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USDA Foreign Agricultural Service

GAIN Report

Global Agriculture Information Network

Voluntary Report - public distribution

Date: 6/16/2003

GAIN Report Number: JA3036

Japan

Sanitary / Phytosanitary / Food Safety

New changes in use designation of food additives 2003

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Report Highlights:
Japan has proposed a new designation of l-ascorbic acid 2-glucoside as a food additive, the revision of the use designation for five other additives, potassium pyrosulfite, sodium hydrosulfite, sodium pyrosulfite, sodium sulfite and sulfur dioxide, and the revision of compositional specifications for tar colors.

Includes PSD Changes: No
Includes Trade Matrix: No
Unscheduled Report
Tokyo [JA1]
[JA]

The Ministry of Labor, Health and Welfare (MHLW) invited foreign Embassies in Tokyo today to comment on the proposals. Foreign governments have until June 24, 2003 to comment. MHLW will open the proposal to comments from a wider audience and notify the WTO SPS Committee before final review and adoption.

The proposals include the new designation of one food additive (l-ascorbic acid 2-glucoside), the revision of the use designation standards for five additives (potassium pyrosulfite, sodium hydrosulfite, sodium pyrosulfite, sodium sulfite and sulfur dioxide), and the revision of compositional specifications for tar colors.

All interested parties are encouraged to send their comments well before the deadline for consideration by Foreign Agricultural Service, USDA. The office responsible for the comments is as follows:

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USDA Foreign Agricultural Service
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Following are the document distributed by MHLW.

(Begin document)

THE 83th CONFERENCE FOR PROMOTION OF FOOD IMPORT FACILITATION

(FOOD SAFETY GROUP)

Standards Division
Department of Food Safety
Pharmaceutical and Food Safety Bureau
Ministry of Health, Labour and Welfare

1. Date: June 10 , 2003 (Tuesday) 10:00 ~

2. Place: Central Joint Governmental Office Building, No. 5
   Ministry of Health, Labour and Welfare
   Meeting Room No. 13 (5F)
   Address: 1-2-2, Kasumigaseki, Chiyoda-ku
   Tel: 03-5253-1111

3. Agenda:
(1) Designation of L-Ascorbic Acid 2-Glucosid as a food additive
(2) Revision of the standards for use of potassium pyrosulfite, sodium hydrosulfite, sodium pyrosulfite, sodium sulfite, and sulfur dioxide
(3) Revision of the compositional specifications for tar colors
(4) Miscellaneous

Purpose

This activity is to newly designate a substance as approved food additive and to revise the standards for use and specifications for designated additives.

Under Article 6 of the Food Sanitation Law, food additives may be used or marketed only when designated by the Minister of Health, Labour and Welfare. Where use standards or specifications are established for additives under Article 7 of the law, those additives may be marketed only when they meet the standards or specifications.

In response to consultation by the Minister of Health, Labor and Welfare, the Joint Subcommittee of Toxicity and Food Additives under the Food Sanitation Committee under the Pharmaceutical Affairs and Food Sanitation Council has discussed the designation of L-Ascorbic Acid 2-Glucosid as a food additive, the revision of the standards for use of the five substances (potassium pyrosulfite, sodium hydrosulfite, sodium pyrosulfite, sodium sulfite, and sulfur dioxide), and the revision of the compositional specifications for tar colors.

The subcommittee concluded that the substance may be designated as a food additive and that the specifications given in attachment 1 should be established but use standards were not necessary. The subcommittee also concluded that the use standards for these five substances may be revised, and the compositional specifications for these tar colors should be revised.

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Outline

1) L-Ascorbic Acid 2-Glucosid will be designated by the Minister of Health, Labour and Welfare as not injurious to human health under Article 6 of the Food Sanitation Law. The use standards and compositional specifications will be established under Article 7 of the law. (Attachment 1)

2) The standards for use of Potassium Pyrosulfite, Sodium Hydrosulfite, Sodium Pyrosulfite, Sodium Sulfite, and Sulfur Dioxide will be revised under Article 7 of the law. (Attachment 2)

3) The compositional specifications for these tar colors will be revised under Article 7 of the law. (Attachment 3)

Deadline of submission of opinion

Please submit your opinion on the proposed matters by Tuesday, June 24, 2003. If you do not contact us by the given date, you will be regarded as having no opinion.

Contact

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Standards Division, Department of Food Safety,
Pharmaceutical and Food Safety Bureau,
Ministry of Health, Labour and Welfare
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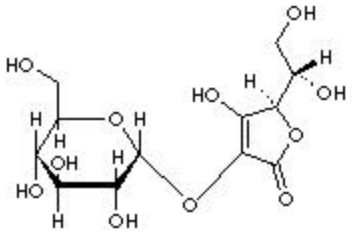
Attachment 1

Draft Compositional Specifications

L-Ascorbic Acid 2-Glucoside



$C_{12}H_{18}O_{11}$[1294-99-78-1] **Molecular weight: 338.26**

Content When calculated on the anhydrous basis, L-Ascorbic Acid 2-Glucoside contains not less than 98.0% of L-ascorbic acid 2-glucoside ($C_{12}H_{18}O_{11}$).

