

Article

New metabolites from the marine sponge *Scopalina hapalia* collected in Mayotte lagoon

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Figure S1. HRMS-ESI ⁺ spectrum of sinularone J (1).	1
Figure S2. ¹ H NMR spectrum of sinularone J (1) (600 MHz, in CD ₃ OD).	2
Figure S3. ¹³ C NMR spectrum of sinularone J (1) (150 MHz, in CD ₃ OD).	3
Figure S4. ¹ H- ¹ H COSY spectrum of sinularone J (1) (600 MHz, in CD ₃ OD).	4
Figure S5. HSQC spectrum of sinularone J (1) (600 MHz, in CD ₃ OD).	5
Figure S6. HMBC spectrum of sinularone J (1) (600 MHz, in CD ₃ OD).	6
Figure S7. HRMS-ESI ⁺ spectrum of sinularone K (2).	7
Figure S8. ¹ H NMR spectrum of sinularone K (2) (600 MHz, in CD ₃ OD).	8
Figure S9. ¹ H- ¹ H COSY spectrum of sinularone K (2) (600 MHz, in CD ₃ OD).	9
Figure S10. HSQC spectrum of sinularone K (2) (600 MHz, in CD ₃ OD).	10
Figure S11. HMBC spectrum of sinularone K (2) (600 MHz, in CD ₃ OD).	11
Figure S12. HRMS-ESI-TOF spectrum of 1-O-octadecyl-2-pentanoyl- <i>sn</i> -glycero-3-phosphocholine (3).	12
Figure S13. ¹ H NMR spectrum of 1-O-octadecyl-2-pentanoyl- <i>sn</i> -glycero-3-phosphocholine (3) (600 MHz, in CD ₃ OD).	13
Figure S14. ¹ H- ¹ H COSY spectrum of 1-O-octadecyl-2-pentanoyl- <i>sn</i> -glycero-3-phosphocholine (3) (600 MHz, in CD ₃ OD).	14
Figure S15. HSQC spectrum of 1-O-octadecyl-2-pentanoyl- <i>sn</i> -glycero-3-phosphocholine (3) (600 MHz, in CD ₃ OD).	14
Figure S16. HMBC spectrum of 1-O-octadecyl-2-pentanoyl- <i>sn</i> -glycero-3-phosphocholine (3) (600 MHz, in CD ₃ OD).	15
Figure S17. HRMS ESI-TOF spectrum of 1-O-(3-methox-tetradecanoyl)- <i>sn</i> -glycero-3-phosphocholine (4).	16
Figure S18. ¹ H NMR spectrum of 1-O-(3-methox-tetradecanoyl)- <i>sn</i> -glycero-3-phosphocholine (4) (600 MHz, in CD ₃ OD).	16
Figure S19. ¹ H- ¹ H COSY spectrum of 1-O-(3-methox-tetradecanoyl)- <i>sn</i> -glycero-3-phosphocholine (4) (600 MHz, in CD ₃ OD).	18
Figure S20. HSQC spectrum of 1-O-(3-methox-tetradecanoyl)- <i>sn</i> -glycero-3-phosphocholine (4) (600 MHz, in CD ₃ OD).	18
Figure S21. HRMS ESI-TOF of 1-O-octadecyl- <i>sn</i> -glycero-3-phosphocholine (5).	19
Figure S22. ¹ H NMR spectrum of compound 1-O-octadecyl- <i>sn</i> -glycero-3-phosphocholine (5) (600 MHz, in CD ₃ OD).	20
Figure S23. HRMS-ESI ⁺ spectrum of compound 1-palmitoyl- <i>sn</i> -glycero-3-phosphocholine (6).	21
Figure S24. ¹ H NMR spectrum of compound 1-palmitoyl- <i>sn</i> -glycero-3-phosphocholine (6) (600 MHz, in CD ₃ OD).	21
Figure S25. HRMS-ESI ⁺ spectrum of compound 1-O-hexadecylglycerol (7).	22
Figure S26. ¹ H NMR spectrum of compound 1-O-hexadecylglycerol (7) (600 MHz, in CD ₃ OD).	22
Figure S27. HRMS-ESI ⁺ spectrum of compound 1-O-octadecylglycerol (8).	24
Figure S28. ¹ H NMR spectrum of compound 1-O-octadecylglycerol (8) (600 MHz, in CD ₃ OD).	25
Figure S29. HRMS-ESI ⁺ spectrum of compound 3-nonadecyloxy-1,2-propanediol (9).	26
Figure S30. ¹ H NMR spectrum of compound 3-nonadecyloxy-1,2-propanediol (9) (600 MHz, in CD ₃ OD).	27
Figure S31. HRMS-ESI ⁺ spectrum of compound 3-icosoxypropane-1,2-diol (10).	28
Figure S32. ¹ H NMR spectrum of compound 3-icosoxypropane-1,2-diol (10) (600 MHz, in CD ₃ OD).	29
Figure S33. HRMS-ESI ⁺ spectrum of compound 5 α ,8 α -epidioxy-24(<i>R/S</i>)-hydroperoxystigmasta-6,28-dien-3 β -ol (11).	30
Figure S34. ¹ H NMR spectrum of compound 5 α ,8 α -epidioxy-24(<i>R/S</i>)-hydroperoxystigmasta-6,28-dien-3 β -ol (11) (600 MHz, in CD ₃ OD).	31
Figure S35. HRMS-ESI ⁺ spectrum of compound 5 α ,8 α -epidioxy-24(<i>R/S</i>)-stigmasta-6,22 <i>E</i> -dien-3 β -ol (12).	32
Figure S36. ¹ H NMR spectrum of compound 5 α ,8 α -epidioxy-24(<i>R/S</i>)-stigmasta-6,22 <i>E</i> -dien-3 β -ol (12) (600 MHz, in CD ₃ OD).	33
Figure S37. ¹ H NMR spectrum of compound cyclo(Val-Leu) (13) (600 MHz, in CD ₃ OD).	34
Figure S38. ¹ H NMR spectrum of compound cyclo(Val-Phe) (14) (600 MHz, in CD ₃ OD).	35
Figure S39. Dereplication of <i>Scopalina hapalia</i> fractions using LC-MS/MS molecular networking.	36
Figure S40. Dereplication of <i>Scopalina hapalia</i> fractions using LC-MS/MS molecular networking.	37

MAY13-165-RP-F07-C56_Mex3 18 (0.440) AM2 (Ar,18000.0,0.00,0.00); Cm (1:20)
[M+Na]⁺

1: TOF MS ES⁺
3.26e6

349.1985

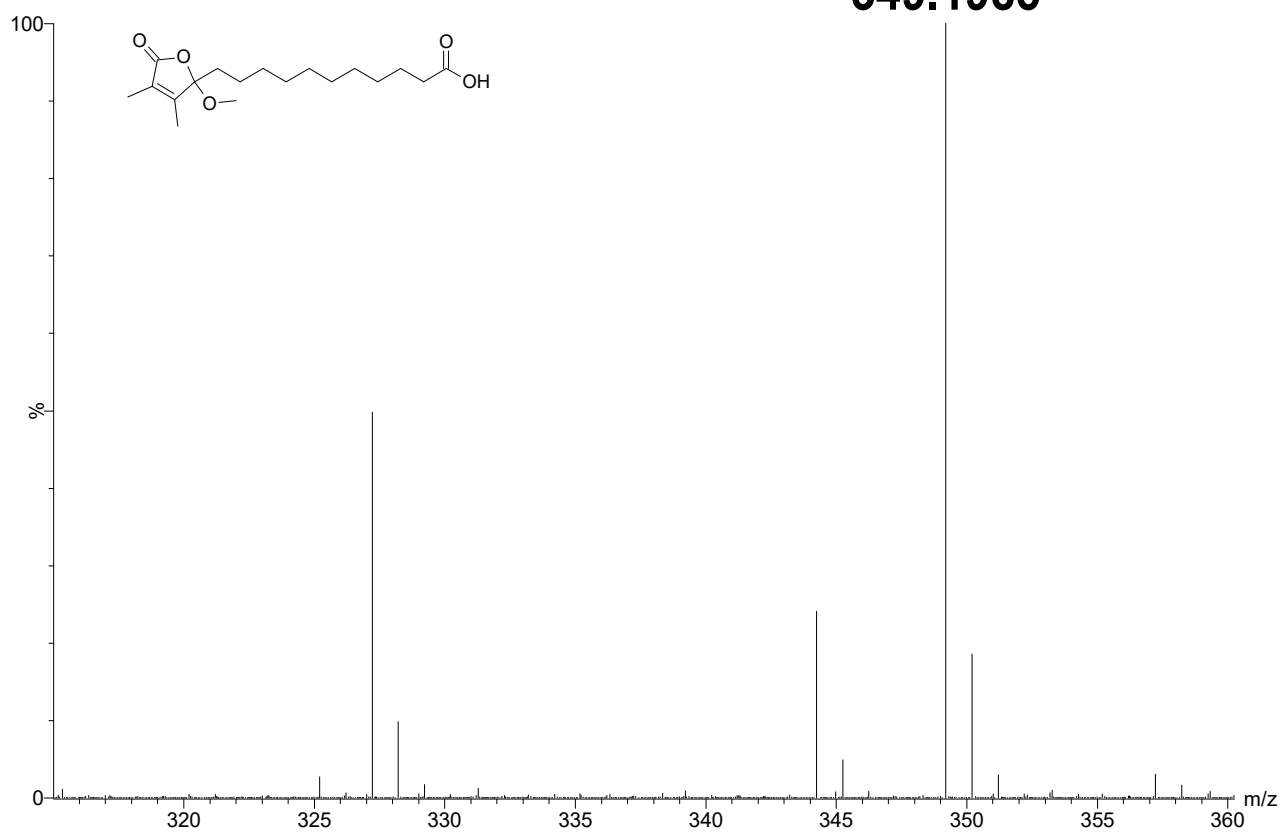
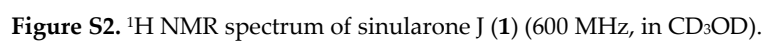


Figure S1. HRMS-ESI⁺ spectrum of sinularone J (1).



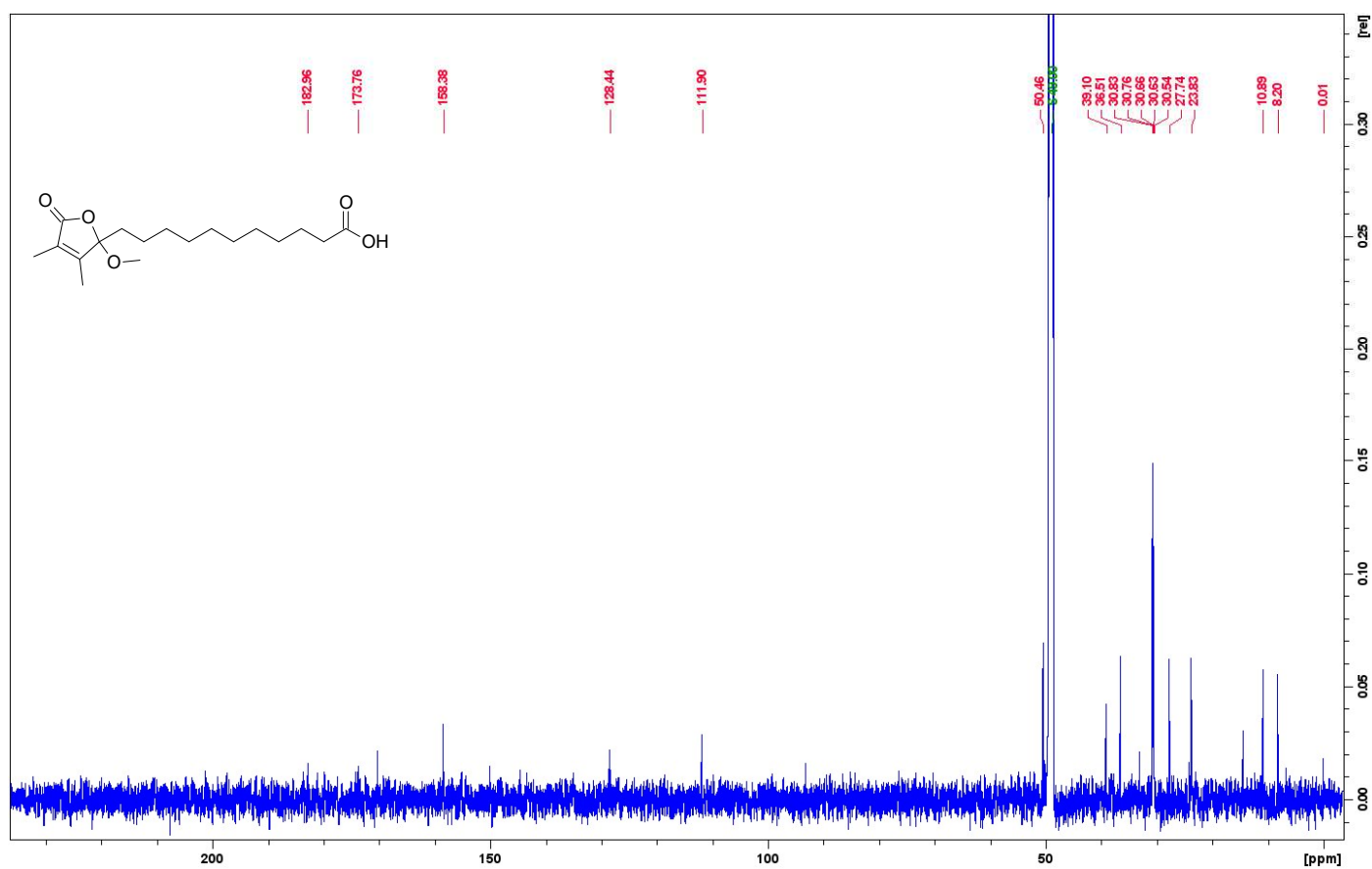


Figure S3. ¹³C NMR spectrum of sinularone J (1) (150 MHz, in CD₃OD).

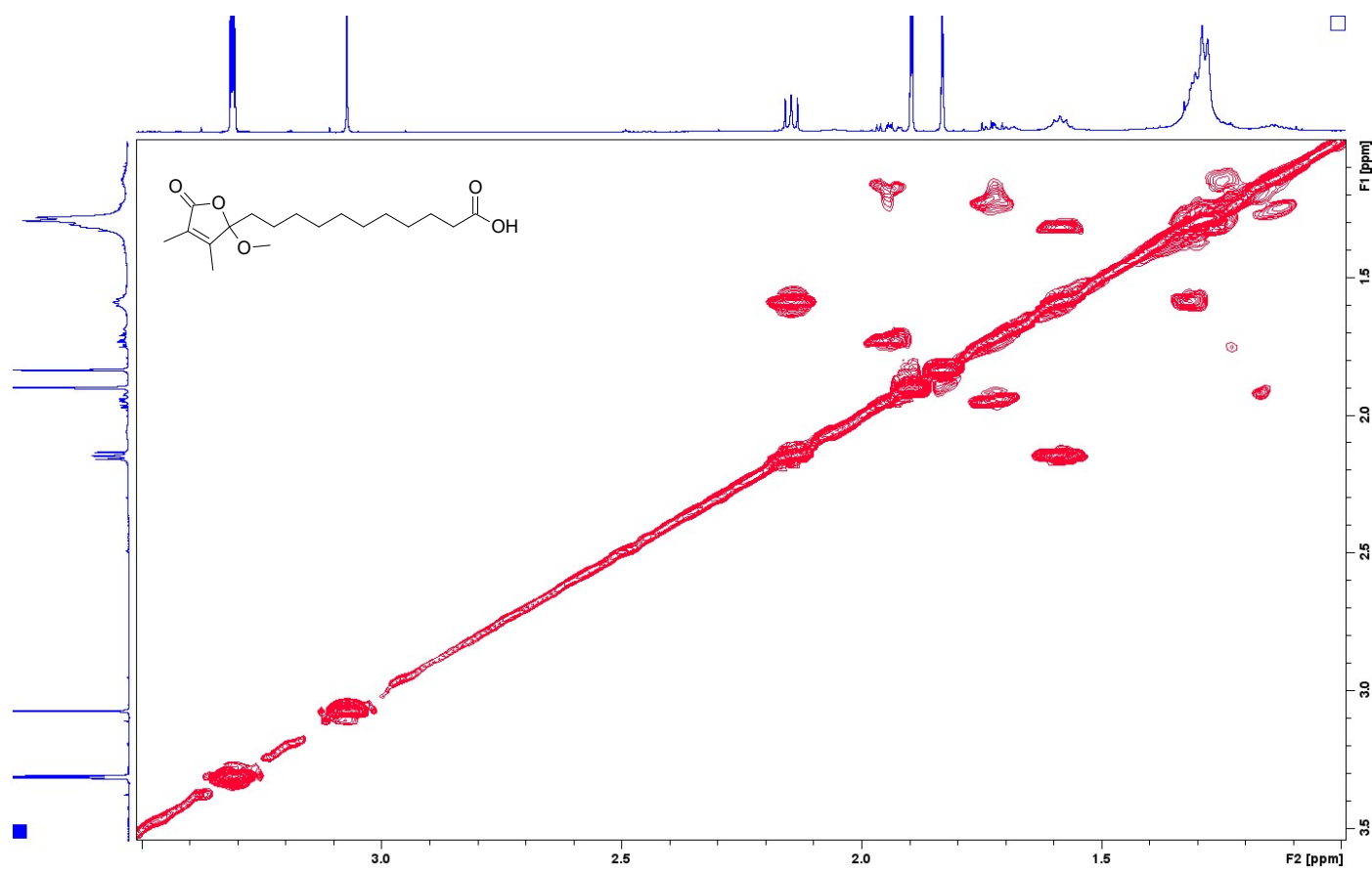


Figure S4. ^1H - ^1H COSY spectrum of sinularone J (1) (600 MHz, in CD_3OD).

