

Bioactive Glycosides from the Twigs of *Litsea cubeba*

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Supplementary Information

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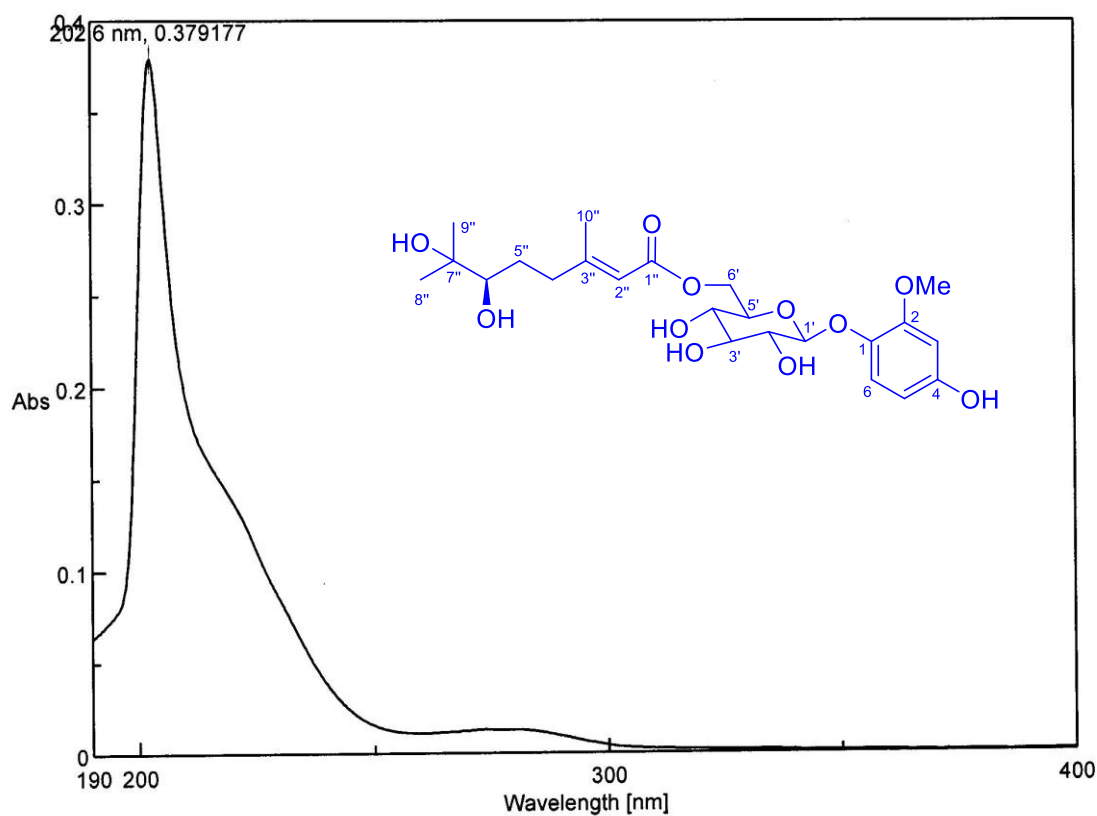
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Table S1. Crystal data of and structure refinement for Compound **1a**

| | |
|-----------------------------------|--|
| Empirical formula | C ₁₀ H ₁₈ O ₄ |
| Formula weight | 202.24 |
| Temperature | 104.2 K |
| Wavelength | 1.5418 Å |
| Crystal system | Orthorhombic |
| space group | P2 ₁ 2 ₁ 2 |
| Unit cell dimensions | a=19.2960(8) Å α = 90 ° b= 8.0943(4) Å β = 90 ° c= 6.9478(3) Å γ = 90 ° |
| Volume | 1085.15(9) Å ³ |
| Z | 4 |
| Calculated density | 1.238 Mg/m ³ |
| Absorption coefficient | 0.784 mm ⁻¹ |
| F(000) | 440 |
| Crystal size | 0.55 × 0.25 × 0.06 mm ³ |
| Theta range for data collection | 9.16 to 142.22 ° |
| Limiting indices | -23 ≤ h ≤ 20 -9 ≤ k ≤ 8 -8 ≤ l ≤ 8 |
| Reflections collected / unique | 3546/2042 [R(int) = 0.0248] |
| Completeness | 0.982 |
| Data / restraints / parameters | 2042/0/133 |
| Goodness-of-fit on F ² | 1.066 |
| Final R indices [I > 2σ(I)] | R ₁ = 0.0349, wR ₂ = 0.0907 |
| R indices (all data) | R ₁ = 0.0371, wR ₂ = 0.0931 |
| Absolute structure parameter | 0.0 (2) |



[Comment]
 Sample Name QYH-26
 Comment 0.02
 User
 Division UV
 Company 324
 [Measurement Information]
 Instrument Name V-650
 Model Name V-650
 Serial No. A034461150

Accessory PSC-718
 Accessory S/N A001761114
 Position 1
 Cell Length 10 mm
 Temperature 19.95 C
 Control Sensor Holder
 Monitor Sensor Holder
 Start Mode Start immediately

Photometric Mode Abs
 Measurement range 400 - 190 nm
 Data pitch 0.2 nm
 Band width(UV/Vis) 2.0 nm
 Response Medium
 Scanning speed 200 nm/min
 Source Change 340 nm
 Light Source D2/WI
 Filter Exchange Step
 Correction Baseline

[Data Information]
 Creation Date 2014-7-15 21:15

Data array type Linear data array
 Horizontal Wavelength [nm]
 Vertical Abs
 Start 400 nm
 End 190 nm
 Data pitch 0.2 nm
 Data points 1051

Figure S1. The UV Spectrum of Compound 1 in MeOH

Single Mass Spectrum Deconvolution Report

Analysis Name: linsh231.d

Method: TEST.MS

Sample Name: QYH-26

Analysis Info:

Instrument: LC-MSD-TraP-SL

Operator: Operator

Print Date: 3/25/2014 2:55:32 PM

Acq. Date: 3/25/2014 2:45:01 PM

Acquisition Parameter:

Mass Range Mode Std/Normal

| | |
|--------------|----------|
| Ion Polarity | Positive |
|--------------|----------|

Ion Source Type

Dry Temp (Set)

Nebulizer (Set)

Dry Gas (Set)

Trap Drive

Octopole RF Amplitude

Capillary Exit

Skimmer

Oct 1 DC

Oct 2 DC

45.5

152.8 V_{pp}

-102.3 Volt

-40.0 Volt

-12.00 Volt

-1.70 Volt

Scan Begin

Scan End

Averages

Max. Accu

ICC Target

Charge Cor

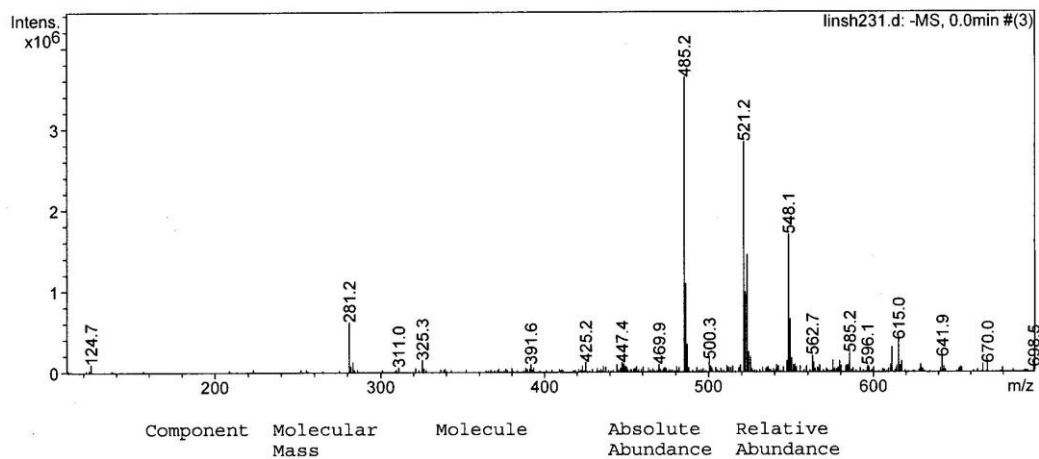
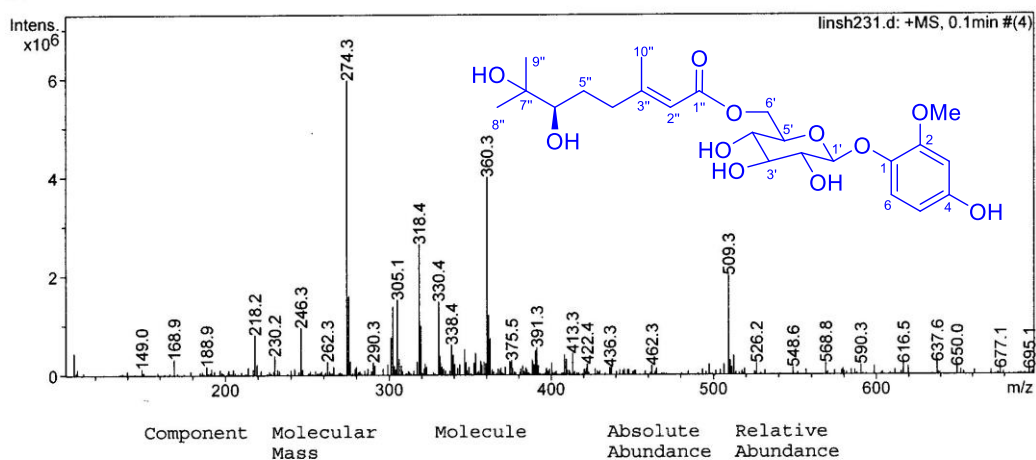
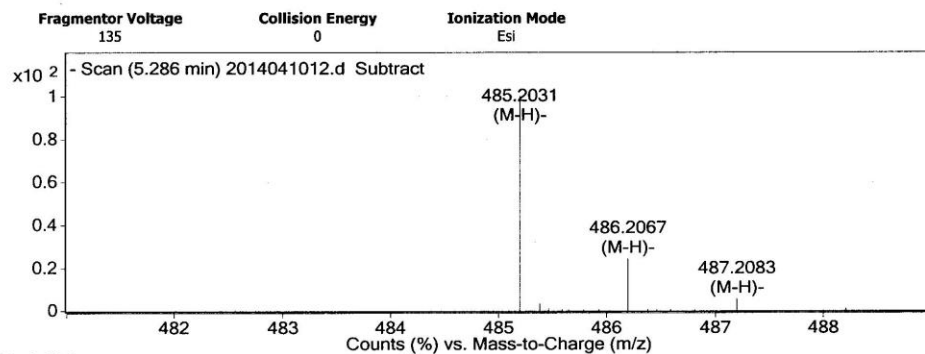


Figure S2. The ESIMS Spectrum of Compound 1

Qualitative Analysis Report



Peak List

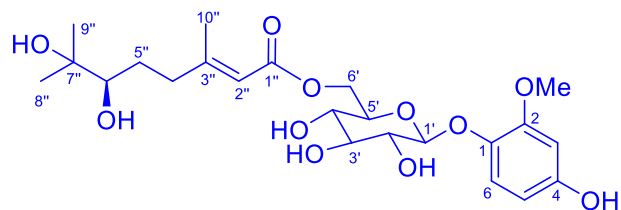
| m/z | z | Abund | Formula | Ion |
|-----------|---|--------|-------------|--------|
| 248.9603 | | 27511 | | |
| 384.9357 | | 25747 | | |
| 485.2031 | 1 | 349415 | C23 H33 O11 | (M-H)- |
| 486.2067 | 1 | 85909 | C23 H33 O11 | (M-H)- |
| 520.9102 | | 81085 | | |
| 553.1896 | | 45205 | | |
| 656.8851 | | 38193 | | |
| 792.8584 | | 25958 | | |
| 966.0001 | | 34307 | | |
| 1033.9882 | | 45086 | | |

Formula Calculator Element Limits

| Element | Min | Max |
|---------|-----|-----|
| C | 3 | 100 |
| H | 0 | 500 |
| O | 0 | 90 |
| N | 0 | 5 |
| S | 0 | 0 |
| Cl | 0 | 0 |
| Br | 0 | 0 |
| Si | 0 | 0 |
| F | 0 | 0 |
| P | 0 | 0 |

Formula Calculator Results

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---------------|------|----------|----------|------------|---------------|-------|
| C23 H34 O11 | TRUE | 486.2104 | 486.2101 | -0.62 | C23 H33 O11 | 99.94 |
| C24 H30 N4 O7 | | 486.2104 | 486.2114 | 2.12 | C24 H29 N4 O7 | 99.61 |



--- End Of Report ---

Figure S3. The HRESIMS Spectrum of Compound 1

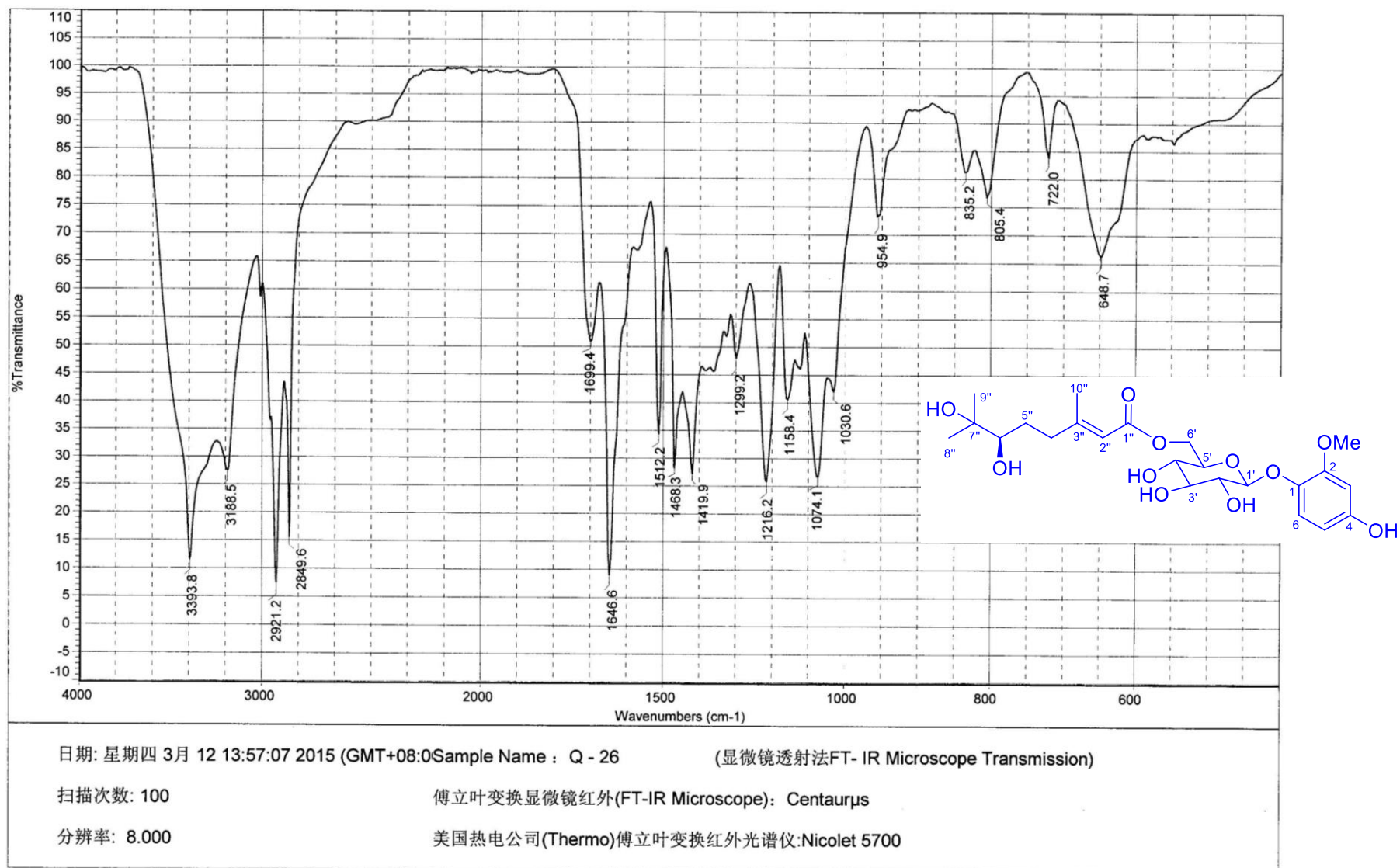


Figure S4. The IR Spectrum of Compound 1

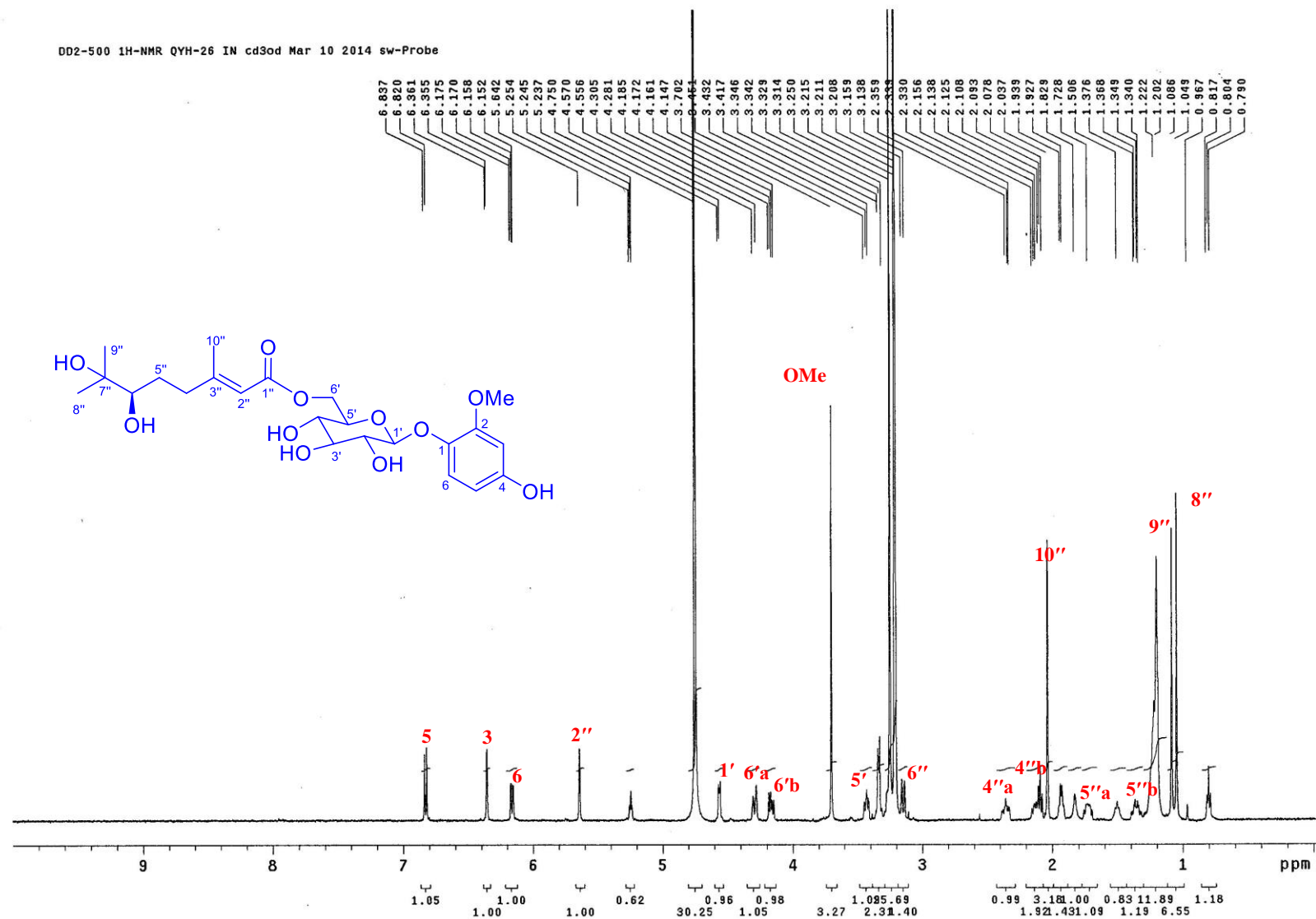


Figure S5. The ^1H NMR Spectrum of Compound 1 in $\text{MeOH-}d_4$ (500MHz)

DD2-500 13C-NMR QYH-26 IN cd3od Mar 11 2014 sw-Probe

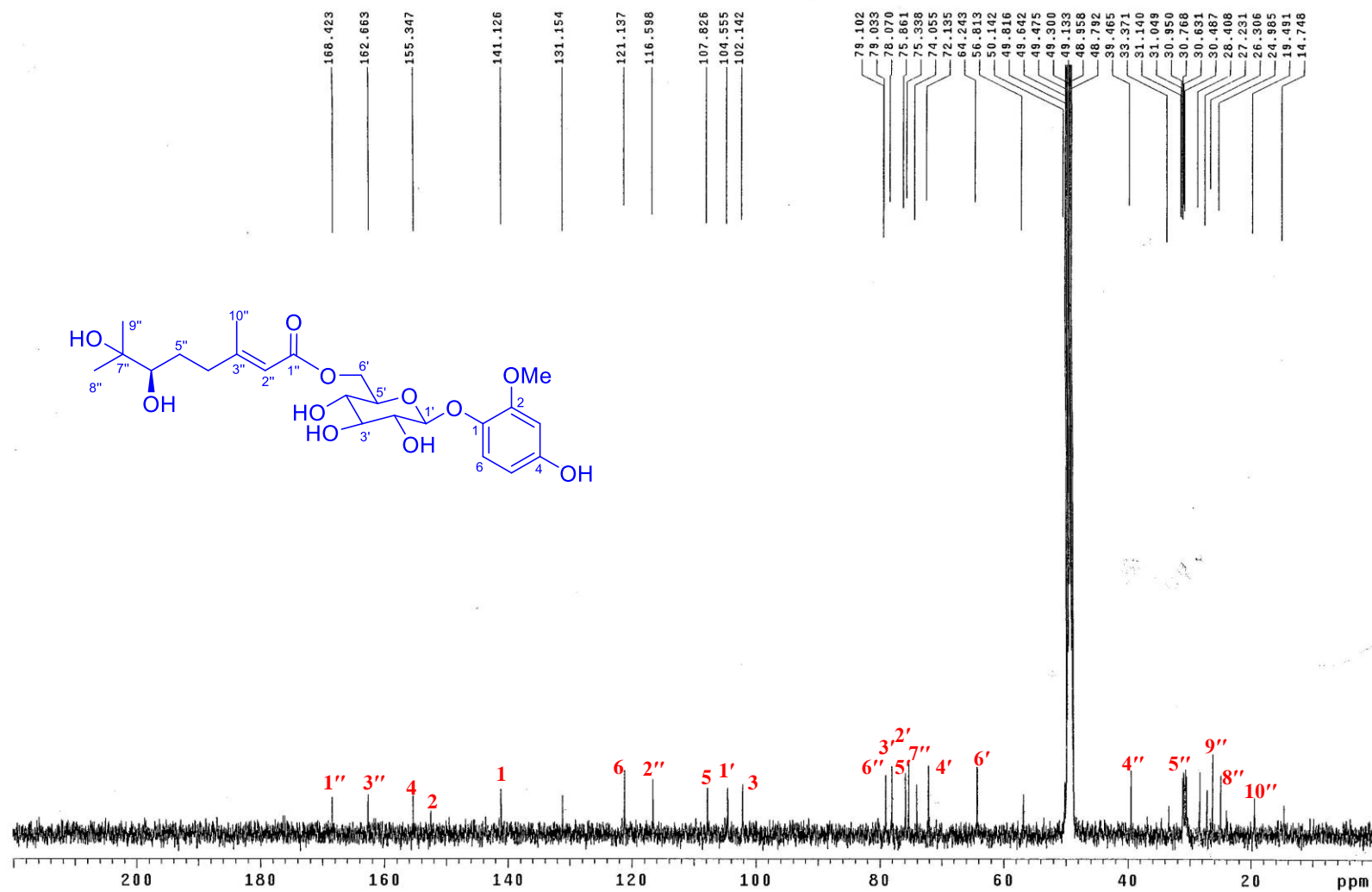


Figure S6. The ^{13}C NMR spectrum of compound 1 in $\text{MeOH-}d_4$ (125MHz)

DD2-500 gCOSY QYH-26 IN cd3od Apr 14 2014 sw

Temp. 25.0 C / 298.1 K
Sample #11, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 3882.0 Hz
2D Width 3882.0 Hz
4 repetitions
200 increments
OBSERVE H1, 499.7694214 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.025 sec
FT size 2048 x 2048
Total time 16 min

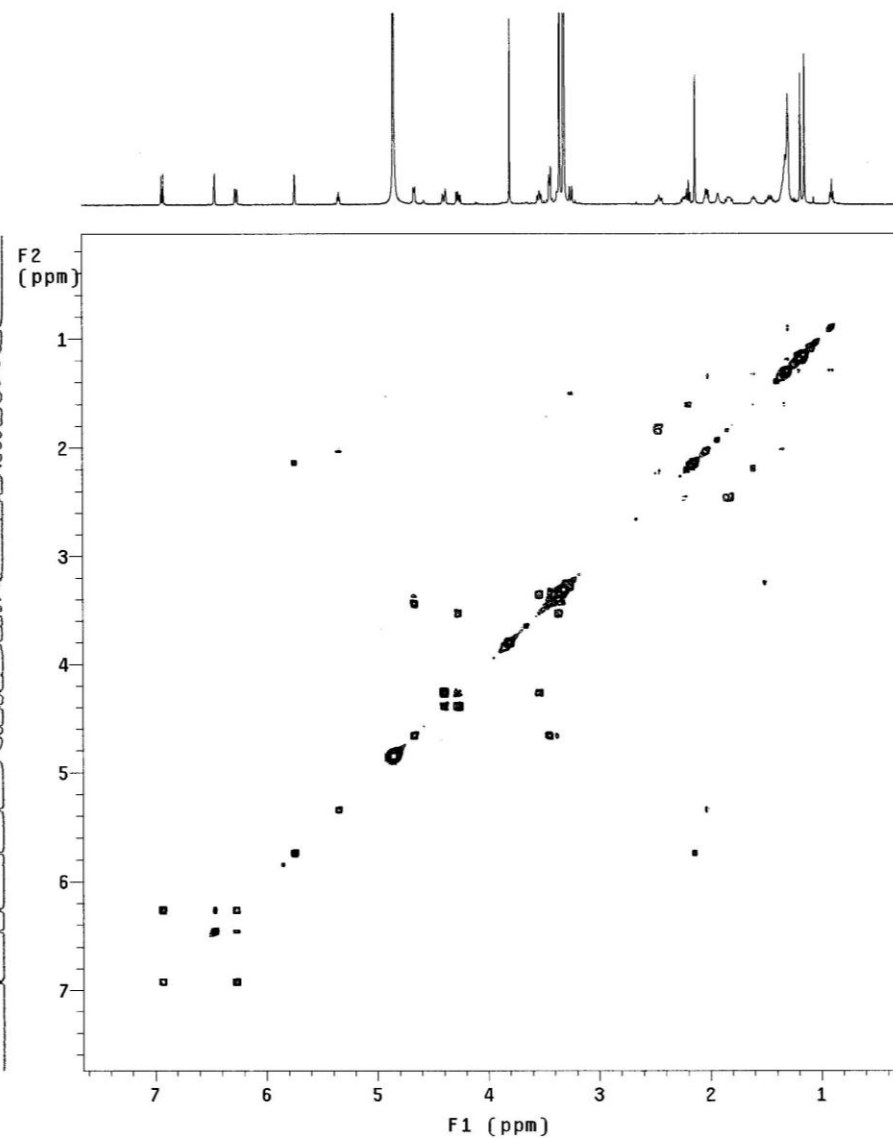
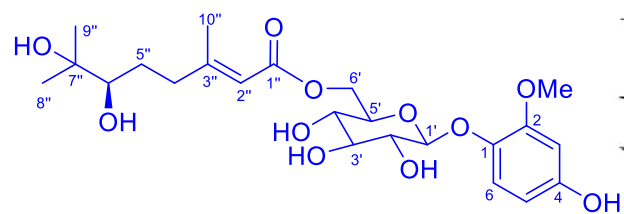


Figure S7. The ^1H - ^1H COSY Spectrum of Compound 1 in $\text{MeOH-}d_4$ (500MHz)

DD2-500 gHSQCAD QYH-76-1 IN cd3od Dec 25 2014 coldprobe

Temp. 25.0 C / 298.1 K
 Sample #5, Operator: vnmr1
 Relax. delay 1.000 sec
 Acq. time 0.262 sec
 Width 4595.6 Hz
 2D Width 25133.5 Hz
 8 repetitions
 2 x 200 increments
 OBSERVE H1, 499.7694110 MHz
 DECOUPLE C13, 125.6784284 MHz
 Power 38 dB
 on during acquisition
 off during delay
 W40_coldprobe modulated
 DATA PROCESSING
 Gauss apodization 0.069 sec
 F1 DATA PROCESSING
 Gauss apodization 0.007 sec
 FT size 4096 x 2048
 Total time 1 hr, 4 min

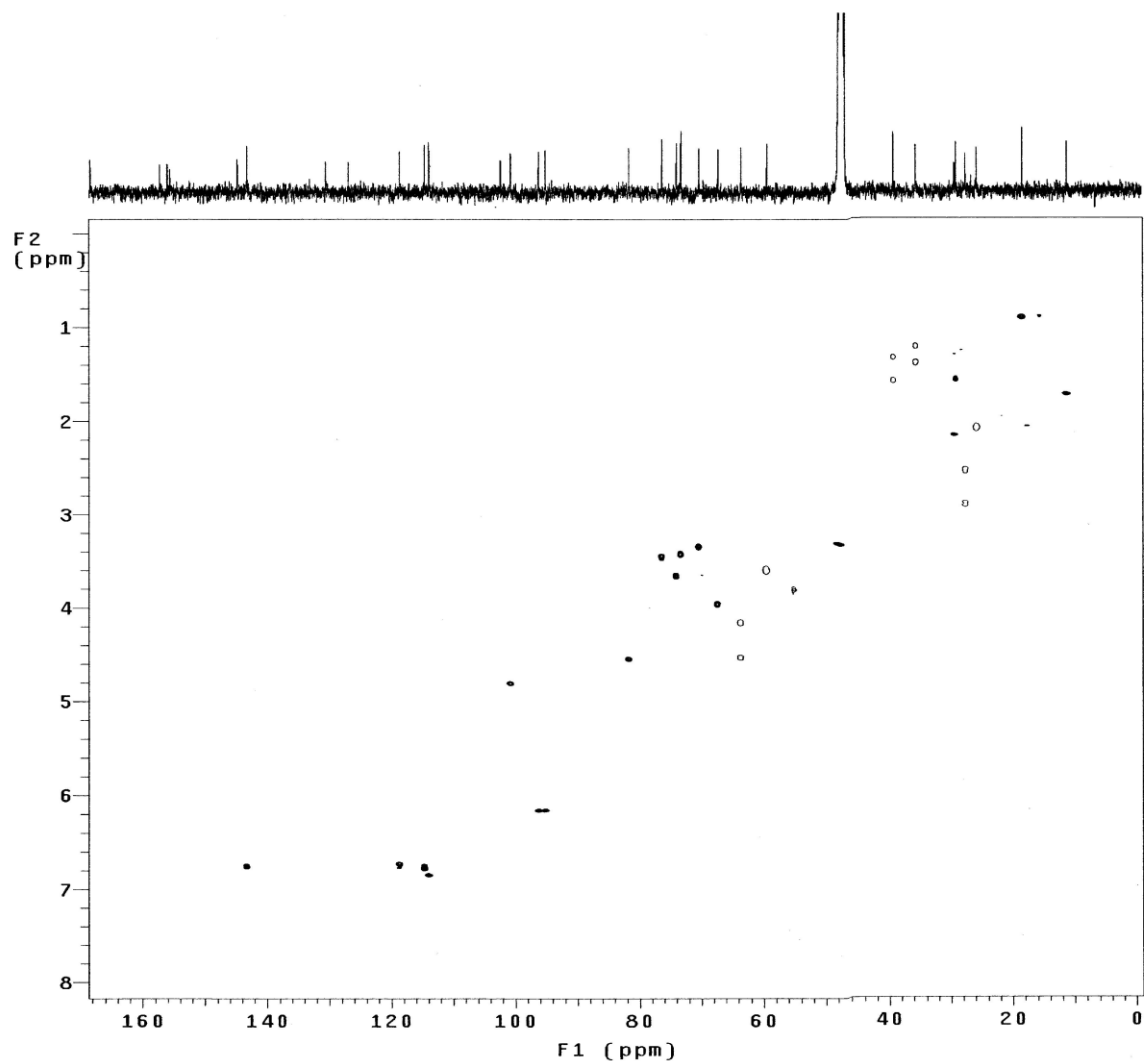
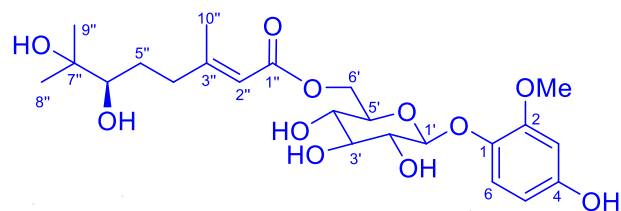


Figure S8. The HSQC Spectrum of Compound 1 in MeOH-*d*₄ (500MHz)

DD2-500 gHMBCAD QYH-26 IN cd3od Apr 14 2014 sw

Temp. 25.0 C / 298.1 K
Sample #11, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.310 sec
Width 3882.0 Hz
2D Width 30154.5 Hz
64 repetitions
2 x 160 increments
OBSERVE H1, 499.7694214 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Gauss apodization 0.005 sec
FT size 4096 x 2048
Total time 7 hr

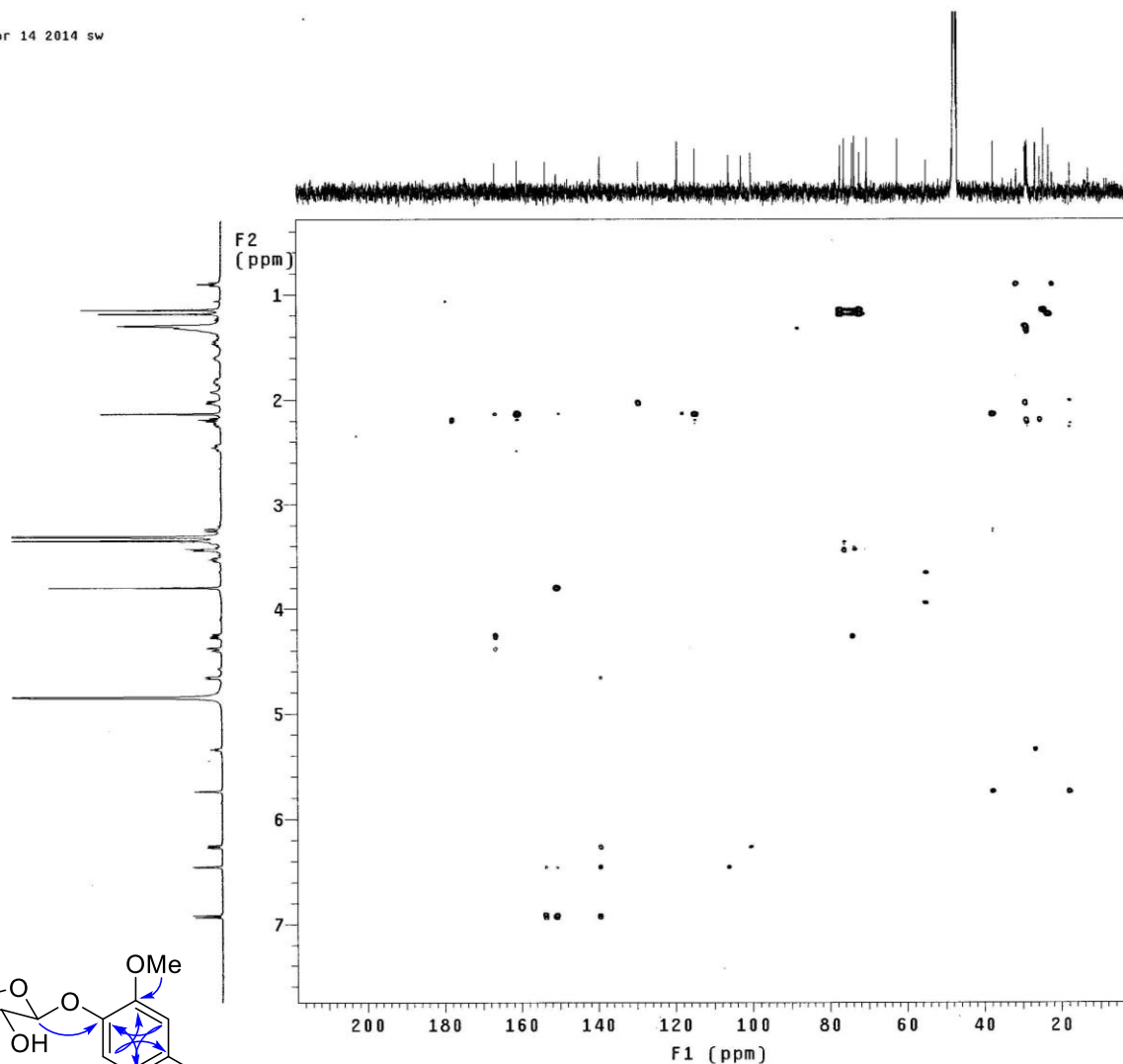
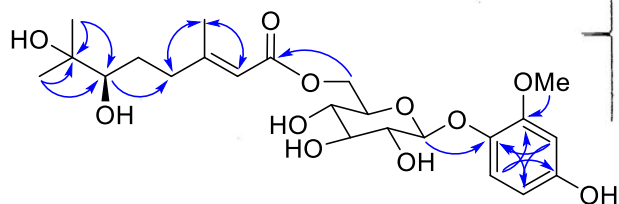
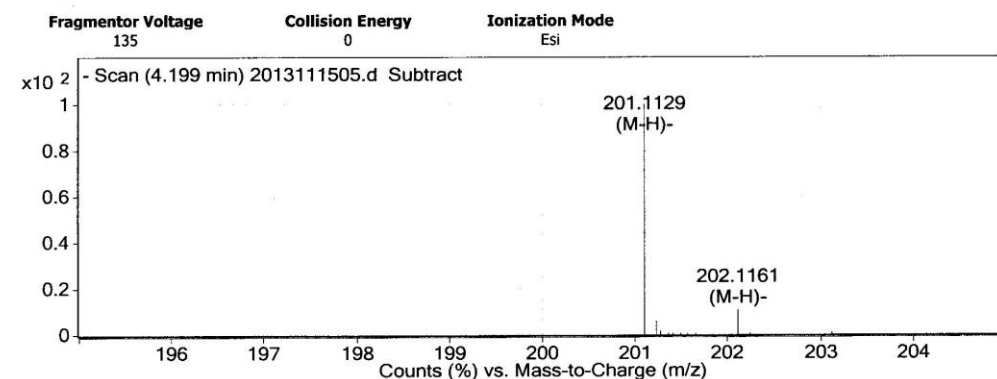


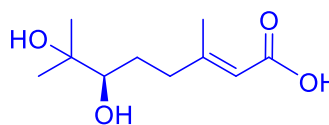
Figure S9. The HMBC Spectrum of Compound 1 in MeOH-*d*₄ (500MHz)

Qualitative Analysis Report



Peak List

| m/z | z | Abund | Formula | Ion |
|----------|---|---------|--|--------|
| 139.1124 | | 61174 | | |
| 201.1129 | 1 | 1183020 | C ₁₀ H ₁₇ O ₄ | (M-H)- |
| 201.2331 | | 69813 | | |
| 202.1161 | 1 | 130005 | C ₁₀ H ₁₇ O ₄ | (M-H)- |
| 247.1182 | | 90459 | | |
| 269.1001 | | 109661 | | |
| 403.2338 | | 248976 | | |



Formula Calculator Element Limits

| Element | Min | Max |
|---------|-----|-----|
| C | 3 | 100 |
| H | 0 | 500 |
| O | 0 | 90 |
| N | 0 | 5 |
| S | 0 | 5 |
| Cl | 0 | 2 |
| Br | 0 | 0 |
| Si | 0 | 0 |
| F | 0 | 0 |
| P | 0 | 0 |

Formula Calculator Results

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|--|------|----------|----------|------------|--|-------|
| C ₁₀ H ₁₈ O ₄ | TRUE | 202.1202 | 202.1205 | 1.68 | C ₁₀ H ₁₇ O ₄ | 99.95 |

--- End Of Report ---

Figure S10. The HRESIMS Spectrum of Compound 1a

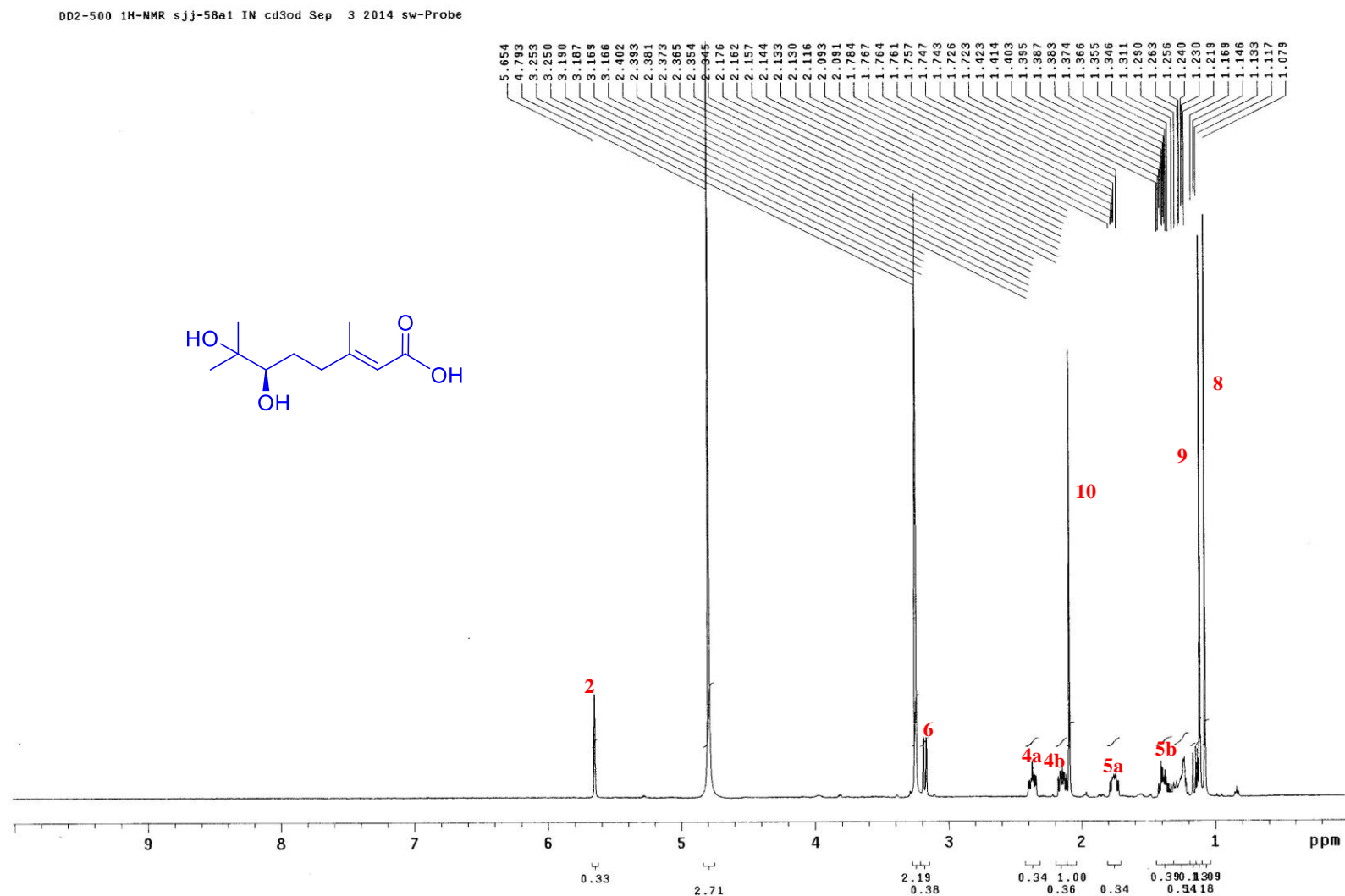


Figure S11. The ^1H NMR spectrum of compound 1a in $\text{MeOH-}d_4$ (500MHz)

DD2-500 13C-NMR sjj-58a IN cd3od Sep 3 2014 sw-Probe

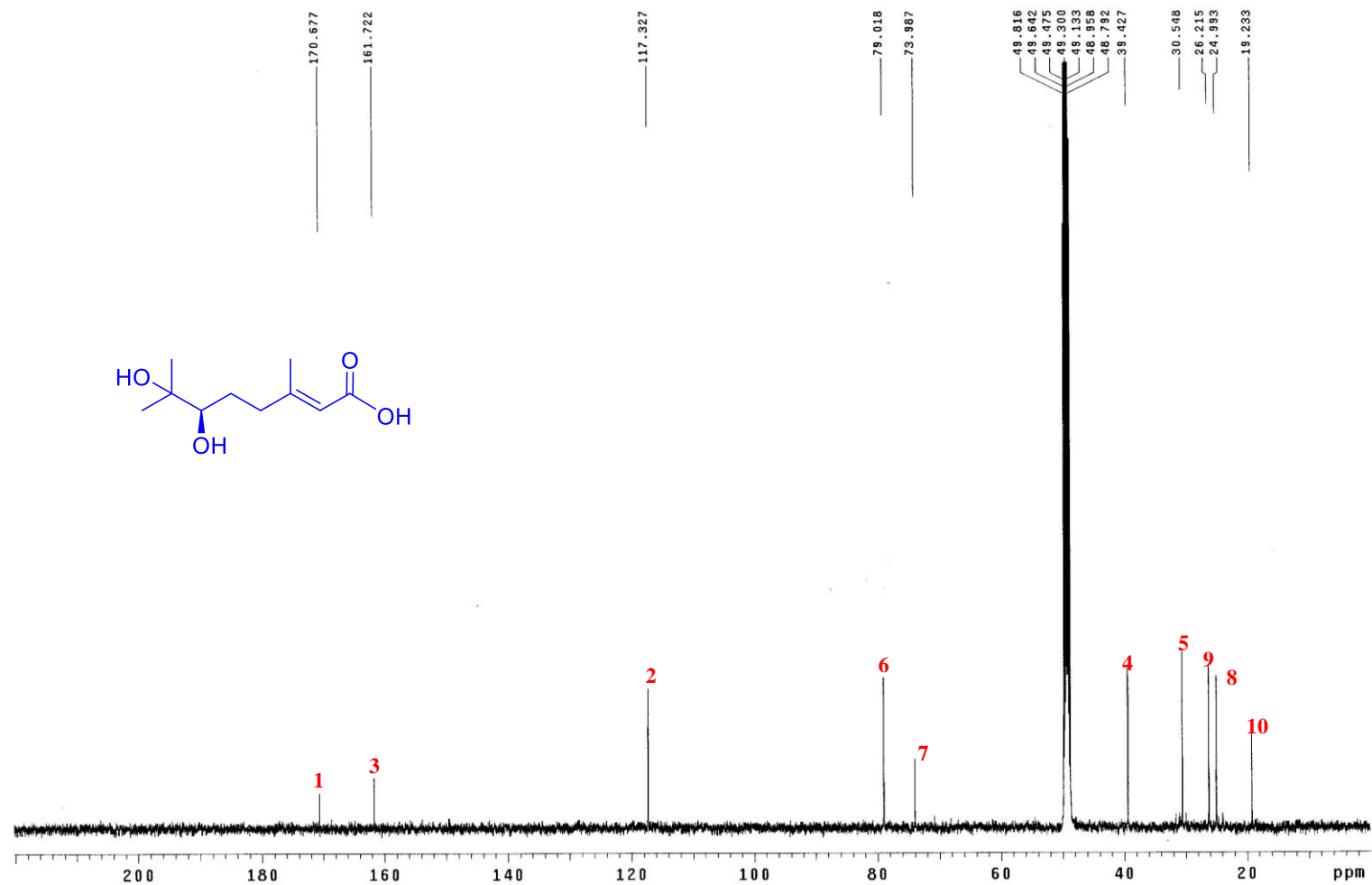
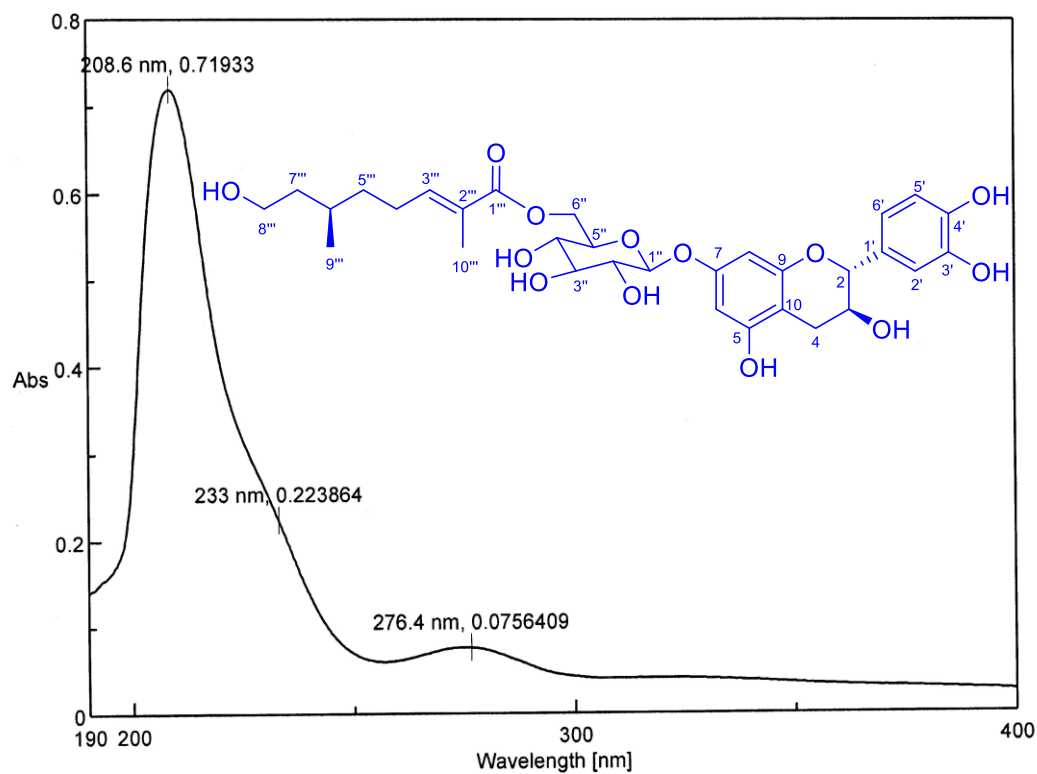


Figure S12. The ^{13}C NMR spectrum of compound 1a in $\text{MeOH-}d_4$ (125 MHz)



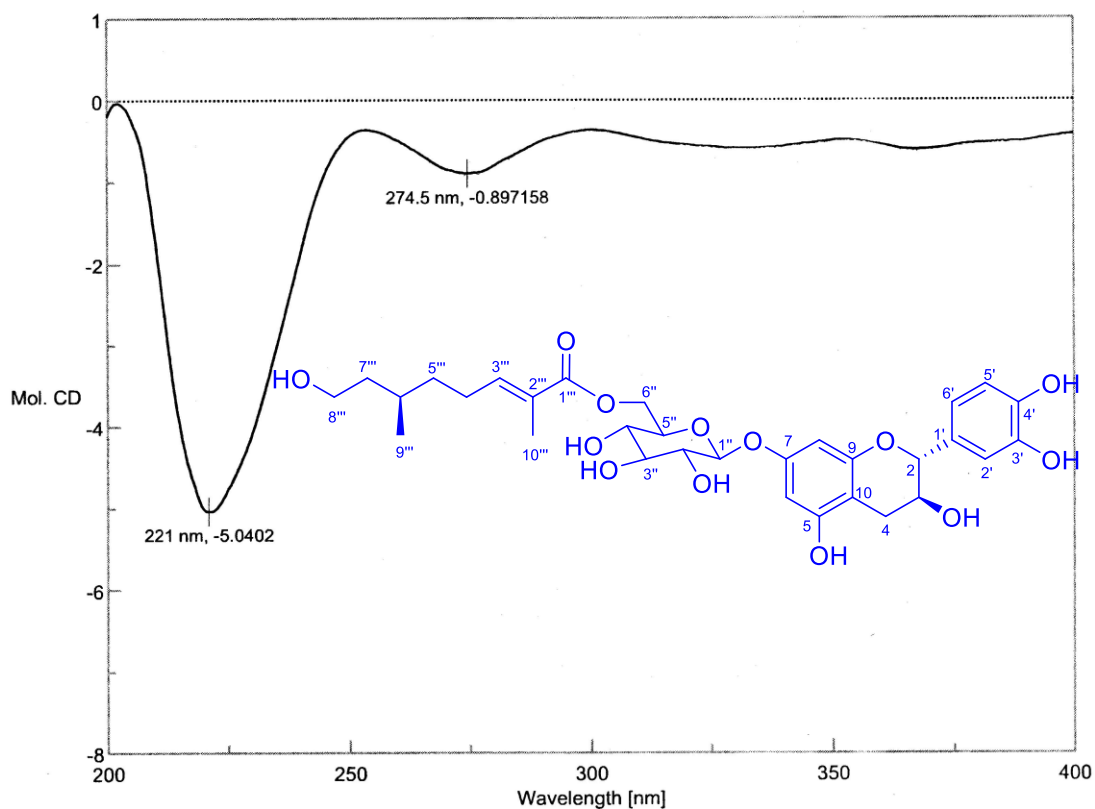
[Comment]
 Sample Name 76-1
 Comment 0.02
 User
 Division UV
 324
 [Measurement Information]
 Instrument Name V-650
 Model Name V-650
 Serial No. A034461150

Accessory PSC-718
 Accessory S/N A001761114
 Position 1
 Cell Length 10 mm
 Temperature 19.98 C
 Control Sensor Holder
 Monitor Sensor Holder
 Start Mode Start immediately

Photometric Mode Abs
 Measurement range 400 - 190 nm
 Data pitch 0.2 nm
 Band width(UV/Vis) 2.0 nm
 Response Medium
 Scanning speed 200 nm/min
 Source Change 340 nm
 Light Source D2/M1
 Filter Exchange Step
 Correction Baseline

