

Status: Currently Official on 14-Feb-2025
Official Date: Official Prior to 2013
Document Type: USP Monographs
DocId: GUID-2879508F-033A-45D1-AE65-AF12E66129DC_2_en-US
DOI: https://doi.org/10.31003/USPNF_M25970_02_01
DOI Ref: i2tm3

© 2025 USPC
Do not distribute

Digitoxin Tablets

» Digitoxin Tablets contain not less than 90.0 percent and not more than 110.0 percent of the labeled amount of $C_{41}H_{64}O_{13}$.

NOTE—Avoid the use of strongly adsorbing substances, such as bentonite, in the manufacture of Digitoxin Tablets.

Packaging and storage—Preserve in well-closed containers.

USP REFERENCE STANDARDS (11).—

[USP Digitoxin RS](#)

Identification—

A: Transfer a quantity of finely powdered Tablets, equivalent to not less than 1 mg of digitoxin, to a suitable flask, add 20 mL of chloroform, and sonicate. Filter, and evaporate the filtrate on a steam bath with the aid of a current of air to dryness. Dissolve the residue in 2 mL of a solution prepared by mixing 0.3 mL of ferric chloride TS and 50 mL of glacial acetic acid, and underlay with 2 mL of sulfuric acid: at the zone of contact of the two liquids a brown color, which gradually changes to light green, then to blue, is produced, and finally the entire acetic acid layer acquires a blue color.

B: The retention time of the major peak in the chromatogram of the *Assay preparation* corresponds to that of the major peak in the chromatogram of the *Standard preparation* as obtained in the *Assay*.

DISSOLUTION (711).—[NOTE—Throughout this procedure, use scrupulously clean glassware, which previously has been rinsed successively with hydrochloric acid, water, and alcohol, and carefully dried. Take precautions to prevent contamination from fluorescent particles and from metal and rubber surfaces.]

Medium: dilute hydrochloric acid (3 in 500); 500 mL. [NOTE—Use the same batch of *Medium* throughout the test.]

Apparatus 1: 120 ± 5 rpm.

Times: 30 minutes; 60 minutes.

Standard stock solution—Weigh accurately about 30 mg of [USP Digitoxin RS](#), dissolve in a minimum amount of alcohol in a 500-mL volumetric flask, add dilute alcohol (4 in 5) to volume, and mix.

Standard solutions—Just prior to use, dilute 5.0 mL of the *Standard stock solution* with *Medium* to 500.0 mL, and mix. Transfer aliquots (2.0 to 10.0 mL) of this solution to individual separators to prepare standards equivalent to 20, 40, 60, 80, and 100% of the labeled amount of digitoxin in 500 mL. Add *Medium* to make 10 mL, and proceed as directed for *Procedure*, beginning with “Extract with three 15-mL portions of chloroform.”

Procedure—Proceed as directed for [Procedure](#) under [Dissolution \(711\)](#). After 30 minutes, accurately timed, withdraw a suitable aliquot of the solution under test from a point midway between the stirring shaft and the wall of the vessel, and approximately midway in depth. Filter the solution promptly after withdrawal, using a suitable membrane filter of not greater than 0.8- μ m porosity, discarding the first 10 mL of the filtrate. Without replacing the *Medium* withdrawn, continue to rotate the basket, and after an additional 30 minutes, accurately timed, similarly withdraw and filter another aliquot. Treat each of these solutions as follows: Assuming dissolution of 100% of the labeled amount of digitoxin, transfer aliquots, equivalent to 6 μ g of digitoxin, to suitable separators. Extract with three 15-mL portions of chloroform, and combine the chloroform extracts in glass-stoppered flasks. Evaporate the combined extracts on a steam bath, with the aid of a current of air, to dryness. In a similar manner, prepare a blank using a suitable volume of *Medium*.

