# Sesquiterpenes and Monoterpenes from the Leaves and Stems of *Illicium simonsii* and their Antibacterial Activity

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[3] School of Pharmaceutical Science, Zhengzhou University, Ke Xue Da Dao 100, Zhengzhou 450001, P.R. China; liujf2009y@126.com
[4] Eastern China Conservation Centre for Wild Endangered Plant Resources, Shanghai Chenshan Botanical Garden, Shanghai 201602, P.R. China
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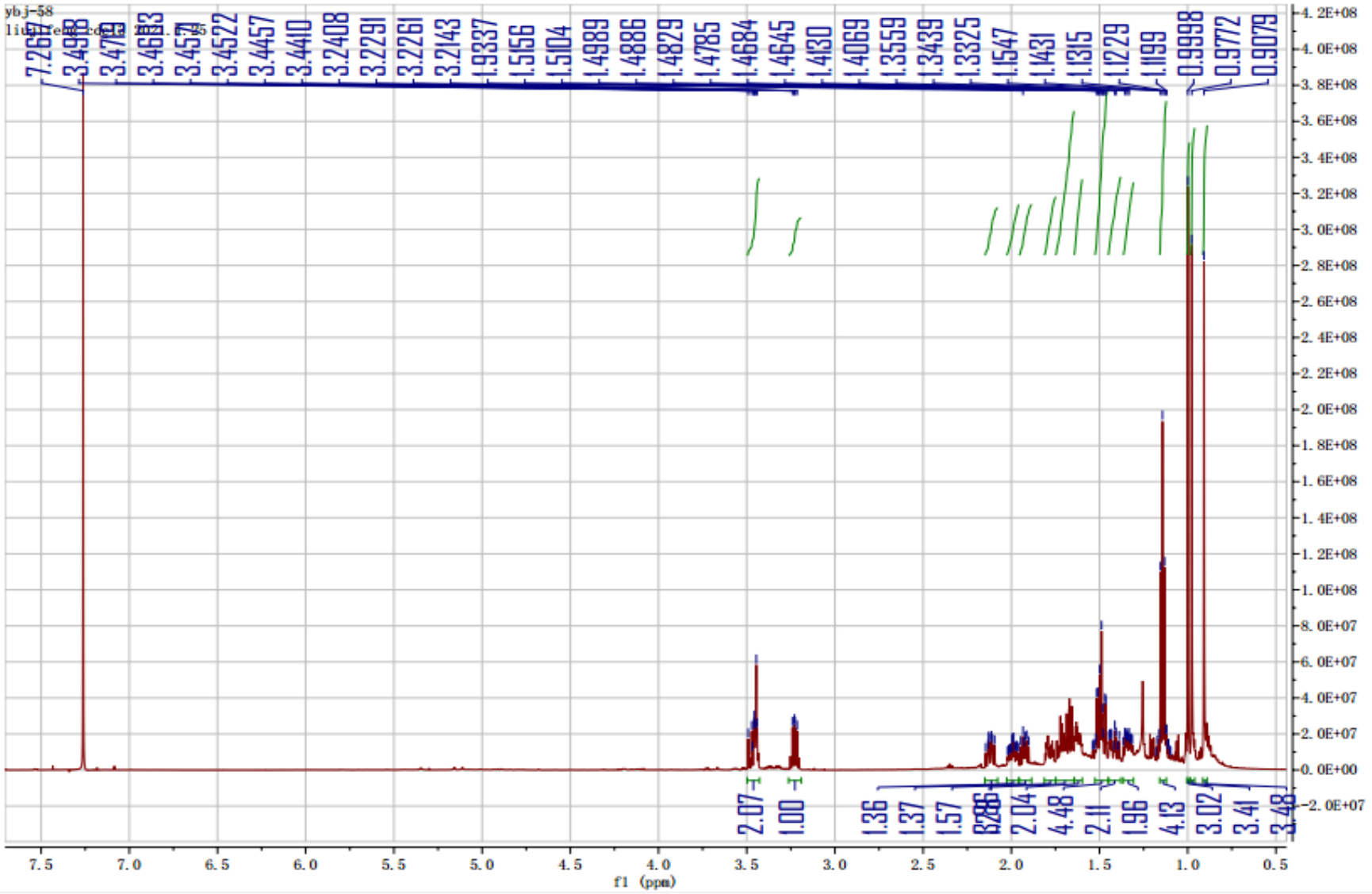
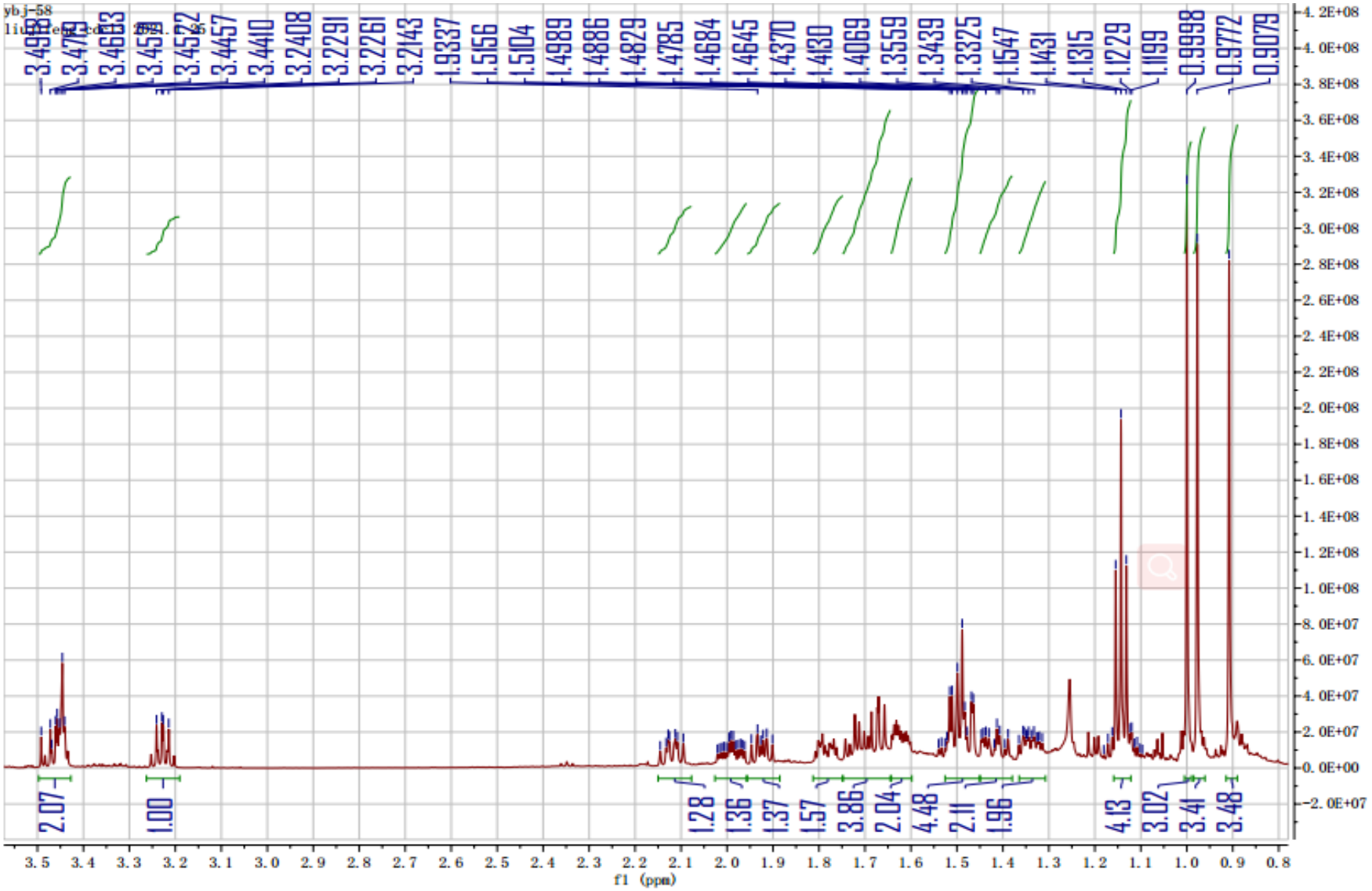
## Supporting information

## Content

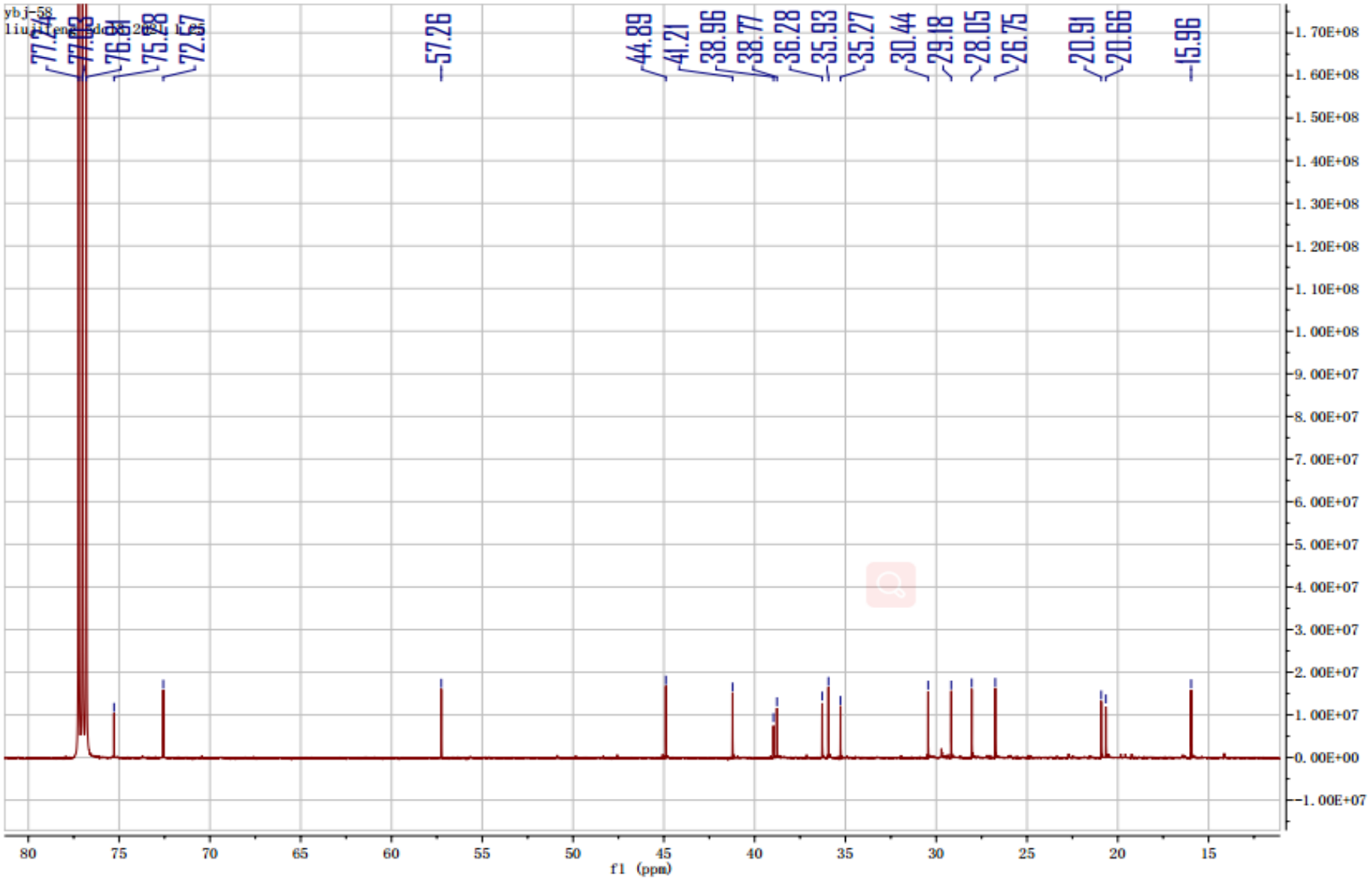
The $^{1}$H NMR spectrum of compound **1**



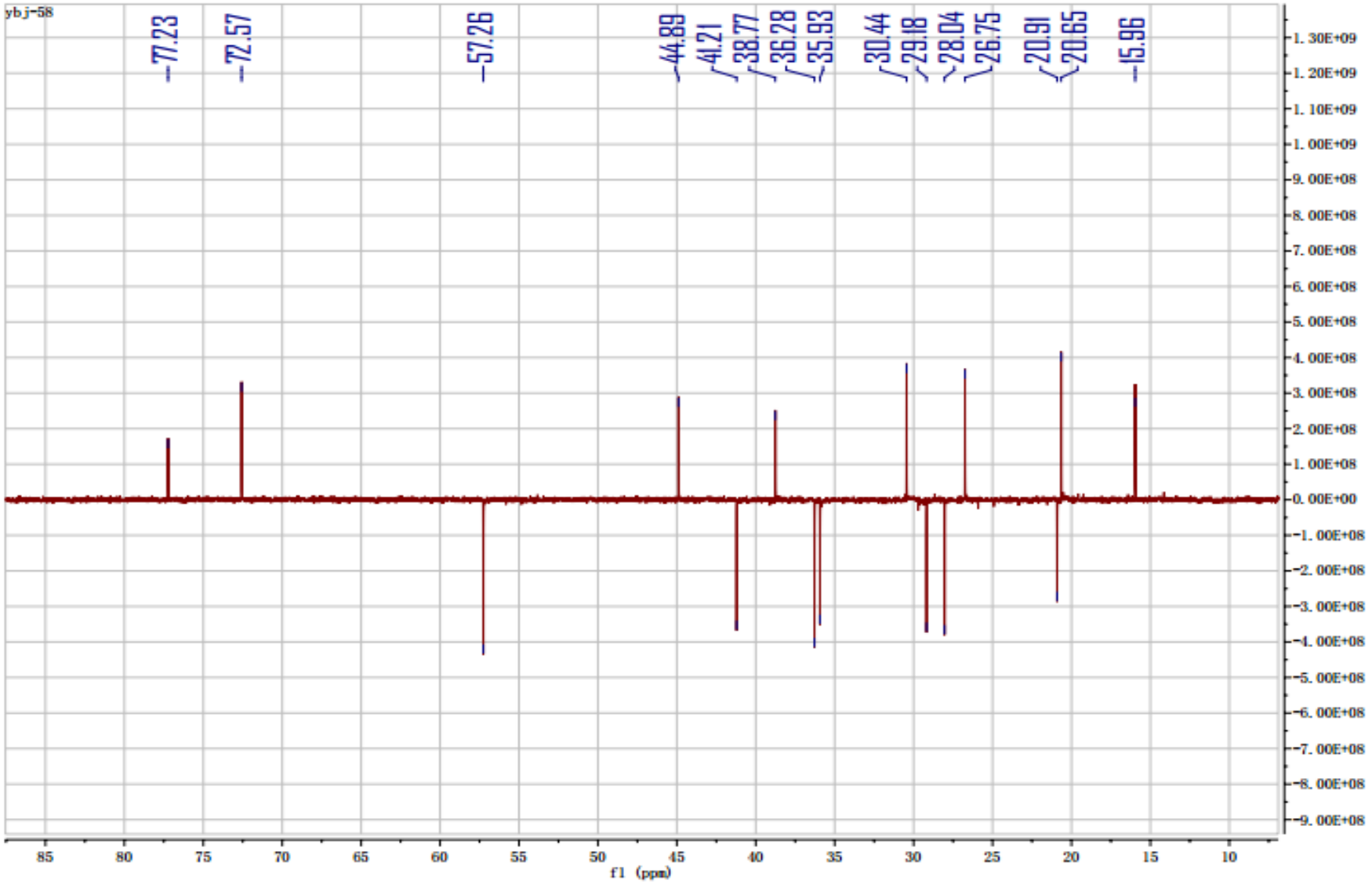
The $^{1}$H NMR spectrum of compound **1**



