Electronic Supplementary Material (ESI) for Soft Matter.

Supporting Information

Surface Properties of Nucleolipids and Photo-Controlled Release of Hydrophobic Guest Molecules from Their Micellar Aggregates

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1. Figure S1

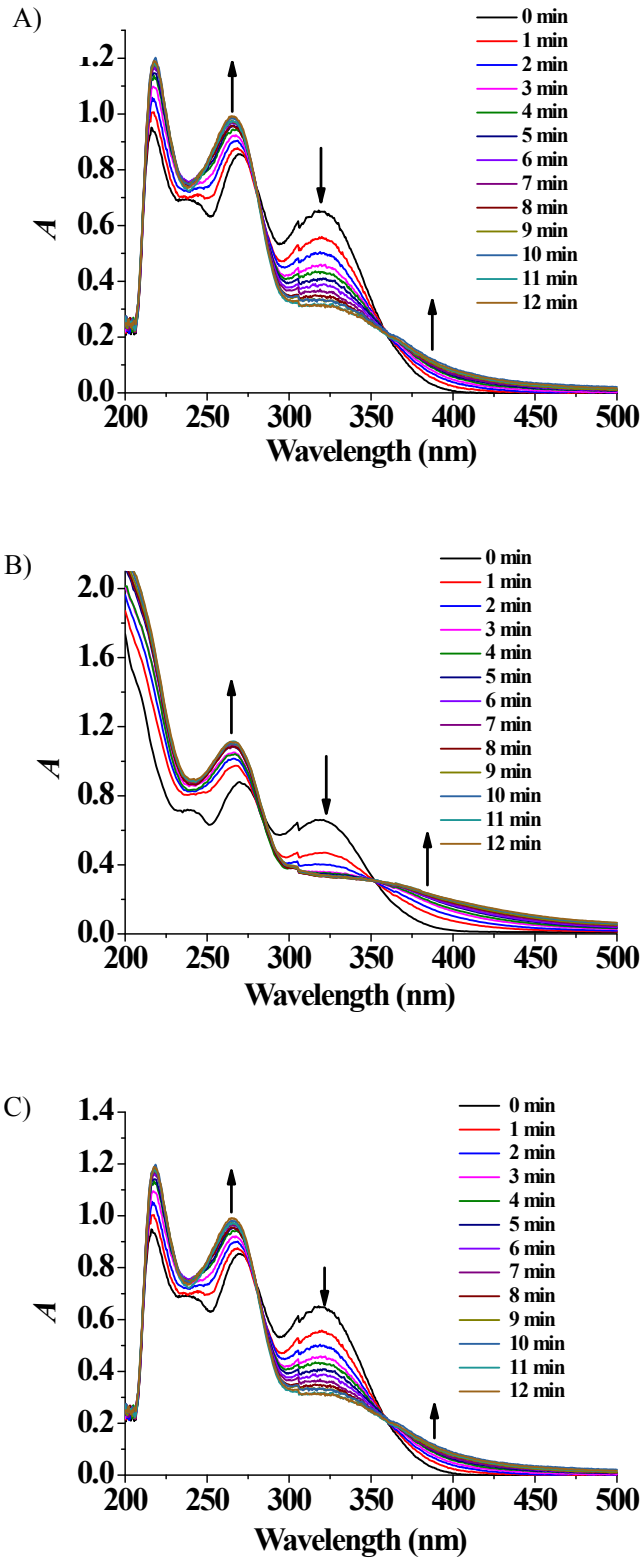


Fig. S1 UV-visible spectra of A) P-dT-C_{14}, B) P-dT-C_{12} and C) P-dT-C_{10} in 0.2 M sodium phosphate solution (pH of 8.0) under UV irradiation (350-380 nm, 50 mW/cm^2)