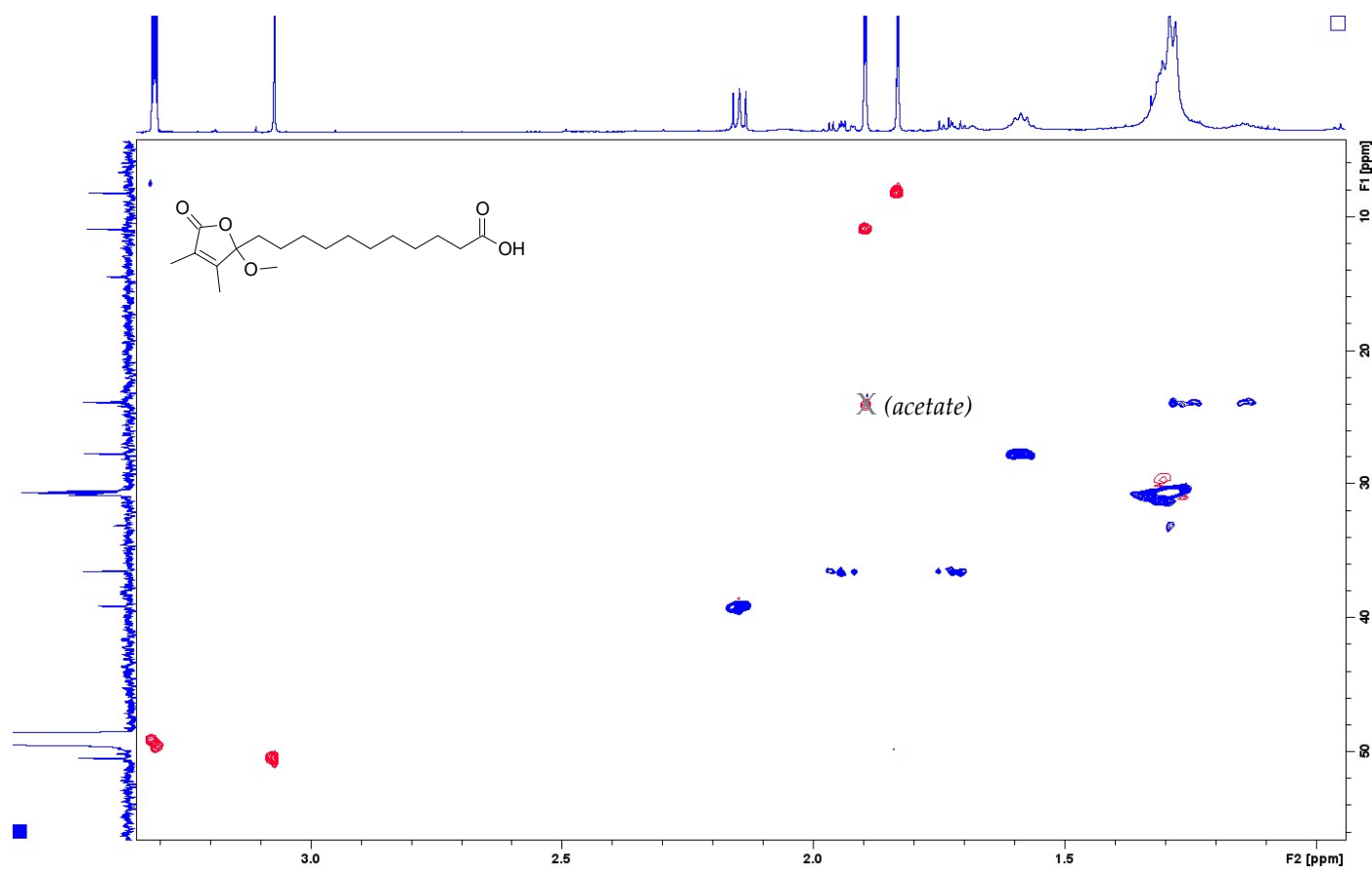


Figure S5. HSQC spectrum of sinularone J (1) (600 MHz, in CD_3OD).

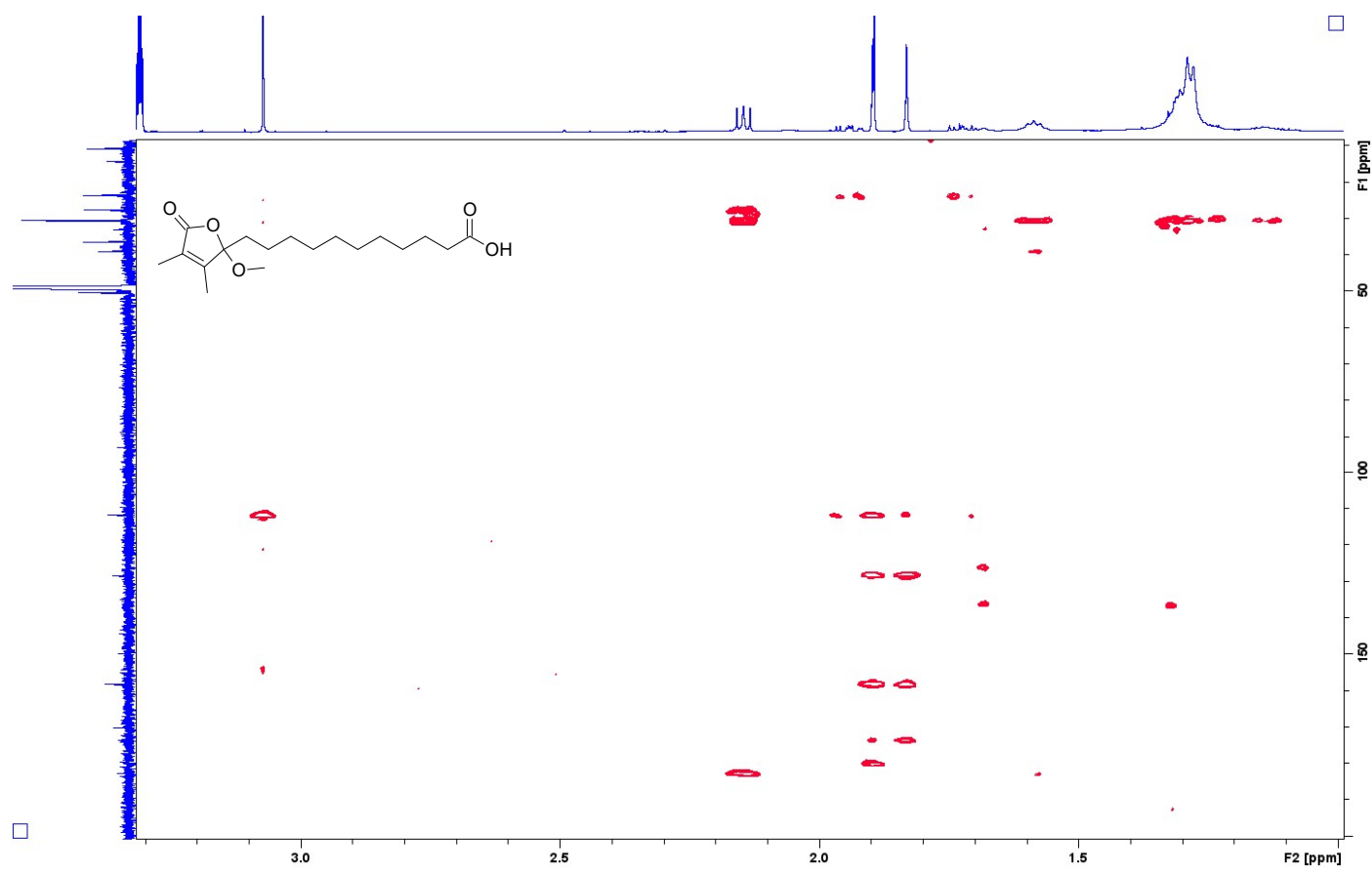


Figure S6. HMBC spectrum of sinularone J (**1**) (600 MHz, in CD₃OD).

1: TOF MS ES+
3.20e6

433.2921

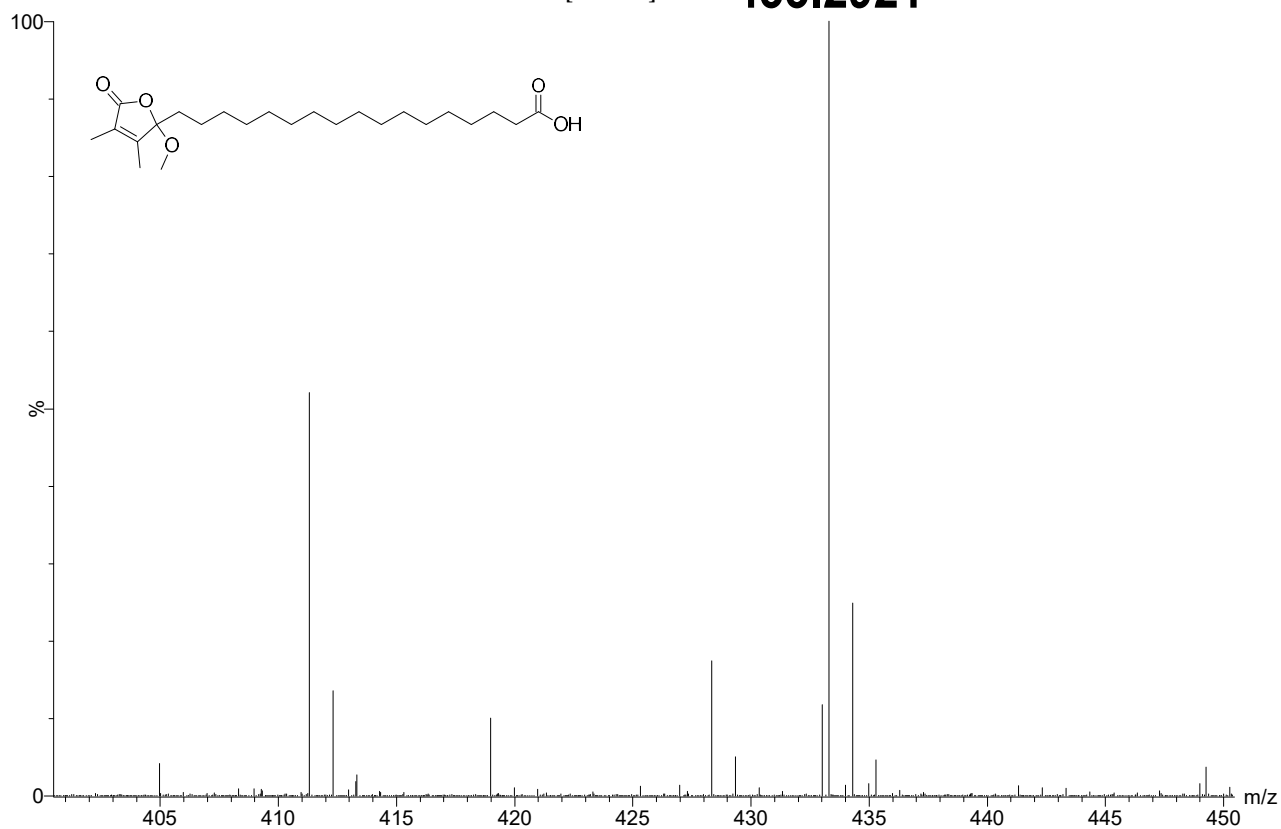


Figure S7. HRMS-ESI⁺ spectrum of sinularone K (2).

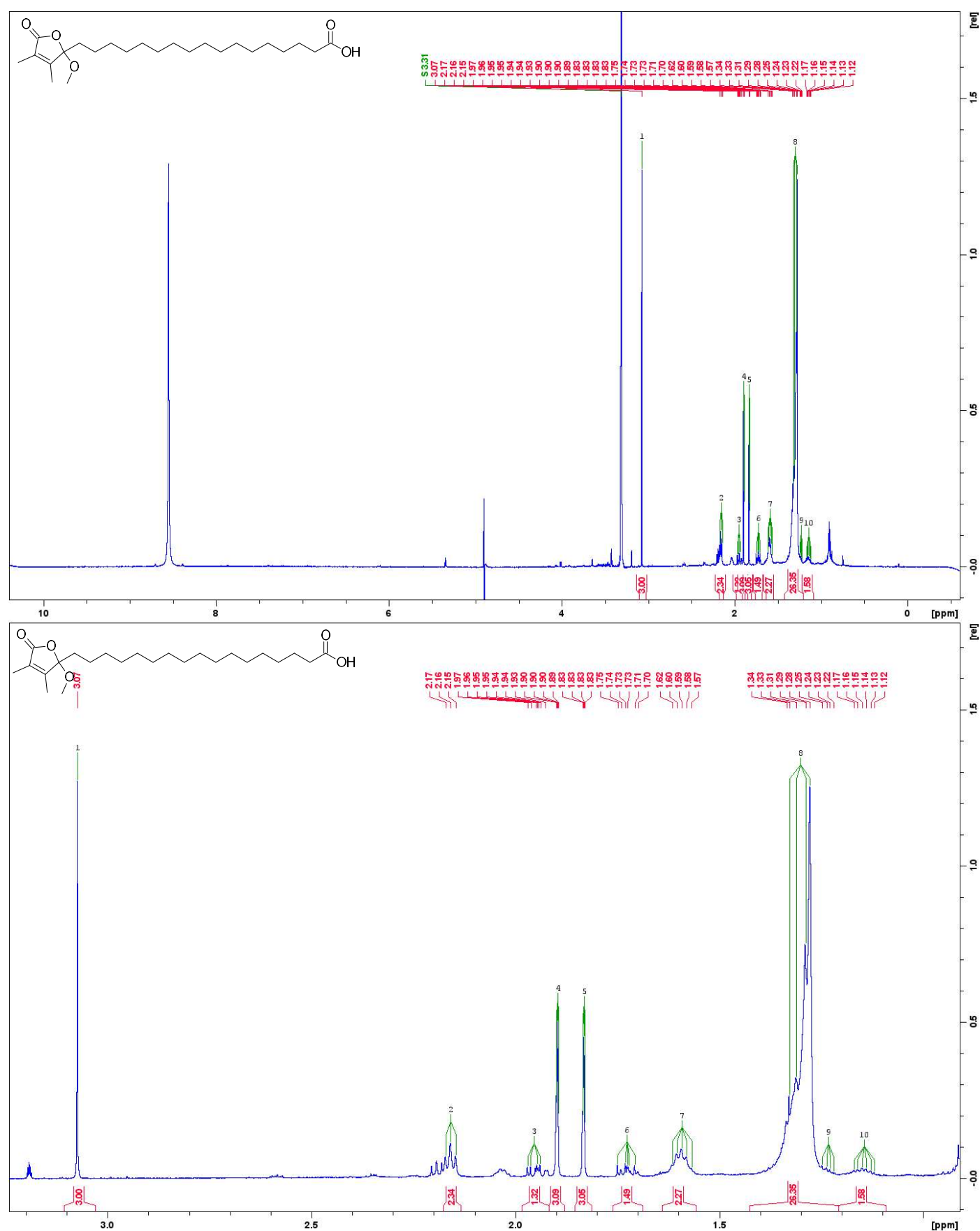


Figure S8. ^1H NMR spectrum of sinularone K (2) (600 MHz, in CD_3OD).

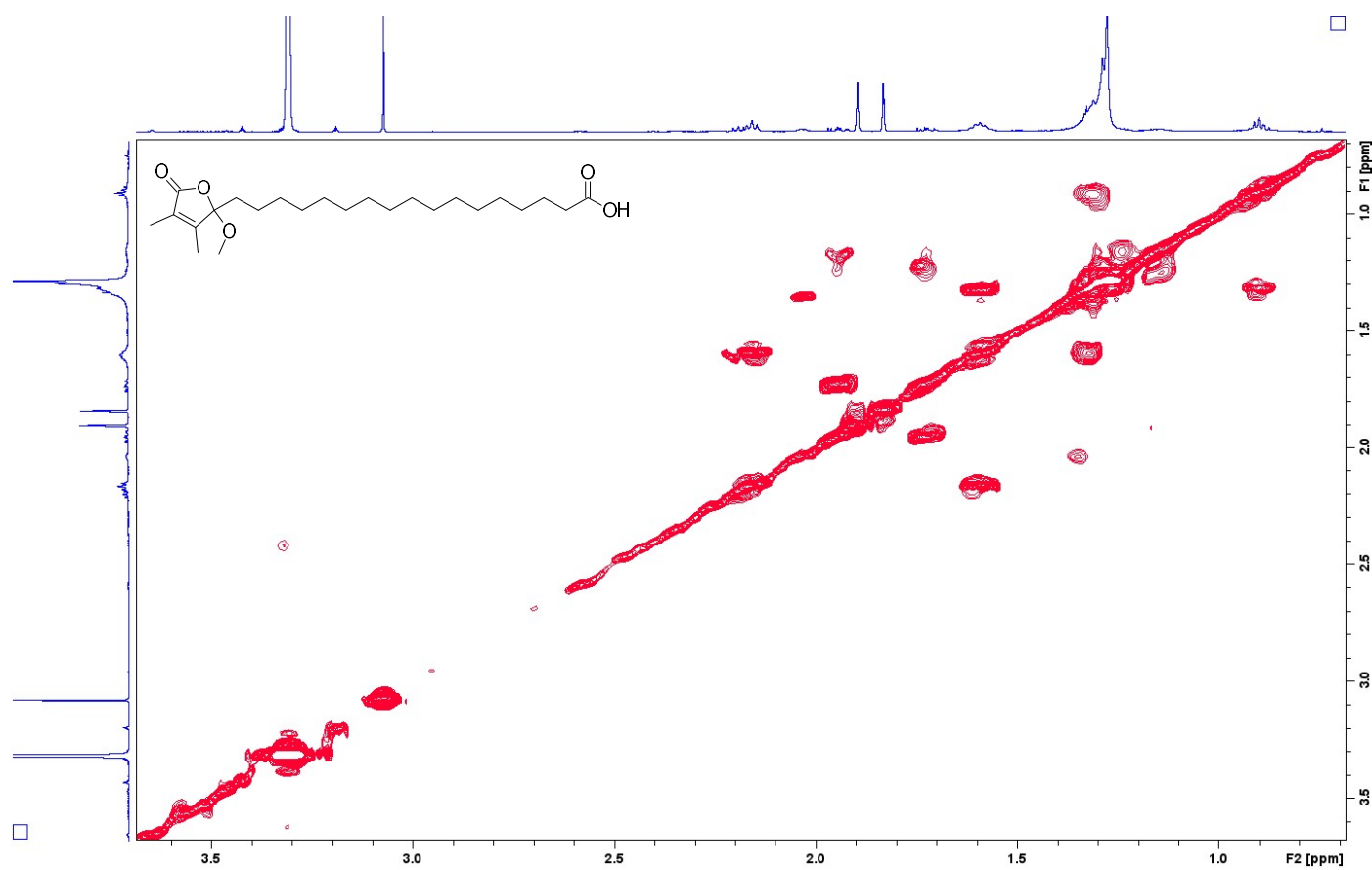


Figure S9. ^1H - ^1H COSY spectrum of sinularone K (**2**) (600 MHz, in CD_3OD).