Measurement of fluorescence—Begin with the *Standard solutions*, and keep all flasks in the same sequence throughout, so that the elapsed time from addition of reagents to reading of fluorescence is the same for each set. Treat 1 flask at a time as follows: Add 10 mL of a solution freshly prepared by dissolving 35 mg of ascorbic acid in 25 mL of methanol and cautiously adding the solution to 100 mL of hydrochloric acid. Mix, and add 1 mL of a solution freshly prepared by diluting 1 mL of 30 percent hydrogen peroxide with water to 500 mL and diluting 1 volume of the resulting solution with 20 volumes of water. Mix, and insert the stopper in the flask. After 45 minutes, measure the fluorescence at about 575 nm, the excitation wavelength being about 395 nm. Correct each reading for the blank, and plot a standard curve of fluorescence versus percentage dissolution. By calculation from the standard curve, determine the percentage dissolution of digitoxin in each Tablet within 30 minutes and the total percentage dissolution of digitoxin within 60 minutes, taking into account the volume of the solution under test removed after the first 30 minutes of the test.

Tolerances—Not less than 60% of the labeled amount of $C_{41}H_{64}O_{13}$ is dissolved within 30 minutes for each Tablet tested, and not less than 85% of the labeled amount is dissolved within 60 minutes for the average of the Tablets tested.

UNIFORMITY OF DOSAGE UNITS (905): meet the requirements.

Procedure for content uniformity—Place 1 Tablet in a suitable glass-stoppered conical flask. Add an accurately measured volume of *Mobile phase* (prepared as directed in the [Assay](#) under [Digitoxin](#)) sufficient to obtain a solution containing about 10 µg of digitoxin per mL, and shake by mechanical means until the Tablet has completely disintegrated (not less than 30 minutes). Centrifuge, and use the clear supernatant as the test solution. Dissolve an accurately weighed quantity of [USP Digitoxin RS](#) in *Mobile phase*, and dilute quantitatively, and stepwise if necessary, with *Mobile phase* to obtain a Standard solution having a known concentration of about 10 µg per mL. Proceed as directed in the *Assay*. Calculate the quantity, in mg, of $C_{41}H_{64}O_{13}$ in the Tablet by the formula:

$$(LC/D)(r_U/r_S)$$

in which *L* is the labeled quantity, in mg, of digitoxin in the Tablet, *C* is the concentration, in µg per mL, of [USP Digitoxin RS](#) in the Standard solution, *D* is the concentration, in µg per mL, of digitoxin in the test solution based on the labeled quantity in the Tablet and the extent of dilution, and *r_U* and *r_S* are the digitoxin peak responses obtained from the test solution and the Standard solution, respectively.

Assay—

Mobile phase, Standard preparation, System suitability preparation, and Chromatographic system—Prepare as directed in the [Assay](#) under [Digitoxin](#).

Assay preparation—Weigh and finely powder not fewer than 20 Tablets. Transfer an accurately weighed quantity of powder, equivalent to about 1 mg of digitoxin, to a 25-mL volumetric flask. Add 15 mL of *Mobile phase*, and sonicate. Dilute with *Mobile phase* to volume, and mix. Filter a portion of this solution, discarding the first few mL of the filtrate. The filtrate is the *Assay preparation*.

Procedure—Proceed as directed for *Procedure* in the [Assay](#) under [Digitoxin](#). Calculate the quantity, in µg, of $C_{41}H_{64}O_{13}$ in the portion of Tablets taken by the formula:

$$25C(r_U/r_S)$$

in which *C* is the concentration, in µg per mL, of [USP Digitoxin RS](#) in the *Standard preparation*, and *r_U* and *r_S* are the peak responses obtained from the *Assay preparation* and the *Standard preparation*, respectively.

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

Topic/Question	Contact	Expert Committee
DIGITOXIN TABLETS	Nam-Cheol Kim Scientific Liaison	BDSHM2020 Botanical Dietary Supplements and Herbal Medicines

Chromatographic Database Information: [Chromatographic Database](#)

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. Information currently unavailable

Current DocID: [GUID-2879508F-033A-45D1-AE65-AF12E66129DC_2_en-US](#)
Previous DocID: [GUID-2879508F-033A-45D1-AE65-AF12E66129DC_1_en-US](#)
DOI: https://doi.org/10.31003/USPNF_M25970_02_01
DOI ref: [i2tm3](#)