

POLYVINYLPIRROLIDONE

Prepared at the 30th JECFA (1986), published in FNP 37 (1986) and in FNP 52 (1992). Metals and arsenic specifications revised at the 63rd JECFA (2004). An ADI of 0-50 mg/kg bw was established at the 30th JECFA (1986)

SYNONYMS

Povidone, PVP; INS No. 1201

DEFINITION

Chemical names

Polyvinylpyrrolidone, poly-[1-(2-oxo-1-pyrrolidinyl)- ethylene]

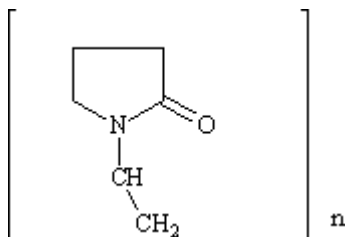
C.A.S. number

9003-39-8

Chemical formula

$(C_6H_9NO)_n$

Structural formula



Formula weight

Lower molecular weight range product: about 40 000
Higher molecular weight range product: about 360 000

Assay

Not less than 12.2% and not more than 13.0% of Nitrogen (N) on the anhydrous basis

DESCRIPTION

White to tan powder; supplied in two molecular weight forms; the molecular weight value is an average molecular weight for the two forms

FUNCTIONAL USES Clarifying agent, stabilizer, bodying agent, tableting adjunct, dispersing agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Soluble in water, in ethanol and in chloroform; insoluble in ether

pH (Vol. 4)

3.0 - 7.0 (5% soln)

Precipitate formation

To 5 ml of a 1 in 50 solution of the sample add 5 ml of dilute hydrochloric acid TS, 5 ml of water and 2 ml of 1 in 10 solution of potassium dichromate. A yellow precipitate forms.

Add 5 ml of a 1 in 50 solution of the sample to 75 mg of cobalt nitrate and 0.3 g of ammonium thiocyanate dissolved in 2 ml of water, mix and acidify with dilute hydrochloric acid TS. A pale blue precipitate forms.

To 5 ml of a 1 in 50 solution of the sample add 1 ml of 25% hydrochloric acid and 5 ml of 5% barium chloride solution and 1 ml of 5% phosphomolybdotungstic acid solution. A voluminous white precipitate is formed which becomes gradually blue on standing in daylight. (Note: The blue colouration on exposure to light distinguishes polyvinylpyrrolidone from polyethylene oxide adducts which give similar precipitates with the same reagents but which retain their white colour in light).

PURITY

<u>Water</u> (Vol. 4)	Not more than 5% (Karl Fischer Method)
<u>Relative viscosity</u>	Between 1.188 and 1.325 for lower molecular weight product, and between 3.225 and 5.662 for higher molecular weight product See description under TESTS.
<u>Total ash</u> (Vol. 4)	Not more than 0.02% Test 10 g of the sample
<u>Aldehyde</u>	Not more than 0.2% (as acetaldehyde) See description under TESTS
<u>Monomer content</u>	Not more than 1% (as vinylpyrrolidone) See description under TESTS
<u>Hydrazine</u>	Not more than 1 mg/kg See description under TESTS
<u>Lead</u> (Vol. 4)	Not more than 2 mg/kg Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, "Instrumental Methods."

TESTS

PURITY TESTS

<u>Relative viscosity</u>	Transfer an accurately weighed portion of the sample, equivalent to 1 g of anhydrous polyvinylpyrrolidone, into a 250 ml conical flask, and calculate the amount of water to be added to make a 1% solution. Allow the mixture to stand at room temperature, with occasional swirling, until solution is complete, and then allow to stand for 1 h. longer. Filter through a dry sintered-glass filter funnel of coarse porosity, then pipet 10 ml of the filtrate into a Cannon-Fenske viscometer, or equivalent, and place the viscometer in a water bath maintained at $25 \pm 0.05^\circ$. After allowing the sample solution and pipet 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 formed from 3 to 4 mm above the upper etched mark. Release the vacuum, and, when the meniscus reaches the upper etched mark, begin timing the flow through the capillary. Record the exact time when the meniscus reaches the lower etched mark, and calculate the flow time to the nearest 0.1 sec. Repeat this operation until at least three readings are obtained. The
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readings must agree within 0.3 sec.; if not, repeat the determination with additional 10 ml portions of the sample solution after recleaning the viscosity pipet with sulfuric acid-dichromate cleaning solution.

Calculate the average flow time for the sample solution, and then obtain the average flow time in similar manner for 10 ml of filtered water for the same viscosity pipet. Calculate the relative viscosity of the sample by dividing the average flow time for the sample solution by that of the water sample.

Aldehyde

Transfer about 10 g of the sample, accurately weighed and dissolved in 300 ml of water, into a 1000 ml round-bottom flask containing 80 ml of 25% sulfuric acid, reflux for about 45 min. under a water-cooled condenser, and then distil about 100 ml into a receiver containing 20 ml of 1 N hydroxylamine hydrochloride previously adjusted to pH 3.1. Titrate the contents of the receiver with 0.1 N sodium hydroxide to a pH of 3.1, using a pH meter. Perform a blank determination and make any necessary correction. Each ml of 0.1 N sodium hydroxide is equivalent to 4.405 mg of C₂H₄O.

Monomer content

Dissolve about 4 g of the sample, accurately weighed, in 30 ml of water in a 125 ml round-bottom flask, add 0.5 g of sodium acetate, mix and begin titrating with 0.1 N iodine. When the iodine colour no longer fades, add additional 3.0 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, 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 5.56 mg of vinylpyrrolidone.

Hydrazine

Transfer 2.5 g of the sample into a 50-ml centrifuge tube, add 25 ml 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 a Standard solution of salicylaldazine in toluene containing 9.38 µg per ml. 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 ultraviolet 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 (1 ppm of hydrazine).

Preparation of Salicylaldazine Standard:

Dissolve 300 mg of hydrazine sulfate in 5 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 vacuum. The crystals have a melting range of 213° to 214°.

**METHOD OF
ASSAY**

Determine as directed under *Nitrogen Determination* in Volume 4, using about 1 g of the sample, accurately weighed.