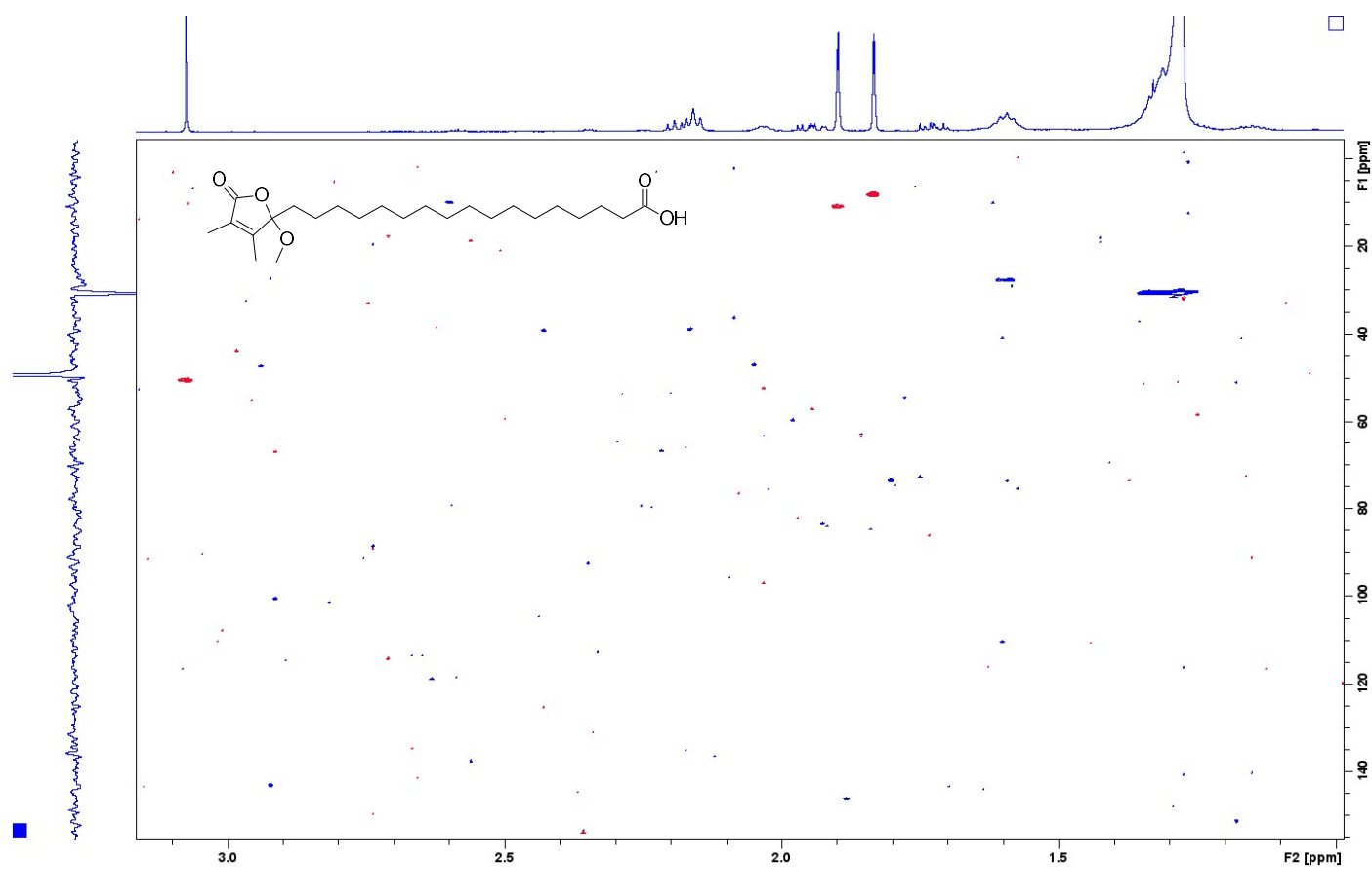


Figure S10. HSQC spectrum of sinularone K (2) (600 MHz, in CD_3OD).

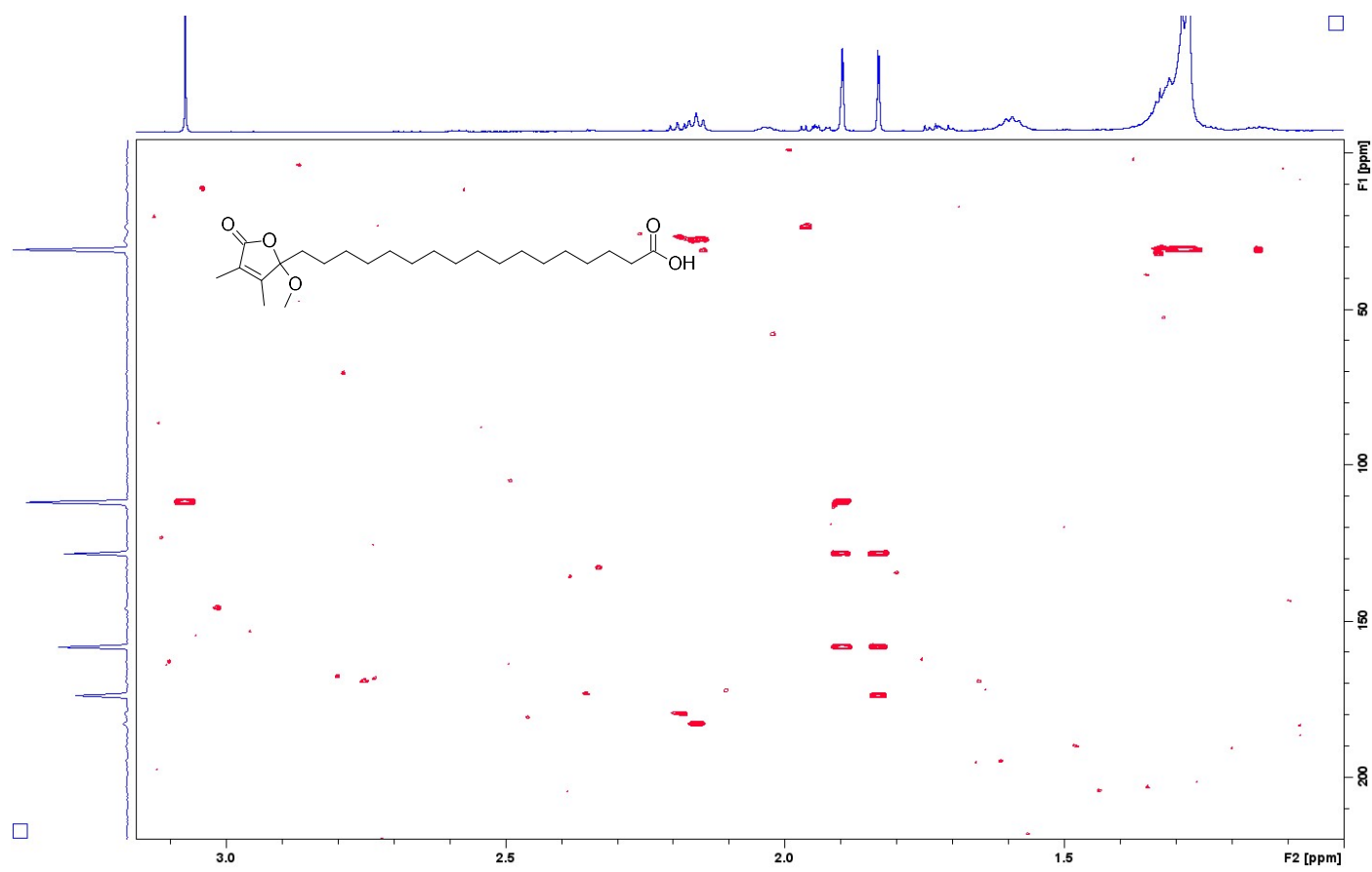
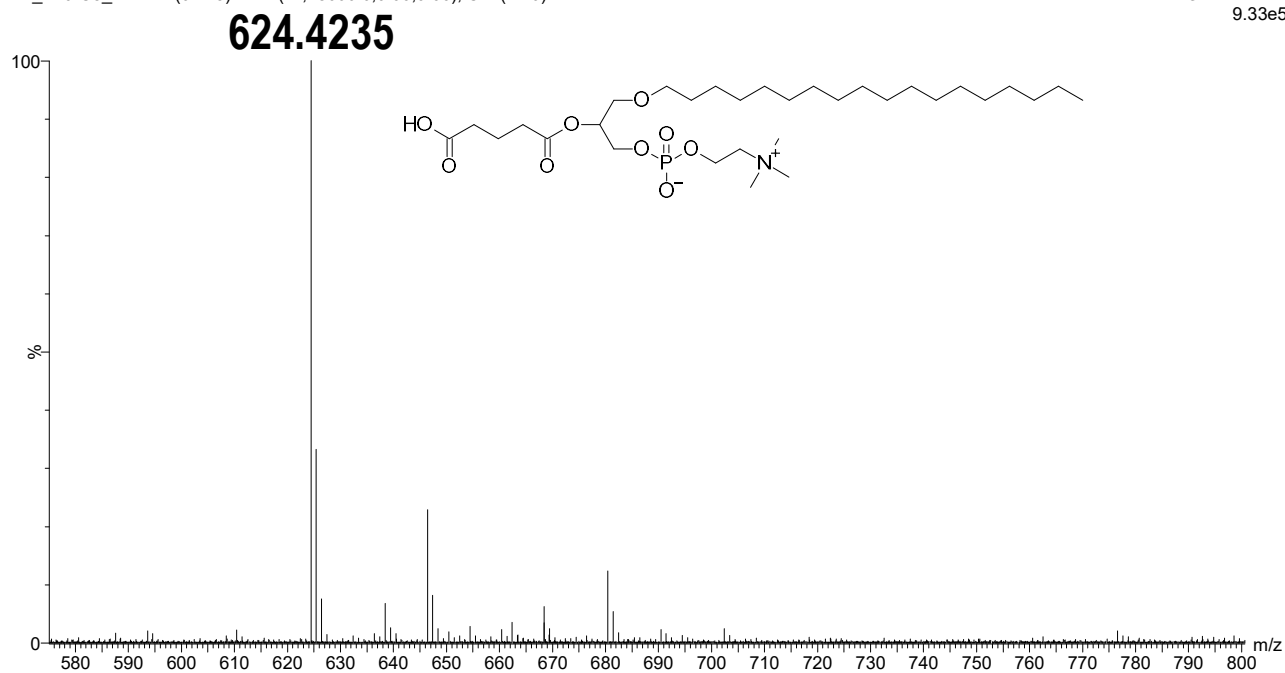


Figure S11. HMBC spectrum of sinularone K (**2**) (600 MHz, in CD₃OD).

NP_F10-C8_Mex2 4 (0.123) AM2 (Ar,18000.0,0.00,0.00); Cm (1:20)

1: TOF MS ES+
9.33e5

NP_F10-C8_DS_624_4_30EV 7 (0.137) Cm (1:22)

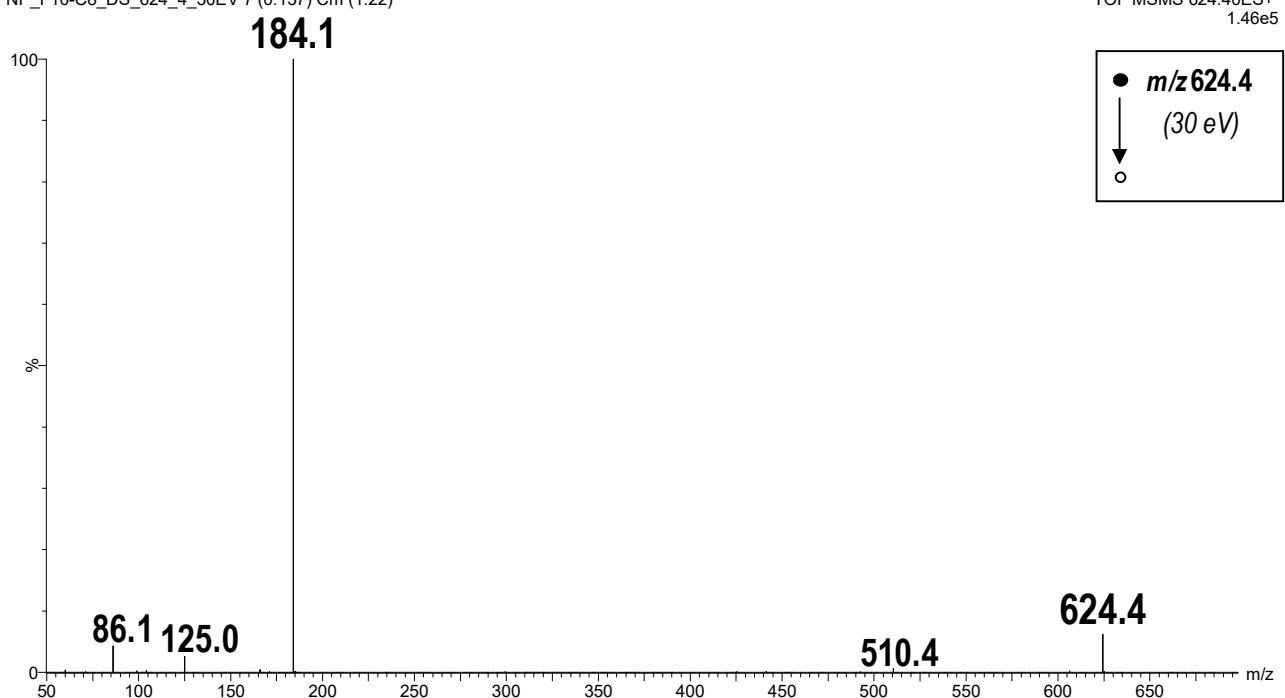
TOF MSMS 624.40ES+
1.46e5

Figure S12. HRMS-ESI-TOF spectrum of 1-*O*-octadecyl-2-pentanoyl-*sn*-glycero-3-phosphocholine (3).

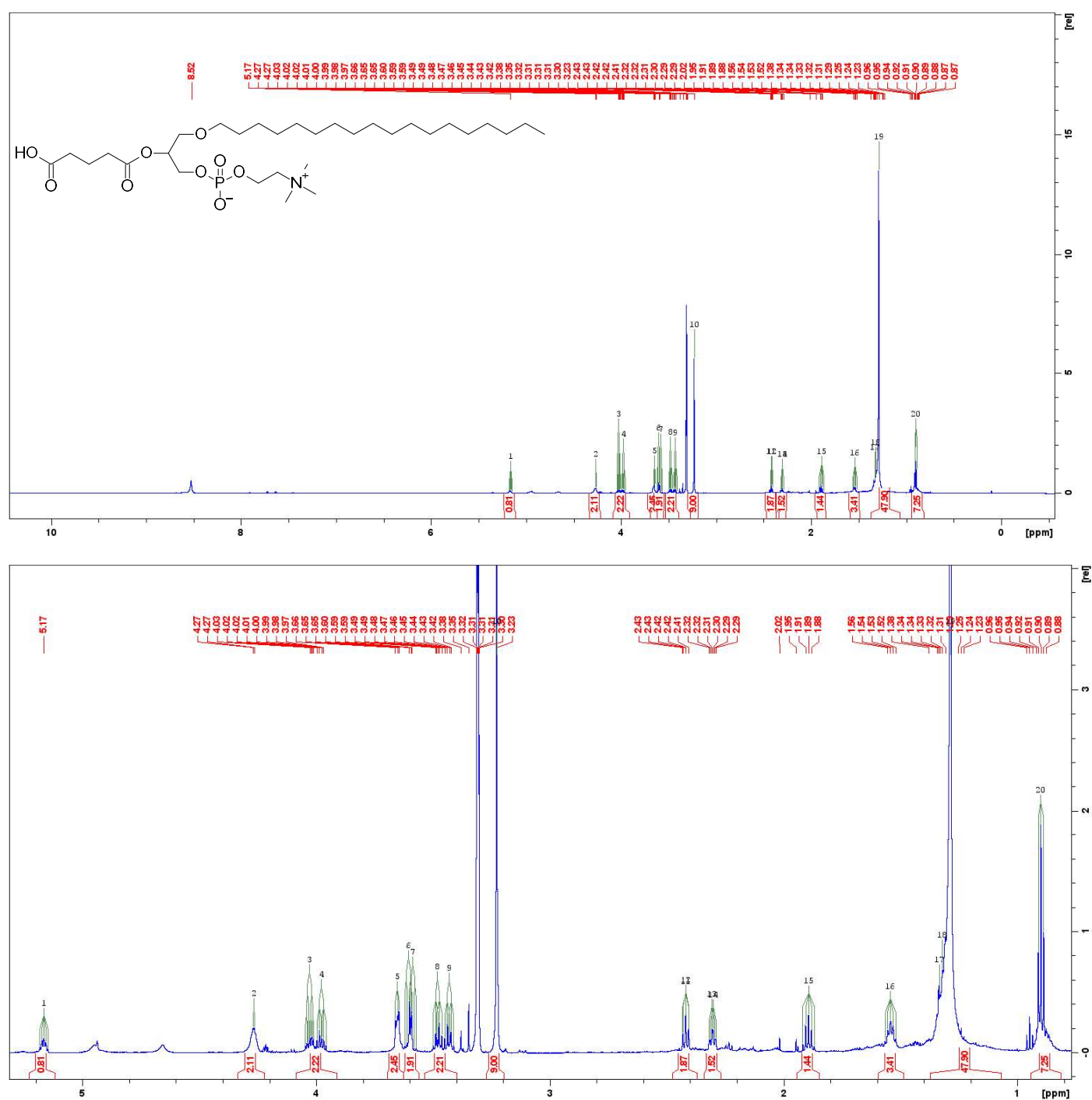


Figure S13. ^1H NMR spectrum of 1-O-octadecyl-2-pentanoyl-*sn*-glycero-3-phosphocholine (3) (600 MHz, in CD_3OD).

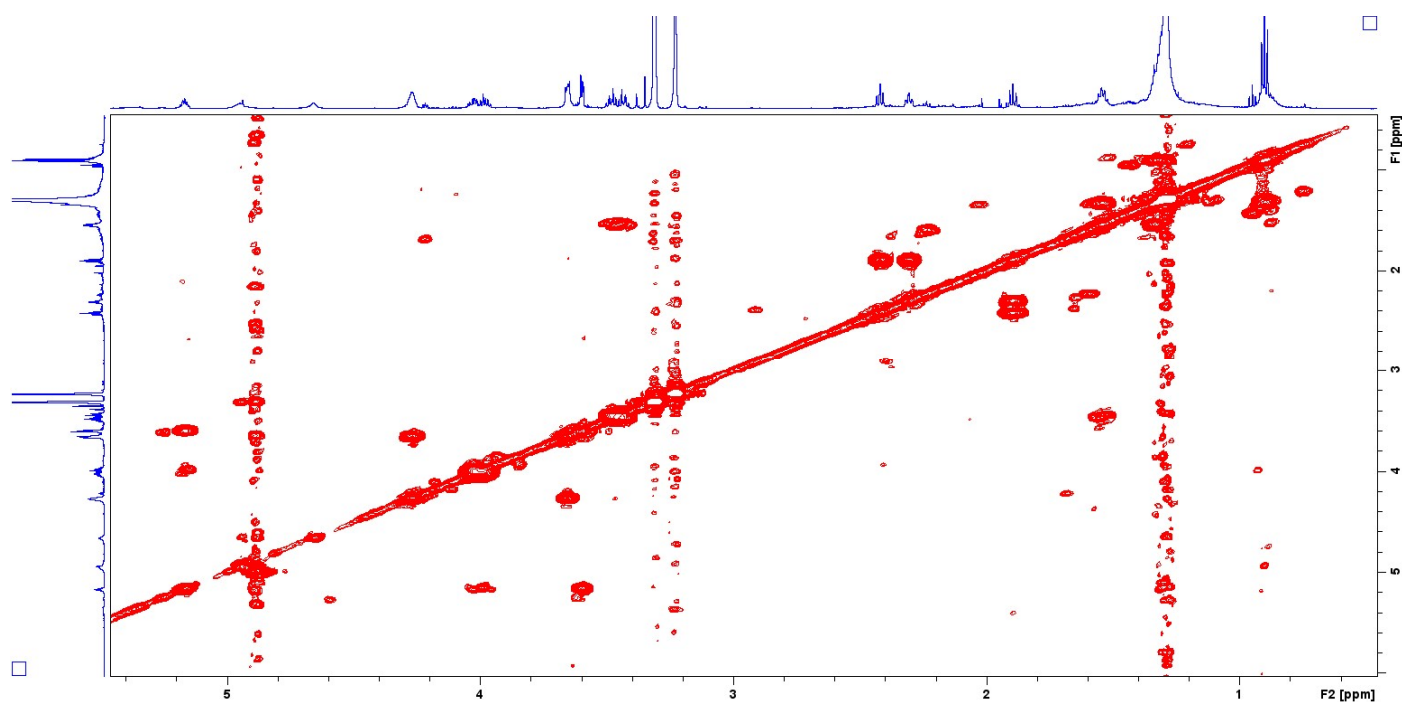


Figure S14. ^1H - ^1H COSY spectrum of 1-*O*-octadecyl-2-pentanoyl-*sn*-glycero-3-phosphocholine (**3**) (600 MHz, in CD_3OD).

