Asparagine Tablets

Asparagine Tablets contain not less than 90.0% and not more than 110.0% of the labelled amount of asparagine ($C_4H_8N_2O_3$).

Description White tablets.

Identification A quantity of the powdered tablets complies with the tests for Identification under Asparagine.

Other requirements Comply with the general requirements for tablets 〈*0101*〉.

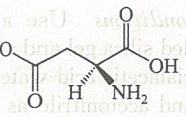
Assay Weigh accurately and powder finely 10 tablets. Accurately weigh a quantity of the powdered tablets equivalent to about 0.15 g of Asparagine (calculated as $C_4H_8N_2O_3$). Carry out the Assay described under Asparagine.

Category As described under Asparagine.

Strength 0.25 g (Calculated as $C_4H_8N_2O_3$)

Storage Preserve in tightly closed containers, stored in a cool place and protected form light.

Aspartic Acid



$C_4H_7NO_4$ 133.10 [56-84-8]

Aspartic Acid is L-2-aminobutanedioic acid. It contains not less than 98.5% of $C_4H_7NO_4$, calculated on the dried basis.

Description White or almost white crystals or a crystalline powder; odourless.

Slightly soluble in water; insoluble in ethanol; soluble in dilute hydrochloric acid solution or in sodium hydroxide solution.

Specific optical rotation +24.0° to +26.0°, a solution containing about 80 mg per ml in 6 mol/L hydrochloric acid solution 〈*0621*〉.

Identification (1) Transfer 10 mg of the substance being examined to a 25 ml volumetric flask, dissolve in 2 ml of ammonia TS, dilute with water to volume, mix well and use as the test solution. Prepare the reference solution in the same way using aspartic acid CRS instead of the substance being examined. Carry out the method described under Other amino acids. The colour and position of the principal spot in the chromatogram obtained with the test solution correspond to those of the principal spot obtained with the reference solution.

(2) The infrared absorption spectrum 〈*0402*〉 is concordant with the reference spectrum (IR Album No. 913).

Acidity Dissolve 0.10 g in 20 ml of water, pH 2.0-4.0 〈*0631*〉.

Transmittance of solution Dissolve 1.0 g in 10 ml of 1 mol/L hydrochloric acid solution, the transmittance at 430 nm is not less than 98.0% 〈*0401*〉.

Chlorides Carry out the limit test for chlorides 〈*0801*〉, using 0.30 g. Any opalescence produced is not more pronounced than that of a reference solution prepared using 6.0 ml of sodium chloride standard solution (0.02%).

Sulfates Carry out the limit test for sulfates 〈*0802*〉, using 1.0 g. Any opalescence produced is not more pronounced than that of a reference solution prepared using 2.0 ml of potassium sulfate standard solution (0.02%).

Ammonium Carry out the limit test for ammonium 〈*0808*〉, using 0.10 g. Any colour produced is not more intense than that of a reference solution prepared using 2.0 ml of ammonium chloride standard solution (0.02%).

Other amino acids Carry out the method for thin-layer chromatography 〈*0502*〉.

Test solution Transfer 0.10 g of the substance being examined to a 10 ml volumetric flask, dissolve in 2 ml of concentrated ammonia solution, dilute with water to volume, and mix well.

Reference solution Transfer 1 ml of the test solution, accurately measured, to a 200 ml volumetric flask, dilute with water to volume and mix well.

System suitability solution Transfer 10 mg of aspartic acid CRS and 10 mg of glutamic acid CRS to a 25 ml volumetric flask, dissolve in 2 ml of ammonia TS, dilute with water to volume, and mix well.

Chromatographic conditions Use silica gel G as the coating substance and a mixture of glacial acetic acid-water-butanol (1 : 1 : 3) as the mobile phase.

Procedure Apply separately to the plate 5 μl of test solution, reference solution and system suitability solution. Develop over a path of 15 cm. Allow the plate to dry in air, spray with a mixture of butanol-2 mol/L acetic acid solution (95 : 5) containing 0.2% ninhydrin, heat at 105℃ for 15 minutes until spots appear and examine immediately.

System suitability requirements The chromatogram obtained with the reference solution shows a clear spot, and system suitability solution shows two clearly separated principal spots.

Limits Any spot other than the principal spot in the chromatogram obtained with the test solution is not more intense than the principal spot obtained with the reference solution (0.5%).

Loss on drying When dried at 105℃ for 3 hours, loses not more than 0.2% of its weight 〈*0831*〉.

Residue on ignition Not more than 0.1% 〈*0841*〉, using 1.0 g.

Iron Carry out the limit test for iron 〈*0807*〉, using 1.0 g. Any colour produced is not more intense than that of a reference solution prepared using 1.0 ml of iron standard solution (0.001%).

Heavy metals Carry out the limit test for heavy metals 〈*0821*, *method 2*〉, using the residue obtained in the test for Residue on ignition; not more than 0.001%.

Arsenic Dissolve 2.0 g in 23 ml of water, add 5 ml of hydrochloric acid, carry out the limit test for arsenic 〈*0822*, *method 1*〉; not more than 0.0001%.

Pyrogens Complies with the test for pyrogens 〈*1142*〉, using 10 ml of a solution of 10 mg per ml in Sodium Chloride Injection per kg of the rabbit's weight (for parenteral use).

