

<b>Chongqing Xingcan Pharmaceutical Technology Co.</b> <b>Finished product quality standards, testing methods</b>			Page 1/4
			Document code: STP-04004-00
Title: Enclomiphene Quality Standards and Test Methods			
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Goal:

Develop quality standards and testing methods for Enclomiphene to ensure the quality of product .

Responsibility

QC operators shall perform according to this regulation;

QC supervisor and QA are responsible for supervision and inspection.

Scope of application

For the testing and release of finished products of Enclomiphene.

Element

#### 1. Testing items and quality standards

Test items		norm	Detection Methods	note
Appearance		White powder	visual assessment	-----
Related substances	Purity (290 nm)	≥99.0 %	HPLC	-----
	Z configuration (233 nm)	≤1.0%		
	Total impurities (290 nm)	≤1.0 %		

Loss on drying	$\leq 1.0\%$	Loss on drying method	Chinese Pharmacopoeia 2020 Edition 0831
Residue on ignition	$\leq 1.0\%$	Residue on ignition Method	Chinese Pharmacopoeia 2020 Edition 0841
Heavy metal	$\leq 20\text{ppm}$	Heavy metal checking method	Chinese Pharmacopoeia 2020 Edition 0821

## 2. Main project testing methods

### 2.1. Visual

inspection of appearance and properties

### 2.2. Detection of substances of concern

#### 2.2.1. Testing Instruments

High performance liquid chromatography (UV detector)

#### 2.2.2. Testing conditions

Column: C4 (5 $\mu\text{m}$ \*4.6mm\*150mm)

Mobile Phase A: Methanol

Mobile phase B: 0.01M potassium  
dihydrogen phosphate (PH 2.5)

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Gradient:

times	A (per cent)	B (per cent)
0	5	95
20	75	25
25	75	25
25.01	5	95
35	5	95

Flow rate of mobile phase: 1.0ml/min

Detection wavelength:

233nm, 290nm Column

temperature: 35°C

Injection

volume: 20μl

Sampling time: 35min

#### 2.2.3. Sample handling and preparation

Weigh about 10mg of the test material into a 100ml volumetric flask, add methanol to dissolve and dilute to the scale, shake well to obtain.

#### 2.2.4.Procedure

The test operation is performed in accordance with the LC-16 HPLC Operation, Maintenance and Repair SOP.

#### 2.3. Loss on drying

Take about 1g of the test sample, accurately weigh it into a constant weight weighing bottle, dry it at 105 for 3 hours, and then weigh and calculate the drying loss.

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#### 2.4. Residue on ignition

Take about 1g of the test sample, weigh it accurately into a constant weight crucible, burn it slowly until it is completely carbonized, let it cool, add 0.5-1.0ml of sulfuric acid, heat it at low temperature until the sulfuric acid vapor is completely removed, and burn it at 700-800 °C for 2 hours until it is completely ashed. Then weigh and calculate the burning residue.

#### 2.5. Heavy metals

Test solution: Weigh approximately 0.5g of the test sample into a colorimetric tube, dissolve it in 10ml of methanol, dilute with water to 25ml, and then add 2ml of lead acetate buffer (pH 3.5) and 2ml of thioacetamide test solution. Shake well and let stand for 2 minutes before observation.

Reference solution: Transfer 1ml of standard lead solution (10ppm) into a colorimetric tube, dilute with water to 10ml, mix with 10ml of methanol, dilute with water to 25ml, then add 2ml of lead acetate buffer (PH3.5) and 2ml of thioacetamide test solution, shake well, and let stand for 2 minutes before observation.

Blank solution: Add 10ml of methanol, dilute with water to 25ml, then add 2ml of lead acetate buffer (pH 3.5) and 2ml of thioacetamide test solution, shake well, leave for 2 minutes, and observe.

Compare the above three solutions and visually observe from top to bottom. The control solution should be darker than the blank solution, and the color of the test solution should not be darker than the control solution.

**Related records:** Record Name: Enclomiphene Quality test record  
Record number : STP-04004-R01-00