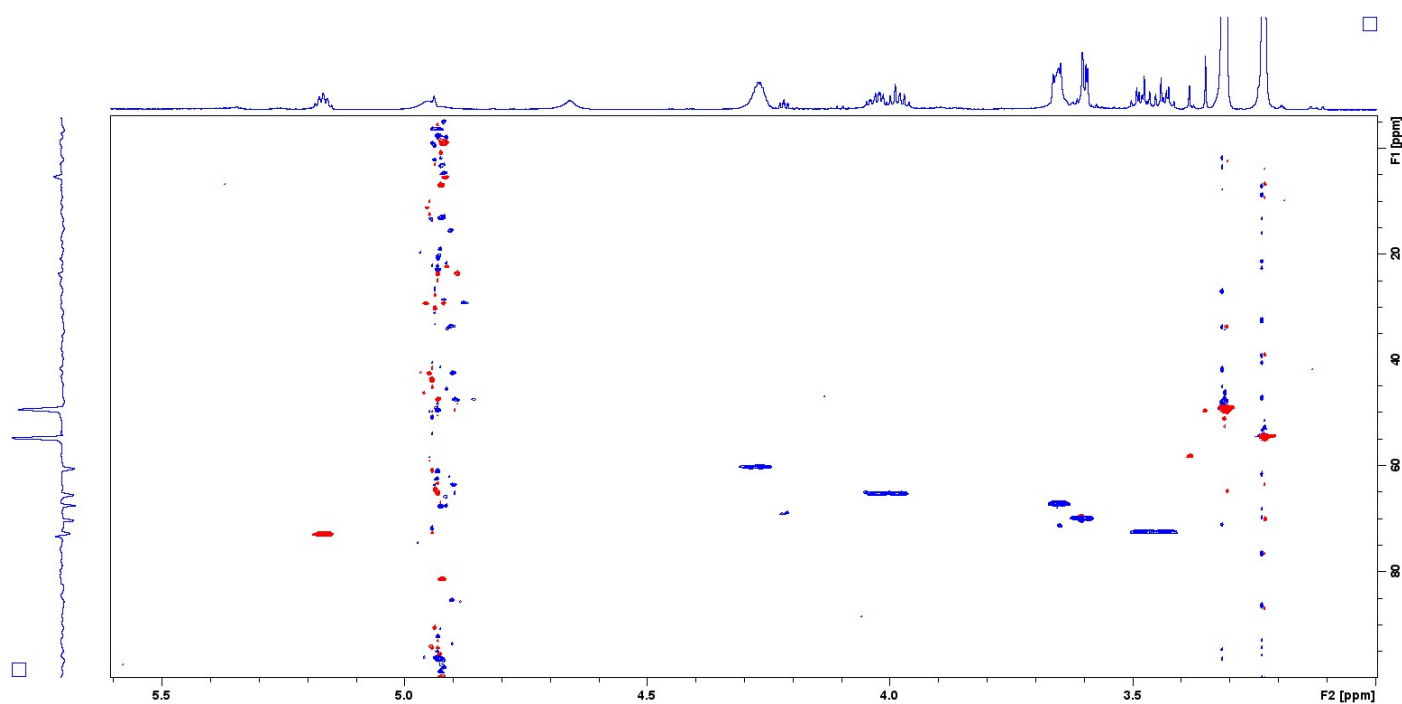


Figure S15. HSQC spectrum of 1-*O*-octadecyl-2-pentanoyl-*sn*-glycero-3-phosphocholine (**3**) (600 MHz, in CD_3OD).

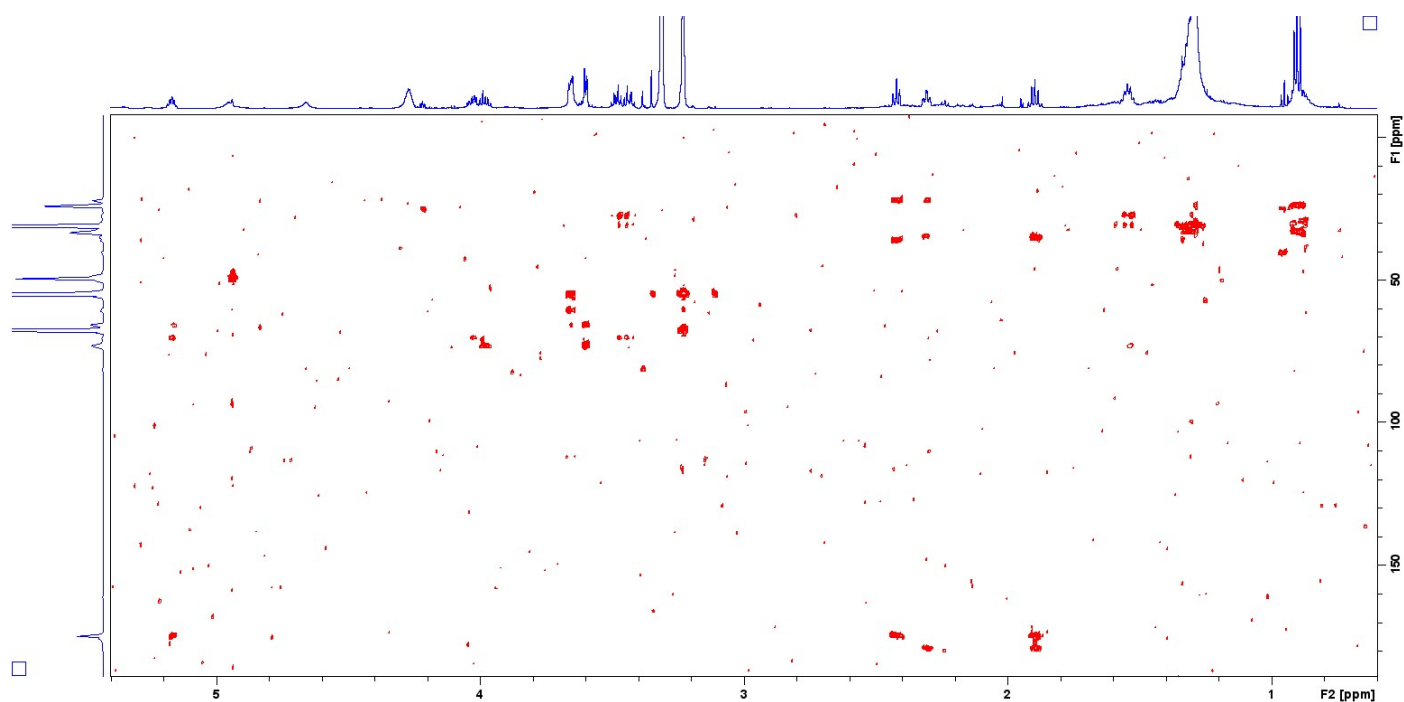


Figure S16. HMBC spectrum of 1-*O*-octadecyl-2-pentanoyl-*sn*-glycero-3-phosphocholine (**3**) (600 MHz, in CD₃OD).

NP_F10-C2_Mex3 6 (0.157) AM2 (Ar,18000.0,0.00,0.00); Cm (1:20)

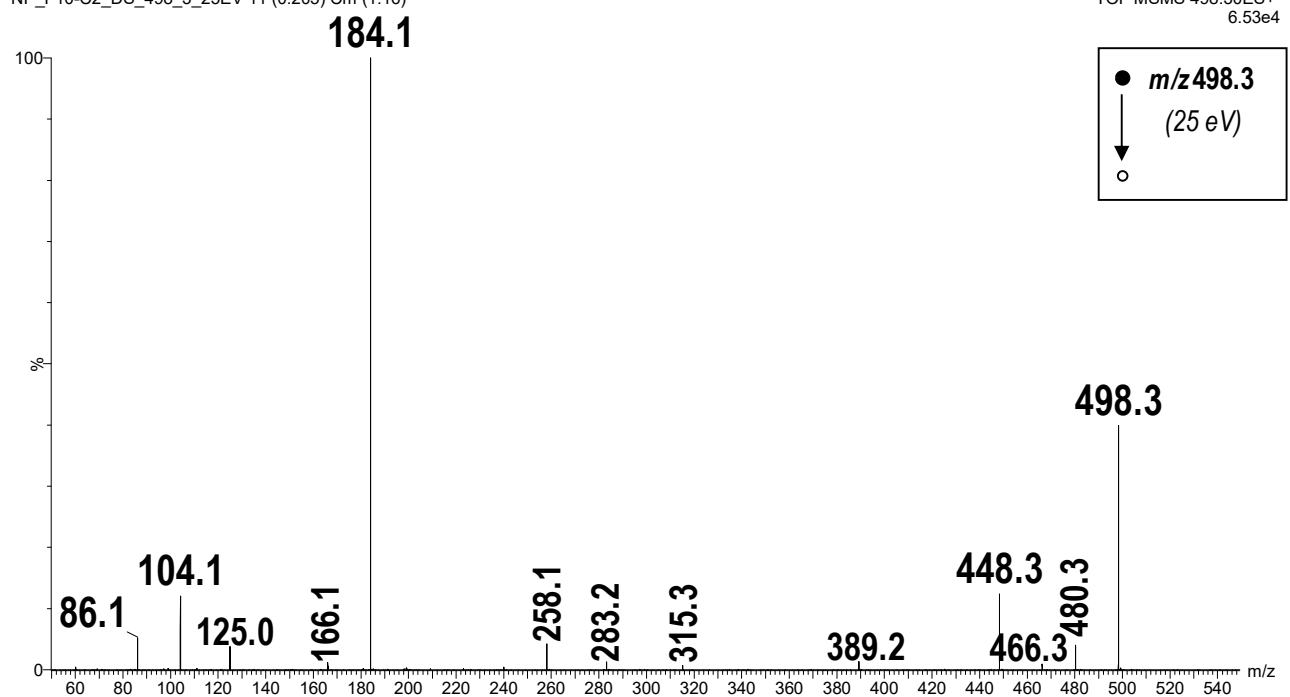
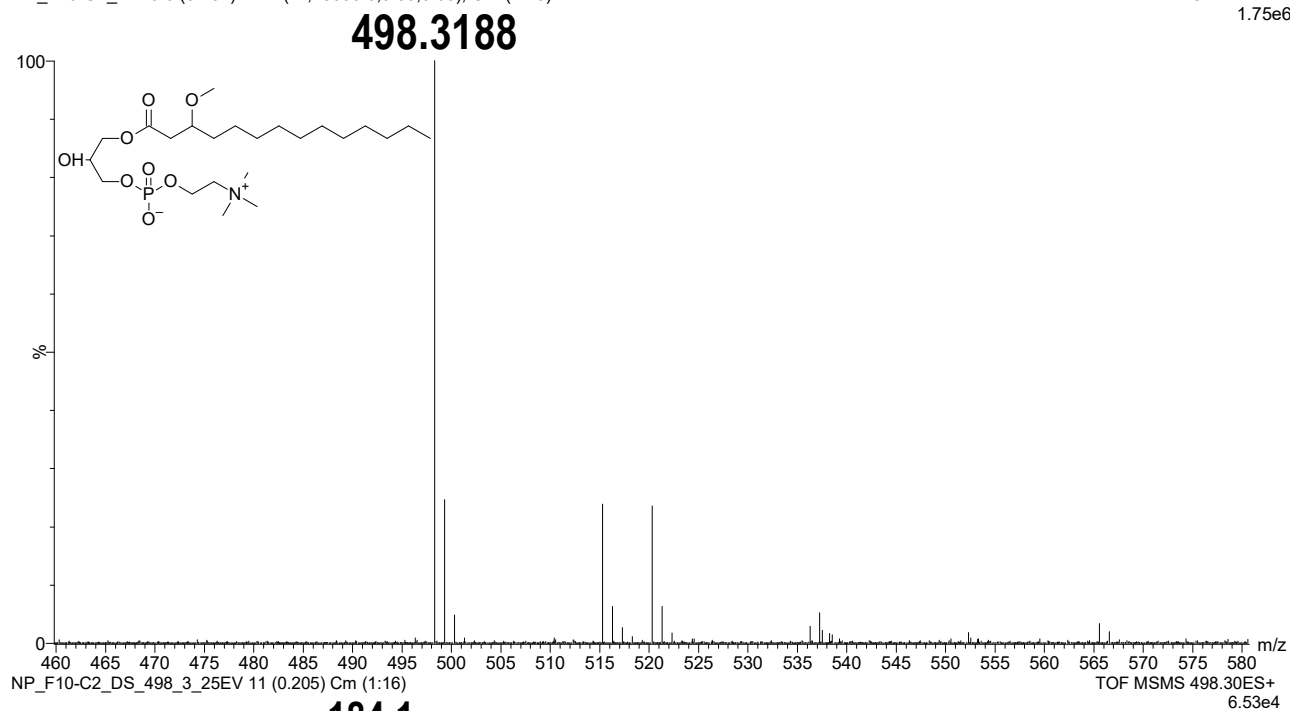
1: TOF MS ES+
1.75e6

Figure S17. HRMS ESI-TOF spectrum of 1-O-(3-methoxy-tetradecanoyl)-*sn*-glycero-3-phosphocholine (4).

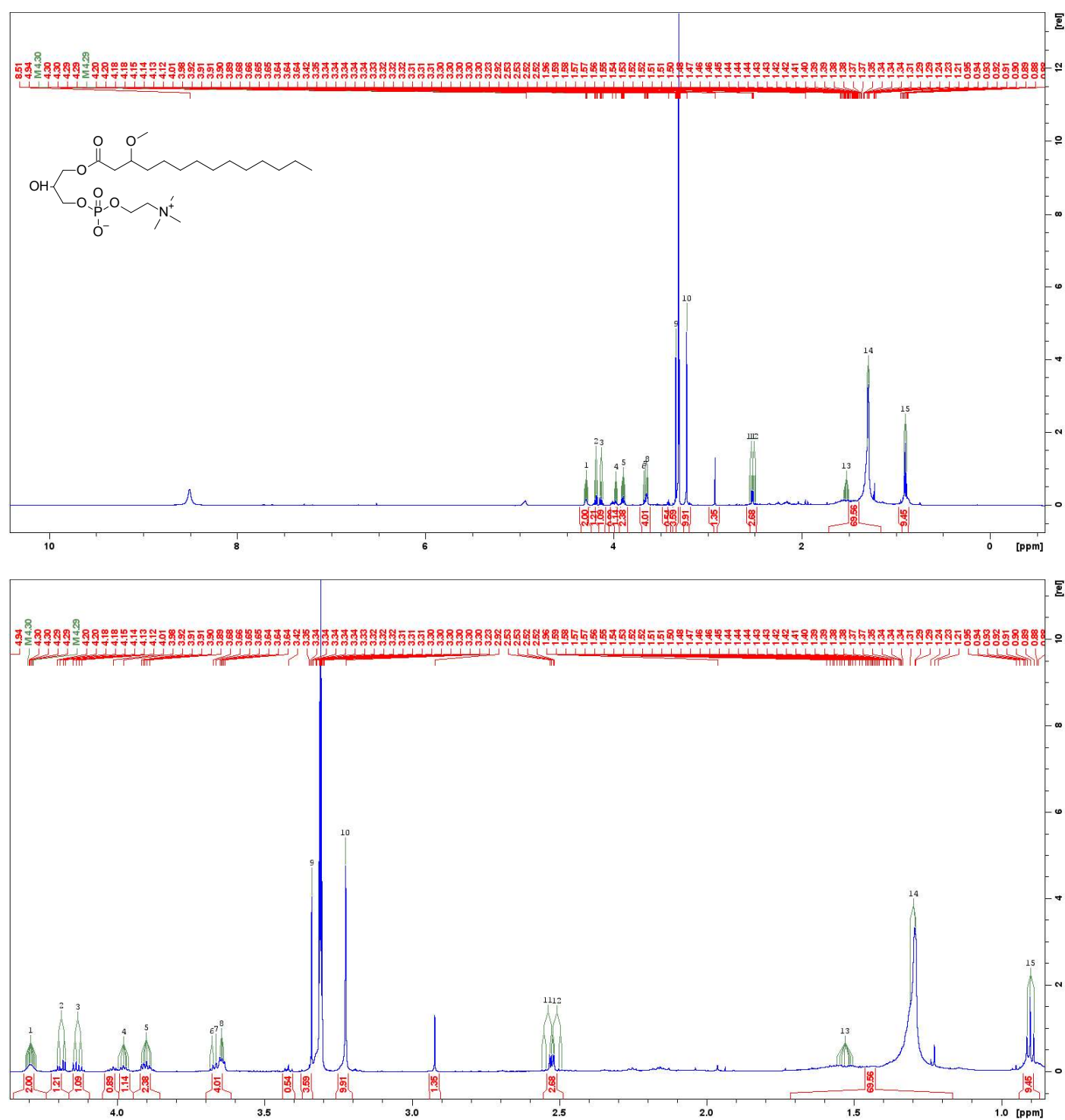


Figure S18. ^1H NMR spectrum of 1-O-(3-methoxy-tetradecanoyl)-*sn*-glycero-3-phosphocholine (4) (600 MHz, in CD_3OD).

