The 259th Conference for Promotion of Food Import Facilitation Items and Contact Information

Item 1. Establishment of the Maximum Residue Limits for Agricultural Chemicals in Foods

The Food Sanitation Act authorizes the Ministry of Health, Labour and Welfare (MHLW) to establish residue standards (maximum residue limits, "MRLs") for pesticides, feed additives, and veterinary drugs (hereafter referred to as "agricultural chemicals") that may remain in foods. Any food for which standards are established pursuant to the provisions in Article 13, Paragraph 1 of the act is not permitted to be marketed in Japan unless it complies with the established standards.

On May 29, 2006, Japan introduced the Positive List System* for agricultural chemicals in food. All foods distributed in the Japanese marketplace are subject to regulation of the system.

The MHLW is going to modify or newly set MRLs in some commodities for the following substances, including modification of MRLs in some commodities that were provisionally set at the introduction of the Positive List System:

Pesticides : Flonicamid, Fluoxastrobin, Polyoxorim-zinc, Prothiofos
Feed additive : 3-Nitrooxypropanol

(See the *MRL table* for details; pages 5-15)

*: The aim of the positive list system on "agricultural chemicals" is to prohibit the distribution of any foods which contain agricultural chemicals at amounts exceeding a certain level (0.01 ppm) in the Japanese marketplace unless specific maximum residue limits (MRLs) have been set.

Item 2. Designation of a Food Additive and Revision of Use Standards

The government of Japan will designate Polyvinyl alcohol as a food additive.

Summary

Japan prohibits the sale of food additives that are not designated by the Minister of Health, Labour and Welfare ("the Minister") under Article 12 of the Food Sanitation Act (Act No. 233 of 1947; "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 of 1959) pursuant to Article 13 of the Act, the sale of those additives is

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prohibited unless they meet the specifications or the standards.
On July 19, 2023, the Committee on Food Additives of the Food Sanitation Council established under the Pharmaceutical Affairs and Food Sanitation Council ("the Committee") deliberated on Polyvinyl alcohol and concluded that it is appropriate for this substance to be designated by the Minister as a food additive that is unlikely to cause harm to human health pursuant to Article 12 of the Act. The Committee also concluded that it is appropriate for specifications and standards to be established for the additive pursuant to Article 13 of the Act. See *Attachment* for the details (pages 16-19).

The Ministry of Health, Labour and Welfare is taking necessary steps to designate Polyvinyl alcohol as a food additive and establish specifications and standards for the additive.

Notes

Polyvinyl alcohol was developed in Germany in 1924. In Japan and overseas, it is widely used as an industrial raw material for paper, fibers, and films; and used as a processing agent for adhesion, emulsification, and suspension. Polyvinyl alcohol is listed both in the Japanese Pharmaceutical Excipients 2018 and the Japanese Standards of Quasi-drug Ingredients, and is also used as an additive for cosmetics.

The European Union permits the use of polyvinyl alcohol in solid food supplements. Specifically, it is permitted only in capsule- and tablet-form products, excluding chewable forms. The maximum use level is 18,000 mg/kg.

In the United States, polyvinyl alcohol is approved as GRAS (generally recognized as safe). This substance is permitted for use in packaging materials that come into direct contact with food.

The Codex Alimentarius Commission lists polyvinyl alcohol as glazing agents and thickeners in the Codex General Standards for Food Additives (GSFA). It is permitted for use in food supplements (food category 13.6) at a maximum use level of 45,000 mg/kg.

Item 3. Revision of the Ministerial Order on Milk and Milk Products Concerning Compositional Standards, Etc.

Summary

Pursuant to the stipulation in Article 13, Paragraph (1) of the Food Sanitation Act, and from the viewpoint of public health, the Minister of Health, Labour and Welfare may establish the criteria for the methods of producing, processing, using, cooking, or preserving food or additives to be served for the purpose of marketing; or may establish standards for the ingredients of food or additives to be served for the purpose of marketing by hearing the opinions of the Pharmaceutical Affairs and Food Sanitation Council ("the PAFSC").

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Standards and criteria for milk, milk products, and food mostly made from milk and milk products are stipulated in the Ministerial Order on Milk and Milk Products Concerning Compositional Standards, Etc. ("the Ministerial Order on Milk, Etc.") pursuant to the stipulation in Article 13, Paragraph (1) of the Food Sanitation Act.