[Data Information]
 Creation Date 2015-3-10 19:31
 Data array type Linear data array
 Horizontal Wavelength [nm]
 Vertical Abs
 Start 400 nm
 End 190 nm
 Data pitch 0.2 nm
 Data points 1051

Figure S13. The UV Spectrum of Compound 2



[Comments]
 Sample name QYH-76-1
 Comment
 User
 Division
 Company dell

[Measurement Information]
 Instrument Name J-815
 Model Name J-815
 Serial No. A024461168

Accessory Standard
 Accessory S/N A024461168
 Cell Length 1 mm

Photometric Mode CD, HT, Abs
 Measure Range 400 - 200 nm
 Data pitch 0.5 nm
 Sensitivity Standard
 D.I.T. 1 sec
 Band width 2.00 nm
 Start Mode Immediately
 Scanning Speed 100 nm/min
 Baseline Correction Baseline
 Shutter Control Auto
 PMT Voltage Auto
 Accumulations 2
 Solvent MEOH
 Concentration 0.192 (w/v)%

[Detailed Information]
 Creation date 2015-3-24 14:32

Data array type Linear data array * 3
 Horizontal axis Wavelength [nm]
 Vertical axis(1) Mol. CD
 Vertical axis(2) HT [V]
 Vertical axis(3) Abs
 Start 400 nm
 End 200 nm
 Data interval 0.5 nm
 Data points 401

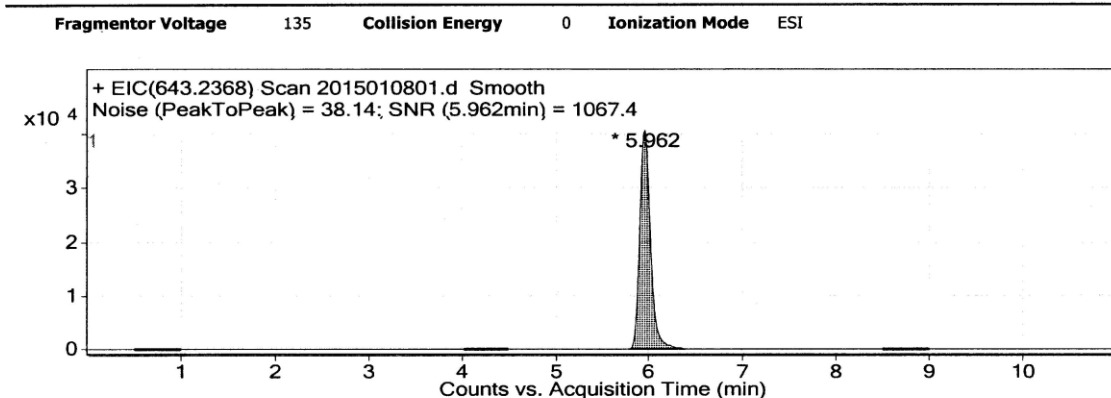
Figure S14. CD Spectrum of Compound 2 in MeOH

Qualitative Analysis Report

Data Filename 2015010801.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method
DA Method TEST LCMS.m

Sample Name QYH-76-1
Position P1-C2
User Name
IRM Calibration Status
Comment

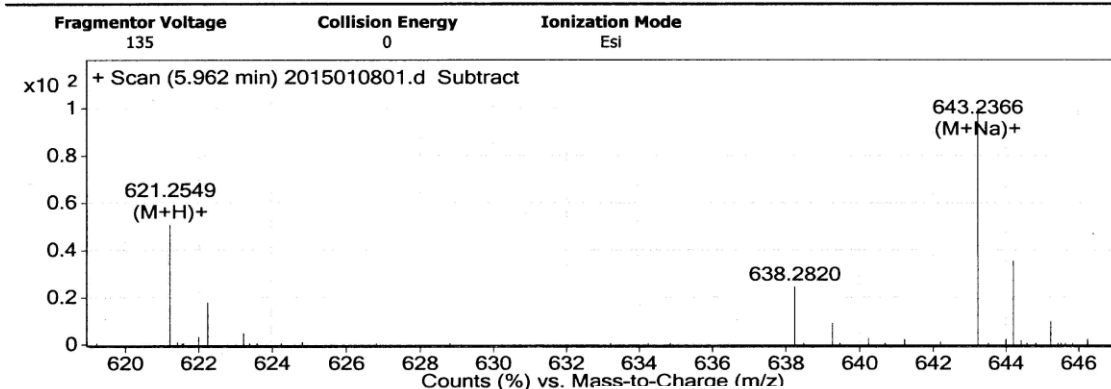
User Chromatograms



Integration Peak List

| Peak | Start | RT | End | Height | Area | Area % | Signal To Noise |
|------|-------|-------|-------|--------|--------|--------|-----------------|
| 1 | 5.785 | 5.962 | 6.397 | 40710 | 324002 | 100 | 1067.4 |

User Spectra



Peak List

| m/z | z | Abund | Formula | Ion |
|----------|---|-------|----------------|---------|
| 125.986 | | 36790 | | |
| 143.0326 | | 18347 | | |
| 158.9643 | | 19062 | | |
| 187.1263 | | 14522 | | |
| 469.206 | 1 | 14495 | | |
| 603.2436 | 1 | 12328 | | |
| 621.2549 | 1 | 29255 | C31 H41 O13 | (M+H)+ |
| 638.282 | 1 | 14140 | | |
| 643.2366 | 1 | 57739 | C31 H40 Na O13 | (M+Na)+ |
| 644.2394 | 1 | 20346 | C31 H40 Na O13 | (M+Na)+ |

Formula Calculator Element Limits

| Element | Min | Max |
|---------|-----|-----|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |

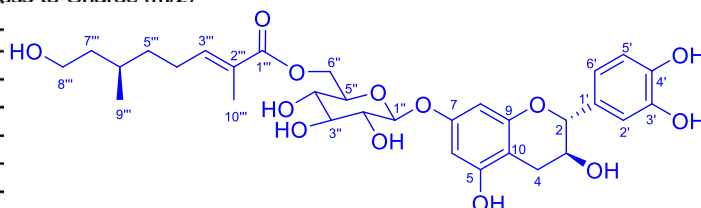
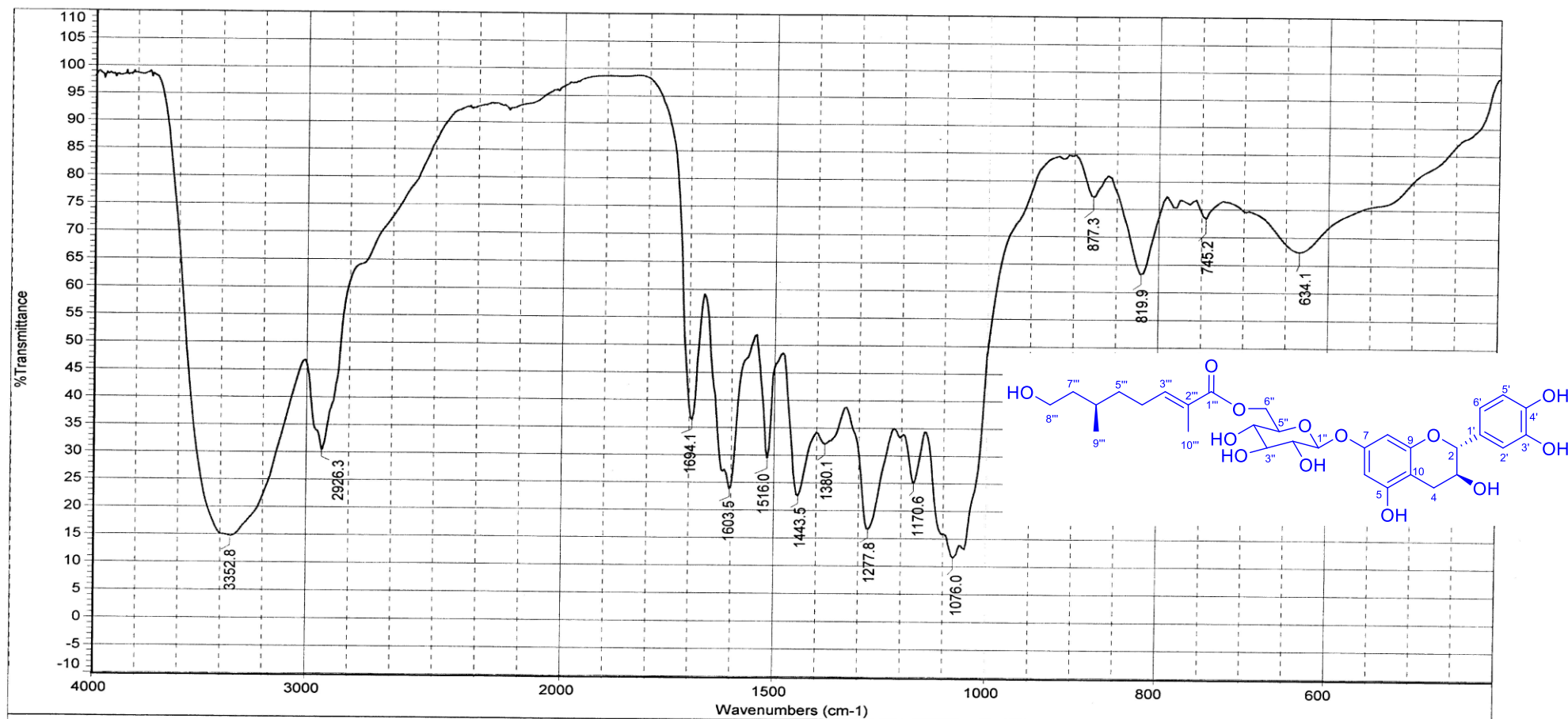


Figure S15. The HRESIMS Spectrum of Compound 2 in MeOH



日期: 星期四 3月 12 15:06:36 2015 (GMT+08:00) Sample Name : 76 - 1

(显微镜透射法FT- IR Microscope Transmission)

扫描次数: 100

傅立叶变换显微镜红外(FT-IR Microscope): Centaurus

分辨率: 8.000

美国热电公司(Thermo)傅立叶变换红外光谱仪:Nicolet 5700

Figure S16. The IR Spectrum of Compound 2

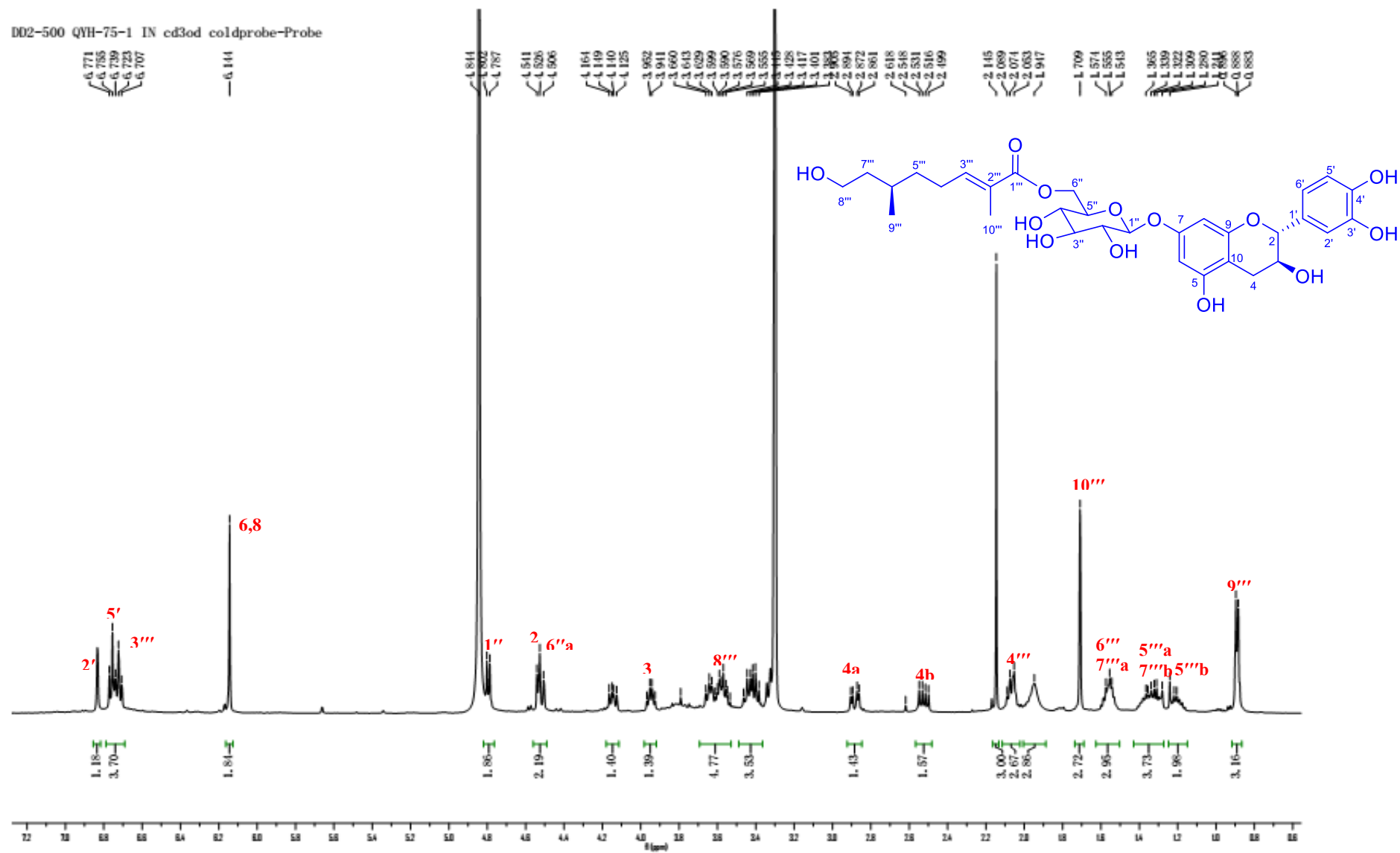


Figure S17. The ^1H NMR Spectrum of Compound 2 in $\text{MeOH-}d_4$ (500MHz)

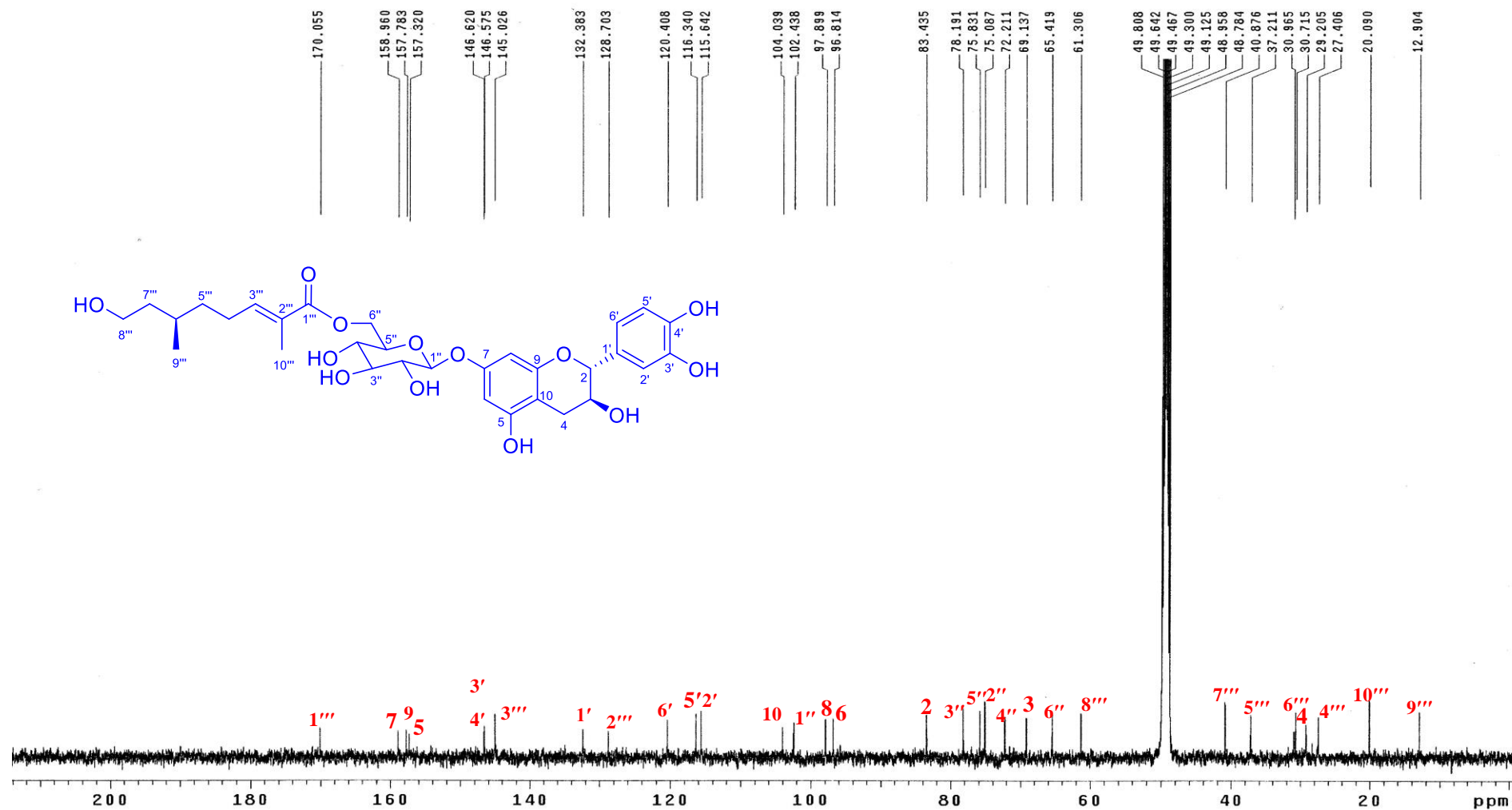


Figure S18. The ^{13}C NMR Spectrum of Compound 2 in $\text{MeOH-}d_4$ (125MHz)

DD2-500 gCOSY

Temp. 25.0 C
Sample #5, 0p

Relax. delay
Acq. time 0.
Width 459
2D Width 459
4 repetition
128 incremer

OBSERVE H1,
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.028 sec
FT size 2048 x 2048
Total time 10 min

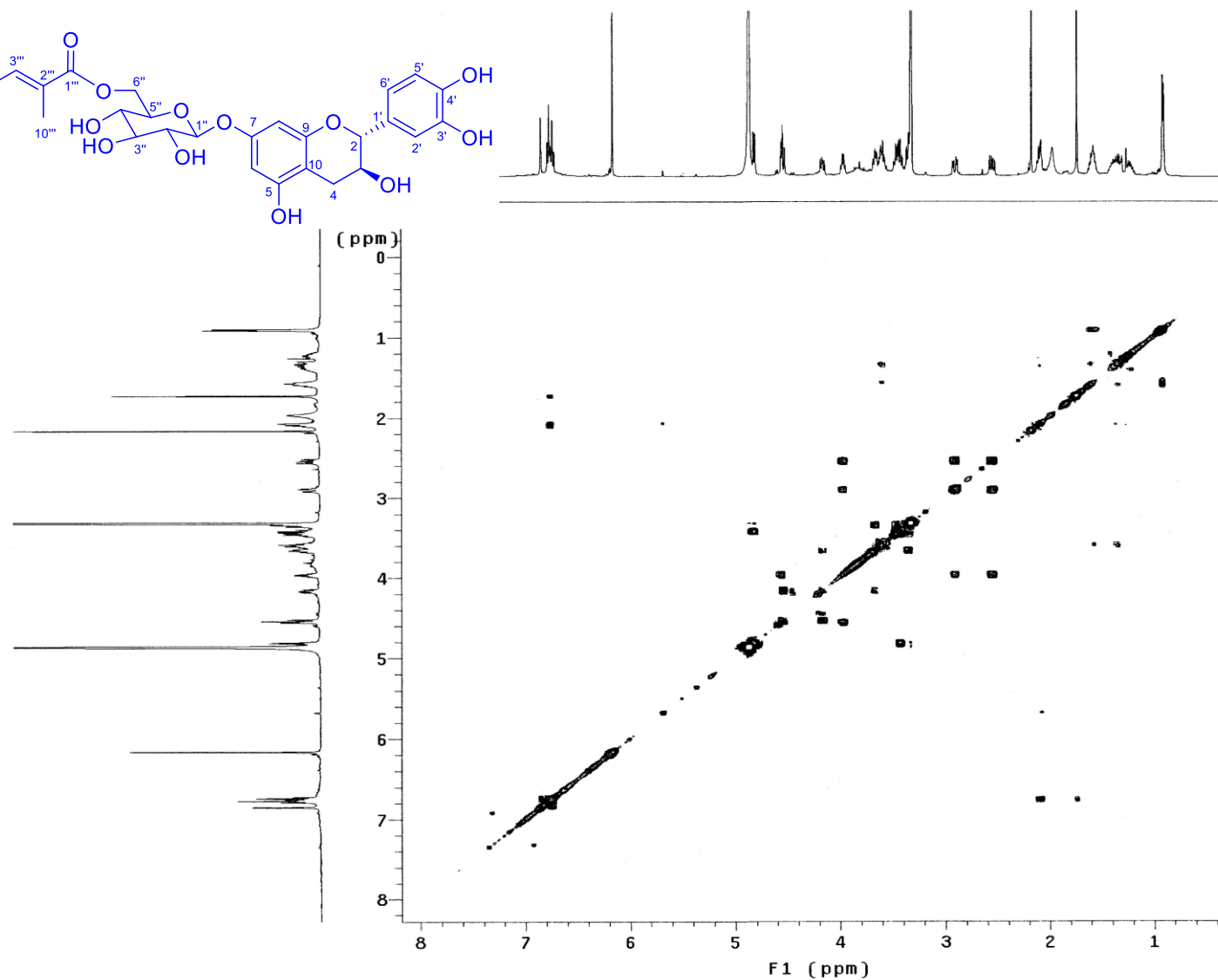
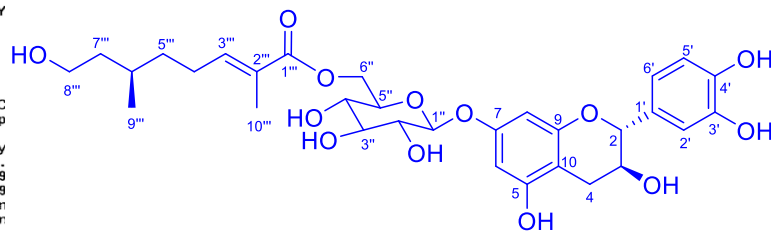


Figure S19. The ^1H - ^1H COSY Spectrum of Compound 2 in $\text{MeOH-}d_4$ (500MHz)

DD2-500 gHSQCAD QYH-76-1 IN cd3od Dec 25 2014 coldprobe

Temp. 25.0 C / 298.1 K
Sample #5, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.262 sec
Width 4595.6 Hz
2D Width 25133.5 Hz
8 repetitions
2 x 200 increments
OBSERVE H1, 499.7694110 MHz
DECOUPLE C13, 125.6784284 MHz
Power 38 dB
on during acquisition
off during delay
W40_coldprobe modulated
DATA PROCESSING
Gauss apodization 0.069 sec
F1 DATA PROCESSING
Gauss apodization 0.007 sec
FT size 4096 x 2048
Total time 1 hr, 4 min

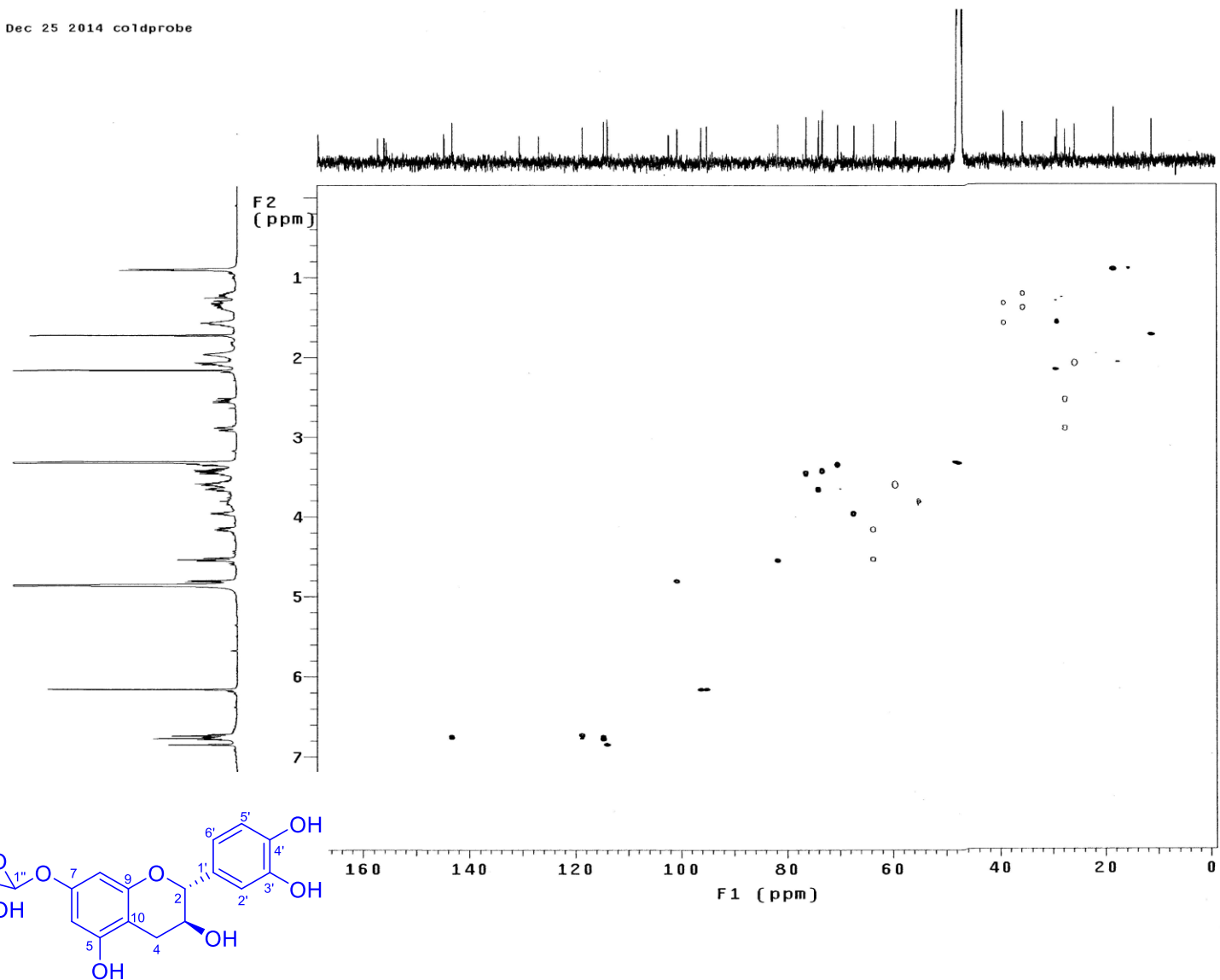


Figure S20. The HSQC Spectrum of Compound 2 in $\text{MeOH-}d_4$ (500MHz)

Temp. 25.0 C / 298.1 K
Sample #5, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.262 sec
Width 4595.6 Hz
2D Width 30154.5 Hz
16 repetitions
2 x 200 increments
OBSERVE H1, 499.7694110 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Gauss apodization 0.006 sec
FT size 4096 x 2048
Total time 2 hr, 12 min

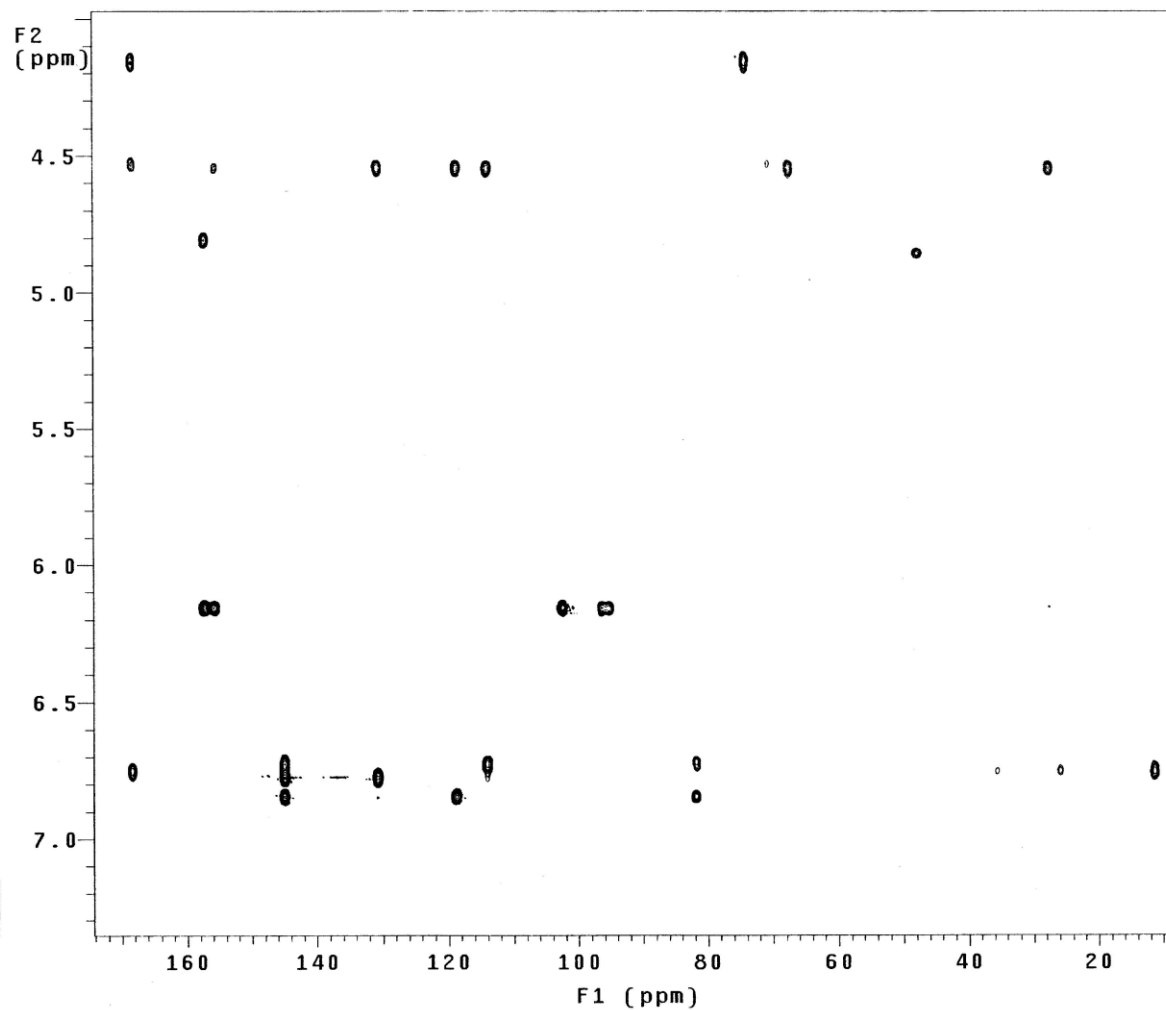
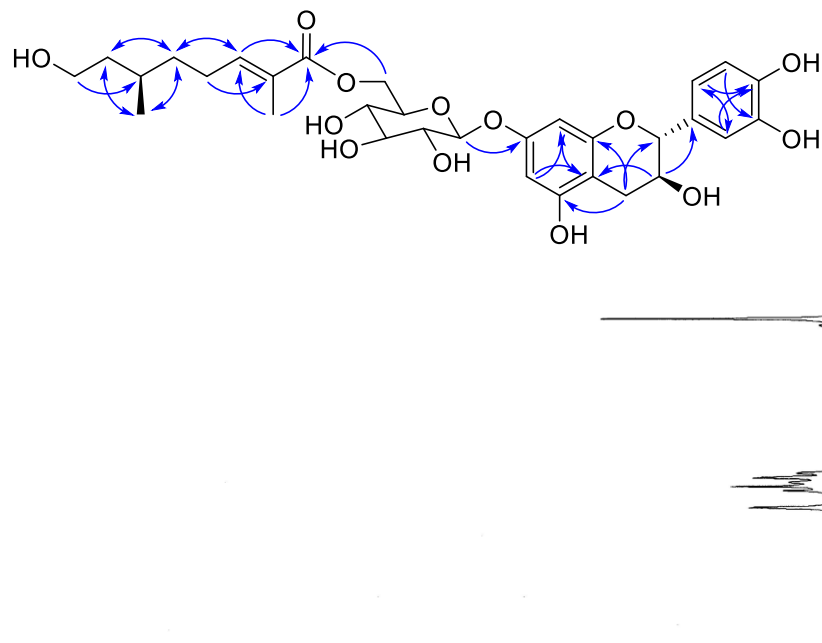


Figure S21. The HMBC Spectrum of Compound 2 in MeOH-*d*₄ (500MHz)

DD2-500 NOESY QYH-76-1 IN cd3od Jan 4 2015 coldprobe

Temp. 25.0 C / 298.1 K
 Sample #1, Operator: vnmr1
 Relax. delay 1.600 sec
 Acq. time 0.150 sec
 Width 4595.6 Hz
 2D Width 4595.6 Hz
 8 repetitions
 2 x 200 increments
 OBSERVE H1, 499.7694110 MHz
 DATA PROCESSING
 Gauss apodization 0.056 sec
 F1 DATA PROCESSING
 Gauss apodization 0.040 sec
 FT size 2048 x 2048
 Total time 2 hr, 18 min

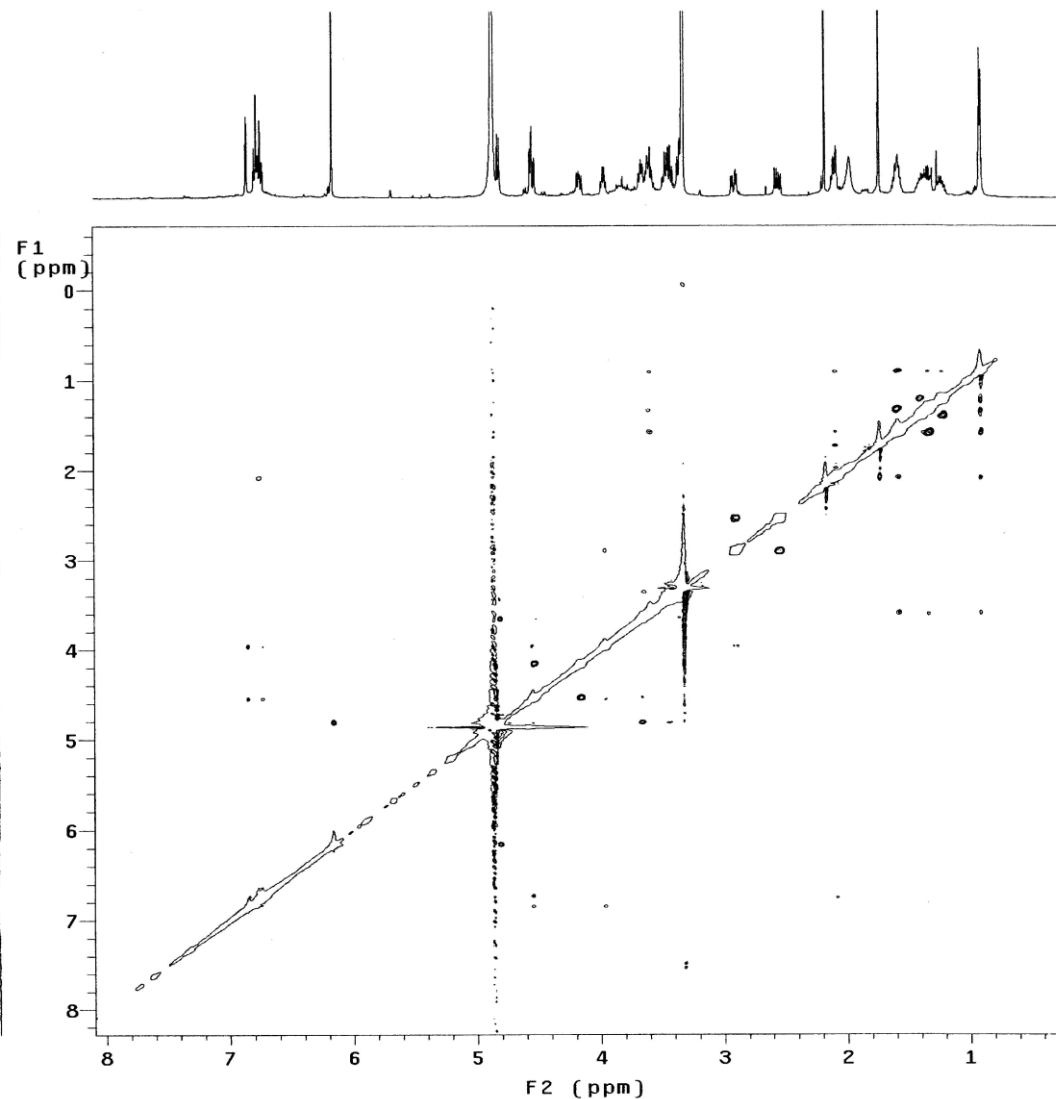
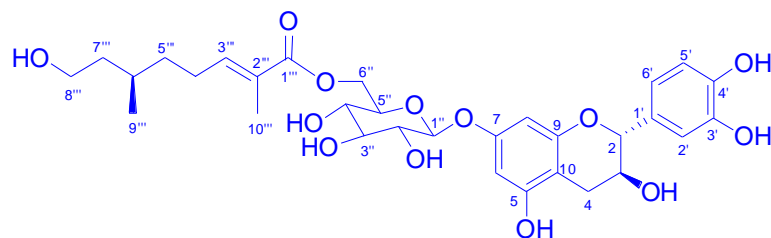
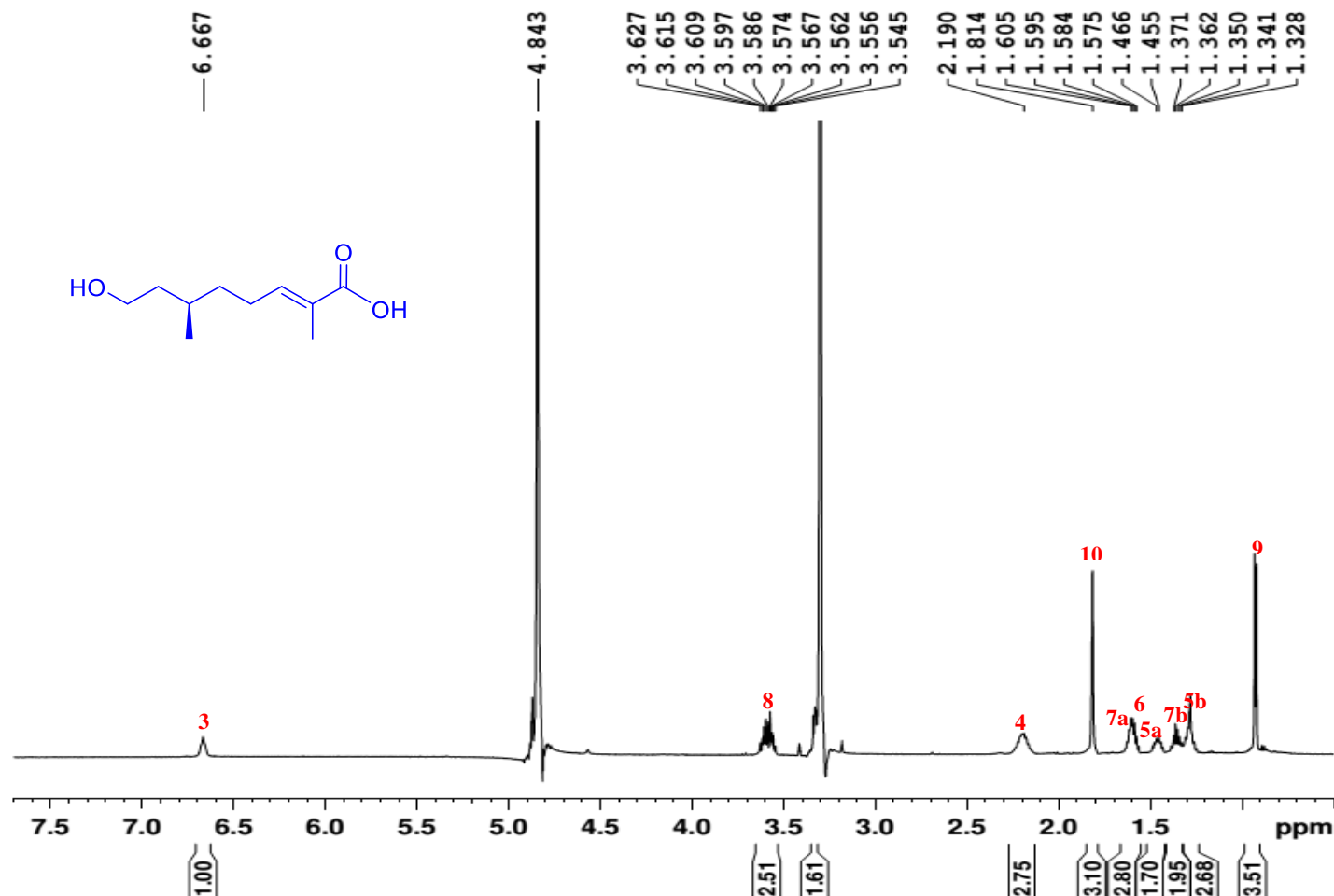


Figure S22. The NOESY Spectrum of Compound 2 in MeOH- d_4 (500MHz)

PROTON CD3OD D:\ DATA2015 4



NAME 20151231 qyh-
EXPNO 1
PROCNO 1
Date_ 20151231
Time 22.25
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG zg30
TD 65536
SOLVENT CD3OD
NS 16
DS 2
SWH 12019.230
FIDRES 0.183395
AQ 2.7263477
RG 57
DW 41.600
DE 20.00
TE 298.0
D1 1.00000000
TD0 1

===== CHANNEL f1 =====
SFO1 600.2537068
NUC1 1H
P1 11.50
SI 65536
SF 600.2499873
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00

Figure S23. The ^1H NMR Spectrum of Compound 2a in $\text{MeOH-}d_4$ (600MHz)

Bruker AV111HD 600 20160105
C13 CD3OD D:\ DATA2015 24

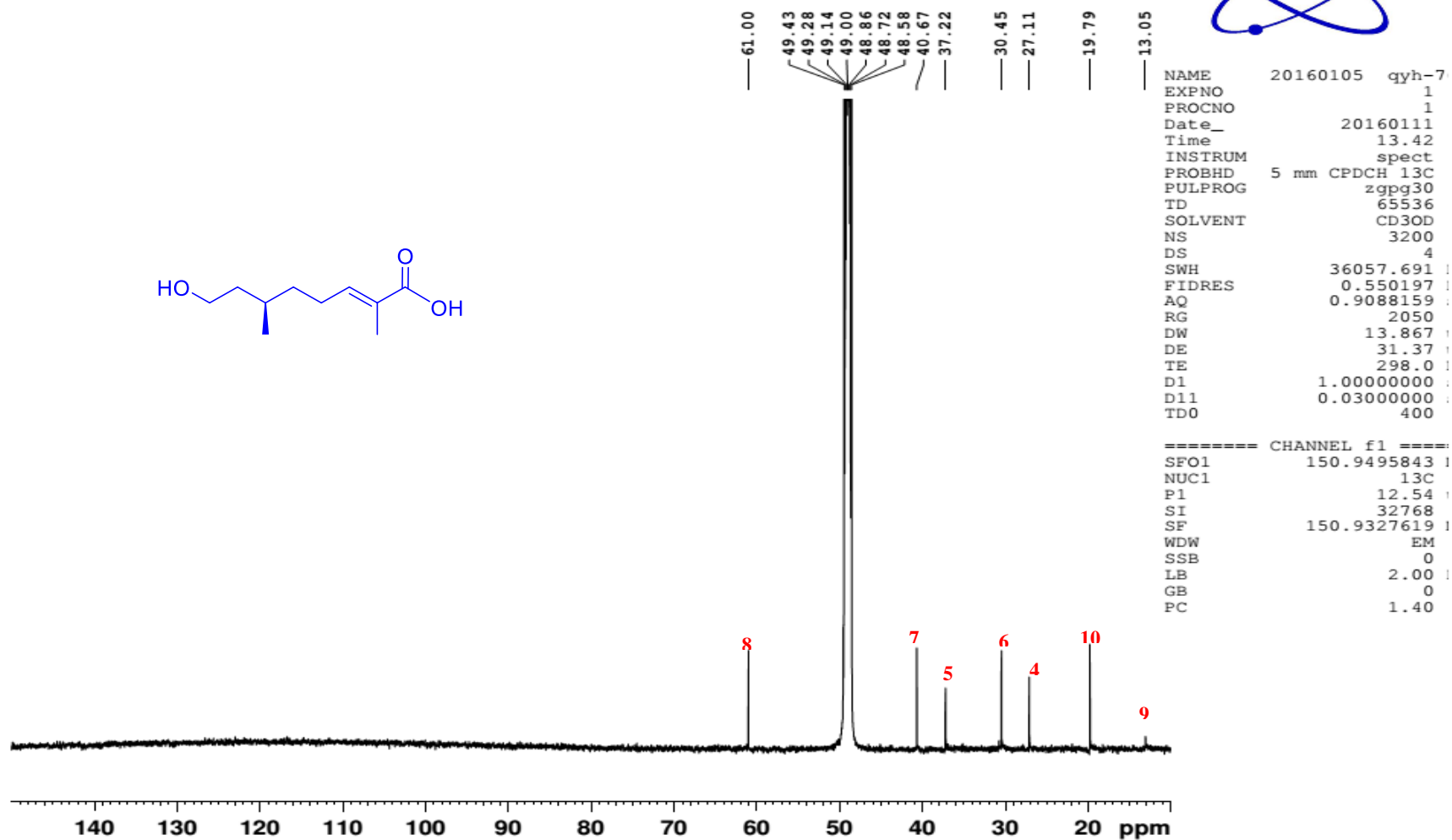
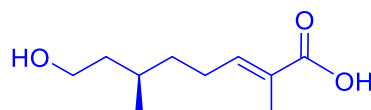
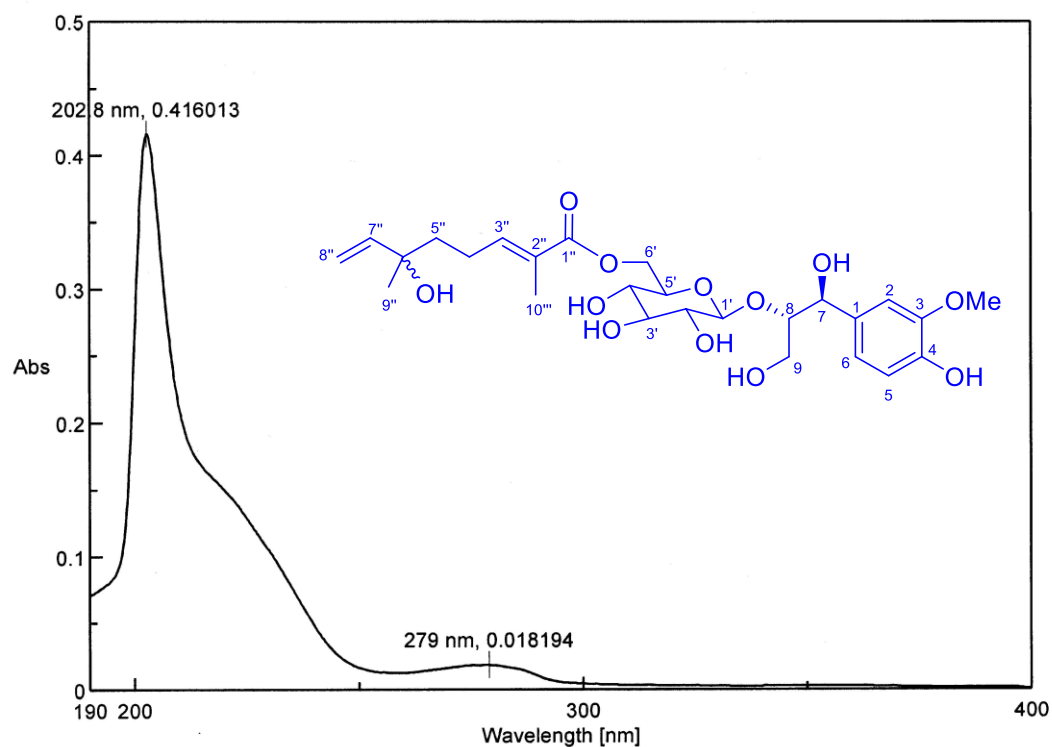


Figure S24. The ^{13}C NMR Spectrum of Compound 2a in $\text{MeOH-}d_4$ (150MHz)



[Comment]
 Sample Name QYH-40
 Comment 0.02
 User
 Division UV
 Company 324
 [Measurement Information]
 Instrument Name V-650
 Model Name V-650
 Serial No. A034461150

Accessory PSC-718
 Accessory S/N A001761114
 Position 1
 Cell Length 10 mm
 Temperature 19.94 C
 Control Sensor Holder
 Monitor Sensor Holder
 Start Mode Start immediately

Photometric Mode Abs
 Measurement range 400 - 190 nm
 Data pitch 0.2 nm
 Band width(UV/Vis) 2.0 nm
 Response Medium
 Scanning speed 200 nm/min
 Source Change 340 nm
 Light Source D2/WI
 Filter Exchange Step
 Correction Baseline

[Data Information]
 Creation Date 2014-7-15 21:19

Data array type Linear data array
 Horizontal Wavelength [nm]
 Vertical Abs
 Start 400 nm
 End 190 nm
 Data pitch 0.2 nm
 Data points 1051

Figure S25. The UV Spectrum of Compound 3 in MeOH

Single Mass Spectrum Deconvolution Report

Analysis Name: quyh0005.d **Instrument:** LC-MSD-Trap-SL **Print Date:** 9/23/2014 10:58:57 AM
Method: standby.m **Operator:** Operator **Acq. Date:** 9/23/2014 10:48:10 AM
Sample Name: QYH-40
Analysis Info:

Acquisition Parameter:

| | | | | | |
|-----------------|------------|-----------------------|-------------|----------------|-----------|
| Mass Range Mode | Std/Normal | Trap Drive | 53.0 | Scan Begin | 100 m/z |
| Ion Polarity | Positive | Octopole RF Amplitude | 171.0 Vpp | Scan End | 800 m/z |
| Ion Source Type | ESI | Capillary Exit | -115.0 Volt | Averages | 5 Spectra |
| Dry Temp (Set) | 330 °C | Skimmer | -40.0 Volt | Max. Accu Time | 200000 µs |
| Nebulizer (Set) | 40.00 psi | Oct 1 DC | -12.00 Volt | ICC Target | 20000 |
| Dry Gas (Set) | 9.00 l/min | Oct 2 DC | -1.70 Volt | Charge Control | on |

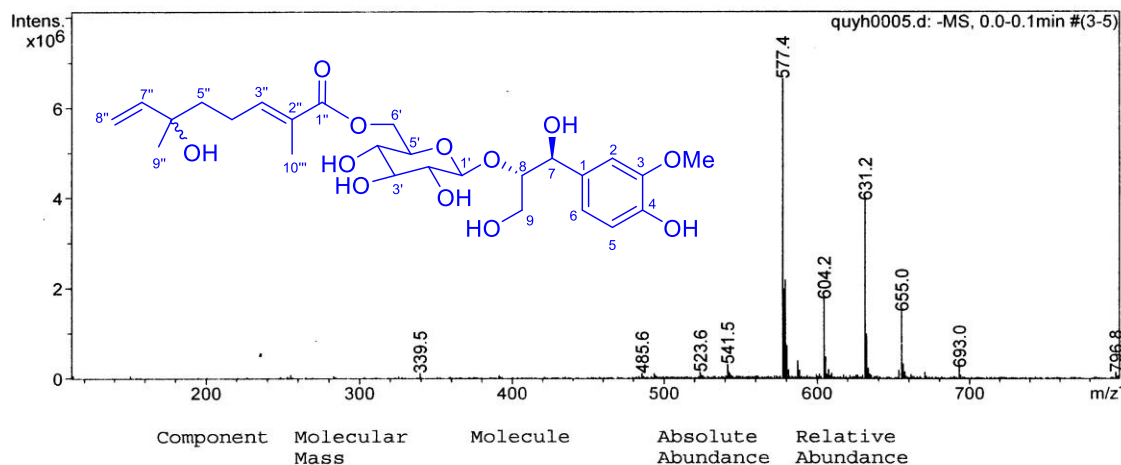
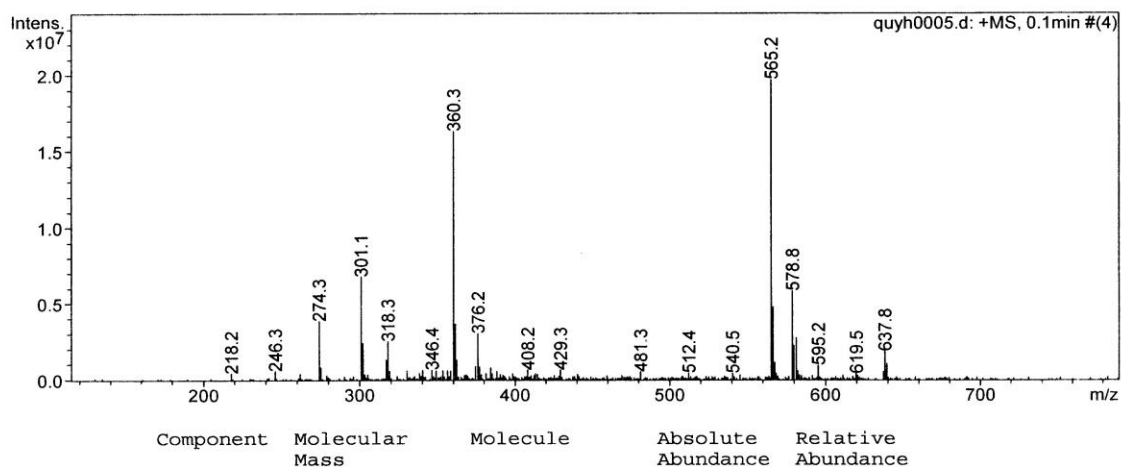


