

Natural Nitric Oxide (NO) inhibitors from the rhizomes of *Curcuma phaeocaulis*

Supplementary Information

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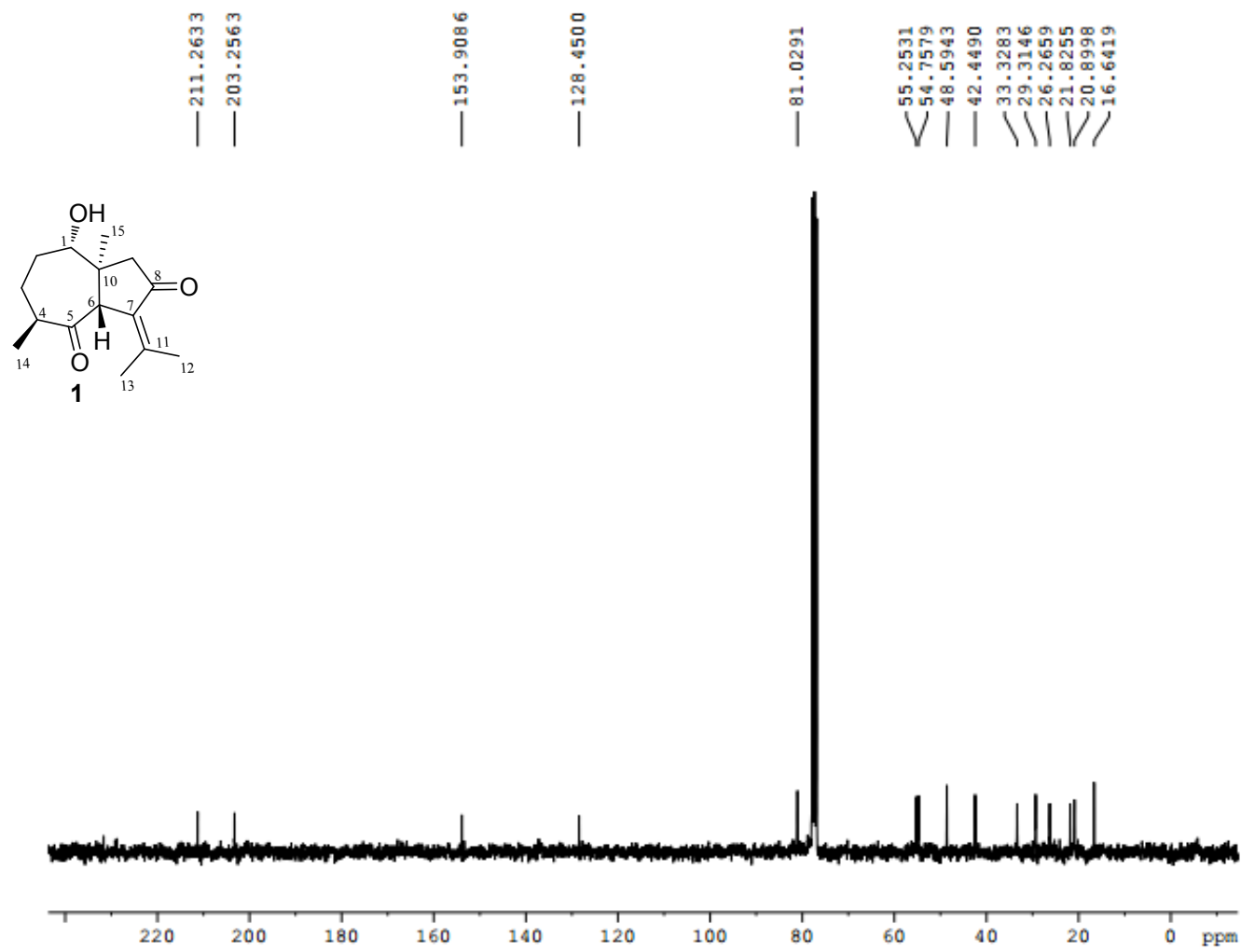
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Figure S1. ¹³C NMR spectra of phasalvione (1)



NAME JE-16
 EXPNO 2
 PROCNO 1

Figure S2. ¹H NMR spectra of phasalvione (1)

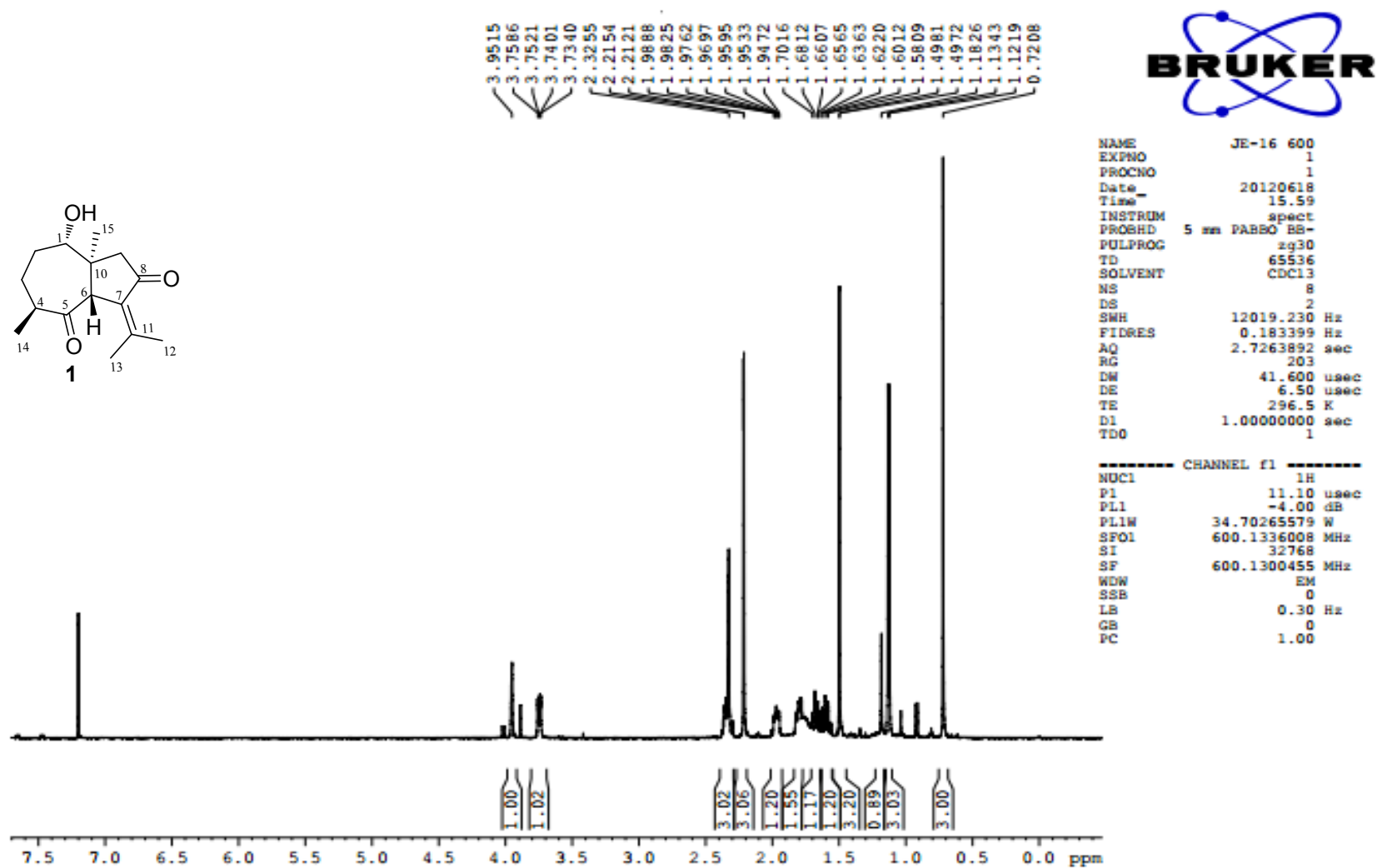
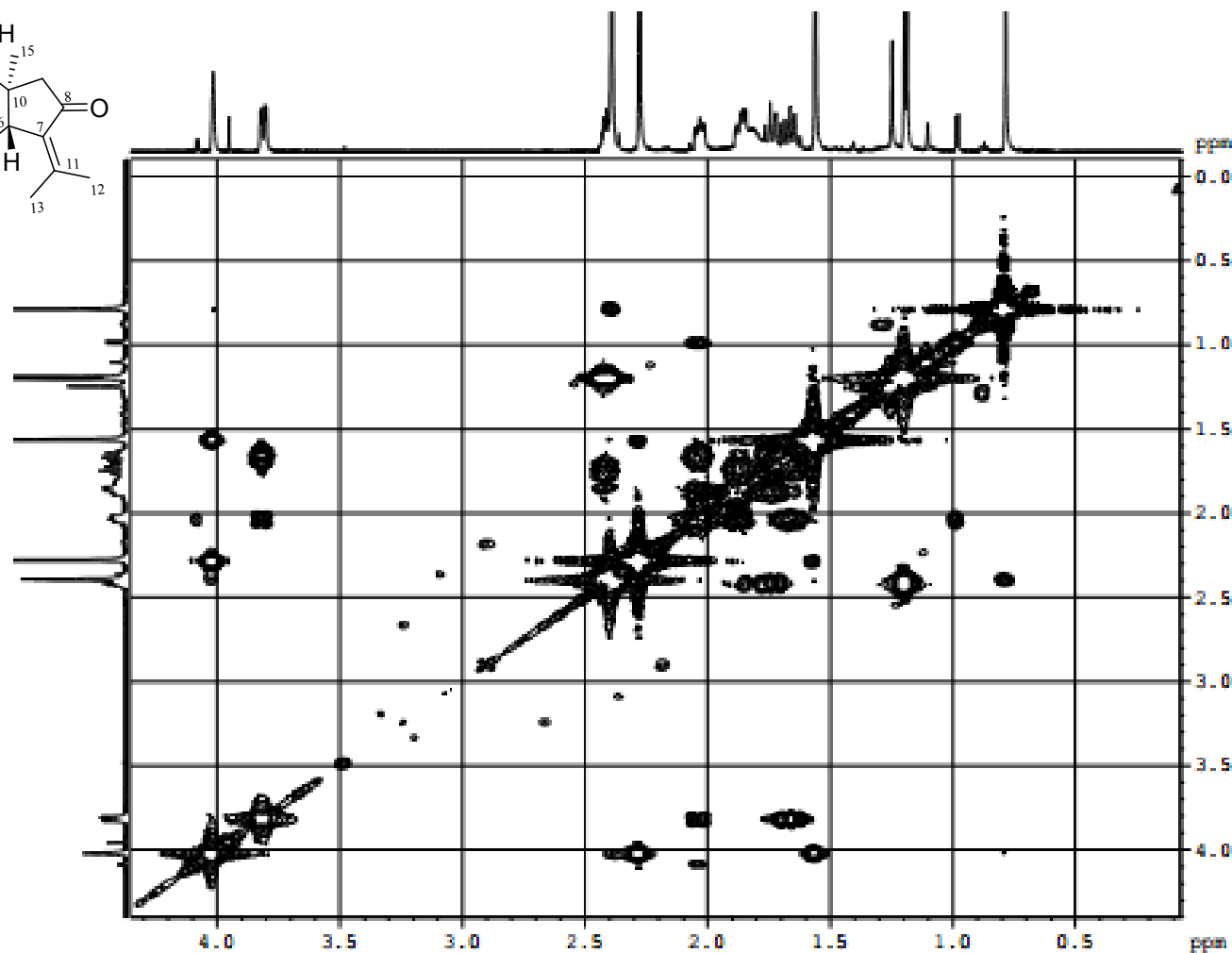
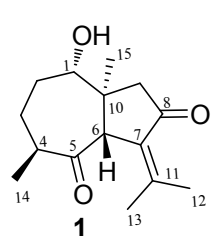


Figure S3. ^1H - ^1H COSY spectra of phasalvione (**1**)



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PROCNO        1
Date_         20130629
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INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       cosyppqf
TD            1634
SOLVENT       CDCl3
NS            4
DS            8
SWH           6009.615 Hz
FIDRES        5.868765 Hz
AQ            0.0653300 sec
RG            181
DN            83.200 usec
DE            6.50 usec
TE            296.3 K
D0            0.0000300 sec
D1            2.0000000 sec
D13           0.0000400 sec
D16           0.0002000 sec
IND           0.00016640 sec
    
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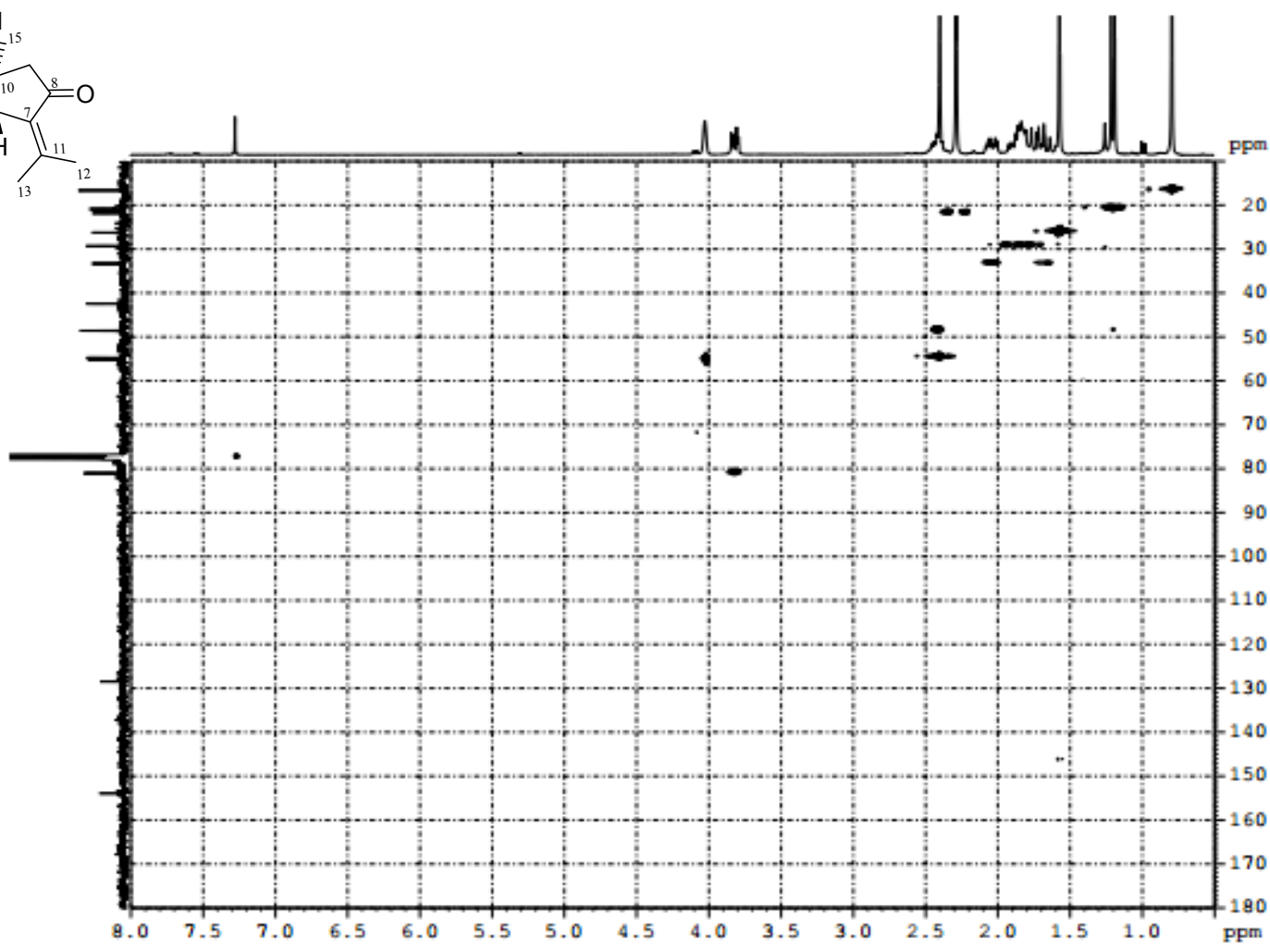
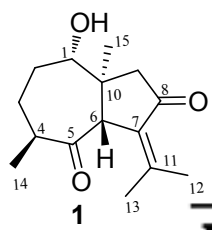
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P0            11.10 usec
P1            11.10 usec
PC1           -4.00 dB
PC1W          34.7626579 W
SFO1          600.1374805 MHz
    
```

```

***** GRADIENT CHANNEL *****
GPRAMP1      SINE.100
GPE1         16.00 %
PL1          1000.00 usec
MDS          1
TD           356
SFO1         600.1374 MHz
FIDRES       23.475823 Hz
SN           10.014 ppm
PRANDR       QP
SI           1634
SF           600.1374805 MHz
WDW          SINE
SSB          0
LB           0.00 Hz
GB           0
PC           1.40
SI           1634
SF           QP
WDW          0
SSB          0
LB           0
GB           0
    
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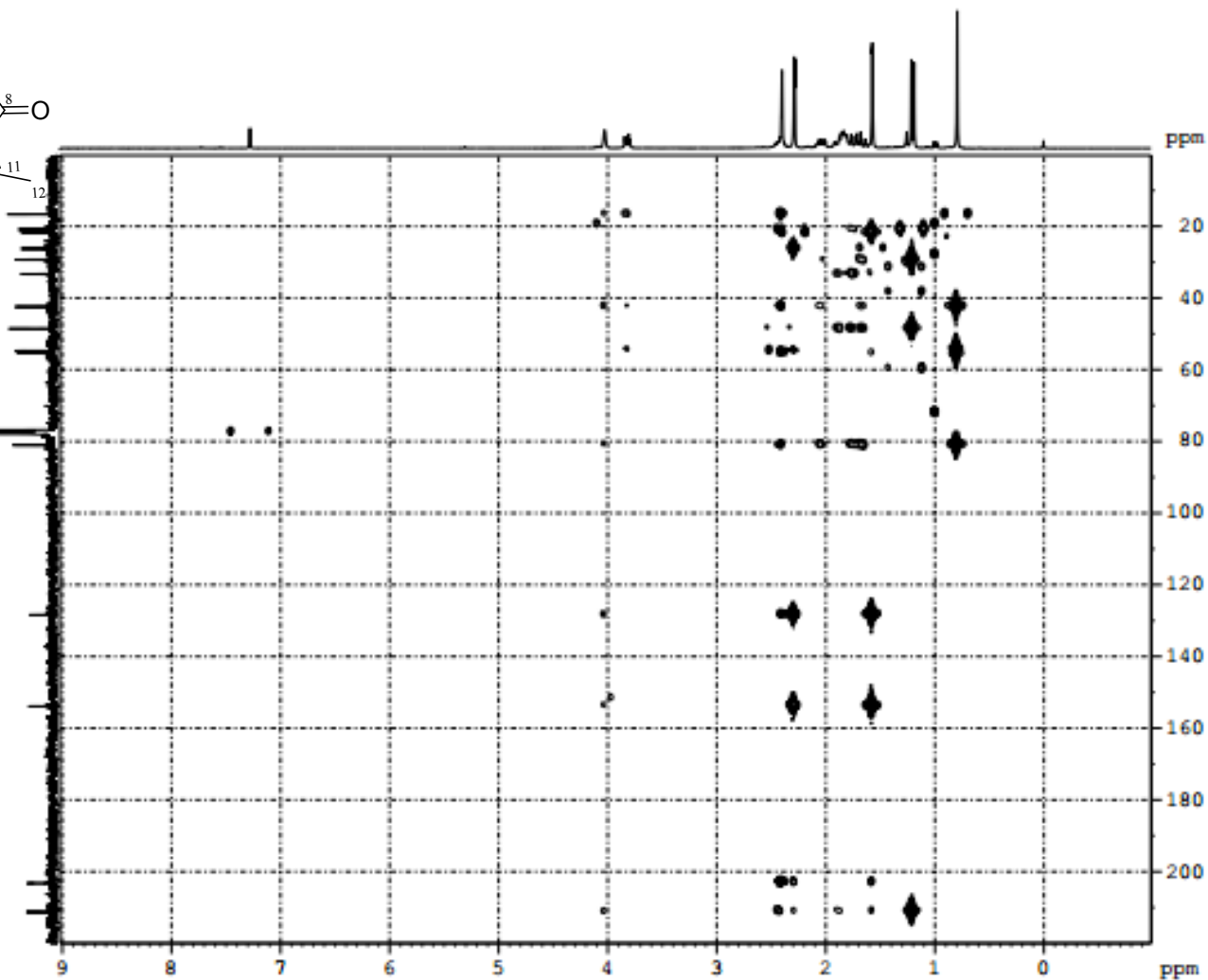
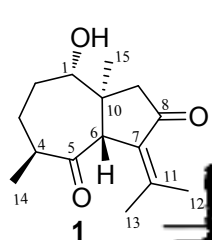
Figure S4. HSQC spectra of phasalvione (1)



```

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PROCNO        1
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Time          14.03
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            1024
SOLVENT       CDCl3
NS            2
DS            4
SWH           4029.413 Hz
FIDRES       0.388748 Hz
AQ           0.0001900 sec
RG           256.00
DM           63.200 umsec
DE           6.30 umsec
TE            296.2 K
CHRG1        143.0000000 umsec
DO           0.0000100 umsec
D1           1.30000000 umsec
d11          0.00170111 umsec
d12          0.01000000 umsec
d13          0.00000100 umsec
d14          0.00010000 umsec
d15          0.00110000 umsec
180
NAME
===== CHANNEL f1 =====
NUC1          13C
P1            11.10 umsec
PC1           22.20 umsec
PL1           1000.00 umsec
PL12          -1.00 dB
PL1M          31.70218379 W
SFO1          100.6261000 MHz
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
P2            6.30 umsec
PC2           17.40 umsec
PL2           80.00 umsec
PL22          1.00 dB
PL2M          20.17 dB
PL2M          33.20218379 W
PL2M          1.00701721 W
SFO2          400.1460000 MHz
===== GRABBER CHANNEL =====
OPBPRG1      ZINE_100
OPBPRG2      ZINE_100
OPB1         80.00 %
OPB2         20.10 %
P16          1000.00 umsec
NS2          2
TD2          256
SFO1         100.6261000 MHz
FIDRES       100.110001 Hz
SWH           180.000 ppm
F2FREQ       Beta-Antimony
Z1           1024
ZF           400.1460000 MHz
NS2          2
DS2          2
SWH2         0.00 Hz
DM2          0
PC2          1.10
ET           1024
MCO         beta-antimony
ZF           150.9434000 MHz
MCM          QZINE
S2B          2
LB          0.00 Hz
DM           0
PC           1.10
ET           1024
MCO         beta-antimony
ZF           400.1460000 MHz
S2B          2
LB          0.00 Hz
DM           0
    
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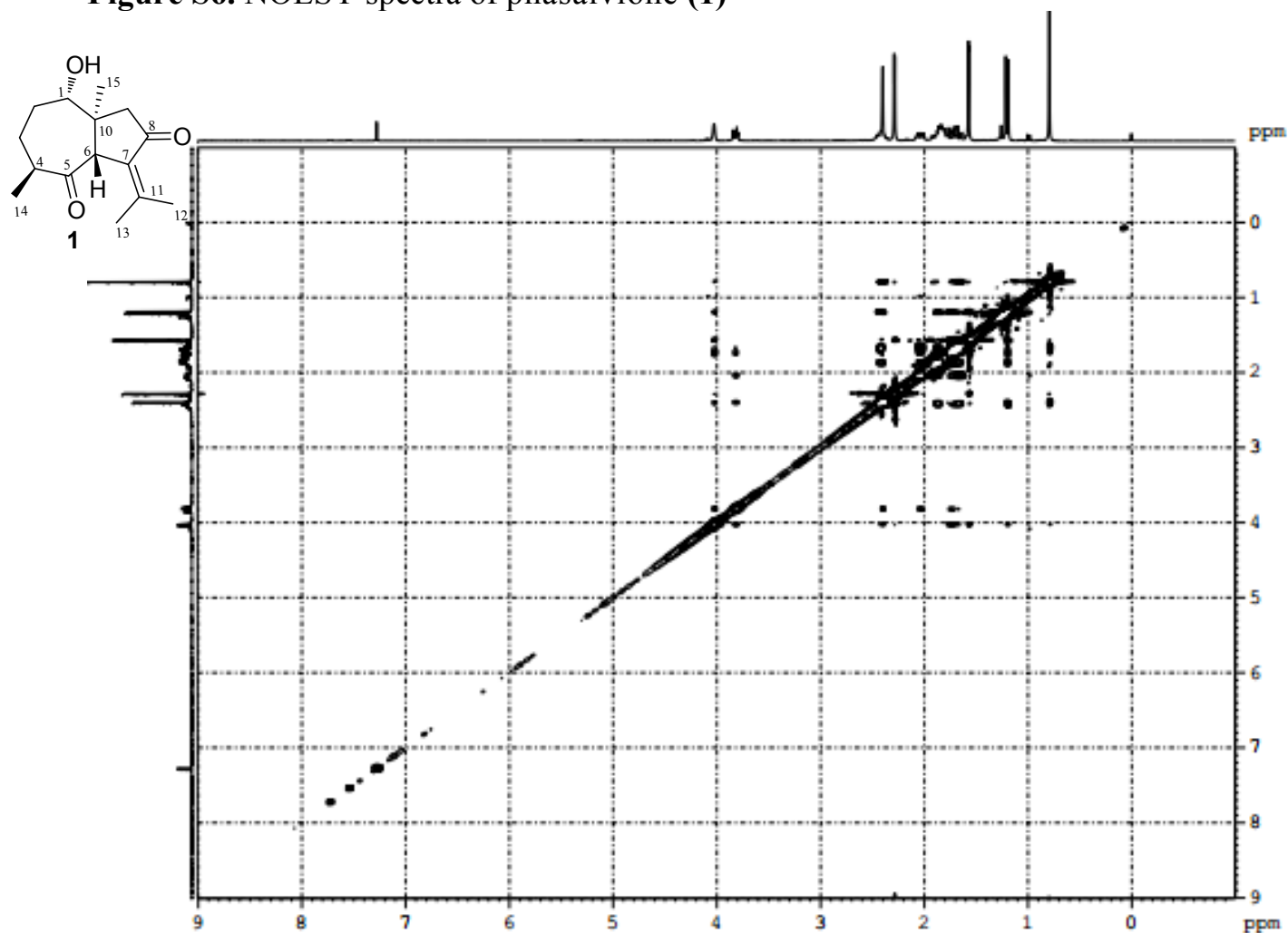
Figure S5. HMBC spectra of phasalvione (1)



