

national standards of People's Republic of China

GB 6781—201X

National food safety standards

Food nutrition enhancer ferrous lactate

(draft for comments)

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Foreword

This standard replaces the "food additive ferrous lactate" (GB 6781-2007).

Compared with the "food additive ferrous lactate" (GB 6781-2007), the main changes are as follows:

- modified the standard name;
- increased ferrous lactate dihydrate;
- modified the sensory requirements;
- Delete the weight loss of drying and its detection method;
- Increased moisture and its detection methods.

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Food nutrition enhancer ferrous lactate

1 Scope

This standard applies to sodium lactate and ferrous sulfate, or sodium lactate and ferrous chloride, or calcium lactate (ammonium lactate) and ferrous ferrous lactate produced by the reaction of ferrous iron or lactic acid and iron powder.

2 Chemical name, molecular formula, structural formula and relative molecular mass

2.1 Chemical Name

Ferrous bishydroxypropionate

2.2 Molecular formula

$C_6H_{10}FeO_6 \cdot nH_2O$ ($n = 2$ or 3)

2.3 Structural formula

2.4 Relative molecular mass

270.02 (dihydrate) (according to 2016 international relative atomic mass)

288.03 (trihydrate) (according to 2016 international relative atomic mass)

3 Technical requirements

3.1 Sensory requirements

Sensory requirements should be in accordance with Table 1.

Table 1 Sensory requirements

item	Head	Want begging	Testing method
Color	White green or yellowish green		Take an appropriate amount of sample in a clean, dry white porcelain dish under natural light. Observe its color, state, smell its smell.
State	Crystal or powder		
Gas smell	Slight characteristic smell		

3.2 Physical and chemical indicators

Physical and chemical indicators should meet the requirements of Table 2.

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Table 2 Physical and chemical indicators

item	Head	MeansStandard	Testing method
Ferrous lactate ($C_6H_{10}FeO_6$) content	content \geq	96.0	Appendix A, A.3
Base), w / %			
Moisture, w / %	\leq	20.0	Appendix A, A.4
pH (20 g / L aqueous solution)		5.0~6.0	GB/T 9724
Ferric iron (in terms of Fe^{3+}), w / %	\leq	0.6	Appendix A, A.5
Chloride (in terms of Cl), w / %	\leq	0.1	Appendix A, A.6
Sulfate (as SO_4), w / %	\leq	0.1	Appendix A, A.7
Lead (Pb) / (mg / kg)	\leq	1.0	GB 5009.12
Total arsenic (as As) / (mg/kg)	\leq	3.0	GB 5009.11

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Appendix A

Testing method

A.1 General provisions

The reagents and water used in this standard refer to the analytical reagents and the tertiary water specified in GB/T 6682 when no other requirements are specified. The standard titration solution used in the test, the standard solution for the determination of impurities, the preparation and its products are all in accordance with GB/T 601, GB/T 602, GB/T 603. The solution used in the test refers to an aqueous solution when it is not indicated which solvent is used.

A.2 Identification test

A.2.1 Reagents and materials

A.2.1.1 Ethanol.

A.2.1.2 Potassium permanganate solution: 3.2 g/L.

A.2.1.3 Sulfuric acid solution: 1+20.

A.2.1.4 Potassium ferricyanide solution: 100 g/L.

A.2.1.5 Hydrochloric acid solution: 1+3.

A.2.1.6 Morpholine solution: 1+4.

A.2.1.7 Sodium nitroferrocyanide solution: 50 g/L.

A.2.1.8 Sodium hydroxide solution: 43 g/L.

A.2.2 Identification method

A.2.2.1 Solubility

Slightly soluble in water, almost insoluble in ethanol.

A.2.2.2 Identification of lactate

Approximately 1.0 g of the sample was weighed and dissolved in 50 mL of water to prepare a sample solution. Take 5 mL of sample solution and mix with 2 mL of sulfuric acid solution. Evenly, add 2 mL of potassium permanganate solution, heat, and acetaldehyde gas should be produced. Acetaldehyde gas is identified using an equal volume of morpholine solution and the mixed solution of the sodium nitroferrocyanide solution is wetted, and the filter paper is blue in contact with the gas.

A.2.2.3 Identification of ferrous salts

Take 10 mL of the sample solution in A.2.2.2, add 2 mL of potassium ferricyanide solution, should form a dark blue precipitate, add hydrochloric acid. The lake is not dissolved; if a sodium hydroxide solution is added, the precipitate should be dissolved. The sample solution is mixed with the sodium hydroxide solution to produce a slightly greenish white precipitate. It turns green quickly and should be brown after shaking.

A.3 Determination of ferrous lactate ($C_6H_{10}FeO_6$) content (on a dry basis)

A.3.1 Reagents and materials

A.3.1.1 Phosphoric acid.

- A.3.1.2 Sulfuric acid solution: 1+15.
- A.3.1.3 Barium sulfate standard titration solution: $c(\text{CeSO}_4) = 0.1 \text{ mol / L}$.
- A.3.1.4 1,10-phenanthroline-ferrous indicator liquid.
- A.3.2 Analysis steps

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Weigh 0.8 g sample, accurate to 0.0001 g, placed in a 300 mL flask, add 160 mL sulfuric acid solution and 5 mL phosphoric acid, necessary Cool to room temperature. Add 1 drop of 1,10-phenanthroline-ferrous indicator solution and immediately titrate to red with 0.1 mol/L barium sulfate standard titration solution Disappeared as the end point. A blank test was also performed.

