

Supporting Information For:

Molecular Library Synthesis Using Complex Substrates: Expanding the Framework of Triterpenoids

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General experimental details

All reactions were run in an atmosphere of dry argon unless otherwise stated. THF was distilled from benzophenone ketyl solution with sodium prior to use. Quick syringe transfers were done with disposable syringes and needles.

Column chromatography was performed with silica gel (particle size 32-63 μm). Analytical and semi-preparative HPLC separations were performed using acetonitrile and water (for HPLC, 99.9%). Analytical thin-layer chromatography (TLC) was carried out using glass-coated silica gel 0.25 mm plates with fluorescent indicator. All reactions that were monitored by TLC were visualized with a 254 nm UV-lamp or using phosphomolybdic acid (PMA) and 1,4-dinitrophenylhydrazine (DNP) stain solutions prepared by well-known protocols.

Chemical shifts of all ^1H and ^{13}C NMR spectra reported in δ units, part per million (ppm) with reference to the residual solvent peak (CDCl_3 , 7.26 ppm for ^1H NMR and 77.16 ppm, center of triplet, for ^{13}C NMR). DEPT, COSY, NOESY, HMQC, HMBC spectra were recorded using standard 2-D NMR pulse sequences.

Complete reference 46

Reference 46. Gaussian 09, Revision A.02,
M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria,
M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci,
G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian,
A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada,
M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima,
Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr.,
J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers,
K. N. Kudin, V. N. Staroverov, R. Kobayashi, J. Normand,
K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi,
M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross,
V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann,
O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski,
R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth,
P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels,
O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski,
and D. J. Fox, Gaussian, Inc., Wallingford CT, 2009.

Figure S1. Mechanistic considerations for the formation of **10** and **11**.

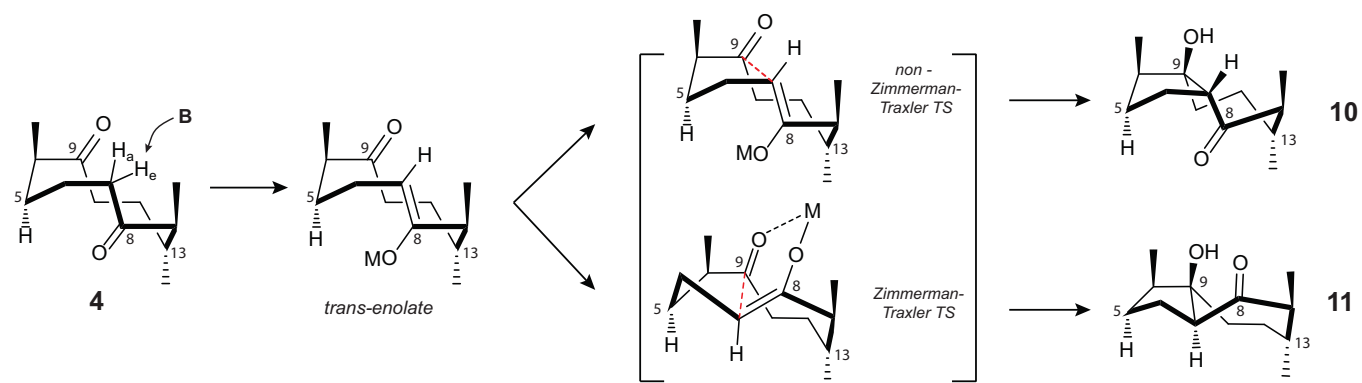


Figure S2. Conformational analyses of polyketones **4**, **6**, **16**.

Conformational analyses were carried out using Gaussian (R) 09 using B3LYP/6-311G(d,p).

Ground state conformations and electronic energies are shown.

BCC = 'boat (with bow at C-5 and stern between C-8 and C-11) -chair-chair'

CCB = 'chair-chair- boat (with bow between C-8 and C-11 and stern at C-13)'

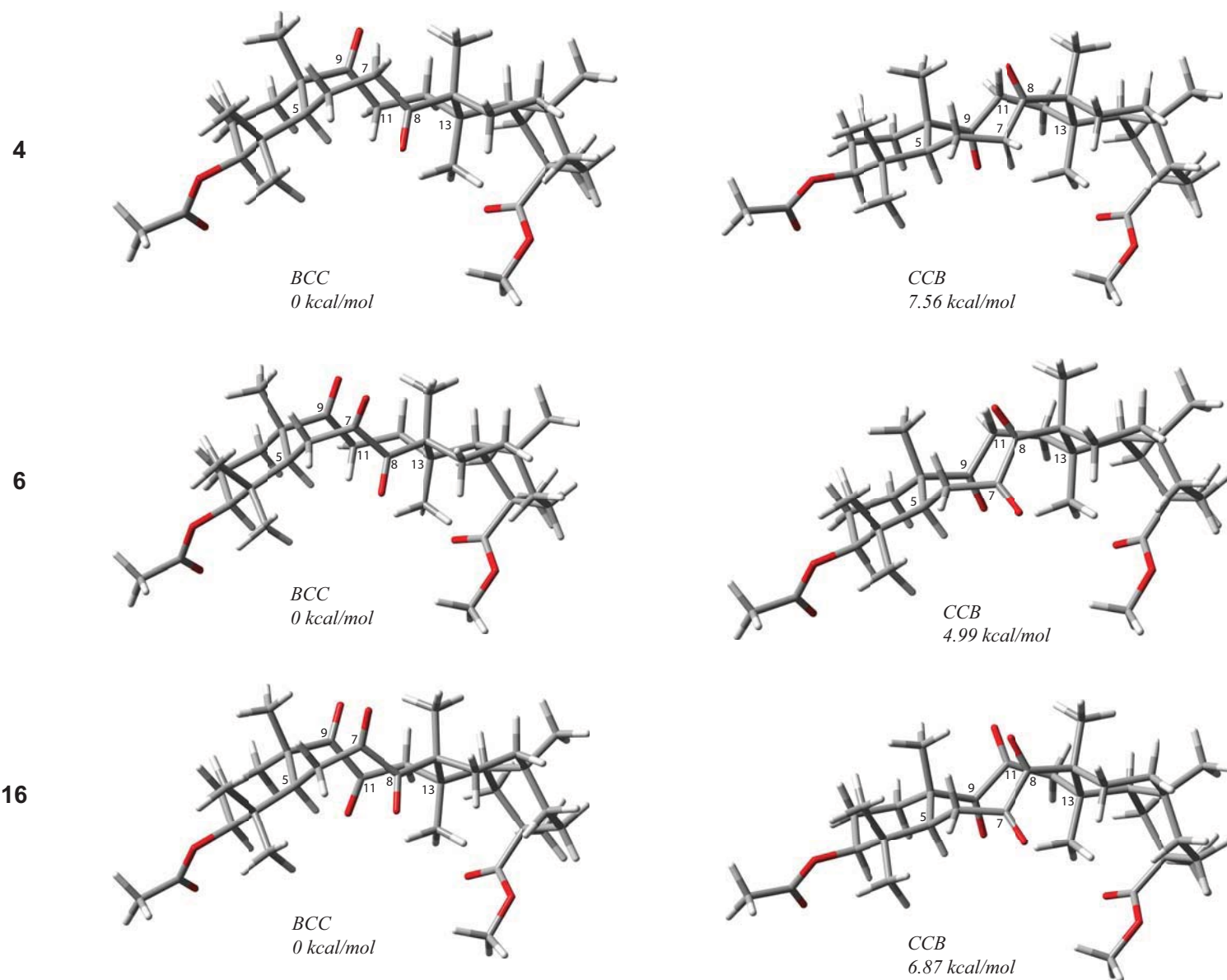
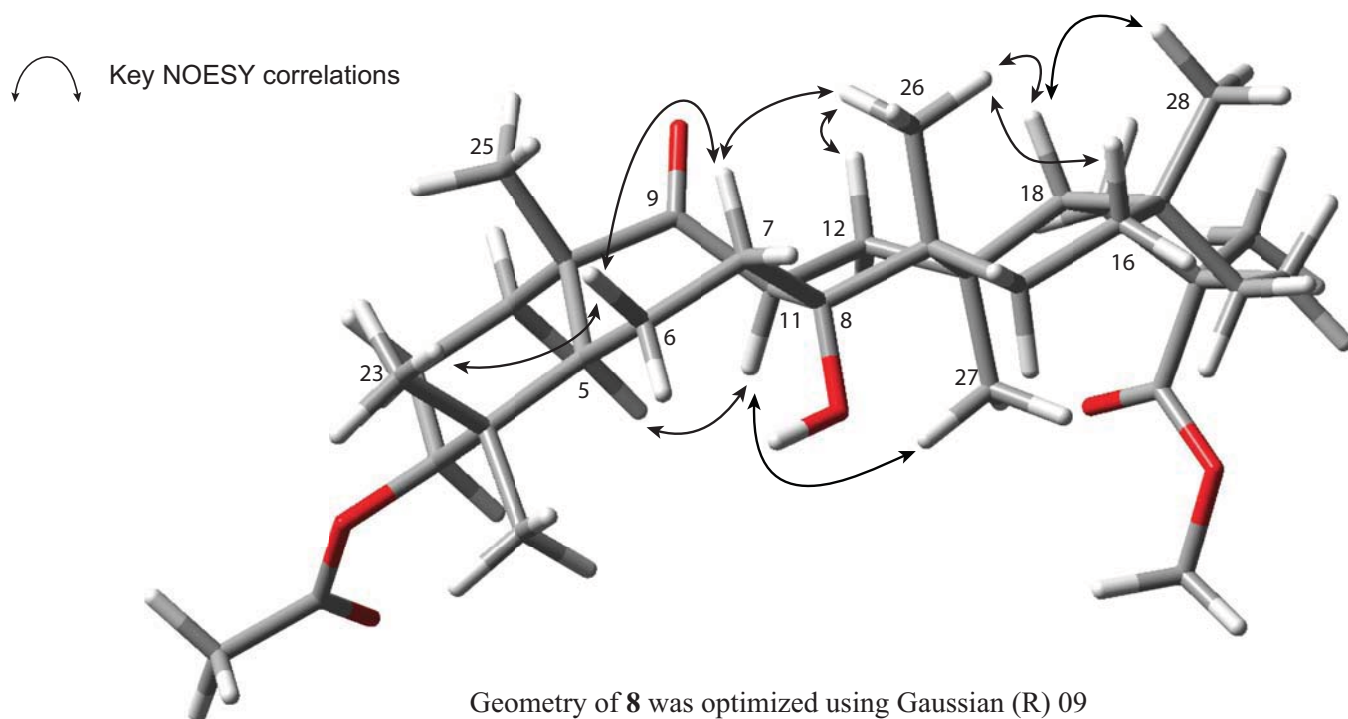
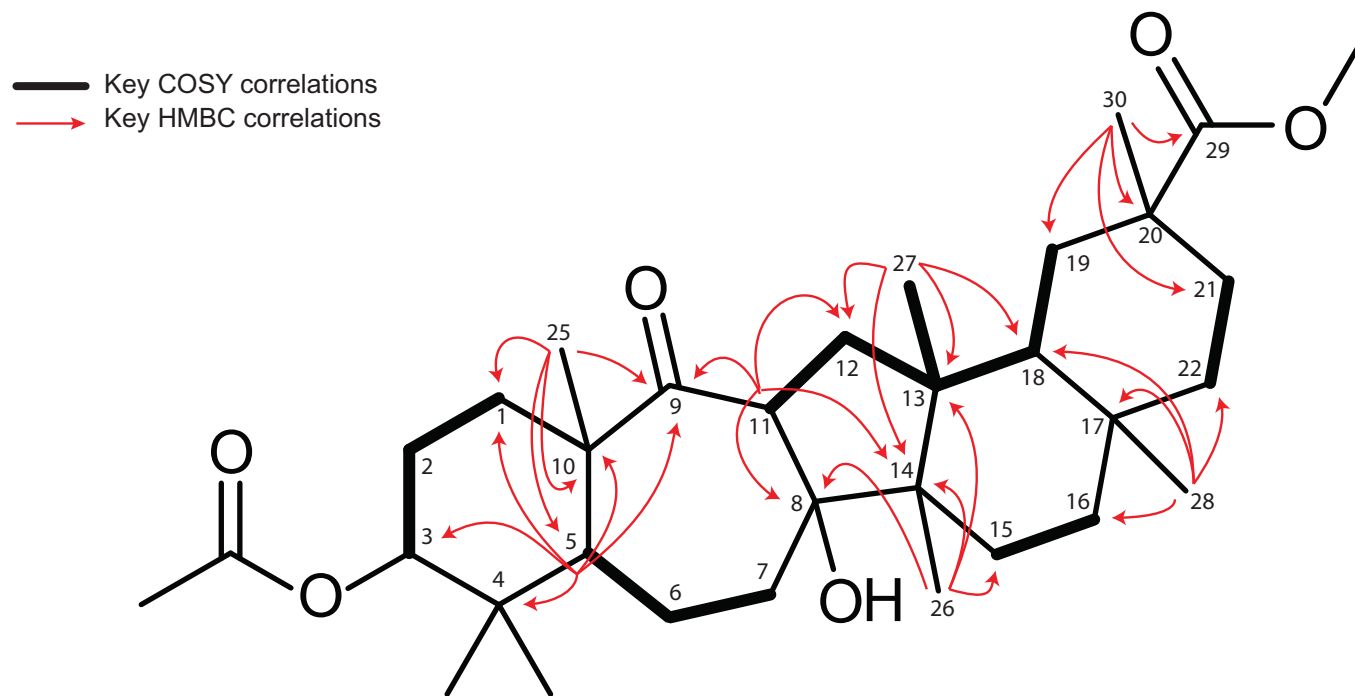
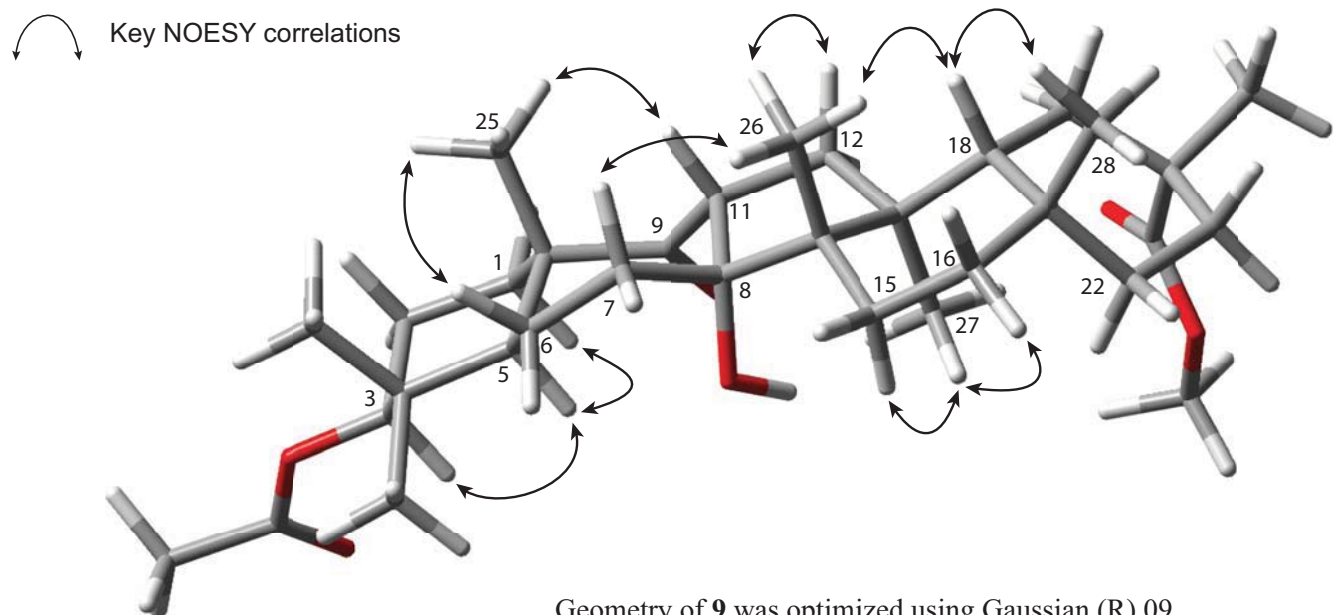
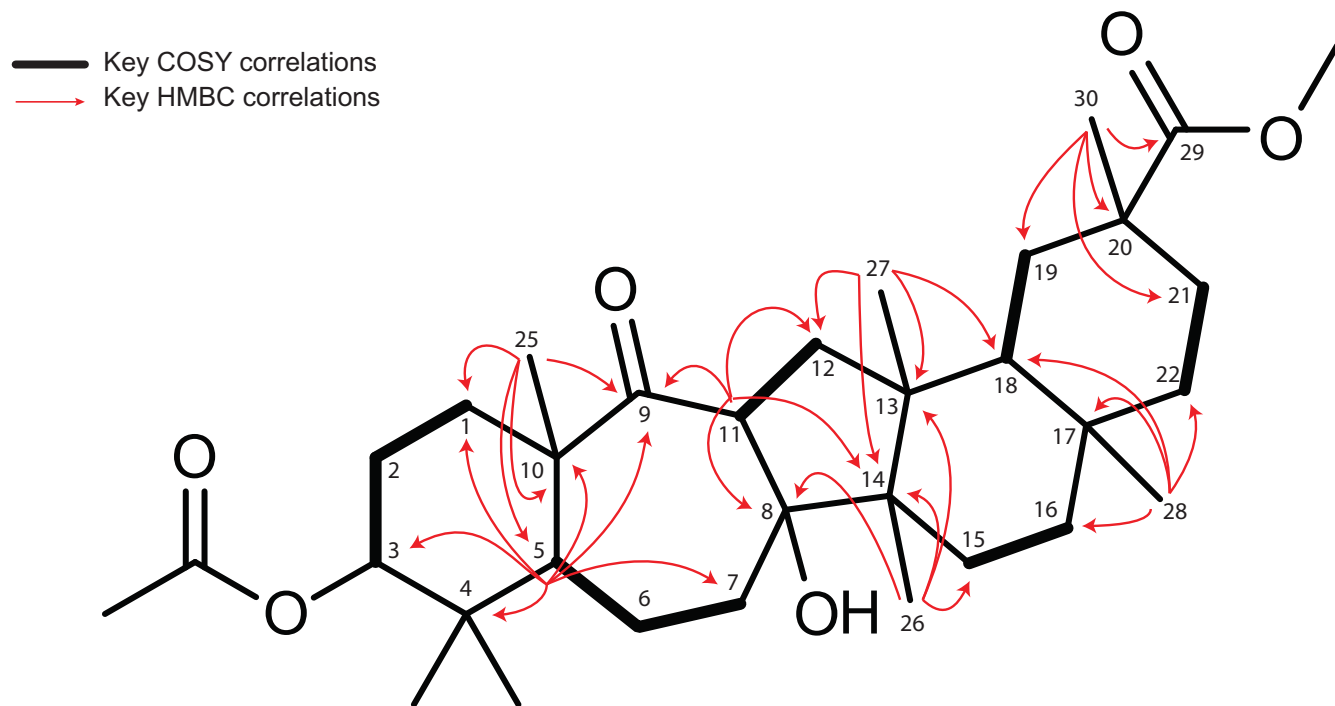


Figure S3. Key COSY, HMBC and NOESY correlations of **8**.



Geometry of **8** was optimized using Gaussian (R) 09 with B3LYP/6-311G(d,p).

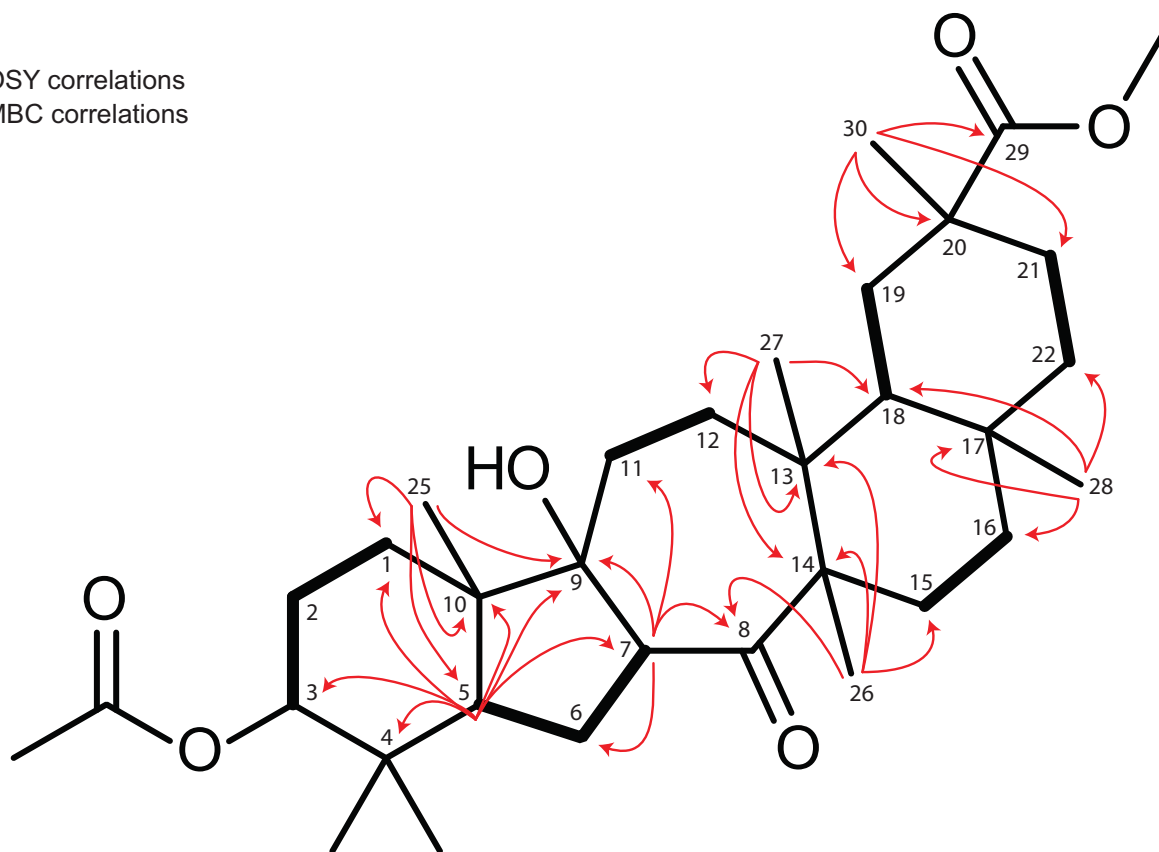
Figure S4. Key COSY, HMBC and NOESY correlations of **9**.



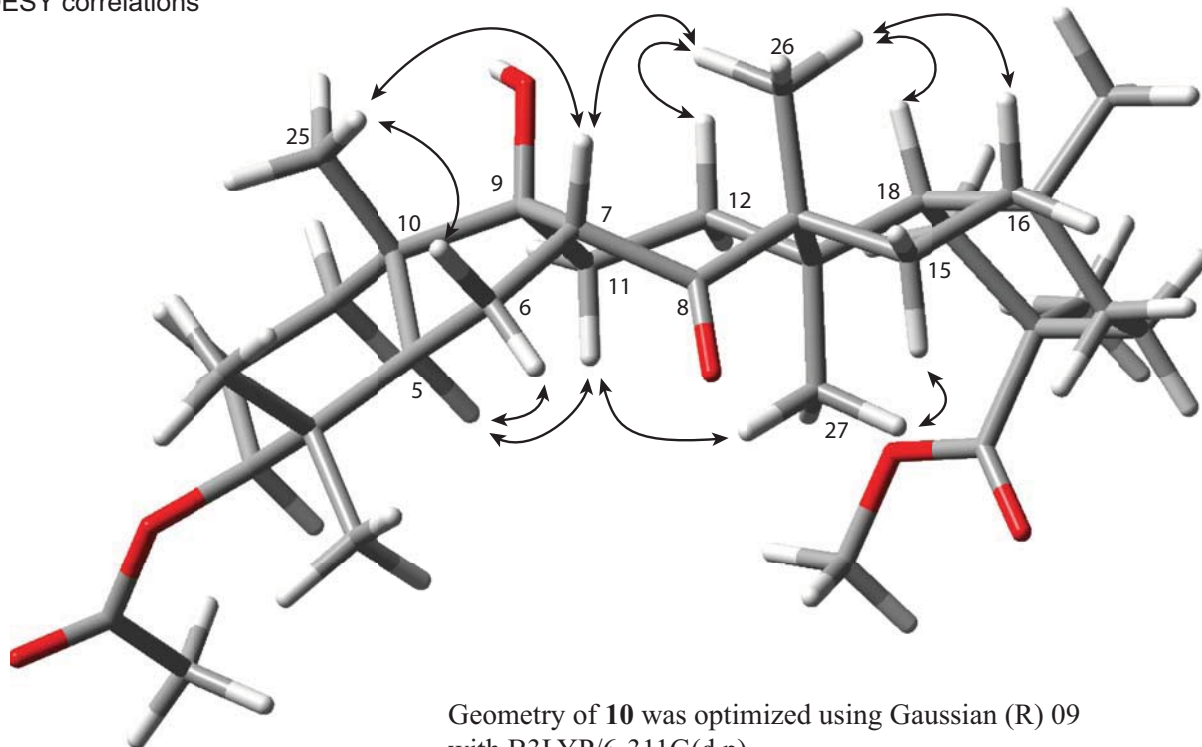
Geometry of **9** was optimized using Gaussian (R) 09 with B3LYP/6-311G(d,p).

Figure S5. Key COSY, HMBC and NOESY correlations of **10**.

— Key COSY correlations
→ Key HMBC correlations



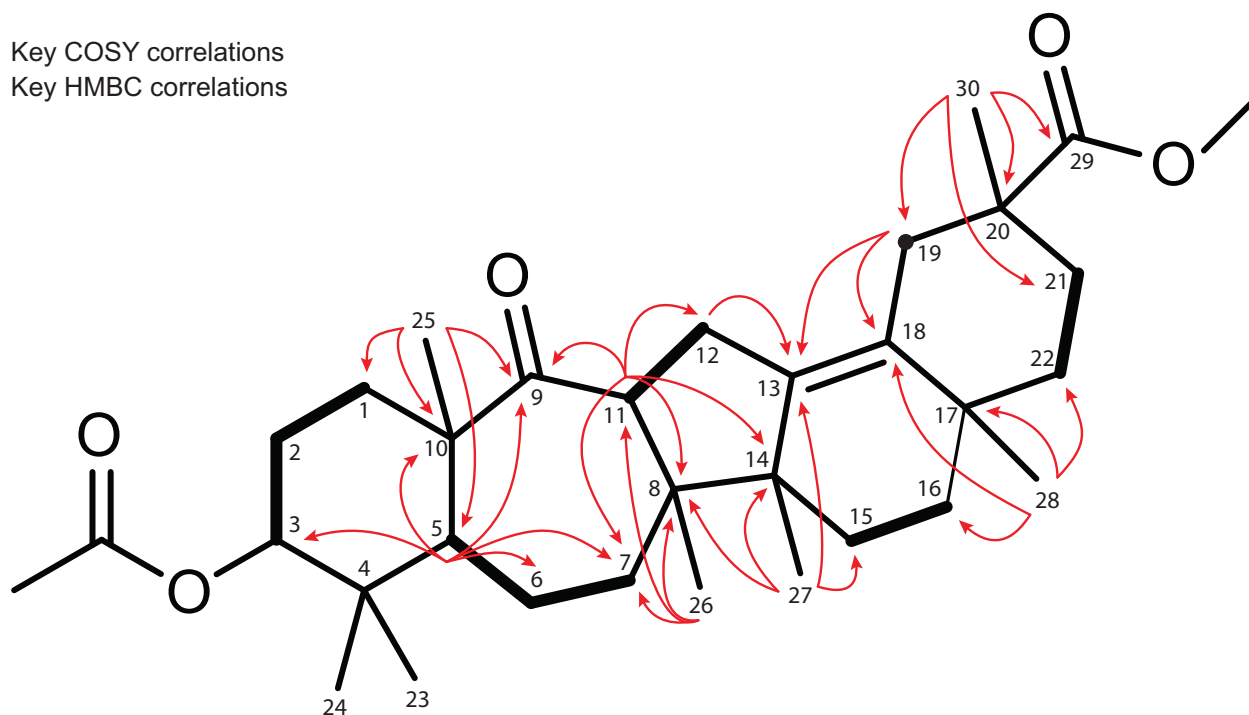
↔ Key NOESY correlations



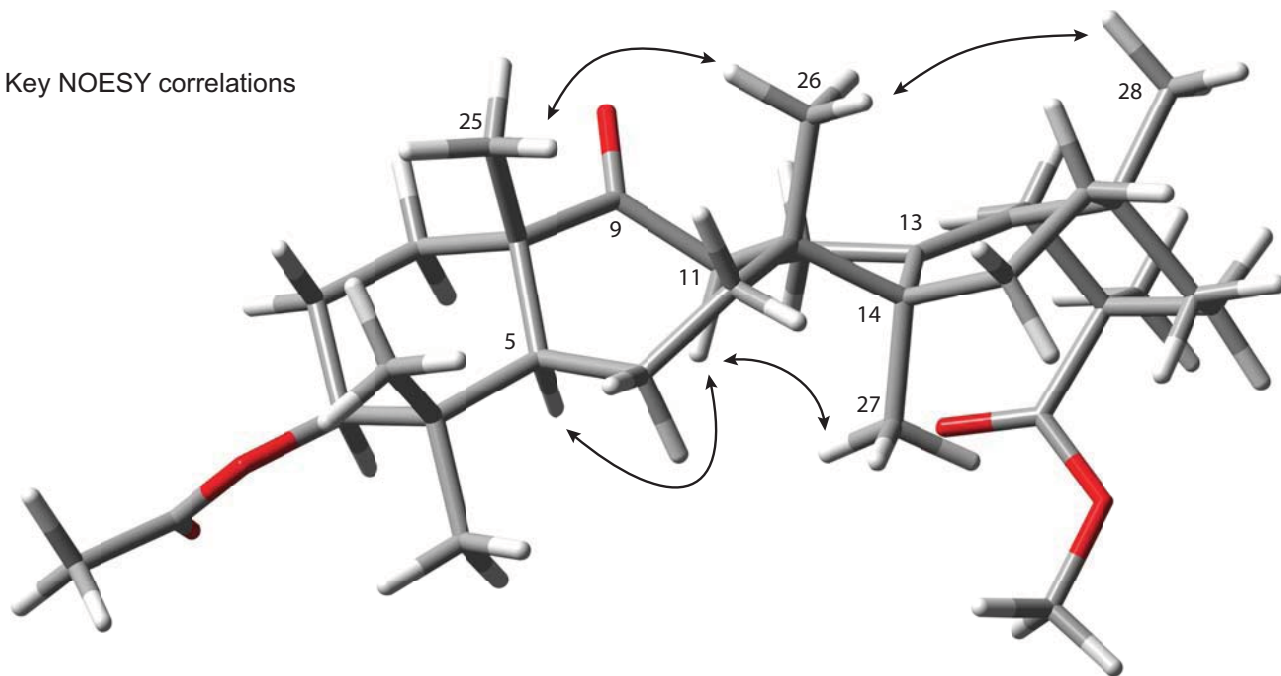
Geometry of **10** was optimized using Gaussian (R) 09 with B3LYP/6-311G(d,p).

Figure S6. Key COSY, HMBC and NOESY correlations of **12**.

— Key COSY correlations
→ Key HMBC correlations

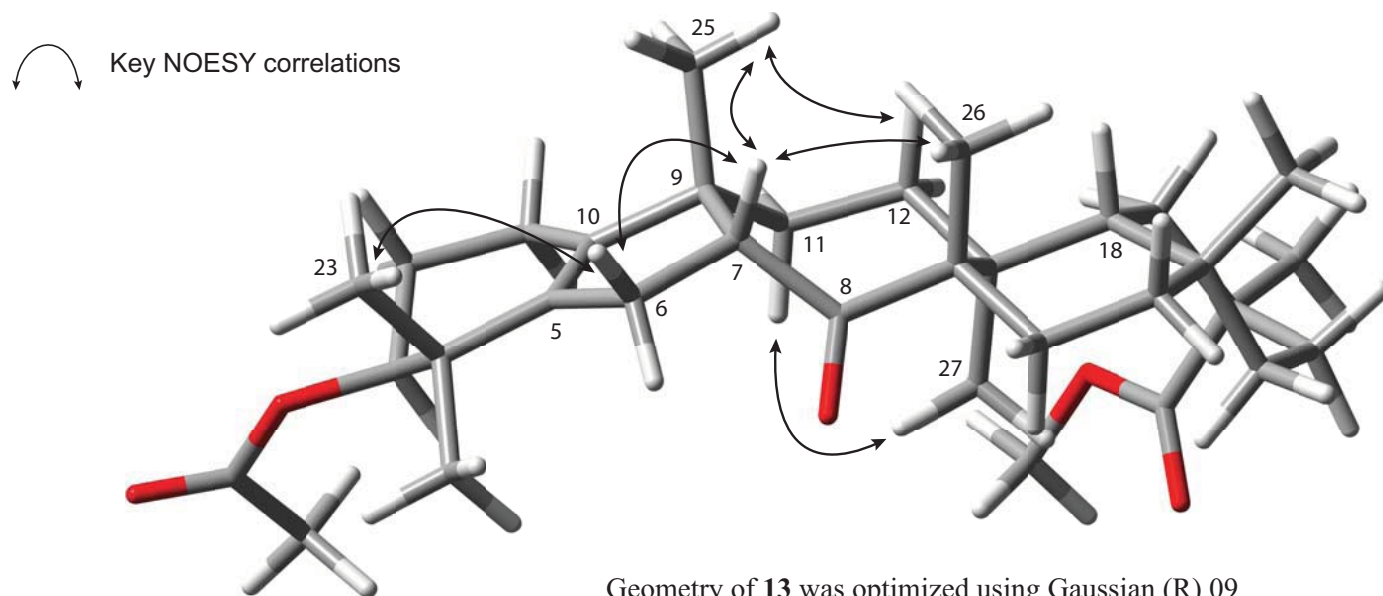
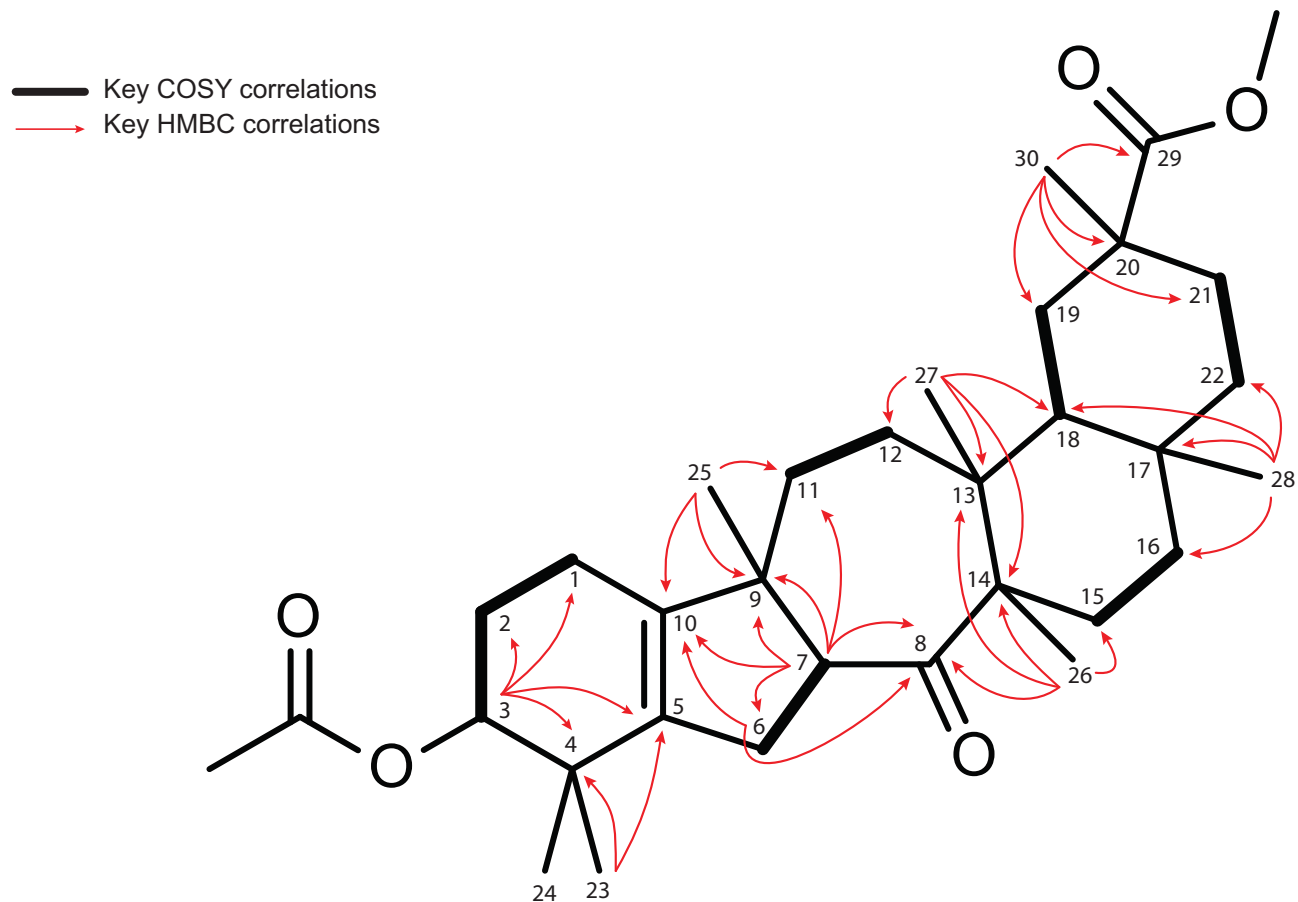


↔ Key NOESY correlations



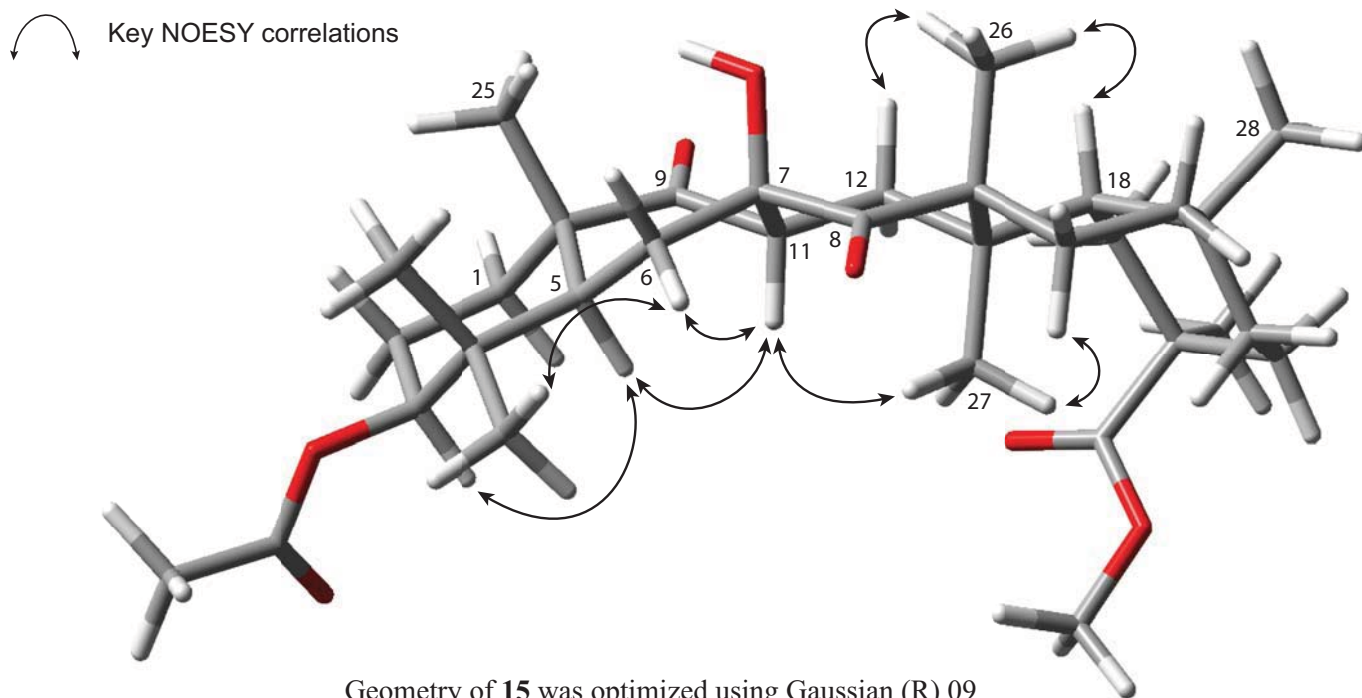
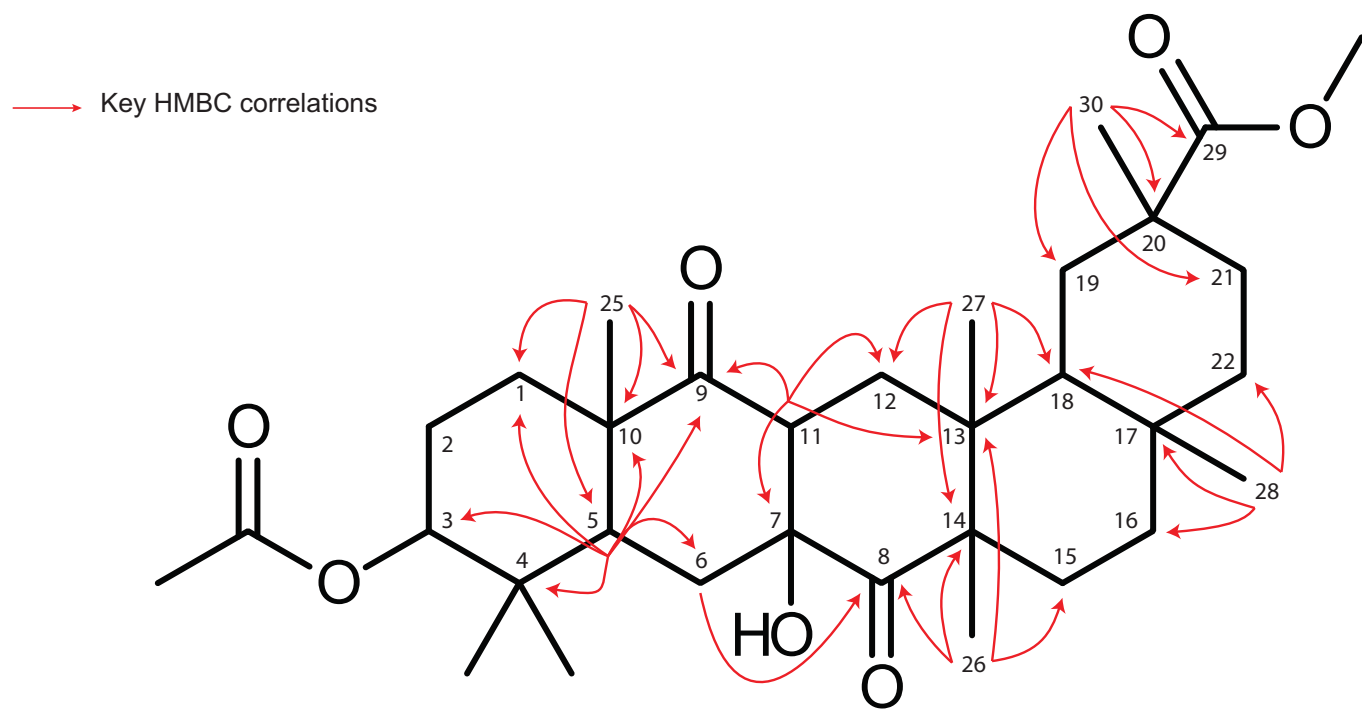
Geometry of **12** was optimized using Gaussian (R) 09 with B3LYP/6-311G(d,p).