Figure S26. The ESIMS Spectrum of Compound 3

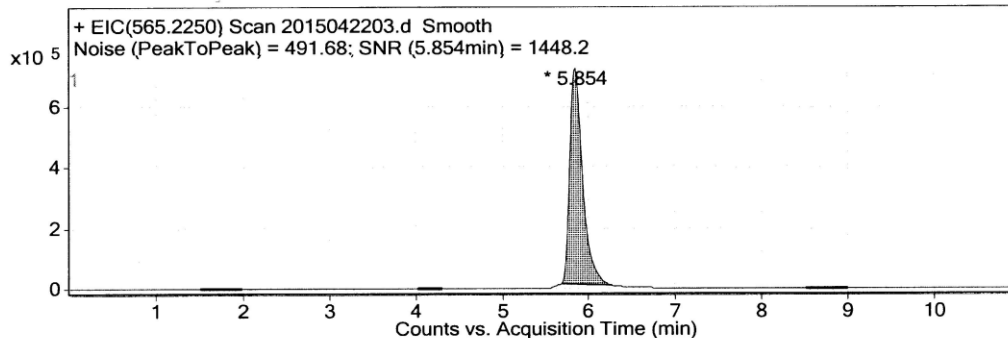
Qualitative Analysis Report

Data Filename 2015042203.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method TEST LCMS.m
DA Method TEST LCMS.m

Sample Name QYH-40
Position P1-C3
User Name
IRM Calibration Status Success
Comment

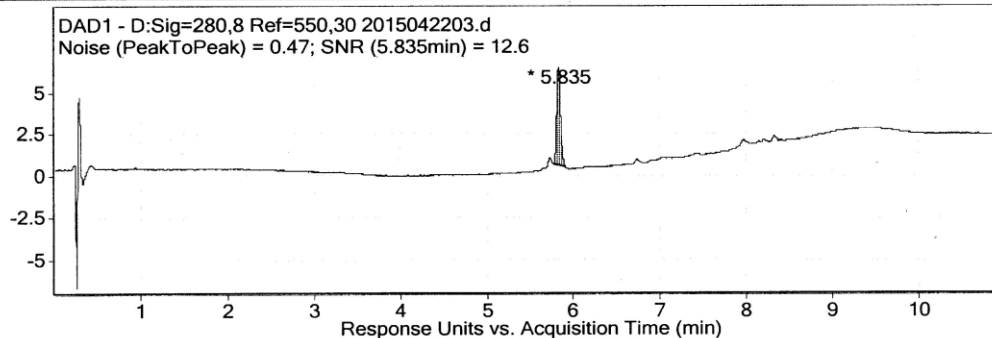
User Chromatograms

Fragmentor Voltage 120 Collision Energy 0 Ionization Mode ESI



Integration Peak List

| Peak | Start | RT | End | Height | Area | Area % | Signal To Noise |
|------|-------|-------|-------|--------|---------|--------|-----------------|
| 1 | 5.693 | 5.854 | 6.273 | 712047 | 7468829 | 100 | 1448.2 |



Integration Peak List

| Peak | Start | RT | End | Height | Area | Area % | Signal To Noise |
|------|-------|-------|-------|--------|-------|--------|-----------------|
| 1 | 5.773 | 5.835 | 5.915 | 5.89 | 18.38 | 100 | 12.6 |

User Spectra

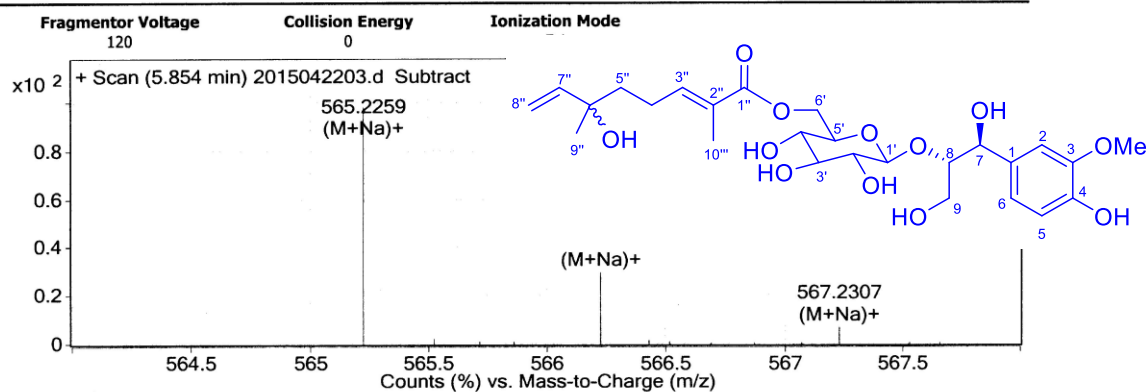
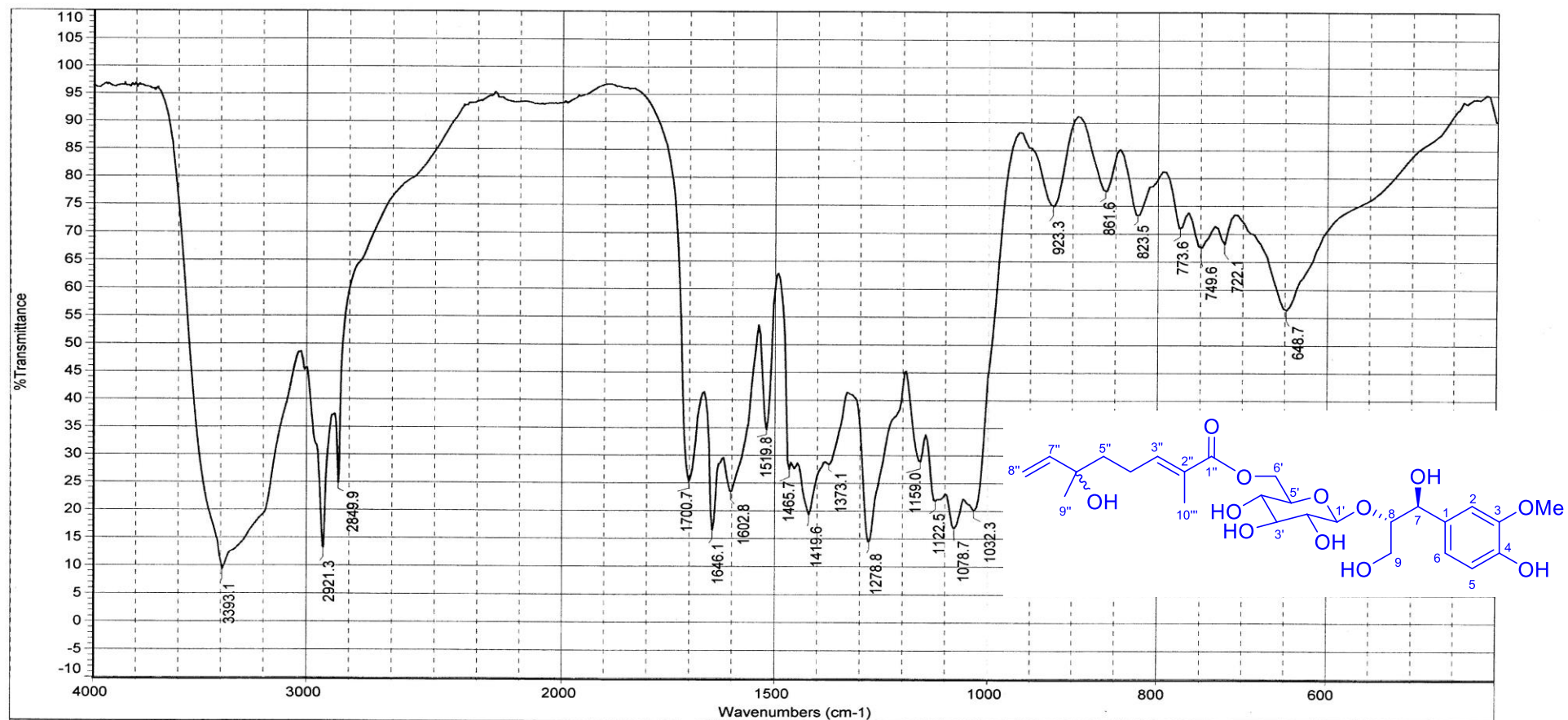


Figure S27. The HRESIMS Spectrum of Compound 3



日期: 星期四 3月 12 14:03:12 2015 (GMT+08:00) Sample Name : Q - 40

(显微镜透射法FT- IR Microscope Transmission)

扫描次数: 100

傅立叶变换显微镜红外(FT-IR Microscope): Centaur μ s

分辨率: 8.000

美国热电公司(Thermo)傅立叶变换红外光谱仪:Nicolet 5700

Figure S28. The IR Spectrum of Compound 3

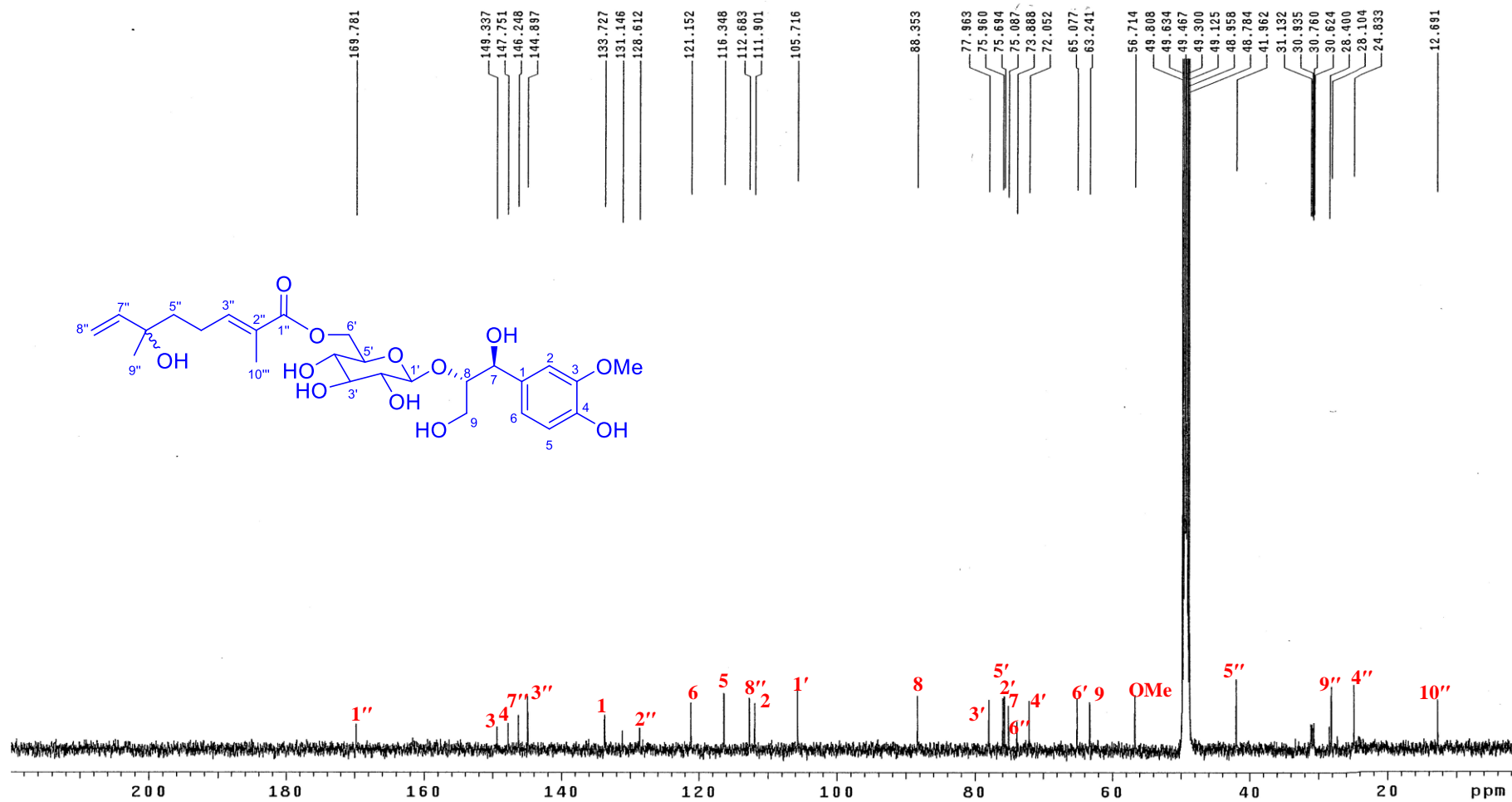


Figure S30. The ^{13}C NMR Spectrum of Compound 3 in $\text{MeOH-}d_4$ (125MHz)

DD2-500 gCOSY QYH-40 IN cd3od May 29 2014 sw

Temp. 25.0 C / 298.1 K
Sample #4, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4464.3 Hz
2D Width 4464.3 Hz
8 repetitions
128 increments
OBSERVE H1, 499.7694214 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.029 sec
FT size 2048 x 2048
Total time 20 min

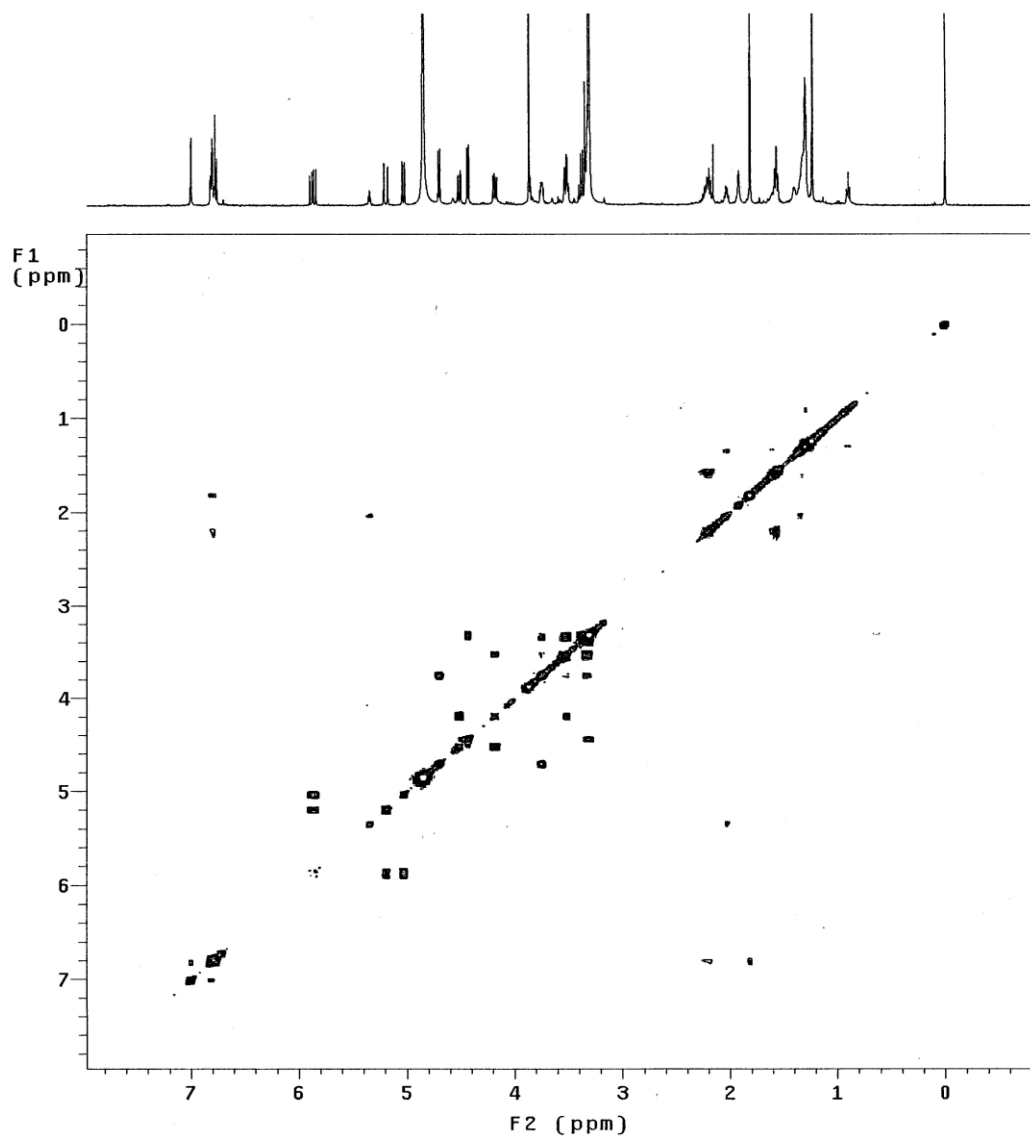
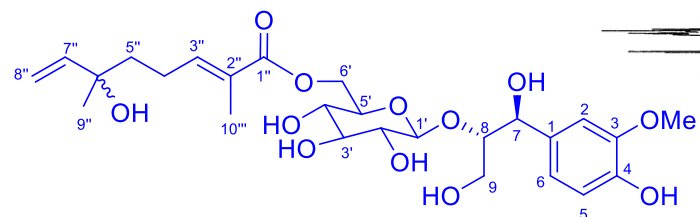


Figure S31. The ^1H - ^1H COSY Spectrum of Compound 3 in $\text{MeOH-}d_4$ (500MHz)

Temp. 25.0 C / 298.1 K
 Sample #4, Operator: vnmr1
 Relax. delay 1.000 sec
 Acq. time 0.269 sec
 Width 4464.3 Hz
 2D Width 25133.5 Hz
 16 repetitions
 2 x 256 increments
 OBSERVE H1, 499.7694214 MHz
 DECOUPLE C13, 125.6784310 MHz
 Power 40 dB
 on during acquisition
 off during delay
 W40_sw modulated
 DATA PROCESSING
 Gauss apodization 0.069 sec
 F1 DATA PROCESSING
 Gauss apodization 0.009 sec
 FT size 4096 x 2048
 Total time 2 hr, 44 min

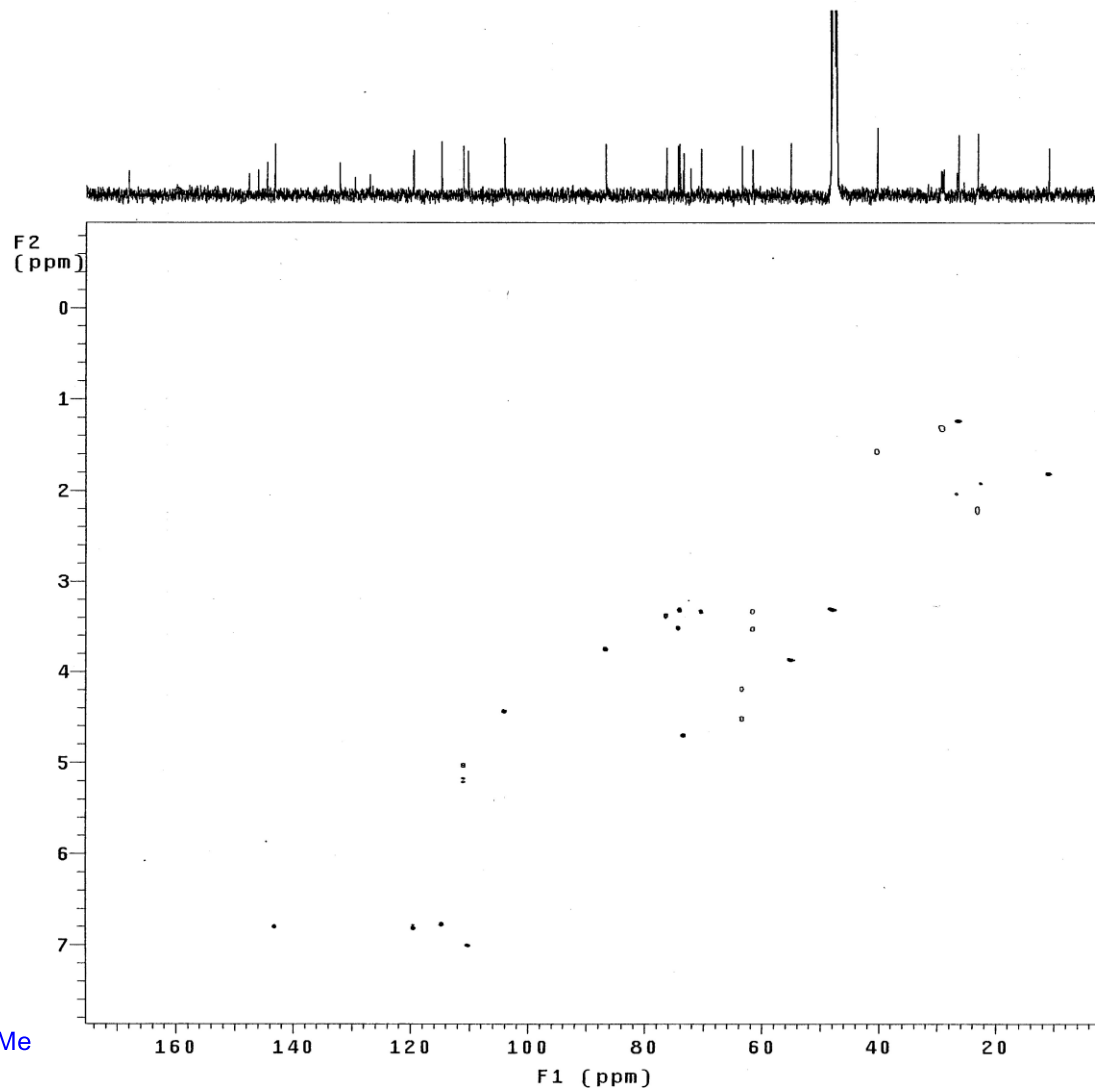
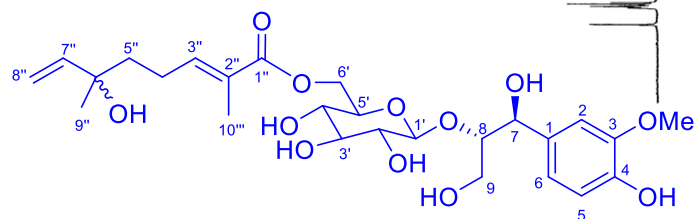


Figure S32. The HSQC Spectrum of Compound 3 in MeOH-*d*₄ (500MHz)

Temp. 25.0 C / 298.1 K
 Sample #4, Operator: vnmr1
 Relax. delay 1.000 sec
 Acq. time 0.269 sec
 Width 4464.3 Hz
 2D Width 30154.5 Hz
 32 repetitions
 2 x 256 increments
 OBSERVE H1, 499.7694214 MHz
 DATA PROCESSING
 Sq. sine bell 0.075 sec
 F1 DATA PROCESSING
 Gauss apodization 0.008 sec
 FT size 4096 x 2048
 Total time 5 hr, 38 min

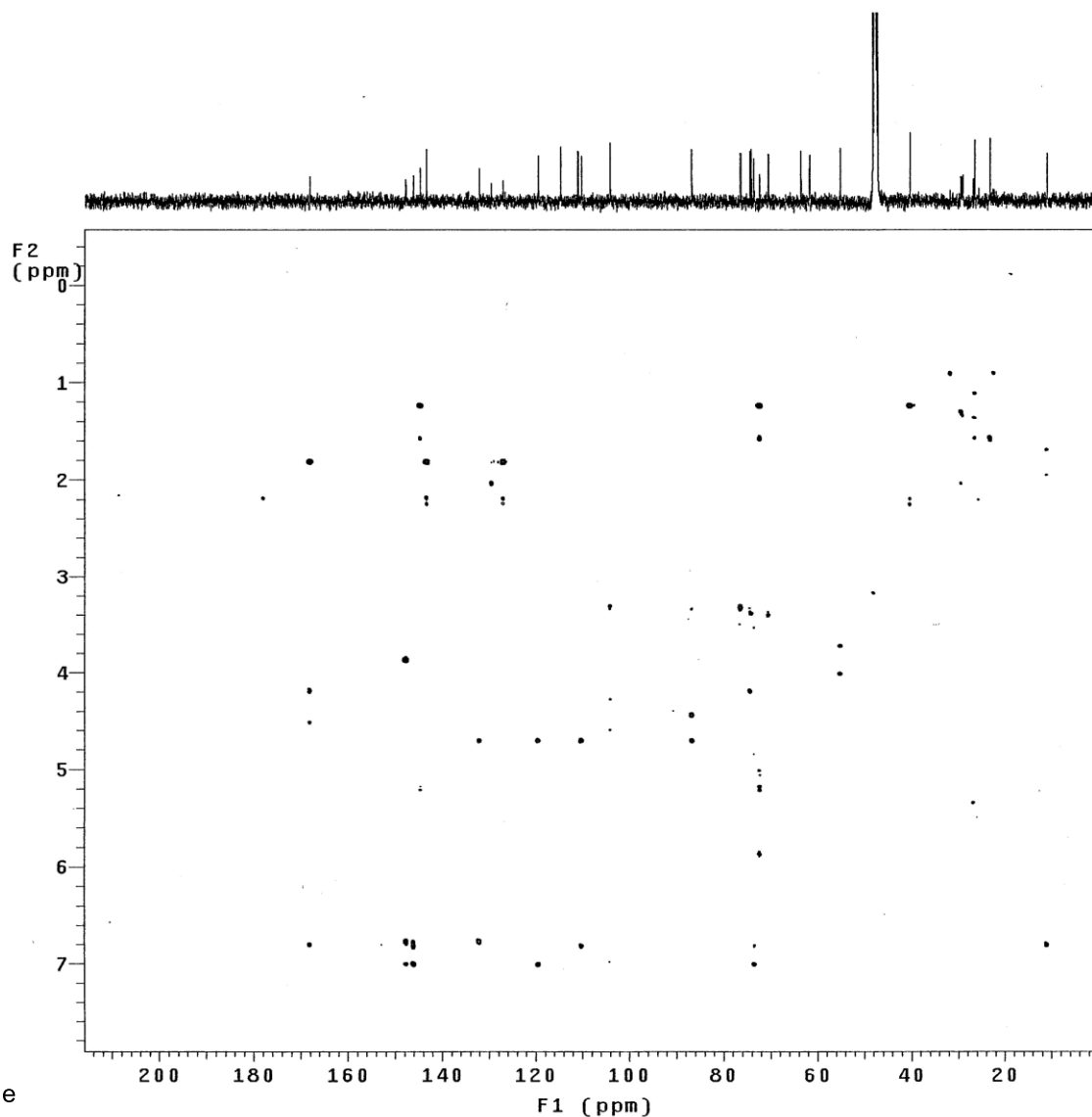
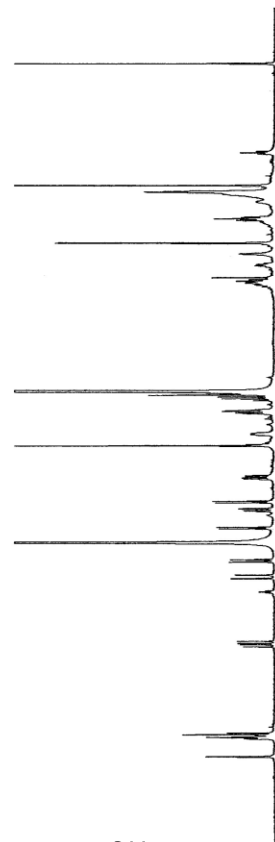
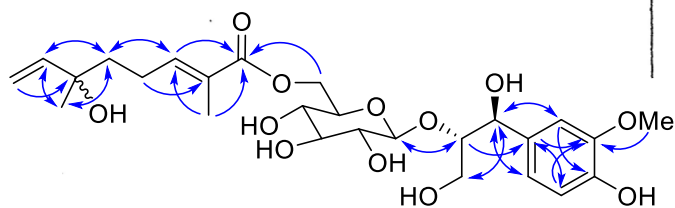
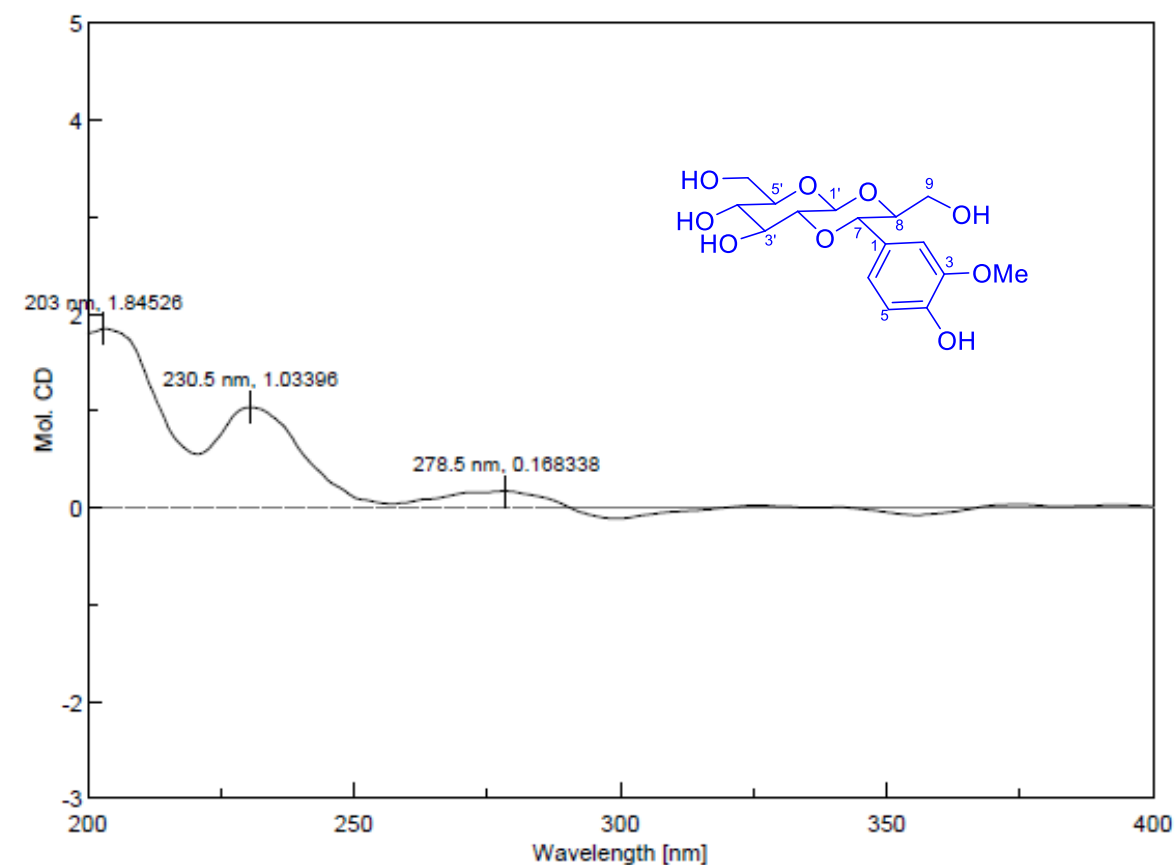


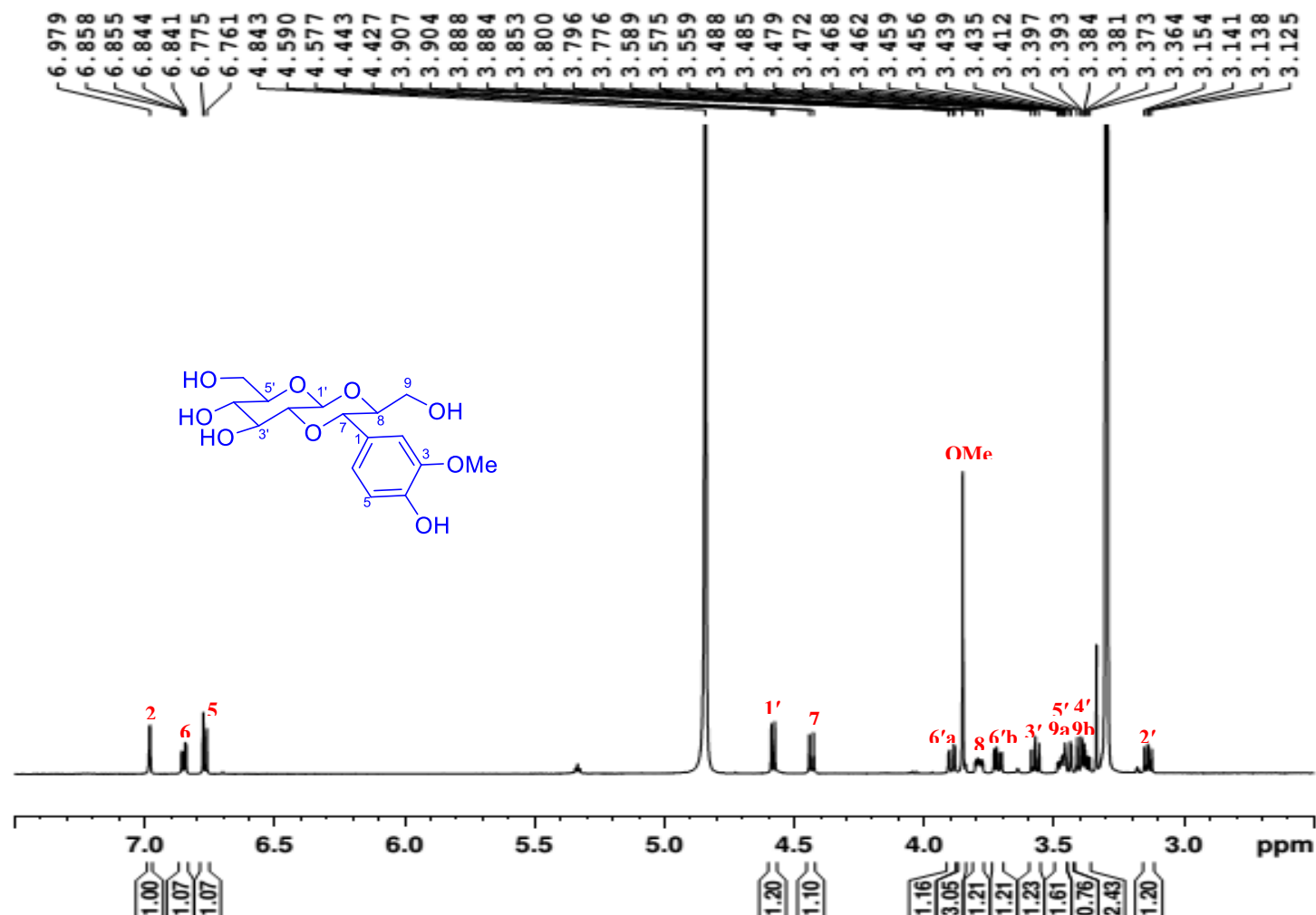
Figure S33. The HMBC Spectrum of Compound 3 in MeOH- d_4 (500MHz)



| | | | |
|---------------------------|-----------------|------------------------|-----------------------|
| [Comments] | | qyh-40-2-s-m.jws | |
| Sample name | qyh-40 | | |
| Comment | | | |
| [Measurement Information] | | | |
| Instrument Name | IMM-CD | | |
| Operator | JMM | | |
| Serial No. | A024461168 | | |
| Accessory | Standard | | |
| Accessory S/N | A024461168 | | |
| Cell Length | 0.1 mm | [Detailed Information] | |
| Measurement date | 2016-8-23 12:04 | Creation date | 2016-8-23 13:45 |
| Photometric Mode | CD, HT, Abs | Data array type | Linear data array * 3 |
| Measure Range | 500 - 200 nm | Horizontal axis | Wavelength [nm] |
| Data pitch | 0.5 nm | Vertical axis(1) | Mol. CD |
| Sensitivity | Standard | Vertical axis(2) | HT [V] |
| D.I.T. | 1 sec | Vertical axis(3) | Abs |
| Bandwidth | 2.00 nm | Start | 500 nm |
| Start Mode | Immediately | End | 200 nm |
| Scanning Speed | 100 nm/min | Data interval | 0.5 nm |
| Baseline Correction | Baseline | Data points | 601 |
| Shutter Control | Auto | | |
| CD Detector | PMT | | |
| PMT Voltage | Auto | | |
| Accumulations | 2 | | |
| Solvent | MEOH | | |
| Concentration | 0.13 (w/v)% | | |

Figure S34. The CD Spectrum of Compound 3a in MeOH

PROTON CD3OD D:\\ DATA2015 5



```

NAME      20151229  qyh-
EXPNO     1
PROCNO    1
Date_     20151229
Time      11.45
INSTRUM   spect
PROBHD    5 mm CPDCH 13C
PULPROG   zg30
TD         65536
SOLVENT   CD3OD
NS         16
DS         2
SWH        12019.230
FIDRES     0.183396
AQ         2.7263477
RG         51
DM         41.600
DE         20.00
TE         298.1
D1         1.00000000
TD0        1

===== CHANNEL f1 =====
SFO1      600.2537068
NUC1       13
P1         11.50
SI         65536
SF         600.2499877
WDW        EM
SSB        0
LB         0.30
GB         0
PC         1.00
  
```

Figure S35. The ^1H NMR Spectrum of Compound 3a in $\text{MeOH-}d_4$ (600MHz)

NAME: 20160119 qyh-4
C13 CD3OD D:\\ DATA2016 28

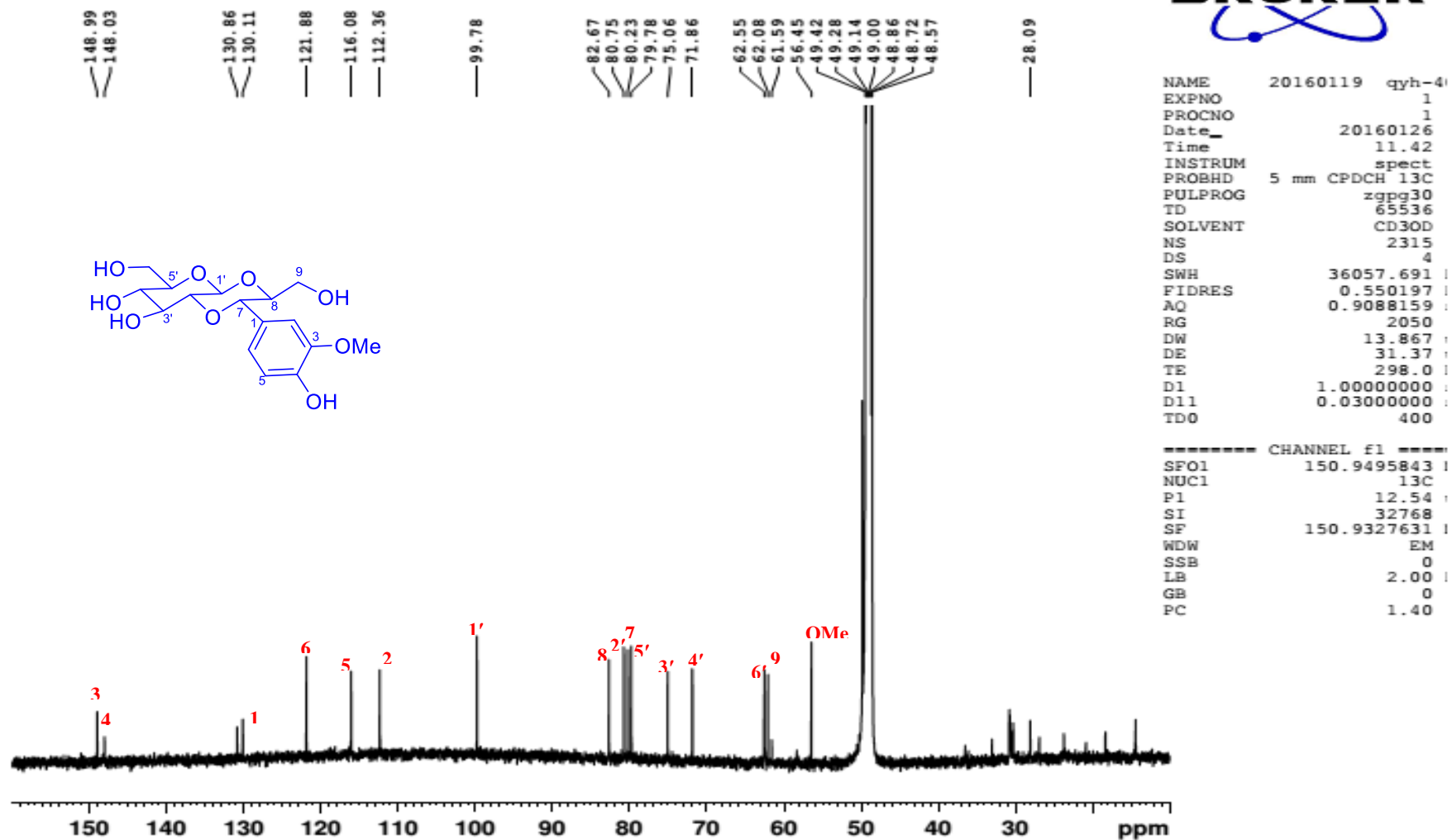


Figure S36. The ¹³C NMR Spectrum of Compound 3a in MeOH-*d*₄ (150MHz)

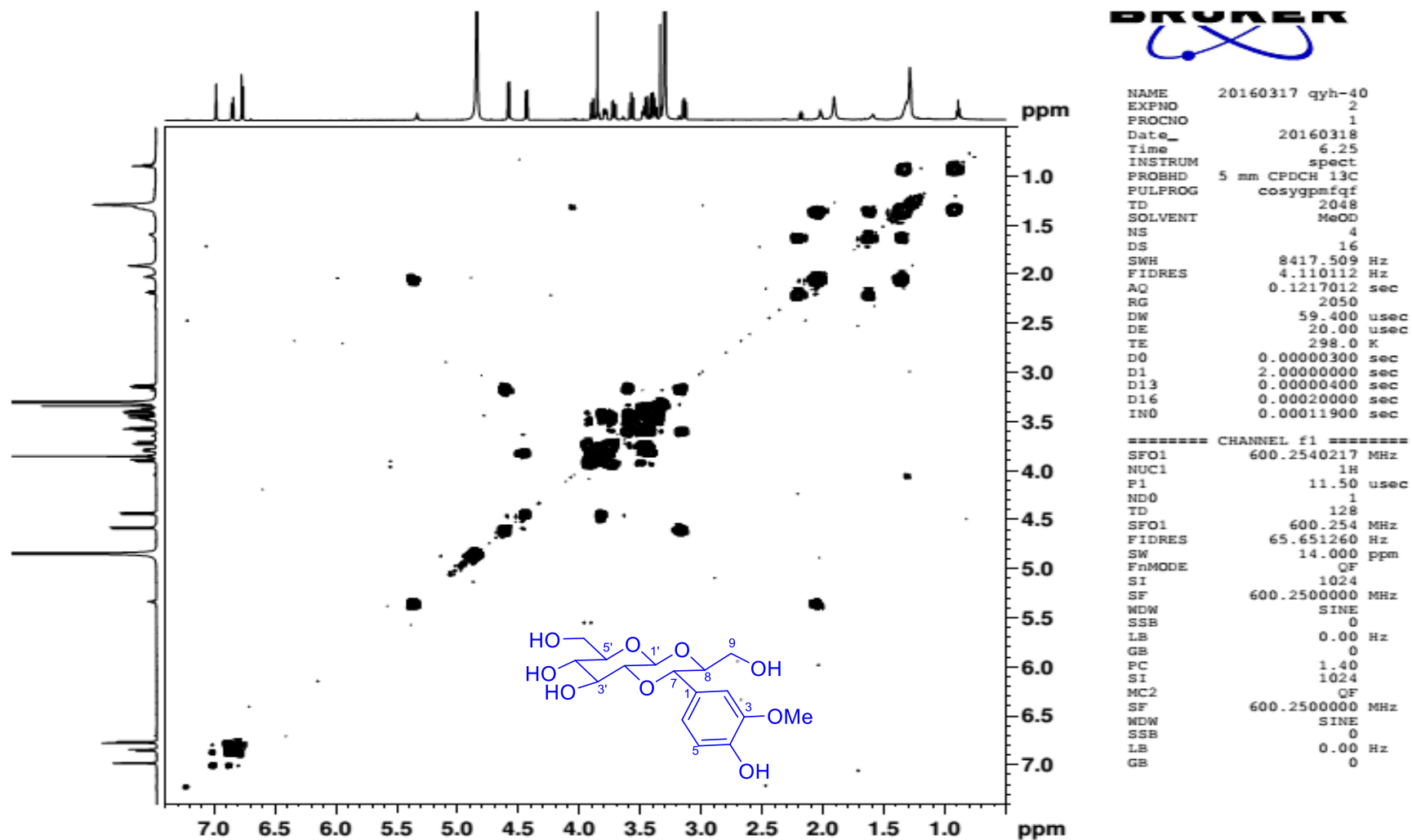


Figure S37. The ^1H - ^1H COSY Spectrum of Compound 3a in $\text{MeOH-}d_4$ (600MHz)

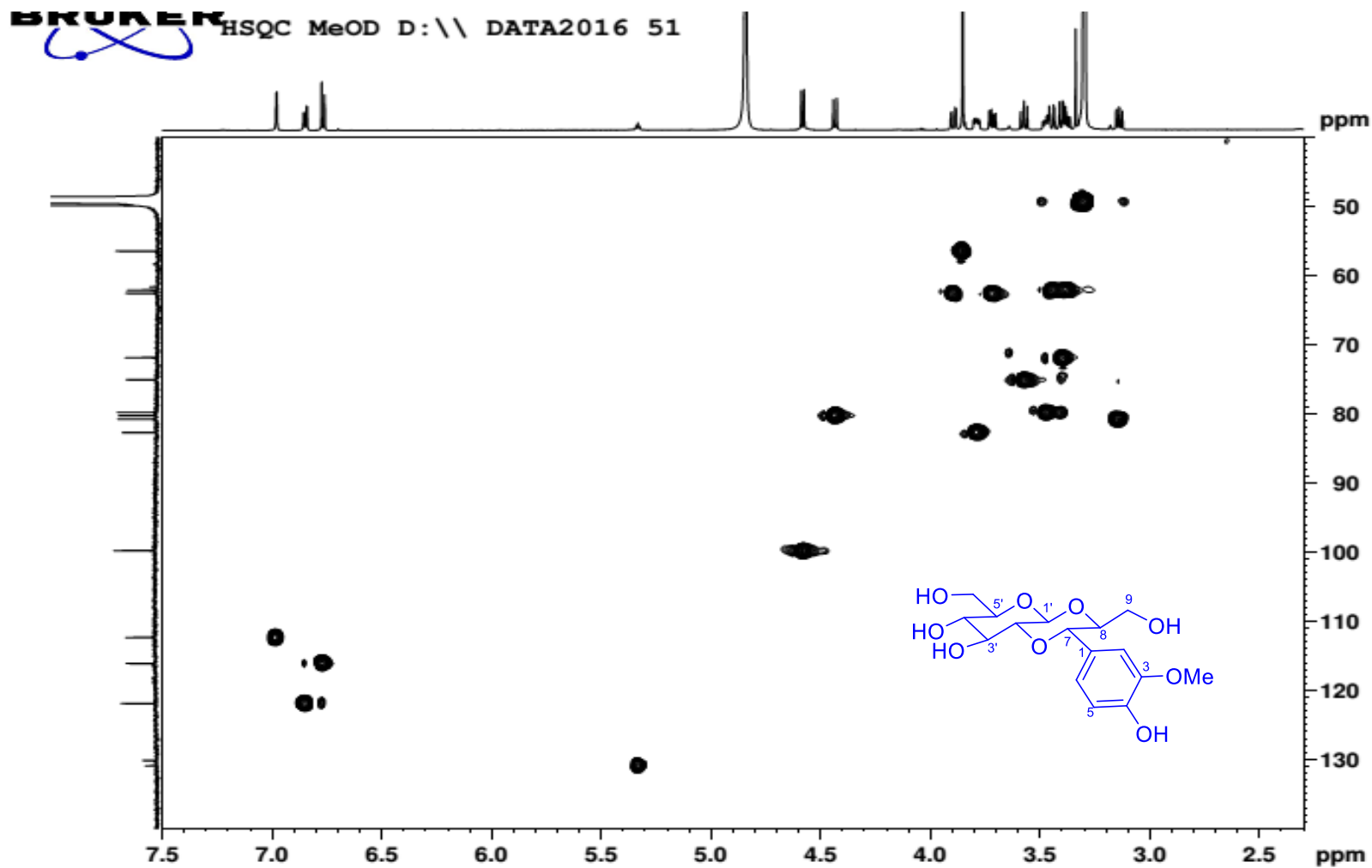


Figure S38. The HSQC Spectrum of Compound 3a in MeOH- d_4 (500MHz)

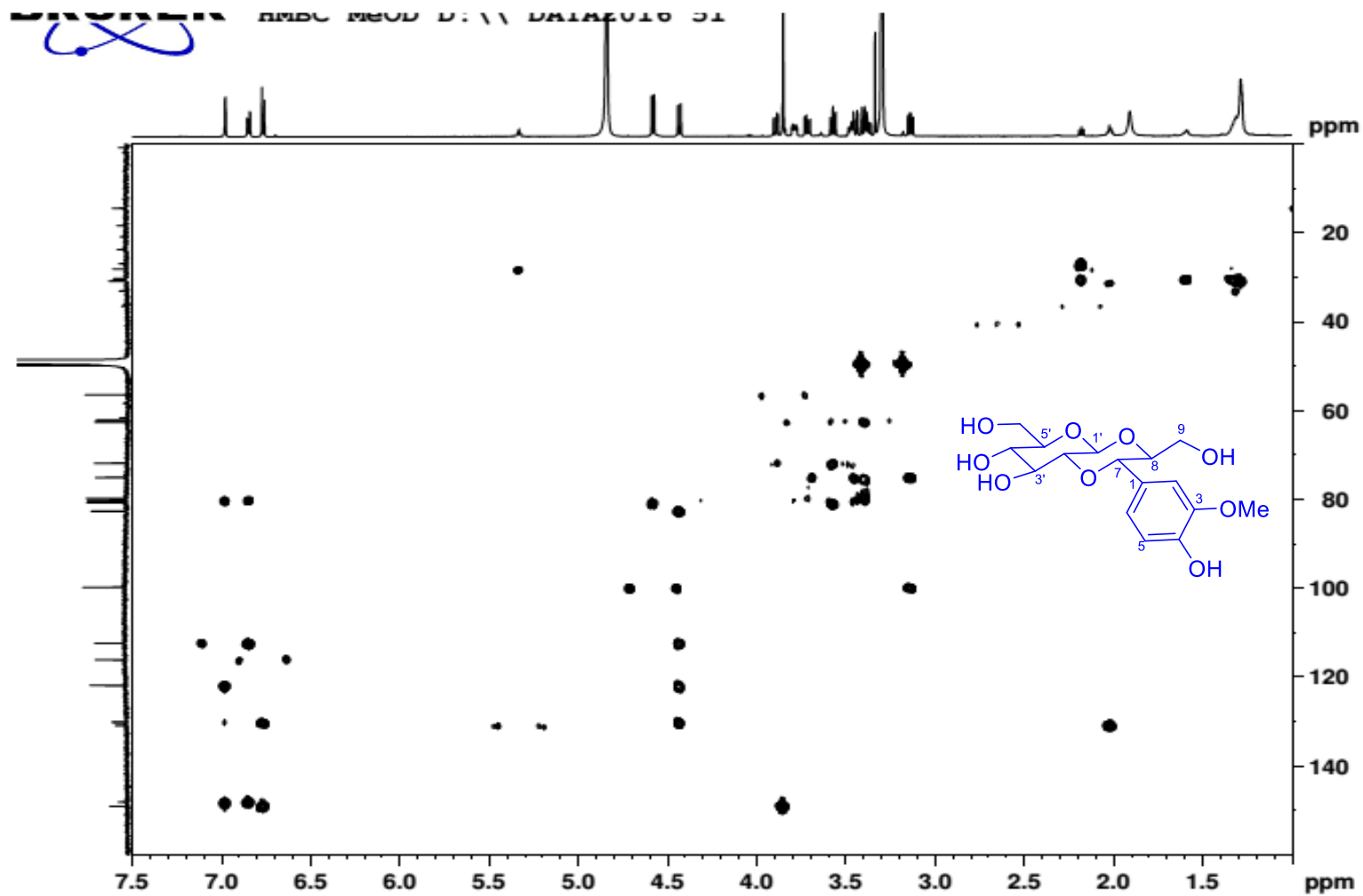
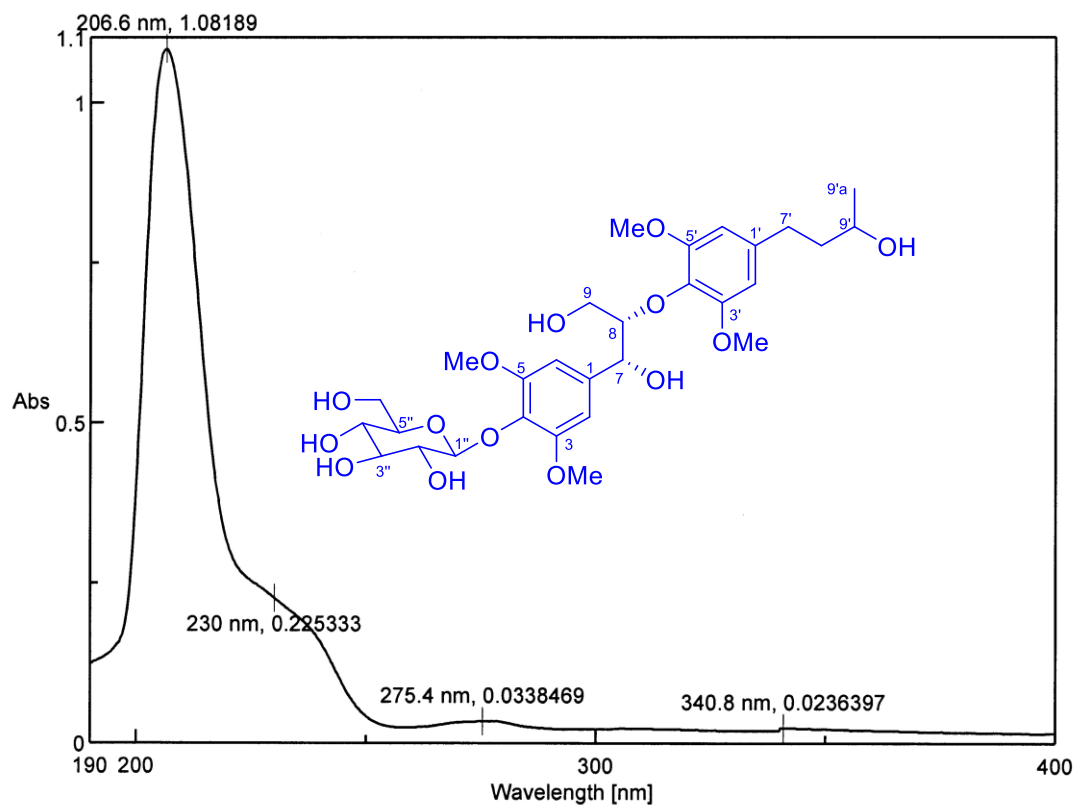


