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

On March 1, 2024, the government of Japan designated Polyvinyl Alcohol as an authorized food additive and established compositional specifications and standards for use of this additive.
See Attachment for the details.

Attachment

Polyvinyl Alcohol

Vinyl Alcohol Polymer

PVOH

PVA

ポリビニルアルコール

Standards for Use

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

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

$\eta = \nu\rho$

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:

$$\text{The ester value} = S - A$$

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.

(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

Equilibrium temperature in the vial: 110°C.

Equilibrium time in the vial: 30 minutes.

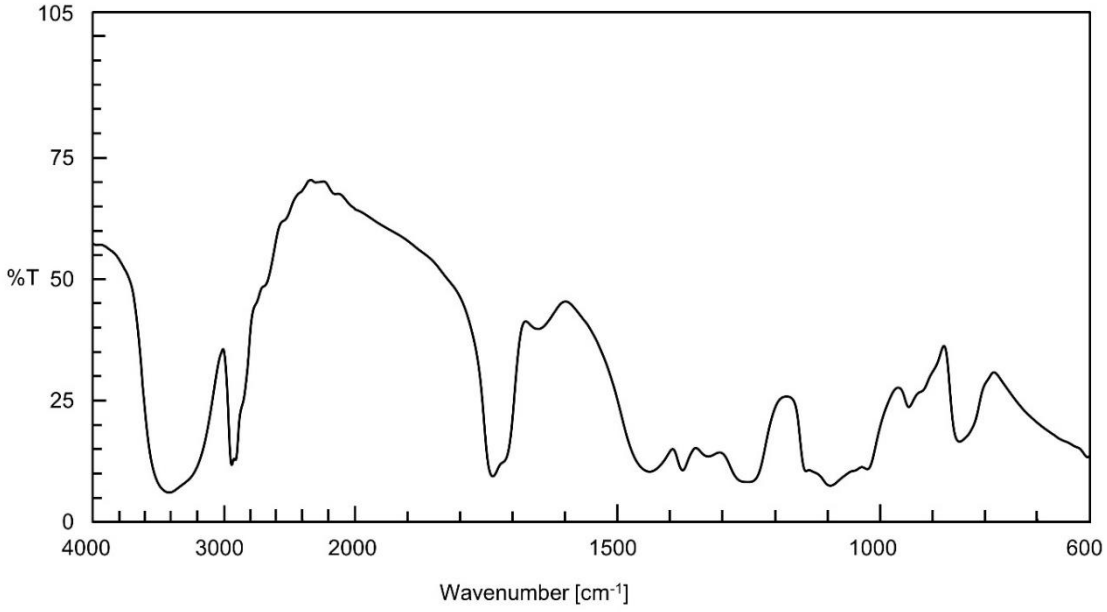
Injection volume: 1.0 mL.

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.