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National Food Safety Standard on Sodium Ferrocyanide

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Report Highlights:
On November 17, 2011, China notified the WTO of National Food Safety Standard: Food Additive Sodium Ferrocyanide as SPS/N/CHN/485. This standard applies to food additive sodium ferrocyanide made from sodium cyanide and ferrous sulfate or ferrum reductum, sodium hydroxide and hydrogen cyanide. It specifies the scope, technical requirements and testing methods for food additive sodium ferrocyanide. The date for submission of final comments to China is January 16, 2012. The proposed date of entry is to be determined. Comments can be sent to China's SPS Enquiry Point at sps@aqsiq.gov.cn. This report is an INFORMAL translation of this document.

General Information:

BEGIN TRANSLATION

GB National Food Safety Standard
GB XXXX－XXXX

National Food Safety Standard

Food Additive Sodium Ferrocyanide (Draft for Soliciting Opinions)

Date of Issue: XXXX-XX-XX

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Issued by the Ministry of Health of the People's Republic of China

National Food Safety Standard

Food Additive Sodium Ferrocyanide

1. Scope

This standard applies to food additive sodium ferrocyanide made from sodium cyanide and ferrous sulfate or ferrum reductum, sodium hydroxide and hydrogen cyanide.

2. Molecular formula, constitutional formula and relative molecular mass

2.1 **Molecular formula**

Na4Fe（CN）6·10H2O

2.2 **Relative molecular mass**

484. 09 (according to international relative molecular mass in 2010).

3. Technical requirements

Organoleptic requirements: should conform to the requirements in table 1.

Table 1: Organoleptic Requirements

Item	Requirement	Method of inspection
Color	Straw yellow	Apply adequate amount of sample into a 50 mL beaker and observe the color and texture status under natural lighting
Texture	Crystallizing particles or crystallizing powders	

The physical and chemical indexes: shall conform to the requirements in Table 2.

Table 2. The physical and chemical indexes

Item	Index	Method of inspection
Sodium ferrocyanide w/% ≥	99.0	A.4 in Appendix A
Chloride (in CI basis) w/% ≤	0.2	A.5 in Appendix A
Cyanide	Test passed	A.6 in Appendix A
Ferricyanide	Test passed	A.7 in Appendix A
Sulfate (in SO4 basis) w/% ≤	0.07	A.8 in Appendix A
Free moisture w/% ≤	1.0	A.9 in Appendix A
Water insoluble w/% ≤	0.03	A.10 in Appendix A
Arsenic（As) / (mg/kg) ≤	3	A.11 in Appendix A

Appendix A

Method of inspection

A.1 Cautions

Some of the reagents as stipulated by the testing method hereof are toxic or corrosive, be careful in operation! If necessary, the operation can be done within laboratory hoods. In case of contact with skin, immediately flush with water. In serious cases, must be treated immediately.

A.2 General Provision

Unless otherwise specified, only the reagents that have been identified as A.R and the Class III water as defined in GB/T6682-2008 can be used.

When no other requirement is noted, all the standard titration solution, the standard solution, preparation and products for determination of impurities that will be used by the testing method shall be prepared as prescribed in GB/T 601, GB/T 602 and GB/T 603.

A.3 Identification test

A.3.1 Reagents and materials

A.3.1.1 Iron chloride （FeCl3·6H2O） solution: 100 g/L.

A.3.2 Identification method

Weigh 0.1 g sample, dissolve it into 1 mL water, and add in 1 mL iron chloride solution, generating blue-black deposition.

A.4 Determination of Sodium ferrocyanide content

A.4.1 Summary of method

In acid medium, an oxidation-reduction reaction occurs between ceric sulfate and Sodium ferrocyanide and the content of Sodium ferrocyanide can be calculated according to the consumption of the standard titration solution of ceric sulfate.

A.4.2 Reagents and materials

A.4.2.1 Sulphuric acid.

A.4.2.2 Standard titration solution of ceric sulfate: c[Ce(SO4)2]≈0.1 mol/L.

A.4.2.3 1, 10-Phenanthroline-ferrous iron indicator solution.

A.4.3 Analysis steps

Weigh 1.5 g of the sample, accurate to 0.0002 g and put it into a 500 mL Erlenmeyer flask, dissolve it in 250 mL water and then slowly add in 25 mL Sulphuric acid; while shaking, add in three drops of 1, 10-Phenanthroline-ferrous iron indicator solution and titrate it by standard titration solution of ceric sulfate until the color becomes from orange to pure yellow.

At the same time, a blank test is conducted, into which the same amount of reagent and the same testing solution in addition to the sample are added and shall be disposed at the same time in the same way as for the sample.