Figure S39. The HMBC Spectrum of Compound 3a in MeOH- d_4 (600MHz)



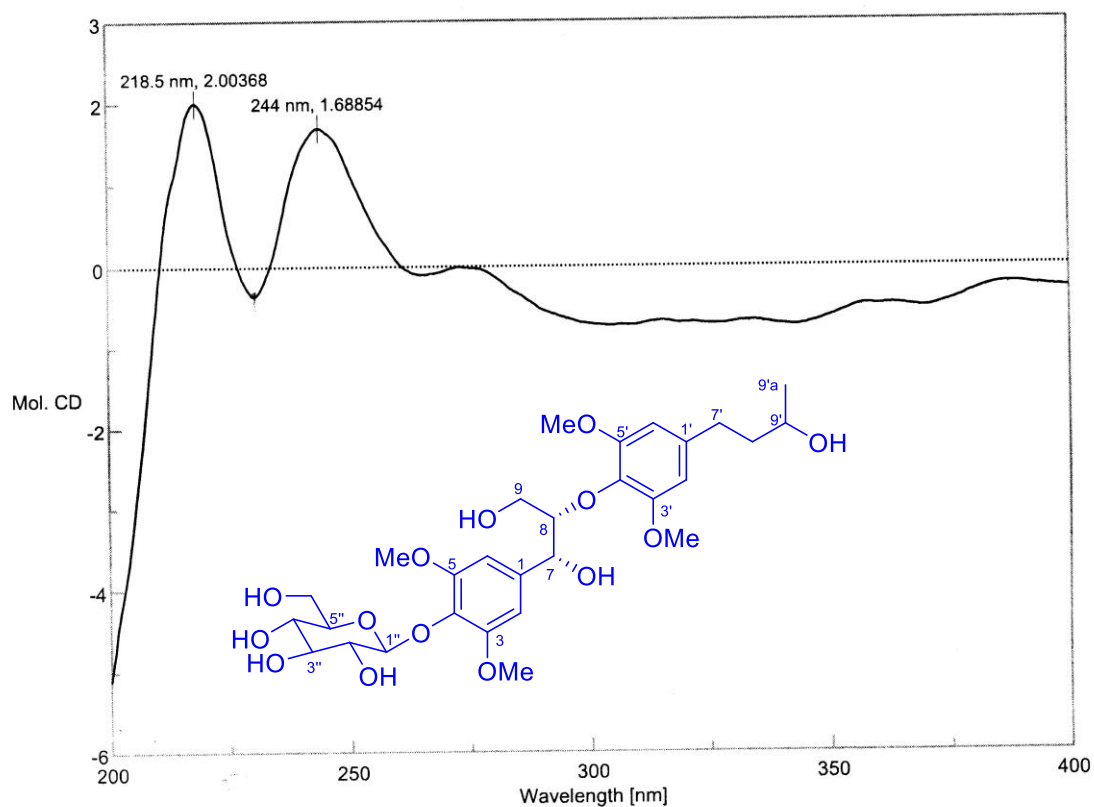
[Comment]
 Sample Name sjj-w37
 Comment 0.02
 User
 Division UV
 Company 324
 [Measurement Information]
 Instrument Name V-650
 Model Name V-650
 Serial No. A034461150

Accessory PSC-718
 Accessory S/N A001761114
 Position 1
 Cell Length 10 mm
 Temperature 19.95 C
 Control Sensor Holder
 Monitor Sensor Holder
 Start Mode Start immediately

Photometric Mode Abs
 Measurement range 400 - 190 nm
 Data pitch 0.2 nm
 Band width(UV/Vis) 2.0 nm
 Response Medium
 Scanning speed 200 nm/min
 Source Change 340 nm
 Light Source D2/WI
 Filter Exchange Step
 Correction Baseline

[Data Information]
 Creation Date 2014-11-18 21:04
 Data array type Linear data array
 Horizontal Wavelength [nm]
 Vertical Abs
 Start 400 nm
 End 190 nm
 Data pitch 0.2 nm
 Data points 1051

Figure S40. The UV Spectra of Compound 4 in MeOH



[Comments]
 Sample name sjj-w37
 Comment
 User
 Division
 Company dell

[Measurement Information]
 Instrument Name J-815
 Model Name J-815
 Serial No. A024461168

Accessory Standard
 Accessory S/N A024461168
 Cell Length 1 mm

Photometric Mode CD, HT, Abs
 Measure Range 400 - 200 nm
 Data pitch 0.5 nm
 Sensitivity Standard
 D.I.T. 1 sec
 Band width 2.00 nm
 Start Mode Immediately
 Scanning Speed 100 nm/min
 Baseline Correction Baseline
 Shutter Control Auto
 PMT Voltage Auto
 Accumulations 2
 Solvent MEOH
 Concentration 0.07 (w/v)%

[Detailed Information]
 Creation date 2015-9-14 16:30

Data array type Linear data array * 3
 Horizontal axis Wavelength [nm]
 Vertical axis(1) Mol. CD
 Vertical axis(2) HT [V]
 Vertical axis(3) Abs
 Start 400 nm
 End 200 nm
 Data interval 0.5 nm
 Data points 401

Figure S41. The CD Spectrum of Compound 4 in MeOH

Single Mass Spectrum Deconvolution Report

Analysis Name: WLYAN000.d

Instrument: LC-MSD-Trap-SL

Print Date: 9/23/2014 11:56:49 AM

Method: standby.m

Operator: Operator

Acq. Date: 9/23/2014 11:52:34 AM

Sample Name: Sjj-w-37

Analysis Info:

Acquisition Parameter:

| | | | | | |
|-----------------|------------|-----------------------|------------|----------------|-----------|
| Mass Range Mode | Std/Normal | Trap Drive | 36.3 | Scan Begin | 100 m/z |
| Ion Polarity | Positive | Octopole RF Amplitude | 171.0 Vpp | Scan End | 800 m/z |
| Ion Source Type | ESI | Capillary Exit | 115.0 Volt | Averages | 5 Spectra |
| Dry Temp (Set) | 330 °C | Skimmer | 40.0 Volt | Max. Accu Time | 200000 µs |
| Nebulizer (Set) | 40.00 psi | Oct 1 DC | 12.00 Volt | ICC Target | 20000 |
| Dry Gas (Set) | 9.00 l/min | Oct 2 DC | 1.70 Volt | Charge Control | on |

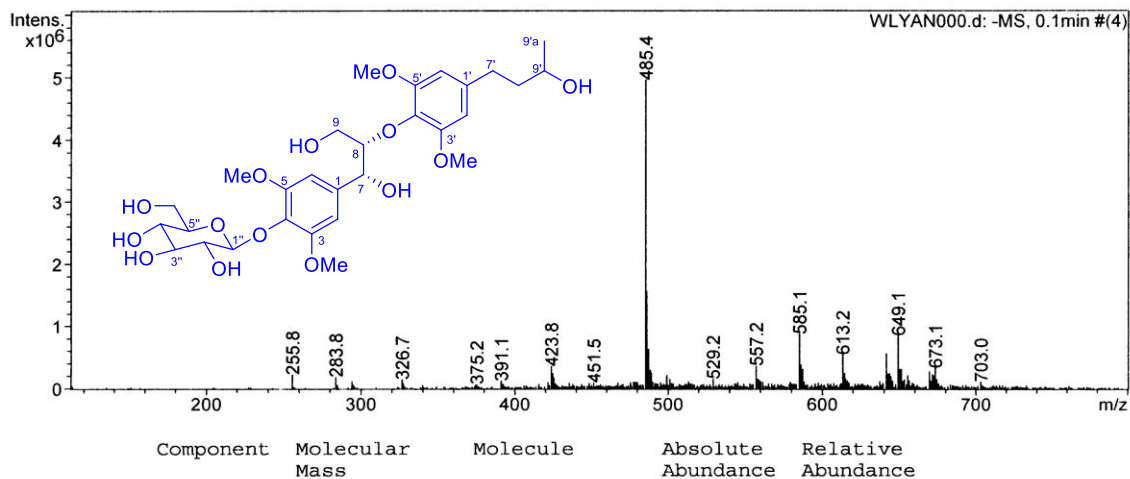
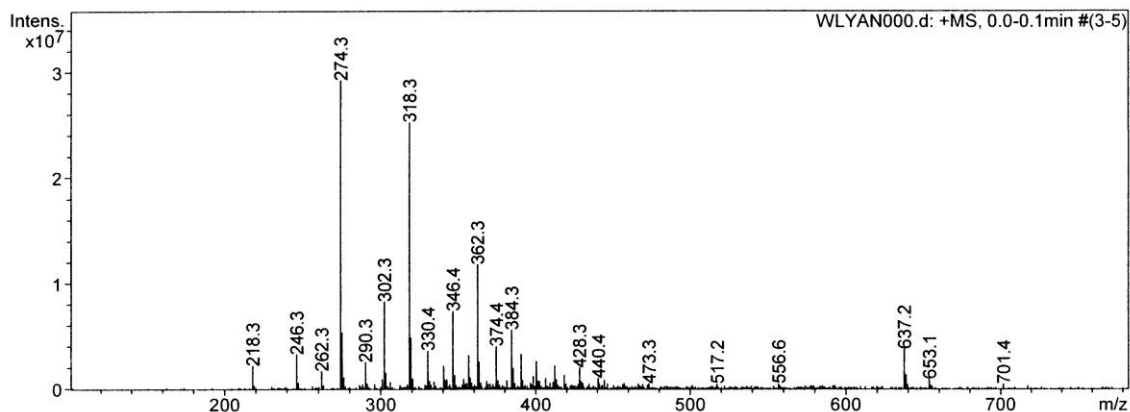
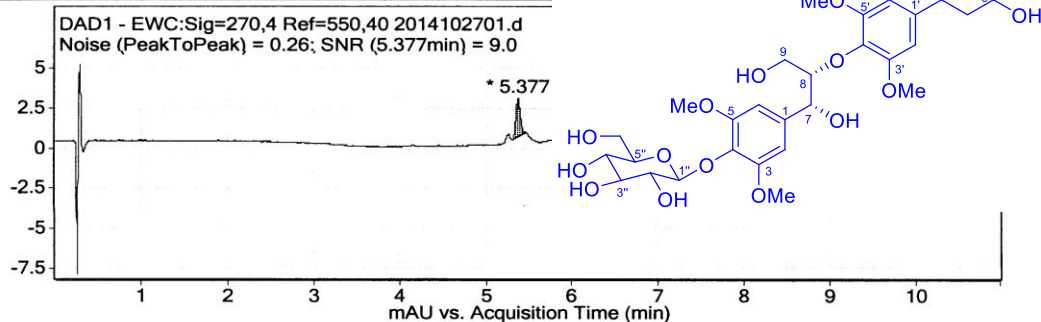


Figure S42. The ESIMS Spectrum of Compound 4

Qualitative Analysis Report

| | | | |
|------------------------|--------------|-------------------------------|---------|
| Data Filename | 2014102701.d | Sample Name | AJJ-W37 |
| Sample Type | Sample | Position | P1-C6 |
| Instrument Name | Instrument 1 | User Name | |
| Acq Method | | IRM Calibration Status | |
| DA Method | TEST LCMS.m | Comment | |

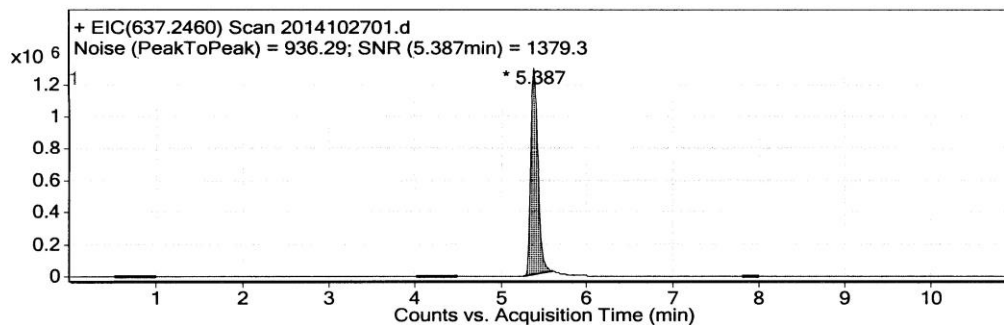
User Chromatograms



Integration Peak List

| Peak | Start | RT | End | Height | Area | Area % | Signal To Noise |
|------|-------|-------|-------|--------|-------|--------|-----------------|
| 1 | 5.323 | 5.377 | 5.476 | 2.36 | 8.762 | 100 | 9 |

Fragmentor Voltage 135 Collision Energy 0 Ionization Mode ESI



Integration Peak List

| Peak | Start | RT | End | Height | Area | Area % | Signal To Noise |
|------|-------|-------|-------|---------|---------|--------|-----------------|
| 1 | 5.258 | 5.387 | 5.612 | 1291433 | 7733713 | 100 | 1379.3 |

User Spectra

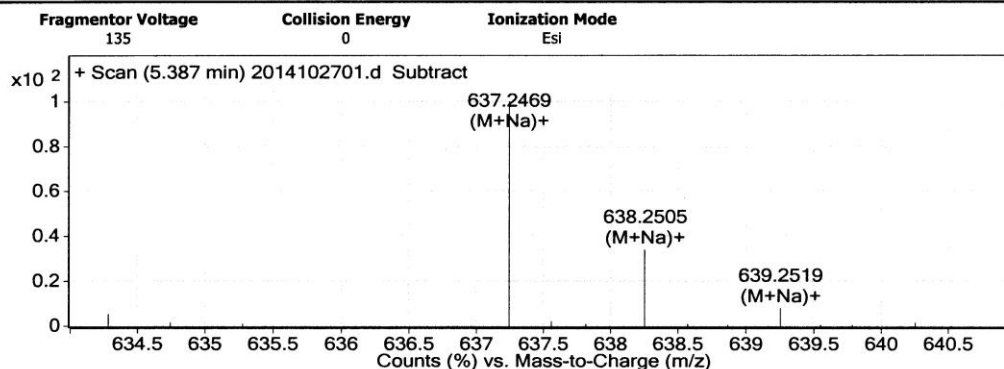


Figure S43. The HRESIMS Spectrum of Compound 4

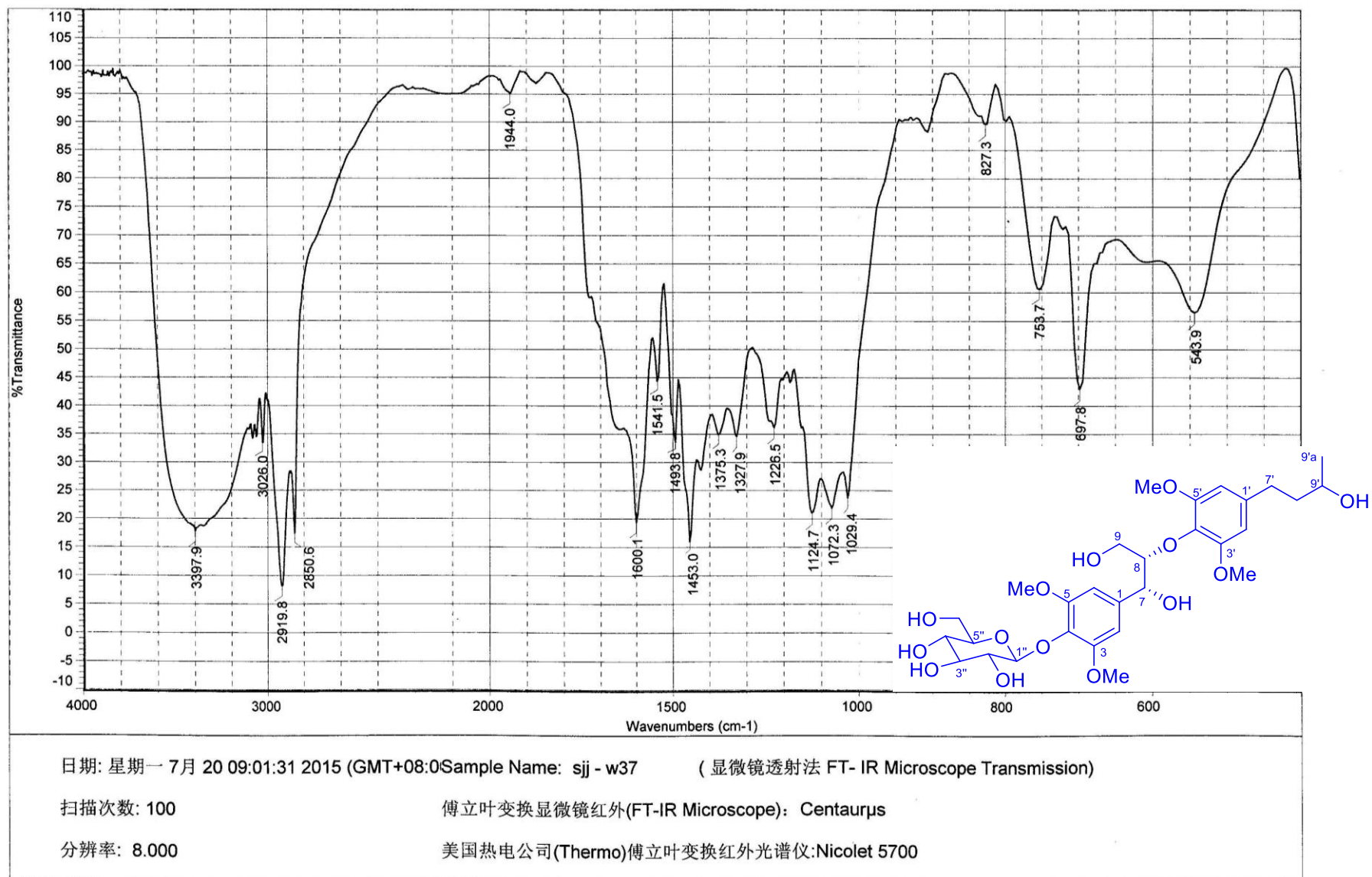


Figure S44. The IR Spectrum of Compound 4

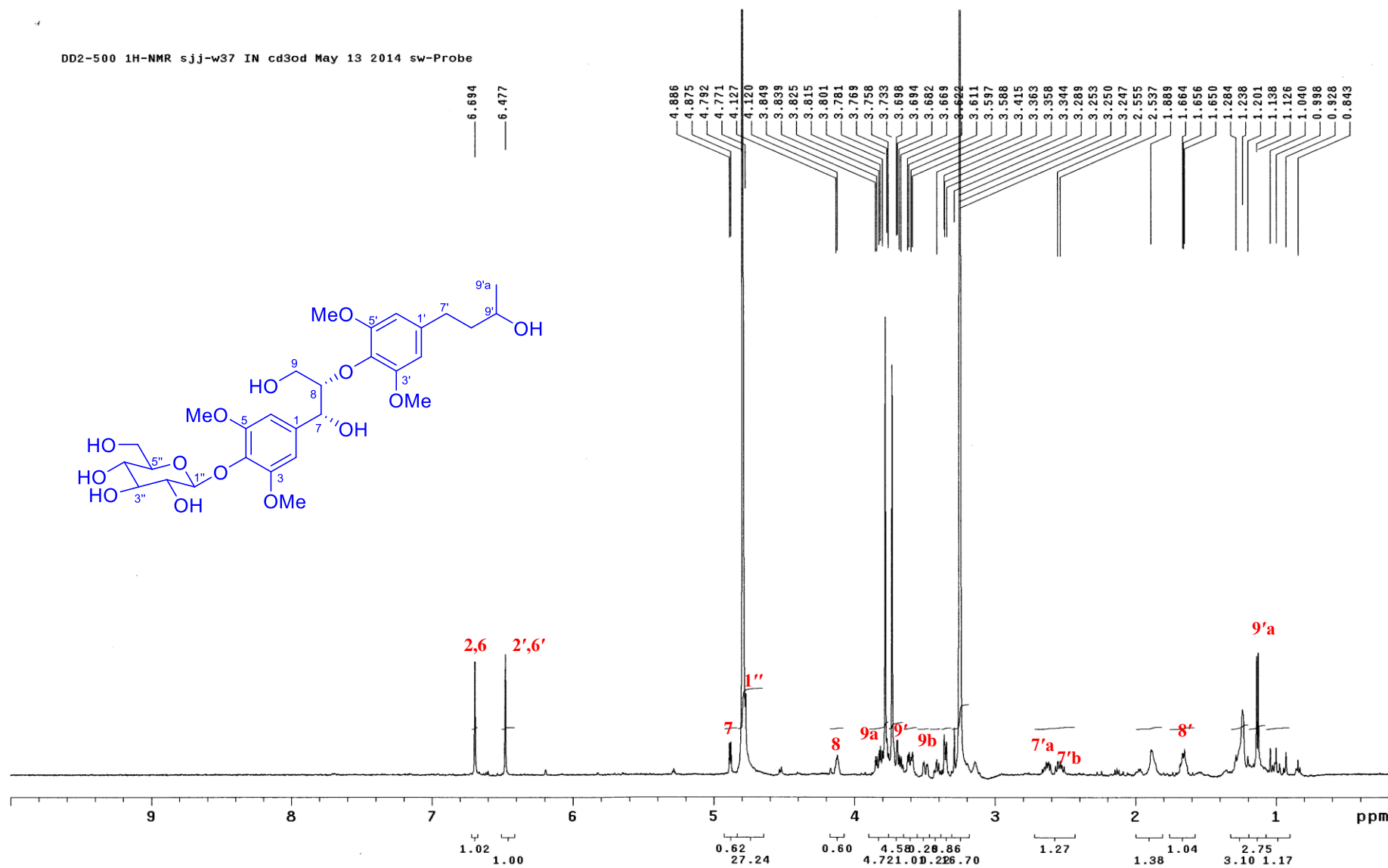


Figure S45. The ¹H NMR Spectrum of Compound 4 in MeOH-*d*₄ (500MHz)

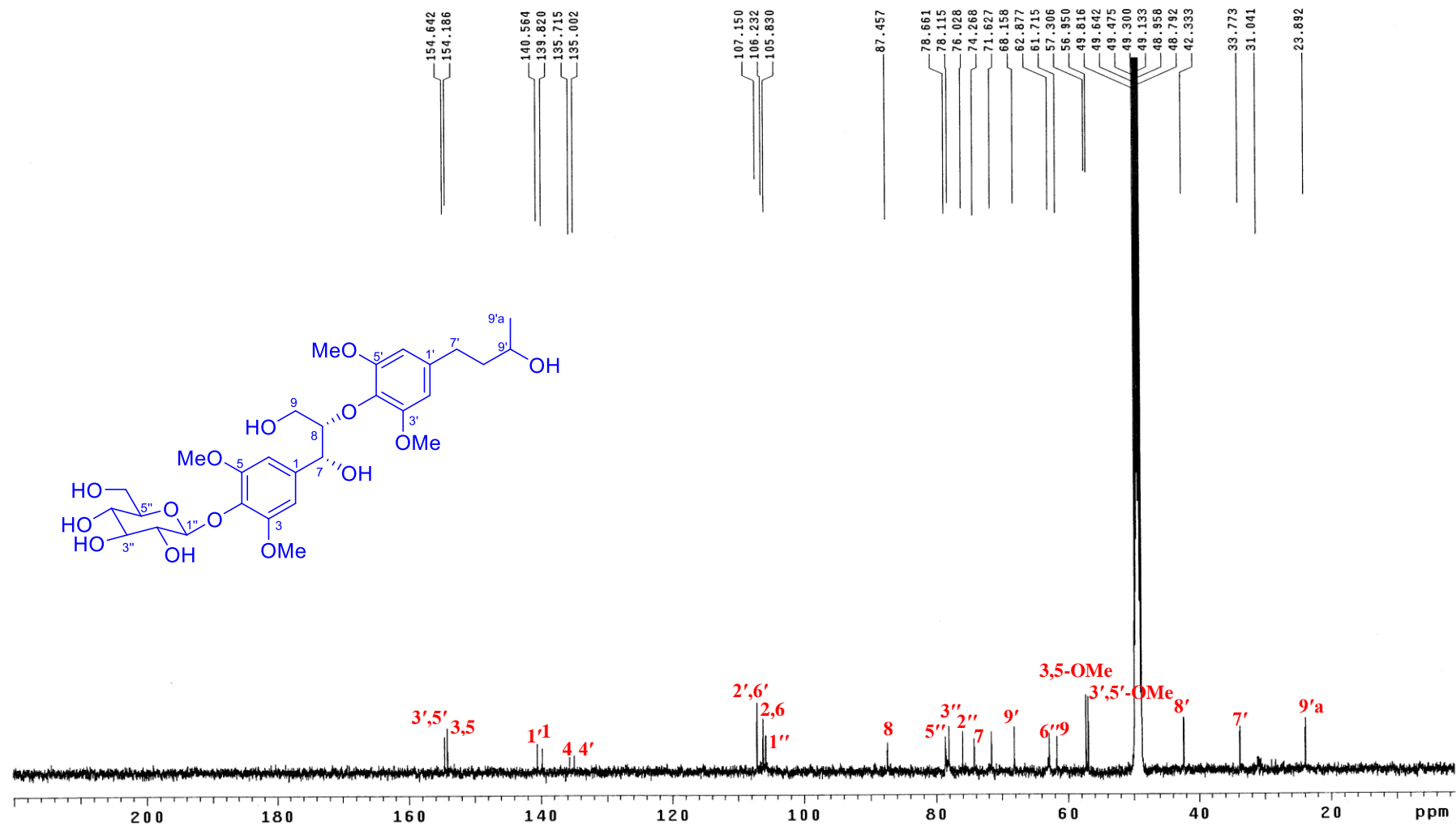


Figure S46. The ^{13}C NMR Spectrum of Compound 4 in $\text{MeOH-}d_4$ (125MHz)

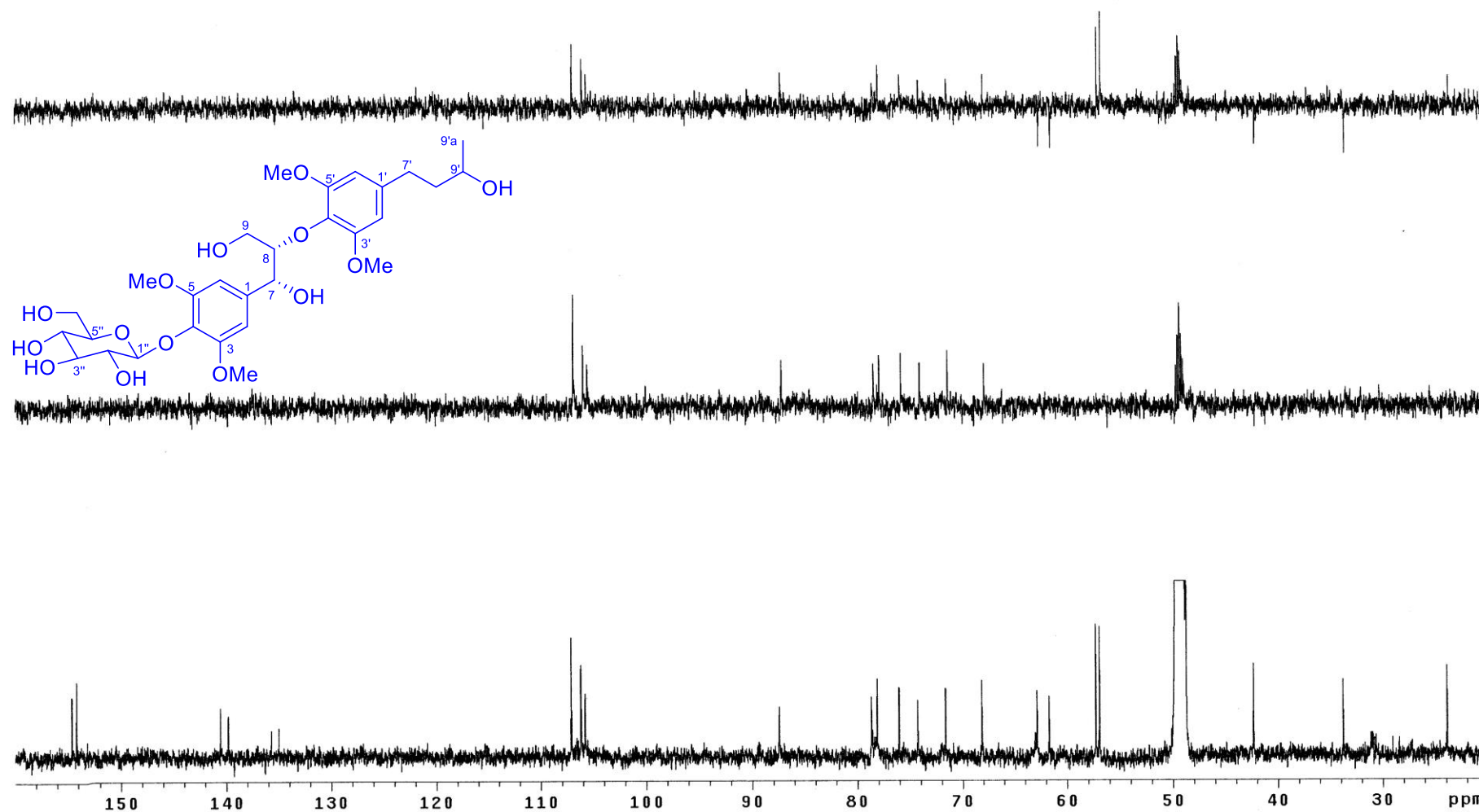
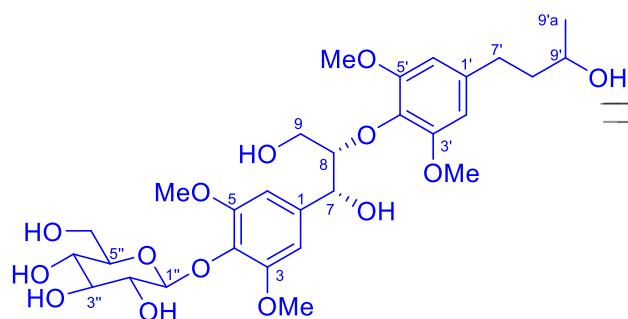


Figure S47. The DEPT NMR Spectrum of Compound 4 in MeOH-*d*₄ (125MHz)

DD2-500 gCOSY sjj-w-37 IN cd3od Oct 31 2014 sw

Temp. 25.0 C / 298.1 K
Sample #9, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 5000.0 Hz
2D Width 5000.0 Hz
4 repetitions
200 increments
OBSERVE H1, 499.7694110 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.025 sec
FT size 2048 x 2048
Total time 16 min



F2
(ppm)

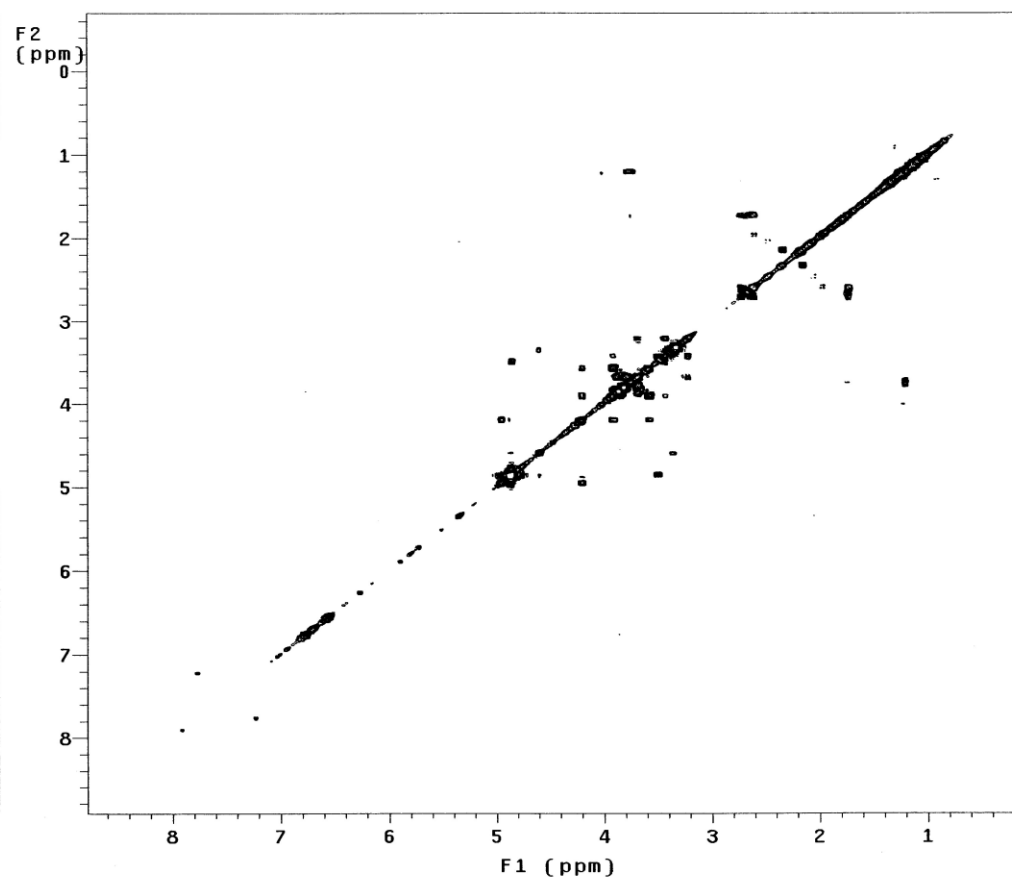


Figure S48. The ^1H - ^1H COSY Spectrum of Compound 4 in $\text{MeOH-}d_4$ (500MHz)

DD2-500 gHSQCAD sjj-w-37 IN cd3od Oct 31 2014 sw

Temp. 25.0 C / 298.1 K
Sample #9, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.240 sec
Width 5000.0 Hz
2D Width 25133.5 Hz
32 repetitions
2 x 200 increments
OBSERVE H1, 499.7694110 MHz
DECOUPLE C13, 125.6784285 MHz
Power 40 dB
on during acquisition
off during delay
W40_sw modulated
DATA PROCESSING
Gauss apodization 0.069 sec
F1 DATA PROCESSING
Gauss apodization 0.007 sec
FT size 4096 x 2048
Total time 4 hr, 16 min

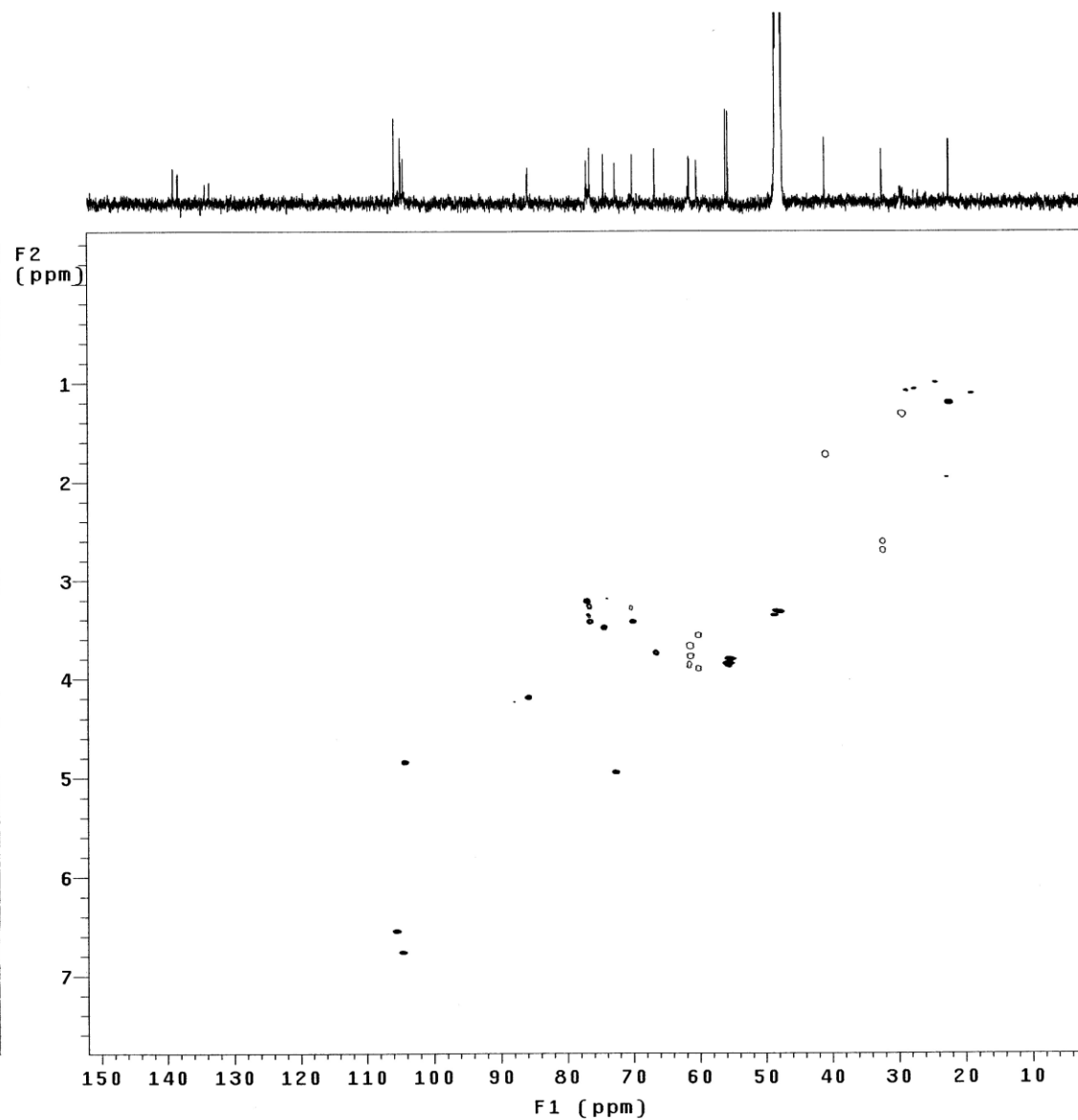
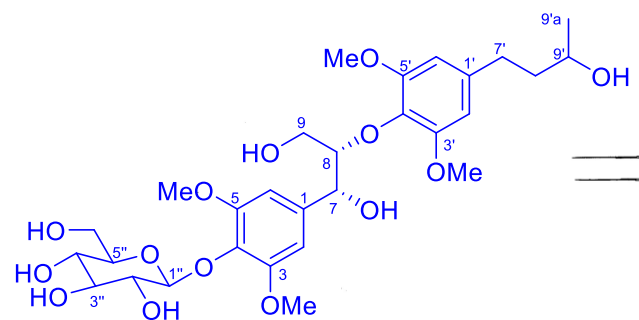


Figure S49. The HSQC Spectrum of Compound 4 in MeOH- d_4 (500MHz)

DD2-500 gHMBCAD sjj-w-37 IN cd3od Nov 1 2014 sw

Temp. 25.0 C / 298.1 K
Sample #9, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.240 sec
Width 5000.0 Hz
2D Width 30154.5 Hz
64 repetitions
2 x 200 increments
OBSERVE H1, 499.7694110 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Gauss apodization 0.006 sec
FT size 4096 x 2048
Total time 8 hr, 47 min

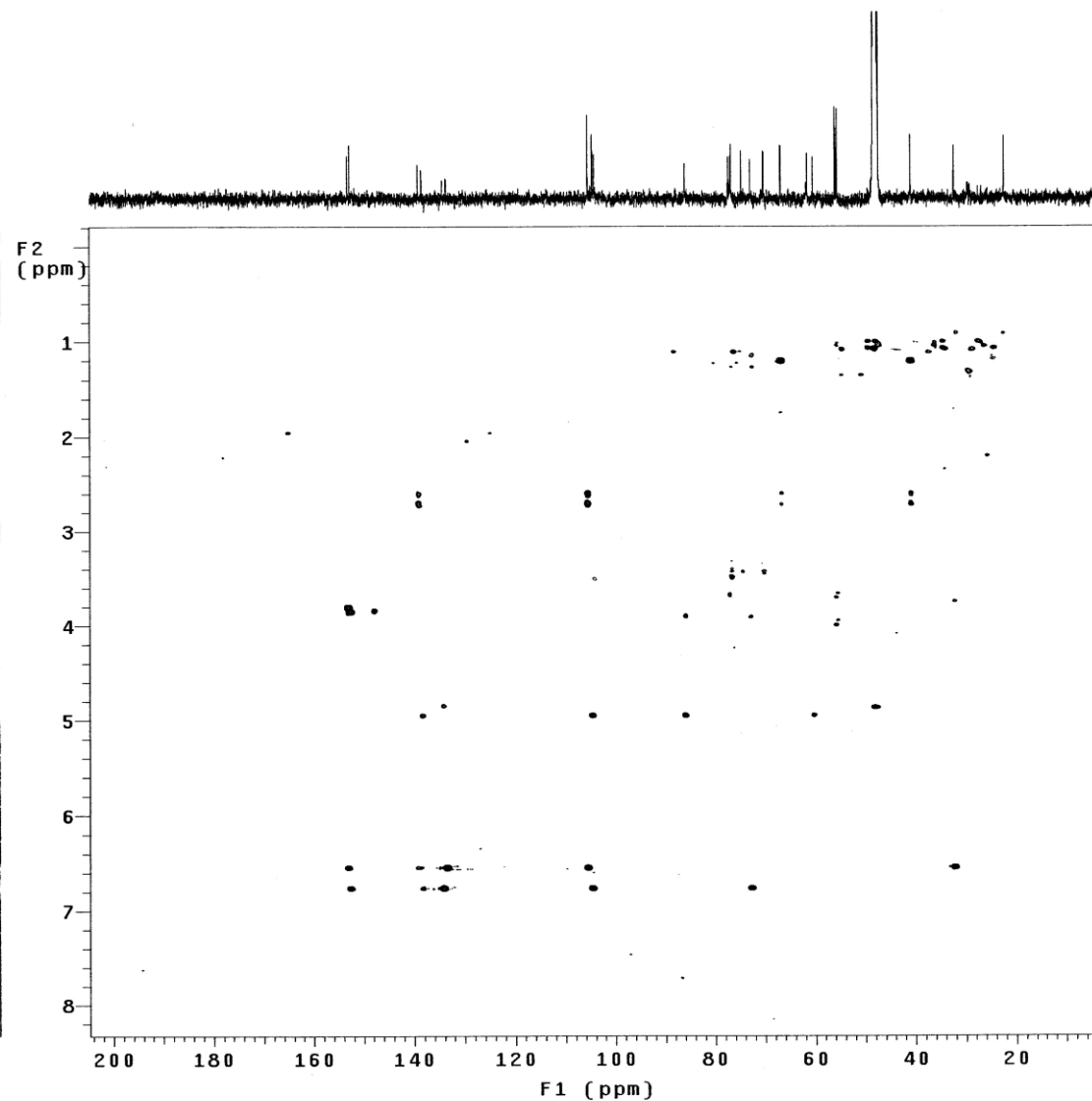
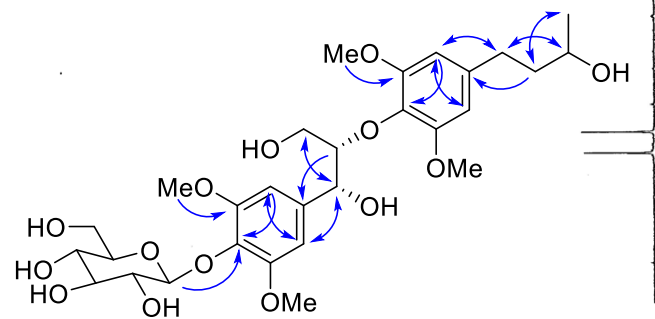
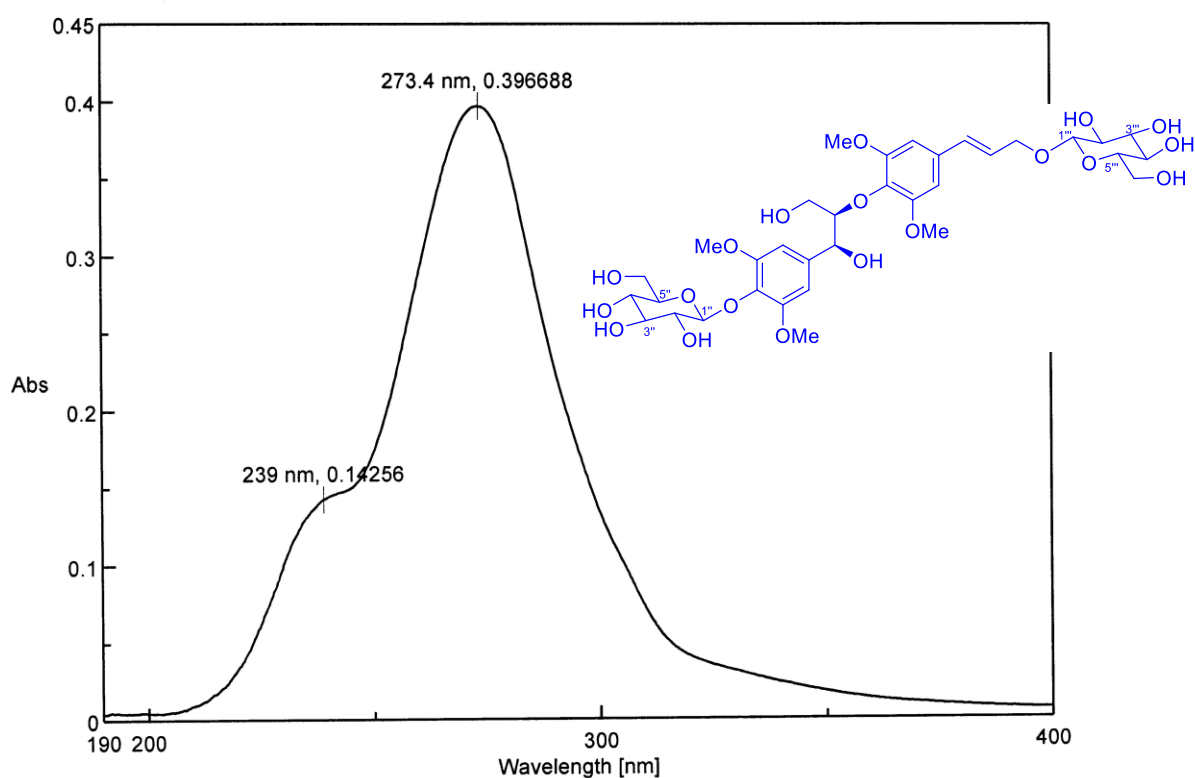


Figure S50. The HMBC Spectrum of Compound 4 in MeOH-*d*₄ (500MHz)



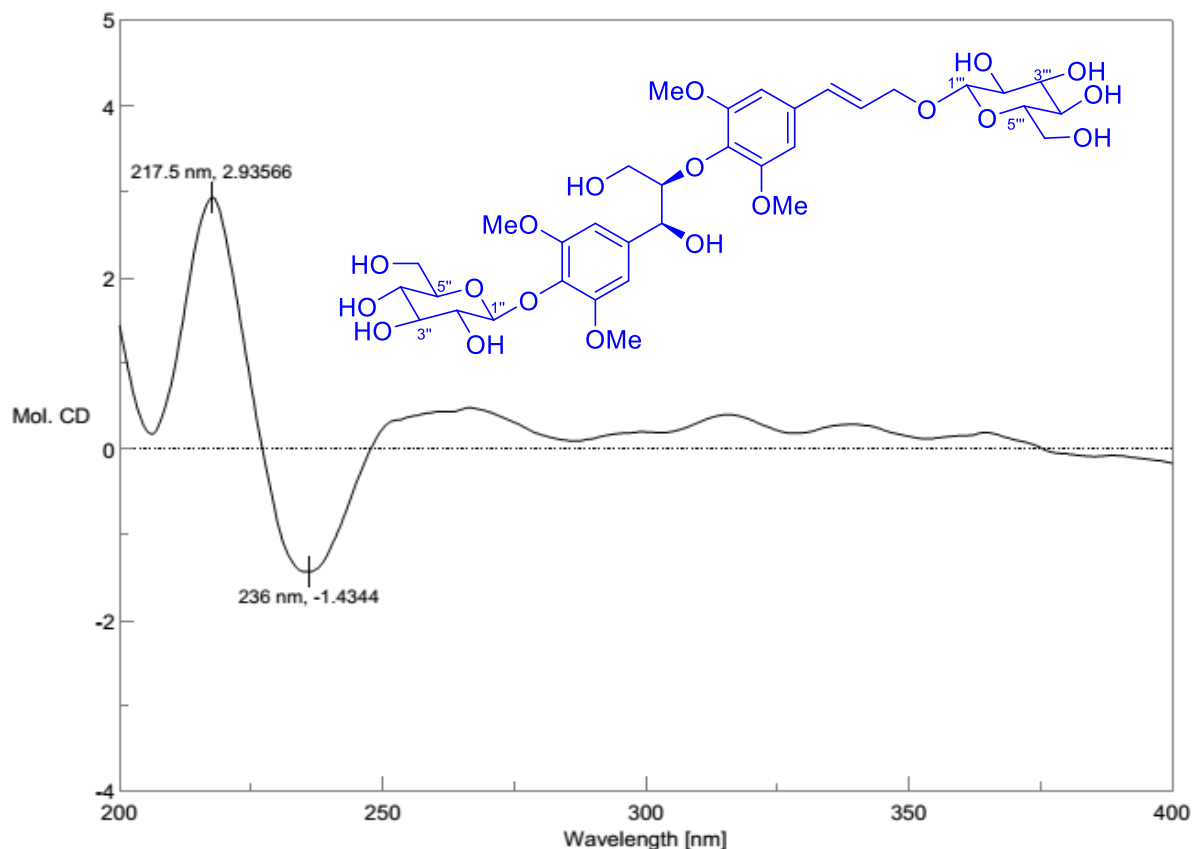
[Comment]
 Sample Name DBT
 Comment 0.02
 User
 Division UV
 Company 324
 [Measurement Information]
 Instrument Name V-650
 Model Name V-650
 Serial No. A034461150

Accessory PSC-718
 Accessory S/N A001761114
 Position 1
 Cell Length 10 mm
 Temperature 20.01 C
 Control Sensor Holder
 Monitor Sensor Holder
 Start Mode Start immediately

Photometric Mode Abs
 Measurement range 400 - 190 nm
 Data pitch 0.2 nm
 Band width(UV/Vis) 2.0 nm
 Response Medium
 Scanning speed 200 nm/min
 Source Change 340 nm
 Light Source D2/WI
 Filter Exchange Step
 Correction Baseline

[Data Information]
 Creation Date 2015-6-30 19:24
 Data array type Linear data array
 Horizontal Wavelength [nm]
 Vertical Abs
 Start 400 nm
 End 190 nm
 Data pitch 0.2 nm
 Data points 1051

Figure S51. The UV Spectrum of Compound 5 in MeOH



[Comments]
 Sample name sjj-w80
 Comment
 User
 Division
 Company dell

[Measurement Information]
 Instrument Name J-815
 Model Name J-815
 Serial No. A024461168

Accessory Standard
 Accessory S/N A024461168
 Cell Length 1 mm

Photometric Mode CD, HT, Abs
 Measure Range 400 - 200 nm
 Data pitch 0.5 nm
 Sensitivity Standard
 D.I.T. 1 sec
 Band width 2.00 nm
 Start Mode Immediately
 Scanning Speed 100 nm/min
 Baseline Correction Baseline
 Shutter Control Auto
 PMT Voltage Auto
 Accumulations 2
 Solvent ME OH
 Concentration 0.086 (w/v)%

[Detailed Information]
 Creation date 2015-7-1 11:23

Data array type Linear data array * 3
 Horizontal axis Wavelength [nm]
 Vertical axis(1) Mol. CD
 Vertical axis(2) HT [V]
 Vertical axis(3) Abs
 Start 400 nm
 End 200 nm
 Data interval 0.5 nm
 Data points 401

Figure S52. The CD Spectrum of Compound 5 in MeOH

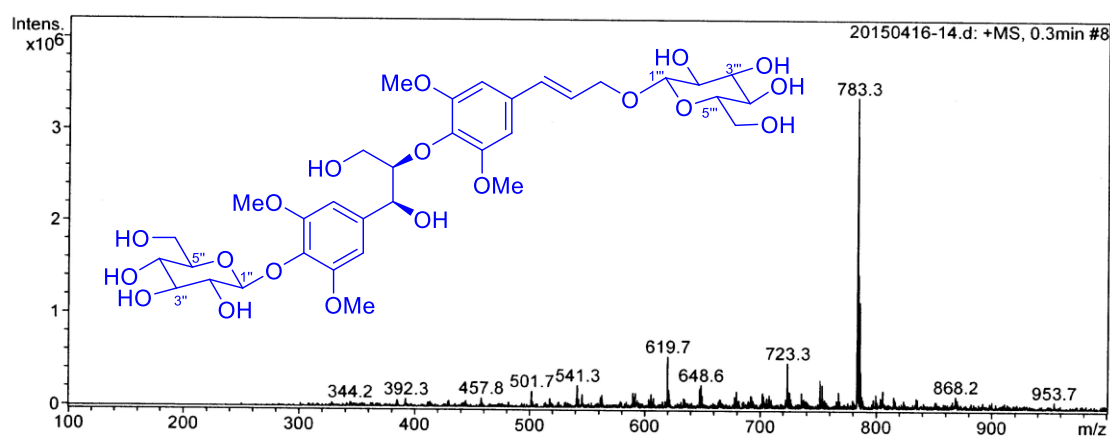
Compound Mass Spectrum Report - MS

Analysis Name: 20150416-14.d **Instrument:** LC-MSD-Trap-SL **Print Date:** 2015-6-8 12:18:04
Method: linsheng.m **Operator:** Operator **Acq. Date:** 2015-4-16 22:52:55
Sample Name: SJJ-w80
Analysis Info:

Acquisition Parameter:

| | | | | | |
|-----------------|------------|-----------------------|-------------|----------------|-----------|
| Mass Range Mode | Std/Normal | Trap Drive | 82.8 | Scan Begin | 100 m/z |
| Ion Polarity | Positive | Skim 1 | -40.0 Volt | Scan End | 1000 m/z |
| Ion Source Type | ESI | Skim 2 | 5.0 Volt | Averages | 7 Spectra |
| Dry Temp (Set) | 330 ℃ | Octopole RF Amplitude | 200.0 Vpp | Max. Accu Time | 200000 釐 |
| Nebulizer (Set) | 15.00 psi | Capillary Exit | -151.0 Volt | ICC Target | 30000 |
| Dry Gas (Set) | 6.00 l/min | | | Charge Control | on |

