

## Supplementary Information for

### New alkaloids from *Daphniphyllum himalense*

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**Figure S54.**  $^1\text{H}$  NMR spectrum for alkaloid **7** in  $\text{CD}_3\text{OD}$ .  
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**Figure S56.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum for alkaloid **7** in  $\text{CD}_3\text{OD}$ .

**Figure S57** HSQC spectrum for alkaloid **7** in  $\text{CD}_3\text{OD}$ .

**Figure S58.** HMBC spectrum for alkaloid **7** in  $\text{CD}_3\text{OD}$ .

**Figure S59.** NOESY spectrum for alkaloid **7** in  $\text{CD}_3\text{OD}$ .

**Figure S60.** IR spectrum for alkaloid **7**.

**Figure S61.** (+)-ESIMS spectrum for alkaloid **7**.

**Figure S62.** (+)-HRESIMS spectrum for alkaloid **7**.

**Figure S63.**  $^1\text{H}$  NMR spectrum for alkaloid **8** in  $\text{CDCl}_3$ .

**Figure S64.**  $^{13}\text{C}$  NMR spectrum for alkaloid **8** in  $\text{CDCl}_3$ .

**Figure S65.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum for alkaloid **8** in  $\text{CDCl}_3$ .

**Figure S66** HSQC spectrum for alkaloid **8** in  $\text{CDCl}_3$ .

**Figure S67.** HMBC spectrum for alkaloid **8** in  $\text{CDCl}_3$ .

**Figure S68.** NOESY spectrum for alkaloid **8** in  $\text{CDCl}_3$ .

**Figure S69.** IR spectrum for alkaloid **8**.

**Figure S70.** (+)-ESIMS spectrum for alkaloid **8**.

**Figure S71.** (+)-HRESIMS spectrum for alkaloid **8**.

**Figure S72.**  $^1\text{H}$  NMR spectrum for alkaloid **9** in  $\text{CDCl}_3$ .

**Figure S73.**  $^{13}\text{C}$  NMR spectrum for alkaloid **9** in  $\text{CDCl}_3$ .

**Figure S74.** HSQC spectrum for alkaloid **9** in  $\text{CDCl}_3$ .

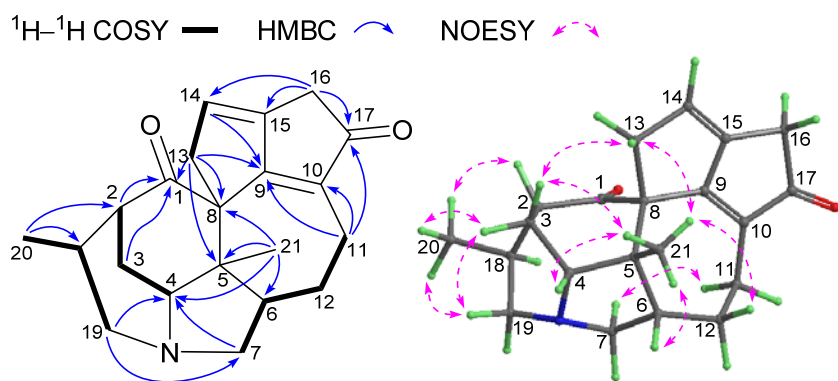
**Figure S75.** HMBC spectrum for alkaloid **9** in  $\text{CDCl}_3$ .

**Figure S76.** NOESY spectrum for alkaloid **9** in  $\text{CDCl}_3$ .

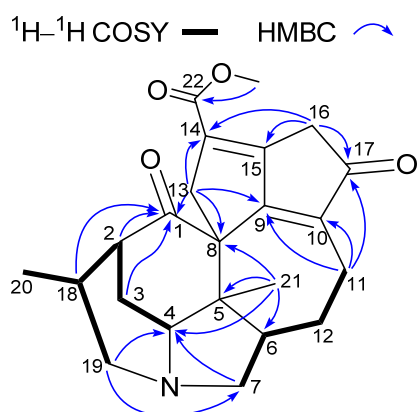
**Figure S77.** IR spectrum for alkaloid **9**.

**Figure S78.** (+)-ESIMS spectrum for alkaloid **9**.

**Figure S79.** (+)-HRESIMS spectrum for alkaloid **9**.

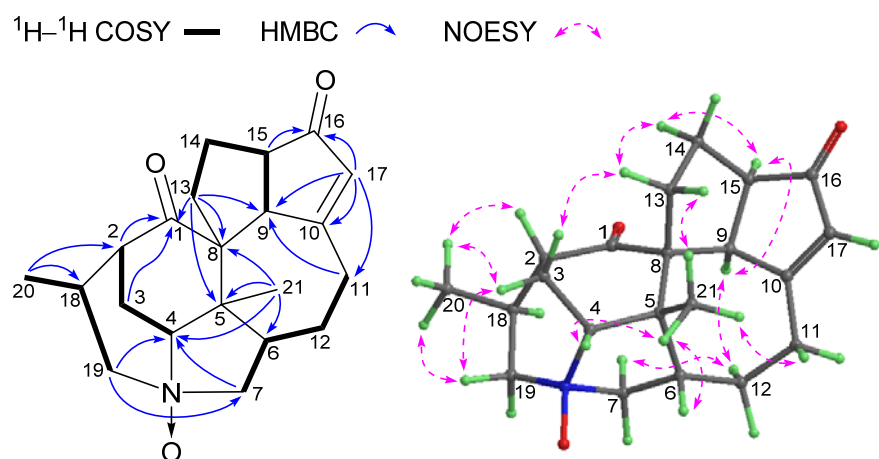


**Figure I.** Key 2D NMR correlations for alkaloid 2.

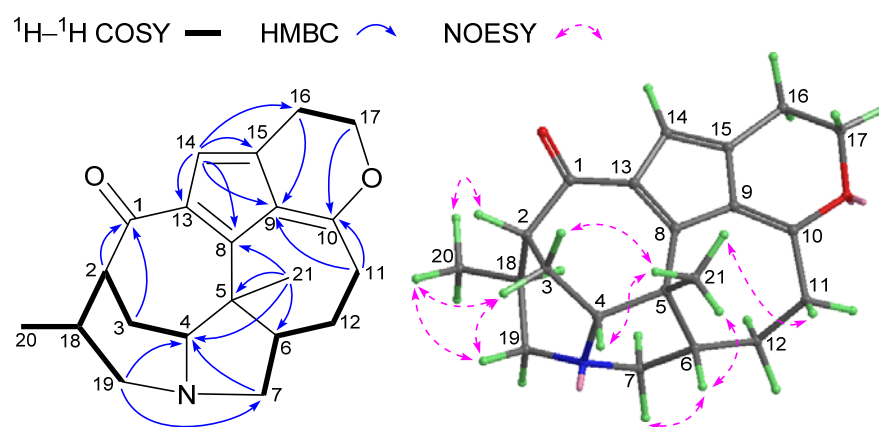


**Figure II.** Key 2D NMR correlations for alkaloid 3.





**Figure III.** Key 2D NMR correlations for alkaloid **7**.



**Figure IV.** Key 2D NMR correlations for alkaloid **8**.

**Table S1.** NMR data for alkaloids **2** and **3** in CD<sub>3</sub>OD.

| No. | <b>2</b>   |   | <b>3</b>   |  |
|-----|------------|---|------------|--|
|     | $\delta_C$ | $\delta_H$ , mult ( <i>J</i> in Hz)                   | $\delta_C$ | $\delta_H$ , mult ( <i>J</i> in Hz)                  |
| 1   | 215.1      |   | 214.0      |  |
| 2   | 45.4       | 2.32, brd (4.3)                                       | 45.1       | 2.40, brd (3.7)                                      |
| 3   | 20.7       | 2.19, brdd (15.3, 4.3)<br>2.12, ddd (5.3, 5.0, 1.7)   | 20.5       | 2.22, brdd (15.3, 4.3)<br>2.16, ddd (15.3, 4.9, 1.4) |
| 4   | 65.7       | 3.33, brd (5.0)                                       | 65.8       | 3.38, brd (4.9)                                      |
| 5   | 50.6       |   | 50.6       |  |
| 6   | 52.8       | 2.33, m   | 52.6       | 2.36, m  |
| 7   | 54.7       | 2.98, dd (13.0, 9.6)<br>2.84, dd (9.6, 6.1)           | 54.6       | 2.97, dd (13.0, 9.8)<br>2.89, dd (9.8, 6.5)          |
| 8   | 65.9       |   | 65.5       |  |
| 9   | 181.9      |   | 178.5      |  |
| 10  | 140.6      |   | 146.3      |  |
| 11  | 18.9       | 2.42, ddd (16.0, 4.8, 2.8)<br>2.02, brdd 16.0, (13.3) | 19.35      | 2.48, m<br>2.06, m                                   |
| 12  | 25.2       | 1.92, m<br>1.71, m                                    | 24.5       | 1.94, m<br>1.75, m                                   |
| 13  | 47.3       | 3.50, brd (18.5)<br>2.85, brd (18.5)                  | 45.6       | 3.63, d (18.1)<br>3.05, d (18.1)                     |
| 14  | 126.8      | 5.88 (m)  | 127.0      |  |
| 15  | 145.2      |   | 154.1      |  |
| 16  | <i>ND</i>  | <i>ND</i>   | <i>ND</i>  | <i>ND</i>  |
| 17  | 208.3      |   | 206.1      |  |
| 18  | 33.6       | 2.75, m   | 33.5       | 2.76, m  |
| 19  | 50.4       | 2.88, dd (14.5, 7.3)<br>2.56, dd (14.5, 10.5)         | 50.3       | 2.92, dd (14.3, 7.5)<br>2.60, dd (14.3, 10.5)        |
| 20  | 19.4       | 1.03, d ( 6.8)  | 19.26      | 1.04, d (6.8)  |
| 21  | 23.2       | 1.22, s   | 23.2       | 1.25, s  |
| 22  |            |   | 166.2      |  |
| OMe |            |   | 52.5       | 3.77 (s)   |

*ND* Signals not detected due to deuteration.

**Table S2.** PTP1B inhibitory assay results.

| Comps no.                | Concentration ( $\mu\text{g/mL}$ ) | Inhibition (%) | Standard error (%) |
|--------------------------|------------------------------------|----------------|--------------------|
| <b>1</b>                 | 20                                 | 48.81          | 3.20               |
| <b>2</b>                 | 20                                 | 39.63          | 3.55               |
| <b>3</b>                 | 20                                 | 26.60          | 11.14              |
| <b>4</b>                 | 20                                 | 26.01          | 12.47              |
| <b>5</b>                 | 20                                 | 1.38           | 19.53              |
| <b>6</b>                 | 20                                 | 0.47           | 11.44              |
| <b>7</b>                 | 20                                 | 27.61          | 5.61               |
| <b>8</b>                 | 20                                 | 27.86          | 3.98               |
| <b>9</b>                 | 20                                 | 37.78          | 6.39               |
| calyciphylline Q         | 20                                 | 27.31          | 4.07               |
| daphniyunnine A          | 20                                 | 39.96          | 6.31               |
| daphniyunnine B          | 20                                 | 0.75           | 7.36               |
| daphniyunnine C          | 20                                 | 25.33          | 7.75               |
| daphniyunnine D          | 20                                 | 41.18          | 4.46               |
| daphniyunnine E          | 20                                 | 2.78           | 4.37               |
| daphlongamine E          | 20                                 | 13.97          | 5.08               |
| daphlongamine F          | 20                                 | 25.13          | 6.66               |
| daphnilongeranin A       | 20                                 | 30.49          | 2.79               |
| daphnilongeranin B       | 20                                 | 20.90          | 11.45              |
| daphnipaxianine A        | 20                                 | 31.72          | 8.04               |
| daphnipaxinin            | 20                                 | 5.52           | 9.51               |
| dehydroxymacropodumine A | 20                                 | 25.08          | 1.91               |
| deoxycalyciphylline B    | 20                                 | 21.27          | 8.93               |
| deoxyisocalyciphylline B | 20                                 | 27.23          | 2.23               |
| longistylumphylline A    | 20                                 | 32.34          | 7.26               |

Oleanolic acid was used as positive control with an  $\text{IC}_{50}$  value of  $1.12 \pm 0.08 \mu\text{g/mL}$ .

**Table S3.** Aurora kinase A inhibitory assay results.

| Comps no.                | Concentration ( $\mu\text{g/mL}$ ) | Inhibition (%) | Standard error (%) |
|--------------------------|------------------------------------|----------------|--------------------|
| <b>1</b>                 | 20                                 | 6.98           | 0.26               |
| <b>2</b>                 | 20                                 | 14.58          | 2.96               |
| <b>3</b>                 | 20                                 | 27.08          | 11.54              |
| <b>4</b>                 | 20                                 | 28.78          | 5.45               |
| <b>5</b>                 | 20                                 | 31.50          | 12.56              |
| <b>6</b>                 | 20                                 | 21.13          | 5.52               |
| <b>7</b>                 | 20                                 | 17.18          | 3.73               |
| <b>8</b>                 | 20                                 | 16.54          | 0.80               |
| <b>9</b>                 | 20                                 | 28.11          | 3.45               |
| calyciphylline Q         | 20                                 | 17.07          | 10.07              |
| daphniyunnine A          | 20                                 | 27.69          | 10.73              |
| daphniyunnine B          | 20                                 | 14.71          | 8.48               |
| daphniyunnine C          | 20                                 | 34.10          | 0.10               |
| daphniyunnine D          | 20                                 | 16.98          | 0.12               |
| daphniyunnine E          | 20                                 | 17.35          | 3.62               |
| daphlongamine E          | 20                                 | 21.85          | 0.84               |
| daphlongamine F          | 20                                 | 17.72          | 6.92               |
| daphnilongeranin A       | 20                                 | 14.21          | 0.54               |
| daphnilongeranin B       | 20                                 | 18.54          | 1.09               |
| daphnipaxianine A        | 20                                 | 18.76          | 4.19               |
| daphnipaxinin            | 20                                 | 14.89          | 1.39               |
| dehydroxymacropodumine A | 20                                 | 18.00          | 4.54               |
| deoxycalyciphylline B    | 20                                 | 25.07          | 0.54               |
| deoxyisocalyciphylline B | 20                                 | 20.31          | 5.61               |
| longistylumphylline A    | 20                                 | 30.08          | 3.87               |

Staurosporine was used as positive control with an  $\text{IC}_{50}$  value of  $31.1 \pm 1.6$  nM.

**Table S4.** HDAC6 inhibitory assay results.

| Compds no.               | Concentration ( $\mu\text{g/mL}$ ) | Inhibition (%) | Standard error (%) |
|--------------------------|------------------------------------|----------------|--------------------|
| <b>1</b>                 | 20                                 | -7.32          | 0.77               |
| <b>2</b>                 | 20                                 | 1.86           | 1.29               |
| <b>3</b>                 | 20                                 | 0.72           | 2.83               |
| <b>4</b>                 | 20                                 | 10.51          | 1.77               |
| <b>5</b>                 | 20                                 | 11.93          | 4.16               |
| <b>6</b>                 | 20                                 | -2.12          | 2.12               |
| <b>7</b>                 | 20                                 | 4.92           | 0.04               |
| <b>8</b>                 | 20                                 | -6.02          | 7.22               |
| <b>9</b>                 | 20                                 | 3.86           | 8.31               |
| calyciphylline Q         | 20                                 | 3.48           | 6.42               |
| daphniyunnine A          | 20                                 | 11.82          | 6.26               |
| daphniyunnine B          | 20                                 | 4.88           | 2.74               |
| daphniyunnine C          | 20                                 | 3.09           | 0.53               |
| daphniyunnine D          | 20                                 | 1.89           | 3.02               |
| daphniyunnine E          | 20                                 | -0.50          | 2.05               |
| daphlongamine E          | 20                                 | 2.44           | 2.74               |
| daphlongamine F          | 20                                 | 0.11           | 1.34               |
| daphnilongeranin A       | 20                                 | -3.95          | 1.92               |
| daphnilongeranin B       | 20                                 | 8.55           | 5.02               |
| daphnipaxianine A        | 20                                 | -1.07          | 3.45               |
| daphnipaxinin            | 20                                 | 6.63           | 1.44               |
| dehydroxymacropodumine A | 20                                 | 8.13           | 6.87               |
| deoxycalyciphylline B    | 20                                 | 2.27           | 1.67               |
| deoxyisocalyciphylline B | 20                                 | -12.23         | 6.21               |
| longistylumphylline A    | 20                                 | 5.45           | 2.96               |

SAHA (Suberoylanilide hydroxamic acid) was used as positive control with an  $\text{IC}_{50}$  value of  $105 \pm 25$  nM.

**Table S5.** IKK- $\beta$  inhibitory assay results.

| Comps no.                | Concentration ( $\mu\text{g/mL}$ ) | Inhibition (%) | Standard error (%) |
|--------------------------|------------------------------------|----------------|--------------------|
| <b>1</b>                 | 20                                 | 8.28           | 4.36               |
| <b>2</b>                 | 20                                 | 11.99          | 4.08               |
| <b>3</b>                 | 20                                 | 21.21          | 4.43               |
| <b>4</b>                 | 20                                 | 18.91          | 2.61               |
| <b>5</b>                 | 20                                 | 28.93          | 2.43               |
| <b>6</b>                 | 20                                 | 21.77          | 2.88               |
| <b>7</b>                 | 20                                 | 16.53          | 3.91               |
| <b>8</b>                 | 20                                 | 17.38          | 3.18               |
| <b>9</b>                 | 20                                 | 27.60          | 5.71               |
| calyciphylline Q         | 20                                 | 18.41          | 2.46               |
| daphniyunnine A          | 20                                 | 19.01          | 3.73               |
| daphniyunnine B          | 20                                 | 8.77           | 5.59               |
| daphniyunnine C          | 20                                 | 17.26          | 0.47               |
| daphniyunnine D          | 20                                 | 13.75          | 2.18               |
| daphniyunnine E          | 20                                 | 10.64          | 1.61               |
| daphlongamine E          | 20                                 | 20.67          | 1.12               |
| daphlongamine F          | 20                                 | 15.20          | 1.21               |
| daphnilongeranin A       | 20                                 | 11.33          | 1.33               |
| daphnilongeranin B       | 20                                 | 20.49          | 5.15               |
| daphnipaxianine A        | 20                                 | 31.45          | 3.68               |
| daphnipaxinin            | 20                                 | 16.19          | 4.59               |
| dehydroxymacropodumine A | 20                                 | 11.57          | 0.21               |
| deoxycalyciphylline B    | 20                                 | 17.71          | 5.95               |
| deoxyisocalyciphylline B | 20                                 | 18.42          | 3.90               |
| longistylumphylline A    | 20                                 | 17.64          | 3.17               |

Staurosporine was used as positive control with an inhibition of  $89.52 \pm 0.72\%$  at  $1 \mu\text{M}$ .

**Table S6.** Crystal data and structure refinement for alkaloid **10**.

|                                   |  |                               |
|-----------------------------------|--|-------------------------------|
| Identification code               | cu_dm16061_0m                                    |                               |
| Empirical formula                 | C <sub>24</sub> H <sub>35</sub> N O <sub>5</sub> |                               |
| Formula weight                    | 417.53   |                               |
| Temperature                       | 293.15 K   |                               |
| Wavelength                        | 1.54178 Å  |                               |
| Crystal system                    | Monoclinic                                       |                               |
| Space group                       | P 1 21 1   |                               |
| Unit cell dimensions              | a = 10.7411(2) Å                                 | $\alpha = 90^\circ$ .         |
|                                   | b = 9.0530(2) Å                                  | $\beta = 93.4430(10)^\circ$ . |
|                                   | c = 11.5845(2) Å                                 | $\gamma = 90^\circ$ .         |
| Volume                            | 1124.43(4) Å <sup>3</sup>                        |                               |
| Z                                 | 2  |                               |
| Density (calculated)              | 1.233 Mg/m <sup>3</sup>                          |                               |
| Absorption coefficient            | 0.689 mm <sup>-1</sup>                           |                               |
| F(000)                            | 452  |                               |
| Crystal size                      | 0.28 x 0.25 x 0.2 mm <sup>3</sup>                |                               |
| Theta range for data collection   | 3.822 to 69.726°.                                |                               |
| Index ranges                      | -13<=h<=12, -10<=k<=10, -13<=l<=13               |                               |
| Reflections collected             | 8067   |                               |
| Independent reflections           | 3732 [R(int) = 0.0311]                           |                               |
| Completeness to theta = 67.679°   | 99.3 %   |                               |
| Absorption correction             | Semi-empirical from equivalents                  |                               |
| Max. and min. transmission        | 0.7532 and 0.5620                                |                               |
| Refinement method                 | Full-matrix least-squares on F <sup>2</sup>      |                               |
| Data / restraints / parameters    | 3732 / 1 / 281                                   |                               |
| Goodness-of-fit on F <sup>2</sup> | 1.074  |                               |
| Final R indices [I>2sigma(I)]     | R1 = 0.0502, wR2 = 0.1568                        |                               |
| R indices (all data)              | R1 = 0.0511, wR2 = 0.1582                        |                               |
| Absolute structure parameter      | 0.19(11)   |                               |
| Extinction coefficient            | 0.022(3)   |                               |
| Largest diff. peak and hole       | 0.228 and -0.332 e.Å <sup>-3</sup>               |                               |

Figure S1. <sup>1</sup>H NMR spectrum for alkaloid **1** in CDCl<sub>3</sub>.

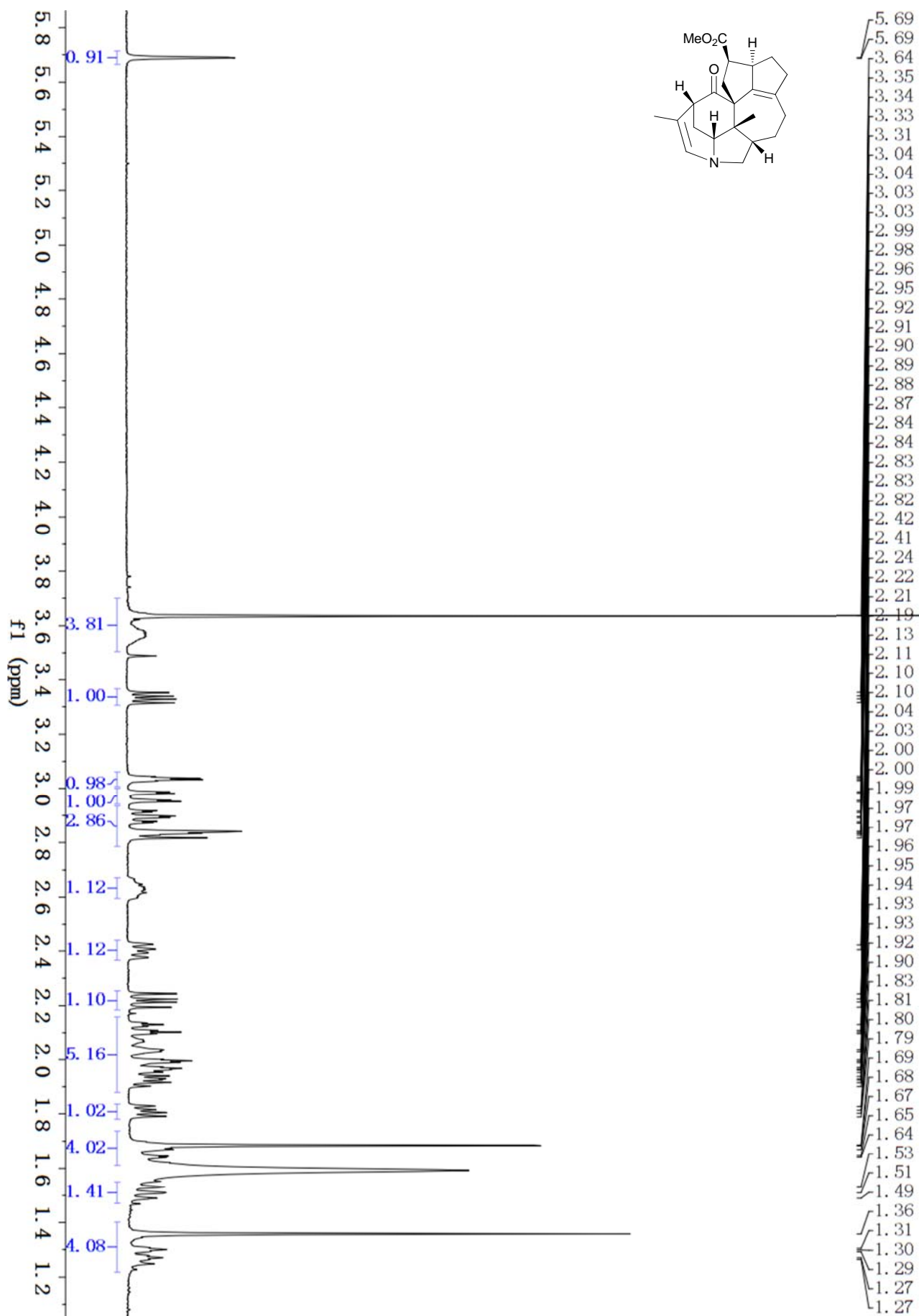
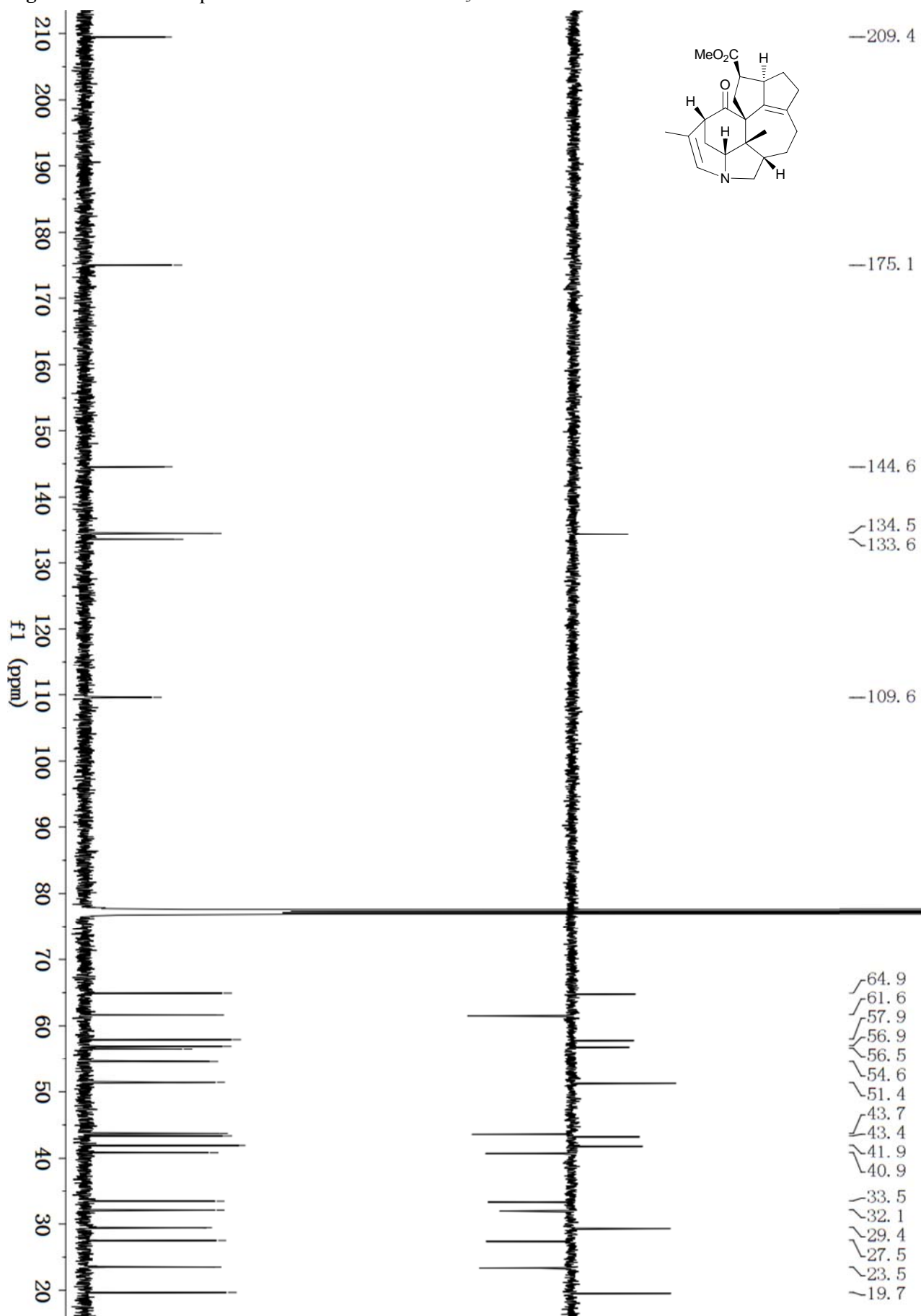




Figure S2.  $^{13}\text{C}$  NMR spectrum for alkaloid **1** in  $\text{CDCl}_3$ .



**Figure S3.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum for alkaloid **1** in  $\text{CDCl}_3$ .

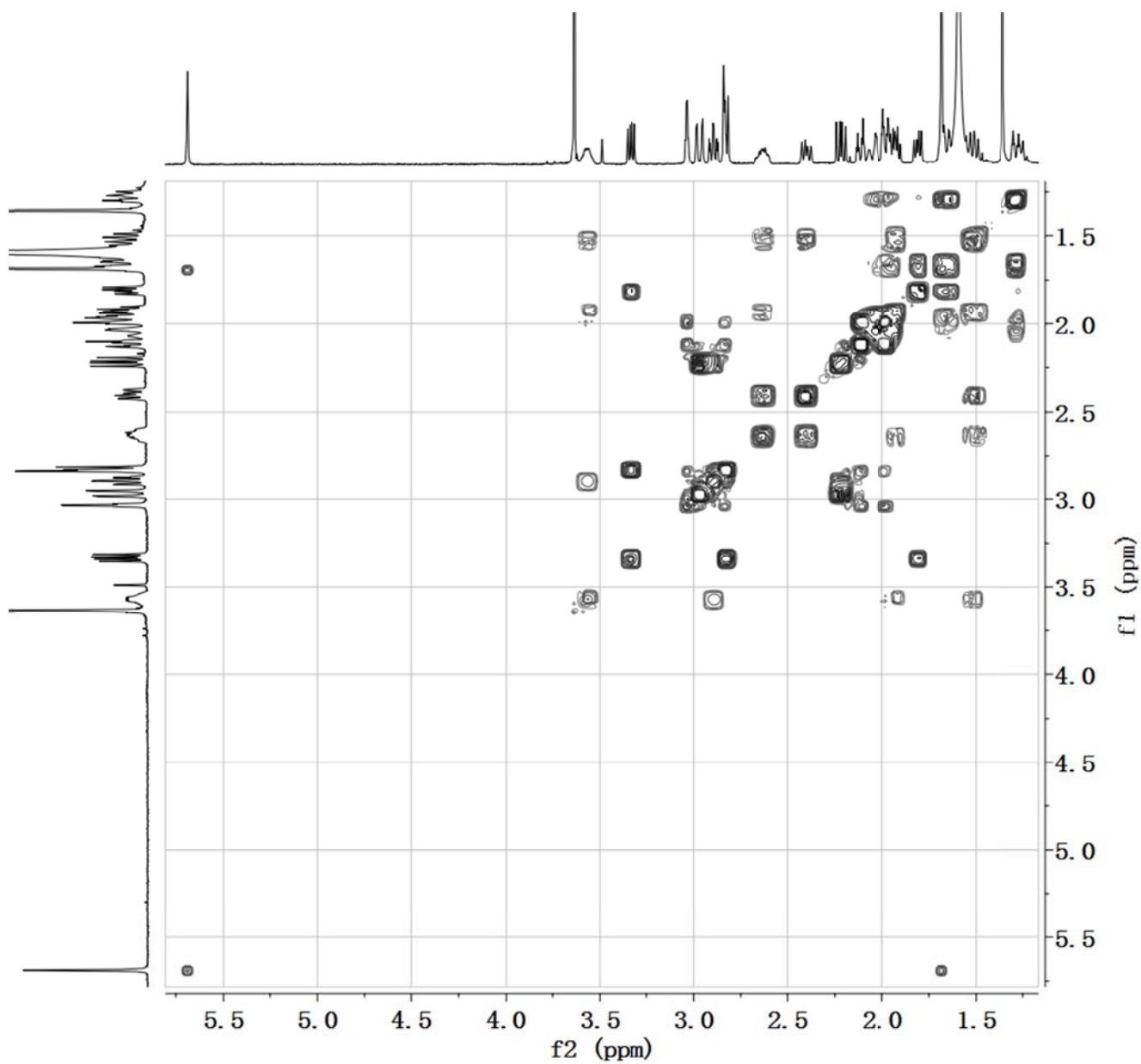


Figure S4. HSQC spectrum for alkaloid 1 in CDCl<sub>3</sub>.

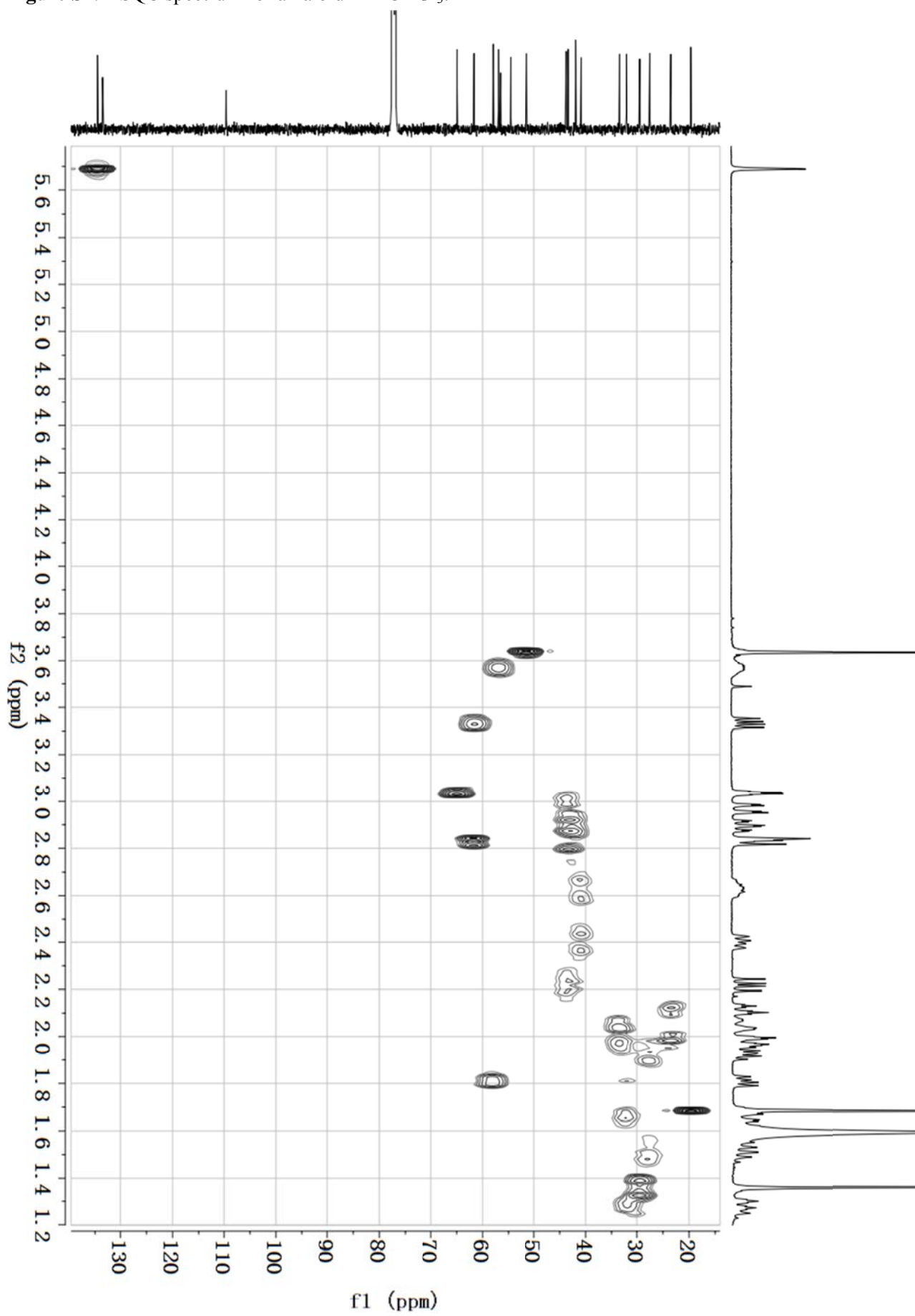
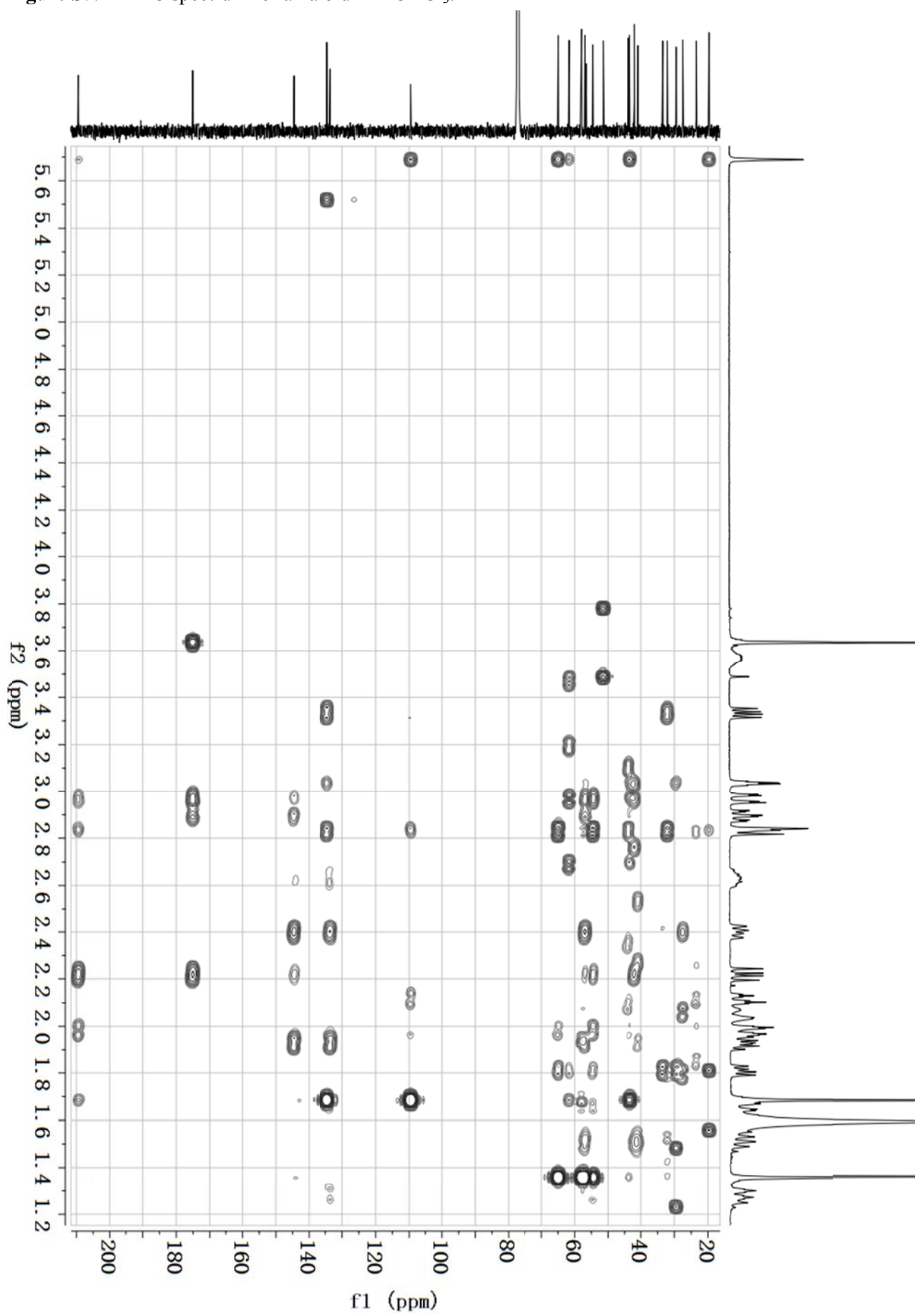


Figure S5. HMBC spectrum for alkaloid **1** in CDCl<sub>3</sub>.



