NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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Replacing GB 31630-2014

National food safety standard - Food additive - Polyvinyl alcohol

食品安全国家标准 食品添加剂 聚乙烯醇

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Table of Contents

1 Scope

1 The document applies to the food additive polyvinyl alcohol prepared by the ethylene process and by the acetylene process.

2 Structural formula and molecular formula

2.1 The structural formula is given as a figure, in which R stands for H or COCH3, randomly distributed.

2.2 The molecular formula is (C2H3OR)n, in which R stands for H or COCH3, randomly distributed.

3 Technical requirements

3.1 The sensory requirements are set in Table 1. The colour is transparent, white or pale yellow and the physical state is a granular powder. The test method is to place a suitable quantity of the sample in a 50 mL beaker and observe its colour and physical state under natural light.

3.2 The physico-chemical requirements are set in Table 2, which gives for each characteristic a limit and the test method. Loss on drying is not to exceed 5.0% by mass, residue on ignition 1.0% by mass and water-insoluble matter 0.1% by mass. The fraction passing a 0.150 mm test sieve is to be not less than 99.0% by mass. Methanol and methyl acetate are each not to exceed 1.0% by mass. The acid value, expressed as KOH, is not to exceed 3.0 mg/g, while the esterification value, also expressed as KOH, is to be 125 mg/g to 153 mg/g. The degree of hydrolysis is to be 86.5% to 89.0% by mass and the viscosity of a 4% solution at 20 °C is to be 4.8 mPa.s to 5.8 mPa.s. All of these are determined by the sub-clauses of Annex A named in the table. Lead is not to exceed 2.0 mg/kg, determined according to GB 5009.75 or GB 5009.12.

A Annex A (normative) Test methods

A A warning states that some of the reagents used in the test methods are toxic, corrosive and flammable and that the operator is to work with care. Splashes on the skin are to be washed off at once with water and serious cases treated immediately, and open flames are not to be used for heating when flammable materials are handled.

A.1 Unless otherwise stated the reagents are of analytical grade and the water is grade three water as defined in GB/T 6682. Standard volumetric solutions, standard solutions for the determination of impurities, preparations and products are prepared according to GB/T 601, GB/T 602 and GB/T 603, and where no solvent is stated the solutions referred to are aqueous.

A.2 Identification comprises six tests. The pH of a solution of 4.0 g of sample in 100 mL of water, heated until dissolution is complete and cooled to room temperature, is to be 5.0 to 6.5. The infrared spectrum of the sample, dispersed in potassium bromide, is to agree with Figure B.1 of Annex B. In colour reaction A, one drop of iodine solution and several drops of boric acid solution are added to 5 mL of a solution prepared from about 0.01 g of sample and the solution is to turn blue. In colour reaction B, one drop of iodine solution is added to 5 mL of a solution prepared from about 1.0 g of sample and the solution is to change from deep red to blue within 1 min to 3 min. In the precipitation reaction, 10 mL of 95% ethanol is added to 5 mL of the solution from colour reaction B and turbidity or a flocculent precipitate is to appear. In the solubility test, about 1.0 g of sample is to dissolve completely in 10 mL to 30 mL of water on stirring for not less than 5 min, and about 1.0 g of sample is to dissolve completely in 30 mL to 100 mL of 95% ethanol on stirring for not less than 5 min.

A.3 Loss on drying is determined on about 5 g of sample weighed to the nearest 0.0002 g in a weighing bottle previously dried to constant mass, the sample being dried for 3 h at 105 °C plus or minus 2 °C and weighed after cooling to room temperature in a desiccator. The mass fraction is calculated by formula (A.1) from the mass of sample and weighing bottle before and after drying and from the sample mass. The mean of parallel determinations is reported, and under repeatability conditions the ratio of the absolute difference between two independent results to their arithmetic mean is not to exceed 2%.

A.4 The residue on ignition is determined on about 2 g of sample weighed to the nearest 0.0002 g in a porcelain crucible previously brought to constant mass at 800 °C plus or minus 25 °C, the sample being heated gently on a hot plate until dry and fully carbonised and then ignited to constant mass in a furnace at 800 °C plus or minus 25 °C. The mass fraction is calculated by formula (A.2) from the mass of the residue and crucible, the mass of the crucible and the sample mass. Under repeatability conditions the absolute difference between two independent results is not to exceed 0.2%.

A.5 Water-insoluble matter is determined on about 10 g of sample weighed to the nearest 0.0002 g, dissolved in 100 mL of freshly boiled water in a 500 mL beaker, stirred and

cooled for 1 h, filtered through a sintered glass crucible of 100 micrometre to 160 micrometre pore size previously dried to constant mass at 105 °C plus or minus 2 °C, and washed three to five times with hot water at 65 °C to 75 °C before drying to constant mass. The mass fraction is calculated by formula (A.3). Under repeatability conditions the absolute difference between two independent results is not to exceed 0.05%.

