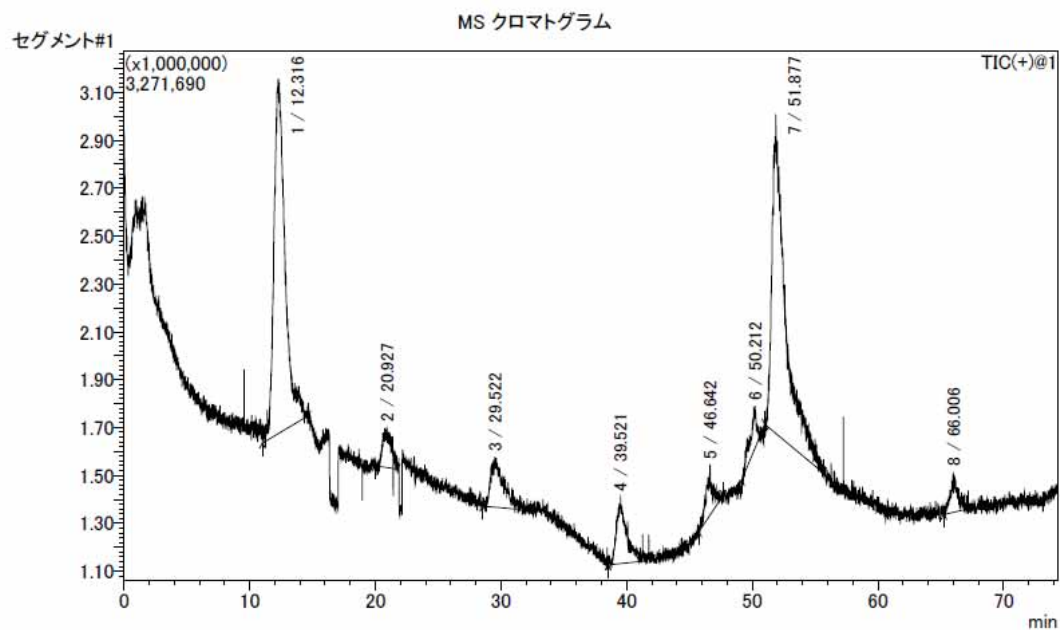


<クロマトグラム>



サンプル: Symphytum officinale, Crude base

カラム: InertSustain C18 5 μ m (250 $\times$ 4.6 mm) (GL Science)

溶離液: (A) 0.2 % ギ酸水溶液
(B) アセトニトリル

グラジエント: 0 min A:B=70:30
15 min A:B=0:100

流速: 0.4 mL/min

カラムオープン: 30 $^{\circ}$ C

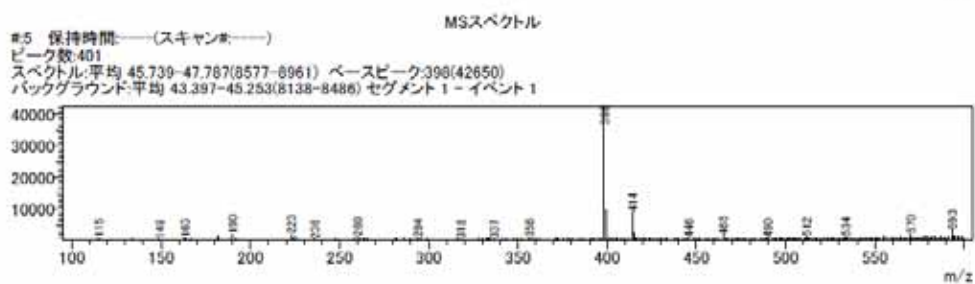
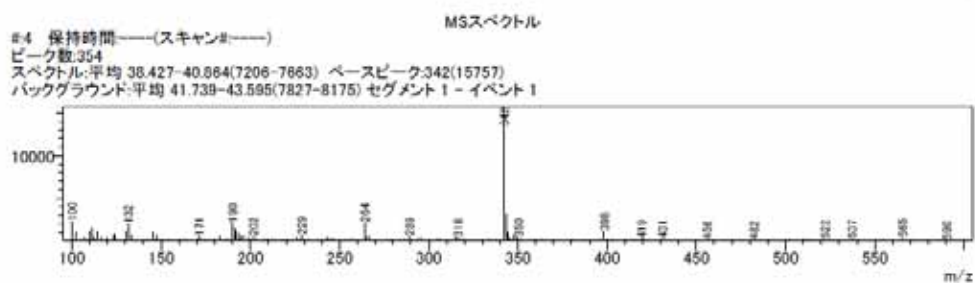
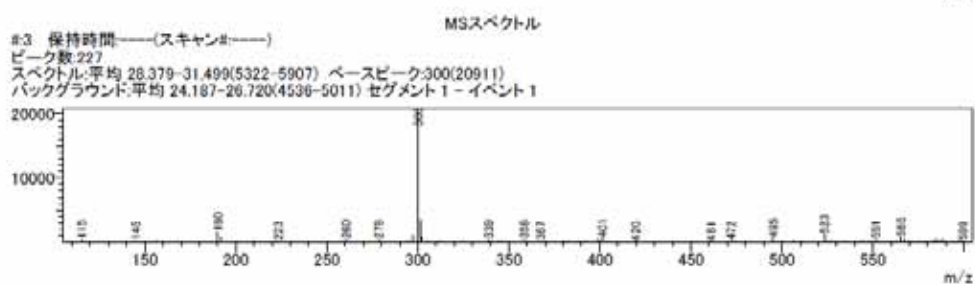
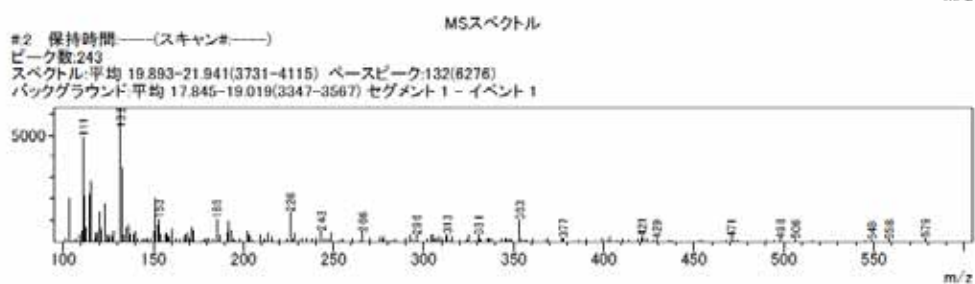
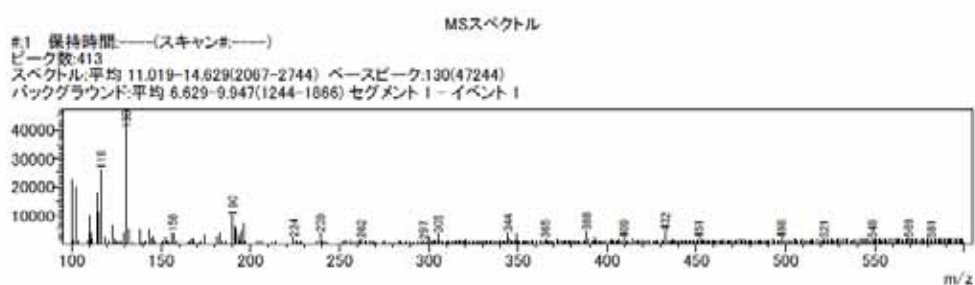
MS: ESI (positive)

m/z

300 $\rightarrow$ Lycopsamine

382 $\rightarrow$ Symphytine, Symviridine, Symlandine

398 $\rightarrow$ Echimidine

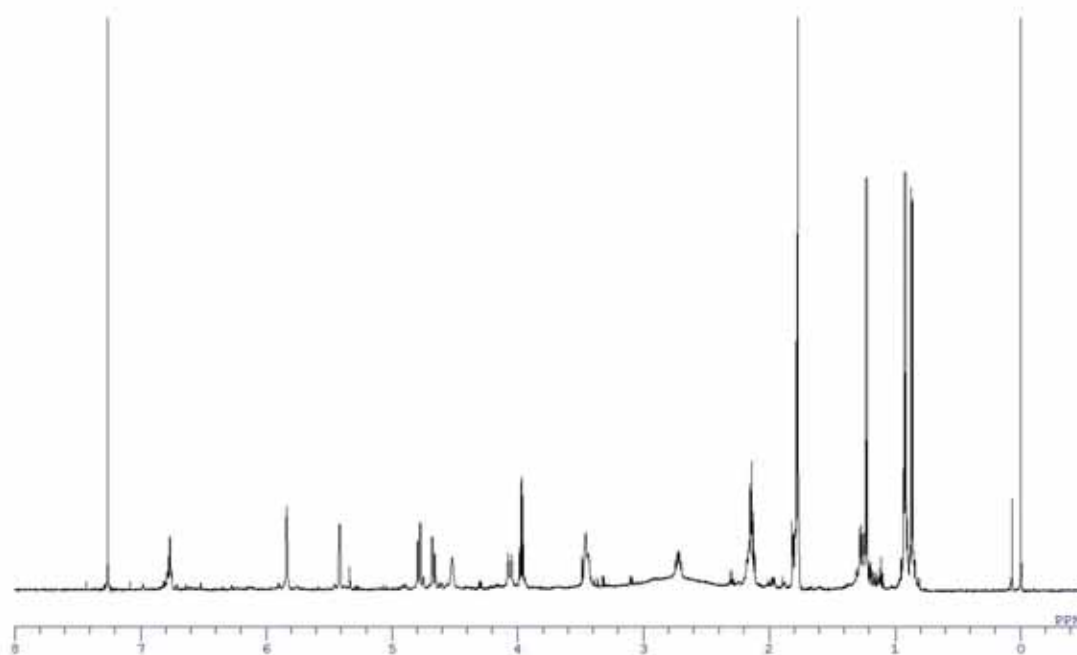


資料3 コンフリーより分離した化合物 **1-4** の NMR スペクトル

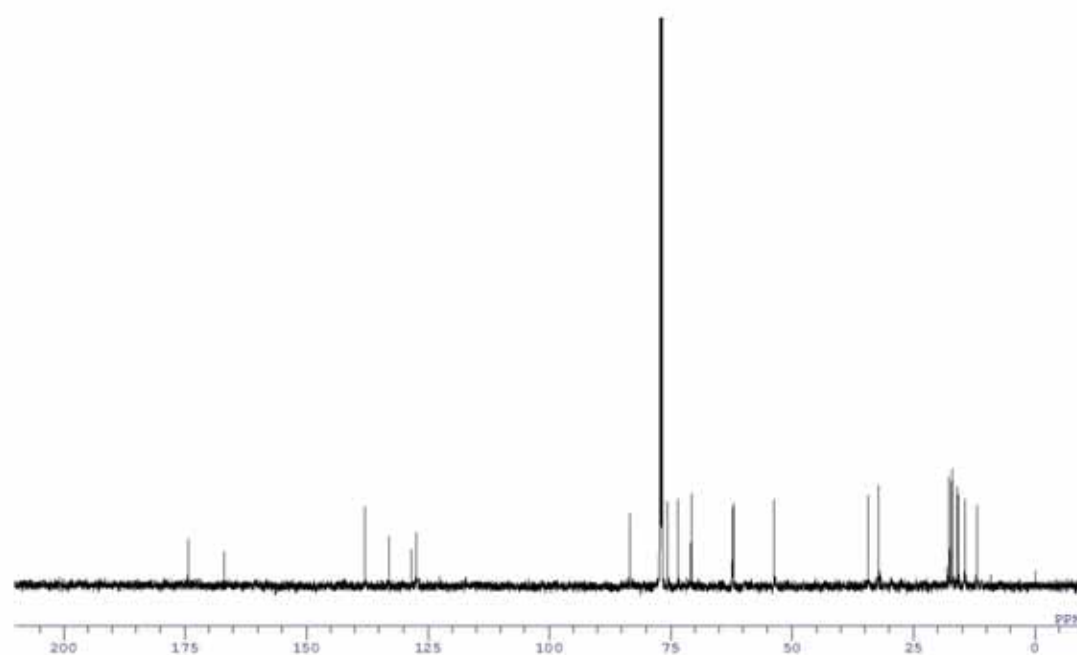
資料 3-1

Symphytine (**1**)

