Voluntary - Public

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China - Peoples Republic of

Post: Beijing

National Food Safety Standard - Erythrosine

Report Categories:

FAIRS Subject Report

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Report Highlights:
On May 5, 2010, China notified the WTO of National Food Safety Standard: Food Additives – Erythrosine as SPS/N/CHN/276. This measure applies to the production, circulation, supervision and management of the food additive erythrosine. It specifies the scope, requirements and testing methods. The date for submission of final comments to China is May 20, 2010. The proposed date of entry is May 30, 2010. Contact information on where to send comments is inside the report. This report is an INFORMAL translation of this document.

Executive Summary:
On May 5, 2010, China notified the WTO of National Food Safety Standard: Food Additives – Erythrosine as SPS/N/CHN/276. This measure applies to the production, circulation, supervision and management of the food additive erythrosine. It specifies the scope, requirements and testing methods. The date for submission of final comments to China is May 20, 2010. The proposed date of entry is May 30, 2010. This report is an INFORMAL translation of this document.

Comments can be sent to the China WTO SPS Enquiry Point at: SPS@aqsiq.gov.cn.

This report contains an UNOFFICIAL translation of National Standard on Determination of Erythrosine in Foods.

General Information:
BEGIN TRANSLATION
GB National Food Safety Standard

GB 17512.1-xxxx

Food Additive - Erythrosine
National Food Safety Standard
(Draft for Comment)

Issued on xx-xx-xxxx
Implemented on xx-xx-xxxx
Issued by the Ministry of Health
of the People's Republic of China

Foreword
This Standard is modified in relation to "Food Red No. 3" in Japan's Specifications and Standards for Food Additives (Edition 8).
Main technical differences between this Standard and "Food Red No. 3" in Japan's Specifications and Standards for Food (Edition 8) are listed in Annex C.
This Standard will supersede GB 17512.1-1998 Food Additive - Erythrosine.
Compared with GB 17512.1-1998, this Standard has main changes as follow:
-- adding CI No., INS No. and CAS No.;
-- modifying appearance from "red to red brown powder" to "red to red brown powder or granule";
-- modifying identification method;
-- modifying permissible difference for parallel determinations by spectrophotometric colorimetric method from 2,0 % to 1,0 %;
-- modifying test method for chloride and sulfate;
-- modifying chemical half-limit method for arsenic test into atomic absorption method;
-- modifying requirements for heavy metals (based on Pb) into control requirements for lead and test method into atomic absorption method; and

-- adding control requirements and test method for zinc.
Annex A and Annex B of this Standard are normative, and Annex C is informative.
This Standard supersedes the following previous edition:
-- GB 17512.1-1998

National Food Safety Standards

Food Additive – Erythrosine

1 Scope

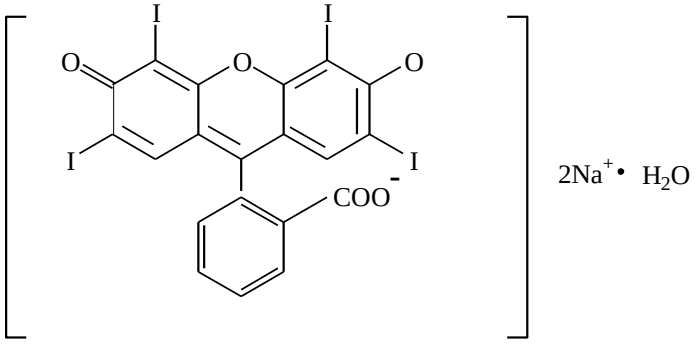
This Standard is applicable to quality control of erythrosine products obtained by iodinating fluorescein.

2 Normative references

Documents referenced in this Standard are indispensable for the application of this Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

3 Chemical name, structural formula, molecular formula, relative molecular mass, INS No. and CAS No.

Chemical name: 9-(o-carboxyphenyl)-6-hydroxy-2,4,5,7-tetraiodo-3H-xanthene-3-one disodium salt monohydrate



Molecular formula: C20H6I4Na2O5 •H2O
Relative molecular mass: 897,87 (based on 2007 International Relative Atomic Mass)
CI No.: C.I.45430
International coding system No.: 127
CAS No.: 16423-68-0 (anhydrous compound)

4 Technical requirements

Technical requirements of erythrosine shall be in accordance with Table 1.
Table 1 Technical requirements of erythrosine