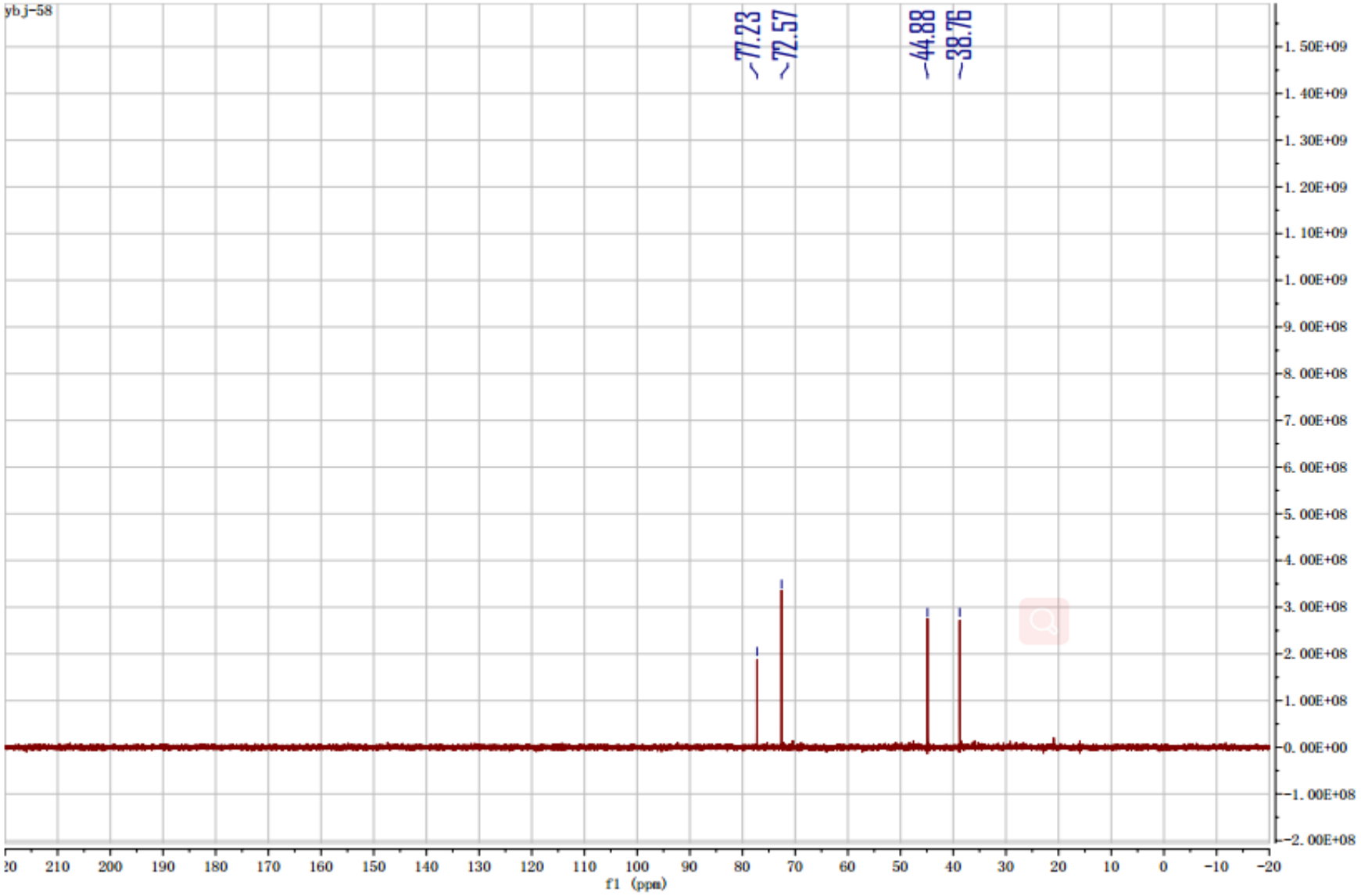
The $^{13}C$ NMR of compound **1**



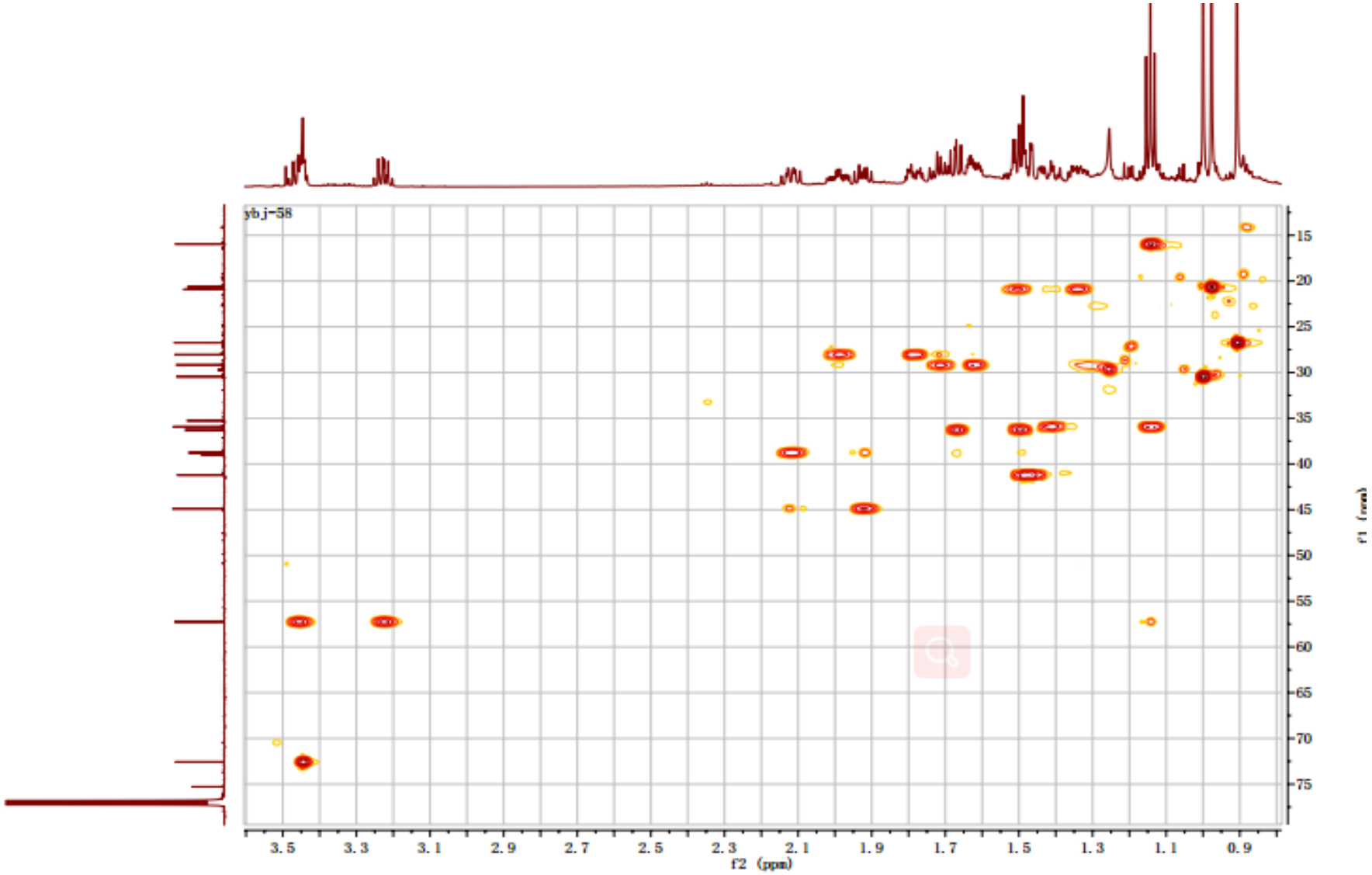
The DEPT-135 spectrum of compound **1**



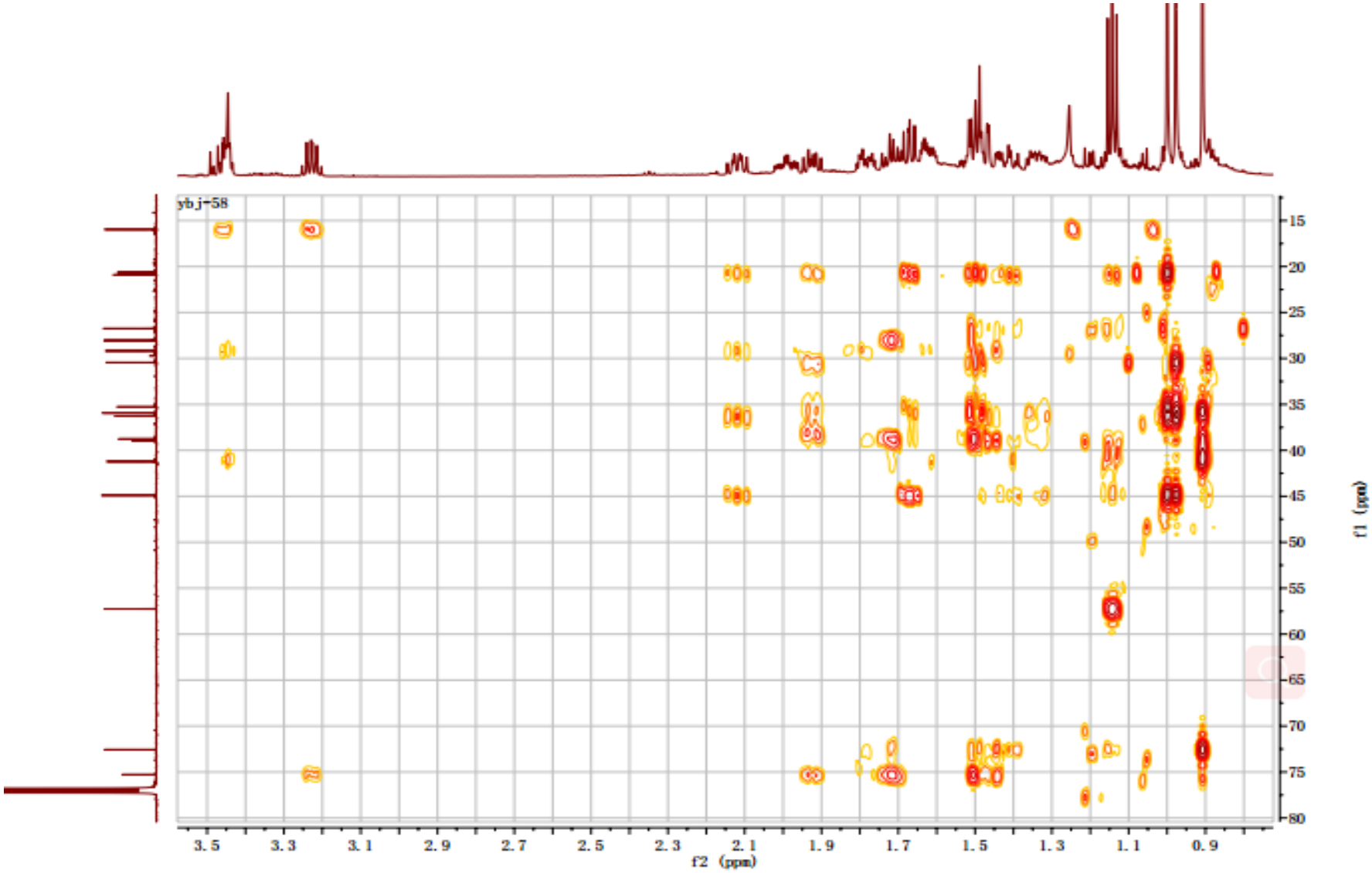
The DEPT-90 spectrum of compound **1**



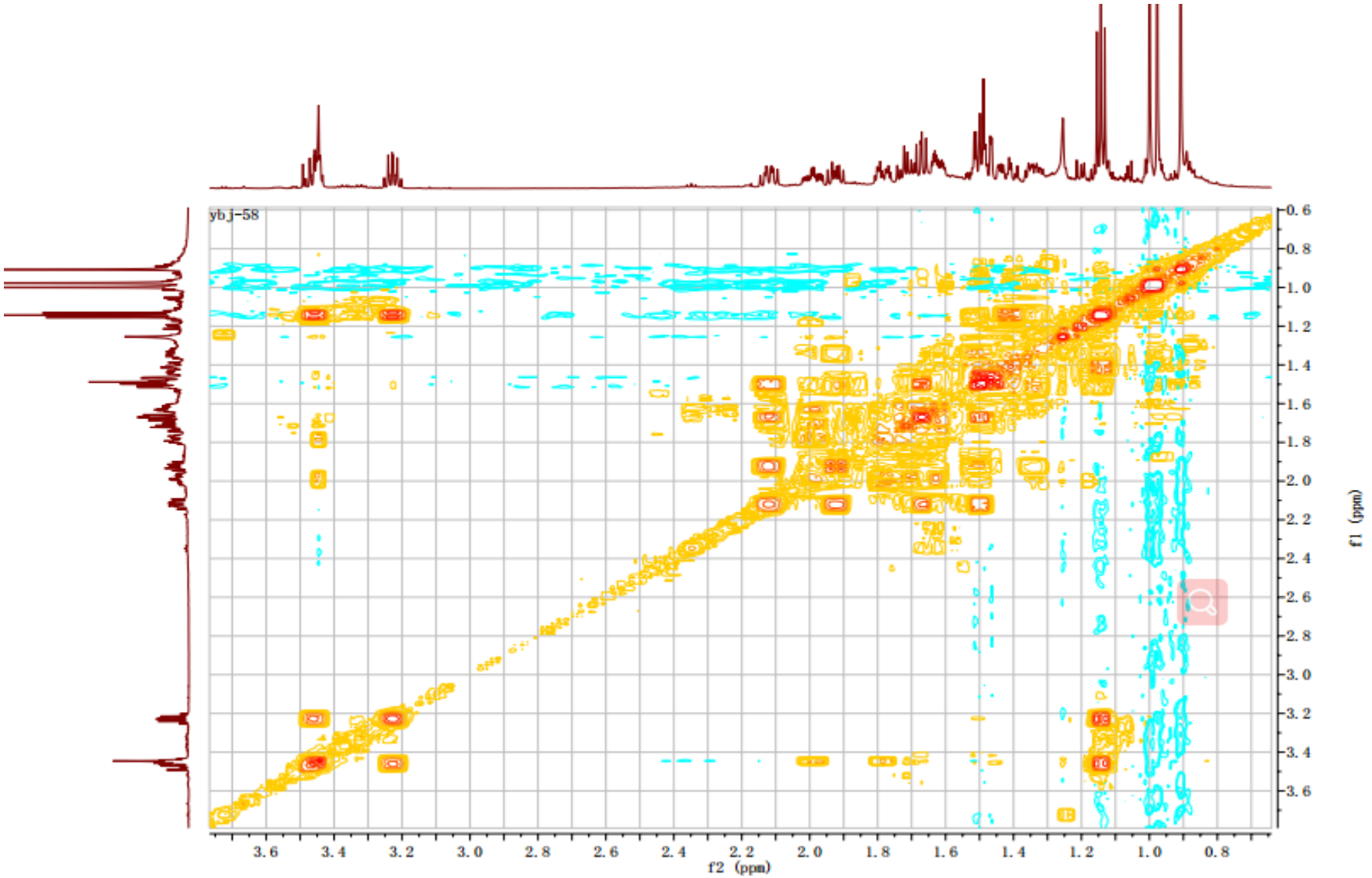
The HSQC spectrum of compound **1**



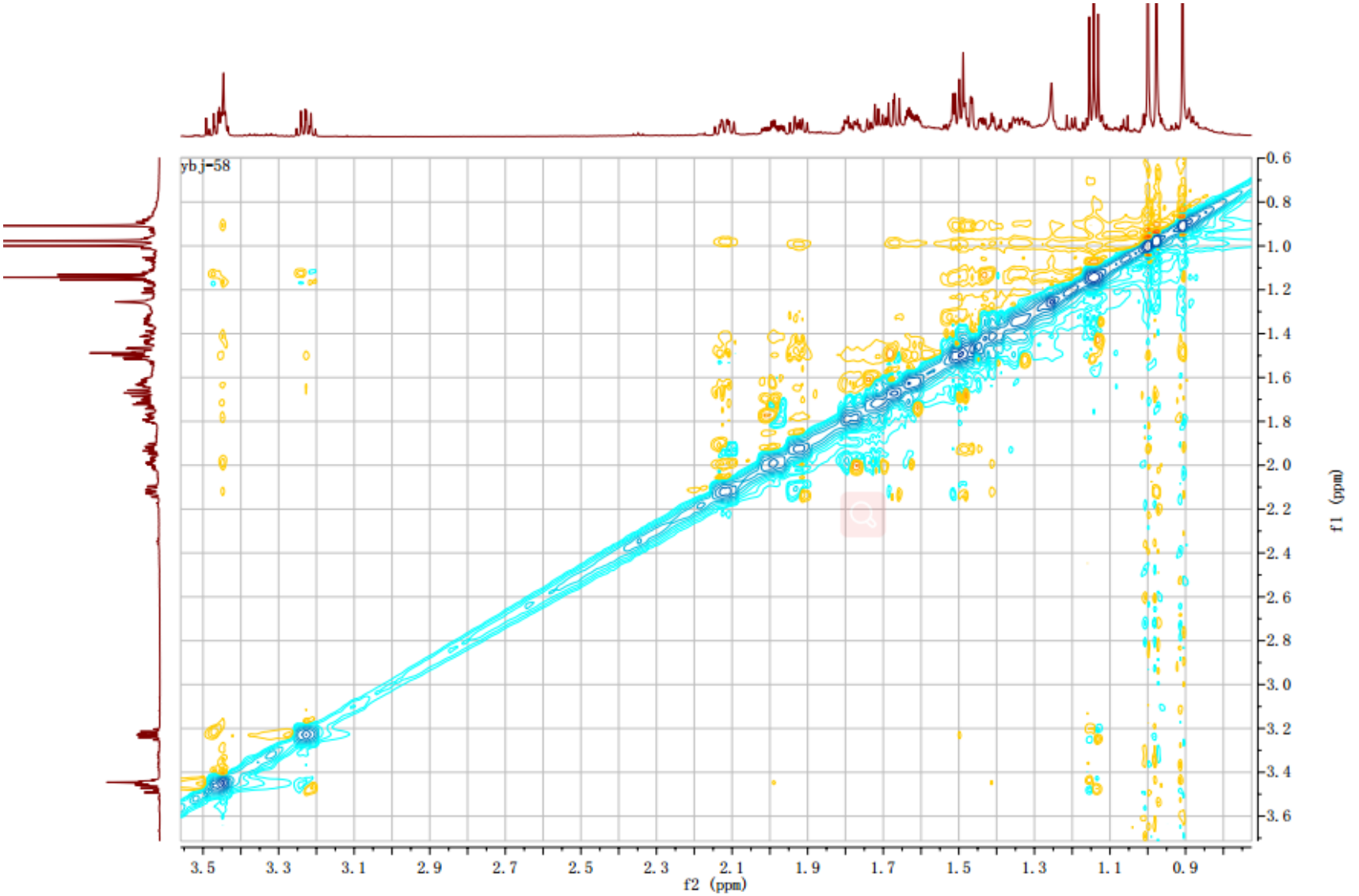
The HMBC spectrum of compound **1**



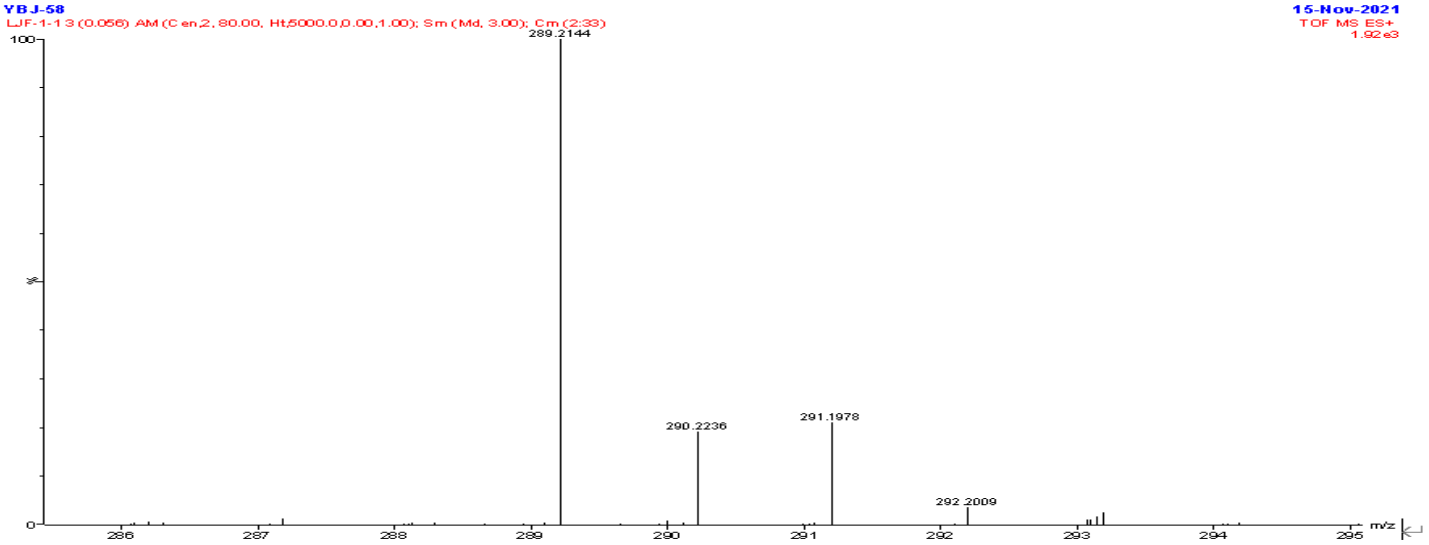
The COSY spectrum of compound **1**



The ROESY spectrum of compound **1**



# The HR-ESI-MS of compound **1**




Elemental Composition Report

Single Mass Analysis

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

Selected filters: None

Monoisotopic Mass, Even Electron Ions

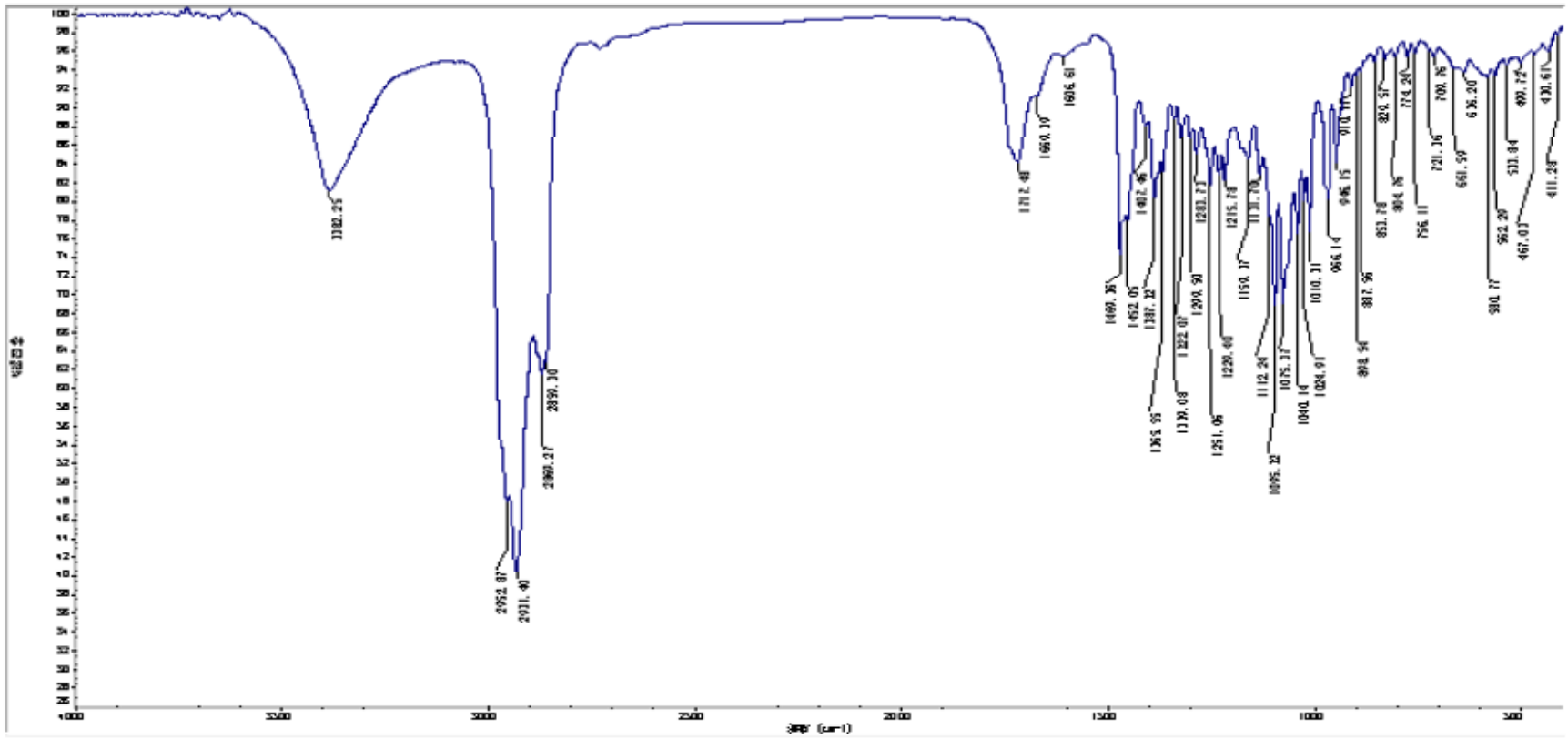
92 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-500 H: 0-1000 O: 0-20 Na: 0-1