Figure S7. Key COSY, HMBC and NOESY correlations of **13** .



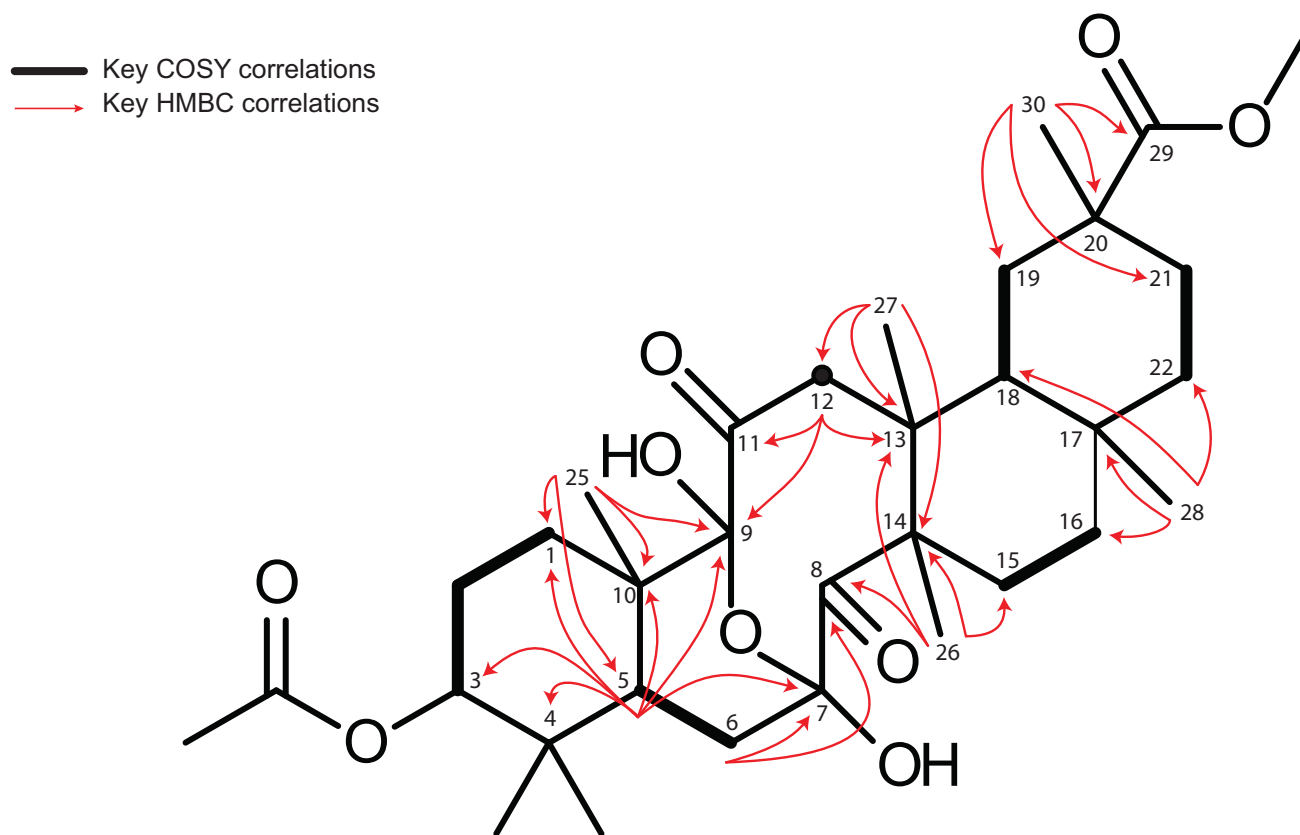
Geometry of **13** was optimized using Gaussian (R) 09 with B3LYP/6-311G(d,p).

Figure S8. Key HMBC and NOESY correlations of **15**.



Geometry of **15** was optimized using Gaussian (R) 09 with B3LYP/6-311G(d,p).

Figure S9. Key COSY, HMBC and NOESY correlations of **19**.



↷ Key NOESY correlations

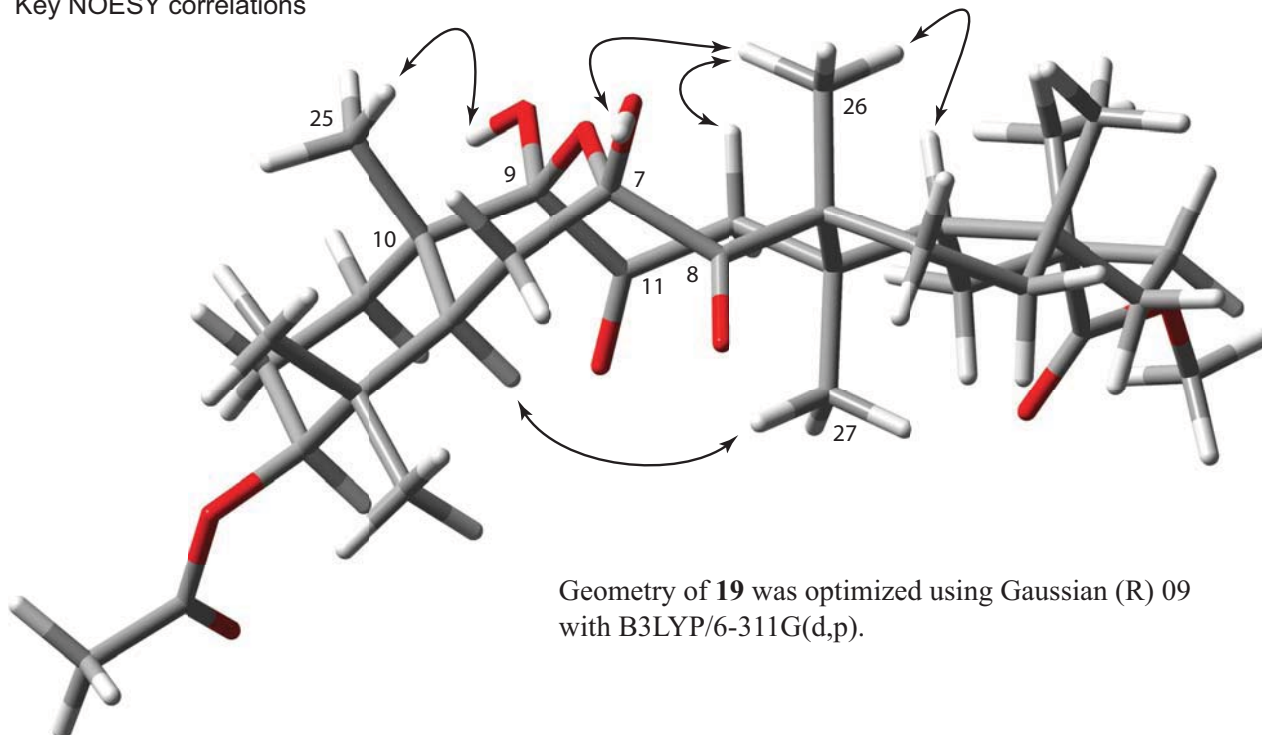
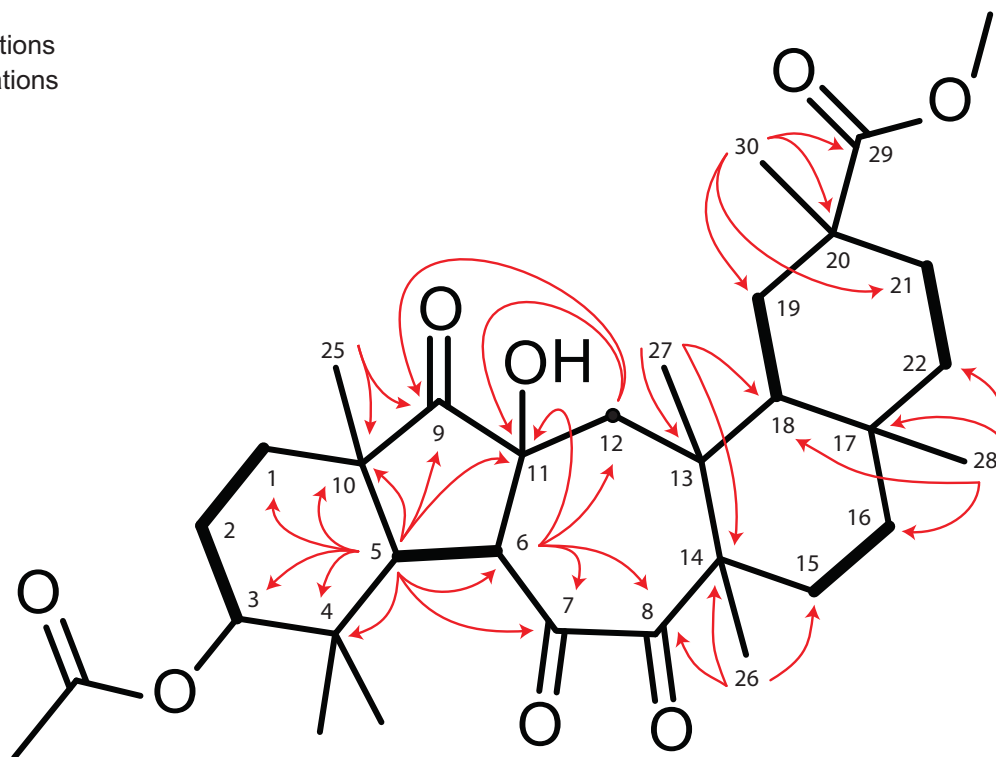
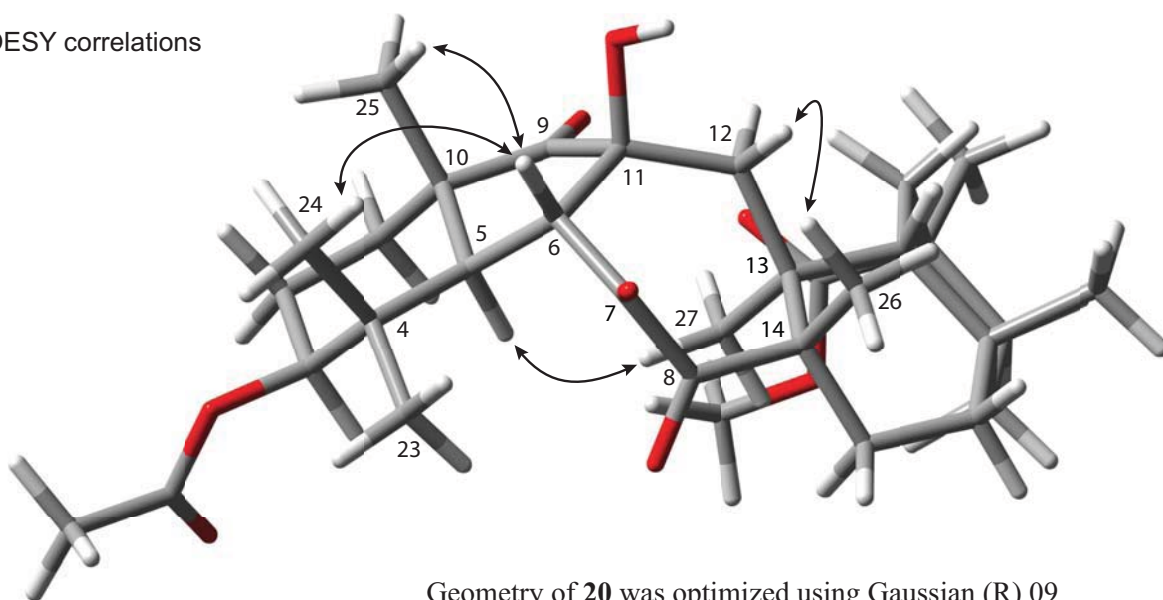


Figure S10. Key COSY, HMBC and NOESY correlations of **20**.

— Key COSY correlations
→ Key HMBC correlations



↷ Key NOESY correlations



Geometry of **20** was optimized using Gaussian (R) 09 with B3LYP/6-311G(d,p).

Figure S11. Key HMBC correlations of 6 .

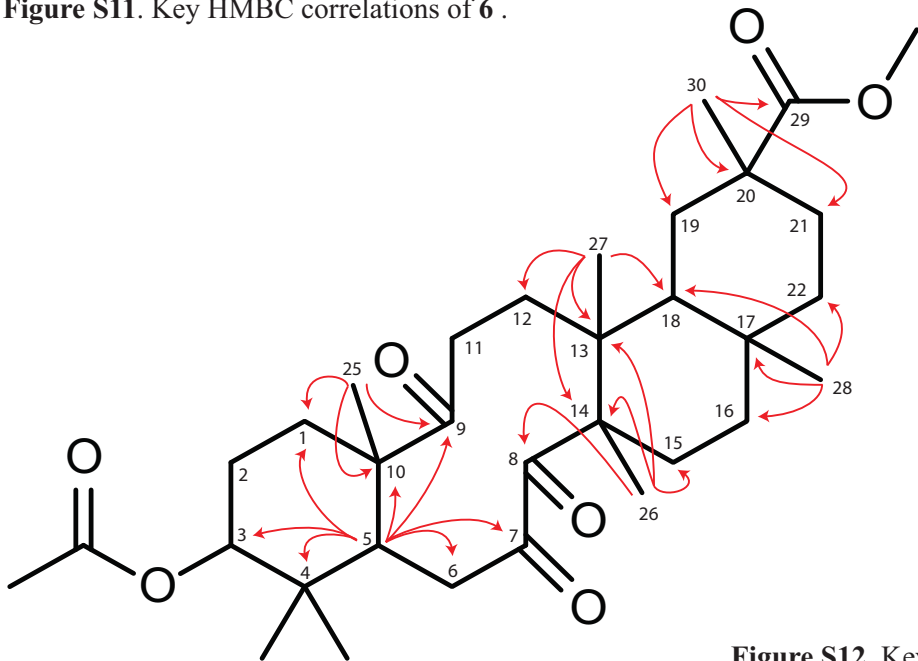


Figure S12. Key HMBC correlations of 14 .

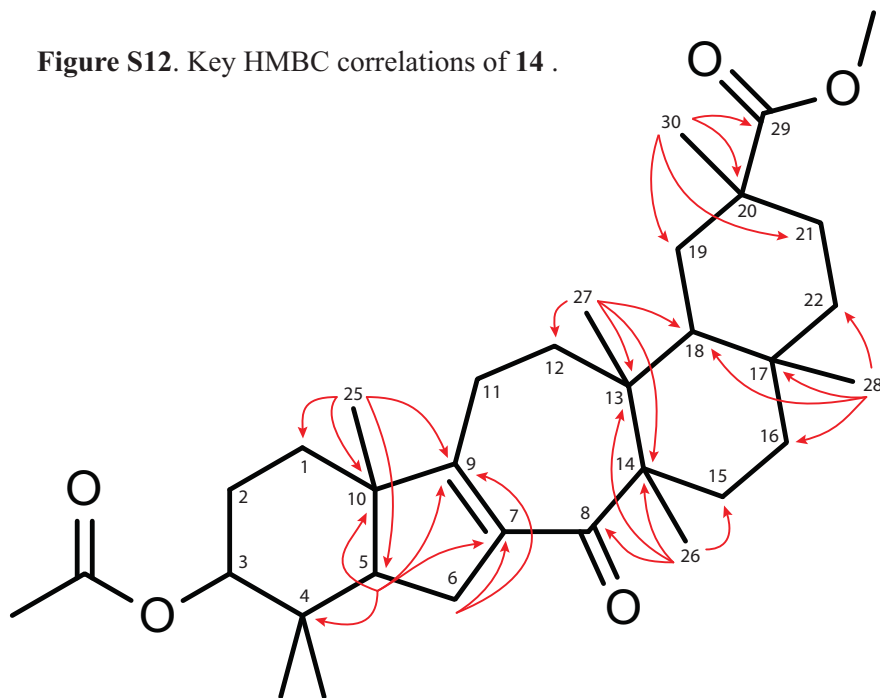


Figure S13. Key COSY and HMBC correlations of 17 .

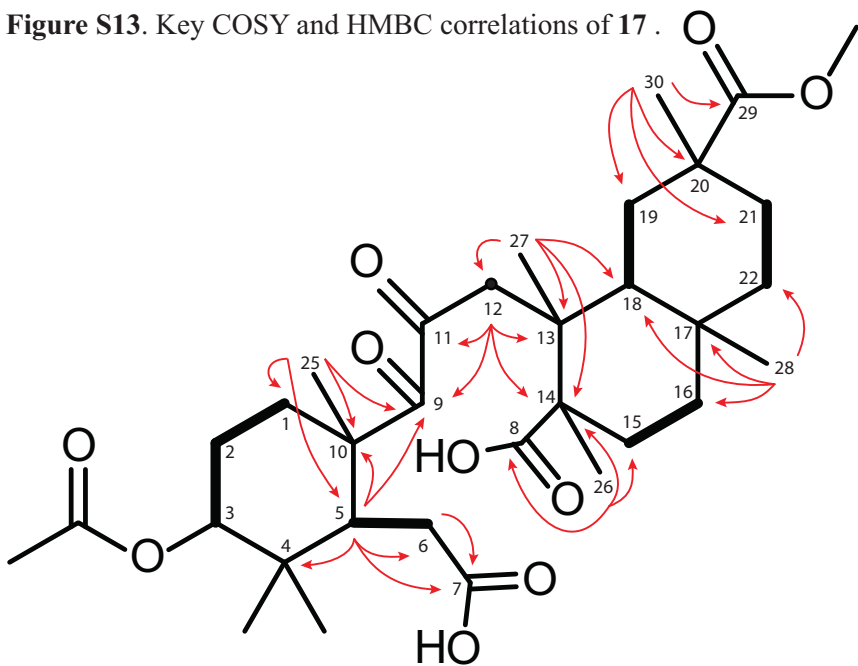


Figure S14. ¹H and ¹³C spectra of 2.

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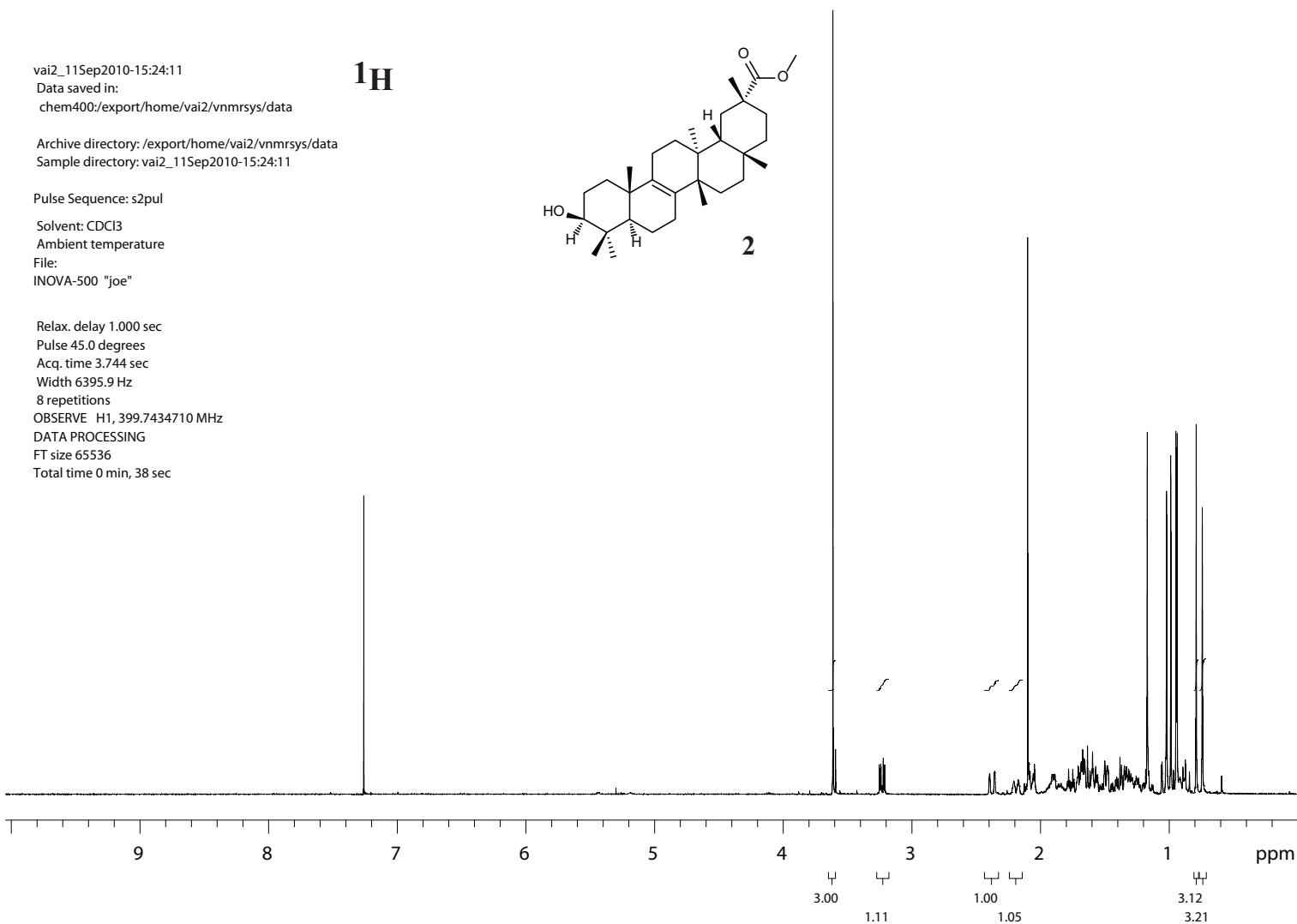
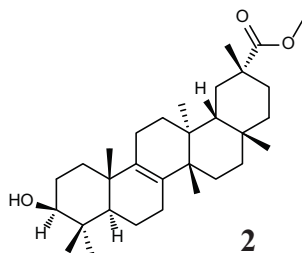
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Solvent: CDCl₃
Ambient temperature
File:
INOVA-500 "joe"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 6395.9 Hz
8 repetitions
OBSERVE H1, 399.7434710 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 38 sec

¹H



STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: C13
INOVA-500 "joe"

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
192 repetitions
OBSERVE C13, 150.8466510 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 44 min, 41 sec

¹³C

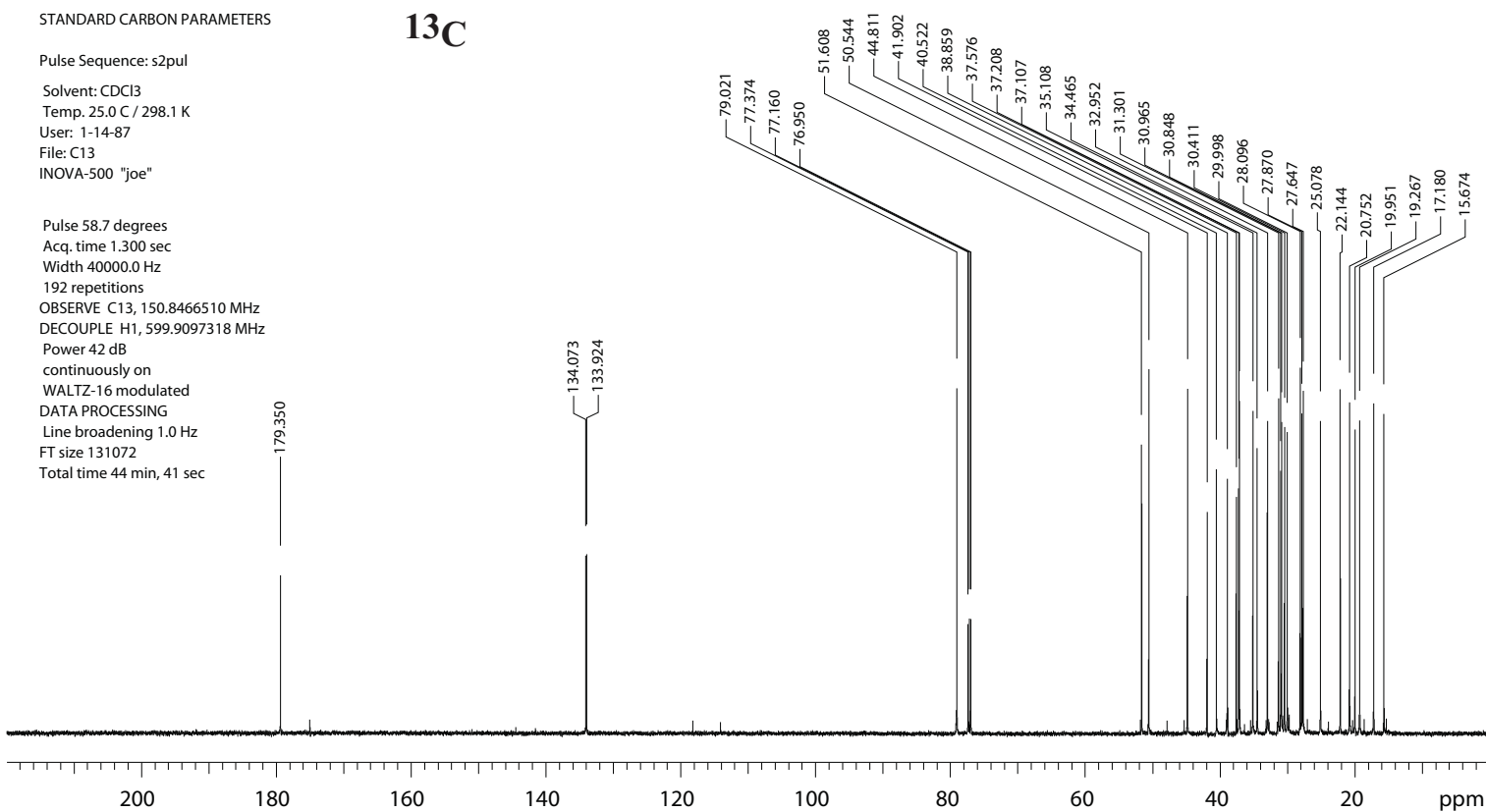


Figure S15. ¹H and ¹³C spectra of 3.

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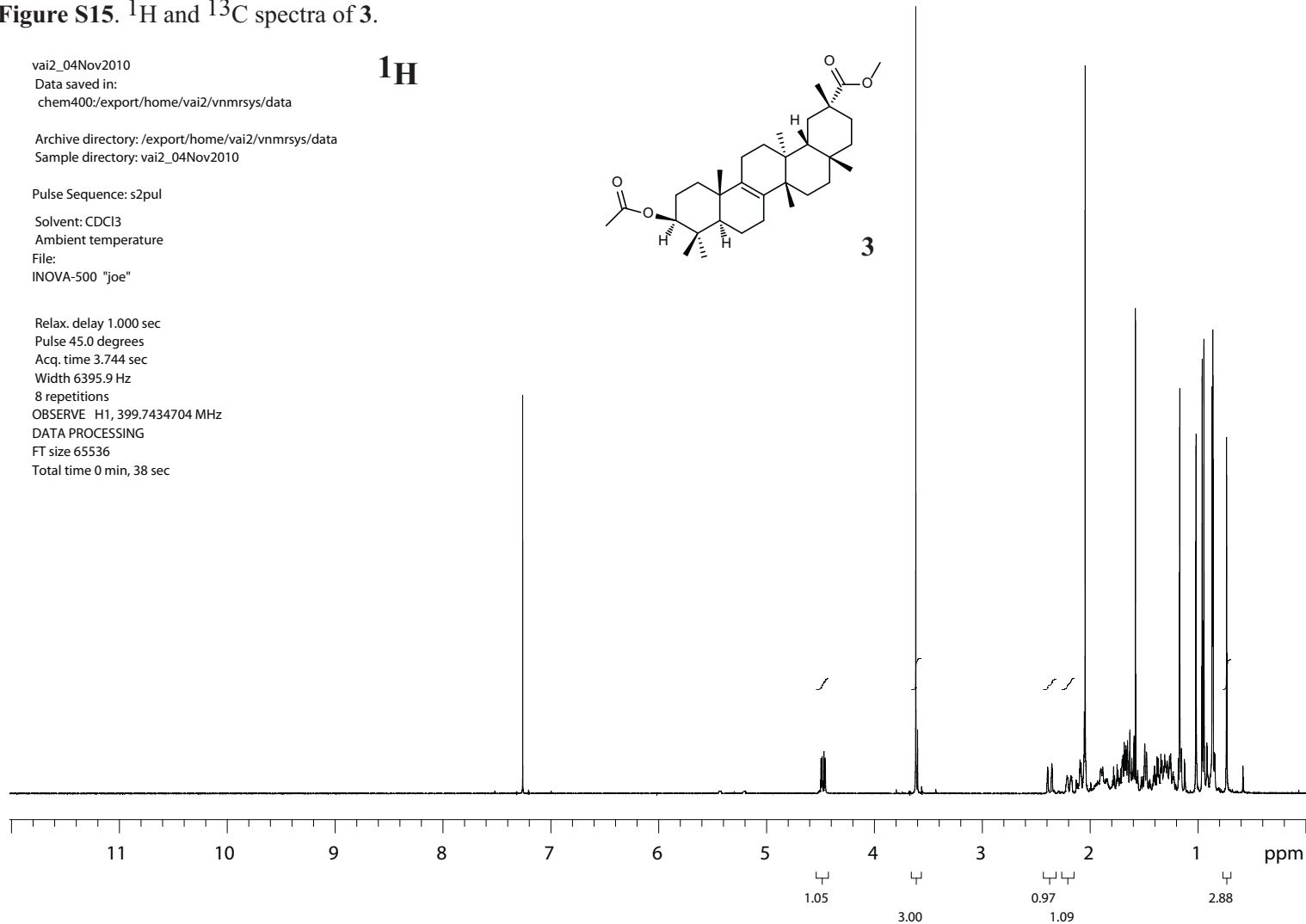
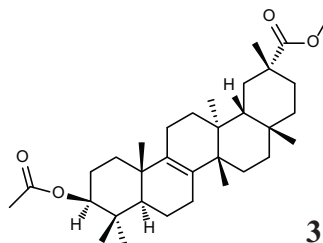
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Solvent: CDCl₃
Ambient temperature
File:
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Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 6395.9 Hz
8 repetitions
OBSERVE H1, 399.7434704 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 38 sec

¹H



vai2_06Jul2010-12:14:41

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Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File:
INOVA-500 "joe"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
256 repetitions
OBSERVE C13, 100.5155623 MHz
DECOUPLE H1, 399.7454756 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 min, 25 sec

¹³C

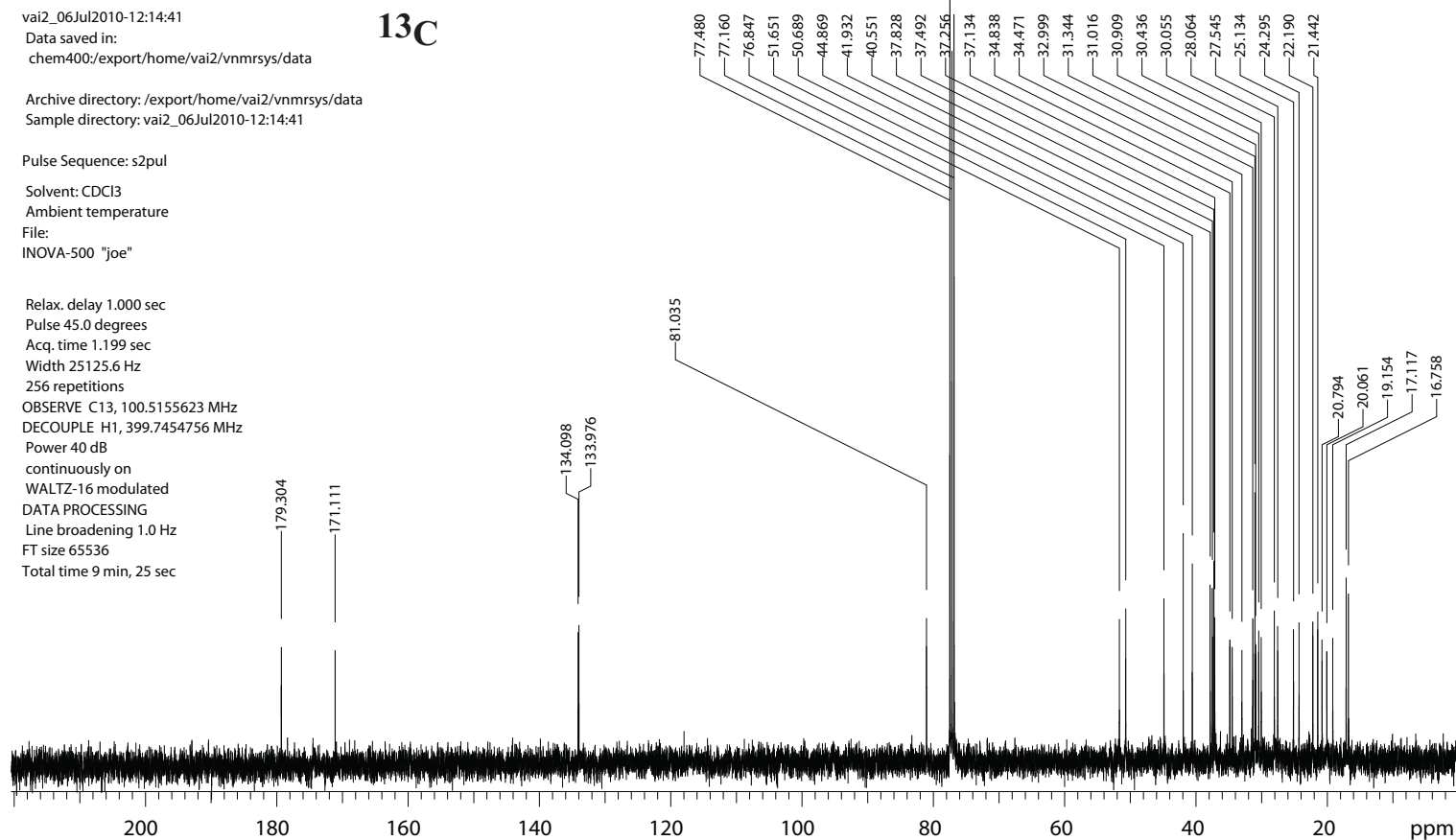


Figure S16. ¹H and ¹³C spectra of 4.

