

Qingning Pills (*Qingning Wan*) – 清宁丸

Sample source

Commercially available Qingning Pills

Chemical reference substances

1. Emodin, 2. Chrysophanol
(National Institute for the Control of Pharmaceutical and Biological Products, Batch number 1. 110756-200210, 2. 110796-200309)

Preparation of test solution

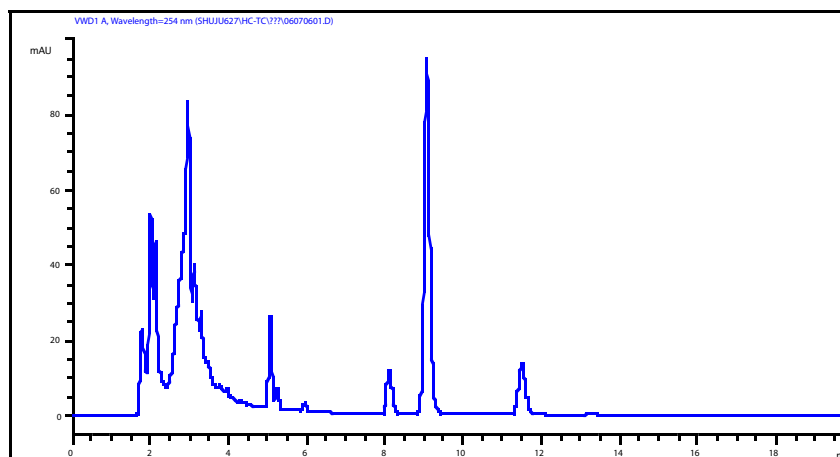
Grind a quantity of the pills to a fine powder, accurately weigh 0.1 g in a conical flask, accurately add 25 mL of a mixture of hydrochloric acid and methanol (1:25) and weigh. Heat under reflux on a water bath for 2 hours, allow to cool, and replenish the lost weight with a mixture of hydrochloric acid and methanol (1:25). Mix well and filter. Accurately measure 5 mL of the filtrate in an evaporating dish, evaporate to dryness. Dissolve the residue in a quantity of methanol, transfer to a 5 mL volumetric flask, dilute to volume with methanol, mix well, filter through a millipore membrane (0.45 μm), and use the filtrate as the test solution.

Chromatographic conditions

- Column: Agilent TC-C18, 4.6 \times 250 mm, 5 μm (518925-902)
- Column temperature: 30 $^{\circ}\text{C}$
- Mobile phase: methanol-0.1% phosphoric acid (85:15)
- Detector wavelength: 254 nm
- Flow rate: 1.0 mL/min
- Injection volume: 5 μL

Chromatographic system

- Agilent 1200 Series quaternary pump with vacuum degasser
- Agilent 1200 Series high-performance autosampler
- Agilent 1200 Series thermostated column compartment
- Agilent 1200 Series variable wavelength detector
- System control through Agilent ChemStation revision B.01.01



Components	Ret Time (min)	Height (mAU)	Area (mAU*s)	n	USP T_r
Emodin	8.112	11.70	108.9	17987	1.07
Chrysophanol	9.077	94.19	945.1	20163	1.08