**Figure S6.** NOESY spectrum for alkaloid **1** in CDCl<sub>3</sub>.

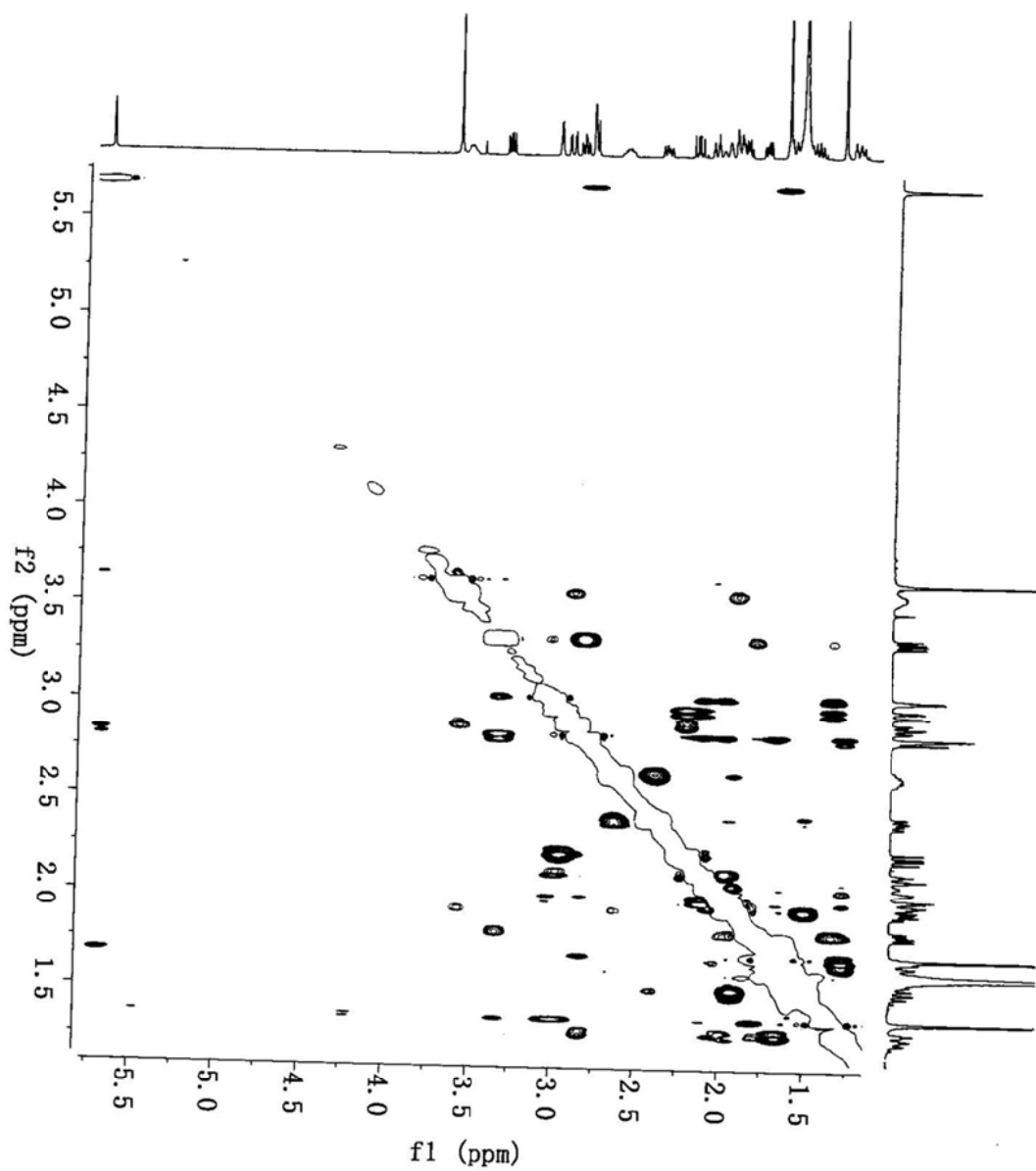


Figure S7. IR spectrum for alkaloid 1.

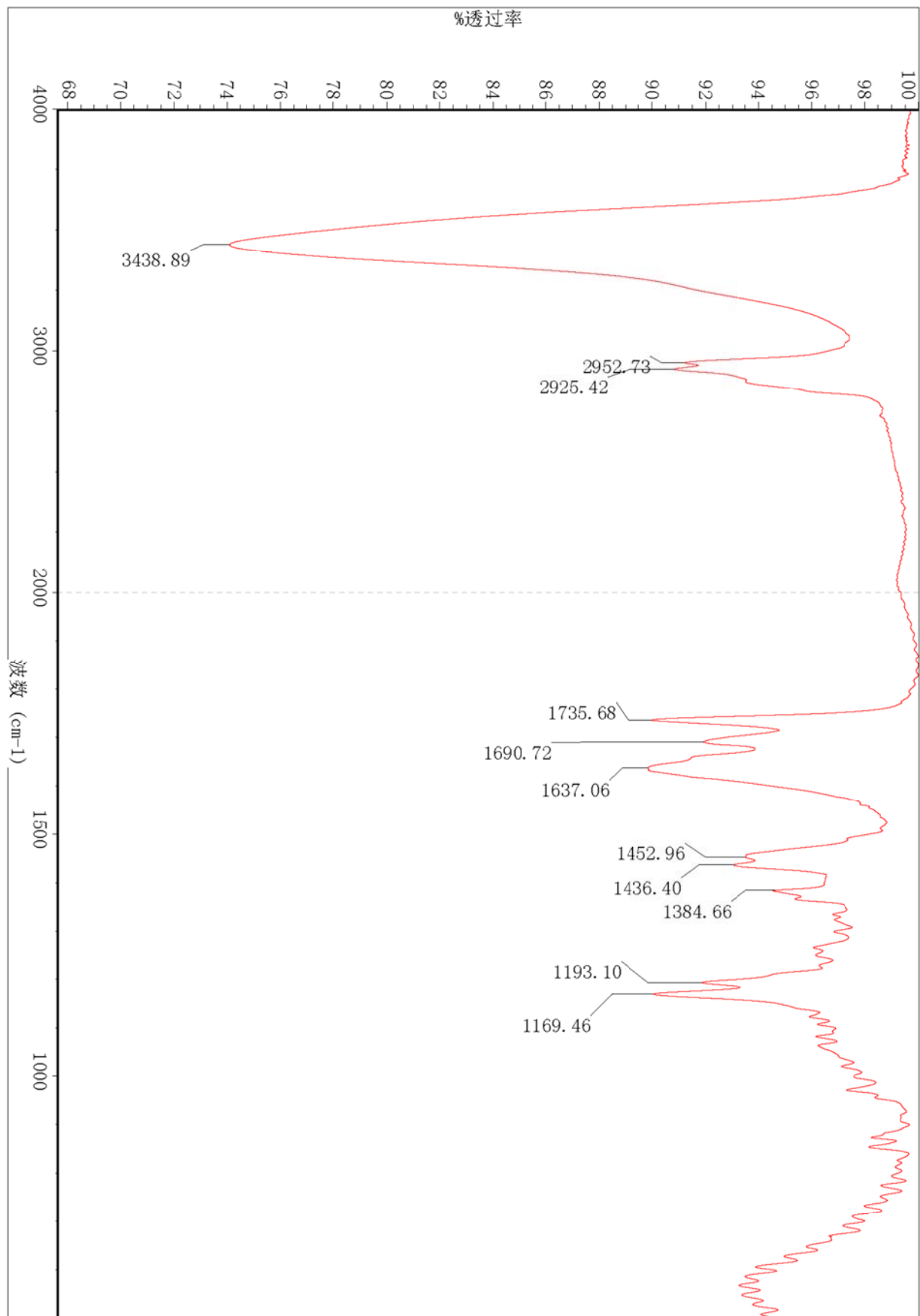


Figure S8. (+)-ESIMS spectrum for alkaloid 1.

### Display Report

**Analysis Info**

Analysis Name 029-2001.D  
Method Copy of DSOPMS2P.M  
Sample Name yjm-D4C1c2  
Comment \_LI

Acquisition Date 07/23/14 14:27:18  
Operator Administrator  
Instrument esquire3000plus

**Acquisition Parameter**

|                   |            |              |           |                          |          |
|-------------------|------------|--------------|-----------|--------------------------|----------|
| Ion Source Type   | ESI        | Ion Polarity | Positive  | Alternating Ion Polarity | off      |
| Mass Range Mode   | Std/Normal | Scan Begin   | 100 m/z   | Scan End                 | 1750 m/z |
| Capillary Exit    | 158.5 Volt | Skim 1       | 40.0 Volt | Trap Drive               | 85.2     |
| Accumulation Time | 15000 程    | Averages     | 3 Spectra | Auto MS/MS               | on       |

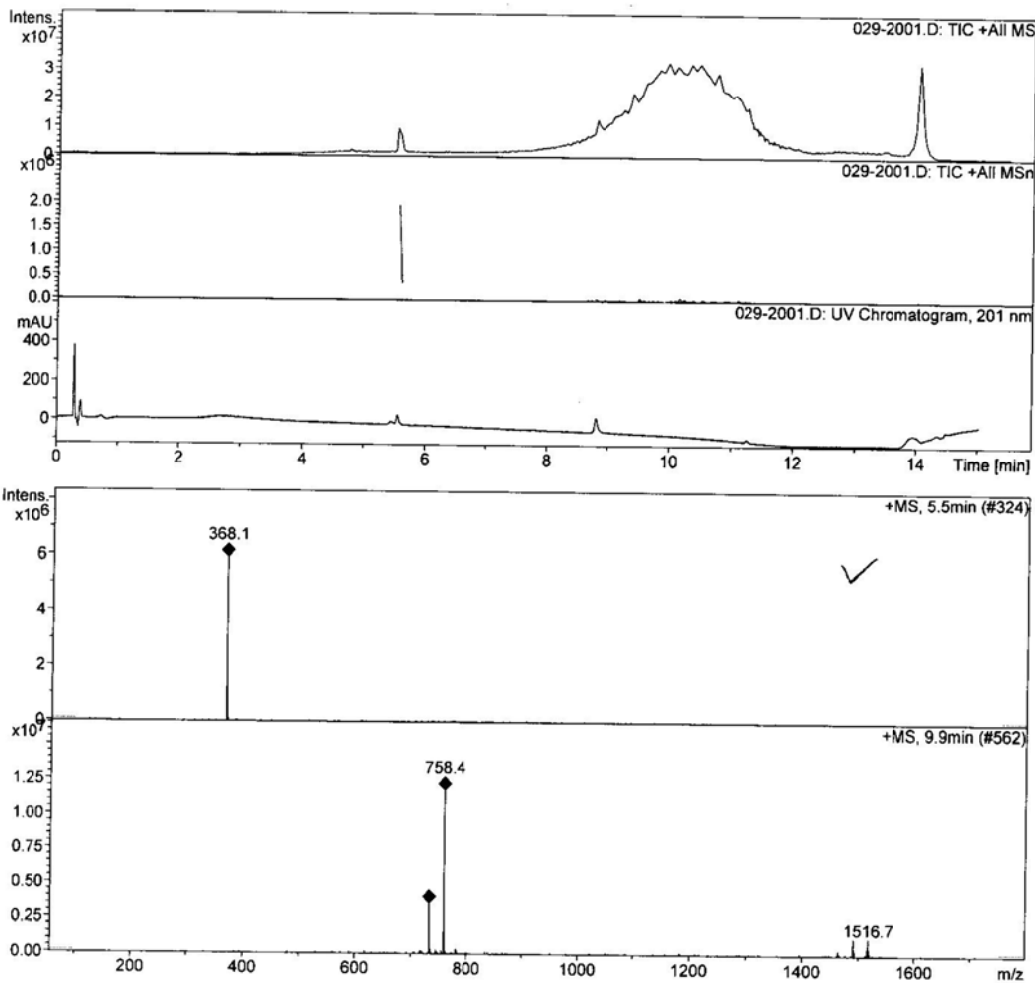


Figure S9. (+)-HRESIMS spectrum for alkaloid 1.

Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0  
 Element prediction: Off  
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

307 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-4 O: 0-20

DH-64

LCT PXE KE324

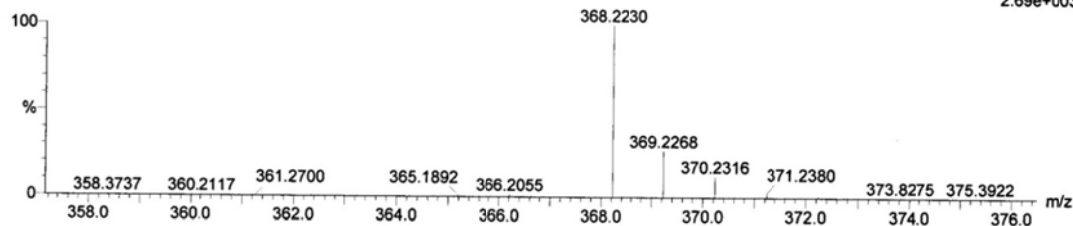
29-Aug-2014

10:46:58

1: TOF MS ES+

2.69e+003

DH-64\_0829 45 (1.005) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (45:56)



Minimum: -1.5  
 Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT | i-FIT (Norm) | Formula      |
|----------|------------|-----|-----|-----|-------|--------------|--------------|
| 368.2230 | 368.2226   | 0.4 | 1.1 | 9.5 | 80.9  | 0.0          | C23 H30 N O3 |



**Figure S10.**  $^1\text{H}$  NMR spectrum for alkaloid **2** in  $\text{C}_5\text{D}_5\text{N}$ .

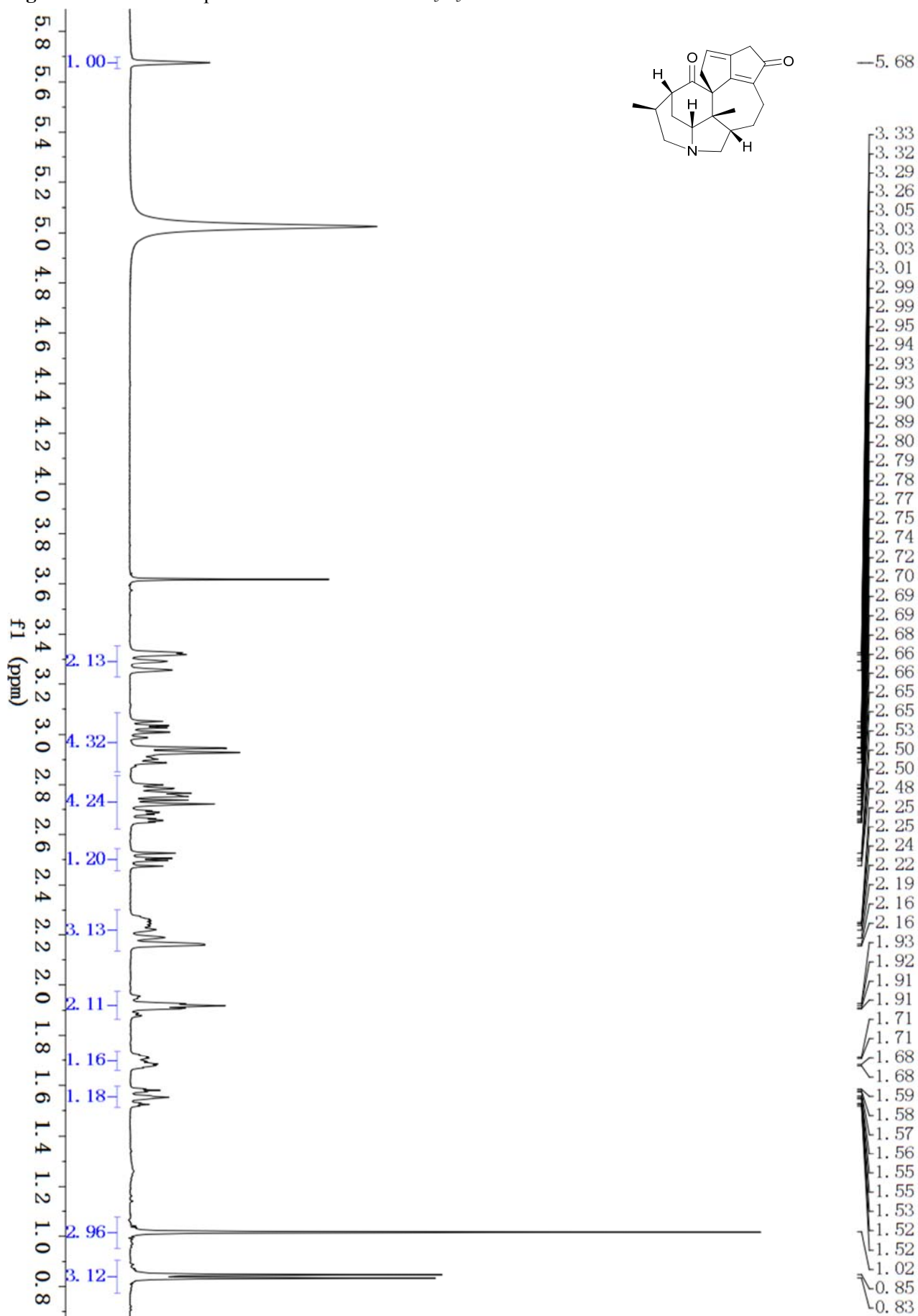
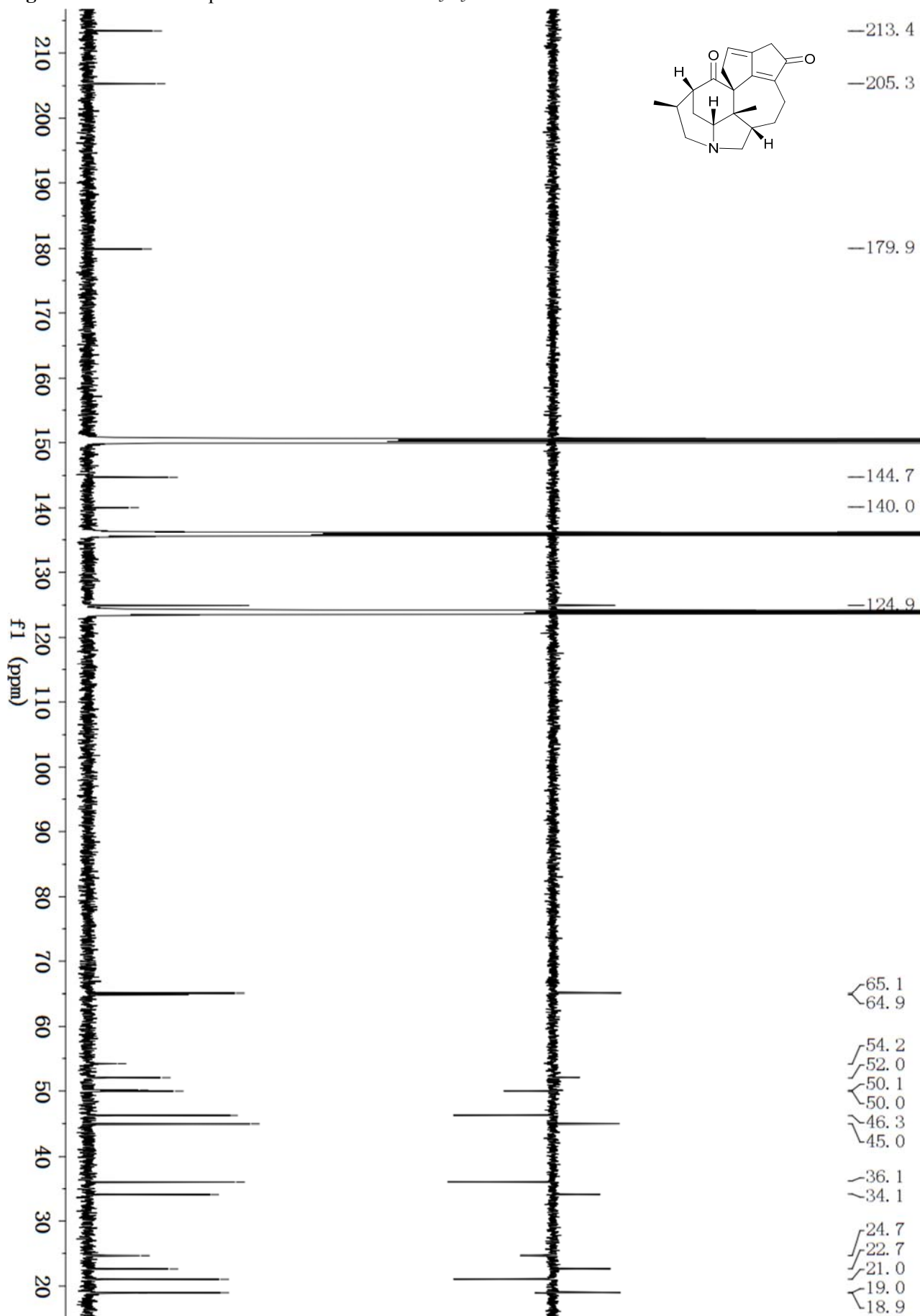
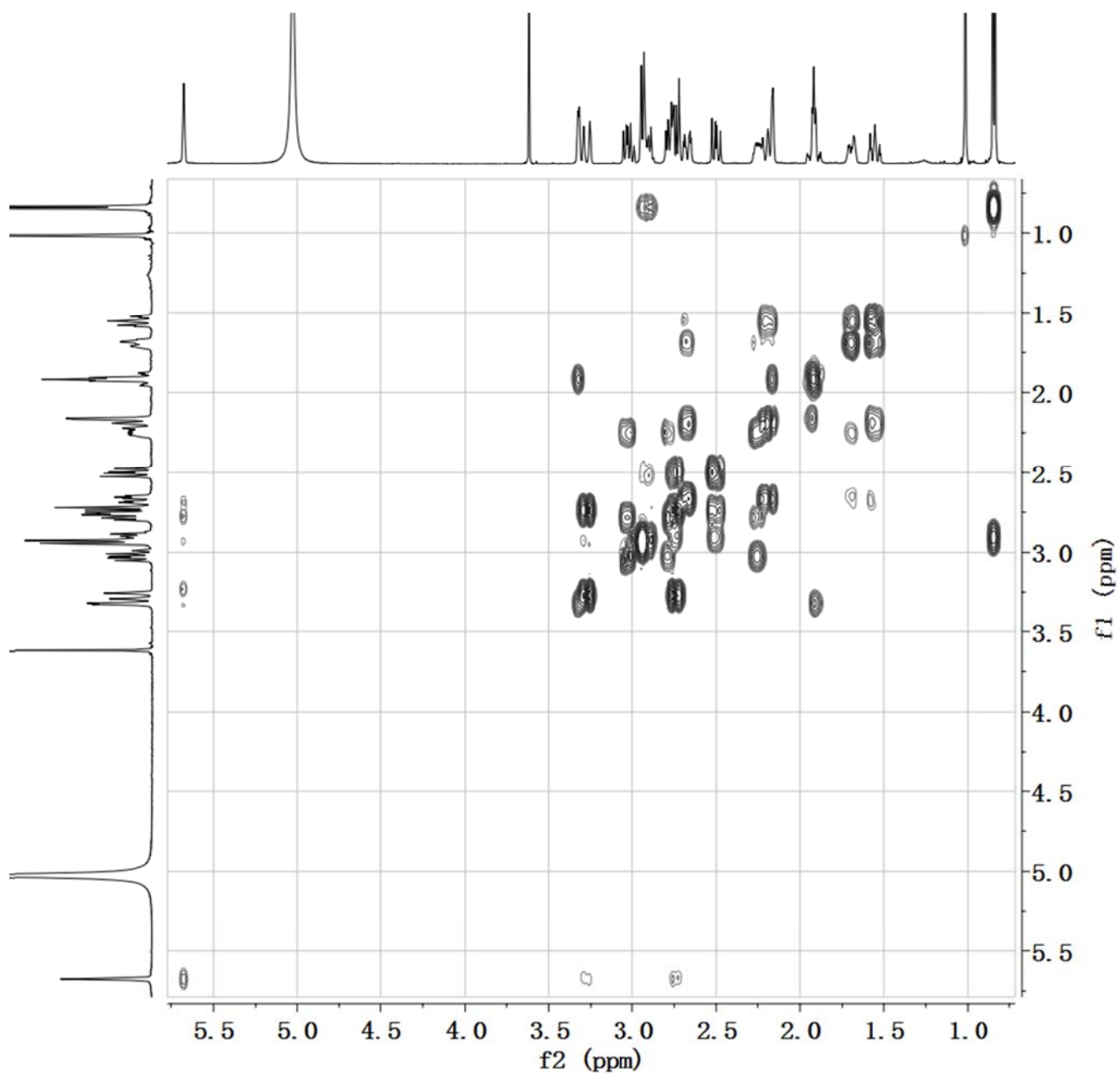


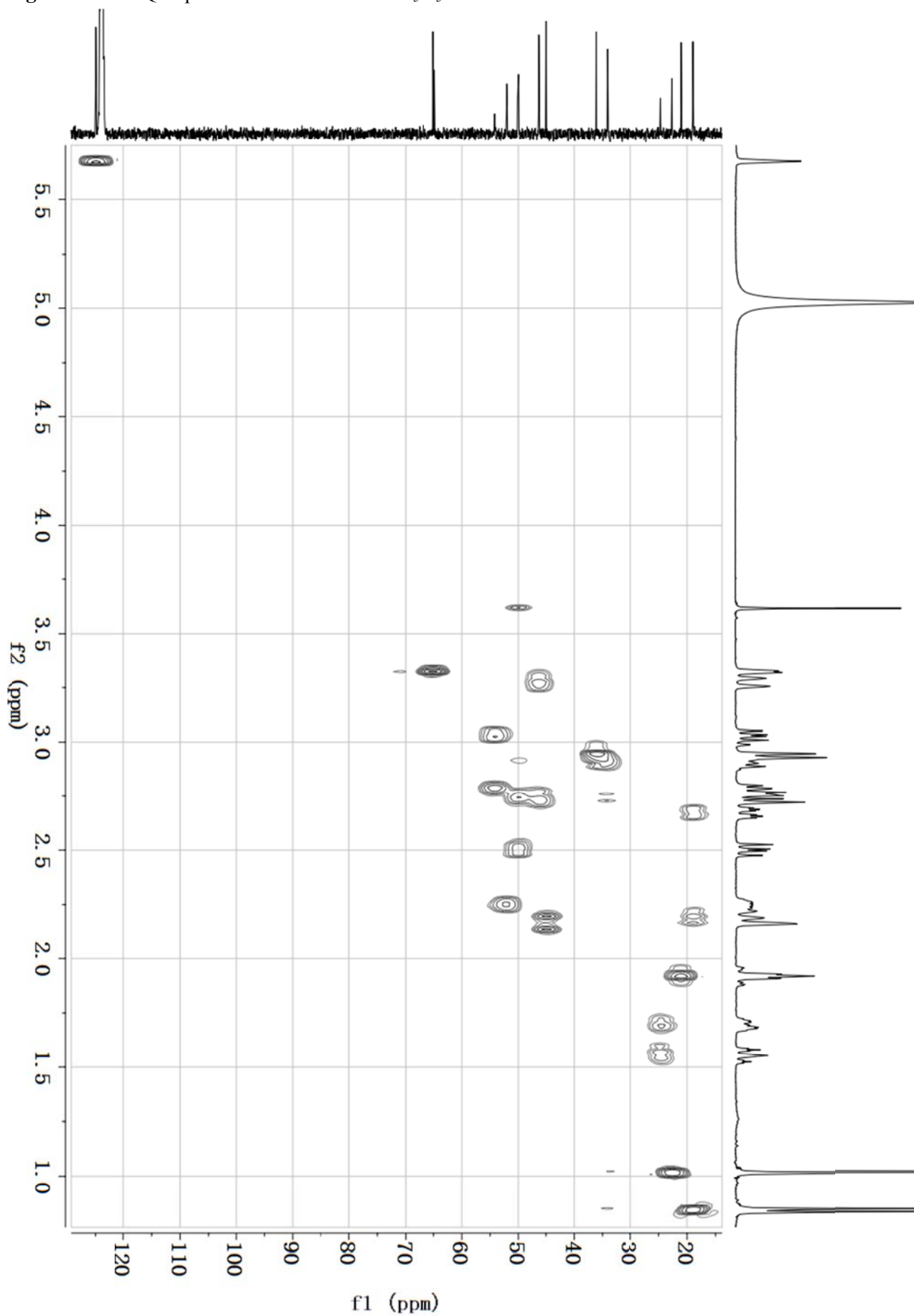
Figure S11.  $^{13}\text{C}$  NMR spectrum for alkaloid **2** in  $\text{C}_5\text{D}_5\text{N}$ .



**Figure S12.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum for alkaloid **2** in  $\text{C}_5\text{D}_5\text{N}$ .



**Figure S13.** HSQC spectrum for alkaloid **2** in  $C_5D_5N$ .



**Figure S14.** HMBC spectrum for alkaloid **2** in  $C_5D_5N$ .

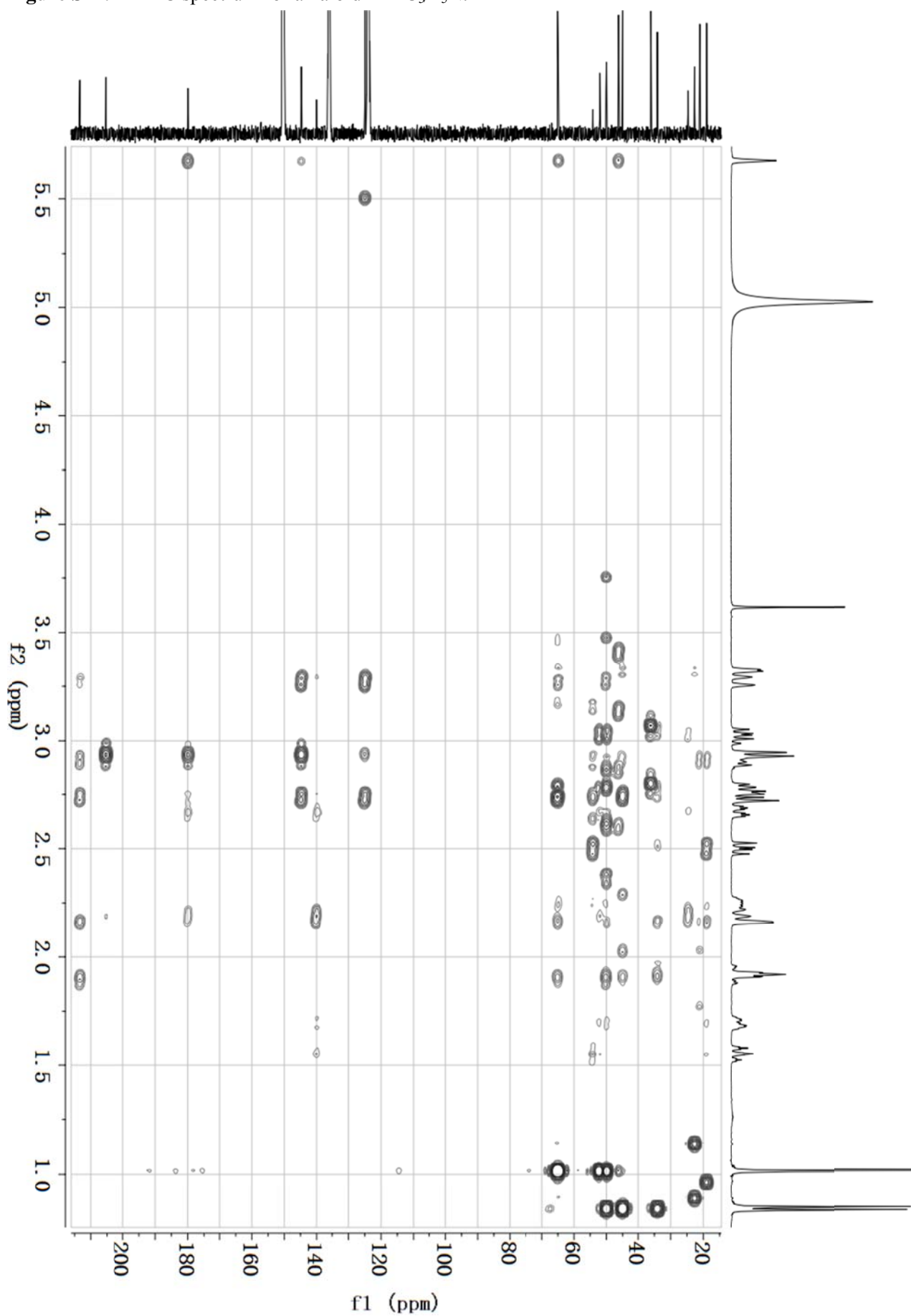


Figure S15. NOESY spectrum for alkaloid 2 in C<sub>5</sub>D<sub>5</sub>N.

