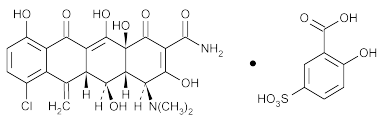


Meclocycline Sulfosalicylate



$C_{22}H_{21}ClN_2O_8 \cdot C_7H_6O_6S$ 695.05

2-Naphthacenecarboxamide, 7-chloro-4-(dimethylamino)-1,4,4a,5,5a,6,11,12a-octahydro-3,5,10,12,12a-pentahydroxy-6-methylene-1,11-dioxo-, [4S-(4 α ,4a α ,5 α ,5a α ,12a α)]-, mono(2-hydroxy-5-sulfobenzoate) (salt).
(4S,4aR,5S,5aR,12aS)-7-Chloro-4-(dimethylamino)-1,4,4a,5,5a,6,11,12a-octahydro-3,5,10,12,12a-pentahydroxy-6-methylene-1,11-dioxo-2-naphthacene carboxamide mono(5-sulfosalicylate) (salt) [73816-42-9].

» Meclocycline Sulfosalicylate has a potency equivalent to not less than 620 μ g of meclocycline ($C_{22}H_{21}ClN_2O_8$) per mg.

Packaging and storage—Preserve in tight containers, protected from light.

USP Reference standards (11)—

USP Meclocycline Sulfosalicylate RS

Identification, Infrared Absorption (197K).

Crystallinity (695): meets the requirements.

pH (791): between 2.5 and 3.5, in a solution containing 10 mg per mL.

Water, Method I (921): not more than 4.0%.

Assay—

0.001 M Ammonium edetate—Transfer 293 mg of edetic acid, accurately weighed, to a 1000-mL volumetric flask, add 1 mL of methanol and 7 mL of ammonium hydroxide, and shake to dissolve the edetic acid. Add 900 mL of water, adjust with glacial acetic acid to a pH of 6.6, dilute with water to volume, and mix.

Mobile phase—Prepare a mixture of 0.001 M Ammonium edetate and tetrahydrofuran (85 : 15). Filter and degas the solution before use.

Standard stock preparation—Dissolve an accurately weighed quantity of USP Meclocycline Sulfosalicylate RS in methanol to obtain a solution having a known concentration of about 0.5 mg of meclocycline per mL.

Standard preparation—Immediately prior to injection, dilute the *Standard stock preparation* quantitatively, and stepwise if necessary, with *Mobile phase* to obtain a solution having a known concentration of about 60 μ g of meclocycline per mL.

Assay stock preparation—Transfer 36 mg of Meclocycline Sulfosalicylate, accurately weighed, to a 50-mL volumetric flask, dilute with methanol to volume, and mix.

Assay preparation—Immediately prior to injection, transfer 3.0 mL of the *Assay stock preparation* to a 25-mL volumetric flask, dilute with *Mobile phase* to volume, and mix to obtain a solution having a nominal concentration of about 60 μ g of meclocycline per mL.

Chromatographic system—The liquid chromatograph is equipped with a 340-nm detector and a 4-mm \times 25-cm column that contains packing L1. The flow rate is about 0.8 mL per minute. Chromatograph the *Standard preparation*, and record the peak responses as directed for *Procedure*: the relative standard deviation of the meclocycline peak for replicate injections is not more than 3.0%.

Procedure—Separately inject equal volumes (about 10 μ L) of the *Standard preparation* and the *Assay preparation* into the chromatograph, and measure the responses for the meclocycline peak. Calculate the quantity in μ g of $C_{22}H_{21}ClN_2O_8$ in each mg of Meclocycline Sulfosalicylate taken by the formula:

$$(C_S / C_U)(r_U / r_S)$$

in which C_S is the concentration, in μ g per mL, of meclocycline in the *Standard preparation*; C_U is the concentration, in mg per mL, of Meclocycline Sulfosalicylate in the *Assay preparation*; and r_U and r_S are the peak responses obtained from the *Assay preparation* and the *Standard preparation*, respectively.

Meclocycline Sulfosalicylate Cream

» Meclocycline Sulfosalicylate Cream contains the equivalent of not less than 90.0 percent and not more than 125.0 percent of the labeled amount of meclocycline ($C_{22}H_{21}ClN_2O_8$).

Packaging and storage—Preserve in tight containers, protected from light.

USP Reference standards (11)—

USP Meclocycline Sulfosalicylate RS

Minimum fill (755): meets the requirements.

Assay—

0.001 M Ammonium edetate—Transfer 293 mg of edetic acid, accurately weighed, to a 1000-mL volumetric flask, add 1 mL of methanol and 7 mL of ammonium hydroxide, and shake to dissolve the edetic acid. Add 900 mL of water, adjust with glacial acetic acid to a pH of 6.6, dilute with water to volume, and mix.

Mobile phase—Prepare a mixture of 0.001 M Ammonium edetate and tetrahydrofuran (85 : 15). Filter and degas the solution before use.

Standard stock preparation—Dissolve an accurately weighed quantity of USP Meclocycline Sulfosalicylate RS in methanol to obtain a solution having a known concentration of about 0.5 mg of meclocycline per mL.

Standard preparation—Immediately prior to injection, dilute the *Standard stock preparation* quantitatively, and stepwise if necessary, with *Mobile phase*, to obtain a solution having a known concentration of about 10 μ g of meclocycline per mL.

Assay stock preparation—Transfer an accurately weighed quantity of Cream, equivalent to about 5 mg of meclocycline, to a glass-stoppered, 50-mL centrifuge tube. Add 20 mL of methanol and 20 mL of 0.025 N sulfuric acid, and shake vigorously for 15 minutes. Transfer the solution to a 50-mL volumetric flask, rinse the centrifuge tube with two 5-mL portions of methanol, and add the rinsings to the flask. Dilute with methanol to volume, and mix.

Assay preparation—Centrifuge a portion of the *Assay stock preparation* for 5 minutes. Immediately prior to injection, transfer 5 mL of the supernatant to a 50-mL volumetric flask, dilute with *Mobile phase* to volume, mix, and filter to obtain a solution having a nominal concentration of about 10 μ g of meclocycline per mL.

Chromatographic system—The liquid chromatograph is equipped with a 340-nm detector and a 4-mm \times 25-cm column that contains packing L1. The flow rate is about 0.8 mL per minute. Chromatograph the *Standard preparation*, and record the peak responses as directed for *Procedure*: the relative standard deviation of the meclocycline peak for replicate injections is not more than 3.0%.

Procedure—Separately inject equal volumes (about 10 μ L) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the meclocycline peak. Calculate the percent label