A.4.4 Result calculation

The Sodium ferrocyanide content is calculated by w1, the mass fraction of sodium ferrocyanide [Na4Fe（CN）6·10H2O], and the numerical value is indicated by %, which is calculated according to formula (A.1):

$$w1 = \frac{c(V-V0)\,M/1000}{m} \times 100\% \quad \text{(A.1)}$$

e accurate numerical value of Standard titration solution of ceric sulfate, in the unit of mole per liter (mol/L).

he numerical value of the volume of Standard titration solution of ceric sulfate that has been consumed by the solution for titration test, in the unit of milliliter (mL).

the numerical value of the volume of Standard titration solution of ceric sulfate that has been consumed by the solution for titration test of the blank test, in the unit of milliliter (mL).

m= the numerical value of the mass of the sample, in the unit of gram (g).

he numerical value of the molar mass of sodium ferrocyanide [Na4Fe（CN）6·10H2O], in the unit of gram per mole (g/mol) (M=484.09).

The arithmetic mean of the result of two measurements is taken as the determined result: the absolute difference between the results of two measurements in the same condition shall not be more than 0.30%.

A.5. Determination of cyanide

A.5.1 Reagents and materials

A.5.1.1 Bluestone solution: 1 g/L.

Dissolve 0.1 g Bluestone solution into 100 mL ammonia spirit (1+15).

A.5.1.2 Filter paper: wet a piece of filter paper with Bluestone solution and place the wetted filter paper into sulfureted hydrogen gas to make the filter paper become brown.

A.5.2 Analysis step

Weigh about 1 g sample to dissolve it into 100 mL water. Get one drop of such solution onto the filter paper (A.5.1.2) and no white ring shall occur.

A.6 Determination of ferricyanide

A.6.1 Reagents and materials

A.6.1.1 Lead nitrate solution: 10 g/L.

A.6.1.2 The acetic acid saturated solution of (1+2) benzidine.

A.6.2 Analysis steps

Weigh about 0.1 g sample to dissolve it into 100 mL water. Get one drop of such solution onto the spot plate, add one drop of Lead nitrate solution and once again add several drops of acetic acid saturated solution of (1+2) benzidine; no blue deposition or blue color shall occur.

A.7 Determination of chloride content

A.7.1 Reagents and materials

A.7.1.1 Nitric acid solution: 1+4

A.7.1.2 Silver nitrate solution：17g/L

A.7.1.3 Copper sulphate solution without chloride：50g/L

Weigh 5g of copper sulphate solution without chloride (prepared by two times of recrystallization of A.R blue vitriol) to dissolve it into water and dilute it to 100 mL.

A.7.1.4 Standard solution of chlorine: one milliliter solution contains 0.01 mg of chlorine (CI);

Use a transfer pipet to get 10 mL of the standard solution of chlorine (CI) prepared according to GB/T 602 into a 100 mL volumetric flask, and dilute it with water to the scale, shaking evenly. This solution shall be instantly prepared and instantly used.

A.7.1.5 Litmus blue test paper

A.7.2 Analysis step

Weigh 0.50 g±0.01 g sample to dissolve it into water, add in 12.5 mL copper sulphate solution without chloride while stirring; When stirred evenly, transfer this solution into a 100 mL volumetric flask, and dilute it with water to the scale, shaking evenly. When the upper solution defecates, it is dryly filtered to abandon the initial 20 mL. Transfer 5.00 mL filtered solution into a 50 mL colorimetric cylinder, add in water to 25 mL, neutralized by nitric acid solution, tested by litmus blue test paper; add in 1 mL nitric acid solution and 1 mL Silver nitrate solution, dilute them with water to the scale, shaking evenly, place in shade for ten min; compare the displayed turbidity with the standard turbidimetry solution.

Preparation of standard turbidimetry solution: Transfer 5.00 mL CI standard solution into a 50 mL colorimetric cylinder, add in water to 25 mL, drop and add in copper sulphate solution without chloride, make the color the same as that of the test solution diluted to 25 mL; add in 1 mL nitric acid solution and 1 mL silver nitrate solution, dilute them with water to the scale, shaking evenly, place in shade for ten min.

A.8 Determination of sulfate content

A.8.1 Reagents and materials

A.8.1.1 Hydrochloric acid solution: 1+3

A.8.1.2 Barium chloride solution: 250 g/L.

A.8.1.3 Standard solution of sulfate: 1 mL solution contains 0.05 mg of sulfate (SO4);

Use a transfer pipet to get 25 mL of the standard solution of sulfate (SO4) prepared according to GB/T 602 into a50 mL volumetric flask, and dilute it with water to the scale, shaking evenly. This solution shall be instantly prepared and instantly used.