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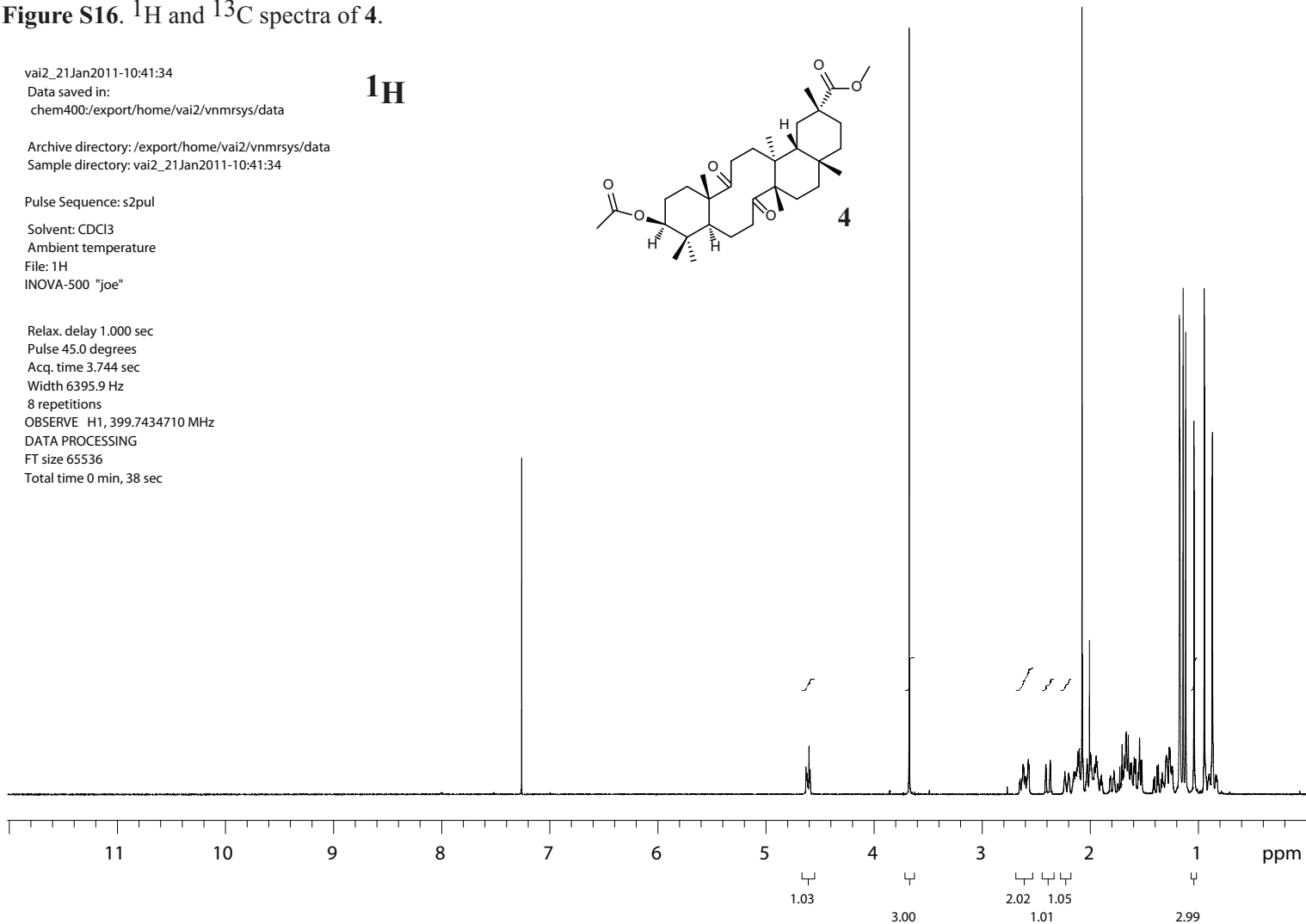
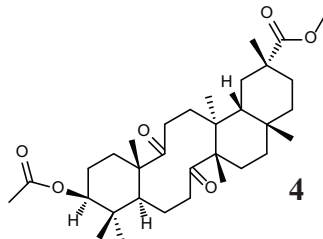
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Solvent: CDCl₃
Ambient temperature
File: 1H
INOVA-500 "joe"

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Pulse 45.0 degrees
Acq. time 3.744 sec
Width 6395.9 Hz
8 repetitions
OBSERVE H1, 399.7434710 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 38 sec

¹H



Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
320 repetitions
OBSERVE C13, 150.8466429 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz

¹³C

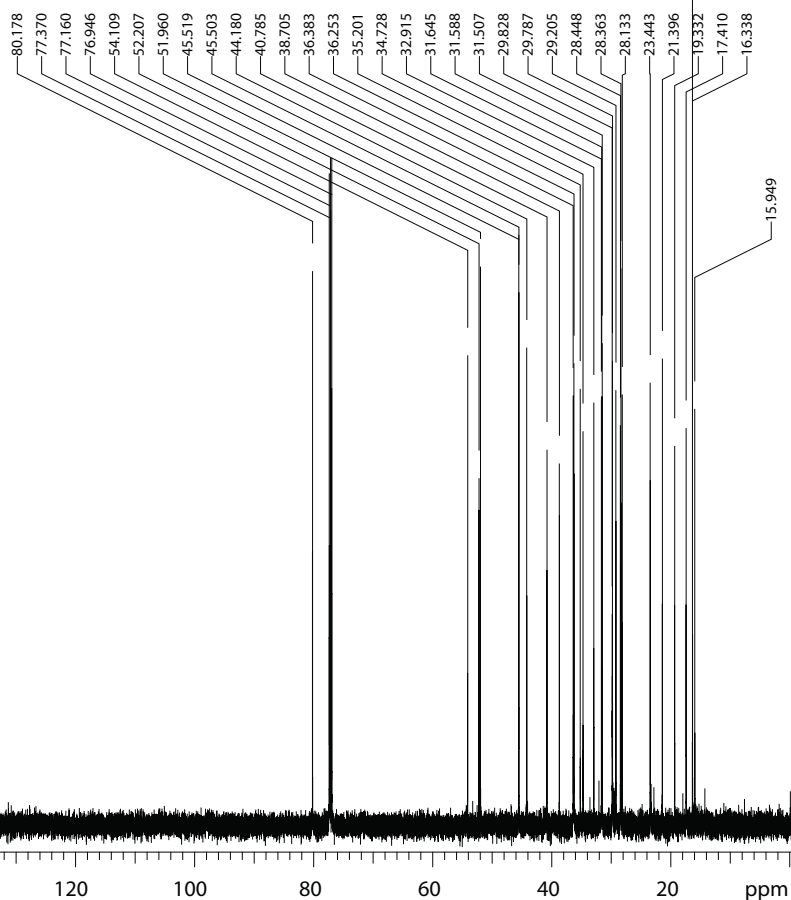


Figure S17. ¹H and ¹³C spectra of 6.

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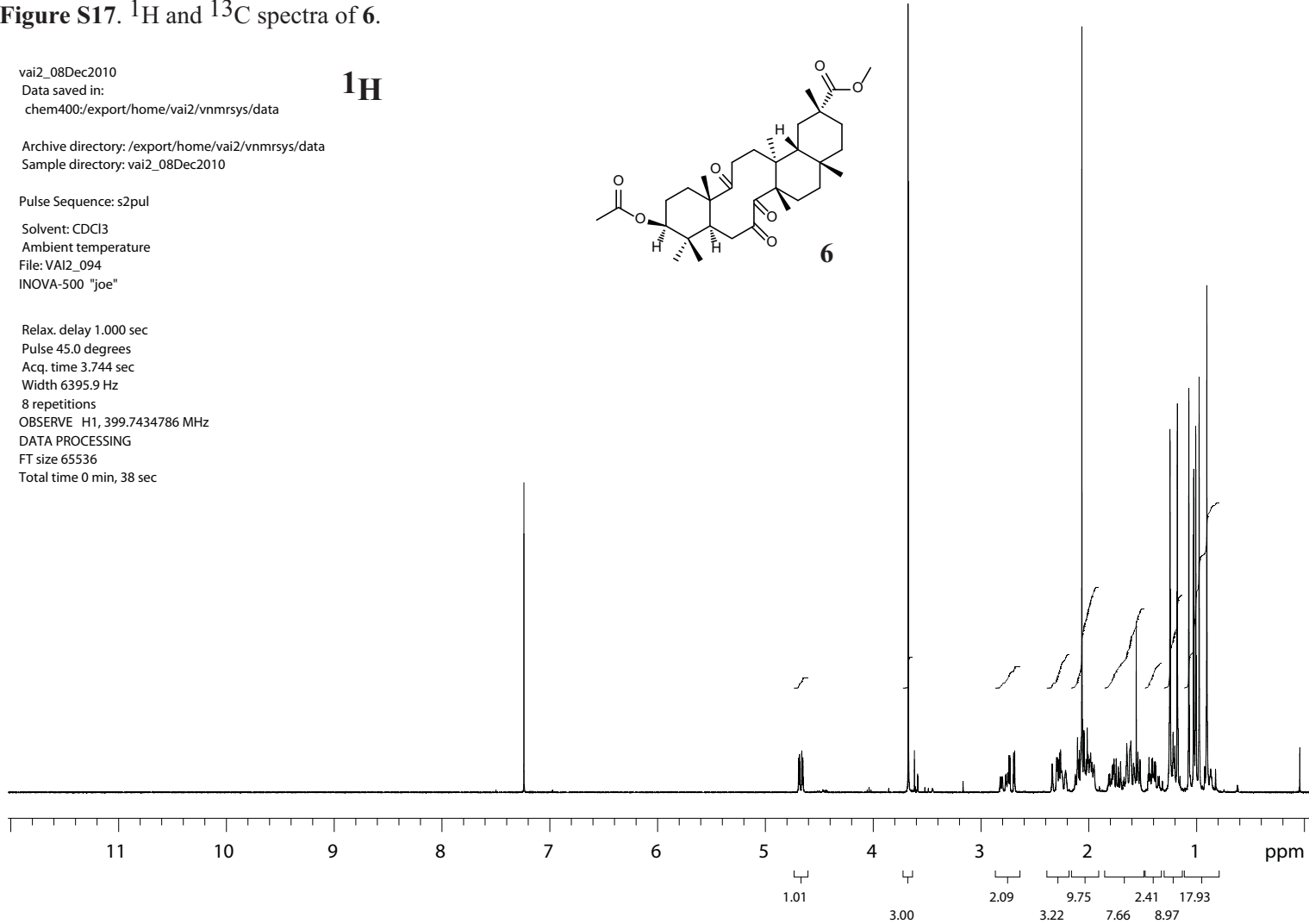
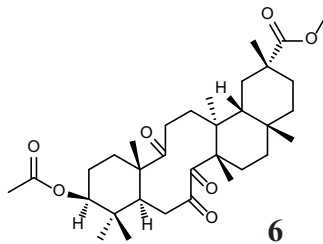
¹H

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Ambient temperature
File: VAI2_094
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8 repetitions
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Total time 0 min, 38 sec



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¹³C

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Solvent: CDCl₃
Ambient temperature

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Pulse 45.0 degrees
Acq. time 1.199 sec
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832 repetitions
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DECOUPLE H1, 399.7454756 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz

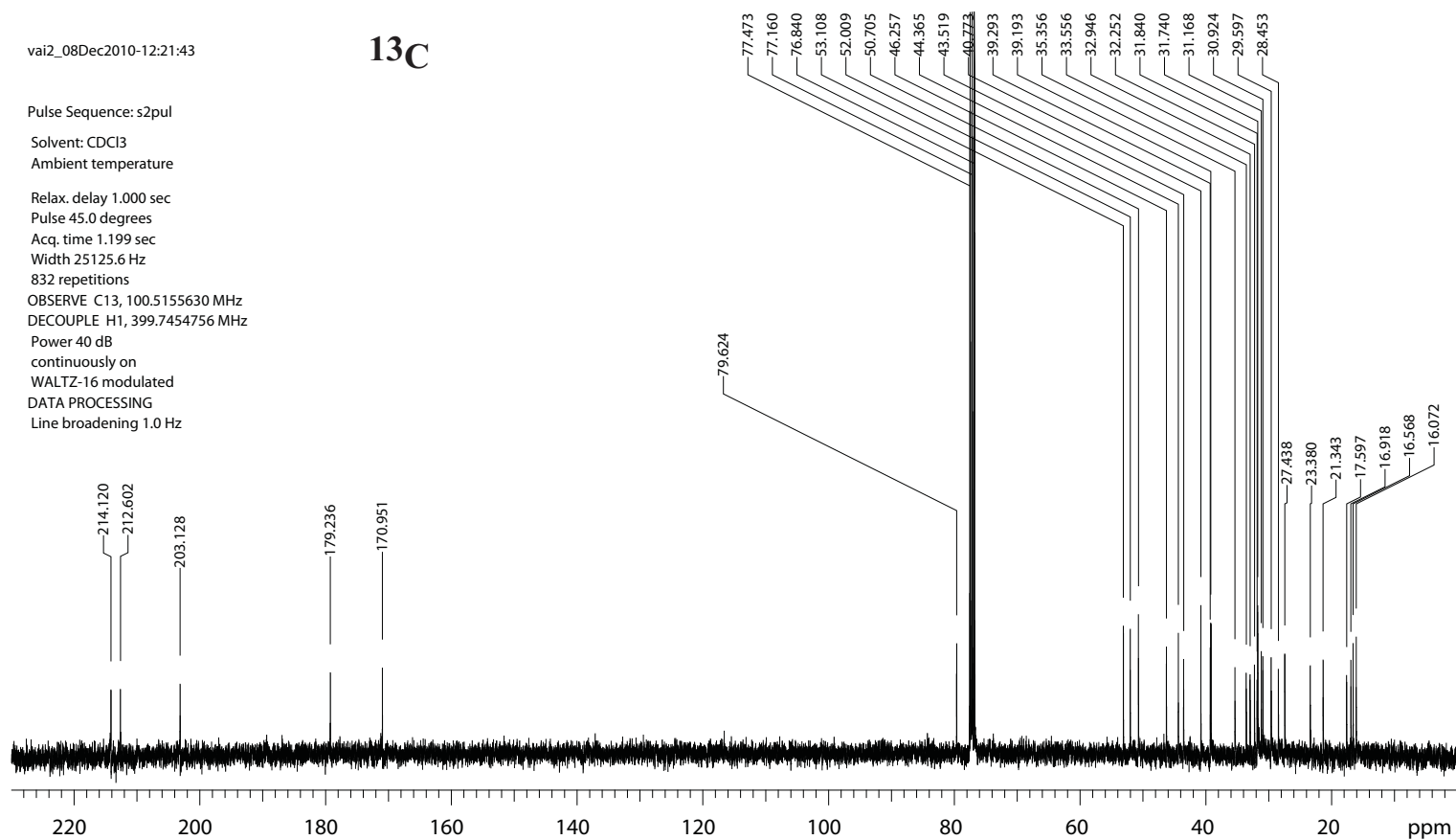
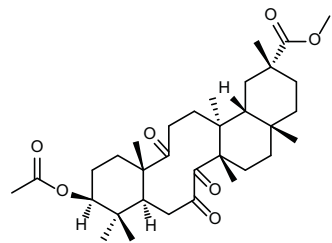


Figure S18. HMQC and HMBC spectra of **6**.



6

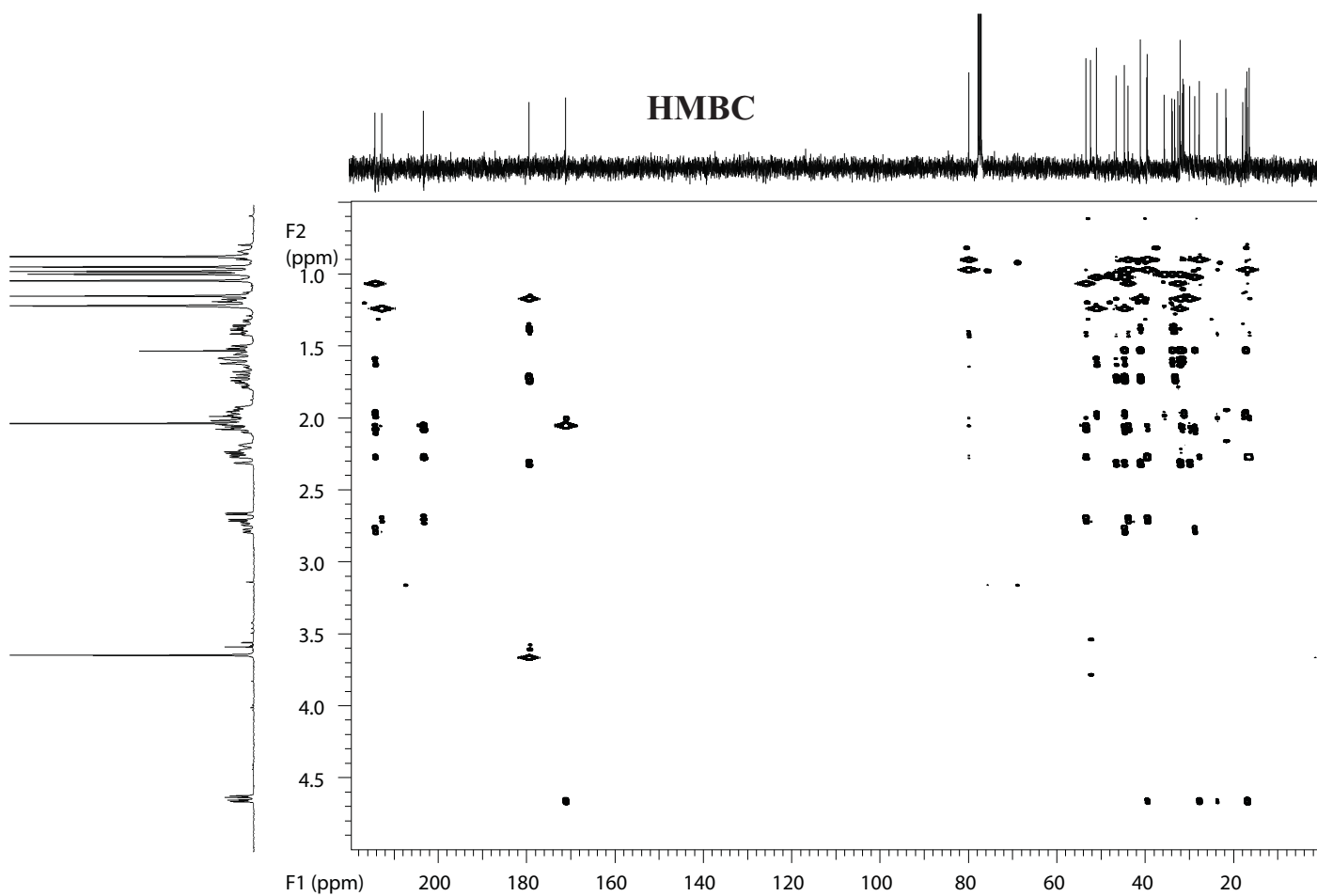
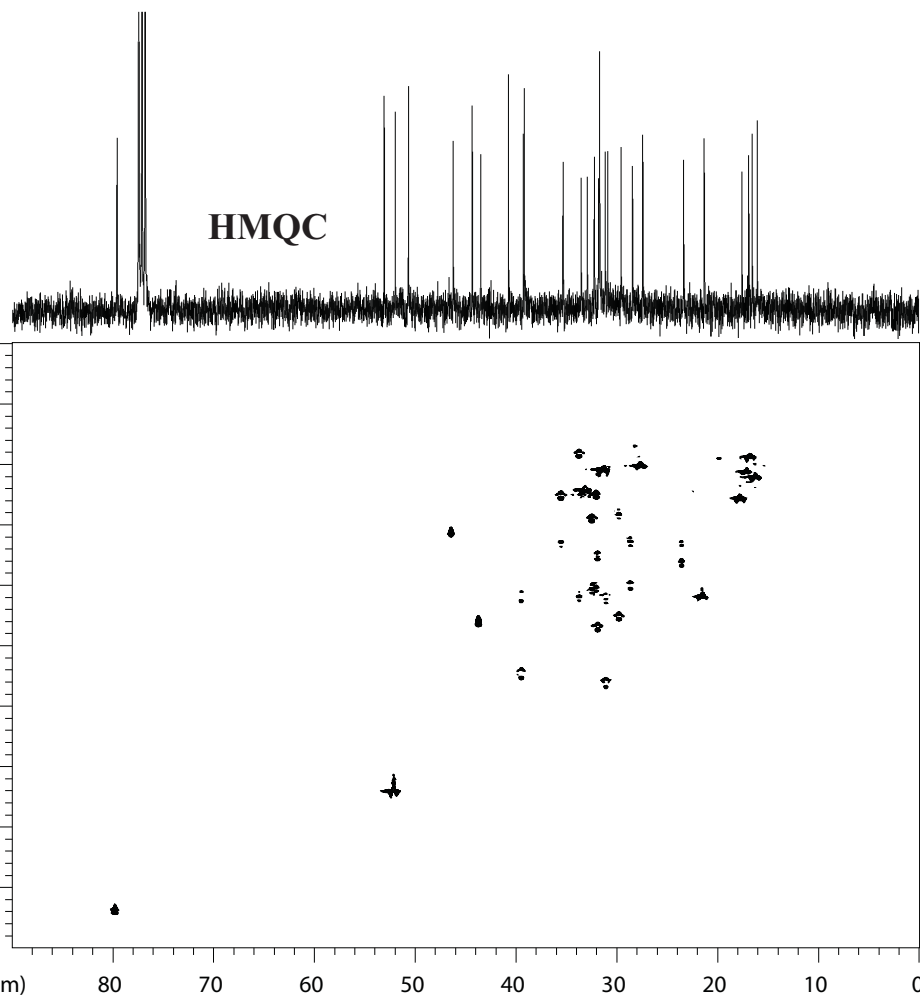


Figure S19. ¹H and ¹³C spectra of 7.

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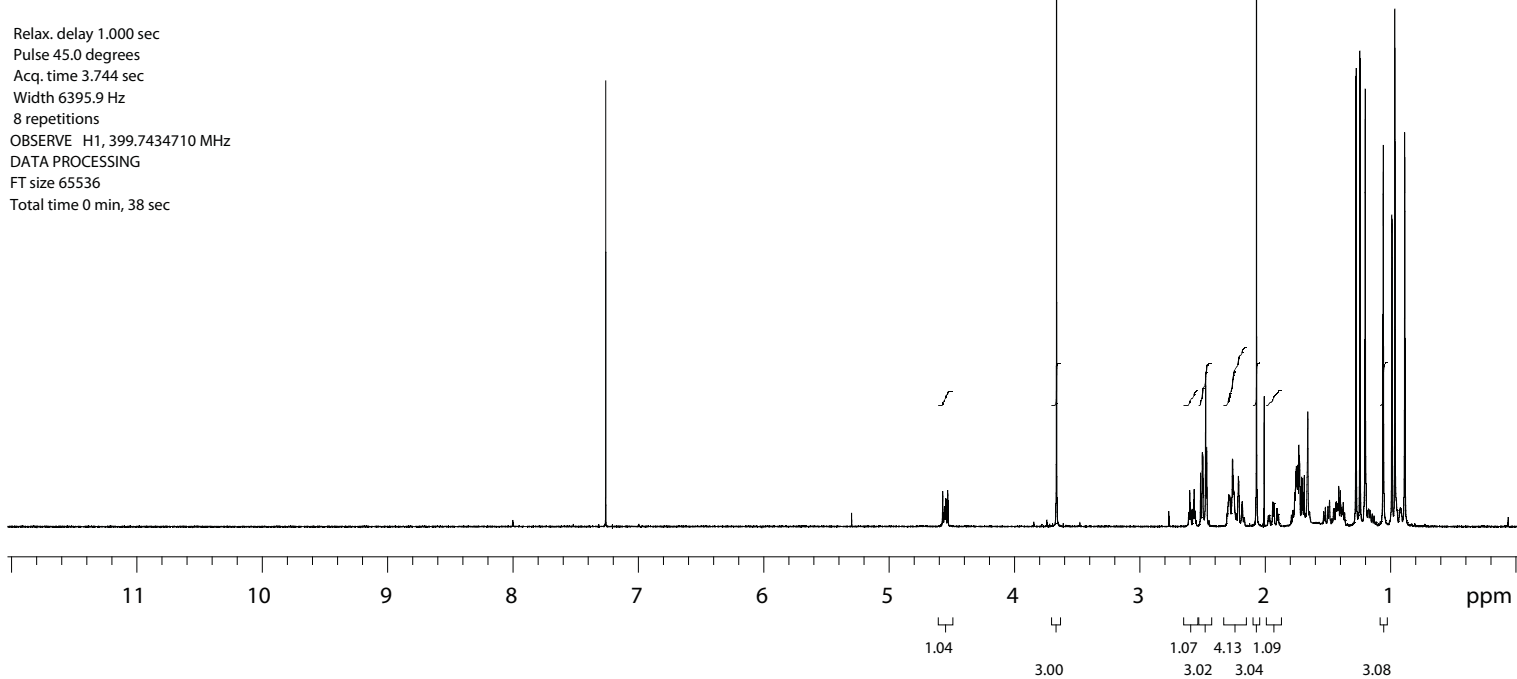
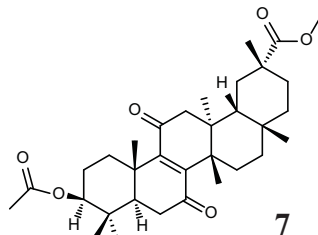
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Ambient temperature
File: 1H
INOVA-500 "joe"

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Pulse 45.0 degrees
Acq. time 3.744 sec
Width 6395.9 Hz
8 repetitions
OBSERVE H1, 399.7434710 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 38 sec

¹H



Sample 101

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: 13C
INOVA-500 "joe"

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
640 repetitions
OBSERVE C13, 150.8466417 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 1 hr, 29 min, 10 sec

¹³C

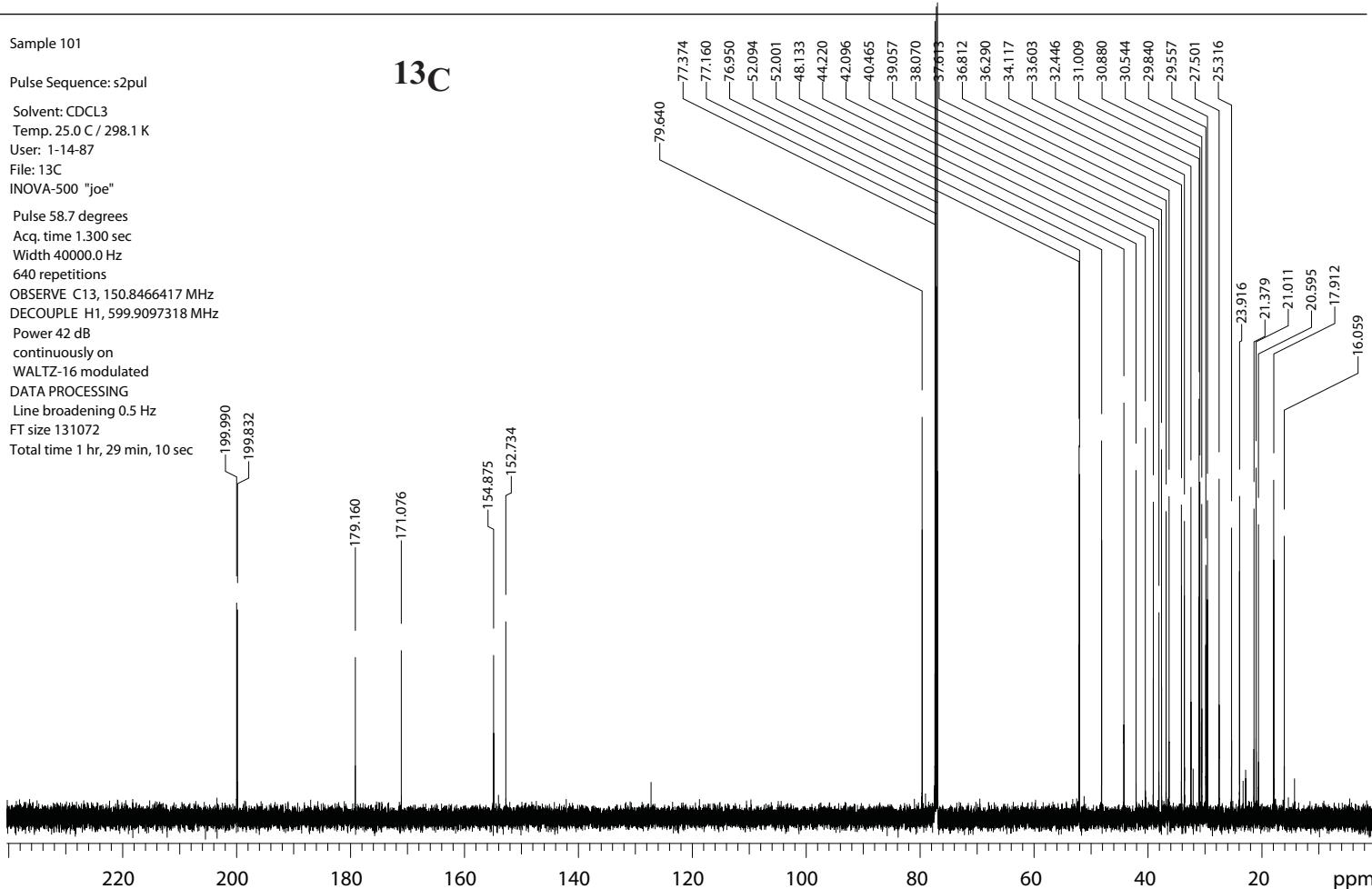


Figure S20. ¹H and ¹³C spectra of 8

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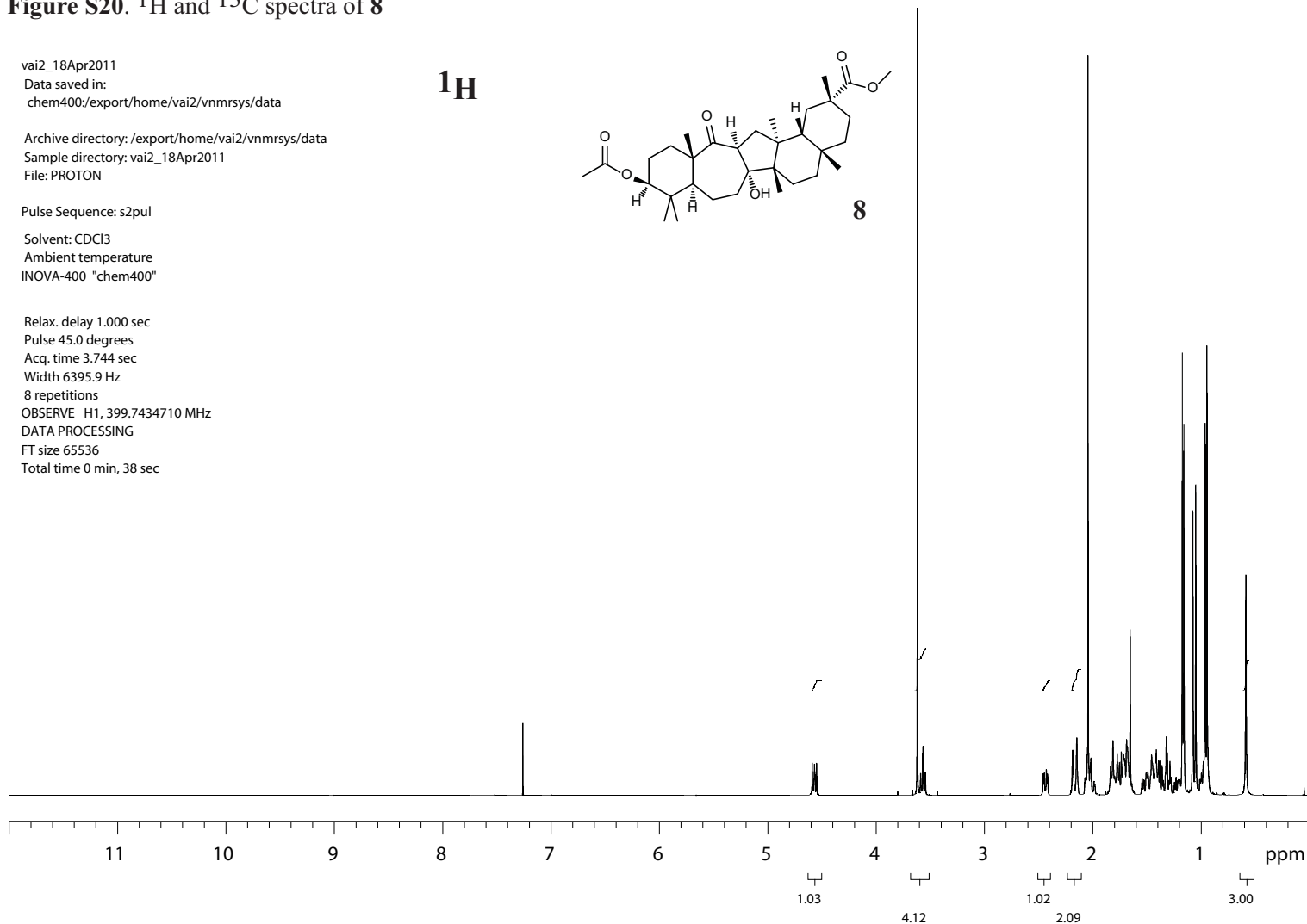
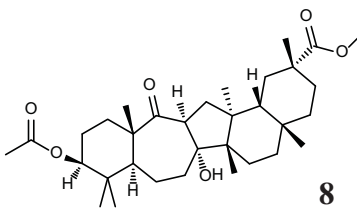
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 File: PROTON

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Solvent: CDCl₃
 Ambient temperature
 INOVA-400 "chem400"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.744 sec
 Width 6395.9 Hz
 8 repetitions
 OBSERVE H1, 399.7434710 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 38 sec

¹H



Pulse Sequence: s2pul

Solvent: CDCl₃
 Temp. 25.0 C / 298.1 K
 Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 160 repetitions
 OBSERVE C13, 150.8466450 MHz
 DECOUPLE H1, 599.9097318 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz

¹³C

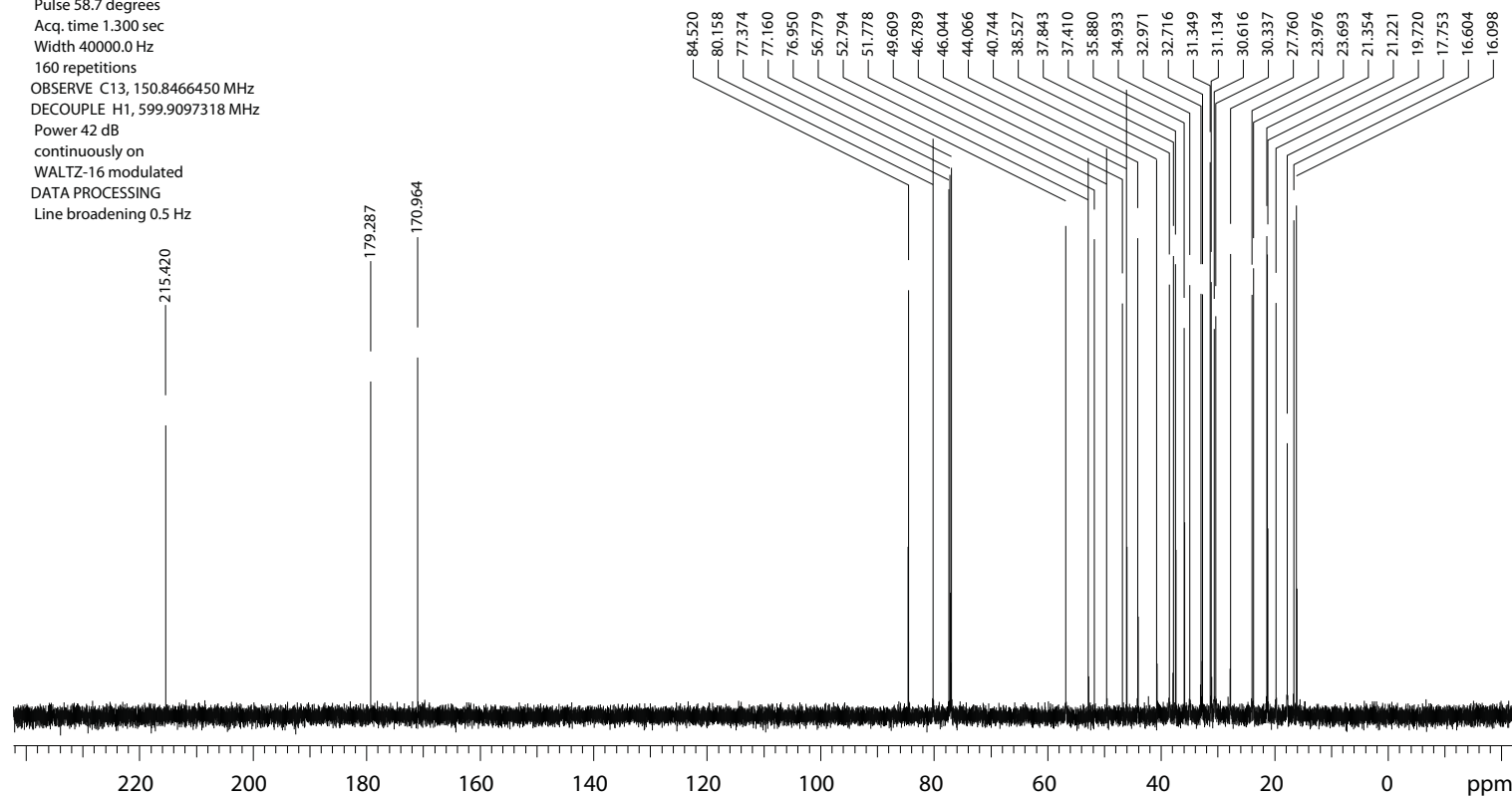


Figure S21. HMQC and HMBC spectra of **8**.