Items	Requirement	Test method
Appearance	Red to red brown powder or granule	Visual inspection under natural light
Erythrosine, *w*/%	≥85,0	A.3 in Annex A
Loss on drying, chloride and sulfate (based on sodium salt), *w*/%	≤14.0	A.4 in Annex A

Water insoluble matters, *w*/%	≤0.20	A.5 in Annex A
Subsidiary colors, *w* /%	≤3.0	A.6 in Annex A
Sodium iodide, *w* /%	≤0.40	A.7 in Annex A
Arsenic, mg/kg	≤1.0	A.8 in Annex A
Lead, mg/kg	≤10.0	A.9 in Annex A
Zinc, mg/kg	≤20.0	A.10 in Annex A

Annex A
(Normative)
Test Method
A.1 General requirements
Reagents and water used in this Standard, unless otherwise stated, are analytically pure reagents and grade III water specified in GB/T 6682-2008. Standard solution, impurity standard solution, preparations and products used in the tests, unless otherwise stated, shall be prepared and calibrated according to requirements of GB/T 601, GB/T 602 and GB/T 603. Test results shall be judged in accordance with 4.3.3 Round-off comparison method in GB/T 8170-2008.

A.2 Identification
A.2.1 Reagents and solutions
a Sulfuric acid;
b. Hydrochloric acid;
c. Ammonium acetate solution: 1.5 g/L.
A.2.2 Apparatus
a Spectrophotometer;
b Cuvette: 10 mm.
A.2.3 Identification method
Weigh about 0.1 g of the sample (accurate to 0.01 g) and dissolve in 100 mL of water, the solution shall develop red clear solution. Add 1 mL of hydrochloric acid to 5 mL of solution to produce red precipitate.
Weigh about 0.2 g of the sample (accurate to 0.01 g) and dissolve in 20 mL of sulfuric acid, the solution shall develop brown yellow. Pipette 2 to 3 drops of the solution, add 5 mL of water to produce orange red precipitate.
Weigh about 0.1 g of the sample (accurate to 0.01 g) and dissolve in 100 mL of ammonium acetate solution. Measure 1 mL of solution and add ammonium acetate solution to make 100 mL of solution. The maximum absorption wavelength of the resulting solution is 526 nm ± 2 nm.

A.3 Determination of erythrosine
A.3.1 Gravimetric method (arbitrary method)
A.3.1.1 Method summary
Dissolve the sample, then dilute, acidify and boil the solution, filter at constant weight, make to constant weight, and then weigh and calculate.
A.3.1.2 Reagents and solutions
a Hydrochloric acid solution: 1 + 49;
b. Hydrochloric acid solution: 1 + 199.
A.3.1.3 Apparatus and instruments
Sintered glass crucible: G4; aperture: 5 μm – 15 μm.
A.3.1.4 Determination procedures
Weigh about 2.5 g of the sample (accurate to 0.0001 g) and put in a beaker, dissolve in water and transfer to a 250 mL volumetric flask, dilute to volume and shake up. Pipette 50 mL of the solution to a 250 mL volumetric flask, and heat to boil, add 20 mL of (1 + 49) hydrochloric acid

solution and boil again, then wash inner wall of the breaker with 5 mL of water and cover watch glass, heat the beaker in water bath for about 5 h, naturally cool to room temperature, filter precipitate by G4 sintered glass crucible baked at 135 ℃ to constant weight and cooled and weighed. After that wash with 15 mL of (1+199) hydrochloric solution twice, then wash with 15 mL of water, bake the precipitate and G4 sintered glass crucible at 135 ℃ to constant weight, cool in a drier for 30 min, and then weigh the matter.

A.3.1.5 Result calculation

Erythrosine is calculated according to formula (A.1) based on mass fraction w1 and its value is expressed in %:

$$w_1 = \frac{m_1 \times 1.074}{m \times 50/250} \times 100 \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (A.1)$$

where:

m1 -value of precipitate mass, expressed in g;

1.074 - transformation coefficient;

m - value of sample mass, expressed in g.

Calculation result is rounded to 0.1.

A.3.1.6 Permissible difference

Absolute difference between two parallel determination results is not more than 0.2 % (mass fraction). Arithmetic mean is taken as determination result.

A.3.2 Spectrophotometric colorimetric method

A.3.2.1 Method summary

Dissolve the sample and standard substance with known erythrosine content in water respectively, measure absorbance at the maximum absorption wavelength, and calculate the content.

A.3.2.2 Reagents and solutions

a) Erythrosine standard substance: ≥85.0 % (mass fraction, content is determined according to A.3.1 in this Standard).