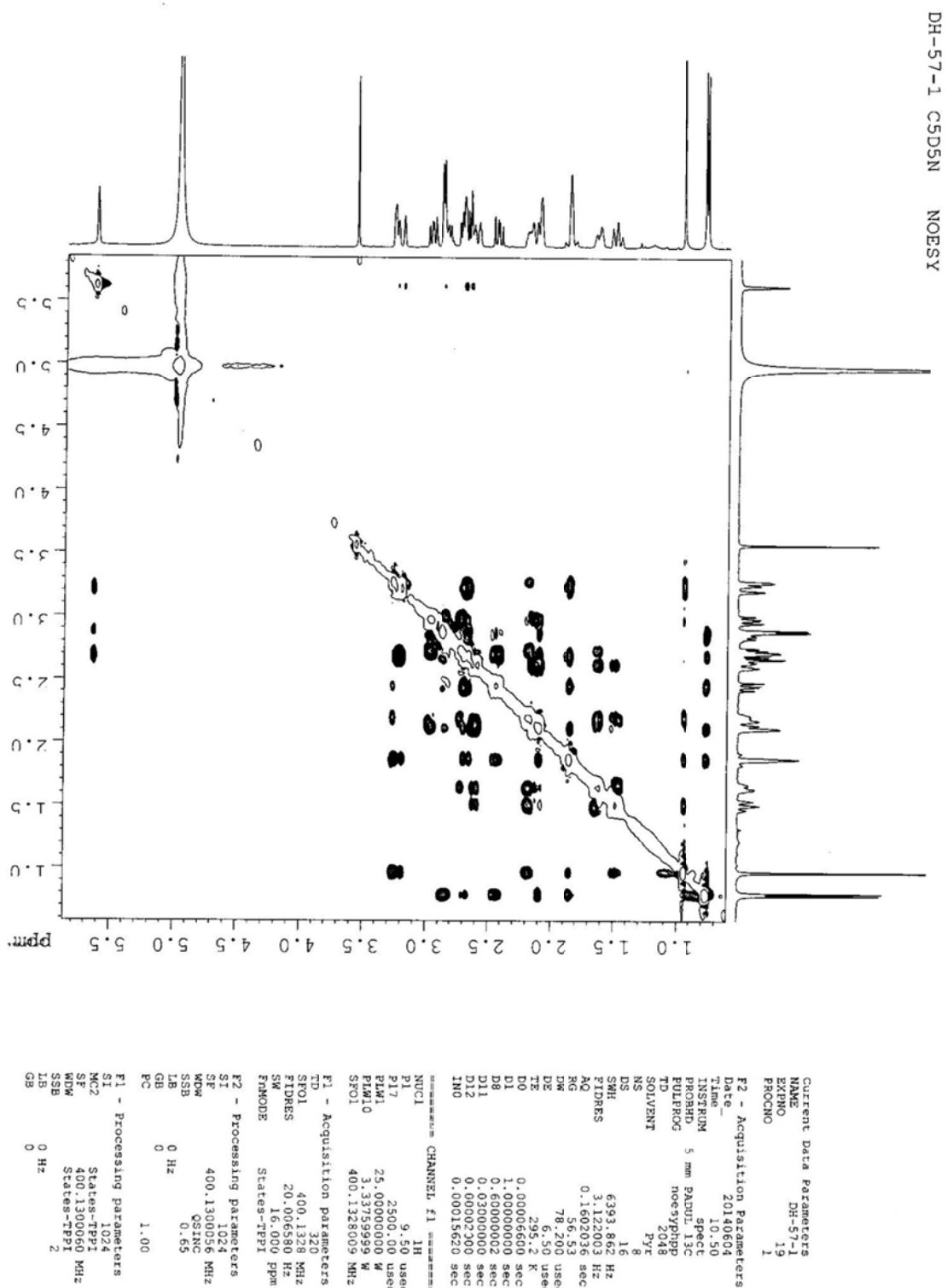


Figure S16. IR spectrum for alkaloid 2.

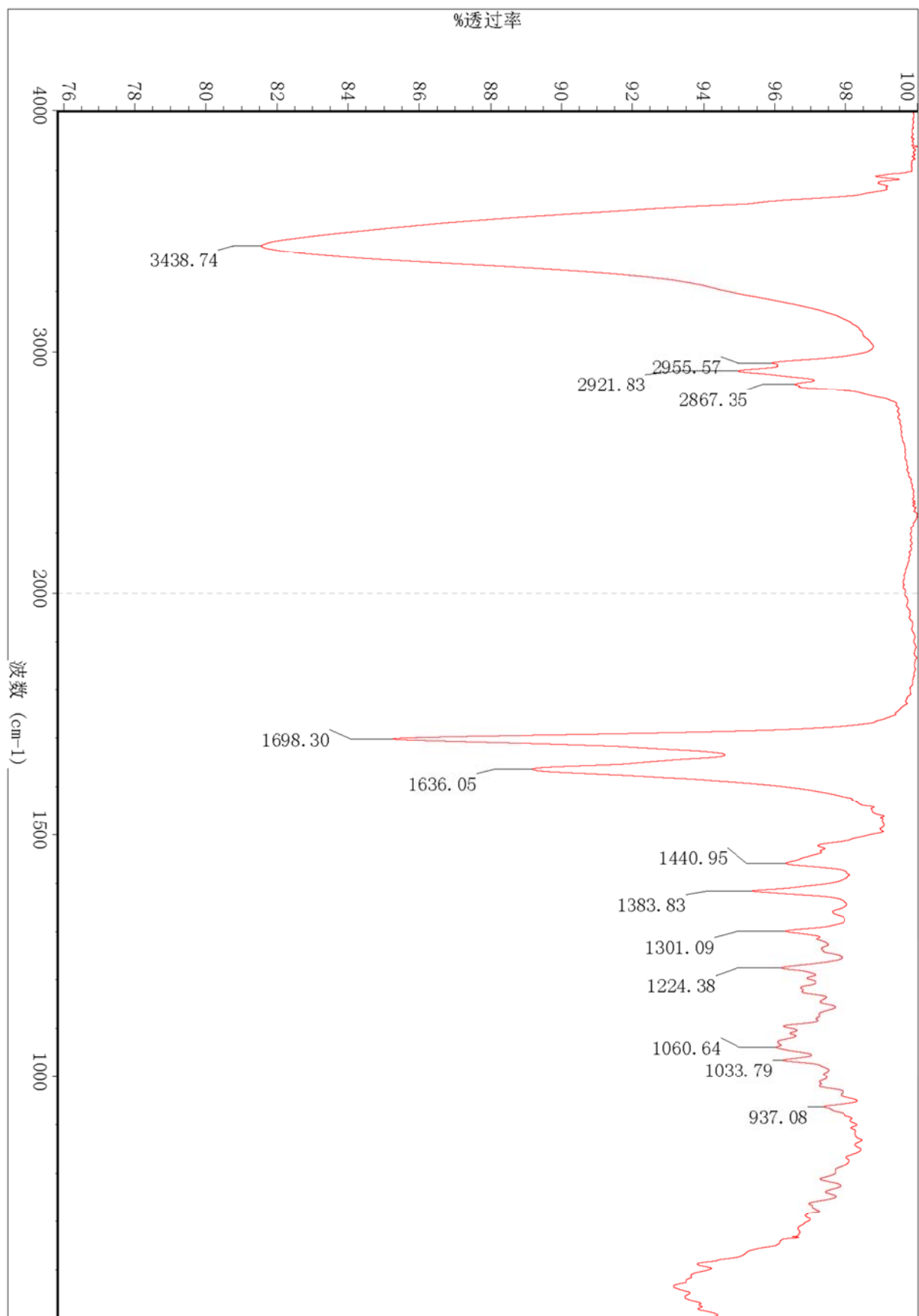


Figure S17. (+)-ESIMS spectrum for alkaloid 2.

### Display Report

**Analysis Info**

Analysis Name 016-1301.D  
 Method Copy of DSOPMS2P.M  
 Sample Name yjm-DH-57-1  
 Comment /

Acquisition Date 05/22/14 12:09:21  
 Operator Administrator  
 Instrument esquire3000plus

**Acquisition Parameter**

|                   |            |              |           |                          |          |
|-------------------|------------|--------------|-----------|--------------------------|----------|
| Ion Source Type   | ESI        | Ion Polarity | Positive  | Alternating Ion Polarity | off      |
| Mass Range Mode   | Std/Normal | Scan Begin   | 100 m/z   | Scan End                 | 1750 m/z |
| Capillary Exit    | 158.5 Volt | Skim 1       | 40.0 Volt | Trap Drive               | 85.2     |
| Accumulation Time | 15000 罫    | Averages     | 3 Spectra | Auto MS/MS               | on       |

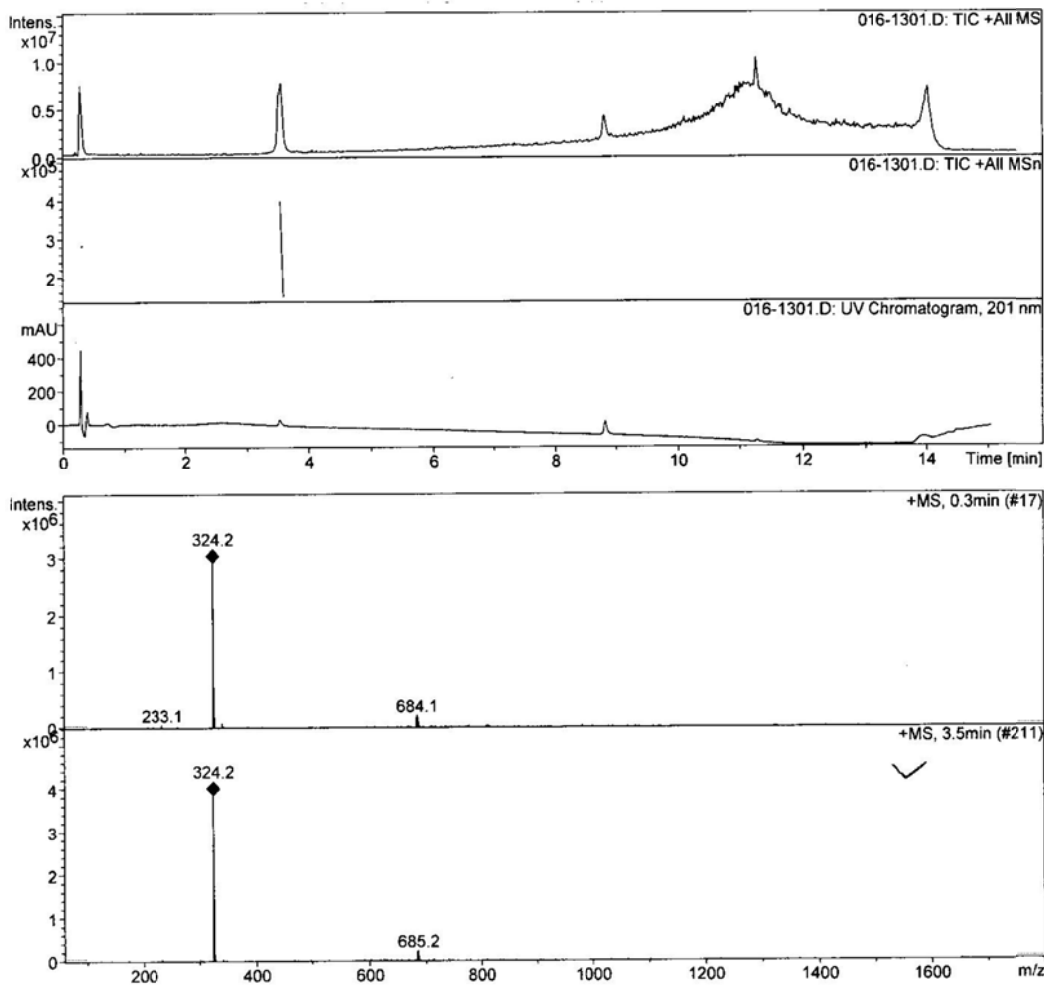




Figure S18. (+)-HRESIMS spectrum for alkaloid 2.

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0  
 Element prediction: Off  
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

156 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-2 O: 0-20

DH-57-1

LCT PXE KE324

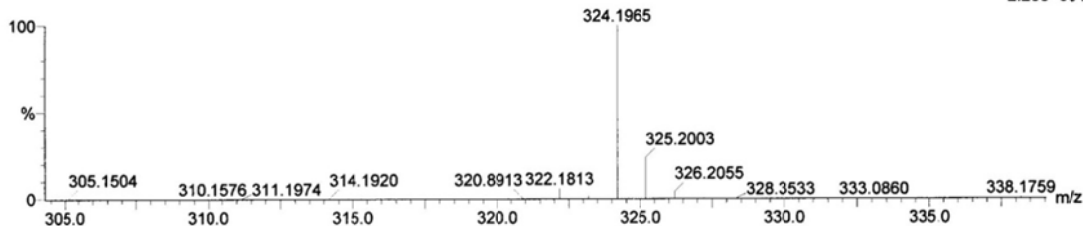
04-Jun-2014

15:23:04

1: TOF MS ES+

2.23e+004

DH-57-1\_0604 25 (0.549) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (13:29)



Minimum: -1.5  
 Maximum: 5.0 5.0 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT | i-FIT (Norm) | Formula      |
|----------|------------|-----|-----|-----|-------|--------------|--------------|
| 324.1965 | 324.1964   | 0.1 | 0.3 | 9.5 | 155.3 | 0.0          | C21 H26 N O2 |

**Figure S19.**  $^1\text{H}$  NMR spectrum for alkaloid **3** in  $\text{C}_5\text{D}_5\text{N}$ .

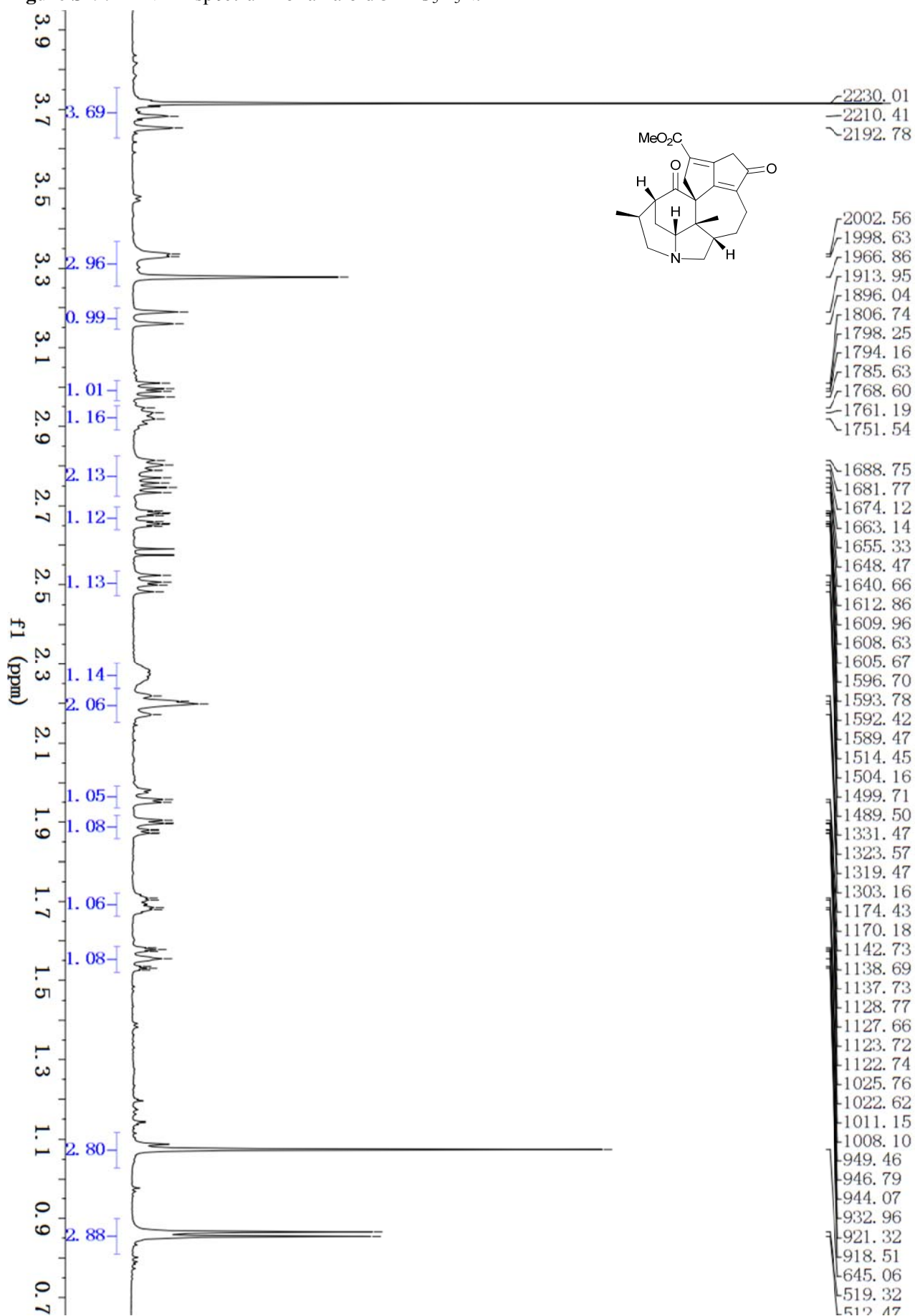
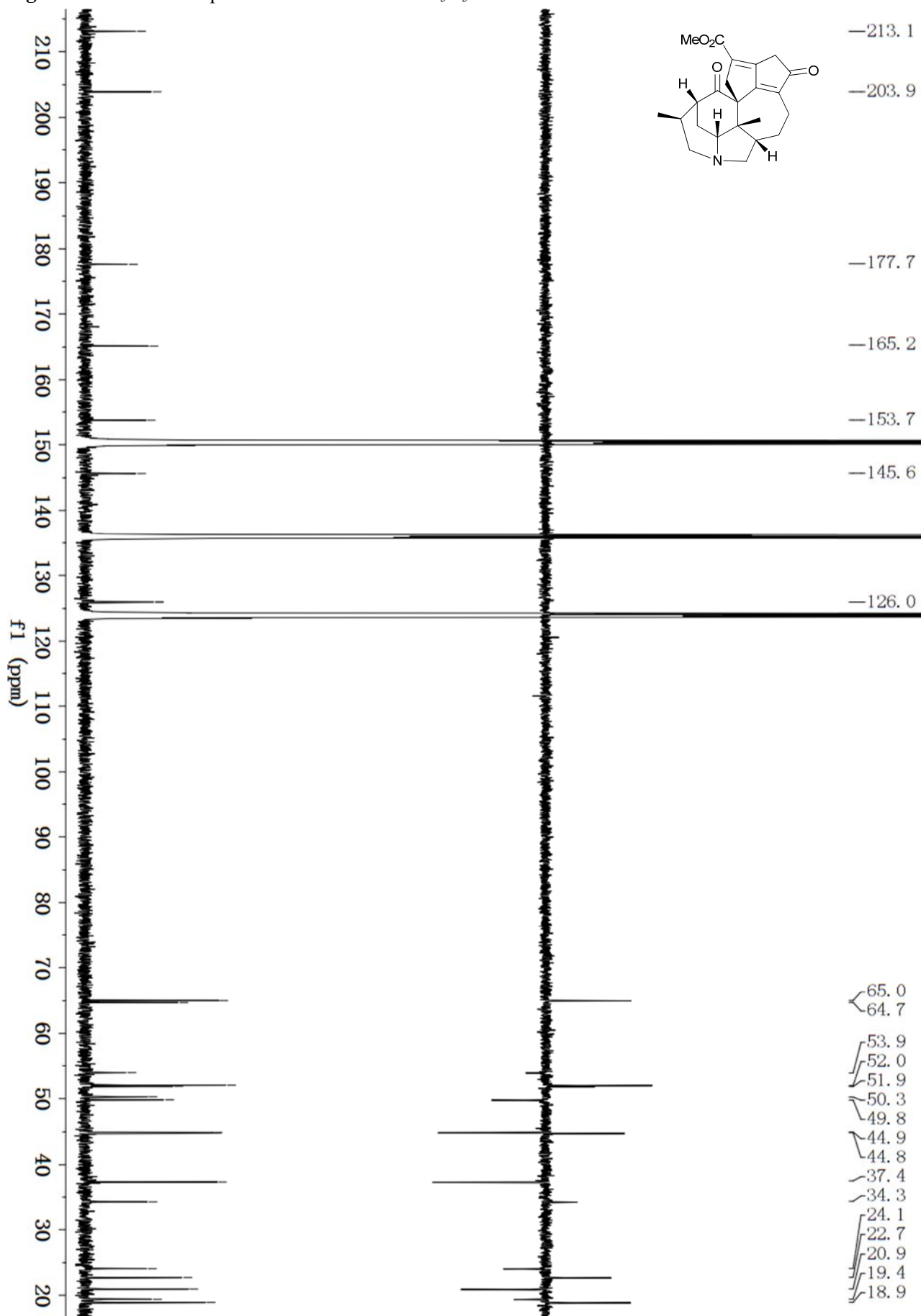
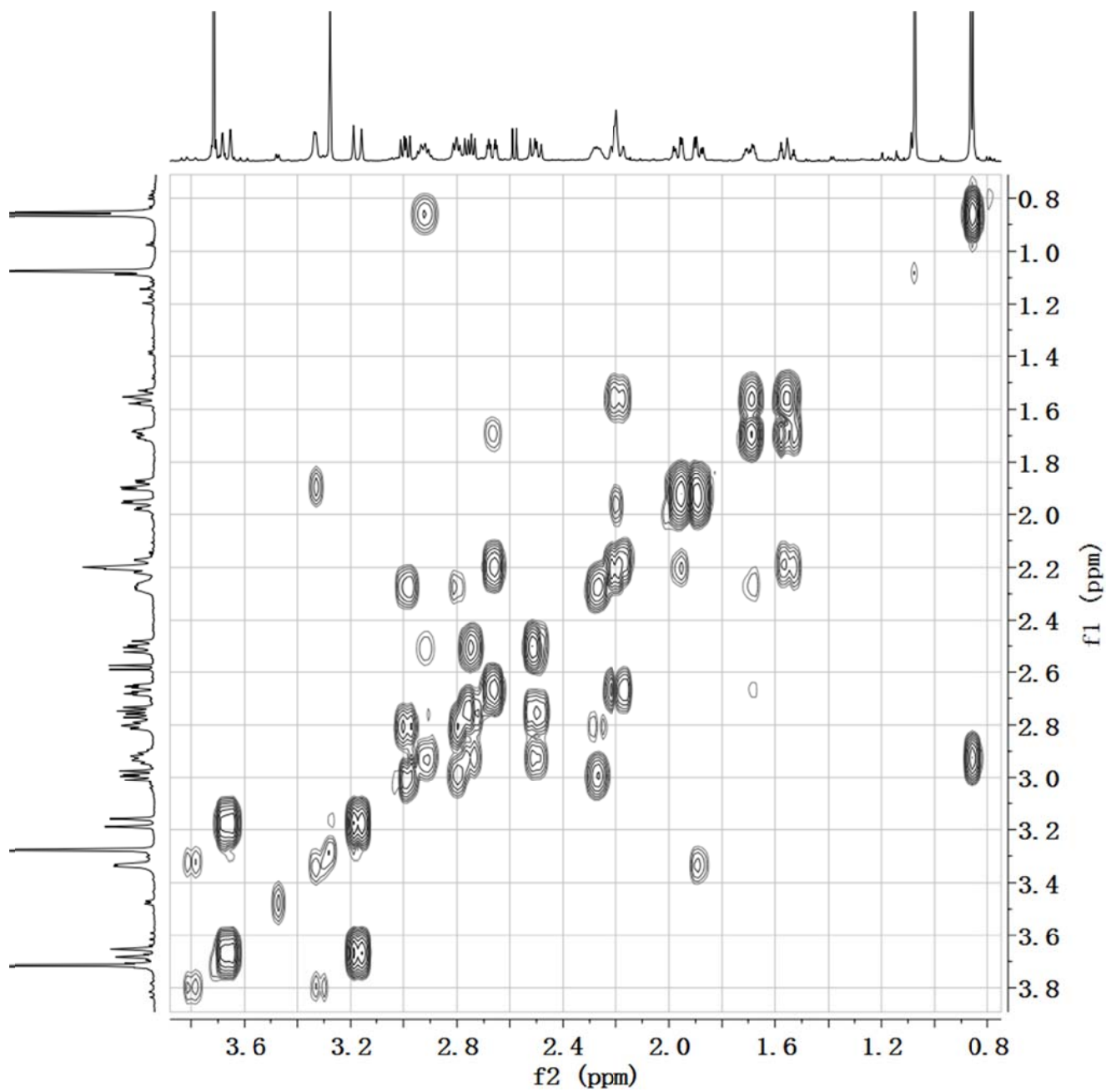


Figure S20.  $^{13}\text{C}$  NMR spectrum for alkaloid **3** in  $\text{C}_5\text{D}_5\text{N}$ .



**Figure S21.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum for alkaloid **3** in  $\text{C}_5\text{D}_5\text{N}$ .



**Figure S22.** HSQC spectrum for alkaloid **3** in  $C_5D_5N$ .

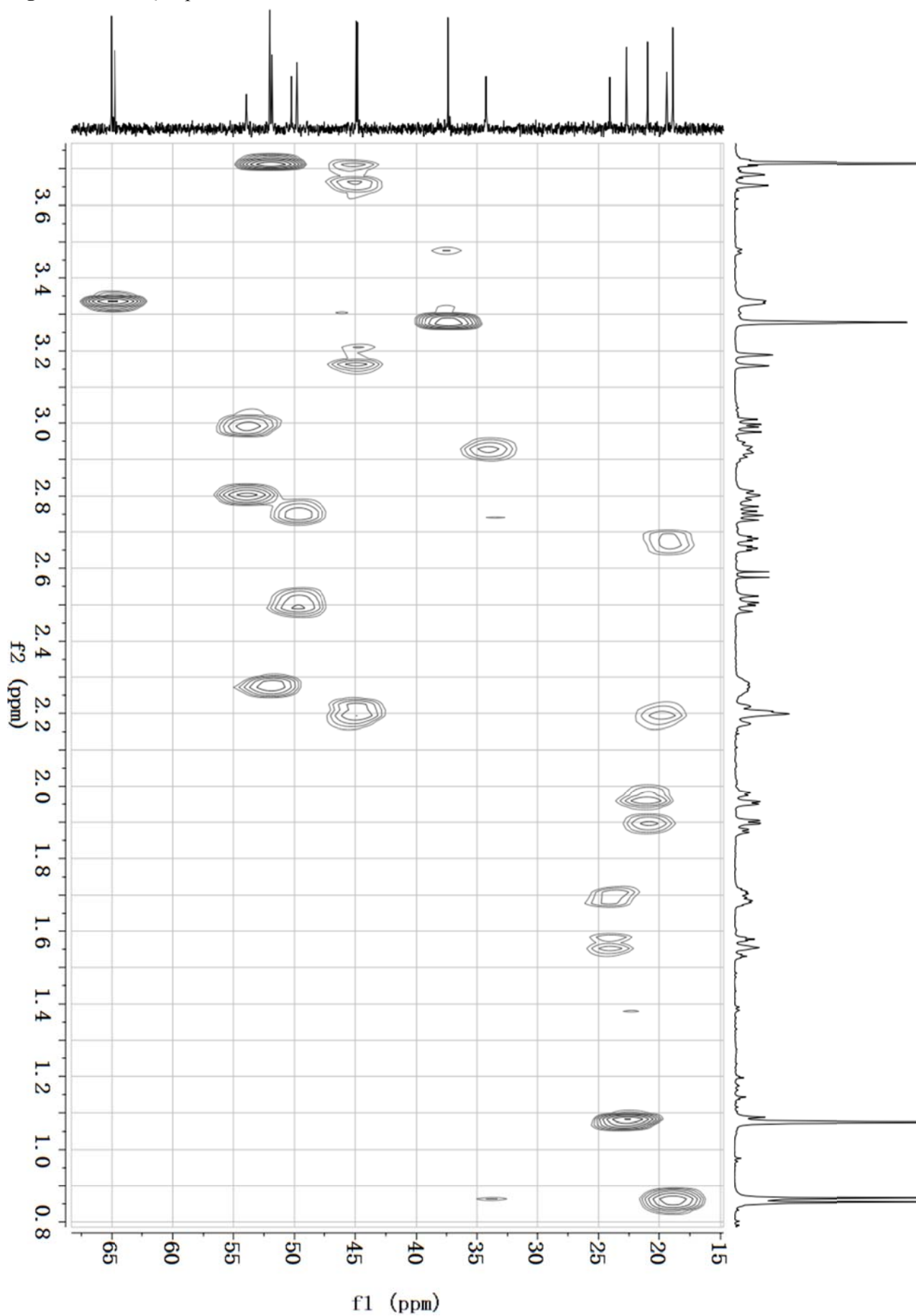


Figure S23. HMBC spectrum for alkaloid 3 in C<sub>5</sub>D<sub>5</sub>N.

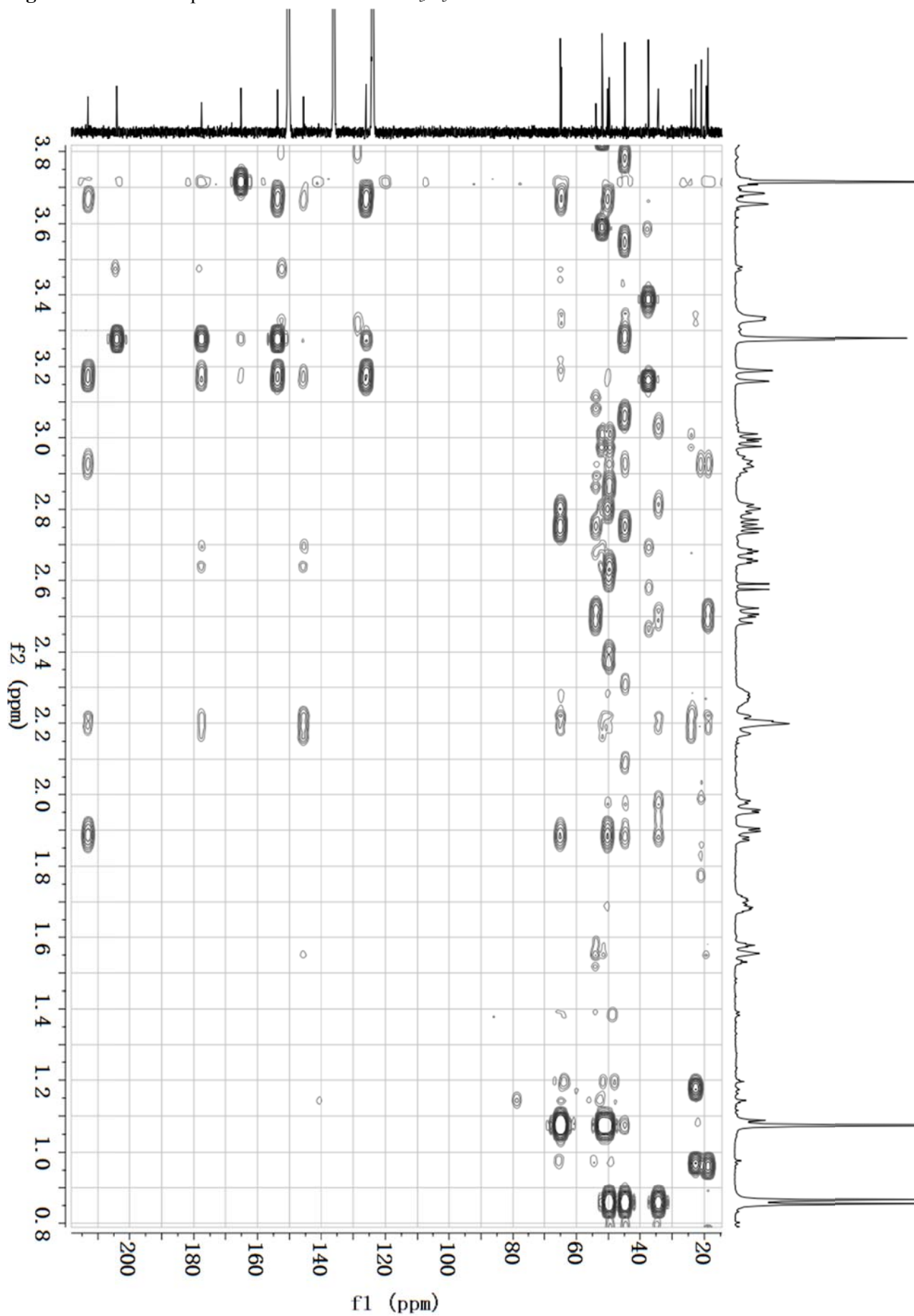


Figure S24. ROESY spectrum for alkaloid 3 C<sub>5</sub>D<sub>5</sub>N.

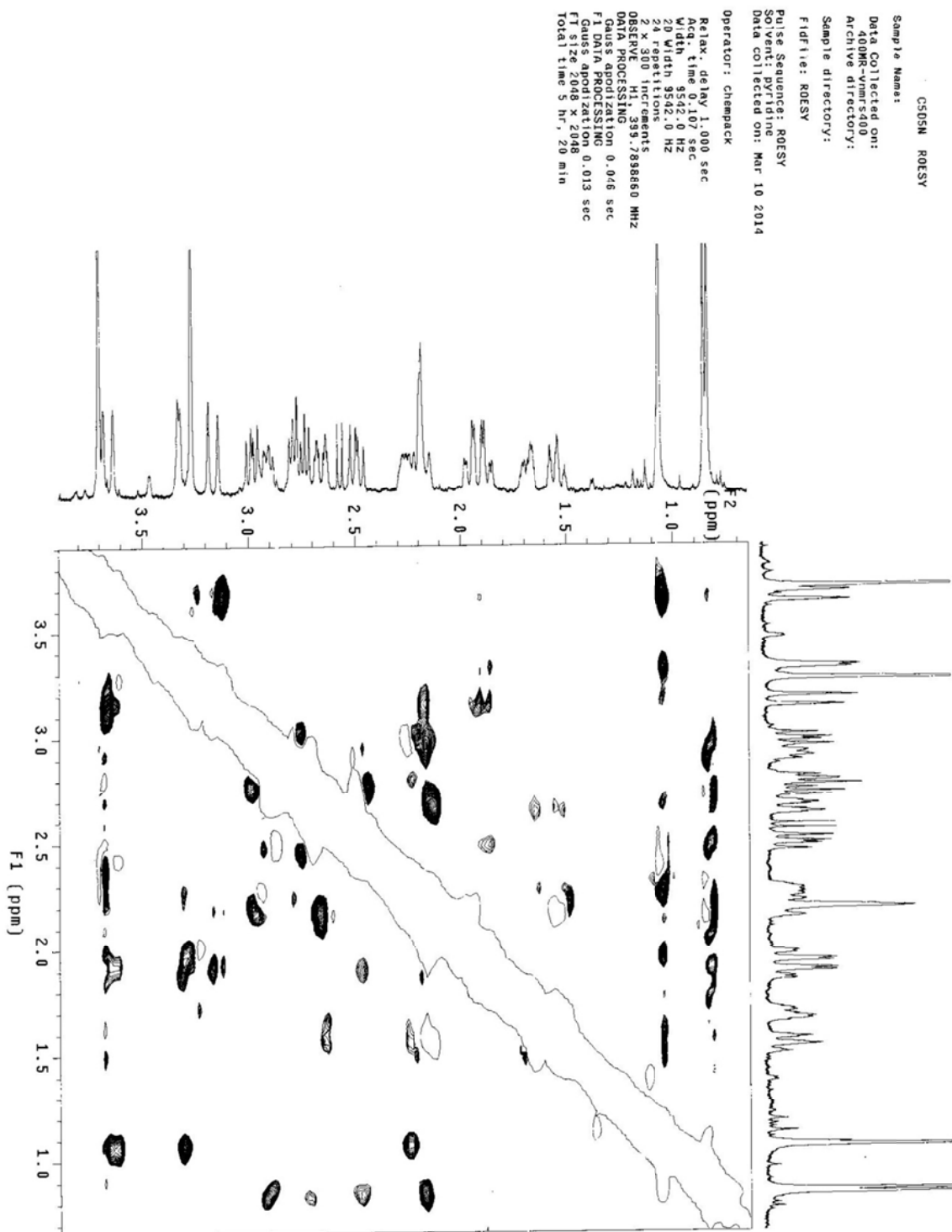


Figure S25 IR spectrum for alkaloid 3.