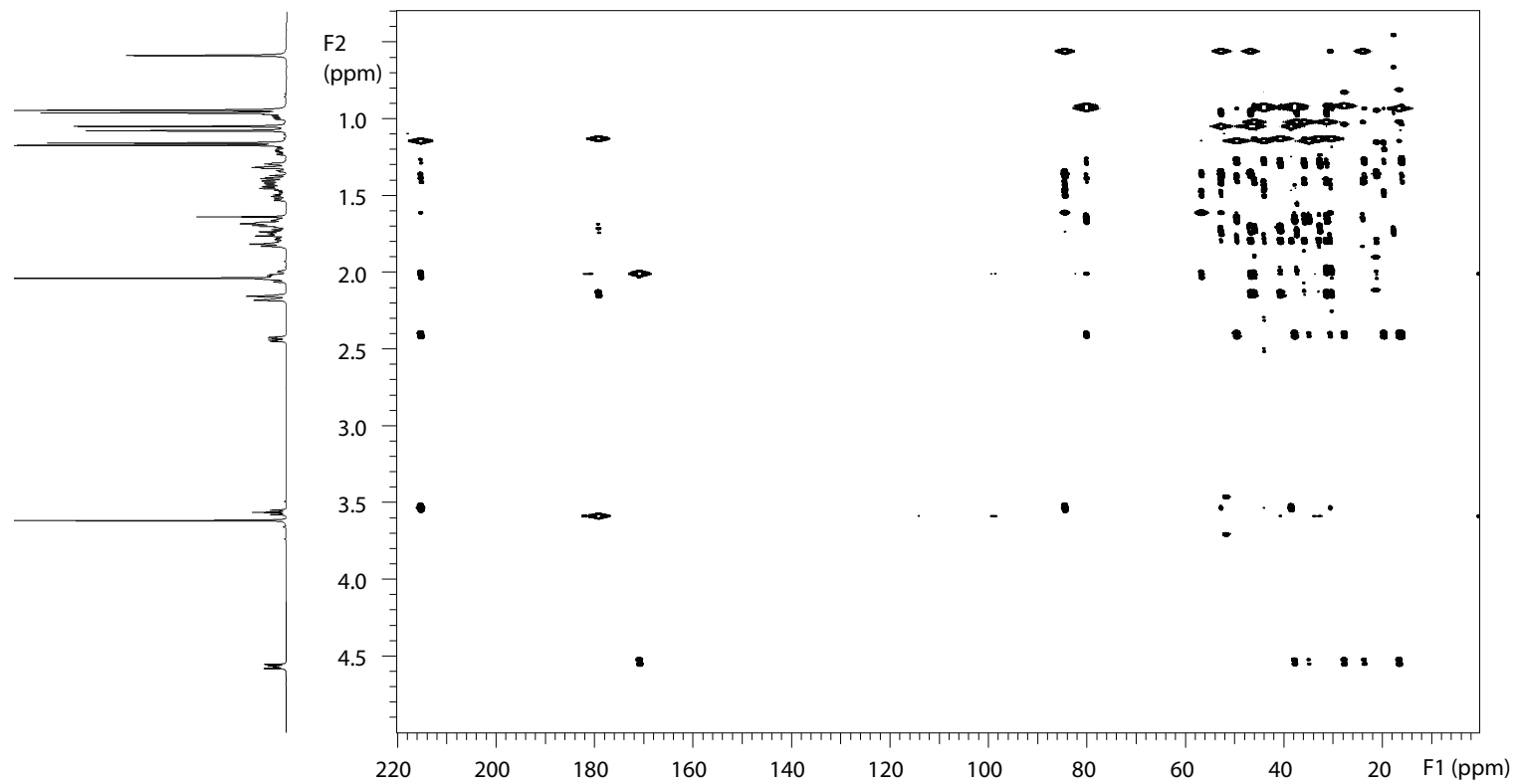
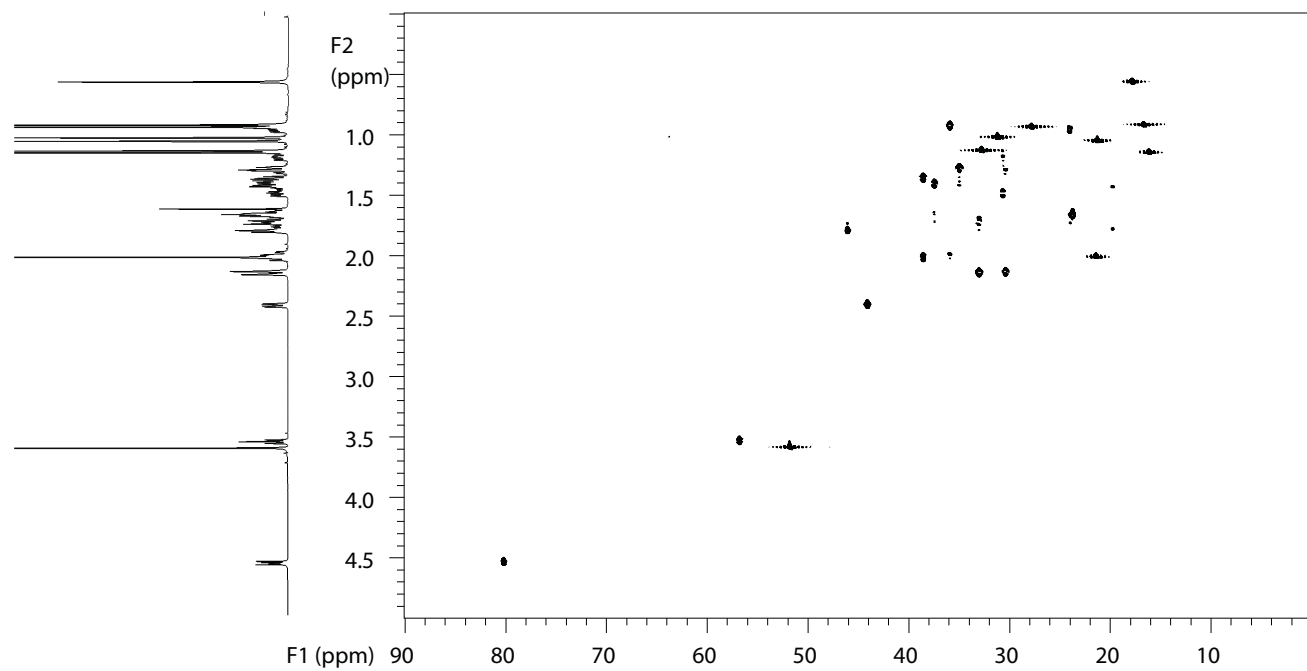
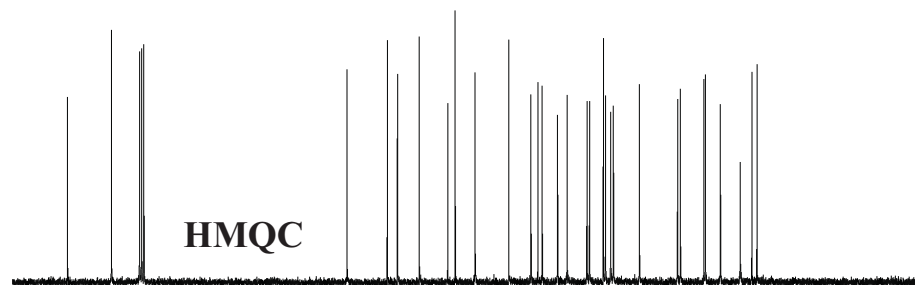
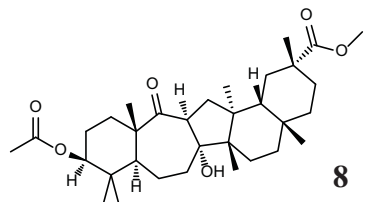
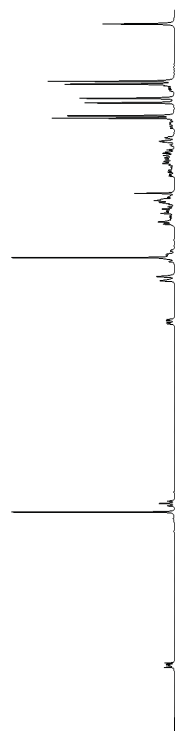
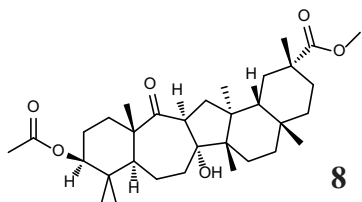
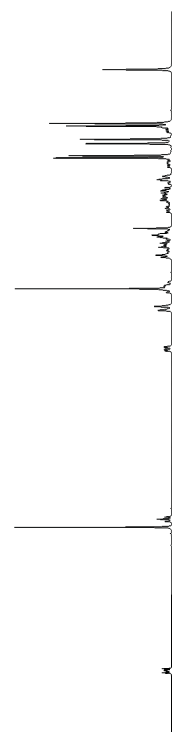
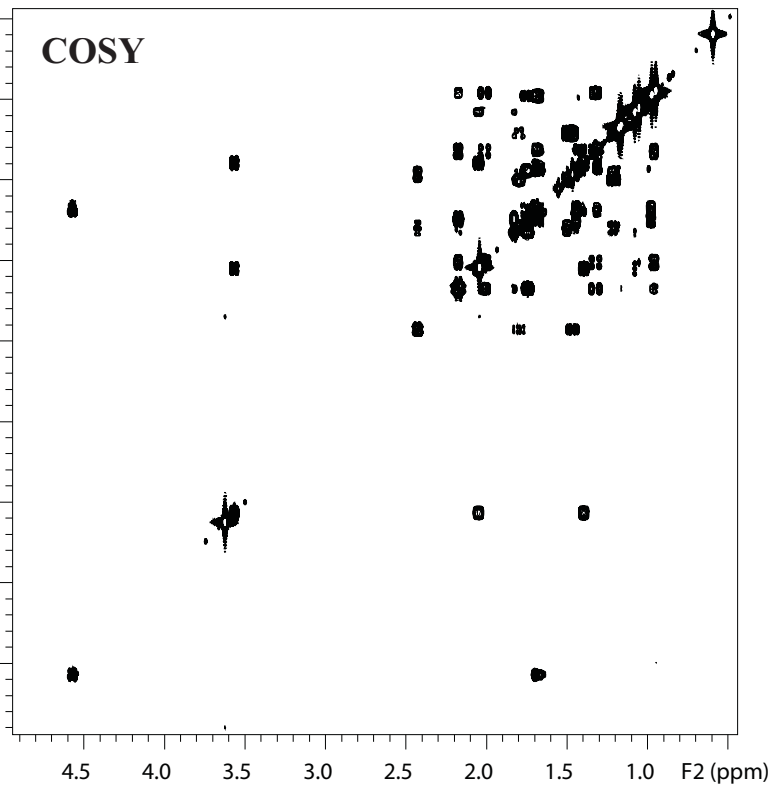


Figure S22. COSY and NOESY spectra of **8**.



F1
(ppm)

COSY



F2
(ppm)

NOESY

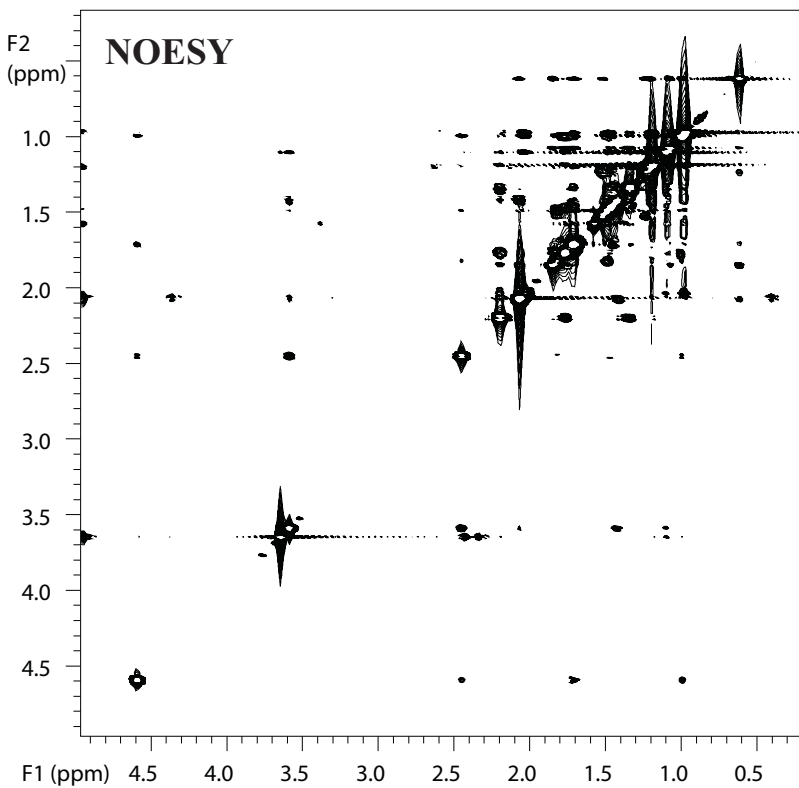


Figure S23. ¹H and ¹³C spectra of **9**

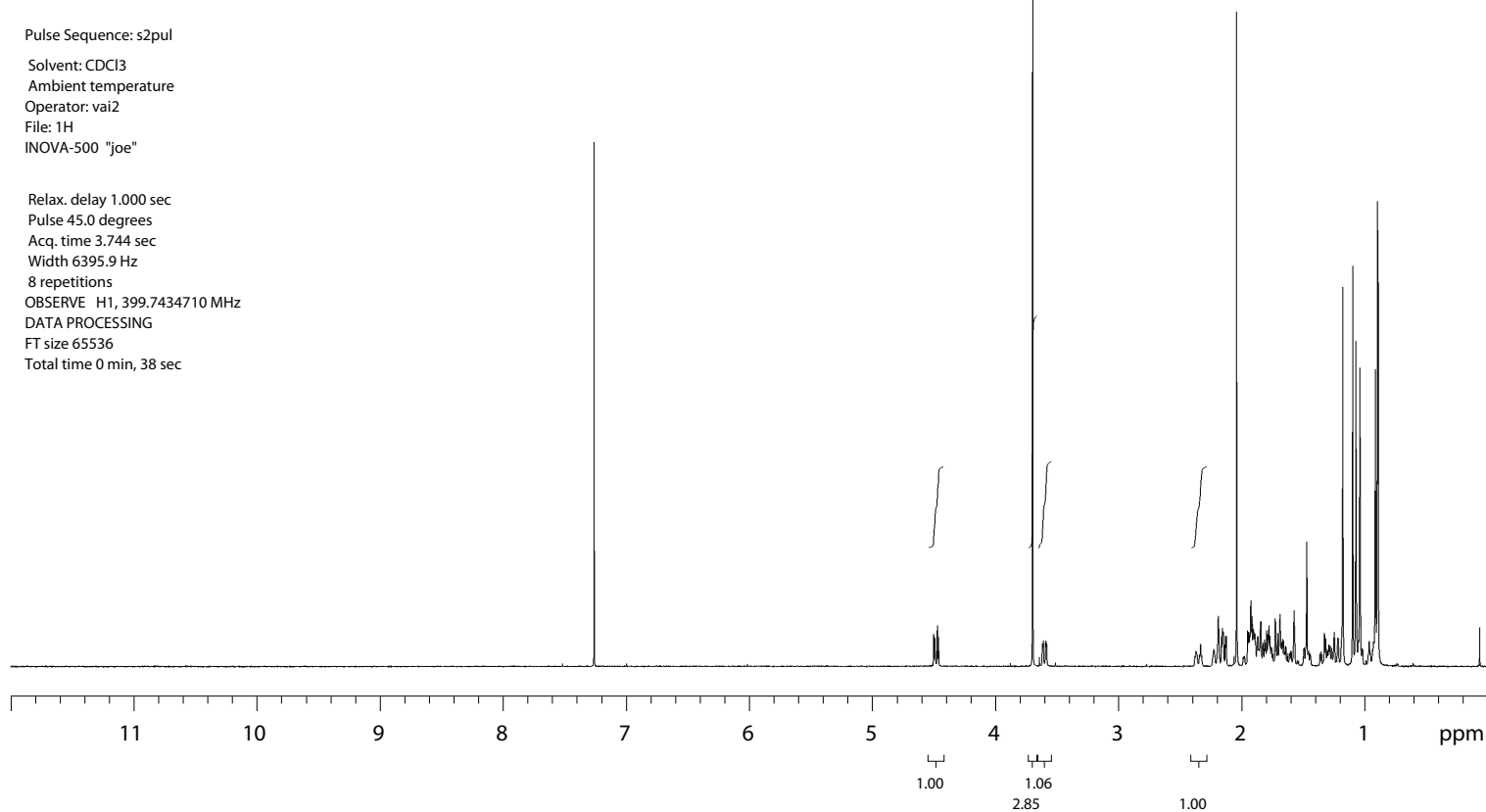
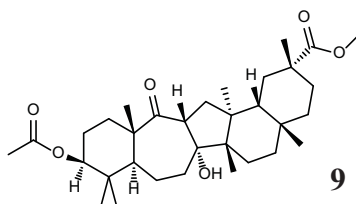
vai_2_18Apr2011-16:47:41
Data saved in:
chem400:/export/home/vai2/vnmrsys/data

Archive directory: /export/home/vai2/vnmrsys/data
Sample directory: vai2_18Apr2011-16:47:41
File: 1H

Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
Operator: vai2
File: 1H
INOVA-500 "joe"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 6395.9 Hz
8 repetitions
OBSERVE H1, 399.7434710 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 38 sec

¹H



Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
1232 repetitions
OBSERVE C13, 150.8466413 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz

¹³C

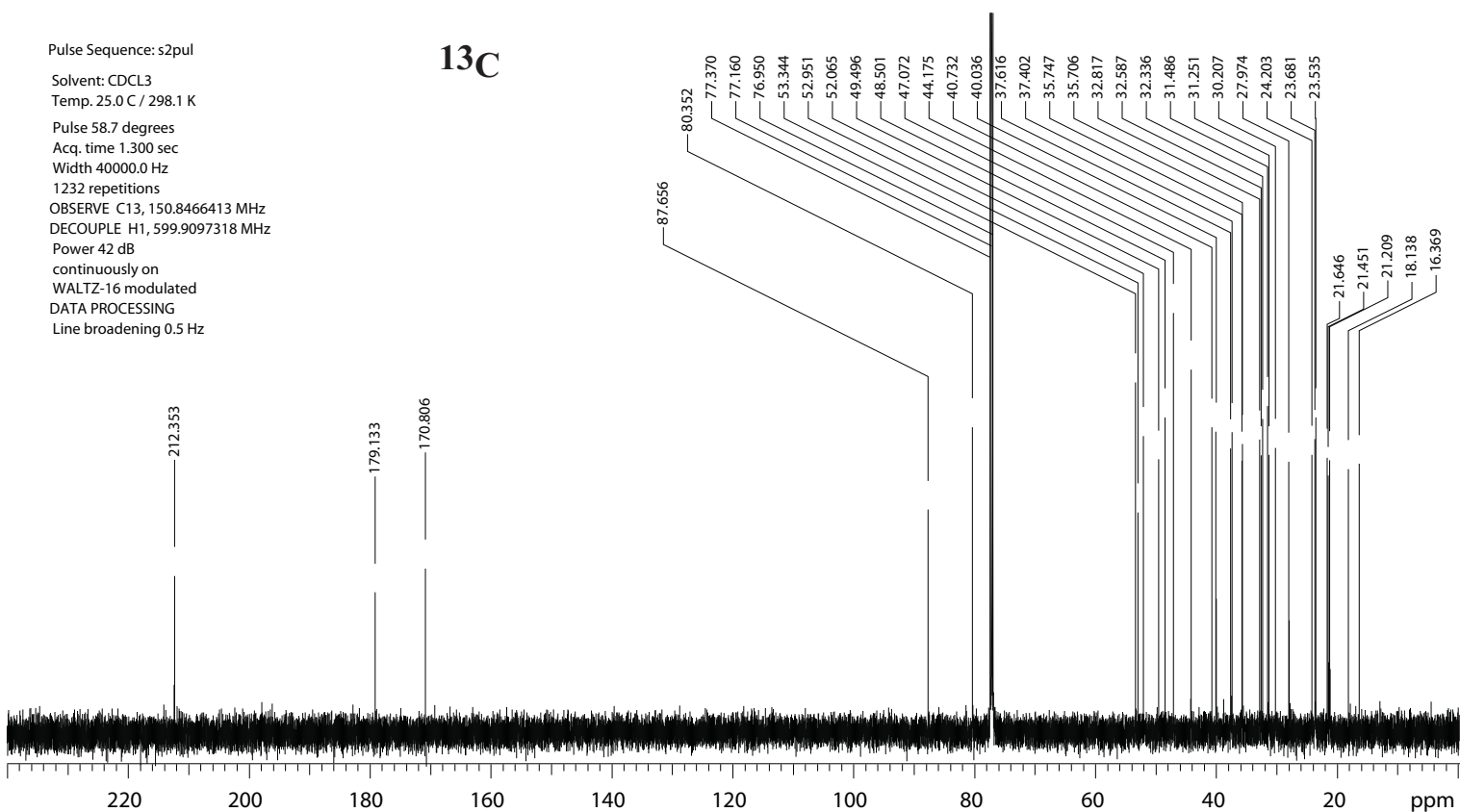


Figure S24. HMQC and HMBC spectra of **9**.

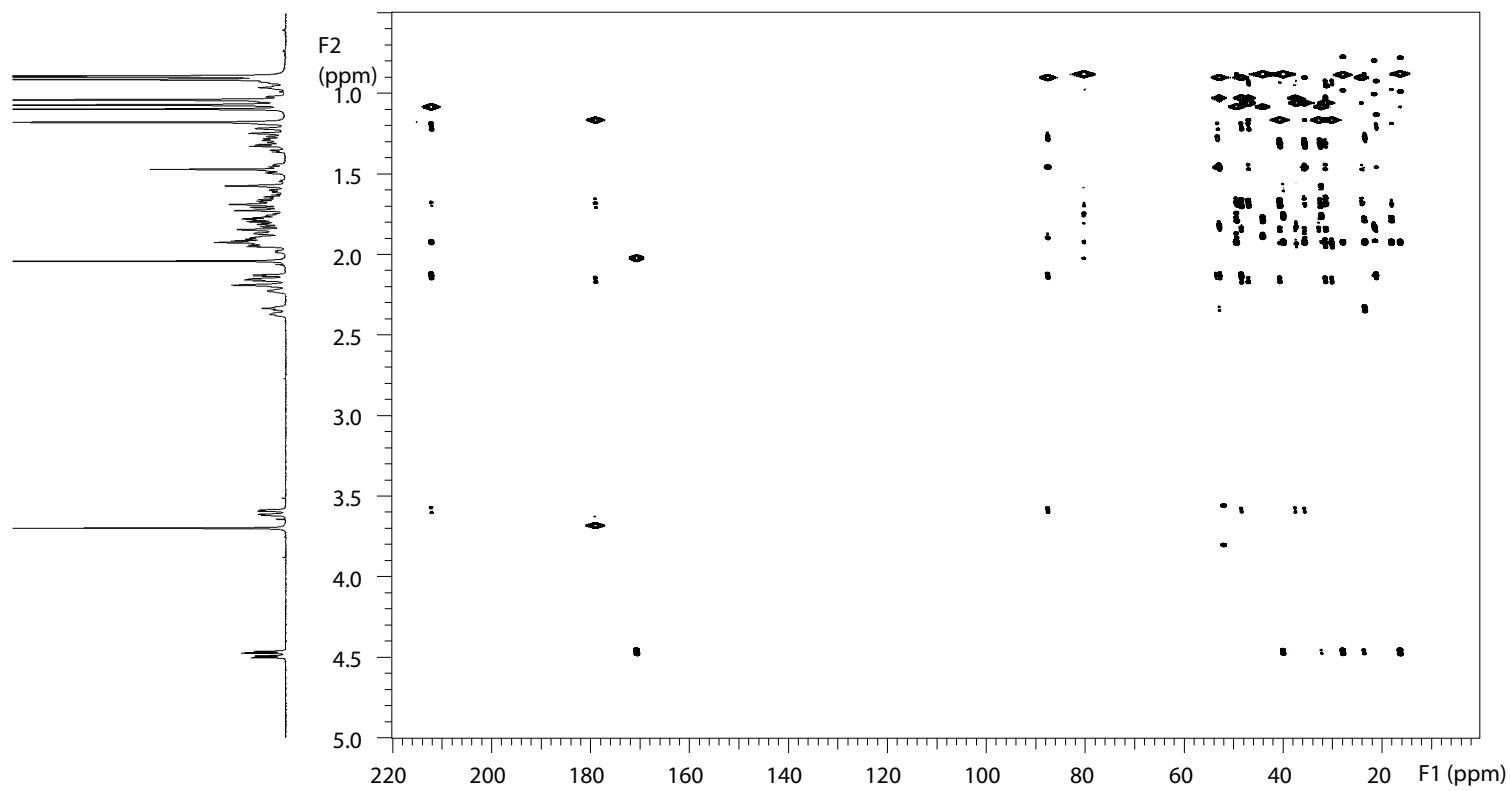
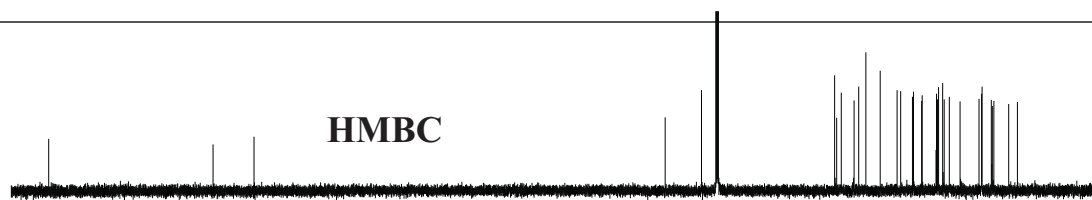
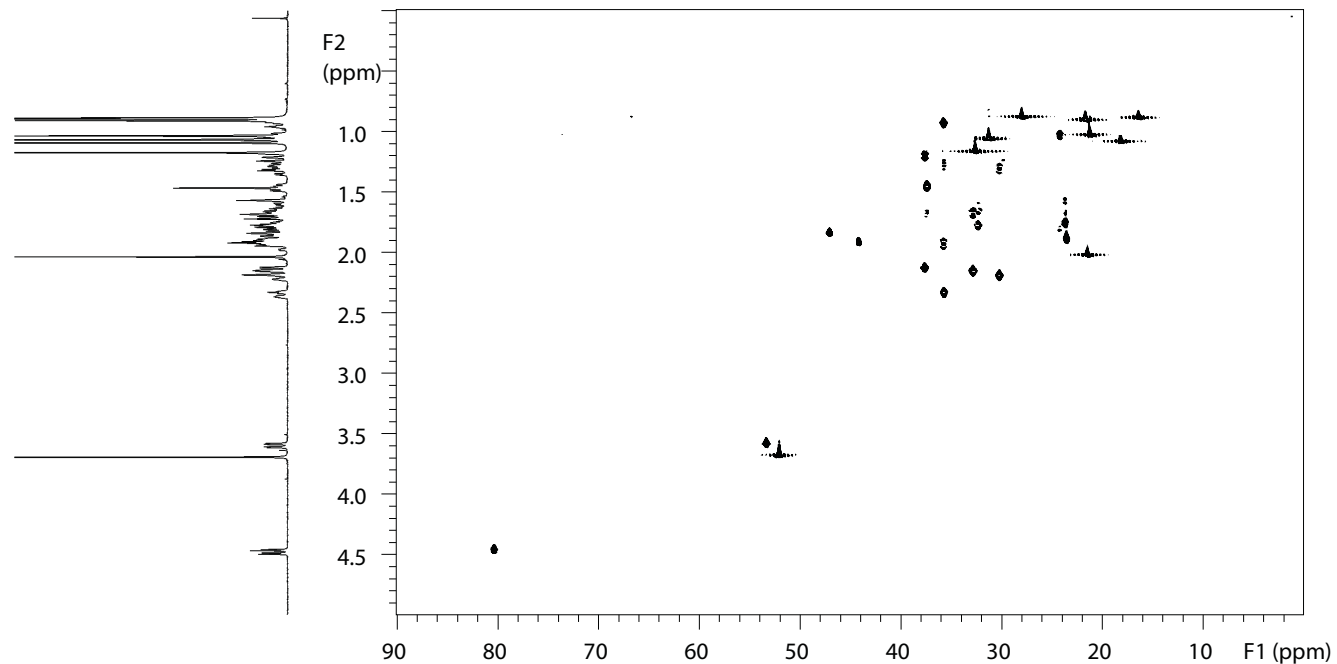
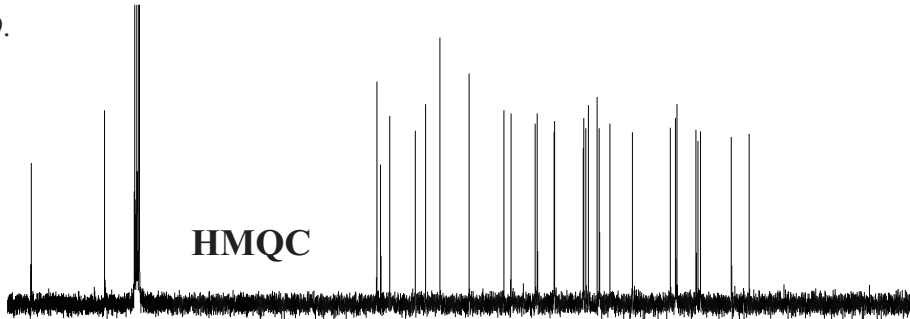
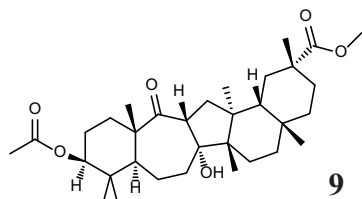
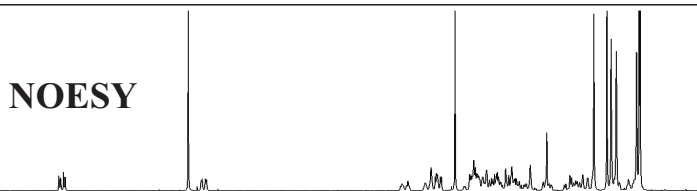
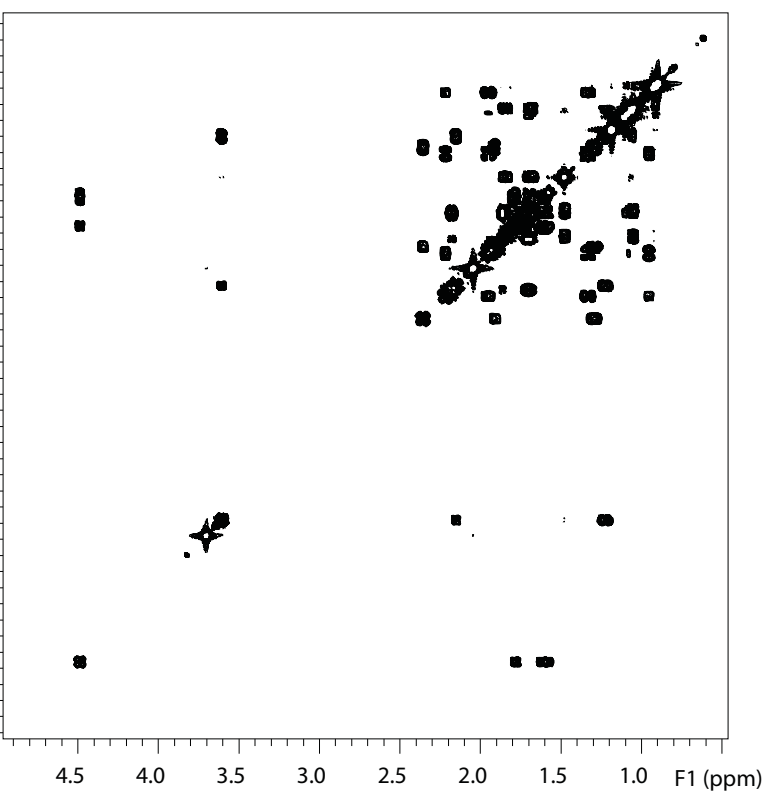
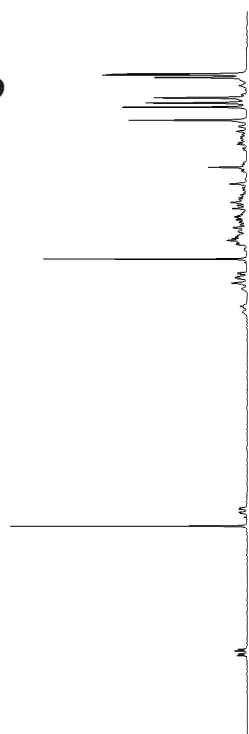
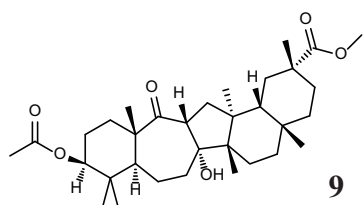


Figure S25. COSY and NOESY spectra of **9**.



NOESY

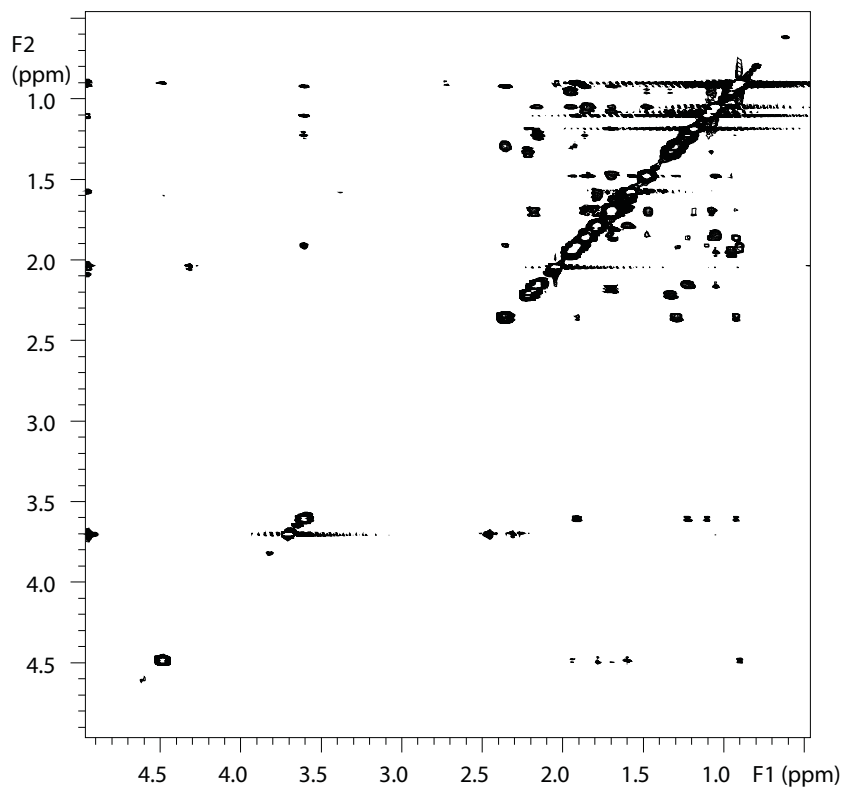
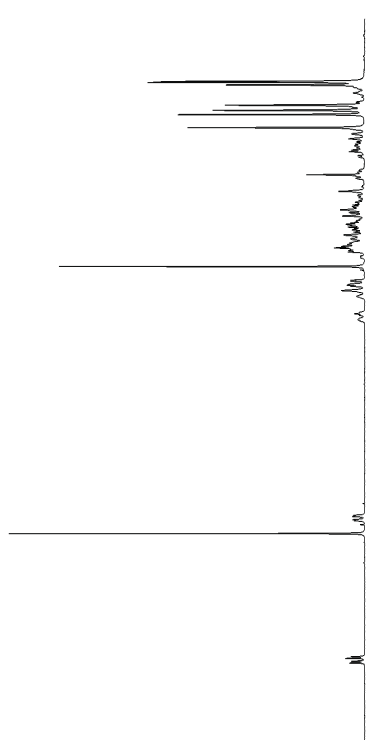


Figure S26. ¹H and ¹³C spectra of 10

vai2_18Apr2011-16:59:19

Data saved in:

chem400:/export/home/vai2/vnmrsys/data

Archive directory: /export/home/vai2/vnmrsys/data

Sample directory: vai2_18Apr2011-16:59:19

File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

INOVA-400 "chem400"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 3.744 sec

Width 6395.9 Hz

8 repetitions

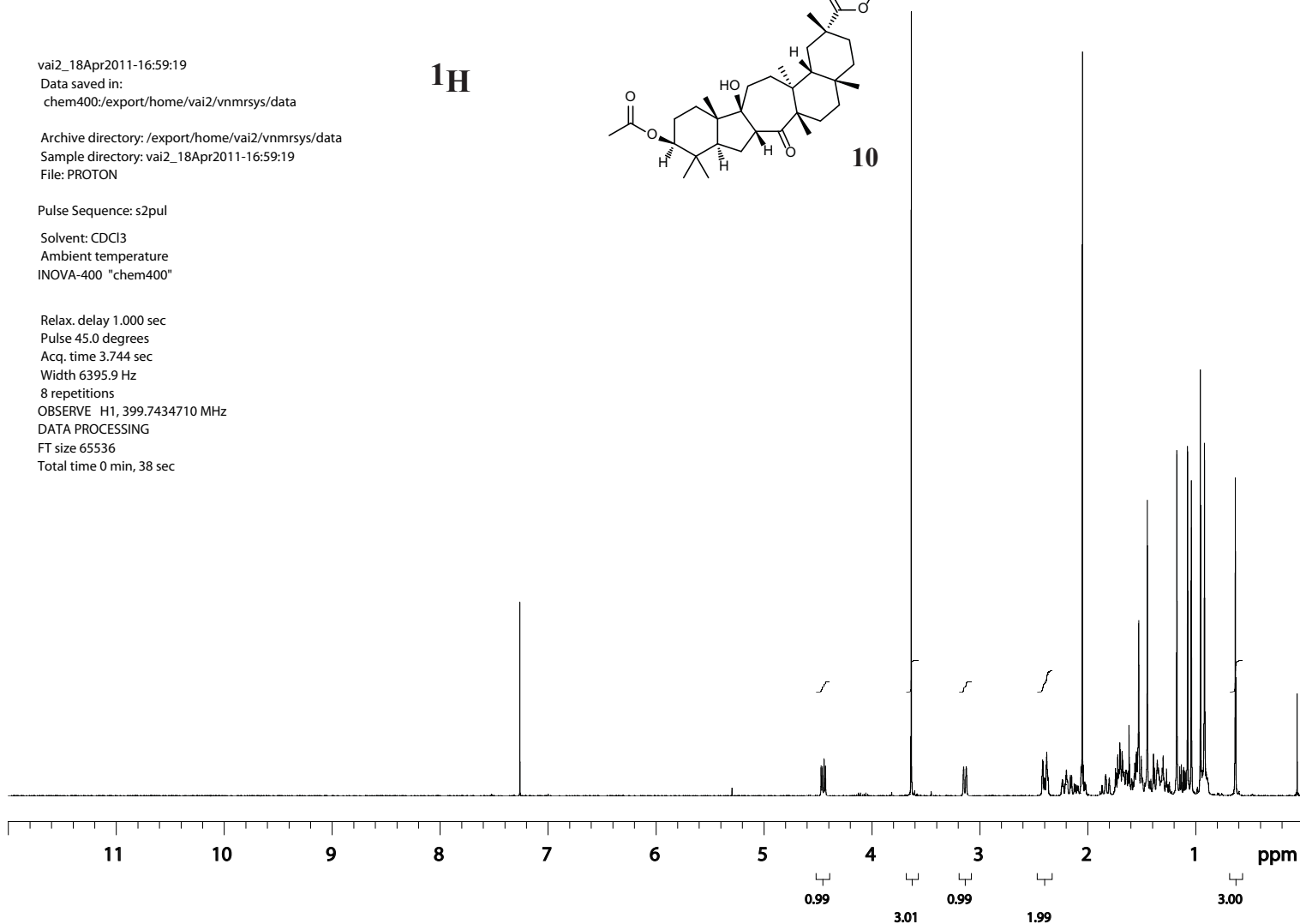
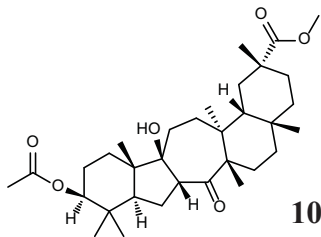
OBSERVE H1, 399.7434710 MHz

DATA PROCESSING

FT size 65536

Total time 0 min, 38 sec

¹H



¹³C

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 40000.0 Hz

288 repetitions

OBSERVE C13, 150.8466425 MHz

DECOUPLE H1, 599.9097318 MHz

Power 42 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

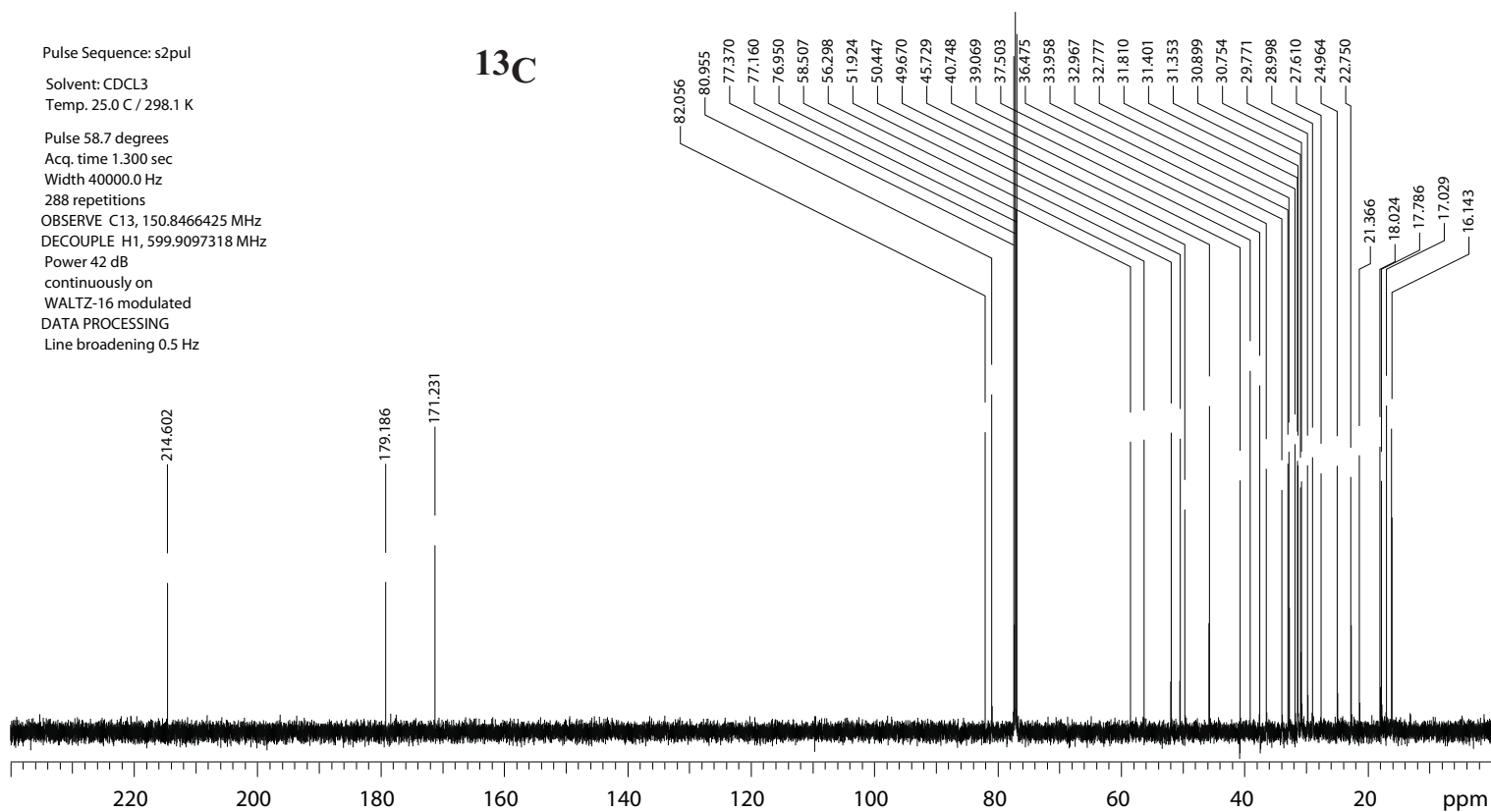


Figure S27. HMQC and HMBC spectra of **10**.