Description It occurs as a while to yellowish white powder or crystallized powder. It is odorless and has an acid taste.

Identification (1) To 5 ml of L-Ascorbic Acid 2-Glucoside solution (1 → 50), add one drop of potassium permanganate solution (1→ 300). The color of the solution disappears immediately. To 5 ml of L-Ascorbic Acid 2-Glucoside solution (1 ? 50), add one to two drops of sodium 2,6-dichlorophenolindophenol. The color of solution disappears immediately.

(2) To 5 ml of boiled Fehling's TS, add 2-3 drops of L-Ascorbic Acid 2-Glucoside solution (5 → 40), and heat for about 5 minutes. A red precipitate is formed.

(3) Determine the infrared absorption spectrum of L-Ascorbic Acid 2-Glucoside, as directed in the potassium bromide disk method under Infrared Spectrophotometry. It exhibits the

absorptions at wave numbers of about 3,300 cm^{-1}, 1,770 cm^{-1}, 1,700 cm^{-1}, 1,110 cm^{-1} and 1,060 cm^{-1}.

Purity (1) Specific rotation [α] = between +186.0 and +188.0 ° (5 g on the dried basis, water 100 ml)

(2) Melting point 158-163?

(3) Heavy metals Not more than 10 μg/g as Pb (2.0g, Method 2? Control solution Lead Standard Solution 2.0ml)

(4) Arsenic Not more than 1.0 μg/g as As_2O_3 (2.0g? Method 3, Apparatus B)

Loss on Drying Not more than 1.0%(105? , 2 hours)

Residue on Ignition Not more than 0.10%

Assay Weigh accurately 0.5 g each of the sample and L-ascorbic acid 2-glucoside for assay, add water to each to dissolve. Add exactly 10 ml of the internal standard solution, then water to make exactly 50 ml. Use these solutions as the test solution and the standard solution, respectively. Measure 20 μl each of the test solution and the standard solution, and perform Liquid Chromatography for each solution under the operating conditions given below. Calculate the ratio of the peak area of L-ascorbic acid 2-glucoside to the peak area of internal standard for each solution, and express as Q_T and Q_S, respectively. Obtain each weight of L-ascorbic acid 2-glucoside for assay and the sample on the anhydrous basis, and determine the content of L-ascorbic acid 2-glucoside in the sample, by the following formula.

Content of L-ascorbic acid 2-glucoside $C_{12}H_{18}O_{11}$) =

$$\frac{\text{Weight of L-ascorbic acid 2-glucoside for assay(mg)}}{\text{Weight (mg) of the sample}} \times \frac{Q_T}{Q_S} \times 100(\%)$$

Internal standard solution: Use 5% w/v of glycerol solution.

Operating conditions

Detector: Differential refractometer

Column packing material: Strongly acidic cation-exchange resin in which sulfonic acid group is combined with styrene-divinyl benzene copolymer

Column: A stainless steel tube 4-8 mm in internal diameter and 20-50 cm in length

Column temperature: Constant temperature about 35?

Mobile phase: Nitric acid(1 → 10,000)

Flow rate: Adjust so that the retention time of L-ascorbic acid 2-glucoside is about 10 minutes.

Regents

L-ascorbic acid 2-glucoside for assay $C_{12}H_{18}O_{11}$

It occurs as white crystals or as a white crystalline powder. It is oderless and has an acid taste.

Content When calculated on the anhyrous basis, it contains not less than 99.9% of L-ascorbic acid 2-glucoside ($C_{12}H_{18}O_{11}$).

Indentification (1) To 5 ml of L-ascorbic acid 2-glucoside for assay solution (1→50), add one drop of potassium permanganate solution (1 → 300). The color of the solution disappears immediately. To 5 ml of L-Ascorbic Acid 2-Glucoside solution (1 → 50), add one to two drops of sodium 2,6-dichlorophenolindophenol. The color of solution disappears immediately

(2) To 5 ml of boiled Fehling's TS, add 2-3 drops of a solution of L-ascorbic acid 2-glucoside solution for assay (5 → 40), and heat for about 5 minutes. A red precipitate is formed.

(3) Determine the infrared absorption spectrum of L-ascorbic acid 2-glucoside for assay, as directed in the potassium bromide disk method under Infrared Spectrophotometry. It exhibits the absorptions at wave numbers of about 3,300 cm^{-1}, 1,770 cm^{-1}, 1,700 cm^{-1}, 1,110 cm^{-1} and 1,060 cm^{-1}.