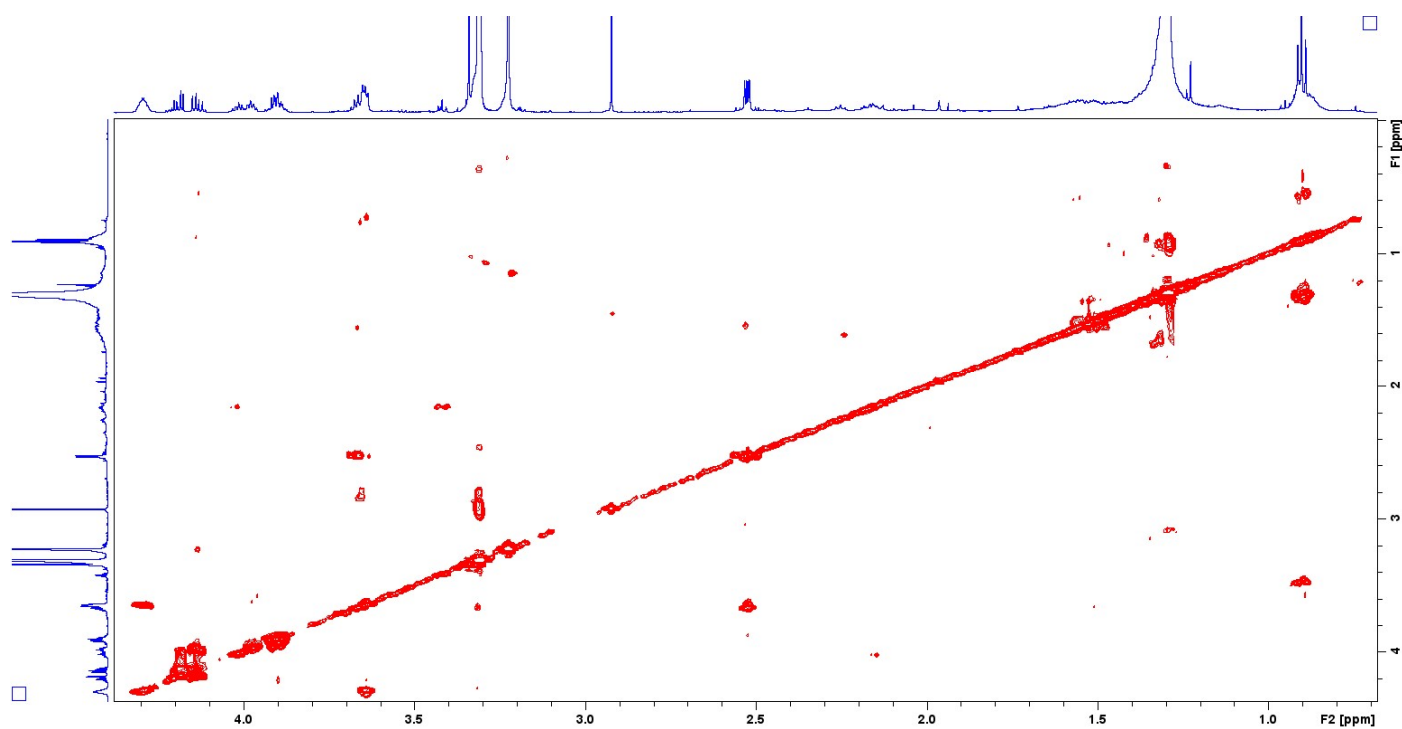


Figure S19. ^1H - ^1H COSY spectrum of 1-O-(3-methoxy-tetradecanoyl)-*sn*-glycero-3-phosphocholine (4) (600 MHz, in CD_3OD).

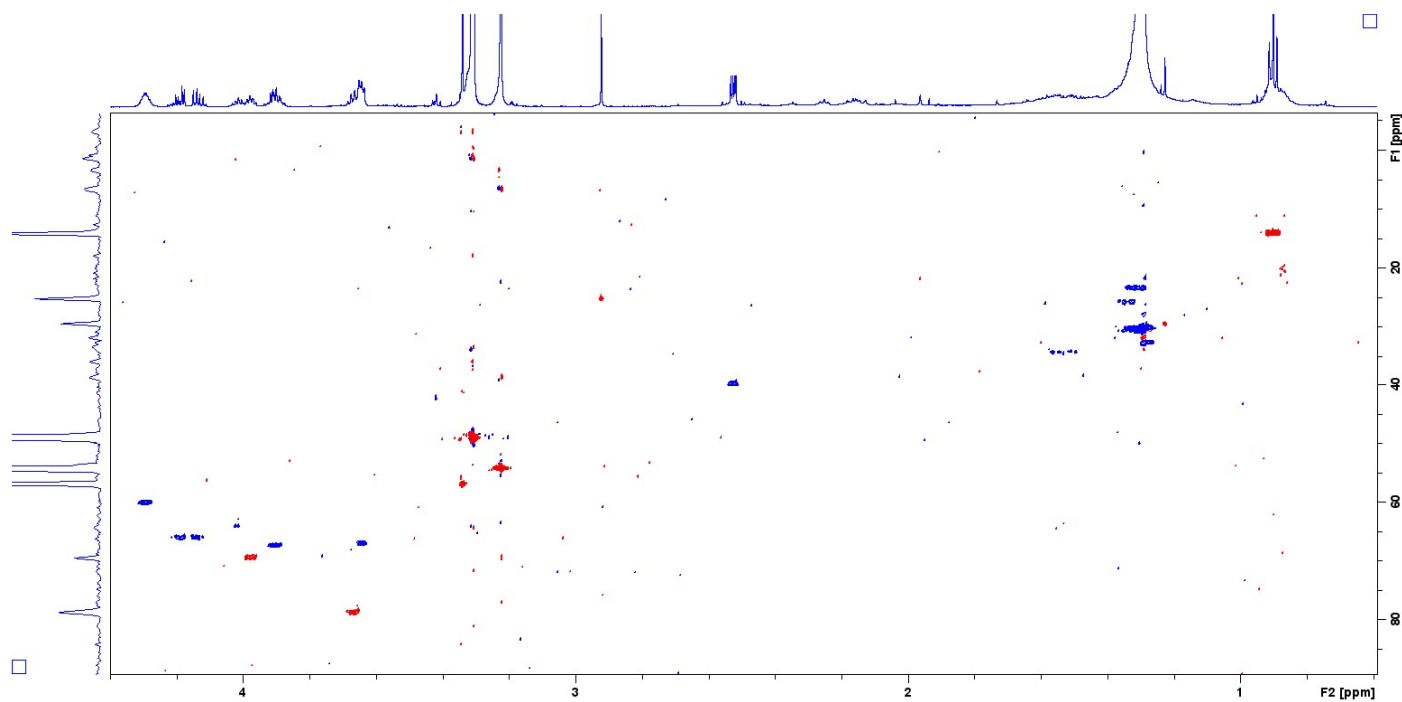
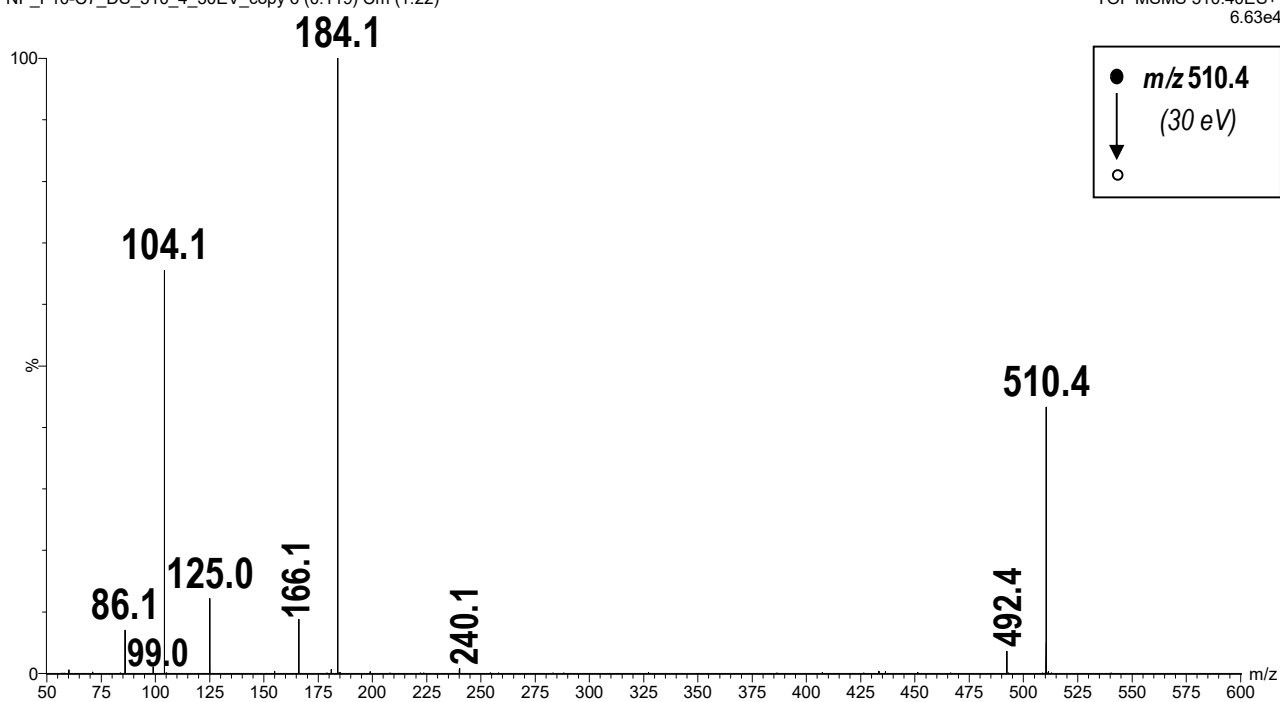
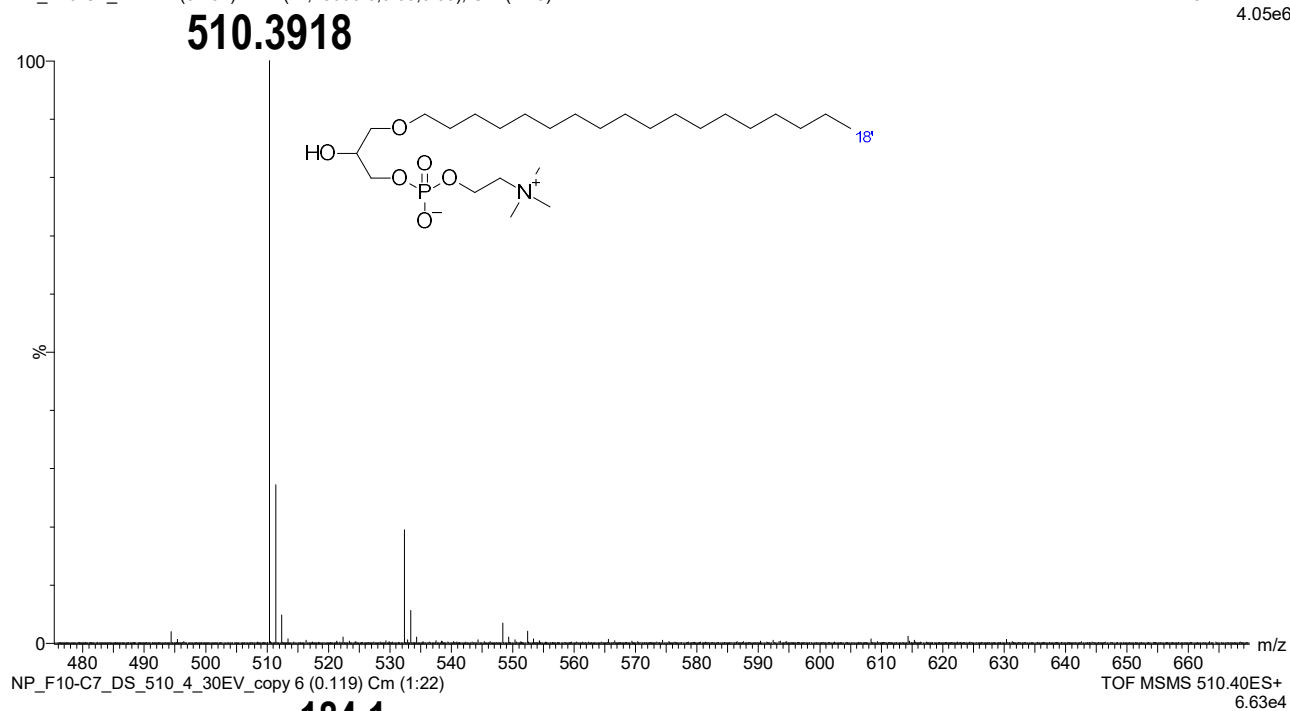
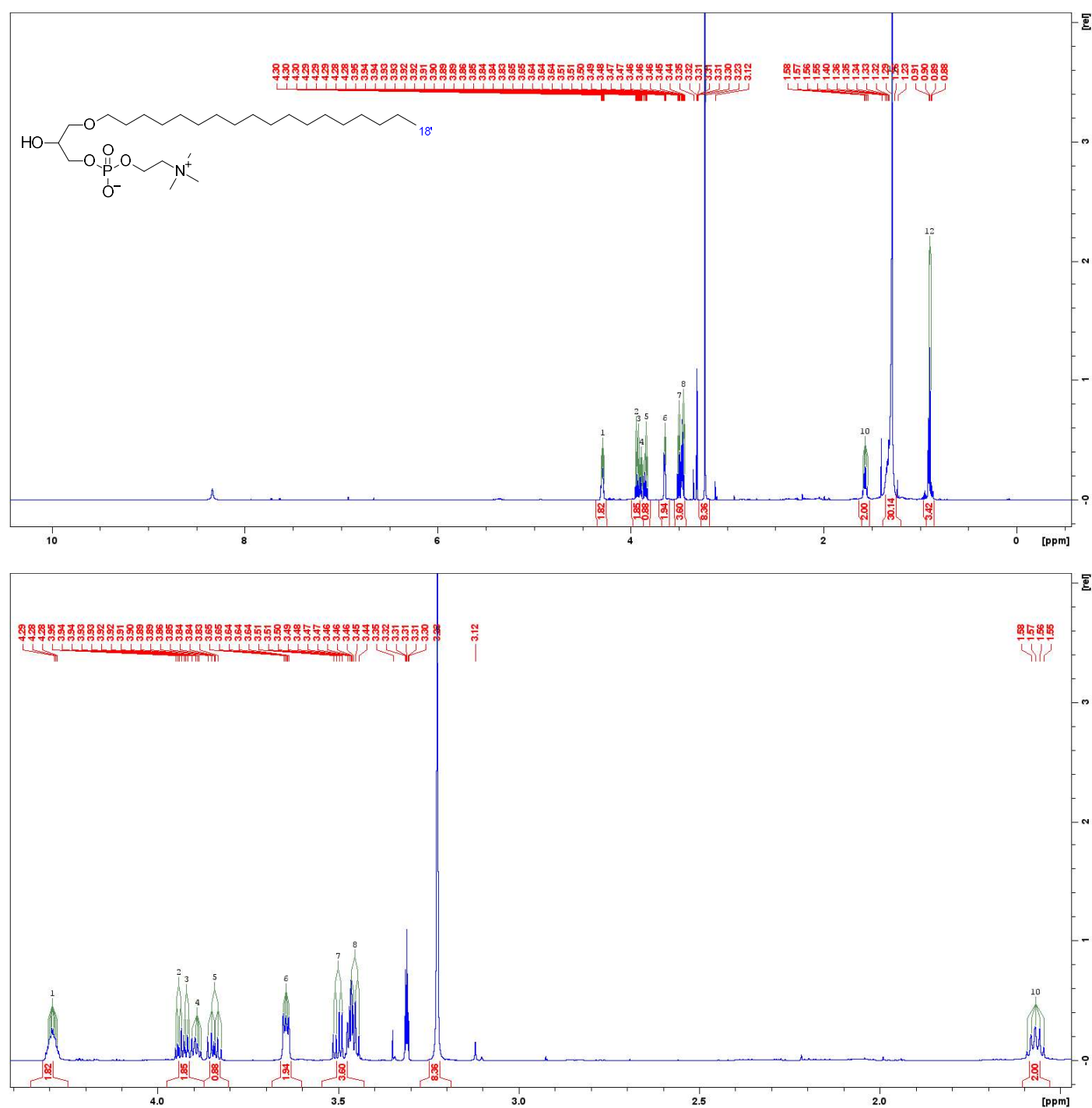


Figure S20. HSQC spectrum of 1-O-(3-methoxy-tetradecanoyl)-*sn*-glycero-3-phosphocholine (4) (600 MHz, in CD_3OD).

NP_F10-C7_Mex1 7 (0.194) AM2 (Ar,18000.0,0.00,0.00); Cm (1:20)

1: TOF MS ES+
4.05e6Figure S21. HRMS ESI-TOF of 1-O-octadecyl-*sn*-glycero-3-phosphocholine (5).



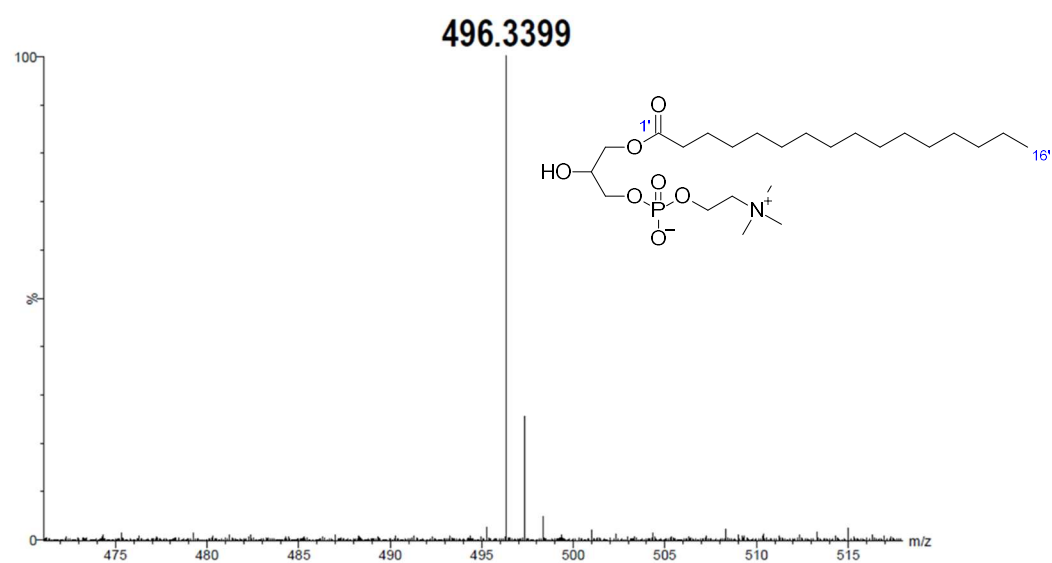


Figure S23. HRMS-ESI⁺ spectrum of compound 1-palmitoyl-*sn*-glycero-3-phosphocholine (6).