Based on the deliberation results of the expert committee of the Food Sanitation Council established under the PAFSC, the Ministry of Health, Labour and Welfare will amend the Ministerial Order on Milk, Etc. to establish standards and criteria for the cow's milk, etc. (i.e., cow's milk, composition modified milk, low fat milk, skimmed milk, processed milk, liquid infant formula, and milk drink) that are not required to be stored at 10°C or below, as given below:

1. Products that are sterilized, and then aseptically filled into containers/packages; and
2. Products that are filled into containers/packages, and then sterilized.

Amendments (draft)

Establishment of standards and criteria for the cow's milk, etc. that are not required to be stored at 10°C or below.

1. Products that are sterilized, and then aseptically filled into containers/packages

(1) Compositional standards
Viable bacterial cells: Negative (the test methods must conform to those for "food packed in containers and sterilized by pressurization and heating.")

(2) Criteria for manufacturing
The manufactures concerned must establish methods (i) and (ii) as given below, and must carry out the methods.

(i) a method to sterilize by heating at 120°C for 4 minutes, which provides sufficient effects to kill bacterial cells in such products that are derived from their raw materials and are viable in these products, or any other method providing a comparable or superior effec
(ii) a method to aseptically fill into pre-sterilized containers/packages

(3) Criteria for storage
The products concerned must be stored at a temperature not exceeding room temperature.

(4) Other criteria
The records on sterilization with automatic recording thermometers must be retained for a reasonable period considering the period until consumption.

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2. Products that are filled into containers/packages, and then sterilized

(1) Compositional standards (excluding liquid infant formula)
Viable bacterial cells: Negative (the test methods shall conform to those for "food packed in containers and sterilized by pressurization and heating.")

(2) Criteria for manufacturing (excluding liquid infant formula and milk drink)
The products concerned must be packed in preservation containers and sterilized by heating at 120°C for 4 minutes or sterilized using any other method providing a comparable or superior effect.

(3) Other criteria
The records on sterilization with automatic recording thermometers must be retained for reasonable period considering the period until consumption.

<The manner of submitting comments>
The MHLW will amend the existing standards and specifications for food as shown in this document. Please provide comments in writing by Tuesday December 19, 2023. After the given date, comments should be directed to the enquiry point in accordance with the WTO/SPS Agreement.

If you wish to request Japan to adopt the same limits as your country's MRLs, you are requested to submit data supporting your country's MRLs, such as risk assessment and residue data.

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Flonicamid

Commodity	MRL (draft) ppm	MRL (current) ppm
Wheat	5	5
Corn (maize, including pop corn and sweet corn)	0.4	0.4
Soybeans, dry	5	5
Beans, dry[1]	5	5
Peas	5	5
Broad beans	0.7	0.7
Other pulses[2]	5	5
Potato	0.3	0.3
Taro	0.2	0.2
Japanese yam (including Chinese yam)	0.2	0.2
Konjac	0.07	0.07
Other potatoes[3]	0.2	0.2
Sugar beet	0.6	0.6
Japanese radish, roots (including radish)	0.7	0.7
Japanese radish, leaves (including radish)	20	20
Turnip, roots (including rutabaga)	0.6	0.6
Turnip, leaves (including rutabaga)	20	20
Horseradish	0.6	0.6
Watercress	20	20
Chinese cabbage	20	20
Cabbage	2	2
Brussels sprouts	2	2
Kale	20	20
Komatsuna (Japanese mustard spinach)	20	20
Kyona	20	20
Qing-geng-cai	20	20
Cauliflower	2	2
Broccoli	5	5
Other cruciferous vegetables[4]	20	20
Burdock	0.6	0.6
Salsify	0.6	0.6
Chicory	0.6	0.6
Endive	3	3
Shungiku	15	15
Lettuce (including cos lettuce and leaf lettuce)	20	20
Other composite vegetables[5]	○ 5	2
Onion	0.3	0.3
Welsh onion (including leek)	3	3
Asparagus	2	2