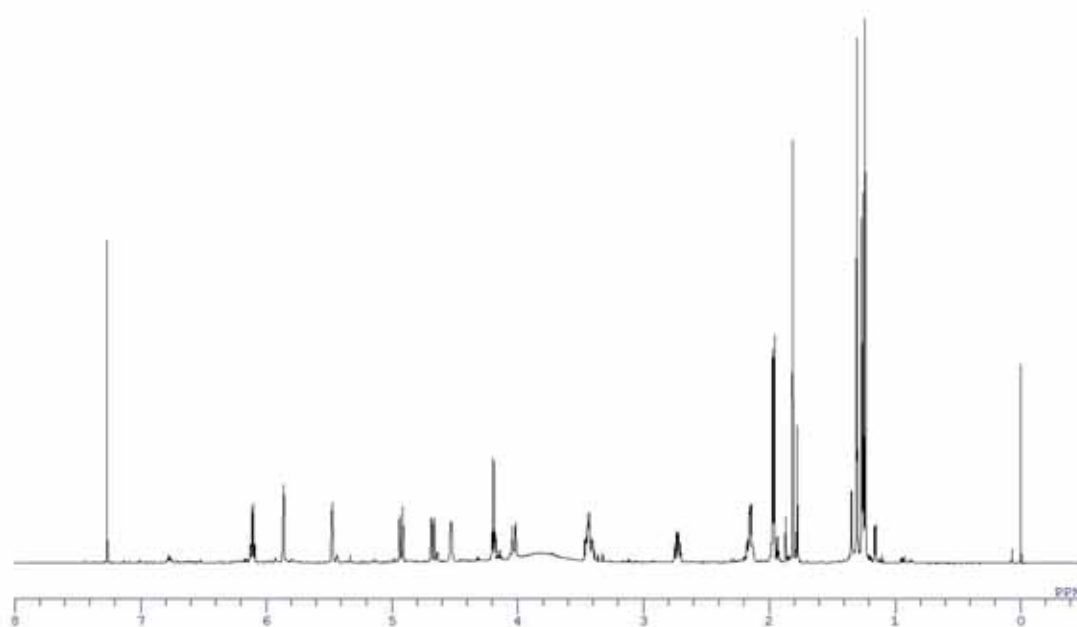
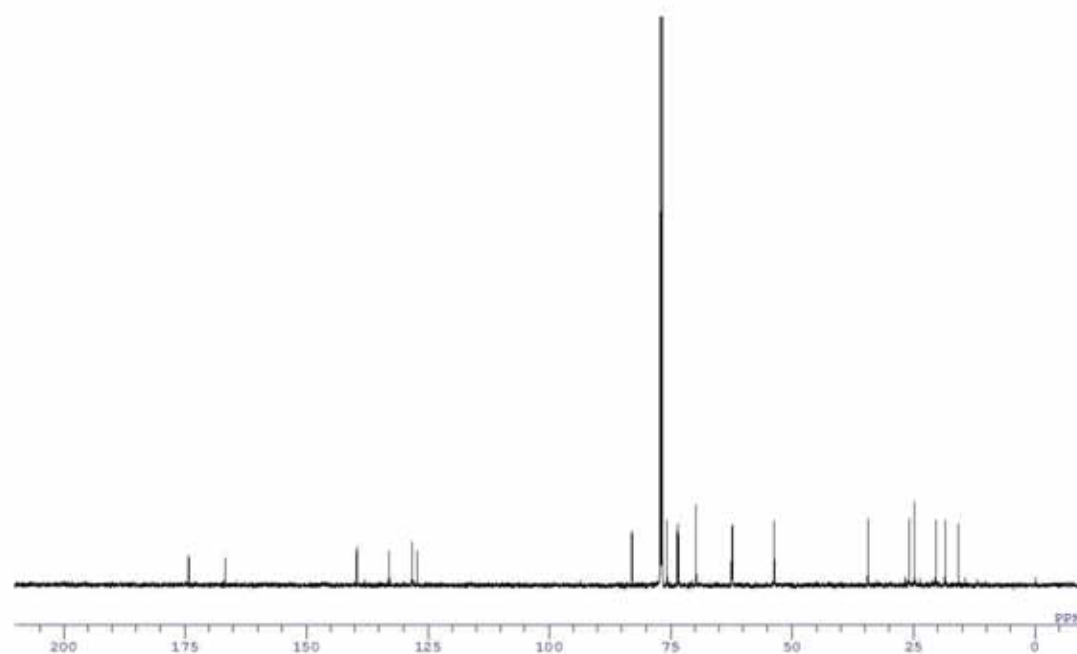
^1H NMR (600 MHz, CDCl_3)

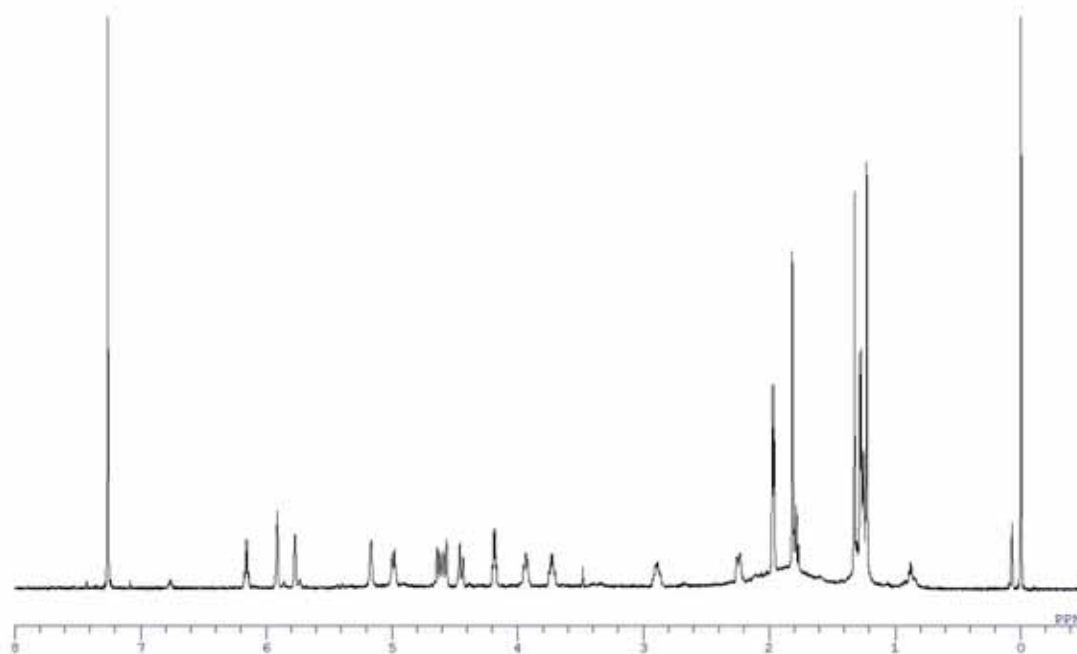
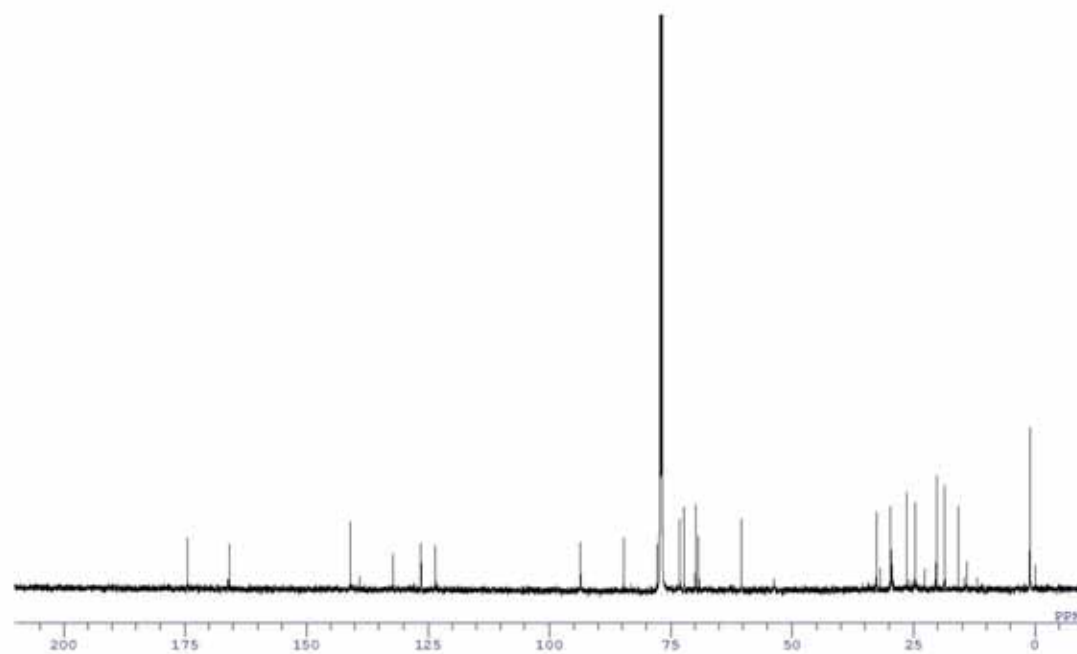


^{13}C NMR (150 MHz, CDCl_3)

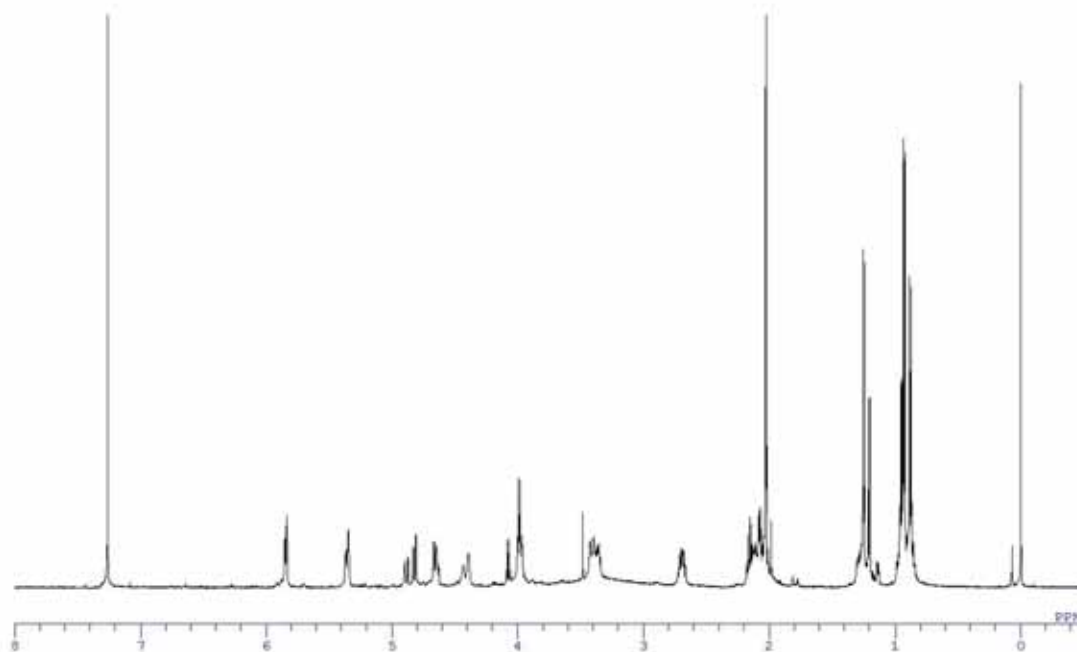
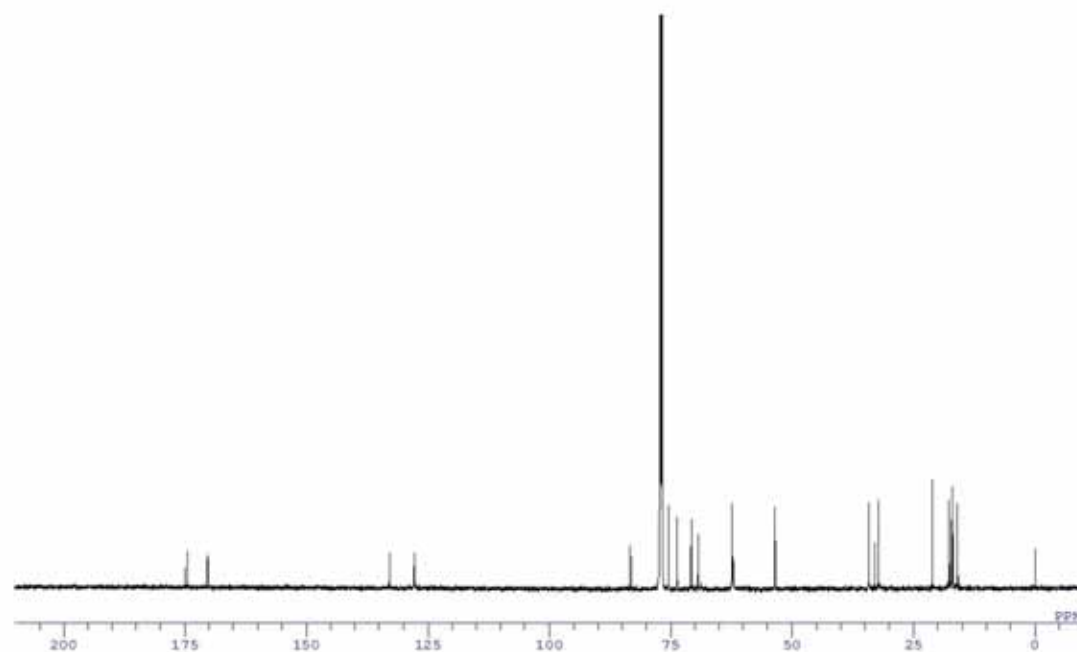


