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Docusate Calcium Capsules

DEFINITION

Docusate Calcium Capsules contain NLT 85.0% and NMT 115.0% of the labeled amount of docusate calcium ($C_{40}H_{74}CaO_{14}S_2$).

IDENTIFICATION

• **A. Identification Tests—General, *Calcium*⟨191⟩**

Sample solution: Ash the contents of 1 Capsule, dissolve the residue in 10 mL of dilute hydrochloric acid (1 in 12), and filter.

Acceptance criteria: The filtrate meets the requirements.

• **B. Thin-Layer Chromatography**

Standard solution: 10 mg/mL of USP Docusate Calcium RS in isopropyl alcohol

Sample solution: Empty the contents of 1 Capsule into a conical flask, and add isopropyl alcohol to obtain a solution containing 10 mg/mL of docusate calcium.

Chromatographic system

(See *Chromatography ⟨621⟩, Thin-Layer Chromatography*.)

Mode: TLC

Adsorbent: 0.25-mm layer of chromatographic silica gel

Application volume: 10 µL. [Note—Apply with the aid of a stream of nitrogen.]

Developing solvent system: Ethyl acetate, ammonium hydroxide, and alcohol (5:2:2)

Analysis

Samples: *Standard solution* and *Sample solution*

Allow the spots to dry, and develop the chromatogram in the *Developing solvent system* until the solvent front has moved 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. Expose the plate to iodine vapors in a closed chamber for about 30 min, and locate the spots.

Acceptance criteria: The R_F value of the principal spot of the *Sample solution* corresponds to that of the *Standard solution*.

ASSAY

• **Procedure**

Basic fuchsin solution: Dissolve 100 mg of basic fuchsin, previously recrystallized from alcohol, in 3 mL of methanol in a 100-mL volumetric flask, and dilute with water to volume. Filter before use.

Standard stock solution: 5 mg/mL of USP Docusate Calcium RS in isopropyl alcohol

Standard solution: Transfer 5 mL of the *Standard stock solution* to a 500-mL volumetric flask containing 90 mL of a mixture of water and isopropanol (1:1) with swirling. Dilute with water to volume. The concentration of USP Docusate Calcium RS in this solution is 50 µg/mL.

Sample stock solution: To 10 Capsules in a 400-mL beaker add 300 mL of hot water. Heat on a steam bath, with occasional stirring, until the Capsules are completely disintegrated. Cool, and transfer with the aid of 100 mL of warm water to a 1000-mL volumetric flask. Rinse the beaker with 100 mL of isopropyl alcohol, add the rinsing to the volumetric flask, and shake to dissolve any previously undissolved particles. Dilute with a mixture of water and isopropyl alcohol (1:1) to volume.

Sample solution: Transfer 10.0 mL of the *Sample stock solution* to a 100-mL volumetric flask, and dilute with water to volume.

Instrumental conditions

(See *Ultraviolet-Visible Spectroscopy ⟨857⟩*.)

Mode: Vis

Analytical wavelength: 545 nm

Cell: 1 cm

Blank: Chloroform

Analysis: Transfer 4.0 mL of the *Standard solution* to a 125-mL separator. To a second 125-mL separator transfer a volume of the *Sample solution*, equivalent to 200–240 µg of docusate calcium, and add a solution of isopropyl alcohol (1 in 100), if necessary, to bring the volume of the solution in the separator to 4.0 mL. To each separator transfer 20.0 mL of *pH 1.2 Hydrochloric Acid Buffer* (see *Reagents, Indicators,*

and Solutions—Solutions) and 2.0 mL of *Basic fuchsin solution*. Add 20 mL of chloroform to each separator, and shake for 1 min. Allow the phases to separate, and transfer the chloroform extracts, through separate pledgets of absorbent cotton, into separate 100-mL volumetric flasks. Extract each of the solutions in the separators in the same manner with additional 20-mL portions of chloroform until no more color is visible in the extract, and pass each extract through the pledget of cotton used for the preceding extract into the flask containing the preceding extract. Dilute the contents of each flask to volume by passing chloroform through the cotton pledgets that had been used to filter the extracts, and mix.

Calculate the percentage of the labeled amount of docusate calcium ($C_{40}H_{74}CaO_{14}S_2$) in the portion of Capsules taken:

$$\text{Result} = (A_U/A_S) \times (C_S/C_U) \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

C_S = concentration of anhydrous USP Docusate Calcium RS in the *Standard solution* (µg/mL)

C_U = nominal concentration of docusate calcium in the *Sample solution* (µg/mL)

Acceptance criteria: 85.0%–115.0%

PERFORMANCE TESTS

• **Dissolution** ⟨711⟩

Medium: Water; 500 mL

Apparatus 2: 50 rpm

Time: 15 min

Analysis: Place 1 Capsule in each vessel, and allow the Capsule to sink to the bottom of the vessel before starting rotation of the blade. Observe the Capsules, and record the time taken for each capsule shell to rupture.

Tolerances: The requirements are met if all of the Capsules tested rupture in NMT 15 min. If 1 or 2 of the Capsules rupture in more than 15 min but NMT 30 min, repeat the test on 12 additional Capsules. NMT 2 of the total of 18 Capsules tested rupture in more than 15 min but NMT 30 min.

• **Uniformity of Dosage Units** ⟨905⟩**:** Meet the requirements for *Content Uniformity* for solid-filled capsules and meet the requirements for *Weight Variation* for solution-filled capsules

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers, and store at controlled room temperature in a dry place.

• **USP Reference Standards** ⟨11⟩

USP Docusate Calcium RS

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

Topic/Question	Contact	Expert Committee
DOCUSATE CALCIUM CAPSULES	Documentary Standards Support	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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