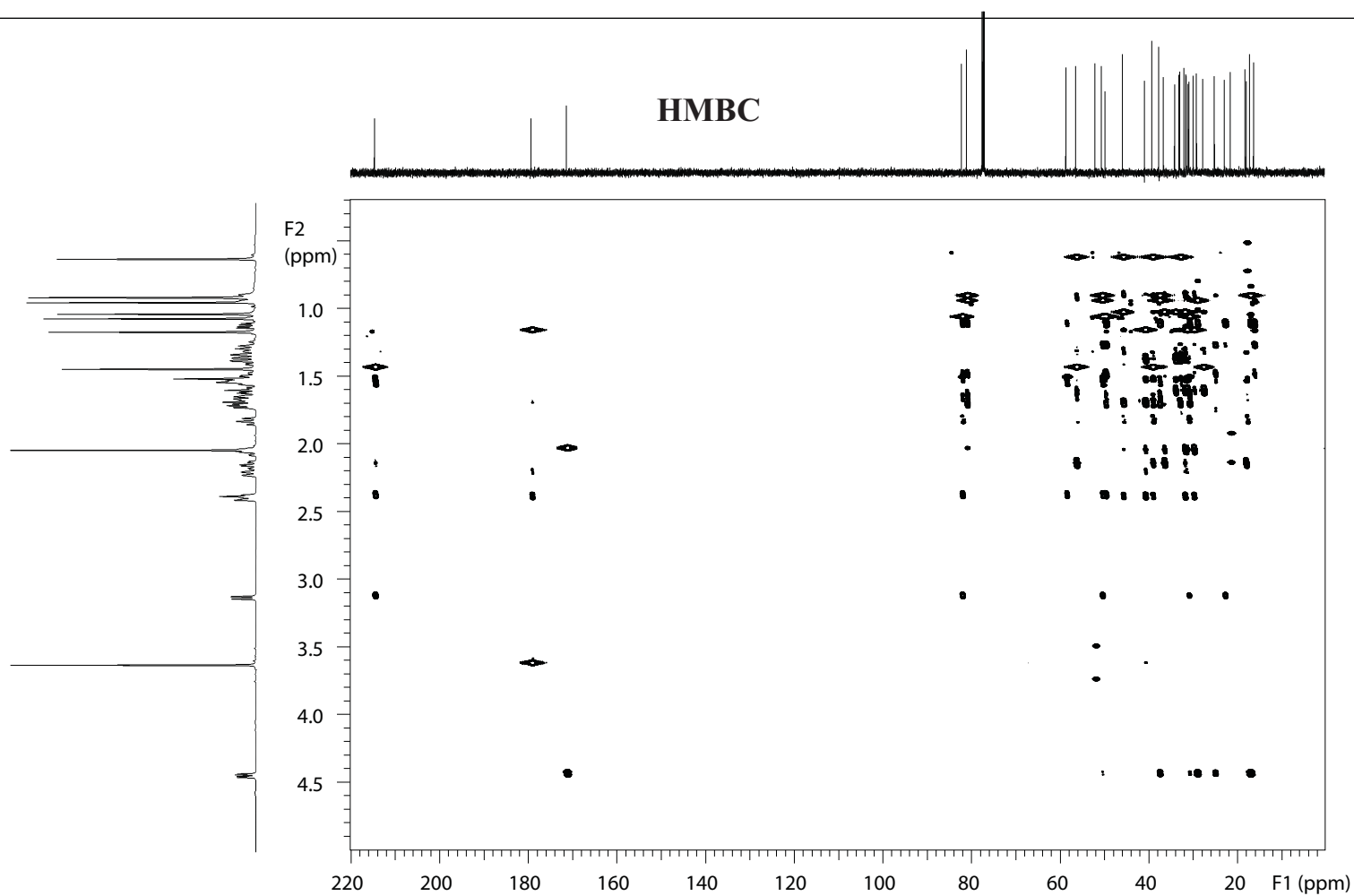
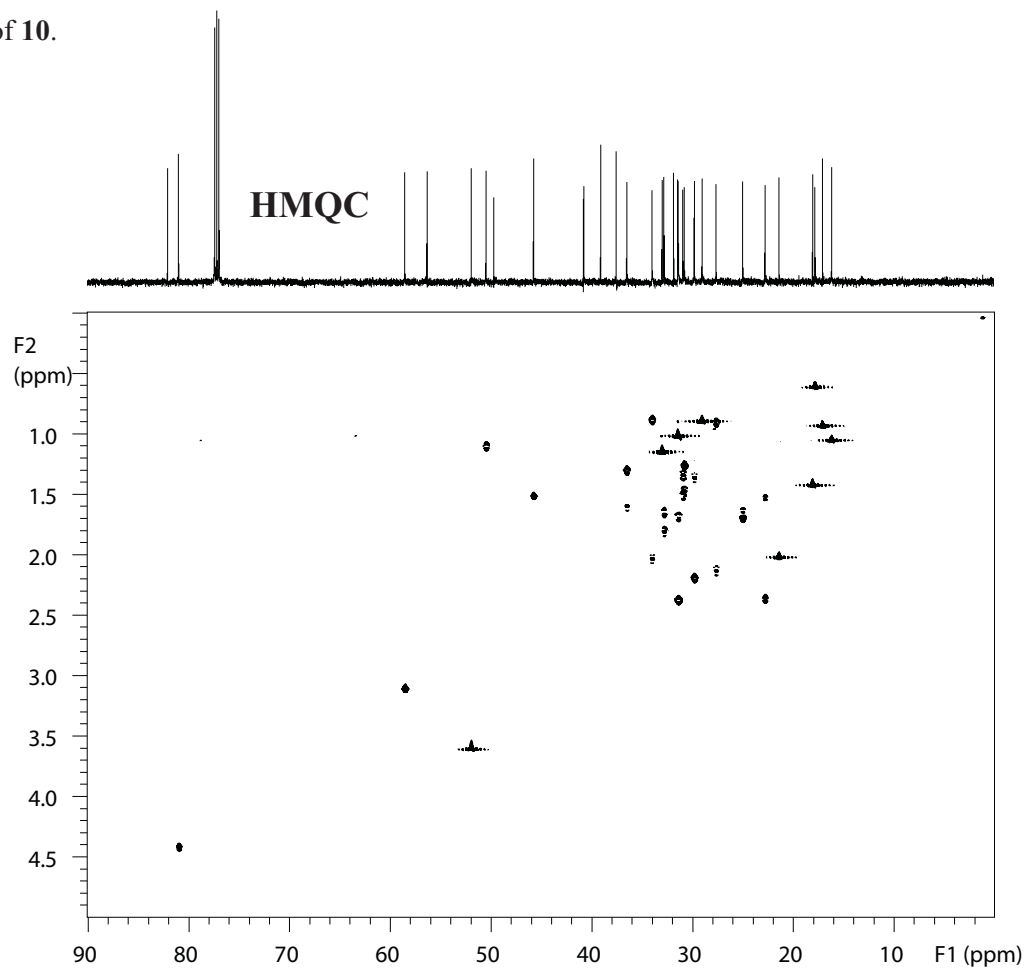
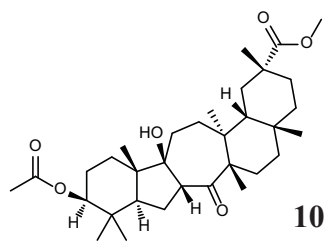
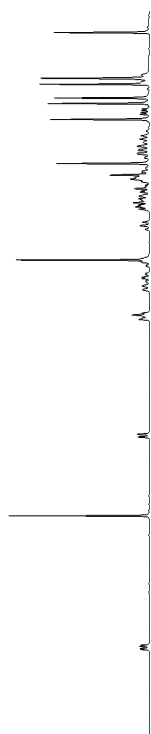
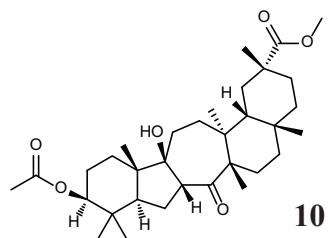
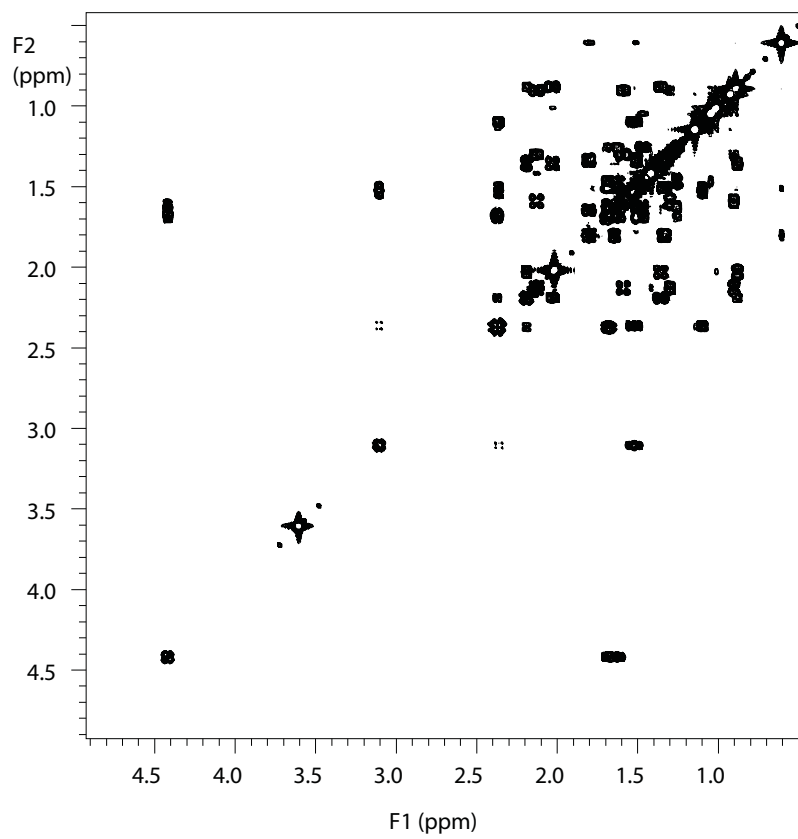
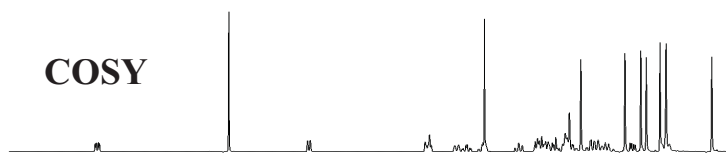


Figure S28. COSY and NOESY spectra of **10**.



COSY



NOESY

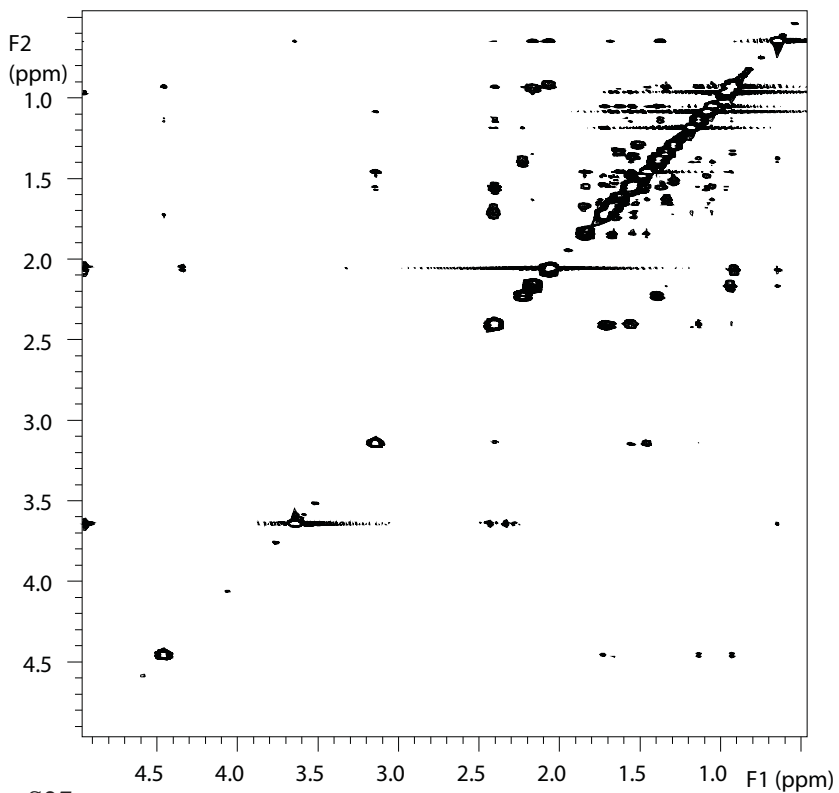
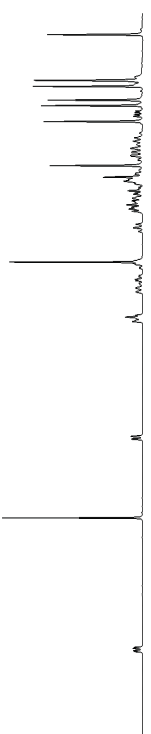
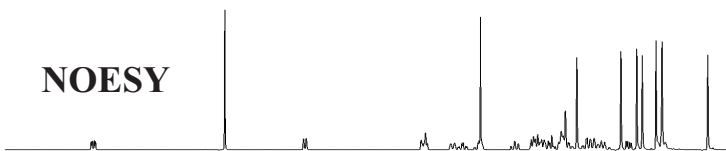


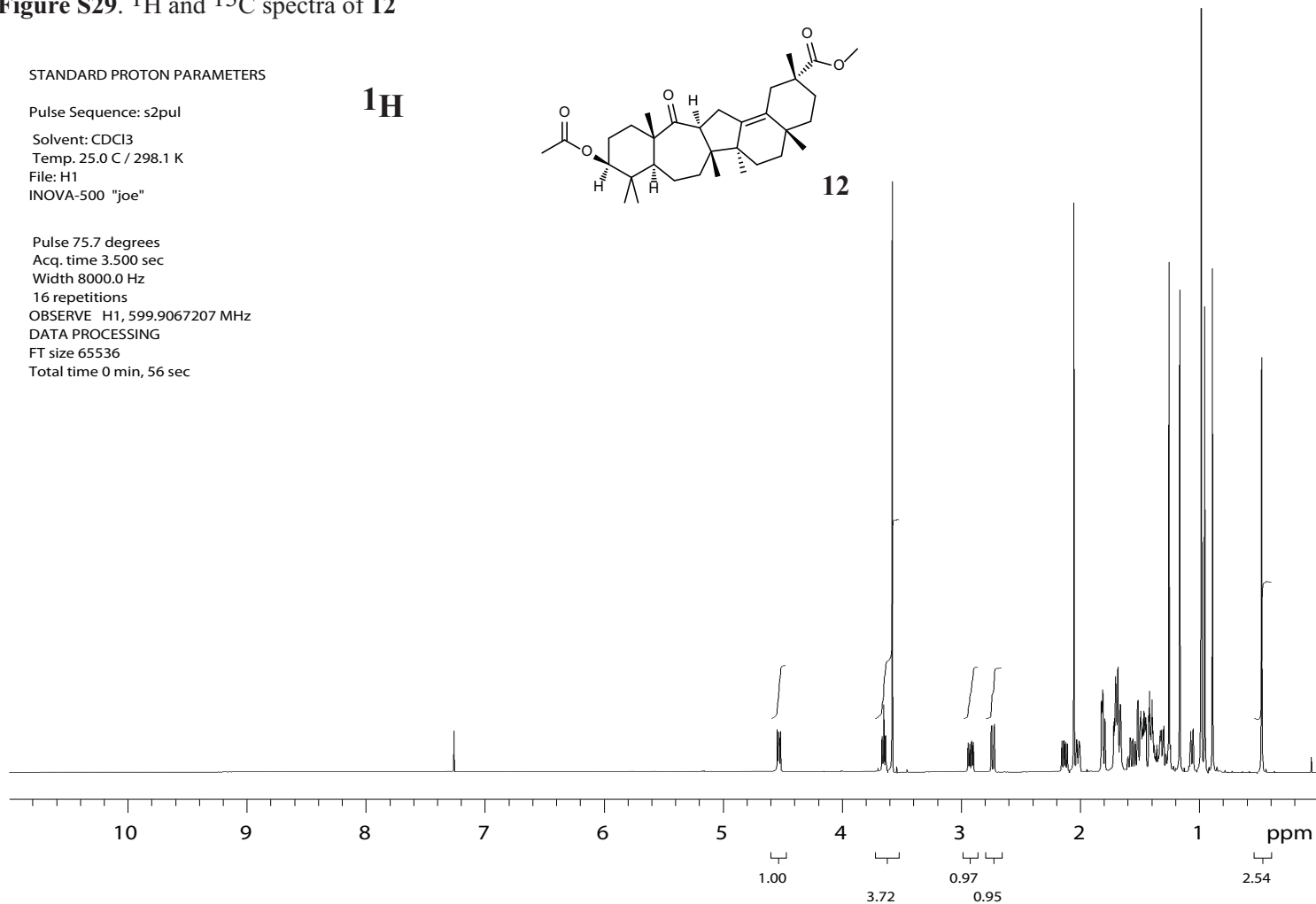
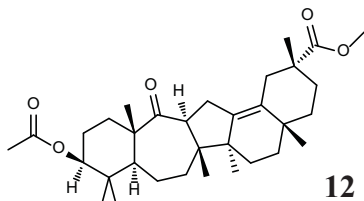
Figure S29. ¹H and ¹³C spectra of 12

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
File: H1
INOVA-500 "joe"

Pulse 75.7 degrees
Acq. time 3.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.9067207 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec

¹H



Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: C13
INOVA-500 "joe"

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
768 repetitions
OBSERVE C13, 150.8466318 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 44 min, 41 sec

¹³C

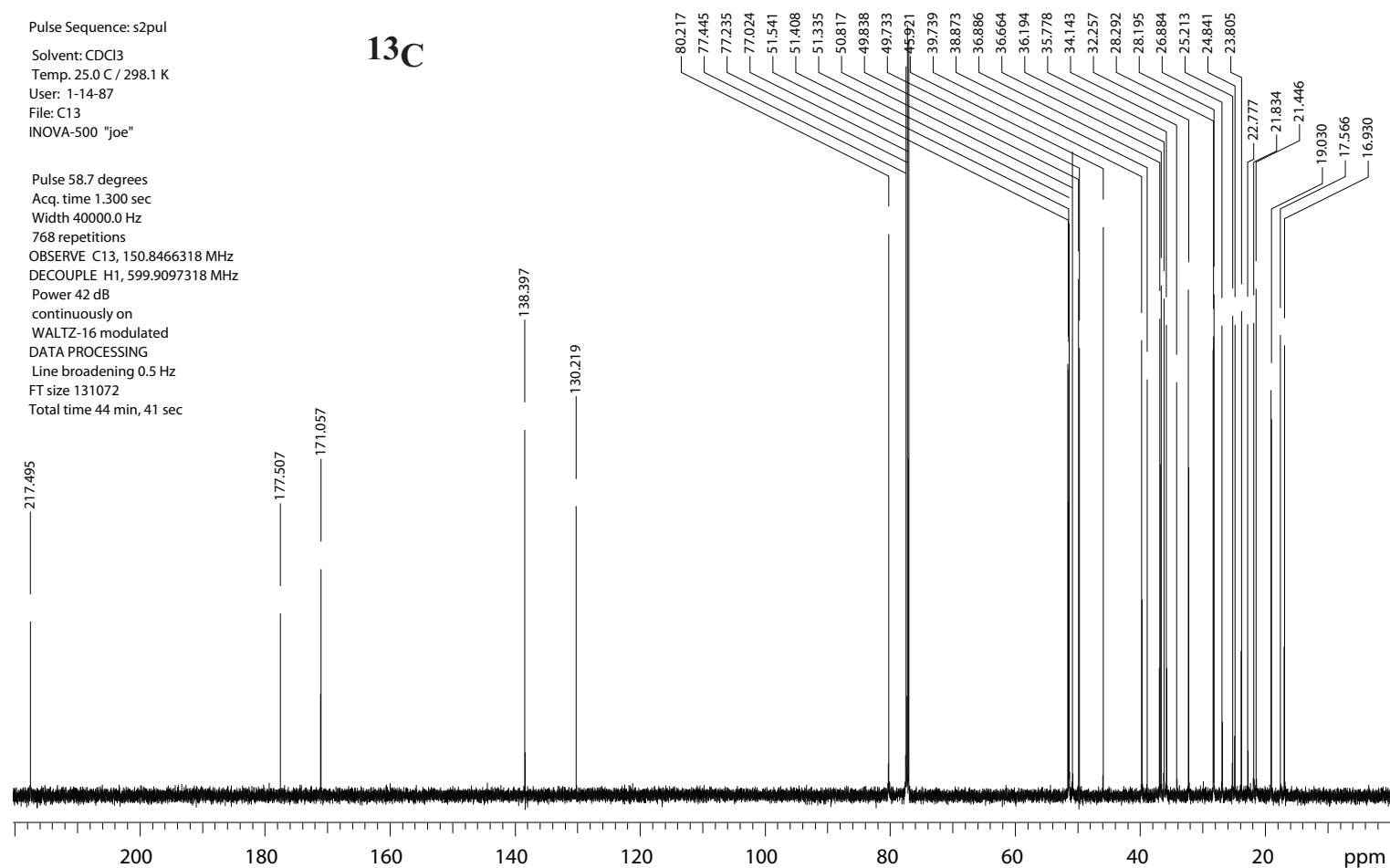


Figure S30. HMQC and HMBC spectra of 12.

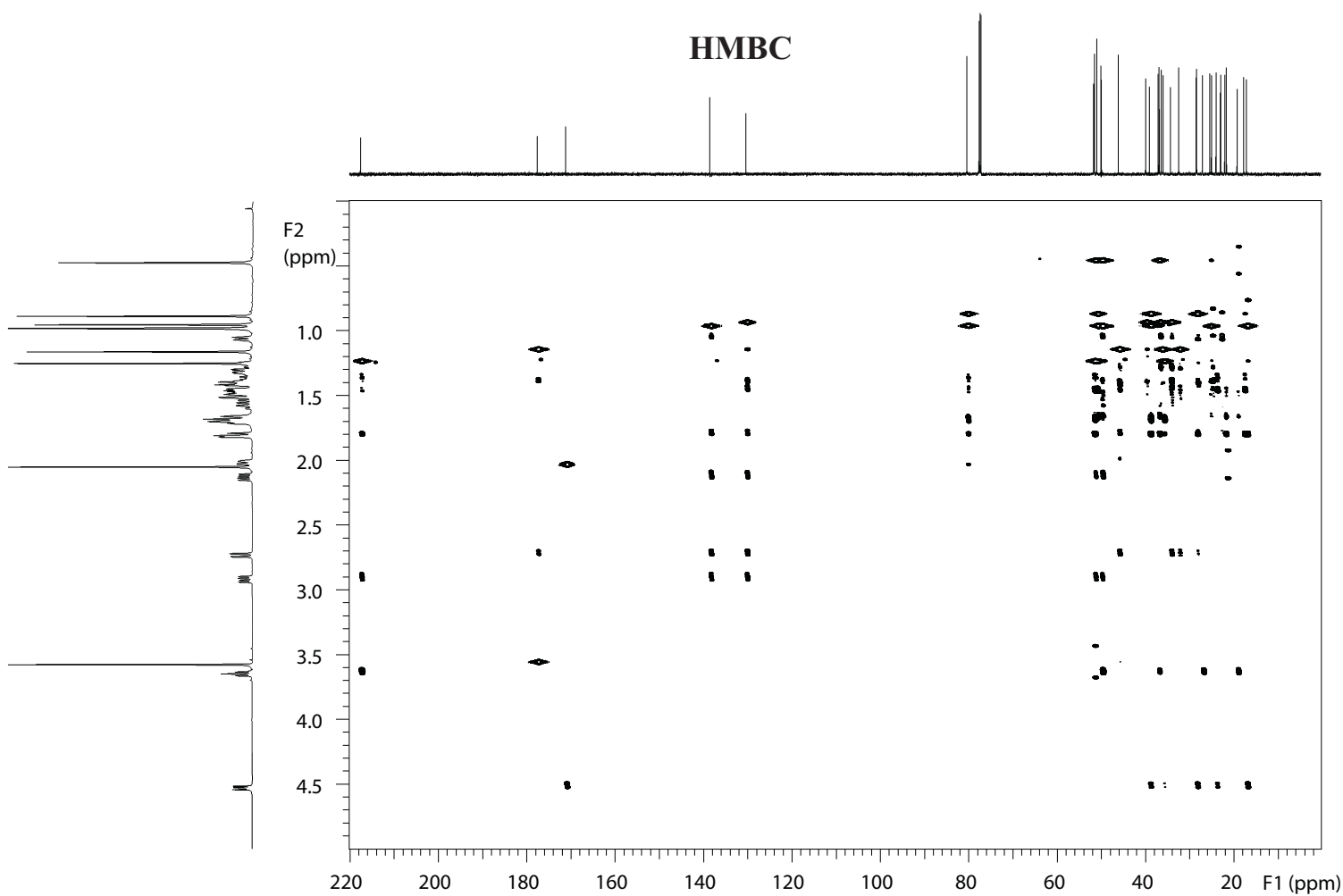
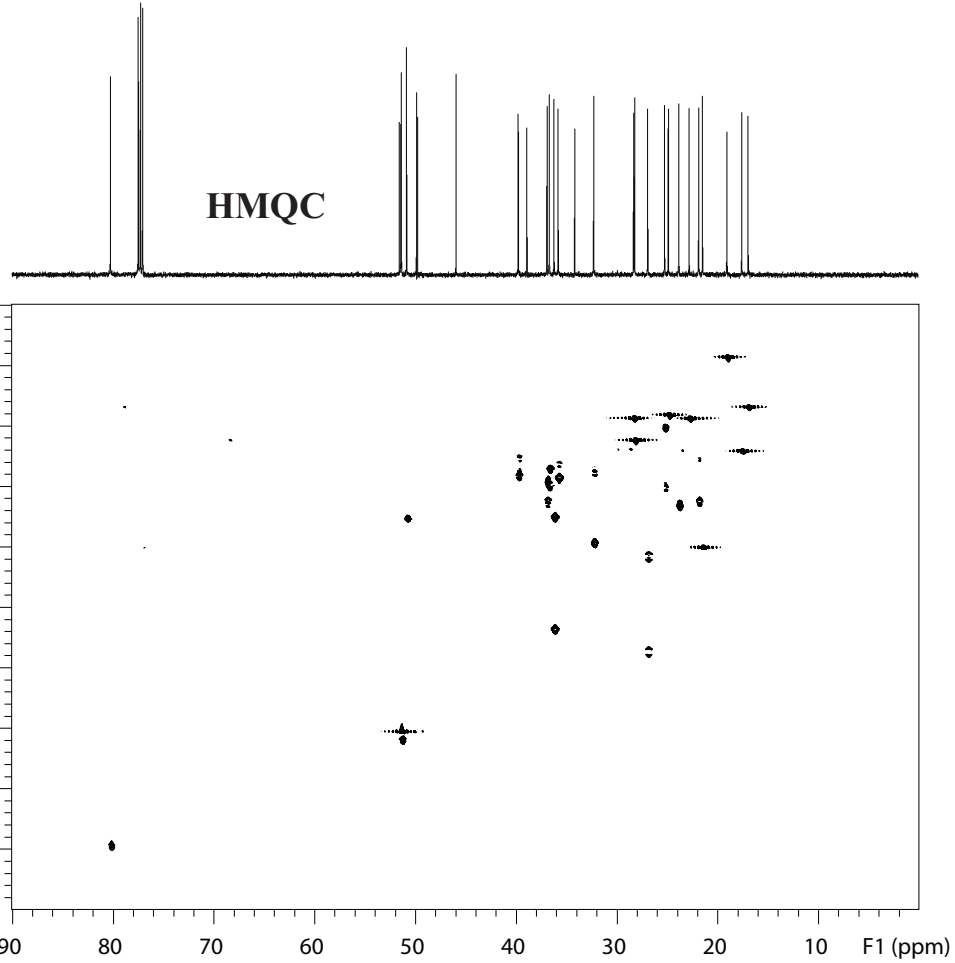
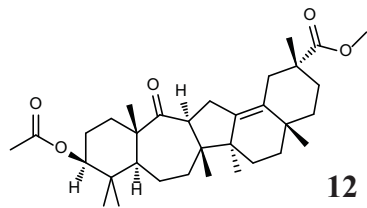
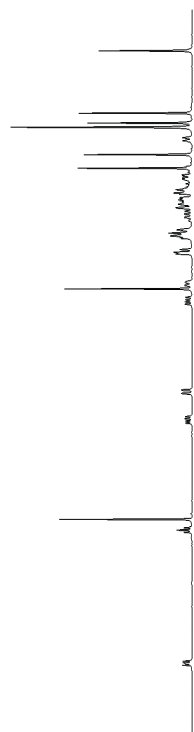
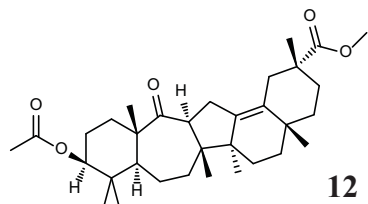
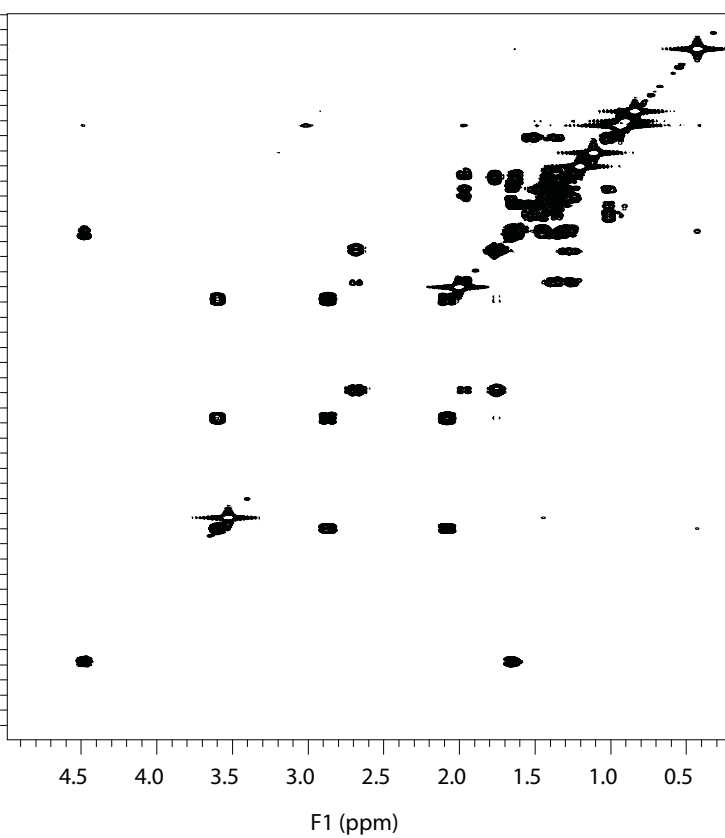


Figure S31. COSY and NOESY spectra of 12.



COSY



NOESY

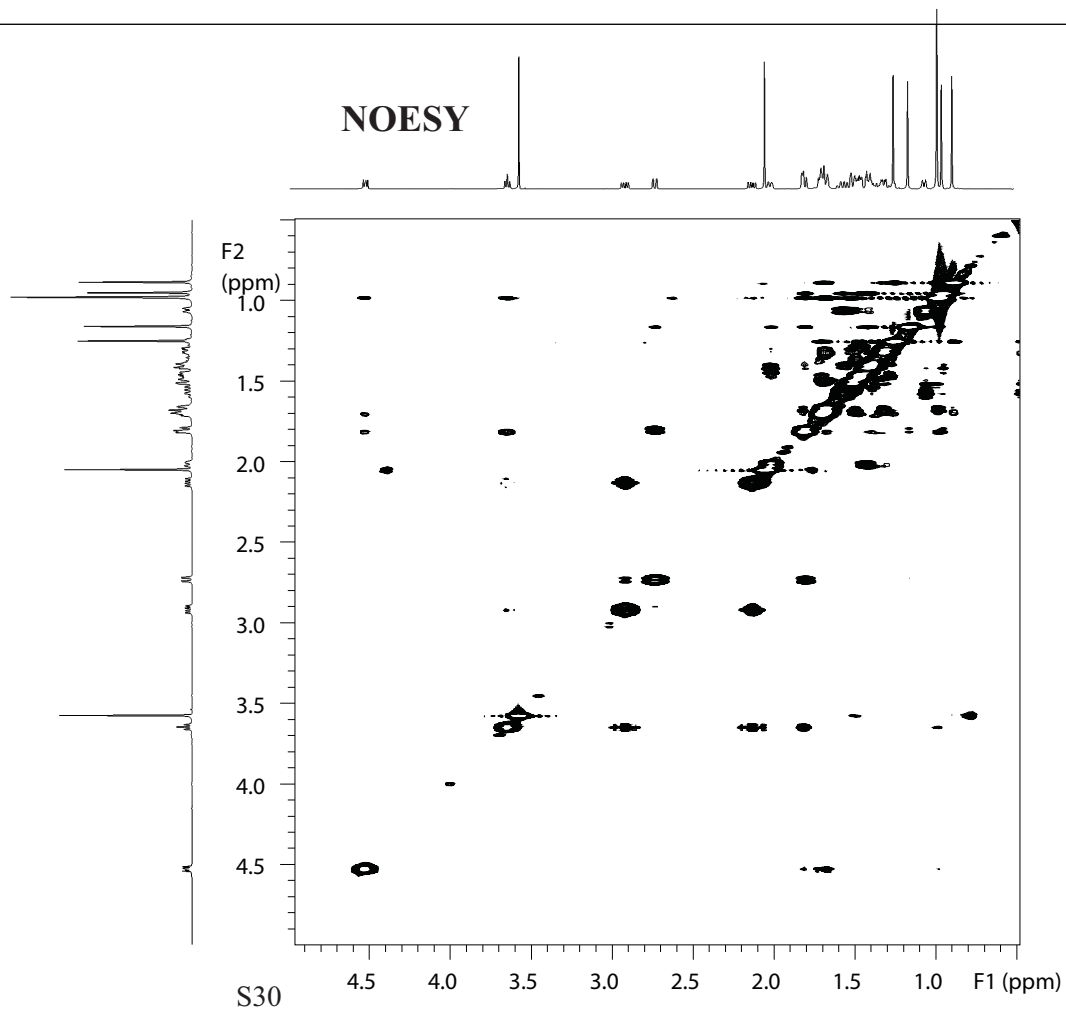


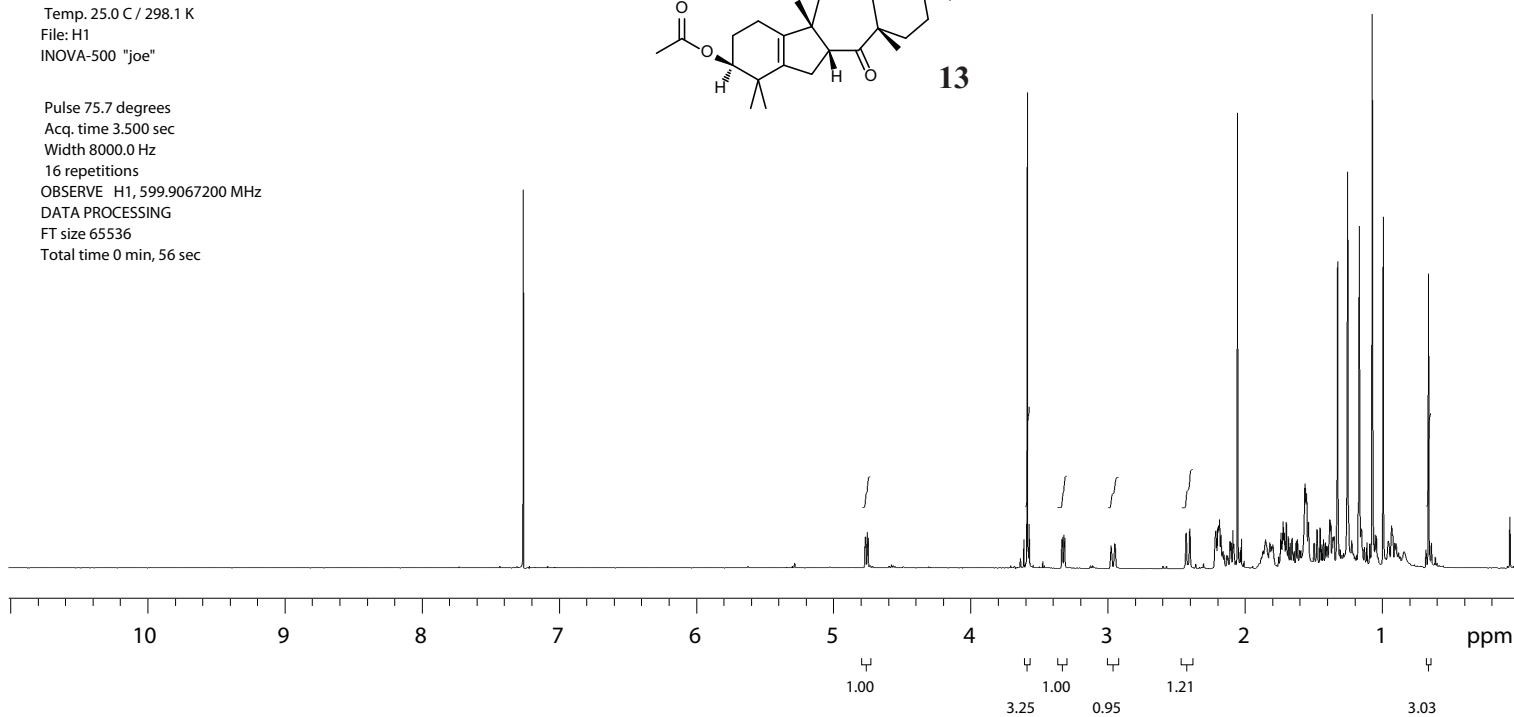
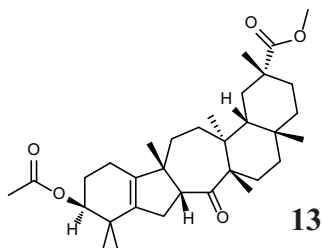
Figure S32. ¹H and ¹³C spectra of 13

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
File: H1
INOVA-500 "joe"

Pulse 75.7 degrees
Acq. time 3.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.9067200 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec

¹H



STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: C13
INOVA-500 "joe"

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
2048 repetitions
OBSERVE C13, 150.8466406 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 44 min, 41 sec

¹³C

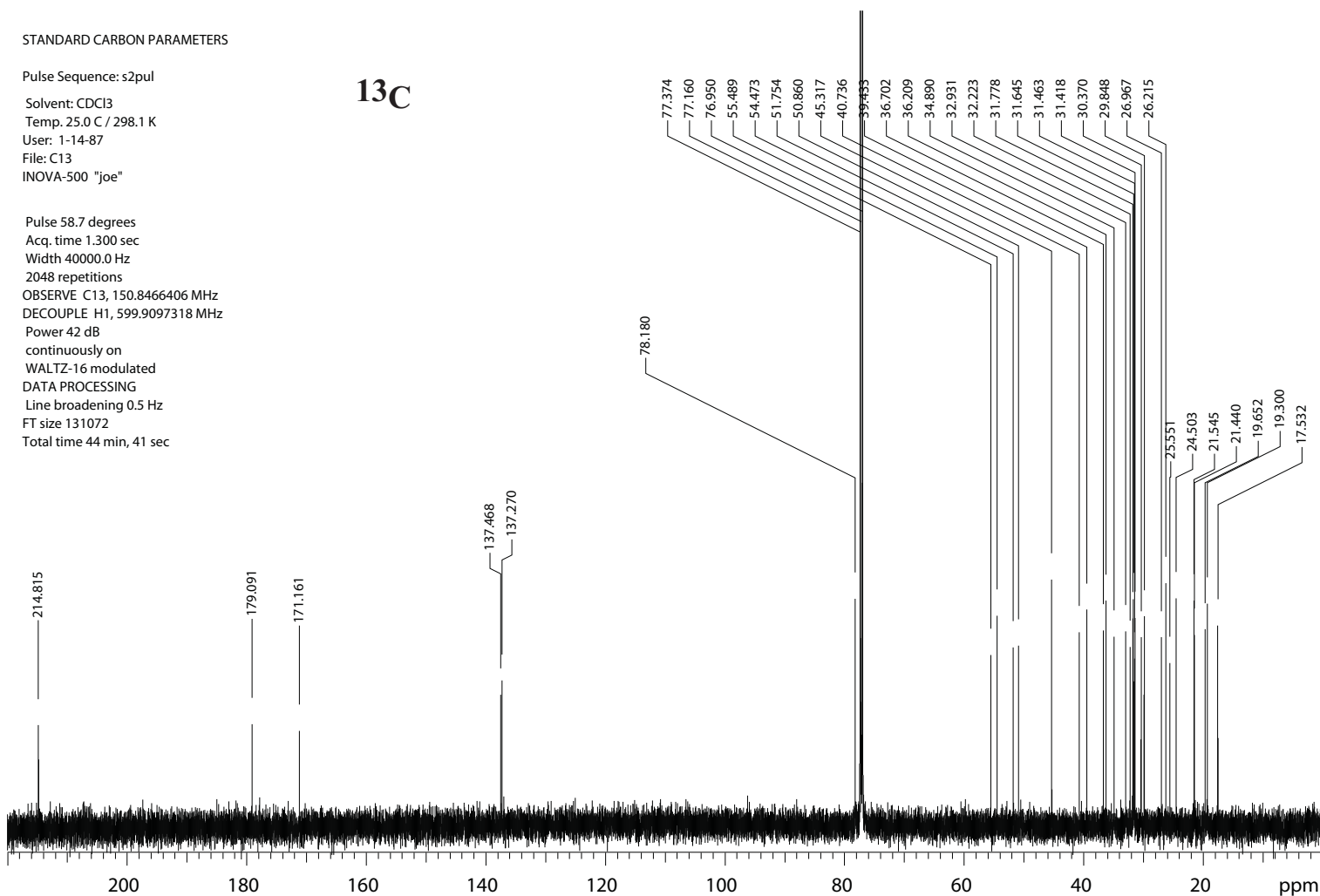


Figure S33. HMQC and HMBC spectra of 13.

