

# Immature Orange Fruit (*Fructus Aurantii Immaturus*) – 枳实

## Sample source

Commercially available Immature Orange Fruit

## Chemical reference substances

Synephrine (National Institute for the Control of Pharmaceutical and Biological Products, Batch number: 110727-200306)

## Preparation of test solution

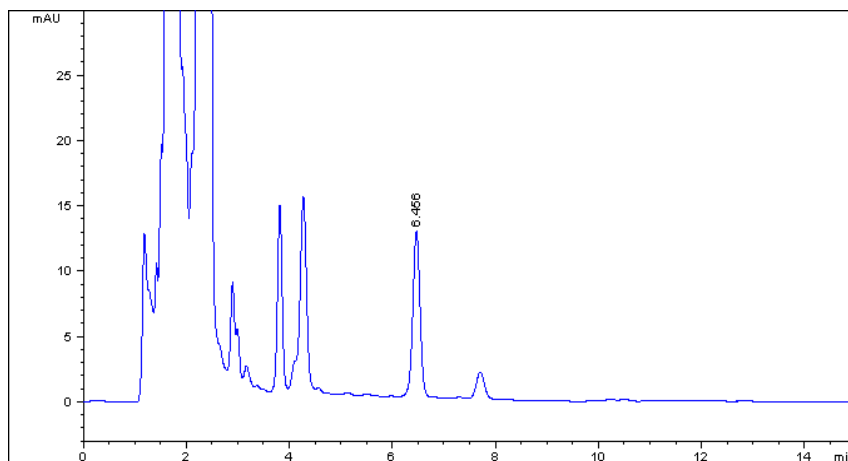
Accurately weigh 1 g of the powder in a stoppered conical flask, add 50 mL of methanol, weigh accurately, heat under reflux for 1.5 hours, allow to cool, weigh again, replenish the lost weight with methanol, mix well and filter. Accurately measure 10 mL of the filtrate and evaporate to dryness. Dissolve the residue in 10 mL of water, mix well, apply to a column (1.5 cm ID) packed with polyamide (60-90 mesh, 2.5 g). Elute with 25 mL of water and collect the eluent, transfer to a 25 mL volumetric flask. Dilute with water to volume and mix well. Filter through a millipore membrane (0.45  $\mu\text{m}$ ) and use the filtrate as the test solution.

## Chromatographic conditions

- Column: ZORBAX XDB C8, 4.6 $\times$ 150 mm, 5  $\mu\text{m}$  (993967-906)
- Column temperature: 30  $^{\circ}\text{C}$
- Mobile phase: methanol-aqueous mono potassium phosphate solution (0.06 % mono potassium phosphate + 0.1 % sodium dodecyl sulfate + 0.1 % glacial acetic acid) (50:50)
- Detector wavelength: 275 nm
- Flow rate: 1.0 mL/min
- Injection volume: 10  $\mu\text{L}$

## 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	k'	Ret Time (min)	Height (mAU)	Area (mAU*s)	n	USP T <sub>r</sub>
Synephrine	3.304	6.456	12.60	120.2	10891	0.99