Amendment to the Ordinance for Enforcement of the Food Sanitation Act and the Specifications and Standards for Foods, Food Additives, Etc.

The government of Japan will designate Copolymer of Vinylimidazole/Vinylpyrrolidone as an authorized food additive and establish compositional specifications and use standards for this additive.

Background

Japan prohibits the sale of food additives that are not designated by the Minister of Health, Labour and Welfare (hereinafter referred to as "the Minister") under Article 12 of the Food Sanitation Act (Act No. 233 of 1947; hereinafter referred to as "the Act"). In addition, when specifications or standards for food additives are stipulated in the Specifications and Standards for Foods, Food Additives, Etc. (Public Notice of the Ministry of Health and Welfare No. 370, 1959), Japan prohibits the sale of those additives unless they meet the specifications or the standards pursuant to Article 13 of the Act.

In response to a request from the Minister, the Committee on Food Additives of the Food Sanitation Council under the Pharmaceutical Affairs and Food Sanitation Council (hereinafter referred to as "the Committee") has discussed the adequacy of the designation of Copolymer of Vinylimidazole/Vinylpyrrolidone as a food additive. The conclusion of the Committee is outlined below.

Outline of conclusion

The Minister should designate Copolymer of Vinylimidazole/Vinylpyrrolidone as a food additive unlikely to cause harm to human health pursuant to Article 12 of the Act and should establish compositional specifications and use standards for this additive pursuant to Article 13 of the Act (see Attachment for the details).

Attachment

Copolymer of Vinylimidazole/Vinylpyrrolidone

PVI/PVP

ビニルイミダゾール・ビニルピロリドン共重合体

Standards for Use (draft)

Permitted for use in grape juice used for wine production and grape wine only.

Shall be used at not more than 0.50 g/L in grape wine as copolymer of vinylimidazole/vinylpyrrolidone. When Copolymer of Vinylimidazole/ Vinylpyrrolidone is used in grape juice used for wine production, this additive is considered to be used in grape wine.

The Copolymer of Vinylimidazole/Vinylpyrrolidone used shall be removed before the completion of the final product.

Compositional Specifications (draft)

Substance Name Copolymer of Vinylimidazole/Vinylpyrrolidone

Definition

Copolymer of Vinylimidazole/Vinylpyrrolidone is produced by polymerization of 1-vinylimidazole and 1-vinyl-2-pyrrolidone, with a ratio of 9：1, in the presence of crosslinking agent 1,3-divinylimidazolin-2-one at a level of less than 2%.

Content

Copolymer of Vinylimidazole/Vinylpyrrolidone, when calculated on the dried basis, contains 26.0–29.0% of nitrogen (N = 14.01).

Description Copolymer of Vinylimidazole/Vinylpyrrolidone occurs as a white to yellowish-white powder.

Identification Determine the absorption spectrum of Copolymer of Vinylimidazole/Vinylpyrrolidone as directed in the Disk Method under Infrared Spectrophotometry, and compare with the Reference Spectrum. Both spectra exhibit similar intensities of absorption at the same wavenumbers.

Purity

(1) Lead Not more than 2 μg/g of as Pb (2.0 g, Method 1, Control Solution: Lead Standard Solution 4.0 mL, Flame Method).

(2) Arsenic Not more than 2 μg/g as As (0.50 g, Method 3, Standard Color: Arsenic Standard Solution 2.0 mL, Apparatus B).

(3) Water-soluble substances Not more than 0.5%.

Weigh 10 g of Copolymer of Vinylimidazole/Vinylpyrrolidone, add it to 100 mL of water, shake, and allow to stand for 24 hours. Filter by suction through a membrane filter (2.5–3.0 μm pore size). Then, filter the filtrate by suction through a membrane filter (0.8 μm pore size), evaporate on a water bath to dryness, and weigh the residue.

(4) Acetic acid/ethanol-soluble substances Not more than 1%.

Weigh 1 g of Copolymer of Vinylimidazole/Vinylpyrrolidone, add 500 mL of a solution of 15 g of acetic acid and 50 mL of ethanol (95) in 500 mL of water, shake, and allow to stand for 24 hours. Filter the filtrate by suction through a membrane filter (2.5–3.0 μm pore size). Then, filter the filtrate by suction through a membrane filter (0.8 μm pore size), evaporate on a water bath to dryness, and weigh the residue.