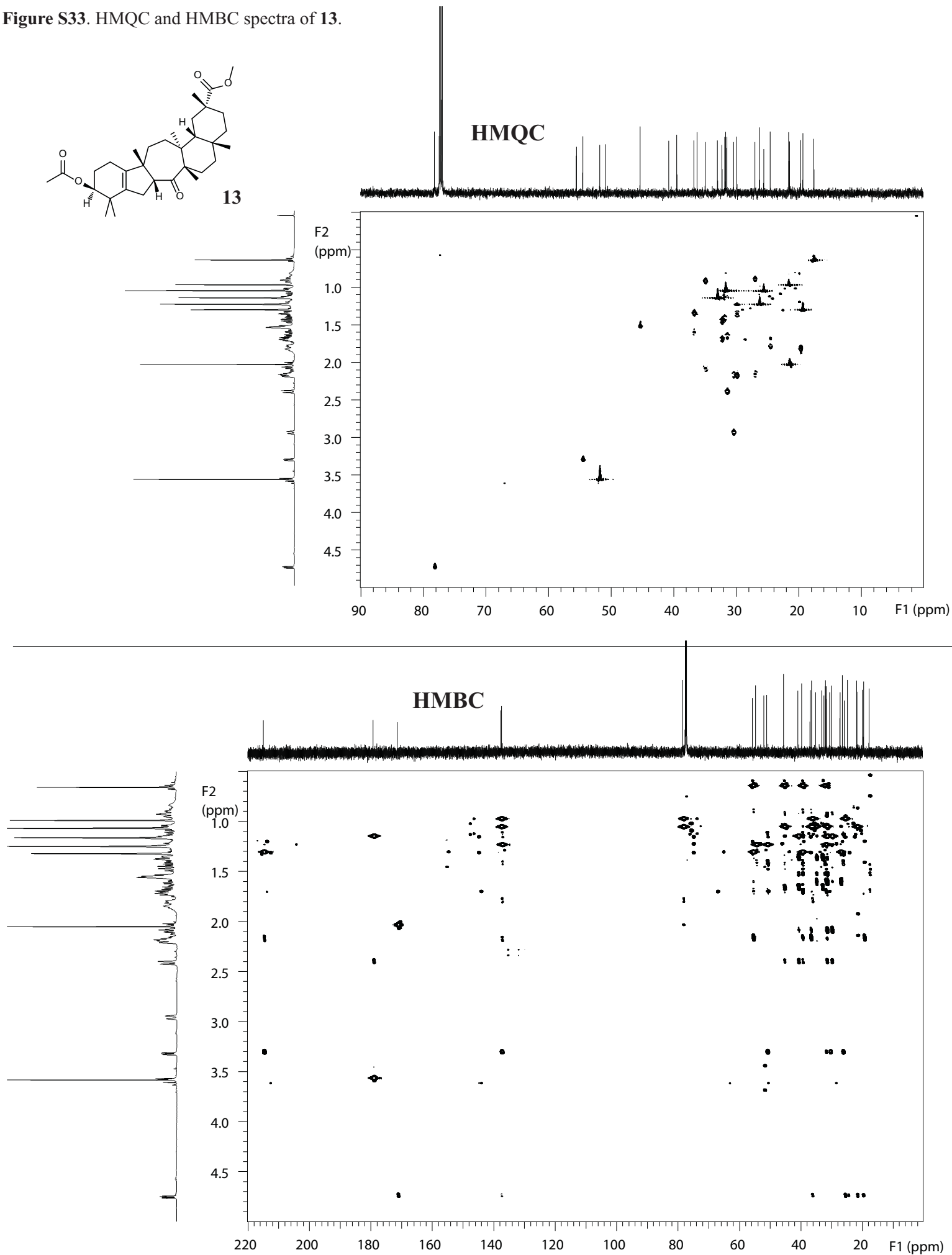


Figure S34. COSY and NOESY spectra of 13.

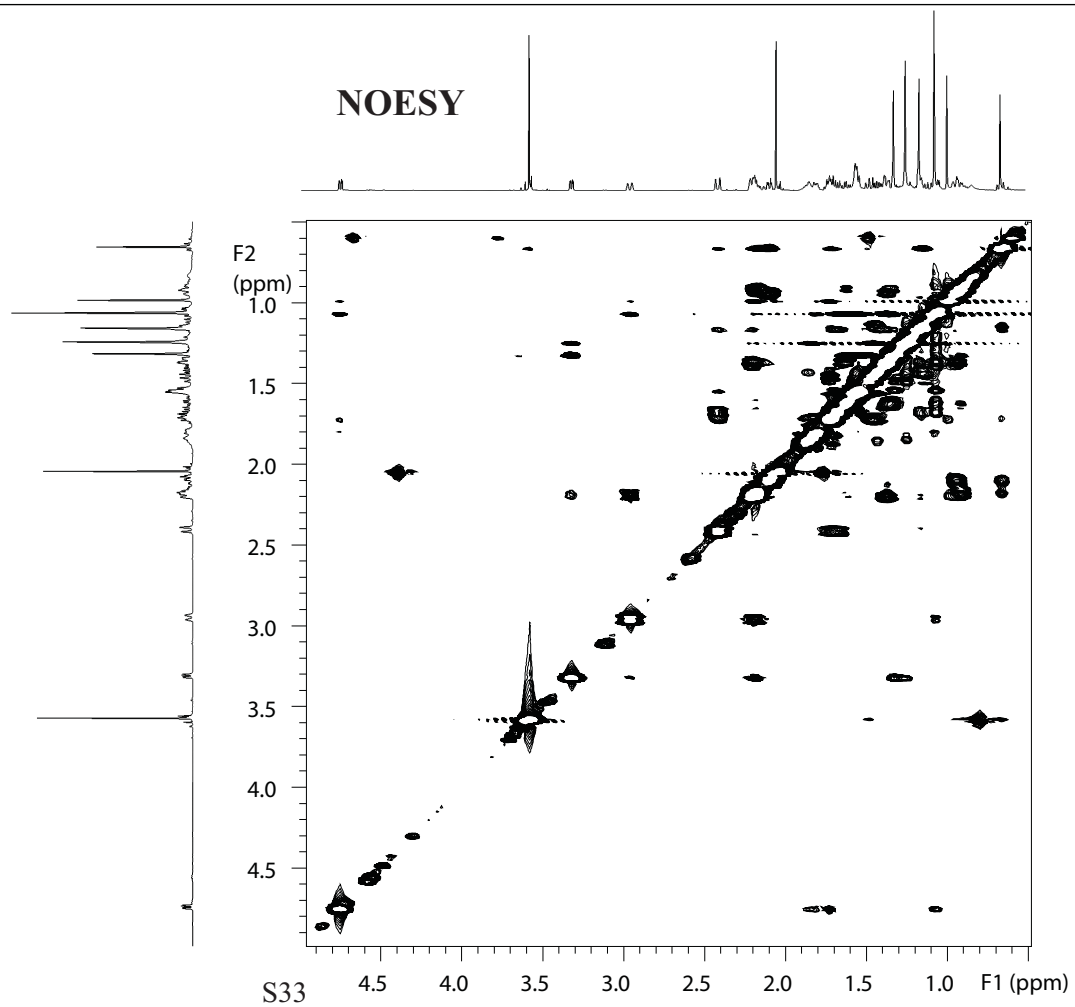
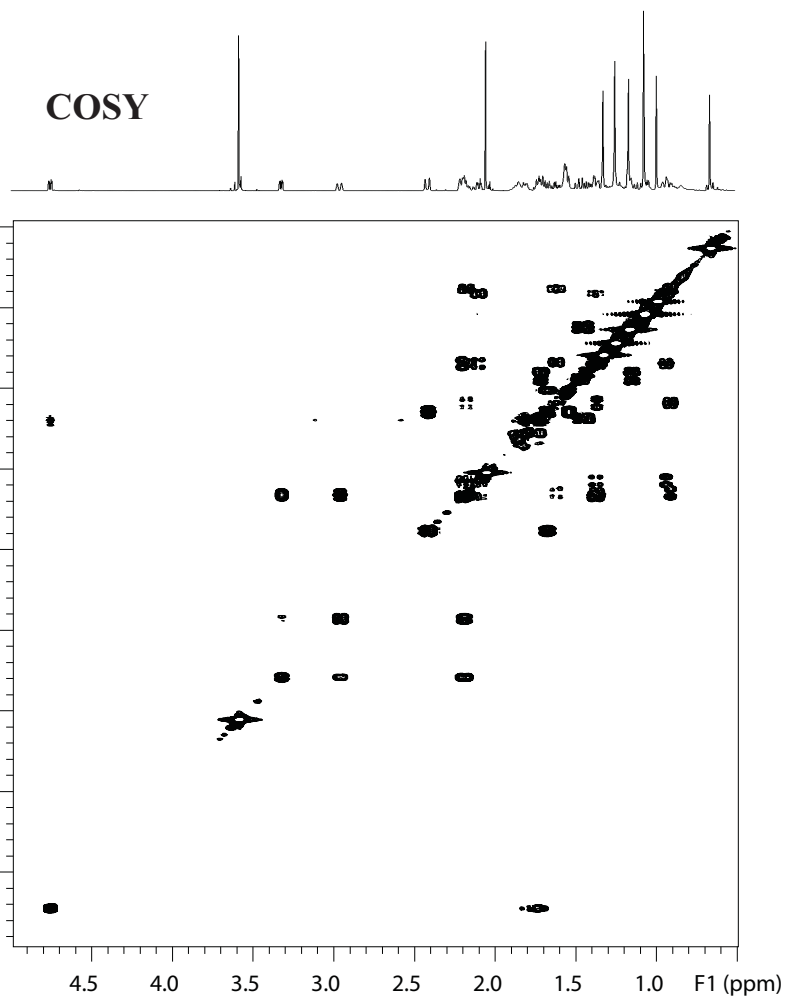
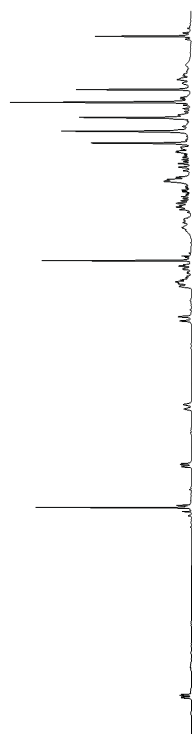
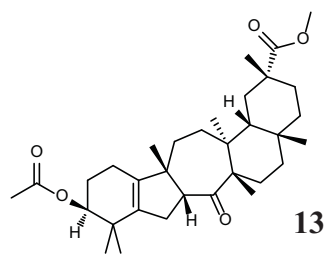


Figure S35. ^1H and ^{13}C spectra of 14

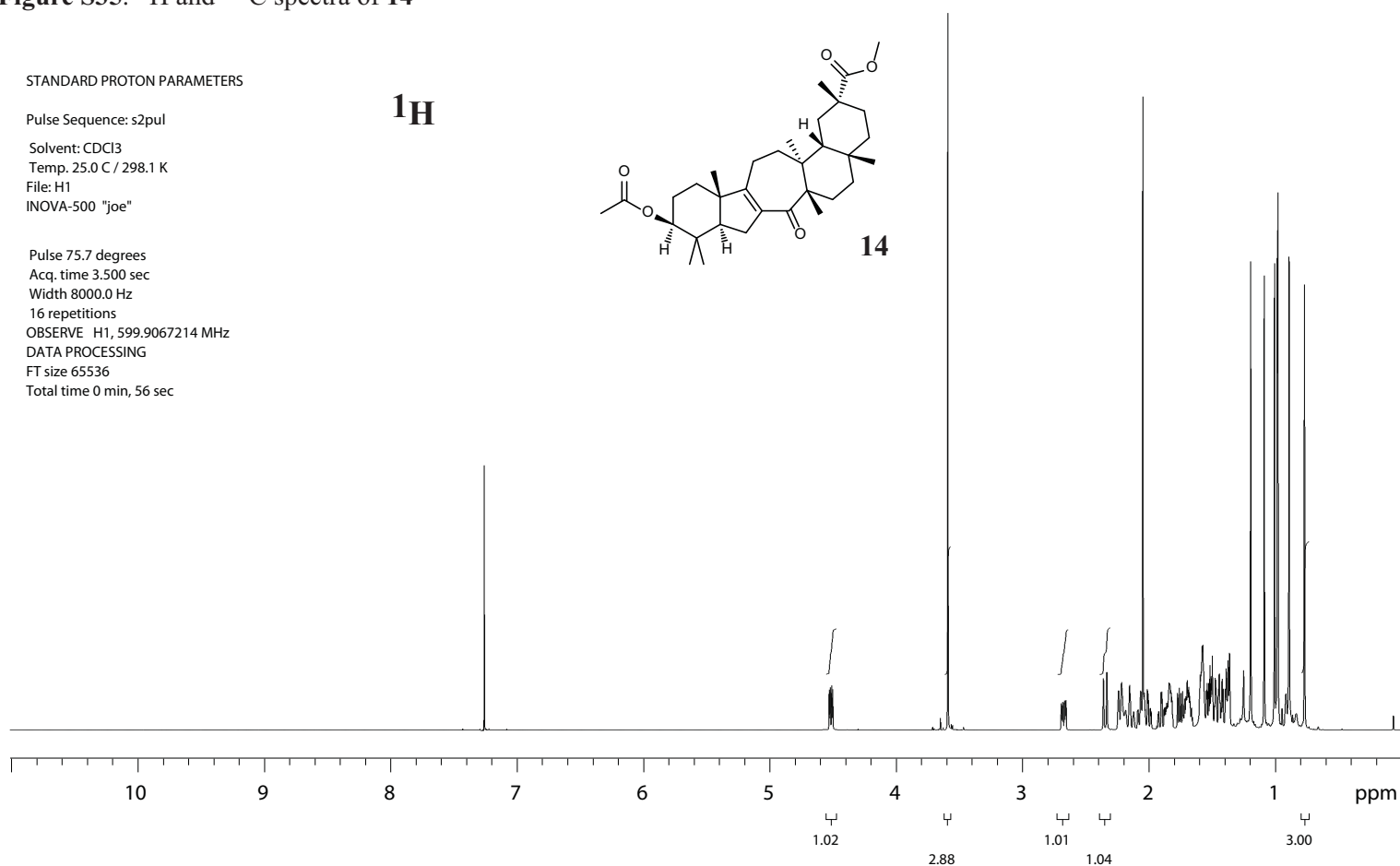
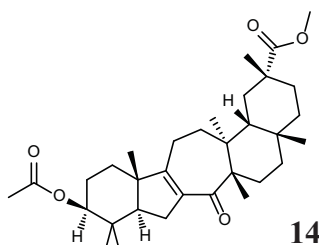
STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl_3
Temp. 25.0 C / 298.1 K
File: H1
INOVA-500 "joe"

Pulse 75.7 degrees
Acq. time 3.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.9067214 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec

^1H



STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl_3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: C13
INOVA-500 "joe"

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
1216 repetitions
OBSERVE C13, 150.8466412 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 44 min, 41 sec

^{13}C

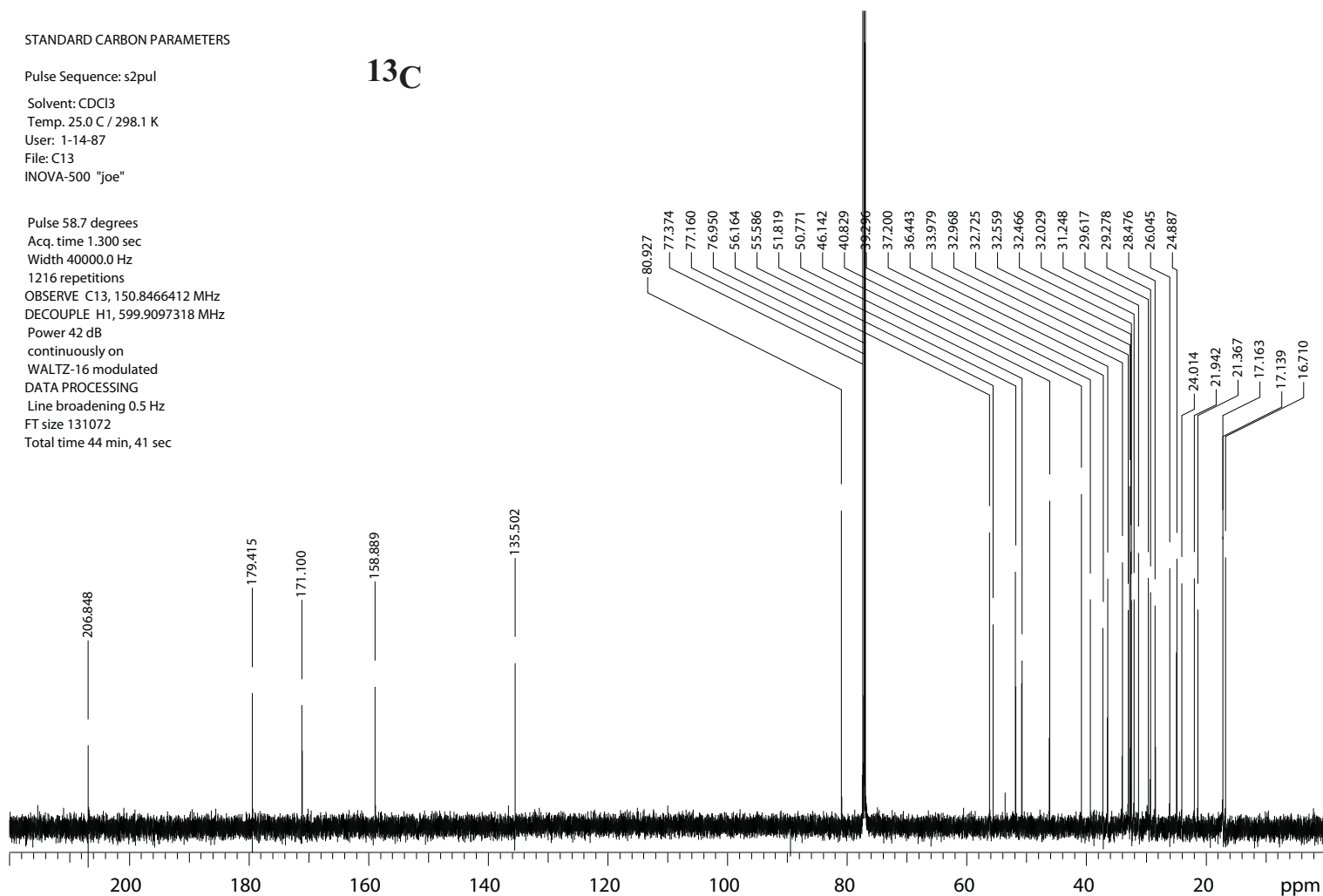


Figure S36. HMQC and HMBC spectra of 14.

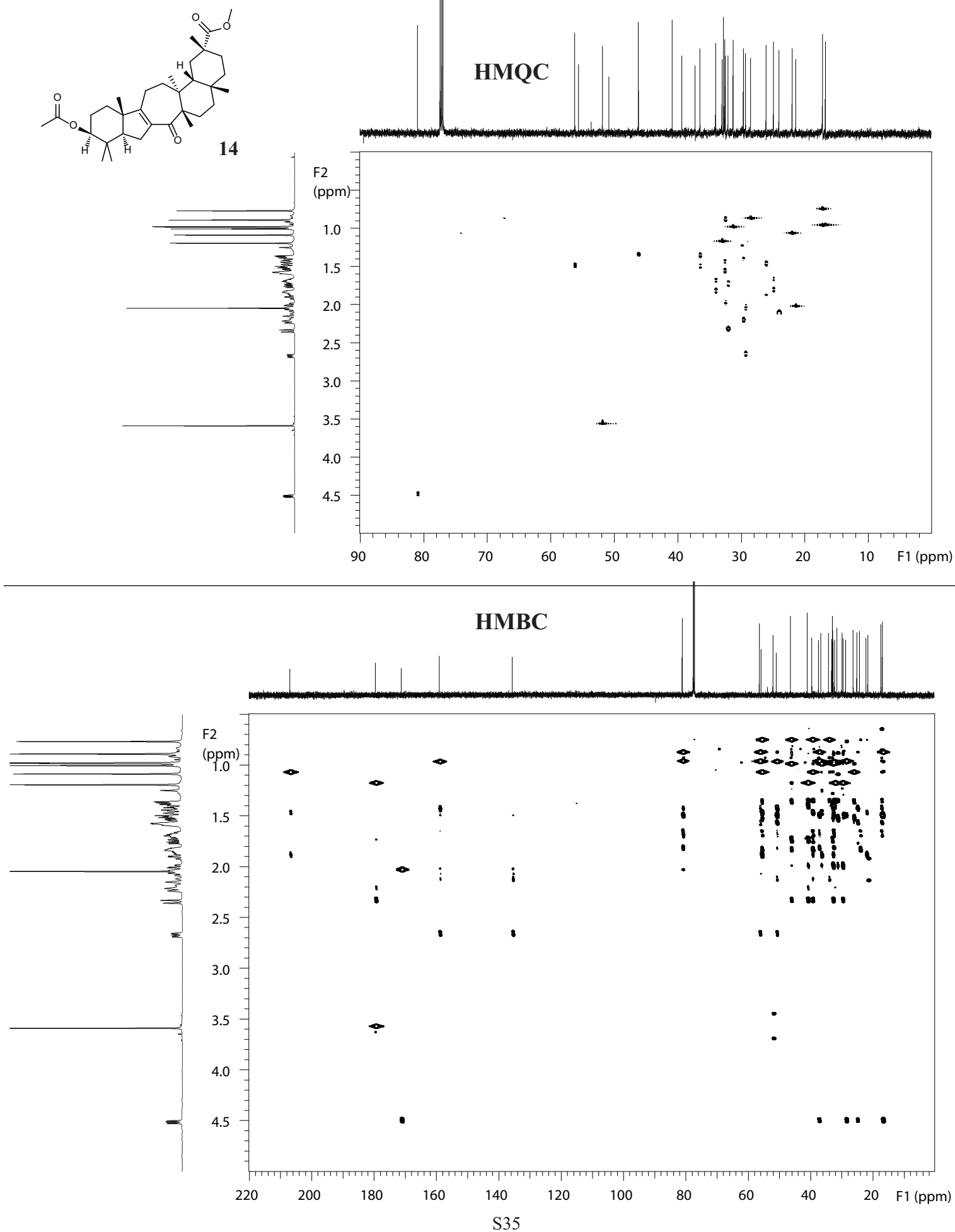


Figure S37. ¹H and ¹³C spectra of 15

vai2_15Dec2010-12:03:37

Data saved in:
chem400:/export/home/vai2/vnmrsys/data

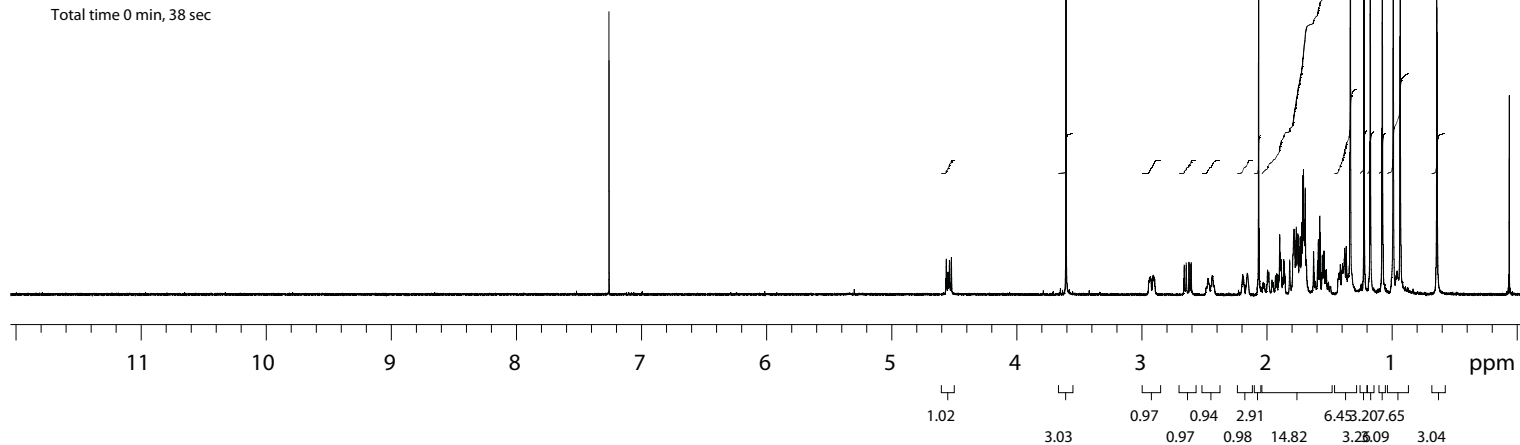
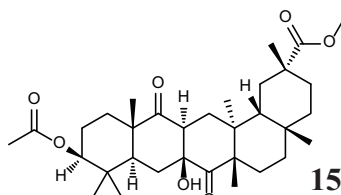
Archive directory: /export/home/vai2/vnmrsys/data
Sample directory: vai2_15Dec2010-12:03:37

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: VAI2_100
INOVA-500 "joe"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 6395.9 Hz
8 repetitions
OBSERVE H1, 399.7434712 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 38 sec

¹H



Sample 101

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: VAI2_098
INOVA-500 "joe"

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
896 repetitions
OBSERVE C13, 150.8466447 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz

¹³C

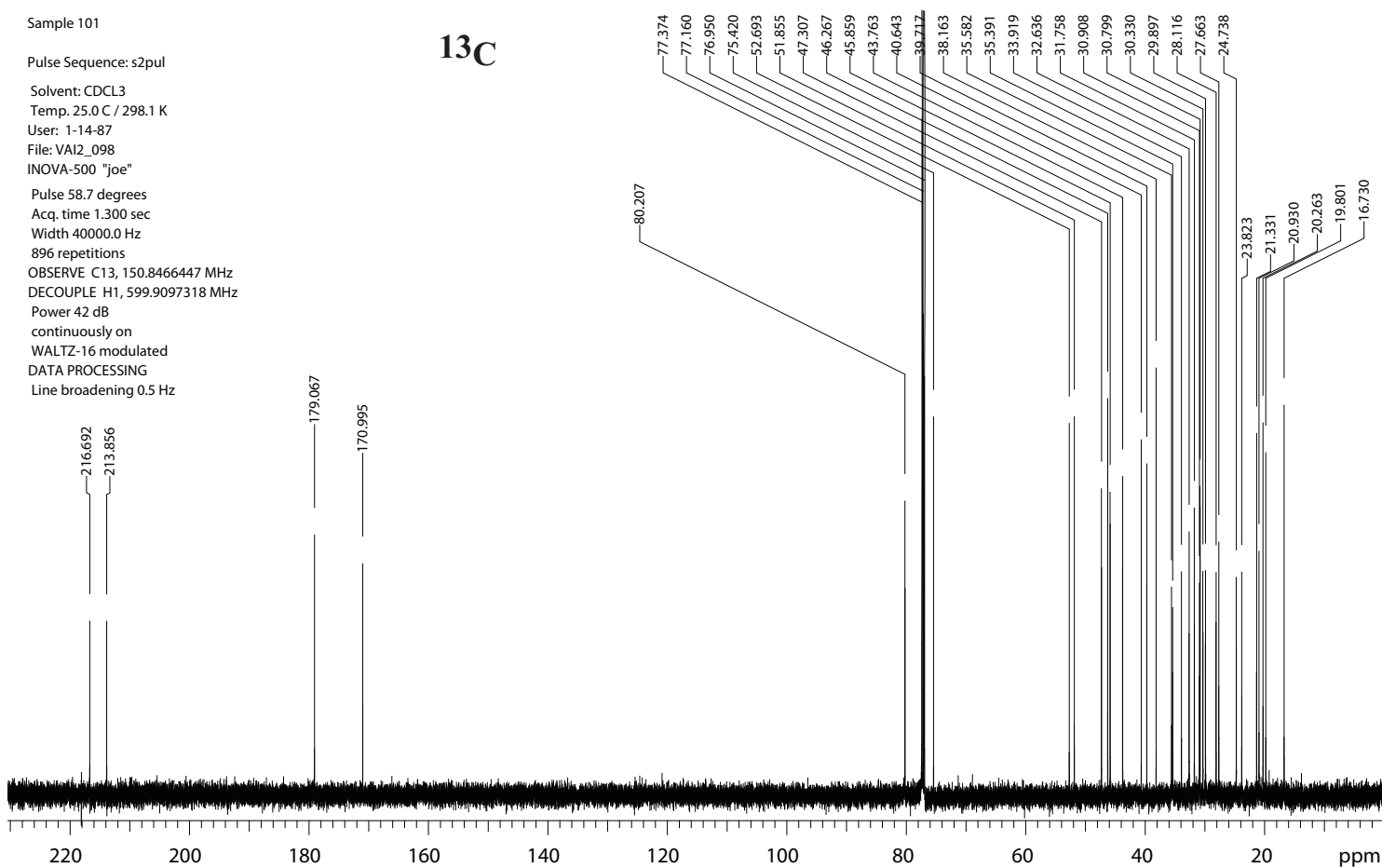


Figure S38. HMQC and HMBC spectra of 15.

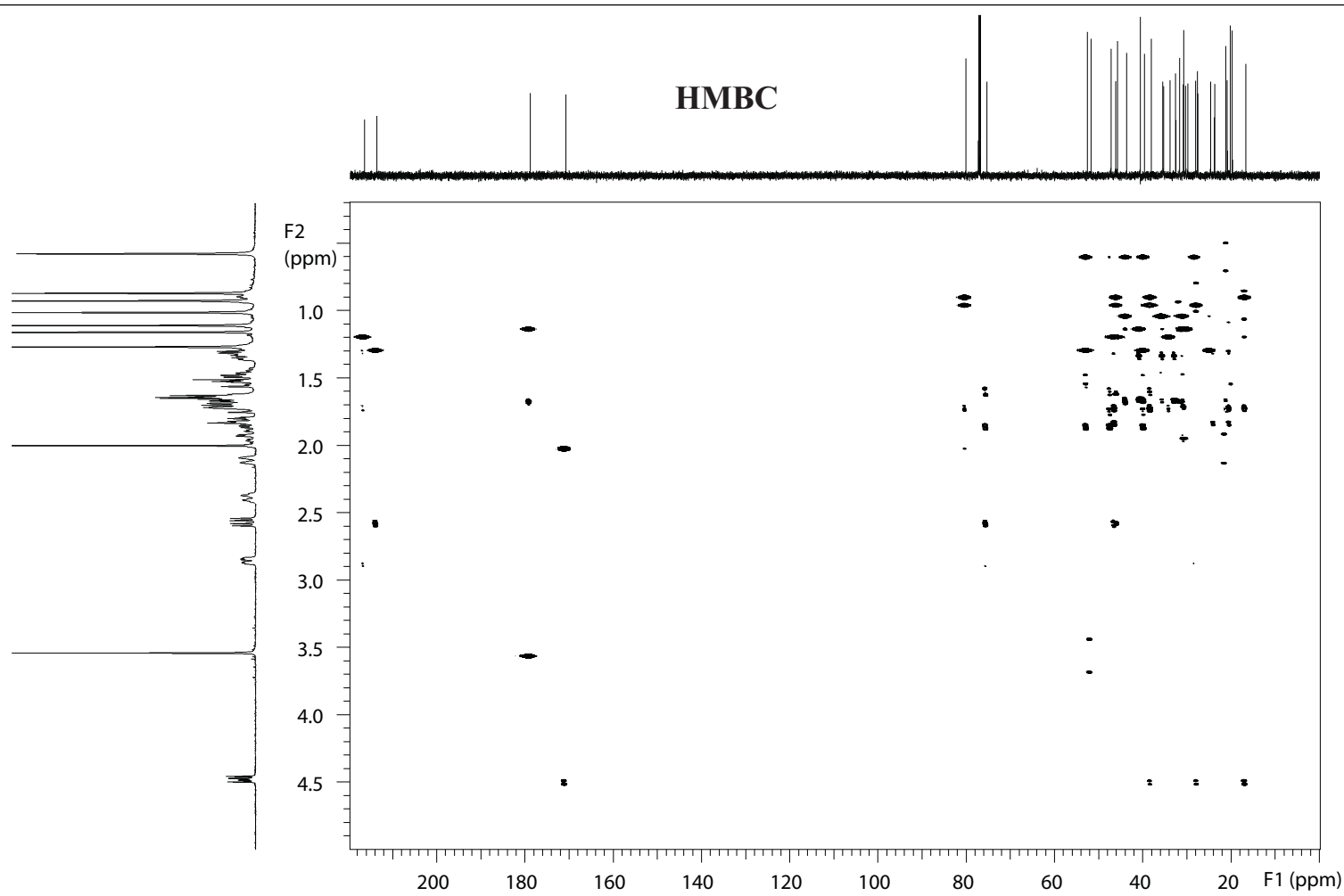
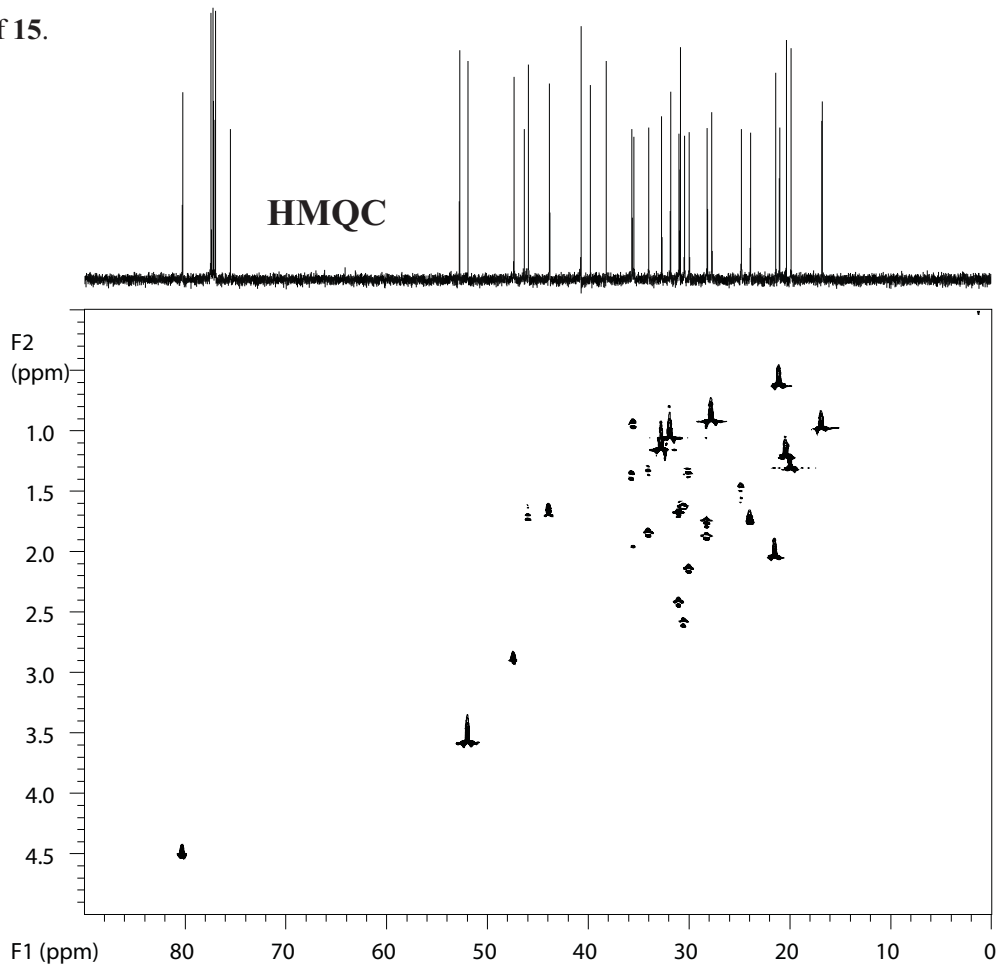
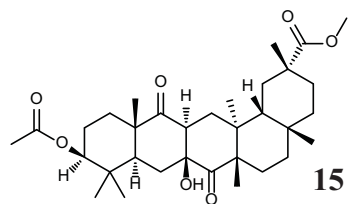
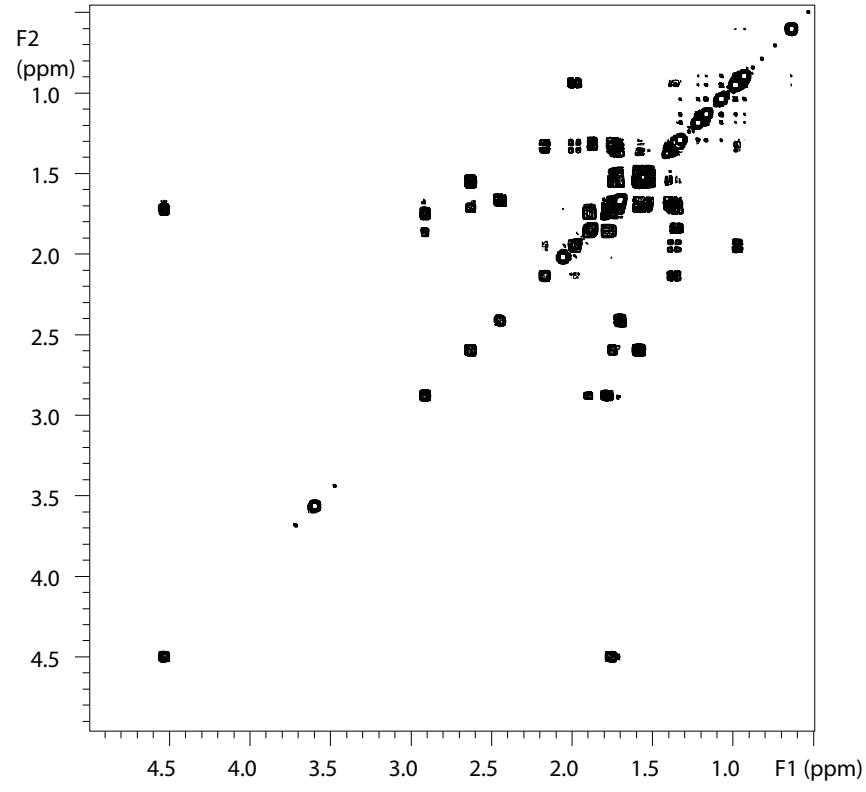
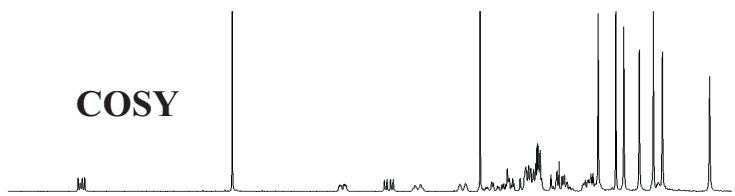
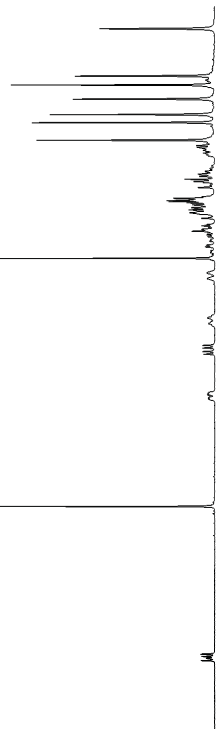
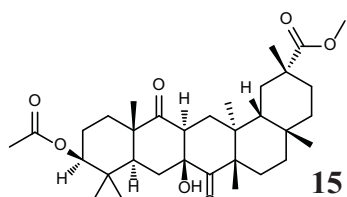


Figure S39. COSY and NOESY spectra of 15.