2. Figure S2

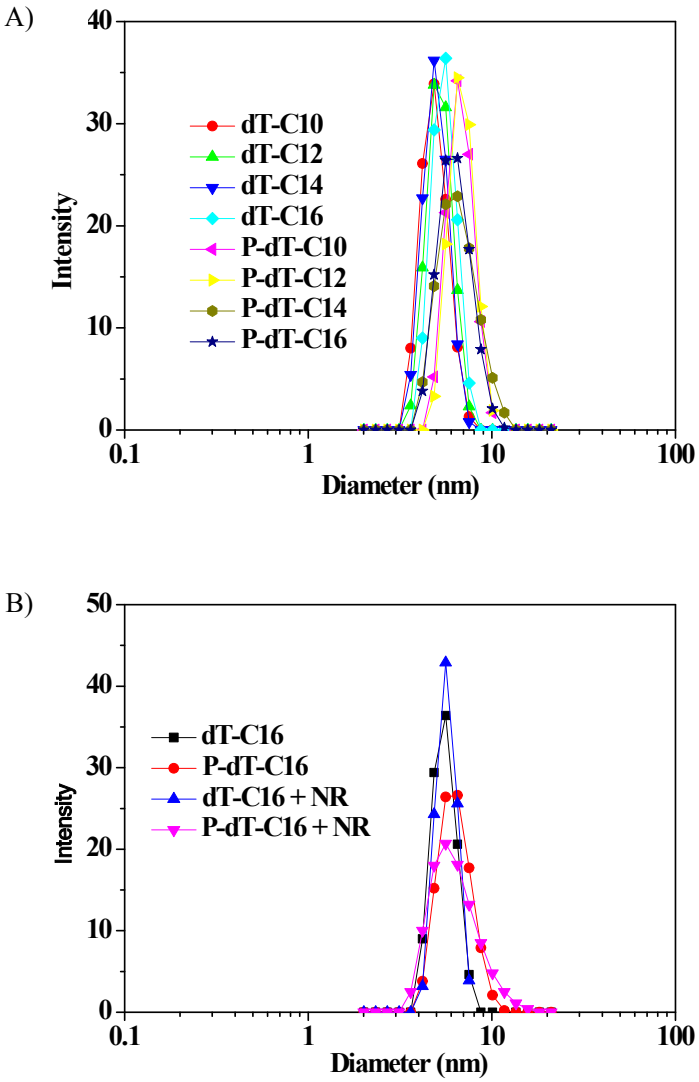


Fig. S2 A) DLS profiles of 10 mM dT-C_n and P-dT-C_n in 0.2 M sodium phosphate solution (pH of 8.0); B) DLS profiles before and after NR loading for 10 mM dT-C_{16} and P-dT-C_{16} in 0.2 M sodium phosphate solution (pH of 8.0)

