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Aspirin Capsules

» Aspirin Capsules contain not less than 93.0 percent and not more than 107.0 percent of the labeled amount of aspirin ($C_9H_8O_4$).

[Note—Capsules that are enteric-coated or the contents of which are enteric-coated meet the requirements for *Aspirin Delayed-Release Capsules*.]

Packaging and storage—Preserve in tight containers.

USP Reference standards ⟨11⟩—

USP Aspirin RS

Identification—

A: Heat about 100 mg of the Capsule contents with 10 mL of water for several minutes, cool, and add 1 drop of ferric chloride TS: a violet-red color is produced.

B: Shake a quantity of the contents of Capsules, equivalent to about 500 mg of aspirin, with 10 mL of alcohol for several minutes. Centrifuge the mixture. Pour off the clear supernatant and evaporate it to dryness. Dry the residue in vacuum at 60° for 1 hour: the residue responds to *Identification* test *B* under *Aspirin*.

Dissolution ⟨711⟩—

Medium: 0.05 M acetate buffer, prepared by mixing 2.99 g of sodium acetate trihydrate and 1.66 mL of glacial acetic acid with water to obtain 1000 mL of solution having a pH of 4.50 ± 0.05; 500 mL.

Apparatus 1: 100 rpm.

Time: 30 minutes.

Procedure—Determine the amount of $C_9H_8O_4$ dissolved from UV absorbances at the wavelength of the isosbestic point of aspirin and salicylic acid at 265 ± 2 nm of filtered portions of the solution under test, suitably diluted with *Medium,* if necessary, in comparison with a Standard solution having a known concentration of USP Aspirin RS in the same *Medium.* [Note—Prepare the Standard solution at the time of use. An amount of alcohol not to exceed 1% of the total volume of the Standard solution may be used to bring the Reference Standard into solution prior to dilution with *Medium.*]

Tolerances—Not less than 80% *(Q)* of the labeled amount of $C_9H_8O_4$ is dissolved in 30 minutes.

Uniformity of dosage units ⟨905⟩: meet the requirements.

Limit of free salicylic acid—

Ferric chloride-urea reagent—Dissolve by swirling, without the aid of heat, 60 g of urea in a mixture of 8 mL of ferric chloride solution (6 in 10) and 42 mL of 0.05 N hydrochloric acid. Adjust the resulting solution, if necessary, with 6 N hydrochloric acid to a pH of 3.2.

Standard preparation—Transfer 75.0 mg of salicylic acid, previously dried over silica gel for 3 hours and accurately weighed, to a 100-mL volumetric flask, add chloroform to volume, and mix. Transfer 10.0 mL of this solution to a second 100-mL volumetric flask, dilute with chloroform to volume, and mix. Transfer 10.0 mL of this last solution to a 50-mL volumetric flask containing 10 mL of methanol, 2 drops of hydrochloric acid, and 10 mL of a 1 in 10 solution of glacial acetic acid in ether, dilute with chloroform to volume, and mix.

Chromatographic column (see *Chromatography ⟨621⟩*)—Proceed as directed under *Column Partition Chromatography*, packing a chromatographic tube with two segments of packing material. The lower segment is a mixture of 1 g of *Solid Support* and 0.5 mL of 5 M phosphoric acid, and the upper segment is a mixture of 3 g of *Solid Support* and 2 mL of freshly prepared *Ferric chloride-urea reagent*.

Test preparation—Weigh accurately a portion of the contents of the Capsules, as determined by the *Assay*, equivalent to 100 mg of aspirin, mix with 10 mL of chloroform by stirring for 3 minutes, and then transfer to the chromatographic column with the aid of a few mL of chloroform. Pass 50 mL of chloroform through the column, rinse the tip of the chromatographic tube with chloroform, and discard the eluate. Prepare as a receiver a 50-mL volumetric flask containing 10 mL of methanol and 2 drops of hydrochloric acid, and elute any salicylic acid from the column by passing 10 mL of a 1 in 10 solution of glacial acetic acid in ether that has been recently saturated with water, followed by 30 mL of chloroform. Dilute the eluate with chloroform to volume, and mix.

Procedure—Concomitantly determine the absorbances of the solutions in 1-cm cells at the wavelength of maximum absorbance at about 306 nm, with a suitable spectrophotometer, using as the blank a solvent mixture of the same composition as that used for the *Standard preparation:* the absorbance of the *Test preparation* does not exceed that of the *Standard preparation* (0.75%, calculated on the labeled aspirin content).

Assay—[Note—In this assay use chloroform recently saturated with water.]

Standard preparation—Transfer about 50 mg of USP Aspirin RS, accurately weighed, to a 50-mL volumetric flask, add 0.5 mL of glacial acetic acid, add chloroform to volume, and mix. Transfer 5.0 mL of this solution to a 100-mL volumetric flask, dilute with a 1 in 100 solution of glacial acetic acid in chloroform to volume, and mix. The concentration of USP Aspirin RS is about 50 µg per mL.

Chromatographic column—Proceed as directed under *Column Partition Chromatography* (see *Chromatography ⟨621⟩*), packing a chromatographic tube with a mixture of 3 g of *Solid Support* and 2 mL of freshly prepared sodium bicarbonate solution (1 in 12).

Assay preparation—Remove, as completely as possible, the contents of not fewer than 20 Capsules, and weigh accurately. Mix the combined contents, and transfer an accurately weighed quantity of the powder, equivalent to about 50 mg of aspirin, to a 50-mL volumetric flask containing 1 mL of a 1 in 50 solution of hydrochloric acid in methanol, add chloroform to volume, and mix. Transfer 5.0 mL of this solution to the column, wash with 5 mL and then with 25 mL of chloroform, and discard the washings. Elute into a 100-mL volumetric flask with about 10 mL of a 1 in 10 solution of glacial acetic acid in chloroform and then with about 85 mL of a 1 in 100 solution of glacial acetic acid in chloroform, dilute with the latter solvent to volume, and mix.

Procedure—Without delay, concomitantly determine the absorbances of the solutions in 1-cm cells at the wavelength of maximum absorbance at about 280 nm, with a suitable spectrophotometer, using chloroform as the blank. Calculate the quantity, in mg, of aspirin ($C_9H_8O_4$) in the portion of Capsules taken by the formula:

$$C(A_U/A_S)$$

in which C is the concentration, in µg per mL, of USP Aspirin RS in the *Standard preparation;* and A_U and A_S are the absorbances of the *Assay preparation* and the *Standard preparation,* respectively.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
ASPIRIN CAPSULES	Documentary Standards Support	SM22020 Small Molecules 2

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