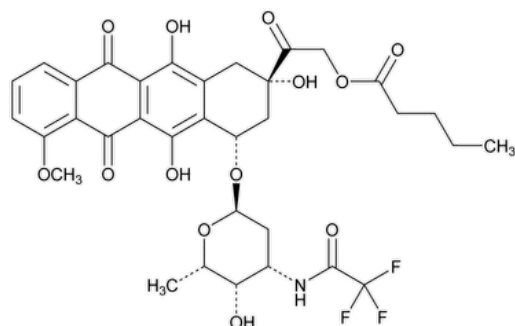


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Valrubicin



$C_{34}H_{36}F_3NO_{13}$ 723.64
 (2S-cis)-2-[1,2,3,4,6,11-Hexahydro-2,5,12-trihydroxy-7-methoxy-6,11-dioxo-4-[[2,3,6-trideoxy-3-[(trifluoroacetyl) amino]- α -L-lyxo-hexopyranosyl]oxy]-2-naphthacenyl]-2-oxoethyl pentanoate;
 (8S,10S)-8-Glycolyl-7,8,9,10-tetrahydro-6,8,11-trihydroxy-1-methoxy-10-[[2,3,6-trideoxy-3-(2,2,2-trifluoroacetamido)- α -L-lyxo-hexopyranosyl]oxy]-5,12-naphthacenedione 8²-valerate CAS RN[®]: 56124-62-0; UNII: 2C6NUM6878.

DEFINITION

Valrubicin contains NLT 98.0% and NMT 102.0% of $C_{34}H_{36}F_3NO_{13}$, calculated on the anhydrous and solvent-free basis.

[CAUTION—Great care should be taken to prevent inhaling particles of Valrubicin and exposing the skin to it.]

IDENTIFICATION

Change to read:

- A. [▲][SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), [Infrared Spectroscopy: 197M](#) [▲] (CN 1-May-2020)
- B. The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the Assay.

ASSAY

PROCEDURE

Solution A: 3.4 mg/mL of monobasic potassium phosphate adjusted with phosphoric acid to a pH of 3.1

Mobile phase: Acetonitrile and *Solution A* (3:2)

Standard solution: 0.5 mg/mL of [USP Valrubicin RS](#) in acetonitrile

Sample solution: 0.5 mg/mL of Valrubicin in acetonitrile

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 5-cm; 1.8- μ m packing L1

Flow rate: 1.5 mL/min

Column temperature: 40°

Injection size: 10 μ L

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of $C_{34}H_{36}F_3NO_{13}$ in the portion of Valrubicin taken:

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

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

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

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

Acceptance criteria: 98.0%–102.0% on the anhydrous and solvent-free basis

IMPURITIES

INORGANIC IMPURITIES

- [RESIDUE ON IGNITION \(281\)](#): NMT 0.2%

ORGANIC IMPURITIES

• PROCEDURE

Solution A: Use *Solution A* as directed in the Assay.

Solution B: Acetonitrile

Mobile phase: See the gradient table below. [NOTE—Analysis time is 55 min. The steps that follow are for column washing and re-equilibration.]

Time (min)	Solution A (%)	Solution B (%)
0	75	25
45	40	60
55	40	60
58	20	80
68	20	80
70	75	25
80	75	25

Solution C: Acetonitrile and water (1:1)

Stock standard solution: 2 mg/mL of [USP Valrubicin RS](#) in acetonitrile and water (1:1). [NOTE—Dissolve first in acetonitrile, using 50% of the final volume and dilute with water to volume.]

Standard solution: 0.01 mg/mL of valrubicin in *Solution C*

System suitability solution: 2 mg/mL of [USP Valrubicin Resolution Mixture RS](#) in acetonitrile and water (1:1). [NOTE—Dissolve first in acetonitrile, using 50% of the final volume and dilute with water to volume.]

Sample solution: 2 mg/mL of Valrubicin in acetonitrile and water (1:1). [NOTE—Dissolve first in acetonitrile, using 50% of the final volume and dilute with water to volume.]

Chromatographic system

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

Mode: LC

Detector: 254 nm

Column: 4.6-mm × 5-cm; 1.8-μm packing L1

Flow rate: 1.5 mL/min

Temperature

Column: 40°

Autosampler: 4°

Injection size: 10 μL