Echimidine (2)

 ^1H NMR (600 MHz, CDCl_3) ^{13}C NMR (150 MHz, CDCl_3)

Echimidine *N*-oxide (**3**) ^1H NMR (600 MHz, CDCl_3) ^{13}C NMR (150 MHz, CDCl_3)

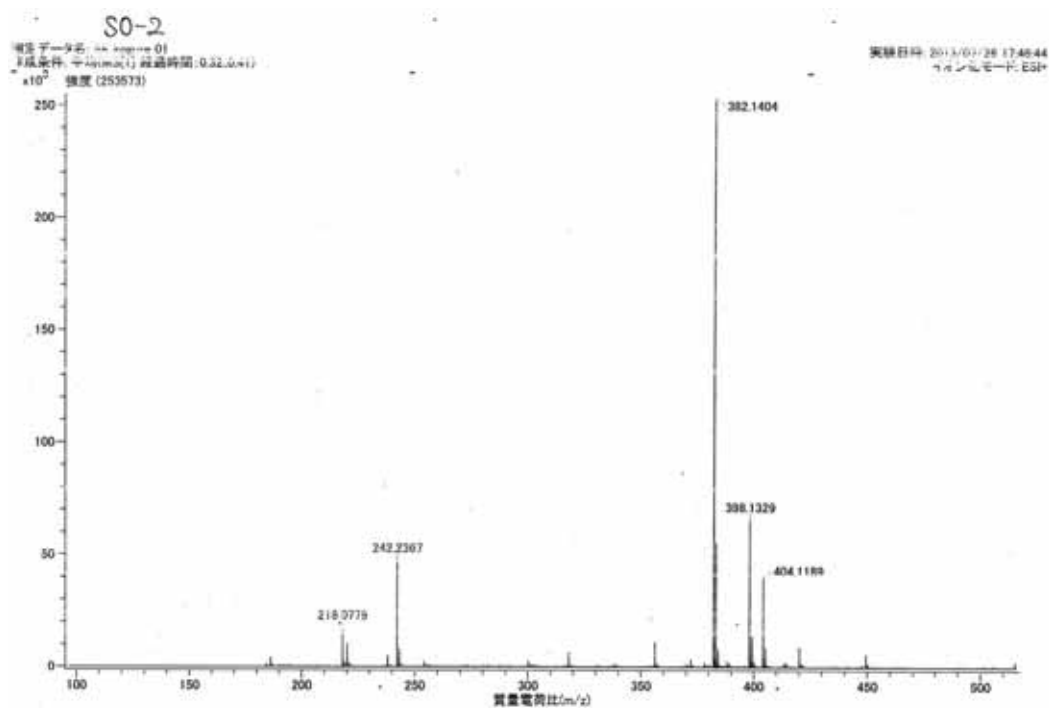
Lycopsamine (4)

 ^1H NMR (600 MHz, CDCl_3) ^{13}C NMR (150 MHz, CDCl_3)

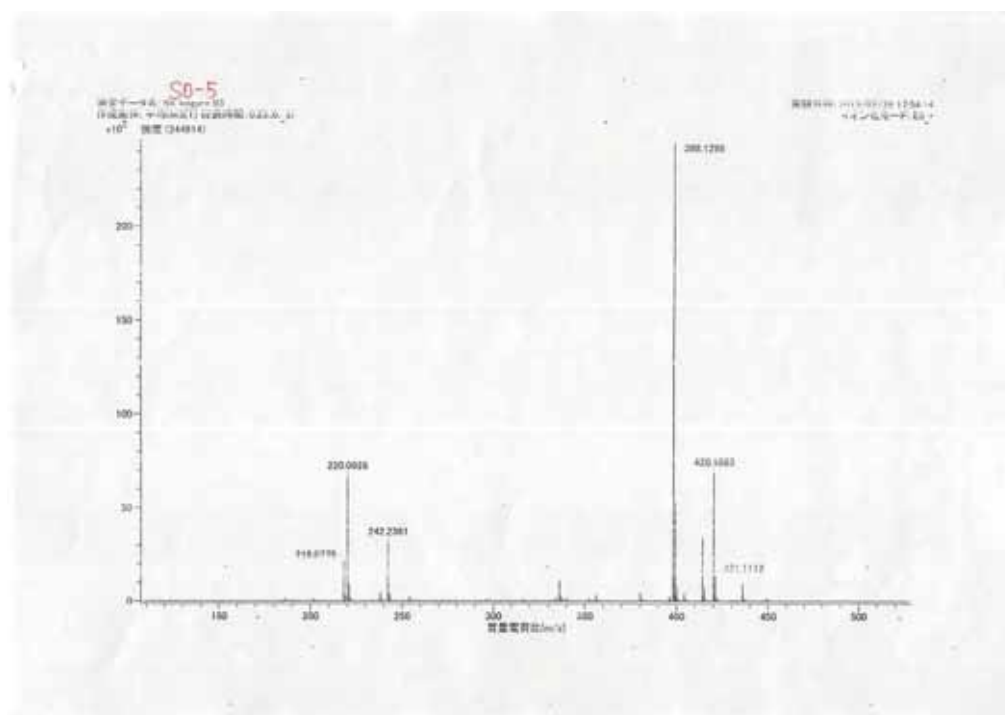
資料4 コンフリーより分離した化合物 **1-3** の ESI-MS スペクトル

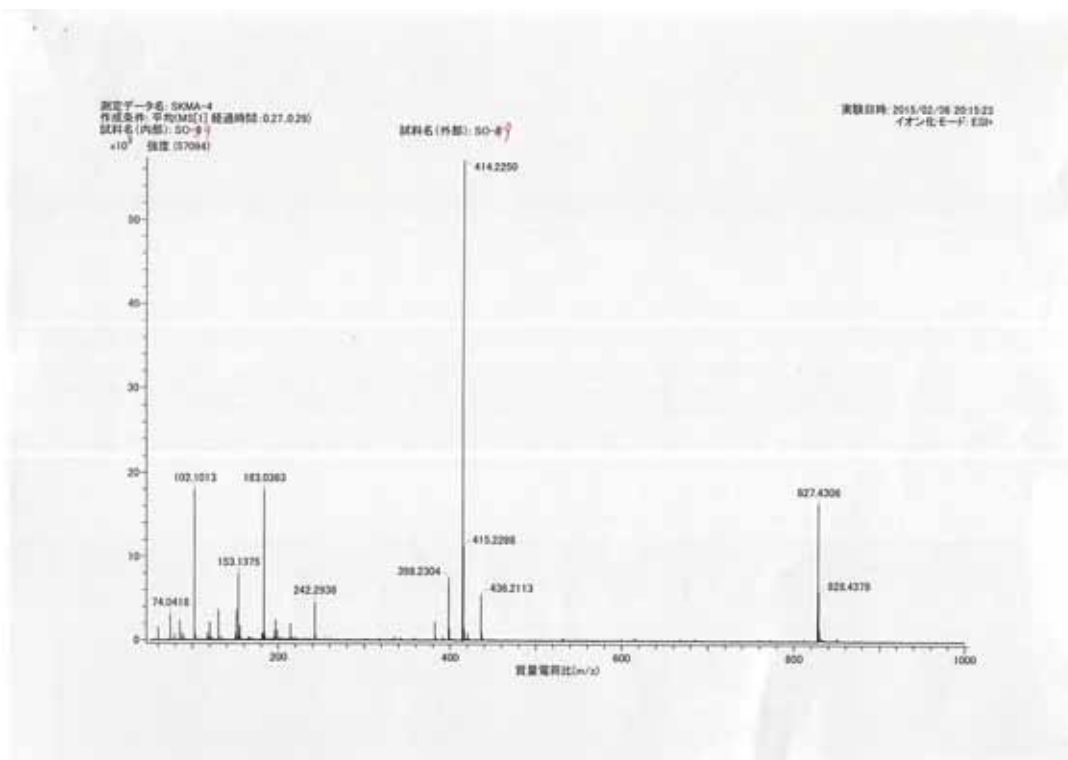
資料 4-1

Symphytine (1)



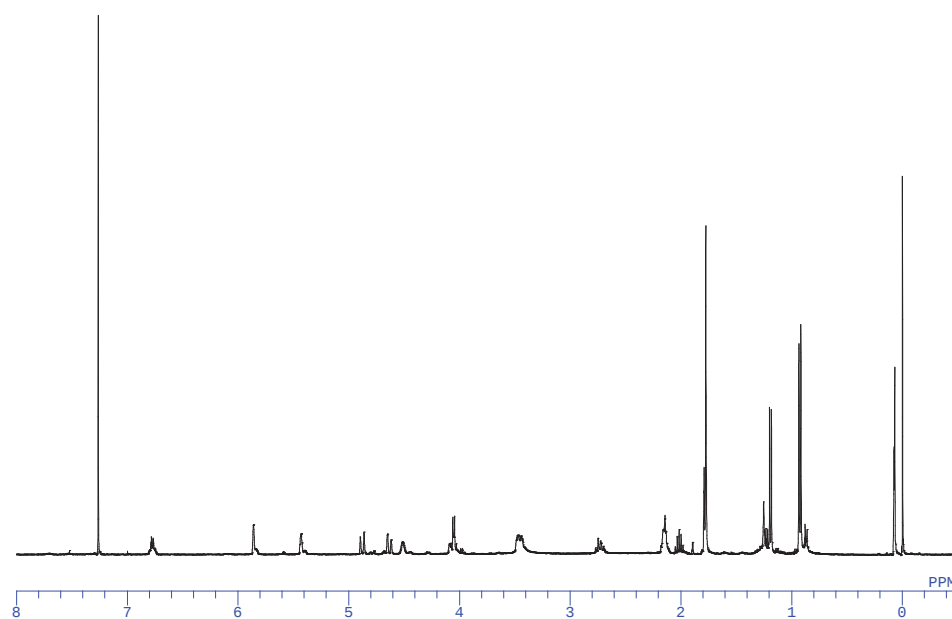
Echimidine (2)



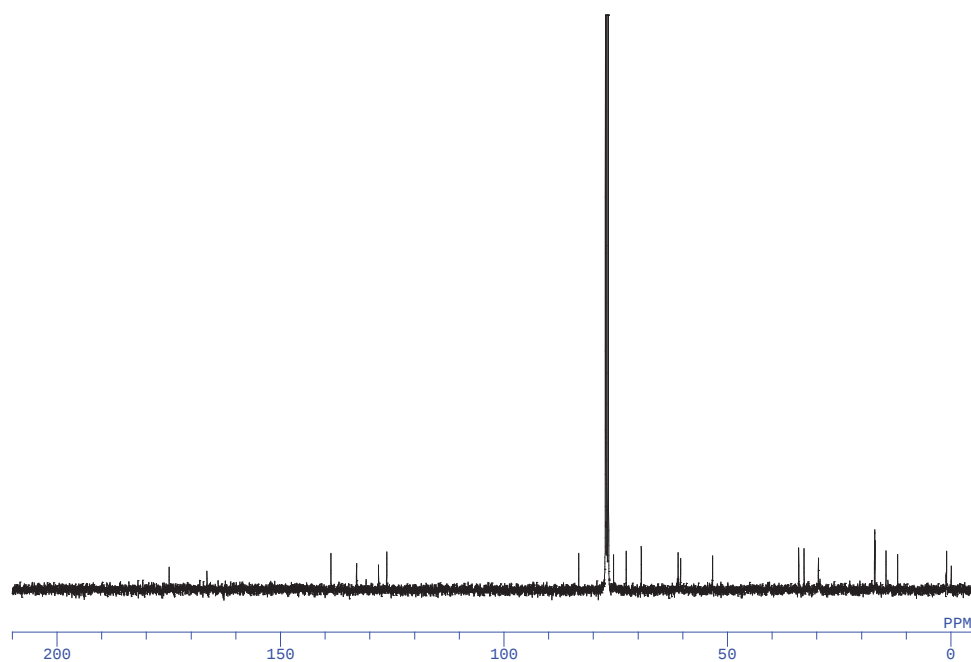
Echimidine *N*-oxide (3)