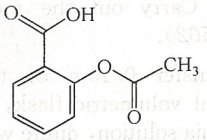
Assay Dissolve about 0.1 g, accurately weighed, in 5 ml of anhydrous formic acid, add 30 ml of glacial acetic acid. Carry out the method for potentiometric titration 〈*0701*〉,

titrate with perchloric acid (0.1 mol/L) VS. Perform a blank determination and make any necessary correction. Each ml of perchloric acid (0.1 mol/L) VS is equivalent to 13.31 mg of $C_4H_7NO_4$.

Category Amino acid.

Storage Preserve in tightly closed containers.

Aspirin



$C_9H_8O_4$ 180.16 [50-78-2]

Aspirin is 2-(acetyloxy) benzoic acid. It contains not less than 99.5% of $C_9H_8O_4$.

Description White crystals or a white crystalline powder; odourless or with a faint acetic acid odour; gradually hydrolyses in contact with moisture to form salicylic acid and acetic acid.

Freely soluble in ethanol; soluble in chloroform and in ether; slightly soluble in water and in anhydrous ether; soluble in solutions of sodium hydroxide and in sodium carbonate with decomposition.

Identification (1) To about 0.1 g add 10 ml of water, boil and cool. Add 1 drop of ferric chloride TS; a violet colour is produced.

(2) To about 0.5 g add 10 ml of sodium carbonate TS, boil for 2 minutes and cool. Add excessive dilute sulfuric acid; a white precipitate is produced and an odour of acetic acid is perceptible.

(3) The infrared absorption spectrum 〈*0402*〉 is concordant with the reference spectrum (IR Album No. 5).

Clarity of solution A solution of 0.50 g in 10 ml of sodium carbonate TS previously heated to about 45℃ is clear.

Free salicylic acid Carry out the method for high performance liquid chromatography 〈*0512*〉. Prepare the solutions immediately before use.

Solvent 1% glacial acetic acid solution in methanol.

Test solution Weigh accurately 0.1 g in a 10 ml volumetric flask, add a quantity of the solvent, shake to dissolve and dilute to volume, mix well.

Reference solution Weigh accurately 10 mg of salicylic acid CRS in a 100 ml volumetric flask, add a quantity of the solvent, shake to dissolve and dilute to volume, mix well. Transfer accurately 5 ml into a 50 ml volumetric flask, dilute to volume with the solvent and mix well.

Chromatographic conditions Use a column packed with octadecylsilane bonded silica gel and a mixture of acetonitrile-tetrahydrofuran-glacial acetic acid-water (20 : 5 : 5 : 70) as the mobile phase. The detection wavelength is 303 nm. The injection volume is 10 μl.

System suitability requirements The number of theoretical plates is not less than 5000, calculated with reference to the peak of salicylic acid. The resolution factor between the peaks of aspirin and salicylic acid complies with the related requirement.

Procedure Separately inject the test solution and reference solution in the chromatograph, record the chromatograms.

Limits Calculate the content of salicylic acid with respect to the peak area obtained in the chromatogram by the external standard method. The content of salicylic acid is not more than 0.1%.

Readily carbonizable substances Carry out the limit test for readily carbonizable substances 〈*0842*〉, using 0.5 g; any colour produced is not more intense than that of a reference preparation (mix 0.25 ml of standard cobalt chloride CS, 0.25 ml of standard potassium dichromate CS, and 0.40 ml of standard copper sulfate CS, with water to produce 5 ml).

Related substances Carry out the method for high performance liquid chromatography 〈*0512*〉.

Solvent 1% glacial acetic acid solution in methanol.

Test solution Weigh accurately 0.1 g in a 10 ml volumetric flask, add a quantity of the solvent, shake to dissolve and dilute to volume, mix well.

Reference solution Transfer accurately 1 ml of the test solution, to a 200 ml volumetric flask, dilute to volume with the solvent, mix well.

Salicylic acid reference solution Proceed as described in Reference Solution under Free Salicylic Acid.

Sensitivity solution Transfer 1 ml of the reference solution, accurately measured, to a 10 ml volumetric flask, dilute with the solvent to volume, and mix well.

Chromatographic conditions Use a column packed with octadecylsilane bonded silica gel and a mixture of acetonitrile-tetrahydrofuran-glacialacetic acid-water (20 : 5 : 5 : 70) as mobile phase A, and acetonitrile as mobile phase B. Carry out the gradient elution as described below. The detection wavelength is 276 nm. The injection volume is 10 μl.

Time(minute)	Mobile phase A(%)	Mobile phase B(%)
0	100	0
60	20	80

System suitability requirements The retention time of the aspirin peak is about 8 minutes. The resolution factor between the peaks of aspirin and salicylic acid complies with the related requirement. In the chromatogram of the sensitivity solution, the signal-to-noise ratio of the principal peak is greater than 10.

Procedure Inject separately the test solution, the reference solution, the sensitivity solution and the salicylic acid reference solution into the chromatograph and record the chromatograms.

Limits In the chromatogram of the test solution, disregard any peak with an area less than the area of principal peak obtained with the sensitivity solution, the sum of the areas of all the peaks other than the principal peak and salicylic acid peak is not greater than the peak area of principal peak in the chromatogram obtained with the reference solution (0.5%).

Loss on drying When dried in vacuum over phosphorous pentoxide to constant weight at 60℃, loses not more than 0.5% of its weight 〈*0831*〉.

Residue on ignition Not more than 0.1% 〈*0841*〉.

Heavy metals Dissolve 1.0 g in 23 ml of ethanol, add 2 ml of acetate BS (pH 3.5), carry out the limit test for heavy metals 〈*0821*, *method 1*〉: not more than 0.001%.

Assay Dissolve about 0.4 g, accurately weighed, in 20 ml