Purity (1) Clarity of solution Clear(1.0g? Water 50ml)

(2) Free ascorbic acid and free glucose

Test soluton Dissolve 0.5 g of L-ascorbic acid 2-glucoside for assay in the mobile phase specified in the operating conditions to make exactly 25 ml.

Standard solutions Dissolve 0.5 g of L-ascorbic acid in the mobile phase to make exactly 25 ml. Take exactly 1.0 ml of this solution, and add the mobile phase to make exactly 100 ml. Use this solution as the ascorbic acid standard stock solution. One point zero milliliter of this solution contains 0.2 mg of ascorbic acid. Separately, dissolve 0.5 g of glucose into the mobile phase to make exactly 25 ml. Take exactly 1.0 ml of this solution, and add the mobile phase to make exactly 100 ml. Use this solution as the glucose standard stock solution. One point zero milliliter of this solution contains 0.2 mg of glucose. Measure exactly 10 ml each of the ascorbic acid standard stock solution and the glucose standard stock solution, and add the mobile phase to make exactly 100 ml each. Use these solutions as the ascorbic acid standard solution and the glucose standard solution.

Procedure Measure 10µl each of the test solution, the ascorbic acid standard solution, and the glucose standard solution, and perform Liquid Chromatography under the operating conditions below. Determine the peak areas of ascorbic acid and glucose for each solution. The peak areas for the test solution at the same retention times as for ascorbic acid and as for

glucose are not greater than the corresponding peak areas of ascorbic and glucose for the ascorbic acid standard solution and the glucose standard solution.

Operating conditions

Detector: Differential refractometer
Column packing material: 5-10 µm dimethylaminopropylsilyl silica gel dimethylaminopropyl silylated silica gel for Liquid Chromatograph
Column: A stainless steel 4-5 mm in internal diameter and 15-30 in length
Column temperature: 40?
Mobile phase: A mixture of acetonitrile and 0.5% (vol) potassium dihydrogen phosphate solution in phosphoric acid
Flow rate: Constant rate about 0.7 ml/minute

Loss on Drying Not more than 1.0%(105? ? 2 hours)

Assay Weigh accurately about 1.0 g of the sample, dissolve in 30 ml of water, and add two drops of phenolphthalein TS. Titrate with 0.2 mol/l sodium hydroxide solution until the slight red color of the solution maintains about for 30 seconds.

One ml of 0.2 mol/l sodium hydroxide is 67.654 mg of $C_{12}H_{18}O_{11}$.

Attachment 2

Summary of draft revised use standards

Target substances: Potassium pyrosulfite, Sodium hydrosulfite, Sodium pyrosulfite, Sodium sulfite, and Sulfur dioxide.

1. Dried raisin is newly added to the target foods.
2. "Dried mashed potatoes" is replaced with "dried potatoes." Dried potetos include any kind of dried potetos including mash potatoes and cut potetoes.

Target foods	As residue limit of SO_2
Kanpyo: dried gourd strips	Less than: 5.0g/kg
Dried fruits (excluding dried raisin)	2.0g/kg
Dried raisin	**1.5g/kg**
Konnyaku-ko(powdered konjac)	0.90g/kg
Dijon mustard, ~~**Dried mashed potato**~~ **Dried potatoes**, gelatin	**0.50g/kg**
Wines (any kind of fruit wine, excluding squeezed fruit juice and its concentrate that are used for wine brewing and that contain 1% or more alcohol by volume)	0.35g/kg
Candied cherries? Food molasses	0.30g/kg
Tapioca starch for saccharification	0.25g/kg
Mizuame (starch syrup)	0.20g/kg
Natural fruit juice (confined to foods to be consumed in 5-fold or more dilution)	0.15g/kg
Amanatto (bean glaces), simmered beans	0.10g/kg
Frozen crab, Prawn	0.10g/kg of shelled prawn
Other foods (excluding cherry used for candied cherries, hop used for brewing beer, fruit juice used for manufacturing wine, and squeezed fruit juice containing alcohol of not less than 1% by volume, and a concentrate of the same).*	0.030g/kg

*When other foods (excluding *konnyaku*) manufactured or processed using foods listed in this table in which an additive listed in the left column is used according to the standards of use contain a residue (as SO_2) of not less than 0.030 g/ kg, the amount of residue shall be the maximum residue limit.

Attachment 3

Revision of testing methods and compositional specifications for coloring matter tests (draft)

General tests

8. Subsidiary colors

Test Solution Weigh accurately the specified amount of the sample, dissolve in the specified solution to make exactly 100ml.