| | | | | | | |
|---|---|---|---|---|---|---|
| Minimum: | | | | -1.5 | | |
| Maximum: | | 10.0 | 50.0 | 50.0 | | |
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
| 289.2144 | 289.2168 | -2.4 | -8.3 | 5.5 | 167.1 | C19 H29 O2 |
| | 289.2144 | 0.0 | 0.0 | 2.5 | 169.8 | C17 H30 O2 Na |
| | 289.2015 | 12.9 | 44.6 | 1.5 | 175.6 | C15 H29 O5 |

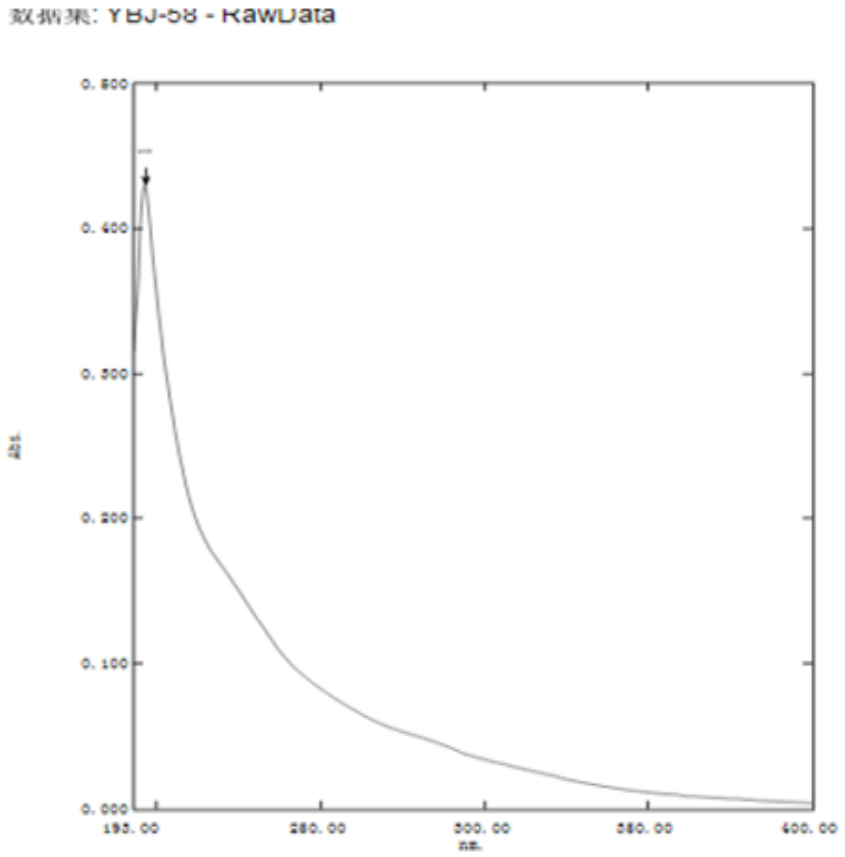
The IR (KBr) of compound **1**



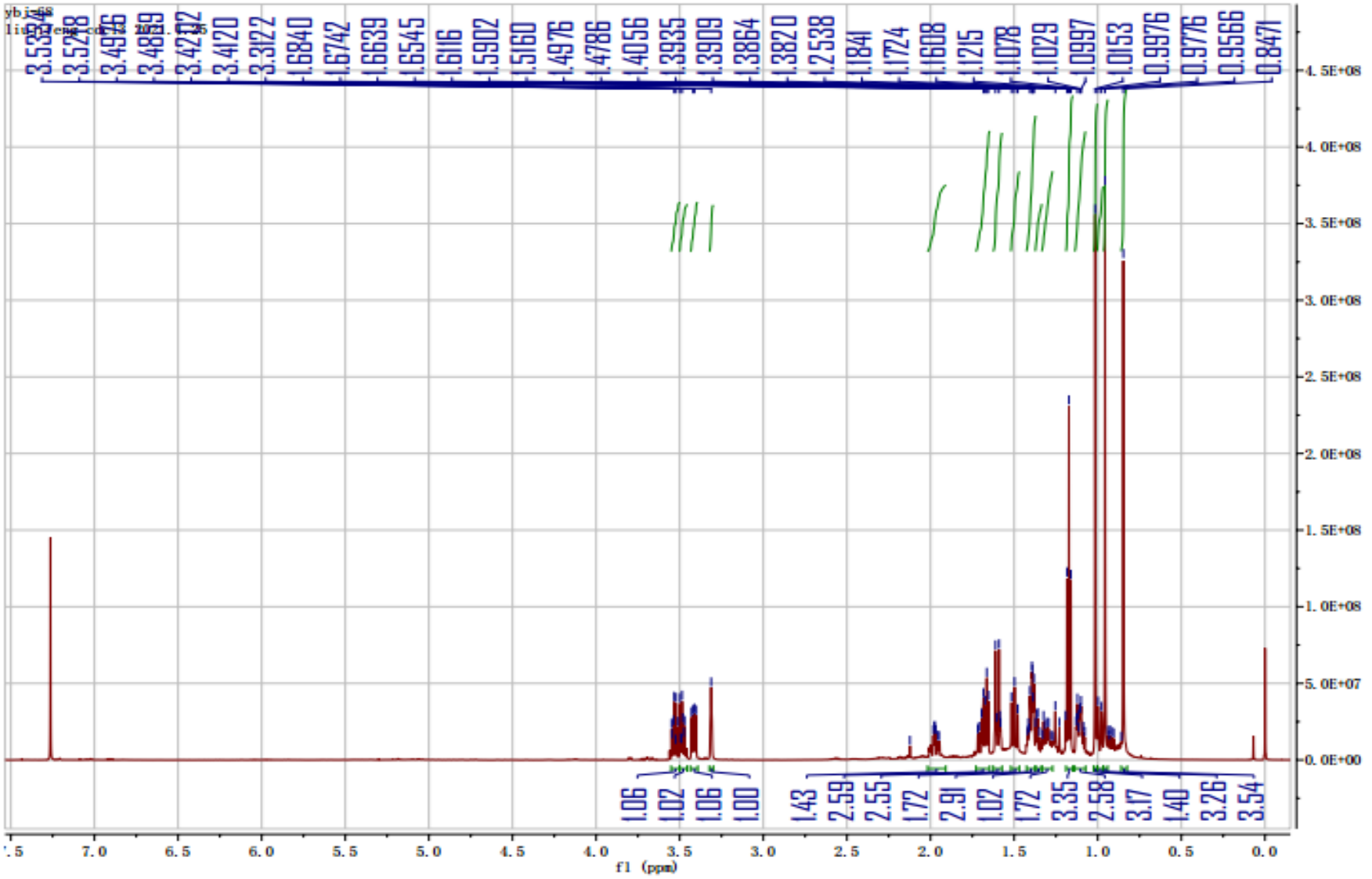

Sample Name: YBJ-58
KBr压片
采集时间: 星期四 10月 14 15:31:30 2021 (GMT+08:00) (Measured on)
仪器型号: NICOLET iS10 (Instrument)

(Sample scan) 样品扫描次数: 16
背景扫描次数: 16
(Resolution) 分辨率: 4.000
采样增益: 1.0
动镜速度: 0.4747
(Acquisition) 光阑: 80.00

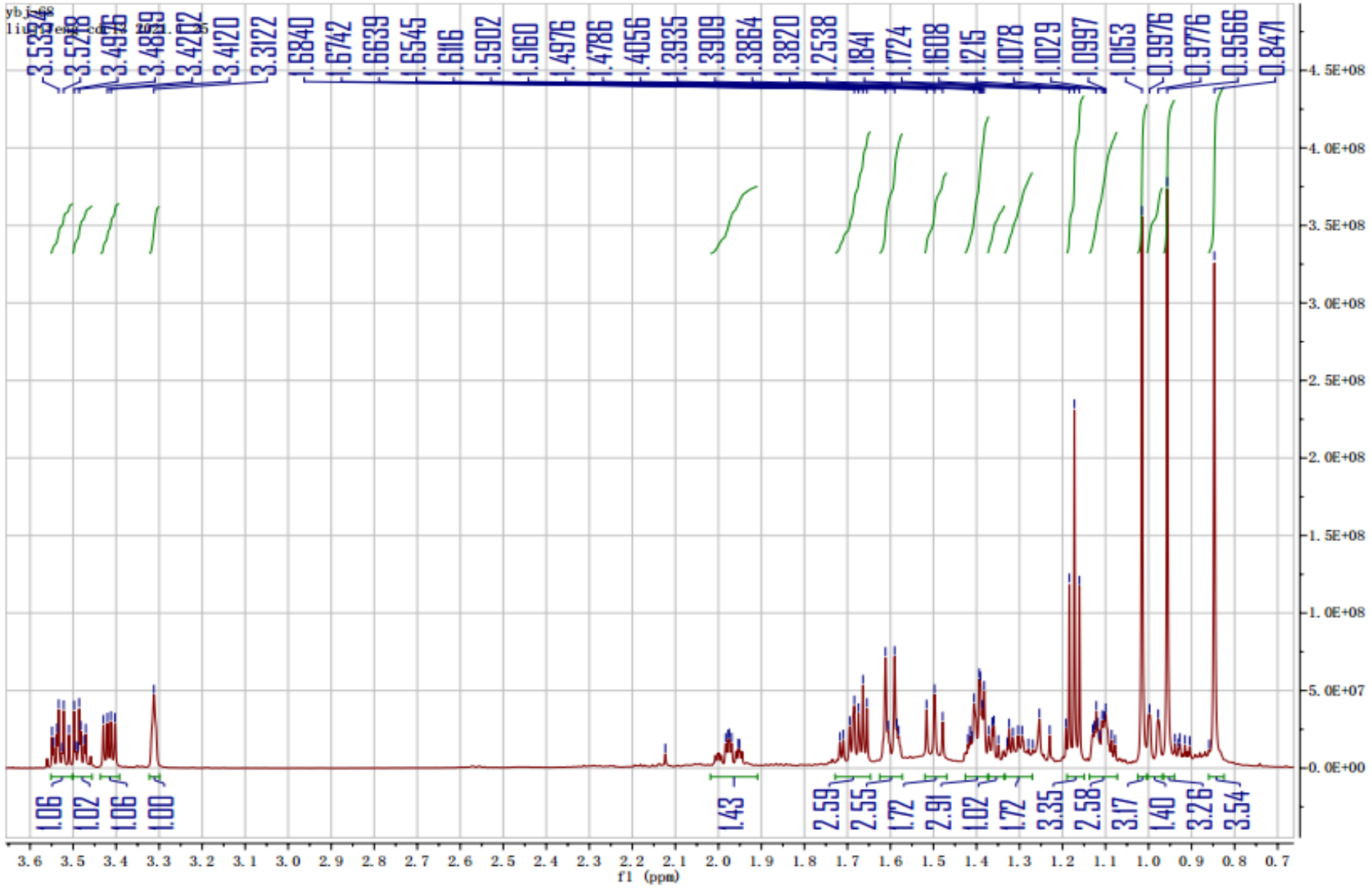
The UV spectrum of compound **1**



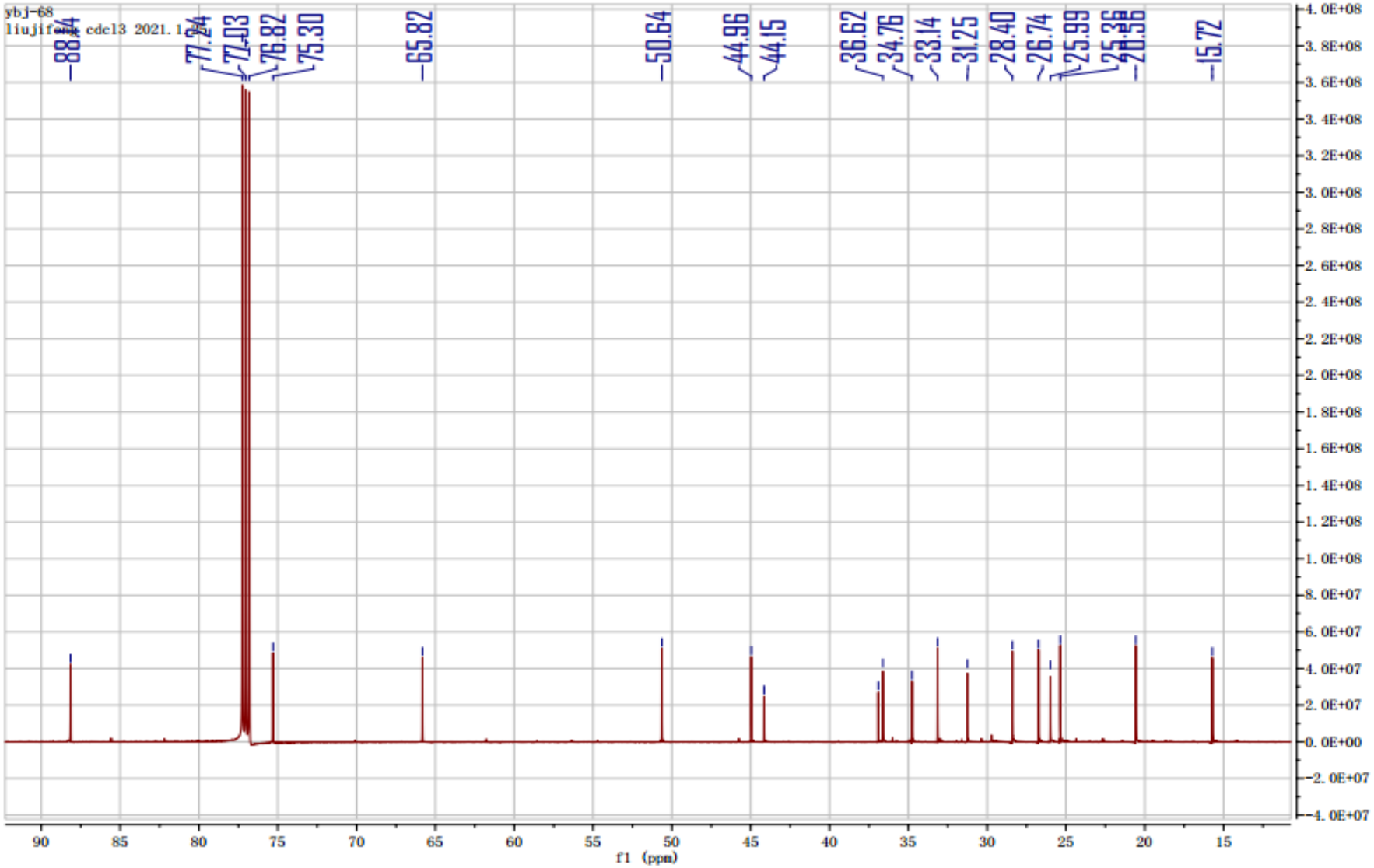
The $^{1}$H NMR spectrum of compound **3**