A.3.2.3 Apparatus

a) Spectrophotometer;

b) Cuvette: 10 mm.

A.3.2.4 Preparation of erythrosine standard sample solution

Weigh about 0.25 g of erythrosine standard sample (accurate to 0.0001g) and dissolve in a proper amount of water, transfer the resulting solution to a 1000 mL brown volumetric flask, dilute to volume and shake up. Accurately pipette 10 mL of the solution to a 500 mL brown volumetric flask, dilute to volume and shake up.

A.3.2.5 Preparation of erythrosine sample solution

Weigh and operate according to procedures stipulated in A.3.2.4 Preparation of sample solution of this Standard.

A.3.2.6 Determination procedures

Put erythrosine standard sample solution and erythrosine sample solution in 10 mm cuvettes respectively and measure absorbance by the spectrophotometer at the maximum absorption wavelength, with water as reference solution.

A.3.2.7 Result calculation

Erythrosine is calculated according to formula (A.2) based on mass fraction w1 and its value is expressed in %:

$$w_1 = \frac{Am_0}{A_0 m} \times w_0 \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (A.2)$$

where:

A - absorbance value of erythrosine sample solution;

m0 - value of mass of erythrosine standard substance; expressed in g;

w0 - value of content (gravimetric method) of erythrosine standard substance; expressed in %;

A0 - absorbance value of erythrosine standard solution;
m - value of mass of sample; expressed in g.
Calculation result is rounded to 0.1.
A.3.2.8 Permissible difference
Absolute difference between two parallel determination results is not more than 1.0 % (mass fraction). Arithmetic mean is taken as determination result.
Gravimetric method is arbitrary method for above analytical methods. Determination can be made by any method can for daily inspection according to situation.

A.4 Determination of total of loss on drying, chloride (based on NaCl) and sulfate (based on Na2S04)
A.4.1 Determination of loss on drying
A.4.1.1 Determination procedures
Weigh about 2 g of the sample (accurate to 0.001 g), put in a weighing bottle made to constant weight, and bake the weighing bottle in a 135℃ constant temperature oven to constant weight.
A.4.1.2 Result calculation
Loss on drying is calculated according to formula (A.3) based on mass fraction w2 and its value is expressed in %:

$$w_2 = \frac{m_2 - m_3}{m_2} \times 100 \quad \text{.....} (A.3)$$

where:
m2 - value of mass of sample before drying, expressed in g;
m3 - value of mass of sample after drying to constant weight, expressed in g.
Calculation result is rounded to 0.1.
A.4.1.3 Permissible difference
Absolute difference between two parallel determination results is not more than 0.2 % (mass fraction). Arithmetic mean is taken as determination result.
A.4.2 Determination of chloride (based on NaCl)
A.4.2.1 Reagents and solutions
a) Nitrobenzene;
b) Activated carbon: type 767 injection powder;
c) Nitric acid solution:1+1;
d) Silver nitrate solution: c(AgNO3)=0.1 mol/L;
e) Ammonium ferric sulfate solution;
Preparation method: weigh about 14 g of ammonium ferric sulfate and dissolve in 100 mL of water, filter, add 10 mL of nitric acid and store in a brown bottle;
f) standard titration solution of ammonium thiocyanate: c(NH4CNS)=0.1 mol/L.
A.4.2.2 Preparation of sample solution
Weigh about 2 g of the sample (accurate to 0.001 g), dissolve in 150 mL of water, add about 15 g of activated carbon, mildly boil for 2 to 3 min, add 1 mL of nitric acid solution, frequently shake up and place 30 min (shake occasionally), filter by dry filter paper, add 5 g of activated carbon again in case of color filtrate, place for one hour while shaking occasionally, filter by dry filter paper again (in case of color filtrate again, follow previous procedures until the filtrate is colorless). Clean activated carbon by a 10 mL of water each time for three times, combine the filtrate and transfer to a 200 mL volumetric flask, add water to volume and shake up. The resulting solution is used to determine contents of chloride and sulfate.
A.4.2.3 Determination procedures
Transfer 50 mL of sample solution to a 500 mL conical flask, add 2 mL of nitric acid solution,10 mL of silver nitrate solution (add more silver nitrate solution in case of more chloride) and 5 mL of nitrobenzene, shake vigorously till silver chloride condenses, add 1 mL of ammonium ferric sulfate solution, and titrate excessive silver nitrate with ammonium thiocyanate standard titration solution to the end point and keep 1 min. Meanwhile, perform a blank test by the same method.