3. Figure S3

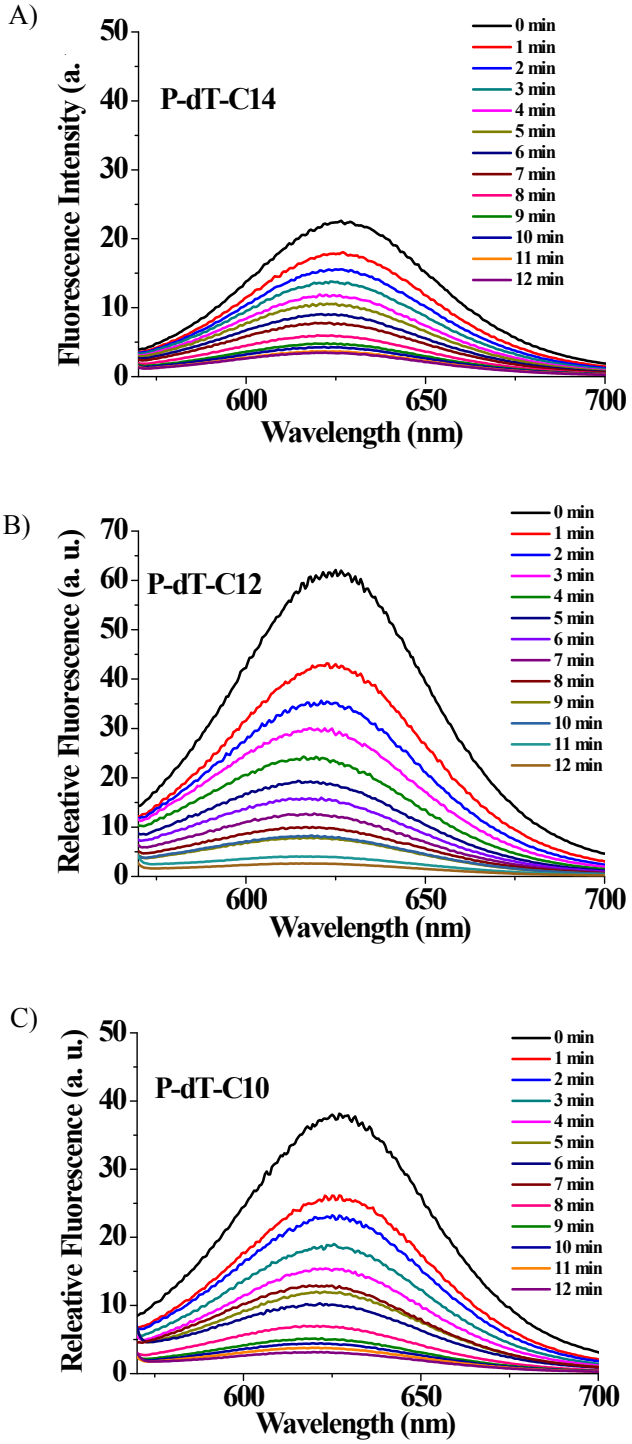


Fig. S3 Fluorescence emission spectra (λ_{exc}= 550 nm) of the NR loaded aqueous solutions of A) P-dT-C_{14}, B) P-dT-C_{12} and C) P-dT-C_{10} (10 mM) under UV irradiation (350-380 nm, 50 mW/cm^2).

4. Figure S4

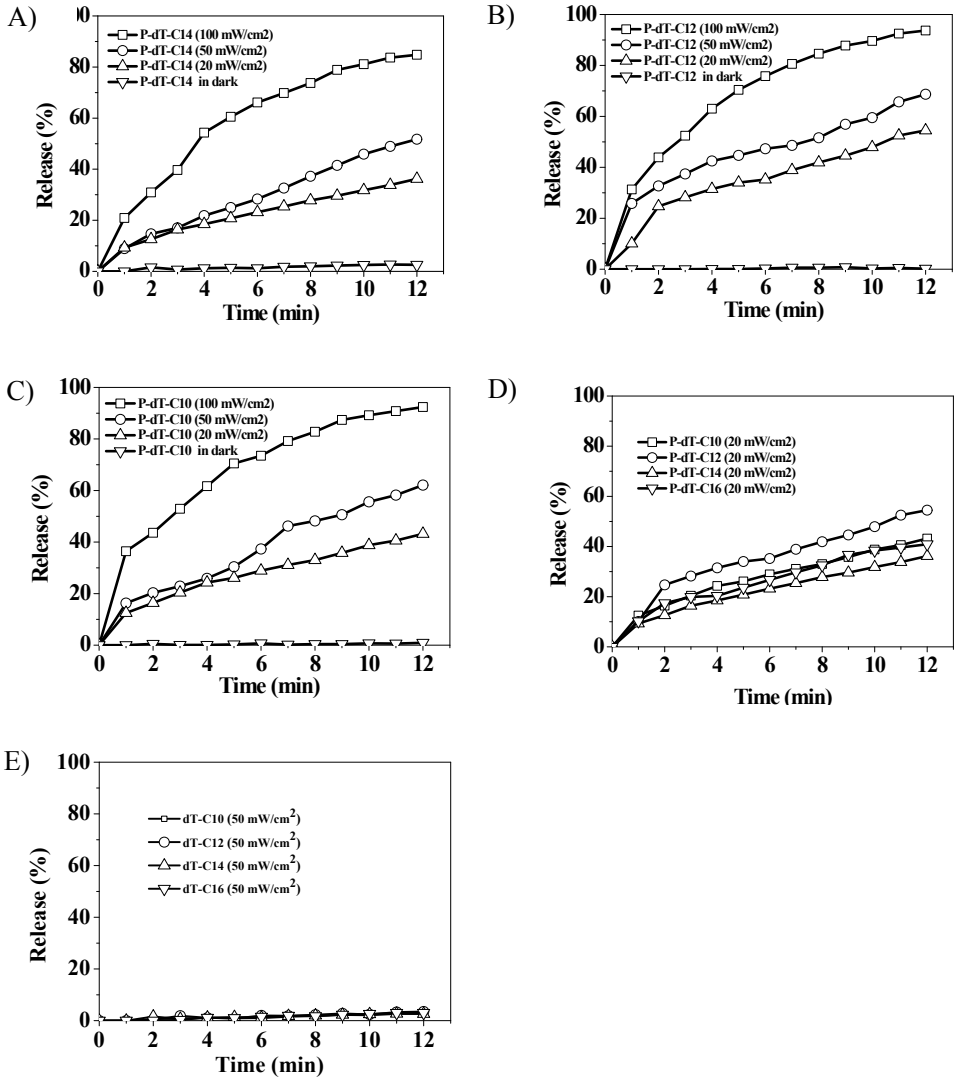


Fig. S4 NR Release profiles of the NR loaded A) P-dT-C_{14}, B) P-dT-C_{12} and C) P-dT-C_{10} solutions under UV irradiation at light intensities of 20, 50, and 100 mW/cm^2 and in the dark; D) Release profiles of NR in the P-dT-C_n solutions under UV irradiation of 20 mW/cm^2; E) Release profiles of NR in the dT-C_n solutions under UV irradiation of 50 mW/cm^2.