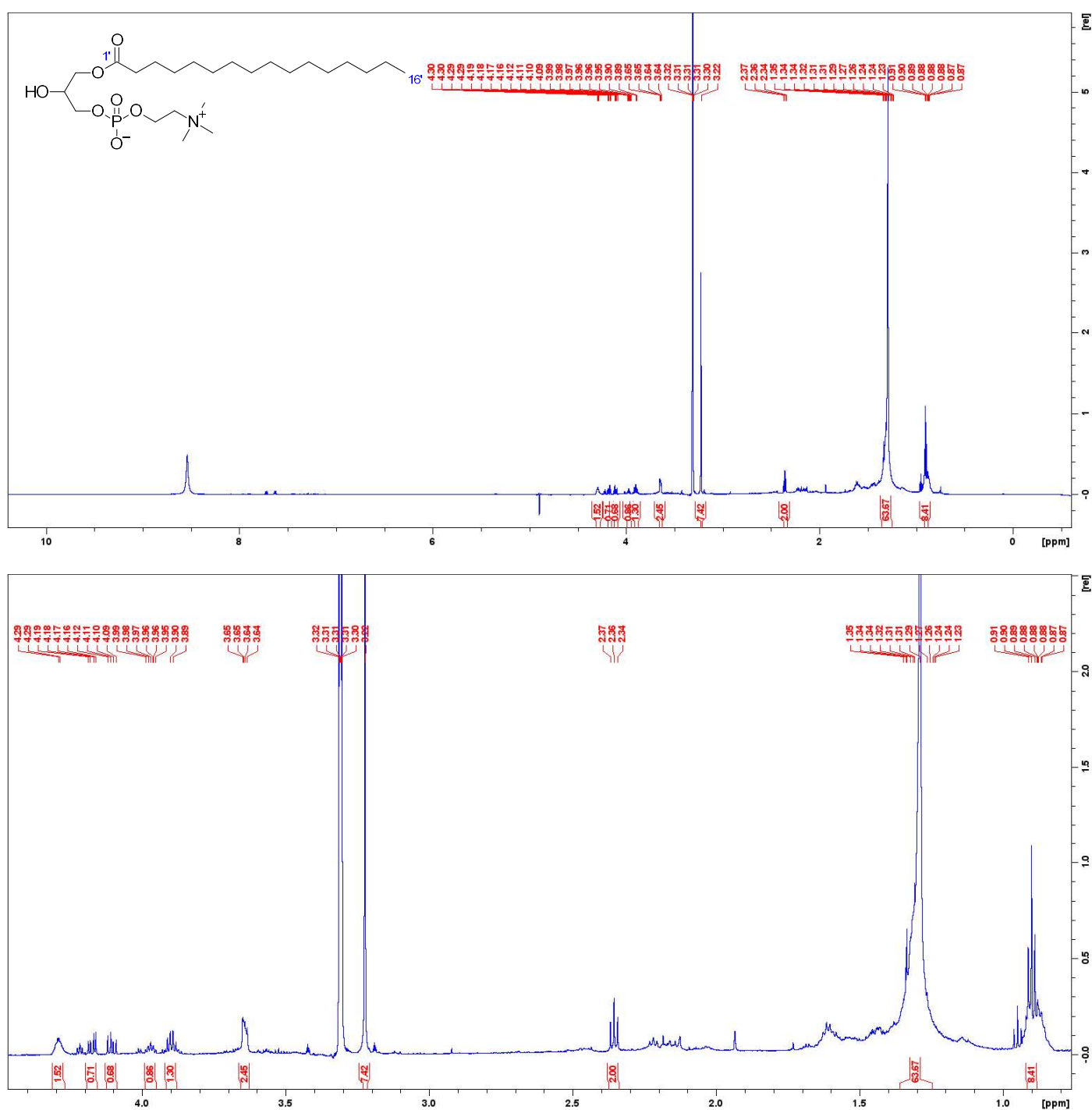
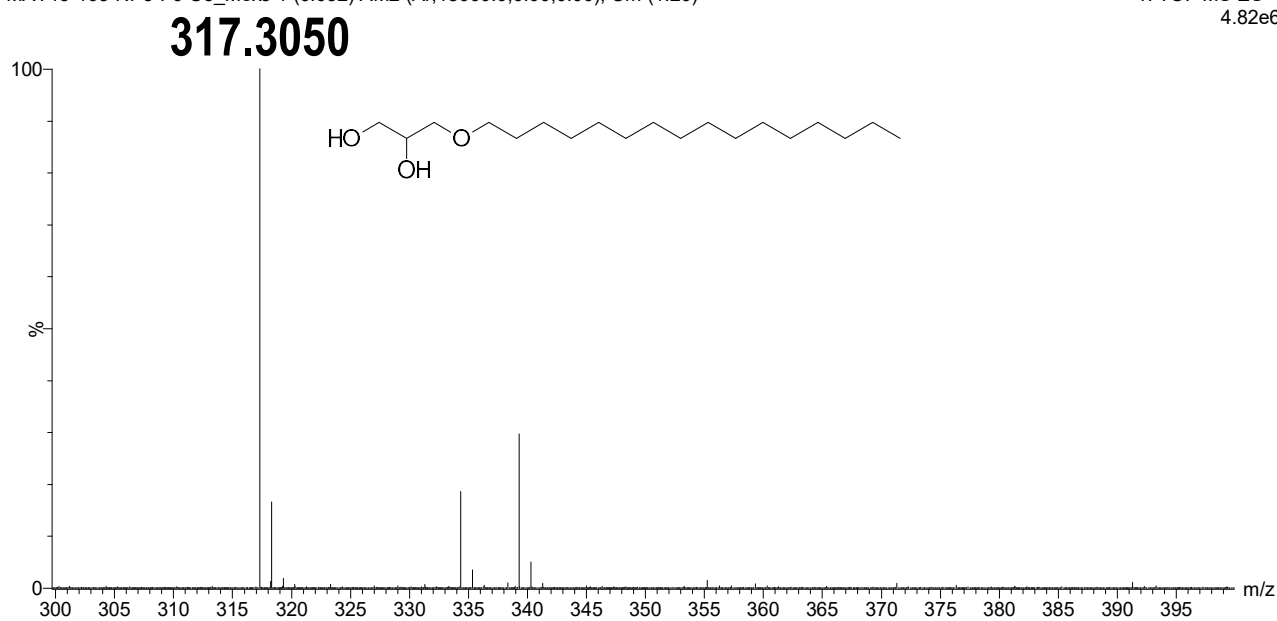


Figure S24. ^1H NMR spectrum of compound 1-palmitoyl-*sn*-glycero-3-phosphocholine (6) (600 MHz, in CD_3OD).

MAY13-165-NP3-F6-C3_Mex3 1 (0.052) AM2 (Ar,18000.0,0.00,0.00); Cm (1:20)

1: TOF MS ES+
4.82e6**Figure S25.** HRMS-ESI⁺ spectrum of compound 1-O-hexadecylglycerol (7).

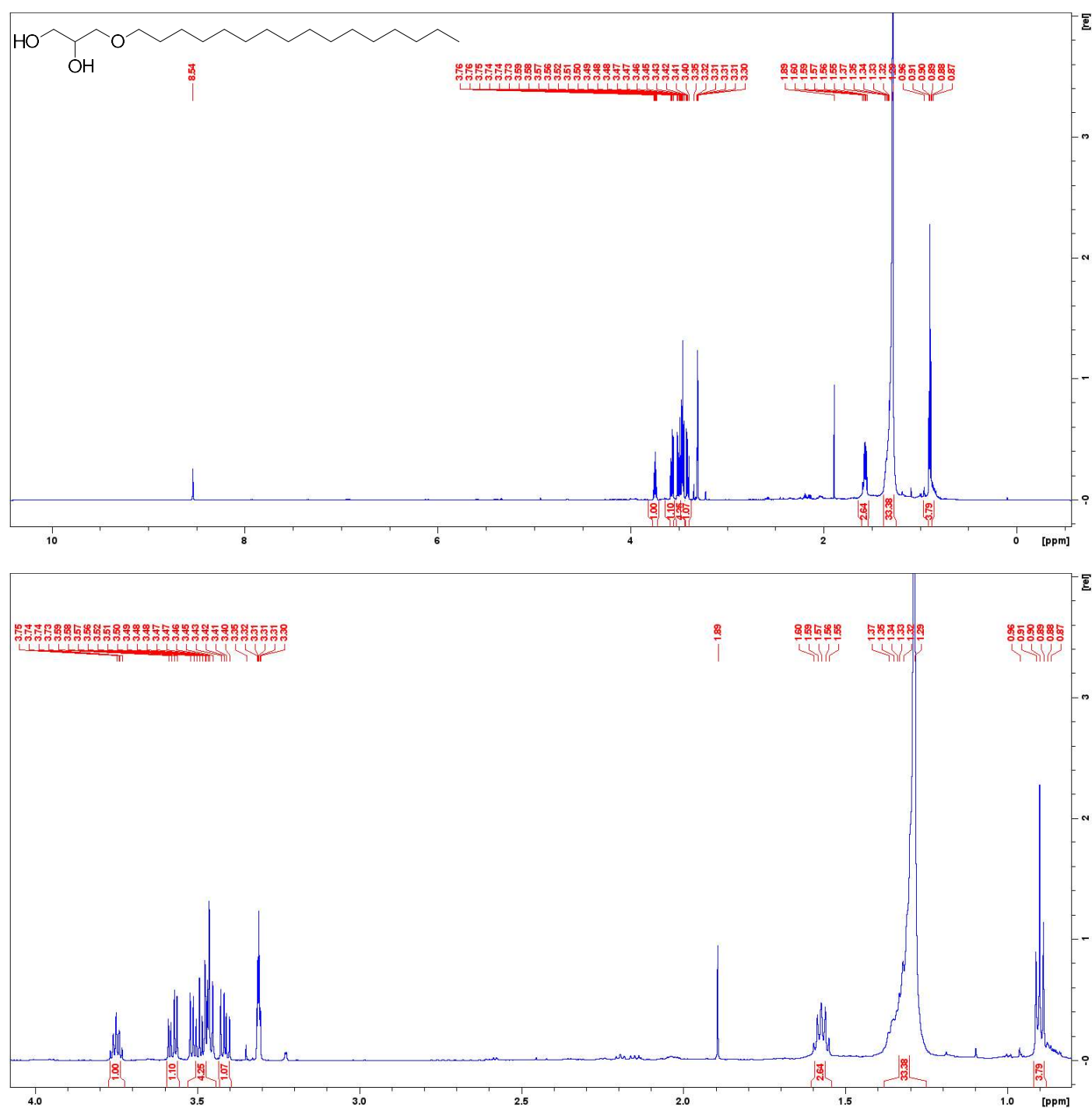
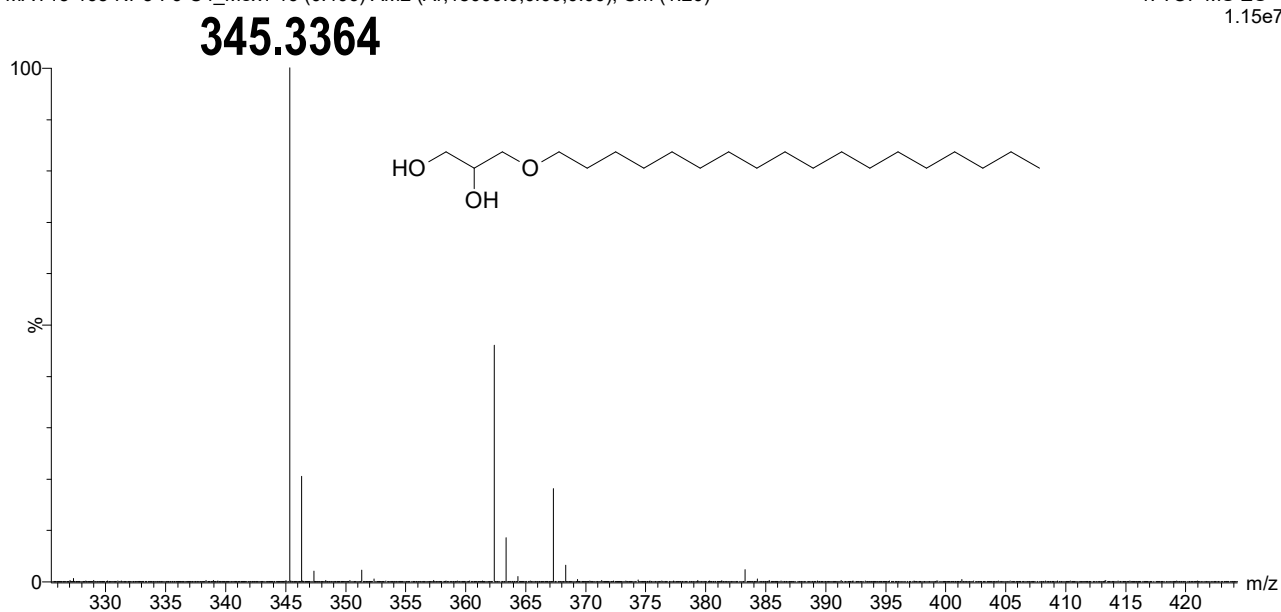


Figure S26. ^1H NMR spectrum of compound 1-O-hexadecylglycerol (7) (600 MHz, in CD_3OD).

MAY13-165-NP3-F6-C4_Mex1 16 (0.406) AM2 (Ar,18000.0,0.00,0.00); Cm (1:20)

1: TOF MS ES+
1.15e7**Figure S27.** HRMS-ESI⁺ spectrum of compound 1-O-octadecylglycerol (8).

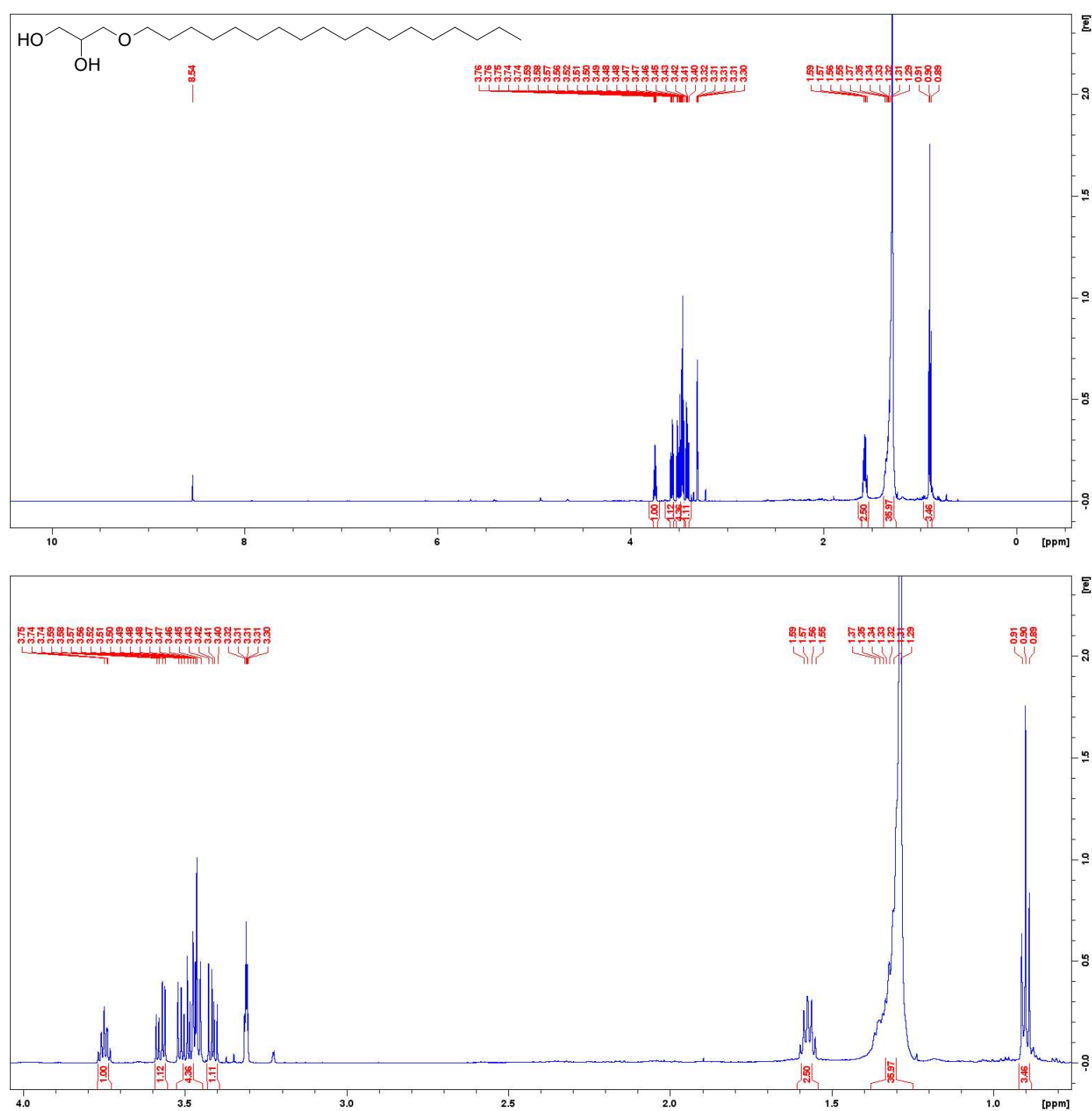


Figure S28. ¹H NMR spectrum of compound 1-O-octadecylglycerol (8) (600 MHz, in CD₃OD).

NP3-F6-C5_Mex1 12 (0.299) AM2 (Ar,18000.0,0.00,0.00); Cm (1:20)

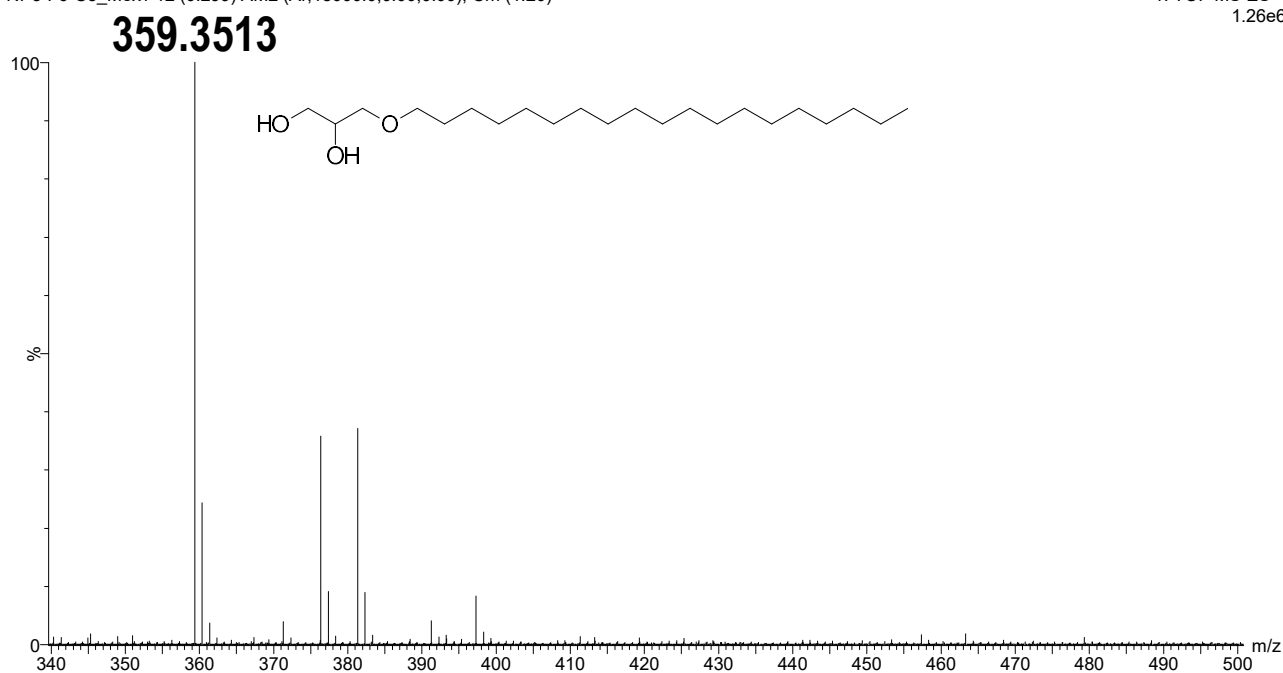
1: TOF MS ES+
1.26e6

Figure S29. HRMS-ESI⁺ spectrum of compound 3-nonadecyloxy-1,2-propanediol (9).