NOESY

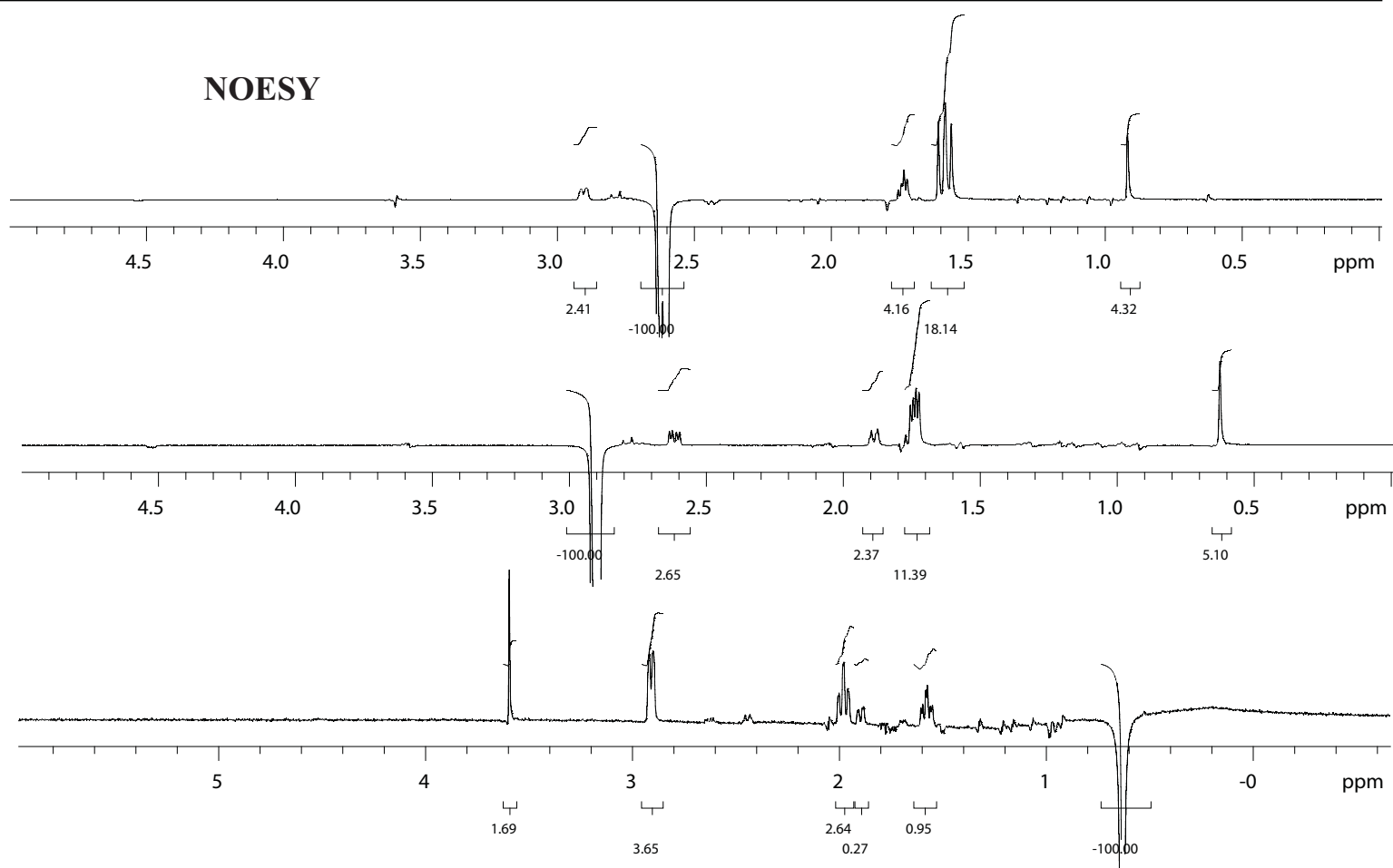


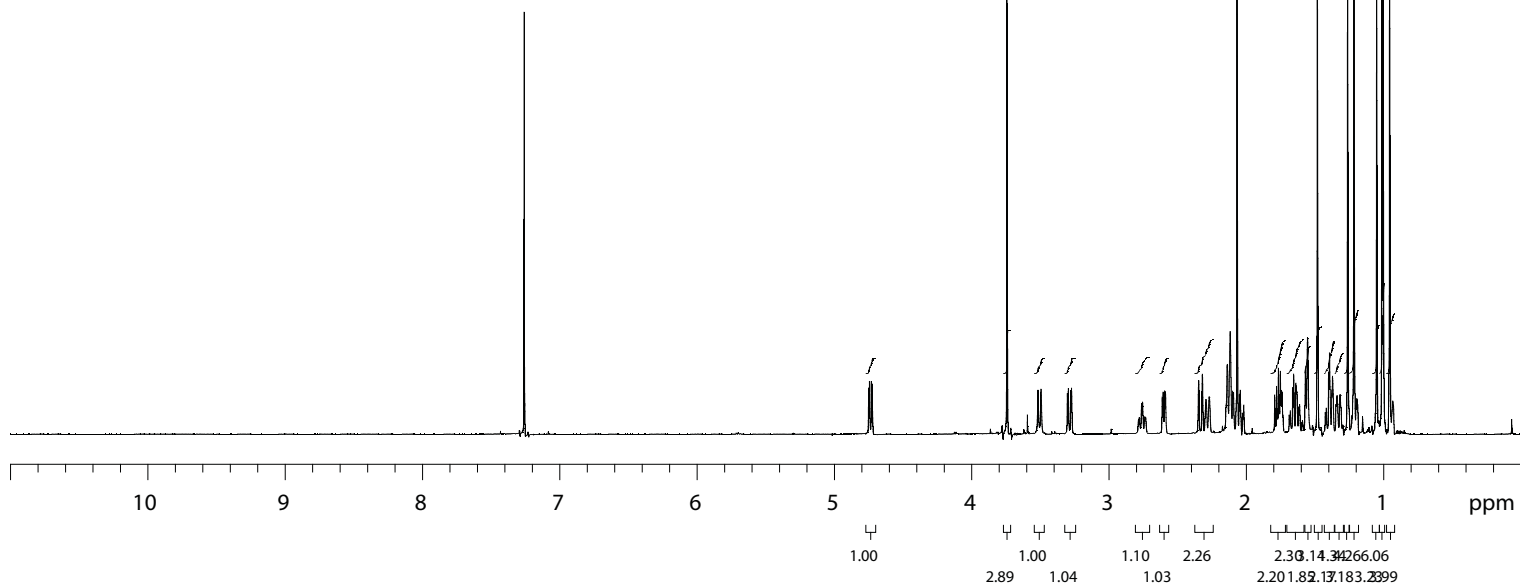
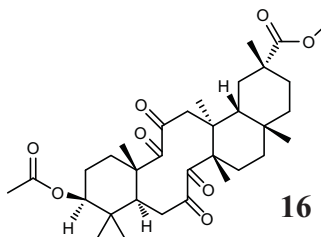
Figure S40. ^1H and ^{13}C spectra of **16**

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
 Solvent: CDCl₃
 Temp: 25.0 C / 298.1 K
 File: 1H
 INOVA-500 "joe"

Pulse 75.7 degrees
 Acq. time 3.500 sec
 Width 8000.0 Hz
 8 repetitions
 OBSERVE H1, 599.9067222 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 28 sec

^1H



Sample6

Pulse Sequence: s2pul
 Solvent: CDCl₃
 Temp: 25.0 C / 298.1 K
 User: 1-14-87
 File: 13C
 INOVA-500 "joe"

Pulse 58.7 degrees
 Acq. time 1.300 sec
 Width 40000.0 Hz
 4096 repetitions
 OBSERVE C13, 150.8466417 MHz
 DECOUPLE H1, 599.9097318 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz

^{13}C

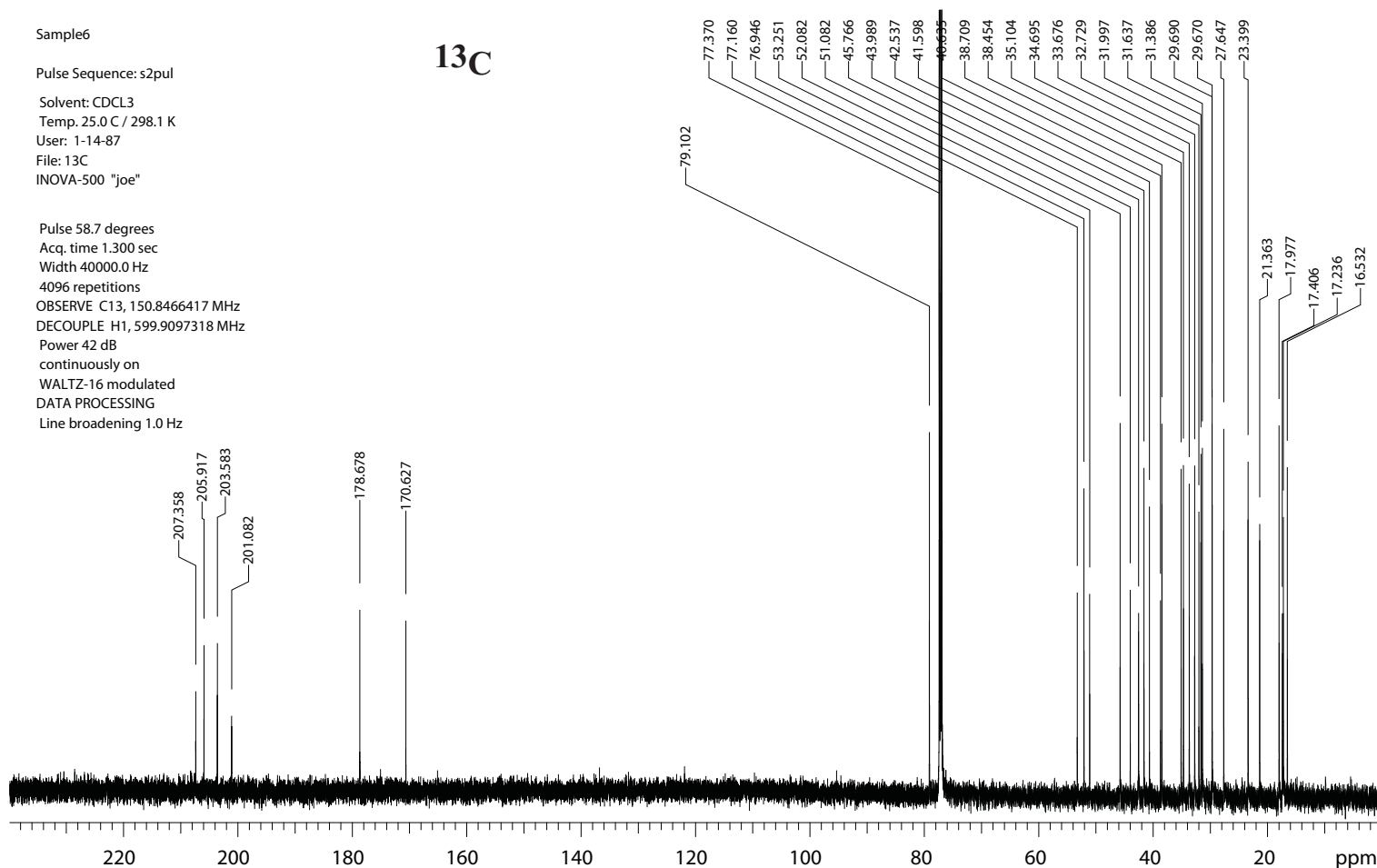


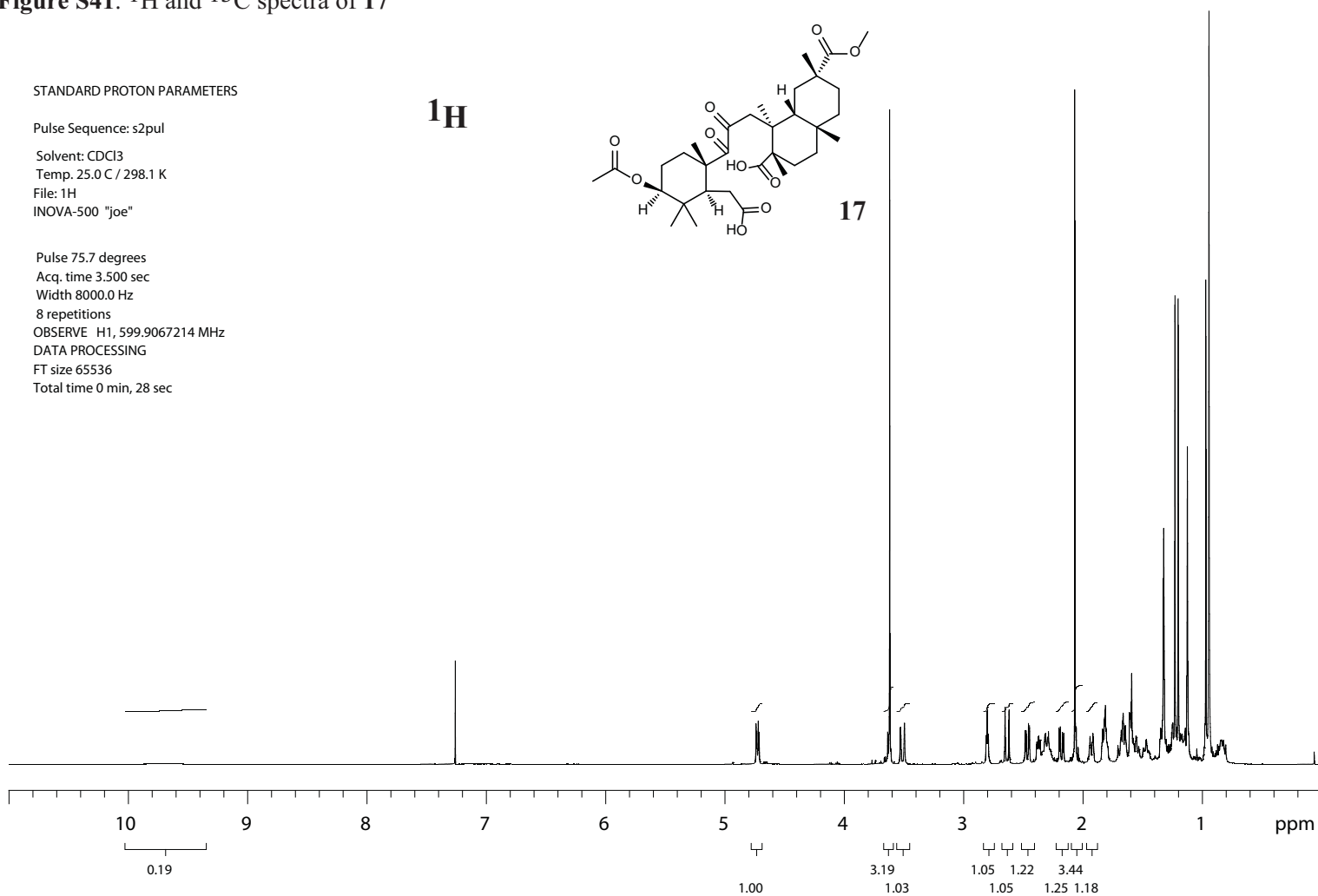
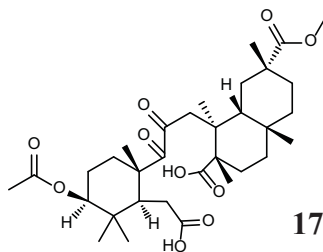
Figure S41. ¹H and ¹³C spectra of 17

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
File: 1H
INOVA-500 "joe"

Pulse 75.7 degrees
Acq. time 3.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 599.9067214 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 28 sec

¹H



Sample6

Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: 13C
INOVA-500 "joe"

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
4096 repetitions
OBSERVE C13, 150.8466429 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz

¹³C

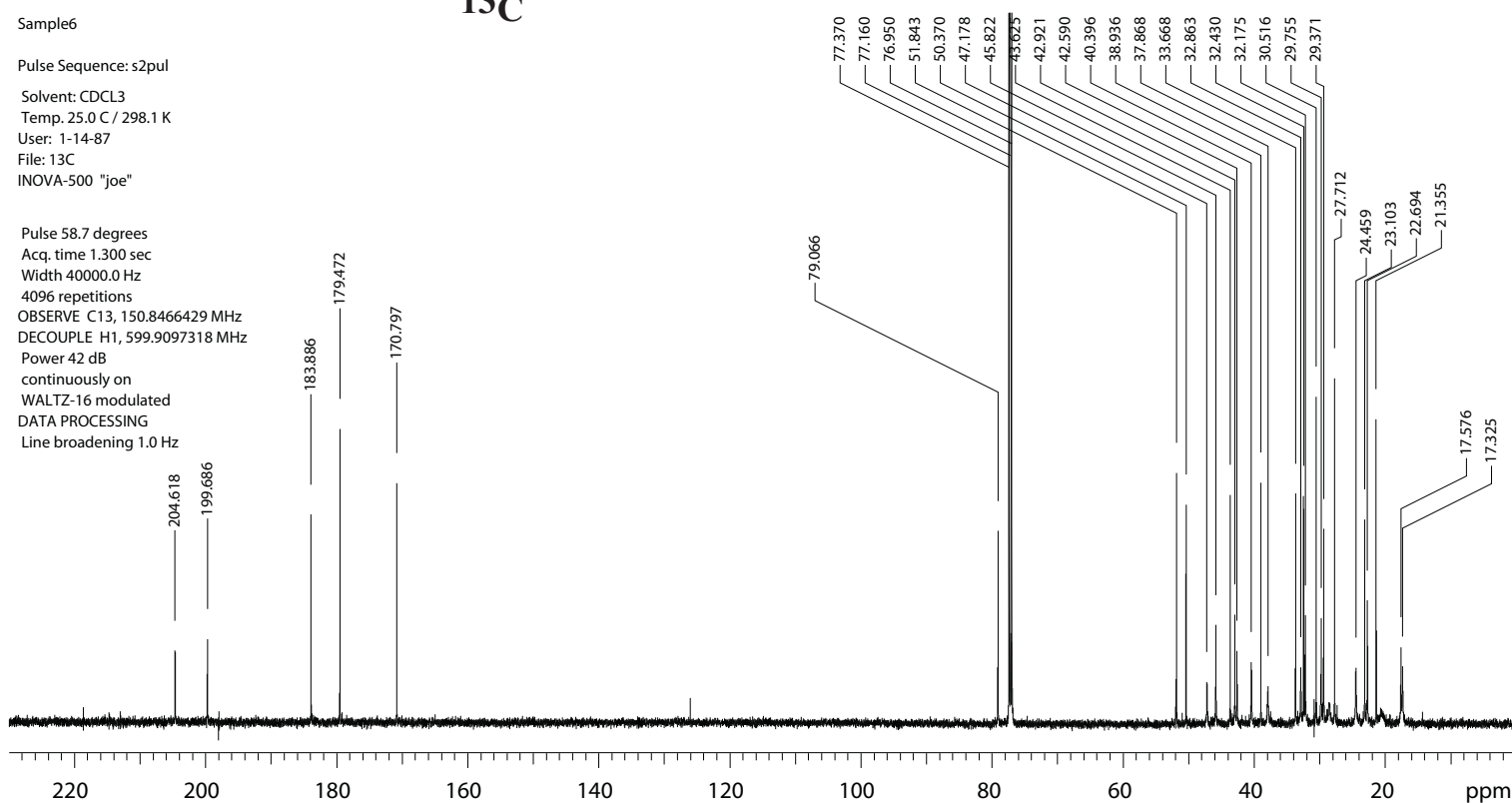


Figure S42. HMQC and HMBC spectra of 17.

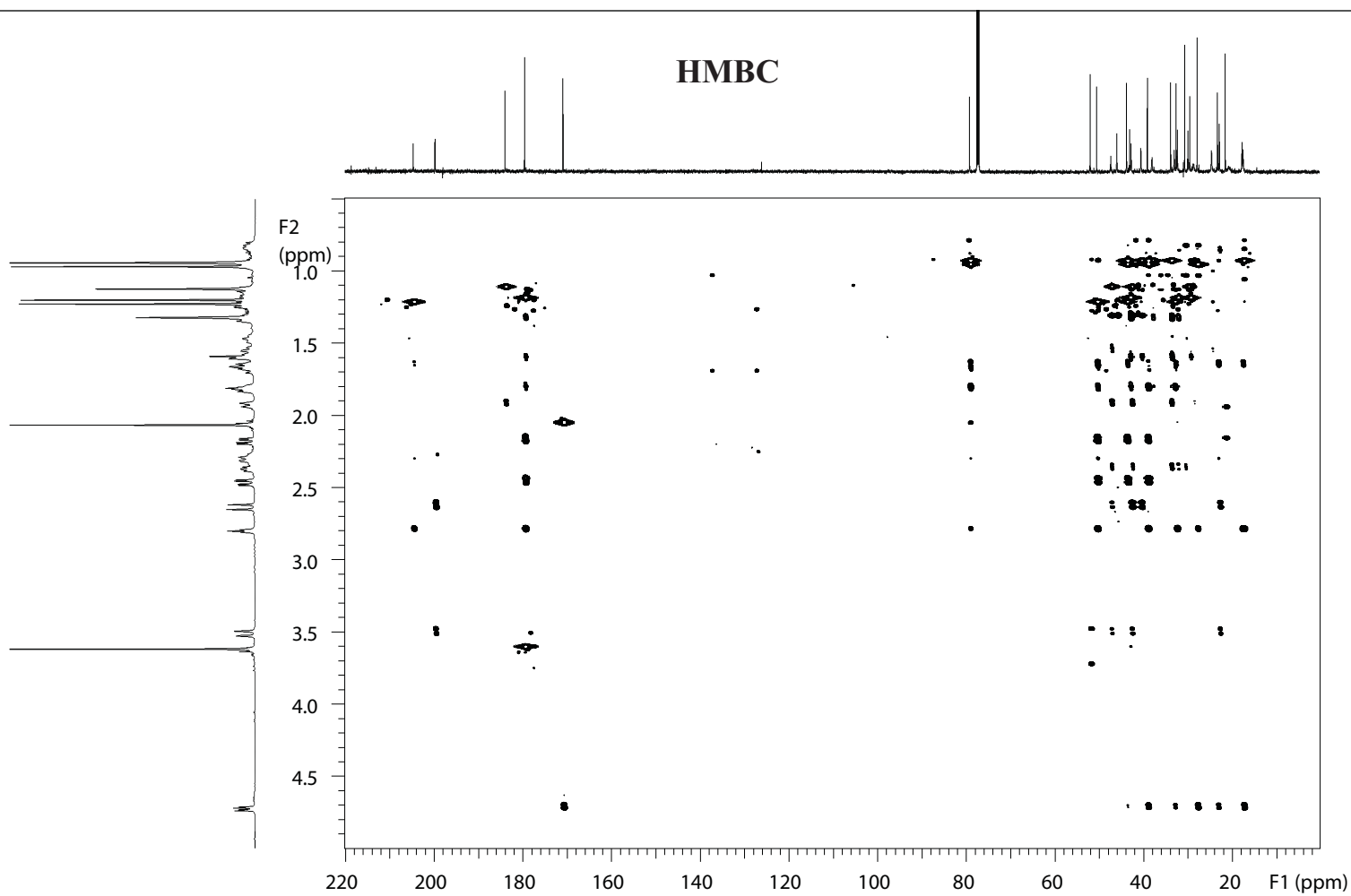
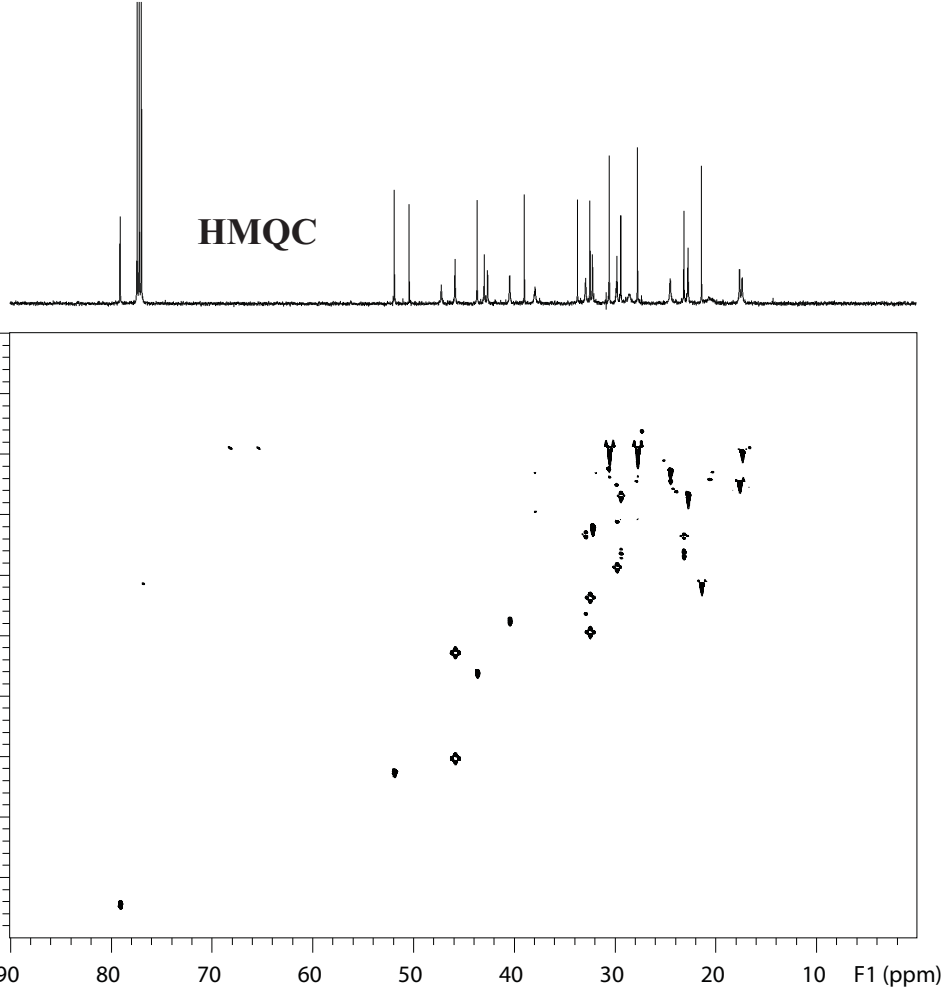
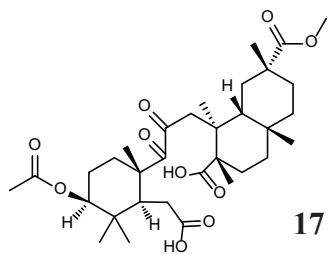


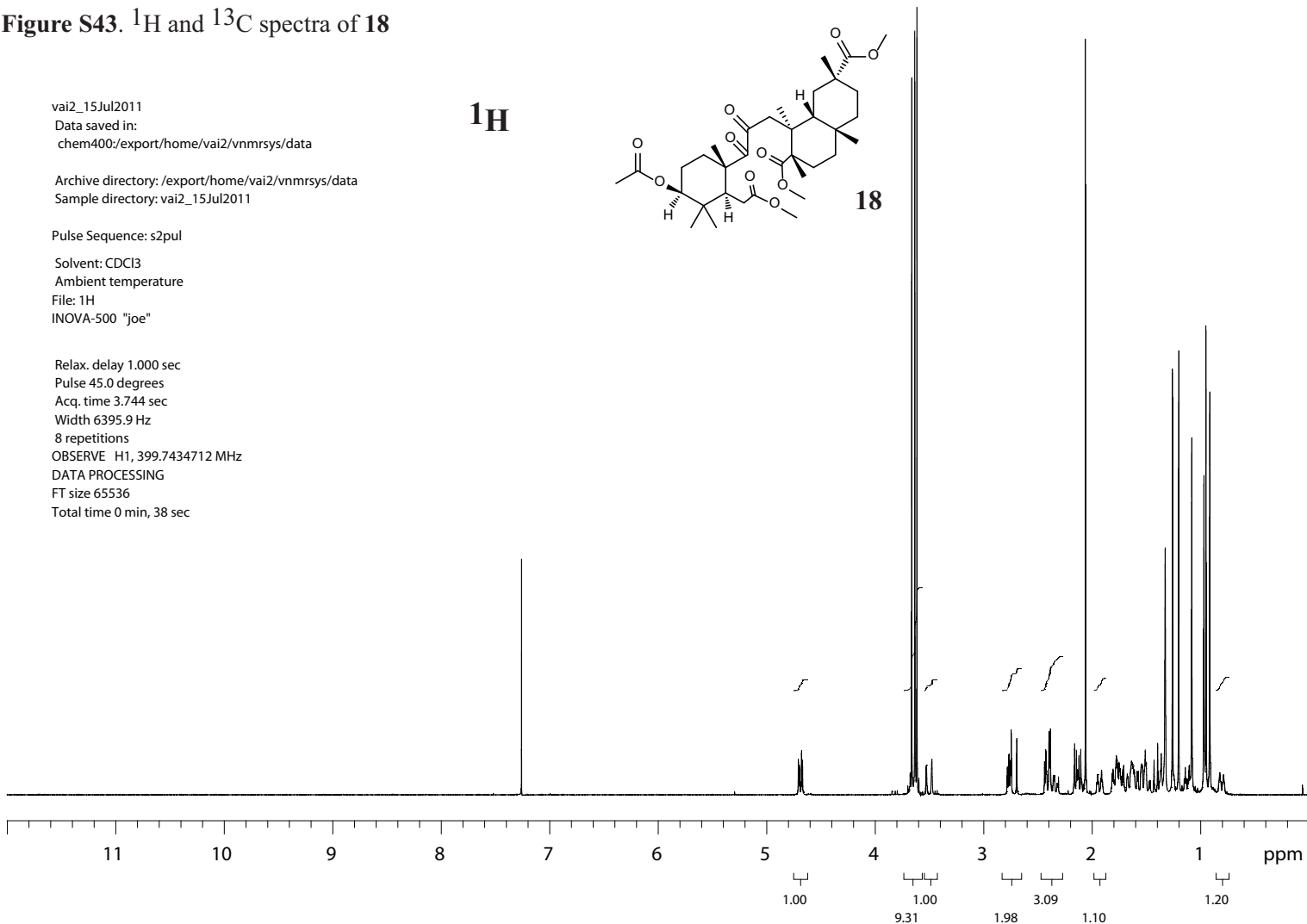
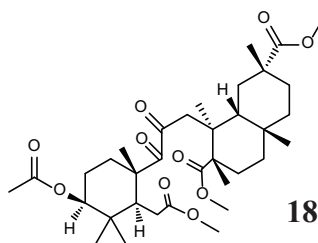
Figure S43. ¹H and ¹³C spectra of 18

vai2_15Jul2011
Data saved in:
chem400/export/home/vai2/vnmrsys/data
Archive directory: /export/home/vai2/vnmrsys/data
Sample directory: vai2_15Jul2011

Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File: 1H
INOVA-500 "Joe"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 6395.9 Hz
8 repetitions
OBSERVE H1, 399.7434712 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 38 sec

¹H



Sample6
Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-600 "chem600"

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
4096 repetitions
OBSERVE C13, 150.8466425 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 29 min, 23 sec

¹³C

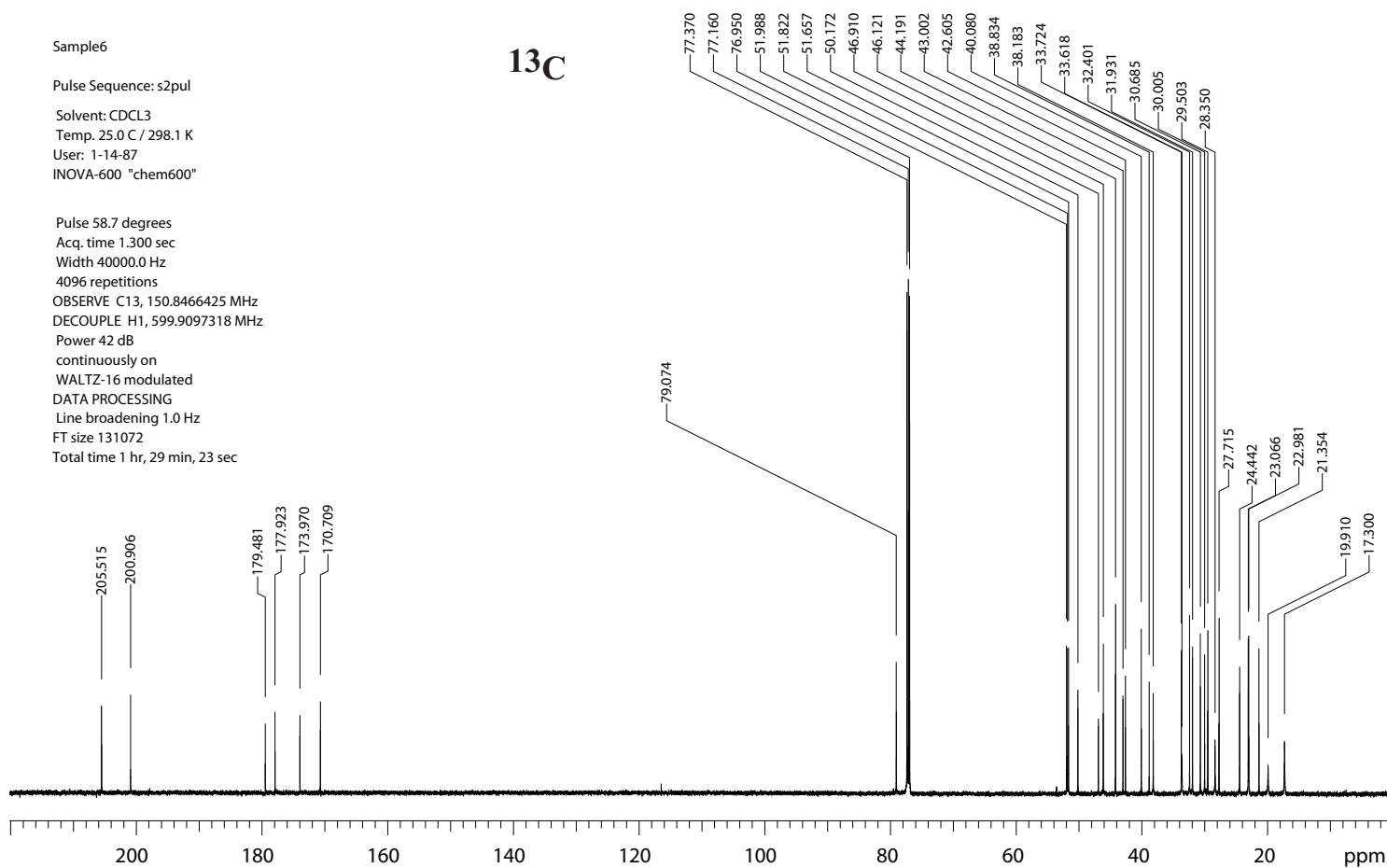


Figure S44. ^1H and ^{13}C spectra of 19

Jan28_2011

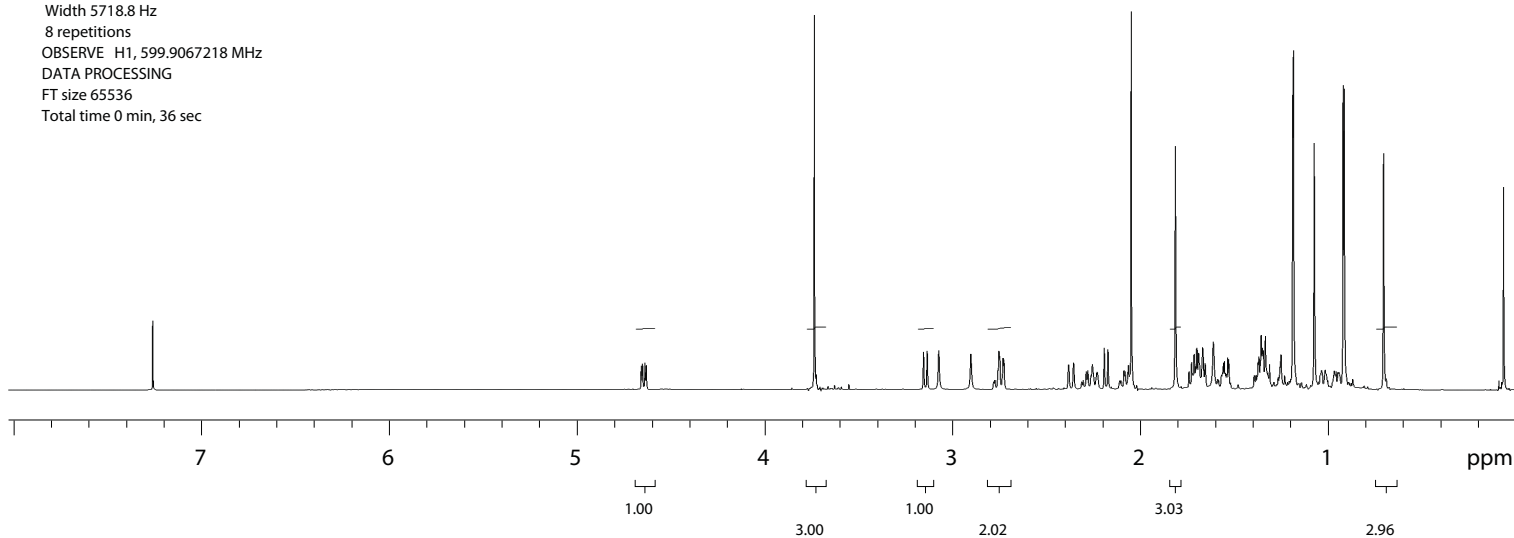
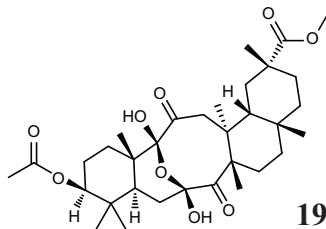
Archive directory: /export/home/greg/vnmrsys/data
Sample directory: Sample2_25Jan2011
File: H11D

