

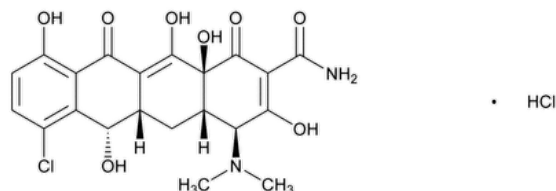
Status: Currently Official on 14-Feb-2025
 Official Date: Official as of 01-Nov-2020
 Document Type: USP Monographs
 DocId: GUID-319E5346-09EF-4543-855D-4D95A45EE058_4_en-US
 DOI: https://doi.org/10.31003/USPNF_M22720_04_01
 DOI Ref: 517d8

© 2025 USPC
 Do not distribute

Demeclocycline Hydrochloride

Change to read:

▲



▲ (USP 1-May-2020)

$C_{21}H_{21}ClN_2O_8 \cdot HCl$ 501.31

2-Naphthacenecarboxamide, 7-chloro-4-(dimethylamino)-1,4,4a,5,5a,6,11,12a-octahydro-3,6,10,12,12a-pentahydroxy-1,11-dioxo-, monohydrochloride, 4S-(4 α ,4a α ,5a α ,6 β ,12a α)-;

7-Chloro-4-(dimethylamino)-1,4,4a,5,5a,6,11,12a-octahydro-3,6,10,12,12a-pentahydroxy-1,11-dioxo-2-naphthacenecarboxamide monohydrochloride CAS RN[®]: 64-73-3; UNII: 29O079NTYT.

DEFINITION

Demeclocycline Hydrochloride has a potency of NLT 900 µg/mg of demeclocycline hydrochloride ($C_{21}H_{21}ClN_2O_8 \cdot HCl$), calculated on the dried basis.

IDENTIFICATION

Change to read:

- **A.** ▲ **SPECTROSCOPIC IDENTIFICATION TESTS** (197), *Infrared Spectroscopy*: (197K) or (197A) ▲ (USP 1-May-2020)

Change to read:

- **B.** ▲ The retention time 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. ▲ (USP 1-May-2020)

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: 1 mg/mL of Demeclocycline Hydrochloride in *Diluent*

Chromatographic system

(See [Chromatography \(621\)](#), *System Suitability*.)

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 25-cm; ▲8-μm▲ (USP 1-May-2020) 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 0.7 and 1.0, respectively.]

Suitability requirements

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

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

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the quantity, in μg/mg, of demeclocycline hydrochloride ($C_{21}H_{21}ClN_2O_8 \cdot HCl$) in the portion of Demeclocycline Hydrochloride taken:

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

r_U = peak response of demeclocycline from the *Sample solution*

r_S = peak response of demeclocycline from the *Standard solution*

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

C_U = concentration of Demeclocycline Hydrochloride in the *Sample solution* (mg/mL)

P = potency of demeclocycline hydrochloride in [USP Demeclocycline Hydrochloride RS](#) (μg/mg)

Acceptance criteria: NLT 900 μg/mg on the dried basis

SPECIFIC TESTS

• [CRYSTALLINITY \(695\)](#): Meets the requirements

• [pH \(791\)](#)

Sample solution: 10 mg/mL

Acceptance criteria: 2.0–3.0

Change to read:

• [LOSS ON DRYING \(731\)](#)

Sample: 100 mg

Analysis: Dry the *Sample* ▲ in a capillary-stoppered bottle in vacuum▲ (ERR 1-May-2020) at 60° for 3 h.

Acceptance criteria: NMT 2.0%

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in tight, light-resistant containers.

• [USP REFERENCE STANDARDS \(11\)](#)
[USP Demeclocycline Hydrochloride RS](#)

Auxiliary Information - Please [check for your question in the FAQs](#) before contacting USP.

Topic/Question	Contact	Expert Committee
DEMECLOCYCLINE HYDROCHLORIDE	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-319E5346-09EF-4543-855D-4D95A45EE058_4_en-US