A.4.2.4 Result calculation

Chloride is calculated according to formula (A.4) based on mass fraction w3 and its value is expressed in %:

$$w_3 = \frac{c_1[(V_1 - V_0)/1000]M_1}{m_4(50/200)} \times 100 \quad \text{.....(A.4)}$$

where:

c1--accurate value of concentration of ammonium thiocyanate standard titration solution; expressed in mol/L;

V 1 --accurate value of volume of ammonium thiocyanate standard titration solution consumed for titrating blank solution; expressed in mL;

V0--accurate value of volume of ammonium thiocyanate standard titration solution consumed for titrating sample solution; expressed in mL;

M1--value of molar mass of sodium chloride; expressed in g/mol [M(NaCl)=58.4];

m4--value of mass of sample; expressed in g.

Calculation result is rounded to 0.1.

A.4.2.5 Permissible difference

Absolute difference between two parallel determination results is not more than 0.3 % (mass fraction). Arithmetic mean is taken as determination result.

A.4.3 Determination of sulfate (based on Na2S04)

A.4.3.1 Reagents and solutions

a) Sodium hydroxide solution: 0.2 g/L;

b) Hydrochloric acid solution:1 + 1999;

c) Barium chloride standard titration solution: c(1/2BaCl2)=0.l mol/L (see Annex C for preparation method);

d) Phenolphthalein indicator solution:10 g/L;

e) Rhodizonic acid disodium salt indicator solution: weigh 0.1 g of rhodizonic acid disodium salt and dissolve in 10 mL of water (prepared freshly).

A.4.3.2 Determination procedures

Pipette 25 mL of sample solution (A.4.2.2 in this Standard) to a 250 mL conical flask, add a drop of phenolphthalein indicator solution, add hydrochloric acid solution dropwise till pink disappears, shake up, dissolve and titrate with barium chloride standard titration solution while shaking constantly, take rhodizonic acid disodium salt indicator solution as external indicator solution, and consider rose red spot that reaction solution and indicator solution generate at intersection on filter paper and keeps 2 min without fading, then the rose red spot is the end point. Meanwhile, perform a blank test by the same method.

A.4.3.3 Result calculation

Sulfate is calculated according to formula (A.5) based on mass fraction w4 and its value is expressed in %:

$$w_4 = \frac{c_2[(V_2 - V_3)/1000](M_2/2)}{m_4(25/200)} \times 100 \quad \text{.....(A.5)}$$

where:

c2--accurate value of concentration of barium chloride standard titration solution; expressed in mol/L;

V2--accurate value of volume of barium chloride standard titration solution consumed for titrating sample solution; expressed in mL;

V3--accurate value of barium chloride standard titration solution consumed for titrating blank solution; expressed in mL;

M2--value of molar mass of sodium sulphate; expressed in g/mol [M(Na2SO4)=142.04];

m4--value of sample mass; expressed in g.

Calculation result is rounded to 0.1.

A.4.3.4 Permissible difference

Absolute difference between two parallel determination results is not more than 0.2 % (mass fraction). Arithmetic mean is taken as determination result.

A.4.4 Result calculation of total of loss on drying, chloride (based on NaCl) and sulfate (based on Na2S04)

Total of loss on drying, chloride (based on NaCl) and sulfate (based on Na2S04) is calculated according to formula (A.6) based on mass fraction w5 and its value is expressed in %:

$$w_5 = w_2 + w_3 + w_4 \quad \text{(A.6)}$$

where:

--content of loss on drying; expressed in % (mass fraction);

--chloride content (based on NaCl); expressed in % (mass fraction);

--sulfate content (based on Na2SO4); expressed in % (mass fraction).

Calculation result is rounded to 0.1.

A.5 Determination of water insoluble matters

A.5.1 Apparatus

a) Sintered glass crucible : G4 , aperture: 5 μm - 15 μm;

b) Constant temperature oven.

A.5.2 Determination procedures

Weigh about 3 g of sample (accurate to 0.001 g), place in a 500 mL beaker, add 250 mL of 50 ℃ - 60 ℃ hot water for dissolving, filter by G4 sintered glass crucible baked to constant weight at 135 ℃, clean completely with hot water till cleaning solution is colorless, and bake in 135℃ constant temperature oven to constant weight.