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Commodity	MRL (draft) ppm	MRL (current) ppm
Carrot	0.6	0.6
Parsnip	0.6	0.6
Parsley	15	15
Celery	4	4
Mitsuba	5	5
Other umbelliferous vegetables[6]	2	2
Tomato	2	2
Pimiento (sweet pepper)	3	3
Egg plant	3	3
Other solanaceous vegetables[7]	5	5
Cucumber (including gherkin)	2	2
Pumpkin (including squash)	2	2
Oriental pickling melon (vegetable)	1	1
Water melon (whole commodity after removal of stems)	2	2
Melons	2	2
Makuwauri melon (whole commodity after removal of stems)	0.5	0.5
Other cucurbitaceous vegetables[8]	3	3
Spinach	20	20
Okra	10	10
Peas, immature (with pods)	2	2
Kidney beans, immature (with pods)	4	4
Green soybeans	5	5
Other vegetables[9]	3	3
Unshu orange (whole commodity)	2	2
Citrus natsudaidai, whole	2	2
Lemon	3	3
Orange (including navel orange)	2	3
Grapefruit	2	3
Lime	3	3
Other citrus fruits[10]	3	3
Apple	0.8	0.8
Japanese pear	0.8	0.8
Pear	0.8	0.8
Quince	0.8	0.8
Loquat (whole commodity after removal of stems)	0.8	0.8
Peach (whole commodity after removal of stems and stones but the residue calculated and expressed on the whole commodity without stems)	0.8	0.8
Nectarine	1	1
Apricot	2	2
Japanese plum (including prune)	0.3	0.3

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Commodity	MRL (draft) ppm	MRL (current) ppm
Mume plum	2	2
Cherry	2	2
Strawberry	2	2
Cranberry	2	2
Other berries[11]	2	2
Grape	6	6
Japanese persimmon	0.8	0.8
Mango	3	3
Other fruits[12]	0.8	0.8
Cotton seeds	0.7	0.7
Rapeseeds	2	2
Chestnut	0.1	0.1
Pecan	0.1	0.1
Almond	0.1	0.1
Walnut	0.1	0.1
Other nuts[13]	0.5	0.5
Tea	40	40
Hop	20	20
Other spices[14]	8	8
Other herbs[15]	20	20
Cattle, muscle	0.2	0.2
Pig, muscle	0.2	0.2
Other terrestrial mammals[16], muscle	0.2	0.2
Cattle, fat	0.05	0.05
Pig, fat	0.05	0.05
Other terrestrial mammals, fat	0.05	0.05
Cattle, liver	0.2	0.2
Pig, liver	0.2	0.2
Other terrestrial mammals, liver	0.2	0.2
Cattle, kidney	0.4	0.4
Pig, kidney	0.4	0.4
Other terrestrial mammals, kidney	0.4	0.4
Cattle, edible offal[17]	0.4	0.4
Pig, edible offal	0.4	0.4
Other terrestrial mammals, edible offal	0.4	0.4
Milk	0.2	0.2
Chicken, muscle	0.1	0.1
Other poultry[18], muscle	0.1	0.1
Chicken, fat	0.07	0.07
Other poultry, fat	0.07	0.07
Chicken, liver	0.1	0.1
Other poultry, liver	0.1	0.1

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Commodity	MRL (draft) ppm	MRL (current) ppm
Chicken, kidney	0.1	0.1
Other poultry, kidney	0.1	0.1
Chicken, edible offal	0.1	0.1
Other poultry, edible offal	0.1	0.1
Chicken eggs	0.2	0.2
Other poultry, eggs	0.2	0.2
Honey (including royal-jelly)	○ 0.05	

● : Commodities for which MRLs are to be lowered.

○ : Commodities for which MRLs are to be raised.

(*It should be noted that the residue definition (for agricultural / animal products) will be changed.)

NOTE: The residue definition for agricultural products and honey will be sum of Flonicamid, metabolite C 【*N*-(4-trifluoromethylnicotinoyl)glycine】 and metabolite E 【4-(trifluoromethyl)nicotinic acid】. The residue definition for animal products will be the sum of Flonicamid, metabolite D 【4-trifluoromethylnicotinamide】 and metabolite E 【4-(trifluoromethyl)nicotinic acid】, expressed as Flonicamid.

* The uniform limit 0.01 ppm will be applied to commodities not listed above.