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
File: H11D
INOVA-500 "joe"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 5718.8 Hz
8 repetitions
OBSERVE H1, 599.9067218 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 36 sec

^1H



Sample 4

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: C13
INOVA-500 "joe"

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
1044 repetitions
OBSERVE C13, 150.8466417 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 29 min, 10 sec

^{13}C

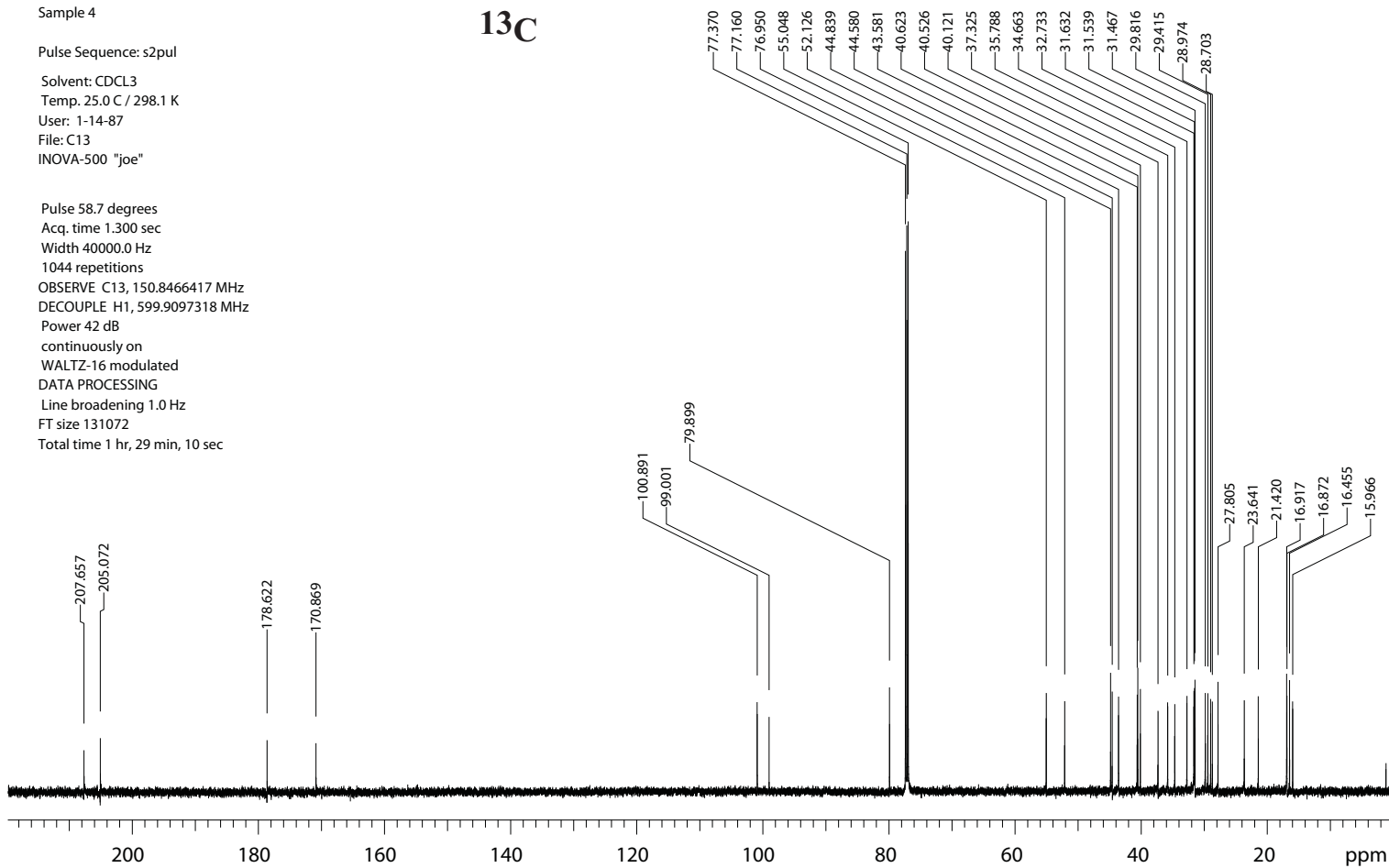


Figure S45. HMQC and HMBC spectra of 19.

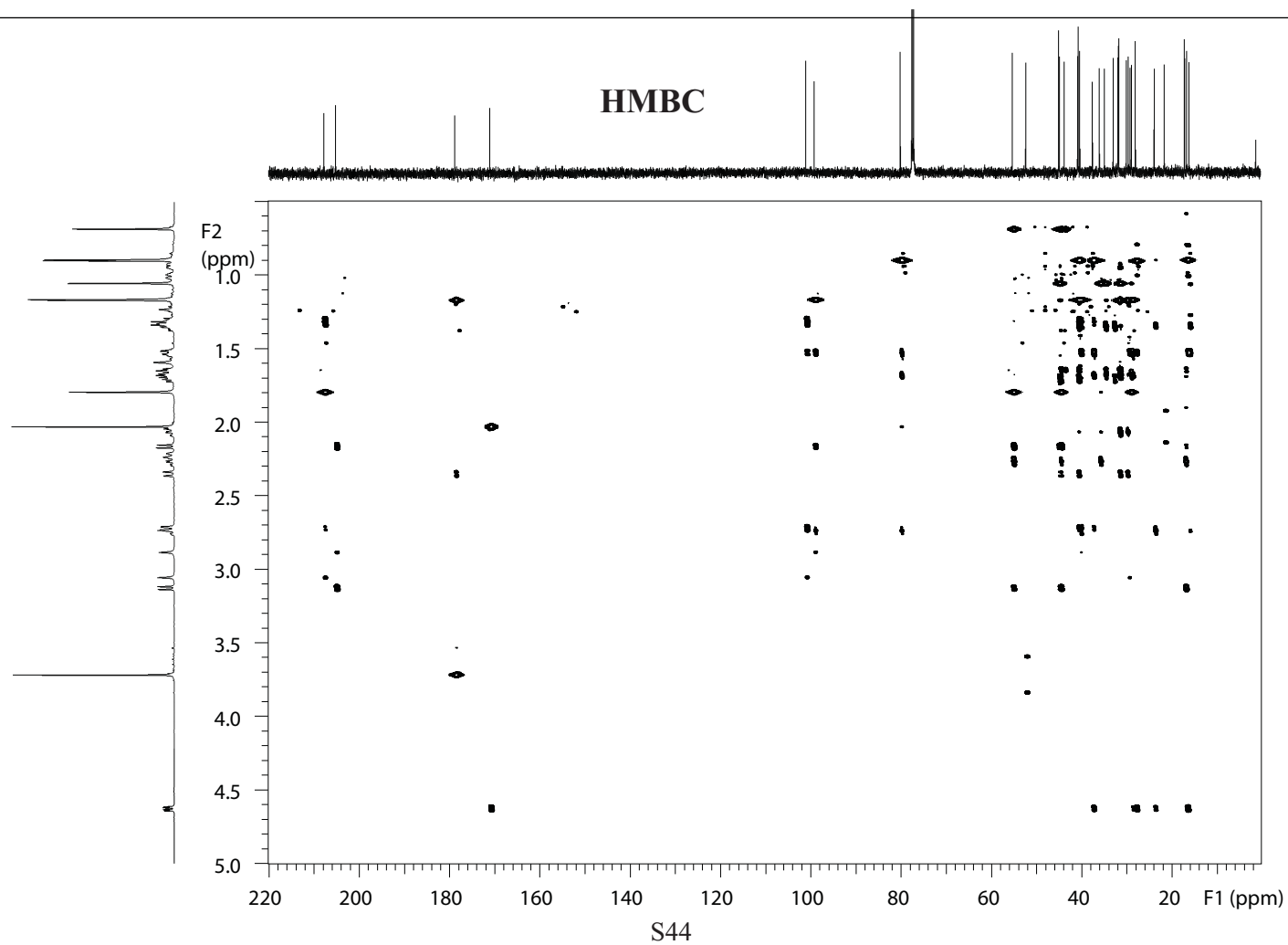
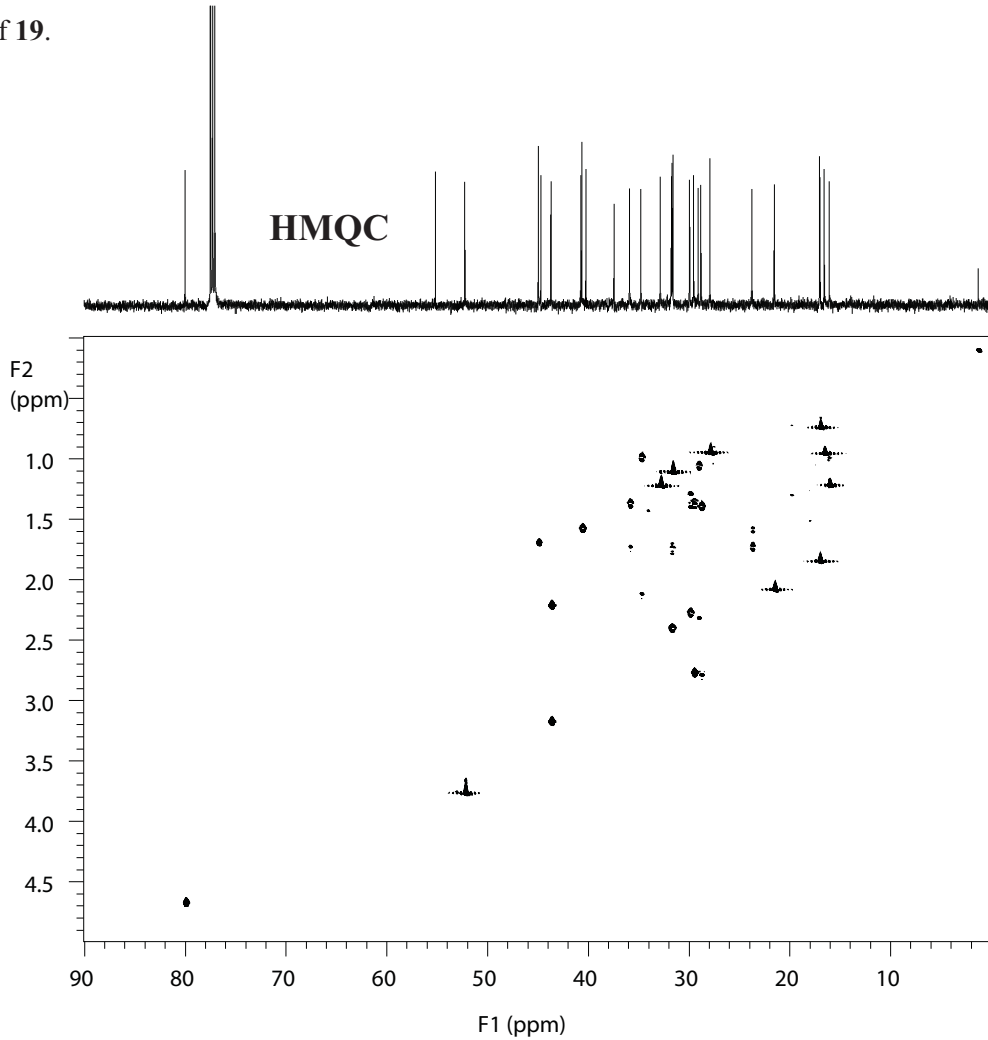
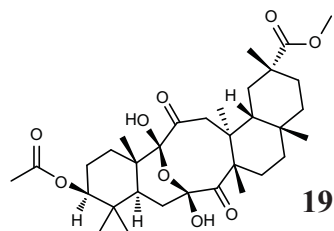


Figure S46. COSY and NOESY spectra of 19.

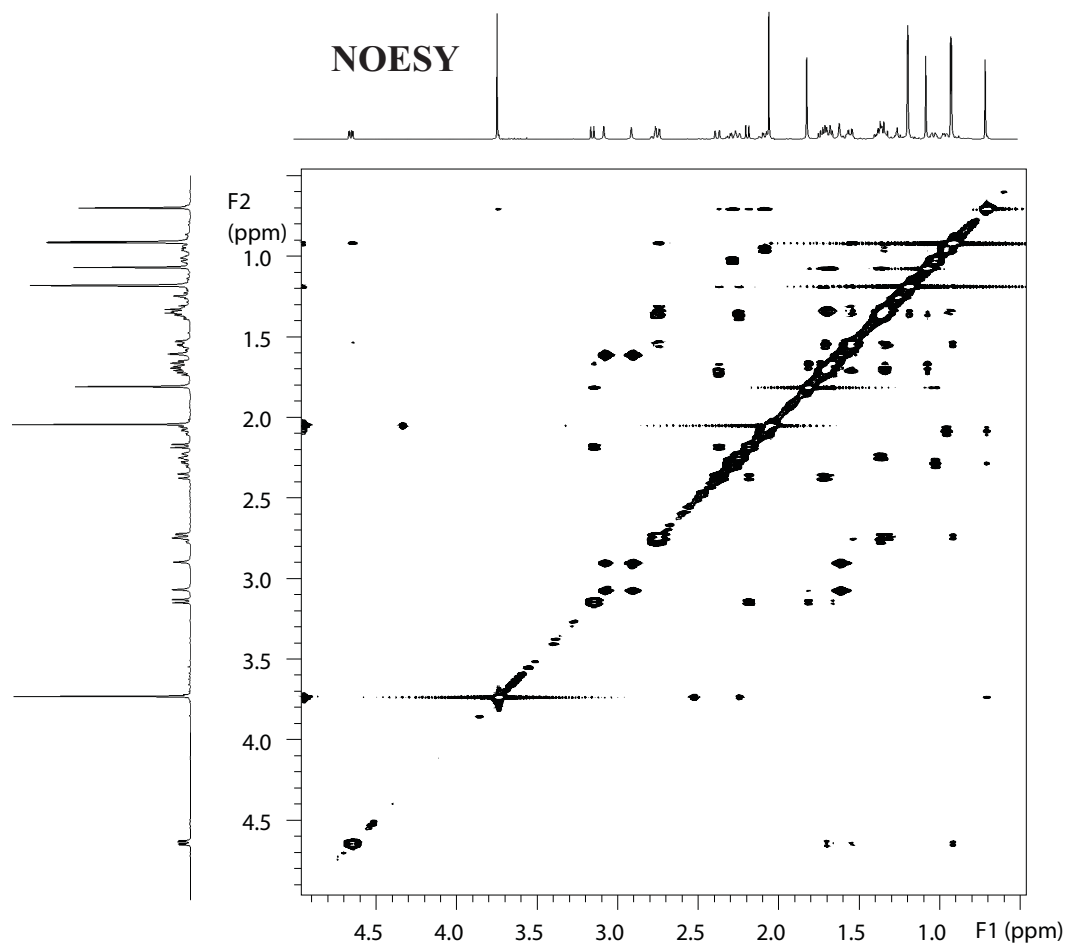
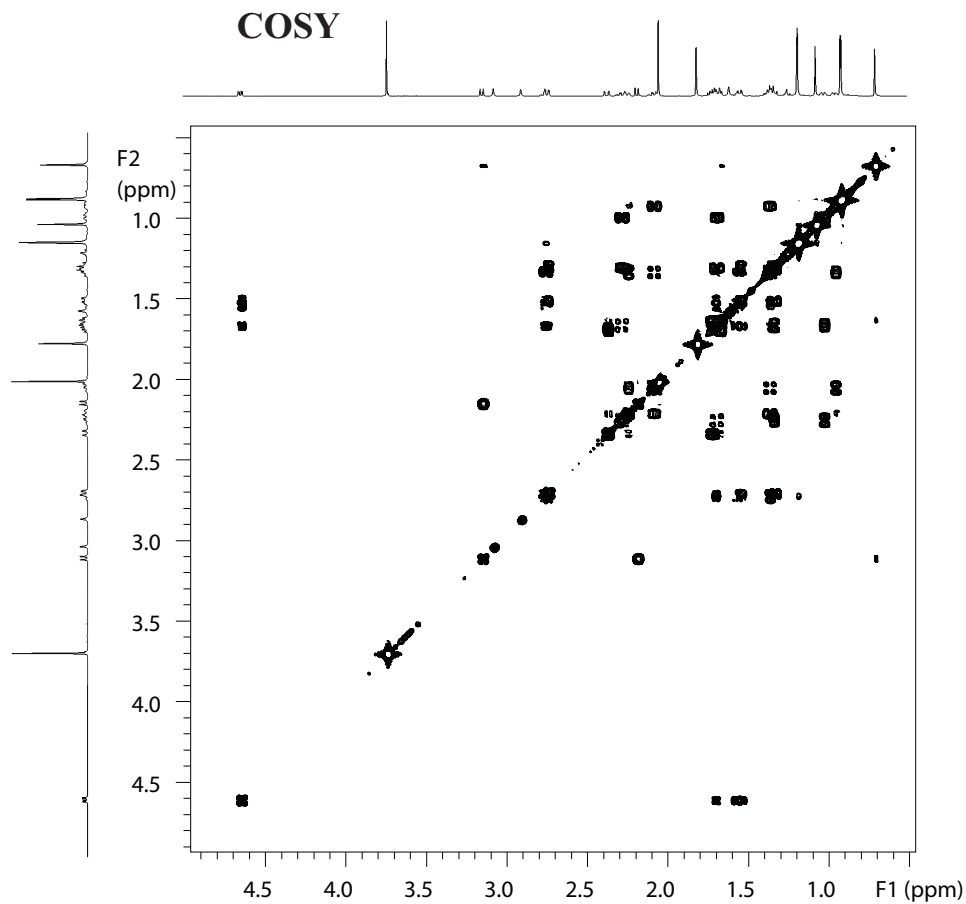
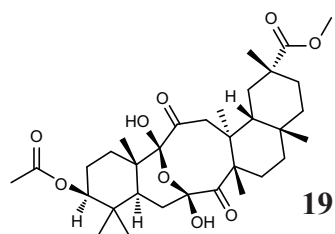


Figure S47. ^1H and ^{13}C spectra of **20**

STANDARD PROTON PARAMETERS

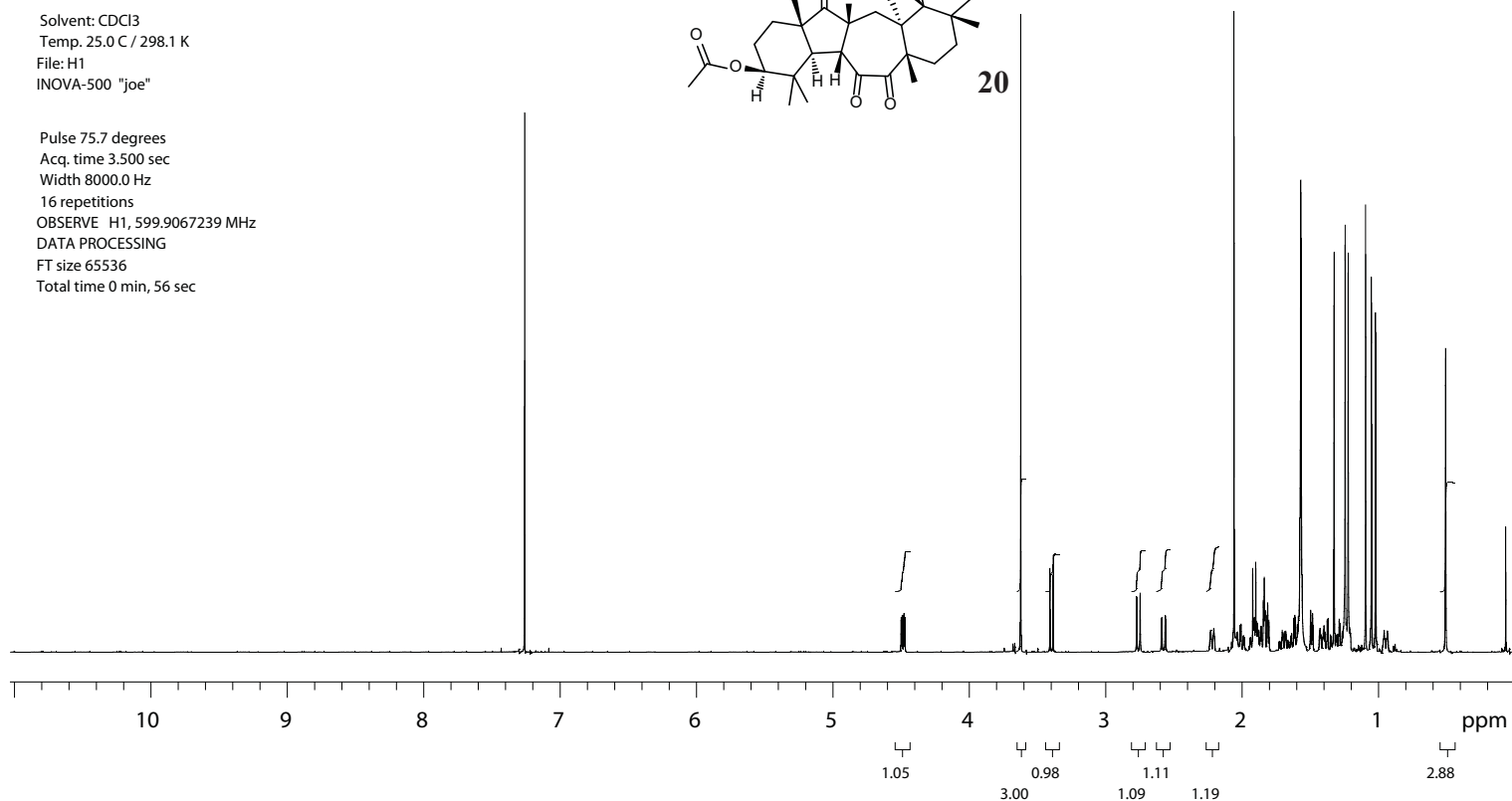
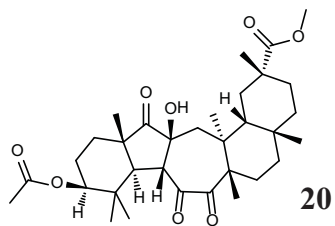
Pulse Sequence: s2pul

Solvent: CDCl_3
Temp. 25.0 C / 298.1 K

File: H1
INOVA-500 "joe"

Pulse 75.7 degrees
Acq. time 3.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.9067239 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec

^1H



^{13}C

Sample6

Pulse Sequence: s2pul

Solvent: CDCl_3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-600 "chem600"

Pulse 58.7 degrees
Acq. time 1.300 sec
Width 40000.0 Hz
4096 repetitions
OBSERVE C13, 150.8466407 MHz
DECOUPLE H1, 599.9097318 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 29 min, 23 sec

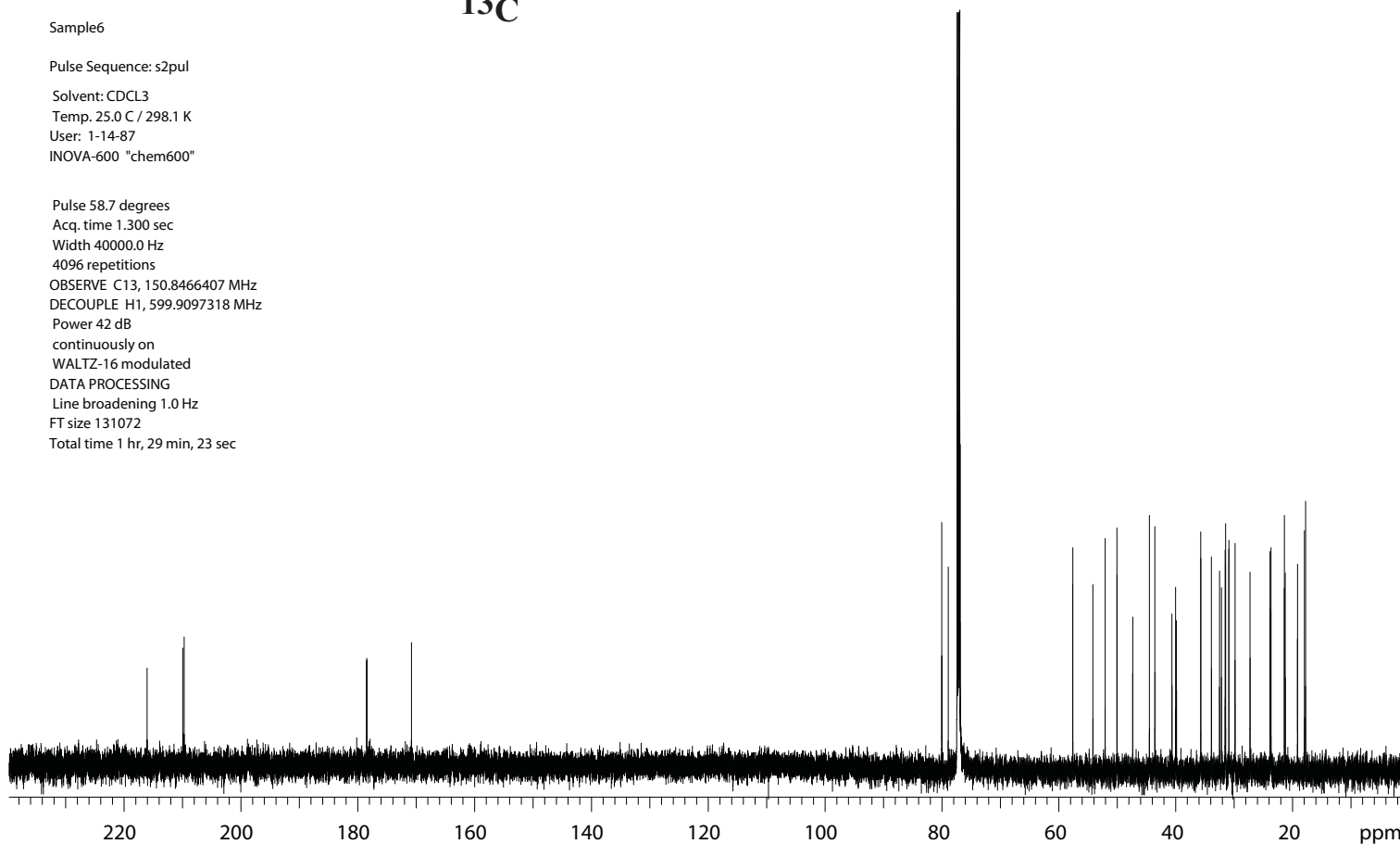


Figure S48. HMQC and HMBC spectra of **20**.

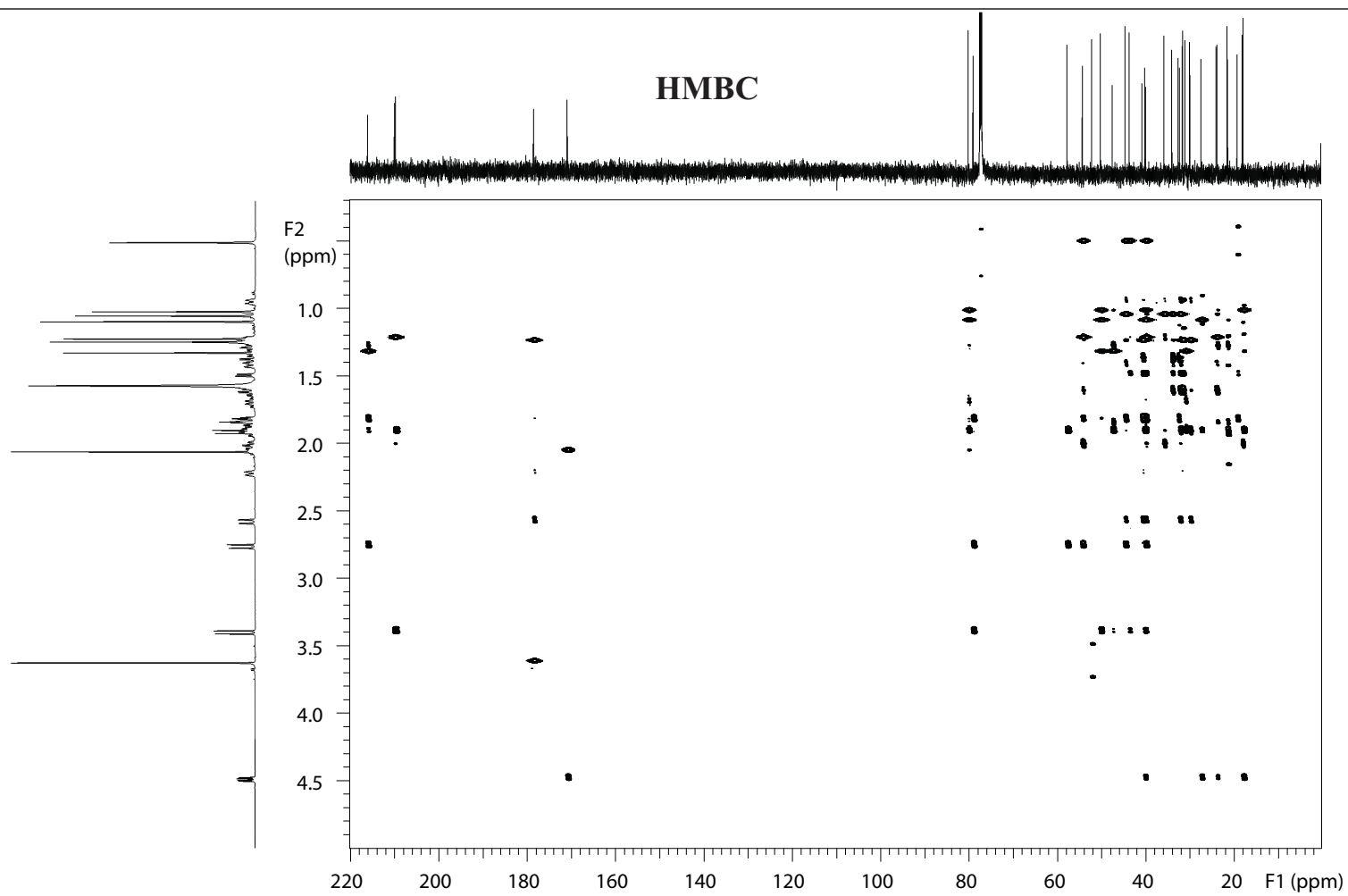
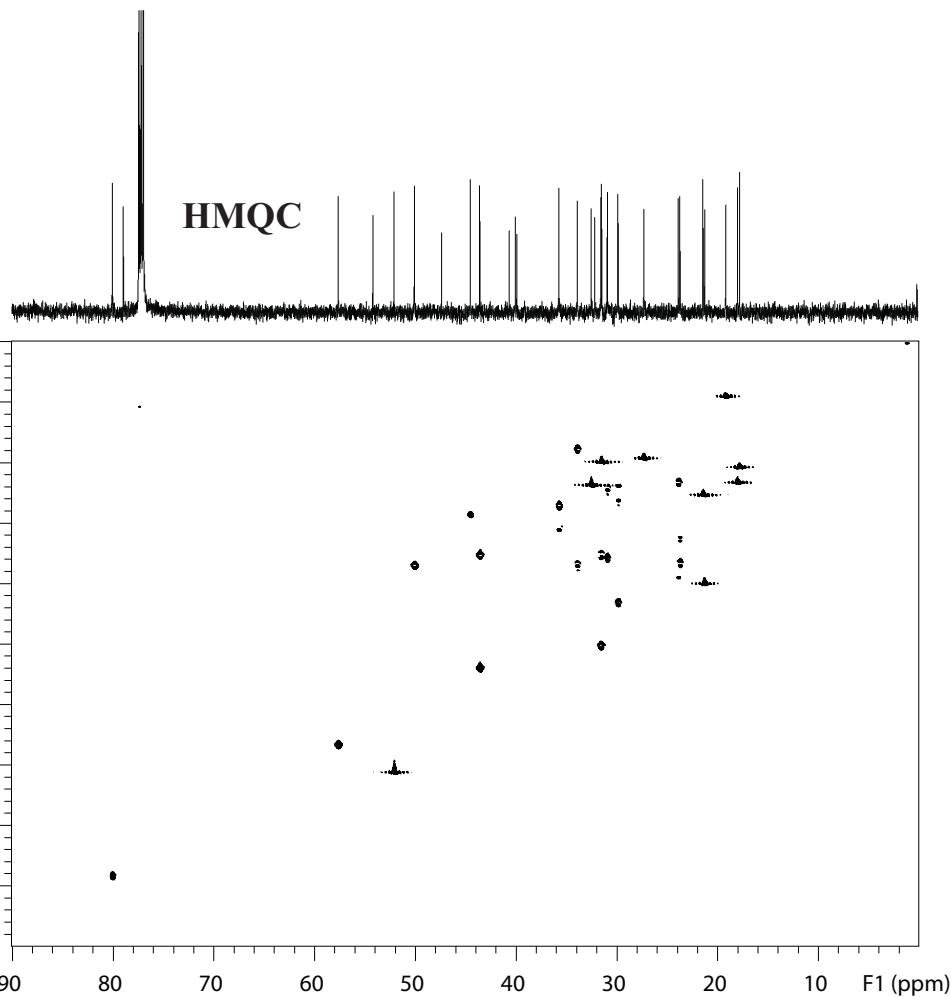
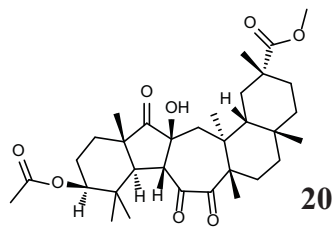
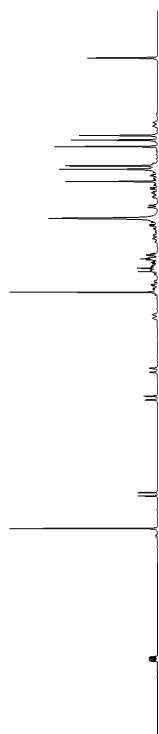
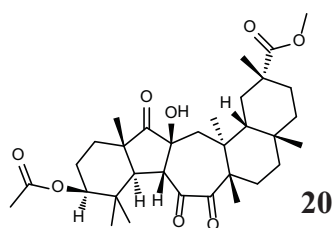
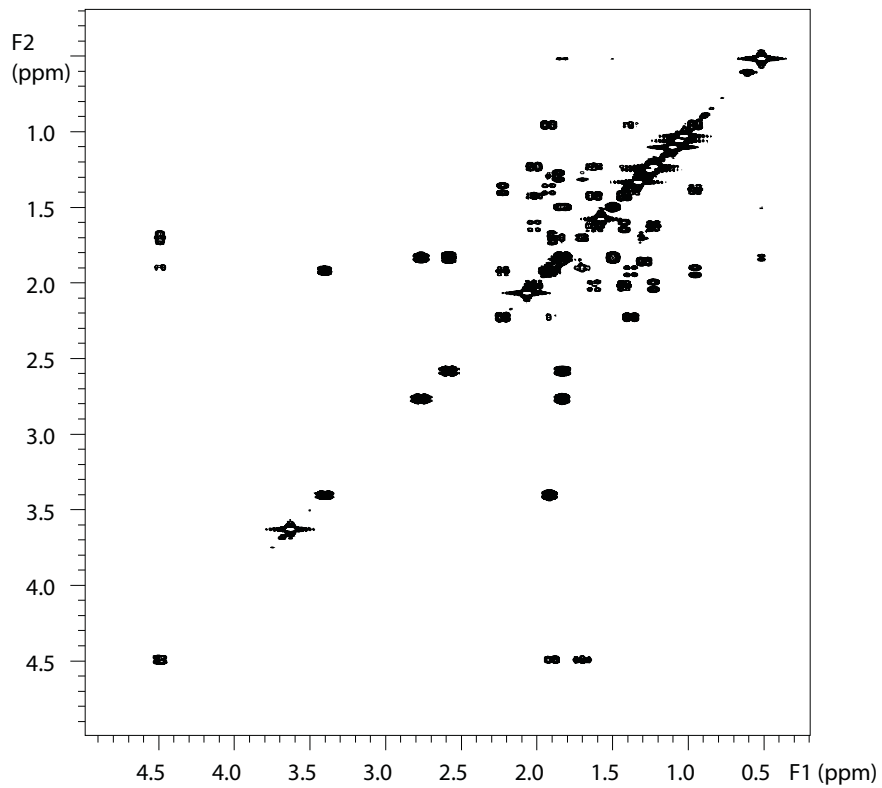


Figure S49. COSY and NOESY spectra of **20**.



COSY



NOESY

