tube, and add water to 10mL; start the same treatment as the sample from "add 1mL nitric acid ..."at the same time.

The turbidity of the sample solution shall not be higher than the standard turbidity solution, otherwise it is regarded as unqualified.

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

#### A.6.1 Method summary

Add hydrochloric acid solution 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 in suspension within a certain period of time, making the solution turbid. Add sulfate-ethanol solution as a seed solution to make the particle size appropriate and uniform, so it can be used for the visual turbidimetric determination of sulfate.

#### A.6.2 Reagents and materials

A.6.2.130% hydrogen peroxide solution.

A.6.2.2Hydrochloric acid.

A.6.2.3 Hydrochloric acid solution: 1+1(V+V).

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

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

A.6.2.5Barium chloride solution: 250 g/L

A.6.2.6Standard sulfate solution: 1mL solution contains 0.10 mg sulfate( $SO_4^{2-}$ ).

#### A.6.3 Analysis steps

Weigh 1.00 g sample with an accuracy to 0.01g, place in a 100mL beaker, and add 10mL water;

add hydrochloric acid solution dropwise until the sample is completely dissolved, and add 30% hydrogen peroxide to fade the dark color. Heat and boil for 2 min, cool at room temperature, and transfer to a 100mL volumetric flask; dilute to the mark with water, and shake well. Transfer 2.0mL of test solution to a 50mL beaker with a pipette, add water to 25mL, and add 0.5mL of hydrochloric acid solution. Standard turbidity solution: pipette 1.0mL of standard sulfate solution into a beaker, add water to 25mL, and add 0.5mL of hydrochloric acid solution.

Add 0.25mL of sulfate-ethanol solution and 1mL of barium chloride solution to two colorimetric tubes, and place them accurately for 1min. Pour the test solution and the standard solution in the beaker into the colorimetric tubes and shake for 10 min.

The turbidity of the sample solution shall not be higher than the standard turbidity solution, otherwise it is regarded as unqualified.

END TRANSLATION

#### Attachments:

No Attachments.