Standard Solutions Dry the specified subsidiary colors for 24 hours in a vacuum desiccator, weigh 10.0 mg of each color, dissolve in the specified solution to make exactly 100ml, respectively. Use them as the standard stock solutions. Measure exactly 1 ml, 2ml, 5ml, and 10ml of each standard stock solution, place each in a flask, and add the specified solution (the solution used to prepare the standard stock solution) to make exactly a 100ml-solution each.

Procedure Measure 20 μl of each of the test solution and the standard solutions. For each solution, perform Liquid Chromatography under the operating conditions below. Then, measure the peak area for each of the standard solutions, and make a calibration curve for each coloring matter. Measure the peak area of each subsidiary color in the test solution. Obtain the content of each color using the calibration curves, and calculate total amount of the subsidiary colors.

Operating Conditions

Detector: Visible range absorption detector (measurement wavelength directed in the individual monograph).

Column packing material: 5-μm chemical-bond type octadecylsilanized silica gel.

Column: A stainless steal tube of 4.6 mm in internal diameter and 25 cm in length.

Column temperature: 30? .

Flow rate: 1 ml/min.

9. Unreacted raw materials and products of side reactions

Test Solution Weigh accurately the specified amount of the sample, dissolve in the specified solution to make exactly 100ml.

Standard Solutions Dry the specified unreacted raw materials and products of side reactions for 24 hours in a vacuum desiccator, weigh 10.0 mg of each substance, dissolve in the specified solution to make exactly 100ml, respectively. Use them as the standard stock solutions, respectively. Measure exactly 1 ml, 2ml, 5ml, and 10ml of each standard stock

solution, place each in a flask, and add the specified solution (the solution used to prepare the standard stock solution) to make exactly a 100ml-solution each.

Procedure Measure 20 μl of each of the test solution and the standard solutions. For each solution, perform Liquid Chromatography under the operating conditions below. Then, measure the peak area for each standard solution, and make a calibration curve for each substance. Measure the peak area of each of the unreacted raw materials and products of side reactions in the test solution. Obtain the content of each unreacted raw material and reaction intermediate using the calibration curve.

Operating Conditions

Detector: Ultraviolet region absorption detector (measuring wavelength: directed in the Monographs)

Column packing material: 5-μm chemical-bond type octadecylsilanized silica gel.

Column: A stainless steel tube of 4.6 mm in inner diameter and 25 cm in length

Column temperature: 30?

Flow rate: 1 ml/min

10. Unsulfonated primary aromatic amines

(1) Hereinafter in the Monographs, such a specification as "not more than 0.010% as aniline (Coloring Matter Tests)" indicates that when determined as directed in the following procedure, the content of unsulfonated primary aromatic amines is not more than 0.010% as aniline.

Procedure *Test Solution* Weigh 2.0 g of the sample, transfer into a separating funnel containing 100 ml of water, add 50 ml of water to dissolve. Add 5 ml of sodium hydroxide solution (4 → 100) and 50 ml of ethyl acetate, shake, and extract. Separate the ethyl acetate layer. Add 50 ml of ethyl acetate into the water layer shake, and extract. Combine the two ethyl acetate extracts, and wash with sodium hydroxide solution (4 → 1000) until the color of the solution disappears. Extract three times from the ethyl acetate extract with three 10-ml portions of diluted hydrochloric acid (3 → 10), combine the hydrochloric acid extract, and add water to make exactly 100 ml. Use this solution as the sample solution. Transfer 10 ml of the sample solution into a test tube, and cool in ice for 10 minutes. Add 1 ml of potassium bromide solution (1 → 2) and 0.05 ml of sodium nitrite solution (1 → 30), mix, and allow to stand for 10 minutes in ice. Transfer this mixed solution with water into a 25-ml volumetric flask containing 1 ml of 0.05 mol/l disodium salt of 3-hydroxy-2,7-naphthalenedisulfonic acid solution and 10 ml of sodium carbonate solution (1 → 10), and add water to make exactly 25 ml. Allow this solution to stand for 15 minutes in a dark place.

Control Solution Weigh 10. mg of aniline, add 30 ml of diluted hydrochloric acid (3 → 10) to dissolve, add water to make exactly 100 ml. Measure exactly 2 ml of this solution and add 30 ml

of diluted hydrochloric acid (3 → 10) and water to make 100 ml. For his solution proceed in the same manner as directed for the test solution.

Reference Solution To measure the absorbance of the test solution, use the following reference solution: Transfer 10 ml of the sample solution into a 25-ml volumetric flask, add 1 ml of 0.05 mol/l disodium salt of 3-hydroxy-2,7-naphthalenedisulfonic acid solution and 10 ml of sodium carbonate solution (1 → 10), and add water to make exactly 25 ml.

