INSOLUBLE POLYVINYLPYRROLIDONE


SYNONYMS Crospovidone, cross linked polyvidone, insoluble PVP, polyvinylpolypyrrolidone, cross linked homopolymer of 1-ethenyl-2-pyrrolidone, insoluble cross linked homopolymer of N-vinyl-1-pyrrolidone, INS No. 1202

DEFINITION A poly-[1-(2-oxo-1-pyrrolidinyl) ethylene], cross-linked in a random fashion produced by the polymerization of N-vinyl-2-pyrrolidone in the presence of either caustic catalyst or N,N'-divinyl-imidazolidone; due to its insolubility in all common solvents the molecular weight range is not amenable to analytical determination.

Assay Not less than 11.0% and not more than 12.8% nitrogen calculated on the anhydrous basis

DESCRIPTION White hygroscopic powder with a faint, non-objectable odour

FUNCTIONAL USES Colour stabilizer, colloidal stabilizer, clarifying agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Insoluble in water, ethanol and ether

Absorption of iodine Add 0.1 ml of 0.1 N iodine to a suspension of 1 g of the sample in 10 ml of water and shake for 30 sec. There should be no blue colouration on shaking up with 1 ml of starch TS.

PURITY

Water (Vol. 4) Not more than 6% (Karl Fischer Method)

pH (Vol. 4) 5 - 8 (1% w/v aqueous suspension)

Sulfated ash (Vol. 4) Not more than 0.4%

Test 2 g of the sample (Method I)

Water-soluble matter Not more than 1.5%

Transfer about 25 g, accurately weighed, to a 500-ml flat bottom flask, add 225 ml of water and a 5 cm magnetic stirring bar, and place on a combination heater-stirrer. Attach a water-cooled condenser, and reflux gently, with stirring, for 20 h. Allow the slurry to cool, transfer to a 250-ml volumetric flask, with the aid of 25 ml of water, add water to volume, and mix. Allow the bulk of the solids to settle for about 15 min, decant the liquid into centrifuge tubes, and centrifuge until clear. Typically, at least 1 h at 12,000 rpm is required. Transfer 50.0 ml of the clear supernatant liquid to a
tared 250 ml beaker, evaporate, and dry to constant weight in a forced air oven at about 90°C. Calculate the percentage of water soluble substances by the formula 500 W / G, where W is the weight, in g, of the residue and G is the weight, in g, of the test sample taken.

**Free N-vinyl-pyrrolidone**

Not more than 0.1%

Suspend 4.0 g of the sample with 30 ml of water, stir 15 min., pass through a sintered glass filter of 9-15 µm (= type G 4) in a 250-ml conical flask. Wash the residue with 100 ml of water, add 500 mg of sodium acetate to the combined filtrates, and titrate with 0.1 N iodine until the colour of iodine no longer fades. Add an additional 3.0 ml of 0.1 N iodine, allow to stand for 10 min, and titrate the excess iodine with 0.1 N sodium thiosulfate, adding 3 ml of starch TS as the end-point is approached. Perform a blank determination. Not more than 0.72 ml of iodine is consumed, corresponding to not more than 0.1% vinylpyrrolidone.

**Free N,N'-divinyl imidazolidone**

Not more than 2 mg/kg

See description under TESTS

**Zinc (Vol. 4)**

Not more than 25 mg/kg

Test 2 g of the sample, accurately weighed

**Lead (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**

**Free N,N'-divinyl imidazolidone**

**Principle**

Free N,N'-divinylimidazolidone migrating from insoluble PVP into a solvent (acetone) is determined by capillary column gas chromatography.

**Internal standard solution**

Dissolve 100 mg of heptanoic acid nitrile (acenathic acid nitrile) weighed out to within an accuracy of 0.1 mg in 500 ml of acetone.

**Preparation of the sample**

Weigh out about 2-2.5 g of the polymer to within an accuracy of 0.2 mg into a 50 ml Erlenmeyer flask. By means of a pipette, add 5 ml of internal standard solution. Subsequently, run in about 20 ml of acetone. Shake the mixture for 4 h or let it equilibrate for at least 15 h and analyze the supernatant solution by gas chromatography.

**Calibration solution**

Weigh out about 25 mg of N,N'-Divinylimidazolidone with an accuracy of 0.2 mg into a flask and make up to 100 ml with acetone. By means of a pipette, transfer 2.0 ml of this solution into another 50 ml calibration flask, make up to 50 ml with acetone. Transfer 2 ml of this solution to another
flask, add 5 ml of the internal standard solution (see above) and make up to 25 ml with acetone.

Gas chromatography condition
Column: Capillary (fused silica) "DB-Wax" (cross-linked Carbowax 20 ml), length 30 m, i. d. 0.25 mm, film thickness 0.5 µm.
Column oven temperature: Programmed, 140° - 240°, 4°/min
Injector: Split injector, 220°; split effluent 30 ml
Detector: Thermionic detector (optimized according to manufacturers instructions), 250°.
Carrier gas: Helium, 1 bar (overpressure)
Amount injected: 1 µl of supernatant solution of sample or calibration solution.

Procedure
Obtain a reliable determination of the calibration factor for the specific conditions of analysis by means of repetitive injections of the calibration solution. Analyze the sample. The content of N,N'-divinylimidazolidone in insoluble PVP shall be not more than 2 mg/kg.

Calculation of the calibration factor, f

\[
f = \frac{W_D \times A_{St}}{W_{St} \times A_D}
\]

where

- \( W_D \) = amount of N,N'-divinylimidazolidione taken (mg)
- \( W_{St} \) = amount of internal standard (mg)
- \( A_{St} \) = area of peak of internal standard
- \( A_D \) = area of peak for N,N'-divinylimidazolidone

Calculation of the content of N,N'-divinylimidazolidone, \( C_D \)

\[
C_D = \frac{1000 \times f \times A_D \times W_{St}}{A_{St} \times W_S} \text{ mg/kg}
\]

where

- \( f \) = calibration factor
- \( A_D \) = area of peak for N,N'-divinylimidazolidone
- \( W_{St} \) = amount of internal standard added to the sample (mg)
- \( A_{St} \) = area of peak of internal standard
- \( W_S \) = amount of sample taken (g)

METHOD OF ASSAY

Determine the nitrogen content according to Kjeldahl with a modified digestion. Place about 0.1 g, accurately weighed, in the digestion flask of the apparatus. Add 1 g of a powdered mixture of 10 parts of potassium sulfate and 1 part of cupric sulfate, and wash down any adhering material from the neck of the flask with a fine jet of water. Add 7 ml of sulfuric acid, rinsed down the wall of the flask, then, while swirling the flask, add 1 ml of 30% hydrogen peroxide cautiously down the side of the flask (Do not add hydrogen peroxide during the digestion). Repeat the addition of 1 ml of 30% hydrogen peroxide (usually 3 to 6 times) until a clear, light green solution is obtained on heating the mixture. Heat for an additional 4 h. Cautiously add to the digestion mixture 20 ml of water and proceed to the
steam distillation as directed under *Nitrogen Determination (Kjeldahl Method)* (see Volume 4)