A.6 The fraction passing the 0.150 mm test sieve is determined on about 100 g of sample weighed to the nearest 0.01 g and sieved on a mechanical shaker at an amplitude of 2 mm and a frequency of 150 strokes per minute for 30 min plus or minus 10 s, the material on the receiving pan being weighed; a manual method may also be used. The mass fraction is calculated by formula (A.4) from the mass of the material passing the sieve and the sample mass. Under repeatability conditions the absolute difference between two independent results is not to exceed 0.1%.

A.7 Methanol and methyl acetate are determined by headspace gas chromatography with flame ionisation detection and acetone as internal standard. The reference headspace conditions are a vial equilibration temperature of 80 °C for 30 min, a sample loop temperature of 80 °C and a transfer line temperature of 100 °C. The reference chromatographic conditions are a capillary column of 6% cyanopropylphenyl and 94% polydimethylsiloxane, 30.0 m by 0.530 mm with a film thickness of 3.00 micrometres; high purity nitrogen carrier gas of purity not less than 99.999% at 2 mL/min; automatic injection without split; an inlet temperature of 200 °C; a detector temperature of 250 °C; and an oven programme starting at 40 °C held for 8 min, then rising at 10 °C/min to 150 °C and held for 2 min. A methanol and methyl acetate solution containing 2.5 g/L of each is measured, and a test solution prepared from 0.5 g of sample and 2.00 mL of internal standard solution is measured in the same way. The mass fraction is calculated by formula (A.5) from the concentration of the calibration solution, the volume transferred to the vial, the sample mass and the peak areas of the analyte and of acetone in the two measurements. Under repeatability conditions the absolute difference between two independent results is not to exceed 0.05%.

A.8 The acid value, expressed as KOH, is determined by titration with potassium hydroxide standard volumetric solution of 0.05 mol/L, whose preparation, standardisation against potassium hydrogen phthalate dried to constant mass at 105 °C to 110 °C and calculation by formula (A.6) are given in full. A 10.0 g test portion, weighed to the nearest 0.001 g, is heated and stirred for 30 min in 200 mL of carbon dioxide free water in a round-bottomed flask under a reflux condenser in a boiling water bath, cooled with continued stirring, made up to 250 mL, and a 50.00 mL aliquot is titrated with phenolphthalein indicator to a pink colour persisting for 15 s. A blank is run alongside. The result is calculated by formula (A.7) from the two titration volumes, the concentration of the potassium hydroxide solution, the molar mass of potassium hydroxide taken as 56.10 g/mol, the volumetric flask capacity, the sample mass and the aliquot volume. Under repeatability conditions the absolute difference between two independent results is not to exceed 0.3%.

A.9 The esterification value, expressed as KOH, and the degree of hydrolysis are determined by saponification. A test portion of 1.00 g plus or minus 0.01 g is refluxed for 30 min in a boiling water bath with 25.00 mL of potassium hydroxide ethanol solution, 25 mL of water and five to seven glass beads, then cooled and titrated at once with hydrochloric acid standard volumetric solution of 0.5 mol/L to the disappearance of the pink phenolphthalein colour, a blank being run alongside. The saponification value is calculated by formula (A.8) from the blank and sample titration volumes, the concentration of the hydrochloric acid solution, the molar mass of potassium hydroxide and the sample mass; under repeatability conditions the absolute difference between two independent results is not to exceed 0.3%. The esterification value is obtained by formula (A.9) as the saponification value less the acid value. The degree of hydrolysis is obtained by formula (A.10) from the saponification value on a dry basis together with the conversion factors 7.84 and 0.075, and the saponification value on a dry basis is obtained by formula (A.11) from the saponification value and the loss on drying.

A.10 The viscosity of a 4% solution at 20 °C is determined with an Ostwald capillary viscometer or an Ubbelohde capillary viscometer of hard glass, using a standard viscosity liquid of kinematic viscosity 5 mm2/s. A test portion equivalent to 6.00 g on a dry basis, weighed to the nearest 0.001 g, is added to about 140 mL of water under slow stirring, the stirring is increased once dissolution is complete but without forming bubbles, the solution is heated to 90 °C in a thermostatic water bath and held for 5 min, stirred for a further 1 h after the heating is stopped, made up to a solution mass of 150 g and stirred until uniform, then filtered under suction through a sintered glass crucible into a conical flask and cooled. The efflux times of the sample solution and of the standard viscosity liquid are measured at 20 °C plus or minus 0.5 °C. The viscosity is calculated by formula (A.12) from the kinematic viscosity and efflux time of the standard liquid, the density of the sample solution at 20 °C determined according to 4.3.3 of GB/T 4472-2011 and the efflux time of the sample solution. The mean of two determinations is reported to one decimal place. The lettering of the two viscometers is explained in Figures A.1 and A.2, which give the measuring marks, the capillary, the bulbs and the tubes with their diameters and tolerances.

B Annex B (normative) Reference infrared spectrum of polyvinyl alcohol

B The annex carries the reference infrared spectrum of polyvinyl alcohol as Figure B.1.

C Annex C (normative) Reference gas chromatogram for the determination of methanol and methyl acetate

C The annex carries the reference gas chromatogram for the determination of methanol and methyl acetate as Figure C.1, in which peak 1 is methanol, peak 2 acetone and peak 3 methyl acetate.