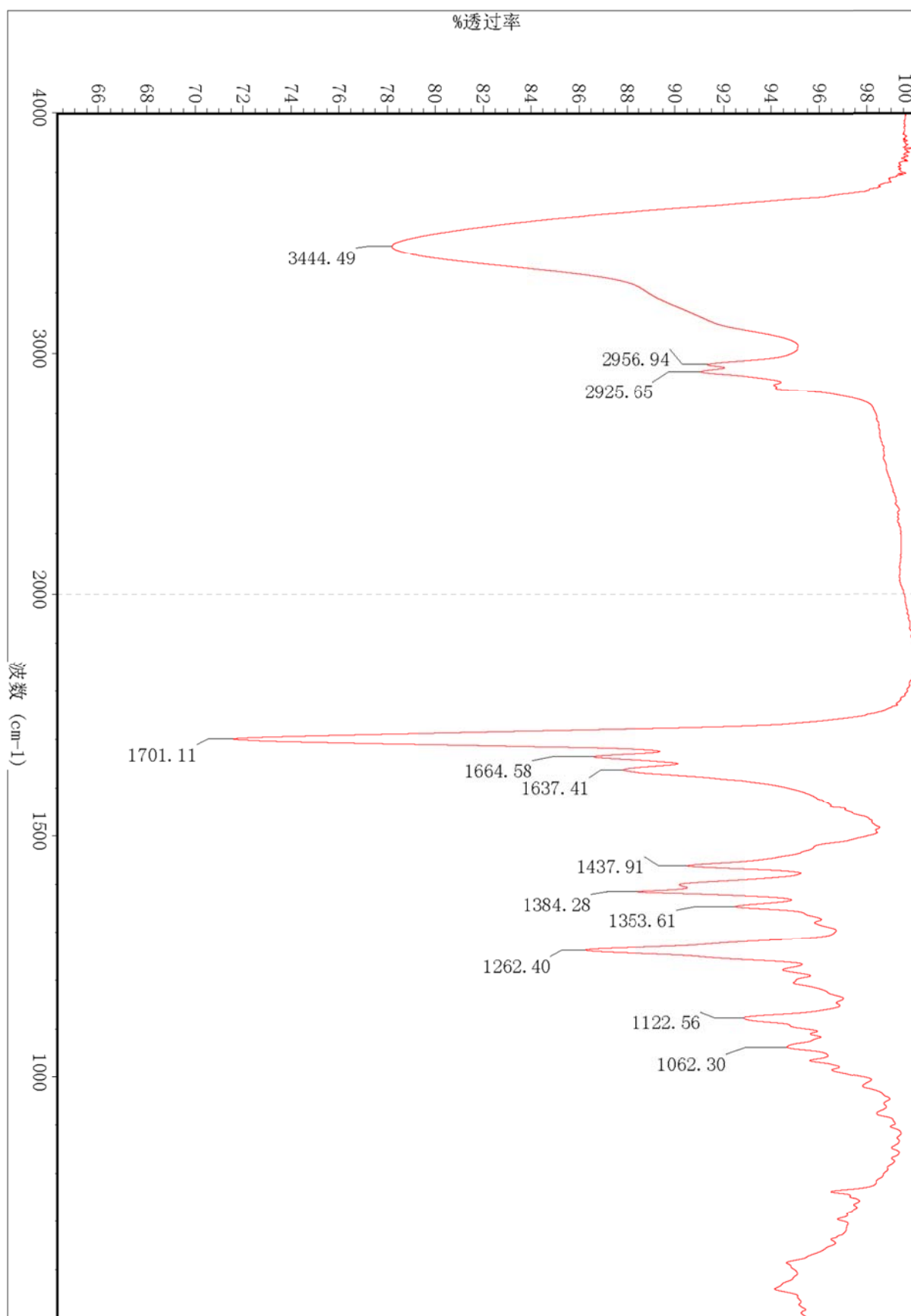




Figure S26. (+)-ESIMS spectrum for alkaloid 3.

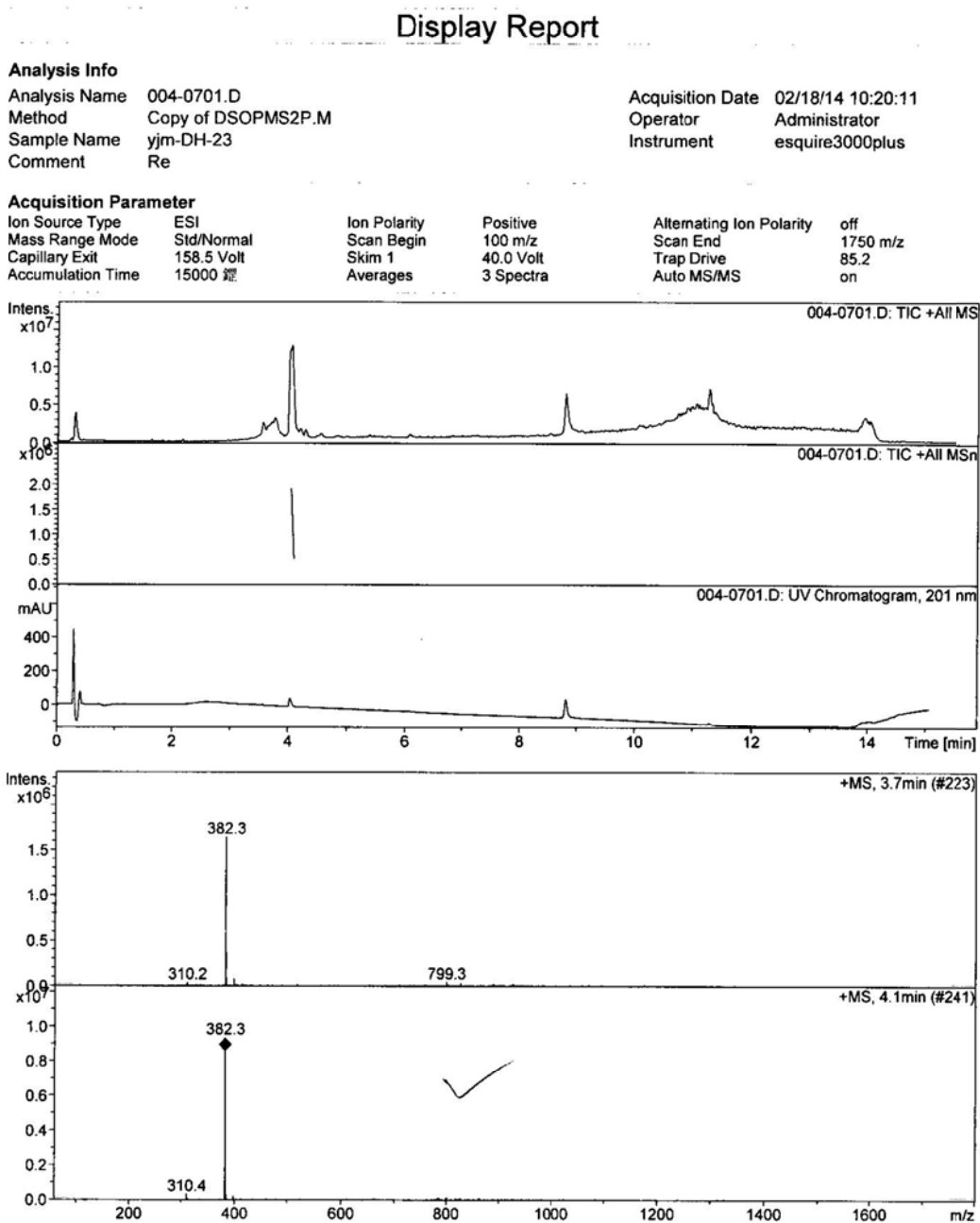


Figure S27. (+)-HRESIMS spectrum for alkaloid 3.

Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0  
 Element prediction: Off  
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

208 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-2 O: 0-20

DH-23

LCT PXE KE324

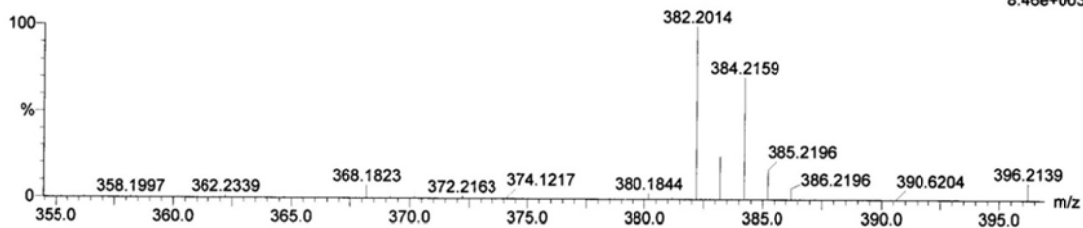
08-May-2014

14:28:23

1: TOF MS ES+

8.46e+003

DH-23\_0508 82 (1.784) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (69:87)



Minimum: -1.5  
 Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE  | i-FIT | i-FIT (Norm) | Formula      |
|----------|------------|------|------|------|-------|--------------|--------------|
| 382.2014 | 382.2018   | -0.4 | -1.0 | 10.5 | 160.0 | 0.0          | C23 H28 N O4 |

Figure S28.  $^1\text{H}$  NMR spectrum for alkaloid **4** in  $\text{CDCl}_3$ .

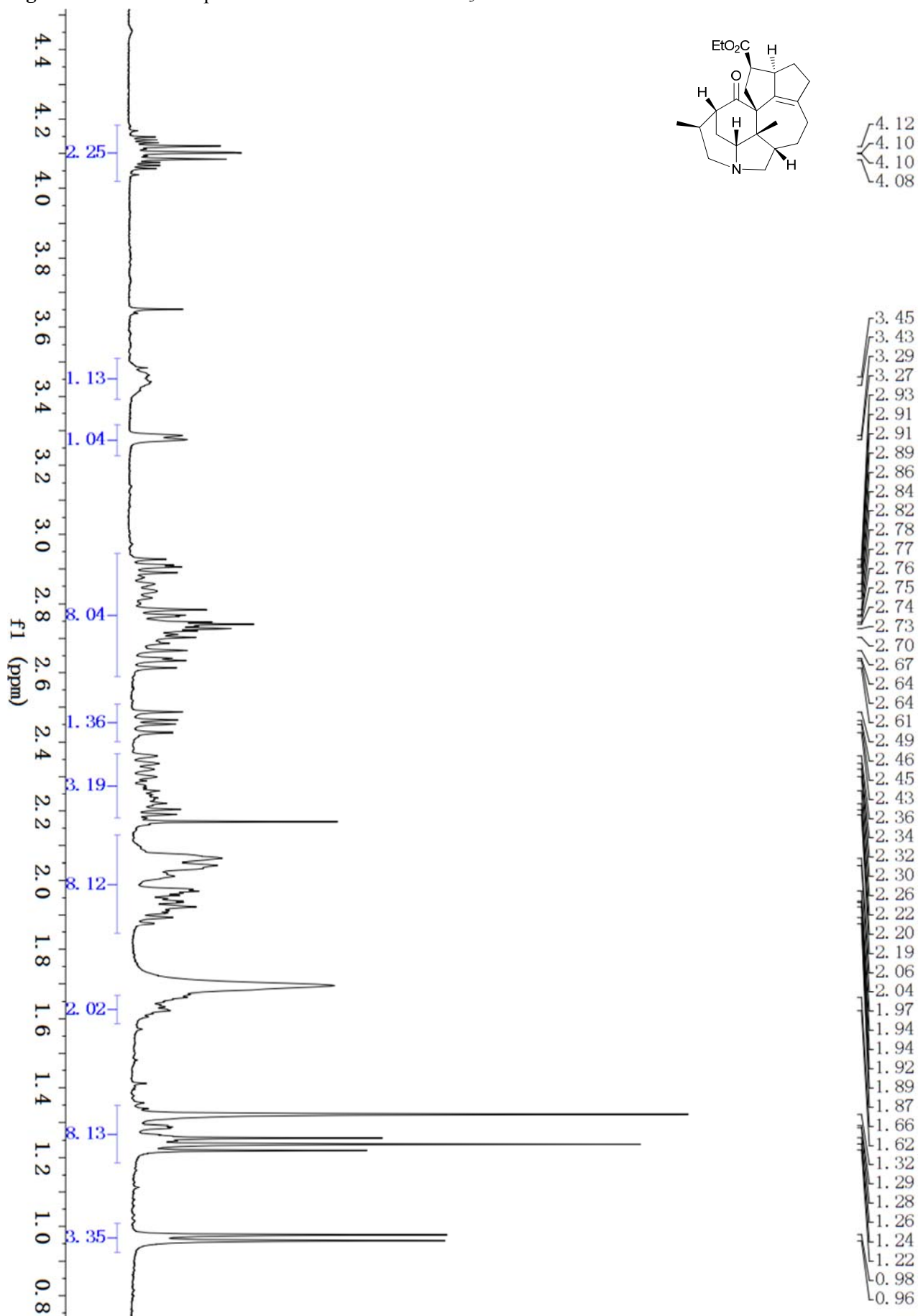


Figure S29.  $^{13}\text{C}$  NMR spectrum for alkaloid **4** in  $\text{CDCl}_3$ .

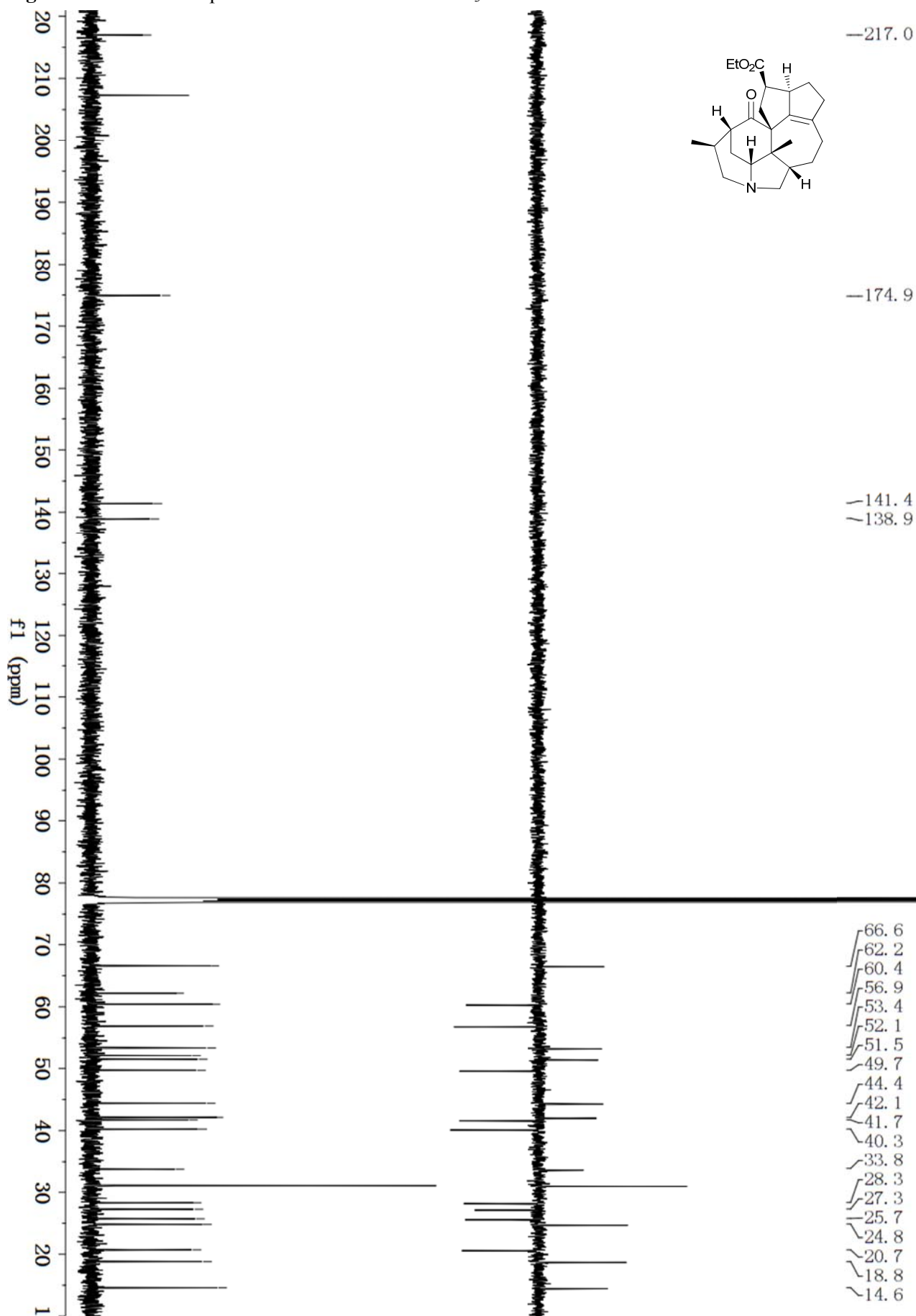


Figure S30. HSQC spectrum for alkaloid 4 in CDCl<sub>3</sub>.

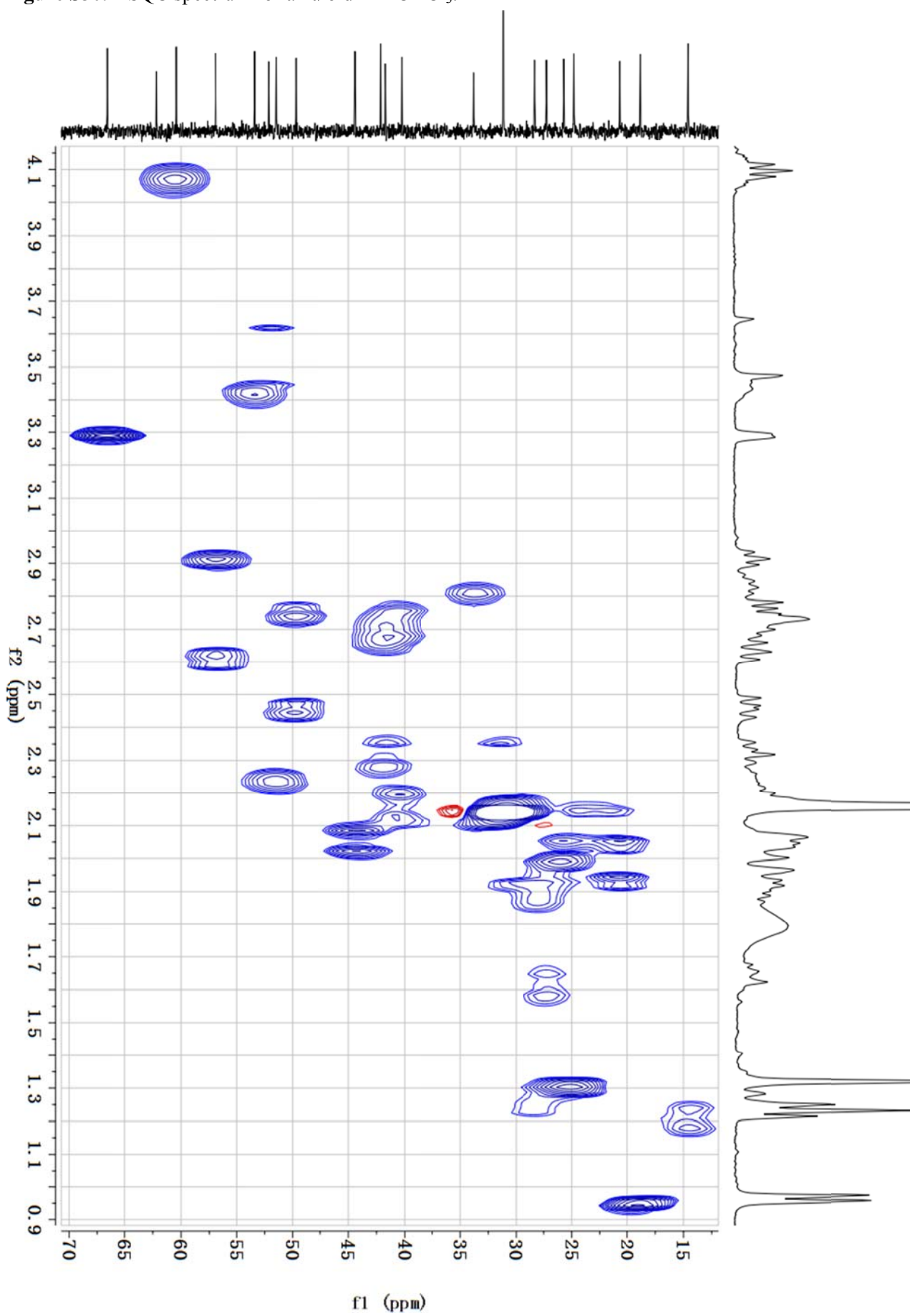


Figure S31. HMBC spectrum for alkaloid **4** in CDCl<sub>3</sub>.

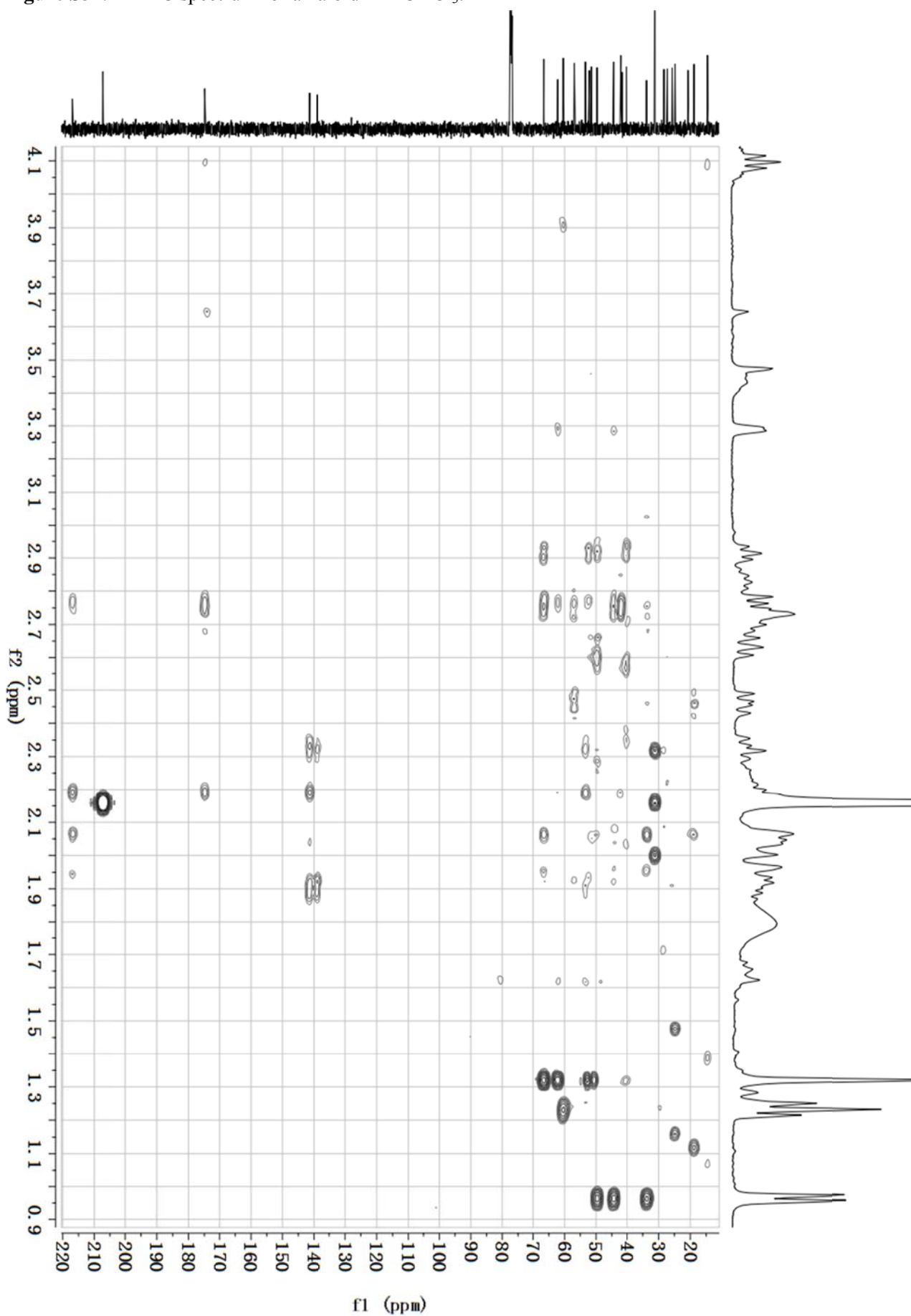
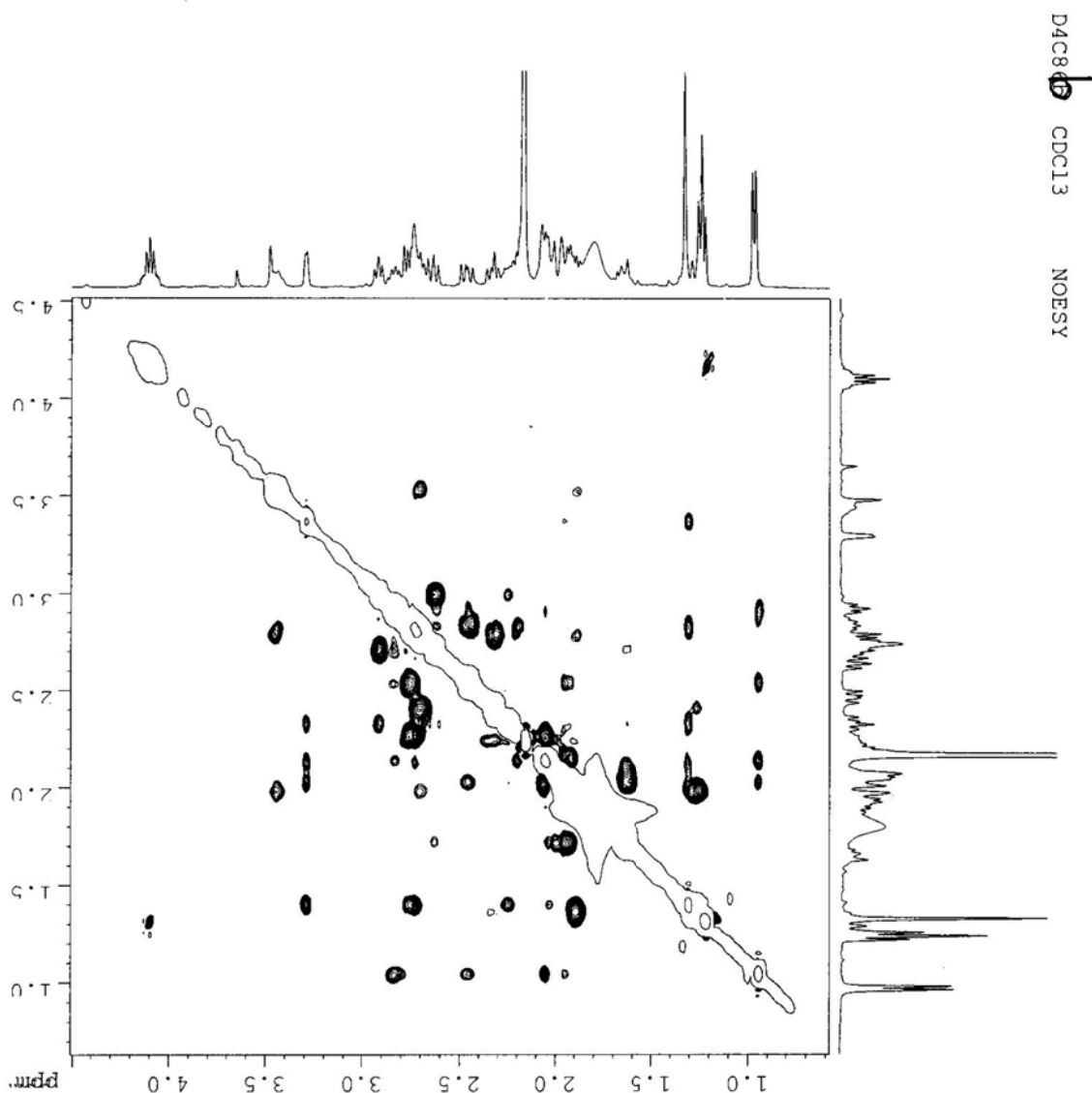


Figure S32. NOESY spectrum for alkaloid 4 in CDCl<sub>3</sub>.



D4C8 CDCl<sub>3</sub> NOESY

```

Current Data Parameters
NAME          D4C8b
EXPNO        15
PROCNO       1

F2 - Acquisition Parameters
Date_         20140718
Time         10.18
INSTRUM      spect
PROBHD       5 mm PABUL13C
PULPROG      noesyphpp
TD           2048
SOLVENT      CDCl3
NS           4
DS           16
SWH          6393.862 Hz
FIDRES       3.122003 Hz
AQ           0.1602036 sec
RG           63.28
DE           78.200 usec
TE           295.2 K
D0           0.00006600 sec
D1           1.00000000 sec
D8           0.60000002 sec
D11          0.03000000 sec
D12          0.00002000 sec
INO          0.00015620 sec

===== CHANNEL f1 =====
NUC1         1H
P17          9.50 usec
PL1          2500.00 usec
PL10         25.00000000 W
PL11         3.33759999 W
SFO1         400.1328009 MHz

F1 - Acquisition parameters
TD           320
SFO1         400.1328 MHz
FIDRES       20.006580 Hz
SM           16.000 ppm
FMODE        States-rppi

F2 - Processing parameters
SI           1024
SF           400.1300034 MHz
WDW          QSINC
SSB          0.65
LB           0 Hz
GB           0
PC           1.00

F1 - Processing parameters
SI           1024
MC2          States-rppi
SF           400.1299746 MHz
WM          States-rppi
SSB         2
LB           0 Hz
GB           0
  
```

Figure S33. IR spectrum for alkaloid 4.

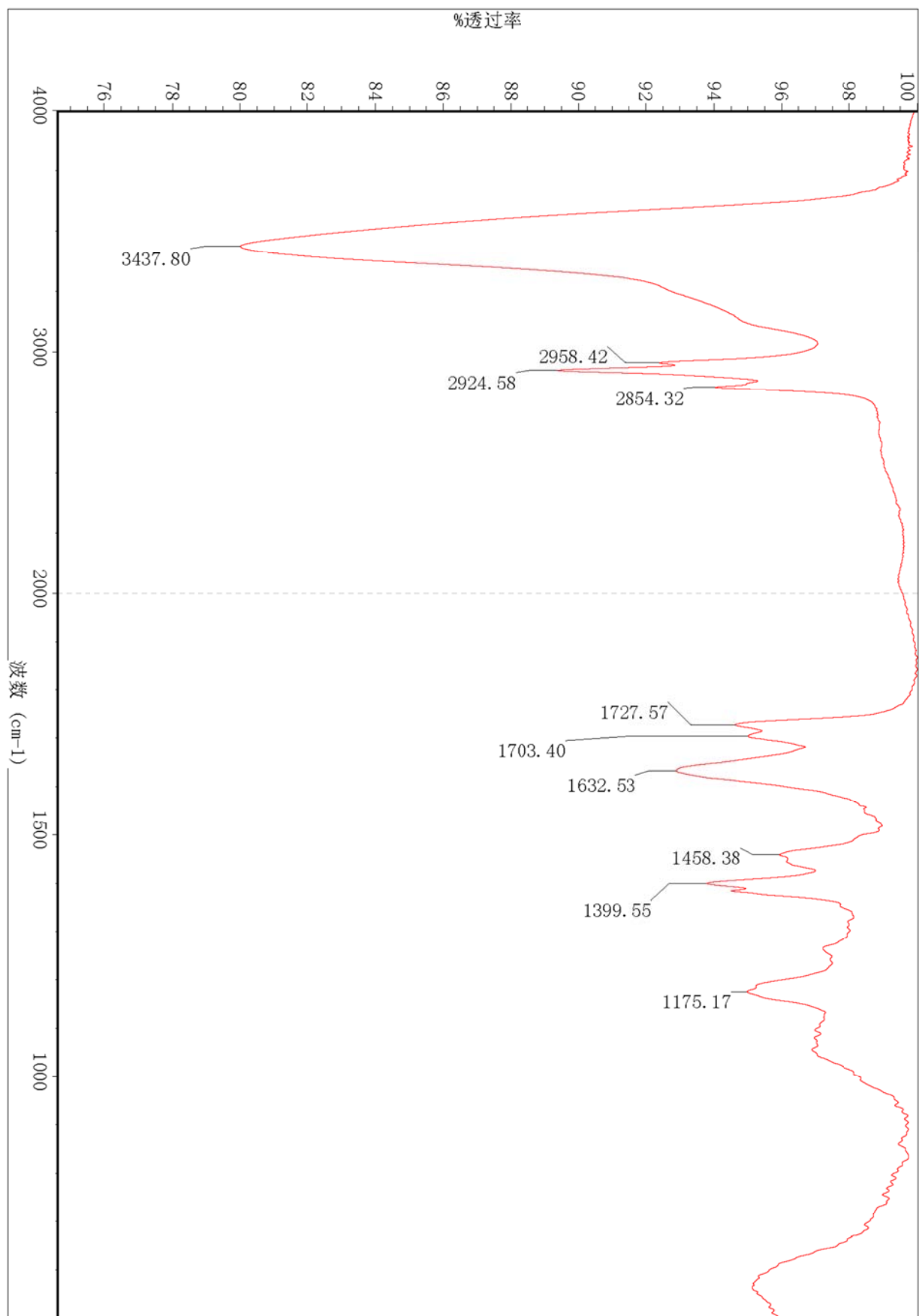




Figure S34. (+)-ESIMS spectrum for alkaloid 4.

### Display Report

**Analysis Info**

Analysis Name 054-3201.D  
 Method Copy of DSOPMS2P.M  
 Sample Name yjm-D4C8b  
 Comment W/

Acquisition Date 07/17/14 20:03:11  
 Operator Administrator  
 Instrument esquire3000plus

**Acquisition Parameter**

|                   |            |              |           |                          |          |
|-------------------|------------|--------------|-----------|--------------------------|----------|
| Ion Source Type   | ESI        | Ion Polarity | Positive  | Alternating Ion Polarity | off      |
| Mass Range Mode   | Std/Normal | Scan Begin   | 100 m/z   | Scan End                 | 1750 m/z |
| Capillary Exit    | 158.5 Volt | Skim 1       | 40.0 Volt | Trap Drive               | 85.2     |
| Accumulation Time | 15000 經    | Averages     | 3 Spectra | Auto MS/MS               | on       |

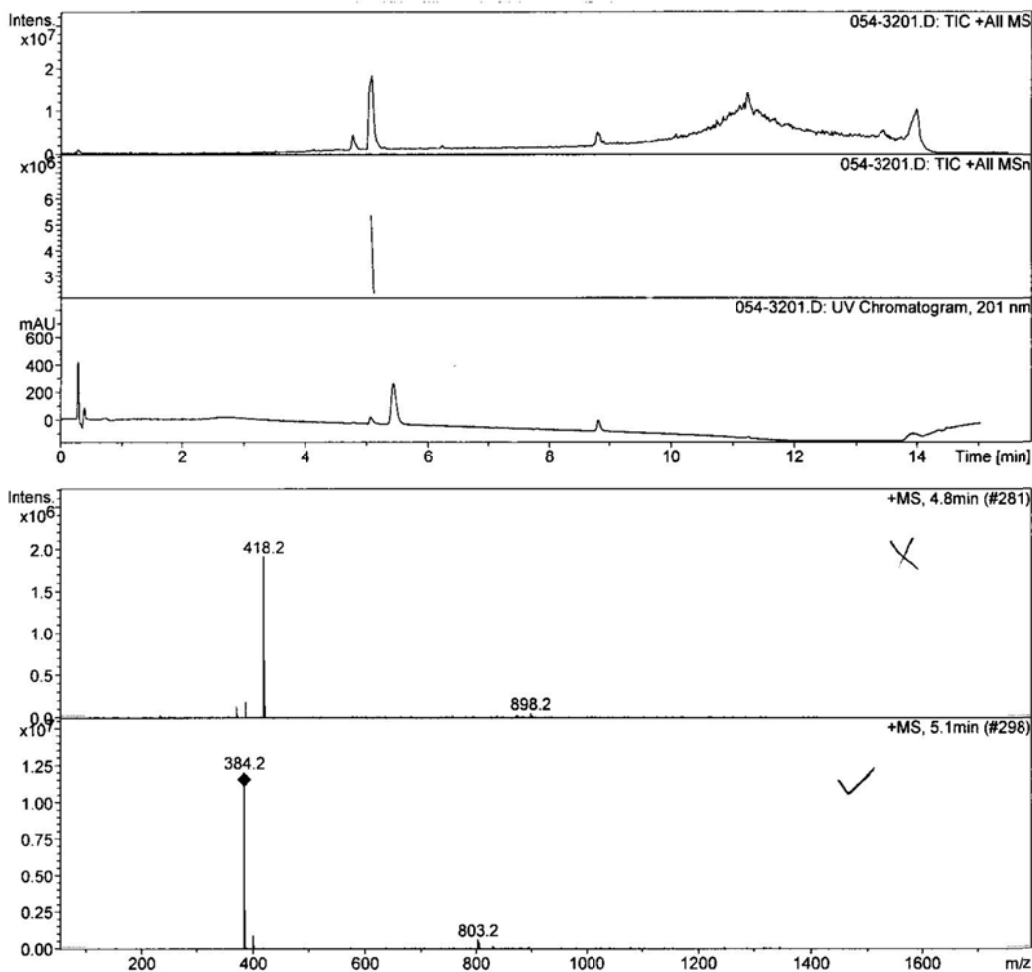


Figure S35. (+)-HRESIMS spectrum for alkaloid 4.

Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0  
 Element prediction: Off  
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

326 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-4 O: 0-20

DH-61

LCT PXE KE324

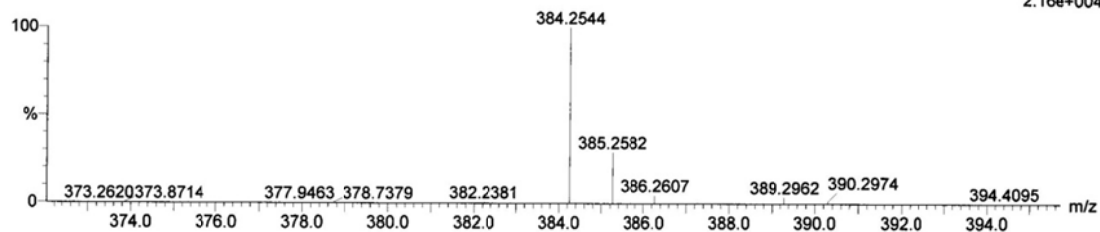
29-Aug-2014

10:41:41

1: TOF MS ES+

2.16e+004

DH-61\_0829 18 (0.371) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (10:24)



Minimum: -1.5  
 Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa | PPM | DBE | i-FIT | i-FIT (Norm) | Formula      |
|----------|------------|-----|-----|-----|-------|--------------|--------------|
| 384.2544 | 384.2539   | 0.5 | 1.3 | 8.5 | 151.5 | 0.0          | C24 H34 N O3 |

Figure S36.  $^1\text{H}$  NMR spectrum for alkaloid **5** in  $\text{CD}_3\text{OD}$ .

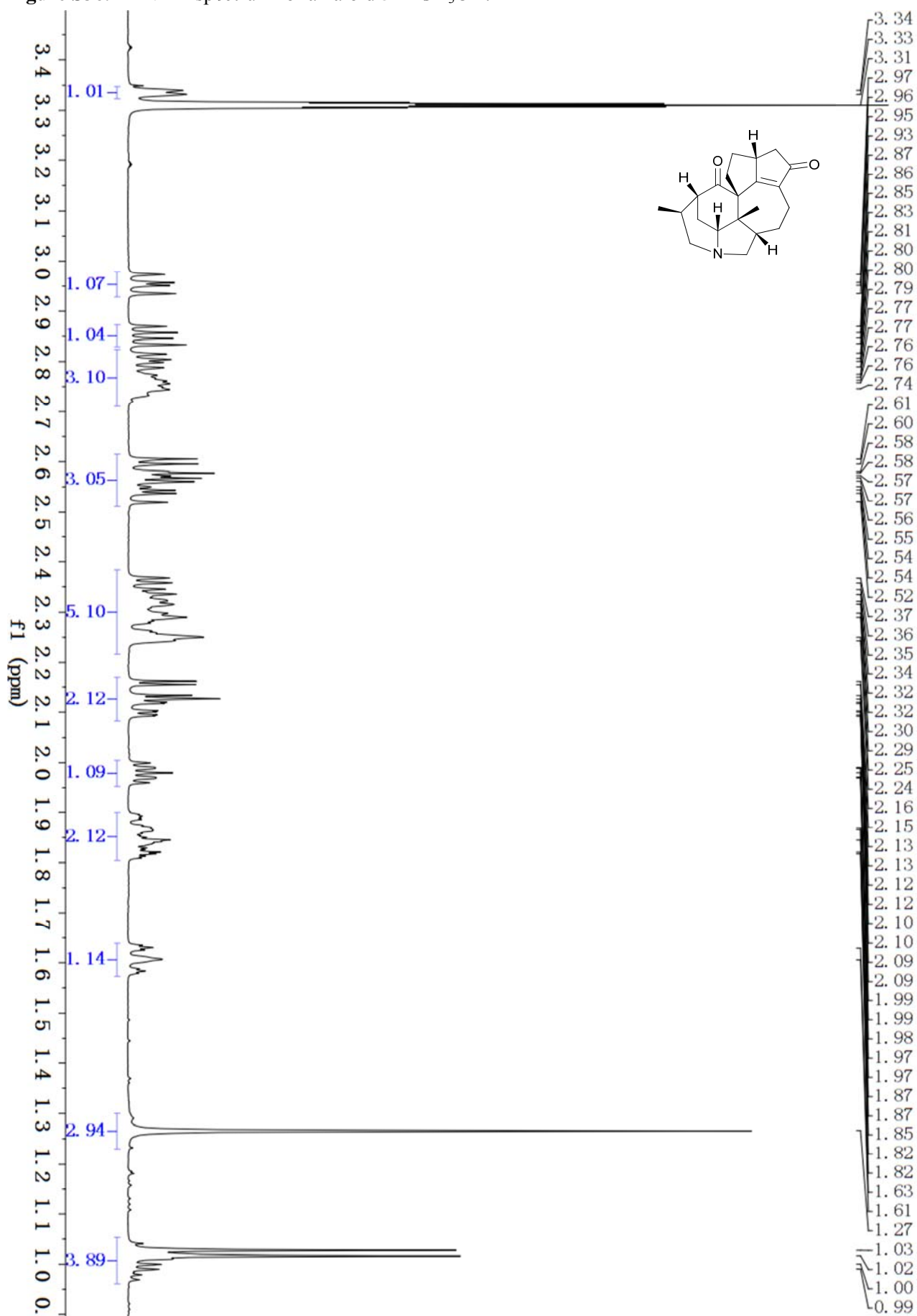
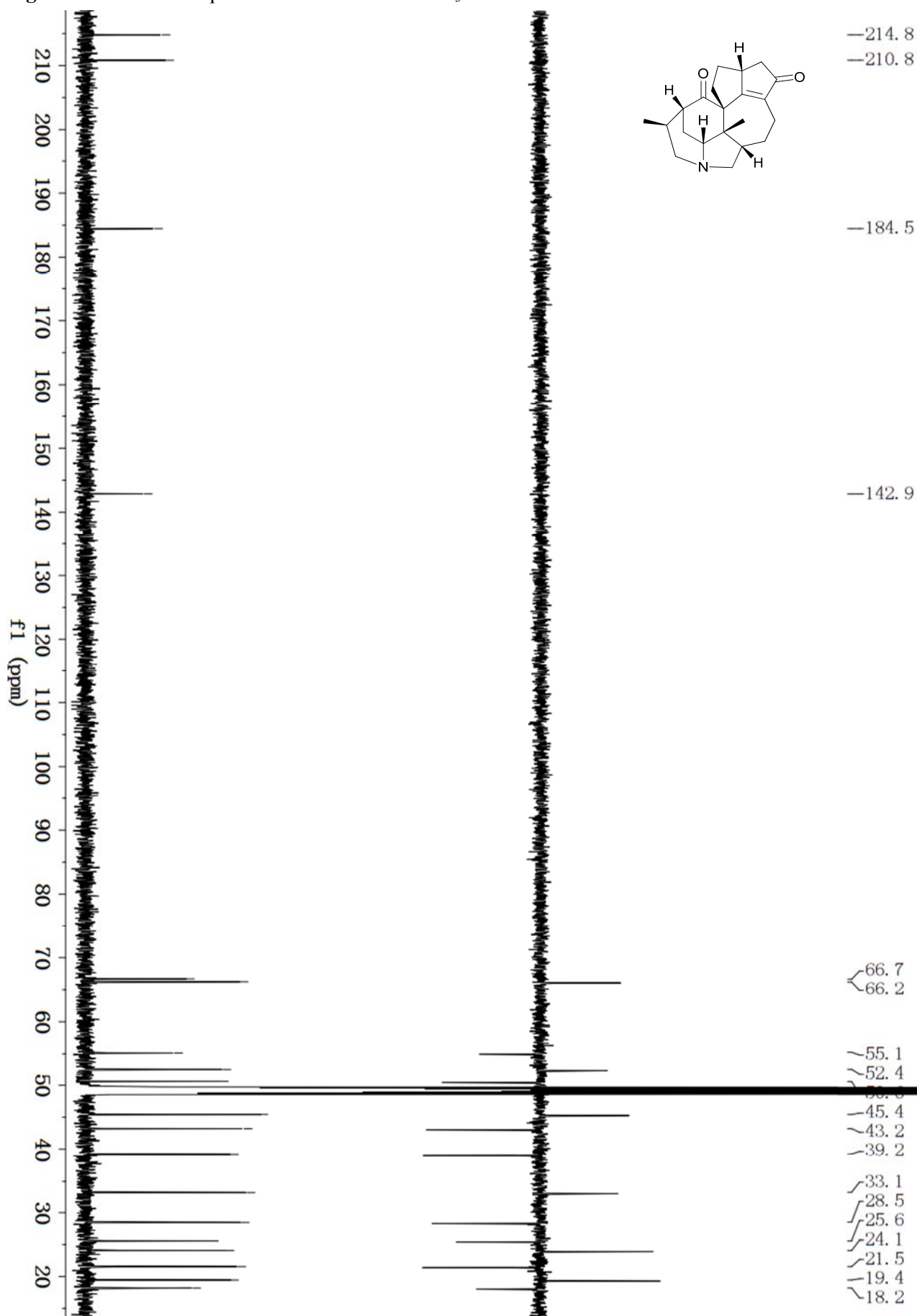


Figure S37.  $^{13}\text{C}$  NMR spectrum for alkaloid 5 in  $\text{CD}_3\text{OD}$ .



**Figure S38.**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum for alkaloid **5** in  $\text{CD}_3\text{OD}$ .

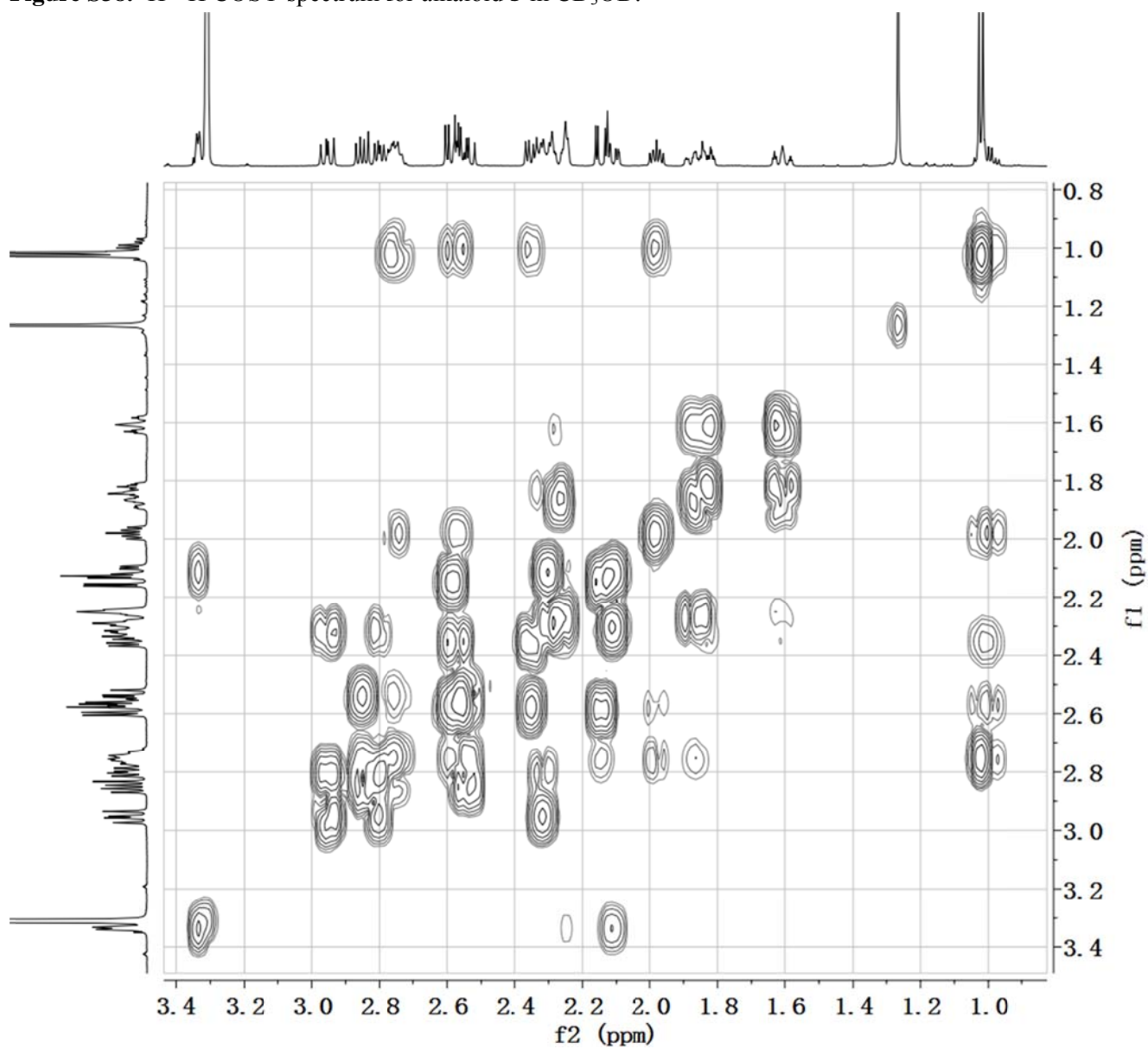
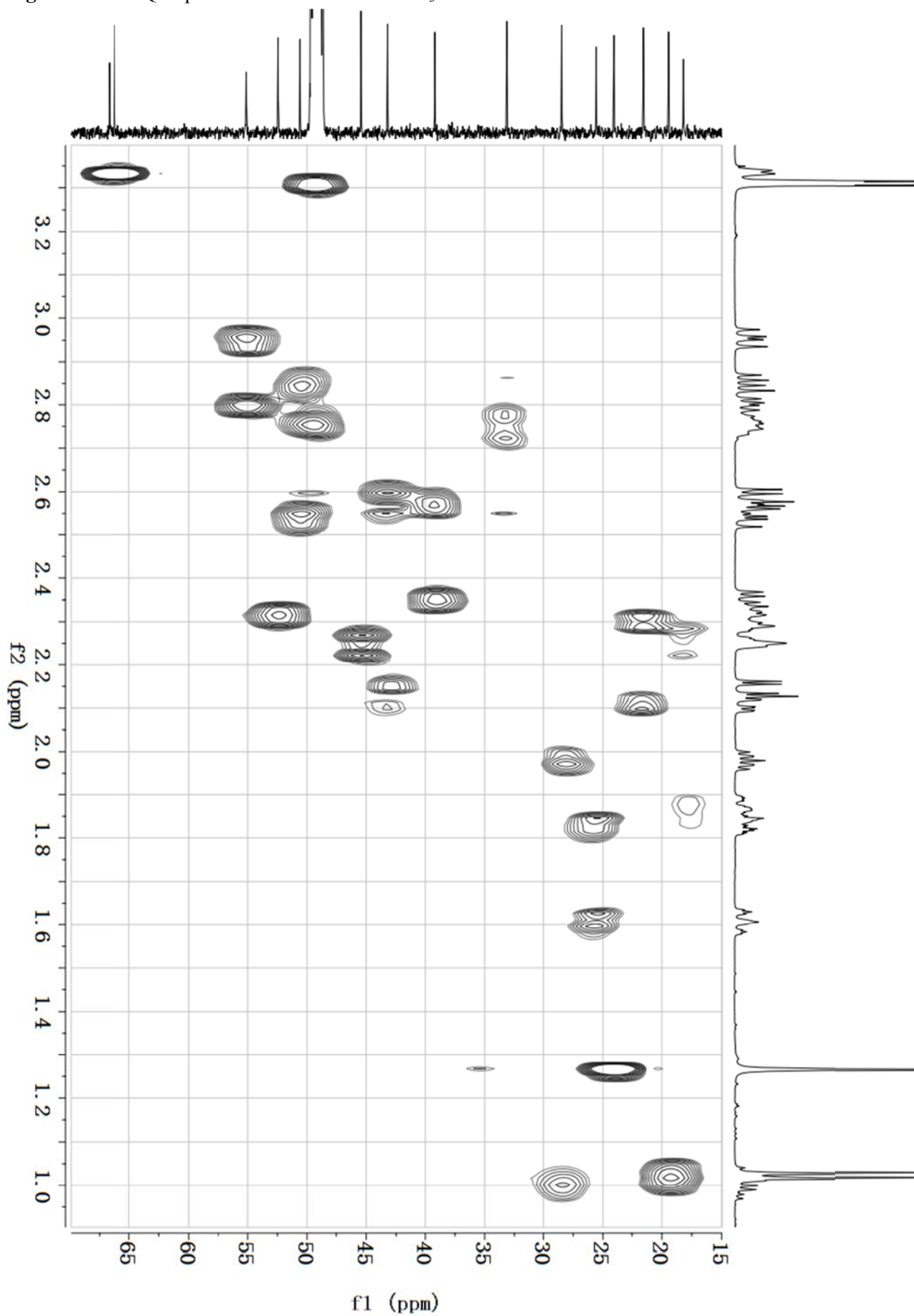


Figure S39. HSQC spectrum for alkaloid 5 in CD<sub>3</sub>OD.



**Figure S40.** HMBC spectrum for alkaloid **5** in CD<sub>3</sub>OD.

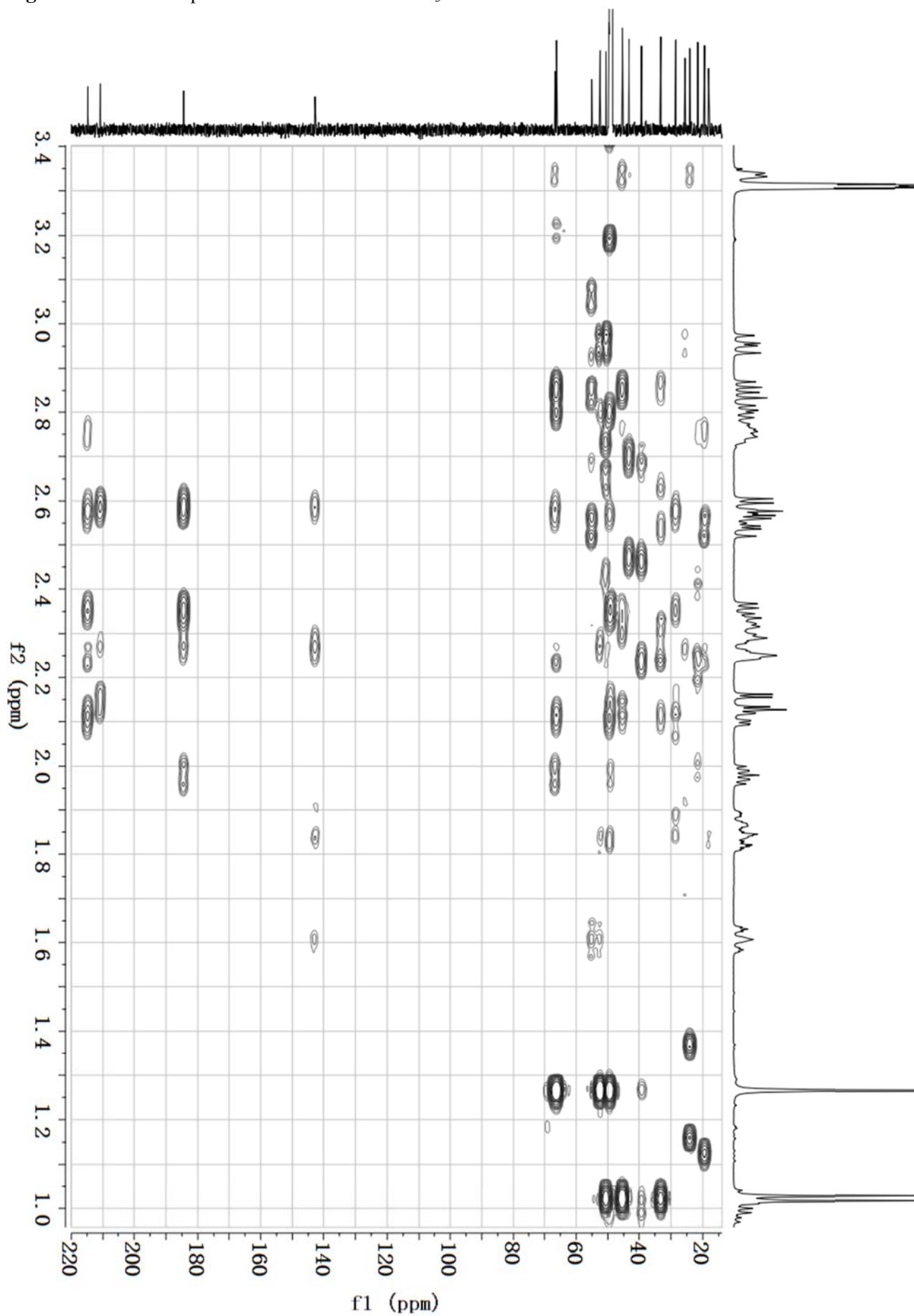


Figure S41. NOESY spectrum for alkaloid 5 in CD<sub>3</sub>OD.

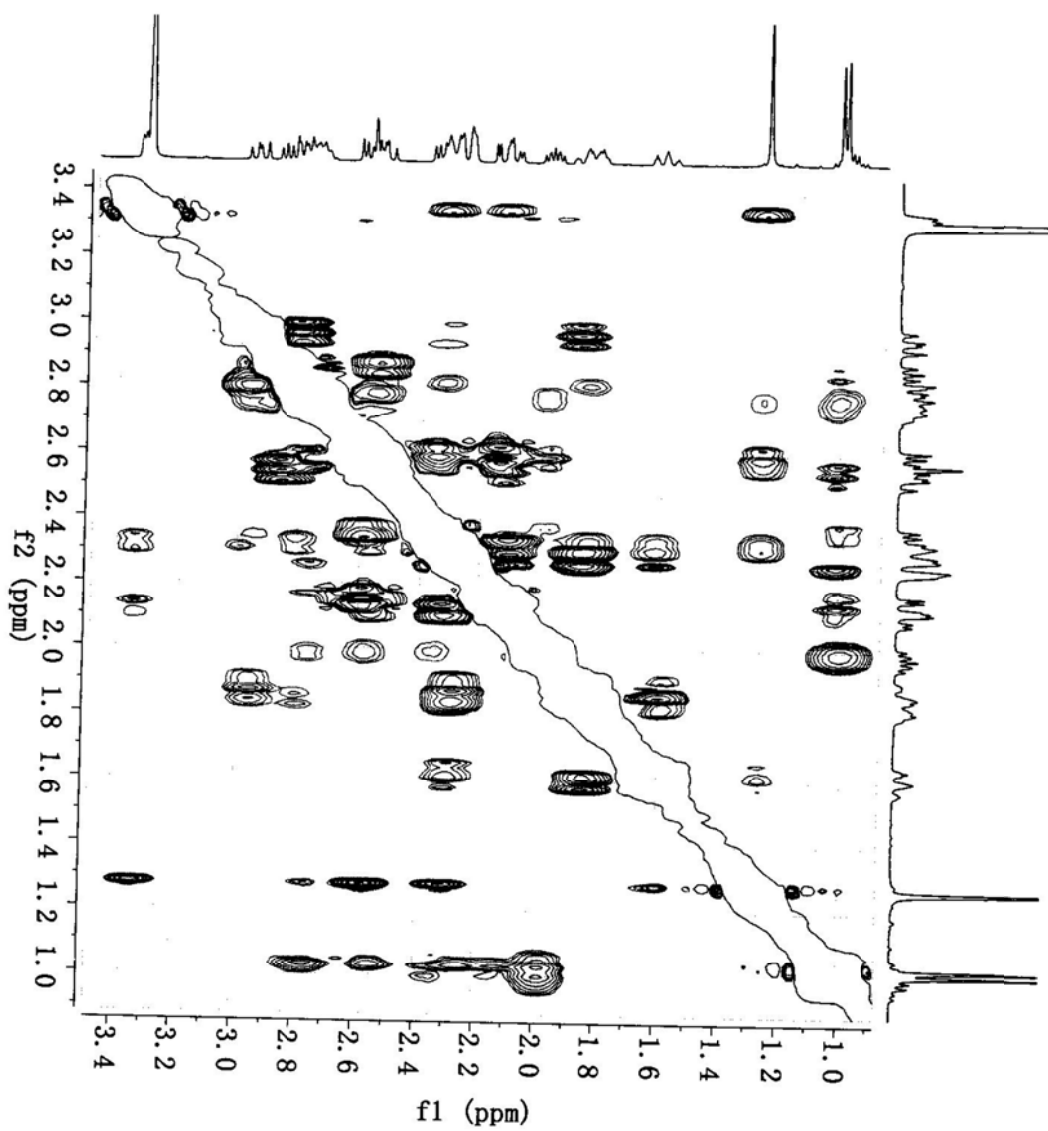




Figure S42. IR spectrum for alkaloid 5.

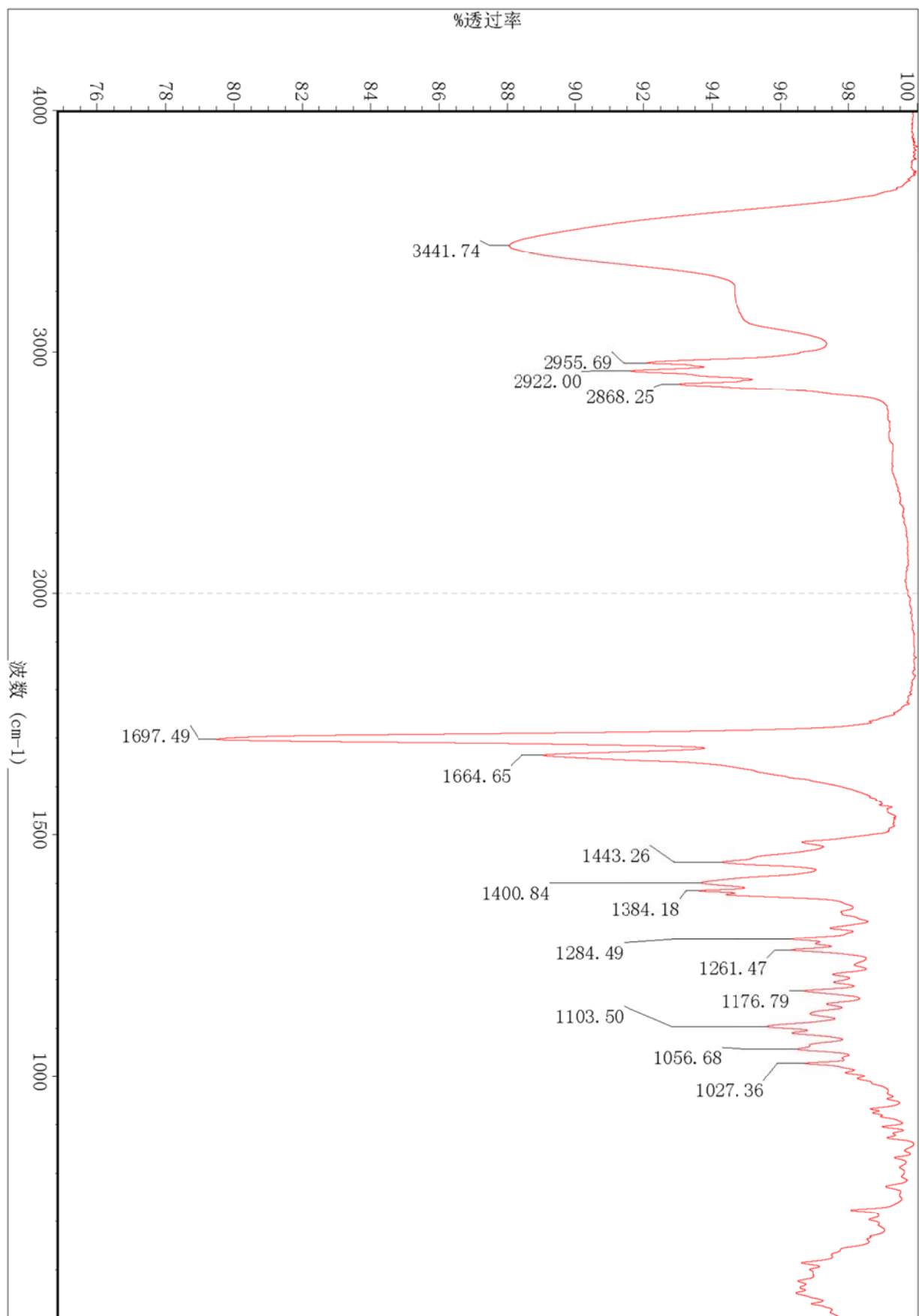


Figure S43. (+)-ESIMS spectrum for alkaloid 5.

### Display Report

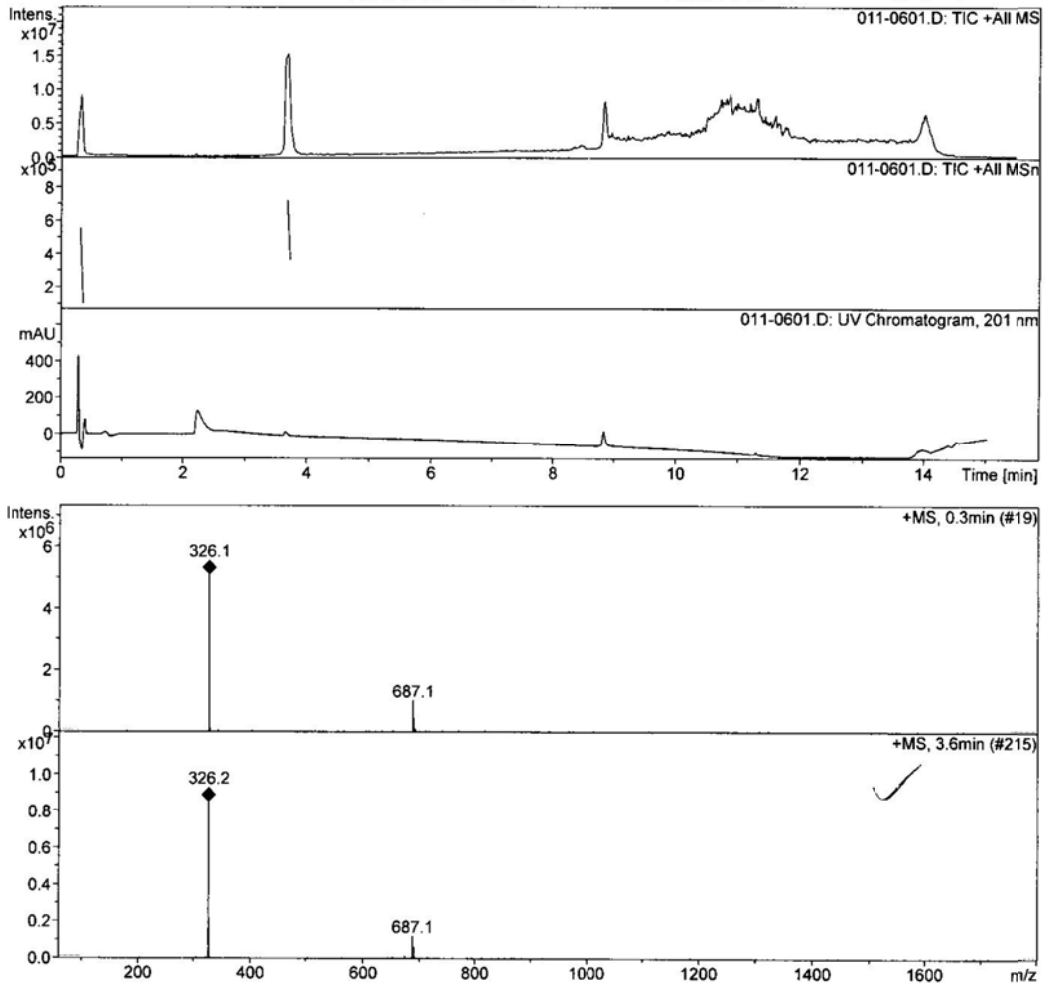
#### Analysis Info

Analysis Name 011-0601.D  
Method Copy of DSOPMS2P.M  
Sample Name yjm-DH-47-1  
Comment ?

Acquisition Date 05/08/14 10:47:11  
Operator Administrator  
Instrument esquire3000plus

#### Acquisition Parameter

|                   |            |              |           |                          |          |
|-------------------|------------|--------------|-----------|--------------------------|----------|
| Ion Source Type   | ESI        | Ion Polarity | Positive  | Alternating Ion Polarity | off      |
| Mass Range Mode   | Std/Normal | Scan Begin   | 100 m/z   | Scan End                 | 1750 m/z |
| Capillary Exit    | 158.5 Volt | Skim 1       | 40.0 Volt | Trap Drive               | 85.2     |
| Accumulation Time | 15000 經    | Averages     | 3 Spectra | Auto MS/MS               | on       |



**Figure S44.** (+)-HRESIMS spectrum for alkaloid **5**.

**Elemental Composition Report**

**Single Mass Analysis**

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

159 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-2 O: 0-20

DH-47-1

LCT PXE KE324

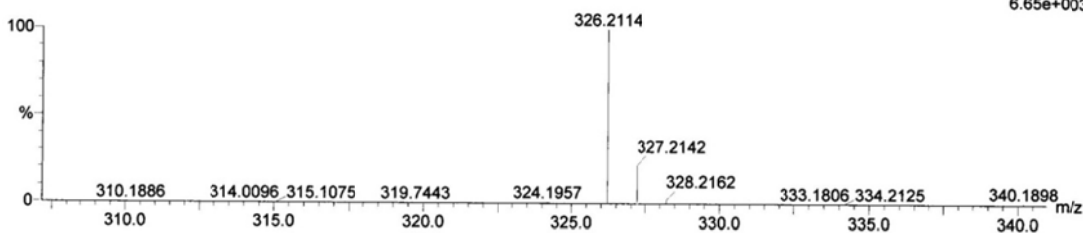
04-Jun-2014

15:05:47

1: TOF MS ES+

6.65e+003

DH-47-1\_0604 29 (0.637) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (17:29)



Minimum:

Maximum: 5.0 10.0 -1.5

50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT | i-FIT (Norm) | Formula      |
|----------|------------|------|------|-----|-------|--------------|--------------|
| 326.2114 | 326.2120   | -0.6 | -1.8 | 8.5 | 94.0  | 0.0          | C21 H28 N O2 |

Figure S45.  $^1\text{H}$  NMR spectrum for alkaloid **6** in  $\text{CD}_3\text{OD}$ .

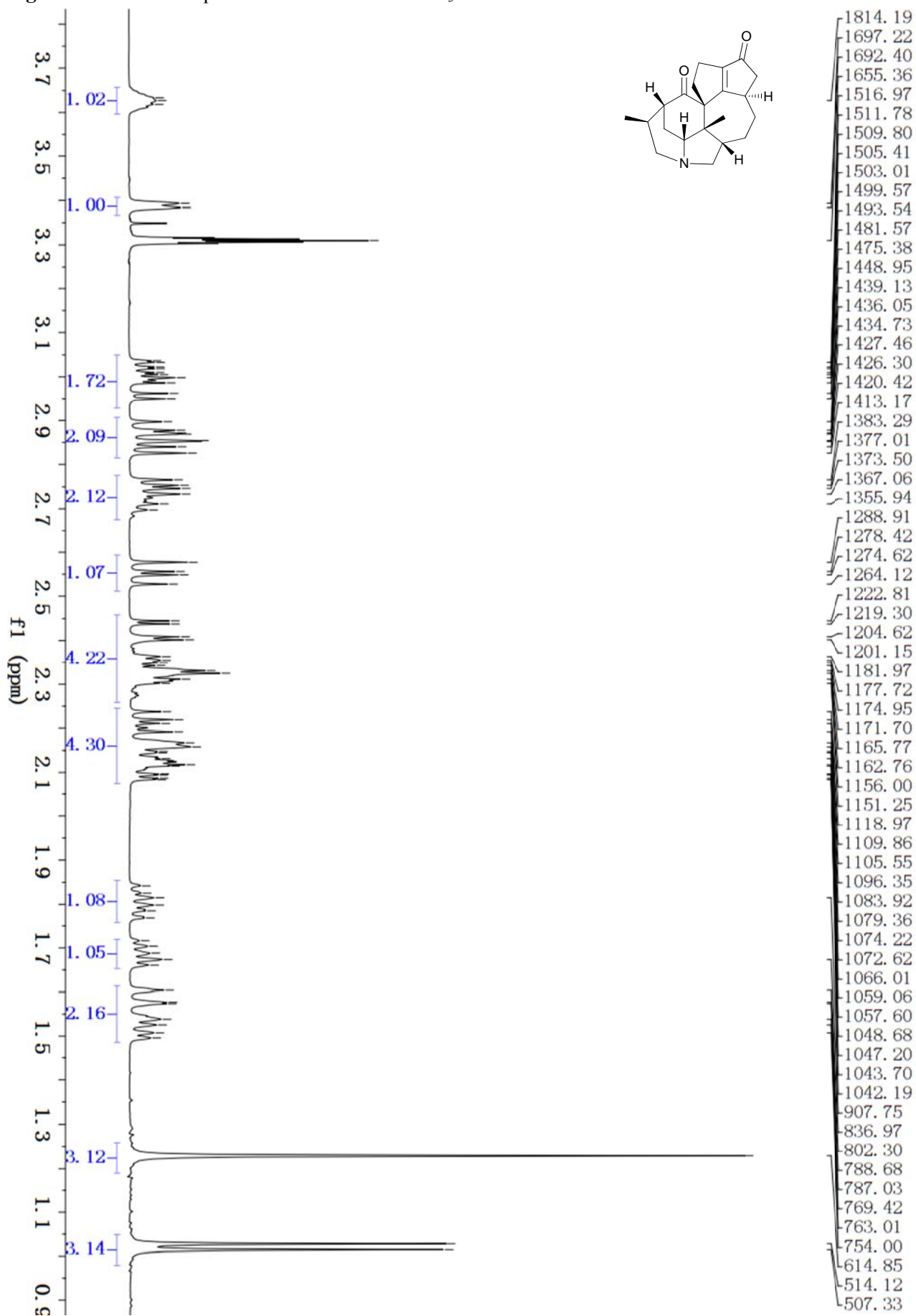


Figure S46.  $^{13}\text{C}$  NMR spectrum for alkaloid **6** in  $\text{CD}_3\text{OD}$ .