+MS, 0.3min #8



-MS, 0.3min #7

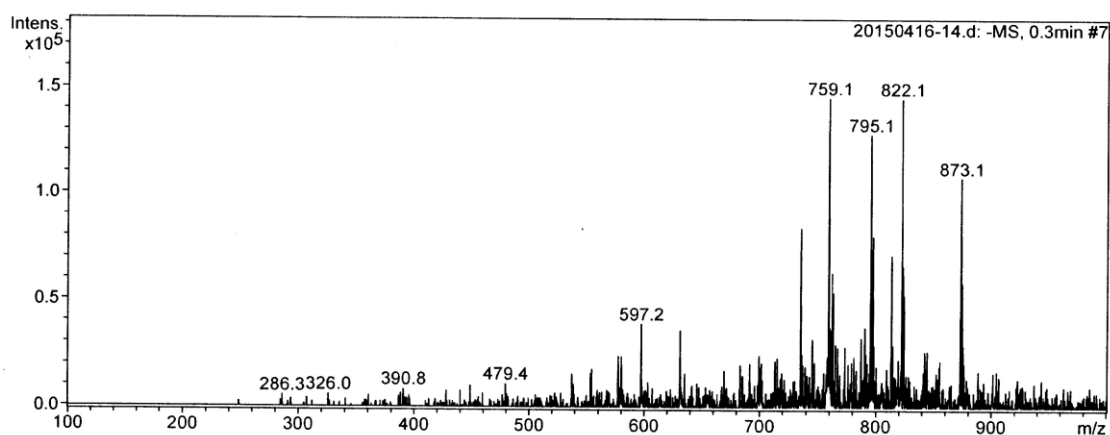


Figure S53. The ESIMS Spectrum of Compound 5

日期:2015-06-17

仪器:API-TOFMS 10000 广州禾信分析仪器有限公司

时间:从0.2900到0.5060

用户单位名称:中国医学科学院药物研究所

样品名称:sjj-w80

离子模式:Positive

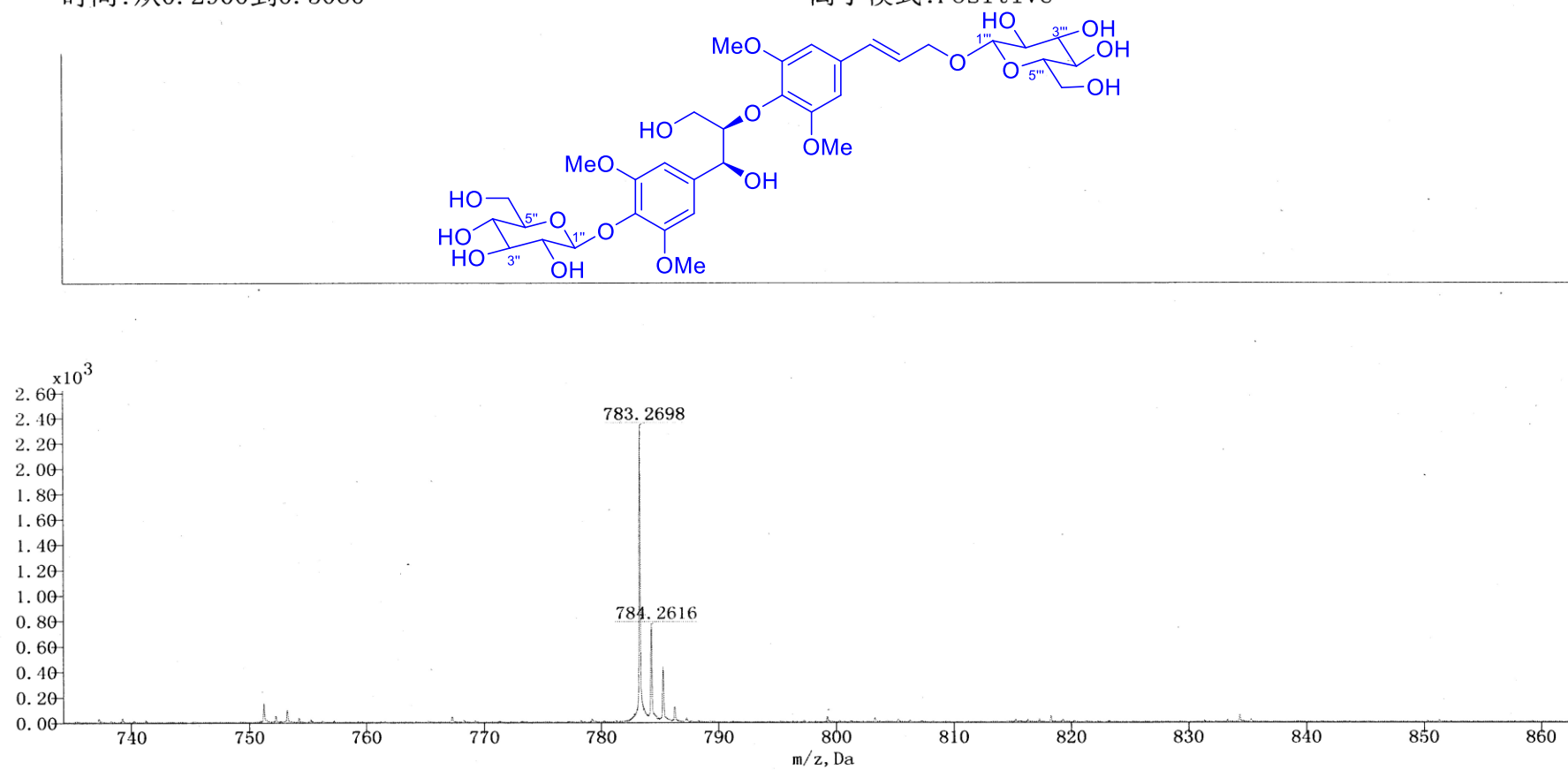
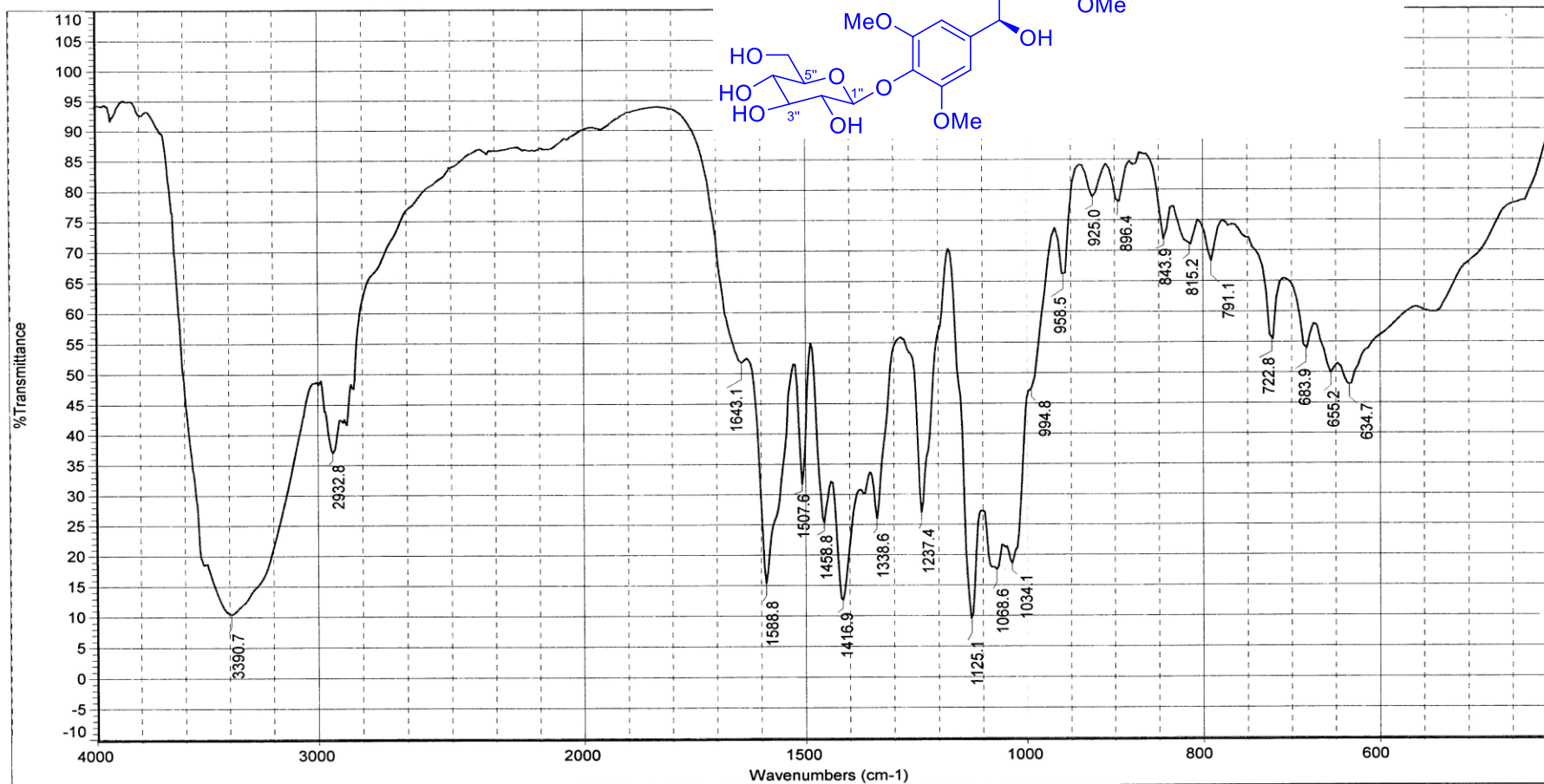


Figure S54. The HRESIMS Spectrum of Compound 5



日期: 星期三 7月 01 10:19:42 2015 (GMT+08:00) Sample Name: sjj - w80

(显微镜透射法 FT- IR Microscope Transmission)

扫描次数: 100

傅里叶变换显微镜红外(FT-IR Microscope): Centaurus

分辨率: 8.000

美国热电公司(Thermo)傅里叶变换红外光谱仪:Nicolet 5700

Figure S55. The IR Spectrum of Compound 5

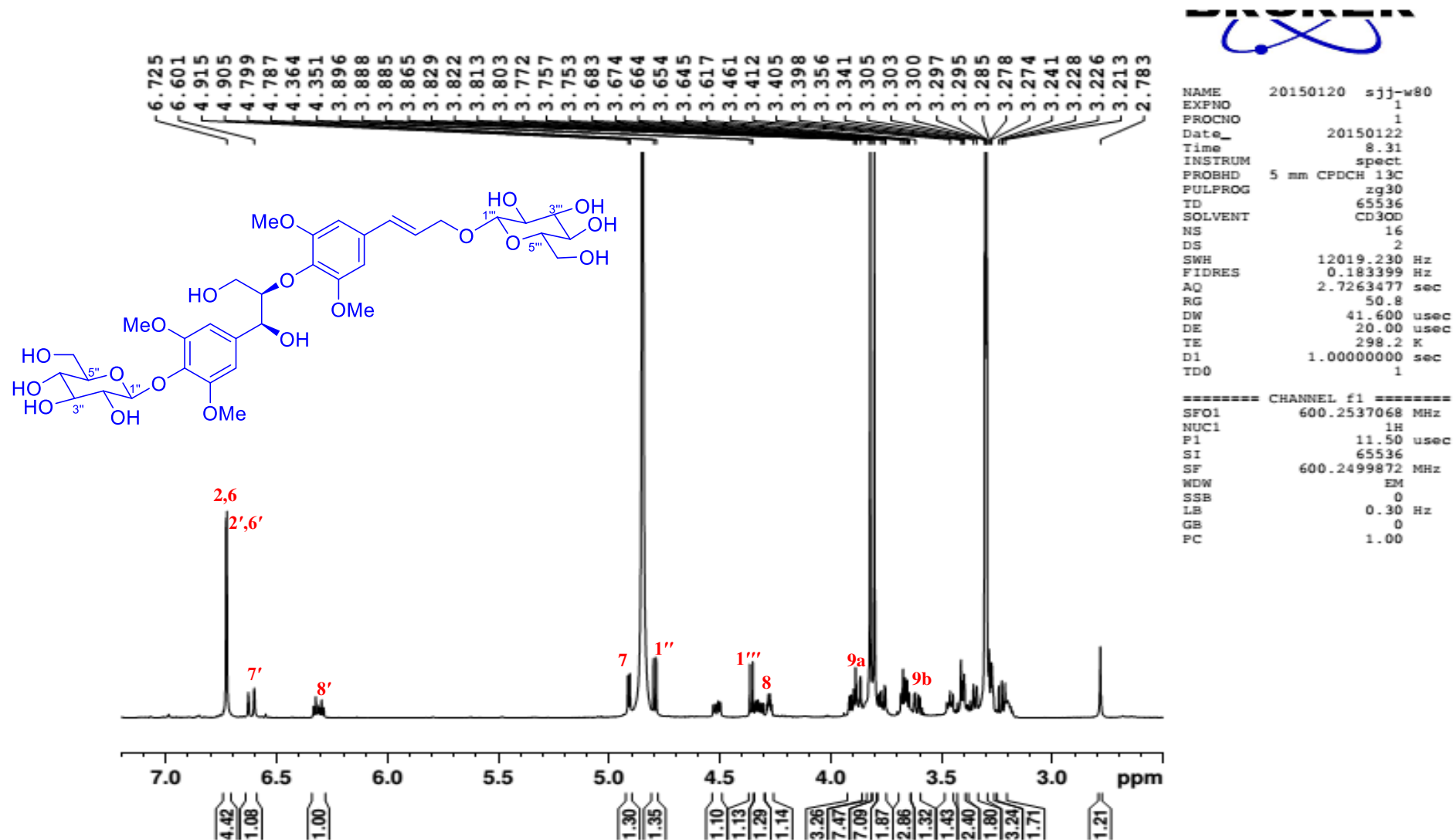


Figure S56. The ^1H NMR Spectrum of Compound 5 in $\text{MeOH-}d_4$ (600MHz)

Bruker AVIIIHD 600 20150112 sjj-w80
 C13 CD3OD D:\ DATA2015 16

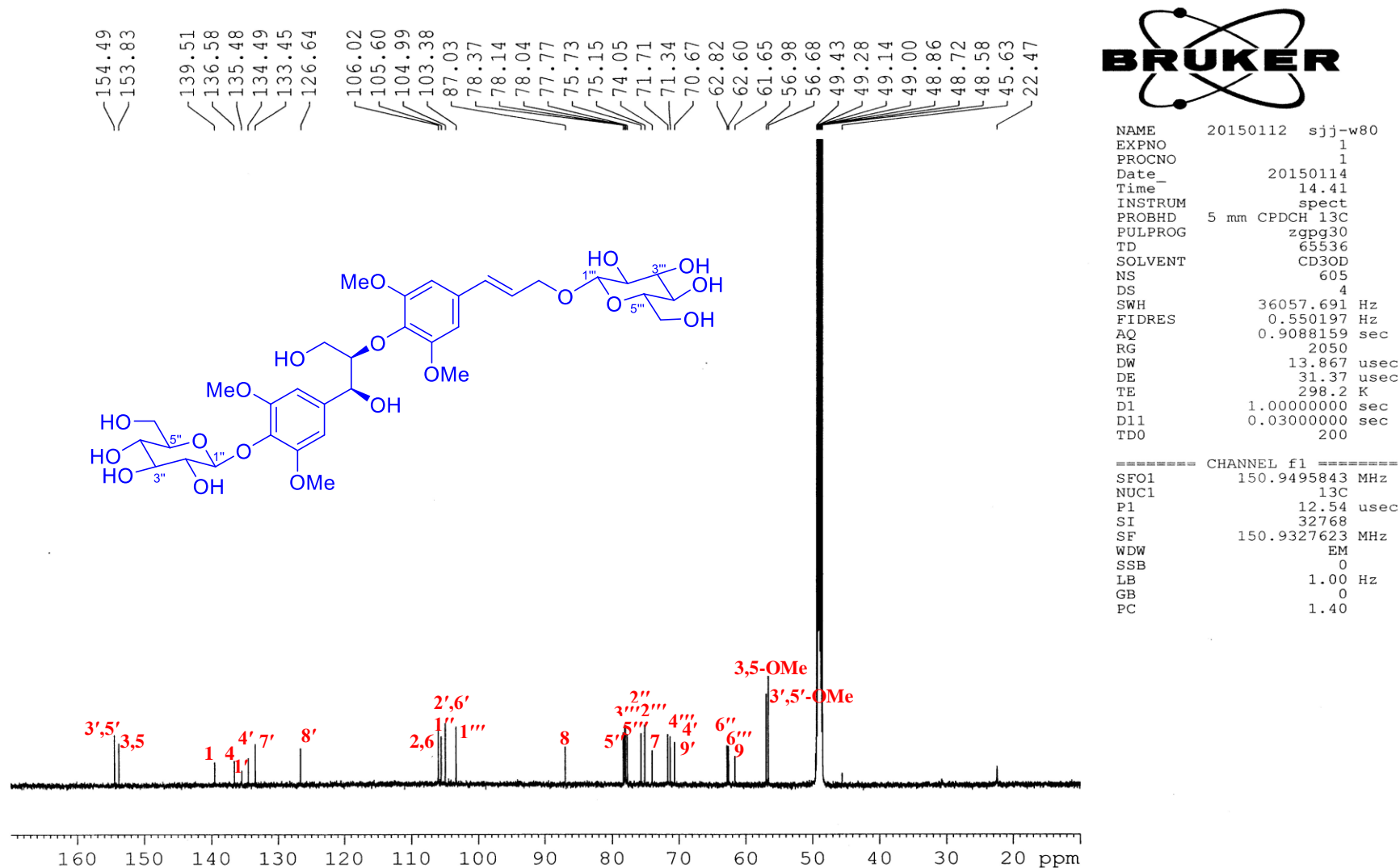


Figure S57. The ^{13}C NMR Spectrum of Compound 5 in $\text{MeOH-}d_4$ (150MHz)

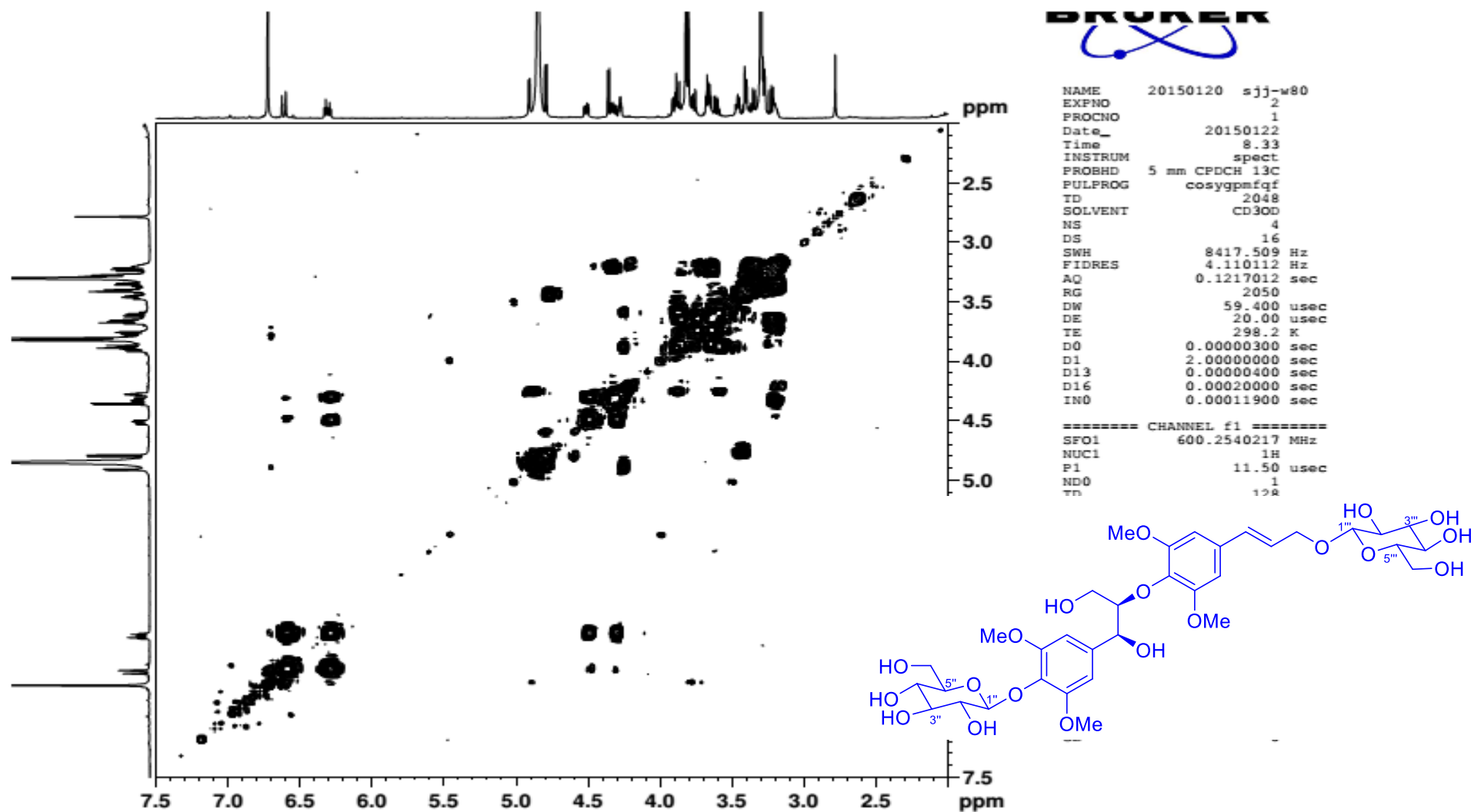


Figure S58. The ^1H - ^1H COSY Spectrum of Compound 5 in $\text{MeOH-}d_4$ (600MHz)

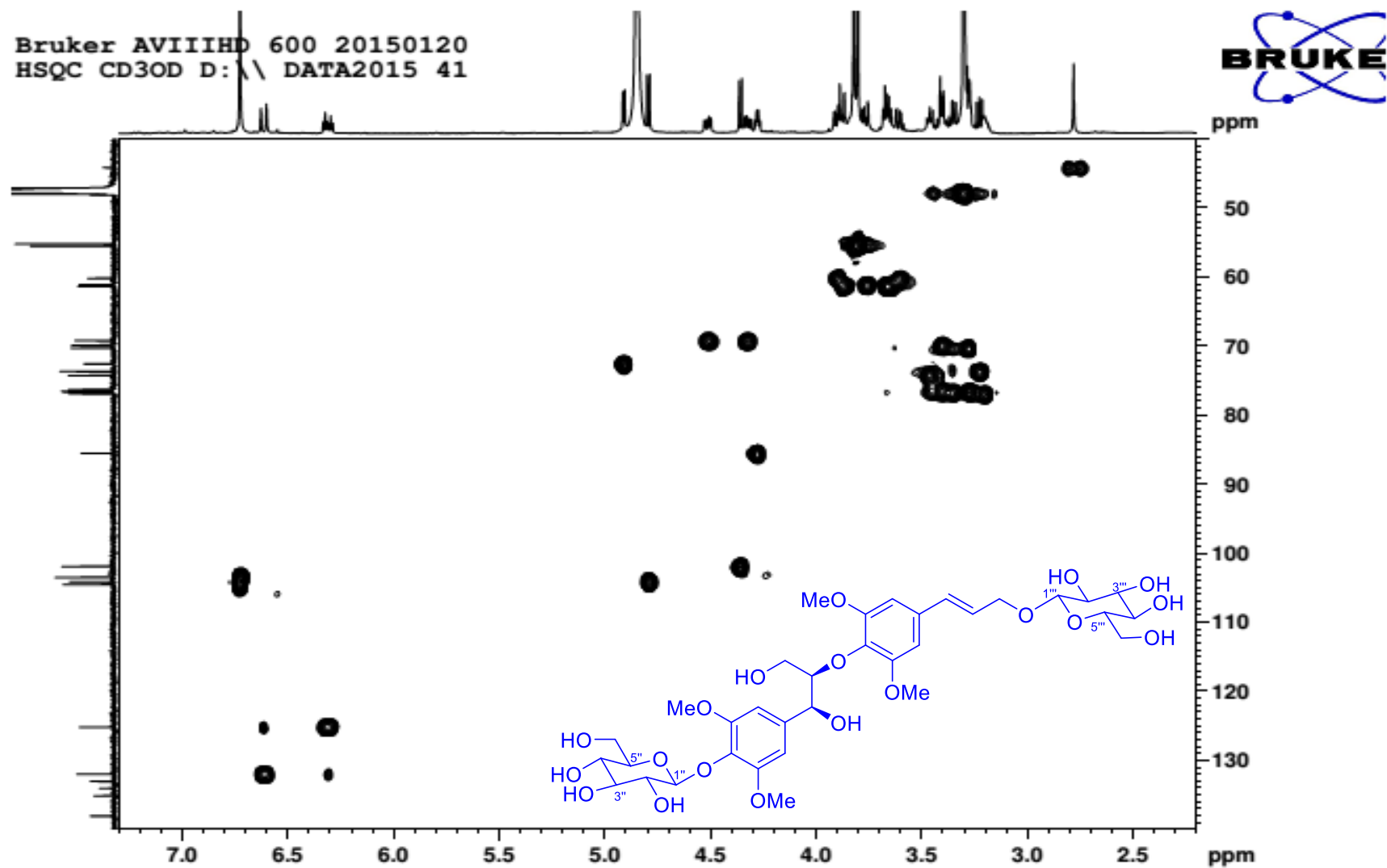


Figure S59. The HSQC Spectrum of Compound 5 in MeOH- d_4 (600MHz)

Bruker AVIIIHD 600 20150120
HMBC CD3OD D: \\ DATA2015 41

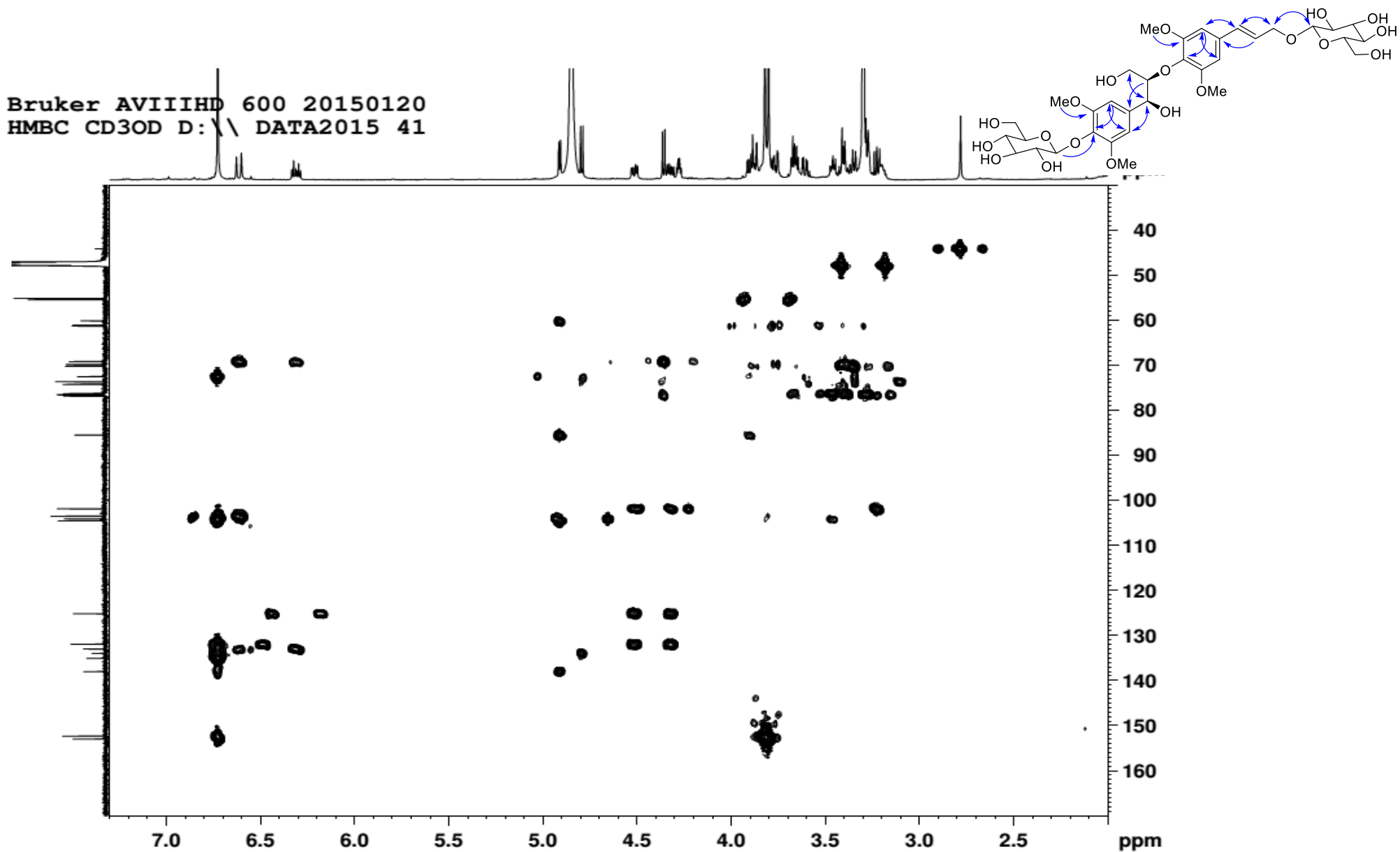
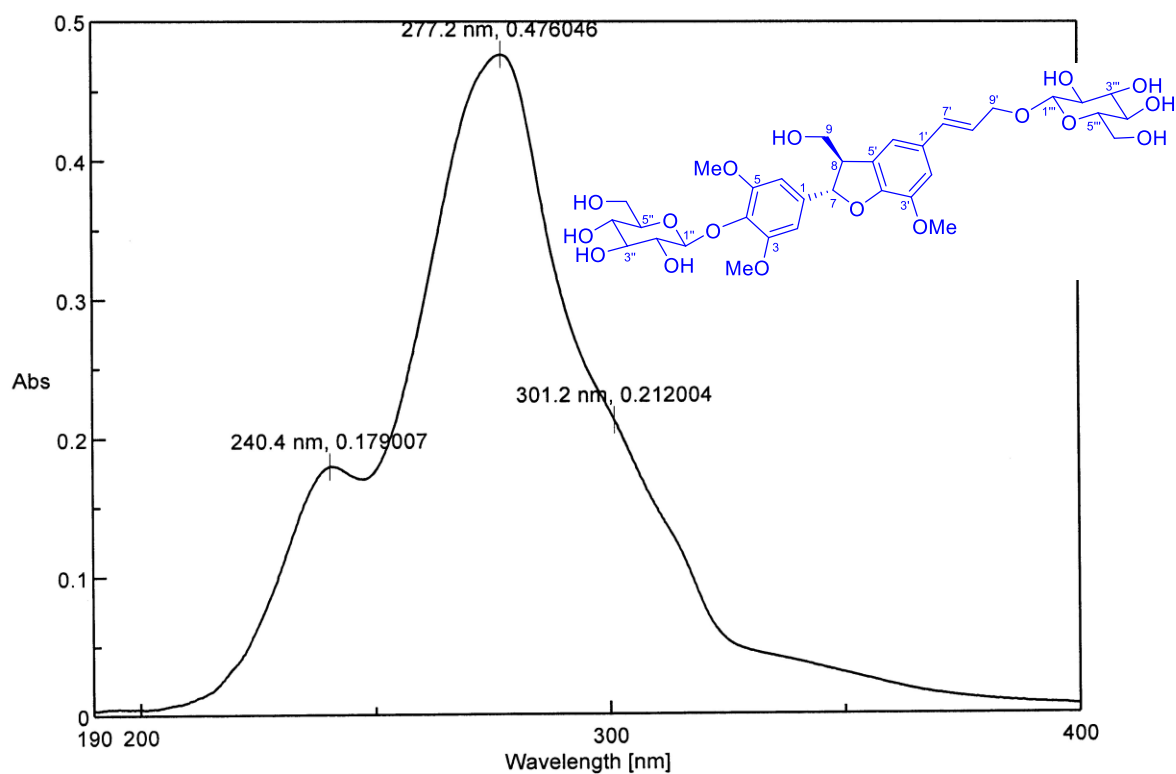


Figure S60. The HMBC Spectrum of Compound 5 in MeOH-*d*₄ (600MHz)



[Comment]
 Sample Name DBT
 Comment 0.02
 User
 Division UV
 Company 324
 [Measurement Information]
 Instrument Name V-650
 Model Name V-650
 Serial No. A034461150

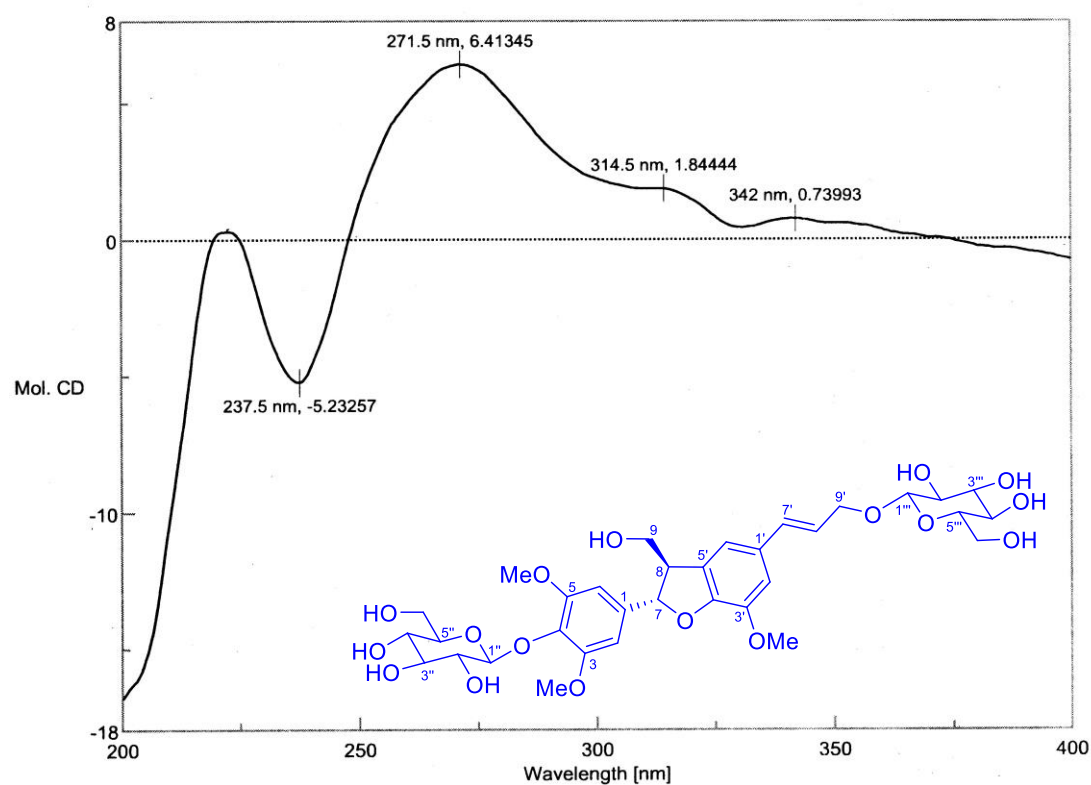
Accessory PSC-718
 Accessory S/N A001761114
 Position 1
 Cell Length 10 mm
 Temperature 19.97 C
 Control Sensor Holder
 Monitor Sensor Holder
 Start Mode Start immediately

Photometric Mode Abs
 Measurement range 400 - 190 nm
 Data pitch 0.2 nm
 Band width(UV/Vis) 2.0 nm
 Response Medium
 Scanning speed 200 nm/min
 Source Change 340 nm
 Light Source D2/WI
 Filter Exchange Step
 Correction Baseline

[Data Information]
 Creation Date 2015-6-30 19:02
 Data array type Linear data array
 Horizontal Wavelength [nm]
 Vertical Abs
 Start 400 nm
 End 190 nm
 Data pitch 0.2 nm
 Data points 1051

SJJ-W78

Figure S61. The UV Spectrum of Compound 6 in MeOH



[Comments]
Sample name sjj-w78
Comment
User
Division
Company dell

[Measurement Information]
Instrument Name J-815
Model Name J-815
Serial No. A024461168

Accessory Standard
Accessory S/N A024461168
Cell Length 1 mm

Photometric Mode CD, HT, Abs
Measure Range 400 - 200 nm
Data pitch 0.5 nm
Sensitivity Standard
D.I.T. 1 sec
Band width 2.00 nm
Start Mode Immediately
Scanning Speed 100 nm/min
Baseline Correction Baseline
Shutter Control Auto
PMT Voltage Auto
Accumulations 2
Solvent MEOH
Concentration 0.028 (w/v)%

[Detailed Information]
Creation date 2015-7-1 10:56

Data array type Linear data array * 3
Horizontal axis Wavelength [nm]
Vertical axis(1) Mol. CD
Vertical axis(2) HT [V]
Vertical axis(3) Abs
Start 400 nm
End 200 nm
Data interval 0.5 nm
Data points 401

Figure S62. The CD Spectrum of Compound 6 in MeOH

Compound Mass Spectrum Report - MS

Analysis Name: 20150416-1.d **Instrument:** LC-MSD-Trap-SL **Print Date:** 2015-6-5 15:46:08
Method: standby.m **Operator:** Operator **Acq. Date:** 2015-4-16 21:33:45
Sample Name: SJJ-w78
Analysis Info:

Acquisition Parameter:

| | | | | | |
|-----------------|------------|-----------------------|-------------|----------------|-----------|
| Mass Range Mode | Std/Normal | Trap Drive | 75.3 | Scan Begin | 100 m/z |
| Ion Polarity | Positive | Skim 1 | -40.0 Volt | Scan End | 1000 m/z |
| Ion Source Type | ESI | Skim 2 | 5.0 Volt | Averages | 5 Spectra |
| Dry Temp (Set) | 330 度 | Octopole RF Amplitude | 200.0 Vpp | Max. Accu Time | 200000 秒 |
| Nebulizer (Set) | 15.00 psi | Capillary Exit | -143.5 Volt | ICC Target | 30000 |
| Dry Gas (Set) | 6.00 l/min | | | Charge Control | on |

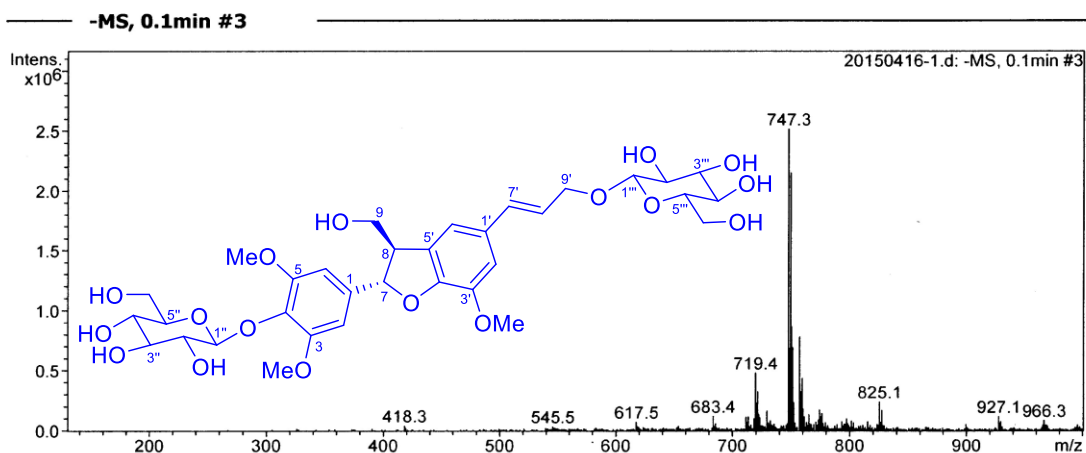
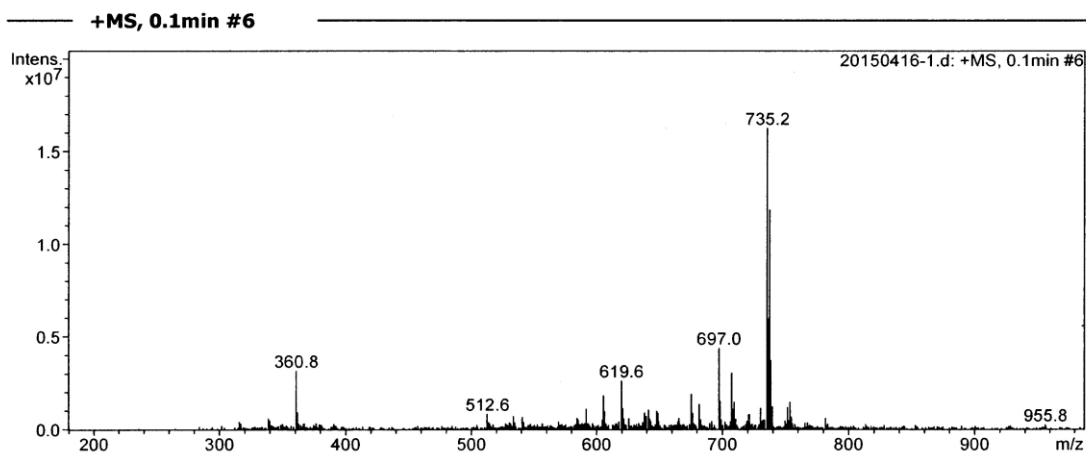


Figure S63. The ESIMS Spectrum of Compound 6

日期:2015-06-17
仪器:API-TOFMS 10000 广州禾信分析仪器有限公司
时间:从0.2900到0.5060

用户单位名称:中国医学科学院药物研究所
样品名称:sjj-w78
离子模式:Positive

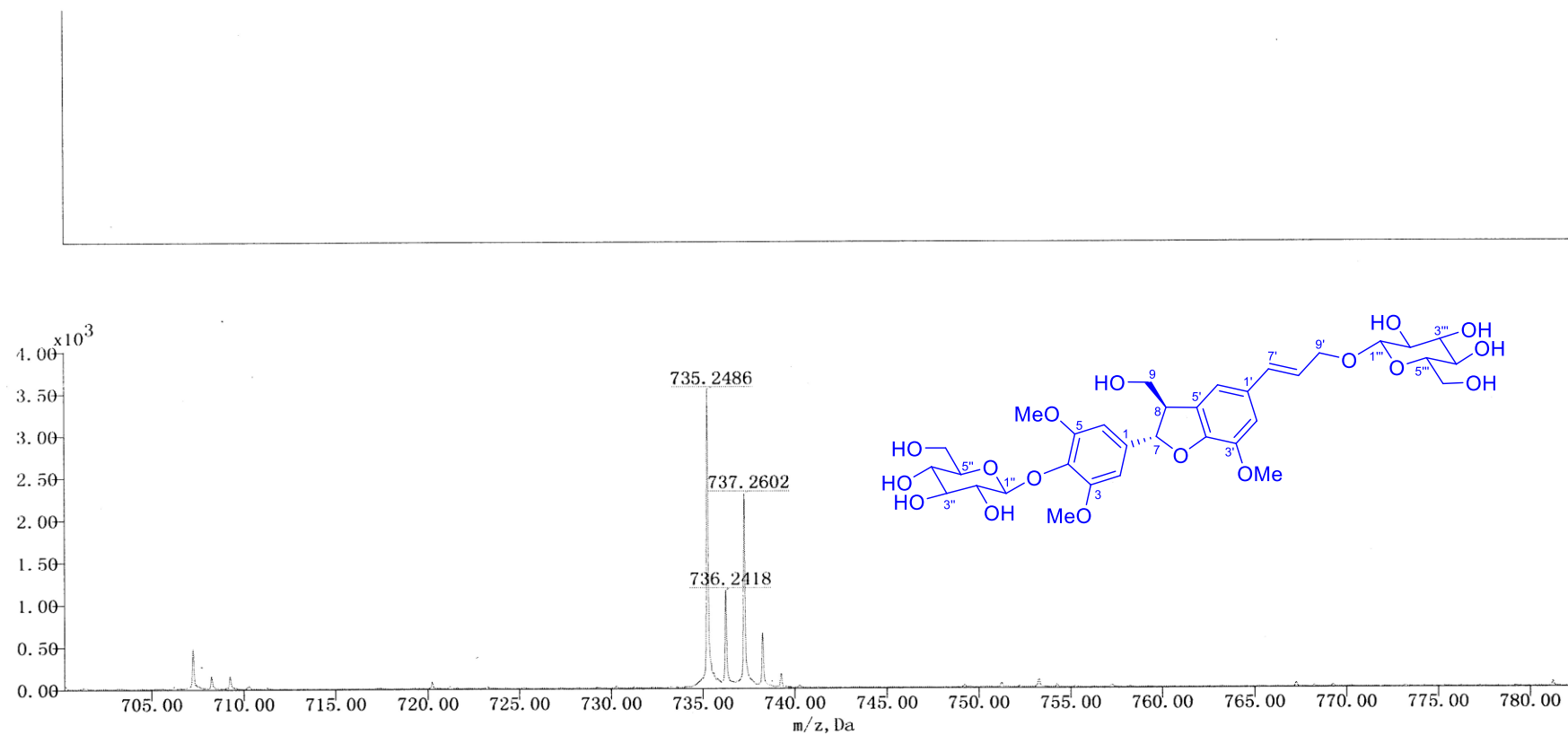
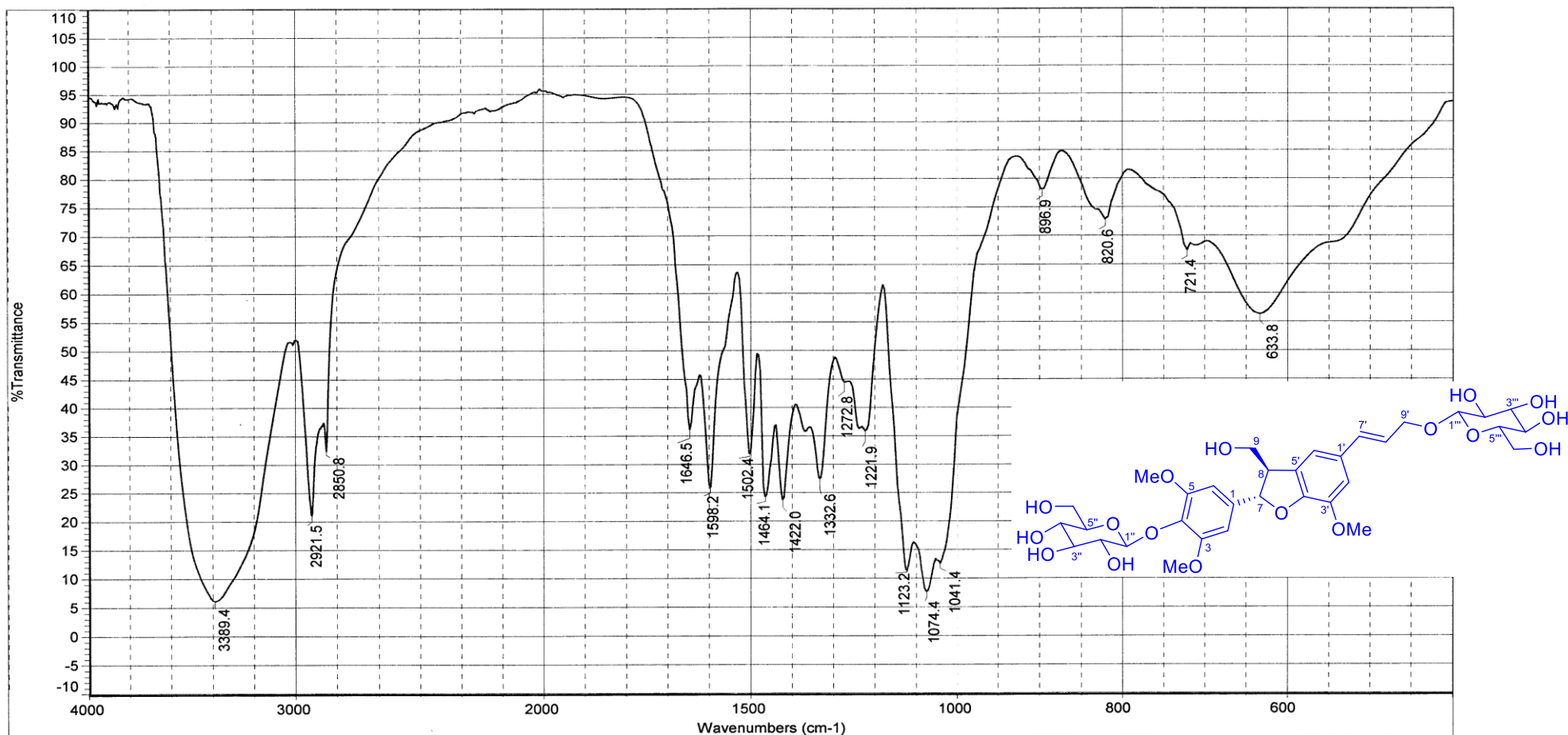


Figure S64. The HRESIMS Spectrum of Compound 6



日期: 星期三 7月 01 10:08:01 2015 (GMT+08:00) Sample Name: sjj - w78

(显微镜透射法 FT- IR Microscope Transmission)

扫描次数: 100

傅里叶变换显微镜红外(FT-IR Microscope): Centaurus

分辨率: 8.000

美国热电公司(Thermo)傅里叶变换红外光谱仪:Nicolet 5700

Figure S65. The IR Spectrum of Compound 6

Bruker AVIIIHD 600 20141229 sjj-w78
PROTON CD3OD D:\\ DATA2014 1

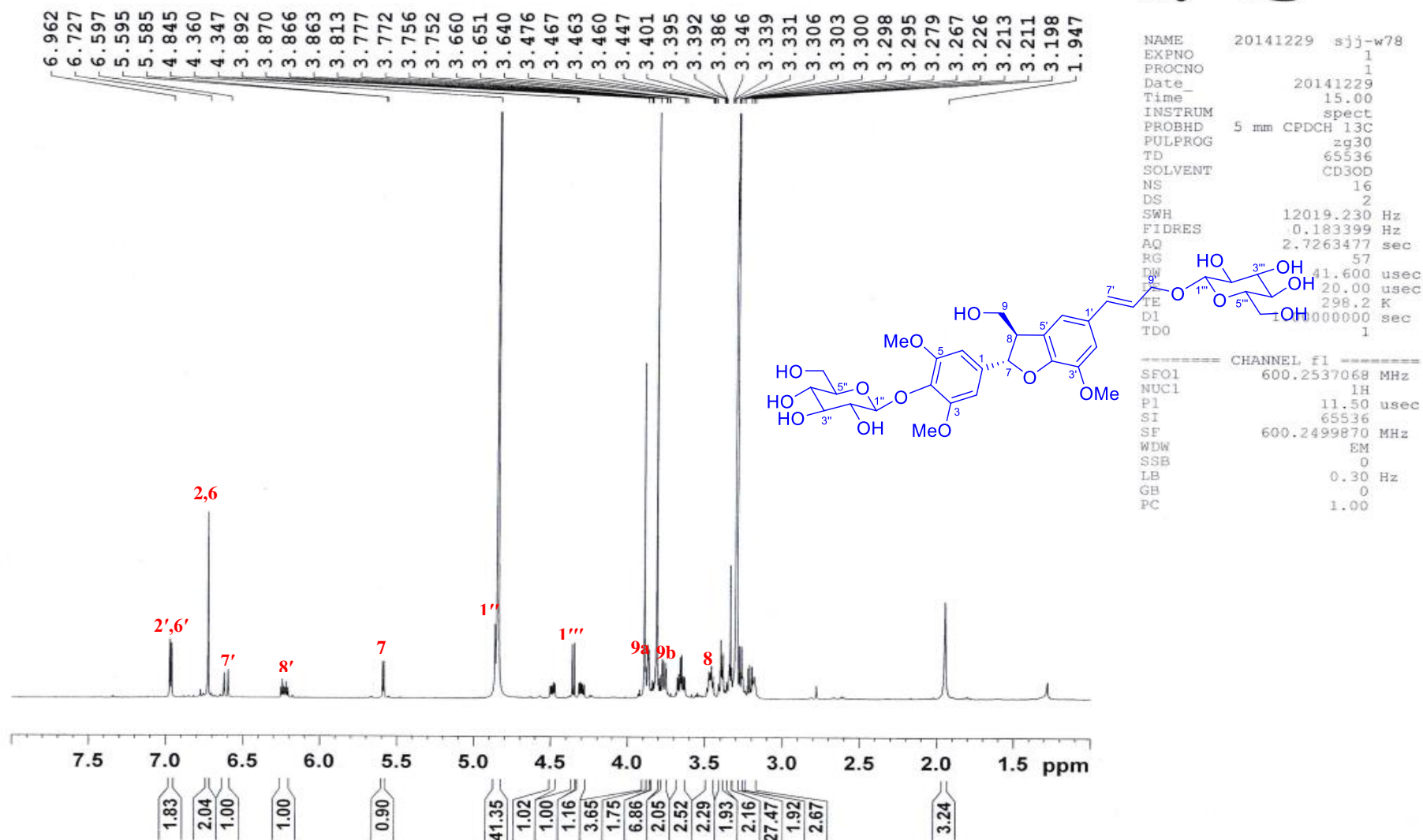


Figure S66. The ^1H NMR Spectrum of Compound 6 in $\text{MeOH-}d_4$ (600MHz)

