Polyvinylpyrrolidone

PVP; Povidone; Poly[1-(2-oxo-1-pyrrolidinyl)ethylene]

INS: 1201 CAS: [9003-39-8]

DESCRIPTION

Polyvinylpyrrolidone is a polymer of purified 1-vinyl-2-pyrrolidinone produced catalytically. It occurs as a white to tan powder, free from objectionable odor. It is soluble in water, in alcohol, and in chloroform, and is insoluble in ether. The pH of a 1 in 20 solution is between 3 and 7.

Functional Use in Foods Clarifying agent; separation/filtration aid; stabilizer; bodying agent; tableting aid; dispersant.

REQUIREMENTS

Labeling Indicate the K-value or the K-value range.

Identification

A. To 10 mL of a 1 in 50 solution of the sample add 20 mL of 1 *N* hydrochloric acid and 5 mL of potassium dichromate TS. An orange yellow precipitate is produced.

B. Add 5 mL of a 1 in 50 solution of the sample to 75 mg of cobalt nitrate and 300 mg of ammonium thiocyanate dissolved in 2 mL of water, mix, and then make the resulting solution acid with 2.7 *N* hydrochloric acid. A pale blue precipitate forms.

C. To 5 mL of a 1 in 200 solution of the sample add a few drops of iodine TS. A deep red color is produced.

Aldehydes (as acetaldehyde) Not more than 0.05%.

Heavy Metals (as Pb) Not more than 10 mg/kg.

Hydrazine Not more than 1 mg/kg.

K-Value Between 27 and 32 for the low-molecular-weight product, and between 81 and 97 for the high-molecular-weight product.

Nitrogen Not less than 11.5% and not more than 12.8%, calculated on the anhydrous basis.

Residue on Ignition Not more than 0.1%.

Unsaturation (as vinylpyrrolidinone) Not more than 0.1%.

Water Not more than 5.0%.

TESTS

Aldehydes

Phosphate Buffer Transfer 50.0 g of potassium pyrophosphate to a 500-mL volumetric flask, and dissolve in 400 mL of water. Adjust, if necessary, with 1 *N* hydrochloric acid to a pH of 9.0, dilute with water to volume, and mix.

Aldehyde Dehydrogenase Solution Transfer a quantity of lyophilized aldehyde dehydrogenase (Sigma A550, or equivalent) to 70 units to a glass vial, dissolve in 10.0 mL of water, and mix.

Note: This solution is stable for 8 h at 4°.

NAD Solution Transfer 40 mg of nicotinamide adenine dinucleotide (B-NAD, Grade III-C, from Sigma Chemical Co.) to a glass vial, dissolve in 10.0 mL of *Phosphate Buffer*, and mix.

Note: This solution is stable for 4 weeks at 4°.

Standard Solution Add about 2 mL of water to a glass weighing bottle, and weigh accurately. Add about 100 mg (about 0.13 mL) of freshly distilled acetaldehyde, and weigh accurately. Transfer this solution to a 100-mL volumetric flask. Rinse the weighing bottle with several portions of water, transferring each rinsing to the 100-mL volumetric flask. Dilute the solution in the 100-mL flask with water to volume, and mix. Store at 4° for about 20 h. Pipet 1 mL of this solution into a 100-mL volumetric flask, dilute with water to volume, and mix.

Test Preparation Transfer about 2 g of Polyvinylpyrrolidone, accurately weighed, to a 100-mL volumetric flask, dissolve in 50 mL of *Phosphate Buffer*, dilute with *Phosphate Buffer* to

volume, and mix. Insert a stopper into the flask, heat at 60° for 1 h, and cool to room temperature.

Procedure Pipet 0.5 mL each of the *Standard Solution*, the *Test Preparation*, and water (to provide the reagent blank) into separate 1-cm cells. Add 2.5 mL of *Phosphate Buffer* and 0.2 mL of *NAD Solution* to each cell. Cover the cells to exclude oxygen. Mix by inversion, and allow to stand for 2 to 3 min at 22° ± 2°. Determine the absorbances of the solutions at a wavelength of 340 nm, using water as the reference. Calculate the percentage of aldehydes, expressed as acetaldehyde, in the Polyvinylpyrrolidone taken by the formula

$$10(C/W)\{[(A_{U2} - A_{U1}) - (A_{B2} - A_{B1})]/[(A_{S2} - A_{S1}) - (A_{B2} - A_{B1})]\},$$

in which C is the concentration, in mg/mL, of acetaldehyde in the *Standard Solution*; W is the weight, in g, of Polyvinylpyrrolidone taken; $AU1$, $AS1$, and A_{B1} are the absorbances of the solutions obtained from the *Test Preparation*, the *Standard Solution*, and the water reagent blank, respectively, before addition of the *Aldehyde Dehydrogenase Solution*; and A_{U2}, A_{S2}, and A_{B2} are the absorbances of the solutions obtained from the *Test Preparation*, the *Standard Solution*, and the water reagent blank, respectively, after addition of the *Aldehyde Dehydrogenase Solution*. Not more than 0.05% is found.

Heavy Metals Prepare and test a 2-g sample as directed in *Method II* under the *Heavy Metals Test*, Appendix IIIB, using 20 μg of lead ion (Pb) in the control (*Solution A*).