```

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EXPNO         1
PROCNO        1
Date_         20120618
Time          16.32
INSTRUM       spect
PROBHD        5 mm QNP1H 2D-
PULPROG       zgpg30
TD            1024
SOLVENT       CDCl3
DE            14
DS            14
EM           600.138 MHz
FIDRES        0.348765 Hz
AQ            0.0011000 sec
RG            2560
SM           81.200 MHz
DE            8.50 MHz
TE            300.2 K
CMT0          149.000000
CMT1          0.000000
D0            0.0000000 sec
D1            1.0000000 sec
D2            0.0010000 sec
D3            0.1000000 sec
D14           0.0000000 sec
RG2           0.0001000 sec
===== CHANNEL f1 =====
NUC1           1H
P1            11.10 MHz
PC            22.20 MHz
PL1           -1.00 dB
PLM           31.7026679 W
SFO1          500.136000 MHz
===== CHANNEL f2 =====
NUC2           13C
P2            8.50 MHz
PC            1.00 dB
PL2           31.7026679 W
SFO2          125.761150 MHz
===== CHANNEL CHANNEL =====
SFO001         510.100 MHz
SFO002         510.100 MHz
SFO003         510.100 MHz
SFO1           50.00 MHz
SFO2           30.00 MHz
SFO3           10.10 MHz
P14           1000.00 MHz
NUC           13
TD            211
SFO1          125.761150 MHz
FIDRES        127.366778 Hz
SM           320.000 ppm
FWDK         GF
SI            1024
SF           500.136000 MHz
RG           0
SZA           0
LB           0.00 MHz
GB           0
PC            1.00
SI            1024
MC2          GF
SF           125.761150 MHz
RG           0
SZA           0
LB           0.00 MHz
GB           0
    
```

Figure S6. NOESY spectra of phasalvione (1)



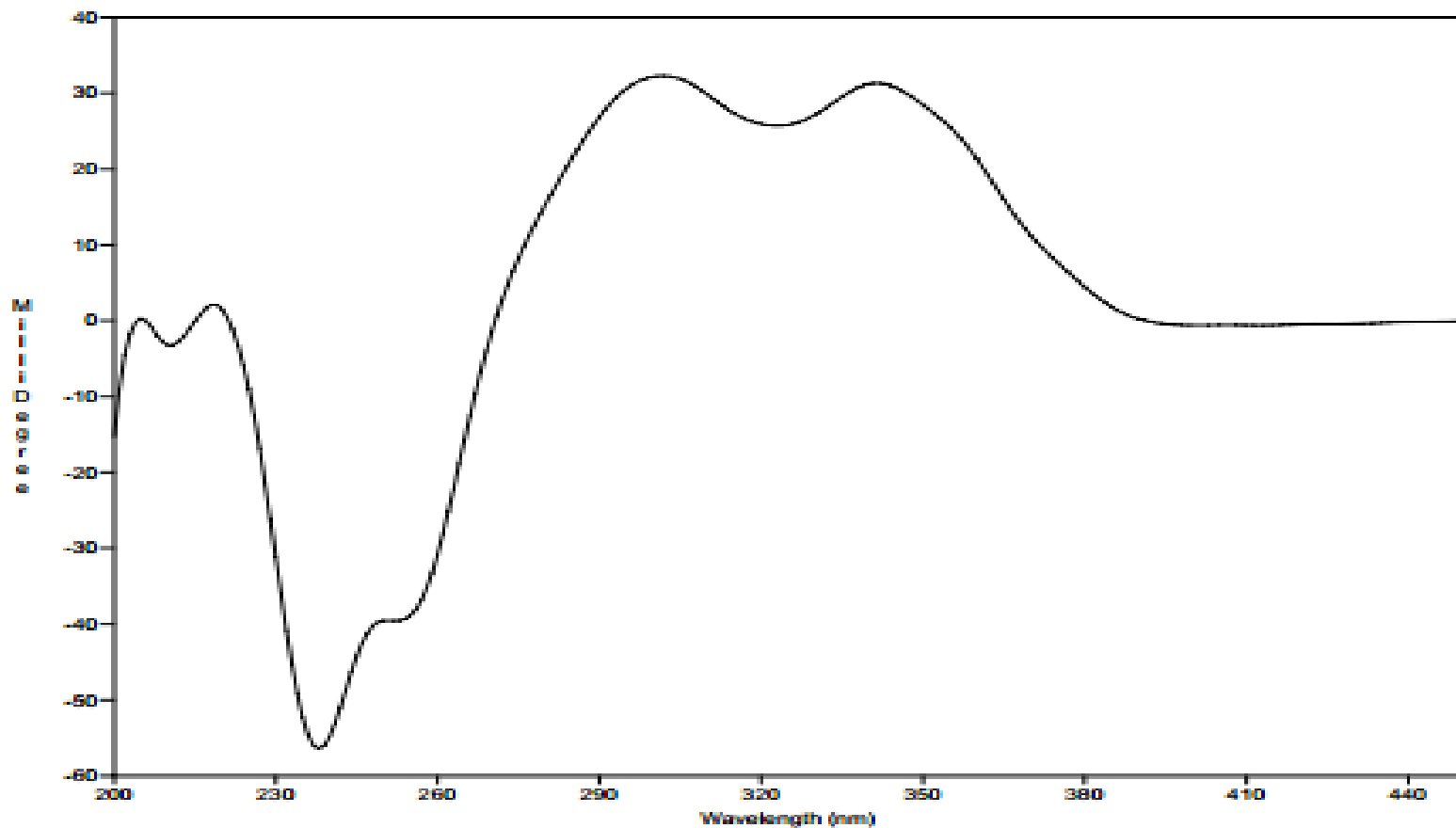
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NAME          JE-16 600
EXPNO         6
PROCNO        1
Date_         20120629
Time          17.11
INSTRUM       spect
PROCBD        5 mm PABBO BB-
PULPROG       noesyph
TD            1024
SOLVENT       CDCl3
NS            4
DS            4
SMH           6009.615 Hz
FIDRES        5.868765 Hz
AQ            0.0853300 sec
RG            64
DW            83.200 usec
DE            6.50 usec
TE            296.1 K
D0            0.00006907 sec
D1            2.00000000 sec
D8            0.60000002 sec
IN0           0.00016640 sec
    
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            11.10 usec
PL1           -4.00 dB
PL1W          34.70265579 W
SFO1          600.1324005 MHz
ND0           1
TD            256
SFO1          600.1324 MHz
FIDRES        23.475023 Hz
SW            10.014 ppm
PRMODE        States-TPPI
SI            1024
SF            600.1300000 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.00
SI            1024
MC2           States-TPPI
SF            600.1300000 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
    
```


Figure S7. Rh₂(OCOFCF₃)₄-induced CD spectra of phasalvione (**1**)



Bio-Kine Software V4.62 Date : 2012-12-28 Time : 17:22:52

COMMENTS :

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Figure S8. HRESIMS spectra of phasalvione (**1**)

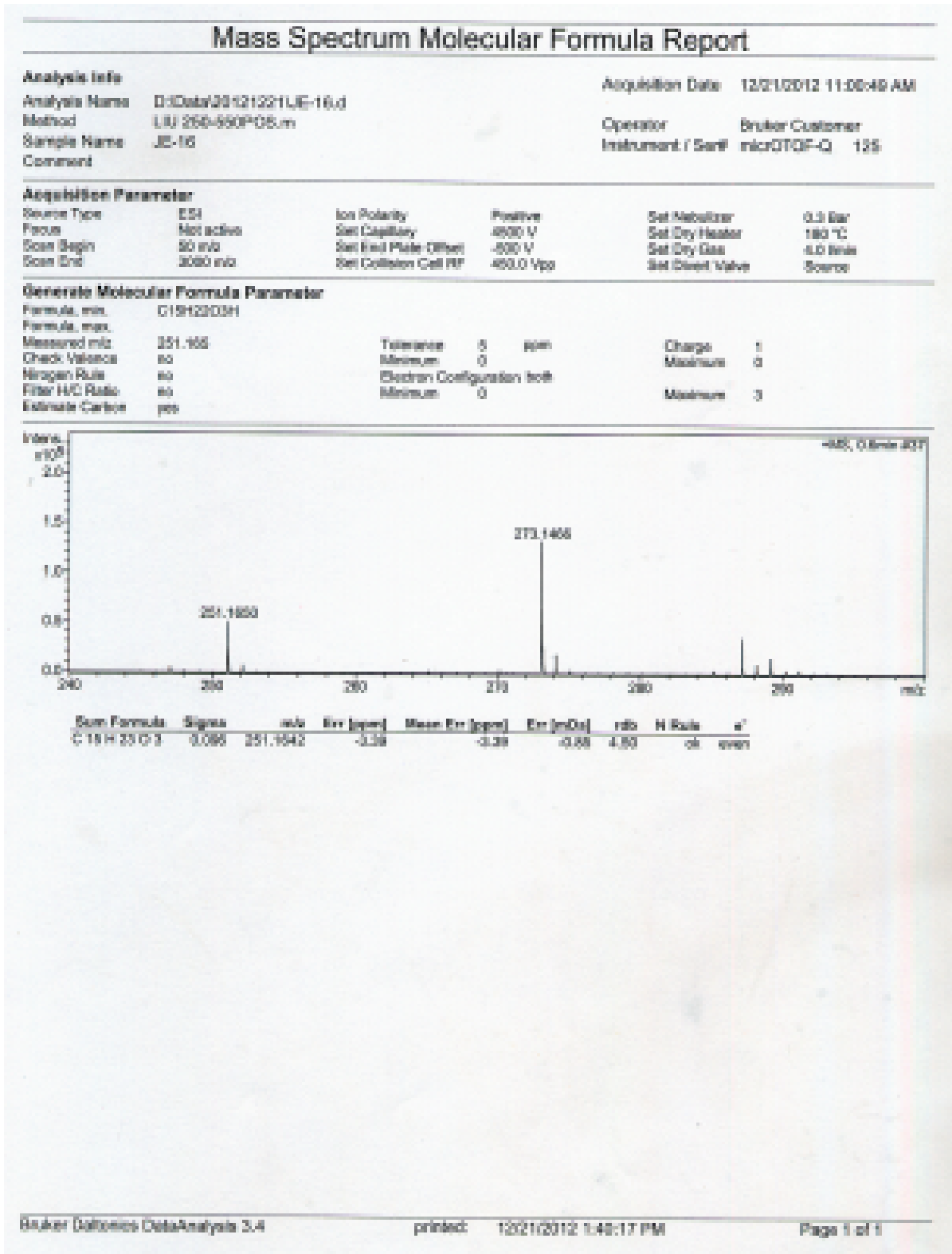


Figure S9. ¹³C NMR spectra of phaeocaudione (2)

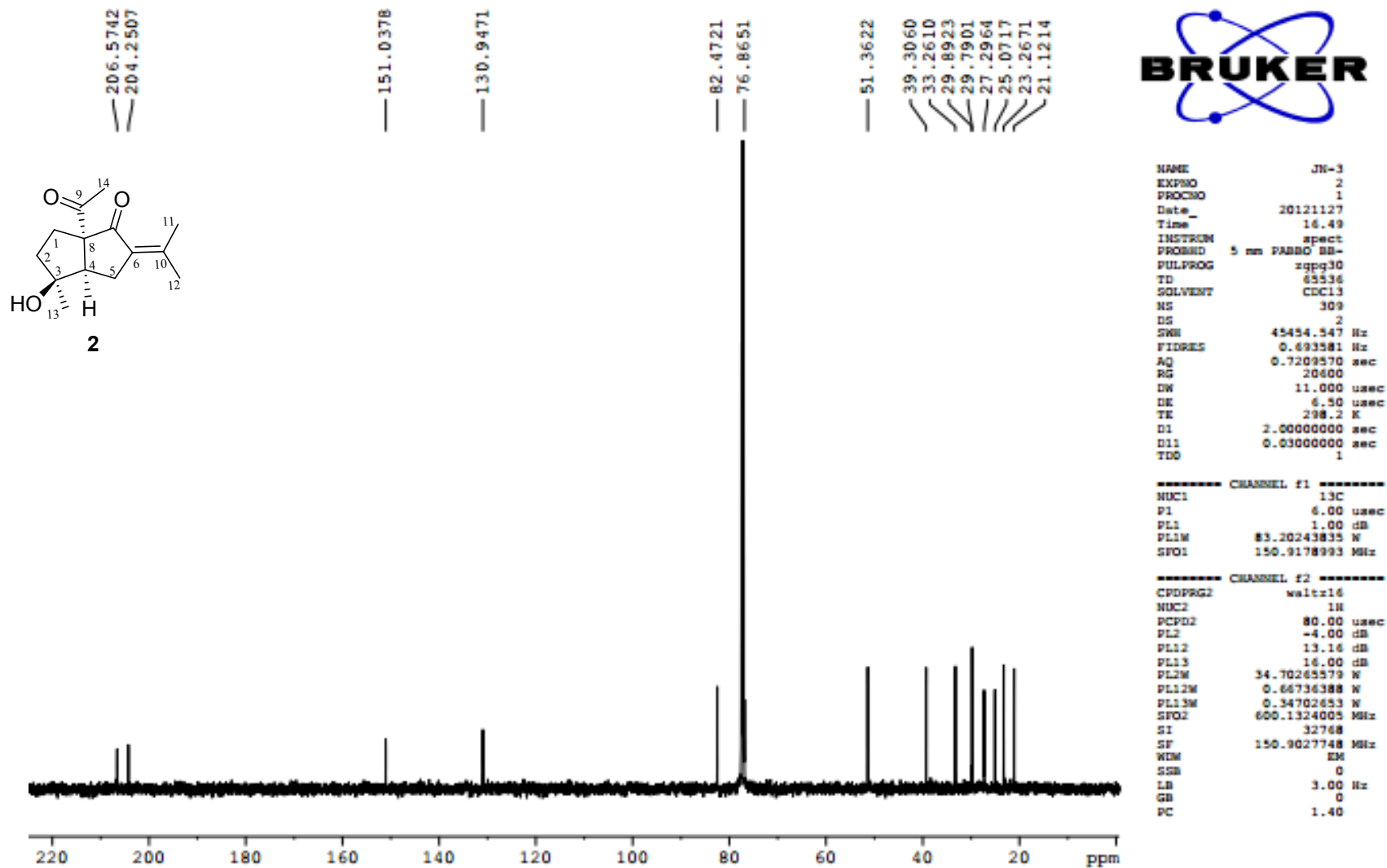
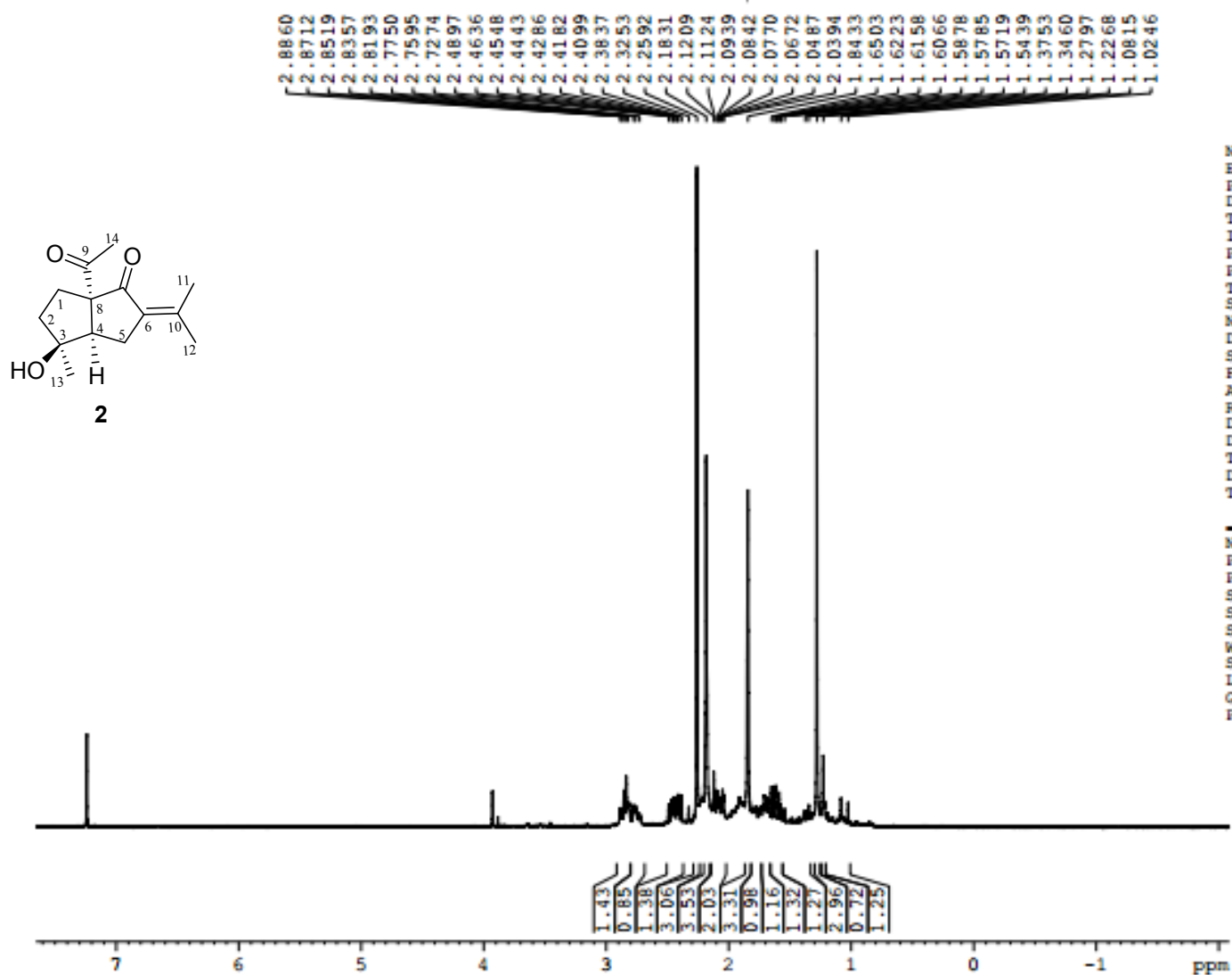


Figure S10. ¹H NMR spectra of phaeocaudione (2)

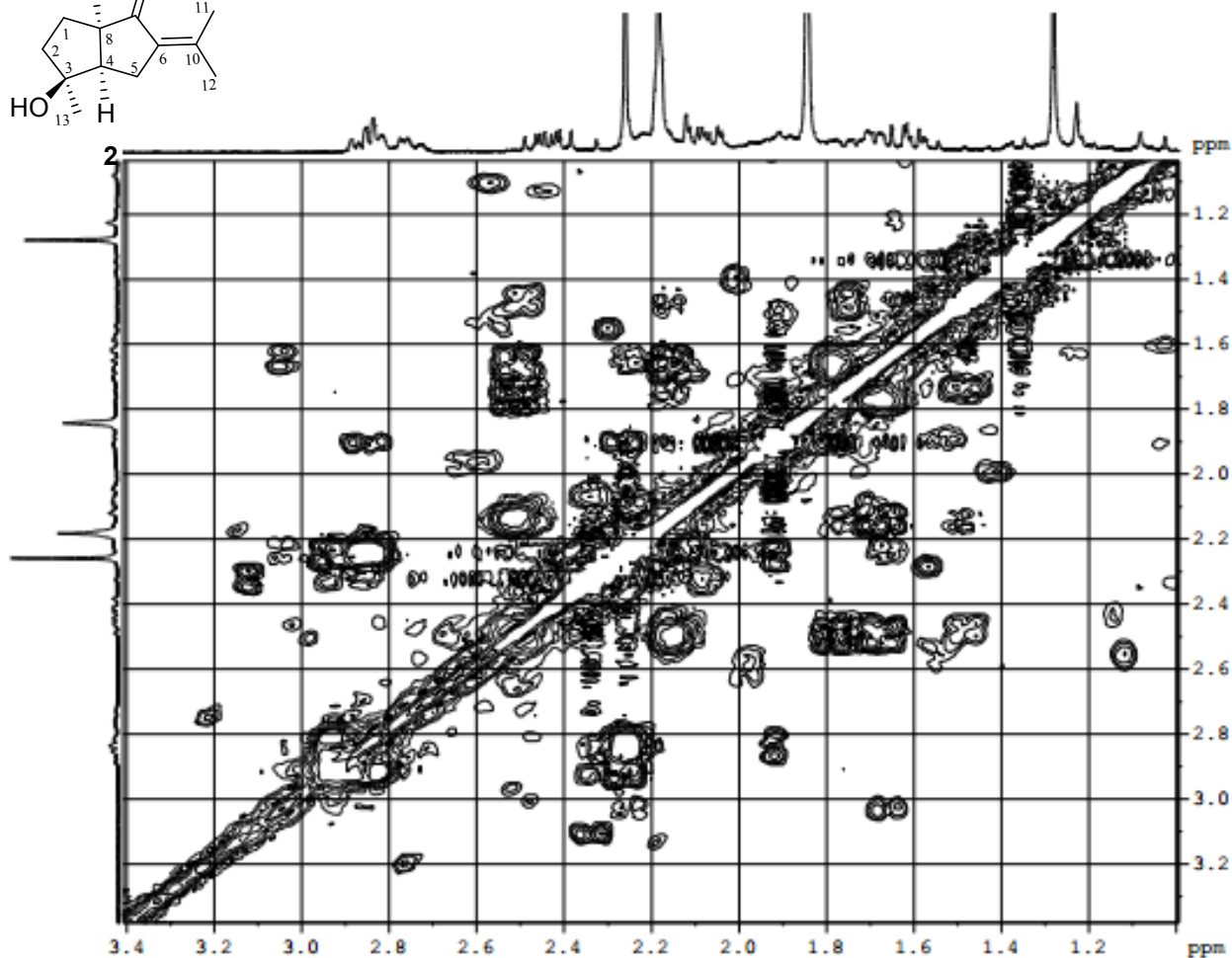
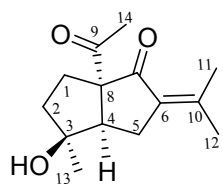


```

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PROCNO        1
Date_         20121126
Time          11.44
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            16
DS            2
SMH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG            812.7
DW            81.000 usec
DE            6.50 usec
TE            673.2 K
D1            1.00000000 sec
TDO           1

----- CHANNEL f1 -----
NUC1          1H
P1            10.80 usec
PL1           1.00 dB
SFO1          300.1318534 MHz
SI            32768
SF            300.1300114 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

Figure S11. ^1H - ^1H COSY spectra of phaeocaudione (2)



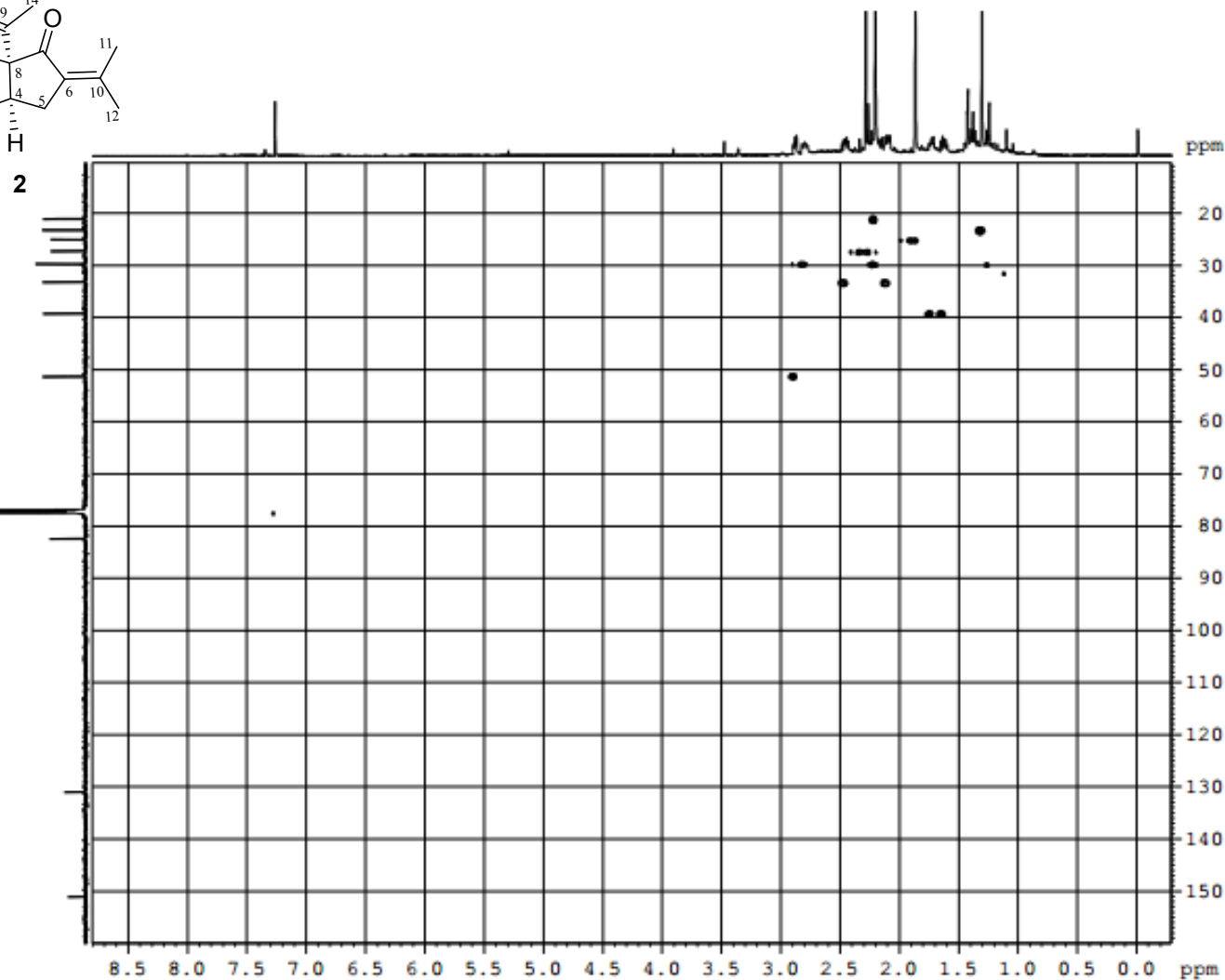
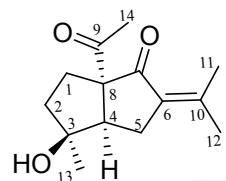
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PROCNO        1
Date_         20130425
Time          11.14
INSTRUM       spect
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PULPROG       coyygpgf
TD            1024
SOLVENT       CDCl3
NS            4
DS            8
SWH           6009.615 Hz
FIDRES        5.868765 Hz
AQ            0.0853300 sec
RG            80.6
DE            83.200 usec
TE            6.50 usec
TK            292.0 K
D0            0.00000300 sec
D1            2.00000000 sec
D13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00016640 sec

----- CHANNEL f1 -----
NUC1          1H
P0            11.10 usec
P1            11.10 usec
PL1           -4.00 dB
PL1W          34.70265579 W
SFO1          600.1324005 MHz

----- GRADIENT CHANNEL -----
GPNAM1        SINE.100
GPR1          16.00 %
P16           1000.00 usec
ND0           1
TD            256
SFO1          600.1324 MHz
FIDRES        23.475023 Hz
SW            10.014 ppm
PRMODE        QF
SI            1024
SF            600.1299740 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            600.1324005 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
```