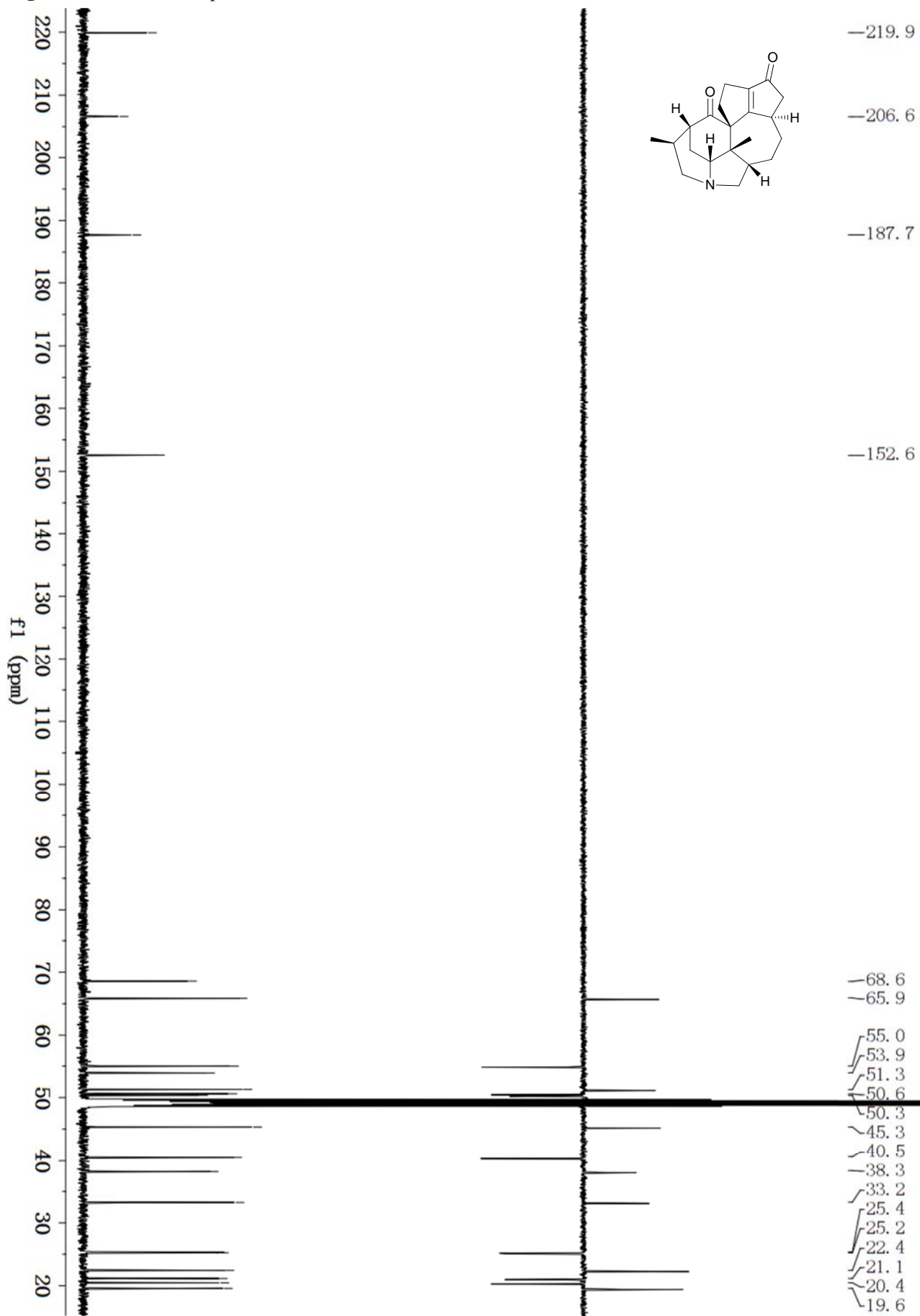




Figure S48 HSQC spectrum for alkaloid 6 in CD<sub>3</sub>OD.

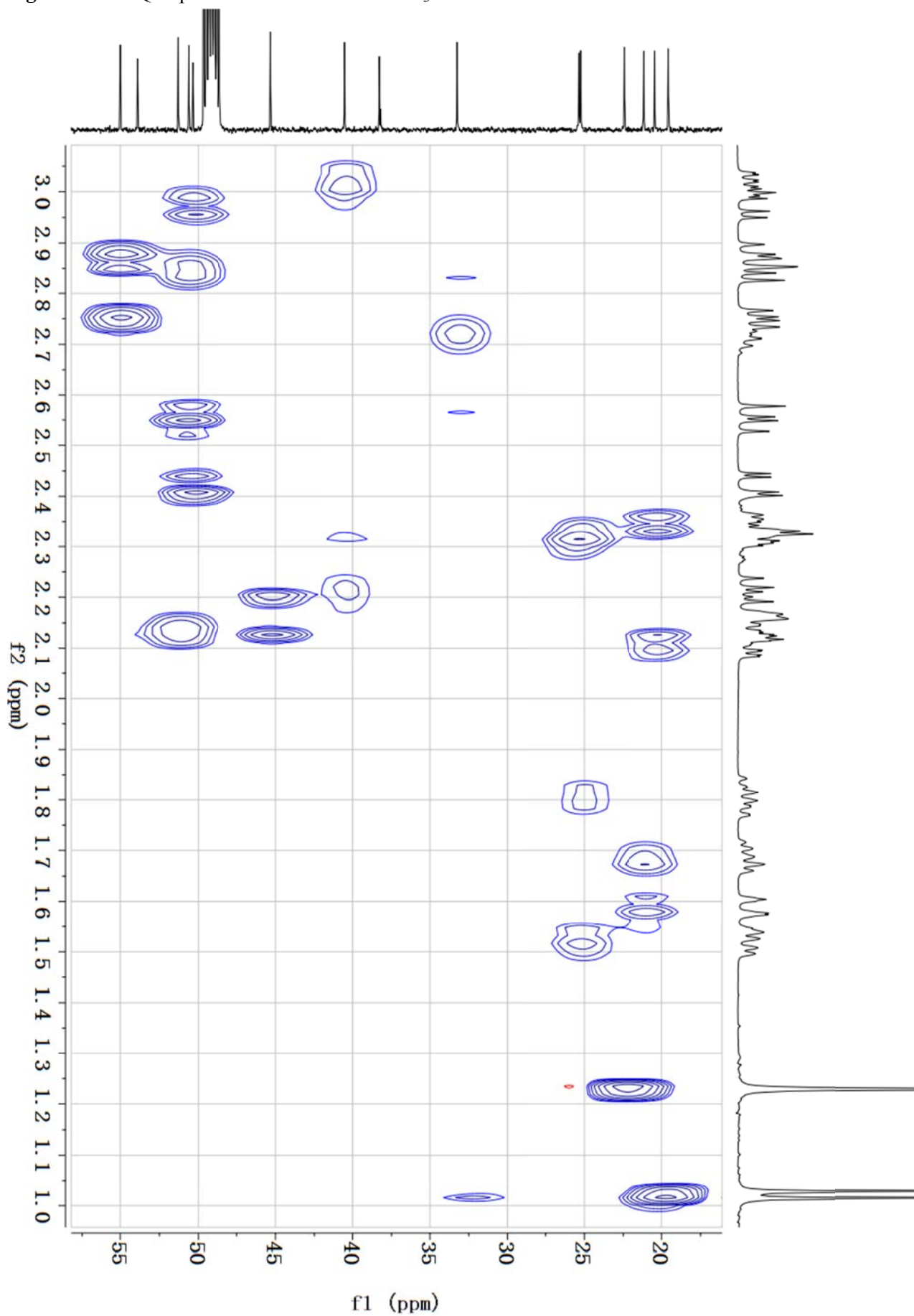


Figure S49. HMBC spectrum for alkaloid 6 in CD<sub>3</sub>OD.

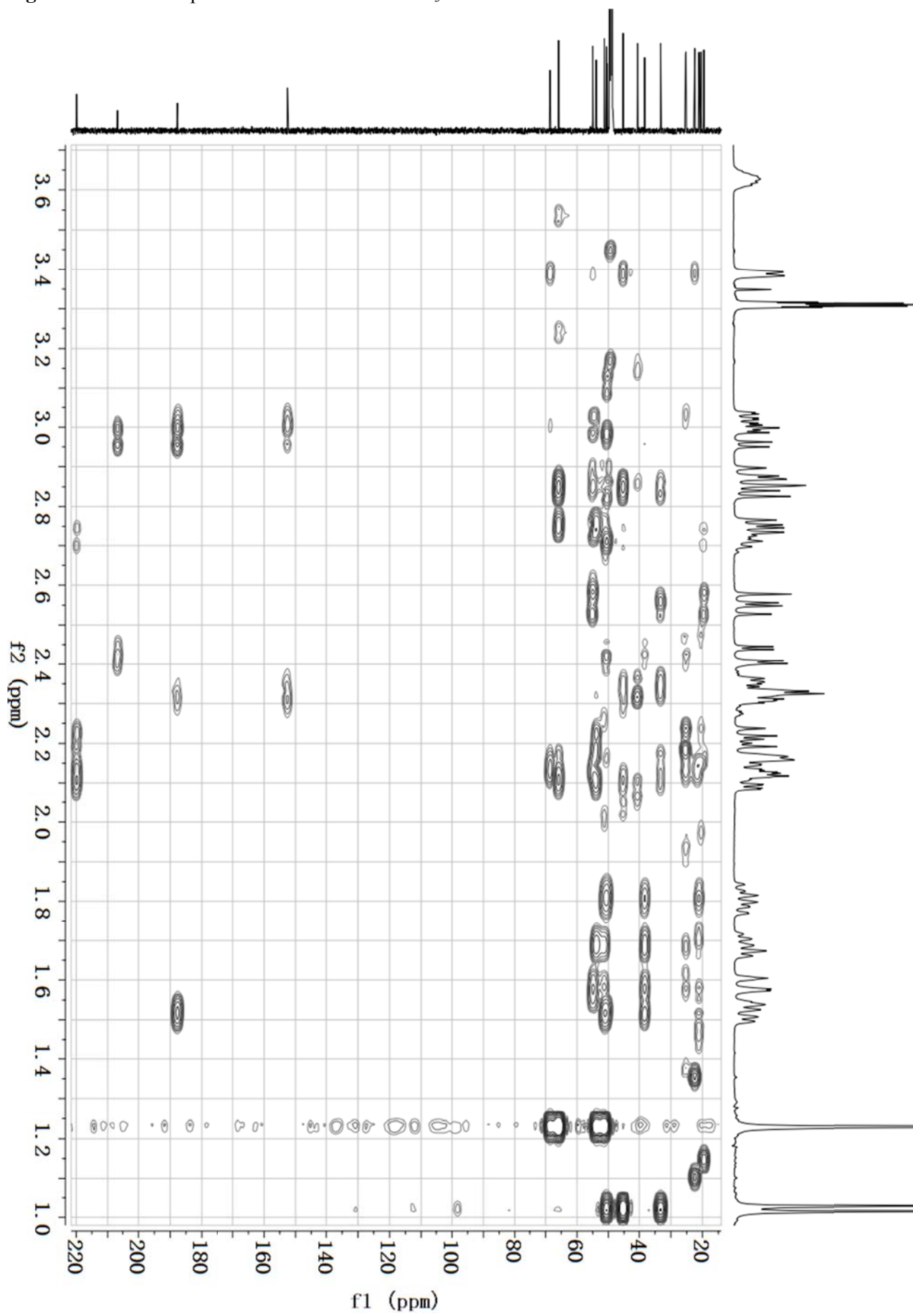




Figure S50. NOESY spectrum for alkaloid 6 in CD<sub>3</sub>OD.

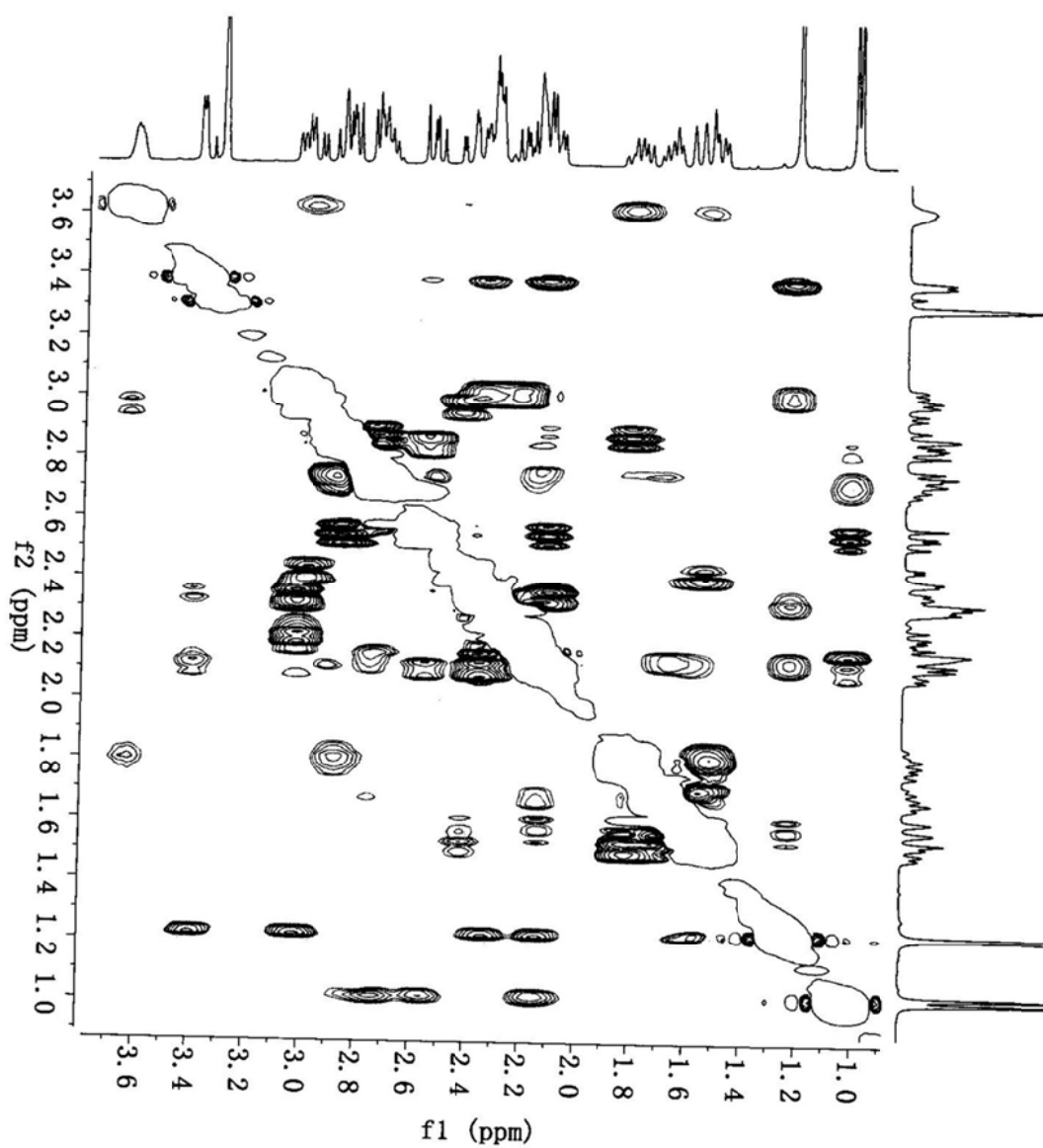


Figure S51. IR spectrum for alkaloid 6.

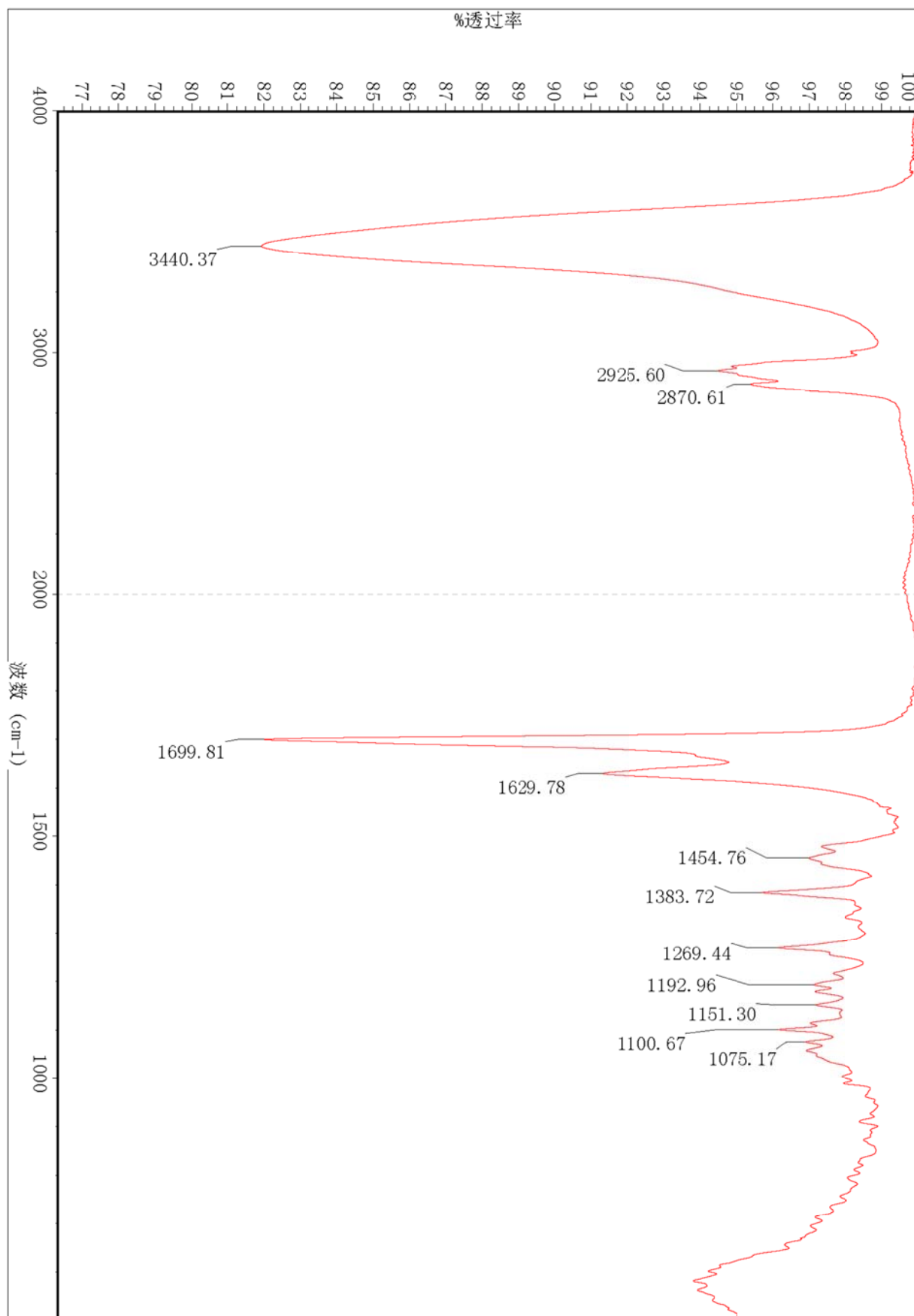


Figure S52. (+)-ESIMS spectrum for alkaloid 6.

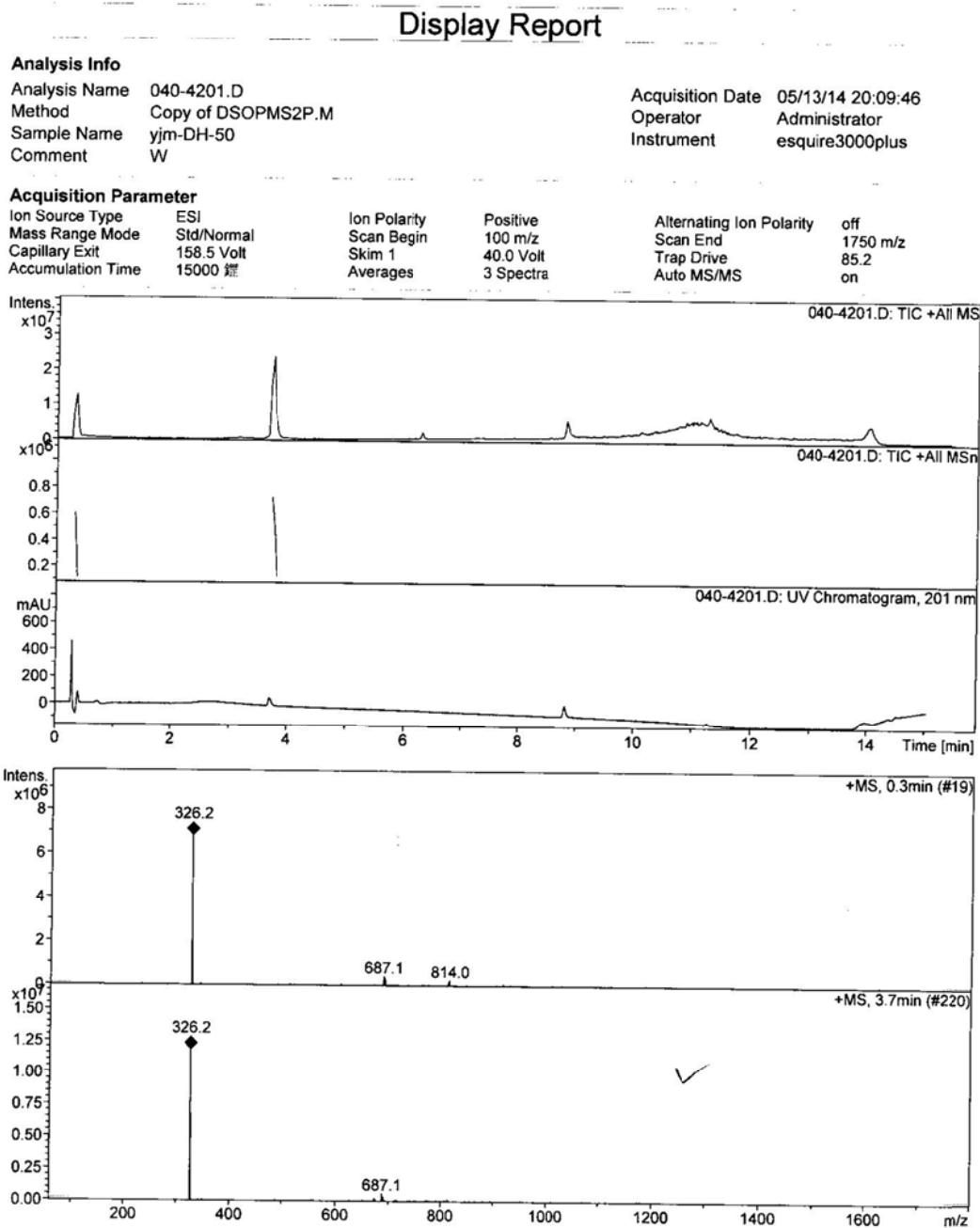


Figure S53. (+)-HRESIMS spectrum for alkaloid 6.

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

159 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-2 O: 0-20

DH-50

LCT PXE KE324

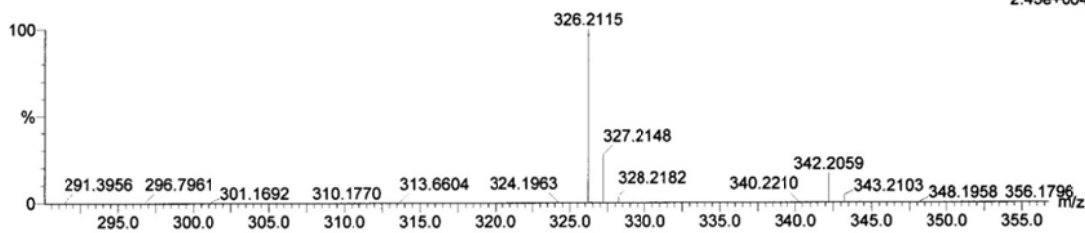
04-Jun-2014

15:34:52

1: TOF MS ES+

2.43e+04

DH-50\_0604 29 (0.636) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (25:41)



Minimum: -1.5  
Maximum: 5.0 5.0 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT | i-FIT (Norm) | Formula      |
|----------|------------|------|------|-----|-------|--------------|--------------|
| 326.2115 | 326.2120   | -0.5 | -1.5 | 8.5 | 156.9 | 0.0          | C21 H28 N O2 |

Figure S54.  $^1\text{H}$  NMR spectrum for alkaloid 7 in  $\text{CD}_3\text{OD}$ .

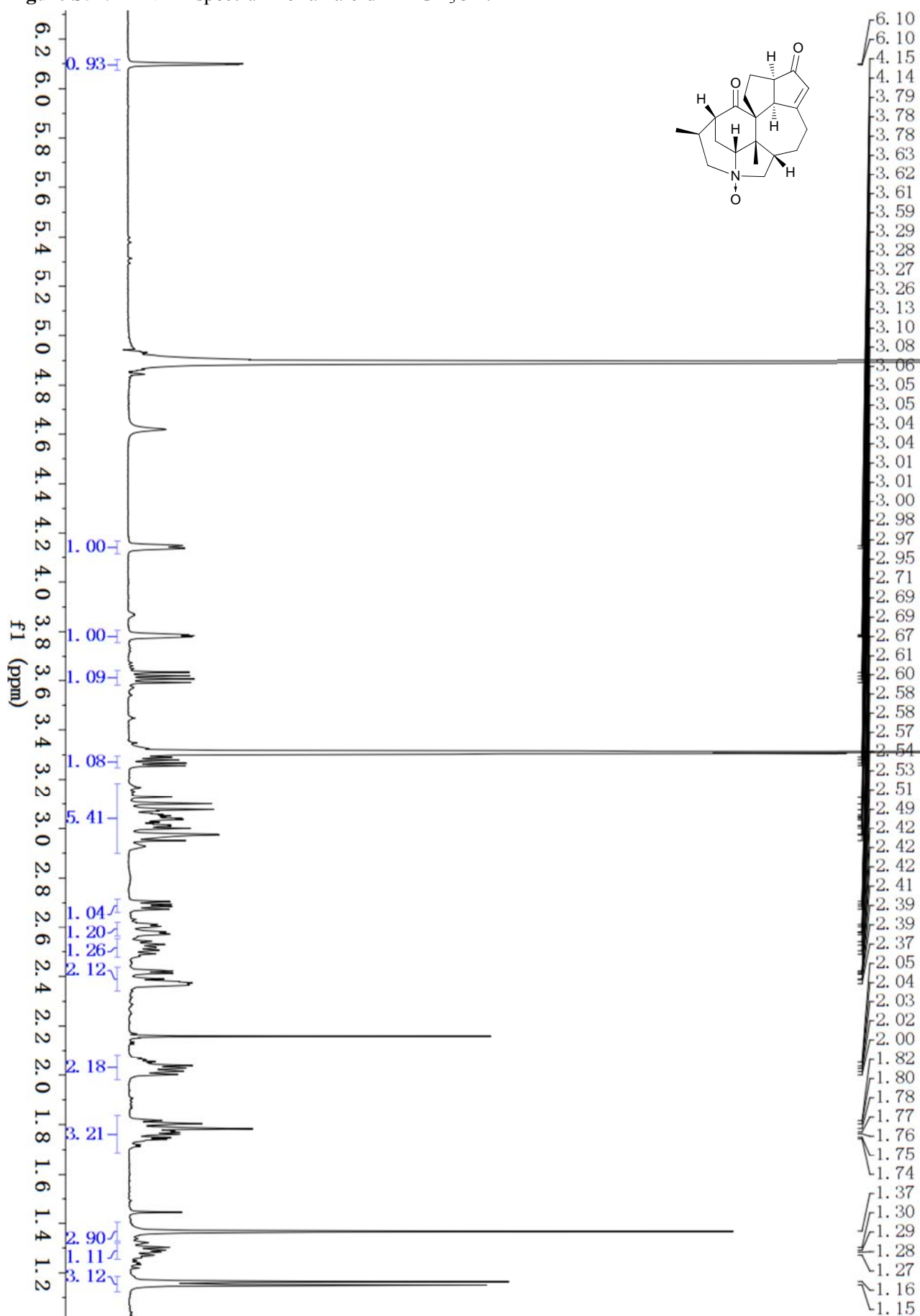


Figure S55.  $^{13}\text{C}$  NMR spectrum for alkaloid **7** in  $\text{CD}_3\text{OD}$ .

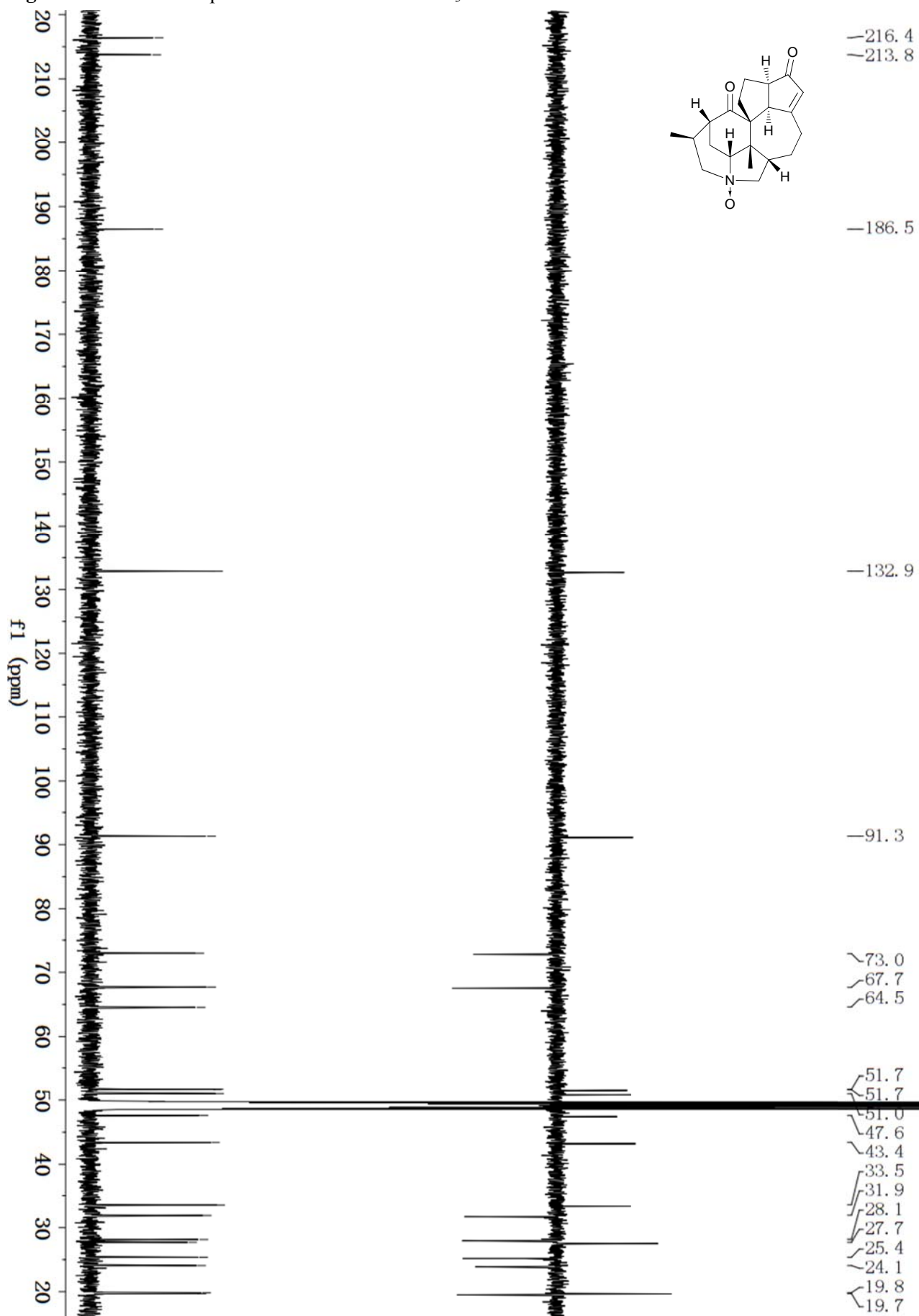




Figure S57 HSQC spectrum for alkaloid 7 in CD<sub>3</sub>OD.

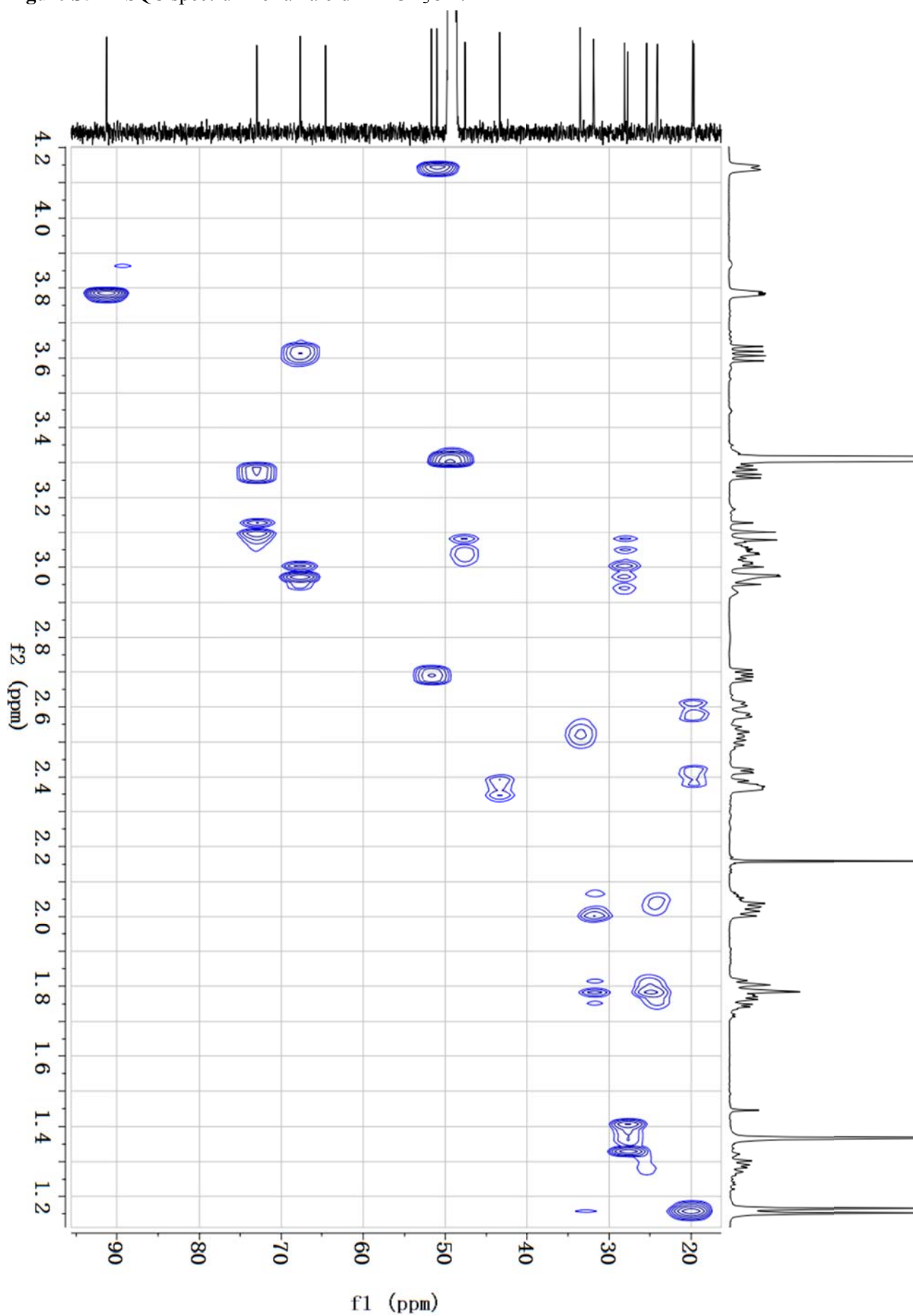




Figure S58. HMBC spectrum for alkaloid 7 in CD<sub>3</sub>OD.