1. "Beans, dry" includes butter beans, cowbeans (red beans), lentil, kidney beans, lima beans, pegia, sultani, sultapya and white beans.

2. "Other pulses" refers to all pulses, except soybeans (dry), beans (dry), peas, broad beans, peanuts (dry) and spices.

3. "Other potatoes" refers to all potatoes, except potato, taro, sweet potato, yam and konjac.

4. "Other cruciferous vegetables" refers to all cruciferous vegetables, except Japanese radish roots and leaves (including radish), turnip roots and leaves, horseradish, watercress, Chinese cabbage, cabbage, brussels sprouts, kale, komatsuna (Japanese mustard spinach), kyona, qing-geng-cai, cauliflower, broccoli and herbs.

5. "Other composite vegetables" refers to all composite vegetables, except burdock, salsify, artichoke, chicory, endive, shungiku, lettuce (including cos lettuce and leaf lettuce) and herbs.

6. "Other umbelliferous vegetables" refers to all umbelliferous vegetables, except carrot, parsnip, parsley, celery, mitsuba, spices and herbs.

7. "Other solanaceous vegetables" refers to all solanaceous vegetables, except tomato, pimiento (sweet pepper) and egg plant.

8. "Other cucurbitaceous vegetables" refers to all cucurbitaceous vegetables, except cucumber (including gherkin), pumpkin (including squash), oriental pickling melon (vegetable), watermelon, melons and makuwauri melon.

9. "Other vegetables" refers to all vegetables, except potatoes, sugar beet, sugarcane, cruciferous vegetables, composite vegetables, liliaceous vegetables, umbelliferous vegetables, solanaceous vegetables, cucurbitaceous vegetables, spinach, bamboo shoots, okra, ginger, peas (with pods, immature), kidney beans (with pods, immature), green soybeans, mushrooms, spices and herbs.

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10. "Other citrus fruits" refers to all citrus fruits, except unshu orange, citrus natsudaidai, citrus natsudaidai (peels), citrus natsudaidai (whole), lemon, orange (including navel orange), grapefruit, lime and spices.

11. "Other berries" refers to all berries, except strawberry, raspberry, blackberry, blueberry, cranberry and huckleberry.

12. "Other fruits" refers to all fruits, except citrus fruits, apple, Japanese pear, pear, quince, loquat, peach, nectarine, apricot, Japanese plum (including prune), mume plum, cherry, berries, grape, Japanese persimmon, banana, kiwifruit, papaya, avocado, pineapple, guava, mango, passion fruit, date and spices.

13. "Other nuts" refers to all nuts, except ginkgo nut, chestnut, pecan, almond and walnut.

14. "Other spices" refers to all spices, except horseradish, wasabi (Japanese horseradish) rhizomes, garlic, peppers chili, paprika, ginger, lemon peels, orange peels (including navel orange), yuzu (Chinese citron) peels and sesame seeds.

15. "Other herbs" refers to all herbs, except watercress, nira, parsley stems and leaves, celery stems and leaves.

16. "Other terrestrial mammals" refers to all terrestrial mammals, except cattle and pig.

17. "Edible offal" refers to all edible parts, except muscle, fat, liver and kidney.

18. "Other poultry" refers to all poultry, except chicken.

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Fluoxastrobin

Commodity	MRL (draft) ppm	MRL (current) ppm
Wheat	0.2	0.2
Barley	0.4	0.4
Corn (maize, including pop corn and sweet corn)	0.02	0.02
Other cereal grains[1]	2	2
Soybeans, dry	0.05	0.05
Beans, dry[2]	0.2	0.2
Peas	0.2	0.2
Broad beans	0.2	0.2
Other pulses[3]	0.2	0.2
Potato	0.01	0.01
Apple	1	1
Japanese pear	0.6	0.6
Pear	0.6	0.6
Cherry	○ 1	
Strawberry	2	2
Grape	2	2
Rapeseeds	0.7	0.7
Cattle, muscle	0.05	0.05
Pig, muscle	0.02	0.02
Cattle, fat	0.1	0.1
Pig, fat	0.03	0.03
Cattle, liver	0.2	0.2
Pig, liver	0.06	0.06
Cattle, kidney	0.2	0.2
Pig, kidney	0.06	0.06
Cattle, edible offal[4]	0.2	0.2
Pig, edible offal	0.06	0.06
Milk	0.03	0.03
Honey (including royal-jelly)	0.05	0.05

○ : Commodities for which MRLs are to be raised.