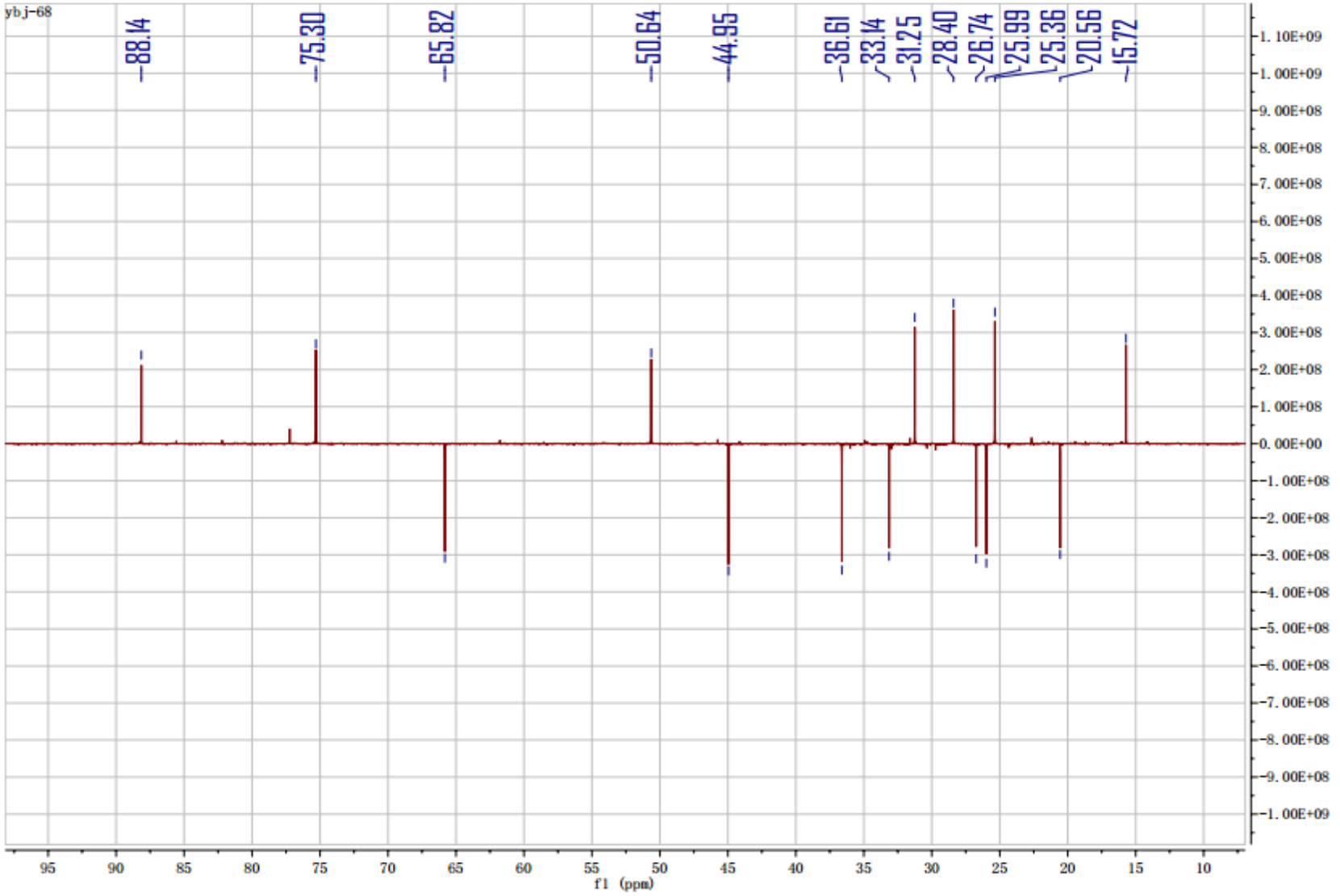
The $^{1}$H NMR spectrum of compound **3**



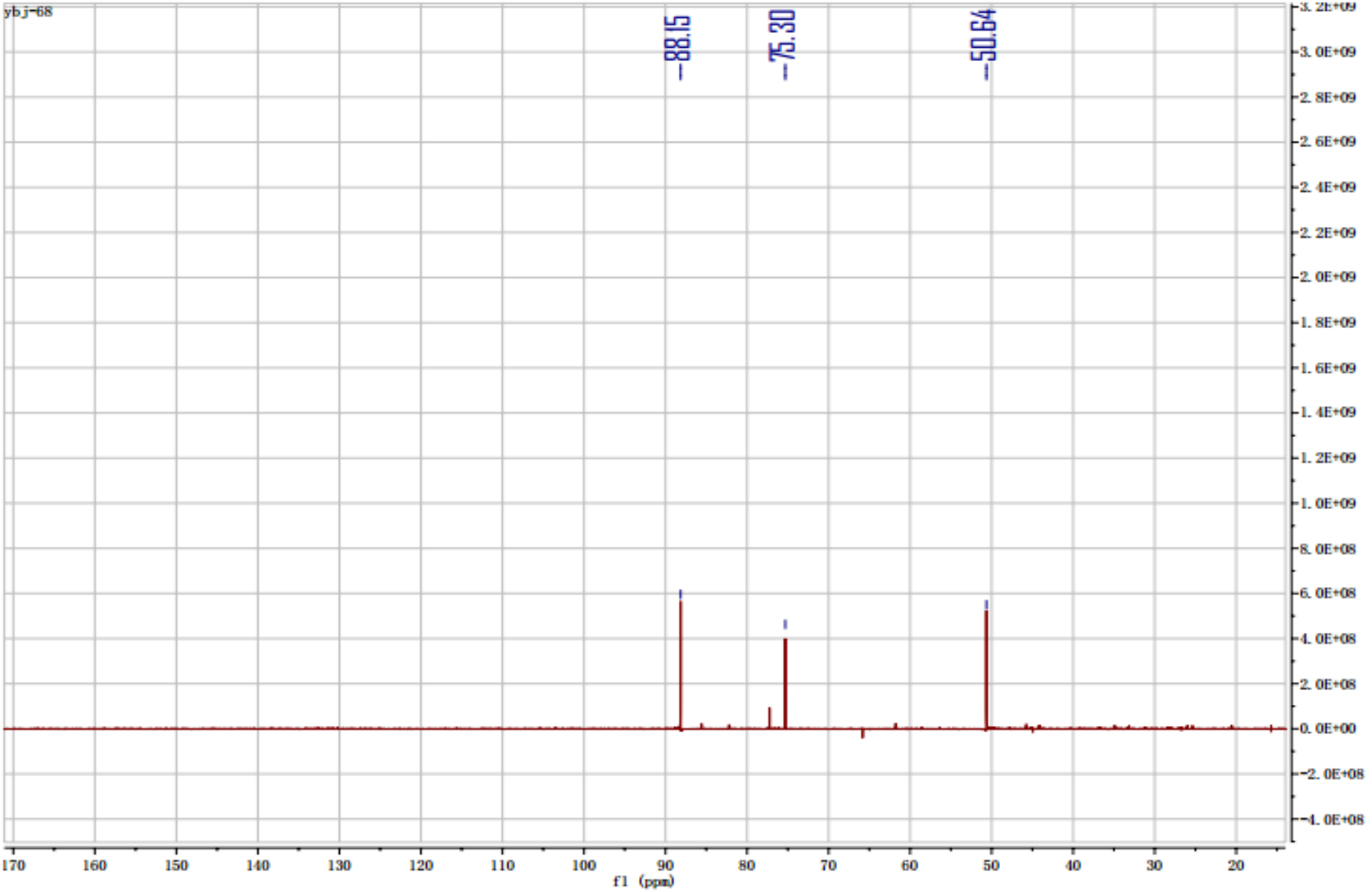
The $^{13}C$ NMR spectrum of compound **3**