(5) Organic impurities

Imidazole	Not more than 50 μg/g.
1,3-Divinylimidazolin-2-one	Not more than 2 μg/g.
1-Vinylimidazole	Not more than 10 μg/g.
1- Vinyl-2-pyrrolidone	Not more than 5 μg/g.
2-Pyrrolidone	Not more than 50 μg/g.

Test Solution Weigh 2.0 g of Copolymer of Vinylimidazole/Vinylpyrrolidone, add exactly 1 mL of internal standard solution, add 24 mL of acetone, and stir for 4 hours with a stirrer. Allow to stand, filter, and use the filtrate as the test solution.

Internal Standard Solution Use a solution of benzonitrile in acetone (1 in 4000).

Standard Solution Weigh separately 80 mg of imidazole, 3.2 mg of 1,3-divinylimidazolin-2-one, 16 mg of 1-vinylimidazole, 8.0 mg of 1-vinyl-2-pyrrolidone, and 80 mg of 2-pyrrolidone in a 200-mL volumetric flask, and add acetone to make 200 mL. Use the solution as the standard solution.

Control Solution Measure exactly 1 mL of the standard solution and 4 mL of the internal standard solution, and add acetone to make100 mL.

Procedure Analyze 1-μL portions of the test solution and the control solution by gas chromatography using the operating conditions given below. Determine the peak area ratio of each organic impurity to benzonitrile for the test solution and the control solution. The peak area ratio of each organic impurity to benzonitrile from the test solution is not greater than the peak area ratio of the corresponding organic impurity to benzonitrile from the control solution.

Operating Conditions

Detector: Nitrogen phosphorous detector.

Column: A fused silica tube (0.25 mm internal diameter and 30 m length) coated with a 0.5-µm thick layer of polyethylene glycol for gas chromatography.

Column temperature: Raise the temperature from 160°C to 210°C at a rate of 5°C/minute, and then maintain at 210°C for 7 minutes.

Injection port temperature: 220°C.

Detector temperature: 250°C.

Carrier gas: Helium.

Flow rate: Adjust so that the peak of benzonitrile appears 4–5 minutes after injection and the peak of each organic impurity is separated.

Injection method: Split.

Split ratio: 1 : 10.

Loss on Drying Not more than 5.0% (140°C, 1 hour).

Ash Not more than 0.3% (800°C, 6 hours).

Assay Weigh accurately about 10 mg of Copolymer of Vinylimidazole/Vinylpyrrolidone, and proceed as directed in the Semi-micro Kjeldahl Method under Nitrogen Determination. Determine the nitrogen content on the dried basis.

Reagents, Solutions, and Other Reference Materials

Benzonitrile C_7H_5N [100-47-0] A colorless, clear liquid.

Purity Related substances Prepare a test solution by dissolving 40 mg of benzonitrile in 25 mL of acetone. Prepare a control solution by diluting exactly 1 mL of the test solution with acetone to make exactly 50 mL. Analyze 1.0 µL each of the test solution and the control solution by gas chromatography using the operating conditions below. Continue the chromatography for two times the retention time of the main peak and measure the peak areas. The sum of the areas of all the peaks, excluding the main peak and solvent peaks, from the test solution is not greater than the area of the main peak from the control solution.

Operating conditions

Detector: Flame-ionization detector.

Column: A fused silica tube (0.25 mm internal diameter and 30 m length) coated with a 0.5-µm thick layer of polyethylene glycol for gas chromatography.

Column temperature: Raise the temperature from 160°C to 210°C at a rate of 5°C/minute, and then maintain at 210°C for 7 minutes.

Injection port temperature: 220°C.

Detector temperature: 250°C.

Carrier gas: Helium.

Flow rate: Adjust so that the peak of benzonitrile appears 4–5 minutes after injection.

Injection method: Split.

Split ratio: 1 : 10.

1,3-Divinylimidazolin-2-one $C_7H_{10}N_2O$ [13811-50-2]

Melting point 65–71°C.

Purity Related substances Prepare a test solution by mixing 6 mg of 1,3-divinylimidazolin-2-one with 2 mL of ethyl acetate. Prepare a control solution by diluting exactly 0.5 mL of the test solution with ethyl acetate to make exactly 10 mL. Analyze 2.0 µL each of the test solution and the control solution by gas chromatography using the operating conditions below. Continue the chromatography for 1.5 times the retention time of the main peak and measure the peak areas. The sum of the areas of all the peaks, excluding the main peak and solvent peaks, from the test solution is not greater than the area of the main peak from the control solution.

