Status: Currently Official on 14-Feb-2025
Official Date: Official as of 01-Nov-2020
Document Type: USP Monographs
DocId: GUID-03B179CE-C91F-4E66-ABA9-3F66F2218C16_2_en-US
DOI: https://doi.org/10.31003/USPNF_M22740_02_01
DOI Ref: px5ji

Demeclocycline Hydrochloride Tablets

DEFINITION

Demeclocycline Hydrochloride Tablets contain NLT 90.0% and NMT 125.0% of the labeled amount of demeclocycline hydrochloride ($C_{21}H_{21}ClN_2O_8 \cdot HCl$).

IDENTIFICATION

• **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

Add the following:

▲• **B.** The UV spectrum of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.▲ (USP 1-May-2020)

ASSAY

Change to read:

• **Procedure**

Solution A: Mix appropriate volumes of 0.2 M dibasic potassium phosphate and 0.2 M monobasic potassium phosphate to prepare a buffer with a pH of 9.0.

Solution B: 0.02 M tetrabutylammonium hydrogen sulfate. Adjust with sodium hydroxide TS to a pH of 9.0.

Solution C: 0.01 M edetate disodium. Adjust with sodium hydroxide TS to a pH of 9.0.

Mobile phase: Transfer 80 g of tertiary butyl alcohol to a 1000-mL volumetric flask with the aid of 200 mL of water. Add 100 mL of *Solution A*, 150 mL of *Solution B*, and 100 mL of *Solution C*. Dilute with water to volume, and degas.

Diluent: 0.01 N hydrochloric acid

System suitability solution: ▲1 mg/mL of▲ (USP 1-May-2020) USP Demeclocycline Hydrochloride RS in *Diluent*. Allow to stand for 3 h.

Standard solution: 1 mg/mL of USP Demeclocycline Hydrochloride RS in *Diluent*

Sample solution: Nominally 1 mg/mL of demeclocycline hydrochloride prepared as follows. Weigh and finely powder NLT 10 Tablets. Transfer a portion, containing nominally 50 mg of demeclocycline hydrochloride, to a 50-mL volumetric flask, and dilute with *Diluent* to volume. Sonicate for 5 min, and centrifuge for 5 min. Pass a portion of the supernatant through a suitable filter of 1.5-µm or finer pore size.

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: UV 254 nm. ▲For *Identification B*, use a diode array detector in the range of 200–400 nm.▲ (USP 1-May-2020)

Column: 4.6-mm × 25-cm; 8-µm packing L21

Column temperature: 60 ± 0.5°

Flow rate: 1 mL/min

Injection volume: 20 µL

System suitability

Samples: *System suitability solution* and *Standard solution*

[Note—The relative retention times for epidemethylchlortetracycline and demeclocycline are about 0.7 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 3.0 between the epidemethylchlortetracycline and demeclocycline peaks, *System suitability solution*

Relative standard deviation: NMT 2.0%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of demeclocycline hydrochloride ($C_{21}H_{21}ClN_2O_8 \cdot HCl$) in the portion of Tablets taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times P \times F \times 100$$

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

C_S = concentration of USP Demeclocycline Hydrochloride RS in the *Standard solution* (mg/mL)

C_U = nominal concentration of demeclocycline hydrochloride in the *Sample solution* (mg/mL)

P = potency of demeclocycline in USP Demeclocycline Hydrochloride RS (µg/mg)

F = conversion factor, 0.001 mg/µg

Acceptance criteria: 90.0%–125.0%

PERFORMANCE TESTS

• **Dissolution ⟨711⟩**

Medium: Water; 900 mL

Apparatus 2: 75 rpm

Time: 45 min

Standard solution: Prepare a solution with a known concentration of USP Demeclocycline Hydrochloride RS in *Medium*.

Sample solution: Sample per *Dissolution ⟨711⟩*. A filtered portion of the solution under test suitably diluted with *Medium*

Instrumental conditions

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

Mode: UV

Analytical wavelength: 274 nm

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of demeclocycline ($C_{21}H_{21}ClN_2O_8$) dissolved:

$$\text{Result} = (A_U/A_S) \times (C_S/L) \times D \times V \times P \times F \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

C_S = concentration of USP Demeclocycline Hydrochloride RS in the *Standard solution* (mg/mL)

L = label claim (mg/Tablet)

D = dilution factor of the *Sample solution*

V = volume of *Medium*, 900 mL

P = potency of demeclocycline in USP Demeclocycline Hydrochloride RS (µg/mg)

F = conversion factor, 0.001 mg/µg

Tolerances: NLT 75% (Q) of the labeled amount of demeclocycline ($C_{21}H_{21}ClN_2O_8$) is dissolved.

• **Uniformity of Dosage Units ⟨905⟩:** Meet the requirements

ADDITIONAL REQUIREMENTS

Change to read:

• **Packaging and Storage:** Preserve in tight, light-resistant containers.▲Store at controlled room temperature.▲ (USP 1-May-2020)

• **USP Reference Standards ⟨11⟩**

USP Demeclocycline Hydrochloride RS

Topic/Question	Contact	Expert Committee
DEMECLOCYCLINE HYDROCHLORIDE TABLETS	Documentary Standards Support	SM12020 Small Molecules 1

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. PF 44(6)

Current DocID: GUID-03B179CE-C91F-4E66-ABA9-3F66F2218C16_2_en-US

DOI: https://doi.org/10.31003/USPNF_M22740_02_01

DOI ref: px5ji