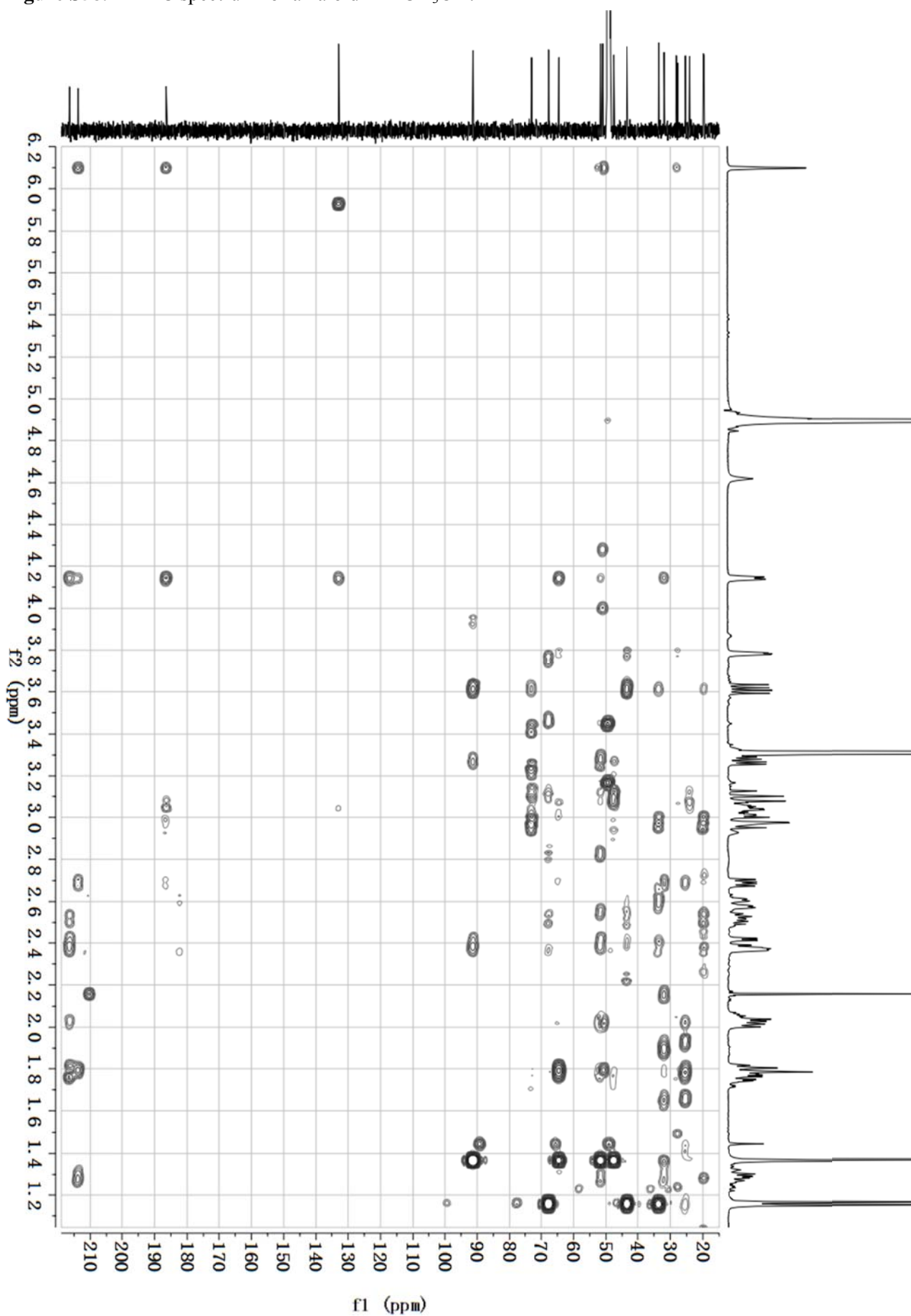


Figure S59. NOESY spectrum for alkaloid 7 in CD<sub>3</sub>OD.

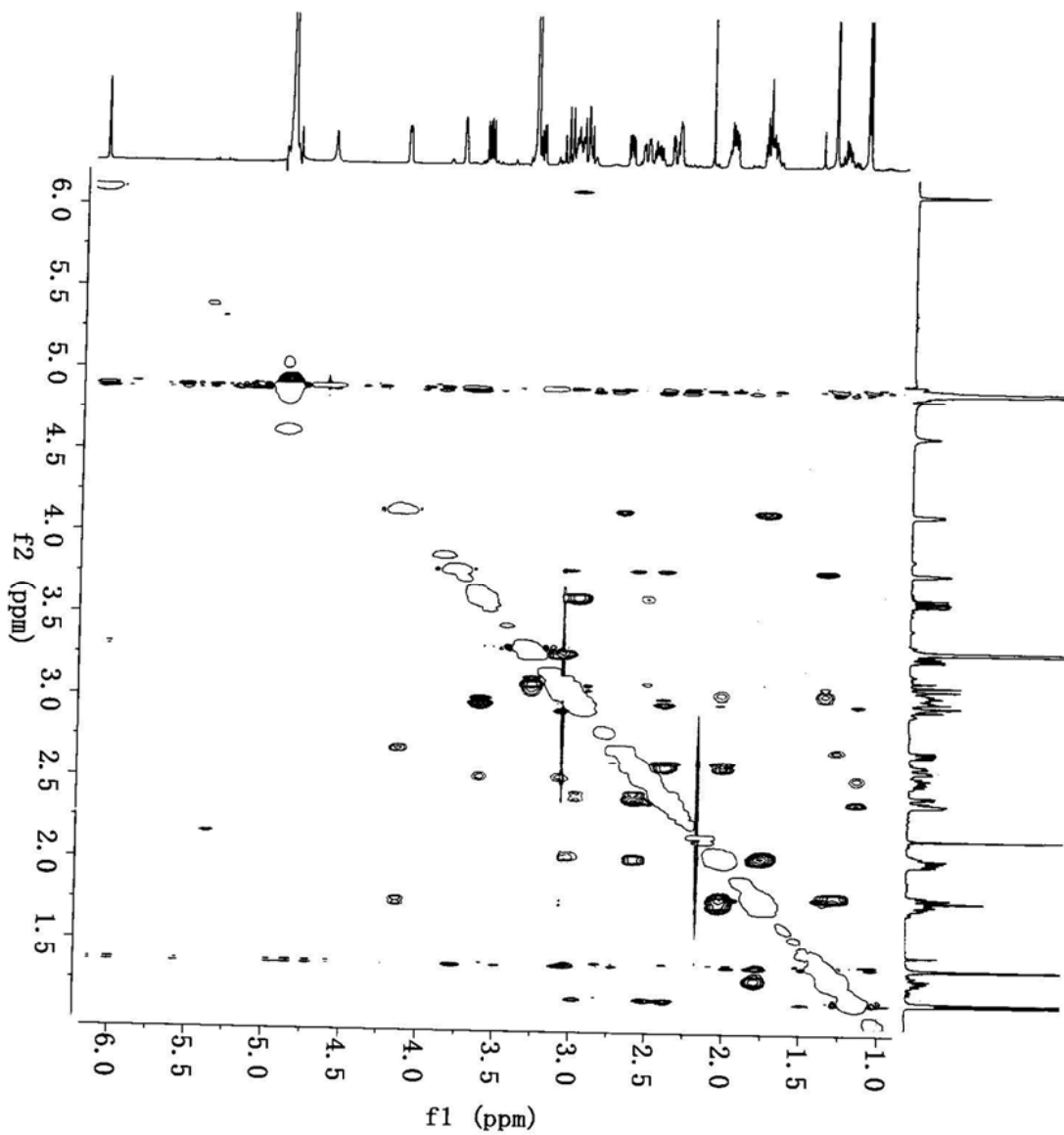


Figure S60. IR spectrum for alkaloid 7.

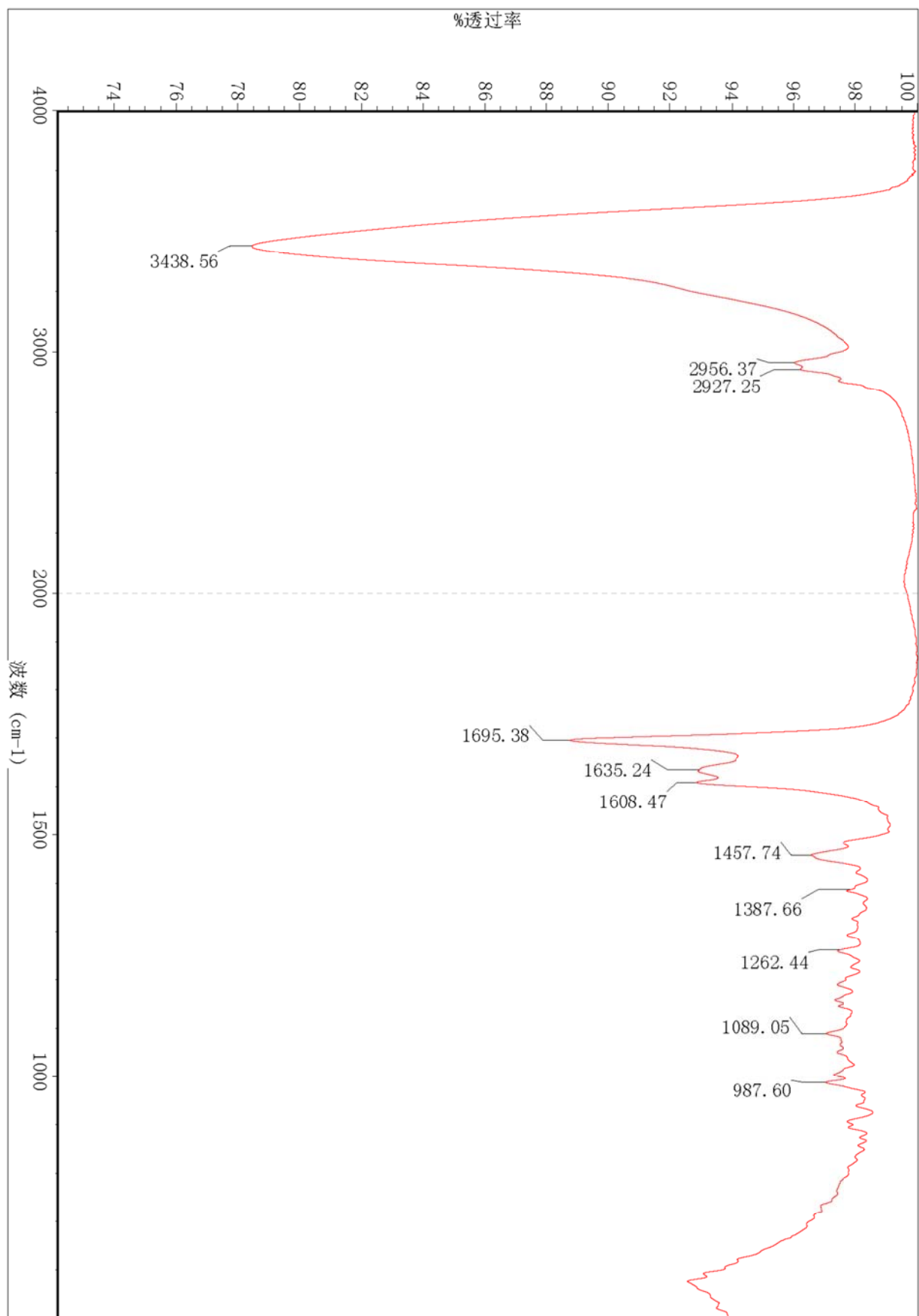
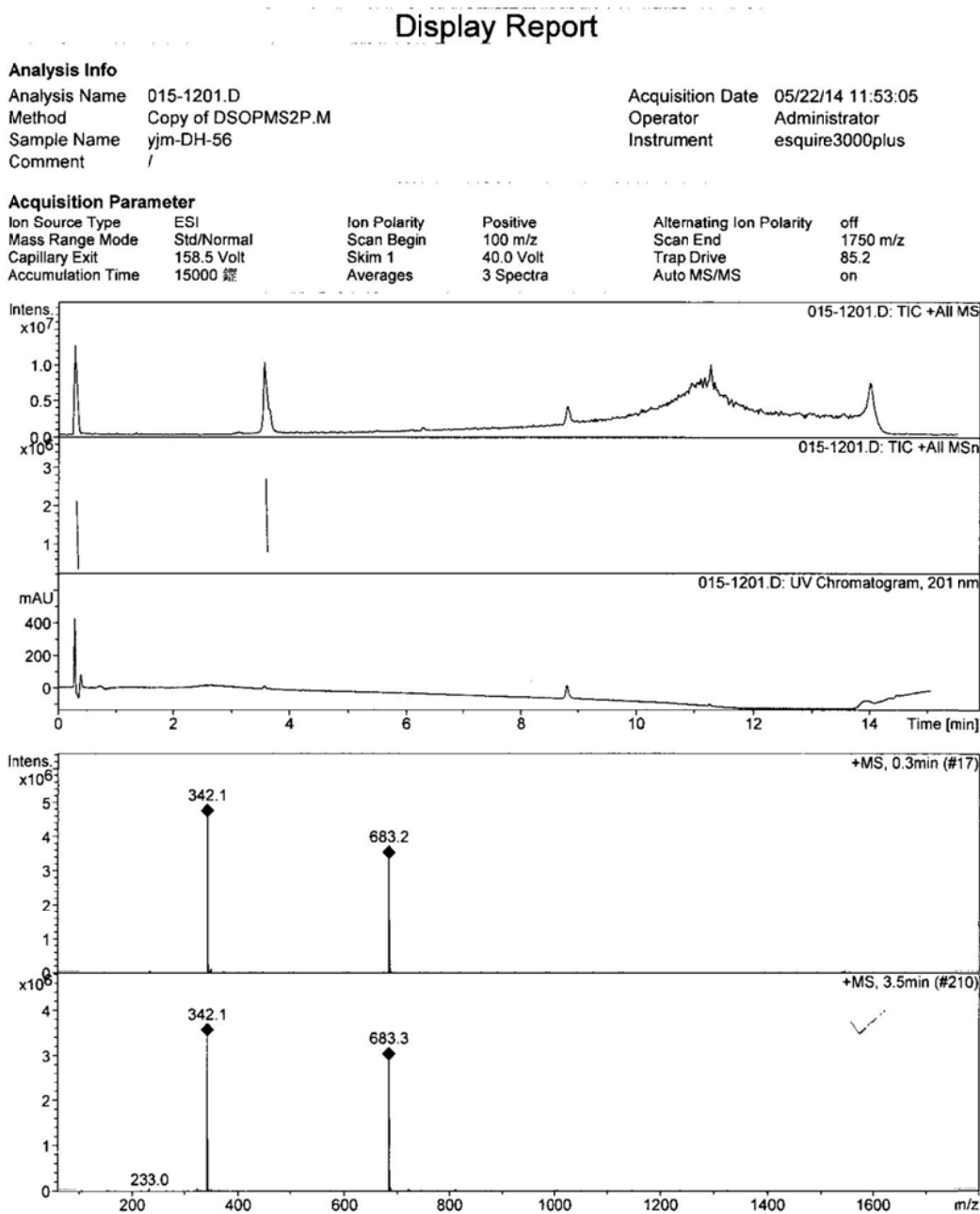


Figure S61. (+)-ESIMS spectrum for alkaloid 7.



**Figure S62.** (+)-HRESIMS spectrum for alkaloid 7.

**Elemental Composition Report**

**Single Mass Analysis**

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0  
 Element prediction: Off  
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions  
 173 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-2 O: 0-20

DH-56

LCT PXE KE324

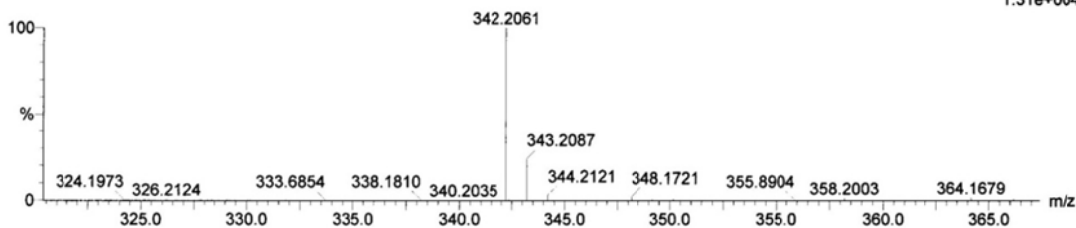
04-Jun-2014

15:17:07

DH-56\_0604 33 (0.724) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (29:44)

1: TOF MS ES+

1.31e+004



Minimum: -1.5  
 Maximum: 5.0 10.0 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT | i-FIT (Norm) | Formula      |
|----------|------------|------|------|-----|-------|--------------|--------------|
| 342.2061 | 342.2069   | -0.8 | -2.3 | 8.5 | 123.0 | 0.0          | C21 H28 N O3 |

Figure S63.  $^1\text{H}$  NMR spectrum for alkaloid **8** in  $\text{CDCl}_3$ .

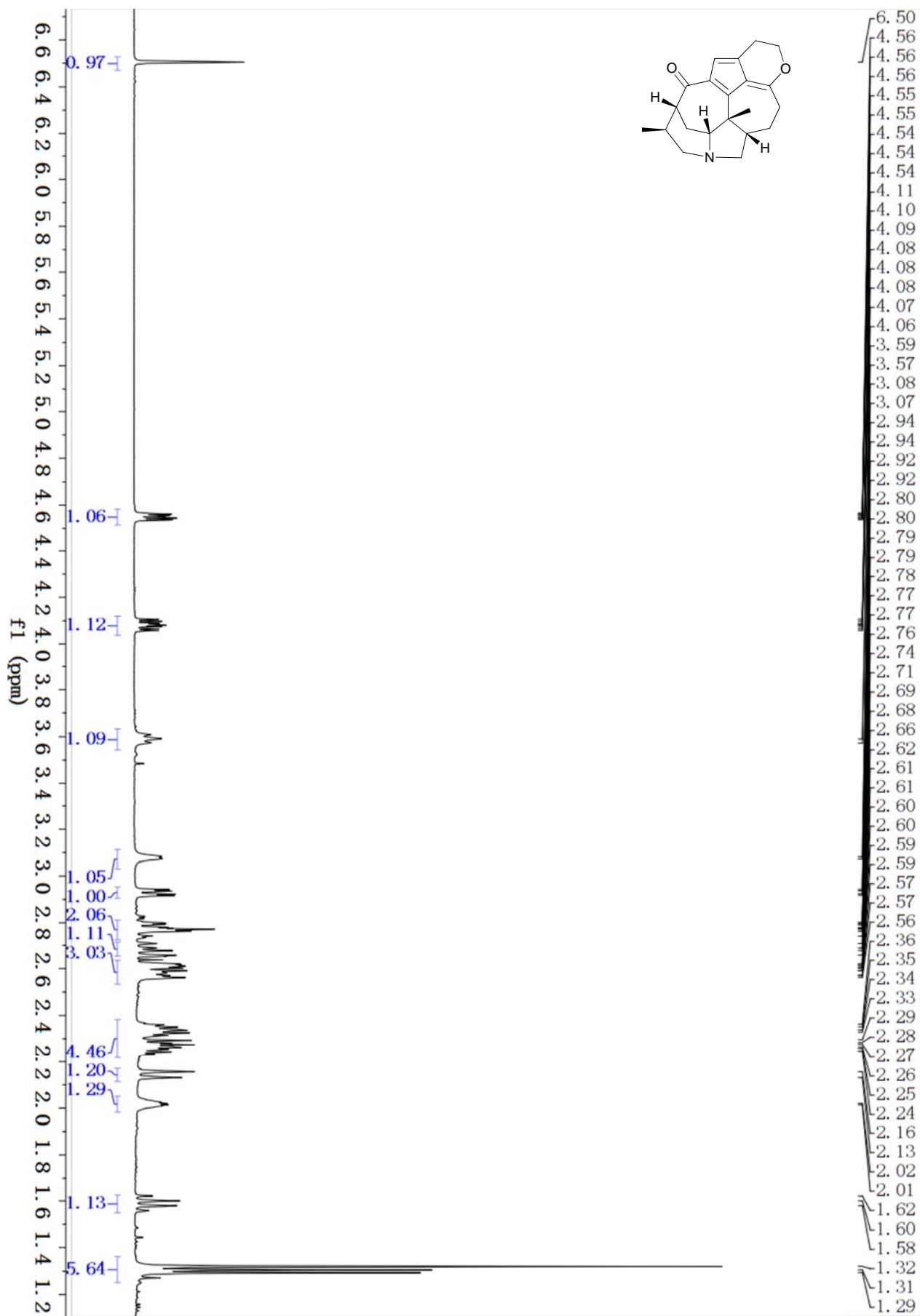


Figure S64.  $^{13}\text{C}$  NMR spectrum for alkaloid **8** in  $\text{CDCl}_3$ .

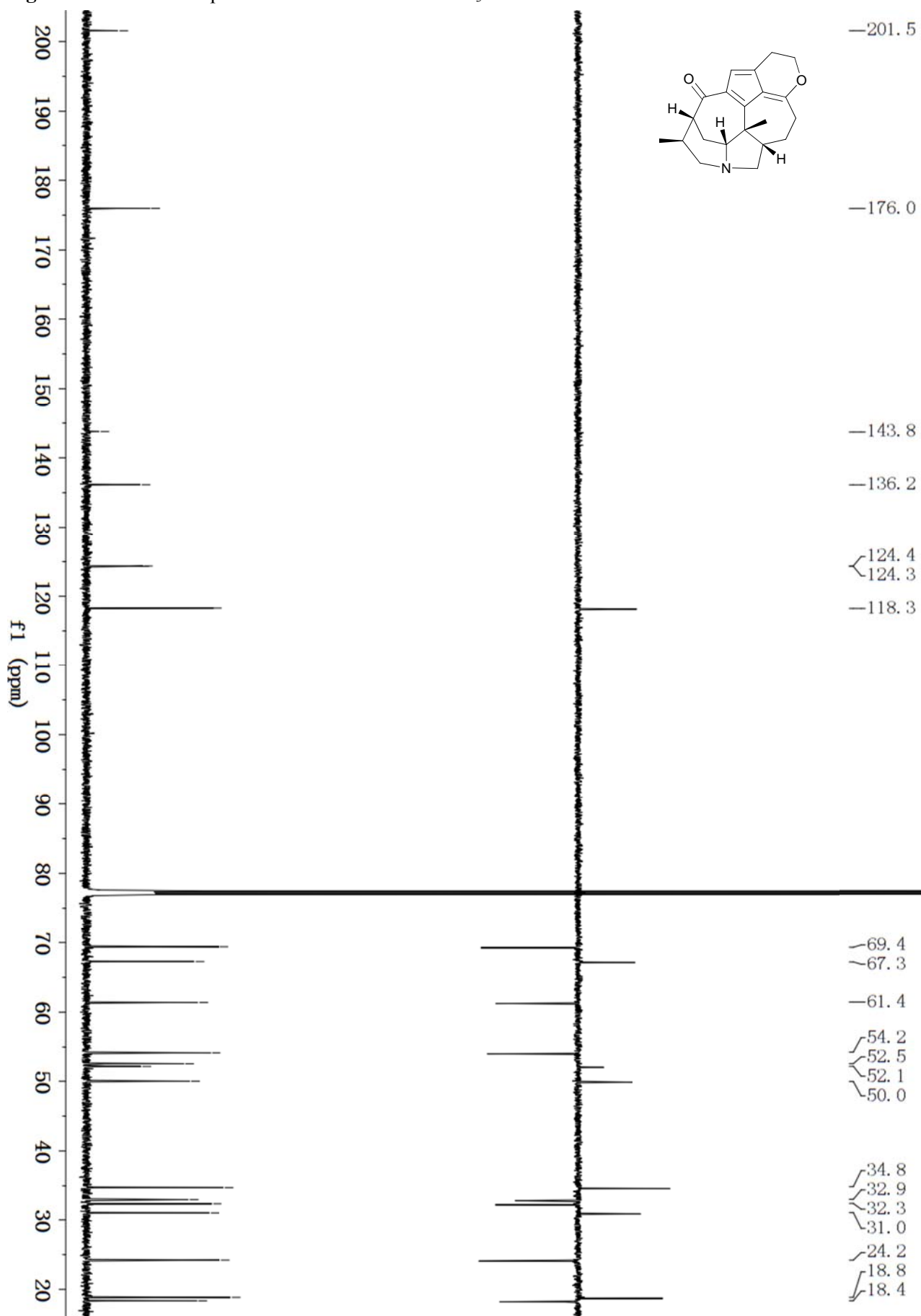


Figure S65.  $^1\text{H}$ - $^1\text{H}$  COSY spectrum for alkaloid **8** in  $\text{CDCl}_3$ .

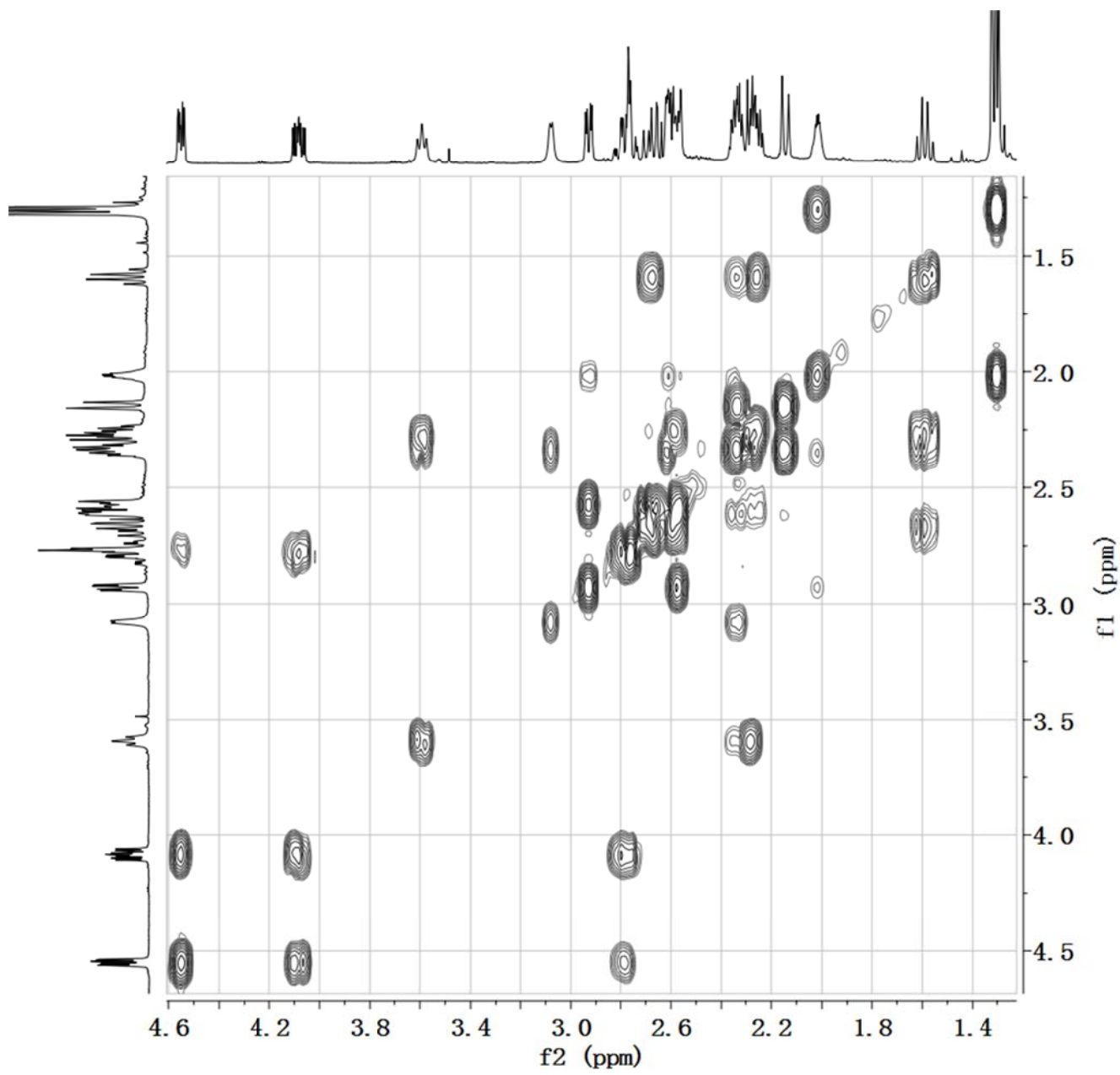




Figure S66 HSQC spectrum for alkaloid **8** in CDCl<sub>3</sub>.

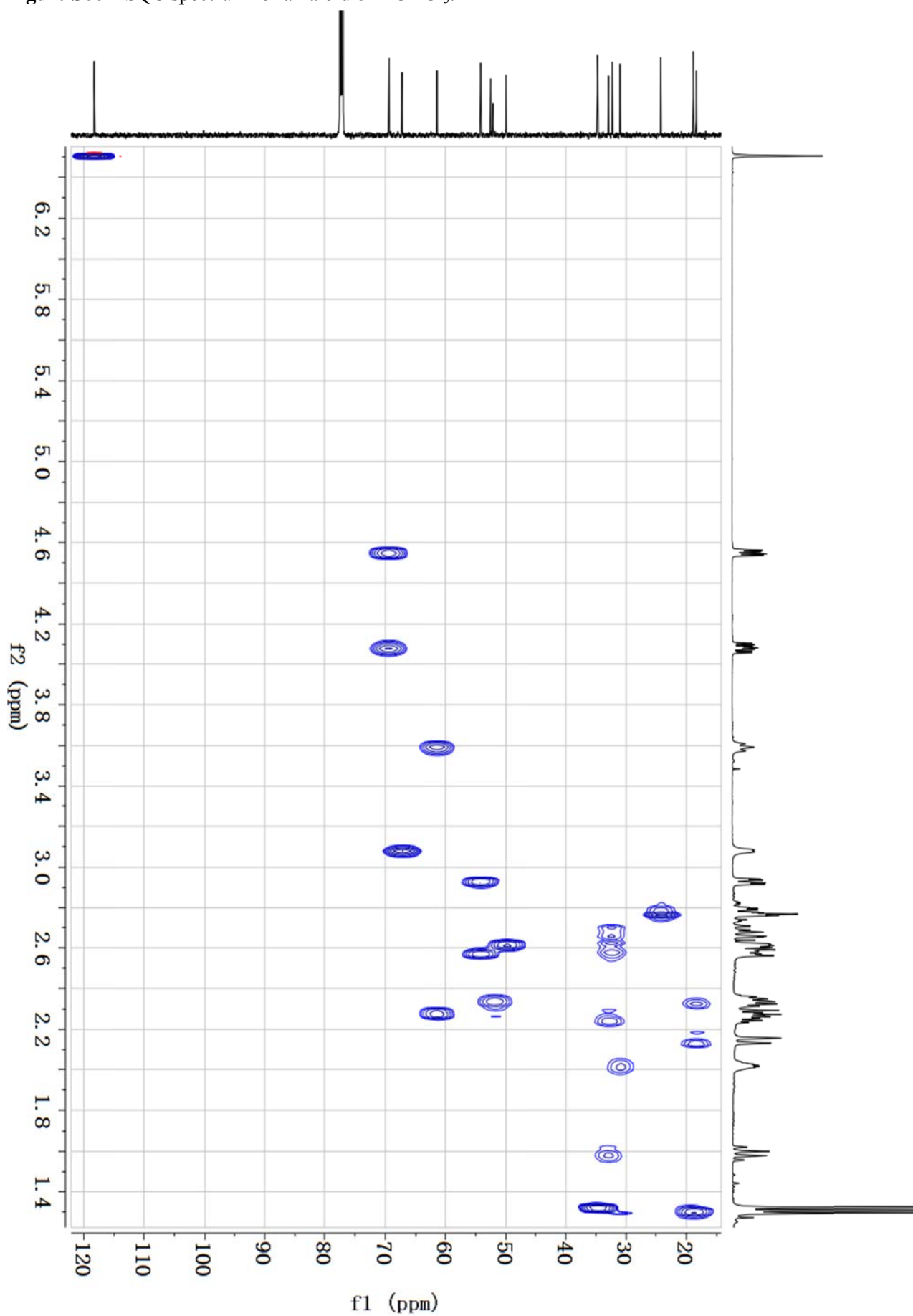


Figure S67. HMBC spectrum for alkaloid 8 in CDCl<sub>3</sub>.

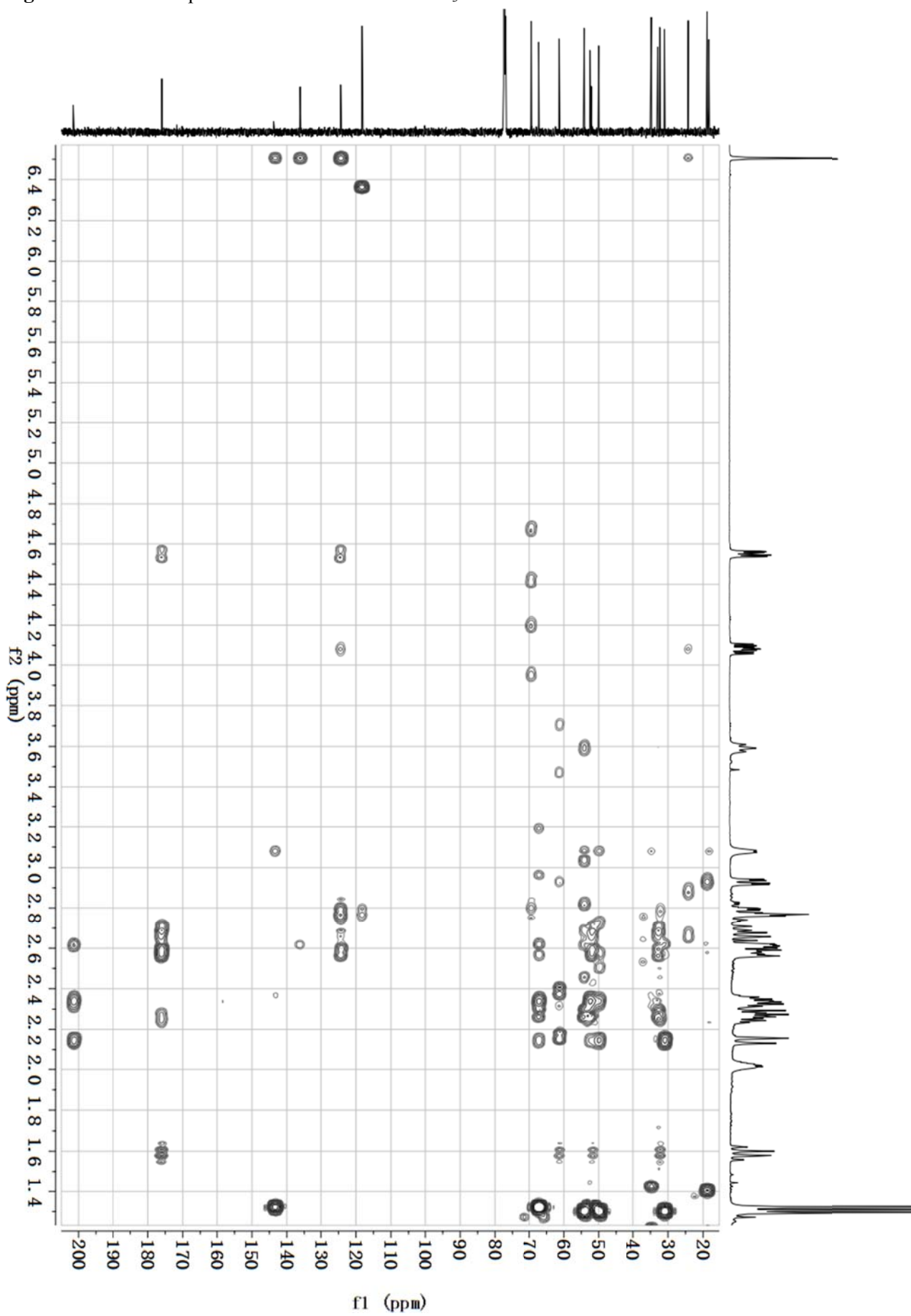


Figure S68. NOESY spectrum for alkaloid **8** in CDCl<sub>3</sub>.