5. Figure S5

Fig. S5 The NR loaded dT-C_{16} solution (10 mM) before (left) and after (right) UV irradiation.

6. The calculation of NR Encapsulation efficiency

The NR encapsulation efficiency was calculated as following (as given in ESI in the revision)

$$Encapsulation\ efficency = \frac{total\ usage\ of\ NR\ (mg) - insolubel\ NR(mg)}{total\ usage\ of\ NR\ (mg)} \times 100\%$$

7. NMR and MS data

Compound 1a

Yield 77%, white solid. ^{1}H NMR (CD_3OD, 400 MHz): δ = 0.78-0.81 (m, C*H*$_3$, 3H), 1.20-1.32 (m, C*H*$_2$, 14H), 1.62-1.65 (m, C*H*$_2$, 2H), 1.62-1.65 (m, C*H*$_2$, 2H), 1.78 (s, C*H*$_3$, 3H), 2.43-2.44 (m, C*H*$_2$O, 2H), 2.80-2.83 (m, C*H*$_2$CN, 2H), 3.81 (m, C*H*$_2$O, 2H), 4.05-4.07 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 4.18-4.20 (m, C*H*O, 1H), 4.98-5.01 (s, C*H*O, 2H), 6.20-6.23 (m, Ar*H*, 1H), 7.70 (m, N*H*, 1H). ESI MS calcd for $C_{23}H_{39}N_3O_8P$,516.54 ($M+H^+$), found 516.25;$C_{23}H_{42}N_4O_8P$,533.58 ($M+NH_4^+$), found 533.27;$C_{23}H_{38}N_3NaO_8P$,538.53($M+Na^+$), found 538.23.

Compound 2a

Yield 71%, white solid.^{1}H NMR (CD_3OD, 400 MHz): δ = 0.89-0.92 (m, C*H*$_3$, 3H), 1.31-1.35 (m, C*H*$_2$, 18H), 1.42-1.44 (m, C*H*$_2$, 2H), 1.79-1.86 (m, C*H*$_2$, 2H), 1.90 (s, C*H*$_3$, 3H), 2.43-2.55 (m, C*H*$_2$O, 2H), 2.93-2.95 (m, C*H*$_2$CN, 2H), 3.29 (b, O*H*, 1H), 3.83 (m, C*H*$_2$O, 2H), 4.17-4.19 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 4.23-4.24 (m, C*H*O, 1H), 4.29-4.31 (m, C*H*$_2$O, 2H), 5.12 (s, C*H*O, 1H), 6.33-6.35 (m, Ar*H*, 1H), 7.82 (m, N*H*, 1H). ESI MS calcd for $C_{25}H_{43}N_3O_8P$,544.59 ($M+H^+$), found 544.27; $C_{25}H_{42}N_3NaO_8P$ 566.58($M+Na^+$), found 566.26.

Compound 3a

Yield 82%, white solid.^{1}H NMR (CD_3OD, 400 MHz): δ = 0.78-0.81 (m, C*H*$_3$, 3H), 1.18-1.22 (m, C*H*$_2$, 22H), 1.61-1.63 (m, C*H*$_2$, 2H), 1.78 (s, C*H*$_3$, 3H), 2.32-2.47 (m, C*H*$_2$O, 2H), 2.79-2.83 (m, C*H*$_2$CN, 2H), 3.29 (b, O*H*, 1H), 3.70-3.71 (m, C*H*$_2$O, 2H), 4.05-4.07 (m, C*H*$_2$O, 2H), 4.11-4.12 (m, C*H*O, 1H), 4.13-4.21 (m, C*H*$_2$O, 2H), 4.98-5.01 (m, C*H*O, 1H), 6.19-6.23 (m, Ar*H*, 1H), 7.70 (m, N*H*, 1H). ESI MS calcd for $C_{27}H_{47}N_3O_8P$,572.65 ($M+H^+$), found 572.31; $C_{27}H_{50}N_4O_8P$,589.68 ($M+NH_4^+$), found 589.33; $C_{27}H_{46}N_3NaO_8P$,594.29 ($M+Na^+$), found 594.63.