To measure the absorbance of the control solution, use the following reference solution: To 3 ml of diluted hydrochloric acid (3 → 10), add 1 ml of 0.05 mol/l disodium salt of 3-hydroxy-2,7-naphthalenedisulfonic acid solution and 10 ml of sodium carbonate solution (1 → 10), and add water to make exactly 25 ml.

Measure the absorption for each solution at a wavelength of 510 nm. The absorption of the test solution is not more than that of the control solution.

Monographs

Food Red No. 2

Purity

(6) Unreacted raw materials and products of side reactions Not more than 0.5% as the total of
monosodium salt of 4-amino-1-naphthalenesulfonic acid,
disodium salt of 7-hydroxy-1,3-naphthalenedisulfonic acid,
disodium salt of 3-hydroxy-2,7-naphthalenedisulfonic acid,
monosodium salt of 6-hydroxy-2-naphthalenesulfonic acid, and
trisodium salt of 7-hydroxy-1,3,6-naphthalenetrisulfonic acid.

Test Solution Weigh accurately about 100 mg of Food Red No. 2, and dissolve in ammonium acetate solution (1.54 → 1,000) to make exactly 100 ml.

Standard Solutions Weigh 10.0 mg each of monosodium salt of 4-amino-1-naphthalenesulfonic acid, disodium salt of 7-hydroxy-1,3-naphthalenedisulfonic acid, disodium salt of 3-hydroxy-2,7-naphthalene- disulfonic acid, monosodium salt of 6-hydroxy-2-naphthalenesulfonic acid, and trisodium salt of 7-hydroxy-1,3,6-naphthalenetrisulfonic acid, dried previously in a vaccumed desiccator for 24 hours, dissolve in ammonium acetate solution (1.54 → 1,000), and make exactly 100 ml, respectively. Use these solutions as the standard stock solutions. Proceed as directed under the Coloring Matter Tests (Unreacted raw materials and

products of side reactions).

Procedure Determine the amount of each salt in the test solution as directed under the Coloring Matter Tests (Unreacted raw materials and products of side reactions) , and calculate the total amount.

Operating Conditions

Determination wavelength: 238 nm

Mobile phase: A. Ammonium acetate solution (1.54 → 1,000), B. Acetonitrile. Concentration gradient: Maintain 100% mobile phase A for 5 minutes, and change linearly the ratio of A:B from 100:0 to 70:30 for 50 minutes.

Food Red No. 40

Purity

(6) Lower sulfonated subsidiary colors Not more than 1.0%.

Test Solution Weigh accurately about 100 mg of Food Red No. 40, and dissolve in ammonium acetate solution (7.7 → 1,000) to make exactly 100 ml.

Standard Solutions Weigh 10.0 mg each of cresidine sulfonic acid azo ß -naphthol and cresidine azo Schaeffer's salt, dried previously in a vaccumed desiccator for 24 hours, dissolve each in ammonium acetate solution (7.7 → 1,000), and make exactly 100 ml. Use each solution as the standard stock solution. Proceed as directed under the Coloring Matter Tests (Subsidiary Colors).

Procedure Determine the amount of cresidine sulfonic acid azo ß -naphthol and cresidine azo Schaeffer's salt in the test solution as directed under the Coloring Matter Tests (Subsidiary Colors) and calculate the total amount.

Operating Conditions

Determination wavelength: 515 nm.

Mobile phase: A. Ammonium acetate solution (7.7 → 1,000), B. Methanol. Concentration gradient: Change linearly the ratio of A:B from 100:0 to 0:100 for 50 minutes.

(7) Higher sulfonated subsidiary colors Not more than 1.0%.

Test Solution Use 20 µl of the test solution prepared under test (6).

Standard Solutions Weigh 10.0 mg each of cresidine sulfonic acid azo G salt and cresidine sulfonic acid azo R salt, dried previously in a vacuumed desiccator for 24 hours, dissolve each in ammonium acetate solution (7.7 → 1,000), and make exactly 100 ml. Use each solution as the standard stock solution. Proceed as directed under the Coloring Matter Tests (Subsidiary

Colors).).

Procedure Perform Liquid Chromatography under the operating conditions specified in (6), as directed under the Coloring Matter Tests (Subsidiary Colors). Determine each amount of cresidine sulfonic acid azo G salt and cresidine sulfonic acid azo R salt in the test solution and calculate the total amount.

(8) Monosodium salt of 6-hydroxy-2-naphthalenesulfonic acid Not more than 0.3%.

Test Solution Use 20 µl of the test solution prepared under test (6).

Standard Solution Weigh 10.0 mg of monosodium salt of 6-hydroxy-2-naphthalenesulfonic acid, dried in a vacuumed desiccator for 24 hours, and dissolve in ammonium acetate solution (7.7 → 1,000) to make exactly 100 ml. Use this solution as the standard stock solution. Proceed as directed under the Coloring Matter Tests (Unreacted raw materials and products of side reactions).