NOTE: The residue definition for agricultural products and honey will be sum of Fluoxastrobin and metabolite *Z*-isomer 【(*Z*)- {2-[6-(2-chlorophenoxy)-5-fluoropyrimidin-4-yloxy]phenyl) (5,6-dihydro-1,4,2-dioxazin-3-yl)methanone *O*-methyl oxime】, expressed as Fluoxastrobin. The residue definition for animal products is sum of Fluoxastrobin, metabolite *Z*-isomer, and metabolite M55 【6-(2-chlorophenoxy)-5-fluoro-4-pyrimidiol】, expressed as Fluoxastrobin. The residue definition will not be changed.

* The uniform limit 0.01 ppm will be applied to commodities not listed above.

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1. "Other cereal grains" refers to all cereal grains, except rice (brown rice), wheat, barley, rye, corn (maize) and buckwheat.

2. "Beans, dry" includes butter beans, cowbeans (red beans), lentil, kidney beans, lima beans, pegia, sultani, sultapya and white beans.

3. "Other pulses" refers to all pulses, except soybeans (dry), beans (dry), peas, broad beans, peanuts (dry) and spices.

4. "Edible offal" refers to all edible parts, except muscle, fat, liver and kidney.

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Polyoxorim-zinc

Commodity	MRL (draft) ppm	MRL (current) ppm
Cabbage	0.1	0.1
Lettuce (including cos lettuce and leaf lettuce)	0.1	0.1
Welsh onion (including leek)	0.1	0.1
Asparagus	○ 0.5	
Cucumber (including gherkin)	0.1	0.1
Apple	0.05	0.05
Honey (including royal-jelly)	○ 0.05	

○ : Commodities for which MRLs are to be raised.

NOTE: The residue definition of a polyoxorim-zinc is polyoxin D only.
The residue definition will not be changed.

* Not the uniform limit of 0.01 ppm but the regulation that foods shall not contain any antibiotics or chemically synthesized substances will be applied to the commodities for which MRLs are not listed above, since this substance is considered to be an antibiotic or chemically synthesized antibacterial substance.

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Prothiofos

Commodity	MRL (draft) ppm	MRL (current) ppm
Soybeans, dry	● 0.03	0.05
Beans, dry[1]	0.03	0.03
Peanuts, dry	0.02	0.02
Potato	0.02	0.02
Sweet potato	0.05	0.05
Sugar beet	0.5	0.5
Sugarcane	0.05	0.05
Cabbage	0.03	0.03
Burdock	● 0.08	0.1
Onion	0.1	0.1
Welsh onion (including leek)	2	2
Garlic	○ 0.1	0.03
Nira	○ 2	0.2
Multiplying onion (including shallot)	○ 2	
Other liliaceous vegetables[2]	○ 2	0.2
Carrot	○ 0.2	
Unshu orange (whole commodity)	○ 3	2
Japanese pear	● 0.1	0.2
Pear	● 0.1	0.2
Strawberry	0.3	0.3
Grape	● 0.8	1
Japanese persimmon	0.2	0.2
Chestnut	0.01	0.01
Tea	5	5
Other spices[3]	○ 15	10
Other herbs[4]	○ 2	
Cattle, muscle	0.01	
Pig, muscle	0.01	
Other terrestrial mammals[5], muscle	0.01	
Cattle, fat	0.01	
Pig, fat	0.01	
Other terrestrial mammals, fat	0.01	
Cattle, liver	0.01	
Pig, liver	0.01	
Other terrestrial mammals, liver	0.01	
Cattle, kidney	0.01	
Pig, kidney	0.01	
Other terrestrial mammals, kidney	0.01	
Cattle, edible offal[6]	0.01	

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Commodity	MRL (draft) ppm	MRL (current) ppm
Pig, edible offal	0.01	
Other terrestrial mammals, edible offal	0.01	
Milk	0.01	
Honey (including royal-jelly)	○ 0.05	

● : Commodities for which MRLs are to be lowered.
○ : Commodities for which MRLs are to be raised.