Operating conditions

Detector: Flame-ionization detector.

Column: A fused silica tube (0.25 mm internal diameter and 30 m length) coated with a 0.25-µm thick layer of 5% diphenyl/95% dimethylpolysiloxane for gas chromatography.

Column temperature: Maintain the temperature at 60°C for 5 minutes, raise to 280°C at a rate of 15°C/minute, and then maintain at 280°C for 1 minute.

Injection port temperature: 150°C.

Detector temperature: 250°C.

Carrier gas: Helium.

Flow rate: Adjust so that the peak of 1,3-divinylimidazolin-2-one appears 11–13 minutes after injection.

Injection method: Splitless.

Imidazole $C_3H_4O_2$ [288-32-4] White to light yellow crystals or powder. Very soluble in water and in methanol.

Content Not less than 98.0%.

Melting point 88–92°C.

Assay Weigh accurately about 0.1 g of imidazole, add 50 mL of acetic acid for nonaqueous titration, and titrate with 0.1 mol/L perchloric acid. To confirm the endpoint, use a potentiometer with a glass indicator electrode and silver-silver chloride reference electrode. A combined electrode can also be used for the indicator and reference electrodes. Separately, perform a blank test to make any necessary correction.

Each mL of 0.1 mol/L perchloric acid = 6.808 mg of $C_3H_4N_2$

2-Pyrrolidone C_4H_7NO [616-45-5] A colorless to pale yellow, clear liquid or white to pale yellow lumps or powder.

Melting point 22–27°C.

Purity Related substances Prepare a test solution by dissolving 1 g of 2-pyrrolidone in 10 mL of methanol. Prepare a control solution by diluting exactly 1 mL of the test solution with methanol to make exactly 50 mL. Analyze 1.0 µL each of the test solution and the control solution by gas chromatography using the operating conditions below. Continue the chromatography for two times the retention time of the main peak and measure the peak areas. The sum of the areas of all the peaks, excluding the main peak and solvent peaks, from the test solution is not greater than the area of the main peak from the control solution.

Operating conditions

Detector: Flame-ionization detector.

Column: A fused silica tube (0.53 mm internal diameter and 30 m length) coated with a 1.0-µm thick layer of polyethylene glycol for gas chromatography.

Column temperature: Maintain the temperature at 80°C for 1 minute, raise to 190°C at a rate of 10°C/minute, and then maintain at 190°C for 20 minutes.

Injection port temperature: A constant temperature of near 200°C.

Detector temperature: 250°C.

Carrier gas: Helium.

Flow rate: Adjust so that the peak of 2-pyrrolidone appears about 10 minutes after injection.

Injection method: Split.

Split ratio: 1 : 20.

1-Vinylimidazole $C_5H_6N_2$ [1072-63-5] A colorless to light yellow liquid.

Purity Related substances Prepare a test solution by dissolving 100 mg of 1-vinylimidazole in 25 mL of acetone. Prepare a control solution by diluting exactly 1 mL of the test solution with acetone to make exactly 50 mL. Analyze 1.0 µL each of

the test solution and the control solution by gas chromatography using the operating conditions below. Continue the chromatography for two times the retention time of the main peak and measure the peak areas. The sum of the areas of all the peaks, excluding the main peak and solvent peaks, from the test solution is not greater than the area of the main peak from the control solution.

Operating conditions

Detector: Flame-ionization detector.

Column: A fused silica tube (0.25 mm internal diameter and 30 m length) coated with a 0.5-µm thick layer of polyethylene glycol for gas chromatography.

Column temperature: Raise the temperature from 160°C to 210°C at a rate of 5°C/minute, and then maintain at 210°C for 7 minutes.

Injection port temperature: 220°C.

Detector temperature: 250°C.

Carrier gas: Helium.

Flow rate: Adjust so that the peak of 1-vinylimidazole appears 4–5 minutes after injection.

Injection method: Split.

Split ratio: 1 : 10.

Infrared Reference Spectrum

Copolymer of Vinylimidazole/Vinylpyrrolidone