Procedure Determine the amount of monosodium salt of 6-hydroxy-2-naphthalenesulfonic acid in the test solution as directed under the Coloring Matter Tests (Unreacted raw materials and products of side reactions).

Operating Conditions

Determination wavelength: 290 nm.

Mobile phase: A. Ammonium acetate solution (7.7→ 1,000), B. Methanol. Concentration gradient: Change linearly the ratio of A:B from 100:0 to 0:100 for 50 minutes.

(9) 4-Amino-5-methoxy-2-methylbenzenesulfonic acid Not more than 0.2%.

Test Solution Use 20 µl of the test solution prepared in test (6).

Standard Solution Weigh 10.0 mg of 4-amino-5-methoxy-2-methylbenzenesulfonic acid, dried in a vacuumed desiccator for 24 hours, dissolve in ammonium acetate solution (7.7 → 1,000) to make exactly 100 ml. Use this solution as the standard stock solution. Proceed as directed under the Coloring Matter Tests (Unreacted raw materials and products of side reactions).

Procedure Perform Liquid Chromatography under the operating conditions specified in test (8), as directed in the Coloring Matter Tests (Unreacted raw materials and products of side reactions) and determine the amount of 4-amino-5-methoxy-2-methylbenzenesulfonic acid in the test solution.

(10) Disodium salt of 6,6'-oxybis(2-naphthalenesulfonic acid) Not more than 1.0%.

Test Solution Use 20 µl of the test solution prepared under tests (6).

Standard Solution Weigh 10.0 mg of disodium salt of 6,6'-oxybis(2-naphthalenesulfonic

acid), dried in a vacuumed desiccator for 24 hours, dissolve in ammonium acetate solution (7.7 → 1,000) to make exactly 100 ml. Use this solution as the standard stock solution. Proceed as directed under to the Coloring Matter Tests (Unreacted raw materials and products of side reactions). .

Procedure Perform Liquid Chromatography under the operating conditions specified in test (8), as directed in the Coloring Matter Tests (Unreacted raw materials and products of side reactions and determine the amount of disodium salt of 6,6-oxybis(2-naphthalenesulfonic acid) in the test solution.

Food Red No. 40 Aluminum Lake

(Alurared AC Aluminum Lake)

Identification

(2) To 0.1 g of Food Red No. 40 Aluminum Lake, add 60 ml of aqueous ammonia (4 → 100), heat to boil, and concentrate until about 40 ml. Cool and centrifuge the liquid, and take the supernatant. To the residue add 10 ml of water, mix, and centrifuge again. Combine both supernatants, and add ammonium acetate solution (7.7 → 1,000) to make 100 ml. Measure 1 to 10 ml of this solution so that the absorbance to be measured will be within a range of 0.2- 0.7, and add ammonium acetate solution (7.7 → 1,000) to make exactly 100 ml. The solution exhibits absorption maximum at a wavelength of 497- 501 nm.

Purity

(6) Lower sulfonated subsidiary colors Not more than 1.0% (as the content is 85.0%).

Weigh 0.10 g of Food Red No. 40 Aluminum Lake, add 60 ml of aqueous ammonia (4 → 100), heat to boil, and concentrate to about 40 ml. Cool and centrifuge the liquid, and take the supernatant. To the residue add 10 ml of water, mix, and centrifuge again. Combine both supernatants, and add ammonium acetate solution (7.7 → 1,000) to make 100 ml. Use this solution as the test solution. Proceed as directed in Purity (6) for Food Red No. 40.

(11) Unsulfonated primary aromatic amines Not more than 0.01% as aniline (as the content is 85.0%).

Weigh 0.85 g as tar color, add 70 ml of ethyl acetate, allow to stand for 1 hour while shaking occasionally, and filter through a dry filter paper (5C) for quantitative analysis. Wash the residue on the filter paper three times with 10 ml of ethyl acetate each time, and combine the filtrate with the washings. Extract three times from this solution with 10 ml of diluted hydrochloric acid (3 → 100) each time, combine the extracts, add water to make exactly 50 ml.

Use this solution as the sample solution. Proceed as directed in Purity (11) for Food Red No. 40.

Food Red No. 102

Purity

(6) Unreacted raw materials and products of side reactions Not more than 0.5% as the total of monosodium salt of 4-amino-1-naphthalenesulfonic acid,
disodium salt of 7-hydroxy-1,3-naphthalenedisulfonic acid,
disodium salt of 3-hydroxy-2,7-naphthalenedisulfonic acid,
monosodium salt of 6-hydroxy-2-naphthalenesulfonic acid, and
trisodium salt of 7-hydroxy-1,3,6-naphthalenetrisulfonic acid.