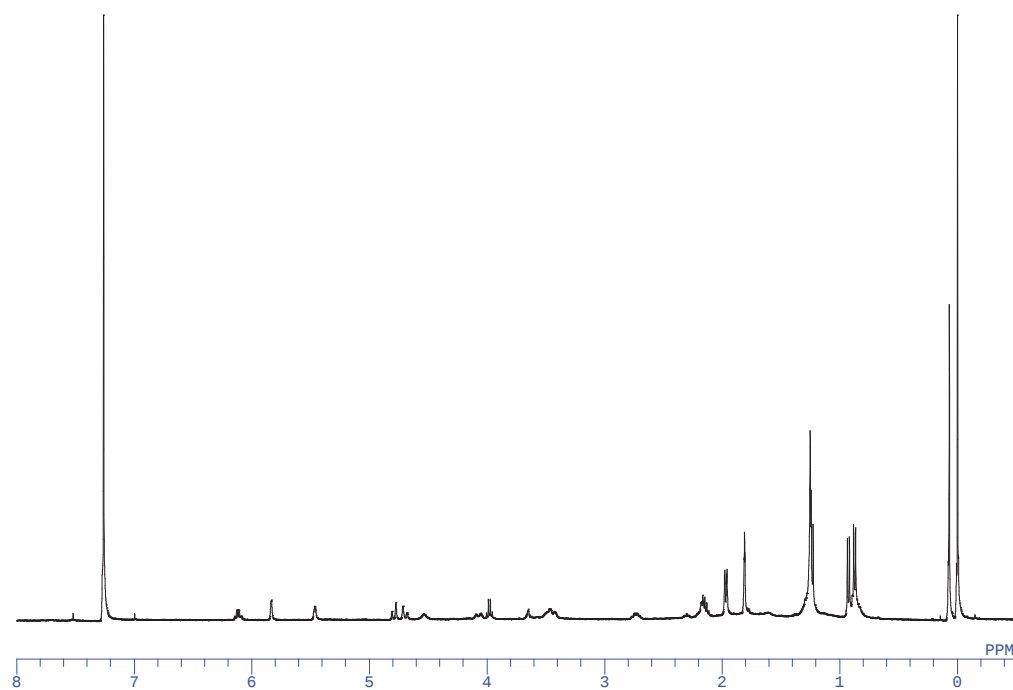
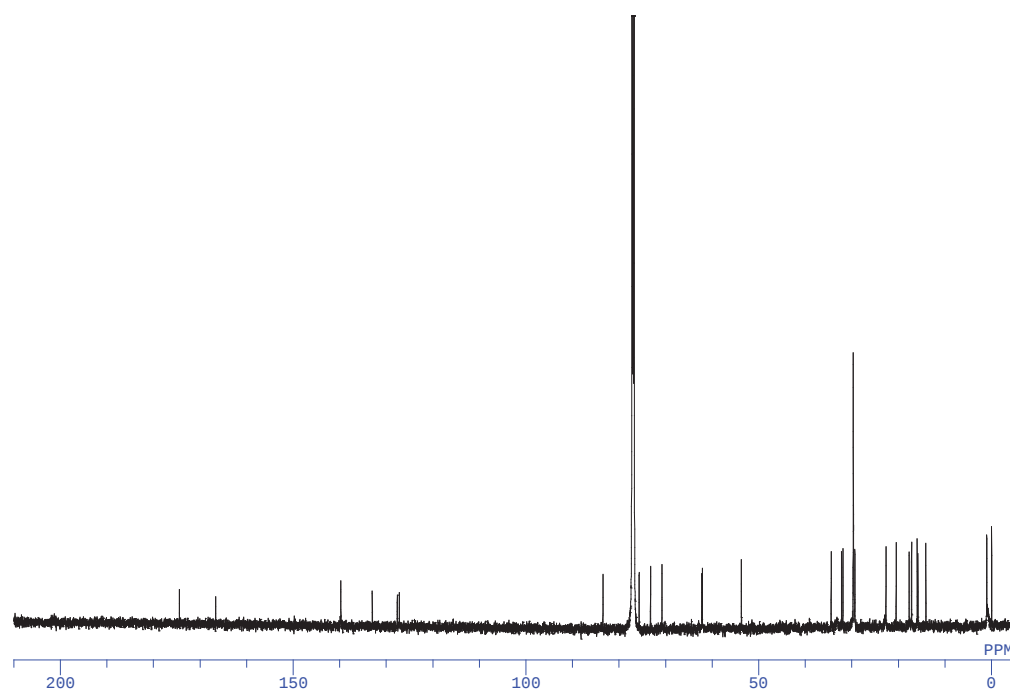
Myoscorpine (**10**)

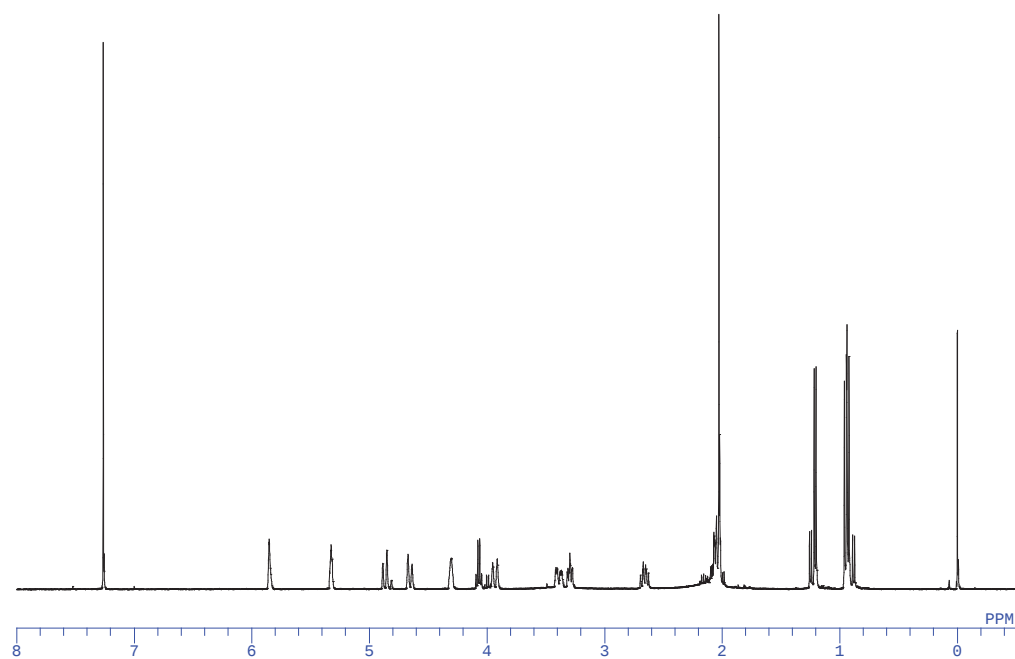
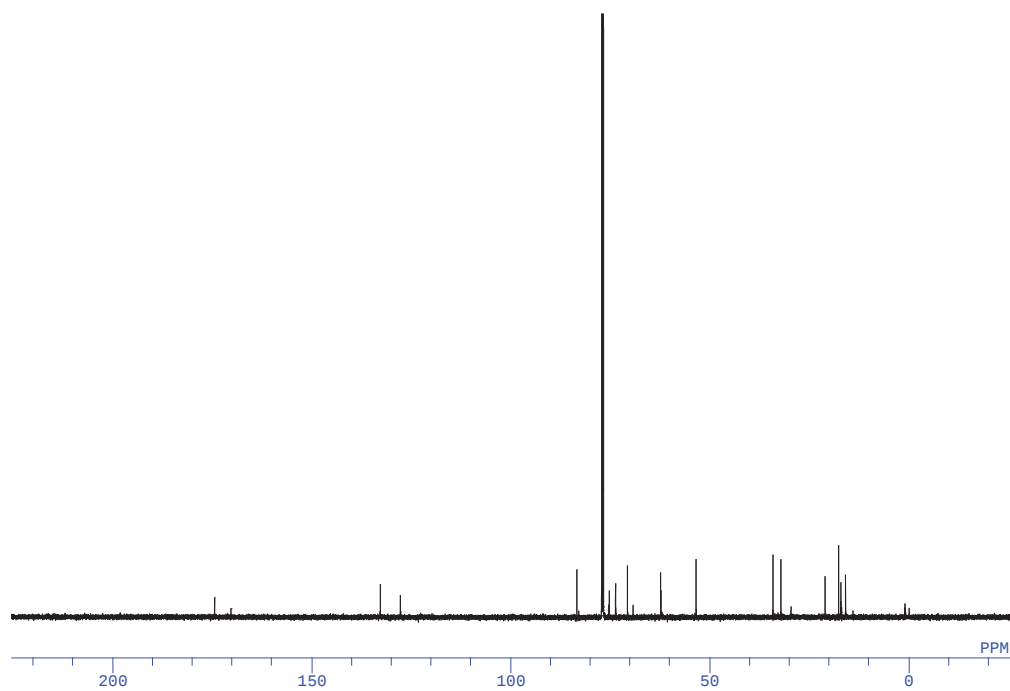
^1H NMR (400 MHz, CDCl_3)

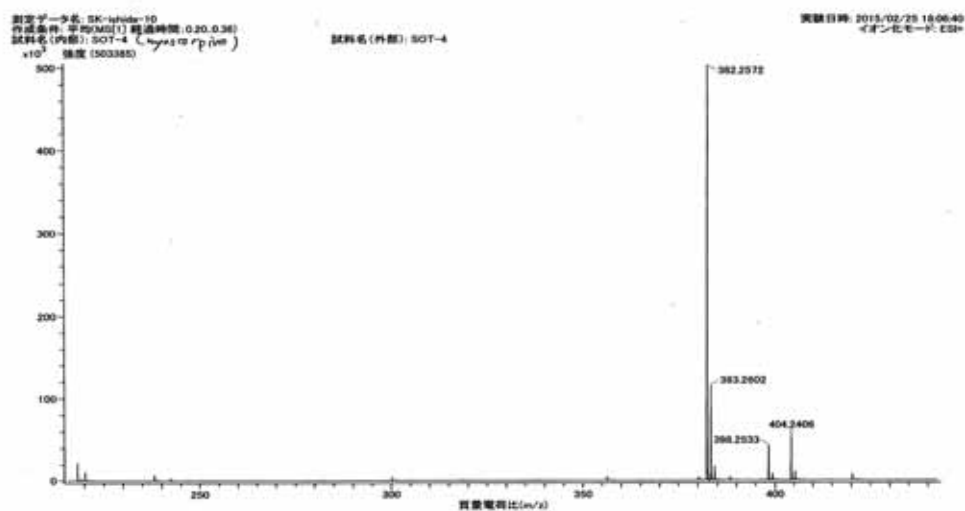
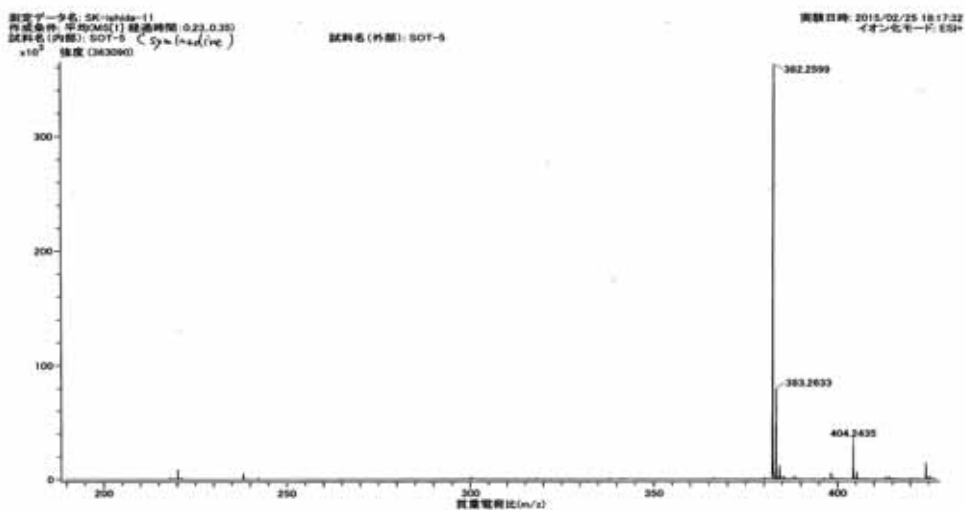