A.5.3 Result calculation

Water insoluble matters are calculated according to formula (A.7) based on mass fraction w6 and its value is expressed in %:

$$w_6 = \frac{m_6}{m_5} \times 100 \quad \text{(A.7)}$$

where:

m6--value of mass of water insoluble matters after drying; expressed in g;

m5--value of sample mass; expressed in g.

Calculation result is rounded to 0.01.

A.5.4 Permissible difference

Absolute difference between two parallel determination results is not more than 0.05 % (mass fraction). Arithmetic mean is taken as determination result.

A.6 Determination of subsidiary colors

A.6.1 Method summary

Separate and elute components by paper chromatography, and determine by spectrophotography.

A.6.2 Reagents

a) Absolute ethyl alcohol;

b) N-butyl alcohol;

c) Acetone solution: 1 + 1,

d) Ammonia solution: 4 + 96;

e) Sodium bicarbonate solution: 4 g/L.

A.6.3 Apparatus and instruments

a) Spectrophotometer;

b) Chromatography filter paper: No. 1 medium speed, 150 mm × 250 mm;

c) Chromatography tank: φ240 mm × 300 mm;

d) Micro sample injector: 100 μL;

e) Nessler tub with ground glass stopper: 50 mL;

f) Sintered glass funnel: G3; aperture: 15 μm - 40 μm;
g) 50 mm cuvette;
h) 10 mm cuvette.
A.6.4 Determination procedures
A.6.4.1 Conditions for paper chromatography
a) Developing solvent: n-butyl alcohol + absolute ethyl alcohol + ammonia solution = 6 + 2 + 3;
b) Temperature: 20 ℃ - 25 ℃.
A.6.4.2 Preparation of sample solution
Weigh about 1 g of sample (accurate to 0.001 g), place in a beaker, add a proper amount of water for dissolving, transfer to a 100 mL volumetric flask, dilute to volume and shake up for use. Concentration of the sample solution is 1 %.
A.6.4.3 Preparation of sample eluate
Pipette 100 μL of sample solution with a micro sample injector, evenly inject on a baseline 25 mm away from bottom edge of filter paper to form a straight line with width not more than 5 mm and the length of 130 mm on the filter paper, blow dry with an air blower. Develop the filter paper in the chromatography tank with pre-prepared developing solvent. Immerse the bottom edge of filter paper 10 mm below the developing solvent till front line of the developing solvent rises to 150 mm or subsidiary colors are separated to satisfaction, take out chromatography filter paper, and blow dry with cold air.
Develop blank filter paper under the same condition. The blank filter paper shall be cut from adjacent part of the same filter paper as the filter paper used in previous developing procedures. Schematic diagram of paper chromatography of subsidiary colors is shown in Fig. A.2.

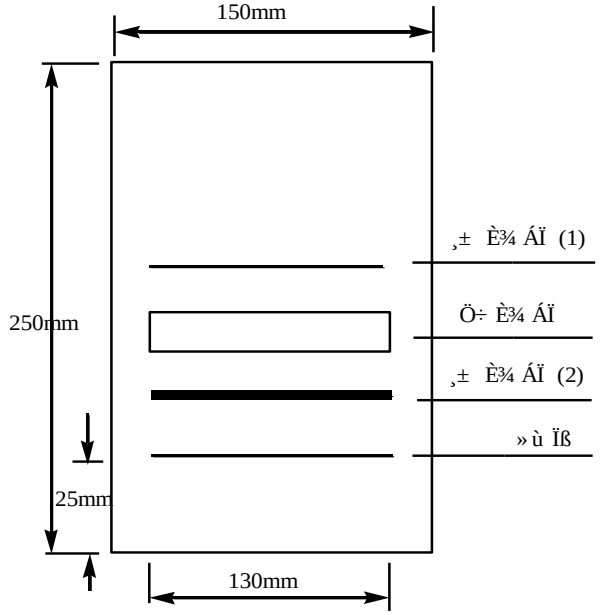


Fig. A.2 Schematic diagram of paper chromatography of subsidiary colors
Cut filter paper of various developed subsidiary colors and parts of filter paper corresponding to subsidiary colors on the blank filter paper at the same size, cut into 5 mm × 15 mm strips, place in 50 mL Nessler tubes respectively, accurately add 5 mL of acetone solution and shake for 3 min - 5 min, accurately add 20 mL of sodium hydrocarbonate solution again and shake thoroughly, and filter the resulting solution by sintered glass funnel respectively, the resulting filtrate must be clear and free of suspension. Add water to volume, and obtain eluates of subsidiary colors and blank solution respectively. Use 50 mm cuvette to measure absorbance of eluates of subsidiary colors by the spectrophotometer at the maximum absorption wavelength of subsidiary colors. Use mixture of 5 mL of acetone solution and 20 mL of sodium hydrocarbonate solution as reference solution when measuring absorbance by the spectrophotometer.