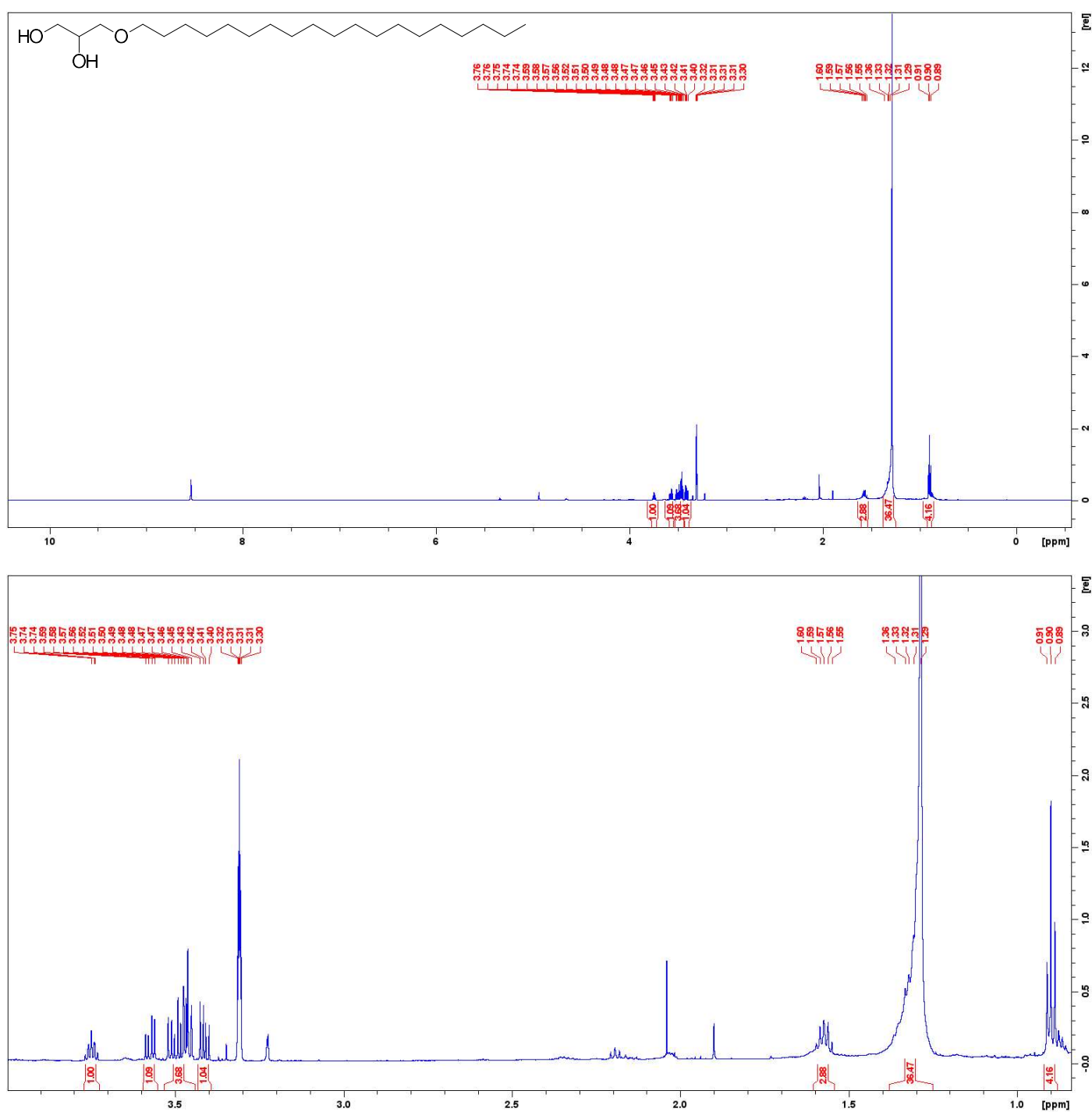
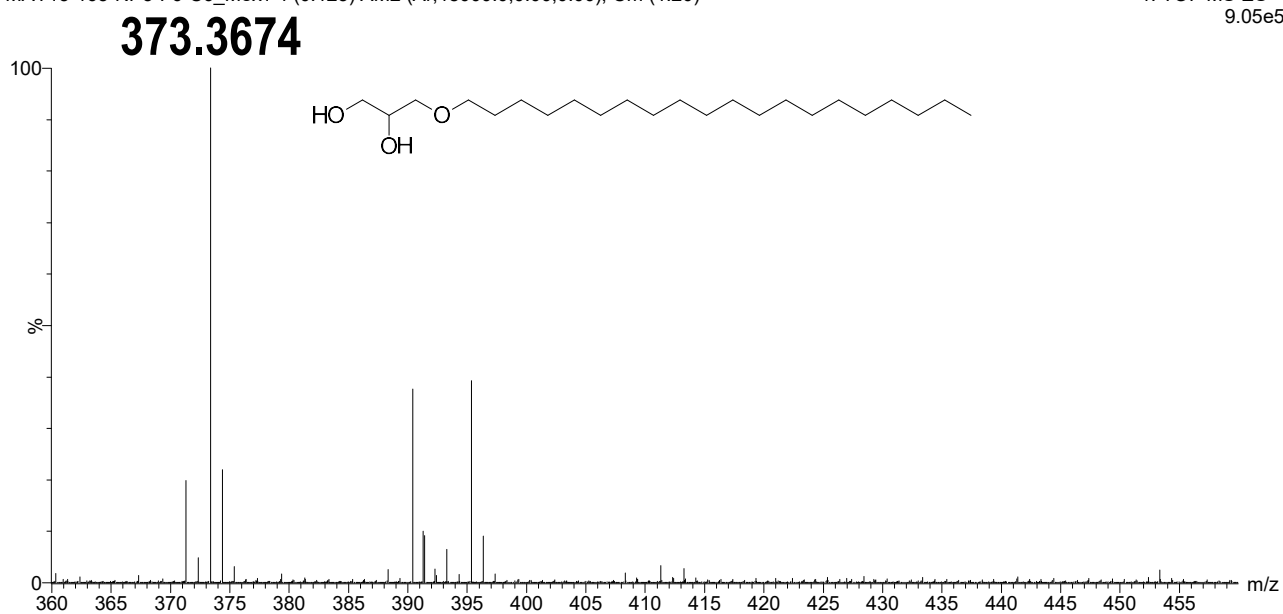


Figure S30. ^1H NMR spectrum of compound 3-nonadecyloxy-1,2-propanediol (9) (600 MHz, in CD_3OD).

MAY13-165-NP3-F6-C6_Mex1 4 (0.123) AM2 (Ar,18000.0,0.00,0.00); Cm (1:20)

1: TOF MS ES+
9.05e5**Figure S31.** HRMS-ESI⁺ spectrum of compound 3-icosoxypropane-1,2-diol (**10**).

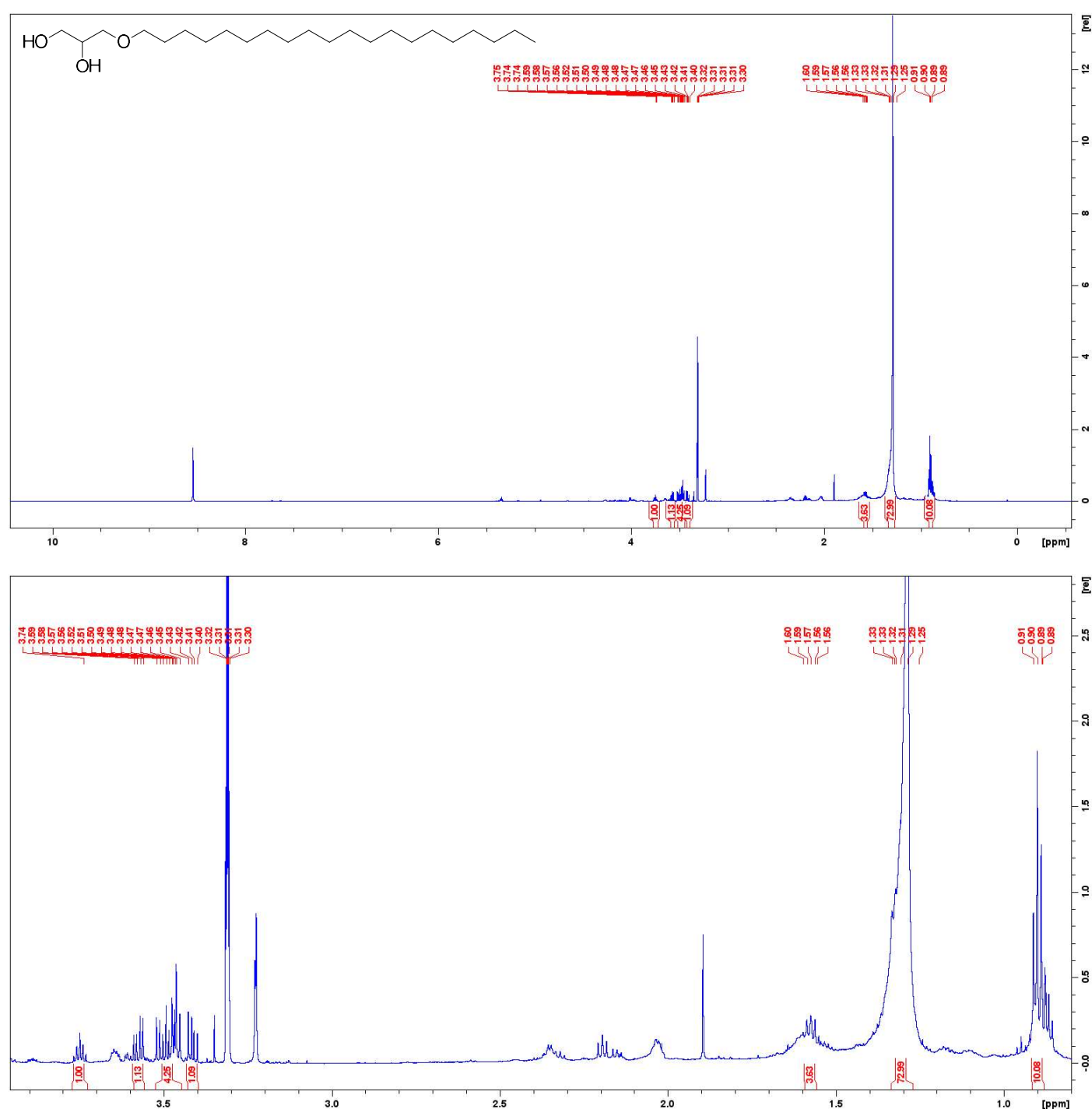


Figure S32. ^1H NMR spectrum of compound 3-icosoxypropane-1,2-diol (10) (600 MHz, in CD_3OD).

NP_F4F5-F11F12-C2_Mex3 3 (0.086) AM2 (Ar,18000.0,0.00,0.00); Cm (1:20)

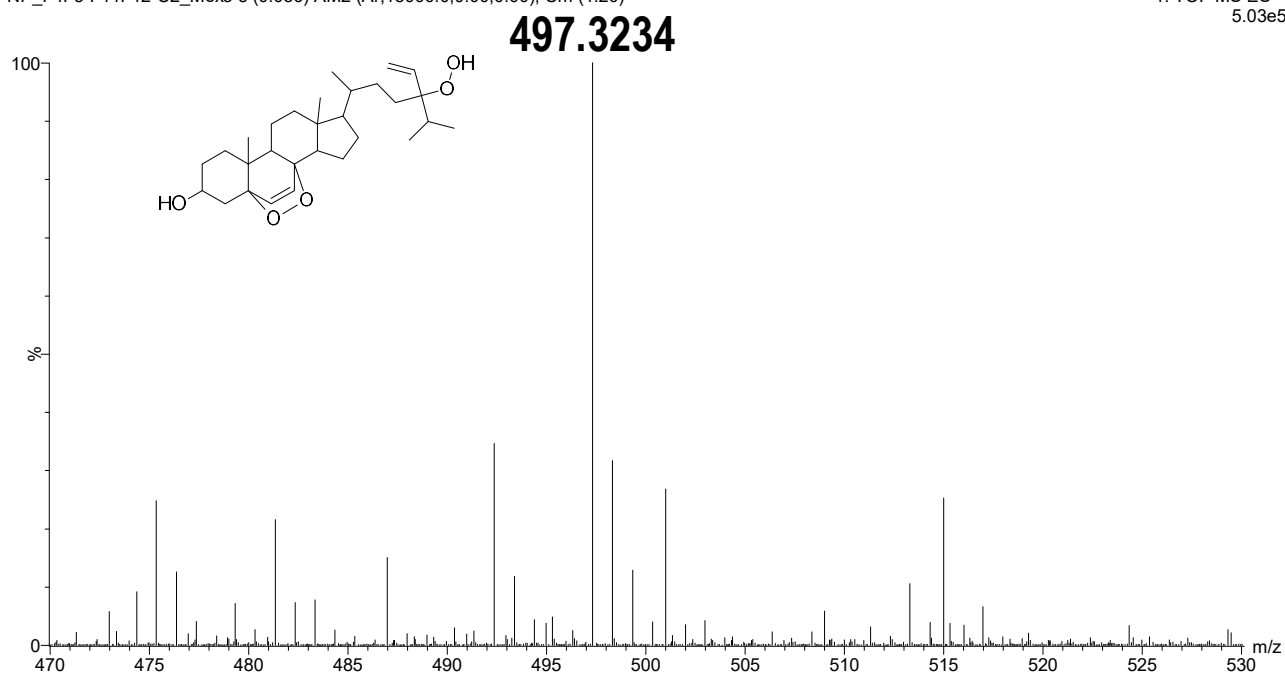
1: TOF MS ES+
5.03e5

Figure S33. HRMS-ESI⁺ spectrum of compound 5 α ,8 α -epidioxy-24(*R/S*)-hydroperoxystigmasta-6,28-dien-3 β -ol (**11**).

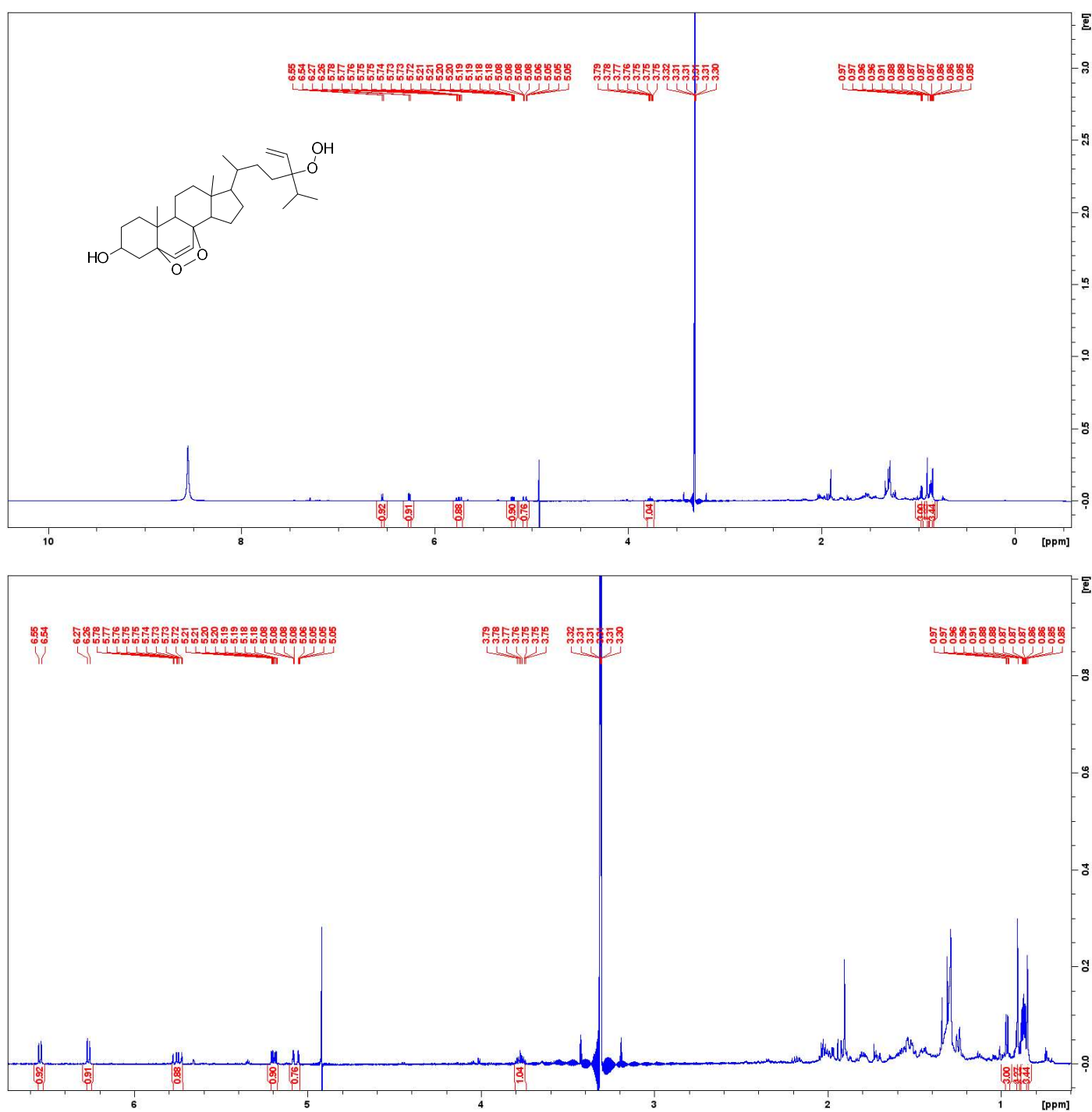


Figure S34. ^1H NMR spectrum of compound 5 α ,8 α -epidioxy-24(R/S)-hydroperoxystigmasta-6,28-dien-3 β -ol (11) (600 MHz, in CD_3OD).