Hydrazine

Salicylaldazine Standard Solution Dissolve 300 mg of hydrazine sulfate in 5 mL of water, add 1 mL of glacial acetic acid and 2 mL of a freshly prepared 20% (v/v) solution of salicylaldehyde in isopropyl alcohol, mix, and allow to stand until a yellow precipitate forms. Extract the mixture with two 15-mL portions of methylene chloride. Combine the methylene chloride extracts, and dry over anhydrous sodium sulfate. Decant the methylene chloride solution, and evaporate it to dryness. Recrystallize the residue of salicylaldazine from a mixture of warm toluene and methanol (60:40) with cooling. Filter, and dry the crystals in a vacuum. The crystals have a melting range of 213° to 219°, but the range between the beginning and end of melting is not to exceed 1°. Prepare a salicylaldazine solution containing 9.38 μg/mL of toluene.

Procedure Transfer 2.5 g to a 50-mL centrifuge tube, add 25 mL of water, and mix to dissolve. Add 500 μL of a 1 in 20 solution of salicylaldehyde in methanol, swirl, and heat in a water bath at 60° for 15 min. Allow to cool, add 2.0 mL of toluene, insert a stopper in the tube, shake vigorously for 2 min, and centrifuge. On a suitable thin-layer chromatographic plate coated with a 0.25-mm layer of dimethylsilanized chromatographic silica gel mixture, apply 10 μL of the clear upper toluene layer in the centrifuge tube and 10 μL of the *Standard Solution* of salicylaldazine. Allow the spots to dry, and develop the chromatogram in a solvent system of methanol and water (2:1) until the solvent front has moved about three-fourths of the length of the plate. Remove the plate from the developing chamber, mark the solvent front, and allow the solvent to evaporate. Locate the spots on the plate by examination under UV light at a wavelength of 365 nm: Salicylaldazine appears as a fluorescent spot having an R_f value of about 0.3, and the fluorescence of

any salicylaldazine spot from the test specimen is not more intense than that produced by the spot obtained from the *Standard Solution*.

K-Value The molecular weight of the sample is characterized by its viscosity in aqueous solution, relative to that of water, expressed as a K-value. Determine the relative viscosity, z, as follows: Transfer an accurately weighed portion of the as-is sample, equivalent to approximately 1 g on the anhydrous basis, to a 100-mL volumetric flask, dissolve in about 50 mL of water, dilute to volume, mix thoroughly, and allow to stand for 1 h, then pipet 15 mL of filtrate into a clean, dry Ubbelholde-type viscometer, and place the viscometer in a water bath maintained at 25° ± 0.2°. After allowing the viscometer and the sample solution to warm in the water bath for 10 min, draw the solution by means of very gentle suction up through the capillary until the meniscus is above the upper etched mark. Release suction and begin timing the flow through the capillary. After the meniscus reaches the upper etched mark, record the exact time when the meniscus reaches the lower etched mark, and calculate the flow time to the nearest 0.01 s. Repeat this operation until at least three readings are obtained. The readings must agree within 0.1 s; if not, repeat the determination with additional 15-mL portions of the sample solution after recleaning the viscometer with sulfuric acid–dichromate cleaning solution or with a suitable laboratory cleaning compound that will remove oils, greases, waxes, and other impurities. Calculate the average flow time for the sample solution, and then obtain the flow time in a similar manner for 15 mL of water for the same viscosity pipet. Calculate the relative viscosity, z, of the sample by dividing the average flow time of the sample solution by that of the water sample, and then calculate the K-value by the formula

$$[\sqrt{300c \log z + (c + 1.5c \log z)^2} + 1.5c \log z - c]/(0.15c + 0.003c^2),$$

in which c is the weight, in g, on the anhydrous basis, of the sample in each 100.0 g of solution, and z is as defined above.

Nitrogen Determine as directed in *Method II* under *Nitrogen Determination*, Appendix IIIC, using a 100-mg sample. In the wet-digestion step, omit the use of hydrogen peroxide, and use 5 g of a mixture of potassium sulfate, cupric sulfate, and titanium dioxide (33:1:1) instead of the potassium sulfate and cupric sulfate mixture (10:1). Heat until a clear, light green solution is obtained, heat for an additional 45 min, and continue as directed, beginning with "Cautiously add 20 mL of water, cool, then. . . ," except use 70 mL of water instead of 20.

Residue on Ignition Weigh accurately about 2 g, and proceed as directed in the general method, Appendix IIC.

Unsaturation Dissolve about 10 g of the sample, accurately weighed, in 80 mL of water in a 125-mL, round-bottom flask, add 1.0 g of sodium acetate, mix, and begin titrating with 0.1 *N* iodine. When the iodine color no longer fades, add 3 additional mL of the titrant, and allow the solution to stand for 5 to 10 min. Add starch TS, and titrate the excess iodine with 0.1 N sodium thiosulfate. Perform a blank determination (see *General Provisions*), using the same volume of 0.1 *N* iodine, accurately measured, as was used for the sample. Each mL of 0.1 *N* iodine is equivalent to 5.556 mg of vinylpyrrolidinone.

Water Determine by the *Karl Fischer Titrimetric Method*, Appendix IIB.

Packaging and Storage Store in tight containers.