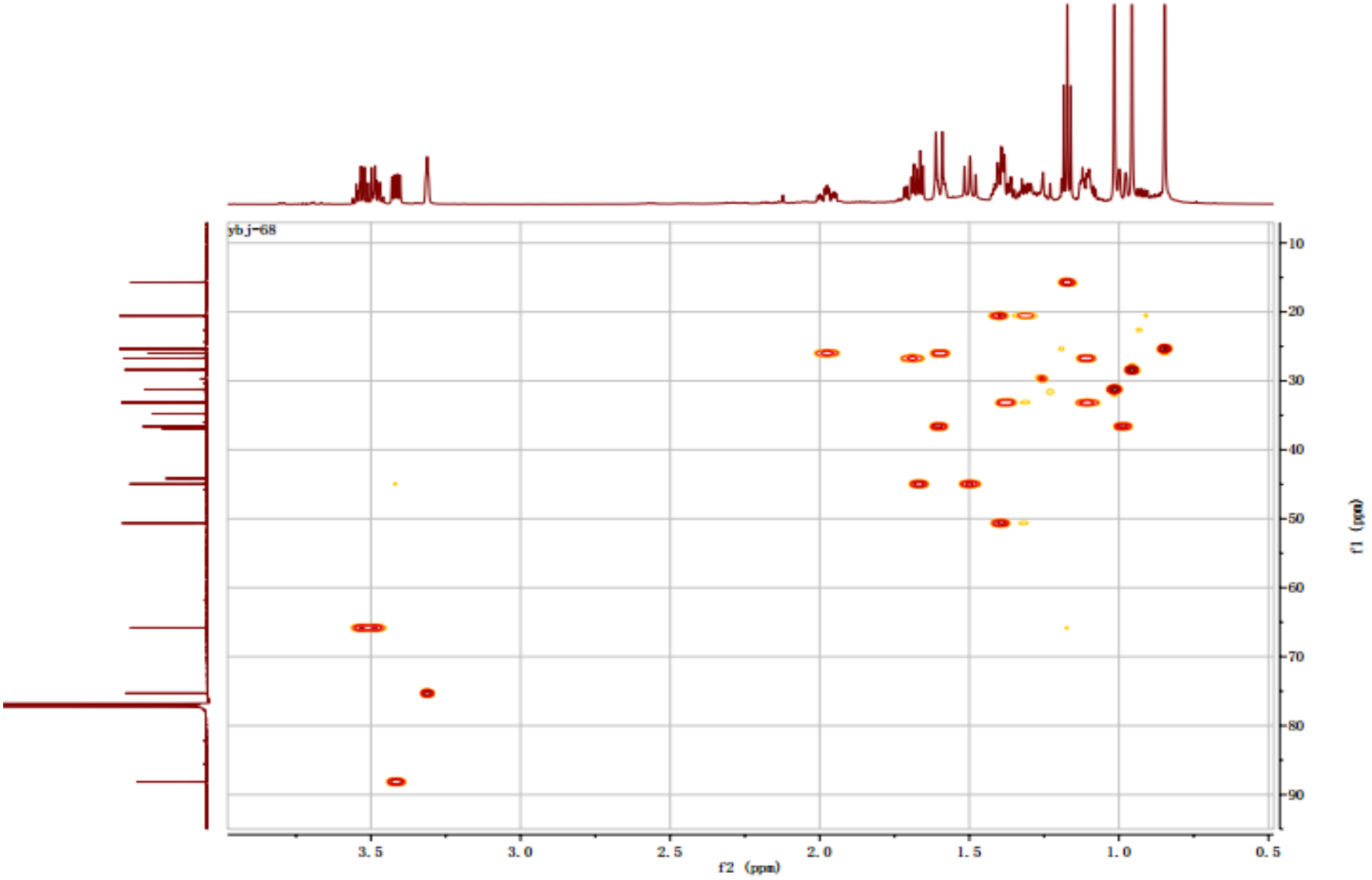
The DEPT-135 spectrum of compound **3**



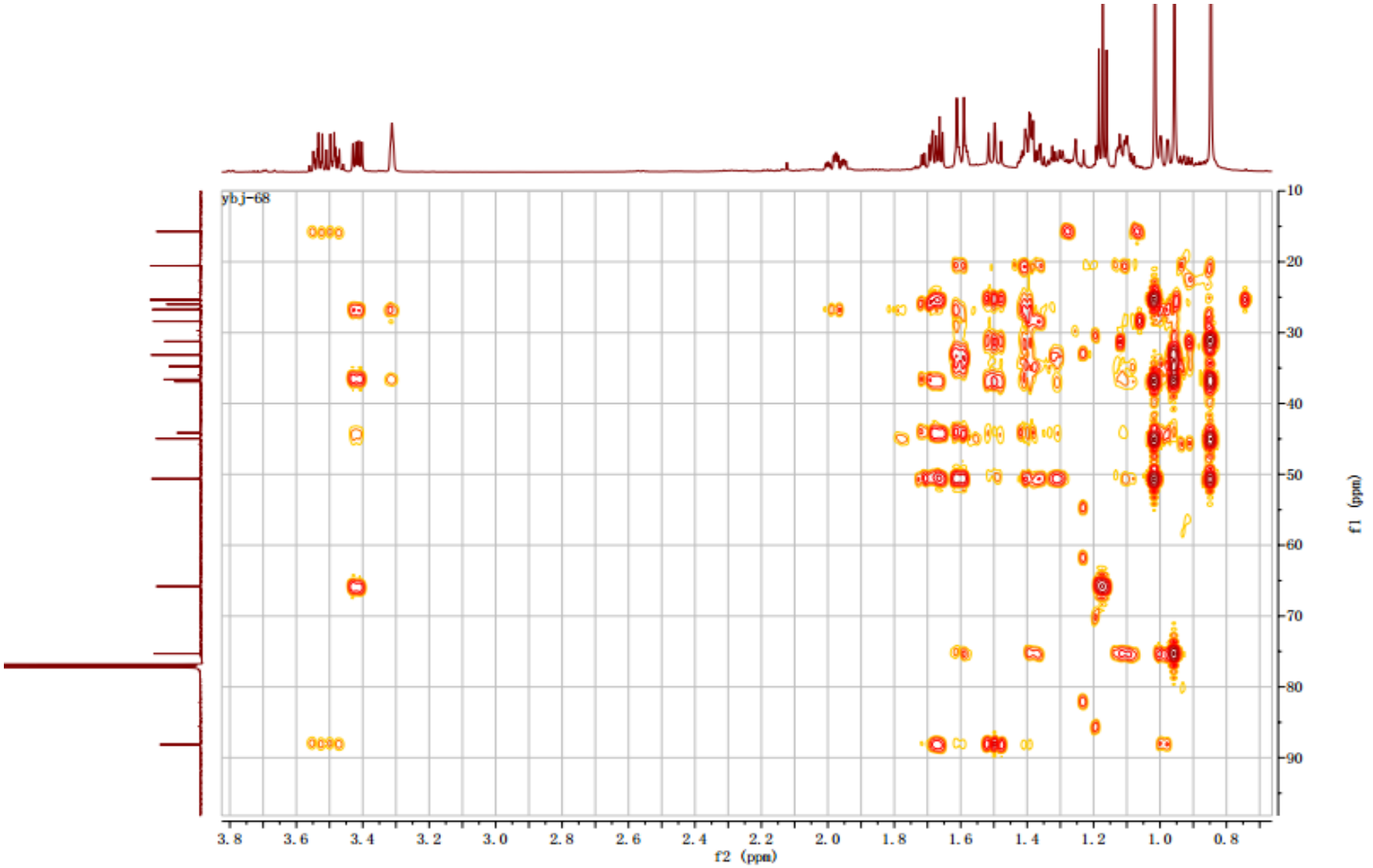
The DEPT-90 spectrum of compound **3**



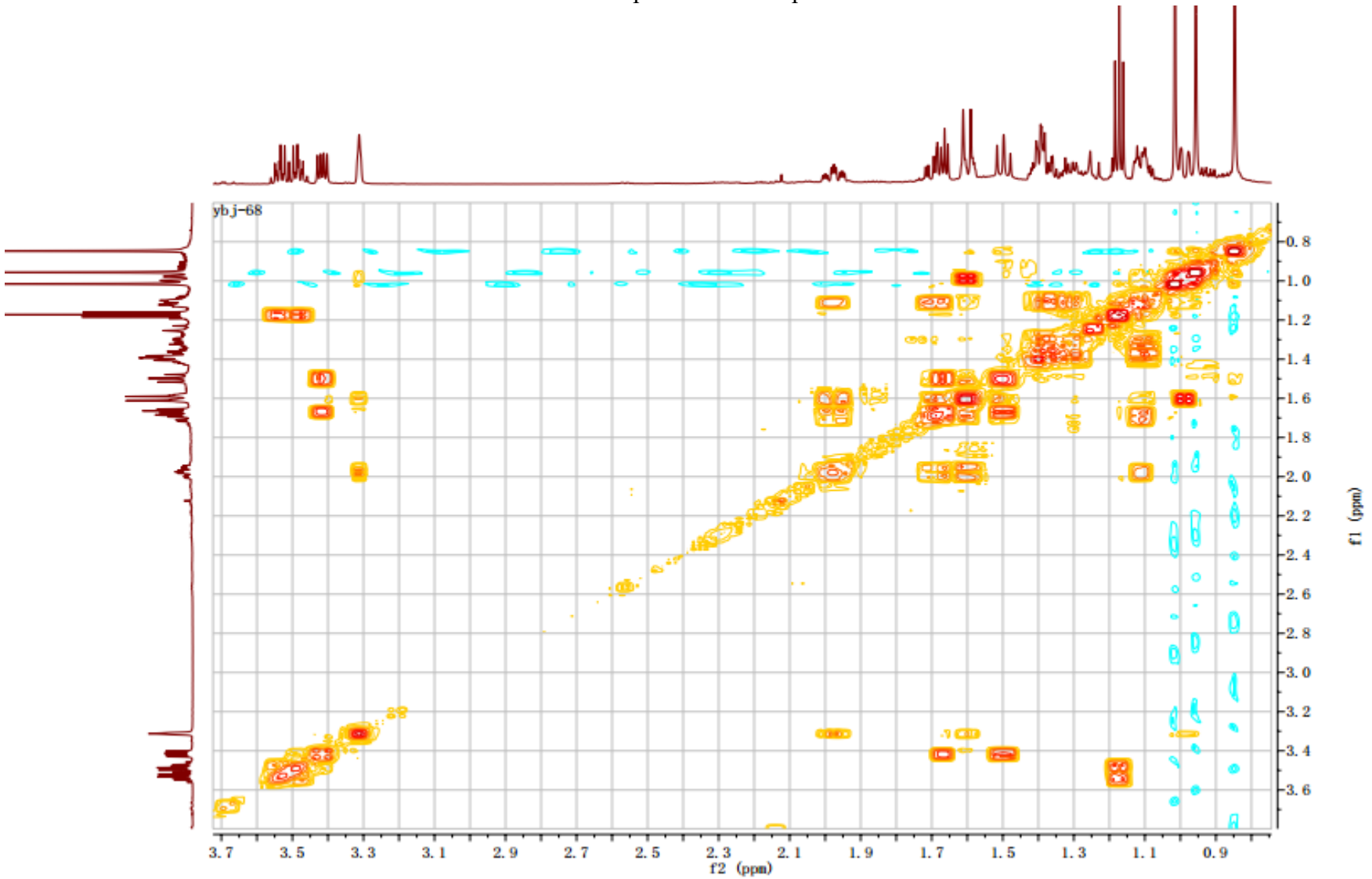
The HSQC spectrum of compound **3**



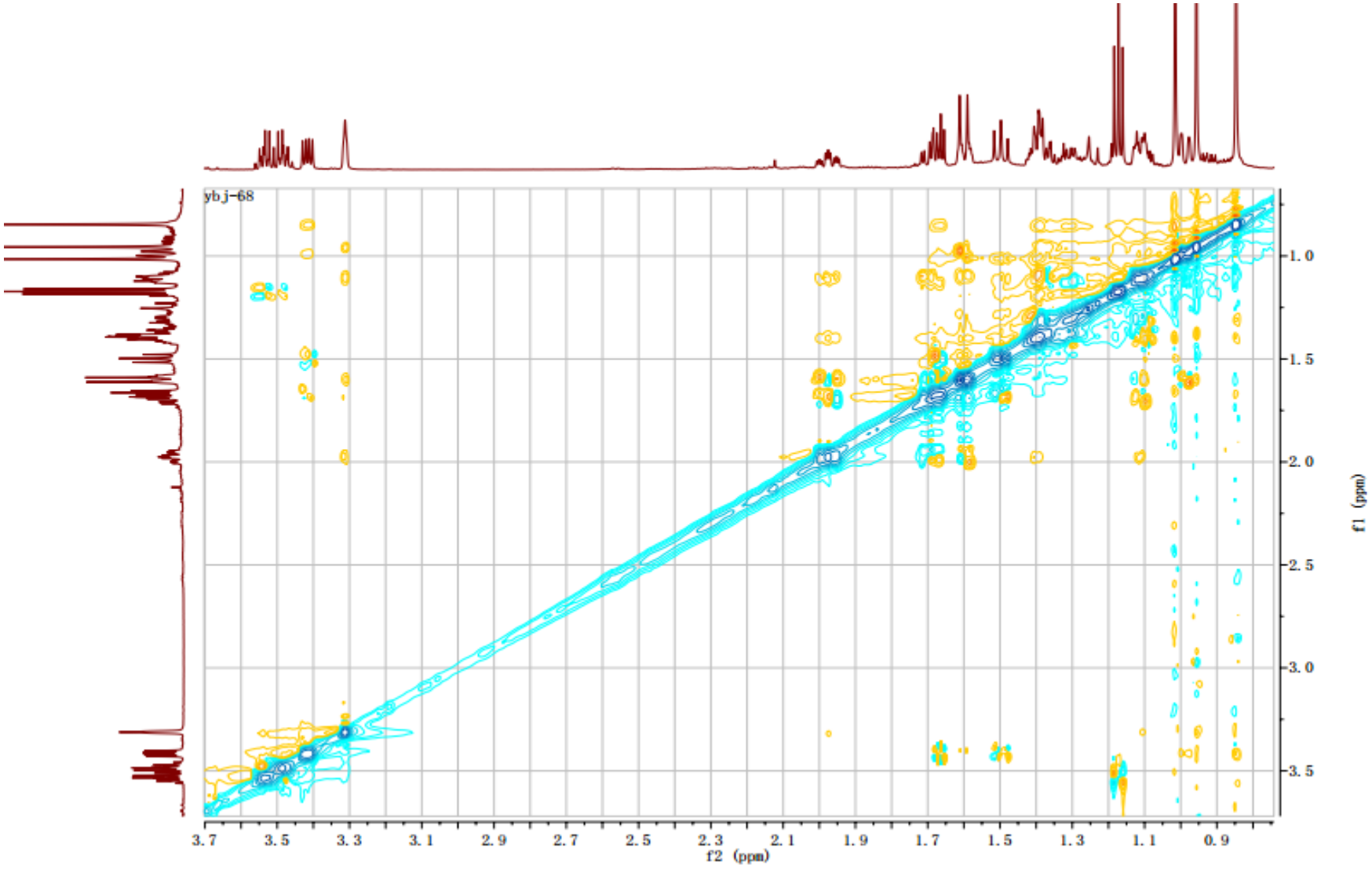
The HMBC spectrum of compound **3**



The COSY spectrum of compound **3**



The ROESY spectrum of compound **3**



Formula Predictor Report - YBJ-68.lcd



Data File: E:\DATA\2021\1011\YBJ-68.lcd

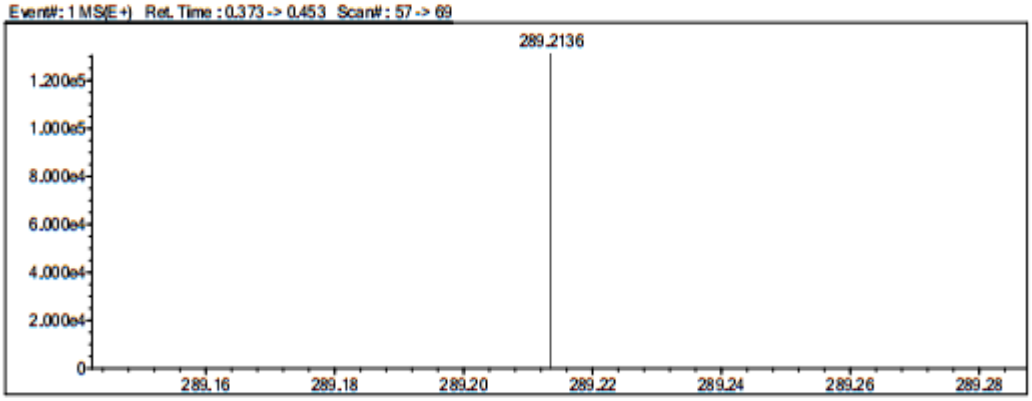
| Elmt | Val. | Min | Max | Elmt | Val. | Min | Max | Elmt | Val. | Min | Max | Elmt | Val. | Min | Max | Use Adduct |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | 1 | 5 | 100 | F | 1 | 0 | 0 | Cl | 1 | 0 | 0 | Ag | 1 | 0 | 0 | Na |
| 2H | 1 | 0 | 0 | Na | 1 | 0 | 0 | Co | 2 | 0 | 0 | I | 3 | 0 | 0 | |
| B | 3 | 0 | 0 | Mg | 2 | 0 | 0 | Cu | 2 | 0 | 0 | Ir | 3 | 0 | 5 | |
| C | 4 | 5 | 100 | Si | 4 | 0 | 0 | Se | 2 | 0 | 0 | | | | | |
| N | 3 | 0 | 20 | P | 3 | 0 | 0 | Br | 1 | 0 | 0 | | | | | |
| O | 2 | 0 | 30 | S | 2 | 0 | 0 | Pd | 2 | 0 | 0 | | | | | |

Error Margin (ppm): 5
HC Ratio: unlimited
Max Isotopes: all
MSn Iso RI (%): 75.00

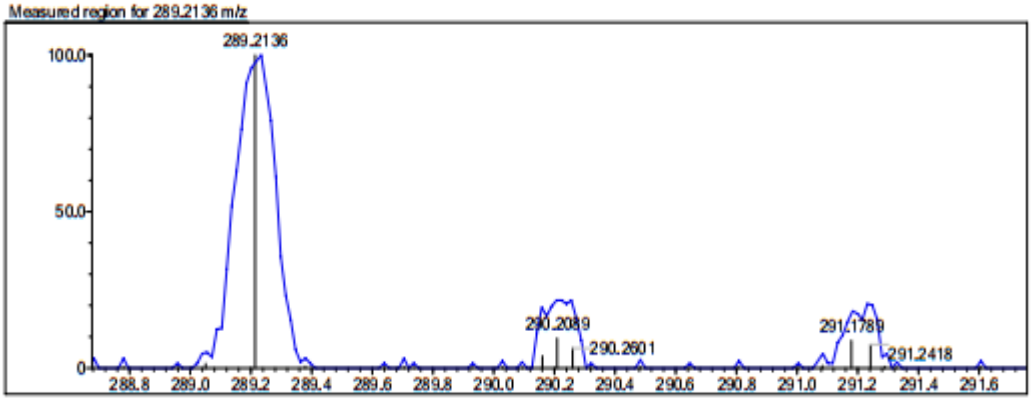
DBE Range: not fixed
Apply N Rule: no
Isotope RI (%): 1.00
MSn Logic Mode: OR

Electron Ions: both
Use MSn Info: yes
Isotope Res: 10000
Max Results: 20

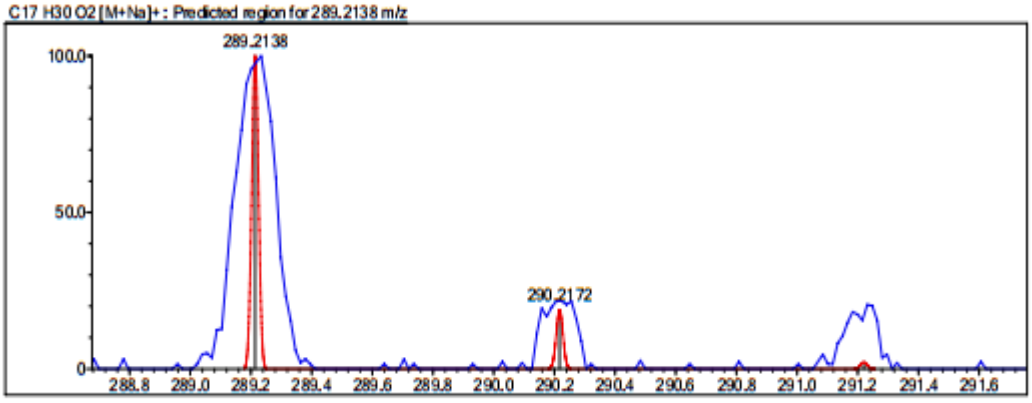
Event#: 1 MS(E+) Ret. Time : 0.373 -> 0.453 Scan# : 57 -> 69




Measured region for 289.2136 m/z



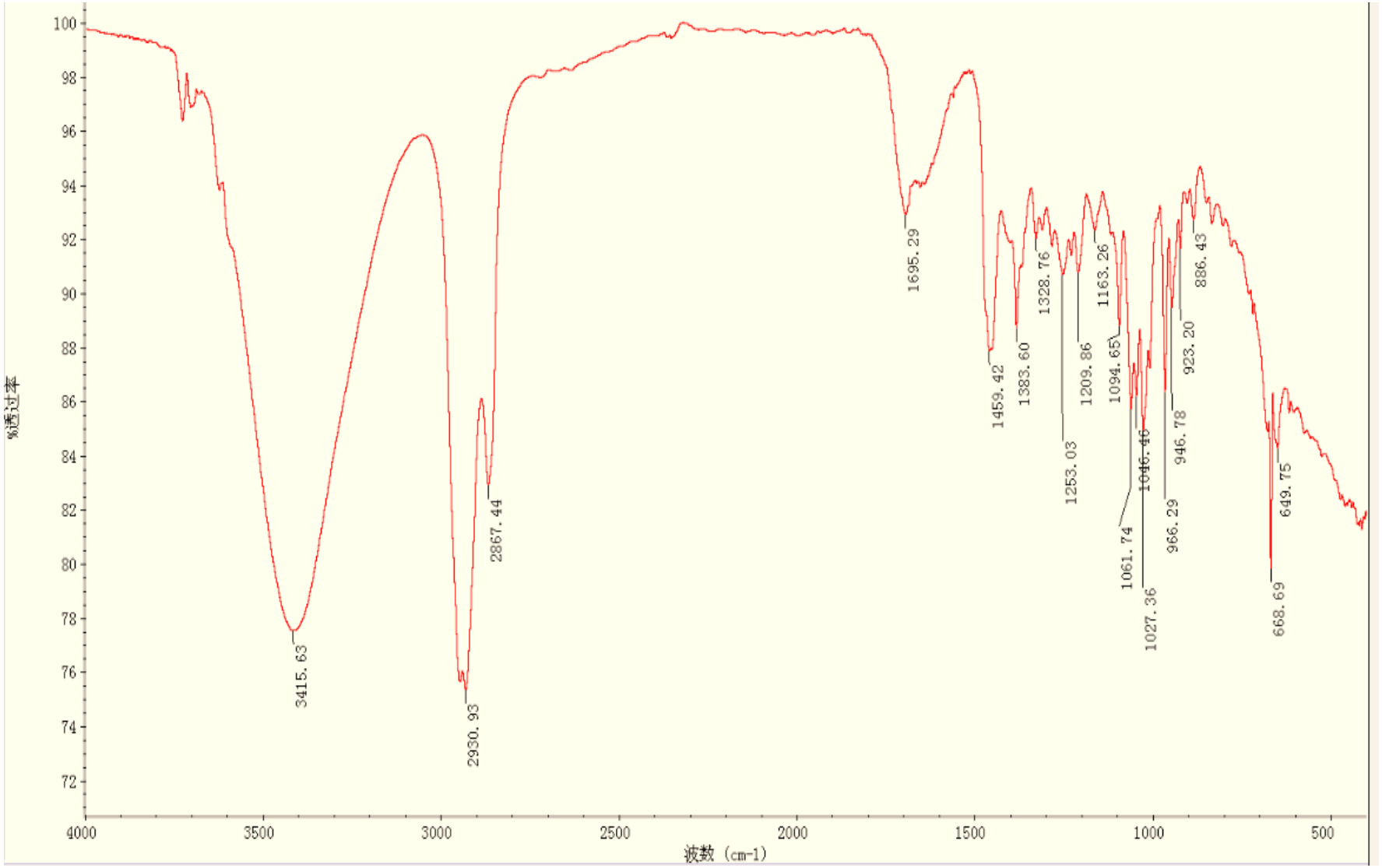

C17 H30 O2 [M+Na]+ : Predicted region for 289.2138 m/z



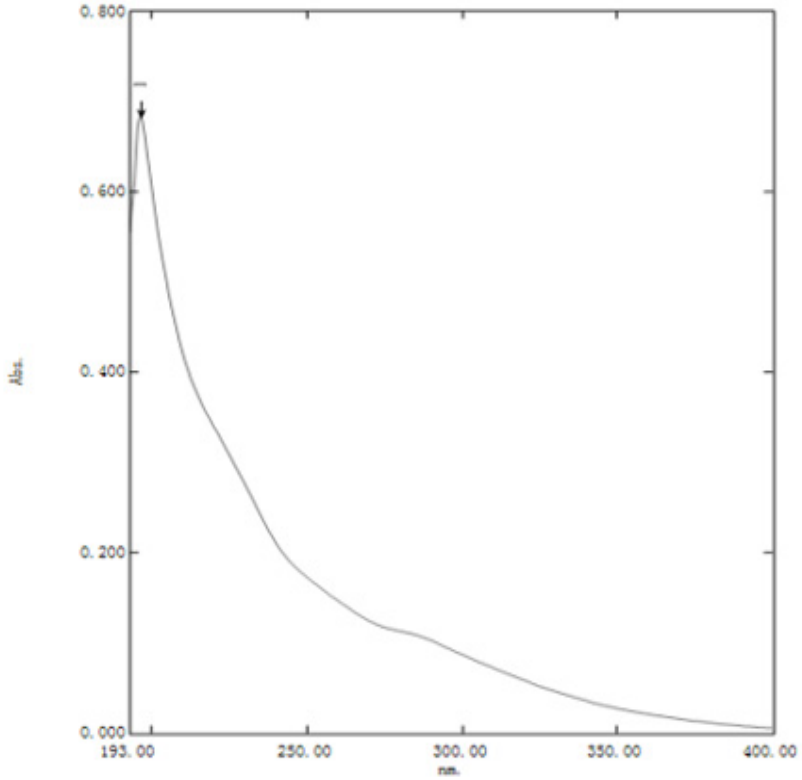

| Formula (M) | Ion | Meas. m/z | Pred. m/z | Df. (mDa) | Df. (ppm) | DBE |
|---|---|---|---|---|---|---|
| C17 H30 O2 | [M+Na]+ | 289.2136 | 289.2138 | -0.2 | -0.69 | 3.0 |

The HR-ESI-MS spectrum of compound **3**

The IR (KBr) spectrum of compound **3**



The UV spectrum of compound **3**




【测定属性】
波长范围(nm): 190.00 到 600.00
扫描速度: 中速
采样间隔: 0.5
自动采样间隔: 停用
扫描模式: 单个

【仪器属性】
仪器类型: UV-2700 系列
测定方式: 吸收值
狭缝宽: 5.0 nm
积分时间: 0.1 秒
光源转换波长: 325.0 nm
检测器单元: 直接
S/R 转换: 标准
阶梯校正: Off