A.6.4.4 Preparation of standard solution
Pipette 2 mL of 1 % sample solution to a 100 mL volumetric flask, dilute to volume, and shake up. The resulting solution is taken as standard solution.
A.6.4.5 Preparation of standard eluate
Pipette 100 μL of standard solution by the micro sample injector, evenly inject on a baseline 25 mm away from the bottom edge of filter paper, and blow dry with an air blower. Put filter paper in a chromatography tank with pre-prepared developing solvent for developing, take out and cool the filter paper with cold air after front line of developing solvent rises to 40 mm, cut all parts with developed colors and extract by the method in A.6.4.3 of this Standard to obtain standard eluate. Use 10 mm cuvette to measure absorbance value at the maximum absorption wavelength.
Meanwhile, develop blank filter paper under the same conditions and measure absorbance value of eluate according to the same procedures.
A.6.4.6 Result calculation
Subsidiary colors are calculated according to formula (A.8) based on mass fraction w7 and its value is expressed in %:

$$w_7 = \frac{[(A_1 - b_1) + \cdots\cdots + (A_n - b_n)]5}{(A_s - b_s)(100/2)} \times S \qquad (A.8)$$

where:
A1…, An--absorbance values of eluates of subsidiary colors measured at 50 mm beam path distance;
b1…,bn--absorbance values of control blank eluates of subsidiary colors measured at 50 mm beam path distance;
As--absorbance value of standard eluate measured at 10 mm beam path distance;
bs--absorbance value of standard control blank eluate measured at 10 mm beam path distance;
5--ratio of being converted into 10 mm beam path distance;
100/2--ratio of standard eluate converted into 1 % sample solution;
S--sample content; expressed in % (mass fraction).
Calculation result is rounded to 0.1.
A.6.4.7 Permissible difference
Absolute difference between two parallel determination results is less than 0.2 % (mass fraction). Arithmetic mean is taken as determination result.

A.7 Determination of sodium iodide
A.7.1 Method summary
Titrate content of sodium iodide in sample solution with silver nitrate standard titration solution by potentiometric titration.
A.7.2 Reagent
Silver nitrate standard titration solution: c(AgNO3) = 0.001 mol/L.
A.7.3 Apparatus and instruments
a) Digital millivoltmeter;
b) Iodine ion selective electrode;
c) Reference electrode;
d) Electromagnetic stirrer.
A.7.4 Preparation of sample solution
Weigh about 4.0 g of sample (accurate to 0.0001 g), place in a beaker, add accurately measured 100 mL of water, and stir with the electromagnetic stirrer to dissolve, the resulting solution is taken as sample solution.
A.7.5 Determination procedures
Insert iodine ion selective electrode and reference electrode into dissolved sample solution, adjust readings of millivoltmeter, and titrate with silver nitrate standard titration solution while stirring thoroughly. Titrate 0.5 mL at beginning of titration, and increase titer gradually, observe electric

potential change after each titration and record reading. Lower titer to 0.1 mL when closing to end point, draw a curve with electric potential millivoltmeter readings and titer volume of relevant silver nitrate standard titration solution, consider maximum jump point of the curve as titration end point and obtain volume of silver nitrate standard titration solution.

A.7.6 Result calculation

Sodium iodide is calculated according to formula (A.9) based on mass fraction w8 and its value is expressed in %:

$$w_8 = \frac{c_2(V_4/1000)M_3}{m_7} \times 100 \cdots\cdots\cdots\cdots\cdots\cdots (A.9)$$

where:

c2--accurate value of concentration of silver nitrate standard titration solution; expressed in mol/L;

V4--value of volume of silver nitrate standard titration solution consumed for titrating sample; expressed in mL;

M3--value of molar mass of sodium iodide; expressed in g/mol [M(NaI) =149.89];

m7--value of sample mass; expressed in g.

Calculation result is rounded to 0.1.

A.8 Determination of arsenic

A.8.1 Method summary

Digest erythrosine by wet method, prepare into sample solution, and determine arsenic content by atomic absorption spectrometry.