^{13}C NMR (100 MHz, CDCl_3)

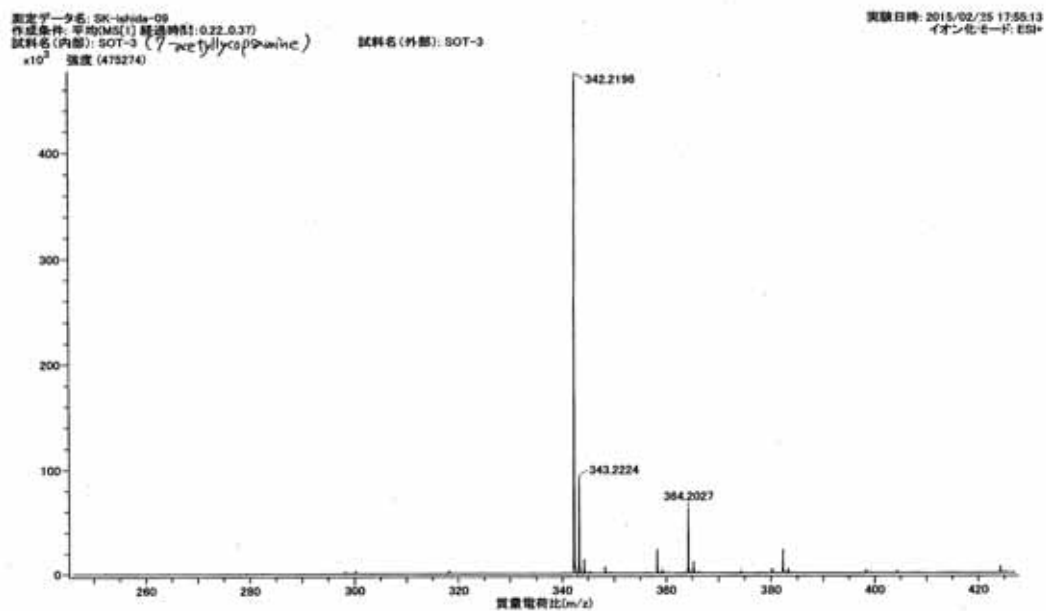


Symlandine (**11**) ^1H NMR (400 MHz, CDCl_3) ^{13}C NMR (150 MHz, CDCl_3)

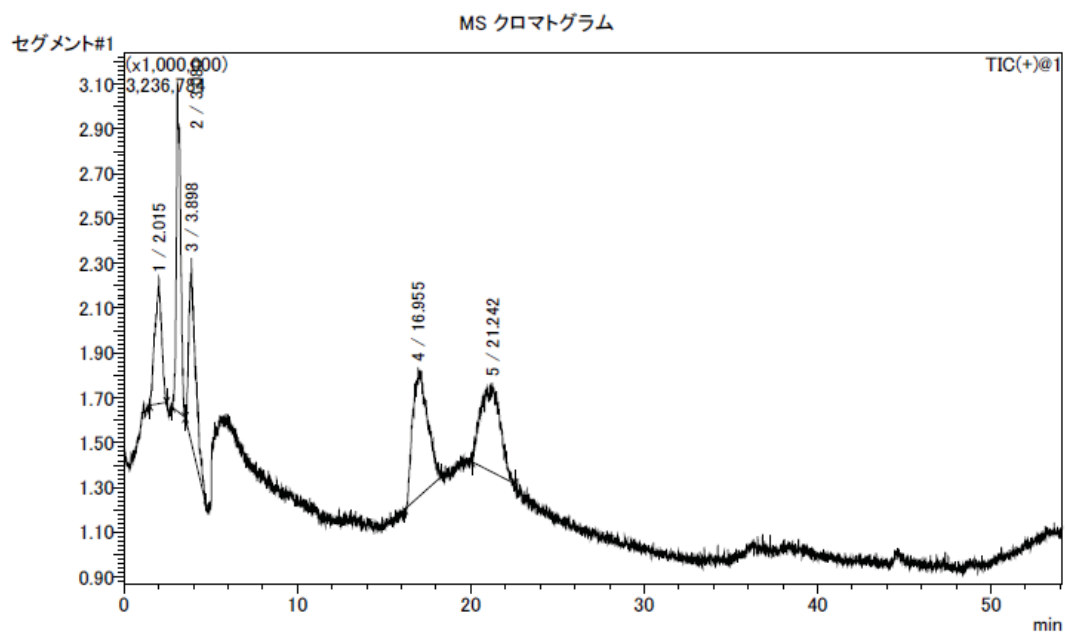
7-Acetyllycopsamine (**12**) ^1H NMR (400 MHz, CDCl_3) ^{13}C NMR (150 MHz, CDCl_3)

Myoscorpine (**10**)Symlandine (**11**)

7-Acetyllycopamine (12)



〈クロマトグラム〉



サンプル: フキ(Petasites japonicus), Crude base

カラム: InertSustain C18 5 μ m (250 $\times$ 4.6 mm) (GL Science)

溶離液: (A) 0.2 % ギ酸水溶液
(B) アセトニトリル

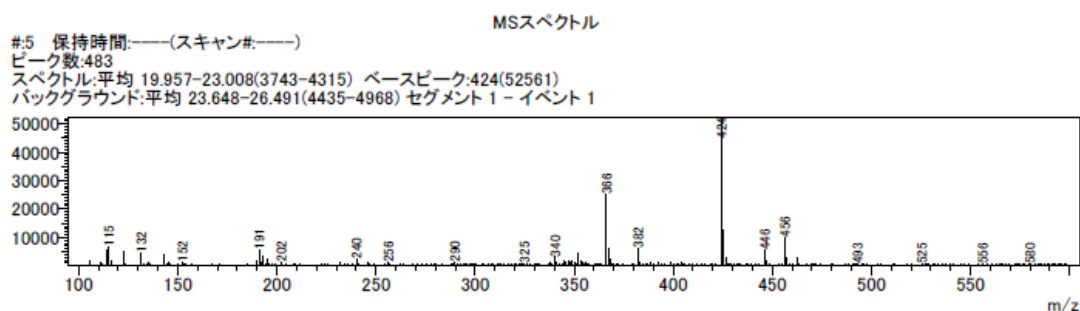
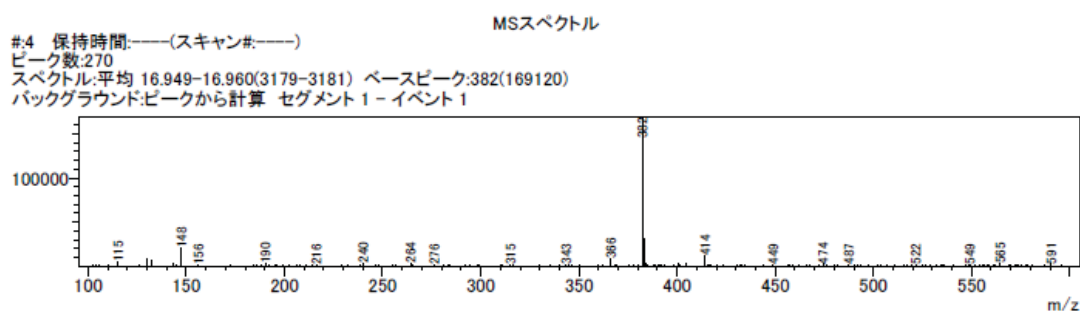
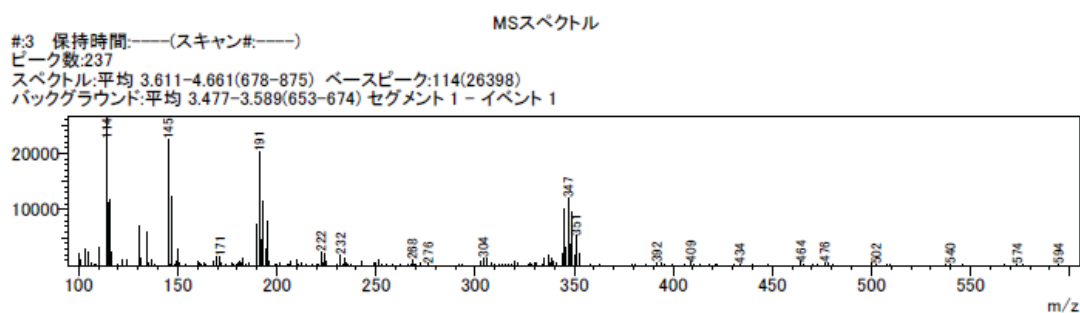
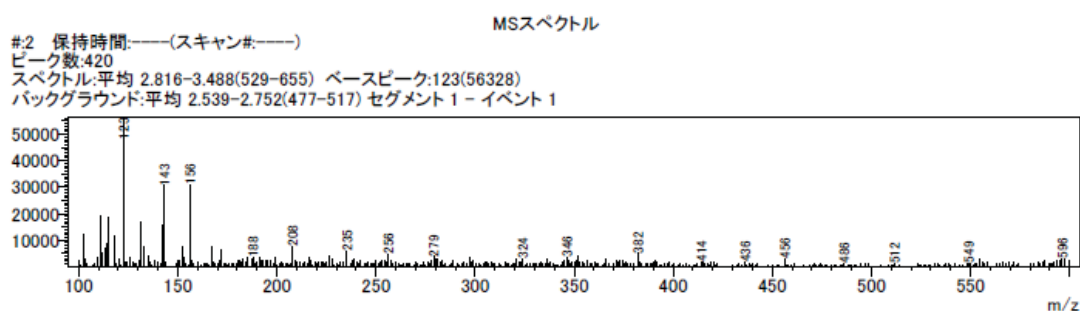
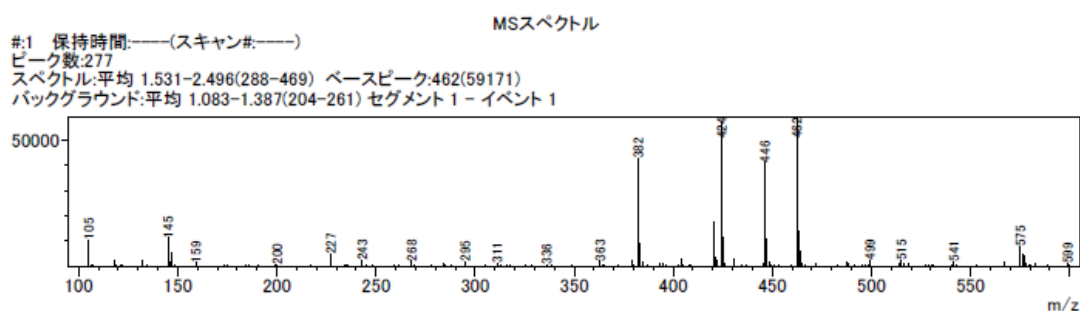
グラジエント: 0 min A:B=70:30
15 min A:B=0:100

流速: 0.4 mL/min

カラムオーブン: 30 $^{\circ}$ C

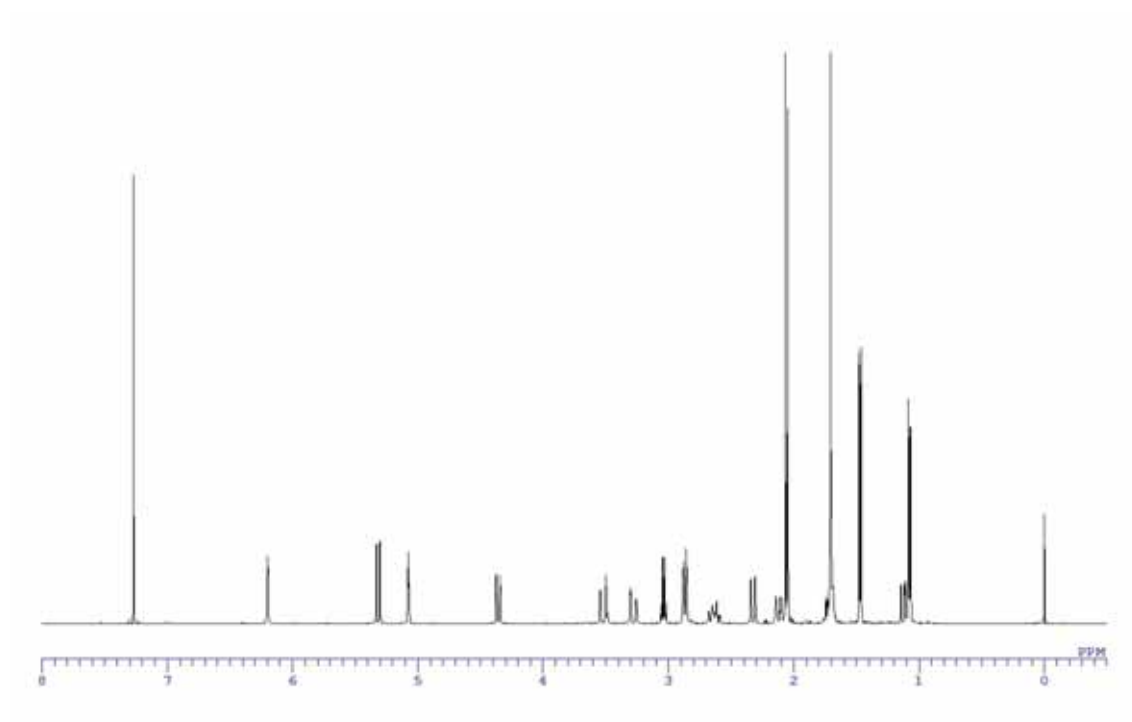
MS: ESI (positive)

m/z
366 $\rightarrow$ Senkirkine
382 $\rightarrow$ Petasitenine(Fukinotoxin)
424 $\rightarrow$ Neopetasitenine

