Dahuang Zhechong Pills *(Dahuang Zhechong Wan)* – 大黄蛴虫丸

Sample source

Commercially available Dahuang Zhechong Pills

Chemical reference substances

Emodin (National Institute for the Control of Pharmaceutical and Biological Products, Batch number 0756-200210)

Preparation of test solution

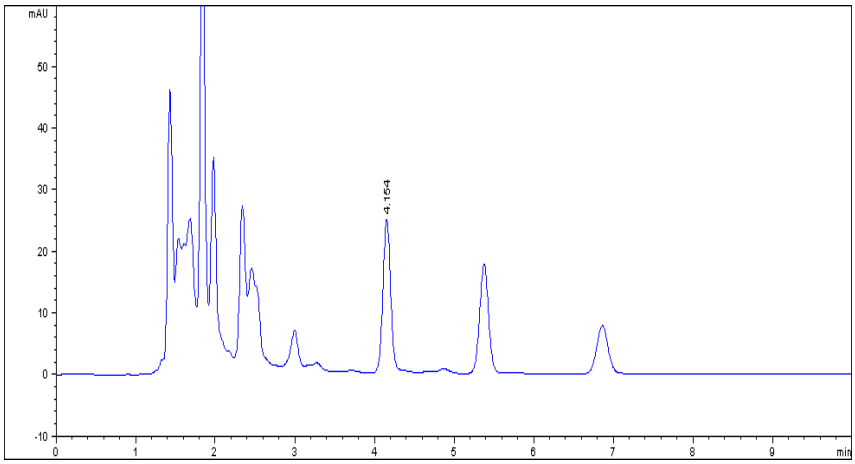
Cut a quantity of pills into pieces and mix well, take a quantity of the pieces, accurately weigh, grind well with an equal quantity of kieselguhr and mix well. Accurately weigh 4 g of the powder in a stoppered conical flask, accurately add 25 mL of methanol, weigh, heat under reflux on a water bath for 1 hour, and allow to cool, weigh again and replenish the lost weight with methanol, mix well, and filter. Accurately measure 5 mL of the successive filtrate in a conical flask, discard the solvent, add 20 mL of 2.5 mol/L sulfuric acid solution, treat ultrasonically for 10 minutes, and heat under reflux for 1 hour, and cool immediately, transfer to a separator. Extract with three 25-mL quantities of ether, combine the ether extracts, wash with 15 mL of water, combine the ether extracts, wash with 15 mL of water, discard the washings, and pass through a funnel packed with anhydrous sodium sulfate. Wash the filter and the container with a little quantity of ether, combine the washings with the filtrate, evaporate to dryness at a low temperature. Dissolve the residue with a quantity of methanol in a 25 mL volumetric flask, dilute with methanol to volume, and mix well.

Chromatographic conditions

- Column: ZORBAX SB C18 4.6×150 mm, 5 µm (883975-902)
- Column temperature: 25 °C
- Mobile phase: methanol-water-phosphoric acid (85:15:0.05)
- Detector wavelength: 289 nm
- Flow rate: 1.0 mL/min

Chromatographic system

- Agilent 1200 Series quaternary pump with vacuum degasser
- Agilent 1200 Series high-performance autosampler
- Agilent 1200 Series thermostatted column compartment
- Agilent 1200 Series diode-array detector
- System control through Agilent ChemStation revision B.01.01



Components	k'	Ret Time (min)	Height (mAU)	Area (mAU*s)	n	USP T_f
Emodin	2.462	4.154	24.82	165.8	9071	1.05