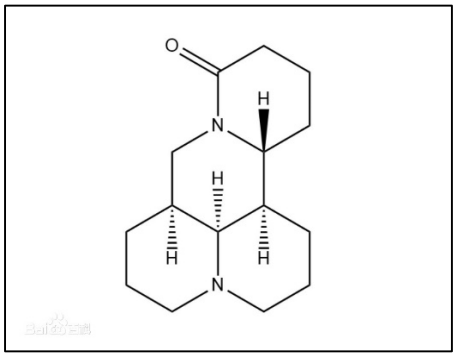
MATRINE



ISO common name	Matrine
Chemical name	(1R,2R,9S,17S)-7,13-diazatetracyclo[7.7.1.02,7.013,17]heptadecan-6-one
Empirical formula	$C_{15}H_{24}N_2O$
RMM	248.36
m.p.	76-77 °C
v.p.	1.67E-06 mmHg at 25°C
Solubility	In water 58 g/l, methanol and acetonitrile > 250 g/l(all at 20°C)
Description	White solid
Stability	Stable in neutral and weak acidic conditions but hydrolysed in alkaline condition
Formulation	Soluble liquid

MATRINE TECHNICAL CONCENTRATE
XXX/TK/M/-

1. **Sampling.** Take at least 100 g.

2. Identity tests

2.1 **HPLC.** Use the HPLC method below. The relative retention time of Matrine in the sample solution should not deviate by more than 1.5% from that of calibration solution.

2.2 **UV spectrometry.** Record the UV spectrum during the HPLC determination from 190 to 400 nm using a diode array detector. The spectrum obtained from the sample should not differ significantly from that of the standard.

3. Matrine

OUTLINE OF METHOD

Matrine is determined by high performance liquid chromatography on a reversed phase column with UV detection and external standardization.

REAGENTS

Acetonitrile: HPLC grade
Methanol:HPLC grade
Water:Ultra-pure
Ammonium acetate: AR grade
Triethylamine: AR grade
Matrine standard: Known purity

APPARATUS

Balance
Ultrasonic water bath
High performance liquid chromatography equipped with a detector suitable for operation at 215nm
Column stainless steel, Inertsustain 150mm × 4.6mm (i.d) columns, C18 packed with octadecyl silane filler (5μm), or equivalent.
Filter pore diameter: 0.45 μm

PROCEDURES

(a) LIQUID CHROMATOGRAPHIC CONDITIONS

Mobile phase: Acetonitrile / Water (0.02% Ammonium acetate + 0.02% Triethylamine) = 23/77(v/v)
Flow rate: 1.0ml/min
Detector wavelength: 215 nm
Injection volume: 10μL
Column temperature: 30°C
Retention time: approximately 11.4 min

(b) Equilibration of the chromatographic system. Inject the calibration solution and repeat the injections until retention times and the response factors calculated from the peak areas vary by less than 1.5% for successive injections.

(c) Preparation of solvent: 500 mL Methanol and 500mL water are measured into a 1000 mL volumetric flask and mixed thoroughly as solvent used to dissolve samples (named as Solution 1).

Preparation of standard solution: Approximate 50 mg (to the nearest 0.2 mg) of analytical standard grade Matrine is weighed into a 25mL volumetric flask, dissolved and made to volume with Solution1 as Matrine stock standard solution. Then transfer 1.0mL of Matrine stock standard solution into a 25mL volumetric flask, dilute to volume with Solution1 and mix thoroughly to prepare Matrine standard solution. The solution should be filtered through 0.45 μm filter film before use.

Preparation of sample solution: Weigh 500mg (to the nearest 0.2mg) sufficient sample to contain about 50 mg Matrine into a 25 mL volumetric flask, dissolved and made to volume with Solution1 as sample stock solution. Then transfer 1.00mL of sample stock solution into a 25mL volumetric flask, dilute to volume with Solution1 and mix thoroughly to prepare sample solution. The solution should be filtered through 0.45 μm filter film before use.

(d)Determination: Inject in duplicate 10μL portions of each sample solution bracketing them by injections of the calibration solutions as follows:
C_A, S_1, S_1, C_B, S_2, S_2, C_A, etc

(e)Calculation

$$f_i = \frac{S \times P}{H_S}$$

Content of $= \frac{H_w \times f}{w}$ g/kg

where:

f_i=individual response factor

f= mean response factor

H_S=peak areas of Matrine in the calibration solution

H_w=peak areas of Matrine in the sample solution

s=mass of Matrine standard (mg)

w=mass of sample taken(mg)

P=purity of Matrine standard (g/kg)