Figure S12. HSQC spectra of phaeocaudione (2)



```

NAME          2n-2
EXPNO         2
PROCNO        1
Date_         20121207
Time          11.27
INSTRUM       spect
PROBHD        5 mm FRANK XH-
PULPROG       zgpg30
TD            16384
SOLVENT       cdcl3
NS            4
DS            16
SWH           6009.615 Hz
FIDRES       0.000745 Hz
AQ           0.0003300 sec
RG            14400
WDW           EM
SSB           0
GB            0
PC            83.300 usec
DE            6.50 usec
TE            298.2 K
CQ12         145.0000000
D0            0.0000000 sec
D1            1.5000000 sec
D2            0.0017244 sec
D3            0.0000000 sec
D4            0.0000000 sec
D5            0.0000000 sec
D6            0.0000000 sec
D7            0.0011000 sec
D8            0.0001950 sec
DECOUPLE2

----- CHANNEL f1 -----
NUC1          13C
P1            11.10 usec
P2            22.20 usec
PC1           1000.00 usec
PL1           -1.00 dB
PL12          24.70245579 N
SFO1          600.1324805 MHz

----- CHANNEL f2 -----
CPDPRG2      g3p
NUC2          13C
P3            8.60 usec
P4            17.40 usec
PCPD2         90.00 usec
PL2           1.00 dB
PL12          28.17 dB
PL12W        83.20243826 N
PL12W        1.00724721 N
SFO2          150.9154357 MHz

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OP1AM2       210K.100
OP11         90.00 %
OP12         28.10 %
PL4          1000.00 usec
SFO          2
TD            32K
SFO1         150.9154 MHz
FIDRES       150.217415 Hz
SW           170.500 ppm
PULPROG      Rho-ARR13ch
SI            1624
SF           600.1298971 MHz
WDW          g30
SSB           2
LA            0.00 Hz
GB            0
PC            1.40
SI            1624
MC2          echo
SF           15
  
```

Figure S13. HMBC spectra of phaeocaudione (2)

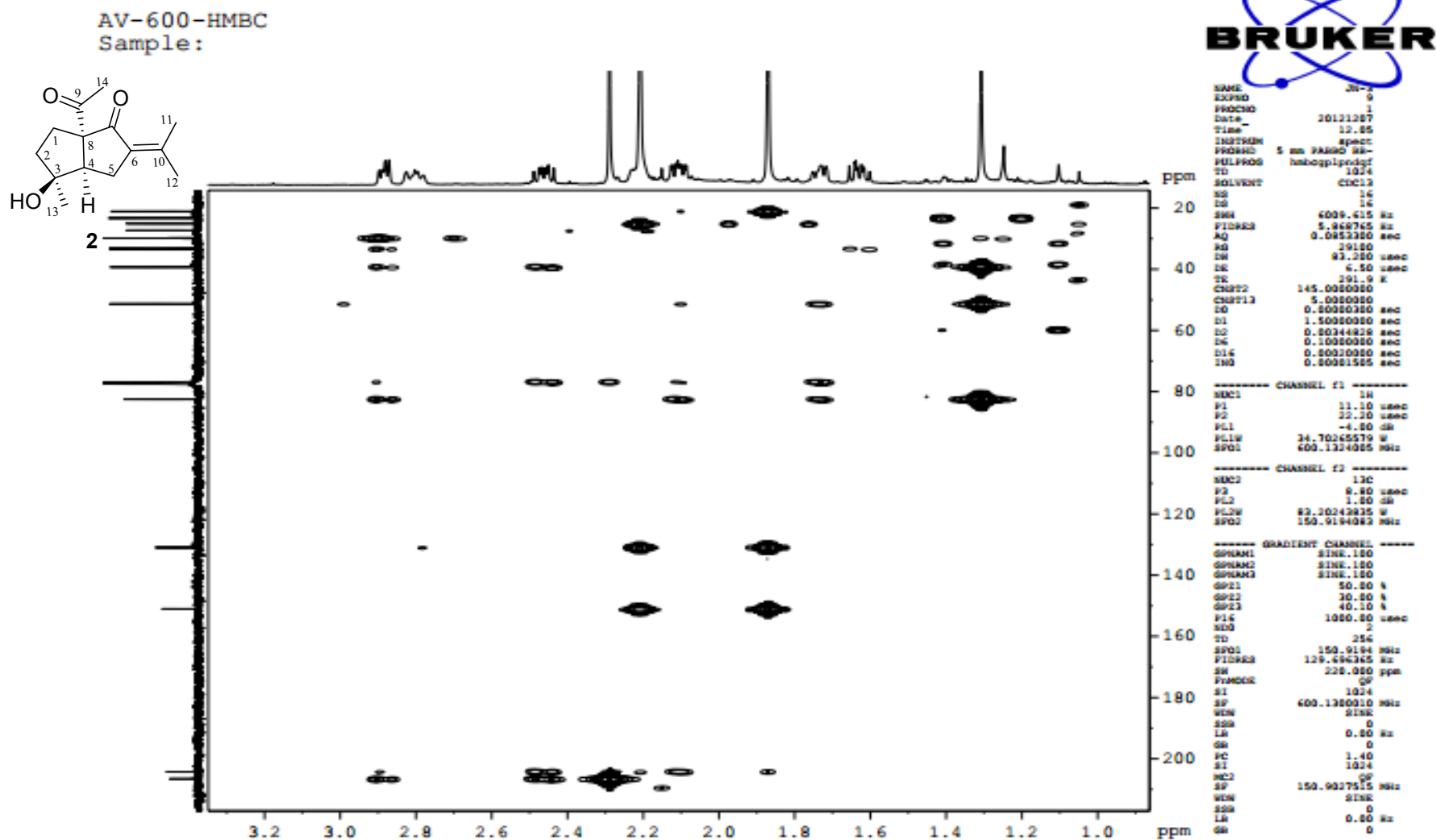
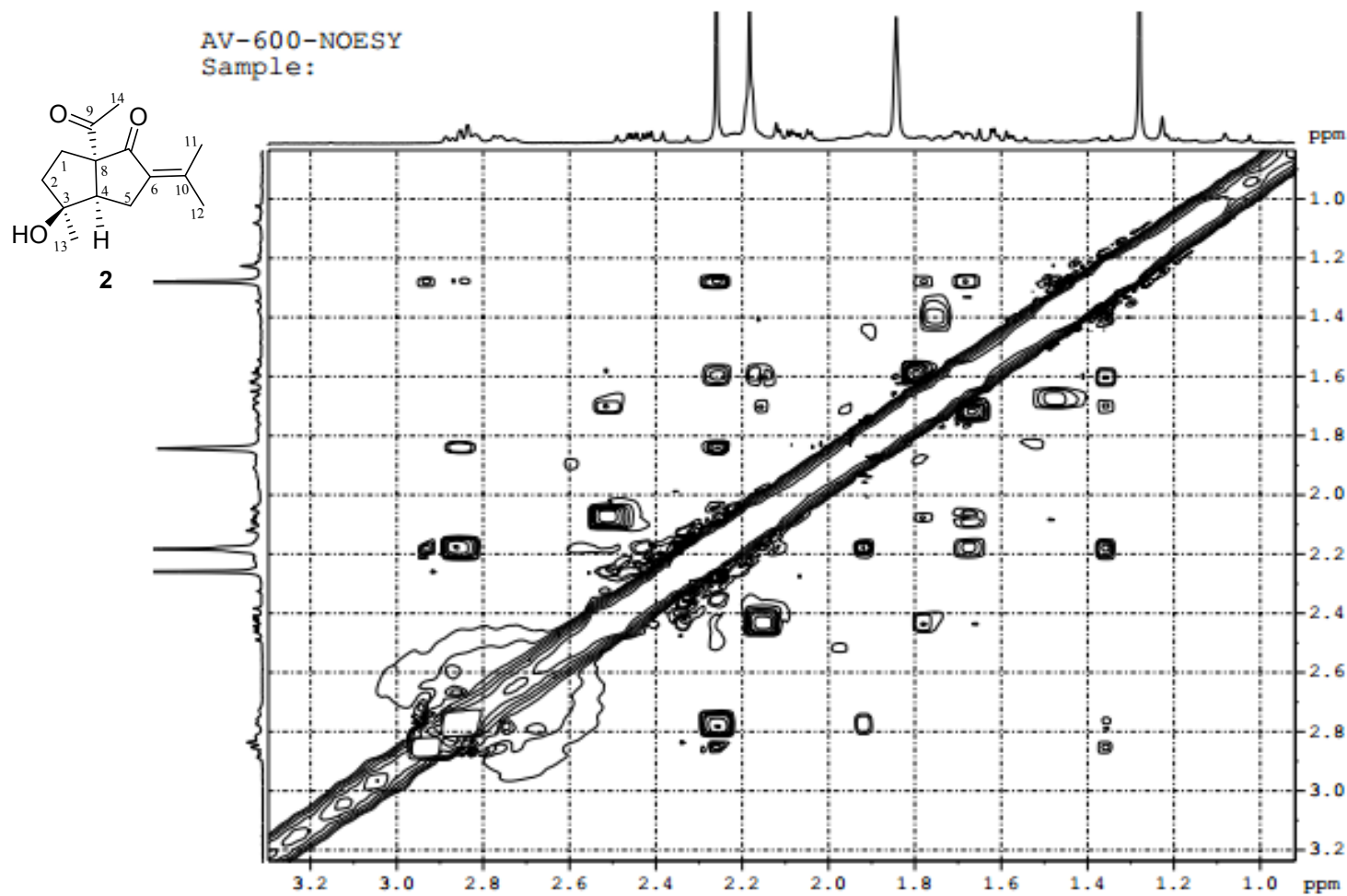


Figure S14. NOESY spectra of phaeocaudione (2)



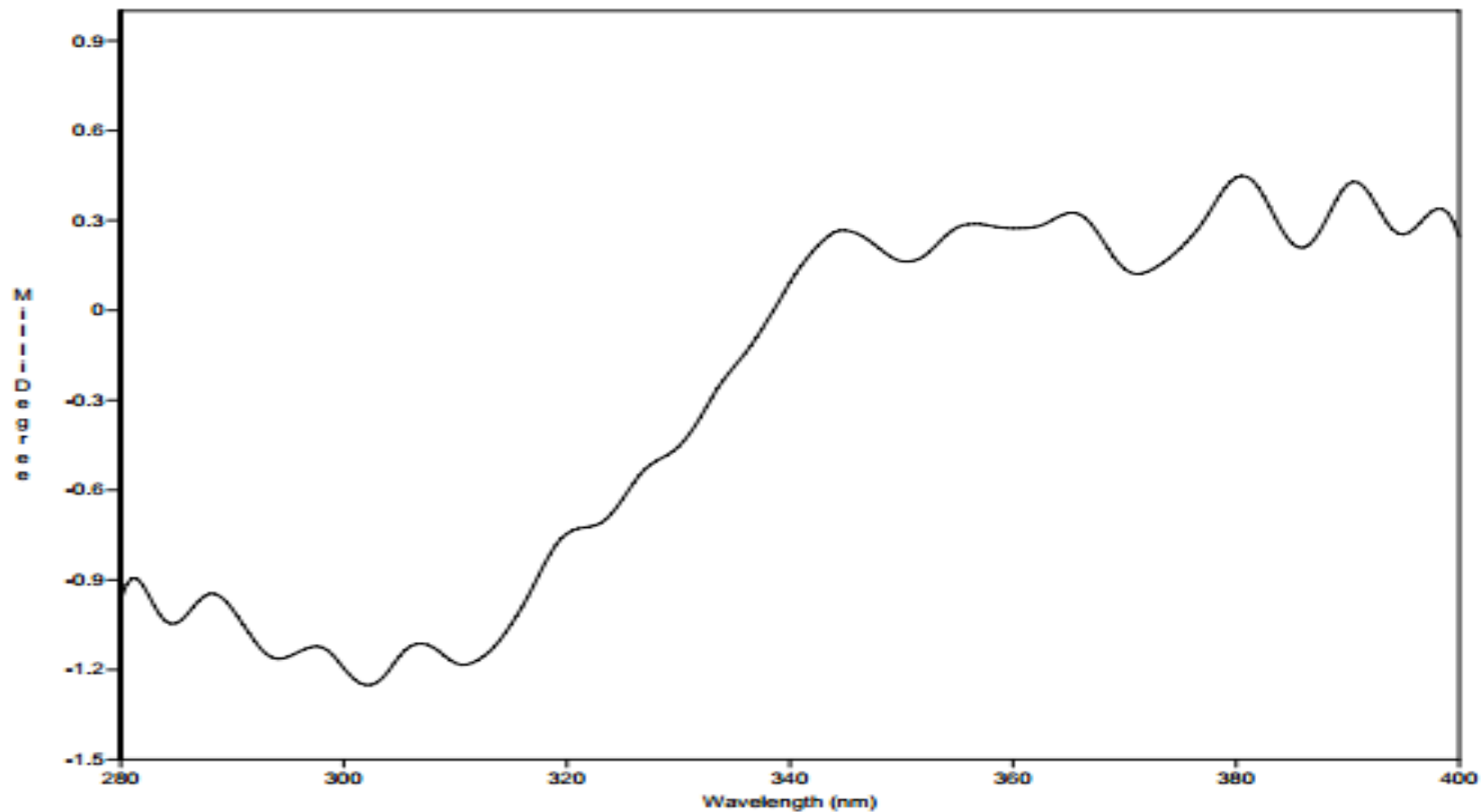
```

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EXPNO         6
PROCNO        1
Date_         20130426
Time         8.33
INSTRUM       spect
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PULPROG       noesyph
TD            1024
SOLVENT       CDCl3
NS            4
DS            4
SWH           6009.615 Hz
FIDRES        5.868765 Hz
AQ            0.0853300 sec
RG            64
DW            83.200 usec
DE            6.50 usec
TE            292.0 K
D0            0.00006907 sec
D1            2.00000000 sec
D8            0.60000002 sec
IN0           0.00016640 sec
    
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            11.10 usec
PL1           -4.00 dB
PL1W          34.70265579 W
SFO1          600.1324005 MHz
ND0           1
TD            256
SFO1          600.1324 MHz
FIDRES        23.475023 Hz
SW            10.014 ppm
FnMODE        States-TPPI
SI            1024
SF            600.1299722 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.00
SI            1024
MC2           States-TPPI
SF            600.1300197 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
    
```


Figure S15. Rh₂(OCOCF₃)₄-induced CD spectra of phaeocaudione (2)



Bio-Kine Software V4.71 Date : 2014-12-21 Time : 11:29:11

COMMENTS :

File name : sav-golay
Savitzky-Golay Smooth of sav-golay
Window Points=15
Polynomial Order=3
Derivative=0

Figure S16. HRESIMS spectra of phaeocaudione (2)

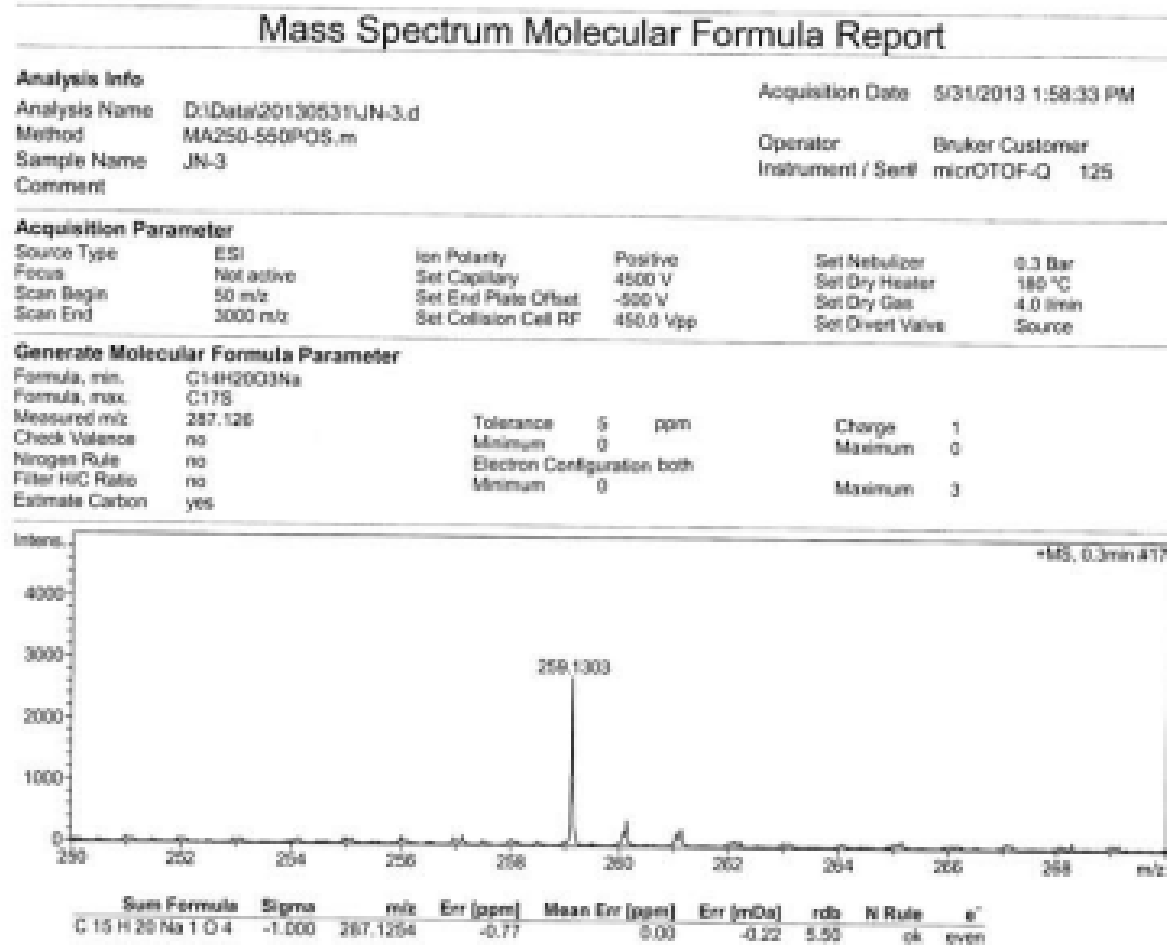


Figure S17. ¹³C NMR spectra of phaeocaoune (3)

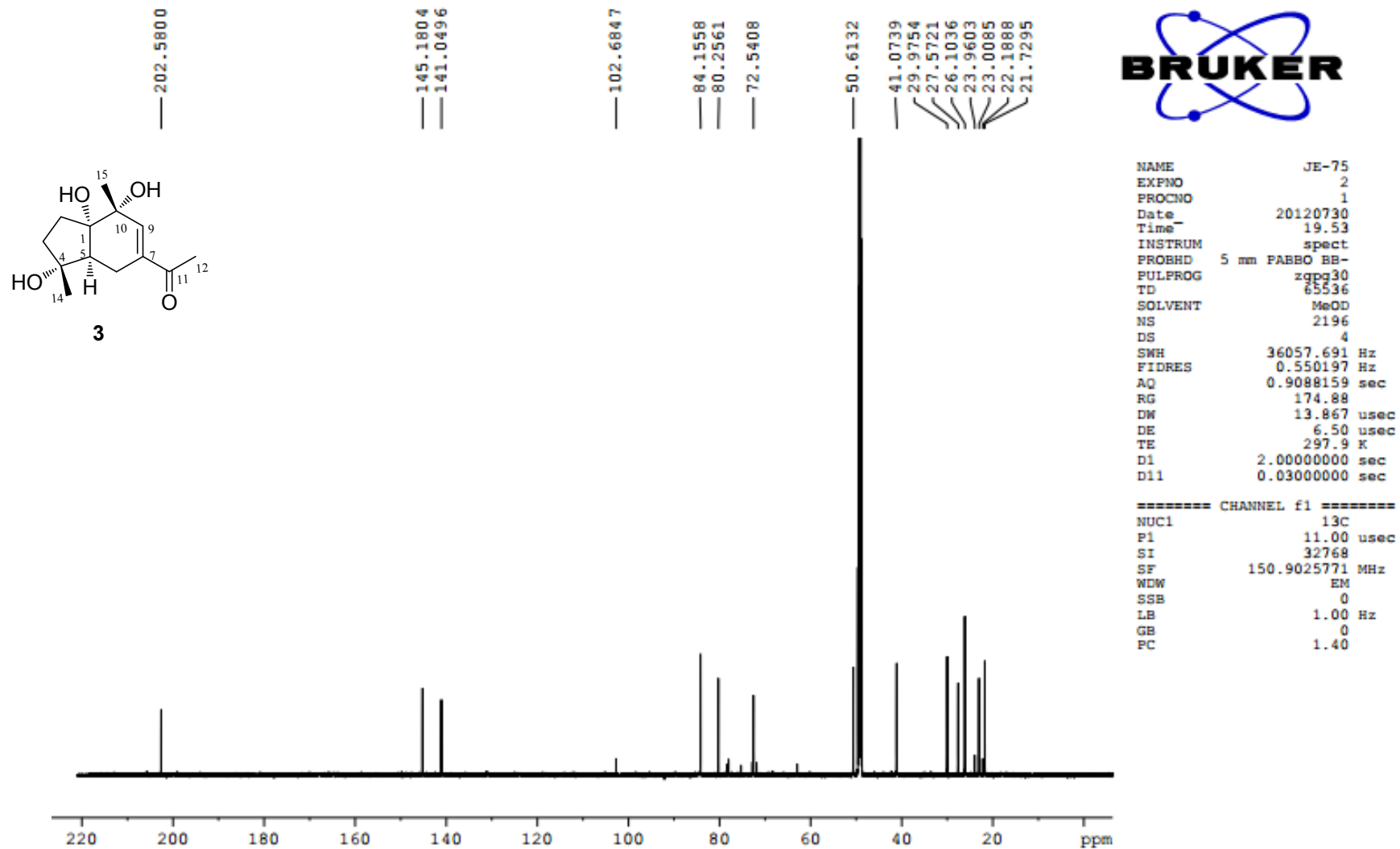


Figure S18. ¹H NMR spectra of phaeocaoune (3)

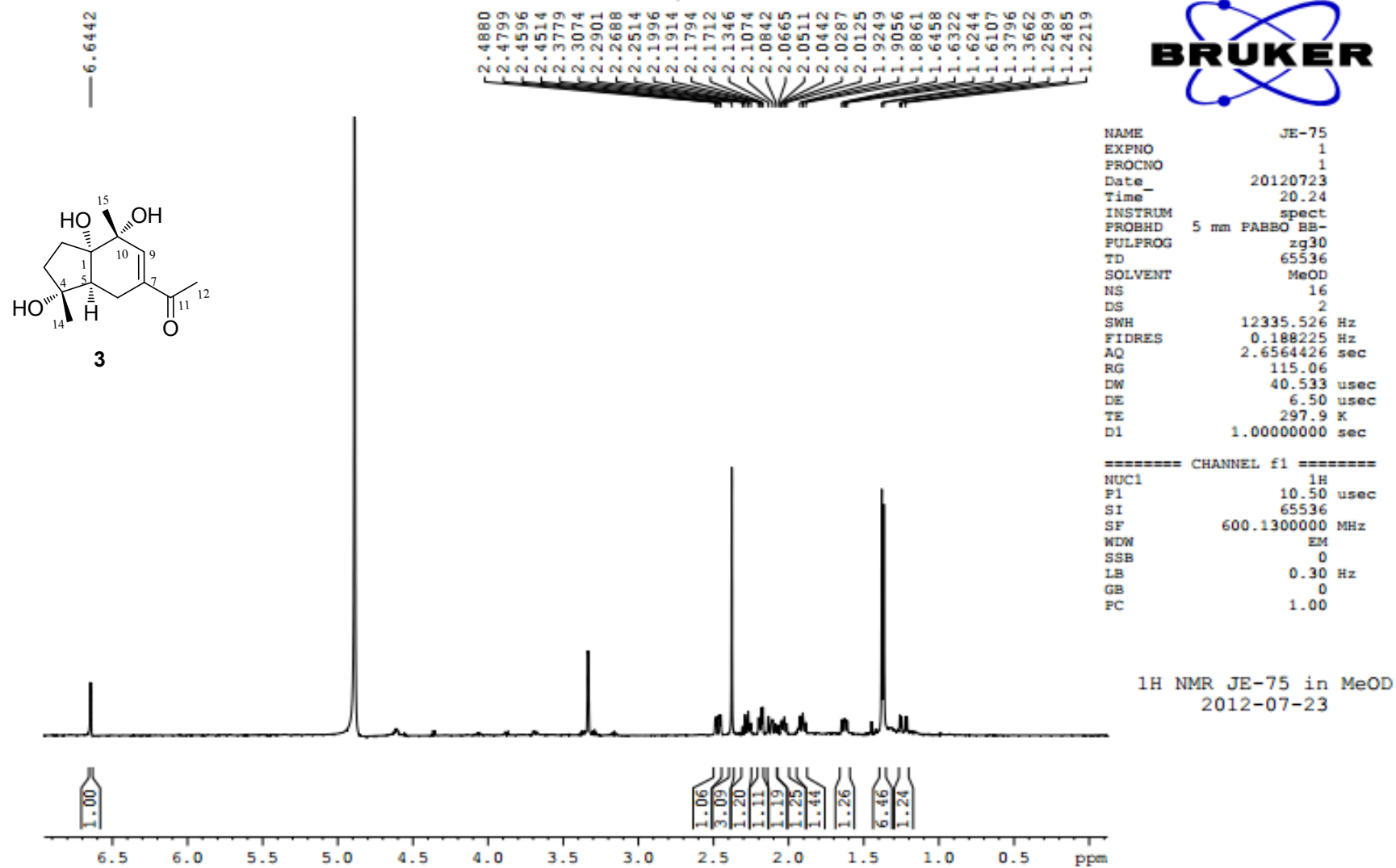
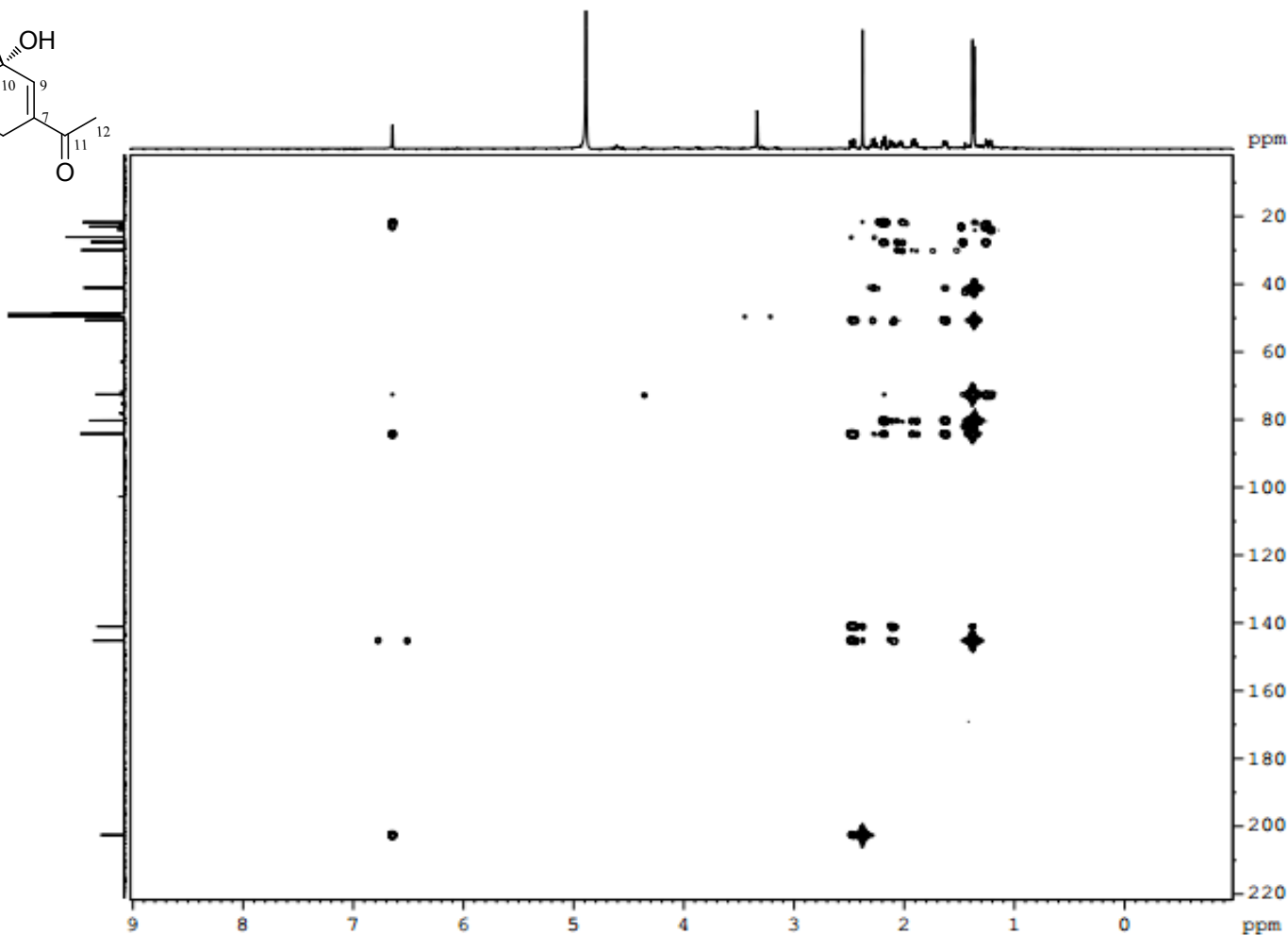
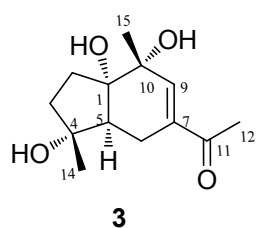