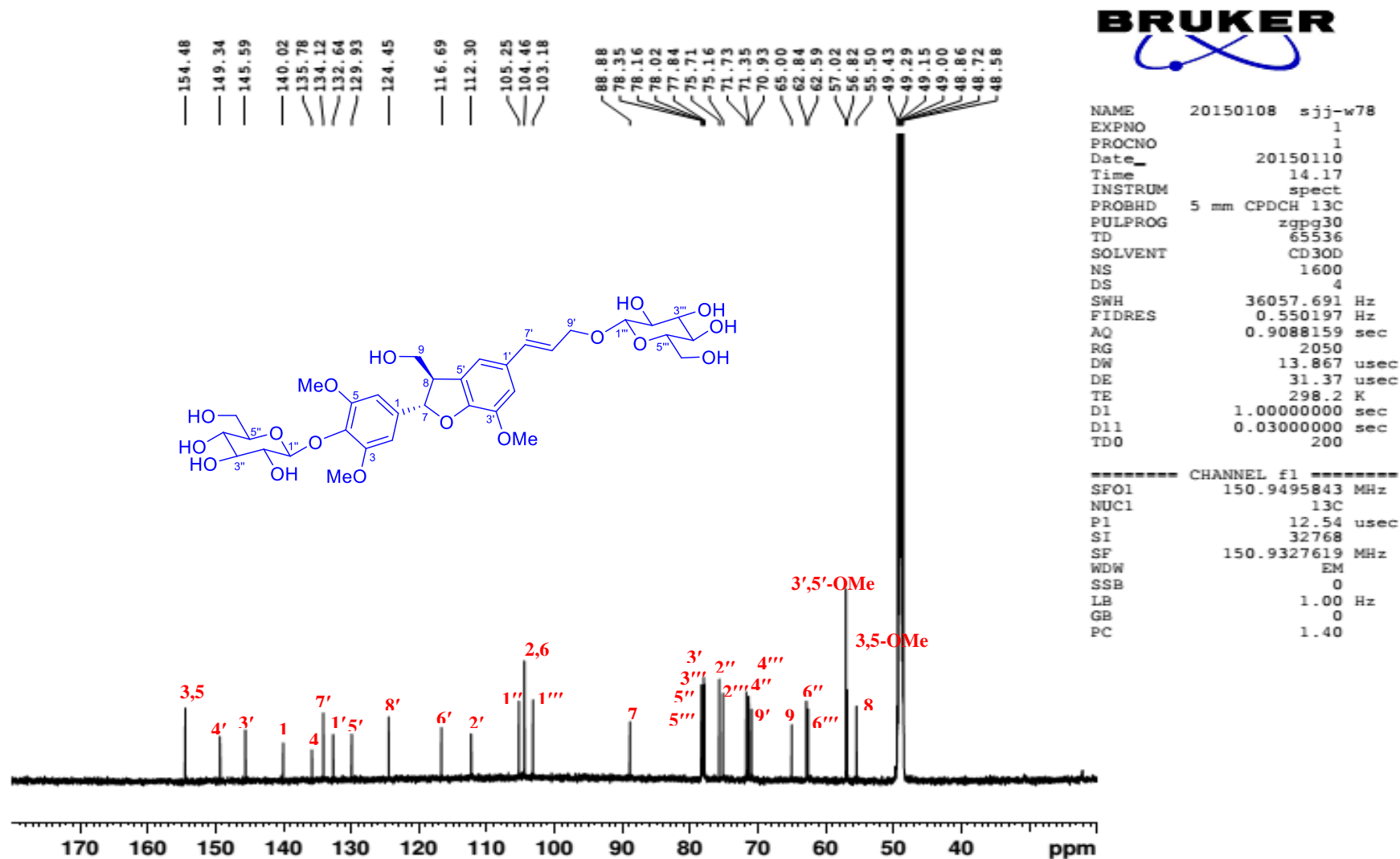
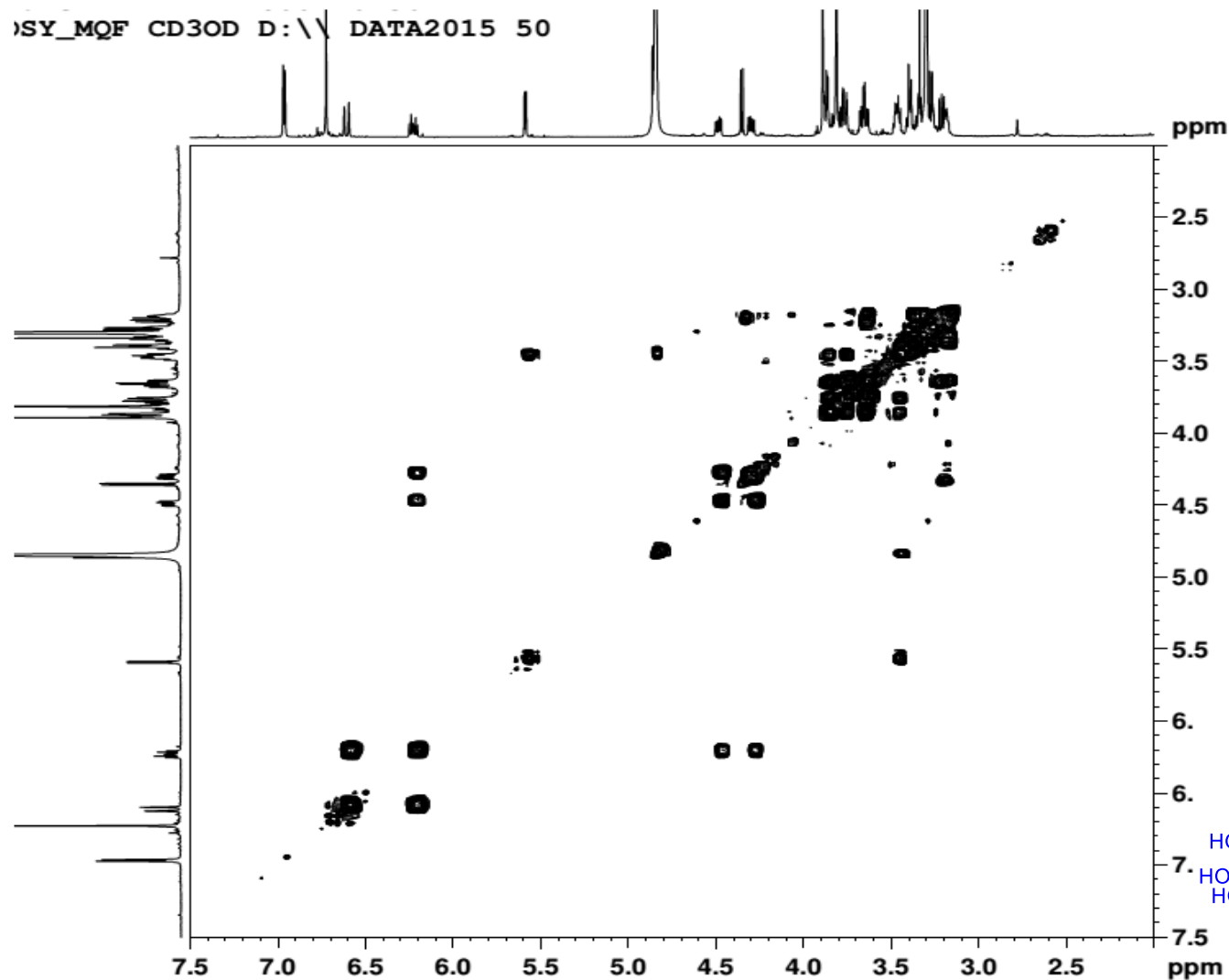


Figure S67. The ¹³C NMR Spectrum of Compound 6 in MeOH-*d*₄ (150MHz)

SY_MQF CD3OD D:\ DATA2015 50



NAME 20150114 sjj-w78
 EXPNO 2
 PROCNO 1
 Date_ 20150118
 Time 12.46
 INSTRUM spect
 PROBHD 5 mm CPDCH 13C
 PULPROG cosygpmfzf
 TD 2048
 SOLVENT CD3OD
 NS 4
 DS 16
 SWH 8417.509 Hz
 FIDRES 4.110112 Hz
 AQ 0.1217012 sec
 RG 2050
 DW 59.400 usec
 DE 20.00 usec
 TE 298.2 K
 D0 0.00000300 sec
 D1 2.00000000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00011900 sec

===== CHANNEL f1 =====
 SFO1 600.2540217 MHz
 NUC1 1H
 P1 11.50 usec
 ND0 1
 TD 128
 SFO1 600.254 MHz
 FIDRES 65.651260 Hz
 SW 14.000 ppm
 FnMODE QF
 SI 1024
 SF 600.2500000 MHz

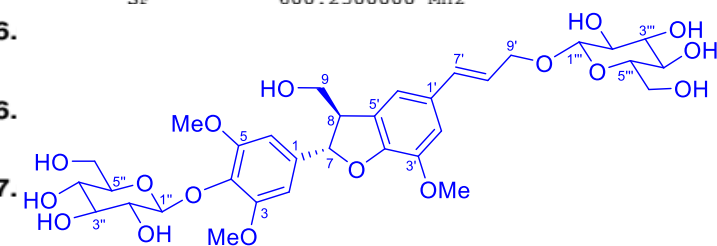


Figure S68. The ^1H - ^1H COSY Spectrum of Compound 6 in $\text{MeOH-}d_4$ (600MHz)

Bruker AVIIIHD 600 20150114
HSQC CD3OD sjj-w78

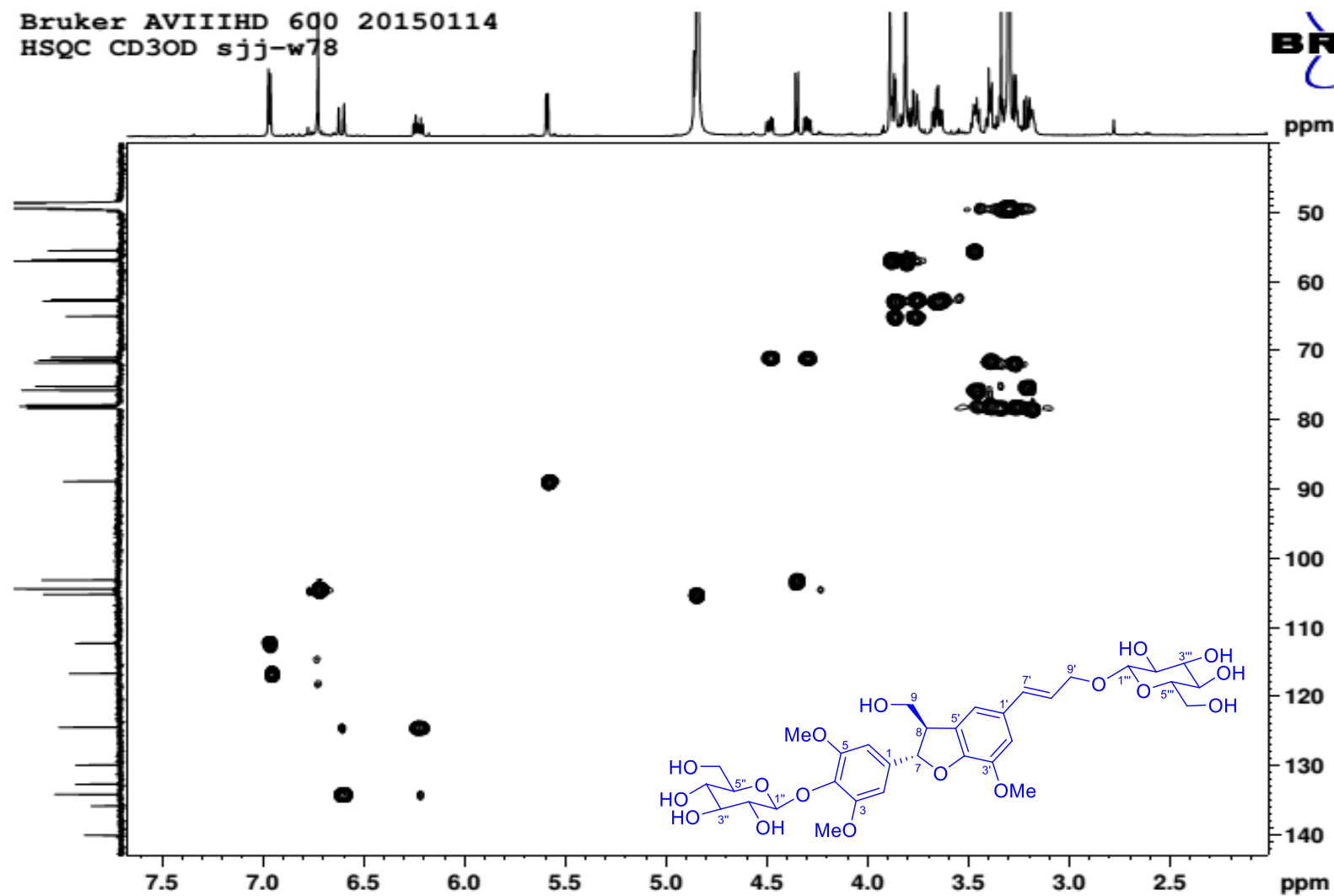


Figure S69. The HSQC Spectrum of Compound 6 in MeOH-*d*₄ (600MHz)

Bruker AVIIIHD 600 20150114
{HMBC etgpl3nd} CD3OD

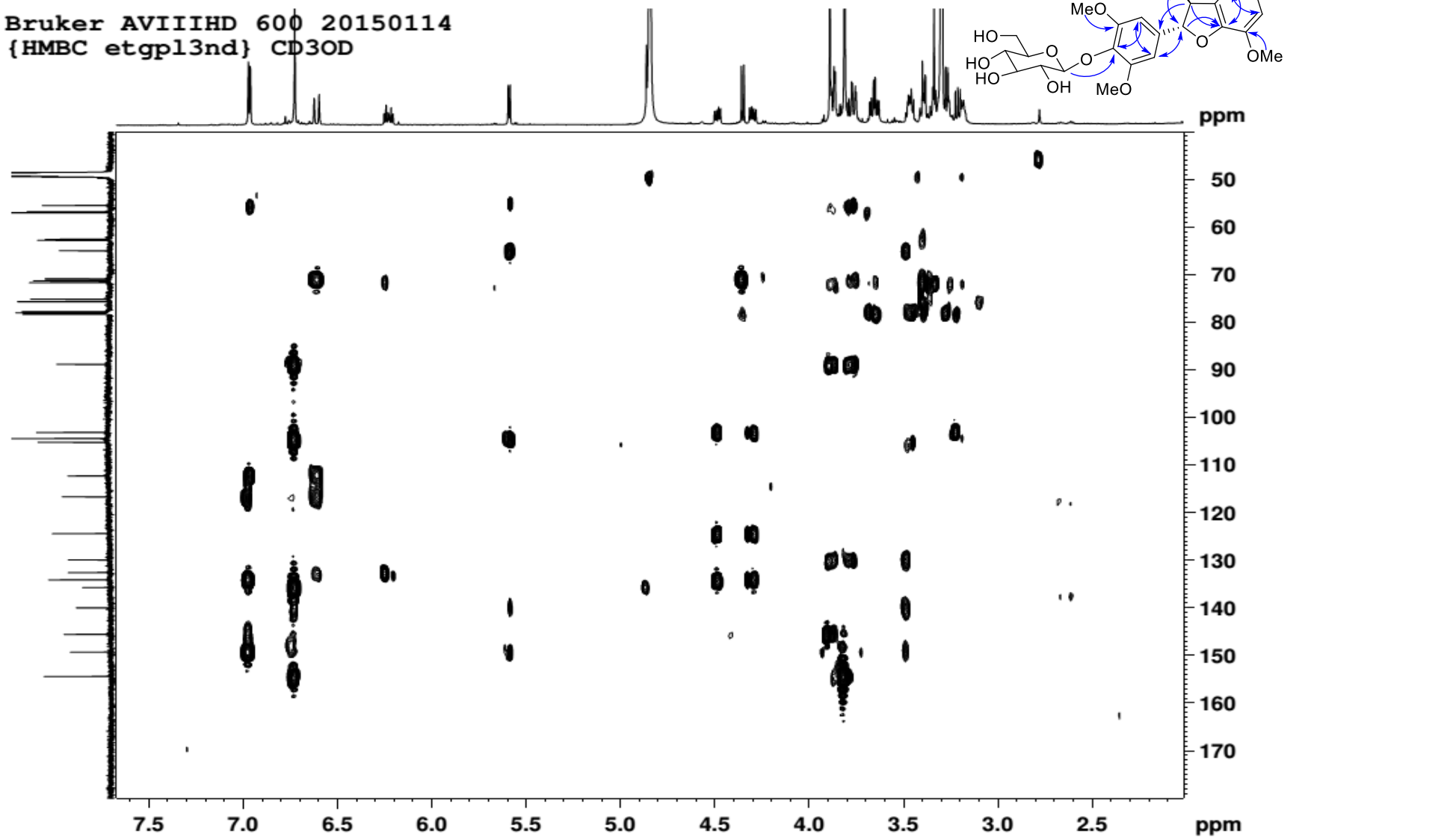
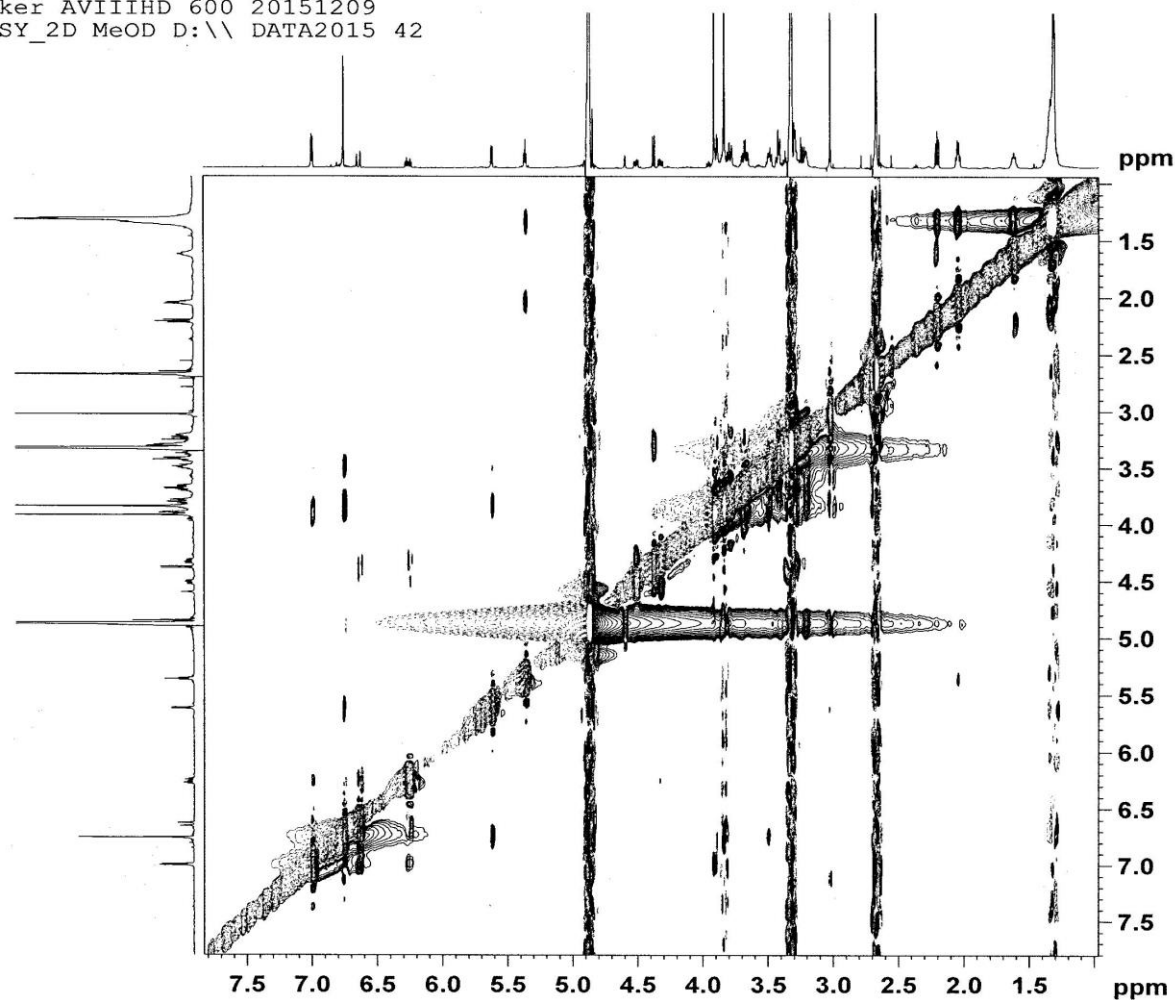


Figure S70. The HMBC Spectrum of Compound 6 in MeOH- d_4 (600MHz)

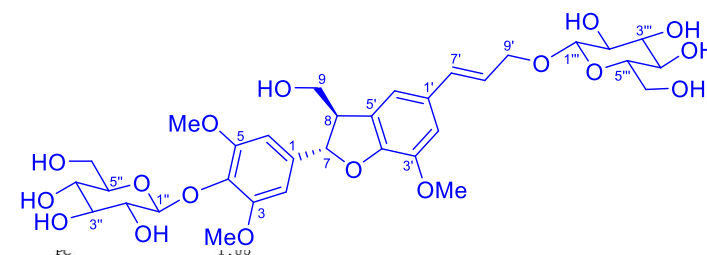
Bruker AVIIIHD 600 20151209
NOESY_2D MeOD D:\ DATA2015 42



Current Data Parameters
NAME 20151210 sjj-w78
EXPNO 2
PROCNO 1

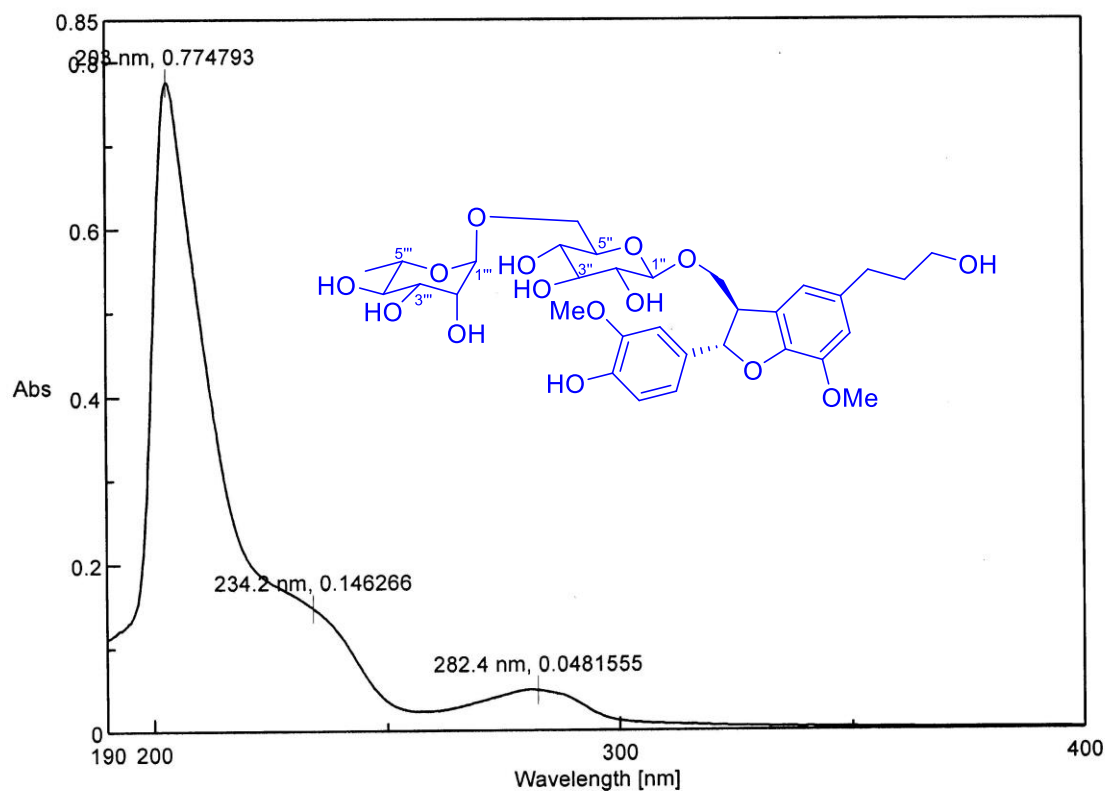
F2 - Acquisition Parameters
Date_ 20151211
Time_ 18.26
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG noesygpphpg
TD 2048
SOLVENT MeOD
NS 32
DS 16
SWH 8417.509 Hz
FIDRES 4.110112 Hz
AQ 0.1216512 sec
RG 144
DW 59.400 usec
DE 20.00 usec
TE 298.0 K
D0 0.00004476 sec
D1 1.20000005 sec
D8 0.60000002 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
INO 0.00011880 sec

----- CHANNEL f1 -----
SFO1 600.2539016 MHz
NUC1 1H
P1 11.50 usec
P2 23.00 usec
P17 2500.00 usec
PLW1 12.93200016 W
PLW10 2.52999997 W



F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 600.2500000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

Figure S71. The NOESY Spectrum of Compound 6 in MeOH- d_4 (600MHz)



[Comment]
 Sample Name sij-w111
 Comment 0.02
 User
 Division UV
 Company 324
 [Measurement Information]
 Instrument Name V-650
 Model Name V-650
 Serial No. A034461150

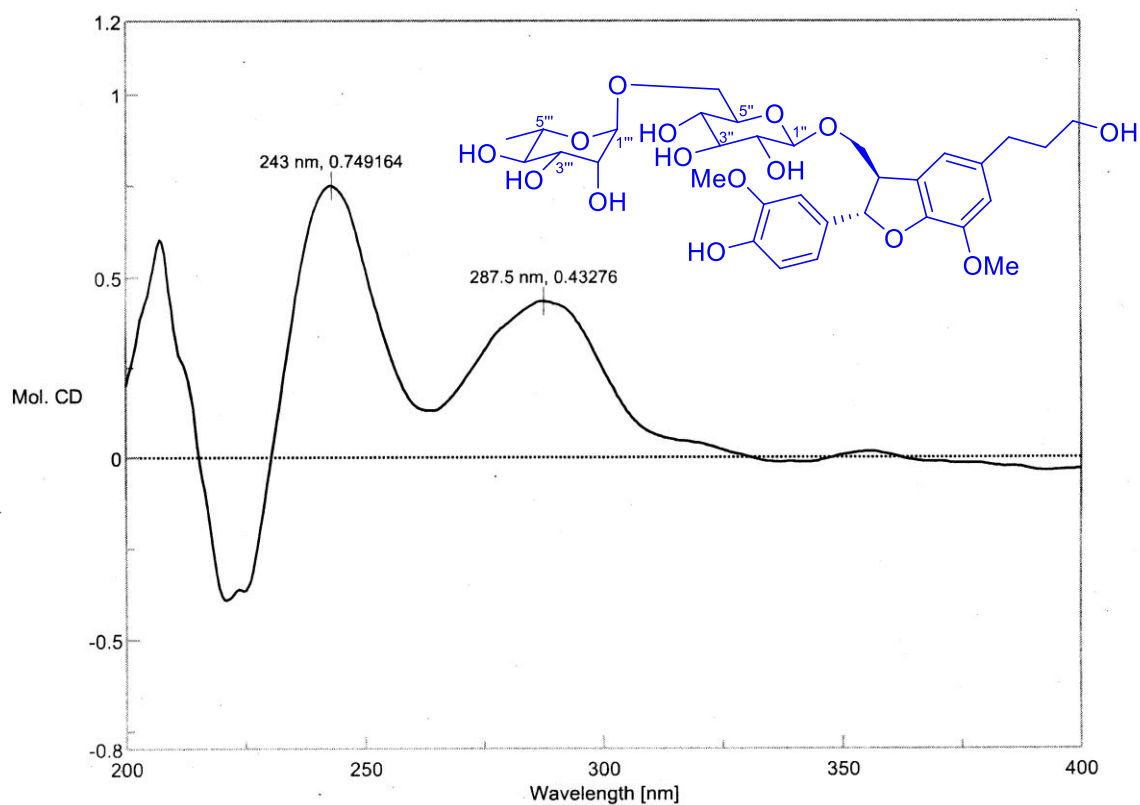
Accessory PSC-718
 Accessory S/N A001761114
 Position 1
 Cell Length 10 mm
 Temperature 19.45 C
 Control Sensor Holder
 Monitor Sensor Holder
 Start Mode Start immediately

Photometric Mode Abs
 Measurement range 400 - 190 nm
 Data pitch 0.2 nm
 Band width(UV/Vis) 2.0 nm
 Response Medium
 Scanning speed 200 nm/min
 Source Change 340 nm
 Light Source D2/M1
 Filter Exchange Step
 Correction Baseline

[Data Information]
 Creation Date 2016-1-19 19:20
 Data array type Linear data array
 Horizontal Wavelength [nm]
 Vertical Abs
 Start 400 nm
 End 190 nm
 Data pitch 0.2 nm
 Data points 1051

DBT-0.02-39

Figure S72. The UV Spectrum of Compound 7 in MeOH



[Comments]
 Sample name sjj-W111
 Comment
 User
 Division
 Company dell

[Measurement Information]
 Instrument Name J-815
 Model Name J-815
 Serial No. A024461168

Accessory Standard
 Accessory S/N A024461168
 Cell Length 1 mm

Photometric Mode CD, HT, Abs
 Measure Range 400 - 200 nm
 Data pitch 0.5 nm
 Sensitivity Standard
 D.I.T. 1 sec
 Band width 2.00 nm
 Start Mode Immediately
 Scanning Speed 100 nm/min
 Baseline Correction Baseline
 Shutter Control Auto
 PMT Voltage Auto
 Accumulations 2
 Solvent MEOH
 Concentration 0.5 (w/v)%

[Detailed Information]
 Creation date 2015-10-19 16:18

Data array type Linear data array * 3
 Horizontal axis Wavelength [nm]
 Vertical axis(1) Mol. CD
 Vertical axis(2) HT [V]
 Vertical axis(3) Abs
 Start 400 nm
 End 200 nm
 Data interval 0.5 nm
 Data points 401

Figure S73. The CD Spectrum of Compound 7 in MeOH

Compound Mass Spectrum Report - MS

Analysis Name: 15092919.d

Instrument: LC-MSD-Trap-SL

Print Date: 2015-10-8 10:23:06

Method: WANGSJ-MS.m

Operator: Operator

Acq. Date: 2015-9-29 11:04:13

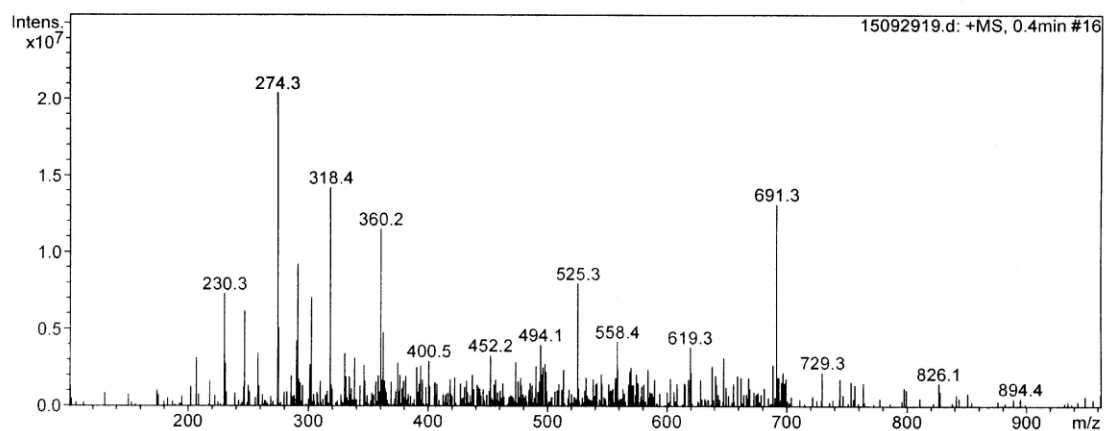
Sample Name: SJJ-w111

Analysis Info:

Acquisition Parameter:

| | | | | | |
|-----------------|------------|-----------------------|-------------|----------------|-----------|
| Mass Range Mode | Std/Normal | Trap Drive | 53.0 | Scan Begin | 100 m/z |
| Ion Polarity | Positive | Skim 1 | -40.0 Volt | Scan End | 1000 m/z |
| Ion Source Type | ESI | Skim 2 | 5.0 Volt | Averages | 5 Spectra |
| Dry Temp (Set) | 300 度 | Octopole RF Amplitude | 171.0 Vpp | Max. Accu Time | 300000 釐 |
| Nebulizer (Set) | 15.00 psi | Capillary Exit | -121.0 Volt | ICC Target | 30000 |
| Dry Gas (Set) | 6.00 l/min | | | Charge Control | on |

+MS, 0.4min #16



-MS, 0.3min #13

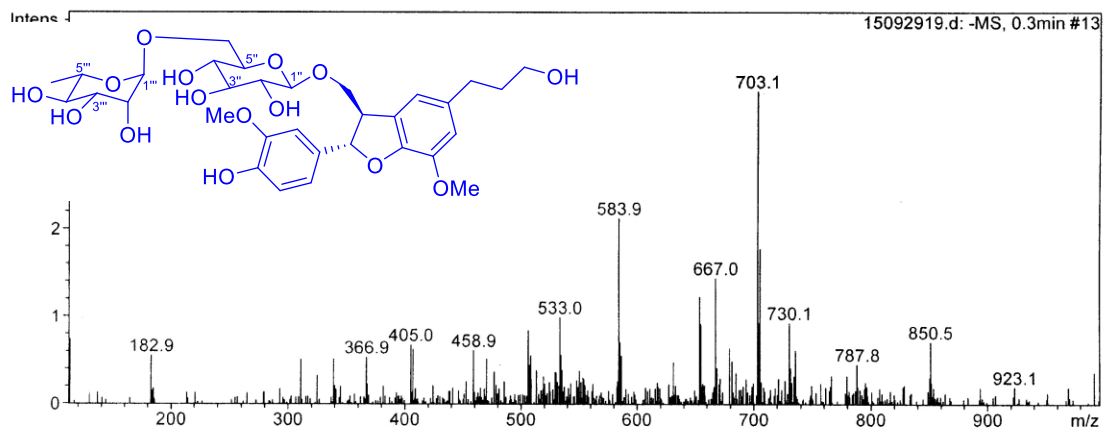


Figure S74. The ESIMS Spectrum of Compound 7

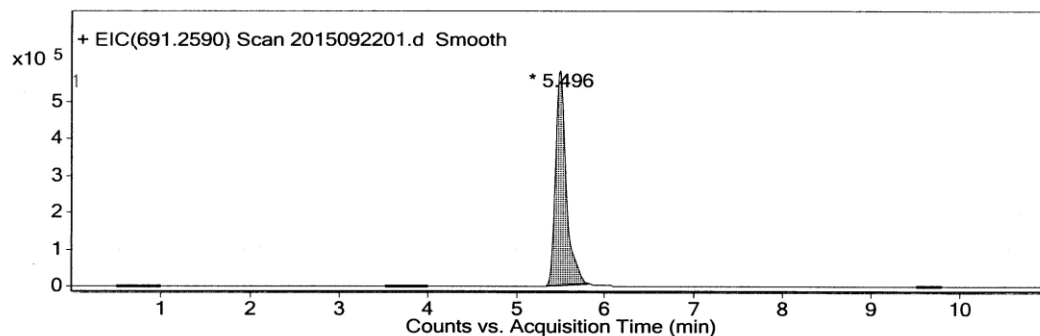
Qualitative Analysis Report

Data Filename 2015092201.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method
DA Method TEST LCMS.m

Sample Name Sjj-WIII
Position P1-C6
User Name
IRM Calibration Status Success
Comment

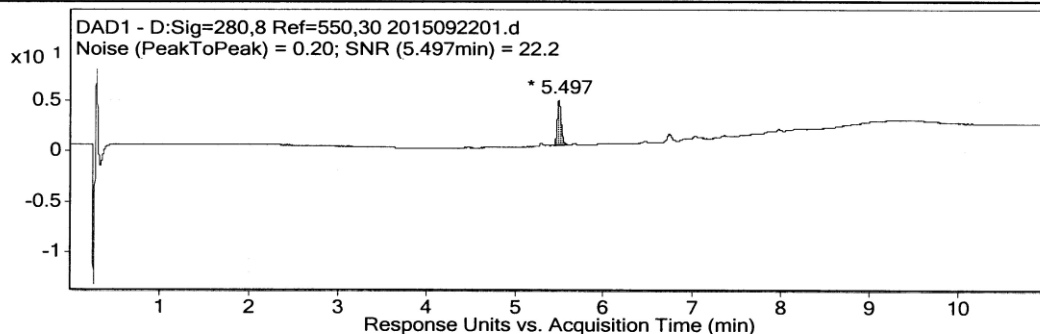
User Chromatograms

Fragmentor Voltage 120 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

| Peak | Start | RT | End | Height | Area | Area % | Signal To Noise |
|------|-------|-------|-------|--------|---------|--------|-----------------|
| 1 | 5.335 | 5.496 | 5.834 | 582268 | 5022024 | 100 | Infinity |



Integration Peak List

| Peak | Start | RT | End | Height | Area | Area % | Signal To Noise |
|------|-------|-------|-------|--------|-------|--------|-----------------|
| 1 | 5.428 | 5.497 | 5.593 | 4.45 | 13.91 | 100 | 22.2 |

User Spectra

Fragmentor Voltage 120 **Collision Energy** 0 **Ionization Mode** ESI

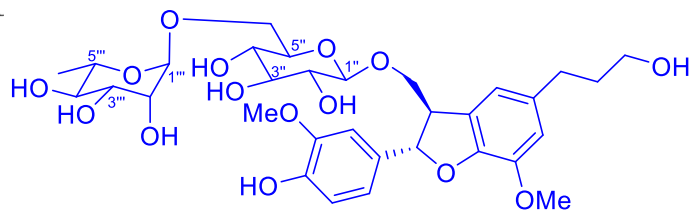
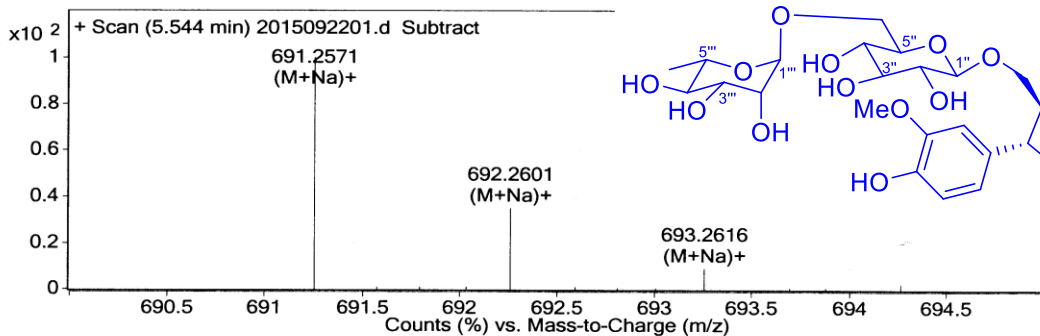


Figure S75. The HRESIMS Spectrum of Compound 7

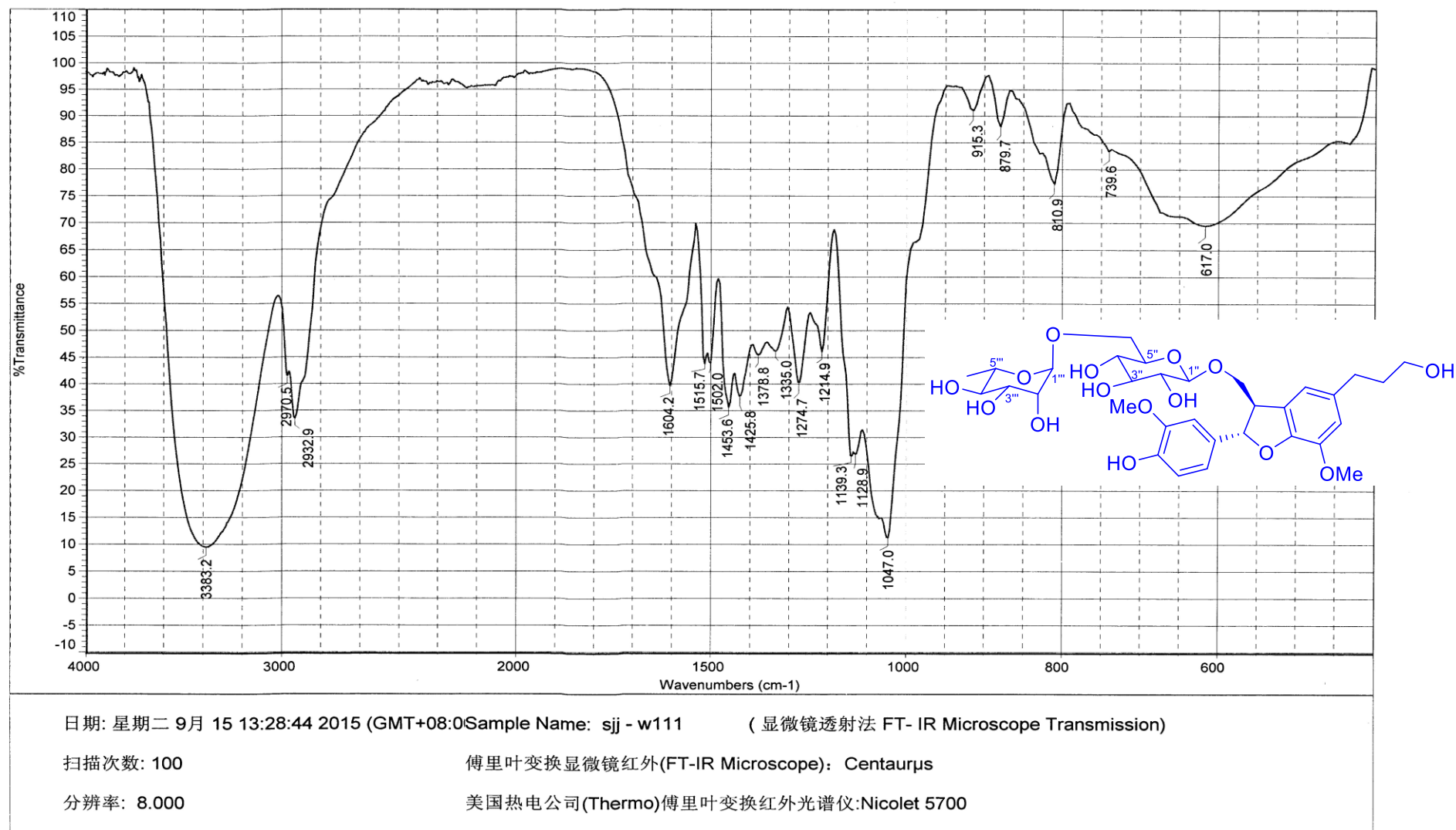


Figure S76. The IR Spectrum of Compound 7

Bruker AVIIIHD 600 20150609
PROTON CD3OD sjj-w111

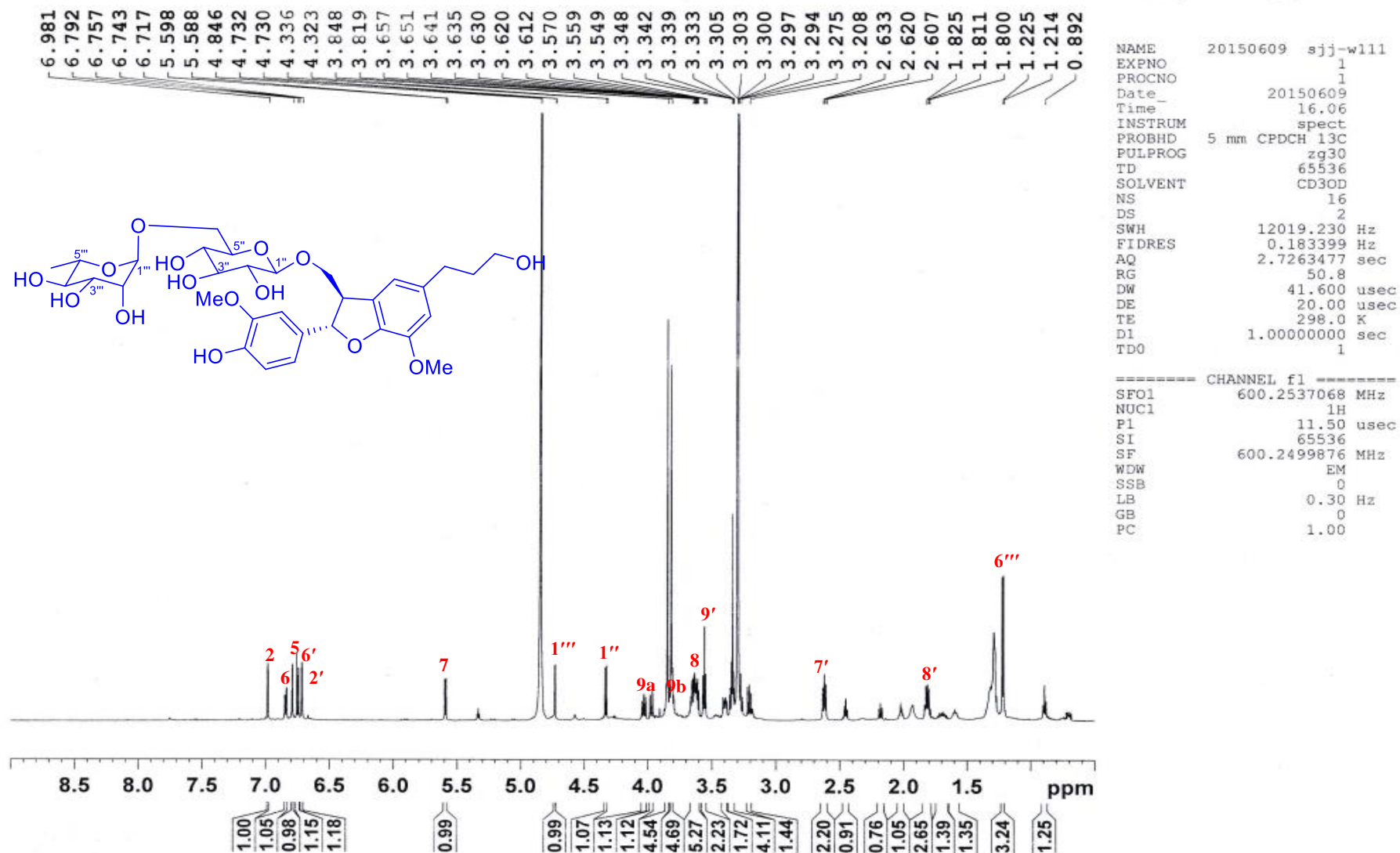


Figure S77. The ¹H NMR Spectrum of Compound 7 in MeOH-d₄ (600MHz)

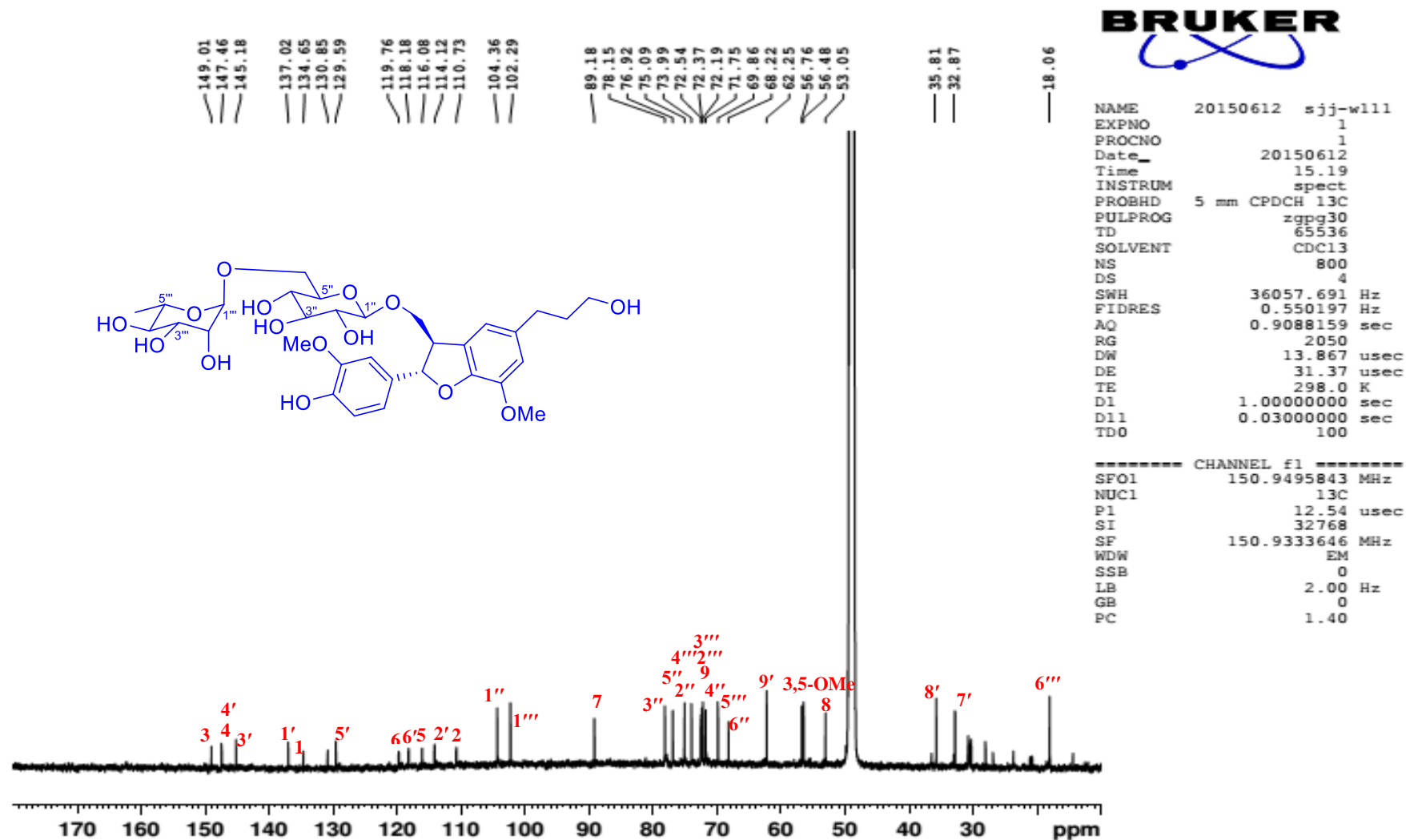


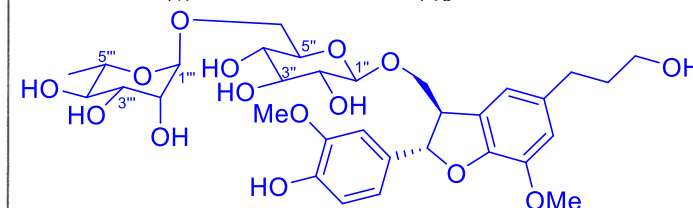
Figure S78. The ^{13}C NMR Spectrum of Compound 7 in $\text{MeOH-}d_4$ (150MHz)

Bruker AVIIIHD 600 20150902
 COSY_MQF CD3OD D:\\ DATA2015 33



NAME 20150902 sjj-w111
 EXPNO 2
 PROCNO 1
 Date_ 20150904
 Time_ 2.27
 INSTRUM spect
 PROBHD 5 mm CPDCH 13C
 PULPROG cosygpmfqf
 TD 2048
 SOLVENT CD3OD
 NS 4
 DS 16
 SWH 8417.509 Hz
 FIDRES 4.110112 Hz
 AQ 0.1217012 sec
 RG 2050
 DW 59.400 usec
 DE 20.00 usec
 TE 298.0 K
 D0 0.00000300 sec
 D1 2.00000000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 IN0 0.00011900 sec

===== CHANNEL f1 =====
 SFO1 600.2540217 MHz
 NUC1 1H
 P1 11.50 usec
 ND0 1
 TD 128



MC2 QF
 SF 600.2500000 MHz
 WDW SINE
 SSB 0
 LB 0.00 Hz
 GB 0

8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm

Figure S79. The ^1H - ^1H COSY Spectrum of Compound 7 in MeOH- d_4 (600MHz)

Bruker AVIIIHD 600 20150902
HSQC CD3OD D:\\ DATA2015 33

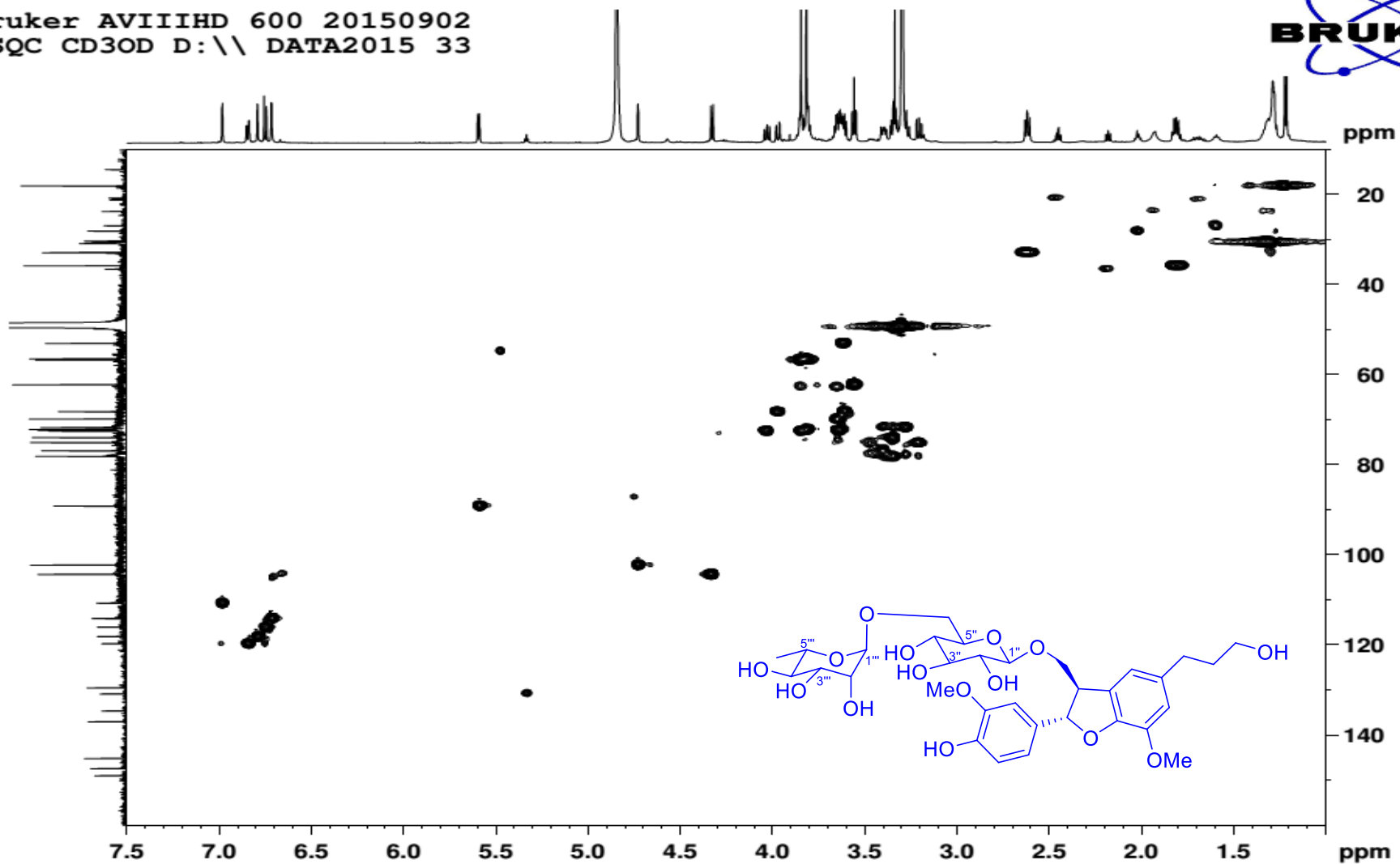


Figure S80. The HSQC Spectrum of Compound 7 in MeOH- d_4 (600MHz)