Test Solution Weigh accurately about 100 mg of Food Red No. 102, dissolve in ammonium acetate solution (1.54 → 1,000), and make exactly 100 ml .

Standard Solutions Weigh 10.0 mg each of monosodium salt of 4-amino-1-naphthalenesulfonic acid, disodium salt of 7-hydroxy-1,3-naphthalenedisulfonic acid, disodium salt of 3-hydroxy-2,7-naphthalenedisulfonic acid, monosodium salt of 6-hydroxy-2-naphthalenesulfonic acid, and trisodium salt of 7-hydroxy-1,3,6-naphthalenetrisulfonic acid, dried previously in a vaccumed desiccator for 24 hours, dissolve in ammonium acetate solution (1.54 → 1,000), and make exactly 100 ml. Use each solution as the standard stock solution. Proceed as directed under the Coloring Matter Tests (Unreacted raw materials and products of side reactions).

Procedure Determine the amount of each salt as directed under the Coloring Matter Tests (Unreacted raw materials and products of side reactions), and calculate the total amount.

Operating Conditions

Determination wavelength: 238 nm.

Mobile phase: A. Ammonium acetate solution (1.54 → 1,000), B. Acetonitrile.

Concentration gradient: Maintain 100% mobile phase A for 5 minutes, and change linearly the ratio of A : B from 100 : 0 to 70 : 30 for 50 minutes.

Food Yellow No. 4

Purity

(6) Unreacted raw materials and products of side reactions Not more than 0.5% as the total of:

4-aminobenzenesulfonic acid,
5-hydroxy-1-(4-sulfophenyl)-3-pyrazolecarboxylic acid,
4-hydrazinobenzenesulfonic acid, and
disodium salt of 4,4'-(diazoamino)-dibenzenesulfonic acid.

Test Solution Weigh accurately about 100 mg of Food Red No. 2, and dissolve in ammonium acetate solution (1.54 → 1,000) to make exactly 100 ml.

Standard Solutions Weigh 10.0 mg each of 4-aminobenzenesulfonic acid, 5-hydroxy-1-(4-sulfophenyl)-3-pyrazolecarboxylic acid, 4-hydrazinobenzenesulfonic acid, and disodium salt of 4,4'-(diazoamino)-dibenzenesulfonic acid, dried previously in a vaccumed desiccator for 24 hours, and dissolve in ammonium acetate solution (1.54 → 1,000), and make exactly 100 ml. Use each solution as the standard stock solution. Proceed as directed under the Coloring Matter Tests (Unreacted raw materials and products of side reactions).

Procedure Determine the amount of each substance in the test solution as directed under the Coloring Matter Tests (Unreacted raw materials and products of side reactions), and calculate the total amount.

Operating Conditions

Determination wavelength:
4-aminobenzenesulfonic acid: 254 nm,
5-hydroxy-1-(4-sulfophenyl)-3-pyrazolecarboxylic acid: 254 nm,
4-hydrazinobenzenesulfonic acid: 254 nm, and
disodium salt of 4,4'-(diazoamino)-dibenzenesulfonic acid: 358 nm.
Mobile phase: A. Ammonium acetate solution (1.54 → 1,000), B. Acetonitrile.
Concentration gradient: Maintain 100% mobile phase A for 5 minutes, and change linearly the ratio of A : B from 100 : 0 to 70 : 30 for 50 minutes.

Food Yellow No. 5

Purity

(5) Subsidiary colors Not more than 5% as the total of
sulfanilic acid azo G salt color,
sulfanilic acid azo R salt color,
sulfanilic acid azo ß -naphtol color, and
aniline azo Shefer's salt color,
provided that colors other than sulfanilic acid azo G salt color is not more than 2%.

Test Solution Weigh accurately about 100 mg of Food Yellow No. 5, and dissolve in ammonium acetate solution (1.54 → 1,000, pH 8.0) to make exactly 100 ml.

Standard Solutions Weigh 10.0 mg each of sulfanilic acid azo G salt color, sulfanilic acid azo R salt color, sulfanilic acid azo ß -naphtol color, and aniline azo Shefer's salt color, dried previously in a vaccumed desiccator for 24 hours, and dissolve in ammonium acetate solution (1.54 → 1,000, pH 8.0) to make exactly 100 ml. Use each solution as the standard stock solution. Proceed as directed under the Coloring Matter Tests (Subsidiary Colors).

Procedure Determine the amount of each color in the test solution as directed under the Coloring Matter Tests (Subsidiary Colors) and calculate the total amount.

Operating Conditions

Detector: Detector for absorbances in the visible region (determination wavelength 482 nm).