Figure S20. HMBC spectra of phaeocauone (3)



```

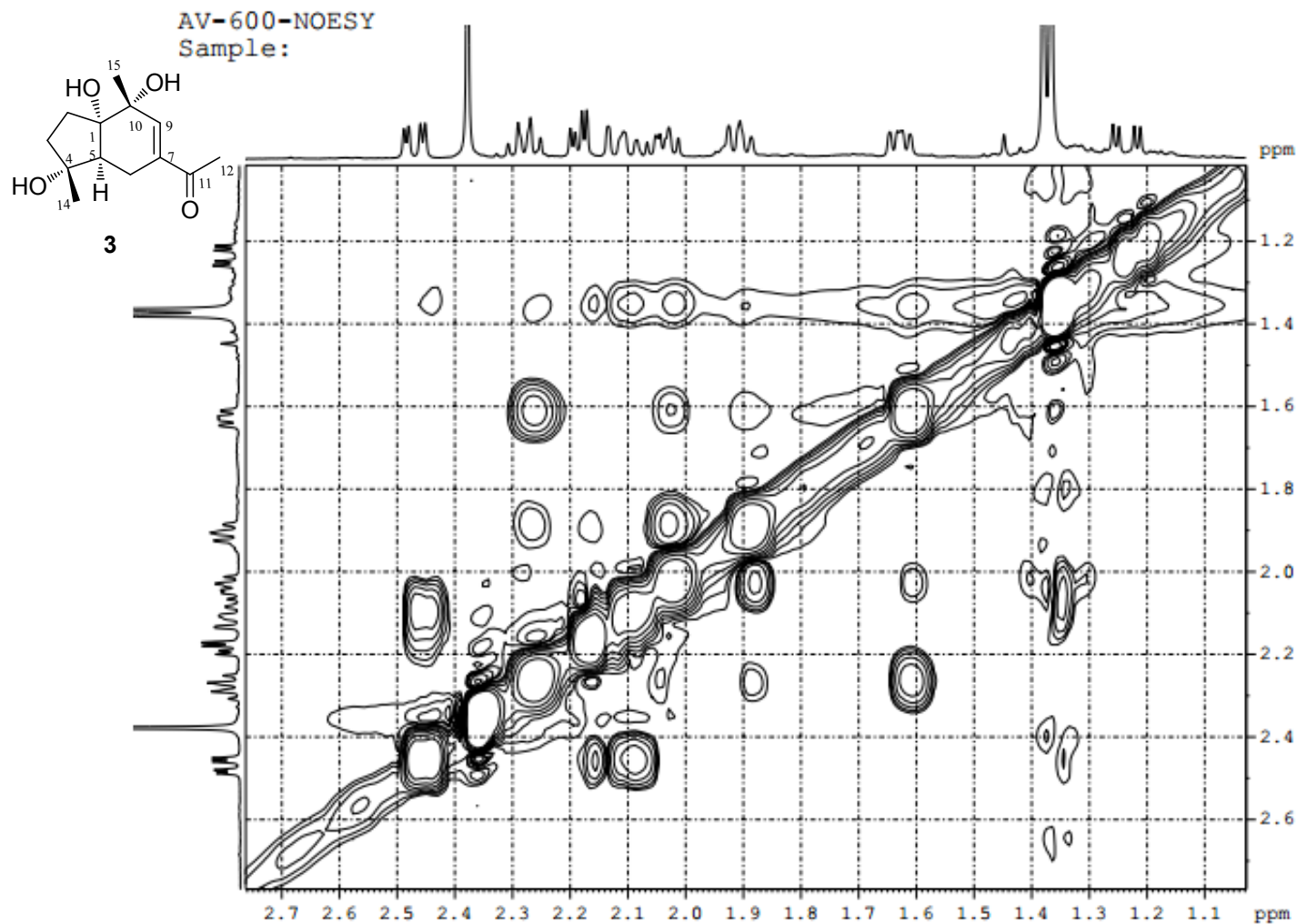
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EXPNO         9
PROCNO        1
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Time          10.09
INSTRUM       spect
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PULPROG       hbhcgplpndqf
TD            1024
SOLVENT       MeOD
NS            12
DS            16
SWH           6009.615 Hz
FIDRES        5.868765 Hz
AQ            0.0853300 sec
RG            29100
DW            83.200 usec
DE            6.90 usec
TE            296.1 K
CHST2         145.0000000
CHST13        5.0000000
DO            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.10000000 sec
D16           0.00020000 sec
IN0           0.00001505 sec

----- CHANNEL f1 -----
NUC1          13C
P1            11.10 usec
P2            22.20 usec
PL1           -4.00 dB
PL1W          34.70265579 W
SFO1          600.1324005 MHz

----- CHANNEL f2 -----
NUC2          13C
P3            8.80 usec
PL2           1.00 dB
PL2W          83.20243835 W
SFO2          150.9194083 MHz

----- GRADIENT CHANNEL -----
GPMAM1        SINE.100
GPMAM2        SINE.100
GPMAM3        SINE.100
GPE1          50.00 %
GPE2          30.00 %
GPE3          40.10 %
PI6           1000.00 usec
NDD           2
TD            256
SFO1          150.9194 MHz
FIDRES        129.696365 Hz
SH            220.000 ppm
F2MODE        QF
SI            1024
SF            600.1299914 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            150.9025562 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
```

Figure S21. NOESY spectra of phaeocaoune (3)



```

NAME          JE-75
EXPNO         6
PROCNO        1
Date_         20130521
Time_         8.56
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       noesyph
TD            1024
SOLVENT       MeOD
NS            4
DS            4
SMH           6009.615 Hz
FIDRES        5.868765 Hz
AQ            0.0853300 sec
RG            322
DW            83.200 usec
DE            6.50 usec
TE            296.3 K
D0            0.00006907 sec
D1            2.00000000 sec
D8            0.60000002 sec
INO            0.00016640 sec
    
```

```

***** CHANNEL f1 *****
NUC1           1H
P1             11.10 usec
PL1            -4.00 dB
PL1W           34.70265579 W
SFO1           600.1324000 MHz
NDO            1
TD             256
SFO1           600.1324 MHz
FIDRES         23.475023 Hz
SW             10.014 ppm
FnMODE         States-TPPI
SI             1024
SF             600.1300000 MHz
WDW            QSINE
SSB            2
LB             0.00 Hz
GB             0
PC             1.00
SI             1024
MC2            States-TPPI
SF             600.1300000 MHz
WDW            QSINE
SSB            2
LB             0.00 Hz
GB             0
    
```

Figure S22. HRESIMS spectra of phaeocauone (3)

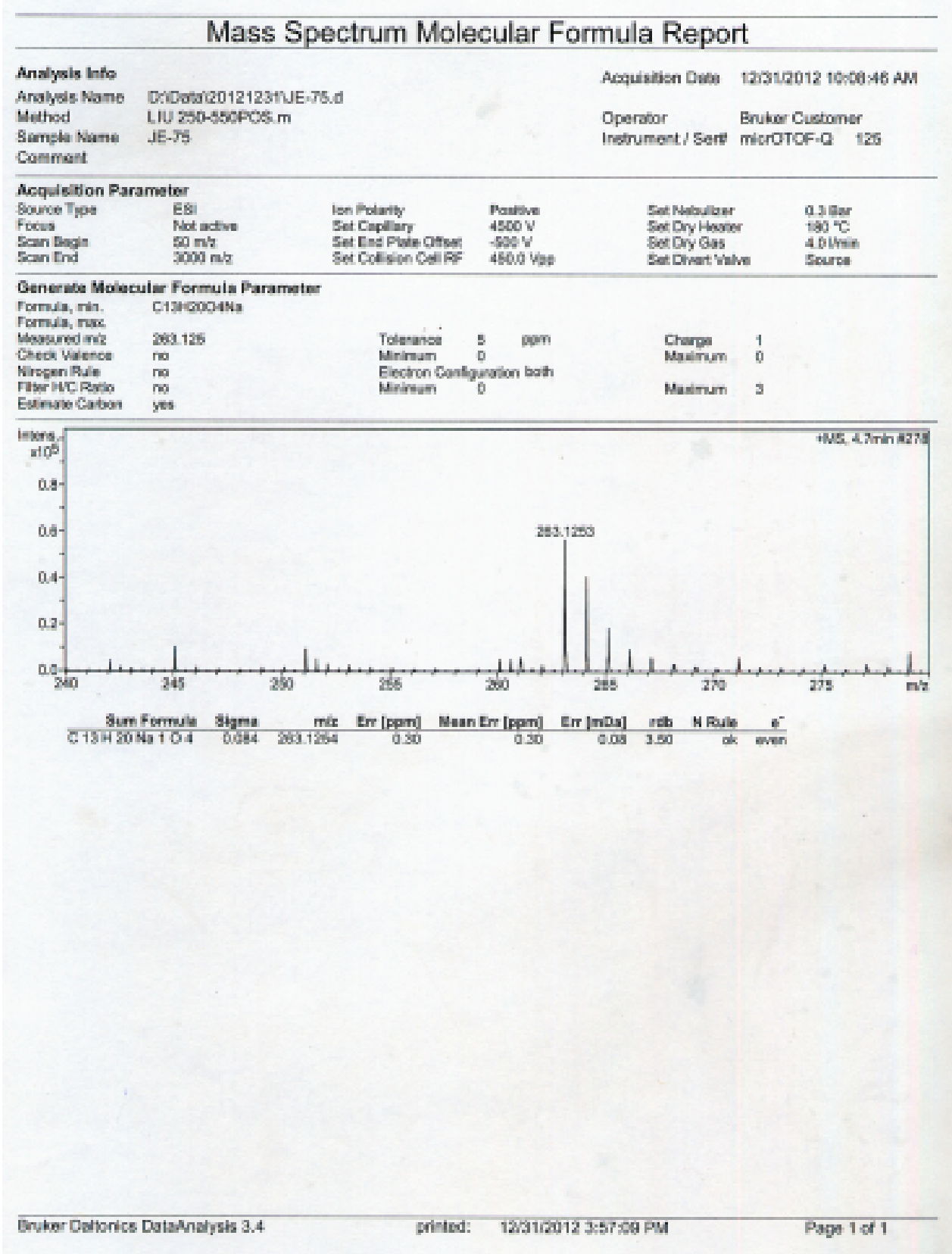
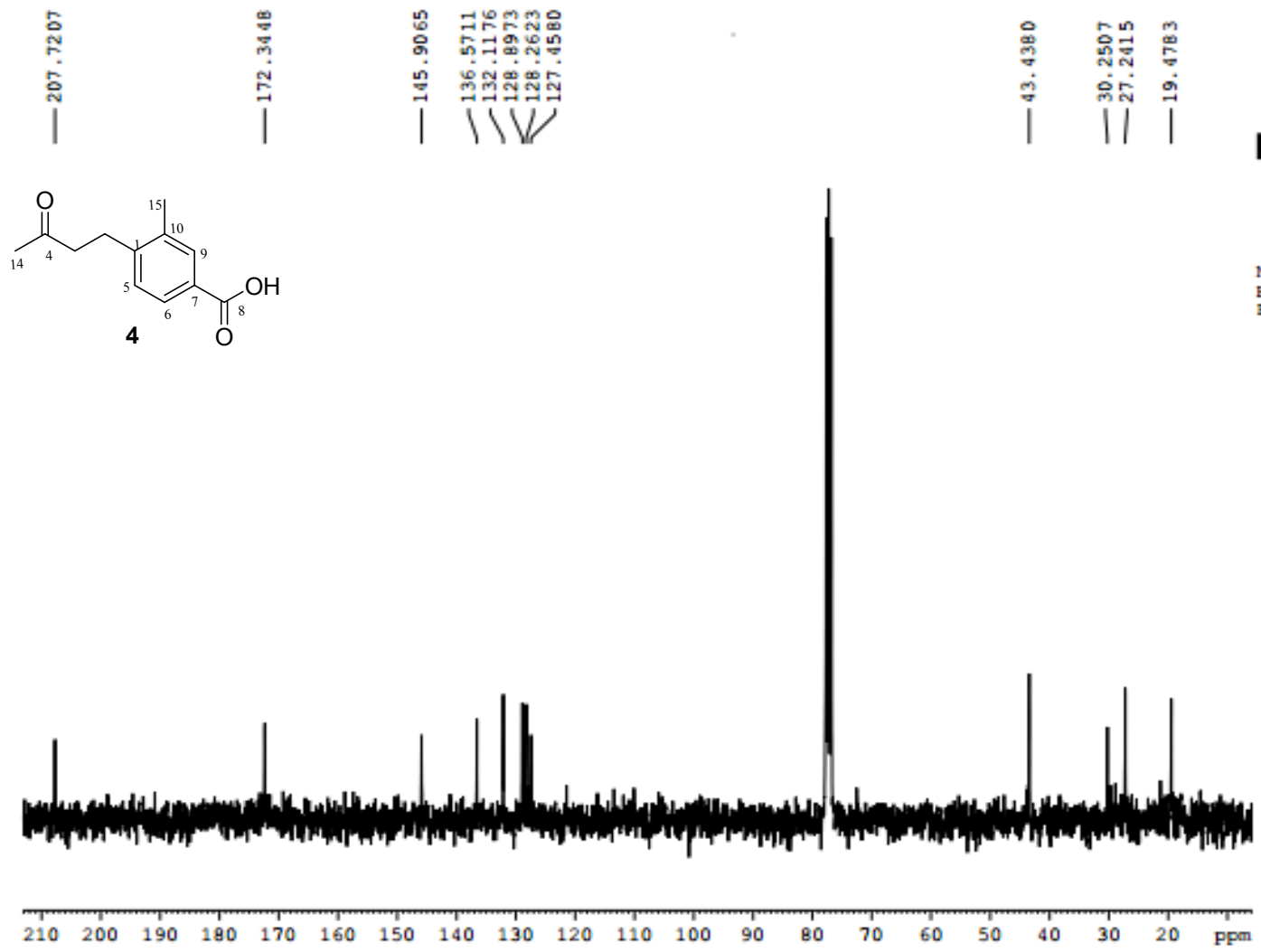
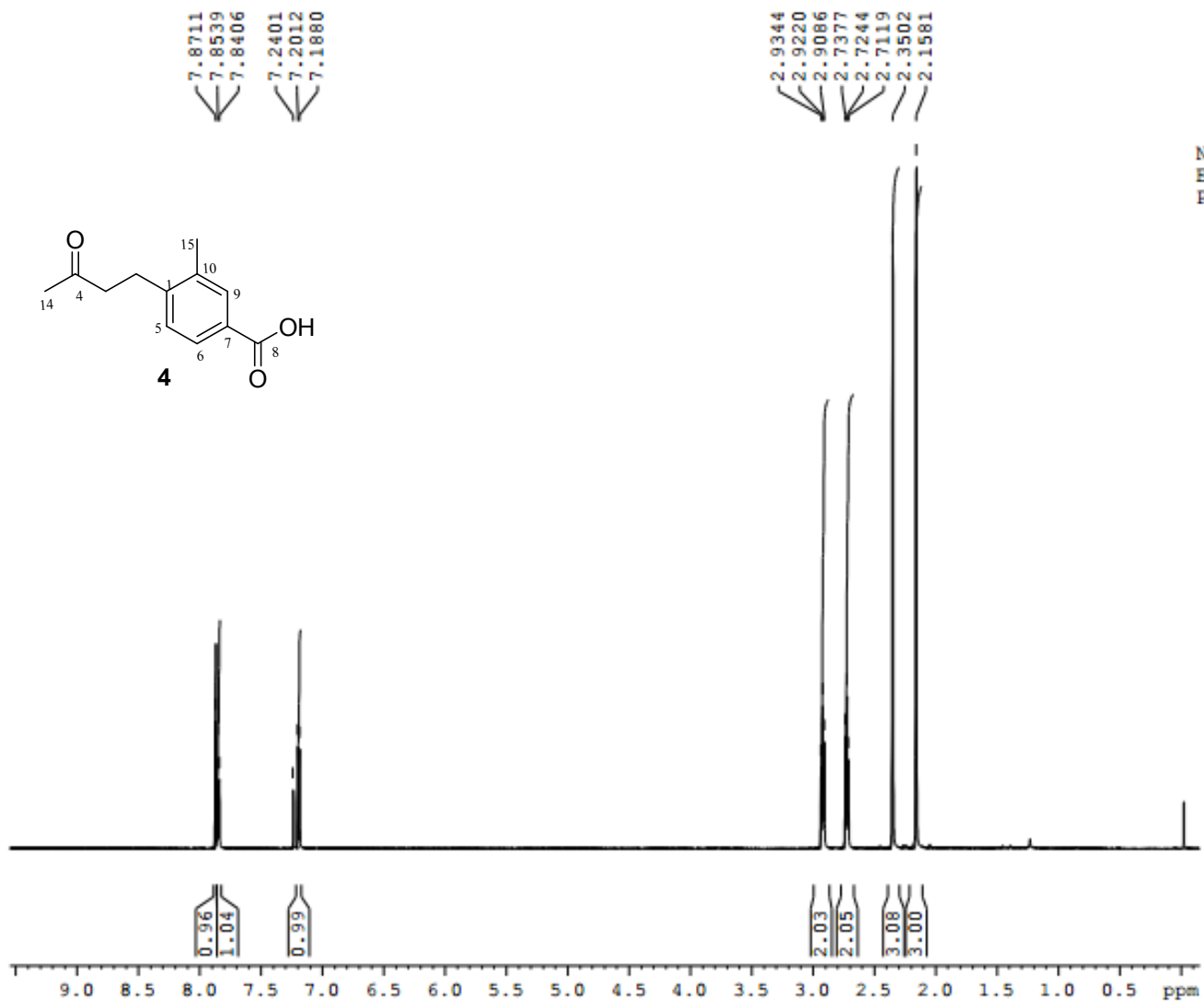


Figure S23. ¹³C NMR spectra of 3-methyl-4-(3-oxobutyl)-benzoic acid (**4**)



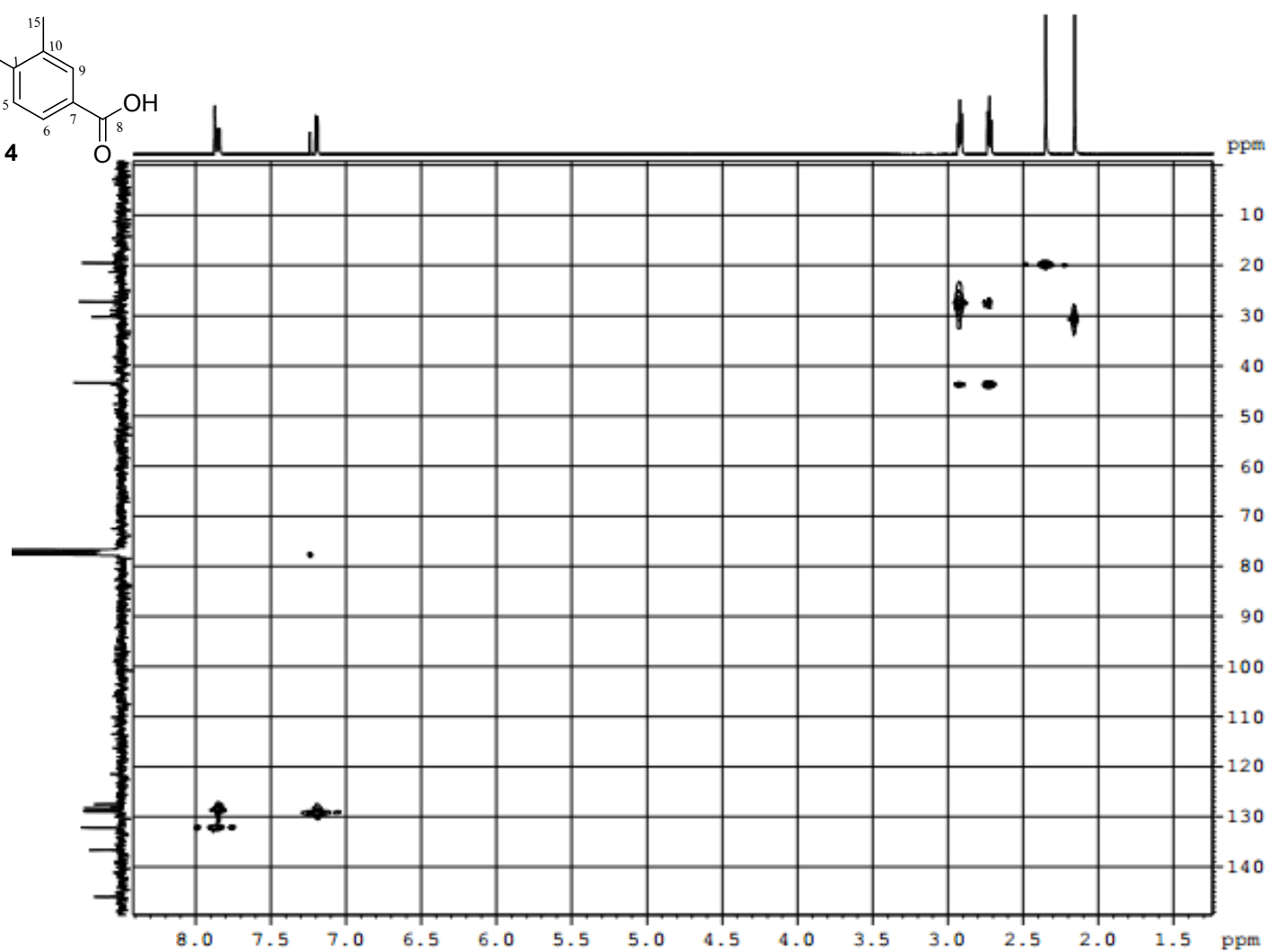
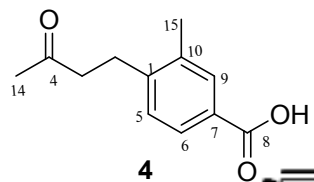
NAME Fr1.10-11J
EXPNO 2
PROCNO 1

Figure S24. ¹H NMR spectra of 3-methyl-4-(3-oxobutyl)-benzoic acid (4)



NAME Fr1.10-11J
EXPNO 1
PROCNO 1

Figure S25. HSQC spectra of 3-methyl-4-(3-oxobutyl)-benzoic acid (4)



NAME
EXPNO 8
PROCNO 1

F=1.10-11J

Figure S26. HMBC spectra of 3-methyl-4-(3-oxobutyl)-benzoic acid (4)