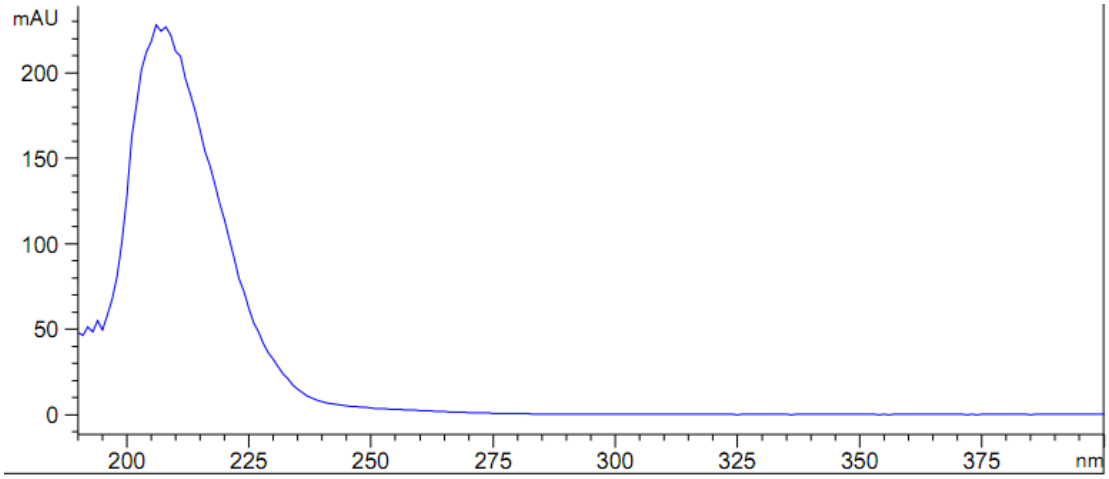


Fig 1 UV spectra of Matrine

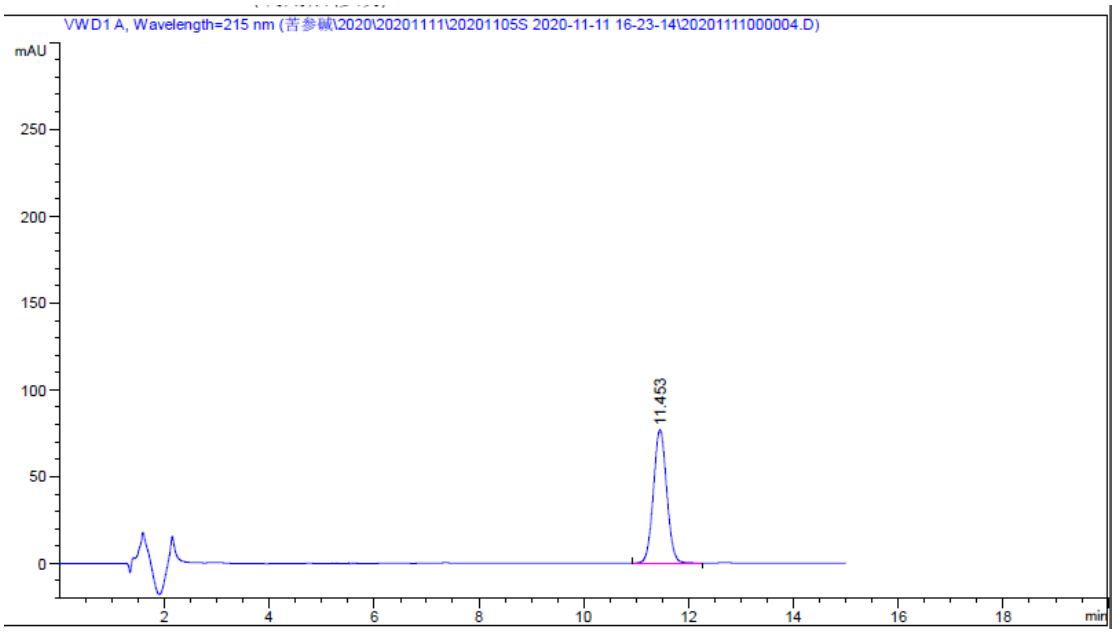


Fig. 2 Chromatogram of Matrine standard

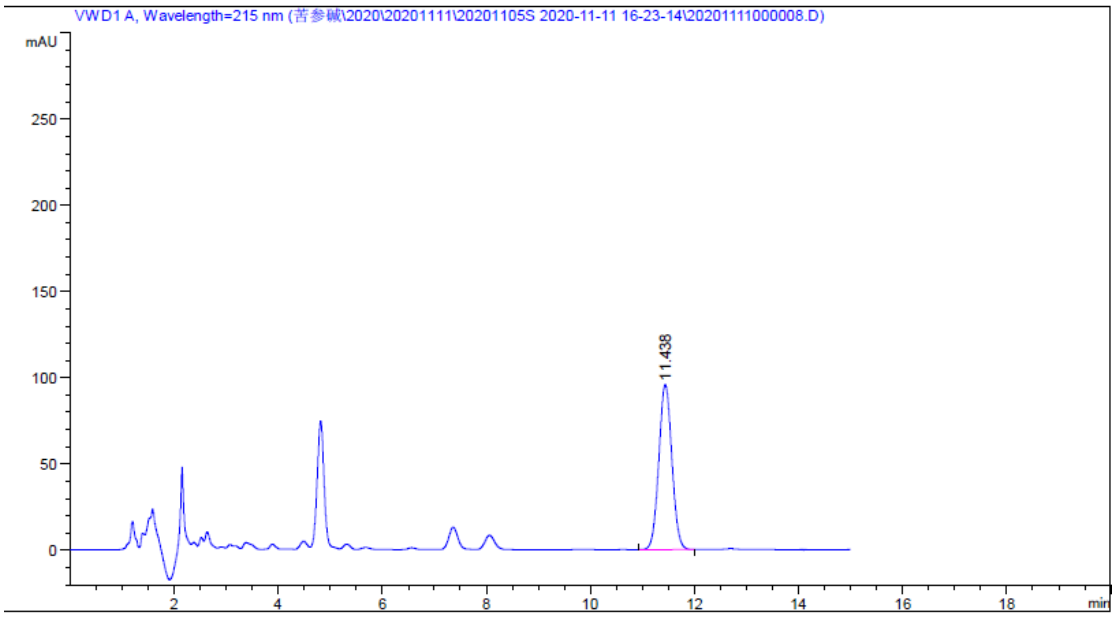


Fig. 3 Chromatogram of Matrine 10% TK sample

MATRINE SOLUBLE LIQUID

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1. **Sampling.** Take at least 1l.

2. **Identity tests.** As for Matrine technical concentrate******

3. **Matrine.** As for Matrine technical concentrate except:

(c)

Preparation of sample solution: Weigh 670mg (to the nearest 0.2mg) sample to contain about 2 mg Matrine into a 25 mL volumetric flask, dissolved and made to volume with Solution1 as sample stock solution. Mix thoroughly, the solution should be filtered through 0.45 μm filter film before use.

