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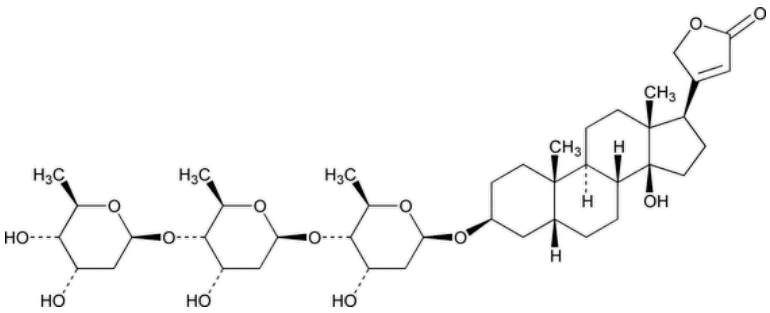
Digitoxin



$C_{41}H_{64}O_{13}$ 764.94

Card-20(22)-enolide, 3-[(*O*-2,6-dideoxy-β-D-*ribo*-hexopyranosyl-(1→4)-*O*-2,6-dideoxy-β-D-*ribo*-hexopyranosyl-(1→4)-2,6-dideoxy-β-D-*ribo*-hexopyranosyl)oxy]-14-hydroxy, (3β,5β)-.

Digitoxin CAS RN®: 71-63-6; UNII: E90NZP2L9U.

» Digitoxin is a cardiotonic glycoside obtained from *Digitalis purpurea* L., *Digitalis lanata* Ehrh. (Fam. Scrophulariaceae), and other suitable species of *Digitalis*. Digitoxin contains not less than 92.0 percent and not more than 103.0 percent of $C_{41}H_{64}O_{13}$, calculated on the dried basis.

[**Caution—**Handle Digitoxin with exceptional care since it is highly potent.]

Packaging and storage—Preserve in tight containers.

USP Reference standards ⟨11⟩—
USP Digitoxin RS

Identification—

Change to read:

A: ▲*Spectroscopic Identification Tests ⟨197⟩, Infrared Spectroscopy: 197K*▲ (CN 1-May-2020) ·

B: Prepare a test solution in methanol containing 1 mg per mL. Apply 1 µL of the test solution and 1 µL of a Standard solution of USP Digitoxin RS in methanol containing 1 mg per mL to a suitable thin-layer chromatographic plate (see *Chromatography ⟨621⟩*) coated with a 0.25-mm layer of chromatographic silica gel mixture. Allow the applications to dry, and develop the chromatogram in a solvent system consisting of a mixture of methylene chloride and methanol (93:7) until the solvent front has moved about three-fourths of the length of the plate. Remove the plate from the developing chamber, mark the solvent front, and dry the plate at 100° to remove the solvent. Spray the plate with a 6 in 10 solution of sulfuric acid in methanol, heat at 105° for 10 minutes, and examine the chromatogram under long-wavelength UV light: the R_F value of the principal spot obtained from the test solution corresponds to that obtained from the Standard solution.

C: 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*.

Loss on drying ⟨731⟩—Dry it in vacuum at 105° for 1 hour: it loses not more than 1.5% of its weight.

Residue on ignition ⟨281⟩: negligible, from 100 mg.

Assay—

Mobile phase—Prepare a filtered and degassed mixture of water and acetonitrile (55:45). Make adjustments if necessary (see *System Suitability* under *Chromatography ⟨621⟩*).

Standard preparation—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 solution having a known concentration of about 40 µg per mL.

Assay preparation—Transfer about 50 mg of Digitoxin, accurately weighed, to a 200-mL volumetric flask. Dissolve in and dilute with *Mobile phase* to volume, and mix. Pipet 4 mL of this solution into a 25-mL volumetric flask, dilute with *Mobile phase* to volume, and mix.

System suitability preparation—Prepare a solution in *Mobile phase* containing about 40 µg each of digitoxin and digoxin per mL.

Chromatographic system (see *Chromatography ⟨621⟩*)—The liquid chromatograph is equipped with a 218-nm detector and a 3.9-mm × 30-cm column that contains packing L1. The flow rate is about 1 mL per minute. Chromatograph the *Standard preparation* and the *System suitability preparation*, and record the peak responses as directed for *Procedure:* the relative retention times are about 0.35 for digoxin and 1.0 for digitoxin; the resolution, *R,* between the digoxin and digitoxin peaks is not less than 2.0; the tailing factor for the analyte peak is not more than 2.0; and the relative standard deviation for replicate injections of the *Standard preparation* is not more than 2.0%.

Procedure—Separately inject equal volumes (about 50 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity, in mg, of $C_{41}H_{64}O_{13}$ in the portion of Digitoxin taken by the formula:

$$1.25C(r_U/r_S)$$

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	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

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