NAME Fri.10-11J
EXPNO 9
PROCNO 1

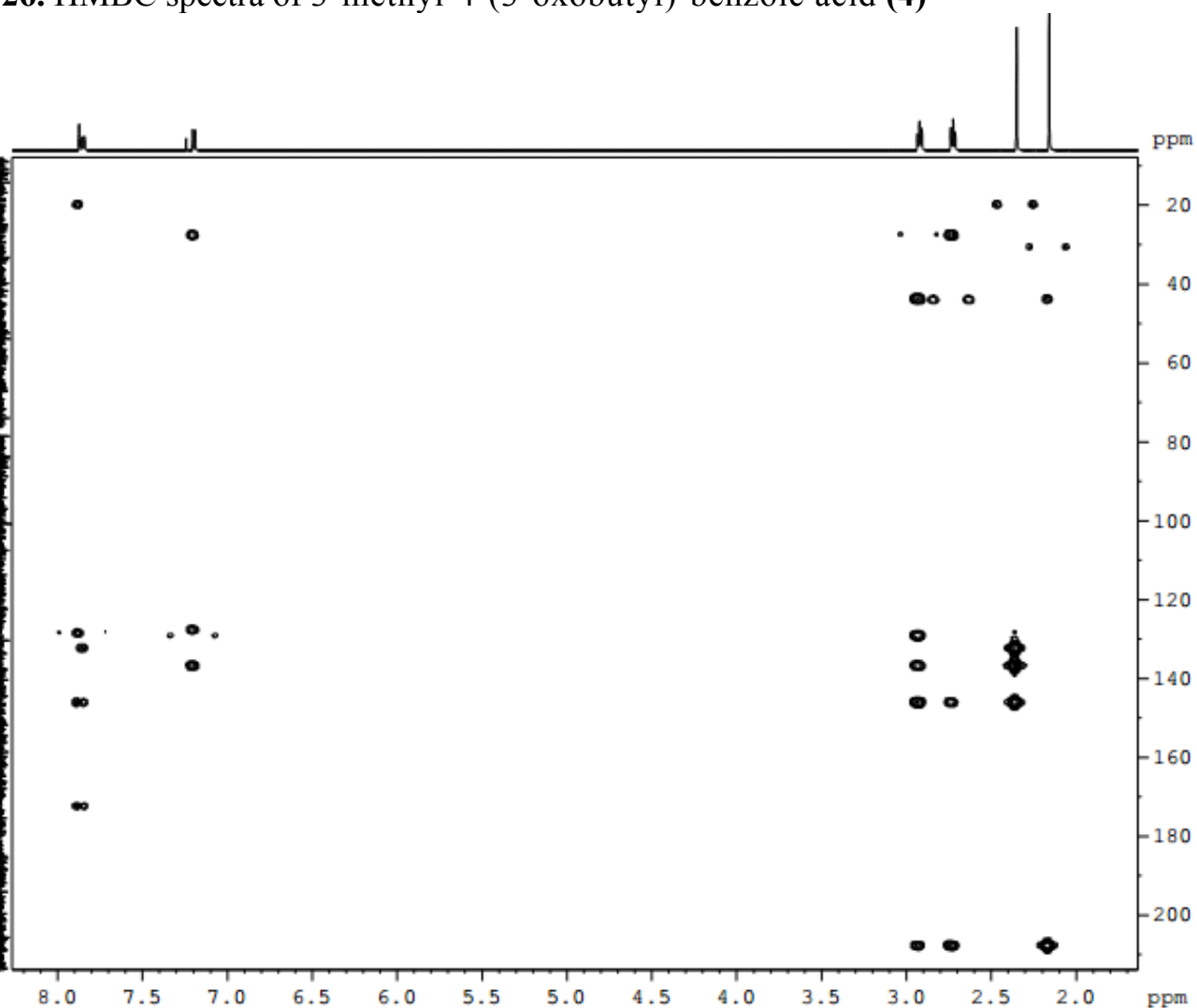
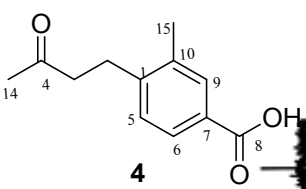


Figure S27. HRESIMS spectra of 3-methyl-4-(3-oxobutyl)-benzoic acid (4)

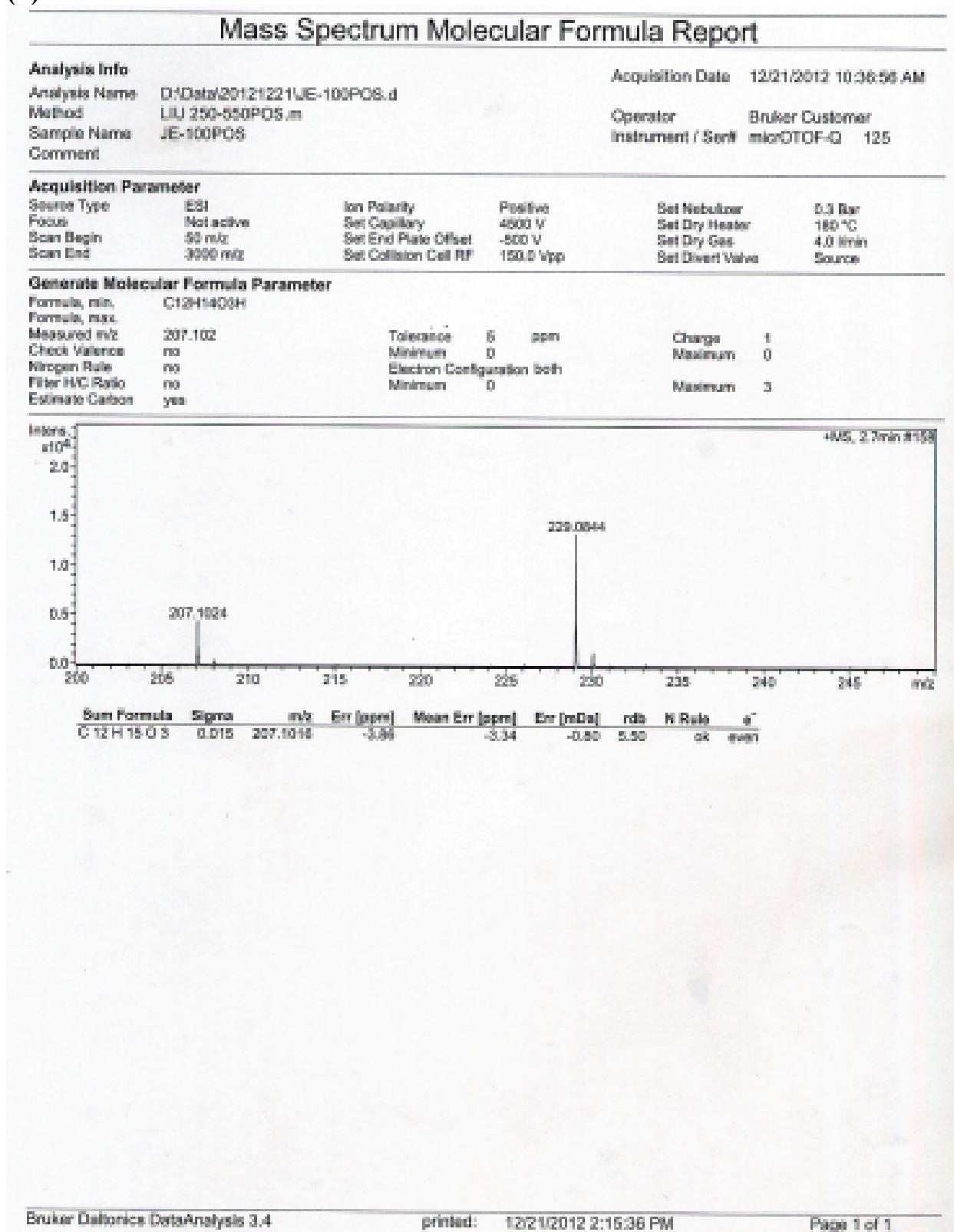


Figure S28. ^{13}C NMR spectra of $8\beta(\text{H})$ -elema-1,3,7(11)-trien-8,12-lactam (**5**)

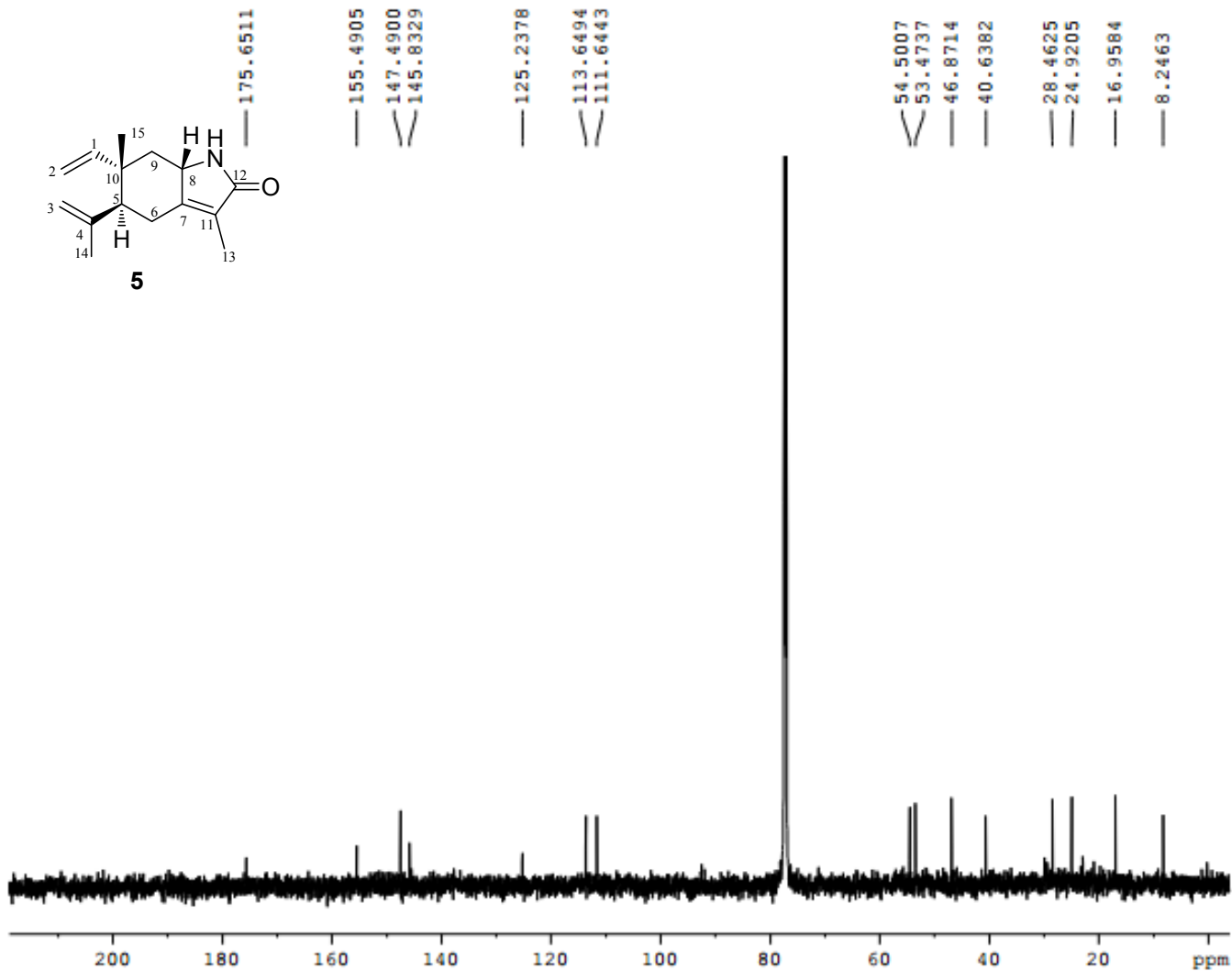


Figure S29. ^1H NMR spectra of $8\beta(\text{H})$ -elema-1,3,7(11)-trien-8,12-lactam (**5**)

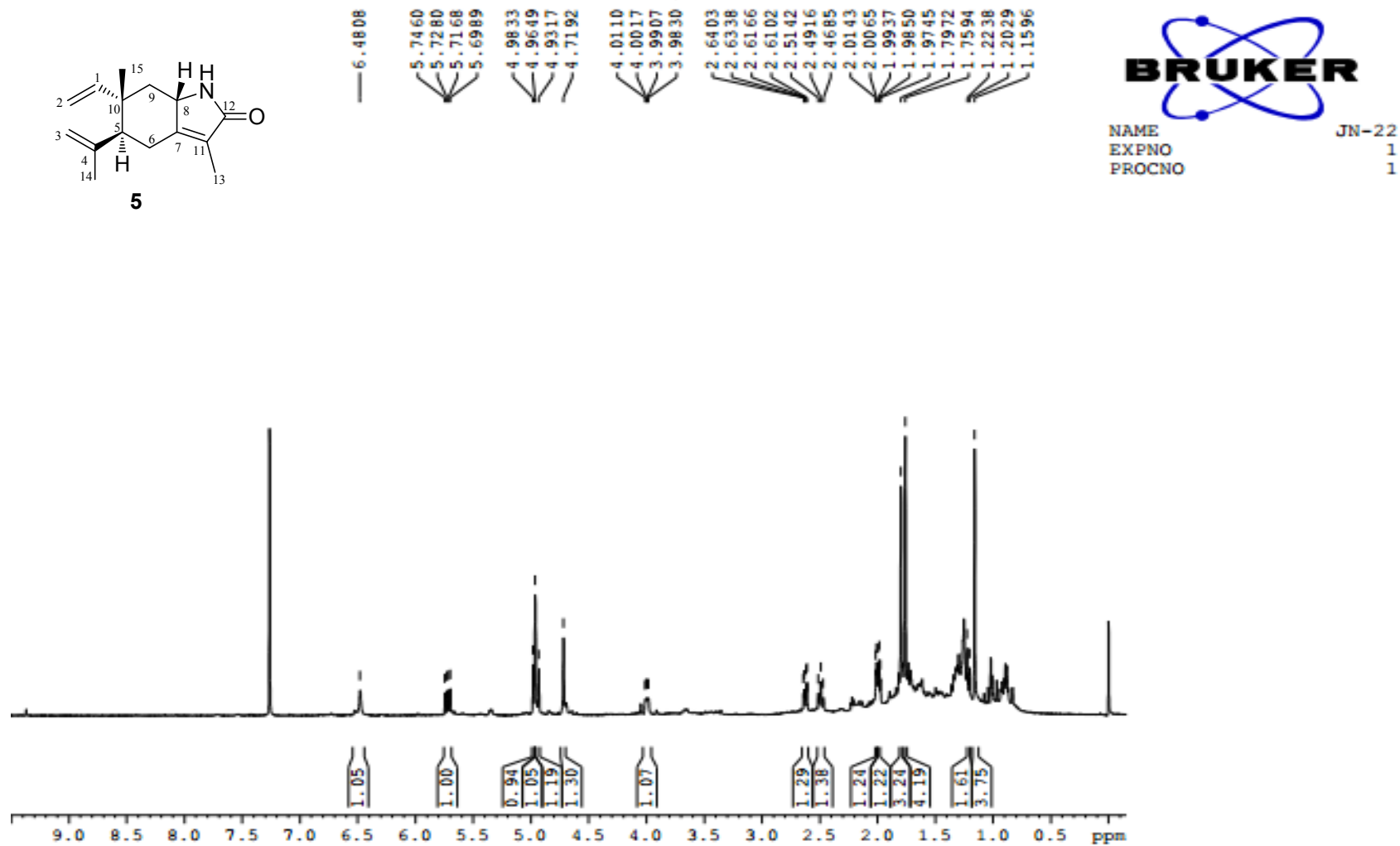
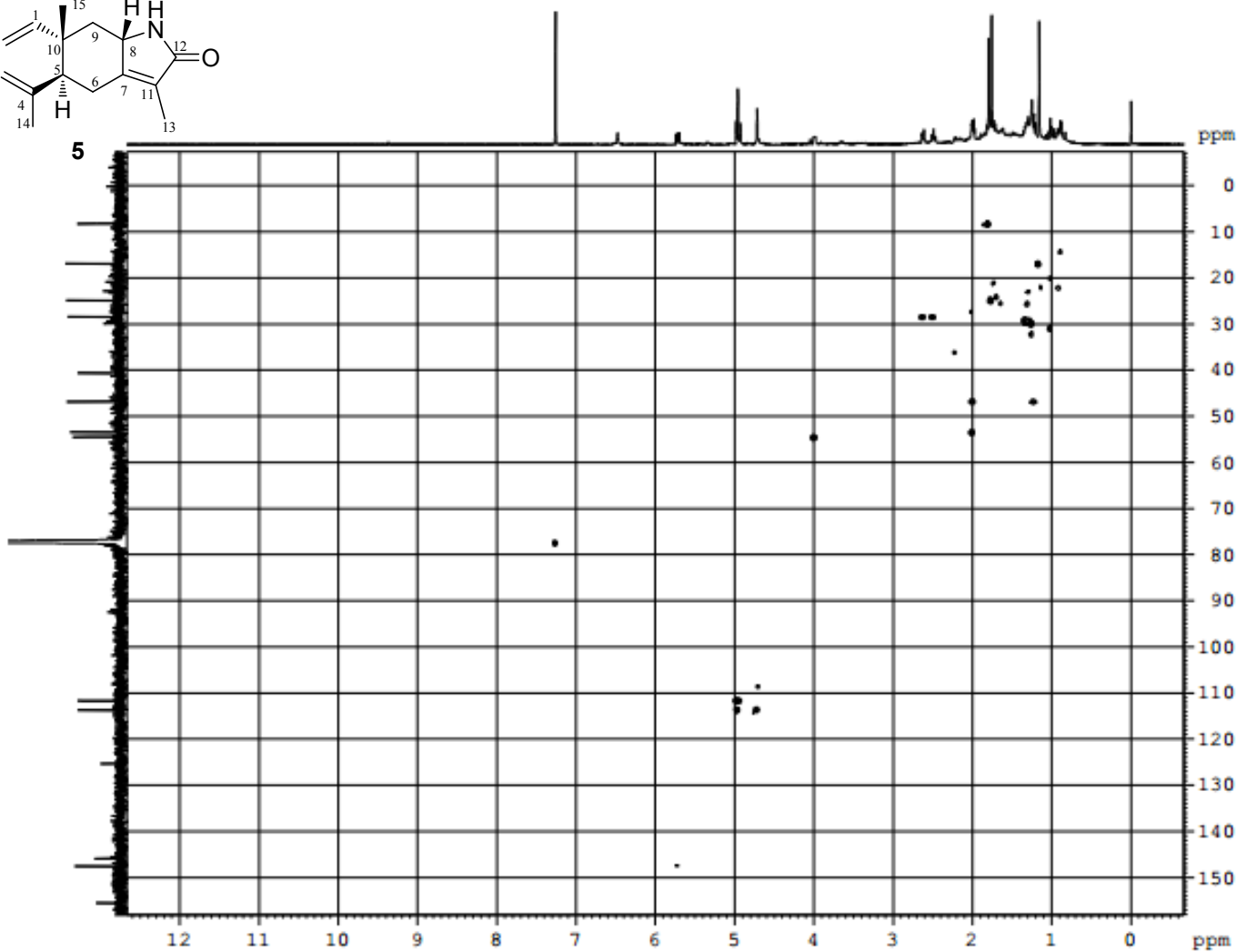
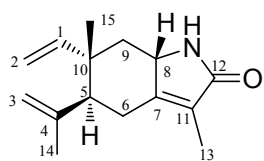


Figure S30. HSQC spectra of 8β (H)-elema-1,3,7(11)-trien-8,12-lactam (**5**)



```

NAME          JN-22
EXPNO         618
PROCNO        1
DATE_         20130820
Time_         14.25
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            1634
SOLVENT       CDCl3
SS            8
DS            16
SFO1          401.250 MHz
FIDRES        7.81950 Hz
AQ            0.0619476 sec
RG            174.68
DSW           62.400 usec
DE            6.50 usec
TE            298.2 K
CHST2         145.000000
D0            0.0000300 sec
D1            1.5000000 sec
D4            0.00172414 sec
D11           0.0200000 sec
D13           0.0000400 sec
D14           0.0020000 sec
D16           0.0006207 sec
D19           0.0002000 sec
ENDPT00

----- CHANNEL f1 -----
NUC1          13
P1            12.65 usec
P2            25.70 usec
P39           1000.00 usec
RG            2
TD            654
SFO1          150.9141 MHz
FIDRES        97.652046 Hz
SW            165.650 ppm
PRGMRG        Echo-antiecho
SI            1624
SF            400.1380112 MHz
WDW           GQ1MC
SSB           2
LA            0.00 Hz
GB            0
PC            1.40
SI            1624
ME2           echo-antiecho
SF            150.9027636 MHz
WDW           GQ1MC
SSB           2
LA            0.00 Hz
GB            0
    
```


Figure S31. HMBC spectra of 8 β (H)-elema-1,3,7(11)-trien-8,12-lactam (5)

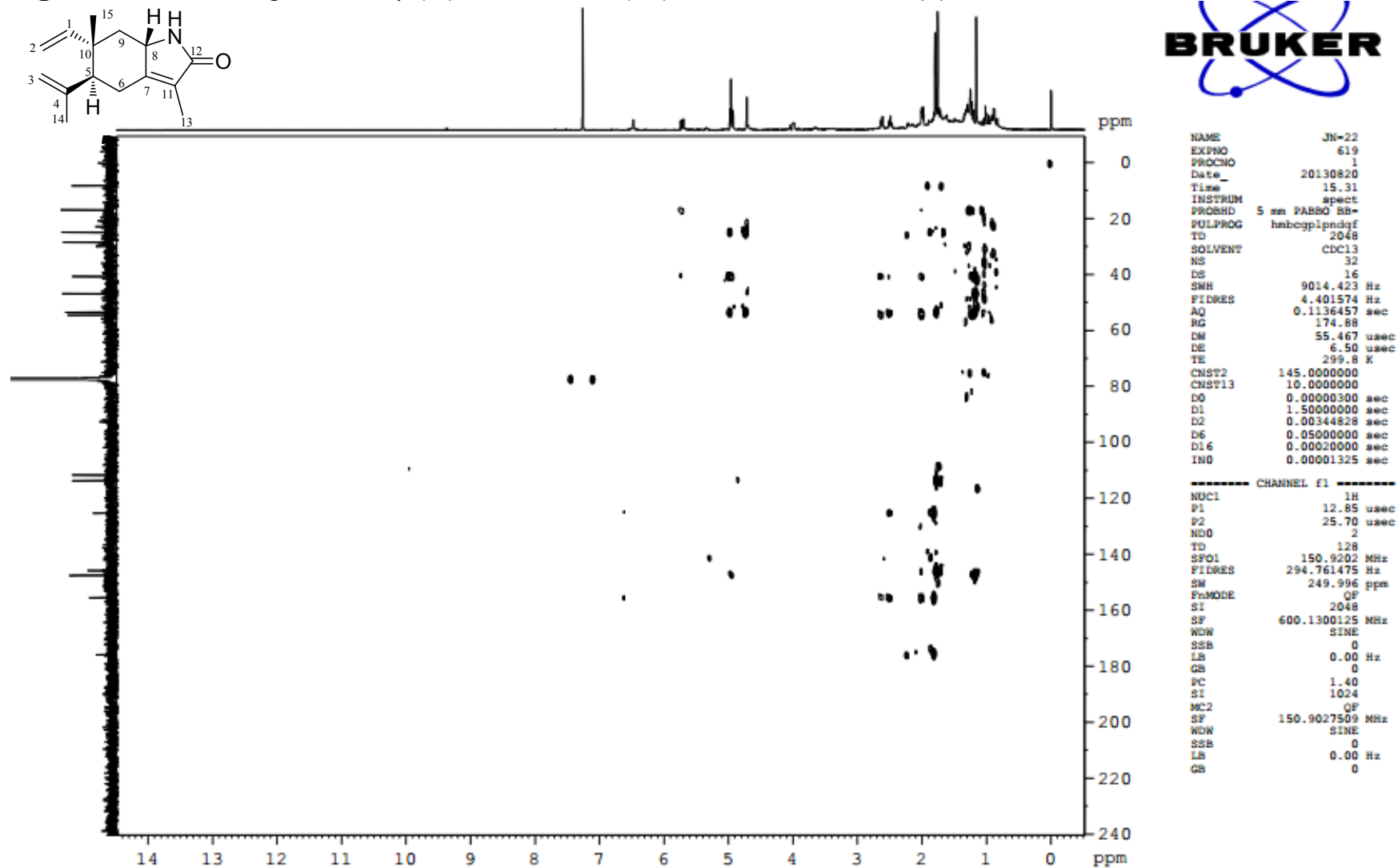
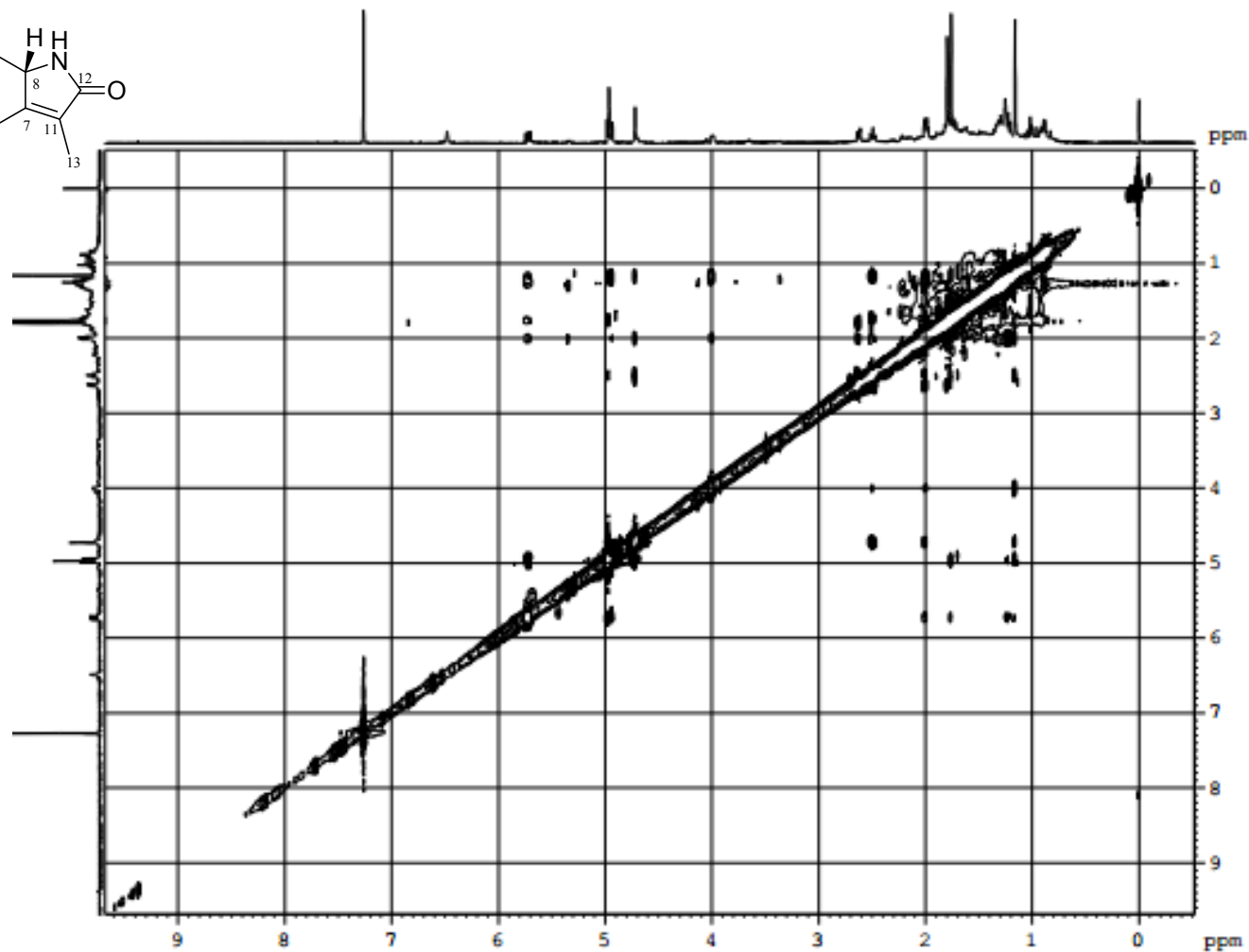
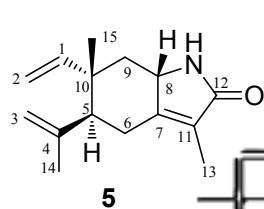


Figure S32. NOESY spectra of 8 β (H)-elema-1,3,7(11)-trien-8,12-lactam (**5**)



```

NAME          JM-22
EXPNO         1012
PROCNO        1
Date_         20140101
Time          7.45
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       noesypphpgp
TD            2048
SOLVENT       CDCl3
NS            16
DS            32
SFO1          6127.451 Hz
FIDRES        2.991920 Hz
AQ            0.1671668 sec
RG            88.31
CW            81.600 usec
DE            6.50 usec
TE            299.8 K
D0            0.00006866 sec
D1            2.00000000 sec
D8            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00002000 sec
IN0           0.00016320 sec
    
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            10.16 usec
P2            20.32 usec
P17           2500.00 usec
ND0           1
TD            256
SFO1          600.1328 MHz
FIDRES        23.935356 Hz
SN            10.210 ppm
PnMODE        States-TPP1
SI            1024
SF            600.1300155 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.00
SI            1024
MC2           States-TPP1
SF            600.1300127 MHz
WDW           QSINE
SSB
LB
GB
    
```

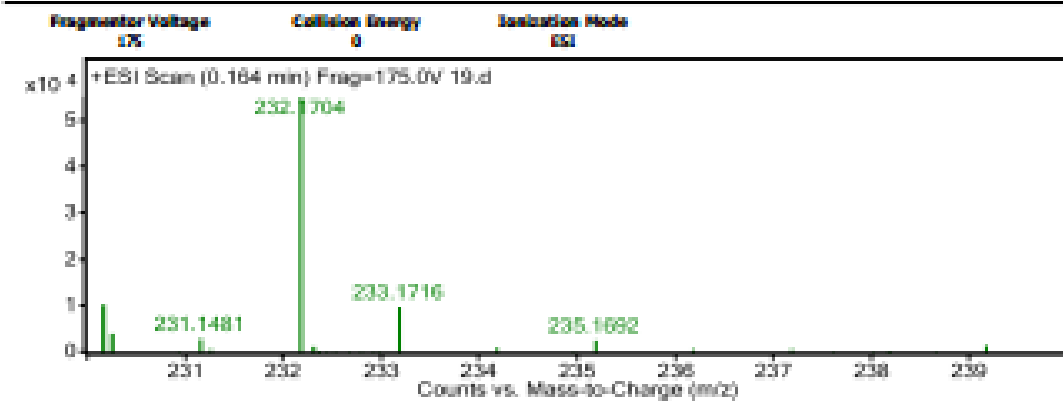
Figure S33. HRESIMS spectra of 8 β (H)-elema-1,3,7(11)-trien-8,12-lactam (5)

Qualitative Analysis Report

Data Filename:	100	Sample Name:	17
Sample Type:	Sample	Position:	F1-F1
Instrument Name:	Instrument 1	User Name:	
Acq Method:	HR(+).m	Acquired Time:	5/17/2014 10:30:24 AM
IRM Calibration Status:	Success	DA Method:	1.m
Comment:			
Sample Group:	Info.		