Mobile phase: A. Ammonium acetate solution (1.54 → 1,000), B. Acetonitrile

Concentration gradient: Change linearly the ratio of A:B from 100:0 to 60:40 for 50 minutes.

(6) Unreacted raw materials and products of side reactions Not more than 0.5% as the total of:

4-aminobenzenesulfonic acid,
disodium salt of 7-hydroxy-1,3-naphthalenedisulfonic acid,
disodium salt of 3-hydroxy-2,7-naphthalenedisulfonic acid,
monosodium salt of 6-hydroxy-2-naphthalenesulfonic acid,
disodium salt of 6,6'-oxybis(2-naphthalenesulfonic acid), and
disodium salt of 4,4'-(diazoamino)-dibenzenesulfonic acid.

Test Solution Weigh accurately about 100 mg of Food Yellow No. 5, dissolve in ammonium acetate solution (1.54 → 1,000, pH 8.0) to make exactly 100 ml.

Standard Solutions Weigh 10.0 mg each of 4-aminobenzenesulfonic acid, disodium salt of 7-hydroxy-1,3-naphthalenedisulfonic acid, disodium salt of 3-hydroxy-2,7-naphthalenedisulfonic acid, monosodium salt of 6-hydroxy-2-naphthalenesulfonic acid, disodium salt of 6,6'-oxybis(2-naphthalenesulfonic acid), and disodium salt of 4,4'-(diazoamino)-dibenzenesulfonic acid, dried previously in a vaccumed desiccator for 24 hours, and dissolve in sodium hydroxide solution (4 → 1,000) for 4,4'-(diazoamino)-dibenzenesulfonic acid or in ammonium acetate solution (1.54 → 1,000, pH 8.0) for other salts. Use each solution as the standard stock solution. Proceed as directed under the Coloring Matter Tests (Unreacted raw materials and products of side reactions).

Procedure Determine the amount of each substance in the test solution as directed under the Coloring Matter Tests (Unreacted raw materials and products of side reactions), and

calculate the total amont.

Operating Conditions

Determination wavelength:

4-Aminobenzenesulfonic acid 232 nm,

Disodium salt of 7-hydroxy-1,3-naphthalenedisulfonic acid 232 nm,

Disodium salt of 3-hydroxy-2,7-naphthalenedisulfonic acid 232 nm,

Monosodium salt of 6-hydroxy-2-naphthalenesulfonic acid 232 nm,

Disodium salt of 6,6'-oxybis(2-naphthalenesulfonic acid) 232 nm, and

Disodium salt of 4,4'-(diazoamino)-dibenzenesulfonic acid 358 nm.

Mobile phase: A. Ammonium acetate solution (1.54 → 1,000), B. Acetonitrile.

Concentration gradient: Change linearly the ratio of A : B from 100 : 0 to 60 : 40 for 50 minutes.

Food Yellow No. 5 Aluminum Lake

(Sunset Yellow FCF Aluminum Lake)

Purity

(5) Subsidiary colors Not more than 5% as the total of

sulfanilic acid azo G salt color,

sulfanilic acid azo R salt color,

sulfanilic acid azo ß -naphtol color, and

aniline azo Shefer's salt color,

provided that colors other than sulfanilic acid azo G salt color is not more than 2% (as the content is 85.0%).

Weigh 0.1 g of Food Yellow No. 5 Aluminum Lake, add 60 ml of aqueous ammonia (4 → 100), heat to boil, and concentrate to about 40 ml. Cool and centrifuge the liquid, and take the supernatant. To the residue add 10 ml of water, mix, and centrifuge again. Combine both supernatants, and add ammonium acetate solution (7.7 → 1,000) to make exactly 100 ml. Use this solution as the test solution. Proceed as directed in Purity (5) for Food Yellow No. 5.

C. Reagents and solutions

Disodium Salt of 4,4'-(Diazo-amino)-dibenzenesulfonic Acid $C_{12}H_9N_3Na_2O_6S_2$

Weigh 10.0 mg of Disodium Salt of 4,4'-(Diazo-amino)-dibenzenesulfonic Acid, previously dried for 24 hours in a desiccator under a reduced pressure, add sodium hydroxide solution (4 →

1,000) to dissolve, and make exactly 100 ml. Use this solution as solution A. Measure exactly 10 ml of solution A, and add ammonium acetate solution (3 → 2,000) to make exactly 100 ml. Measure absorptions of this solution. The solution exhibits absorption maxima at wavelengths of 240 nm and 358 nm.

Purity Other aromatic compounds Measure exactly 10 ml of solution A, and add ammonium acetate solution (3 → 2,000) to make exactly 100 ml. Measure 20 µl of this solution, and perform Liquid Chromatography under the operating conditions specified in Purity (6) for Food Yellow No. 4 in the Monographs, JSFA-? . Only one peak is observed.

(End document)