MAY13-165-RP-F16-C64_Mex1 11 (0.282) AM2 (Ar,18000.0,0.00,0.00); Cm (1:20)

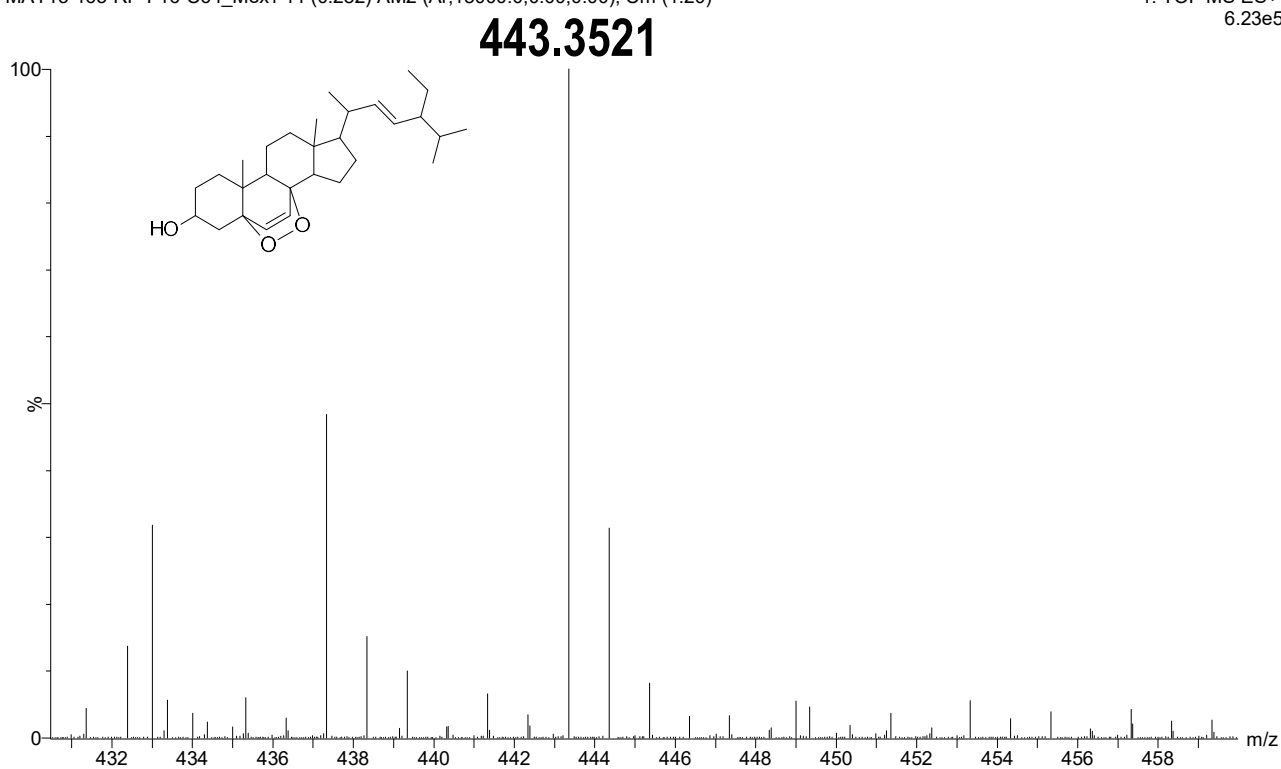
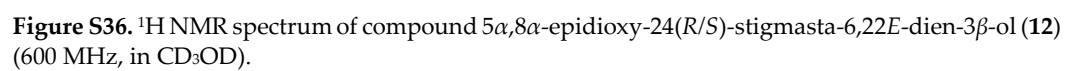
1: TOF MS ES+
6.23e5

Figure S35. HRMS-ESI⁺ spectrum of compound 5α,8α-epidioxy-24(R/S)-stigmasta-6,22E-dien-3β-ol (**12**).



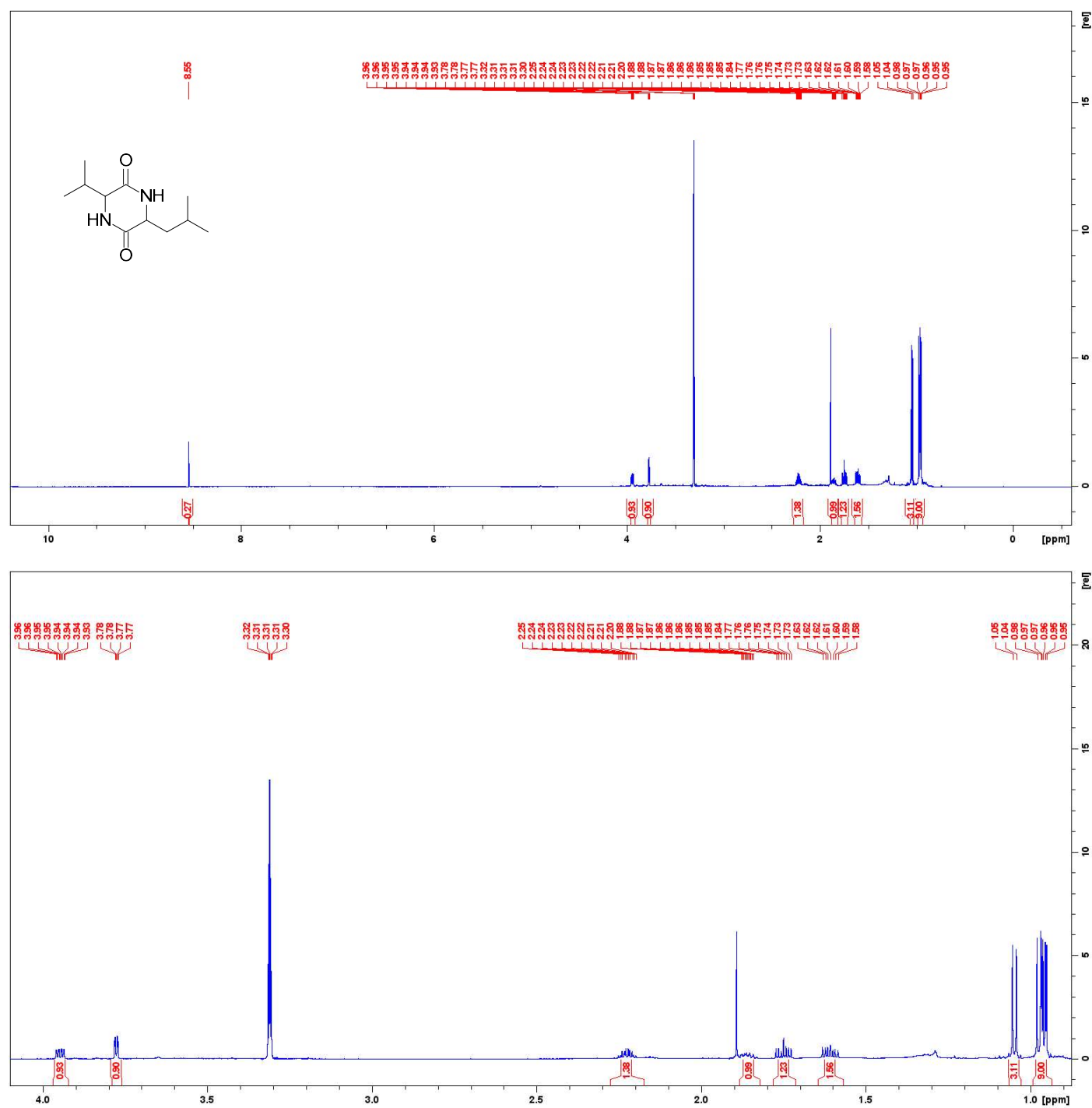


Figure S37. ^1H NMR spectrum of compound cyclo(Val-Leu) (13) (600 MHz, in CD_3OD).

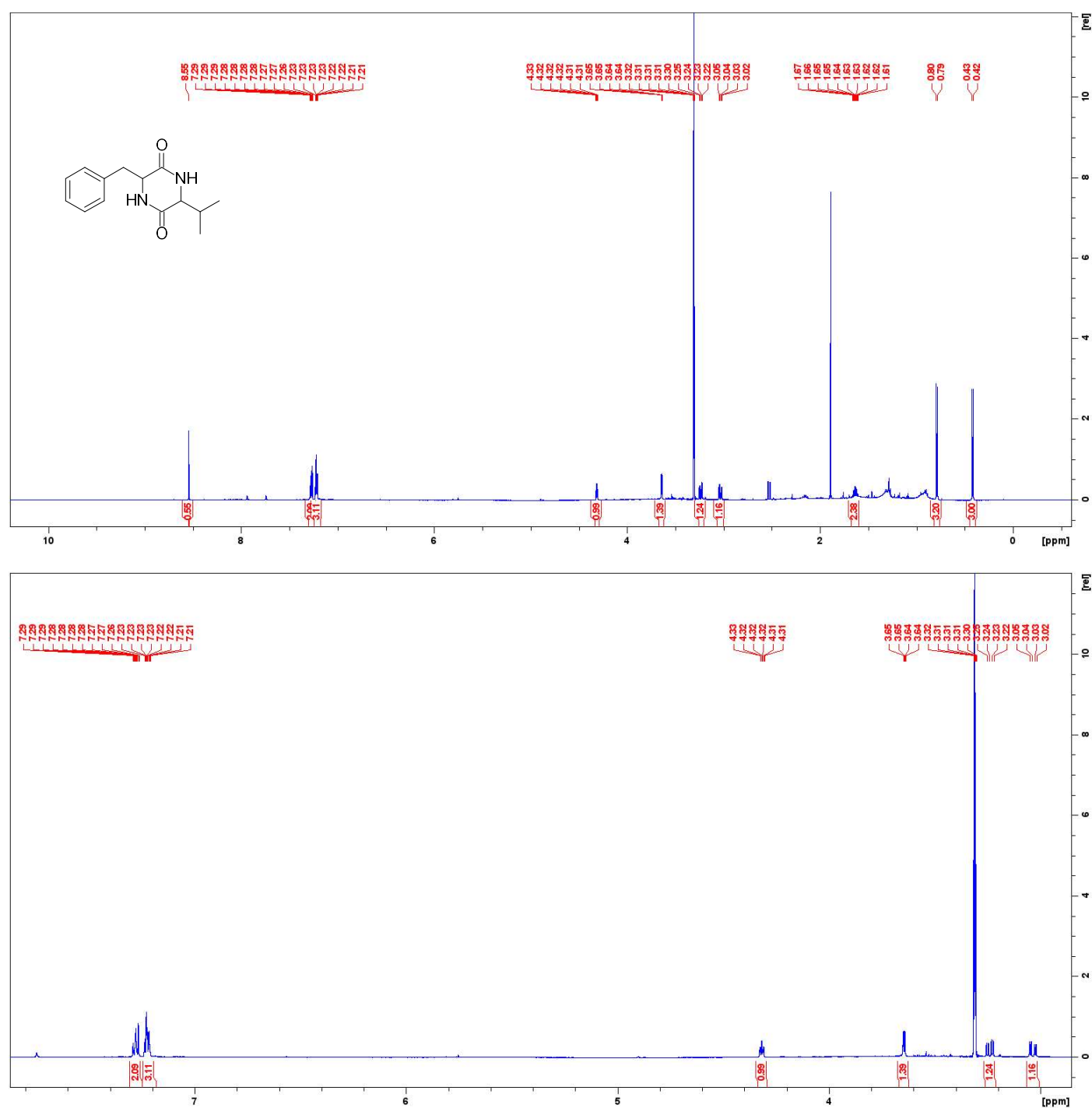


Figure S38. ^1H NMR spectrum of compound cyclo(Val-Phe) (**14**) (600 MHz, in CD_3OD).

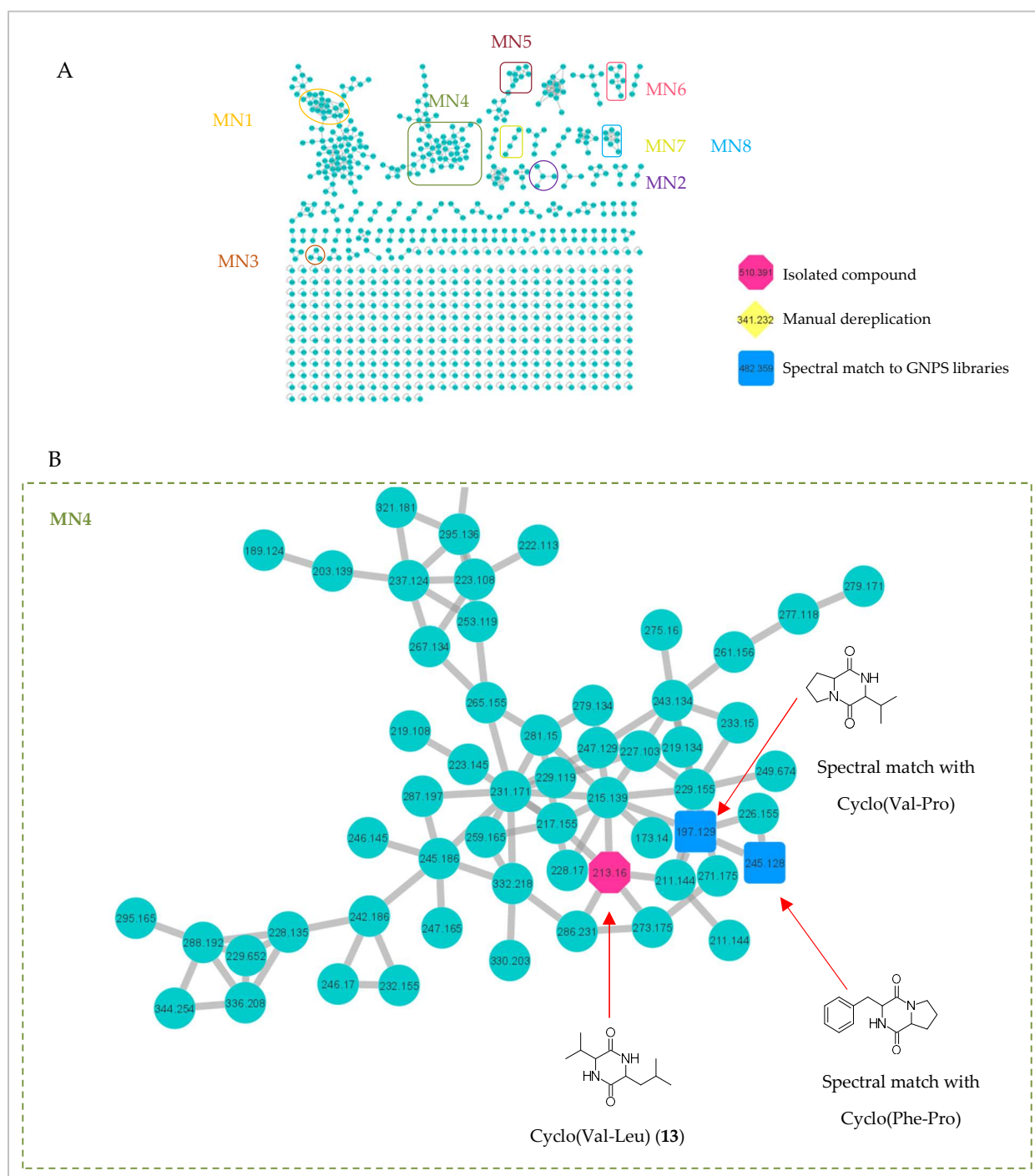


Figure S39. Dereplication of *Scopalina hapalia* fractions using LC-MS/MS molecular networking. (A) Molecular Network constructed using MS/MS data from fractions of *Scopalina hapalia*, obtained after the removal of non-polar lipids of organic crude extract, with a cosine similarity cutoff of 0.7. Edge thickness corresponds to relative cosine score similarity between nodes. Annotated clusters are enlarged. (B) Cluster MN4 related to diketopiperazines.

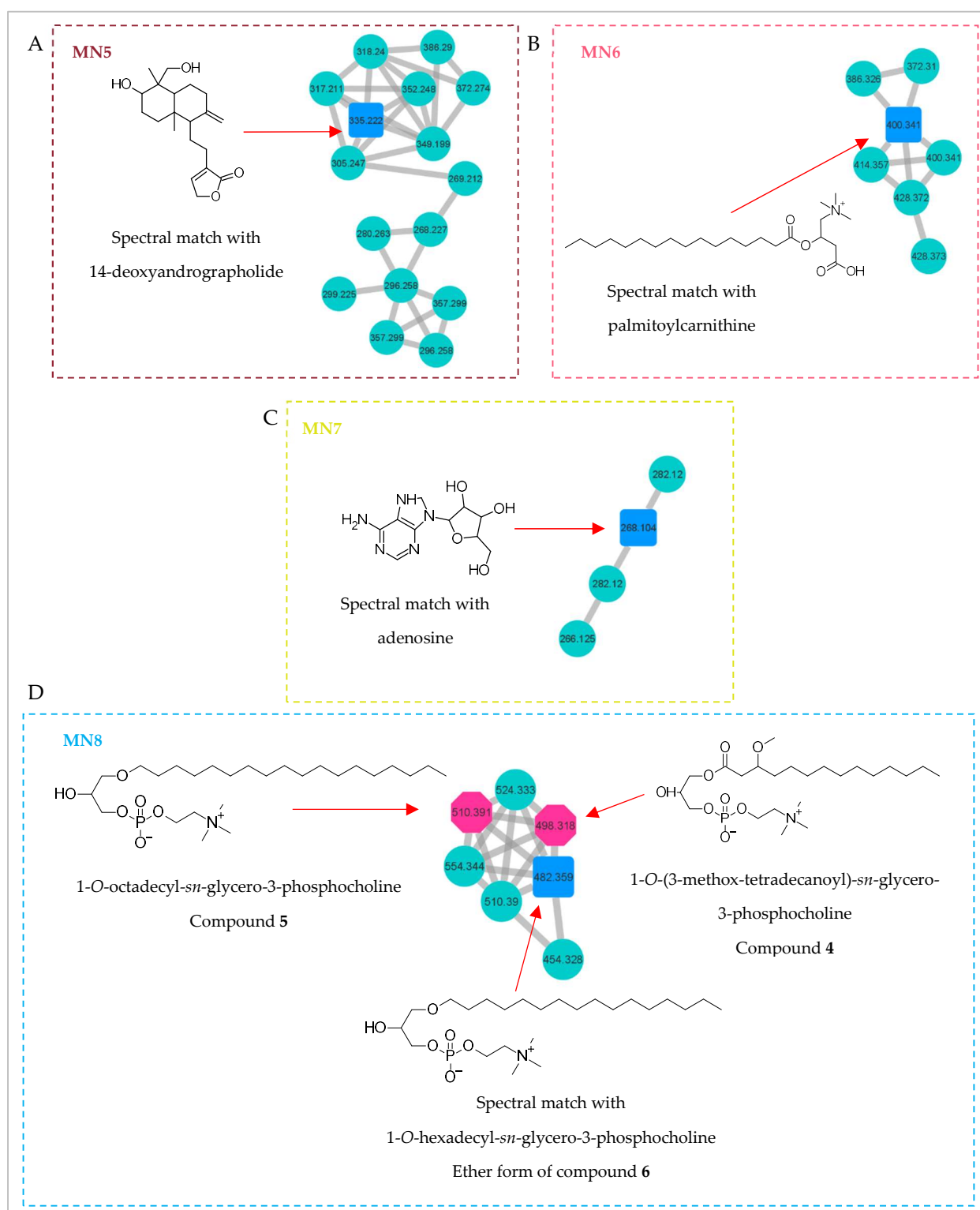


Figure S40. Dereplication of *Scopalina hapalia* fractions using LC-MS/MS molecular networking. (A) Cluster MN5 related to terpenoids. (B) Cluster MN6 related to palmitoylcarnithine. (C) Cluster MN7 related to purine nucleosides. (D) Cluster MN8 related to lysophospholipids.