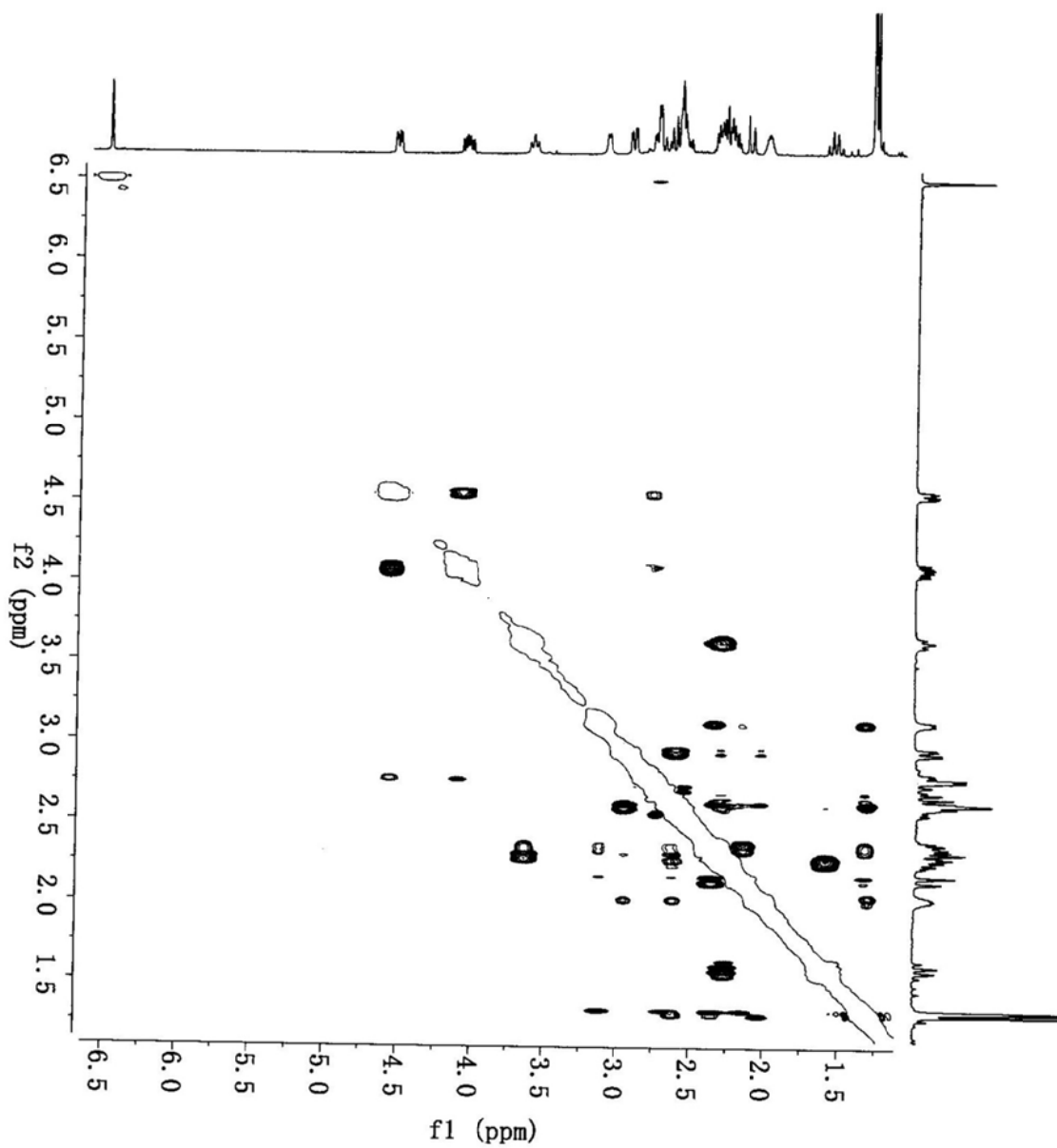


Figure S69. IR spectrum for alkaloid 8.

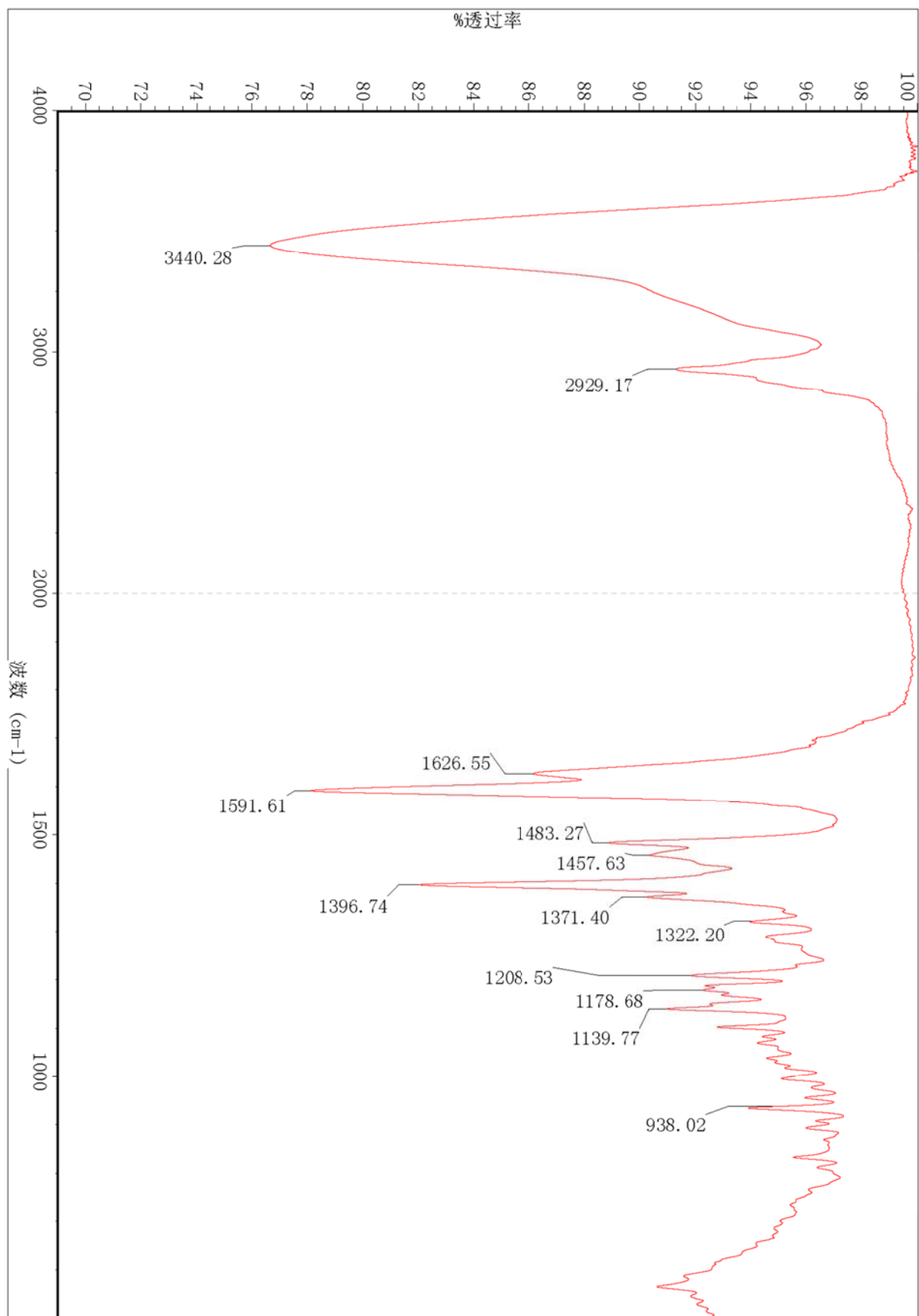


Figure S70. (+)-ESIMS spectrum for alkaloid 8.

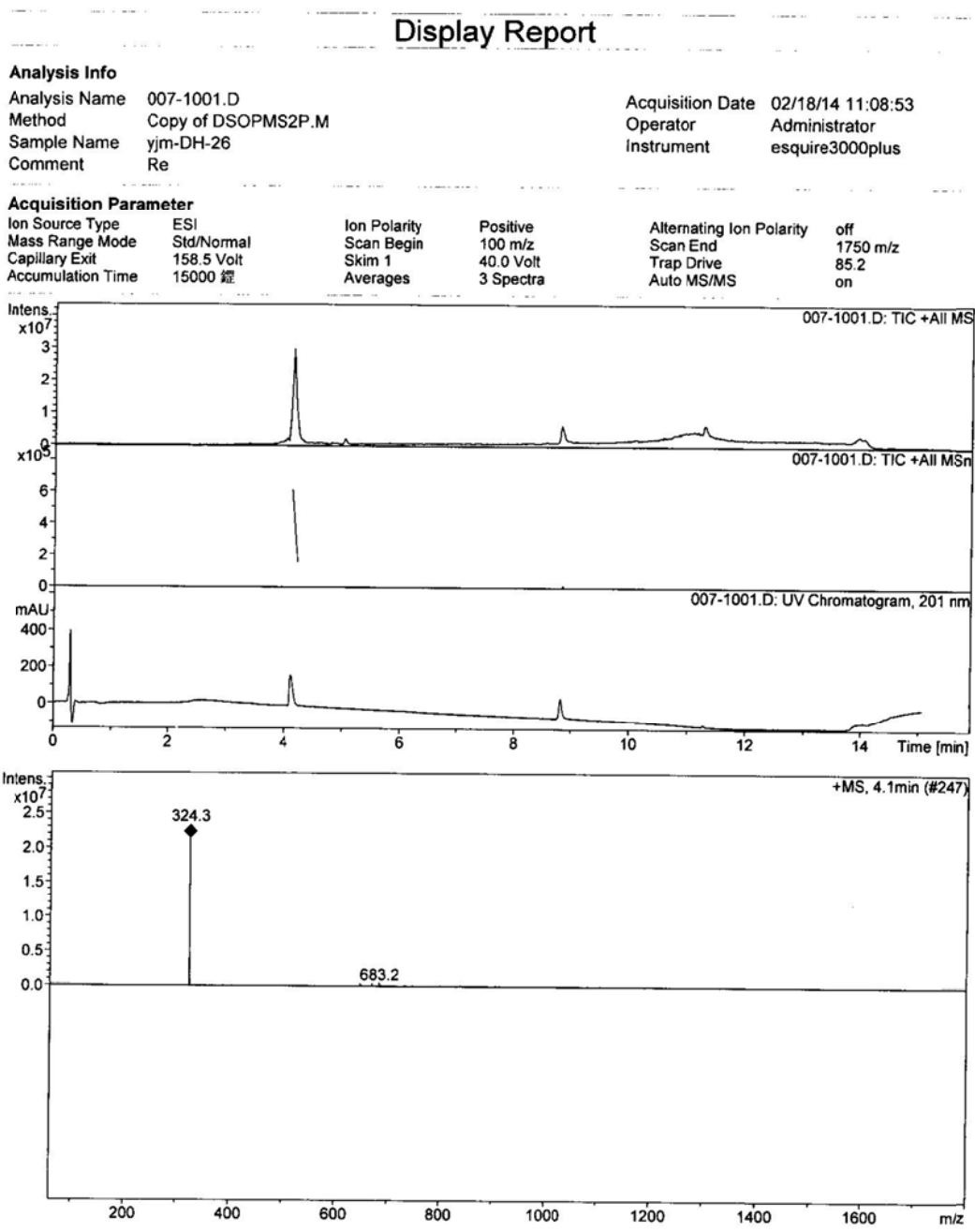


Figure S71 (+)-HRESIMS spectrum for alkaloid 8.

Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

156 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-2 O: 0-20

DH-26

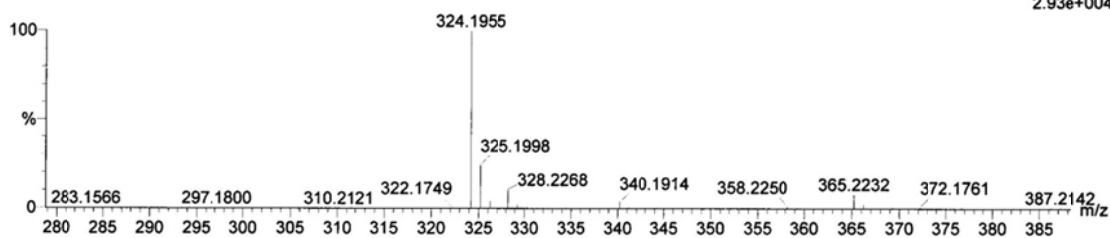
LCT PXE KE324

04-Jun-2014

14:55:28

DH-26\_0604 34 (0.726) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (33:48)

1: TOF MS ES+  
2.93e+004



Minimum: -1.5  
Maximum: 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT | i-FIT (Norm) | Formula      |
|----------|------------|------|------|-----|-------|--------------|--------------|
| 324.1955 | 324.1964   | -0.9 | -2.8 | 9.5 | 196.4 | 0.0          | C21 H26 N O2 |

**Figure S72.**  $^1\text{H}$  NMR spectrum for alkaloid **9** in  $\text{CDCl}_3$ .

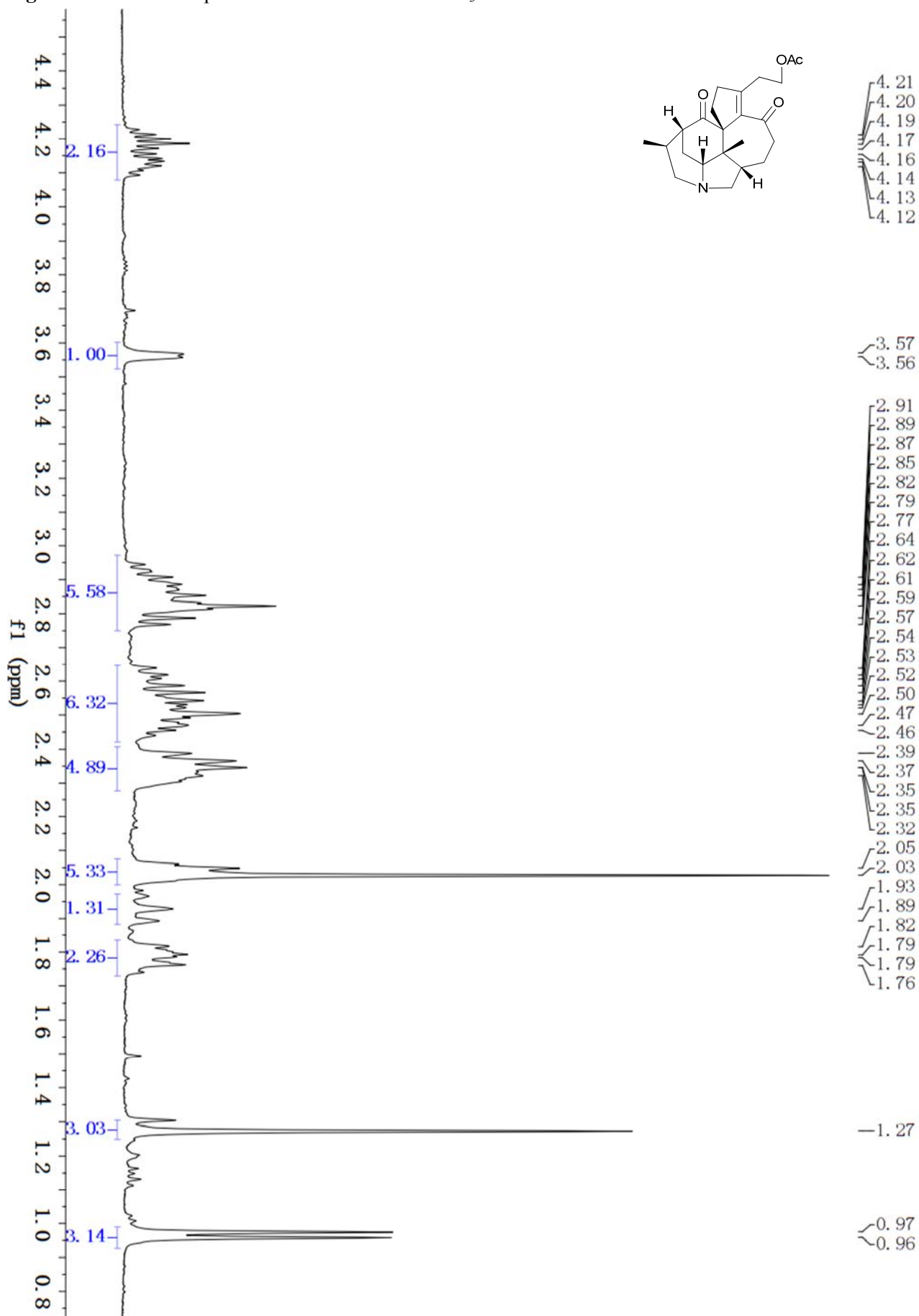


Figure S73.  $^{13}\text{C}$  NMR spectrum for alkaloid **9** in  $\text{CDCl}_3$ .

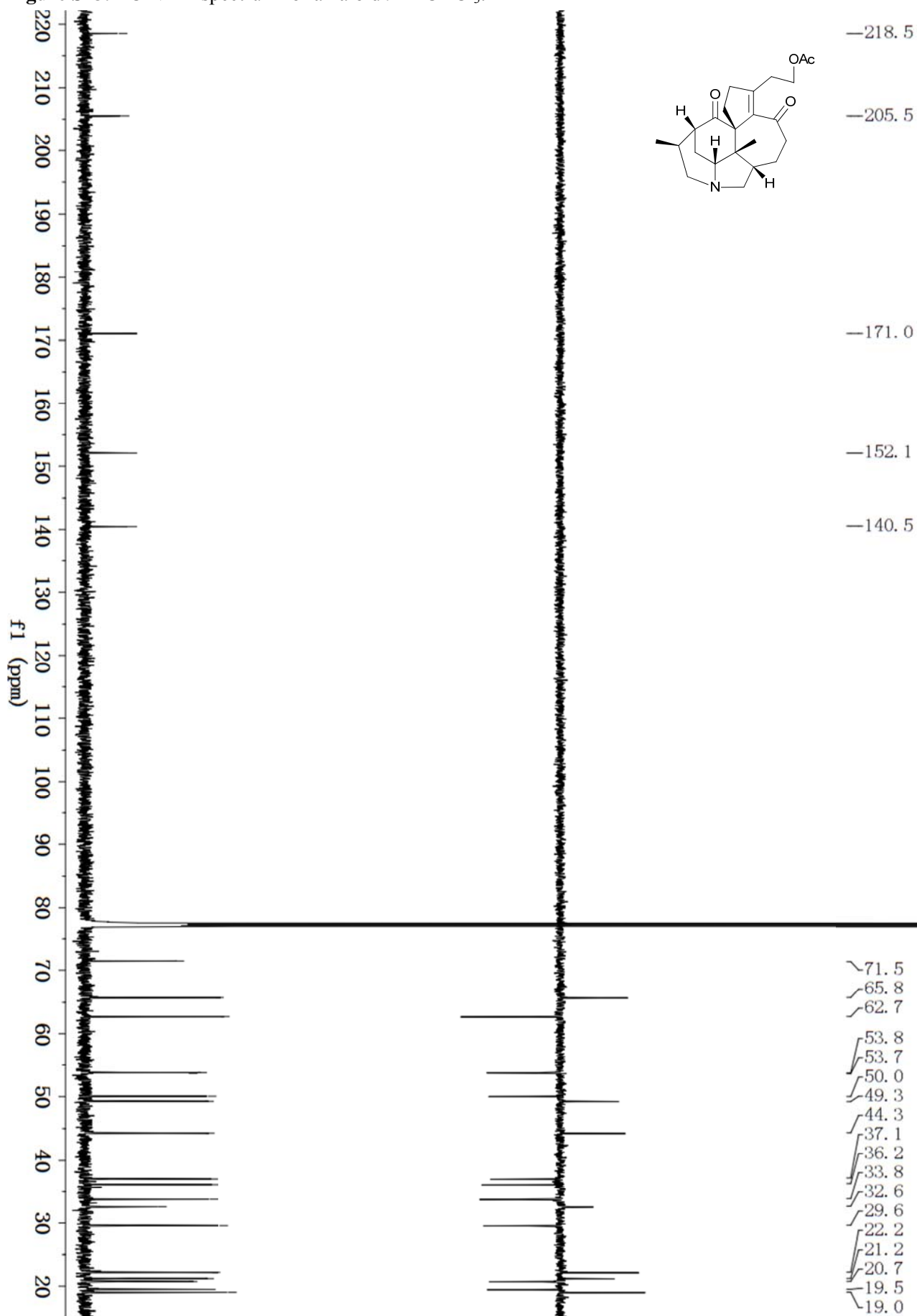






Figure S75. HMBC spectrum for alkaloid **9** in CDCl<sub>3</sub>.

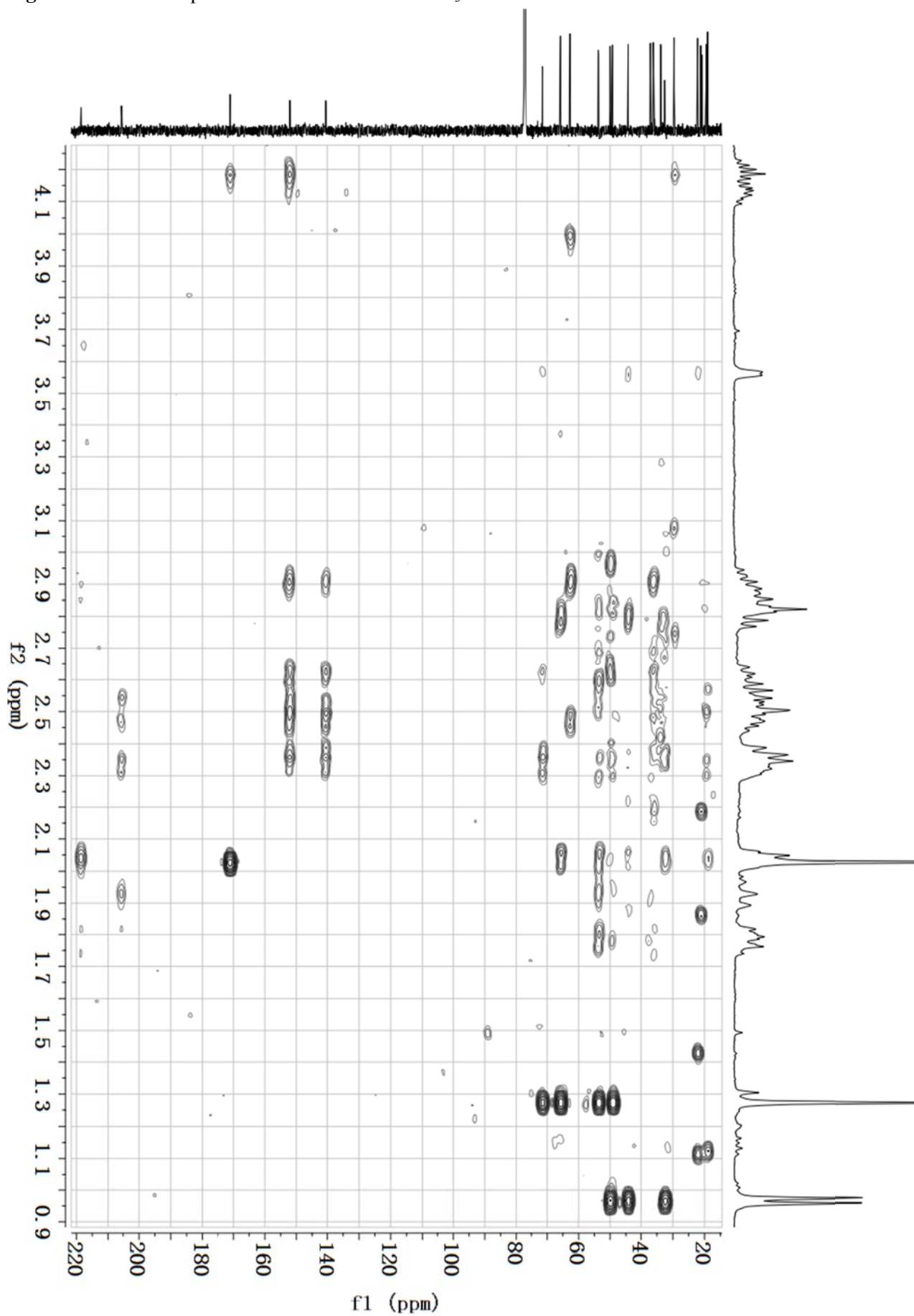


Figure S76. NOESY spectrum for alkaloid 9 in CDCl<sub>3</sub>.

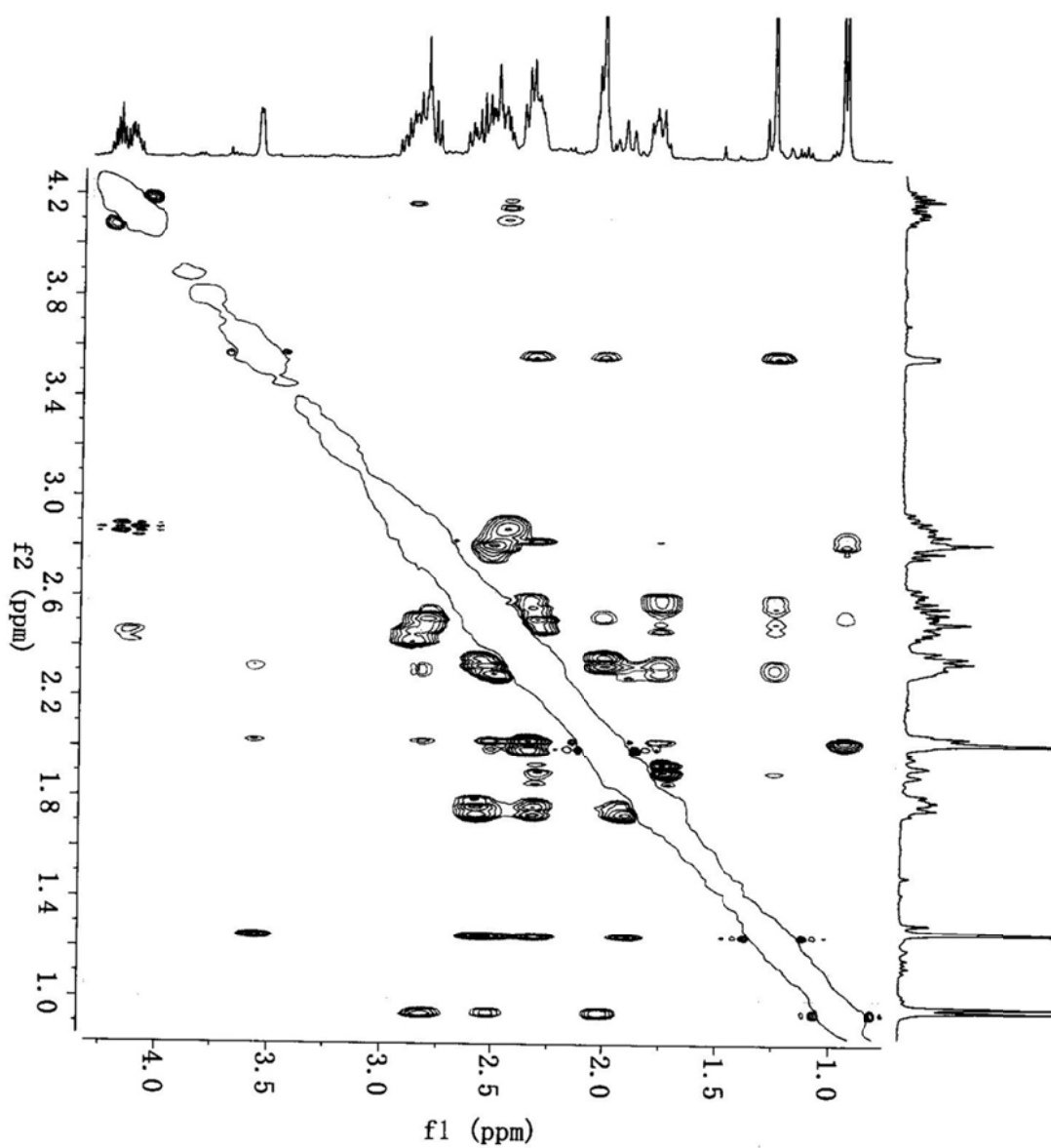


Figure S77. IR spectrum for alkaloid 9.

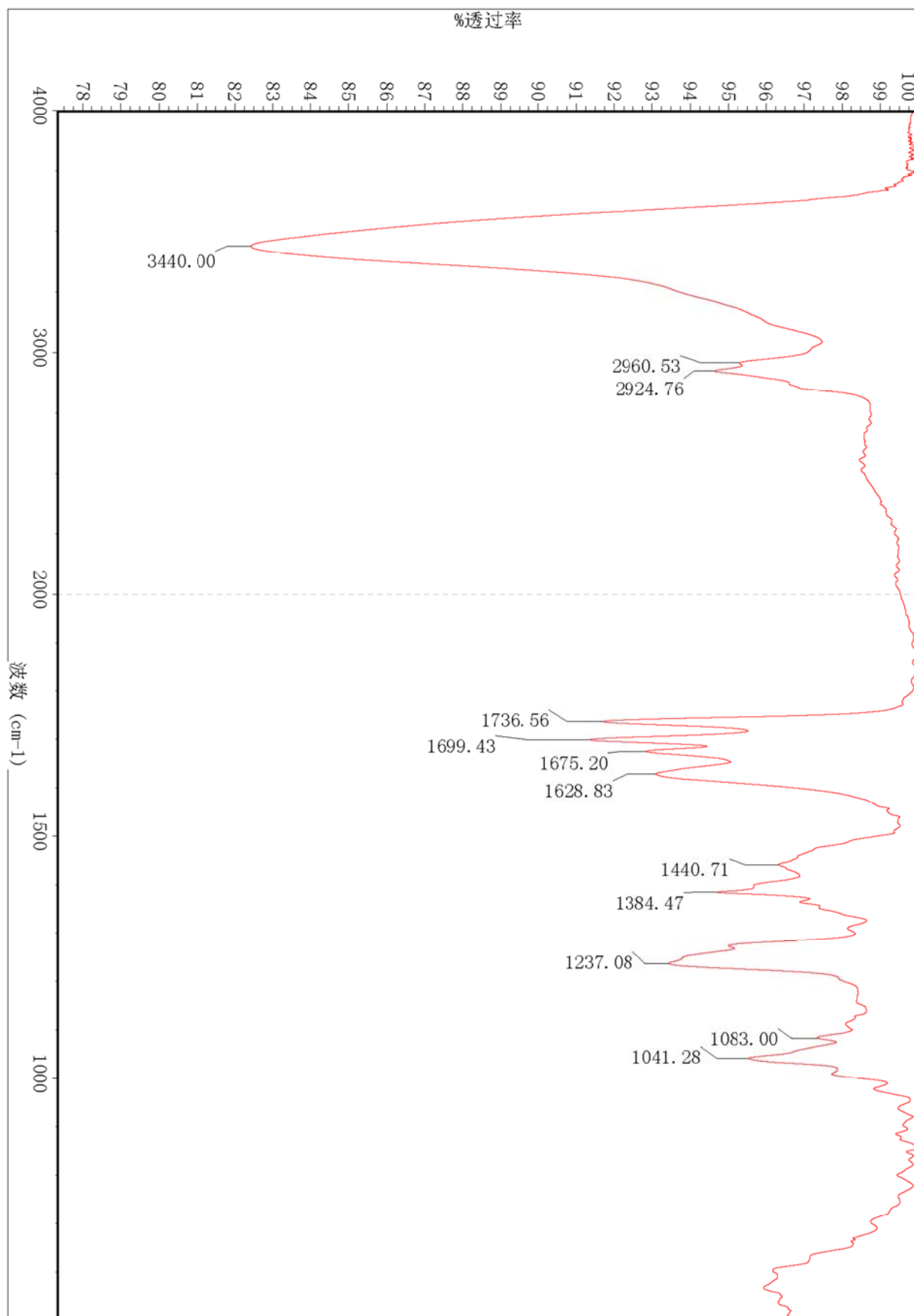


Figure S78. (+)-ESIMS spectrum for alkaloid 9.

### Display Report

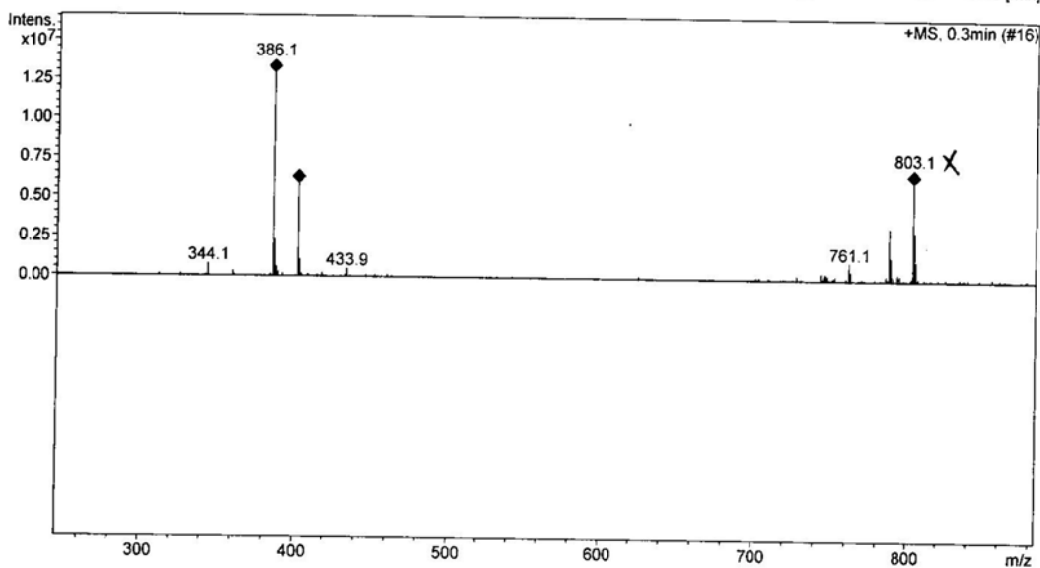
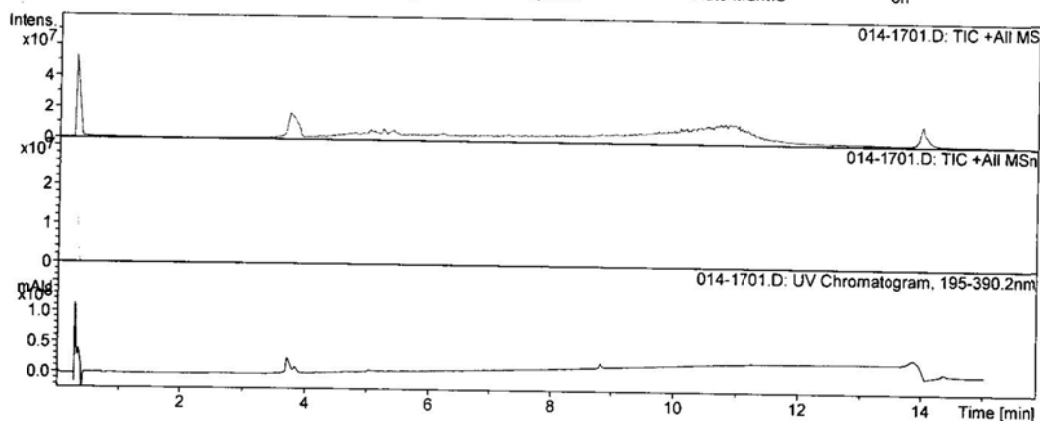
#### Analysis Info

Analysis Name 014-1701.D  
Method Copy of DSOPMS2P.M  
Sample Name yjm-DH-43  
Comment

Acquisition Date 06/21/15 22:52:45  
Operator Administrator  
Instrument esquire3000plus

#### Acquisition Parameter

|                   |            |              |           |                          |          |
|-------------------|------------|--------------|-----------|--------------------------|----------|
| Ion Source Type   | ESI        | Ion Polarity | Positive  | Alternating Ion Polarity | off      |
| Mass Range Mode   | Std/Normal | Scan Begin   | 100 m/z   | Scan End                 | 1750 m/z |
| Capillary Exit    | 158.5 Volt | Skim 1       | 40.0 Volt | Trap Drive               | 85.2     |
| Accumulation Time | 15000 罫    | Averages     | 3 Spectra | Auto MS/MS               | on       |



**Figure S79.** (+)-HRESIMS spectrum for alkaloid **9**.

**Elemental Composition Report**

**Single Mass Analysis**

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

207 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 N: 0-2 O: 0-20

DH-43

LCT PXE KE324

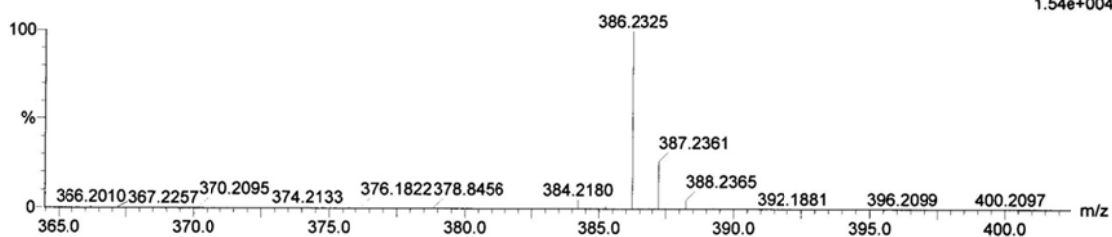
04-Jun-2014

15:28:50

1: TOF MS ES+

1.54e+004

DH-43\_0604 29 (0.636) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (24:39)



Minimum: -1.5  
Maximum: 5.0 5.0 50.0

| Mass     | Calc. Mass | mDa  | PPM  | DBE | i-FIT | i-FIT (Norm) | Formula      |
|----------|------------|------|------|-----|-------|--------------|--------------|
| 386.2325 | 386.2331   | -0.6 | -1.6 | 8.5 | 108.5 | 0.0          | C23 H32 N O4 |