User Chromatograms

User Spectra



Formula Calculator Results

Formula	Best	Mass	Tgt. Mass	Diff (ppm)	Ion Species	Score
C15 H21 N O	TRUE	231.1631	231.1623	-3.45	C15 H21 N O	91.63

--- End Of Report ---

Figure S34. ¹³C NMR spectra of 8β-methoxy-isogermafurenolide (6)

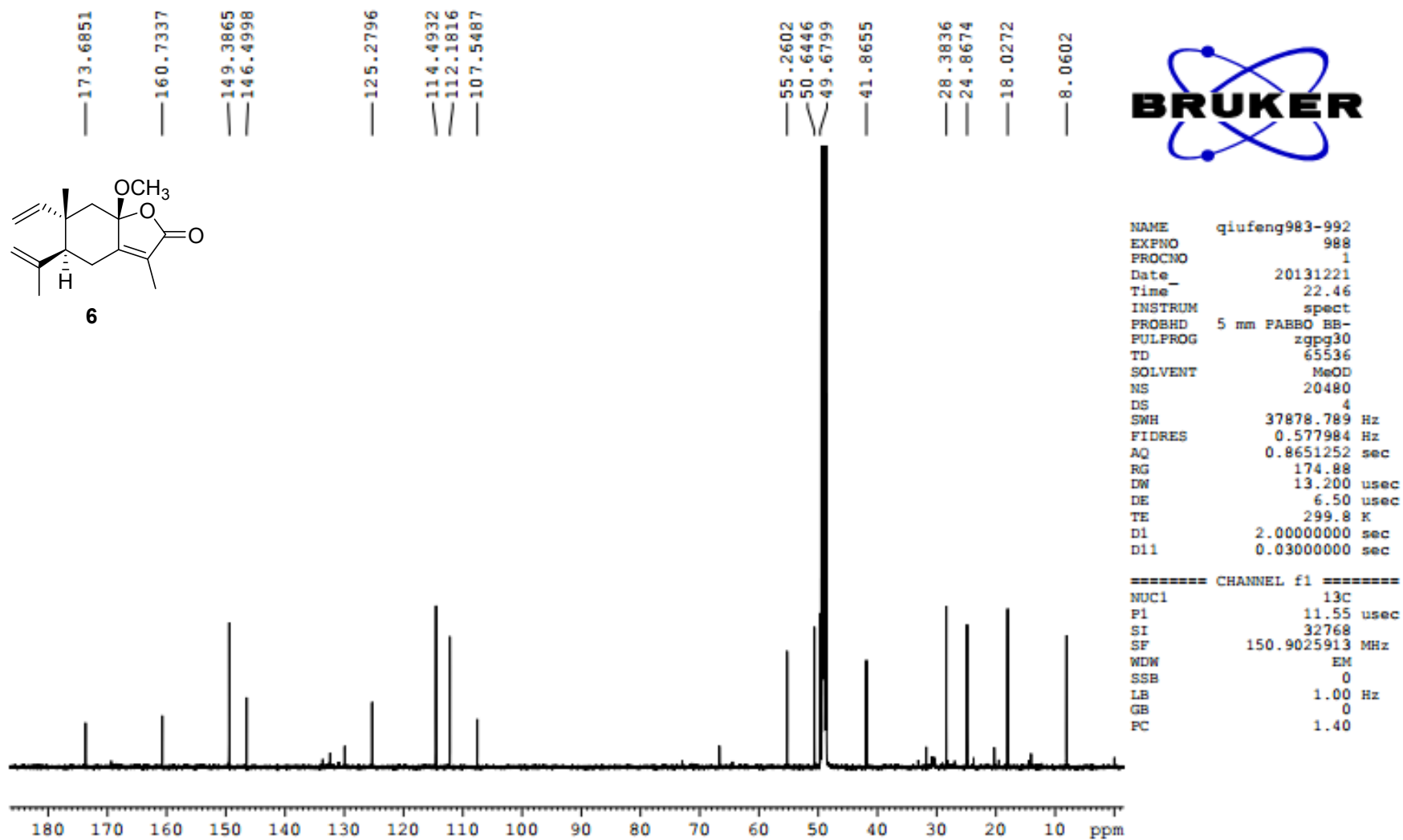
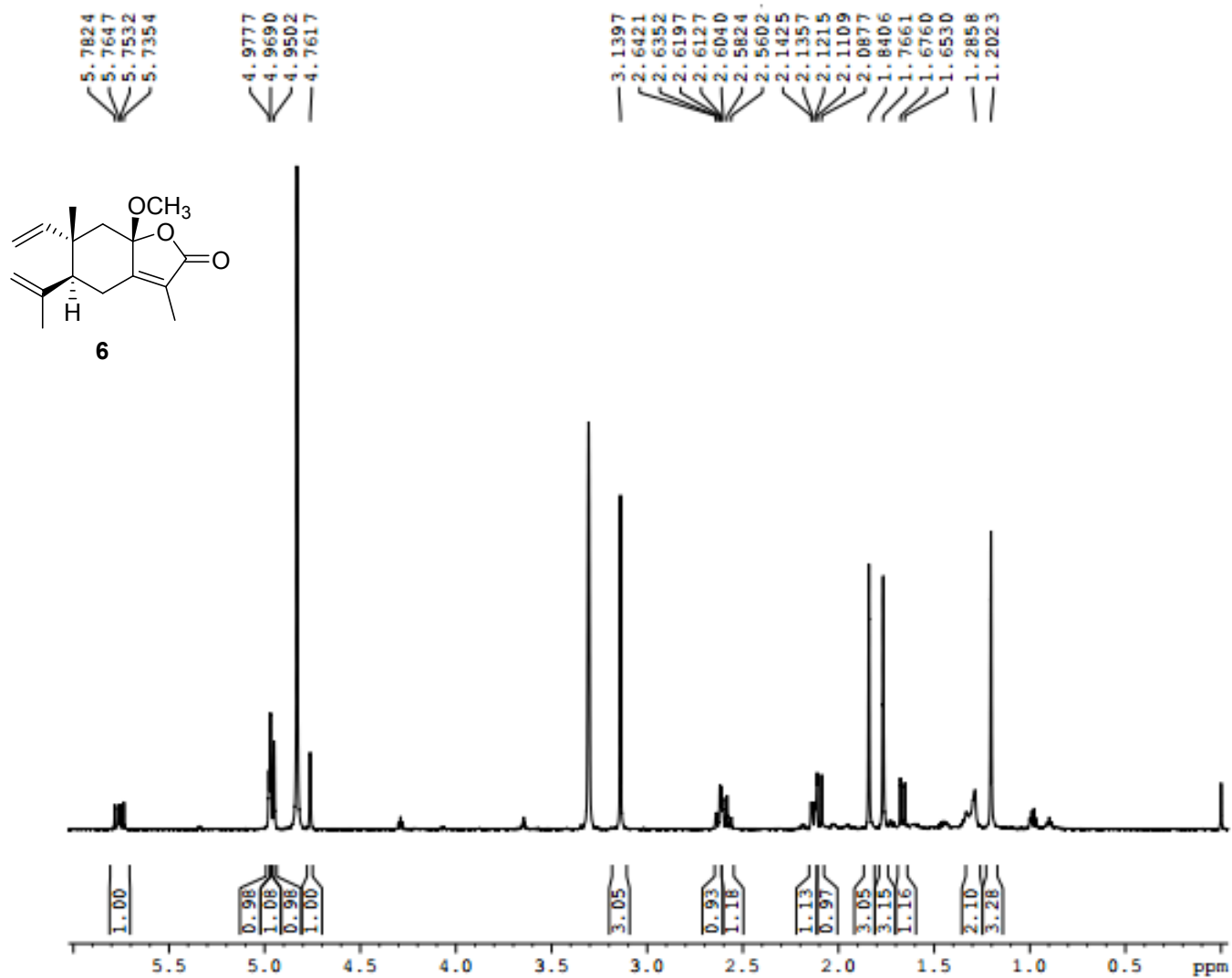


Figure S35. ¹H NMR spectra of 8β-methoxy-isogermafurenolide (6)



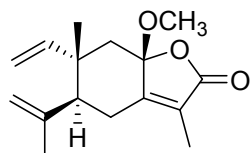
```

NAME      qiufeng931-954
EXPNO     940
PROCNO    1
Date_     20131216
Time      9.21
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   MeOD
NS         16
DS         2
SWH        12335.526 Hz
FIDRES     0.188225 Hz
AQ         2.6564426 sec
RG         174.88
DW         40.533 usec
DE         6.50 usec
TE         299.8 K
D1         1.00000000 sec
    
```

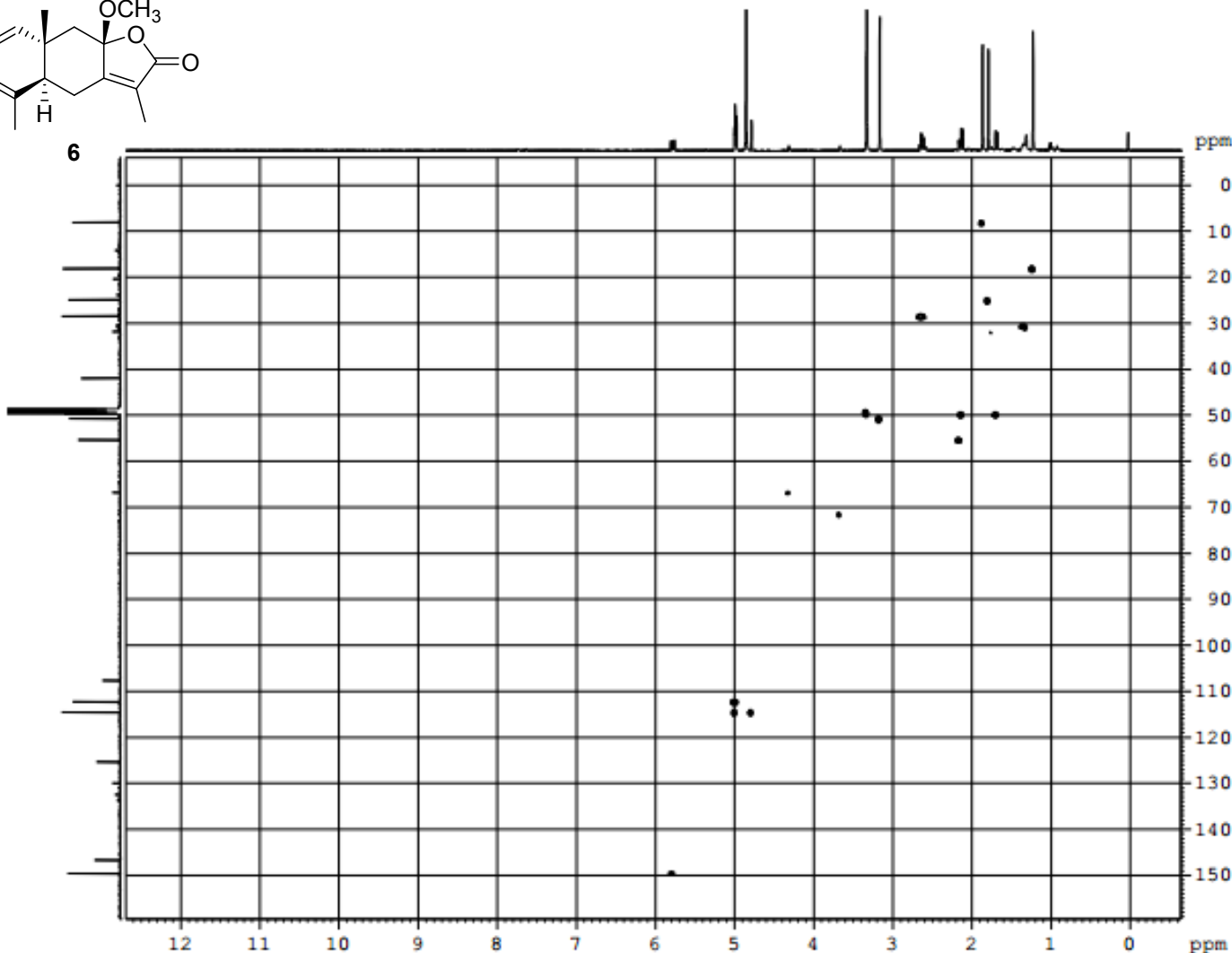
```

----- CHANNEL f1 -----
NUC1      1H
P1        12.85 usec
SI        65536
SF        600.1300165 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```

Figure S36. HSQC spectra of 8 β -methoxy-isogermafurenolide (6)



6

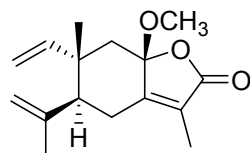


```

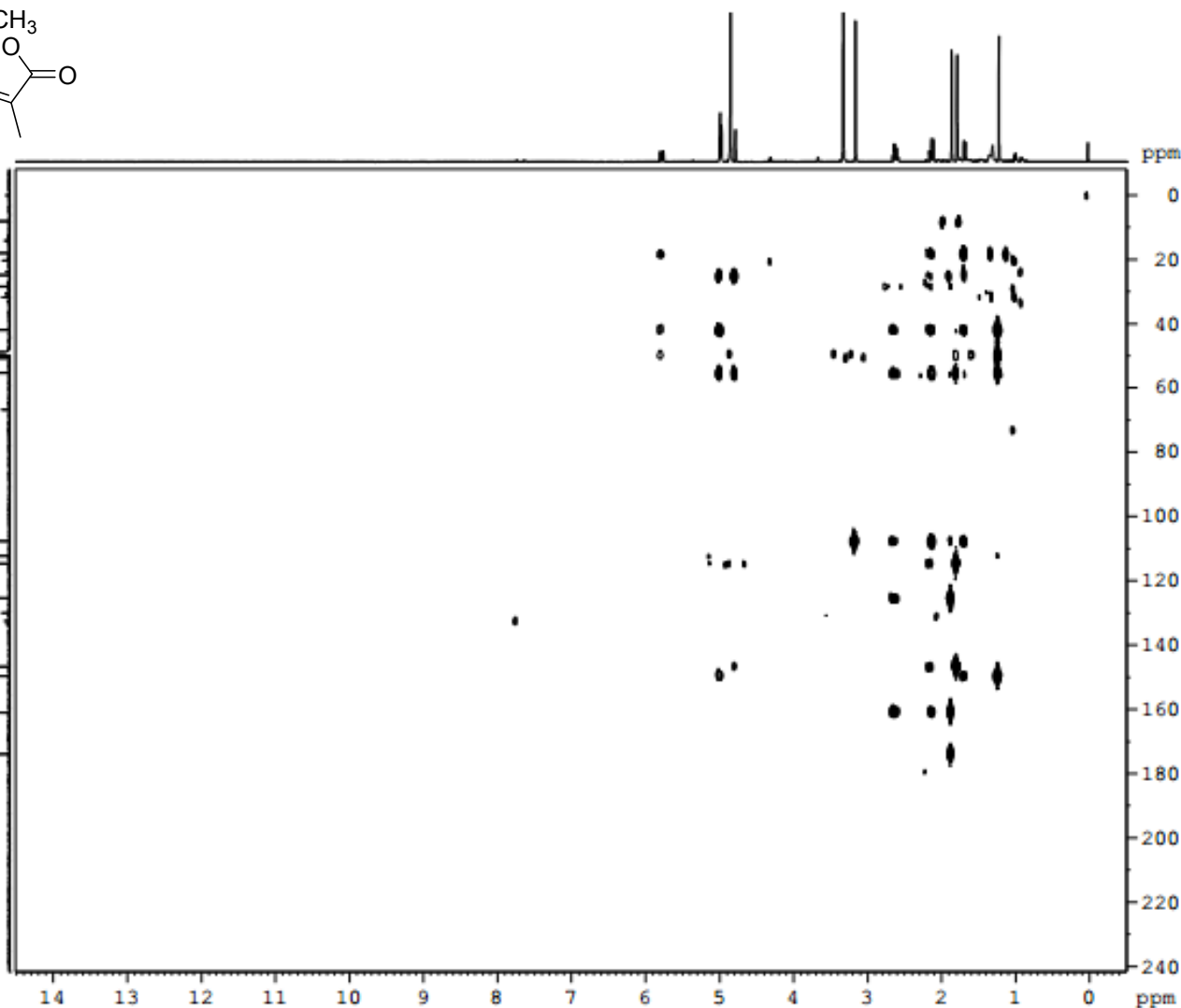
NAME          mjb-22
EXPNO         1013
PROCNO        1
Date_         20140102
Time         14.53
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            1024
SOLVENT       MeOD
NS            4
DS            16
SWH           8012.820 Hz
FIDRES        7.825800 Hz
AQ            0.0639476 sec
RG            174.88
SR            61.403 usec
DE            6.50 usec
TE            299.9 K
CHFT2         145.0000000
D0            0.0000000 sec
D1            1.5000000 sec
D4            0.00172414 sec
D11           0.02000000 sec
D13           0.00004000 sec
D14           0.00010000 sec
D24           0.00082107 sec
D30           0.00002000 sec
SFOPT20

----- CHANNEL f1 -----
NUC1          13C
P1            12.85 usec
P2            25.70 usec
PCPD          1000.00 usec
NSD           2
TD            256
SFO1          150.9141 MHz
FIDRES        97.652046 Hz
SW            145.650 ppm
PRMODE        Echo-antiecho
SI            1024
SF            400.1299949 MHz
WDW           GQ1MC
SSB           2
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           echo-antiecho
SF            150.9025561 MHz
WDW           GQ1MC
SSB           2
LB            0.00 Hz
GB            0
    
```

Figure S37. HMBC spectra of 8β-methoxy-isogermafurenolide (6)



6

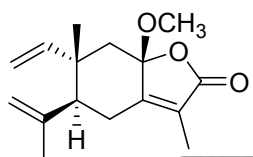


```

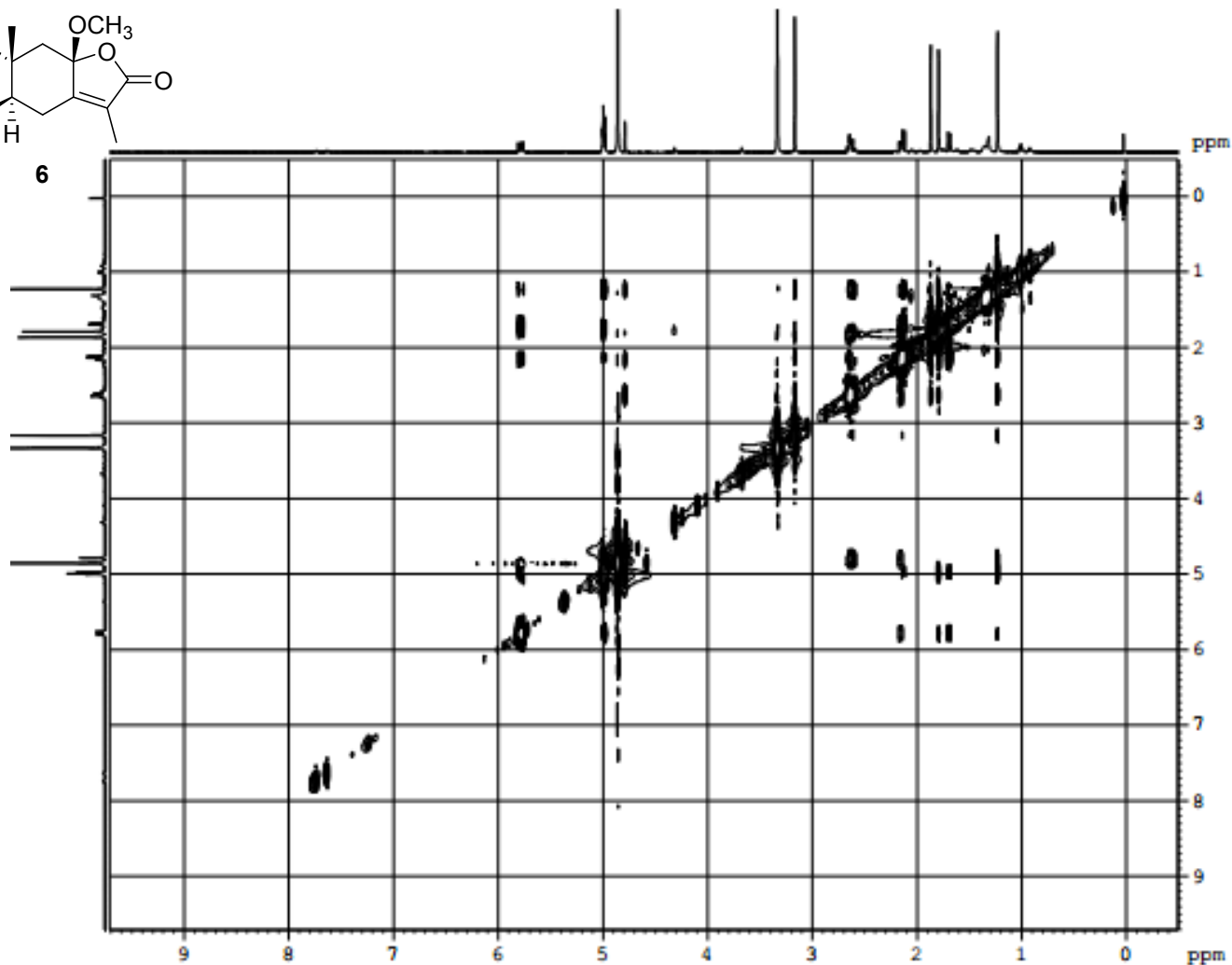
NAME          mjb-32
EXPNO         1014
PROCNO        1
Date_         20140102
Time_         17.49
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       hbhcgplpndqf
TD            2048
SOLVENT       MeOD
NS            32
DS            16
SFO1          9014.423 Hz
FIDRES        4.401574 Hz
AQ            0.1136437 sec
RG            174.88
SM            55.467 usec
DE            6.50 usec
TE            299.8 K
CNST2         145.0000000
CNST13        10.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.05000000 sec
D16           0.00020000 sec
IND           0.00001325 sec

----- CHANNEL f1 -----
NUC1          1H
P1            12.85 usec
P2            25.70 usec
NUC2          13C
TD            128
SFO1          150.9202 MHz
FIDRES        294.761475 Hz
SM            249.996 ppm
P2MODE        QF
SI            2048
SF            600.1299978 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            150.9025641 MHz
WDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
```

Figure S38. NOESY spectra of 8 β -methoxy-isogermafurenolide (6)



6



```

NAME          njh-32
EXPNO         1049
PROCNO        1
Date_         20140108
Time          8.12
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       noesygpphpc
TD            2048
SOLVENT       MeOD
NS            8
DS            32
SMH           6127.451 Hz
FIDRES        2.991920 Hz
AQ            0.1671668 sec
RG            88.31
DM            81.600 usec
DE            6.50 usec
TE            299.8 K
D0            0.00006571 sec
D1            2.00000000 sec
D8            1.00000000 sec
D11           0.03000000 sec
D12           0.00020000 sec
D16           0.00020000 sec
IN0           0.00016320 sec
    
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            12.48 usec
P2            24.96 usec
P17           2500.00 usec
ND0           1
TD            128
SFO1          600.1328 MHz
FIDRES        47.870712 Hz
SN            10.210 ppm
FnMODE        States=TPPI
SI            1024
SF            600.130000 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.00
SI            1024
MC2           States=TPPI
SF            600.130000 MHz
WDW           QSINE
SSB           2
LB            0
GB
    