A.8.2 Reagents and solutions

a) Nitric acid;

b) Sulfuric acid solution: 1 + 1;

c) Nitric acid-perchloric acid mixed solution: 3 + 1;

d) Arsenic (As) standard solution: after preparation and calibrating in accordance with GB/T 602, dilute and prepare into three standard solutions with relevant arsenic concentrations according to requirements of used apparatus;

e) Sodium hydroxide solution: 1 g/L;

f) Sodium borohydride solution: 8 g/L (solvent is 1 g/L sodium hydroxide solution);

g) Hydrochloric acid solution: 1 + 10;

h) Potassium iodide solution: 200 g/L.

A.8.3 Apparatus

Atomic absorption spectrometer

Reference conditions of apparatus: analysis line wavelength of arsenic hollow cathode lamp: 193.7 nm; slit: 0.5 nm – 1.0 nm; lamp current: 6 mA-10 mA;

Flow rate of carrier gas: 250 mL/min, argon gas ;

Temperature of atomizer: 900 ℃.

A.8.4 Determination procedures

A.8.4.1 Sample digestion

Weigh about 1.0 g of sample (accurate to 0.001 g), place in a 250 mL conical or round bottomed flask, add 10 mL – 15 mL of nitric acid and 2 mL of sulfuric acid, shake up, and heat with low fire to remove nitrogen dioxide gas, stop heating when solution develops brown, cool naturally, add 5 mL of nitric acid-perchloric acid mixed solution, heat with strong fire till the solution is colorless and transparent or yellowish. In case of opaque solution, cool naturally, add 5 mL of nitric acid-perchloric acid mixed solution again, keep heating till solution is colorless and transparent or yellowish and produces white smoke (avoid carbonization due to burning out), stop heating, cool naturally, add 5 mL of water, heat to boil to remove residual acid-perchloric acid (add water to boil again when necessary). Keep heating to produce white smoke and keep 10 min, cool naturally, transfer to a 100 mL volumetric flask (filter in case of turbidity, precipitate and mechanical impurities in solution), and dilute to volume with hydrochloric acid solution.

Meanwhile, prepare blank solution by the same method.
A.8.4.2 Determination
Measure 25 mL of digested sample solution to a 50 mL volumetric flask, add 5 mL of potassium iodide solution, dilute to volume with hydrochloric acid solution, shake up, and stand for 15 min. Meanwhile, prepare blank test solution with blank solution by the same method.
Turn on apparatus, after apparatus and arsenic hallow cathode lamp is fully preheated and baseline is stably, use sodium borohydride solution as hydride reducing agent and inject standard blank solution, standard solution, sample blank test solution and sample solution in order according to computer instruction. After test, computer can automatically generate working curve and arsenic concentration of sample solution after deducting sample blank solution. Arsenic content of sample can be automatically calculated after inputting sample information (name, weight, dilution volume, etc.).
A.8.5 Permissible difference
Absolute difference between two parallel determination results is less than 0.1 (mg/kg). Arithmetic mean is taken as determination result.

A.9 Determination of lead
A.9.1 Method summary
Digest erythrosine by wet method, prepare into sample solution, and determine lead content by atomic absorption spectrometry.
A.9.2 Reagents and solutions
a) Lead (Pb) standard solution: after preparation and calibrating in accordance with GB/T 602, dilute and prepare into three standard solutions with relevant lead concentrations according to requirements of used apparatus;
b) Sodium hydroxide solution: 1 g/L;
c) Sodium borohydride solution: 8 g/L (solvent is 1 g/L sodium hydroxide solution);
d) Hydrochloric acid solution: 1 + 10.
A.9.3 Apparatus
Atomic absorption spectrometer
Reference conditions of apparatus: Method 3 - Flame atomic absorption spectrometry in GB 5009.12.
A.9.4 Determination procedures
The sample solution and blank solution in A.8.4.1 of this Standard can be directly used. Operate according to Method 3 - Flame atomic absorption spectrometry in GB 5009.12.
A.9.5 Permissible difference
Absolute difference between two parallel determination results is less than 1.0 (mg/kg). Arithmetic mean is taken as determination result.

A.10 Determination of zinc content
A.10.1 Method summary
Digest erythrosine by wet method, prepare into sample solution and determine zinc content by atomic absorption spectrometry.
A.10.2 Reagents and solutions
a) Zinc (Zn) standard solution: after preparation and calibrating in accordance with GB/T 602, dilute and prepare into three standard solutions with relevant zinc concentrations according to requirements of the apparatus;
b) Sodium hydroxide solution: 1 g/L;
c) Sodium borohydride solution: 8 g/L (solvent is 1 g/L sodium hydroxide solution);
d) Hydrochloric acid solution: 1 + 10.
A.10.3 Apparatus
Atomic absorption spectrometer
Reference conditions of apparatus: Method 1: Atomic absorption spectrometry in GB 5009.14:
A.10.4 Determination procedures

The sample solution and blank solution in A.8.4.1 of this Standard can be directly used. Determine according to Method 1 - Atomic absorption spectrometry in GB 5009.14.