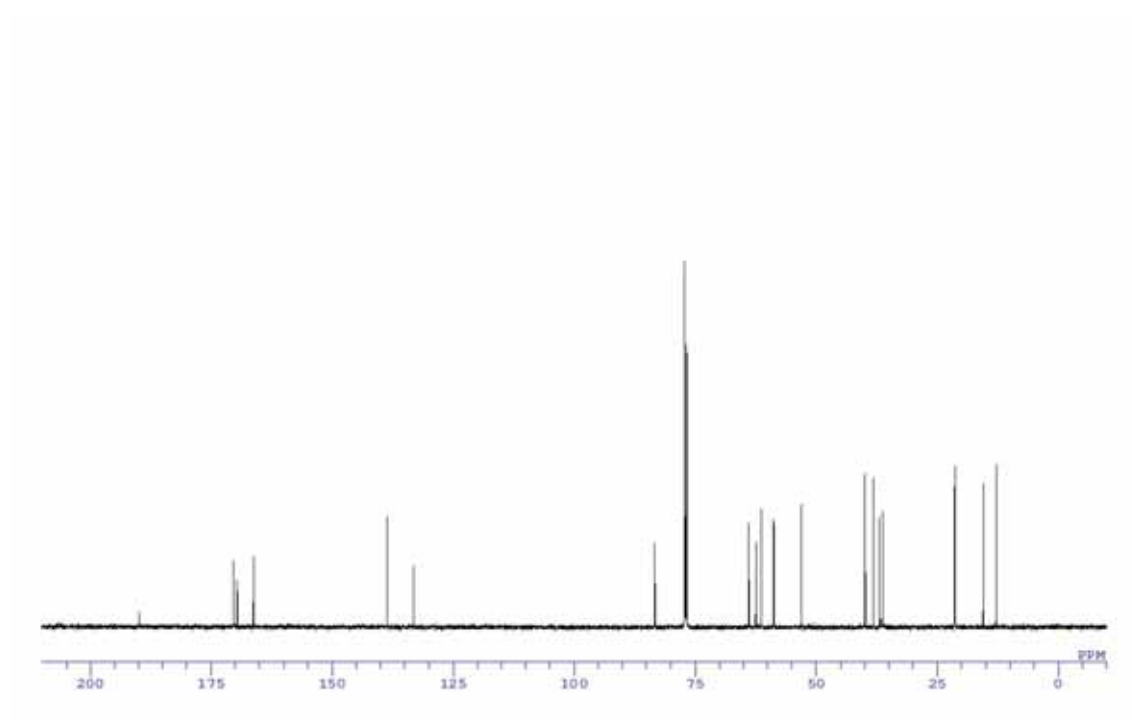


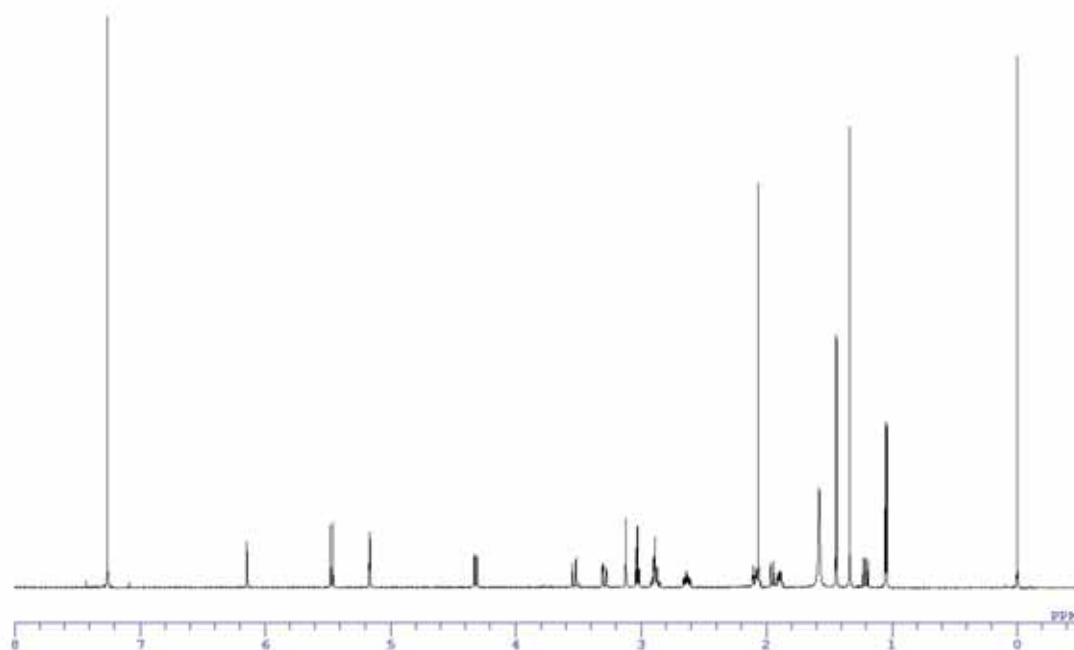
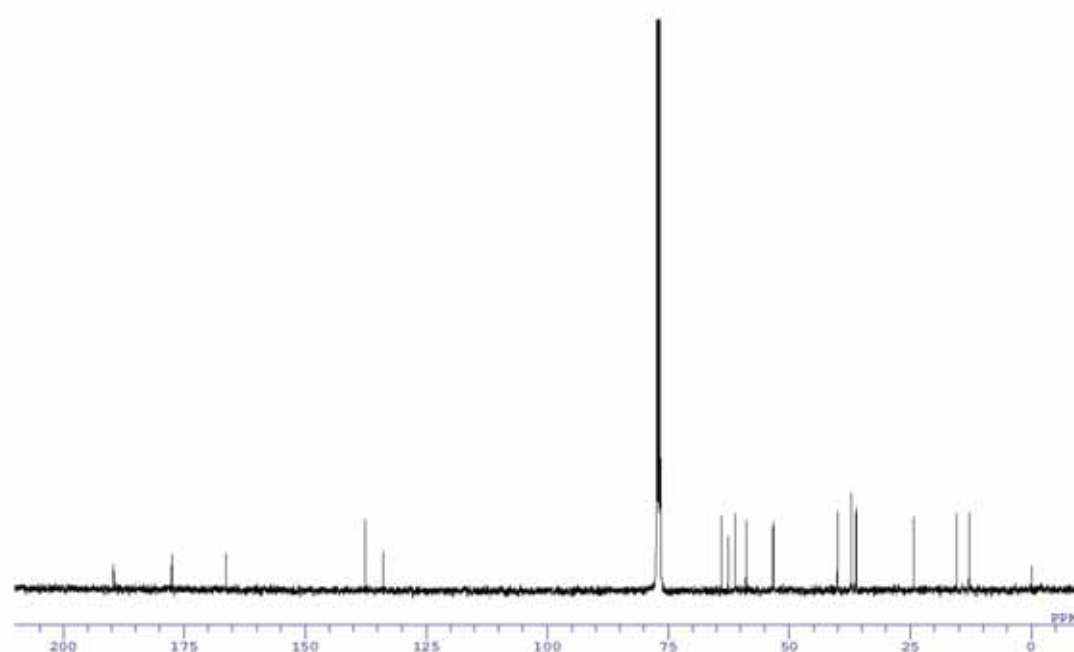
Neopetasitenine (**13**)

^1H NMR (400 MHz, CDCl_3)

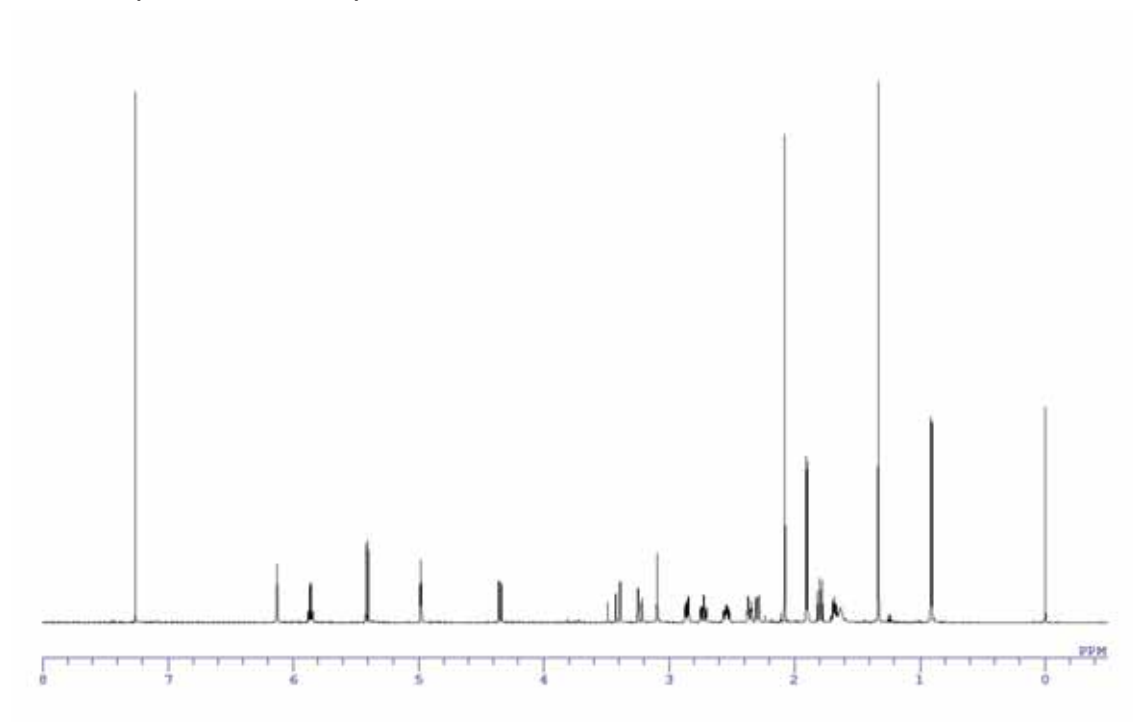
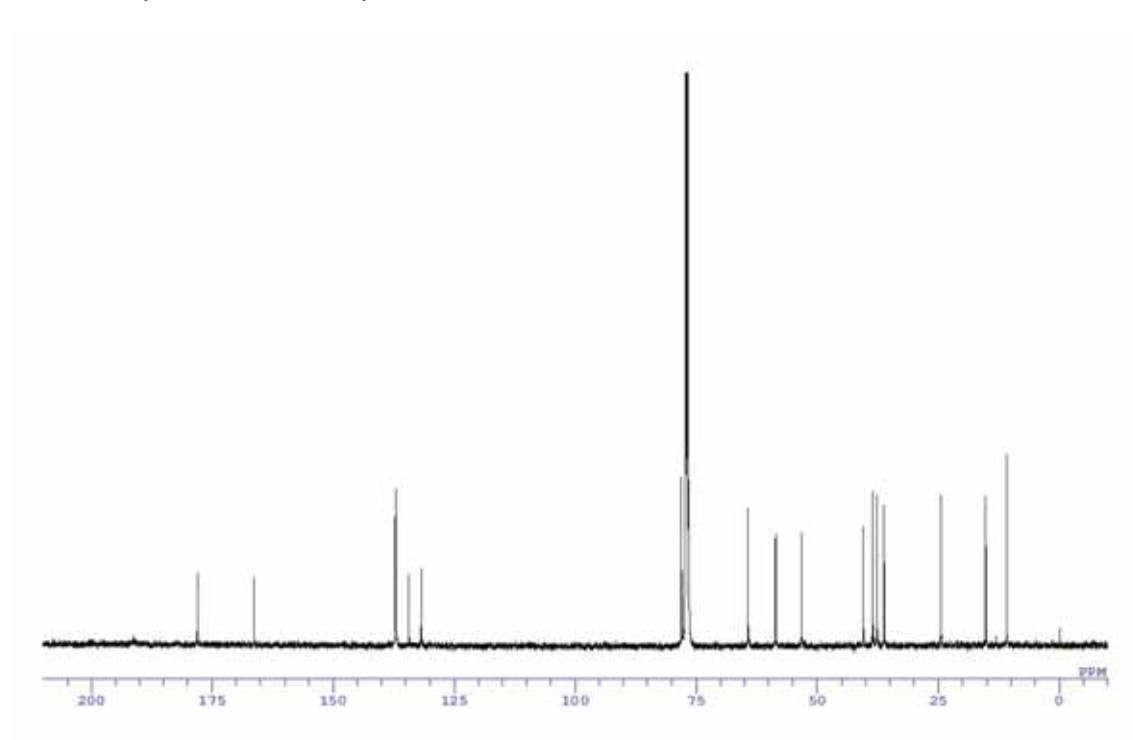