NOTE:The residue definition is Prothiofos only.
The residue definition will not be changed.

* The uniform limit 0.01 ppm will be applied to commodities not listed above.

1. "Beans, dry" includes butter beans, cowbeans (red beans), lentil, kidney beans, lima beans, pegia, sultani, sultapya and white beans.

2. "Other liliaceous vegetables" refers to all liliaceous vegetables, except onion, welsh onion (including leek), garlic, nira, asparagus, multiplying onion and herbs.

3. "Other spices" refers to all spices, except horseradish, wasabi (Japanese horseradish) rhizomes, garlic, peppers chili, paprika, ginger, lemon peels, orange peels (including navel orange), yuzu (Chinese citron) peels and sesame seeds.

4. "Other herbs" refers to all herbs, except watercress, nira, parsley stems and leaves, celery stems and leaves.

5. "Other terrestrial mammals" refers to all terrestrial mammals, except cattle and pig.

6. "Edible offal" refers to all edible parts, except muscle, fat, liver and kidney.

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3-Nitrooxypropanol

Commodity	MRL (draft) ppm	MRL (current) ppm
Cattle, muscle	0.01	
Cattle, fat	0.01	
Cattle, liver	0.01	
Cattle, kidney	0.01	
Cattle, edible offal[1]	0.01	
Milk	0.01	

NOTE: The residue definition is metabolite M2 【3-nitrooxypropionic acid】only.

* The uniform limit 0.01 ppm will be applied to commodities not listed above.

1. "Edible offal" refers to all edible parts, except muscle, fat, liver and kidney.

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Attachment

Polyvinyl Alcohol

Vinyl Alcohol Polymer

PVOH

PVA

ポリビニルアルコール

Standards for Use (draft)

Permitted for use only in foods not in conventional food form (such as capsules and tablets). Must be used at not more than 45 g/kg in food.

Compositional Specifications (draft)

Substance Name Polyvinyl Alcohol

Molecular Formula $(C_2H_3OR)_n$, R = H or $COCH_3$ (randomly distributed)

CAS Number [9002-89-5]

Definition Polyvinyl Alcohol is obtained by partial saponification of vinyl acetate polymer in the presence of an alkaline catalyst.

Description Polyvinyl Alcohol occurs as colorless to white or slightly yellowish white grains or powder. It has no odor.

Identification

(1) Dissolve 0.01 g of Polyvinyl Alcohol in 100 mL of water with warming, and allow to cool. To 5 mL of this solution, add one drop of iodine TS, mix it, and add 5 mL of boric acid solution (1 in 25). A blue color develops.

(2) Dissolve 0.5 g of Polyvinyl Alcohol in 10 mL water with warming, and allow to cool. Use this solution as the sample solution. Add one drop of iodine TS to 5 mL of the sample solution, and allow to stand. A dark red to blue color develops.

(3) Add 10 mL of ethanol (95) to 2 to 5 mL of the sample solution prepared in (2). A white, turbidity or flocculent precipitate is produced.

(4) Proceed as directed in the Disk Method under Infrared Spectrometry. Compare the spectrum obtained with the Reference Spectrum of Polyvinyl Alcohol. Both spectra exhibit similar intensities of absorption at the same wavenumbers.

Viscosity 4.8–5.8 mPa・s.

Weigh an amount of Polyvinyl Alcohol equivalent to 6.00 g on the dried basis, add 140 mL of water, and stir gently to disperse it. Further add water to make the contents 150 g. While constantly stirring, heat it to 90°C in a water bath, maintain it at this temperature for about 5 minutes, and stir for 1 hour at room temperature. Add water to replenish the evaporated water, then measure the kinematic viscosity (ν) of the resulting contents at 20°C as directed under Method 1 in Viscosity. Then, measure the density

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(ρ, g/mL) at 20°C, and obtain the viscosity (η, mPa・s) by the following:

$\eta = v\rho$

pH 5.0– 6.5 (1 g, water 25 mL).

Purity

(1) Acid value Not more than 3.0.