(d)Determination: Inject in duplicate 10μL portions of each sample solution bracketing them by injections of the calibration solutions as follows:

C_A, S_1, S_1, C_B, S_2, S_2, C_A, etc

(e)Calculation

$$f_i = \frac{S \times P}{H_S}$$

$$\text{Content of} = \frac{H_w \times f}{25 \times w} \quad \text{g/kg}$$

where:

fi=individual response factor

f= mean response factor

H_s=peak areas of Matrine in the calibration solution

H_w=peak areas of Matrine in the sample solution

s=mass of Matrine standard (mg)

w=mass of sample taken(mg)

P=purity of Matrine standard (g/kg)

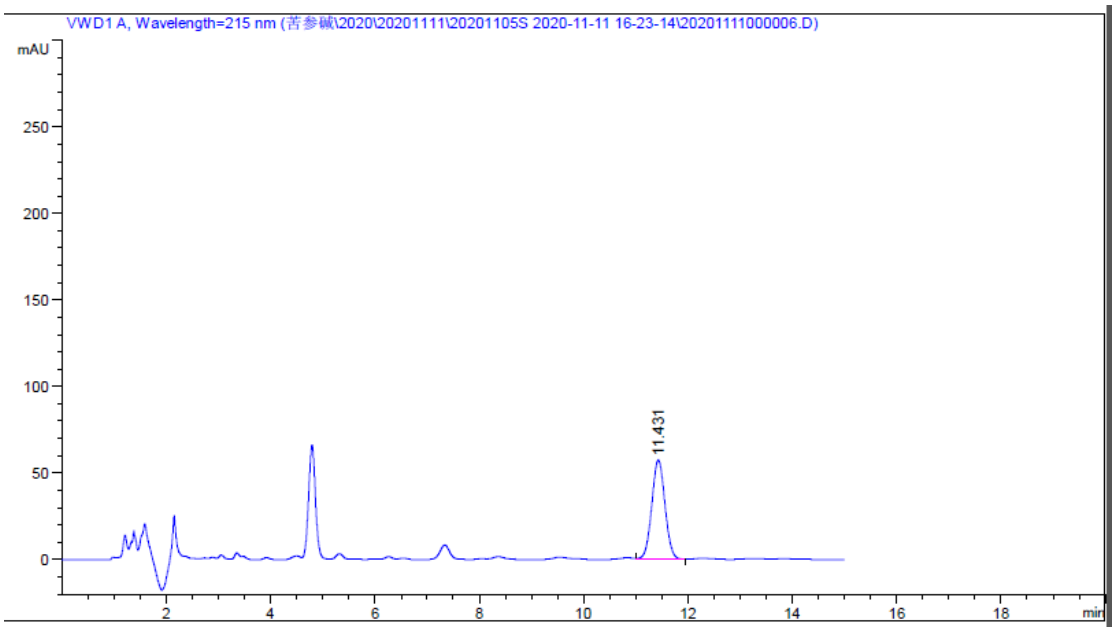

Fig. 4 Chromatogram of Matrine 0.3% SL sample