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

GB 29212-2012

Carbonyl iron powder food additives

食品添加剂 羰基铁粉

(English Translation)

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1 Scope

This standard applies to the sponge iron as raw material, and carbon monoxide reaction synthesis of pentacarbonyl iron, pentacarbonyl iron by thermal decomposition of food additives Carbonyl iron powder.

2.1 Chemical name

Carbonyl iron

2.3 Relative molecular mass

55.85 (according to the 2007 International Relative Atomic Quality)

3.1 Sensory requirements

Shall comply with the provisions of Table 1.

Table 1 sensory requirements

The project requires a test method Color black and gray State powder Take appropriate sample in a 50 mL beaker and observe the color and state under natural light

3.2 Physical and chemical indicators

Shall comply with the provisions of Table 2.

Table 2 Physical and chemical indicators

Item Index Test Method Iron (Fe) content, w /% ≥ 98.0 Appendix A A.4 Arsenic (As)/(mg/kg) ≤ 3 Appendix A A.5 Lead (Pb)/(mg/kg) ≤ 4 Appendix A A.6

Table 2 (continued)

Item Index Test Method Mercury (Hg)/(mg/kg) ≤ 2 Appendix A A.7 Acid insoluble matter, w /% ≤ 0.2 Appendix A A.8 Fineness, w /% Through a 75 μm standard sieve 100 Through the 45 μm standard screen ≥ 95 Appendix A, A.9 Appendix A Testing method A.1 Warning Some of the reagents used in the test methods of this standard are toxic or corrosive and should be operated with appropriate safety and protective measures.

A.2 General provisions the reagents and water used in this standard, when not specified in other requirements, refers to the analysis of pure reagents and GB/T 6682-2008 of the three water.

Standard titration solution used in the test, the standard solution for the determination of impurities, preparations and products, in the absence of other requirements are marked by GB/T 601, GB/T 602, GB/T 603. The solution used refers to an aqueous solution when it is not specified with any solvent formulation.

A.3 Identification test A.3.1 Reagents and materials A.3.1.1 hydrochloric acid solution. 2 8.

A.3.1.2 Potassium ferricyanide solution. 100 g/L.

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

A.3.2 Identification method A small amount of the sample dissolved in hydrochloric acid solution, the hydrogen escape, stop after adding potassium ferricyanide solution, resulting in insoluble hydrochloric acid solution of black blue Color precipitation. This precipitate can be decomposed by sodium hydroxide solution to produce green and white precipitate, the color quickly turned into green, after the oscillation into brown.

A.4 Determination of iron (Fe) content A.4.1 Methodological Summary After the sample was dissolved in the air under the conditions of acid, with 1,10-phenanthroline - ferrous iron as indicator, with cerium sulfate standard titration solution titration, the solution changed from red to light blue, and the iron content was calculated based on the amount of cerium sulfate standard solution consumed.

A.4.2 Reagents and materials A.4.2.1 Sulfuric acid solution. 3 100.

A.4.2.2 Cerium sulfate standard titration solution. $c [\text{Ce} (\text{SO}_4)_2] = 0.1 \text{ mol/L}$.

A.4.2.3 1,10-phenanthroline-ferrous indicator liquid.

A.4.3 Analysis steps Weigh about 0.2g sample, accurate to 0.0002g, placed in 250mL Erlenmeyer flask, add 50mL sulfuric acid solution, covered with the original valve Cork (the production method is to insert a plug in the plug with a short piece of rubber tube with a glass tube, the side of the rubber tube has a long cut Mouth, the other end of the insertion of a glass rod, so that the gas can escape, and the air can not enter). Heat the conical flask on a steam bath to dissolve the sample Solution, cooling, add 50mL boiling and cooling water dilution, add 2 drops of 1,10-phenanthroline-ferrous indicator solution, with cerium

sulfate standard titration solution drops Set to red to light blue, that is the end point.

While the blank test. The blank sample solution is the same as the sample solution except that no sample is added.

A.4.4 Calculation of results the mass fraction w_1 of iron (Fe) is calculated according to formula (A.1) $w_1 = (V_1 - V_0) c_M m \times 1000 \times 100\%$ (A.1) where.

V_1 --- titration sample solution consumed by the volume of cerium sulfate standard titration solution in milliliters (mL);

V_0 --- titration blank sample solution consumed by the volume of cerium sulfate standard titration solution in milliliters (mL);

c - the exact value of the concentration of cerium sulfate standard titration solution in moles per liter (mol/L);

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

M - the molar mass of iron (Fe) in grams per mole (g/mol) ($M = 55.85$);

1000 --- conversion factor.