Weigh accurately about 10 g of Polyvinyl Alcohol, and transfer it into a 500-mL round-bottom flask containing 200 mL of water while stirring. Heat it under a reflex condenser in a water bath for 30 minutes while stirring. After cooling, transfer into a 250-mL volumetric flask. Wash the inside wall of the round-bottom flask with a small amount of water, add the washings to the volumetric flask, and add water to volume. Take exactly 50 mL of this solution, add 1 mL of phenolphthalein TS, titrate with 0.05 mol/L potassium hydroxide until the pink color persists for 15 seconds. Calculate the acid value (A) by the formula:

$$A = MW \times V \times 0.05 \times 5 / M$$

MW = molecular weight of potassium hydroxide (56.11),
V = volume (mL) of 0.05 mol/L potassium hydroxide consumed,
M = amount (g) of the sample taken.

(2) Ester value 125–153.

Weigh accurately about 1 g of Polyvinyl Alcohol into a 250-mL round-bottom flask, and add exactly 25 mL of 0.5mol/L ethanolic potassium hydroxide. Add 25 mL of water and a few glass beads in the flask, and heat under a reflux condenser in a water bath for 30 minutes while occasionally shaking. After cooling, add 1 mL of phenolphthalein TS, and immediately titrate with 0.5 mol/L hydrochloric acid. Separately, perform a blank test in the same manner and calculate the saponification value (S) by the formula:

$$S = MW \times (a - b) \times 0.5 / M$$

MW = molecular weight of potassium hydroxide (56.11),
a = volume (mL) of 0.5 mol/L hydrochloric acid consumed in the blank test,
b = volume (mL) of 0.5 mol/L hydrochloric acid consumed in the test,
M = amount (g) of the sample taken.

Calculate the ester value by the formula:

The ester value = $S - A$
S = the saponification value,
A = the acid value.

(3) Degree of hydrolysis 86.5–89.0 mol%.

Convert the saponification value (S) obtained in Purity (2) to the dried basis to obtain saponification (S_{db}) by the formula:

$$S_{db} = S \times 100 / (100 - \text{loss on drying } (\%))$$

Obtain the degree of hydrolysis by the formula:

$$\text{The hydrolysis degree} = 100 - [7.852 \times S_{db} / (100 - 0.07492 \times S_{db})]$$

S_{db} = the saponification value converted to the dried basis.

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(4) Water insoluble matter Not more than 0.1%.

Weigh accurately about 6 g of Polyvinyl Alcohol, add 140 mL of water, and stir gently to disperse it. Further add water to make the contents 150 g. While constantly stirring, heat to 90°C in a water bath, and maintain it at this temperature for about 5 minutes. Stir for 1 hour at room temperature. Add water to replenish the evaporated water. Filter the resulting liquid through a tarred 100-mesh stainless steel screen. Wash the residue on the screen with about 200 mL of water, and dry the screen with the residue at 105°C for 2 hours, and weigh the mass accurately. Then, obtain the amount of the residue.

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

(6) Methanol Not more than 1.0%.

Methyl acetate Not more than 1.0%.

Test Solution Weigh accurately about 0.2 g of Polyvinyl Alcohol into a 20-mL specified headspace vial, and add exactly 1 mL of the internal standard solution and 4 mL of dimethyl sulfoxide. Place a stirrer in the vial, stopper tightly, and immediately stir at 110°C for 60 minutes.

Internal Standard Solution Dissolve 0.5 g of 1-propanol in dimethyl sulfoxide to make exactly 100 mL.

Standard Solutions Weigh accurately about 5.0 g each of methanol and methyl acetate, add dimethyl sulfoxide to each to make exactly 50 mL, and refer to these solutions as standard solutions A_1 and A_2 respectively. Measure exactly 1 mL each of standard solutions A_1 and A_2, mix them, add dimethyl sulfoxide to make exactly 10 mL, and refer to this solution as standard solution B. Next, measure exactly 5 mL of standard solution B, add dimethyl sulfoxide to make exactly 50 mL, and refer to this as standard solution C. Measure exactly 1 mL, 4 mL, 8 mL, and 10 mL of standard solution C, place them into separate 20-mL volumetric flasks, add exactly 4 mL of the internal standard solution to each, and add dimethyl sulfoxide to volume. Measure exactly 5 mL each of these solutions, and place them in separate vials. Place a stirrer in each vial, stopper tightly, and immediately stir at 110°C for 60 minutes to prepare the standard solutions for the calibration curve.