```


Figure S39. HRESIMS spectra of $\delta\beta$ -methoxy-isogermafurenolide (**6**)



Figure S40. ¹³C NMR spectra of phaeusmane I (7)

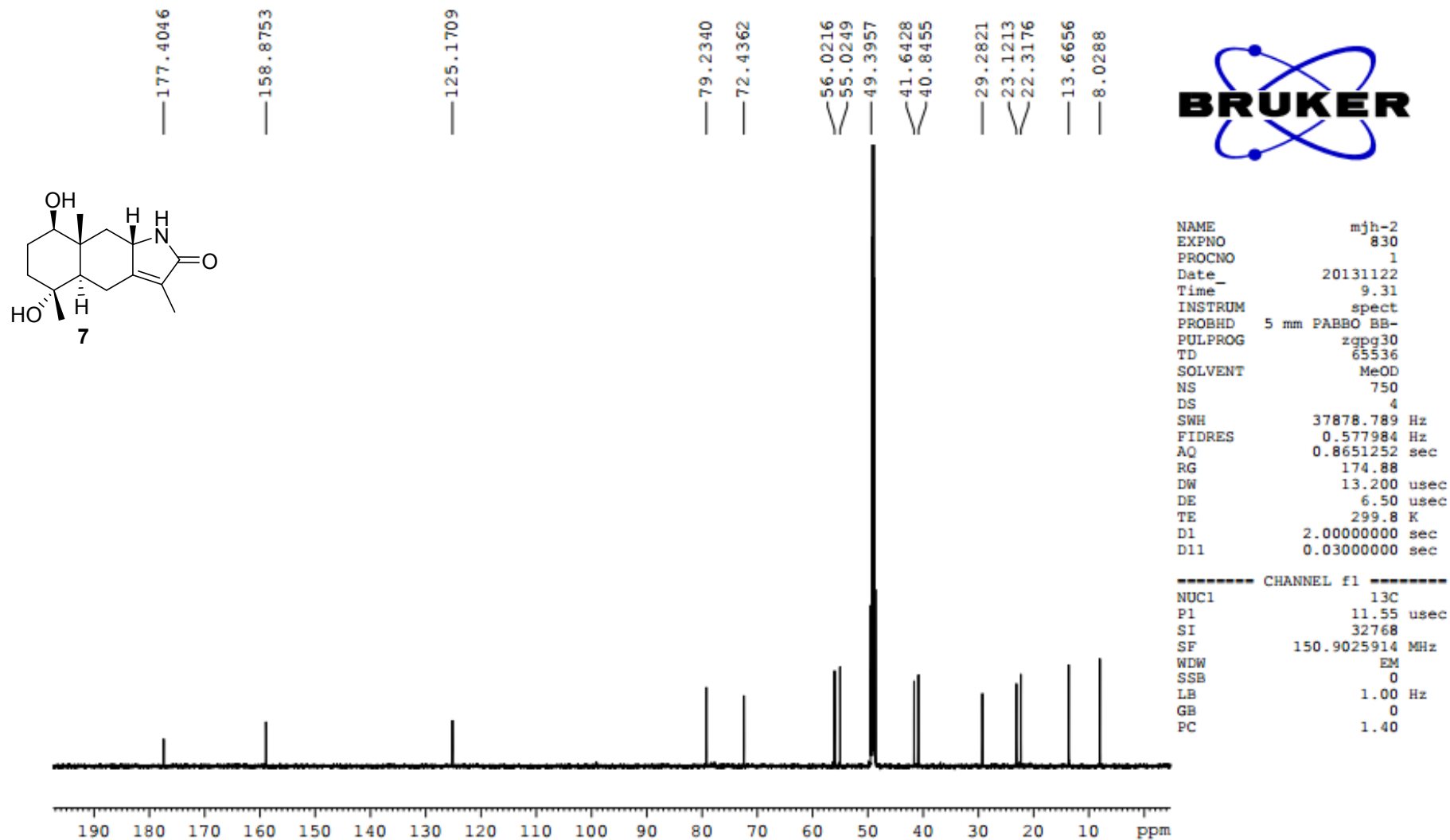
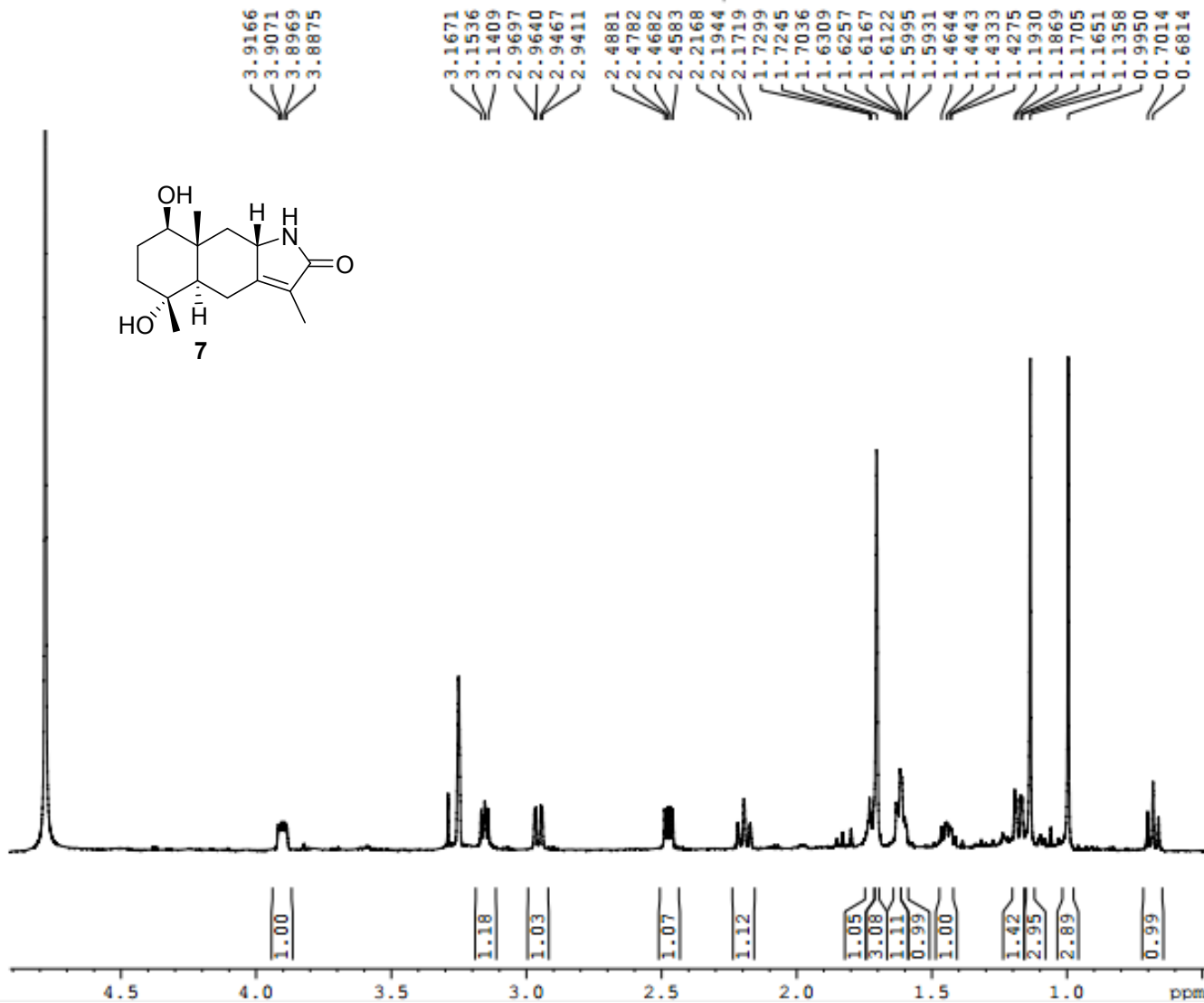


Figure S41. ¹H NMR spectra of phaeusmane I (7)



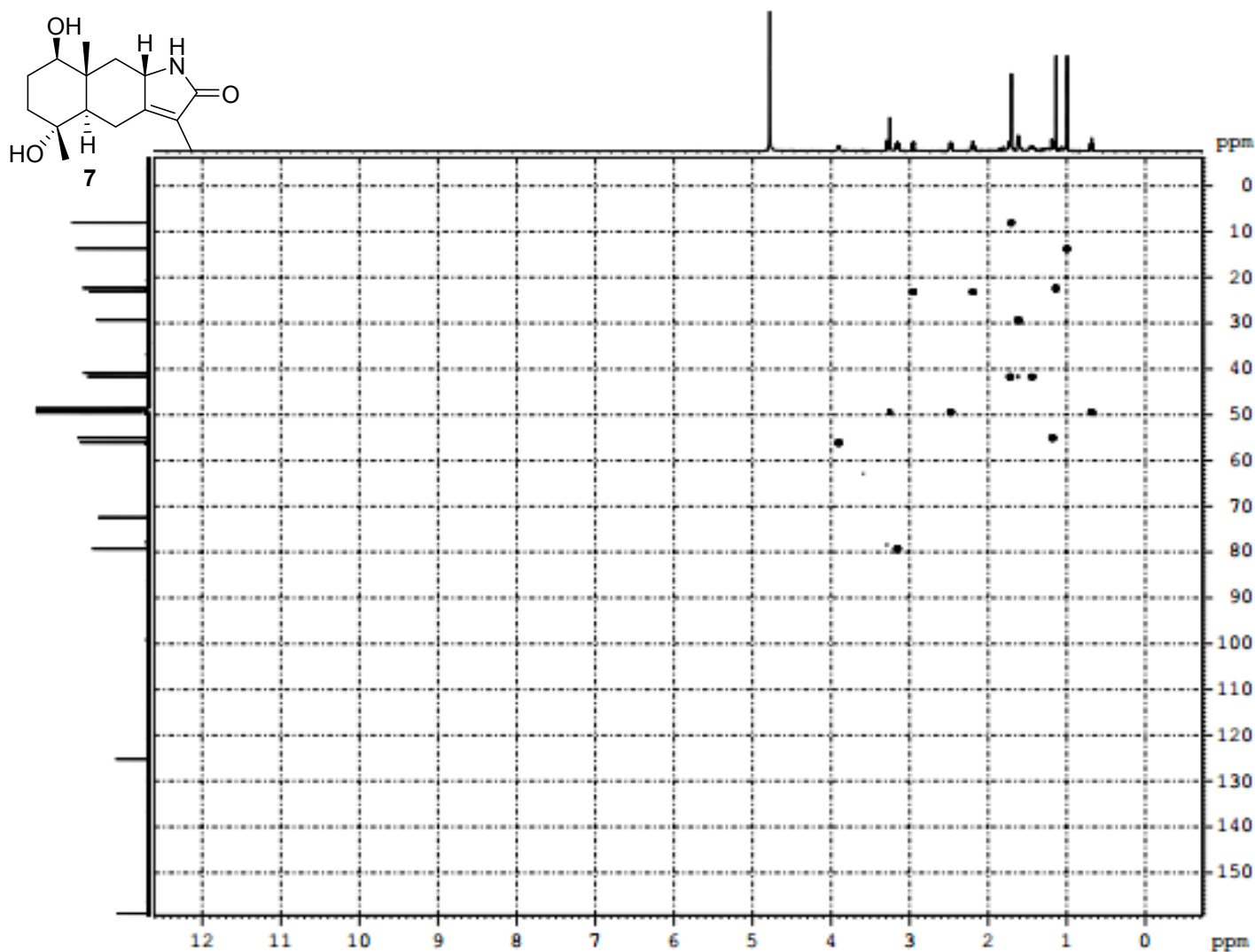
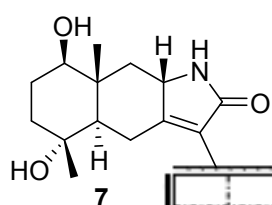
```

NAME           mjh-2
EXPNO          777
PROCNO         1
Date_          20131113
Time_          14.05
INSTRUM        spect
PROBHD         5 mm PABBO BB-
PULPROG        zg30
TD             65536
SOLVENT        MeOD
NS             16
DS             2
SWH            12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG            99.85
DW            40.533 usec
DE            6.50 usec
TE            299.8 K
D1            1.00000000 sec
    
```

```

===== CHANNEL f1 =====
NUC1           1H
P1            12.85 usec
SI            65536
SF            600.1300497 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

Figure S42. HSQC spectra of phaeusmane I (7)



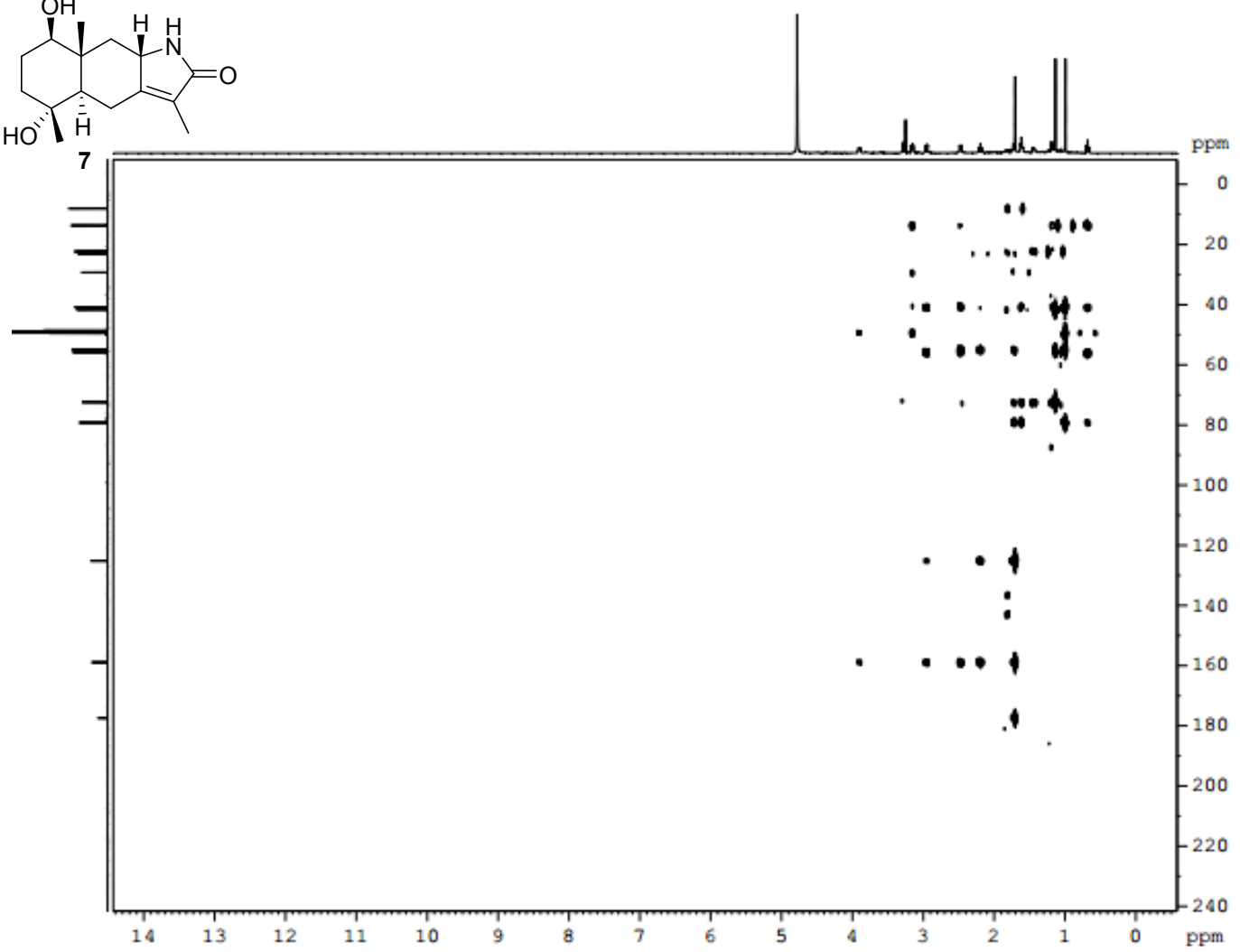
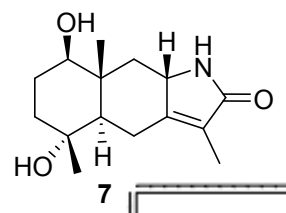
```

NAME      n34-2
EXPNO    608
PROCNO    1
Date_    2011081
Time     11.38
INSTRUM   spect
PROBHD    5 mm TANGO MM-
PULPROG   zgpg30g12
TD        1024
SOLVENT   MeOD
NS        8
DS        16
SWH       8012.800 Hz
F2       7.818000 Hz
AQ        0.0439376 sec
RG        174.88
IN       62.500 usec
DE        6.50 usec
TE        298.15 K
CMT0     149.000000
CO        0.0000000 sec
C1        1.0000000 sec
C2        0.0017216 sec
C3        0.0000000 sec
C4        0.0000000 sec
C5        0.0000000 sec
C6        0.0000000 sec
C7        0.0000000 sec
C8        0.0000000 sec
C9        0.0000000 sec
C10       0.0000000 sec
C11       0.0000000 sec
C12       0.0000000 sec
C13       0.0000000 sec
C14       0.0000000 sec
C15       0.0000000 sec
C16       0.0000000 sec
C17       0.0000000 sec
C18       0.0000000 sec
C19       0.0000000 sec
C20       0.0000000 sec
C21       0.0000000 sec
C22       0.0000000 sec
C23       0.0000000 sec
C24       0.0000000 sec
C25       0.0000000 sec
C26       0.0000000 sec
C27       0.0000000 sec
C28       0.0000000 sec
C29       0.0000000 sec
C30       0.0000000 sec
C31       0.0000000 sec
C32       0.0000000 sec
C33       0.0000000 sec
C34       0.0000000 sec
C35       0.0000000 sec
C36       0.0000000 sec
C37       0.0000000 sec
C38       0.0000000 sec
C39       0.0000000 sec
C40       0.0000000 sec
C41       0.0000000 sec
C42       0.0000000 sec
C43       0.0000000 sec
C44       0.0000000 sec
C45       0.0000000 sec
C46       0.0000000 sec
C47       0.0000000 sec
C48       0.0000000 sec
C49       0.0000000 sec
C50       0.0000000 sec
C51       0.0000000 sec
C52       0.0000000 sec
C53       0.0000000 sec
C54       0.0000000 sec
C55       0.0000000 sec
C56       0.0000000 sec
C57       0.0000000 sec
C58       0.0000000 sec
C59       0.0000000 sec
C60       0.0000000 sec
C61       0.0000000 sec
C62       0.0000000 sec
C63       0.0000000 sec
C64       0.0000000 sec
C65       0.0000000 sec
C66       0.0000000 sec
C67       0.0000000 sec
C68       0.0000000 sec
C69       0.0000000 sec
C70       0.0000000 sec
C71       0.0000000 sec
C72       0.0000000 sec
C73       0.0000000 sec
C74       0.0000000 sec
C75       0.0000000 sec
C76       0.0000000 sec
C77       0.0000000 sec
C78       0.0000000 sec
C79       0.0000000 sec
C80       0.0000000 sec
C81       0.0000000 sec
C82       0.0000000 sec
C83       0.0000000 sec
C84       0.0000000 sec
C85       0.0000000 sec
C86       0.0000000 sec
C87       0.0000000 sec
C88       0.0000000 sec
C89       0.0000000 sec
C90       0.0000000 sec
C91       0.0000000 sec
C92       0.0000000 sec
C93       0.0000000 sec
C94       0.0000000 sec
C95       0.0000000 sec
C96       0.0000000 sec
C97       0.0000000 sec
C98       0.0000000 sec
C99       0.0000000 sec
C100      0.0000000 sec
    
```

```

----- CHANNEL f1 -----
NUC1      13C
PC        12.85 usec
PD        28.70 usec
PDB       1000.00 usec
SFO1     100.626127 MHz
SF        100.626127 MHz
WDW       GATE
SSB       0
LB        0.00 Hz
GB        0
PC        1.00
SI        1024
SCZ       0
SFO2     400.1462017 MHz
SF        400.1462017 MHz
WDW       GATE
SSB       0
LB        0.00 Hz
GB        0
    
```

Figure S43. HMBC spectra of phaeusmane I (7)

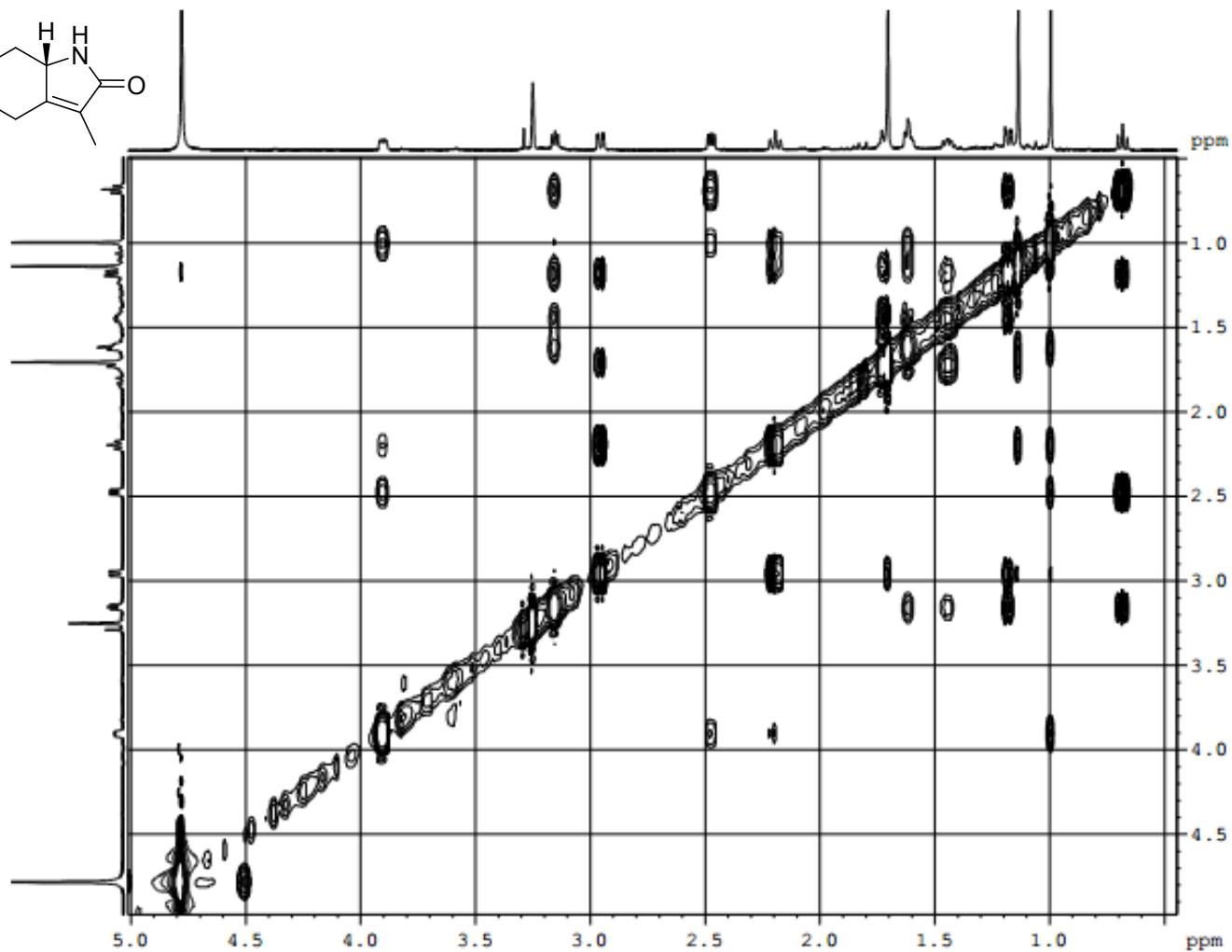
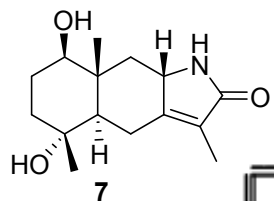


```

NAME          mjb-2
EXPNO         909
PROCNO        1
Date_         20131206
Time          12.34
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       hbhcgplp00qf
TD            2048
SOLVENT       MeOD
NS            16
DS            16
SWH           9014.423 Kz
FIDRES        4.401574 Kz
AQ            0.1136457 sec
RG            174.88
DW            55.467 usec
DE            6.50 usec
TE            299.9 K
CHST2        145.000000
CHST13       19.000000
D0            0.00000300 sec
D1            1.50000000 sec
D2            0.00344828 sec
D6            0.05000000 sec
D16           0.00020000 sec
IND           0.00001325 sec

----- CHANNEL f1 -----
NUC1          13C
P1            12.85 usec
P2            25.70 usec
NUC2          1H
SFO1          150.9202 MHz
FIDRES        294.761475 Kz
SW            249.996 ppm
PRMODE        QF
SI            2048
SF            600.1300477 MHz
WDW           SINE
SSB           0
LB            0.00 Kz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            150.9025729 MHz
WDW           SINE
SSB           0
LB            0.00 Kz
GB            0
    
```

Figure S44. NOESY spectra of phaeusmane I (7)



```

NAME          njh-2
EXPNO         959
PROCNO        1
Date_         20131217
Time          15.27
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       noesygpphpp
TD            2048
SOLVENT       MeOD
NS            8
DS            32
SWH           6127.451 Hz
FIDRES        2.991920 Hz
AQ            0.1671668 sec
RG            174.88
DW            81.600 usec
DE            6.50 usec
TE            299.8 K
DO            0.00006623 sec
D1            2.00000000 sec
D8            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00016320 sec

----- CHANNEL f1 -----
NUC1          1H
P1            12.07 usec
P2            24.14 usec
P17           2500.00 usec
ND0           1
TD            161
SFO1          600.1328 MHz
FIDRES        38.058701 Hz
SW            10.210 ppm
FnMODE        States-TPPI
SI            1024
SF            600.1300471 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.00
SI            1024
MC2           States-TPPI
SF            600.1300454 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
    
```

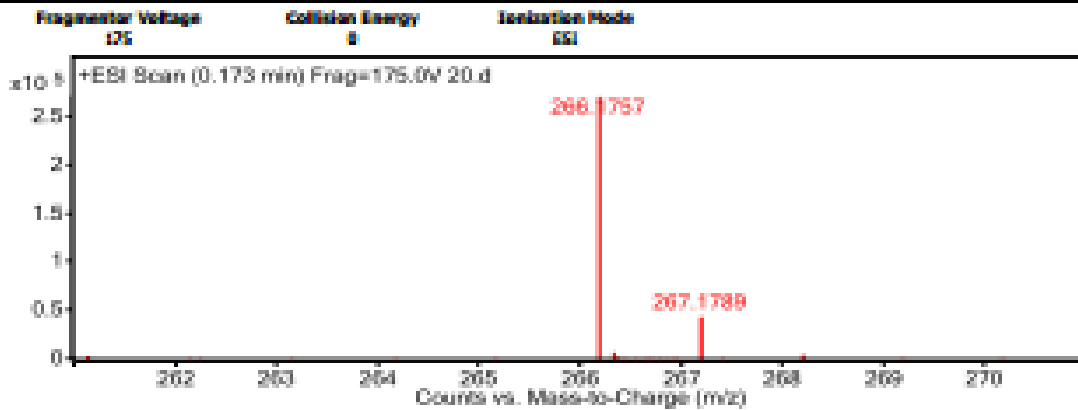
Figure S45. HRESIMS spectra of phaeusmane I (7)

Qualitative Analysis Report

Data Filename:	200	Sample Name:	20
Sample Type:	Sample	Position:	P1-P2
Instrument Name:	Instrument 1	User Name:	
Acq Method:	HR(+).m	Acquired Time:	5/17/2014 10:32:23 AM
IRM Calibration Status:	Success	DA Method:	1.m
Comment:			
Sample Group:	Info.		