Compound 4a

Yield 68%, white solid.^{1}H NMR (CD_3OD, 400 MHz): δ = 0.78-0.82 (m, C*H*$_3$, 3H), 1.18-1.22 (m, C*H*$_2$, 26H), 1.61-1.63 (m, C*H*$_2$, 2H), 1.78 (s, C*H*$_3$, 3H), 2.34-2.49 (m, C*H*$_2$O, 2H), 2.79-2.83 (m, C*H*$_2$CN, 2H), 3.29 (b, O*H*, 1H), 3.79-3.81 (m, C*H*$_2$O, 2H), 4.05-4.07 (m, C*H*$_2$O, 2H), 4.18-4.19 (m, C*H*O, 1H), 4.21-4.23 (m, C*H*$_2$O, 2H), 4.98-

5.01 (m, C*H*O, 1H), 6.19-6.23 (m, Ar*H*, 1H), 7.70 (m, N*H*, 1H). ESI MS calcd for $C_{29}H_{51}N_3O_8P$,600.70 ($M+H^+$), found 600.34; $C_{29}H_{54}N_4O_8P$,617.73 ($M+NH_4^+$), found 617.37; $C_{29}H_{50}N_3NaO_8P$,622.69 ($M+Na^+$), found 622.32.

Compound 5a

Yield 92%, yellow solid.^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.86-0.89 (t, *J* = 7 Hz, C*H*$_3$, 3H), 1.28-1.39 (m, C*H*$_2$, 12H), 1.43-1.48 (m, C*H*$_2$, 2H), 1.80-1.86 (m, C*H*$_2$, 2H), 4.09-4.11 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 7.12-7.15 (dd, J_1 = 3 Hz, J_2 = 9 Hz, Ar*H*, 1H), 7.30-7.31 (d, *J* = 3 Hz, Ar*H*, 1H), 8.14-8.16 (d, *J* = 9 Hz, Ar*H*, 1H), 10.49 (s, ArC*H*O, 1H); ESI MS calcd for $C_{17}H_{26}NO_4$ 308.38 ($M+H^+$), found 308.19; $C_{17}H_{25}NO_4Na$ 330.37 ($M+Na^+$), found 330.17;$C_{17}H_{25}NO_4K$ 346.48 ($M+K^+$), found 346.15.

Compound 6a

Yield 90%, yellow solid.^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.87-0.89 (t, *J* = 7 Hz, C*H*$_3$, 3H), 1.27-1.37 (m, C*H*$_2$, 16H), 1.43-1.48 (m, C*H*$_2$, 2H), 1.80-1.86 (m, C*H*$_2$, 2H), 4.09-4.11 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 7.12-7.14 (dd, J_1 = 3 Hz, J_2 = 9 Hz, Ar*H*, 1H), 7.30-7.31 (d, *J* = 3 Hz, Ar*H*, 1H),8.14-8.16 (d, *J* = 9 Hz, Ar*H*, 1H), 10.49 (s, ArC*H*O, 1H); ESI MS calcd for $C_{19}H_{30}NO_4$, 336.44 ($M+H^+$), found 336.22; $C_{19}H_{29}NO_4Na$, 358.43 ($M+Na^+$), found 358.20.

Compound 7a

Yield 87%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.86-0.89 (t, *J* = 7 Hz, C*H*$_3$, 3H), 1.26-1.37 (m, C*H*$_2$, 20H), 1.45-1.48 (m, C*H*$_2$, 2H), 1.80-1.85 (m, C*H*$_2$, 2H), 4.09-4.11 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 7.12-7.15 (dd, J_1 = 3 Hz, J_2 = 9 Hz, Ar*H*, 1H), 7.30-7.31 (d, *J* = 3 Hz, Ar*H*, 1H),8.14-8.16 (d, *J* = 9.5 Hz, Ar*H*, 1H), 10.48 (s, ArC*H*O, 1H); ESI MS calcd for $C_{21}H_{34}NO_4$, 364.49 ($M+H^+$), found 364.25; $C_{21}H_{33}NO_4Na$, 386.48 ($M+Na^+$), found 386.23.