Bruker AVIIIHD 600 20150713
HMBC CD3OD D:\\ DATA2015 51

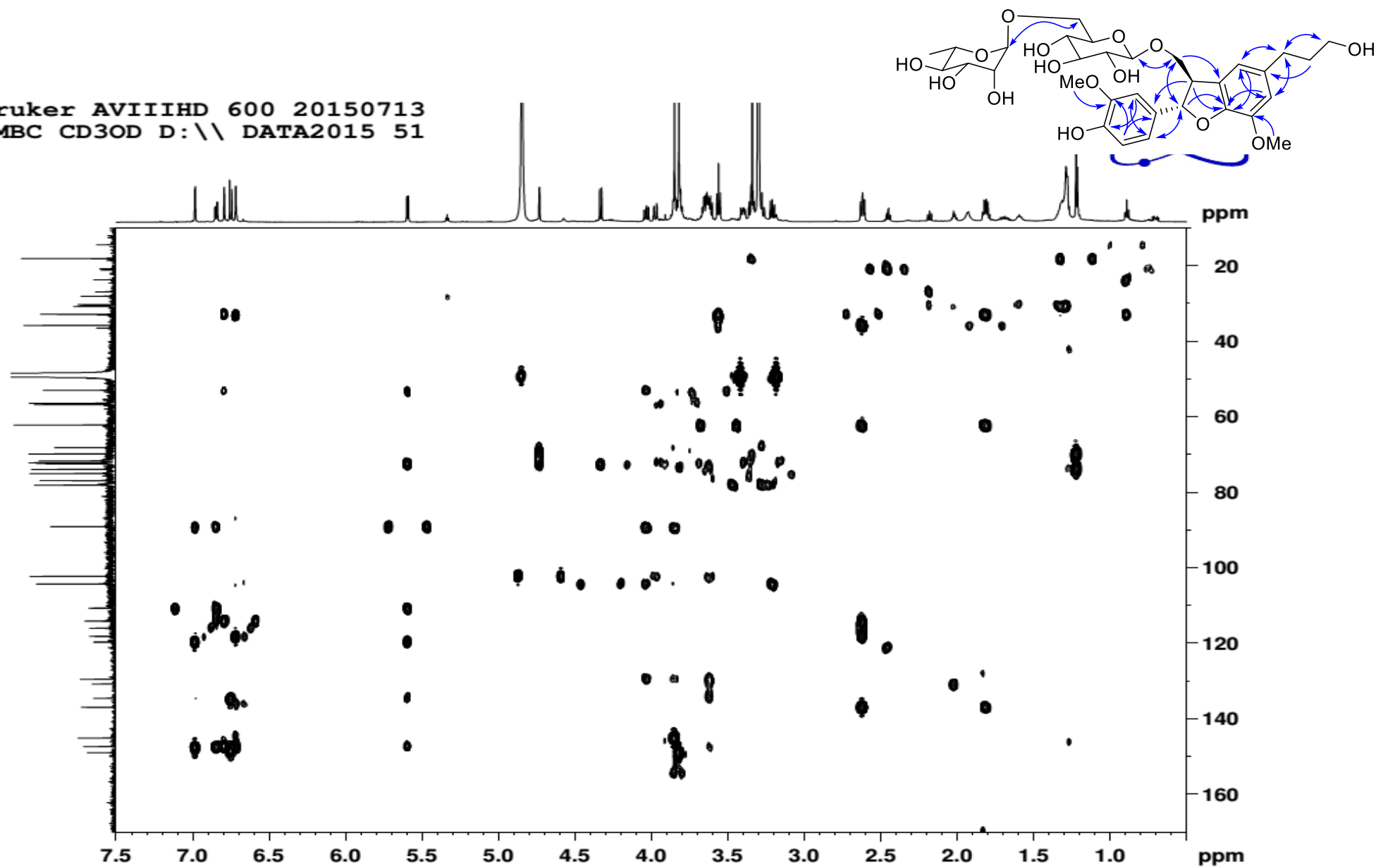


Figure S81. The HMBC Spectrum of Compound 7 in MeOH-*d*₄ (600MHz)

Bruker AVIIIHD 600 20151019
NOESY_2D CD3OD D:\ DATA2015 56



Current Data Parameters
NAME 20151019 sjj-wlll
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151025
Time 23.45
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG noesygpph
TD 2048
SOLVENT CD3OD
NS 16
DS 16
SWH 8417.509 Hz
FIDRES 4.110112 Hz
AQ 0.1216512 sec
RG 144
DW 59.400 usec
DE 20.00 usec
TE 298.0 K
D0 0.0000476 sec
D1 1.20000005 sec
D8 0.60000002 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00011880 sec

===== CHANNEL f1 =====
SF01 600.2539016 MHz
NUC1 1H
P1 11.50 usec
P2 23.00 usec
P17 2500.00 usec
PLW1 12.93200016 W
PLW10 2.52999997 W

===== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPZ1 40.00 %
F16 1000.00 usec

F1 - Acquisition parameters
TD 160
SFO 600.2539016 MHz

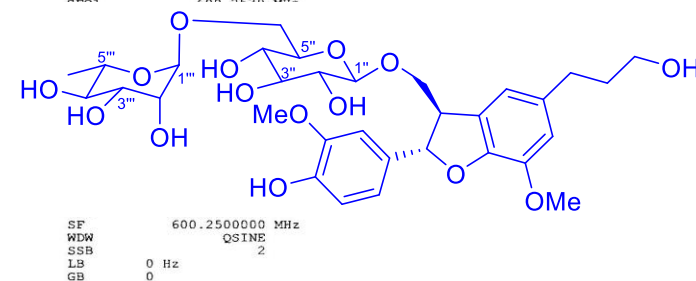
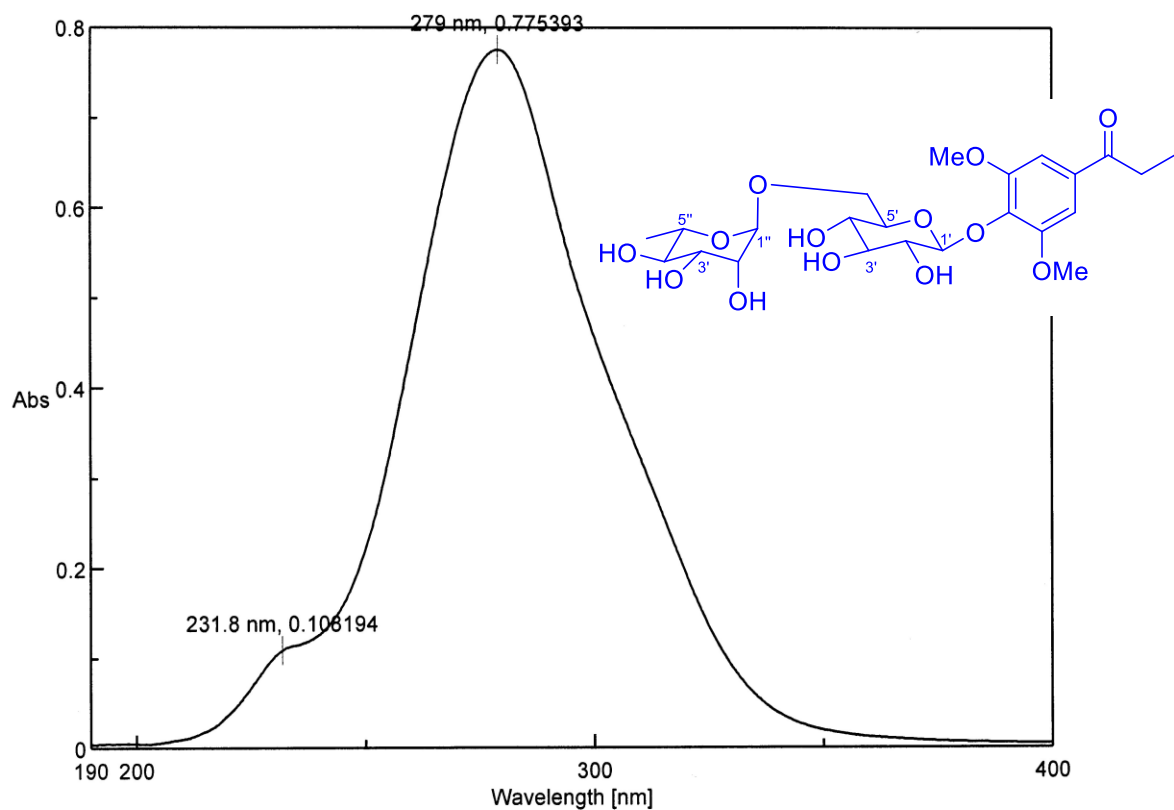


Figure S82. The NOESY Spectrum of Compound 7 in MeOH- d_4 (600MHz)



[Comment]
 Sample Name DBT
 Comment 0.02
 User
 Division UV
 Company 324
 [Measurement Information]
 Instrument Name V-650
 Model Name V-650
 Serial No. A034461150

Accessory PSC-718
 Accessory S/N A001761114
 Position 1
 Cell Length 10 mm
 Temperature 19.96 C
 Control Sensor Holder
 Monitor Sensor Holder
 Start Mode Start immediately

Photometric Mode Abs
 Measurement range 400 - 190 nm
 Data pitch 0.2 nm
 Band width(UV/Vis) 2.0 nm
 Response Medium
 Scanning speed 200 nm/min
 Source Change 340 nm
 Light Source D2/M
 Filter Exchange Step
 Correction Baseline

[Data Information]
 Creation Date 2015-6-30 19:15
 Data array type Linear data array
 Horizontal Wavelength [nm]
 Vertical Abs
 Start 400 nm
 End 190 nm
 Data pitch 0.2 nm
 Data points 1051

Figure S83. The UV Spectrum of Compound 8 in MeOH

Compound Mass Spectrum Report - MS

Analysis Name: 20150416-13.d
Method: linsheng.m
Sample Name: SJJ-w83
Analysis Info:

Instrument: LC-MSD-Trap-SL
Operator: Operator

Print Date: 2015-6-8 12:16:50
Acq. Date: 2015-4-16 22:48:10

Acquisition Parameter:

Mass Range Mode Std/Normal
Ion Polarity Positive
Ion Source Type ESI
Dry Temp (Set) 330 度
Nebulizer (Set) 15.00 psi
Dry Gas (Set) 6.00 l/min

Trap Drive 60.4
Skim 1 -40.0 Volt
Skim 2 5.0 Volt
Octopole RF Amplitude 187.1 Vpp
Capillary Exit -128.5 Volt

Scan Begin 100 m/z
Scan End 1000 m/z
Averages 7 Spectra
Max. Accu Time 200000 秒
ICC Target 30000
Charge Control on

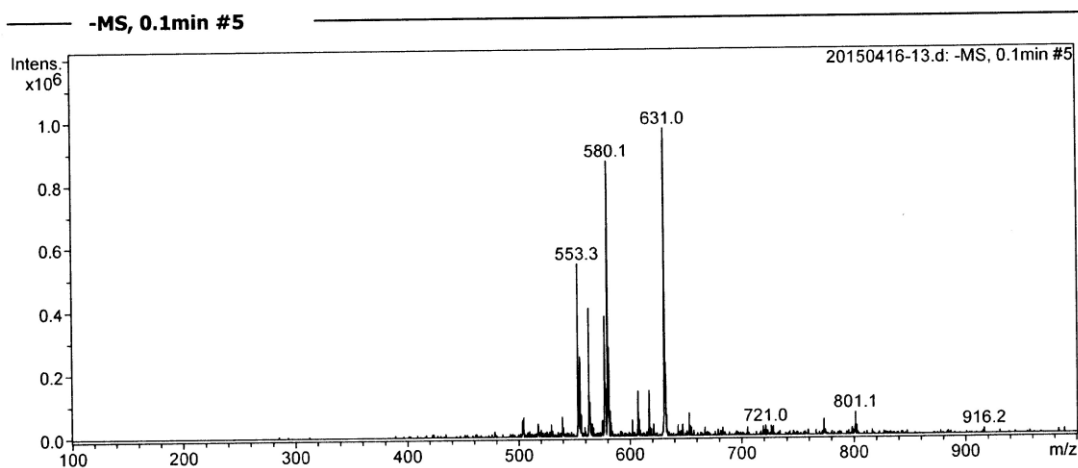
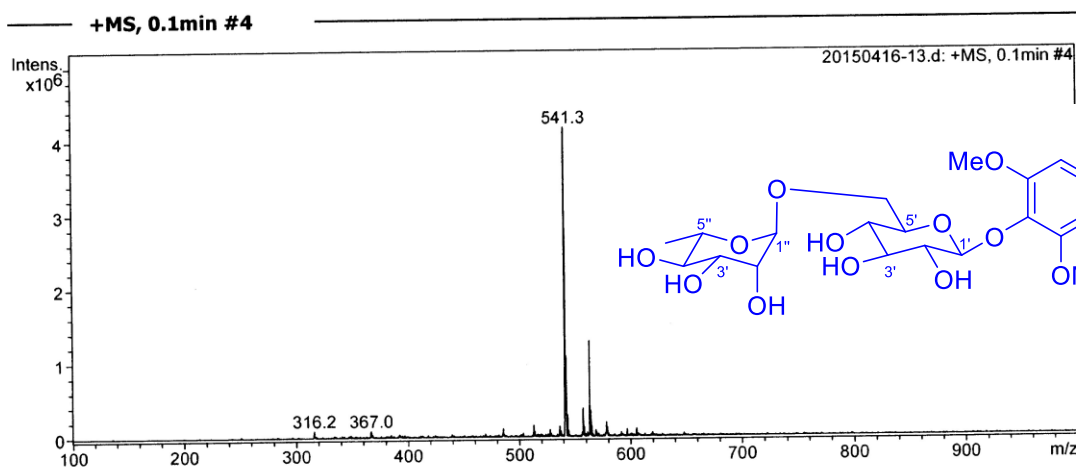


Figure S84. The ESIMS Spectrum of Compound 8

日期:2015-06-17
仪器:API-TOFMS 10000 广州禾信分析仪器有限公司
时间:从0.2900到0.5060

用户单位名称:中国医学科学院药物研究所
样品名称:sjj-w83
离子模式:Positive

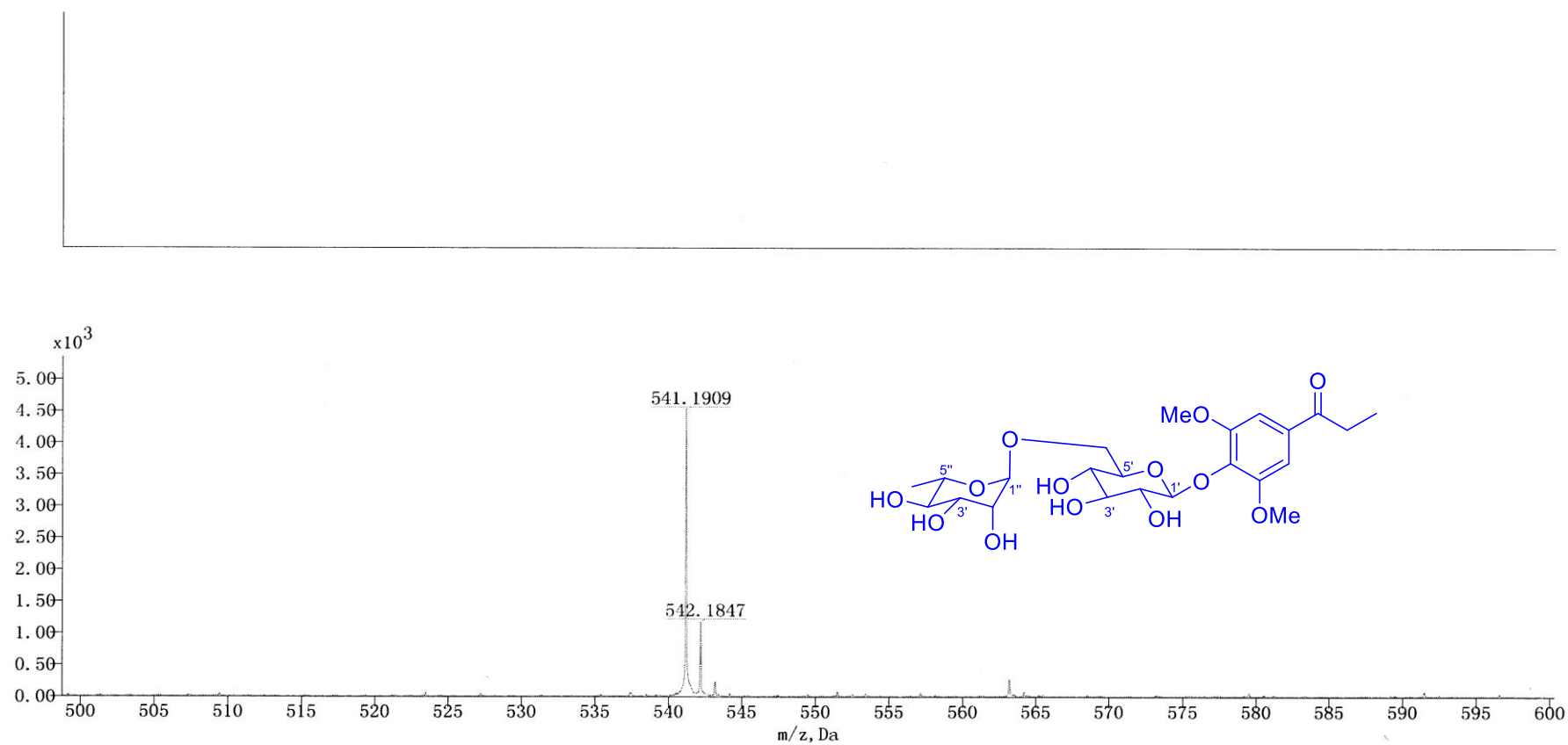


Figure S85. The HRESIMS Spectrum of Compound 8

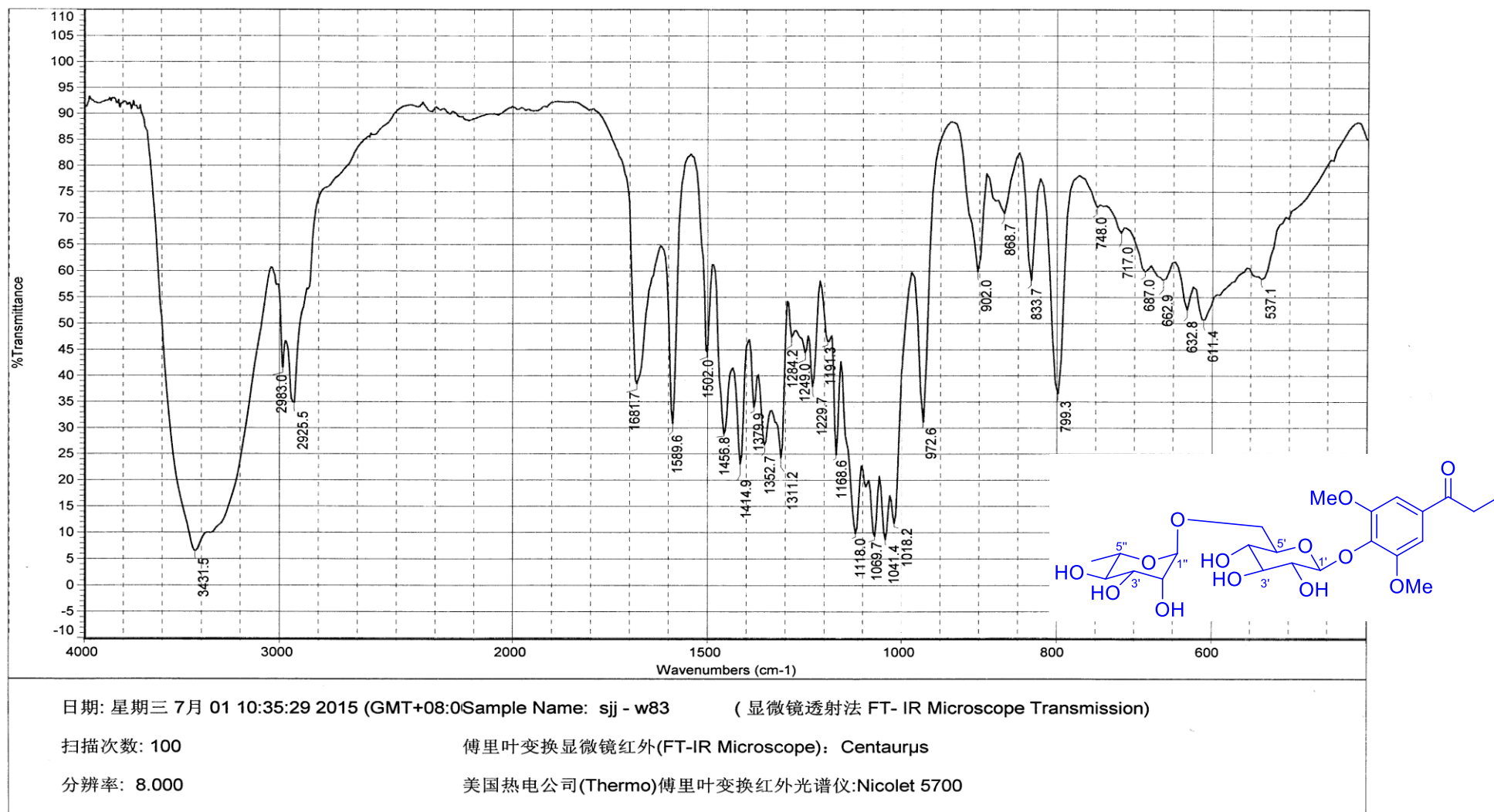


Figure S86. The IR Spectrum of Compound 8

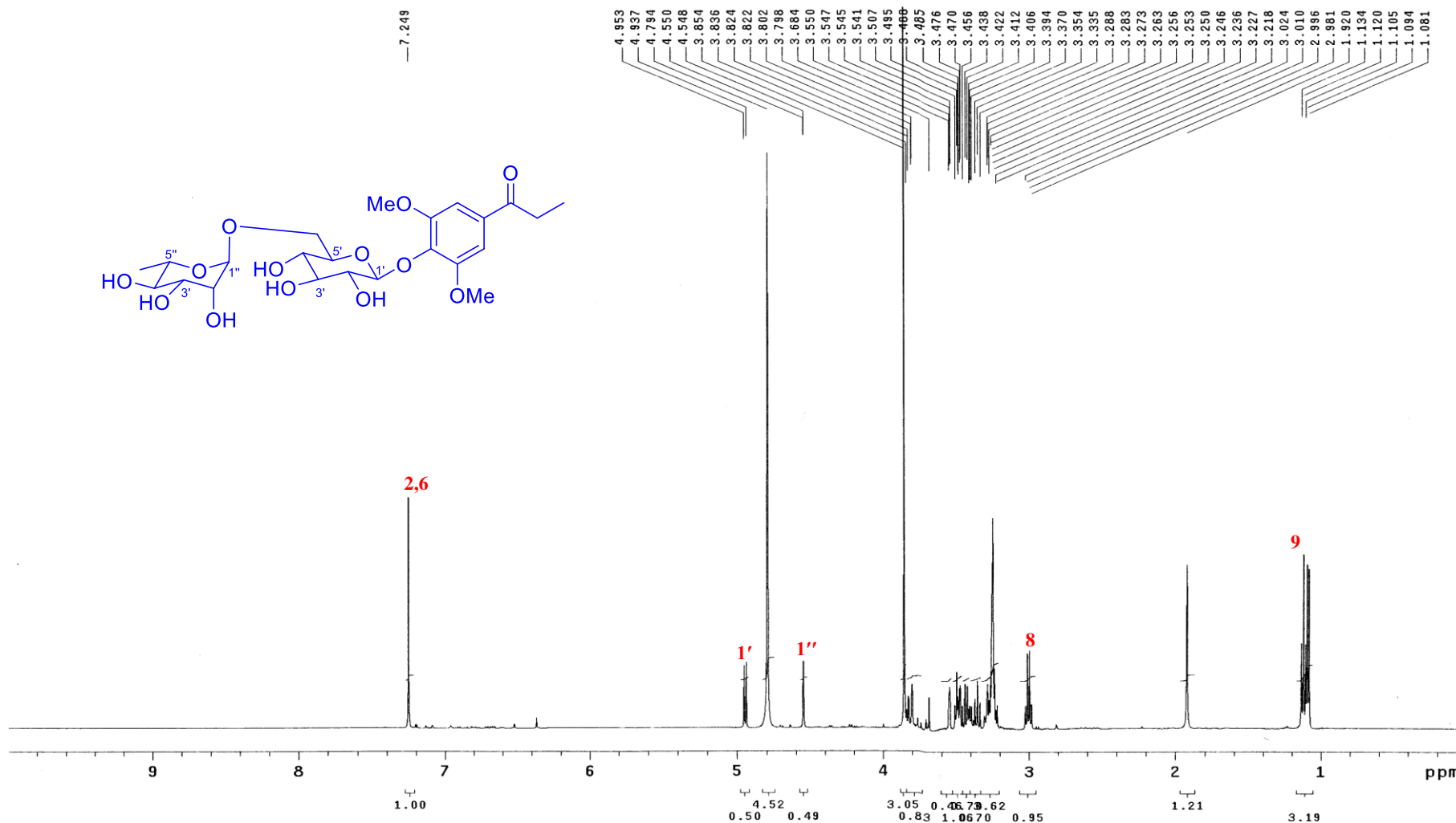


Figure S87. The ^1H NMR Spectrum of Compound 8 in $\text{MeOH-}d_4$ (500MHz)

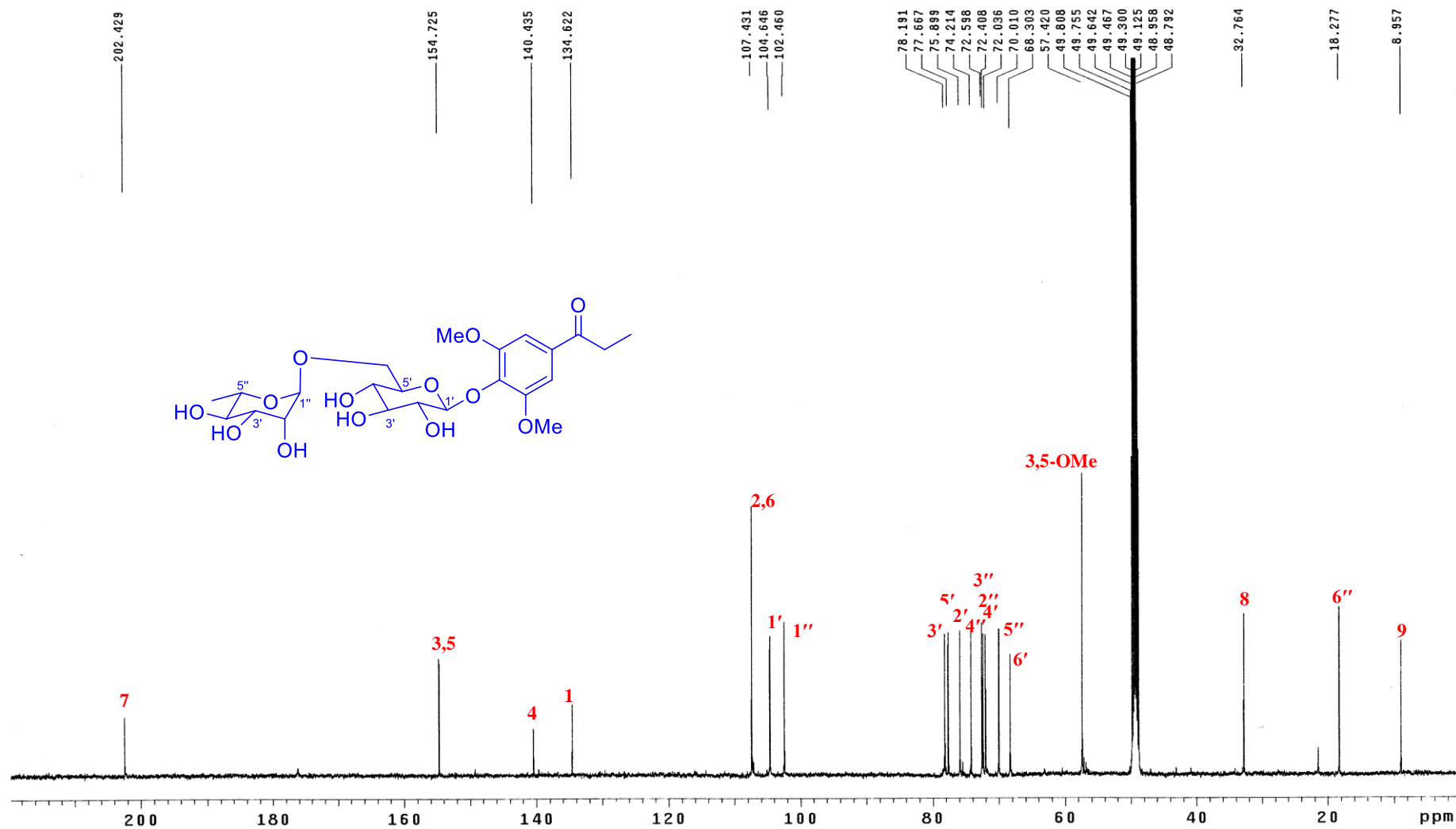


Figure S88. The ^{13}C NMR Spectrum of Compound 8 in $\text{MeOH-}d_4$ (125MHz)

DD2-500 gCOSY sjj-w83 IN cd3od Jan 23 2015 coldprobe

Temp. 25.0 C / 298.1 K
 Sample #8, Operator: vnmr1
 Relax. delay 1.000 sec
 Acq. time 0.150 sec
 Width 4664.2 Hz
 2D Width 4664.2 Hz
 4 repetitions
 128 increments
 OBSERVE H1, 499.7694110 MHz
 DATA PROCESSING
 Sq. sine bell 0.075 sec
 F1 DATA PROCESSING
 Sq. sine bell 0.027 sec
 FT size 2048 x 2048
 Total time 10 min

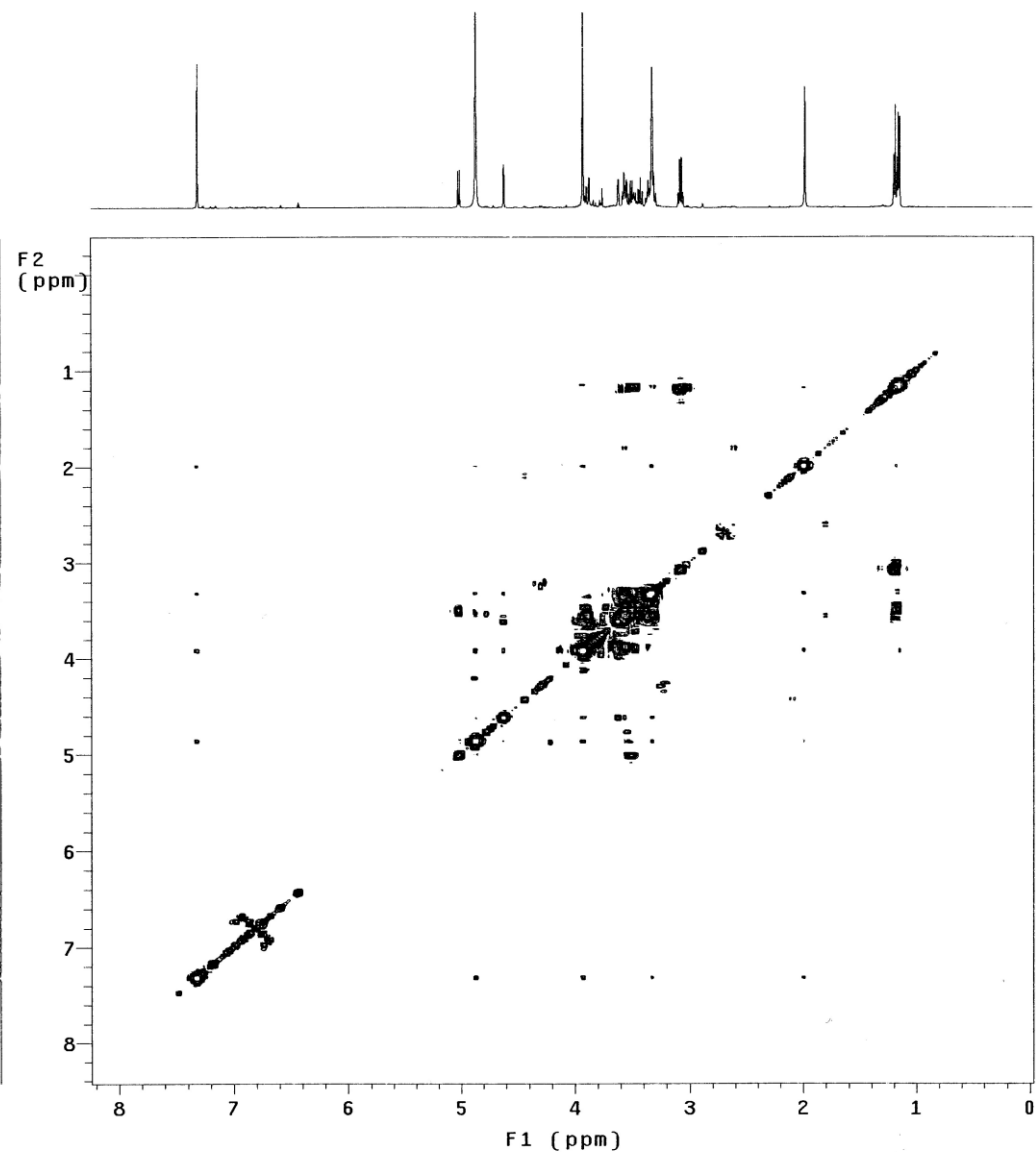
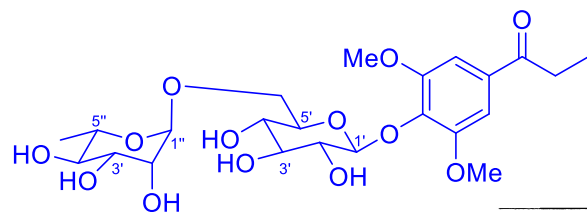


Figure S89. The ^1H - ^1H COSY Spectrum of Compound 8 in $\text{MeOH-}d_4$ (500MHz)

DD2-500 gHSQCAD sjj-w83 IN cd3od Jan 23 2015 coldprobe

Temp. 25.0 C / 298.1 K
Sample #8, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.258 sec
Width 4664.2 Hz
2D Width 25133.5 Hz
8 repetitions
2 x 128 increments
OBSERVE H1, 499.7694110 MHz
DECOUPLE C13, 125.6784284 MHz
Power 38 dB
on during acquisition
off during delay
W40_coldprobe modulated
DATA PROCESSING
Gauss apodization 0.069 sec
F1 DATA PROCESSING
Gauss apodization 0.005 sec
FT size 4096 x 2048
Total time 41 min

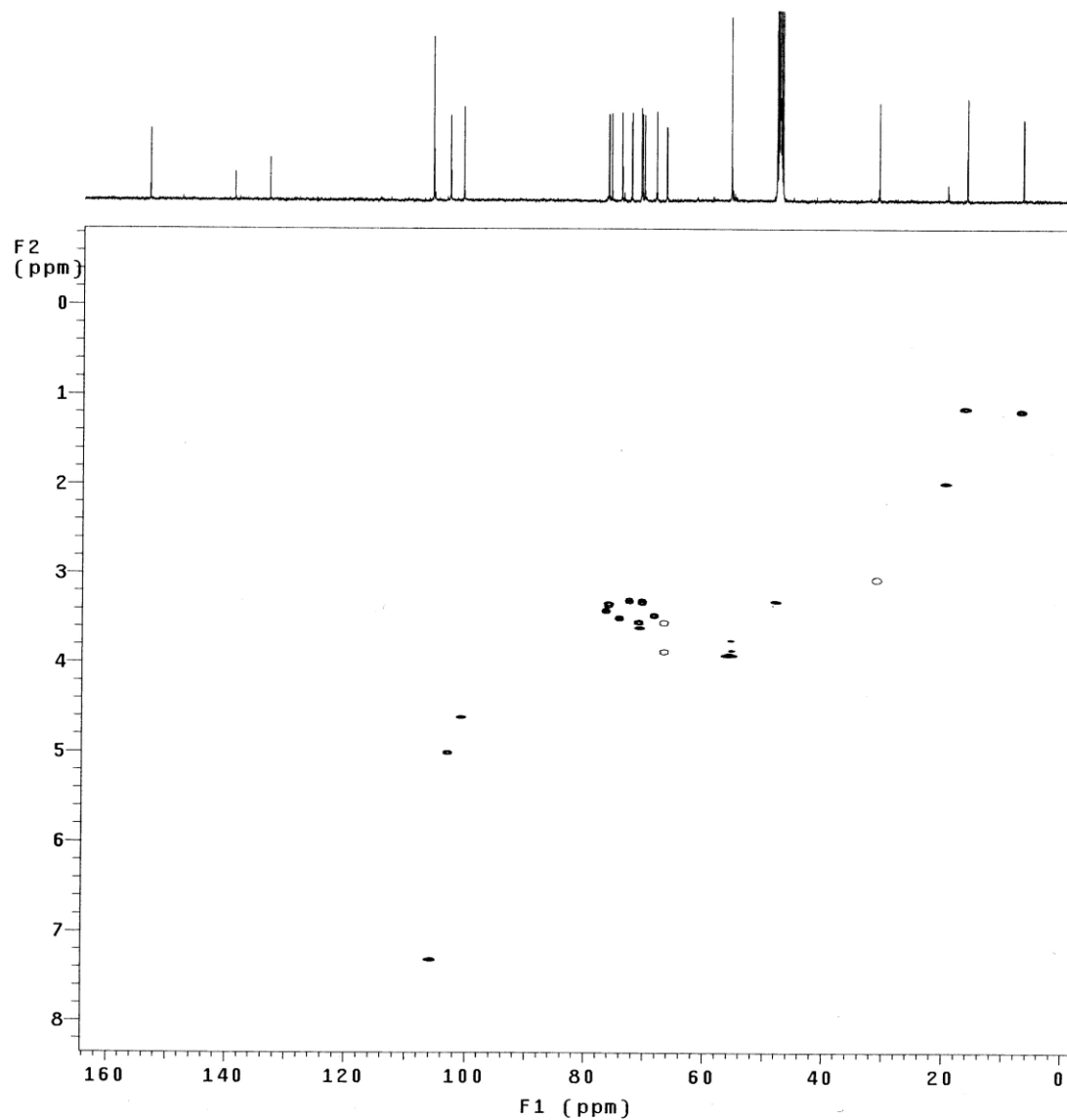
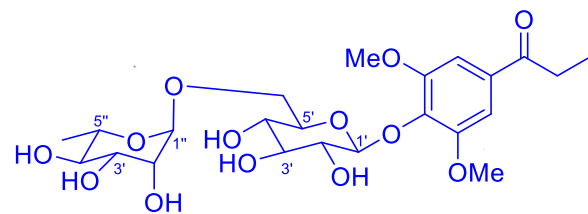


Figure S90. The HSQC Spectrum of Compound 8 in MeOH-*d*₄ (500MHz)

DD2-500 gHMBCAD sjj-w83 IN cd3od Jan 23 2015 coldprobe

Temp. 25.0 C / 298.1 K
Sample #8, Operator: vnmr1
Relax. delay 1.000 sec
Acq. time 0.258 sec
Width 4664.2 Hz
2D Width 30154.5 Hz
16 repetitions
2 x 128 increments
OBSERVE H1, 499.7694110 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Gauss apodization 0.004 sec
FT size 4096 x 2048
Total time 1 hr, 24 min

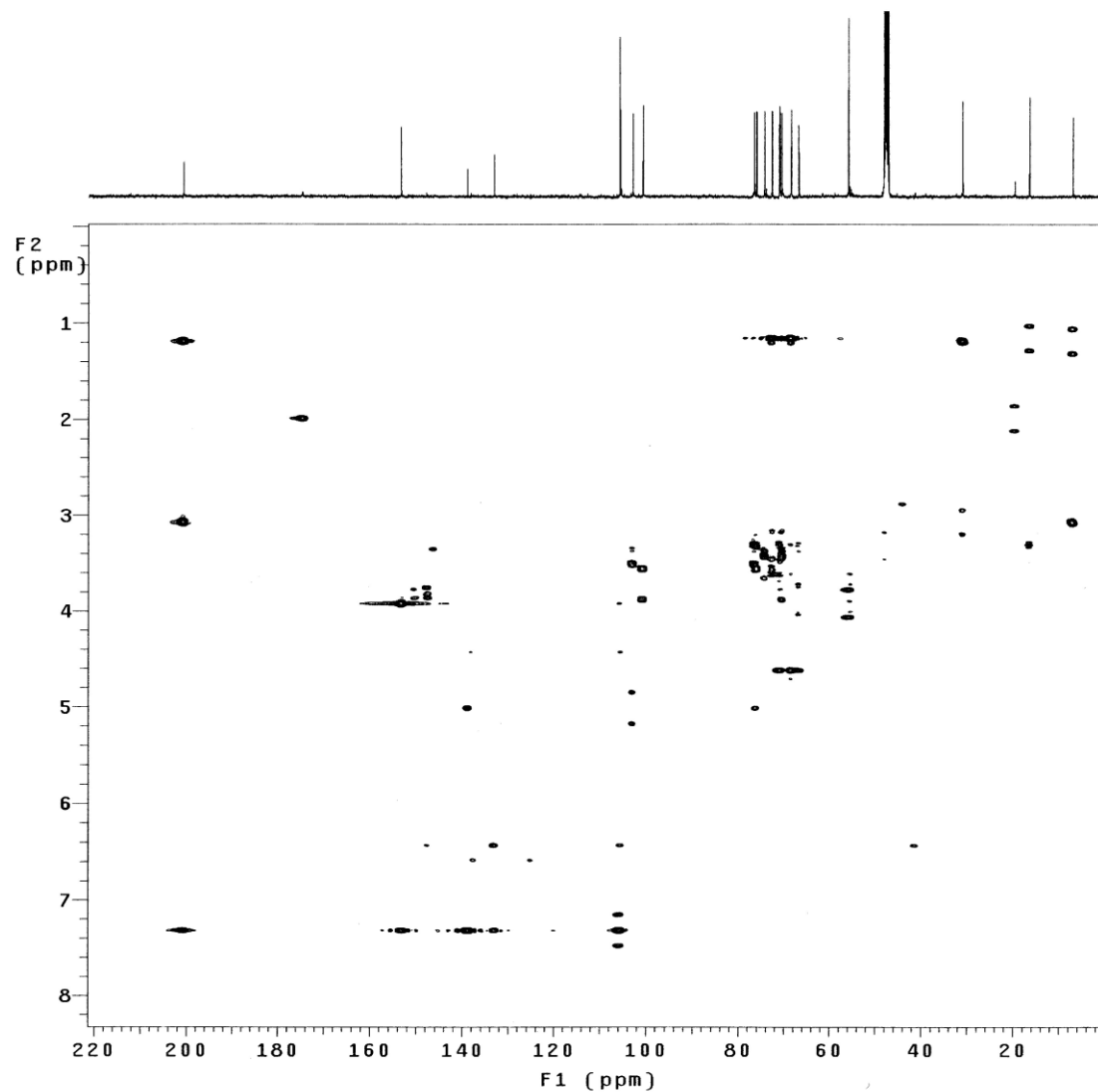
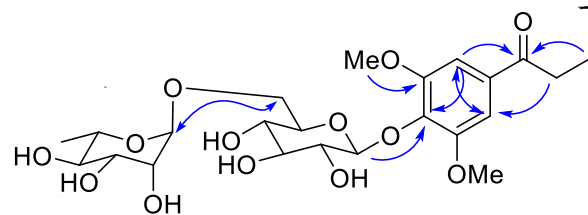
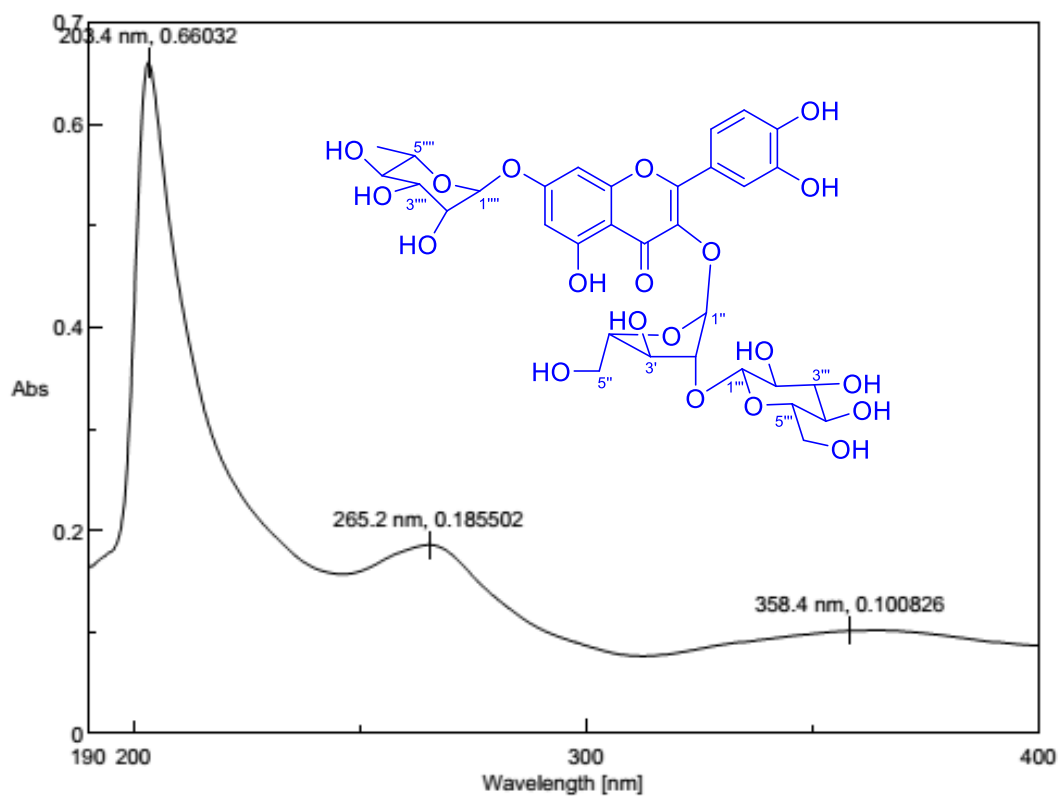


Figure S91. The HMBC Spectrum of Compound 8 in MeOH- d_4 (500MHz)



[Comment]
 Sample Name DBT
 Comment 0.02
 User
 Division UV
 Company 324
 [Measurement Information]
 Instrument Name V-650
 Model Name V-650
 Serial No. A034461150

Accessory PSC-718
 Accessory S/N A001761114
 Position 1
 Cell Length 10 mm
 Temperature 19.99 C
 Control Sensor Holder
 Monitor Sensor Holder
 Start Mode Start immediately

Photometric Mode Abs
 Measurement range 400 - 190 nm
 Data pitch 0.2 nm
 Band width(UV/Vis) 2.0 nm
 Response Medium
 Scanning speed 200 nm/min
 Source Change 340 nm
 Light Source D2/WI
 Filter Exchange Step
 Correction Baseline

[Data Information]
 Creation Date 2016-5-10 19:38
 Data array type Linear data array
 Horizontal Wavelength [nm]
 Vertical Abs
 Start 400 nm
 End 190 nm
 Data pitch 0.2 nm
 Data points 1051

—— SJJ-W96

Figure S92. The UV Spectrum of Compound 9 in MeOH

Compound Mass Spectrum Report - MS

Analysis Name: 20150605-2.d

Instrument: LC-MSD-Trap-SL

Print Date: 2015-6-5 13:27:01

Method: linsheng.m

Operator: Operator

Acq. Date: 2014-5-22 9:23:38

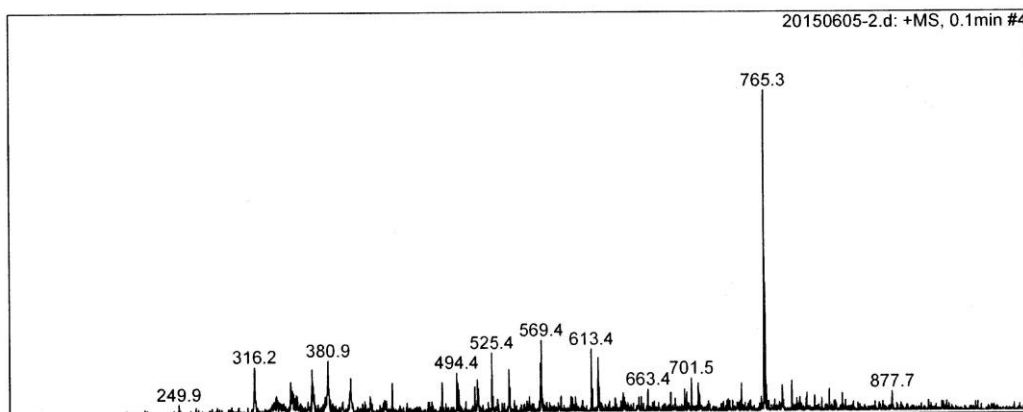
Sample Name: SJJ-w96

Analysis Info:

Acquisition Parameter:

| | | | | | |
|-----------------|------------|-----------------------|-------------|----------------|-----------|
| Mass Range Mode | Std/Normal | Trap Drive | 82.8 | Scan Begin | 100 m/z |
| Ion Polarity | Positive | Skim 1 | -40.0 Volt | Scan End | 1000 m/z |
| Ion Source Type | ESI | Skim 2 | 5.0 Volt | Averages | 7 Spectra |
| Dry Temp (Set) | 330 度 | Octopole RF Amplitude | 200.0 Vpp | Max. Accu Time | 200000 秒 |
| Nebulizer (Set) | 15.00 psi | Capillary Exit | -151.0 Volt | ICC Target | 10000 |
| Dry Gas (Set) | 6.00 l/min | | | Charge Control | on |