User Chromatograms

User Spectra

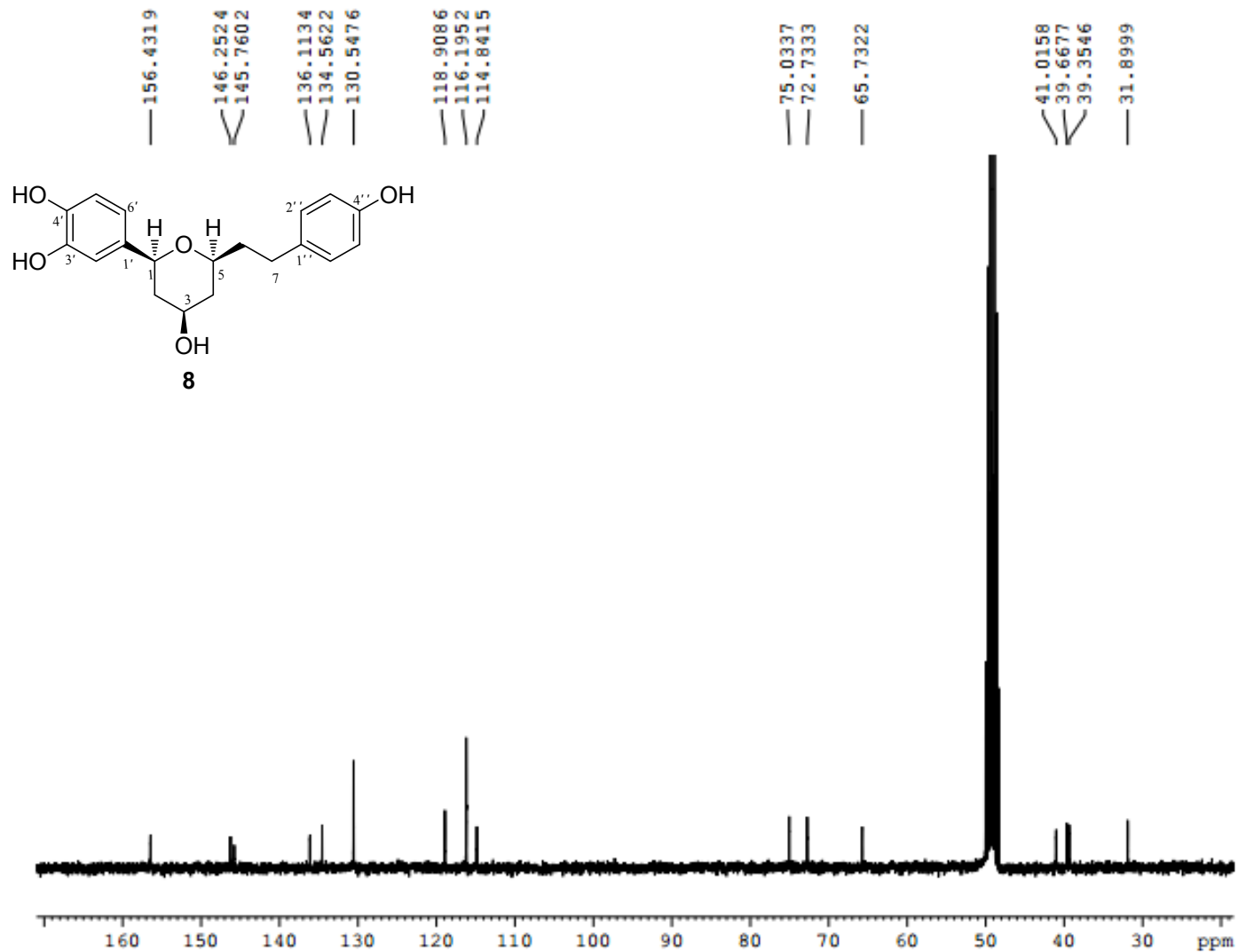


Formula Calculator Results

Formula	Best	Mass	Tgt Mass	Diff (ppm)	Ion Species	Score
C15 H23 N O2	TRUE	265.1684	265.1678	-2.45	C15 H24 N O2	98.22

--- End Of Report ---

Figure S46. ¹³C NMR spectra of phaeoheptanoxide (8)



```

NAME                JE-61
EXPNO                2
PROCNO              1
Date_                20120611
Time_                17.52
INSTRUM             spect
PROBHD              5 mm QNP 1H/13
PULPROG             zgpg30
TD                  65536
SOLVENT             MeOD
NS                   809
DS                   4
SWH                 22675.736 Hz
FIDRES              0.346004 Hz
AQ                  1.4451188 sec
RG                   13004
DW                  22.050 usec
DE                   6.50 usec
TE                  300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                  1
    
```

```

----- CHANNEL f1 -----
NUC1                 13C
P1                   14.00 usec
PL1                   2.00 dB
SFO1                 75.4752953 MHz
    
```

```

----- CHANNEL f2 -----
CPDPRG2             waltz16
NUC2                 1H
PCPD2                90.00 usec
PL2                   2.00 dB
PL12                 20.00 dB
PL13                 20.00 dB
SFO2                 300.1312005 MHz
SI                   32768
SF                   75.4676324 MHz
WDW                  EM
SSB                   0
LB                   1.00 Hz
GB                   0
FC                   1.40
    
```


Figure S47. ¹H NMR spectra of phaeoheptanoxide (8)

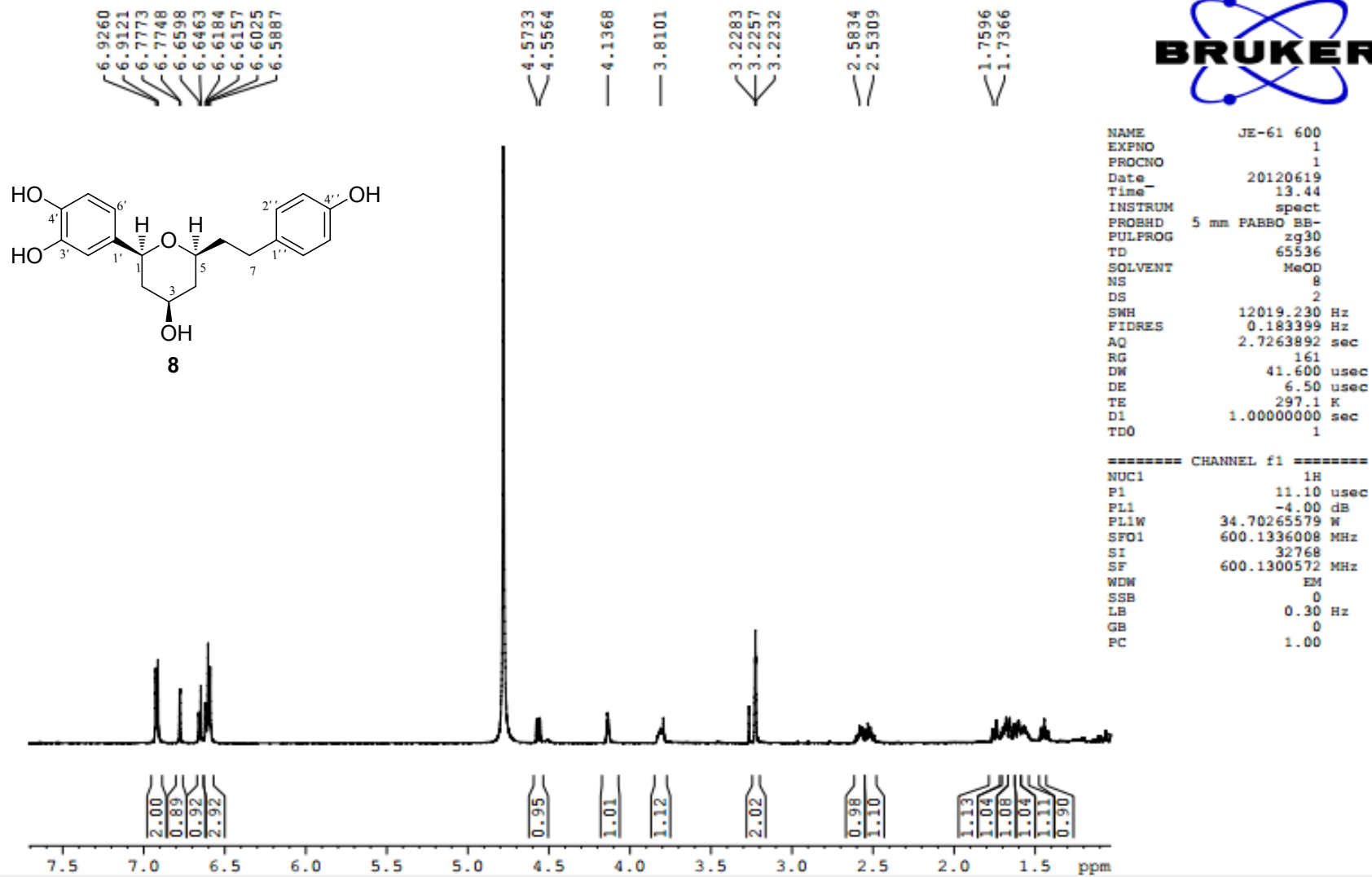
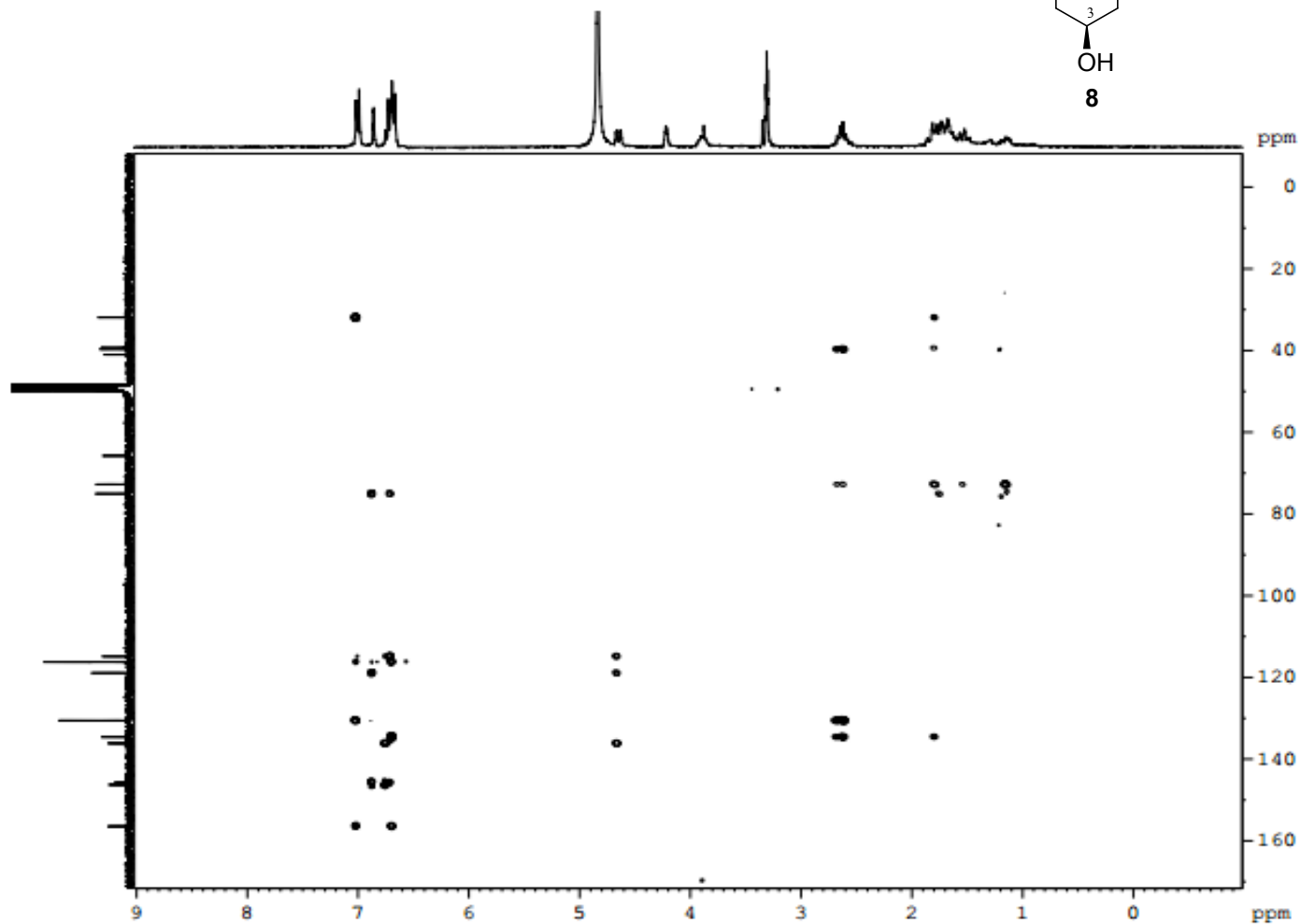
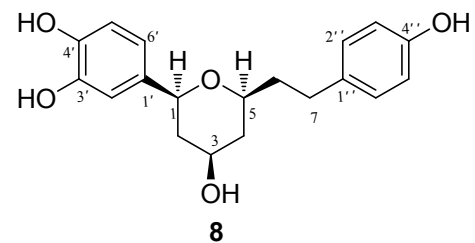


Figure S49. HMBC spectra of phaeoheptanoxide (8)



```

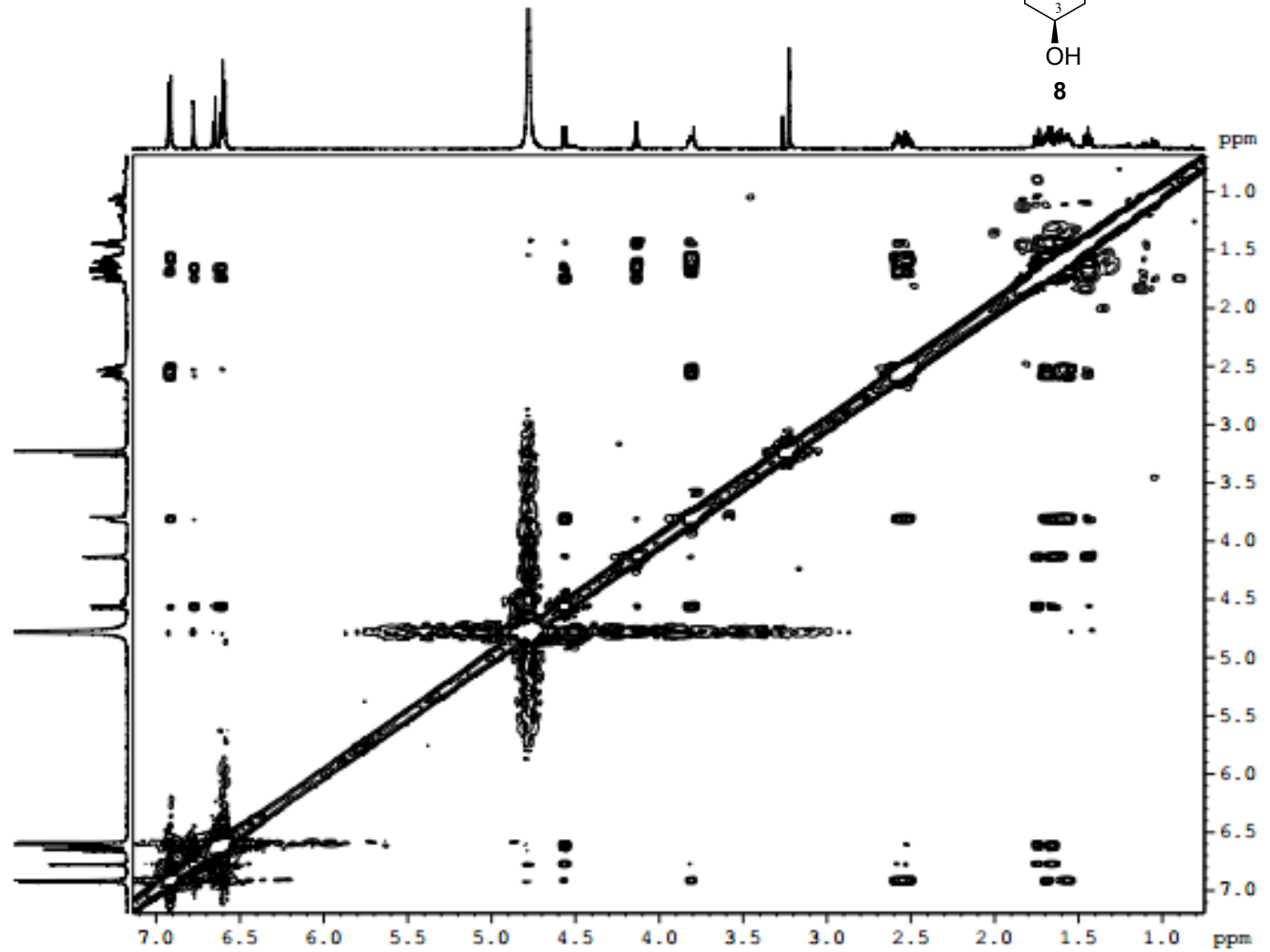
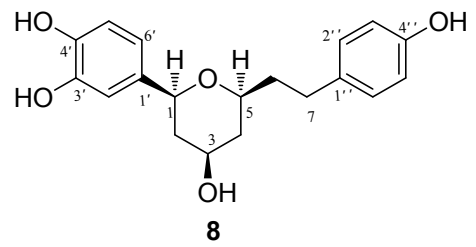
NAME          JK-E1 600
EXPNO         1
PROCNO        1
Date_         20120619
Time          15.03
INSTRUM       spect
PROBHD        5 mm FARGO BB-
PULPROG       hmcgppndqf
TD            1024
SOLVENT       MeOD
NS            16
DS            16
SWH           6009.615 Hz
FIDRES        5.868765 Hz
AQ            0.0833300 sec
RG            29100
DW            83.200 usec
DE            6.50 usec
TE            296.2 K
CHST2         145.0000000
CHST13        5.0000000
D0            0.0000300 sec
D1            1.5000000 sec
D2            0.00344828 sec
D6            0.1000000 sec
D16           0.0002000 sec
TD            0.00001840 sec

----- CHANNEL f1 -----
NUC1           1H
P1            11.10 usec
P2            22.20 usec
PL1           -4.00 dB
PL1W          34.7026579 W
SFO1          600.1324005 MHz

----- CHANNEL f2 -----
NUC2           13C
P3            8.80 usec
PL2           1.00 dB
PL2W          83.20243835 W
SFO2          150.9148812 MHz

----- GRADIENT CHANNEL -----
GPMAM1        SINE.100
GPMAM2        SINE.100
GPMAM3        SINE.100
GPE1          50.00 %
GPE2          30.00 %
GPE3          40.10 %
P16           1000.00 usec
NDD           2
TD            256
SFO1          150.9148 MHz
FIDRES        106.112022 Hz
SW            180.000 ppm
PbMODE        QF
S1            1024
SF            600.1299920 MHz
NDW           SINE
SSB           0
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           QF
SF            150.9025646 MHz
NDW           SINE
SSB           0
LB            0.00 Hz
GB            0
    
```

Figure S50. NOESY spectra of phaeoheptanoxide (8)



```

NAME          JK-61 600
EXPNO         6
PROCNO        1
Date_         20120619
Time          13.47
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            1024
SOLVENT       MeOD
NS            4
DS            4
SWH           6009.615 Hz
FIDRES       5.868765 Hz
AQ           0.0853300 sec
RG            64
DW           83.200 usec
DE            6.50 usec
TE            297.2 K
D0            0.0000007 sec
D1            2.00000000 sec
D8            0.60000002 sec
TD0           0.00016640 sec

----- CHANNEL f1 -----
NUC1          1H
P1            11.10 usec
PL1           -4.00 dB
PL1W          34.78265579 W
SFO1          600.1324005 MHz
NUC           1
TD            256
SFO1          600.1324 MHz
FIDRES       23.475023 Hz
SW           10.014 ppm
F2MODE       States-TFPI
S1           1024
SF           600.1300559 MHz
WDW          QZINE
SSB           2
LR            0.00 Hz
GR            0
PC            1.00
S1           1024
MC2          States-TFPI
SF           600.1300560 MHz
WDW          QZINE
SSB           2
LR            0.00 Hz
GR            0
    
```

Figure S51. HRESIMS spectra of phaeoheptanoxide (8)

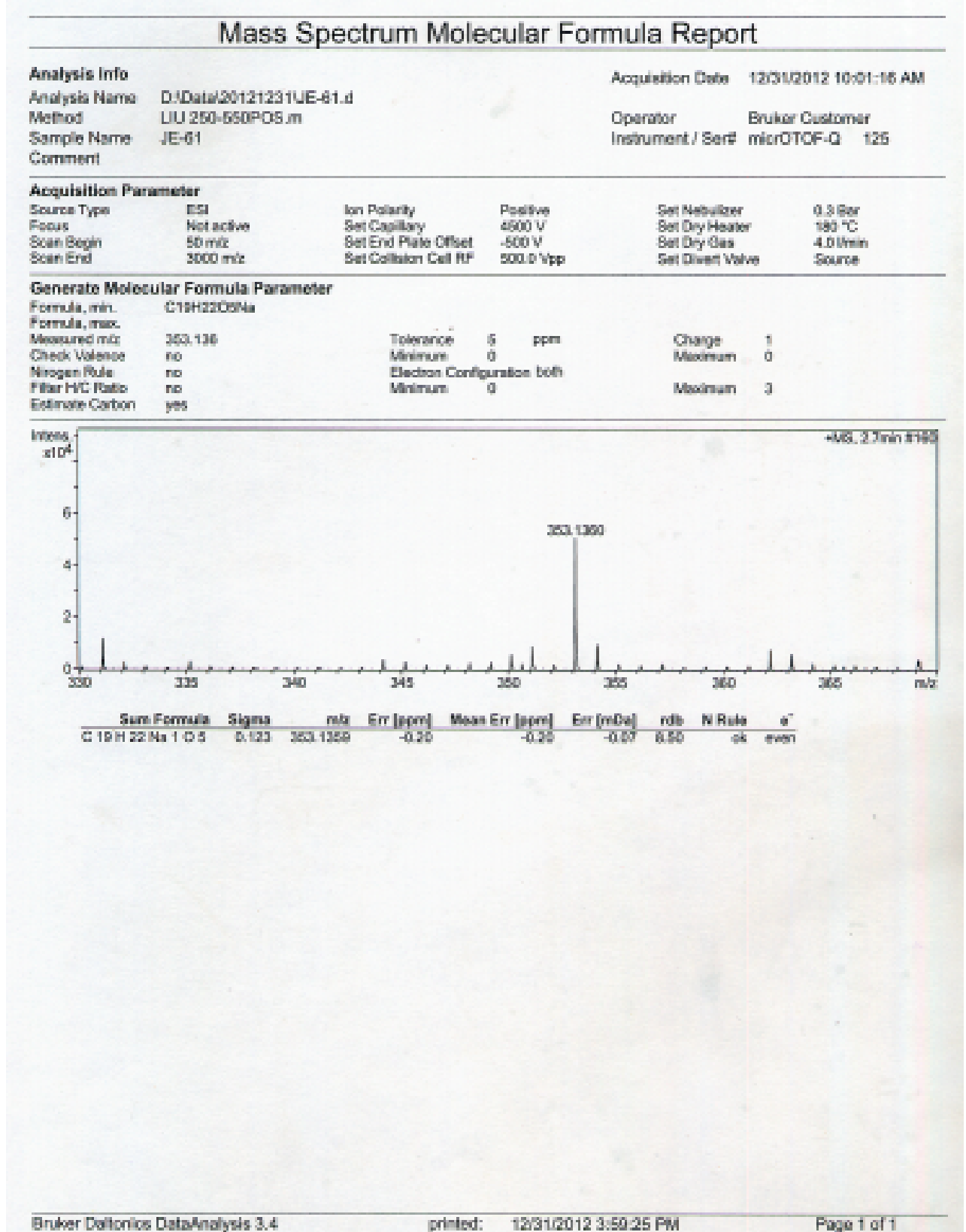


Figure S52. Chiral HPLC analytical chromatograms for compounds **5** and **6**

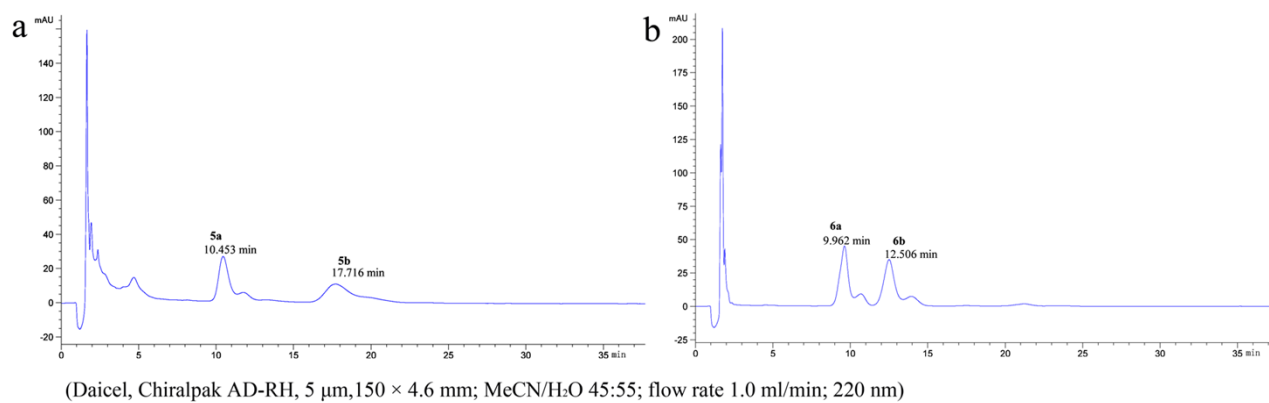


Figure S53. Chiral HPLC analytical chromatogram for compound **7**