【附件属性】
附件: 无

【数据处理参数】
阈值: 0.0010000
点: 4
内插: 停用
平均: 停用

【样品准备属性】
重量:
体积:
稀释:
光程长: 10mm
附加信息: 样品浓度：0.1072毫克/毫升
溶剂：甲醇

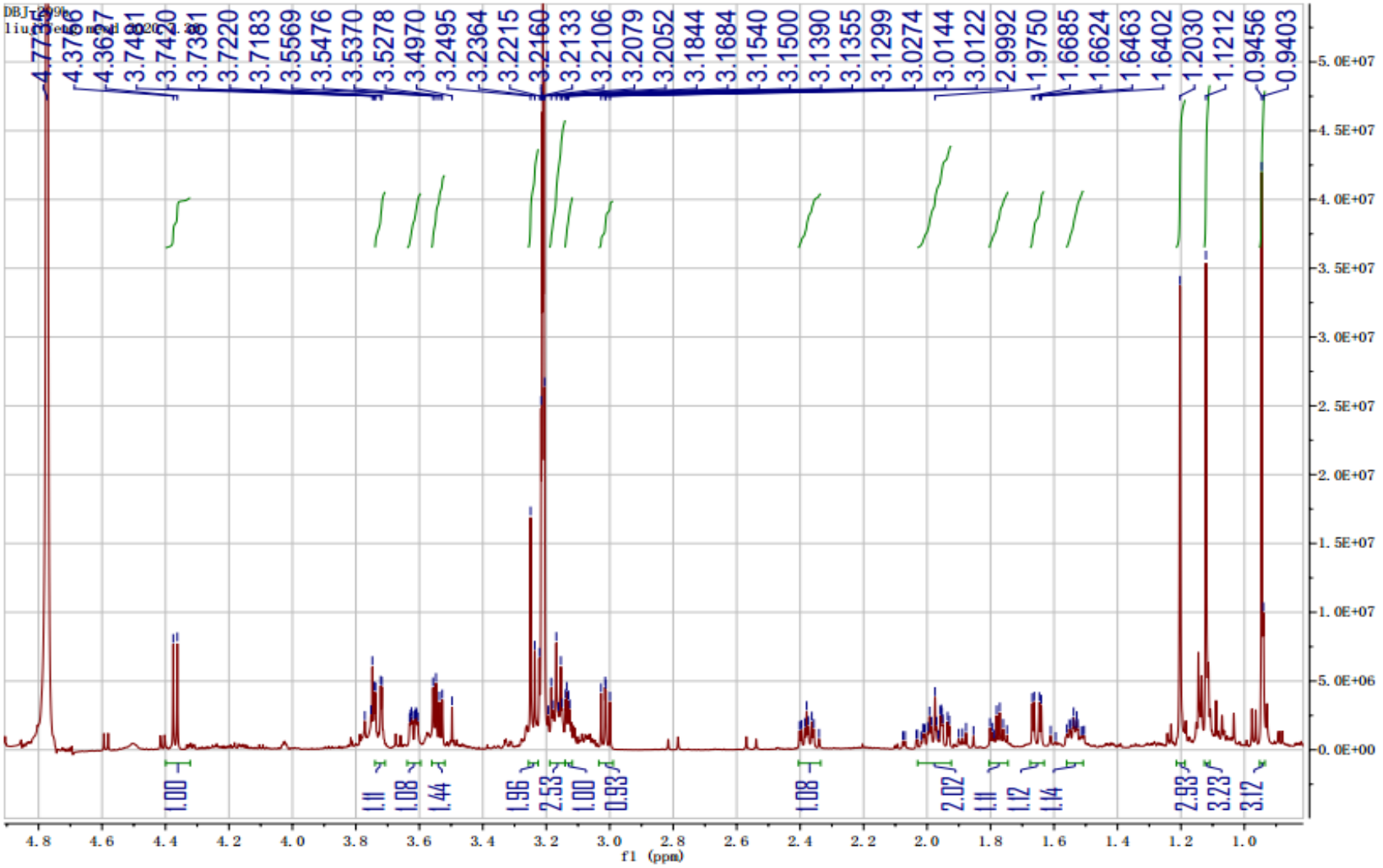
Wavelength Range:1
Scan Speed: Medium
Sampling Interval:0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2700PC Series
Measuring Mode: Absorbance
Slit Width: 5.0 nm
Light Source Change Wavelength: 325.0 nm
S/R Exchange: Normal

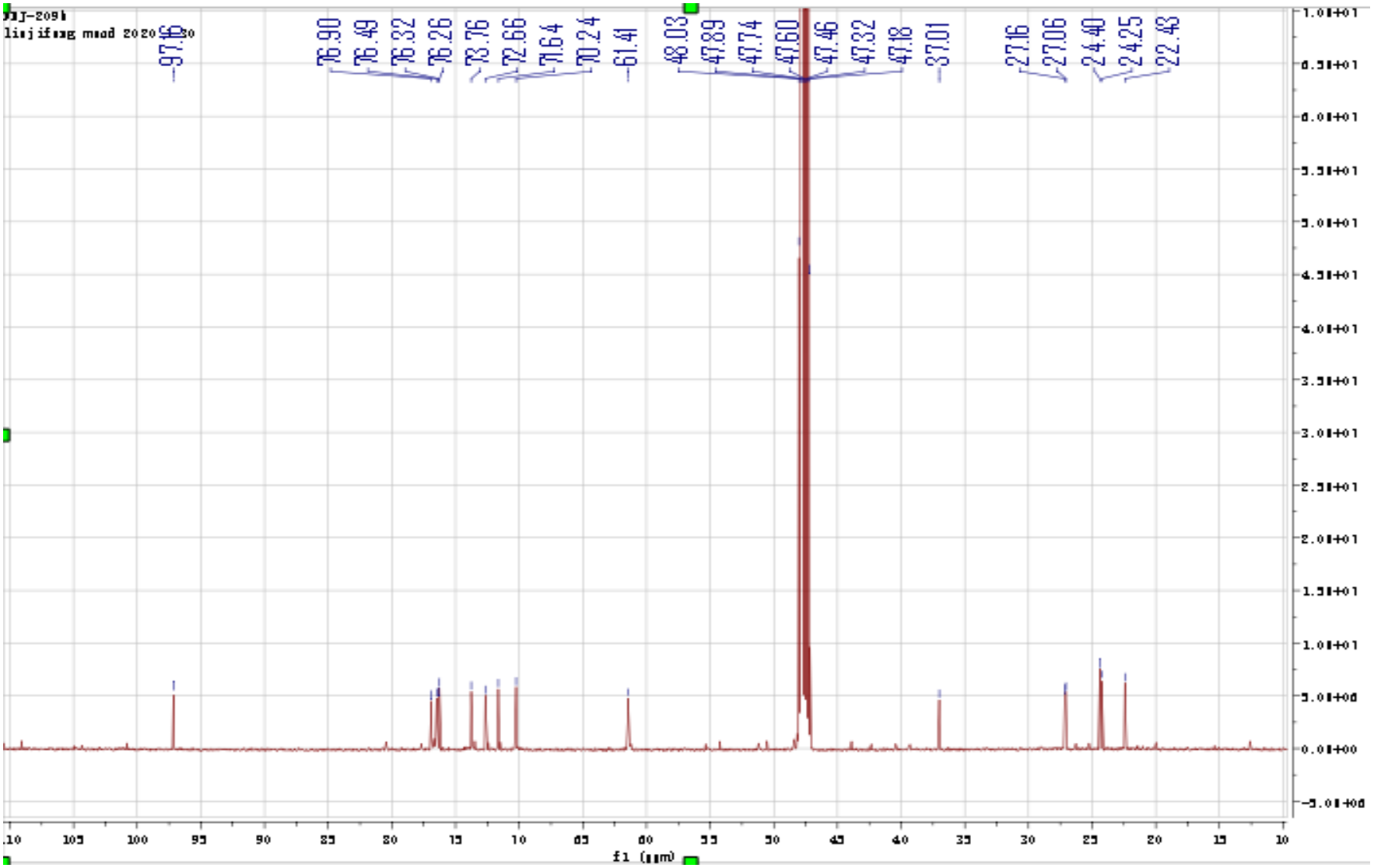
Sample concentration: 0.1072 mg/mL
Solvent: MeOH

| No. | P/V | 波长(nm) | Abs. | 描述 |
|---|---|---|---|---|
| 1 | ↑ | 196.50 | 0.683 | |

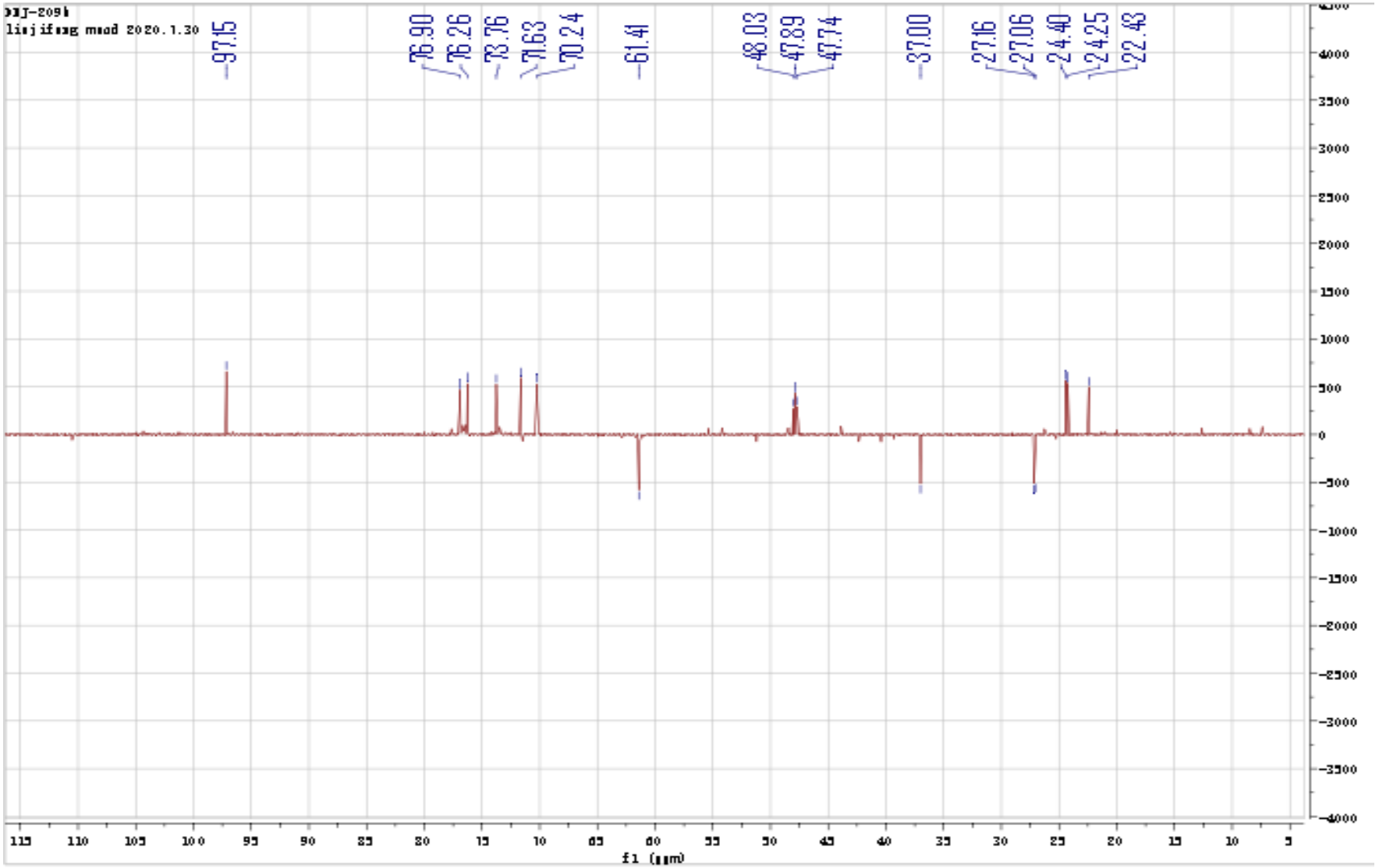
The $^{1}$H NMR spectrum of compound **5**



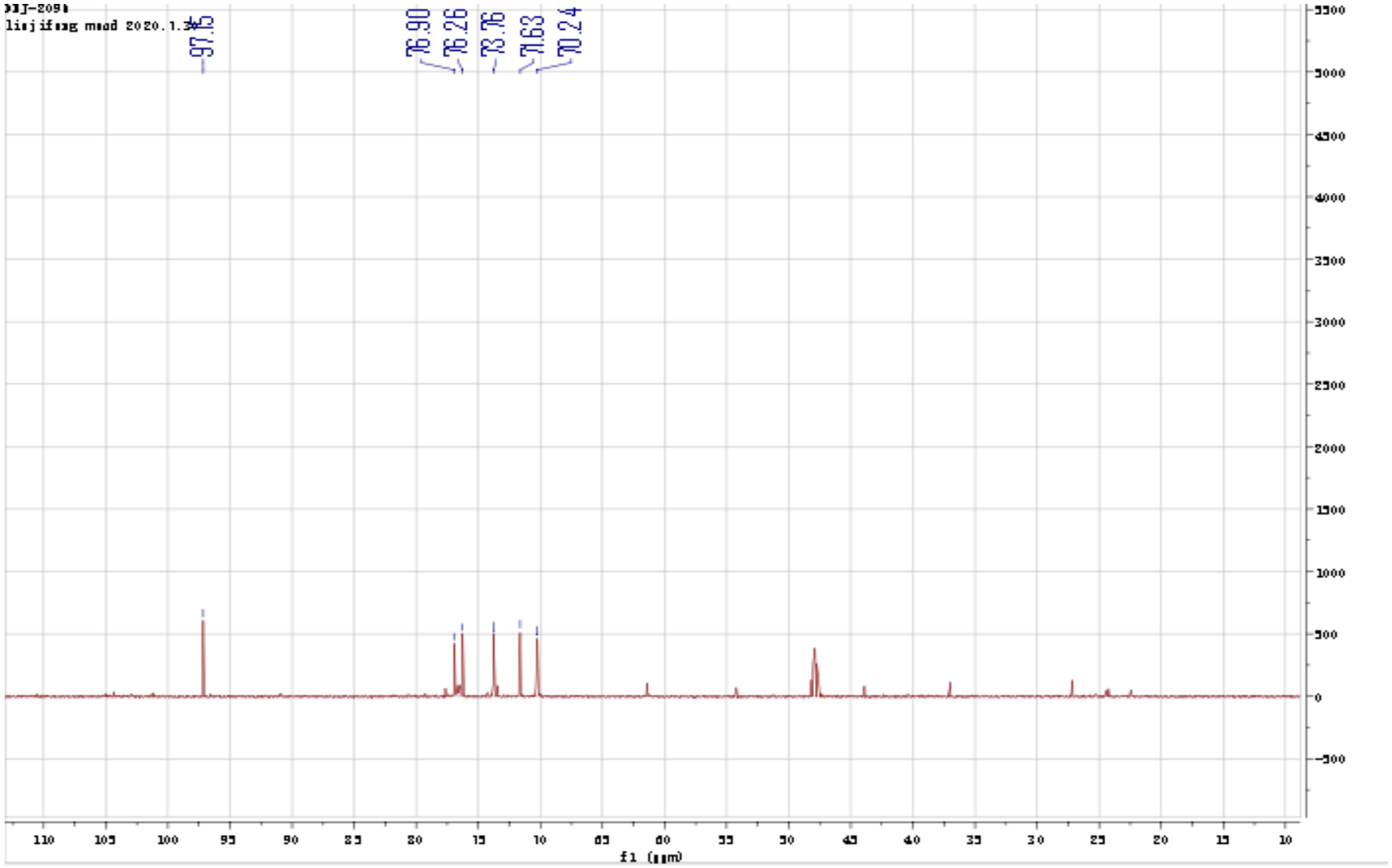
The $^{13}C$ NMR spectrum of compound **5**