A.3.4 Calculation of results

The mass fraction w_1 of the ferrous lactate ($\text{C}_6\text{H}_{10}\text{FeO}_6$) content (on a dry basis) is calculated according to the formula (A.1):

$$w_1 = \frac{(V_1 - V_0) \times c_1 \times M_1}{m_1 \times (1 - w_2)} \times 100\% \dots\dots\dots(\text{A.1})$$

In the formula:

- V_1 - the volume of the standard titration solution of barium sulfate consumed by the titration sample solution, in milliliters (mL);
- V_0 - the volume of the standard titration solution of barium sulfate consumed by the titration blank solution, in milliliters (mL);
- c_1 - the concentration of the standard titration solution of barium sulfate, in moles per liter (mol / L);
- M_1 - the molar mass of ferrous lactate in grams per mole (g / mol) [$M_1(\text{C}_6\text{H}_{10}\text{FeO}_6) = 233.96$];
- m_1 - the mass of the sample in grams (g);
- w_2 - the mass fraction of the actual test sample moisture, %;
- 1000 - conversion factor.

The arithmetic mean of the results of two parallel determinations is taken as the measurement result. The absolute difference between two parallel determinations does not exceed the arithmetic mean 2%.

A.4 Determination of moisture

A.4.1 Reagents

- . A 4.1.1 anhydrous methanol (CH_4O): excellent pure.
- . A 4.1.2 carboxamide: excellent pure.
- . A 4.1.3 Extraction solvent: methanol to (A.4.1.1) and formamide (A.4.1.2) by 1 + 1 (V + V) mixture and shake.
- A. 4.1.4 Karl Fischer reagents.

A.4.2 Instruments and equipment

- A. 4.2.1 Karl Fischer moisture meter.
- . A 4.2.2 balance: a sense of the amount is 0.1 mg.

A.4.3 Analysis steps

Weigh 0.1 g of sample to the nearest 0.0001 g, add to the appropriate amount of extraction solvent (A.4.1.3), and use Karl Fischer reagent under stir Titrate to the end point. The moisture content is determined according to GB/T 606.

A.5 Determination of ferric iron (based on Fe^{3+})

A.5.1 Reagents and materials

- A.5.1.1 Hydrochloric acid.
- A.5.1.2 Potassium iodide.
- A.5.1.3 sodium thiosulfate standard titration solution: $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.1 \text{ mol / L}$.
- A.5.1.4 Starch indicator solution: 10 g / L.

A.5.2 Analysis steps

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Weigh 5 g of sample to the nearest 0.0001 g, place in a 250 mL iodine bottle, add 100 mL of water, dissolve 10 mL of hydrochloric acid, and cool to room temperature, add 3 g of potassium iodide, seal the stopper with water, shake and dissolve, and place in the dark for about 5 min. Titrate the solution with sodium thiosulfate standard. Titration, near the end point, add 2 mL of starch indicator solution, and quickly titrate the solution with sodium thiosulfate standard until the solution becomes colorless. A blank test was also performed.

A.5.3 Calculation of results

The mass fraction w_3 of ferric iron (calculated as Fe^{3+}) is calculated according to formula (A.2):

$$w_3 = \frac{(V_3 - V_2) \times c_2 \times M_2}{m_2 \times 1000} \times 100\% \dots\dots\dots(A.2)$$

In the formula:

- V_3 - the volume of the standard titration solution of sodium thiosulfate consumed by the titration sample solution, in milliliters (mL);
- V_2 - volume of sodium thiosulfate standard titration solution consumed by titration of the blank solution, in milliliters (mL);
- c_2 - the concentration of sodium thiosulfate standard titration solution, in moles per liter (mol / L);
- M_2 - the molar mass of iron in grams per mole (g/mol), [$M_2(Fe) = 55.85$];
- m_2 - the mass of the sample in grams (g);
- 1000 - conversion factor.

The arithmetic mean of the results of two parallel determinations is taken as the measurement result. The absolute difference between two parallel determinations does not exceed the arithmetic mean 10%.

A.6 Determination of chloride (as Cl)

A.6.1 Reagents and materials

- A.6.1.1 Nitric acid solution: 1+9.
- A.6.1.2 Silver nitrate solution: 17 g / L.
- A.6.1.3 Chloride (Cl) standard solution: After being prepared according to GB/T 602, it is diluted to 0.01 mg of chloride ion per 1 mL.

A.6.2 Analysis steps

Weigh 0.1 g of sample to the nearest 0.01 g, place it in a 50 mL Nessler colorimetric tube, add appropriate amount of water and 10 mL of nitric acid. Add 1 mL silver nitrate solution, dilute to 50 mL with water, shake well, place in the dark for 5 min, observe the sample in the axial direction on a black background. The turbidity should not be greater than the standard turbidity solution.

Standard turbidity solution: Measure 10 mL of chloride standard solution and place in a 50 mL colorimetric tube. The same treatment as the sample solution.

A.7 Determination of sulfate (as SO_4)

A.7.1 Reagents and materials

- A.7.1.1 hydrochloric acid solution: 1+4.
- A.7.1.2 Barium chloride solution: 250 g / L.
- A.7.1.3 Sulfate (SO_4) standard solution: After being prepared according to GB/T 602, it is diluted to 0.01 mg of sulfate ion per 1 mL.

A.7.2 Analysis steps

Weigh 0.1 g of sample to the nearest 0.01 g, place it in a 50 mL Nessler colorimetric tube, add appropriate amount of water and 2 mL of hydrochloric acid. Add 5 mL of cesium chloride solution, dilute to 50 mL with water, shake well, place in the dark for 10 min, observe the sample in the axial direction on a black background. The turbidity of the solution shall not be greater than the standard turbidity solution.

Standard turbidity solution: Measure 10 mL of the sulphate standard solution and place in a 50 mL colorimetric tube. The same treatment as the sample solution.