Procedure Analyze the test solution and the four standard solutions by headspace gas chromatography using the following operating conditions. Determine the amounts of methanol and methyl acetate by the Internal Standard Method from the calibration curve.

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 polyethylene glycol for gas chromatography.

Column temperature: Maintain 40°C for 10 minutes, raise the temperature at a rate of 20°C/minute to 180°C, and maintain at 180°C for 4 minutes.

Injection port temperature: 180°C.

Detector temperature: 200°C.

Carrier gas: Nitrogen.

Flow rate: Adjust so that the peak of methyl acetate appears about 4 minutes after injection.

Injection method: Split.

Split ratio: 1 : 10.

Headspace sampler

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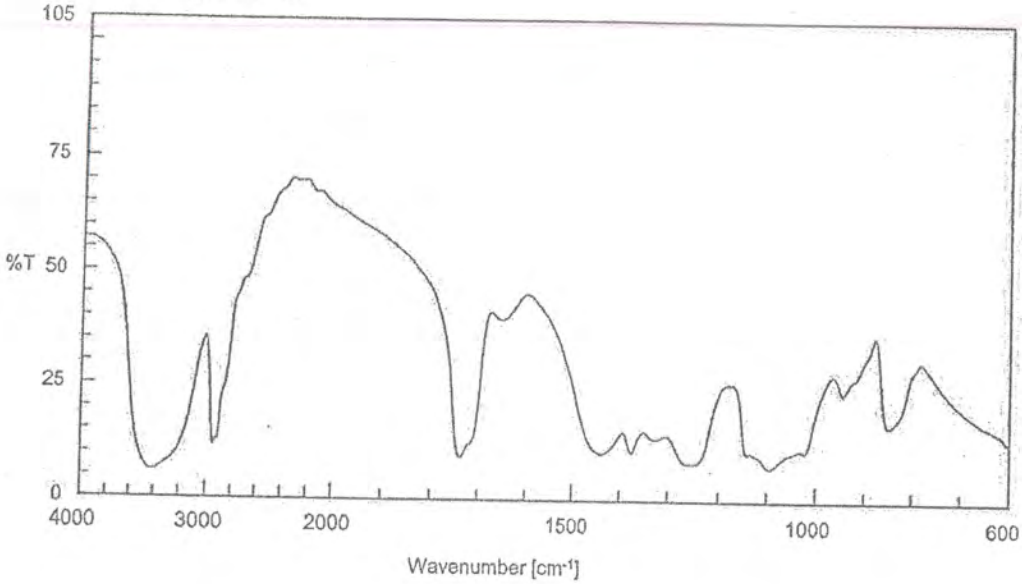
Equilibrium temperature in the vial: 110°C.
Equilibrium time in the vial: 30 minutes.
Injection volume: 1.0 mL.

Loss on Drying Not more than 5.0 % (1 g, 105°C, 3 hours).

Residue on Ignition Not more than 1.0% (1 g, 600 ± 50 °C, 1 hour).

Reference Spectrum

Polyvinyl Alcohol



Reagents, Solutions, and Other Reference Materials

Methyl Acetate CH_3COOCH_3 [K8382, special grade] [79-20-9]

0.05 mol/L Potassium Hydroxide

This solution contains 2.805 g of potassium hydroxide (KOH, molecular weight 56.11) per 1000 mL.

Dilute 1 mol/L potassium hydroxide with water (carbon dioxide-removed) to 20 times its original volume. Do not perform standardization, instead use the factor of 1 mol/L potassium hydroxide; or standardize as directed for 1 mol/L Potassium Hydroxide, using about 0.12–0.13 g of amidosulfuric acid (reference material).

Each mL of 0.05mol/L potassium hydroxide = 4.855mg of $HOSO_2NH_2$

The factor is calculated by the formula:

$f = m/(0.004855 \times V) \times A/100$

f = factor of 0.05mol/L potassium hydroxide,
m = amount (g) of amidosulfuric acid (reference material) taken,
A = content (%) of amidosulfuric acid (reference material),
V = volume (mL) of 0.05mol/L potassium hydroxide consumed.

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