+MS, 0.1min #4



-MS, 0.3min #11

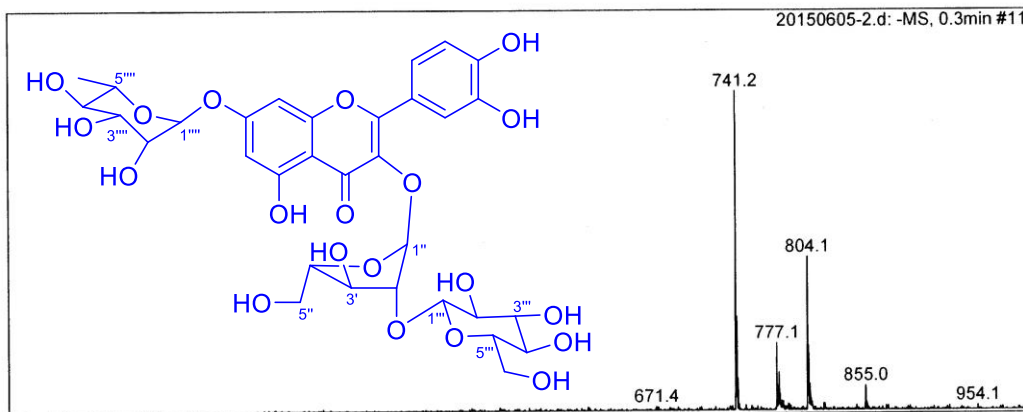


Figure S93. The ESIMS Spectrum of Compound 9

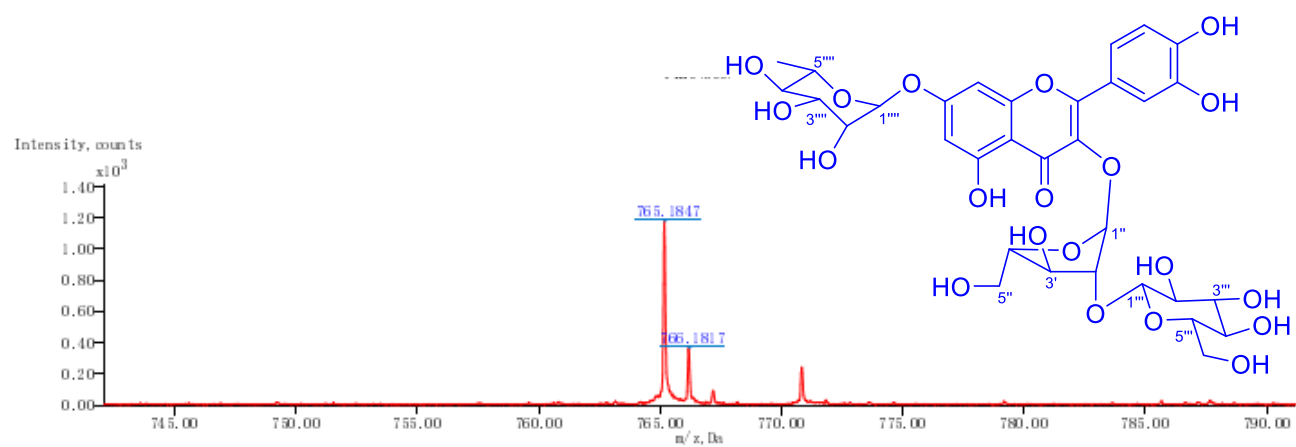


Figure S94. The HRESIMS Spectrum of Compound 9

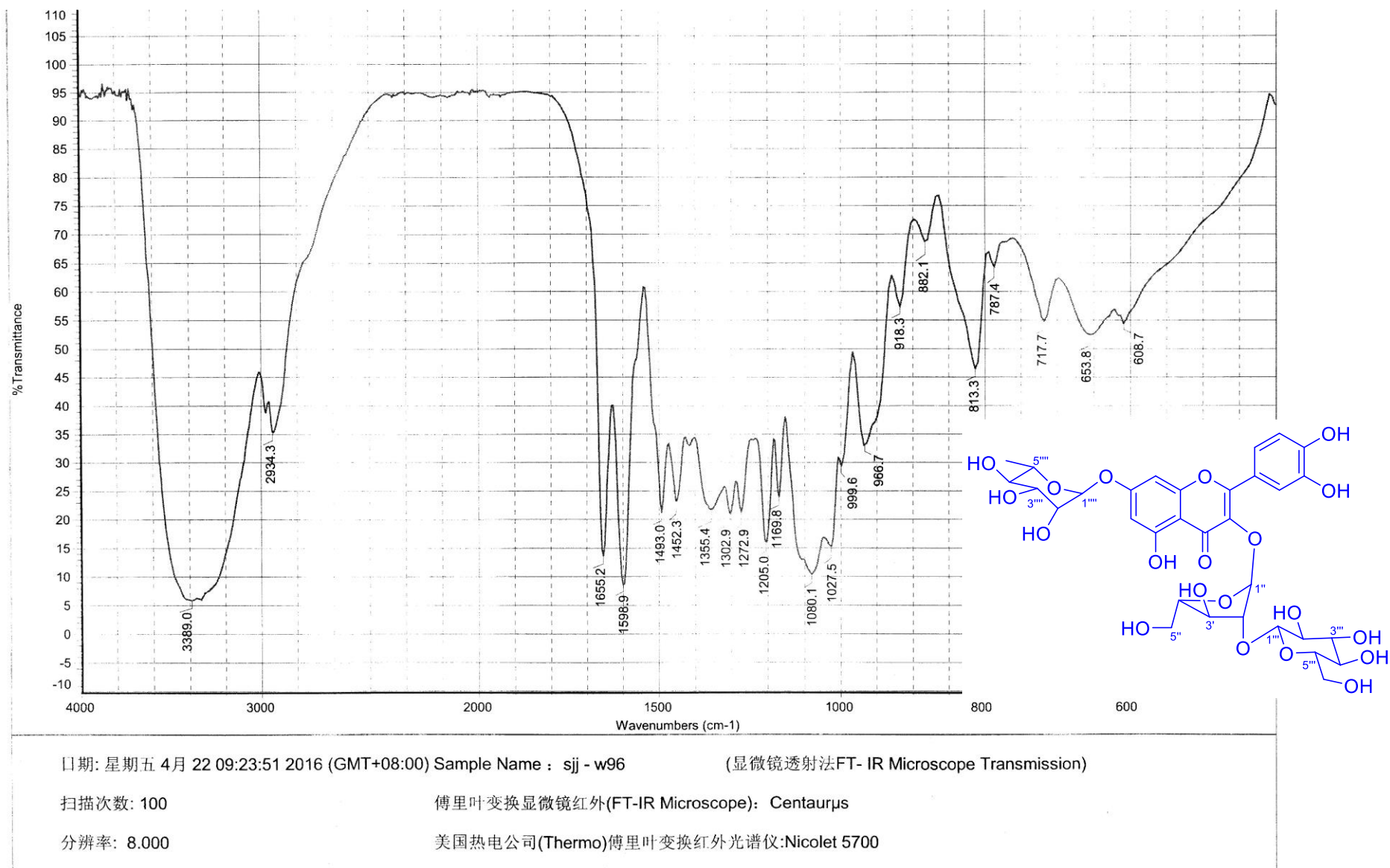


Figure S95. The IR Spectrum of Compound 9

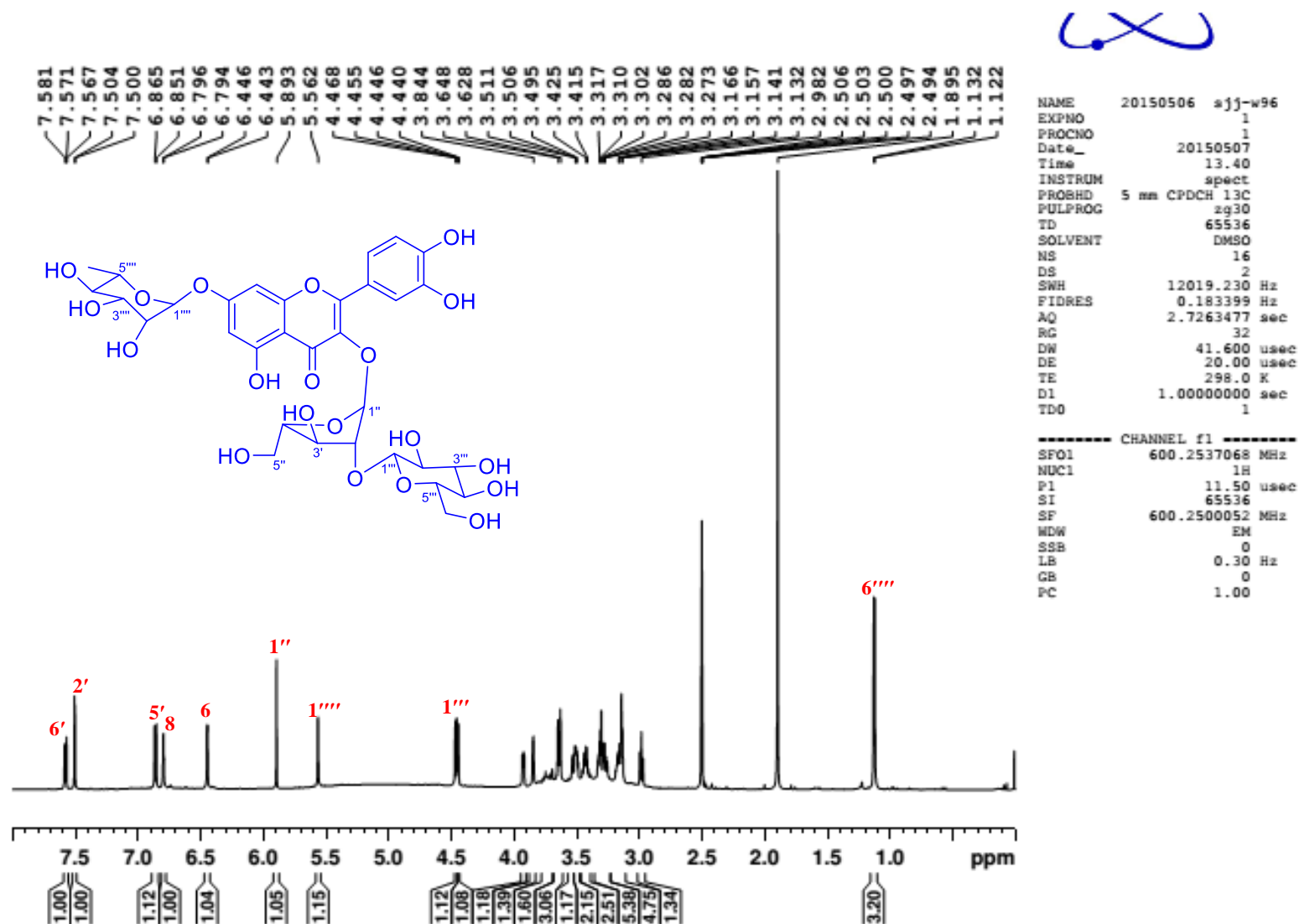


Figure S96. The ^1H NMR Spectrum of Compound 9 in $\text{MeOH-}d_4$ (600MHz)

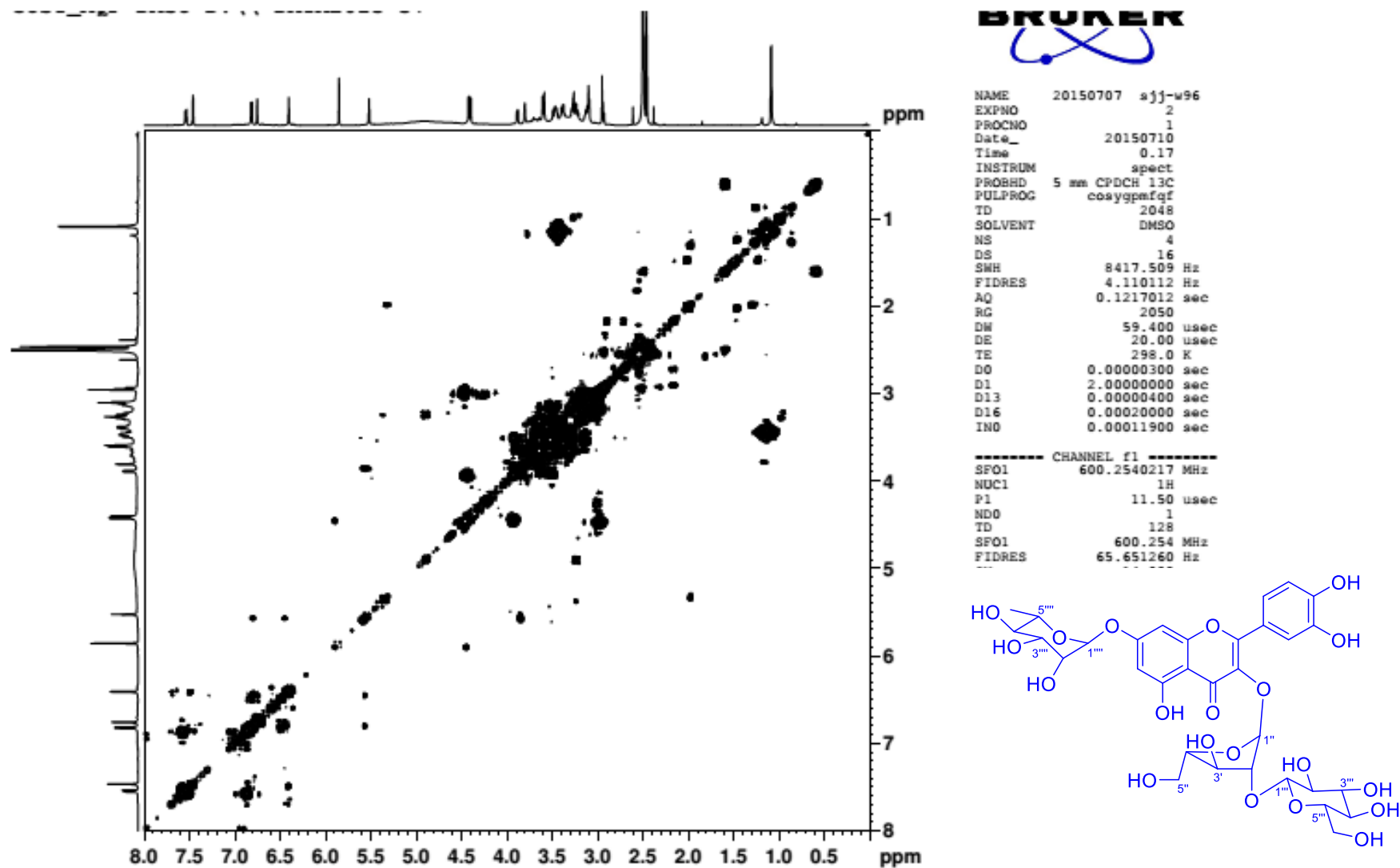


Figure S98. The ^1H - ^1H COSY Spectrum of Compound 9 in $\text{MeOH-}d_4$ (600MHz)

Bruker AVIIIHD 600 20150707
HSQC DMSO D:\ DATA2015 57

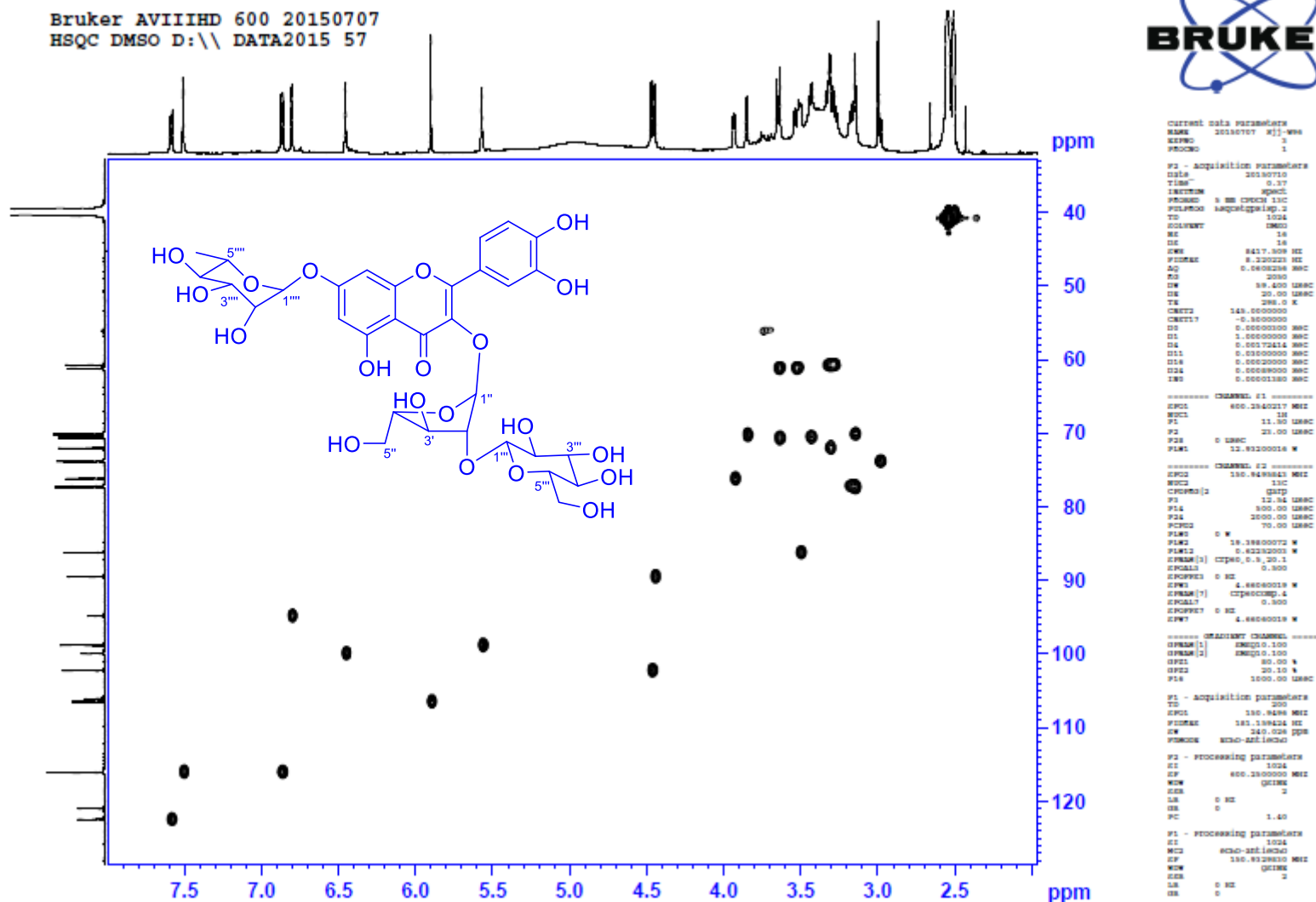


Figure S99. The HSQC Spectrum of Compound 9 in MeOH-*d*₄ (600MHz)

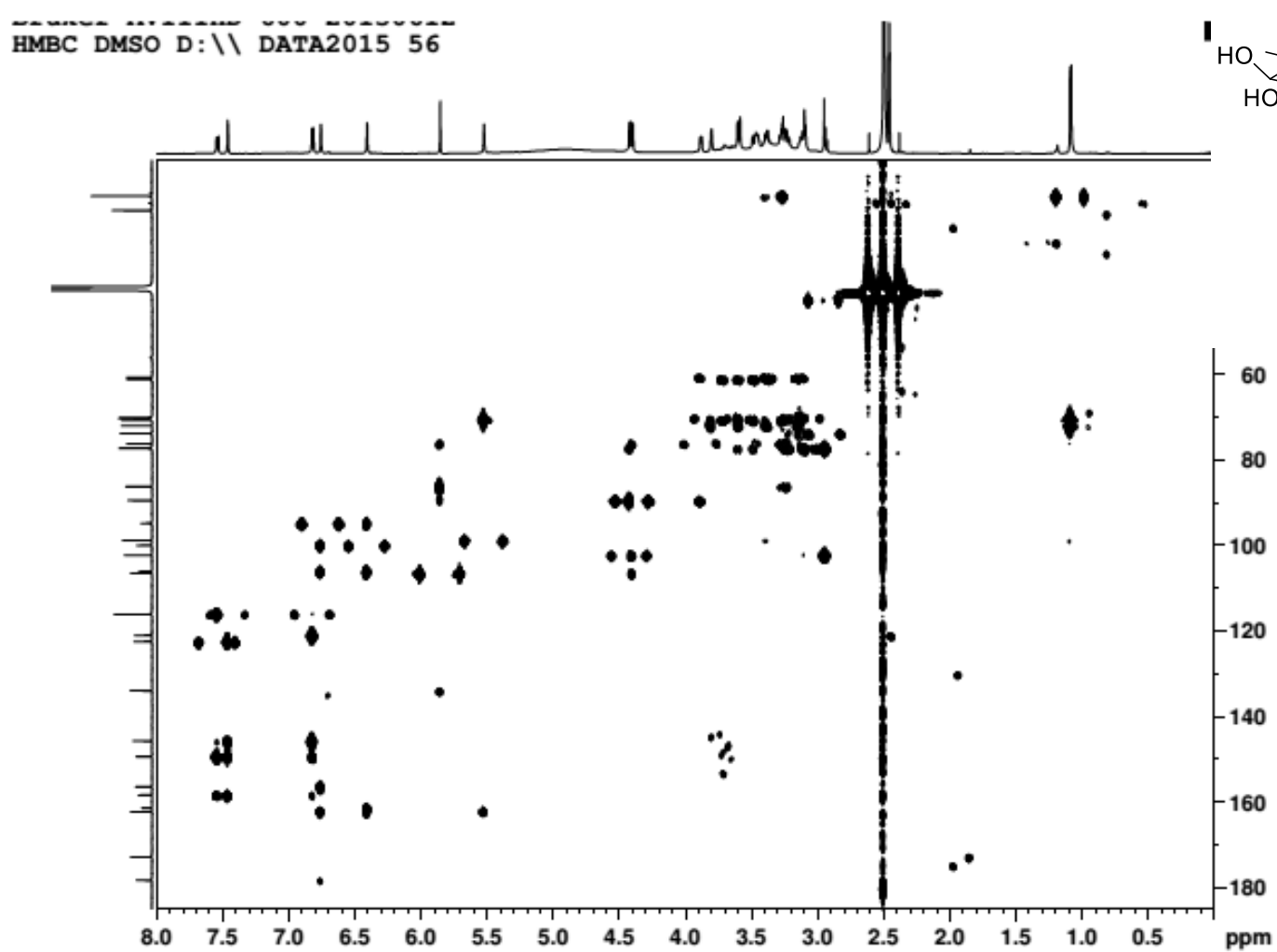
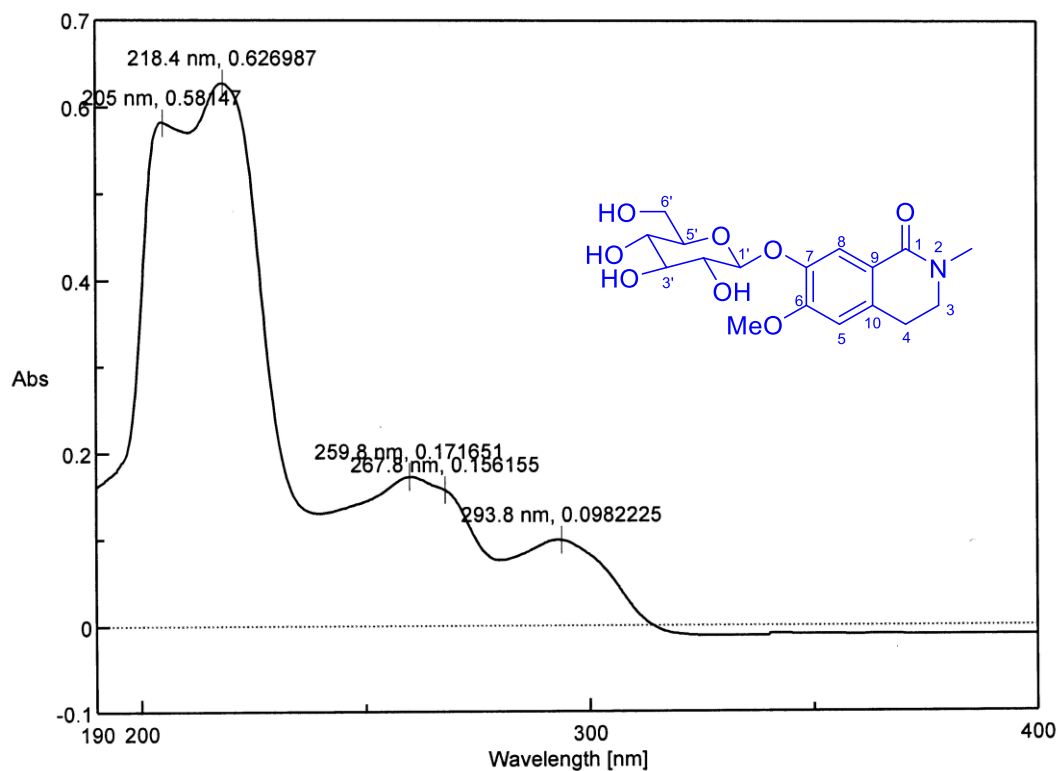


Figure S100. The HMBC Spectrum of Compound 9 in MeOH- d_4 (600MHz)



[Comment]
 Sample Name sij-12
 Comment 0.02
 User
 Division UV
 Company 324
 [Measurement Information]
 Instrument Name V-650
 Model Name V-650
 Serial No. A034461150

Accessory PSC-718
 Accessory S/N A001761114
 Position 1
 Cell Length 10 mm
 Temperature 19.97 C
 Control Sensor Holder
 Monitor Sensor Holder
 Start Mode Start immediately

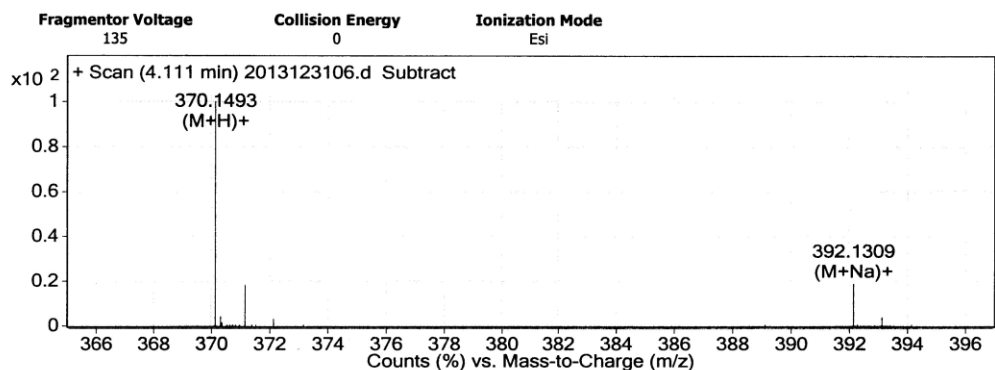
Photometric Mode Abs
 Measurement range 400 - 190 nm
 Data pitch 0.2 nm
 Band width(UV/Vis) 2.0 nm
 Response Medium
 Scanning speed 200 nm/min
 Source Change 340 nm
 Light Source D2/WI
 Filter Exchange Step
 Correction Baseline

[Data Information]
 Creation Date 2014-11-18 20:13

Data array type Linear data array
 Horizontal Wavelength [nm]
 Vertical Abs
 Start 400 nm
 End 190 nm
 Data pitch 0.2 nm
 Data points 1051

Figure S101. The UV Spectrum of Compound 10 in MeOH

Qualitative Analysis Report

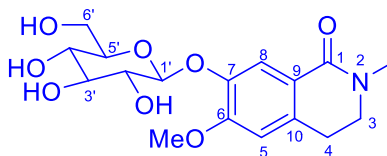


Peak List

| m/z | z | Abund | Formula | Ion |
|----------|---|--------|-----------------|---------|
| 370.1493 | 1 | 610997 | C17 H24 N O8 | (M+H)+ |
| 371.1521 | 1 | 113702 | C17 H24 N O8 | (M+H)+ |
| 392.1309 | | 114552 | C17 H23 N Na O8 | (M+Na)+ |
| 761.2725 | 1 | 277506 | | |
| 762.2752 | 1 | 104815 | | |

Formula Calculator Element Limits

| Element | Min | Max |
|---------|-----|-----|
| C | 3 | 100 |
| H | 0 | 500 |
| O | 0 | 90 |
| N | 0 | 5 |
| S | 0 | 5 |
| Cl | 0 | 2 |
| Br | 0 | 0 |
| Si | 0 | 0 |
| F | 0 | 0 |
| P | 0 | 0 |



Formula Calculator Results

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|-----------------|------|----------|----------|------------|--------------------|-------|
| C17 H23 N O8 | TRUE | 369.142 | 369.1424 | 1 | C17 H24 N O8 | 99.96 |
| C18 H19 N5 O4 | | 369.142 | 369.1437 | 4.62 | C18 H20 N5 O4 | 99.34 |
| C21 H23 N O3 S | | 369.142 | 369.1399 | -5.78 | C21 H24 N O3 S | 98.1 |
| C18 H27 N O3 S2 | | 369.142 | 369.1432 | 3.35 | C18 H28 N O3 S2 | 97.16 |
| C17 H23 N O8 | TRUE | 369.1417 | 369.1424 | 1.8 | C17 H23 N Na O8 | 99.87 |
| C18 H19 N5 O4 | | 369.1417 | 369.1437 | 5.41 | C18 H19 N5 Na O4 | 99.52 |
| C21 H23 N O3 S | | 369.1417 | 369.1399 | -4.98 | C21 H23 N Na O3 S | 98.83 |
| C18 H27 N O3 S2 | | 369.1417 | 369.1432 | 4.14 | C18 H27 N Na O3 S2 | 97.32 |

--- End Of Report ---

Figure S102. The HRESIMS Spectrum of Compound 10

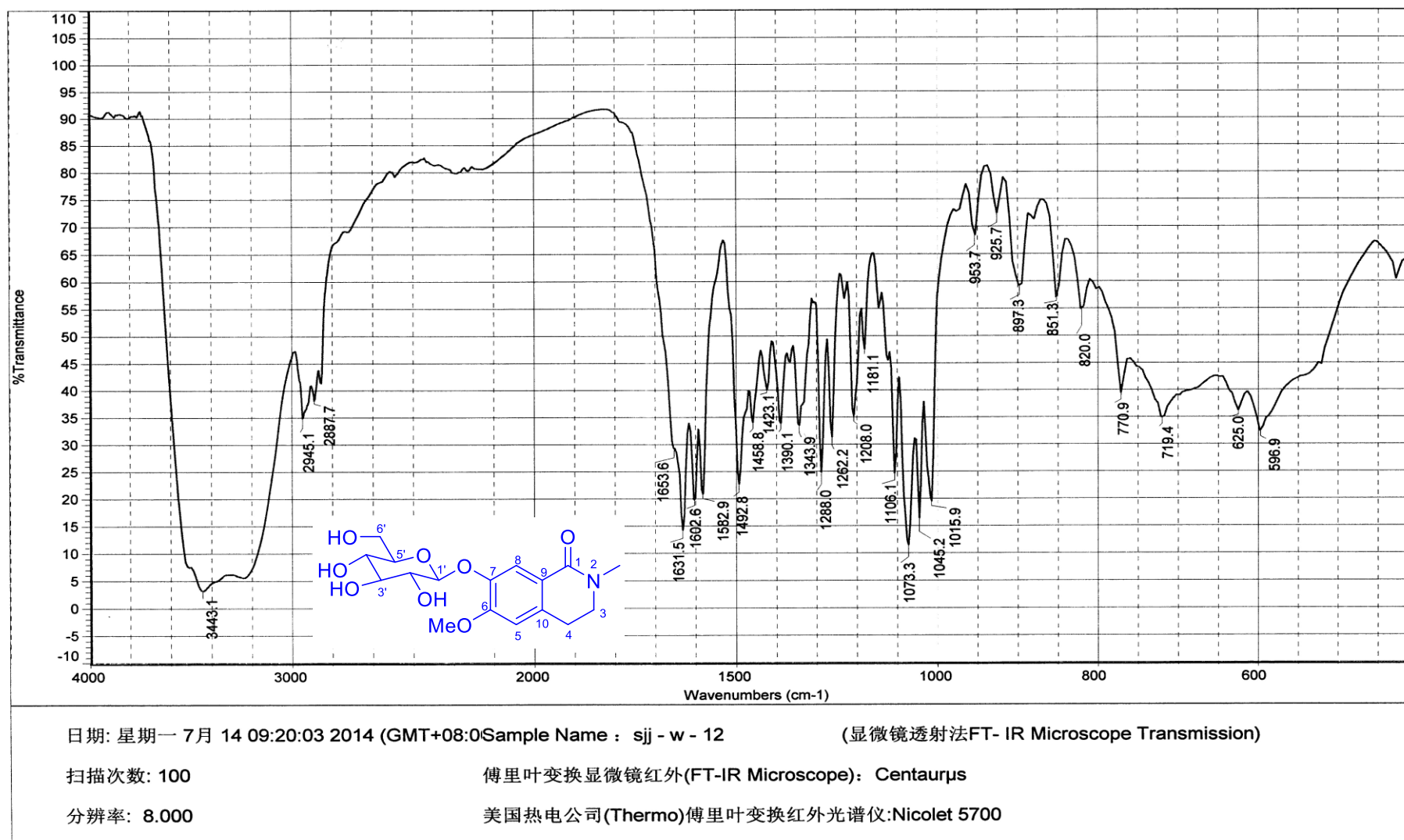


Figure S103. The IR Spectrum of Compound 10

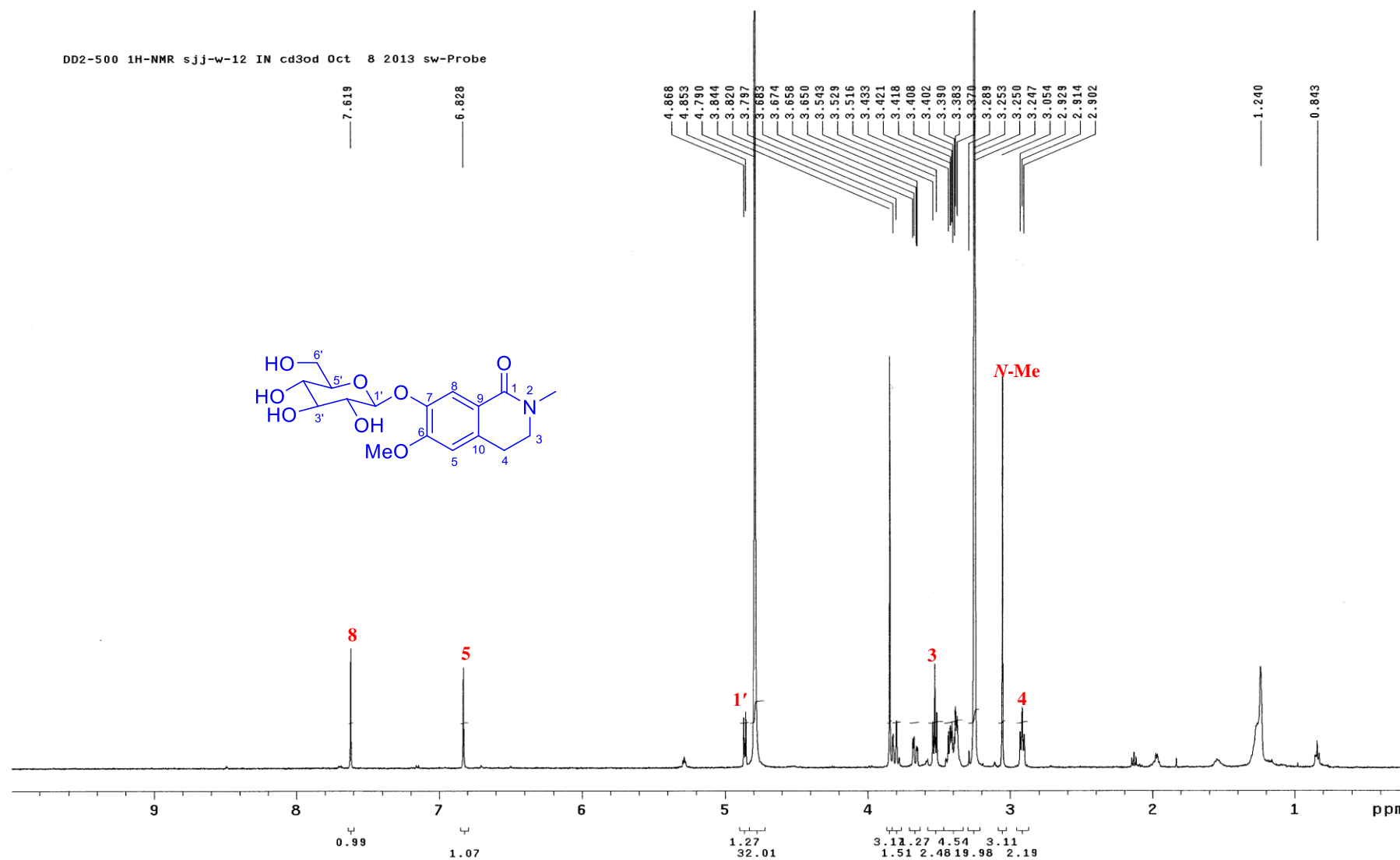


Figure S104.The ^1H NMR Spectrum of Compound 10 in $\text{MeOH-}d_4$ (500MHz)

DD2-500 13C-NMR sjj-w-12 IN acetone Oct 11 2013 sw-Probe

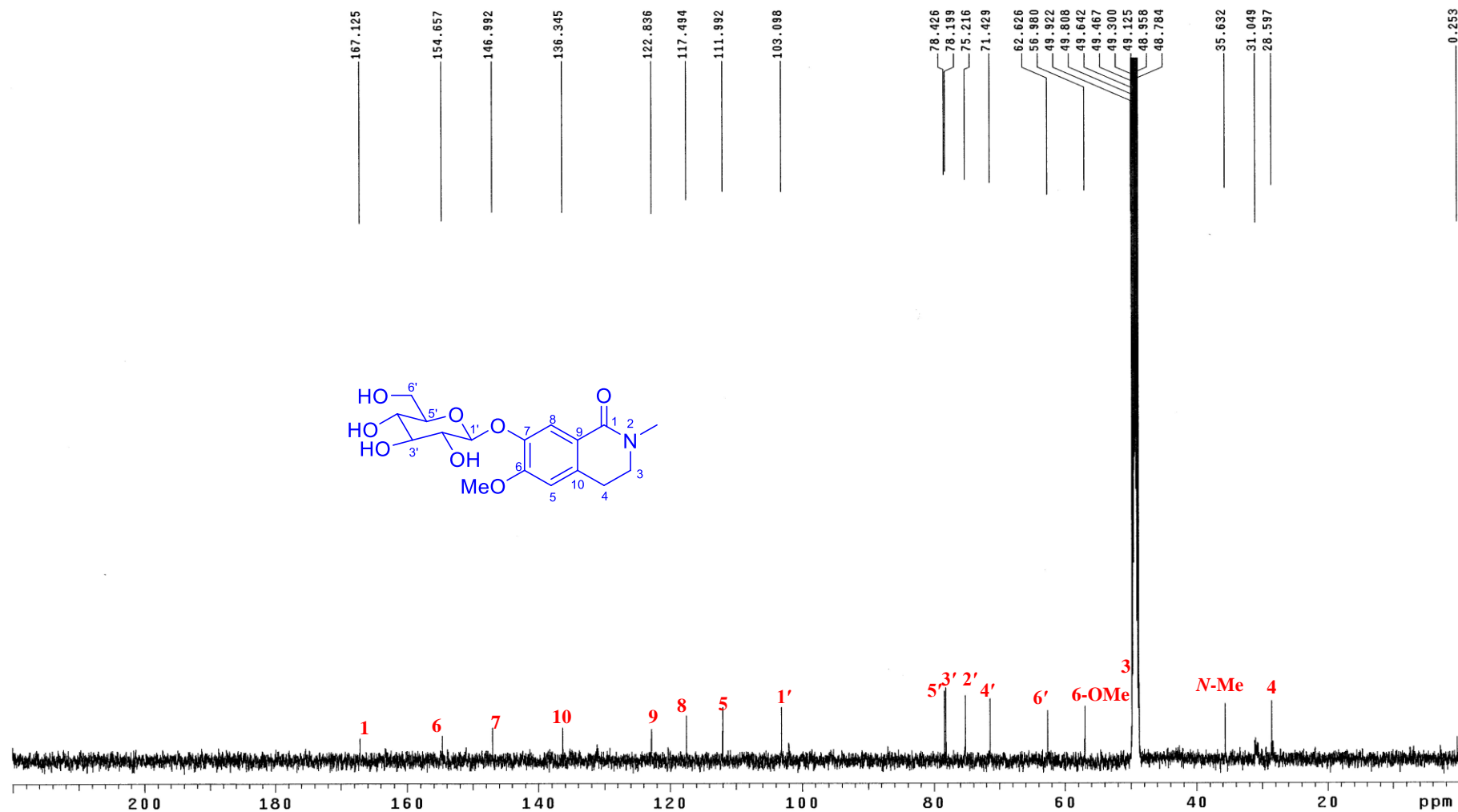


Figure S105. The ^{13}C NMR Spectrum of Compound 10 in $\text{MeOH-}d_4$ (500MHz)

DD2-500 gCOSY sjj-w-12 IN cd3od Jan 2 2014 sw

Temp. 25.0 C / 298.1 K
Sample #10, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4845.0 Hz
2D Width 4845.0 Hz
4 repetitions
200 increments
OBSERVE H1, 499.7694214 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.025 sec
FT size 2048 x 2048
Total time 16 min

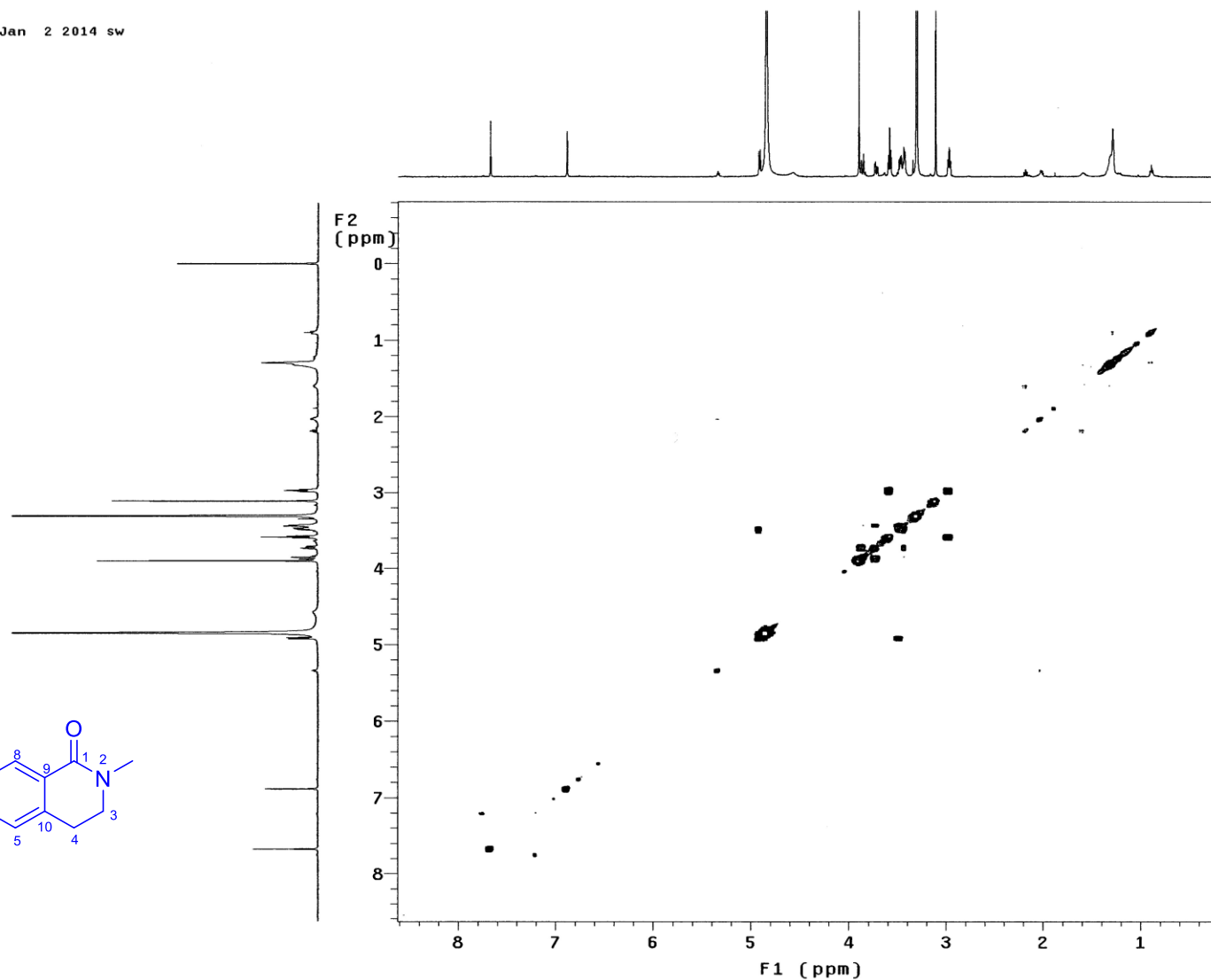
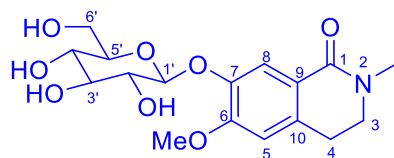


Figure S106. The ^1H - ^1H COSY Spectrum of Compound 10 in $\text{MeOH-}d_4$ (500MHz)

Temp. 25.0 C / 298.1 K
 Sample #10, Operator: vnmr1
 Relax. delay 1.000 sec
 Acq. time 0.248 sec
 Width 4845.0 Hz
 2D Width 25133.5 Hz
 16 repetitions
 2 x 256 increments
 OBSERVE H1, 499.7694214 MHz
 DECOUPLE C13, 125.6784310 MHz
 Power 39 dB
 on during acquisition
 off during delay
 w40_sw modulated
 DATA PROCESSING
 Gauss apodization 0.069 sec
 F1 DATA PROCESSING
 Gauss apodization 0.009 sec
 FT size 4096 x 2048
 Total time 2 hr, 44 min

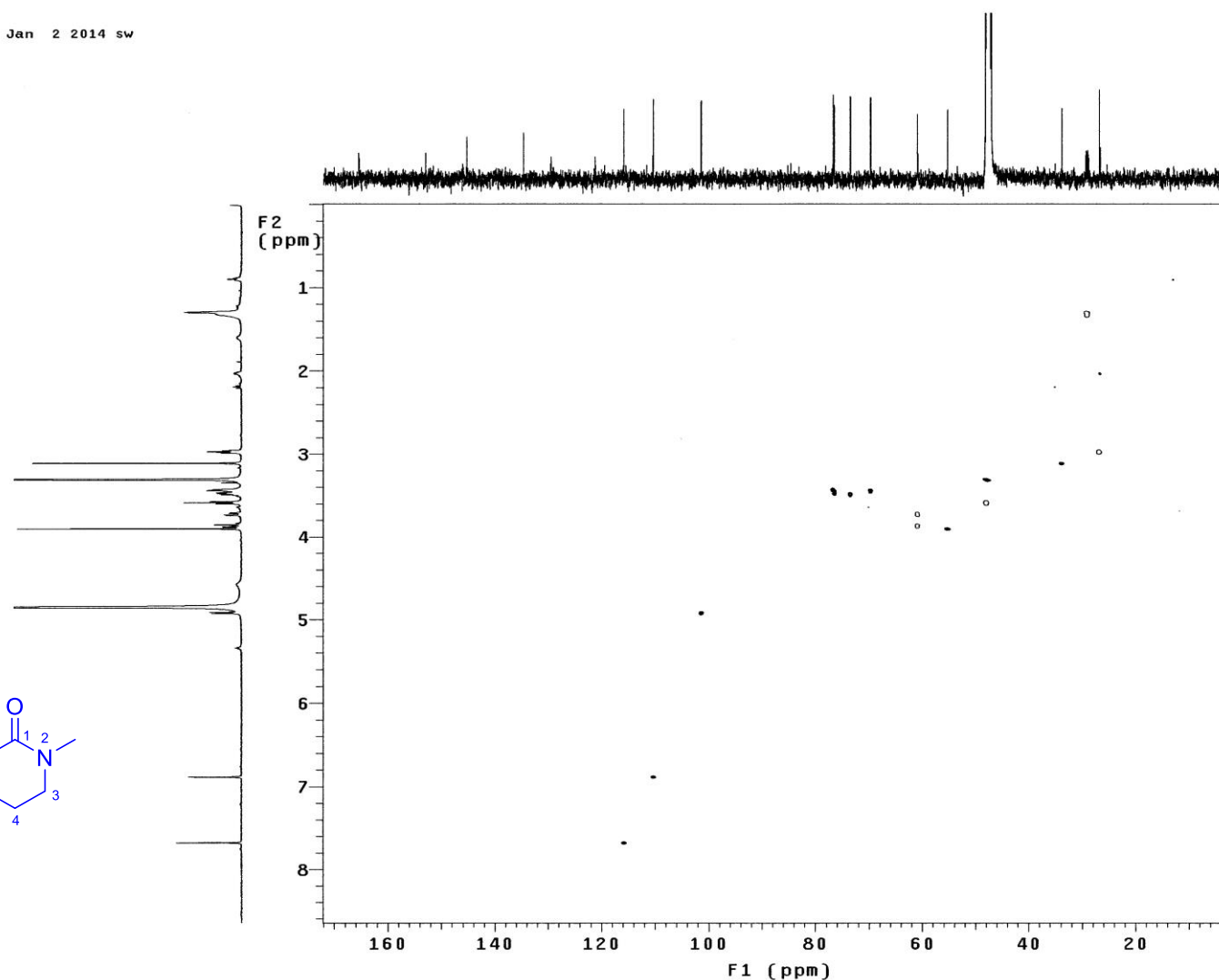
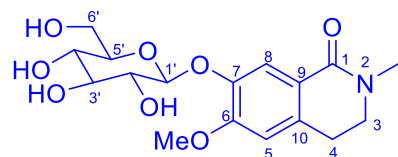


Figure S107. The HSQC Spectrum of Compound 10 in MeOH-*d*₄ (500MHz)

DD2-500 gHMCAD sjj-w-12 IN cd3od Dec 27 2013 sw

Temp. 25.0 C / 298.1 K
Sample #9, Operator: vnmr1

Relax. delay 1.000 sec
Acq. time 0.246 sec
Width 4882.8 Hz
2D Width 30154.5 Hz
32 repetitions
2 x 200 increments
OBSERVE H1, 499.7694214 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Gauss apodization 0.006 sec
FT size 4096 x 2048
Total time 4 hr, 24 min

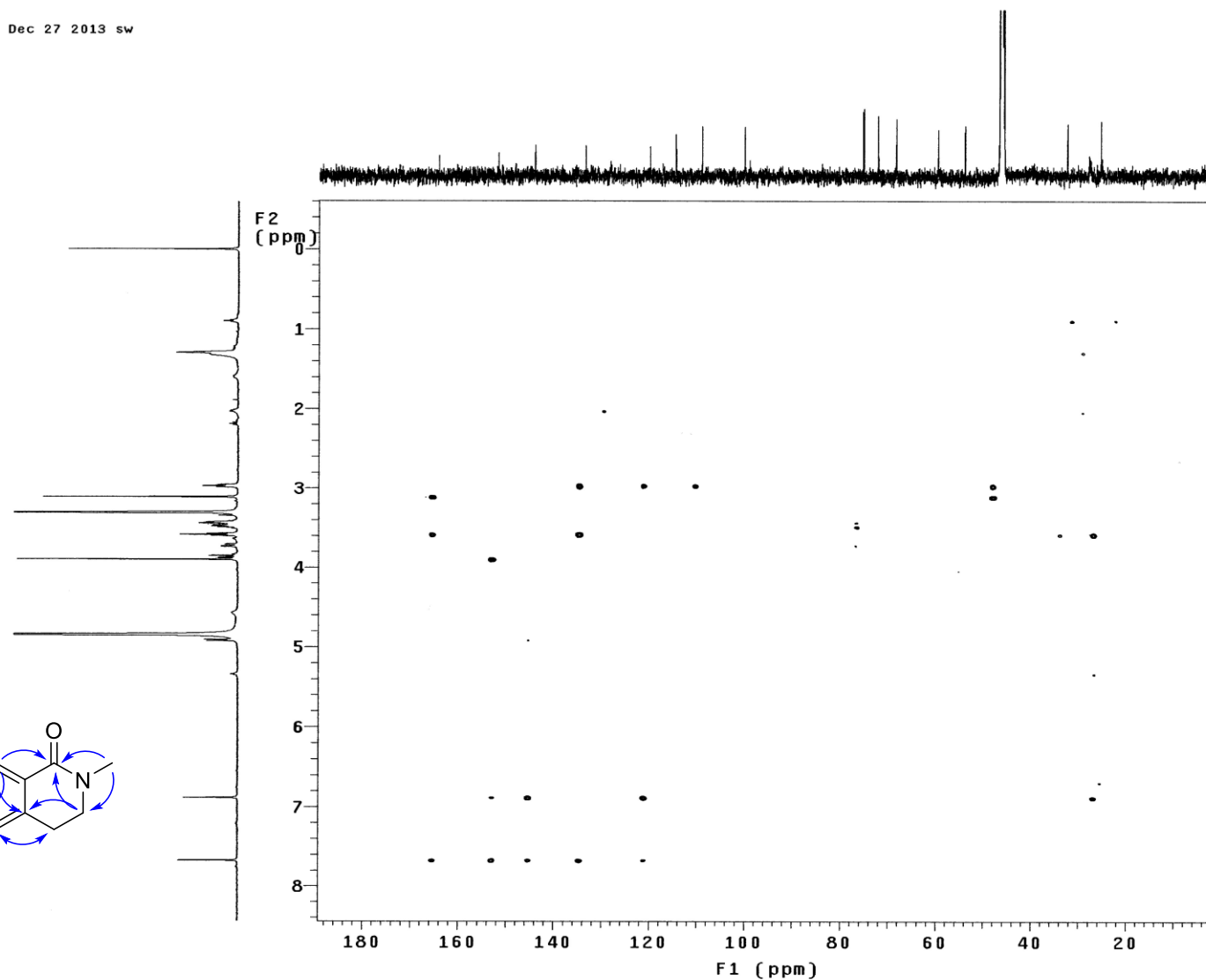
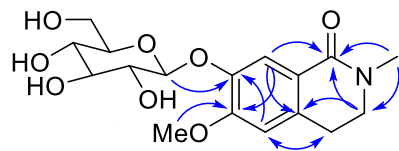


Figure S108. The HMBC Spectrum of Compound 10 in MeOH- d_4 (500MHz)

DD2-500 NOESY1D sjj-w-12 IN cd3od sw-Probe

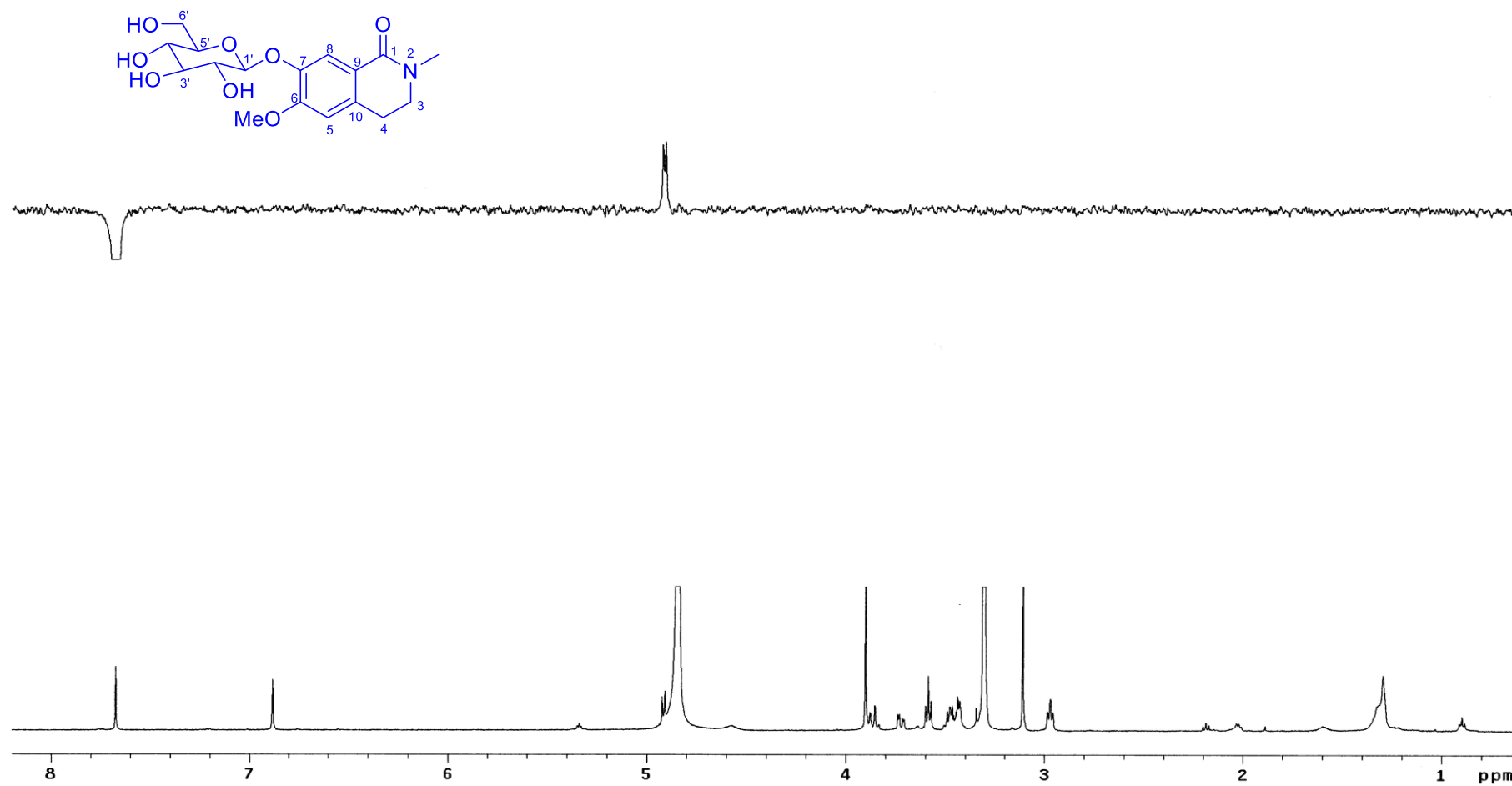


Figure S109. The NOE Difference Spectrum of Compound 10 in MeOH- d_4 (500MHz)