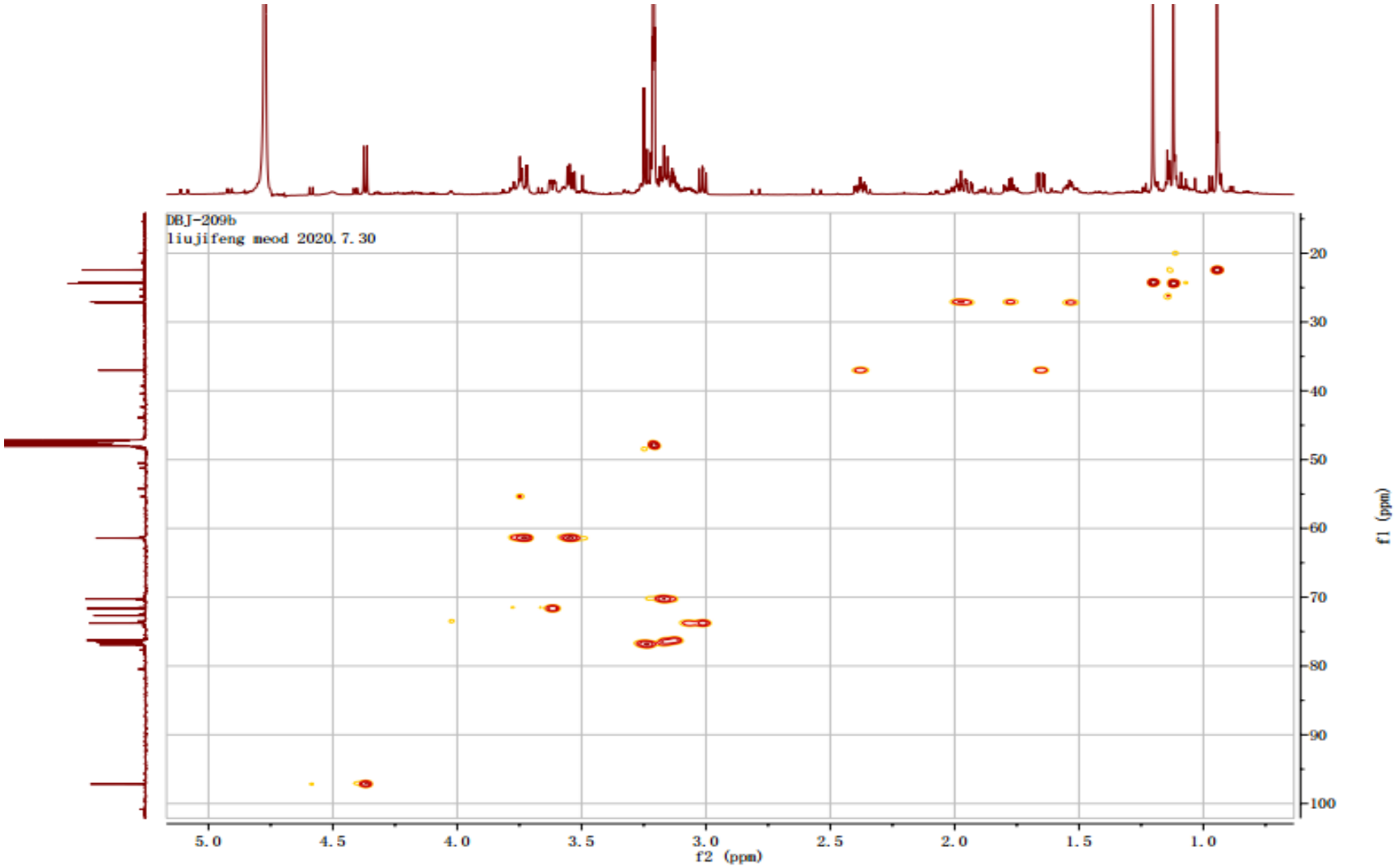
The DEPT-135 spectrum of compound **5**



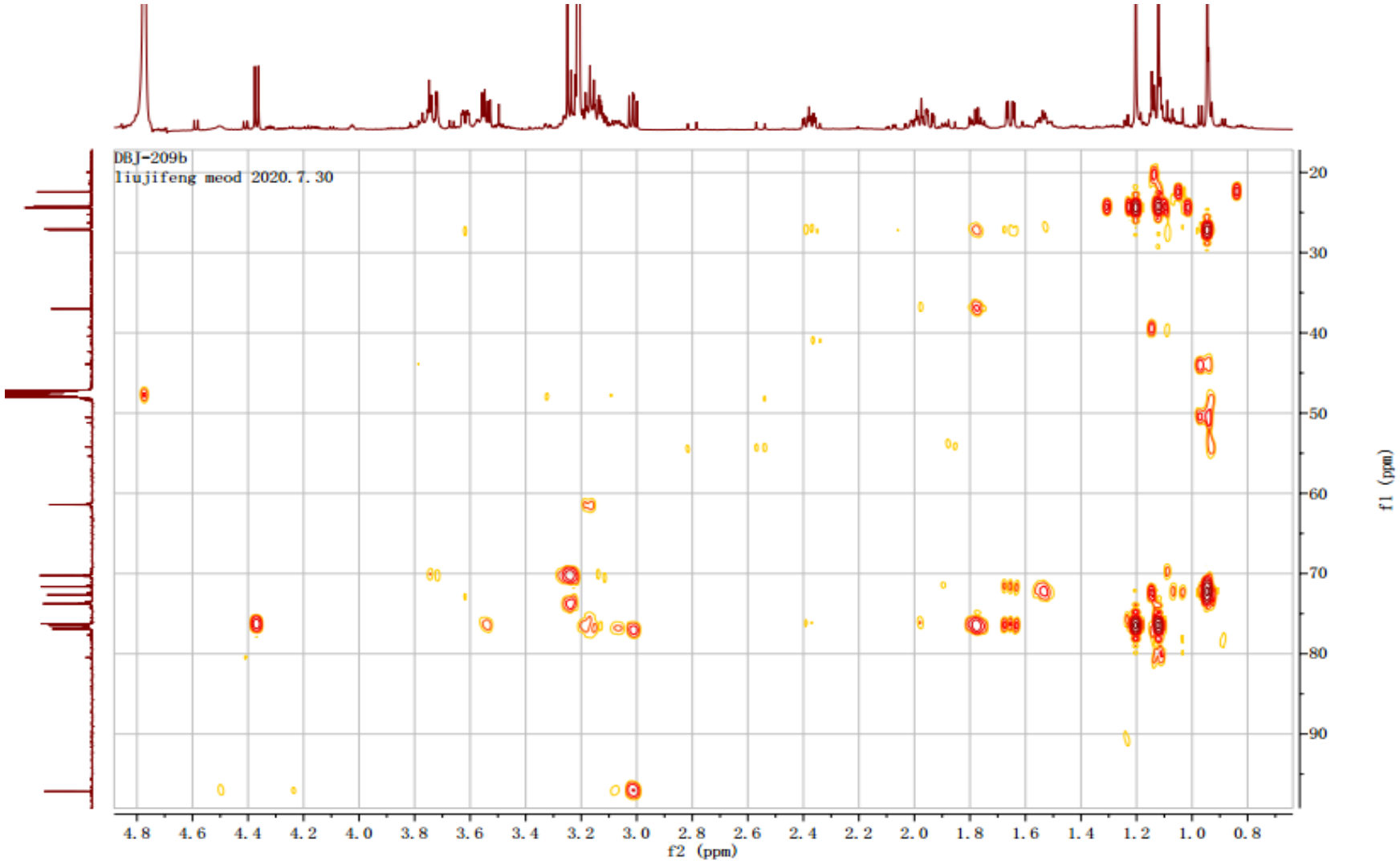
The DEPT-90 spectrum of compound **5**



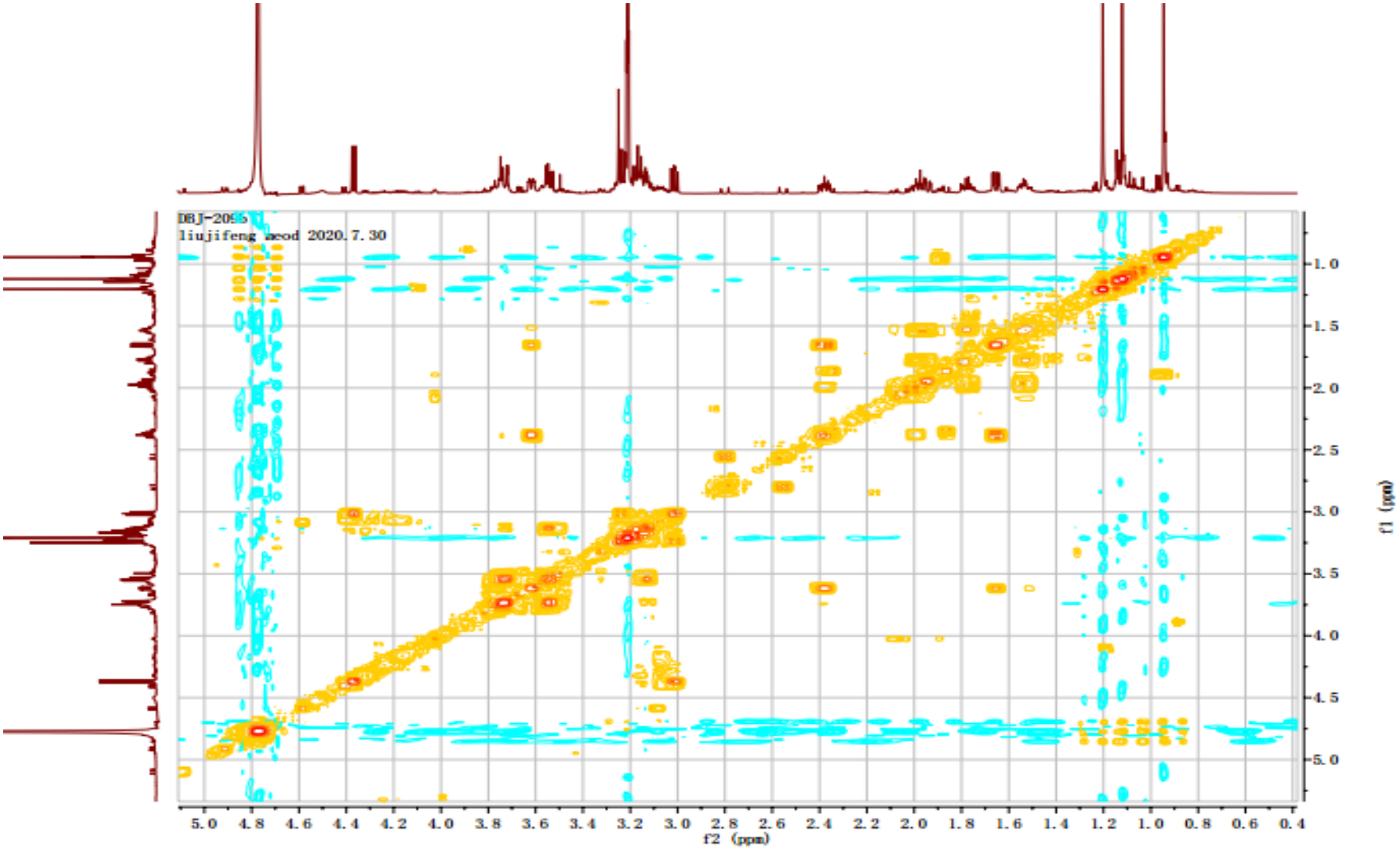
The HSQC spectrum of compound **5**



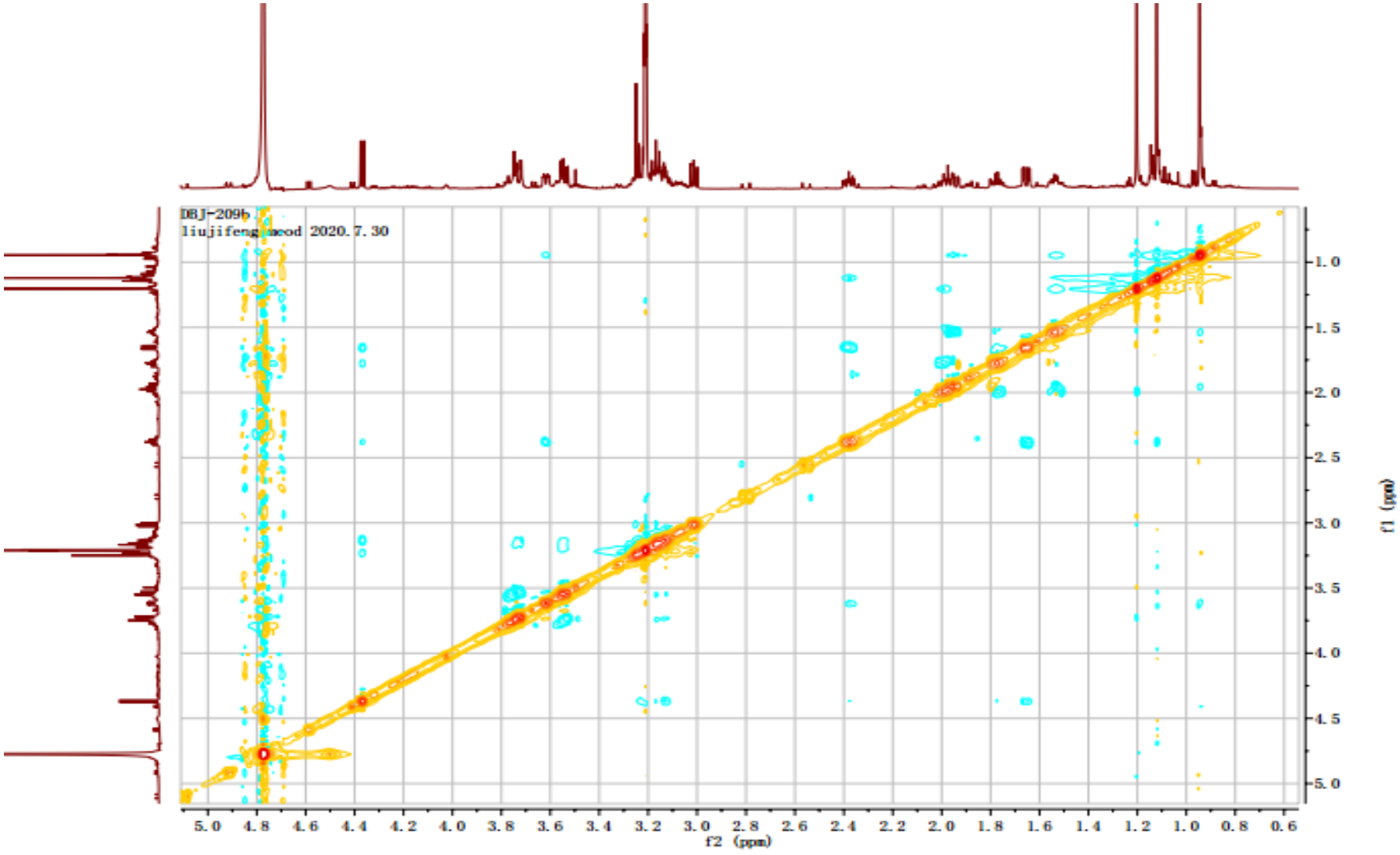
The HMBC spectrum of compound **5**



The $^{1}H$-$^{1}H$ COSY spectrum of compound **5**



The ROESY spectrum of compound **5**



Formula Predictor Report - DBJ-209B_19.lcd

Page 1 of 1

Data File: D:\wendang\2020-10-06\DBJ-209B_19.lcd

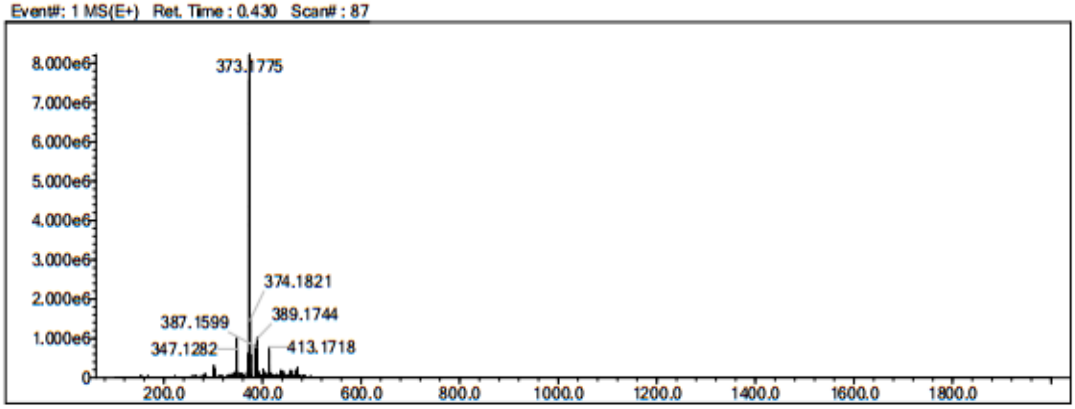
| Elmt | Val. | Min | Max | Elmt | Val. | Min | Max | Use Adduct |
|---|---|---|---|---|---|---|---|---|
| H | 1 | 0 | 300 | O | 2 | 0 | 50 | H |
| C | 4 | 0 | 100 | S | 2 | 0 | 0 | Na |
| N | 3 | 0 | 0 | Br | 1 | 0 | 0 | |

Error Margin (mDa): 20.0
HC Ratio: unlimited
Max Isotopes: all
MSn Iso RI (%): 75.00

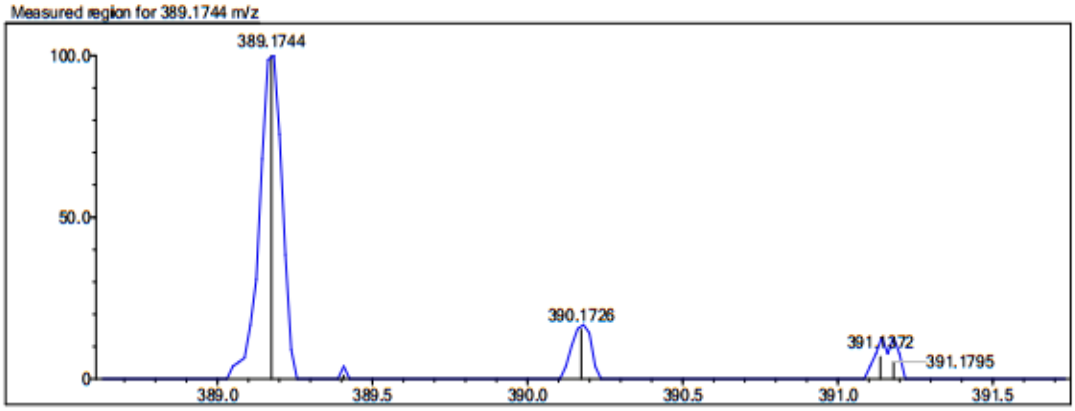
DBE Range: 0.0 - 1000.0
Apply N Rule: yes
Isotope RI (%): 1.00
MSn Logic Mode: AND

Electron Ions: both
Use MSn Info: no
Isotope Res: 10000
Max Results: 500

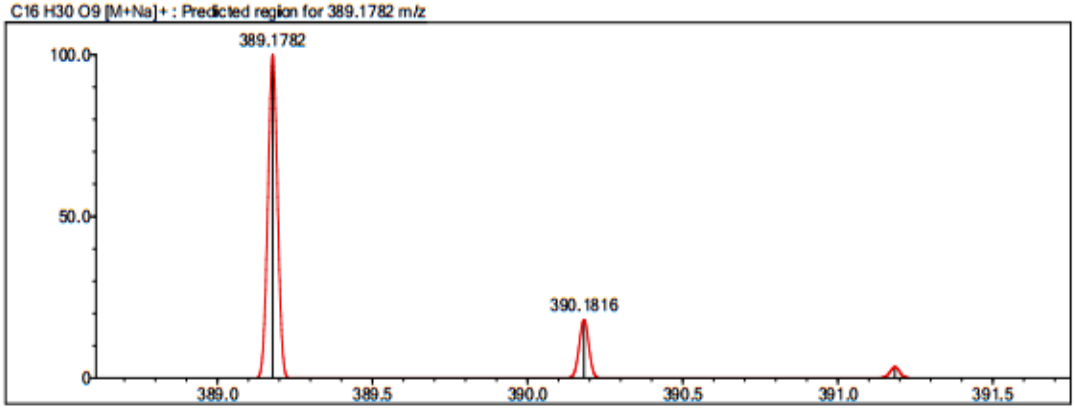
Event#: 1 MS(E+) Ret. Time : 0.430 Scan# : 87




Measured region for 389.1744 m/z



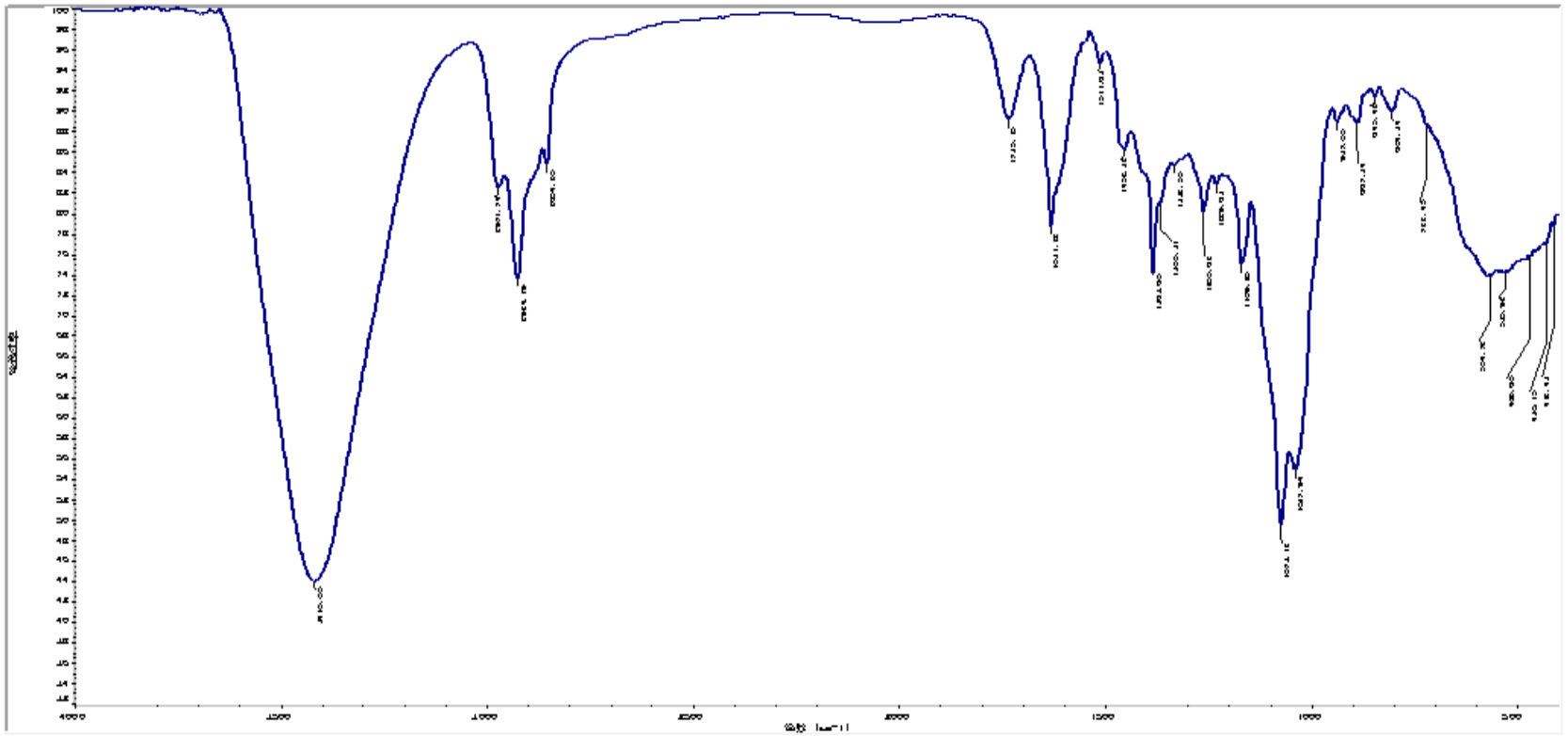

C16 H30 O9 [M+Na]+ : Predicted region for 389.1782 m/z




| Rank | Score | Formula (M) | Ion | Meas. m/z | Pred. m/z | Df. (mDa) | Df. (ppm) | Iso | DBE |
|---|---|---|---|---|---|---|---|---|---|
| 3 | 21.69 | C16 H30 O9 | [M+Na]+ | 389.1744 | 389.1782 | -3.8 | -9.76 | 51.15 | 2.0 |