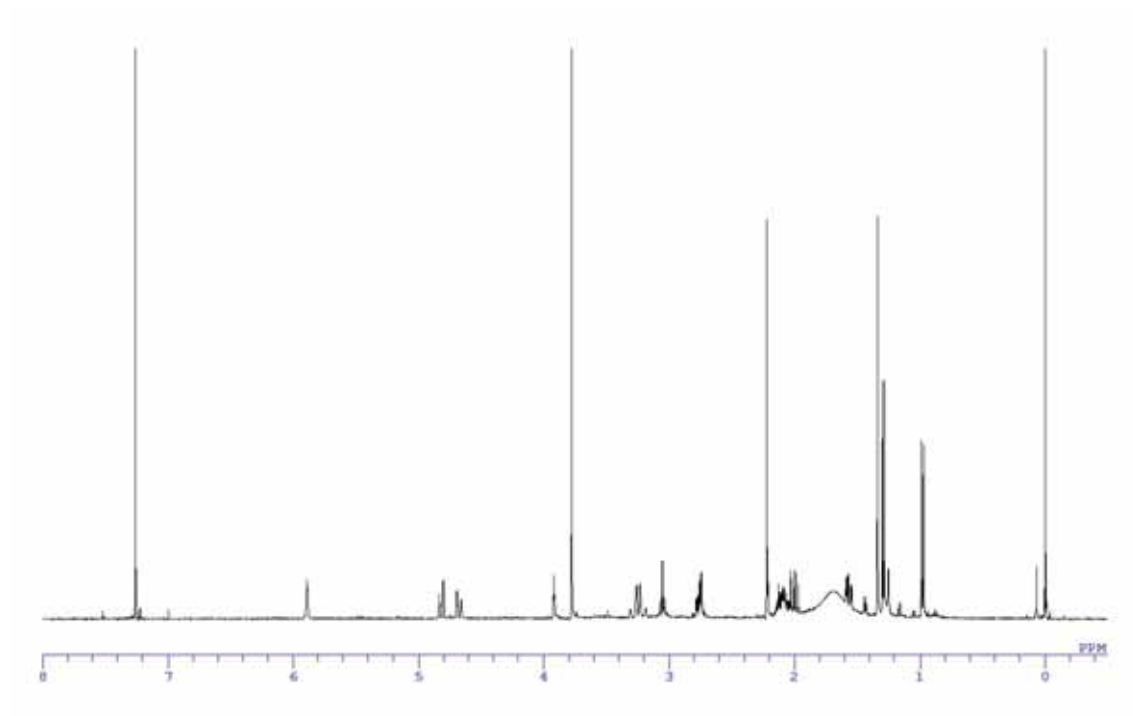
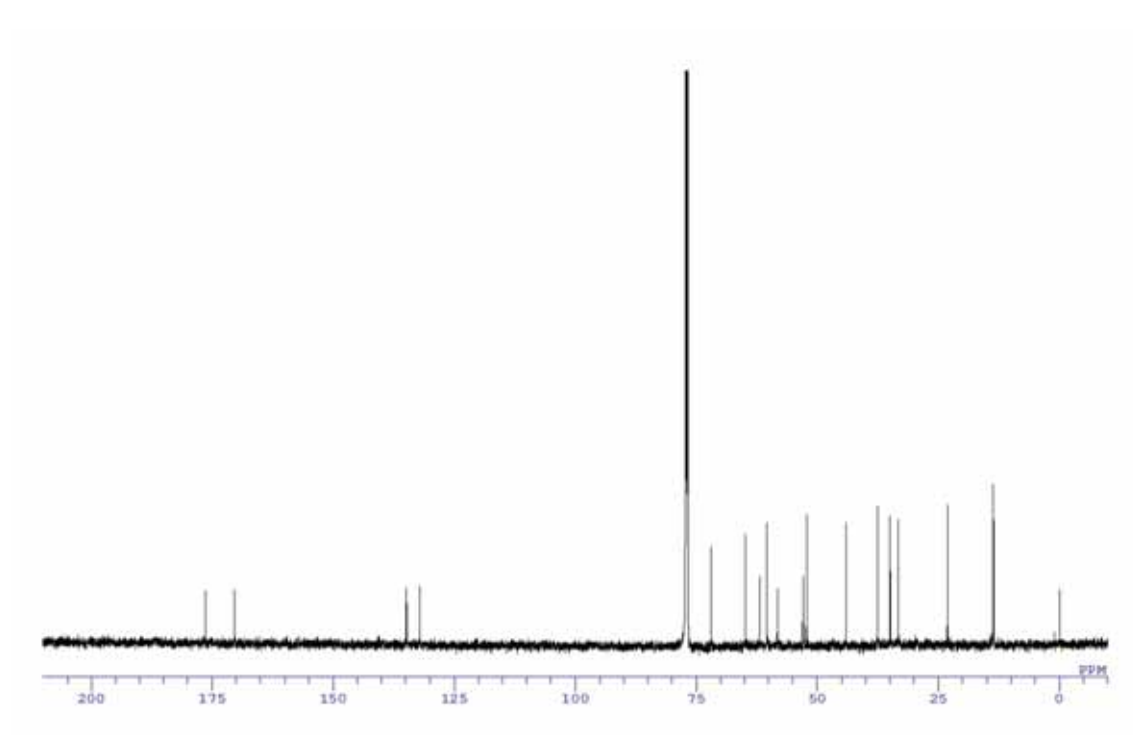
^{13}C NMR (150 MHz, CDCl_3)

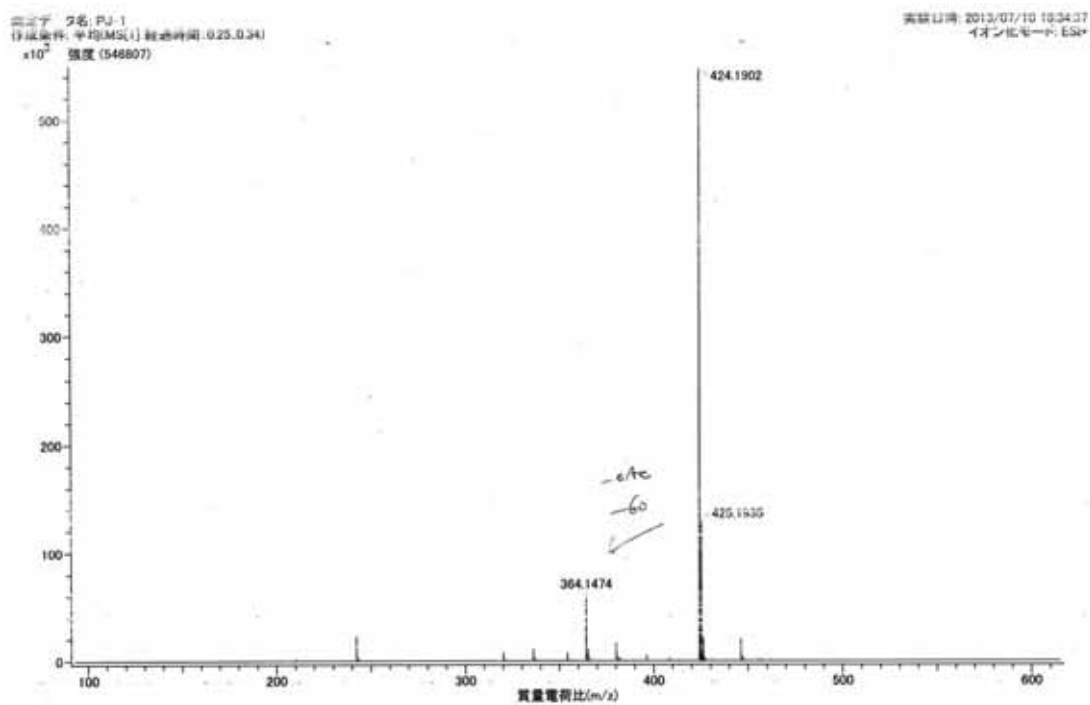
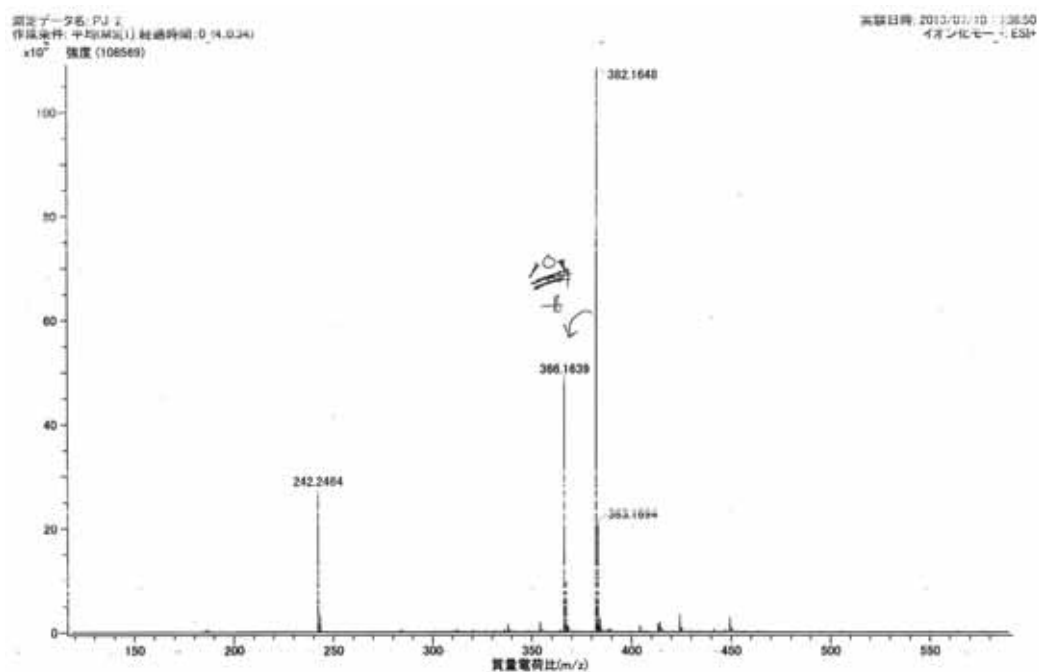


Petasitenine (**14**) ^1H NMR (600 MHz, CDCl_3) ^{13}C NMR (100 MHz, CDCl_3)

