



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

GB 1886.251-2016

**National Food Safety Standard -- Food Additives -- Ferroferric
Oxide**

食品安全国家标准 食品添加剂 氧化铁黑

(English Translation)

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1 Scope

This standard applies to ferrous salt as raw material, the air oxidation method of food additives made of iron oxide black.

2.1 Chemical name

Iron oxide

2.2 Molecular formula

Fe₃O₄

2.3 Relative molecular mass

231.55 (according to 2011 international relative atomic mass)

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 black State powder Take the appropriate amount of sample evenly placed in the white enamel plate, in the natural light Under the observation of its 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 Iron oxide black content (in terms of Fe), w /% ≥ 68 Appendix A
A.3 Water soluble, w /% ≤ 1.0 Appendix A A.4 Total arsenic (in As)/(mg/kg) ≤ 3.0 GB
5009.11 silver salt method a

Table 2 (continued)

Item Index Test Method Lead (Pb)/(mg/kg) ≤ 10.0 Appendix A A.5 Mercury (Hg)/(mg/kg)
 ≤ 1.0 Appendix A A.6 Cadmium (Cd)/(mg/kg) ≤ 1.0 Appendix A A.7 a sample of 0.5g,
plus nitric acid - perchloric acid mixture for the 20mL.

Appendix A

Testing method

A.1 General provisions in addition to the provisions of this standard, the purity of the reagents used are analytical pure, the standard titration solution used, the standard solution for the determination of impurities, preparation and products should be prepared according to GB/T 601, GB/T 602, GB/T 603, test water should be consistent with GB/T 6682 in the three water regulations set. The solution used in the test refers to the aqueous solution when it is not specified in the formulation of the solvent.

A.2 Identification test A.2.1 Reagents and solutions A.2.1.1 hydrochloric acid solution. 1 9.

A.2.1.2 ammonium thiocyanate solution. Weigh 8g ammonium thiocyanate, accurate to 0.01g, dissolved in water and set to 100mL.

A.2.2 Identification method Weigh 0.1g sample, accurate to 0.01g, add hydrochloric acid solution 5mL, boil, cool, add ammonium thiocyanate solution, should show blood red.

A.3 Determination of iron oxide black content (in terms of Fe) A.3.1 Reagents and materials

A.3.1.1 30% Hydrogen Peroxide.

A.3.1.2 Potassium iodide.

A.3.1.3 Hydrochloric acid solution. 9 20.

A.3.1.4 starch indicator solution. take 0.5g soluble starch, add water 5mL stir well, slowly poured into 100mL boiling water, while stirring while stirring, Continue to boil for 2min, let cool, pour the supernatant, that is, too. The liquid should be temporary with the new system.

A.3.1.5 sodium thiosulfate standard titration solution. $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.1\text{mol/L}$.

A.3.2 Analysis steps Weigh 0.2g sample, accurate to 0.0001g, placed in 10mL hydrochloric acid solution has been added to the.200mL Erlenmeyer flask, carefully heated to So that the sample is completely dissolved. After cooling, add 6 drops to 7 drops of hydrogen peroxide, continue to boil for about 3min, cool, add 30mL of water and 2g of iodine Potassium, standing for 5min. Add 30mL of water, add 2.5mL starch indicator solution,

sodium thiosulfate standard titration solution titrated to blue Missed the end. At the same time do blank test.

A.3.3 Calculation of results the mass fraction w_1 of the iron oxide black content (in terms of Fe) is calculated according to the formula (A.1) $w_1 = \frac{V_1 - V_0}{m} \times c \times M \times 1000 \times 100\%$ (A.1) where.

V_1 --- sample solution consumption of sodium thiosulfate standard titration solution volume, in milliliters (mL);

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

c - the actual concentration of sodium thiosulfate standard titration solution in moles per liter (mol/L);

M - the molar mass of iron in grams per mole (g/mol) [$M(\text{Fe}) = 55.85$];

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

1000 --- volume conversion factor.

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

A.4 Determination of water-soluble substances A.4.1 Analysis steps Weigh 5g sample, accurate to 0.01g, placed in 400mL beaker, add.200mL water to boil 5min, stirring, dissolve. After cooling Transferred to a 250 mL volumetric flask, diluted with water to the mark, allowed to stand for 10 min, filtered. Remove the 100 mL filtrate from dry to constant weight Evaporation of the pan, in the boiling water bath carefully dry. The evaporated residue was dried at 105 ° C to 110 ° C for 2 h, cooled in a desiccator to room temperature, Weighing.

A.4.2 Result calculation the mass fraction of water-soluble matter w_2 is calculated according to formula (A.2) $w_2 = \frac{m_2}{m_1} \times 2.5 \times 100\%$ (A.2) where.

m_2 --- the mass of the residue in grams (g);

2.5 --- volume conversion factor;

m_1 --- the quality of the sample, in grams (g).

The results of the test are based on the arithmetic mean of the parallel measurement results. The absolute difference between the two independent determinations obtained under repeatability conditions the value is not more than 0.05%. The result is reserved for two digits after the decimal point.

A.5 Determination of lead (Pb) A.5.1 Reagents and materials A.5.1.1 4-methyl-2-pentanone

(MIBK).

A.5.1.2 Hydrogen peroxide.

A.5.1.3 Hydrochloric acid solution. 1 10.

A.5.1.4 lead standard solution. according to GB/T 602 preparation and calibration, and then according to the requirements of the instrument used to dilute the preparation of lead were 0.5µg/mL, 1µg/mL, 1.5µg/mL three concentrations of standard solution.

A.5.2 Instruments and equipment Atomic Absorption Spectrometer.

A.5.3 Analysis steps Weigh about 2g sample, accurate to 0.01g, placed in 100mL beaker, add 15mL hydrochloric acid solution, heated to the hot plate After dissolving, cooling, add 2 mL of hydrogen peroxide, continue to heat to micro-boiling and hold for 3 min. After cooling, add 25 mL of 4-methyl-2-pentanone and 10 mL of hydrochloric acid solution, the solution was transferred to a 125 mL separatory funnel; shaken for 2 min, allowed to stand for 30 s, the lower aqueous phase into the beaker, Heat to the solution concentrated to about 5mL ~ 10mL, the residual solution is filtered, washed with water residue, combined with the filtrate after the volume to 100mL, prepared use. The blank solution was prepared in the same manner.

The standard solution of lead was prepared according to A.5.1.4 and determined according to GB 5009.12 flame atomic absorption spectrometry.

The results of the test are based on the arithmetic mean of the parallel measurement results. The absolute difference between the two independent determinations obtained under repeatability conditions Value of not more than 1.0mg/kg.

A.6 Determination of mercury (Hg) A.6.1 Reagents and materials A.6.1.1 Sodium hydroxide solution. 1 g/L.

A.6.1.2 sodium borohydride solution. 8g/L (solvent 1g/L sodium hydroxide solution).

A.6.1.3 Mercury standard solution. Prepared and calibrated according to GB/T 602, and then diluted according to the requirements of the instrument used to prepare mercury 0.001µg/mL, 0.002µg/mL, 0.003µg/mL three concentrations of standard solution.

A.6.2 Instruments and equipment Atomic Absorption Spectrometer.

A.6.3 Atomic Absorption Spectrometer Reference Conditions A.6.3.1 Mercury Hollow Cathode Lamp Wavelength. 253.7 nm.

A.6.3.2 Carrier gas. argon.

A.6.3.3 Carrier gas flow rate..200 mL/min.

A.6.3.4 Atomizer temperature. room temperature.

A.6.4 Analysis steps According to A.5.3 preparation of sample solution and blank solution,