The HRESI spectrum of compound **5**

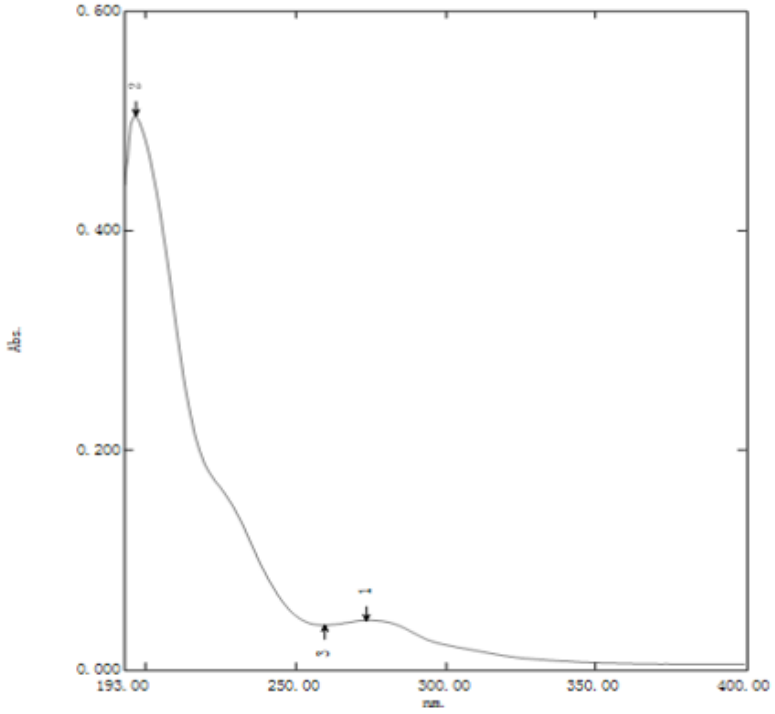
The IR (KBr) spectrum of compound **5**



Sample Name: DBJ-209B
KBr压片
采集时间: 星期四 10月 22 16:24:04 2020 (GMT+08:00)(Measured on)
仪器型号: NICOLET iS10(Instrument)
Software version: OMNIC 9.8.372

(Sample scan) 样品扫描次数: 16
背景扫描次数: 16
(Resolution) 分辨率: 4.000
采样增益: 1.0
动镜速度: 0.4747
(Acquisition) 光阑: 80.00

The UV spectrum of compound **5**




[测定属性]
波长范围(nm): 190.00 到 600.00
扫描速度: 中速
采样间隔: 0.5
自动采样间隔: 停用
扫描模式: 单个

[仪器属性]
仪器类型: UV-2700 系列
测定方式: 吸收值
狭缝宽: 5.0 nm
积分时间: 0.1 秒
光源转换波长: 323.0 nm
检测器单元: 直接
S/R 转换: 标准
阶梯校正: OFF

[附件属性]
附件: 无

[数据处理参数]
阈值: 0.0010000
点: 4
内插: 停用
平均: 停用

[样品准备属性]
重量:
体积:
稀释:
光程长: 10mm
附加信息: 样品浓度：0.0432毫克/毫升
溶剂：甲醇

Wavelength Range:190-600
Scan Speed: Medium
Sampling Interval:0.5
Auto Sampling Interval: Enabled
Scan Mode: Auto

Instrument Properties
Instrument Type: UV-2700PC Series
Measuring Mode: Absorbance
Slit Width: 5.0 nm
Light Source Change Wavelength: 325.0 nm
S/R Exchange: Normal

Sample concentration: 0.0432 mg/mL
Solvent: MeOH

| No. | P/V | 波长(nm) | Abs. | 描述 |
|---|---|---|---|---|
| 1 | ↑ | 273.50 | 0.045 | |
| 2 | ↑ | 196.50 | 0.505 | |
| 3 | ↓ | 259.50 | 0.040 | |

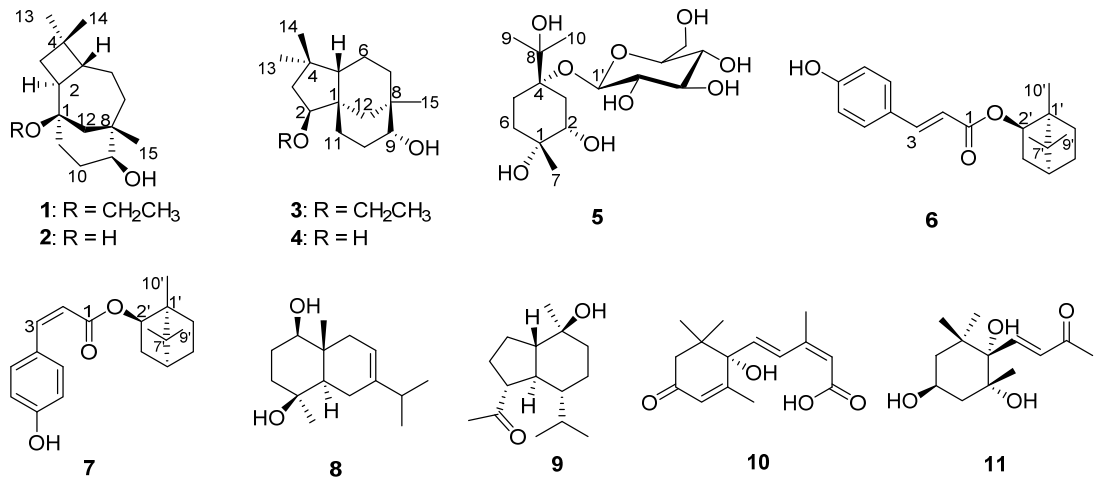



Fig.1. The structure of compounds **1-11**

**Caryolane-1, 9β-diol (2):** White amorphous powder; $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 3.43 (1H, t, *J* = 4.0 Hz, H-9), 2.22 (1H, m, H-2), 2.04 (1H, m, H-10a), 1.88 (1H, m, H-5), 1.76 (1H, m, H-10b), 1.63 (1H, m, H-11a), 1.55 (1H, m, H-3a), 1.52 (1H, m, H-6a), 1.50 (1H, m, H-11b), 1.45 (1H, m, H-3b), 1.40 (3H, m, H-12, H-7a), 1.38 (1H, H-6b), 1.15 (1H, m, H-7b), 1.01 (3H, s, H-14), 1.00 (3H, s, H-13), 0.92 (3H. s, H-15); $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 70.7 (s, C-1), 38.0 (d, C-2), 34.0 (t, C-3), 35.1 (s, C-4), 43.9 (d, C-5), 20.4 (t, C-6), 35.4 (t, C-7), 39.3 (s, C-8), 72.3 (d, C-9), 28.2 (t, C-10), 33.4 (t, C-11), 42.4 (t, C-12), 20.8 (q, C-13), 30.5 (q, C-14), 26.6 (q, C-15); EI-MS *m/z*: 337 ($[M-H]^+$, 5), 286 (10), 150 (100), 149 (50), 69 (100)。

**Clovane-2α,9β-diol (4):** White amorphous powder; $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 3.80 (1H, dd, *J* = 5.6, 10.4 Hz, H-2), 3.33 (1H, br s, H-9), 1.99 (1H, m, H-10a), 1.72 (2H, m, H-11a, H-3a), 1.66 (1H, m, H-10b), 1.55 (1H, m, H-12a), 1.42 (3H, m, H-6a, H-7a, H-5), 1.31 (1H, m, H-6b), 1.12 (1H, m, H-11b), 1.09 (1H, m, H-3b), 1.04 (3H, s, H-14), 0.97 (3H, s, H-15), 0.93 (1H, m, H-12b), 0.86 (3H, s, H-13); $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 44.1 (s, C-1), 80.8 (d, C-2), 47.3 (t, C-3), 37.1 (s, C-4), 50.5 (d, C-5), 20.6 (t, C-6), 33.0 (t, C-7), 34.7 (s,

C-8), 75.2 (d, C-9), 26.3 (t, C-10), 26.7 (t, C-11), 35.5 (t, C-12), 25.4 (q, C-13), 31.4 (q, C-14), 28.4 (q, C-15)。

(-)-Bornyl p-coumarate (**6**): White amorphous powder; $^{1}$H-NMR (600 MHz, $CD_3OD$) δ: 7.62 (1H, d, *J* = 15.9 Hz, H-2), 7.49 (2H, d, *J* = 8.6 Hz, H-5, H-9), 6.82 (2H, d, *J* = 8.6 Hz, H-6, H-8), 6.36 (1H, d, *J* = 15.9 Hz, H-3), 5.01 (1H, m H-2'), 2.43 (1H, m, H-3'b), 2.11 (1H, m, H-6'b), 1.84 (1H, m, H-5'b), 1.72 (1H, t, *J* = 4.5 Hz, H-4'), 1.42 (1H, m, H-6'a), 1.33 (1H, m, H-5'a), 1.06 (1H, dd, *J* = 13.7, 3.6 Hz, H-3'a), 0.99 (3H, s, 9'-$CH_3$), 0.95 (3H, s, 8'-$CH_3$), 0.91 (3H, s, 10'-$CH_3$);$^{13}$C-NMR (150 MHz, $CD_3OD$) δ: 168.3 (s, C-1), 159.9 (s, C-7), 144.9 (d, C-3), 129.8 (d, C-5, C-9), 125.8 (s, C-4), 115.4 (d, C-2), 114.1 (d, C-6, C-8), 79.7 (d, C-2'), 48.5 (s, C-1'), 47.5 (s, C-7'), 44.9 (d, C-4'), 36.5 (t, C-3'), 27.6 (t, C-5'), 26.8 (t, C-6'), 18.7 (q, C-8'), 17.8 (q, C-9'), 12.5 (q, C-10')。

(-)-Bornyl cis-4-hydroxycinnamate **(7):** White amorphous powder; $^{1}$H-NMR (600 MHz, $CD_3OD$) δ: 7.58 (2H, d, *J* = 8.6 Hz, H-5, H-9), 6.89 (1H, d, *J* = 12.7 Hz, H-2), 6.76 (2H, d, *J* = 8.7 Hz, H-6, H-8), 5.81 (1H, d, *J* = 12.7 Hz, H-3), 4.94 (1H, m, H-2'), 2.41 (1H, m, H-3'b), 1.86 (1H, m, H-6'b), 1.77 (1H, m, H-5'b), 1.69 (1H, t, *J* = 4.5 Hz, H-4'), 1.04 (1H, dd, *J* =13.7, 3.6 Hz, H-3'a), 1.29 (1H, m, H-6'a), 1.22 (1H, m, H-5'a), 0.96 (3H, s, 9'-$CH_3$), 0.92 (3H, s, 8'-$CH_3$), 0.82 (3H, s, 10'-$CH_3$);$^{13}$C-NMR (150 MHz, $CD_3OD$) δ: 167.6 (s, C-1), 158.5 (s, C-7), 143.0 (d, C-3), 131.9 (d, C-5, C-9), 126.4 (s, C-4), 116.1 (d, C-2), 114.5 (d, C-6, C-8), 79.8 (d, C-2'), 48.3 (s, C-1'), 47.6 (s, C-7'), 44.9 (d, C-4'), 36.3 (t, C-3'), 27.5 (t, C-5'), 26.7 (t, C-6'), 18.7 (q, C-8'), 17.8 (q, C-9'),12.5 (q, C-10')。

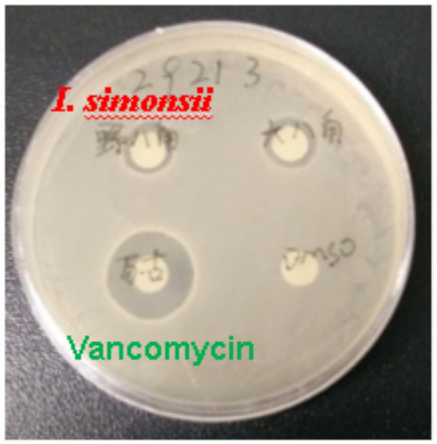
**Oplodiol (8):** White amorphous powder; $^{1}$H-NMR (500 MHz, $CDCl_3$) δ: 5.63 (1H, d, *J* = 5.0 Hz, H-8), 3.32 (1H, dd, *J* = 11.5, 4.0 Hz, H-1), 2.22 (1H, m, H-11), 2.09 (1H, m, H-2a), 2.06 (2H, m, H-6), 1.89 (1H, m, H-9a), 1.86 (1H, m, H-2b), 1.76 (1H, m, H-3a), 1.63 (1H, m, H-9b), 1.59 (1H, m, H-3b), 1.32 (1H, m, H-5), 1.20 (3H, s, H-15), 1.05 (3H, d, *J* = 6.5 Hz, H-12), 1.04 (3H, d, *J* = 6.5 Hz, H-13), 0.98 (3H, s, H-14); $^{13}$C-NMR (125 MHz, $CDCl_3$) δ: 79.7 (d, C-1), 40.7 (t, C-2), 39.4 (t, C-3), 71.0 (s, C-4), 46.2 (d, C-5), 23.0 (t, C-6), 141.9 (s, C-7), 116.1 (d, C-8), 26.9 (t, C-9), 37.7 (s, C-10), 35.0 (d, C-11), 21.8 (q, C-12), 21.2 (q, C-13), 11.7 (q, C-14), 19.8 (q, C-15)。

**Ent-oplopanone (9)**: White amorphous powder; $^{1}$H-NMR (400 MHz, $CDCl_3$) δ: 2.64 (1H, m, H-4), 2.19 (3H, s, H-14), 1.20 (3H, s,

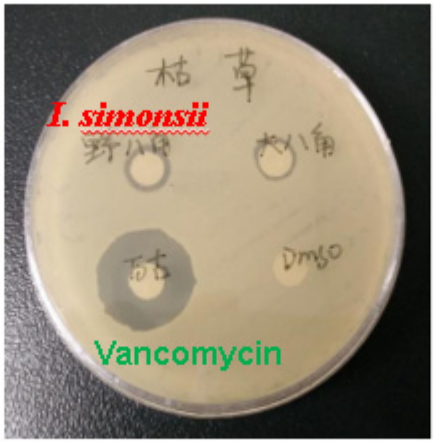
H-15), 0.89 (3H, d, *J* = 6.7 Hz, H-11), 0.68 (3H, d, *J* = 6.7 Hz, H-12); $^{13}$C-NMR (100 MHz, $CDCl_3$) δ: 46.7 (d, C-1), 42.0 (t, C-2), 28.6 (t, C-3), 57.0 (d, C-4), 211.5 (s, C-5), 55.7 (d, C-6), 49.4 (d, C-7), 25.3 (t, C-8), 23.0 (t, C-9), 73.0 (s, C-10), 29.5 (d, C-11), 15.6 (q, C-12), 22.0 (q, C-13), 20.3 (q, C-14), 29.5 (q, C-15)。

**Abscisic acid (10):** White amorphous powder; $^{1}$H-NMR (600 MHz, $CD_3OD$) δ: 7.77 (1H, d, *J* = 16.1 Hz, H-4), 6.24 (1H, d, *J* = 16.1 Hz, H-5), 5.92 (1H, s, H-3′), 5.74 (1H, s, H-2), 2.53 (1H, d, *J* = 17.0 Hz, H-5′a), 2.18 (1H, d, *J* = 17.0, H-5′b), 2.04 (3H, s, H-6), 1.93 (3H, s, H-7′), 1.06 (3H, s, H-8′), 1.03 (3H, s, H-9′); $^{13}$C-NMR (150 MHz, $CD_3OD$) δ:168.0 (s, C-1), 126.2 (d, C-2), 149.7 (s, C-3), 128.0 (d, C-4), 136.6 (d, C-5), 20.0 (q, C-6),79.2 (s, C-1′),165.1 (s, C-2′), 118.2 (d, C-3′), 199.8 (s, C-4′), 49.3 (t, C-5′), 41.5 (s, C-6′), 18.3 (q, C-7′), 22.2 (q, C-8′), 23.3 (q, C-9′).

**(3S,5R,6S,7E)-3,5,6-trihydroxy-7-megastigmen-9-one (11):** White amorphous powder; $^{1}$H-NMR (400 MHz, $CD_3OD$) δ:7.17 (1H, d, *J* = 15.8 Hz, H-8), 6.17 (1H, d, *J* = 15.7 Hz, H-8), 3.76 (1H, m, H-3), 2.40-2.57 (1H, m, H-4a), 2.29 (3H, s, H-10), 1.64-1.68 (1H, m, H-4b), 1.55-1.59 (1H, m, H-2a), 1.23-1.27 (1H, m, H-2b), 1.19 (3H, s, H-12), 1.18 (3H, s, H-13), 0.95 (3H, s, H-11); $^{13}$C-NMR (100 MHz, $CDCl_3$) δ:$^{13}$C-NMR (100 MHz, $CD_3OD$) δ: 34.7 (s, C-1), 46.3 (t, C-2), 63.0 (d, C-3), 39.9 (t, C-4), 67.4 (s, C-5), 69.5 (d, C-6), 144.0 (d, C-7), 132.4 (d, C-8), 198.8 (s, C-9), 26.0 (q, C-10), 23.8 (q, C-11), 128.4 (q, C-12), 8.7 (q, C-13).




*S. aureus* ATCC29213




*B. subtilis* ATCC6633


*E. faecalis* ATCC29212