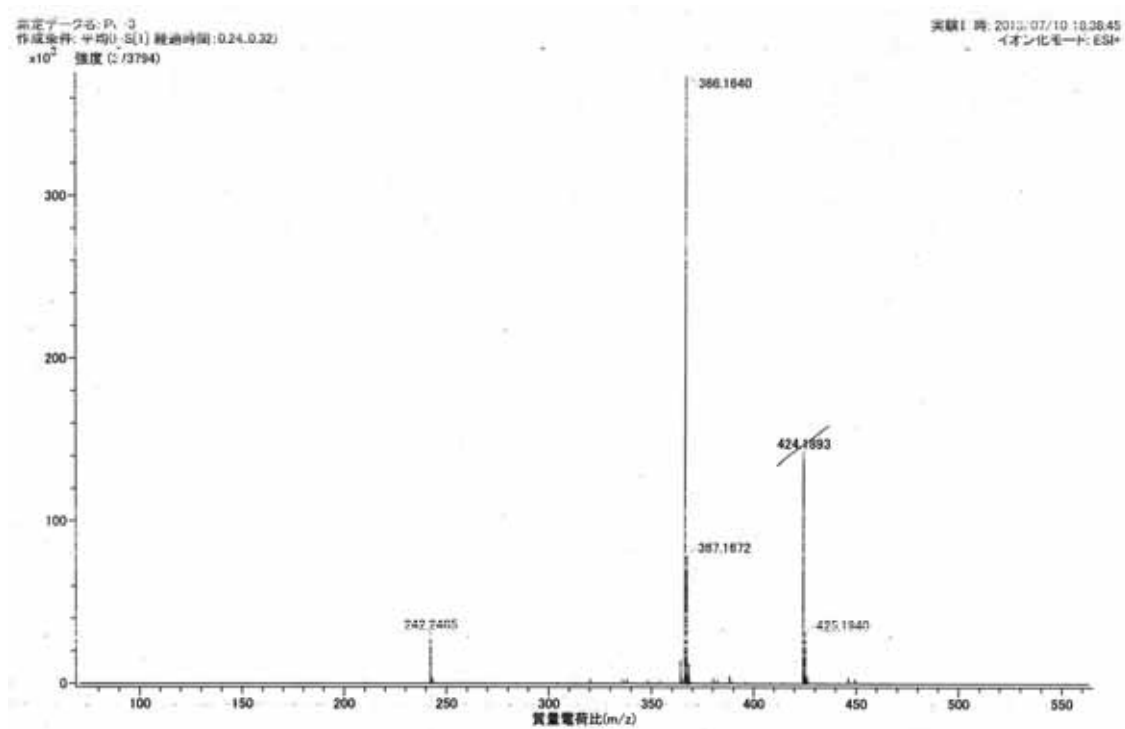
Senkirkine (15)

 ^1H NMR (600 MHz, CDCl_3) ^{13}C NMR (150 MHz, CDCl_3)

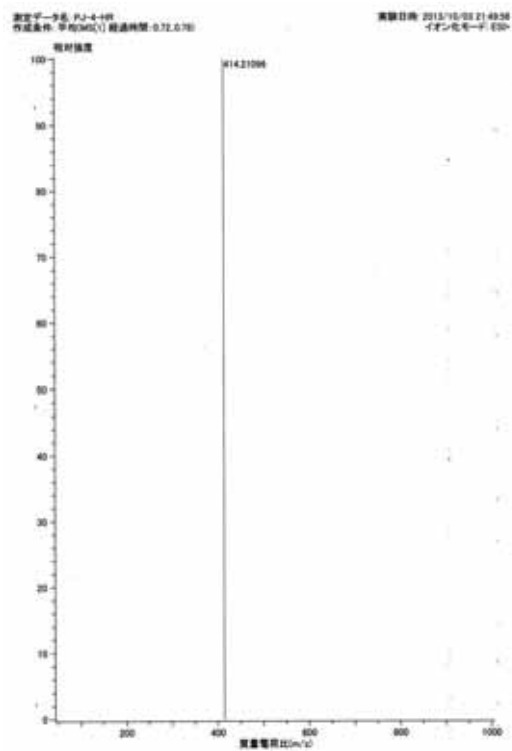
PJ-4 (**16**) (New alkaloid) ^1H NMR (400 MHz, CDCl_3) ^{13}C NMR (150 MHz, CDCl_3)

Neopetasitenine (**13**)Petasitenine (**14**)

Senkirkine (15)



PJ-4 (16)



Condition

Column: Waters Atlantis dC18 5 μ m (250 $\times$ 4.6 mm I.D.)Eluent: A 0.1 v/v% HCO₂H aq., B MeOH; A 55 %, B 45 % (0-20 min)

Flow rate: 0.8 mL/min

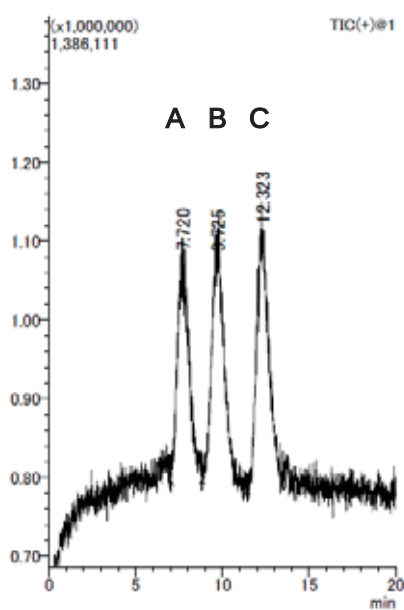
Column temp.: 30 $^{\circ}$ C

Detection: ESI-MS (positive mode, MS analysis)

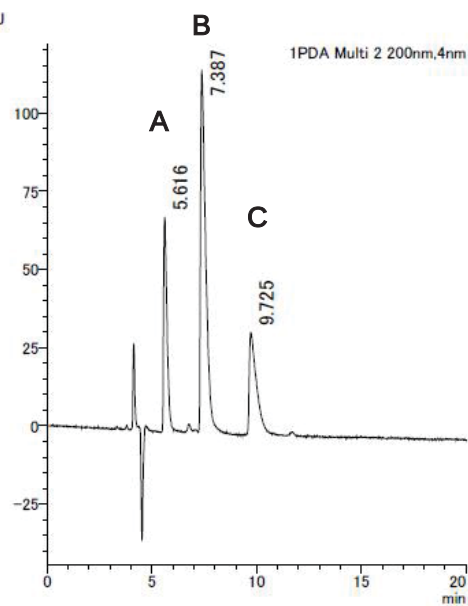
UV (200 nm, PDA analysis)

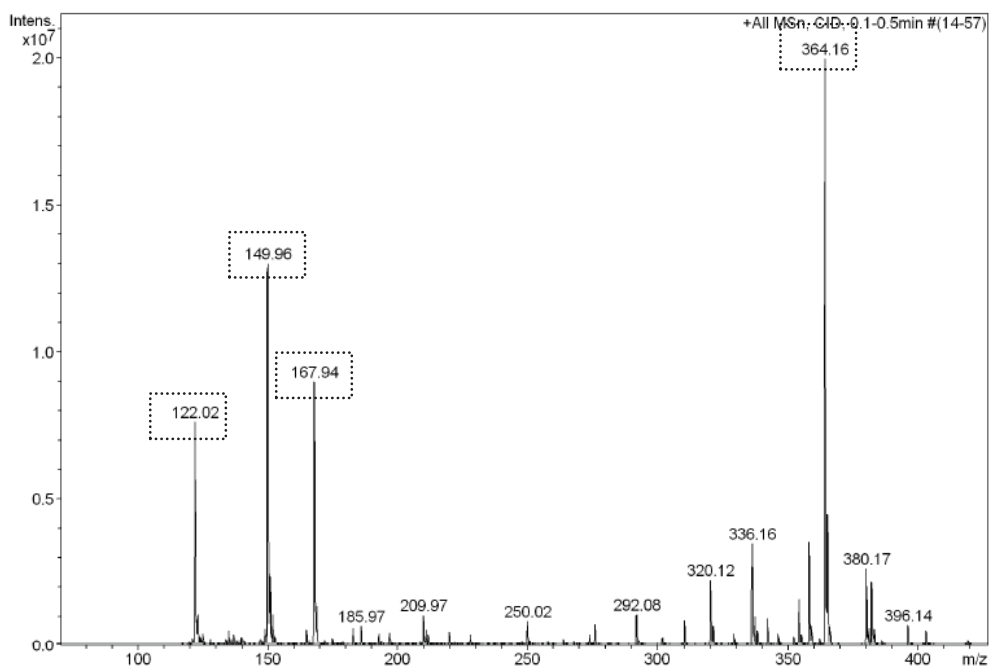
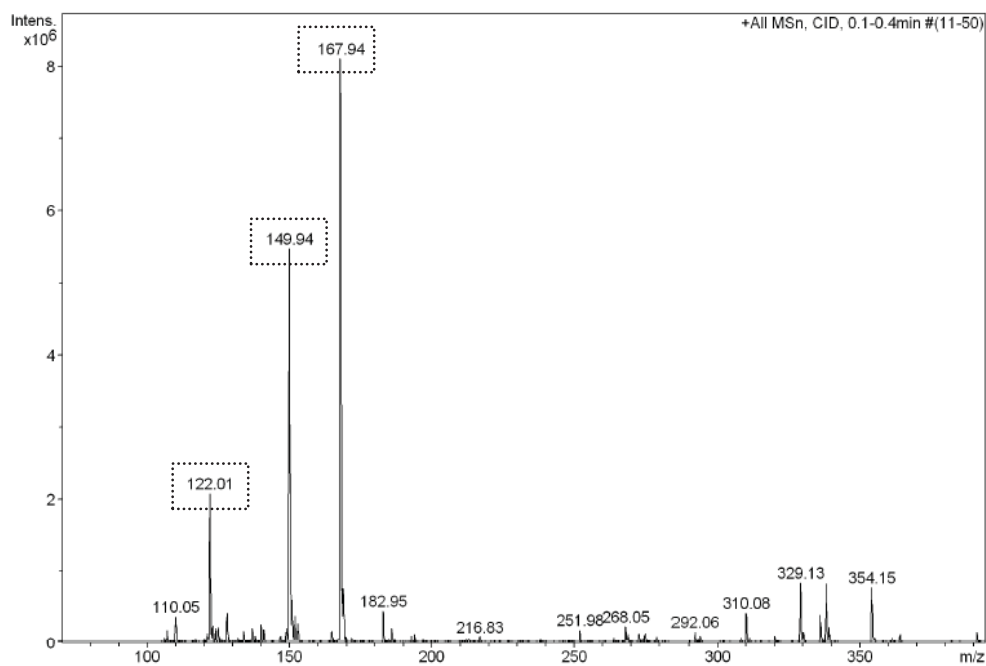
Injection: 3 μ L (10 ng/mL, MS analysis)2 μ L (10 mg/mL, PDA analysis)

MS analysis

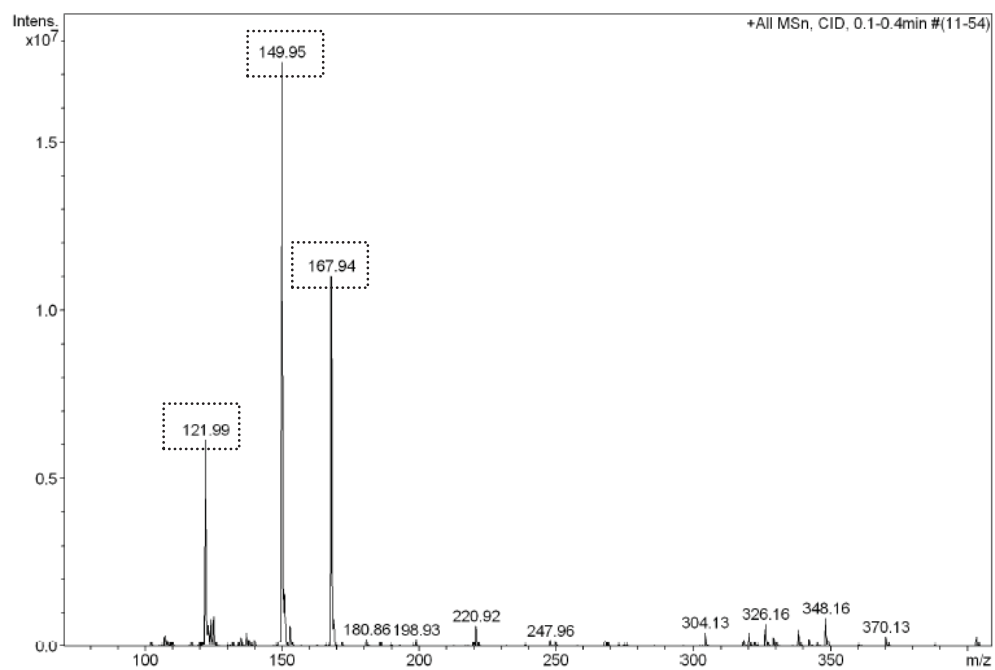


PDA analysis

A, Petasitenine (**14**); B, Senkirkine (**15**); C, Neopetasitenine (**13**).

Neopetasitenine (**13**)Petasitenine (**14**)

Senkirkine (15)



PJ-4 (16) (New alkaloid)