A.8.1.4 Litmus blue test paper

A.8.2 Analysis step

Weigh 0.50 g±0.01 g sample into a 50 mL colorimetric cylinder, add in 30 mL water to dissolve the sample; neutralized by Hydrochloric acid solution until the Litmus blue test paper becomes red and add in 1 mL Hydrochloric acid solution and 3 mL Barium chloride solution, dilute them with water to the scale, shaking evenly, place for ten min; compare the displayed turbidity with the standard turbidimetry solution.

Preparation of standard turbidimetry solution: Transfer 7.00 mL the standard solution of sulfate (SO4) into a 50 mL colorimetric cylinder, add in 1 mL Hydrochloric acid solution, make the color the same as that of the test solution diluted to 25 mL; add in 1 mL nitric acid solution and 3 mL Barium chloride solution, dilute them with water to the scale, shaking evenly, place for ten min.

A.9 Determination of free moisture

A.9.1 Instruments and devices

Electrothermal constant-temperature dry box: temperature in controlled in the range of 105℃±2℃.

A.9.2 Analysis step

Weigh about 2 g of the sample, accurate to 0.0002 g, put it into the weighing bottle that has been dried to invariable mass at 105℃±2℃ in advance and dry it to the invariable mass in the Electrothermal constant-temperature dry box at 105℃±2℃.

A.9.3 Result calculation

The free moisture content is calculated by w2, the mass fraction, and the numerical value is indicated by %, which is calculated according to formula (A.2):

$$W2 = \frac{(m1-m2)-0.3721 \times m \times w1}{m} \times 100\% \quad \text{(A.2)}$$

Where:

he numerical value of the mass of the sample before drying, in the unit of gram (g);

e numerical value of the mass of the weighing bottle and the sample before drying, in the unit of gram (g);

the numerical value of t the mass of sodium ferrocyanide, in the unit of gram (g);

ıe ratio between the molecular mass of the crystal water in sodium ferrocyanide and the molecular mass of sodium ferrocyanide [Na4Fe（CN）6·10H2O].

The arithmetic mean of the result of two measurements in the same condition is taken as the determined result, where the absolute difference between the results of two measurements in the same condition shall not be more than 0.05%.

A.10 Determination of water insoluble content

A.10.1 Reagents and materials

The solution of ammonium ferric sulfate: 5%.

A.10.2 Instruments and devices

A.10.2.1 Sintered glass crucible: the pore diameter of the filter plate is 5 μm～15 μm;

A.10.2.2 Electrothermal constant-temperature dry box: temperature in controlled in the range of 105℃±2℃.

A.10.3 Analysis step

Weigh 50 g of the sample, accurate to 0.01 g, put it into a 400 mL beaker, and add in 300 mL hot water to dissolve it. Before it gets cold, filter the liquor with the sintered glass crucible that has been dried to invariable mass at 105℃±2℃ in advance, and use hot water to wash the liquor until there is no sodium ferrocyanide (tested by solution of ammonium ferric sulfate) in the filtered solution, which is to put into an electrothermal constant-temperature dry box, being dried to invariable mass at 105℃±2℃.

A.10.4 Result calculation

The water insoluble content is calculated by w3, the mass fraction, and the numerical value is indicated by %, which is calculated according to formula (A.3):

$$W3 = \frac{m1-m2}{m} \times 100\% \quad \text{(A.3)}$$

Where:

he numerical value of the mass of the Sintered glass crucible and the residue, in the unit of gram (g);

e numerical value of the mass of the Sintered glass crucible, in the unit of gram (g);

the numerical value of t the mass of the sample, in the unit of gram (g);

The arithmetic mean of the result of two measurements in the same condition is taken as the determined result, where the absolute difference between the results of two measurements in the same condition shall not be more than 0.003%.

A.11 Determination of Arsenic content

A.711.1 Reagents and devices

The reagents are the same as the arsenic stain method, the second method as defined in Chapter IX, GB/T5009.76—2003.

A.11.2 Instruments and devices

The instruments and devices are the same as the arsenic stain method, the second method as defined in Chapter X, GB/T5009.76—2003.

A.11.3 Analysis steps

Weigh 0.50g±0.01g sample and put it into an arsenic test apparatus and add in 200 mL water. The following operation is the same as that described in Chapter XI, GB5009.76—2003, beginning with “add in 5 mL hydrochloric acid” to “ the arsenic stain of the sample shall not be darker than that of the maximum limit standard of arsenic.”

the maximum limit standard of arsenic: transfer 1.50 mL arsenic standard solution (1 mL arsenic contains 1 μg of arsenic); the following operation is the same as that described in Chapter XI, GB5009.76—2003, beginning with “add in 5 mL hydrochloric acid” to “ get out of the arsenic stain for comparison.” It shall be treated at the same time when the sample is treated.

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