The experimental results are based on the arithmetic mean of the parallel measurement results, and the absolute difference between the two independent determinations obtained under the repeatability condition the value is not greater than 0.3%.

A.5 Determination of arsenic (As) Weigh $1.00\text{g} \pm 0.01\text{g}$ sample, placed in a conical flask, add 25mL sulfuric acid solution (3 100), the steam bath heated to the hydrogen stop Only escape. Cooling, add 4mL hydrochloric acid, the following operation in accordance with the provisions of GB/T 5009.76 method for determination.

A.6 Determination of lead (Pb) A.6.1 Reagents and materials A.6.1.1 Hydrochloric acid.

A.6.1.2 Nitric acid.

A.6.1.3 Methyl isobutyl ketone.

A.6.1.4 Hydrochloric acid solution. 4 5.

A.6.1.5 Nitric acid solution. 1 1.

A.6.1.6 ascorbic acid-sodium iodide solution. 20 g of ascorbic acid and 38.5 g of sodium iodide were weighed and dissolved in water and transferred to a capacity of 200 mL Bottle, diluted with water to the mark, shake.

A.6.1.7 Tri-n-octylphosphine oxide solution. Weigh 5.0 g of tri-n-octylphosphine oxide into a 100 mL volumetric flask and use methyl isobutyl ketone Dissolved and diluted to the mark, shake.

WARNING. Tri-n-octylphosphine oxide solution is irritating and avoid contact with eyes, skin

and clothing.

A.6.1.8 Lead (Pb) standard solution. 0.002 mg/mL.

A.6.1.9 Water. in line with GB/T 6682-2008 in the provisions of secondary water.

A.6.2 Instruments and equipment Atomic Absorption Spectrophotometer. With lead hollow cathode lamp.

A.6.3 Analysis steps A.6.3.1 Cleaning of glass instruments all the preparation of the glass container should be soaked in nitric acid solution for 30min, and rinse with tap water, and finally rinse with water clean.

A.6.3.2 Preparation of sample solution A sample of $1.00 \text{ g} \pm 0.01 \text{ g}$ was weighed, placed in a 50 mL beaker, covered with a surface dish, slowly added 8 mL of hydrochloric acid, 2 mL of nitric acid After the reaction is calm, evaporate on the steam bath to dry and cool. Add 10mL hydrochloric acid solution to dissolve the residue, if necessary, may be appropriate heating. Dissolve After which it was washed with 10 mL of water and transferred to a 125 mL separatory funnel, 20 mL of ascorbate-sodium iodide solution, 5 mL of tri-n-octyloxy Phosphine solution, shaking 30s, standing stratification, discard the water phase, the organic phase into the 10mL with a scale tube, spare.

A.6.3.3 Preparation of standard reference solution Take 2mL lead standard solution in 50mL beaker, cover the surface dish, slowly add 8mL hydrochloric acid, 2mL nitric acid, in the steam bath Evaporated to dry and cool. Add 10mL hydrochloric acid solution to dissolve the residue, rinse with 10mL water and transfer to 125mL separatory funnel, add 20mL ascorbic acid-sodium iodide solution, 5mL tri-n-octylphosphine oxide solution, shaking 30s, standing stratified, discarded the water phase, the organic phase into 10mL with a plug scale tube, spare.

A.6.4 Determination

At the wavelength of 283.3nm, the use of air - acetylene flame, the atomic absorption spectrophotometer transferred to the best working state to methyl isobutyl Methyl ketone zero, respectively, the standard reference solution, the sample solution absorbance, sample solution absorbance should not be greater than the standard reference solution Absorbance.

A.7 Determination of mercury (Hg) Weigh $2.00 \text{ g} \pm 0.01 \text{ g}$ sample, placed in 250mL beaker, add 20mL nitric acid solution (1 2), in the steam bath digestion 45min. Add 5 mL of hydrochloric acid solution (1 3) and continue heating on the steam bath until the sample is completely dissolved. Cool to room temperature, filter, other exercises the method was carried out according to the method specified in GB/T 5009.17.

A.8 Determination of acid insoluble matter A.8.1 Reagents and materials A.8.1.1 Sulfuric acid solution. 3 100.