A.10.5 Permissible difference

Absolute difference between two parallel determination results is not more than 2.0 (mg/kg). Arithmetic mean is taken as determination result.

Annex B

(Normative)

Preparation Method of Barium Chloride Standard Solution

B.1 Reagents and solutions

a) Barium chloride;

b) Ammonia;

c) Sulfuric acid standard titration solution: [c(1/2H2SO4)=0.1 mol/L]; prepare and calibrate in accordance with GB/T 601;

d) Rhodizonic acid disodium salt indicator solution (weigh 0.1 g of rhodizonic acid disodium salt, dissolve in 10 mL of water; prepare freshly);

e) Universal pH paper.

B.2 Preparation

Weigh 12.25 g of barium chloride, dissolve in 500 mL of water, transfer to a 1000 mL volumetric flask, dilute to volume, and shake up.

B.3 Calibration method

Pipette 20 mL of sulfuric acid standard titration solution, place in a 250 mL conical flask, add 50 mL of water, neutralize with ammonia to make universal pH paper show 8, and titrate with barium chloride standard titration solution, taken rhodizonic acid disodium salt indicator solution as external indicator solution and rose red spot that reaction solution and indicator solution generate at intersection on filter paper and keeps 2 min without fading, then the rose red spot is the end point.

B.4 Result calculation

Concentration of barium chloride standard titration solution is calculated according to formula (B.1) based on c(1/2BaCl2) and its unit is mol/L:

$$c(\frac{1}{2}BaCl_2)=\frac{c_1V_4}{V_5}.....(B.1)$$

where:

c1--accurate value of concentration of sulfuric acid standard titration solution; expressed in mol/L;

V4--accurate value of volume of sulfuric acid standard titration solution; expressed in mL;

V5--accurate value of volume of consumed barium chloride standard titration solution; expressed in mL.

Calculation result is rounded to 0.0001.

Annex C

(Informative)

Technical Differences Between This Standard and "Food Red No. 3" in Japan's Specifications and Standards for Food (Edition 8)

Table C.1 Technical differences between this Standard and "Food Red No. 3" in Japan's Specifications and Standards for Food (Edition 8)

Chapter of this	Technical differences between this Standard and "Food Red No. 3" in Japan's	Cause

Standard	*Specifications and Standards for Food (*Edition 8*)*	
A.3	Gravimetric method is test method of Japan's standard erythrosine content and arbitrary method, while this Standard adds spectrophotometric method that is a routine test method.	Spectrophotometric method is added to make routine detection more convenient and efficient.
A.4	Japan's standard respectively stipulates requirements for loss on drying not more than 12.0 %, requirement for chloride and sulfate not more than 2.0 % respectively, and uses ion chromatography for test, while this Standard combines loss on drying, chloride (based on NaCl) and sulfate (based on Na_2SO_4) with requirement not more than 14.0 %, and uses chemical titration as its test method.	It is found in actual detection that ion chromatography is complicated, poorly repeated and inaccurate with regard to detection result, while chemical titration, as classical method, is simple and accurate.
A.6	Exact requirements for subsidiary colors are not regulated and spotting method is taken as test method in Japan's standard, while requirement of subsidiary colors is not more than 3,0 %, thin-layer chromatography method and spectrophotometric method after elution treatment are taken as test methods in this Standard.	Thin-layer chromatography method and spectrophotometric method after elution treatment can accurately control subsidiary colors, thus controlling inherent quality of products more efficiently.
A.8	Requirement for arsenic (based on As_2O_3) is not more than 4 mg/kg, and its test method is limit colorimetric method in Japan's standard, while arsenic requirement is not more than 3.0 mg/kg and test method is atomic absorption method in this Standard.	Atomic absorption method can help accurately detect arsenic content.
A.9	Requirement for heavy metal (based on Pb) is not more than 20 mg/kg and test method is limit colorimetric method in Japan's standard, while requirement for lead content is not more than 10 mg/kg and test method is atomic absorption method in this Standard.	Atomic absorption method can help accurately detect lead content.

END TRANSLATION