Compound 8a

Yield 80%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.87-0.89 (t, *J* = 7 Hz, C*H*$_3$, 3H), 1.26-1.37 (m, C*H*$_2$, 24H), 1.44-1.48 (m, C*H*$_2$, 2H), 1.80-1.86 (m, C*H*$_2$, 2H), 4.09-4.11 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 7.12-7.15 (dd, J_1= 3 Hz, J_2 = 9 Hz, Ar*H*, 1H), 7.30-7.31 (d, *J* = 3 Hz, Ar*H*, 1H),8.14-8.16 (d, *J* = 9.5 Hz, Ar*H*, 1H), 10.48 (s, ArC*H*O, 1H); ESI MS calcd for $C_{23}H_{38}NO_4$, 392.54 ($M+H^+$), found 392.28,$C_{23}H_{37}NO_4Na$,

414.53 ($M+Na^+$), found 414.26.

Compound 1b

Yield 66%, white solid.^{1}H NMR (CD_3OD, 400 MHz): δ = 0.79-0.81 (m, C*H*$_3$, 3H), 1.19-1.27 (m, C*H*$_2$+C*H*$_3$, 23H), 1.51-1.53 (m, C*H*$_2$, 2H), 1.78 (s, C*H*$_3$, 3H), 2.17-2.35 (m, C*H*$_2$O, 2H), 3.36-3.38 (m, 6H, C*H*$_2$N), 3.68-3.70 (m, C*H*$_2$O, 2H), 3.75-3.77 (m, C*H*$_2$O, 2H), 4.04-4.05 (m, C*H*O, 1H), 4.71-4.74 (m, C*H*O, 1H), 6.19-6.22 (m, C*H*O, 1H),7.76-7.77 (m, Ar*H*, 1H). ESI MS calcd for $C_{20}H_{34}N_2O_8P$,461.47 ($M-Et_3NH^+$), found 461.20.

Compound 2b

Yield 60%, white solid. ^{1}H NMR (CD_3OD, 500 MHz): δ = 0.79-0.81 (m, C*H*$_3$, 3H), 1.18-1.23 (m, C*H*$_2$+C*H*$_3$, 27H), 1.51-1.54 (m, C*H*$_2$, 2H), 1.88 (s, C*H*$_3$, 3H), 2.17-2.37 (m, C*H*$_2$O, 2H), 3.29 (b, O*H*, 1H), 3.09-3.12 (m, 6H, C*H*$_2$N), 3.67-3.69 (m, C*H*O, 1H), 3.71-3.78 (m, C*H*$_2$O, 2H), 4.04-4.05 (m, C*H*O, 1H), 4.71-4.73 (m, C*H*O, 1H), 6.18-6.22 (m, C*H*O, 1H),7.76-7.77 (m, Ar*H*, 1H). ESI MS calcd for $C_{22}H_{38}N_2O_8P$,489.52 ($M-Et_3NH^+$), found 489.24.

Compound 3b

Yield 58%, white solid. ^{1}H NMR (CD_3OD, 500 MHz): δ = 0.79-0.81 (m, C*H*$_3$, 3H), 1.18-1.27 (m, C*H*$_2$+C*H*$_3$, 31H), 1.51-1.53 (m, C*H*$_2$, 2H), 1.87 (s, C*H*$_3$, 3H), 2.17-2.37 (m, C*H*$_2$O, 2H), 3.20-3.22 (m, 6H, C*H*$_2$N), 3.35-3.40 (m, C*H*O, 1H), 3.69-3.70 (m, C*H*O, 1H), 3.76-3.77 (m, C*H*$_2$O, 2H),4.04-4.05 (m, C*H*O, 1H), 4.71-4.73 (m, C*H*O, 1H), 6.20-6.21 (m, C*H*O, 1H), 7.76-7.77 (m, Ar*H*, 1H). ESI MS calcd for $C_{24}H_{42}N_2O_8P$,517.27 ($M-Et_3NH^+$), found 512.57.

Compound 4b

Yield 55%, white solid. ^{1}H NMR (CD_3OD, 500 MHz): δ = 0.79-0.81 (m, C*H*$_3$, 3H), 1.18-1.23 (m, C*H*$_2$+C*H*$_3$, 35H), 1.52-1.55 (m, C*H*$_2$, 2H), 1.78 (s, C*H*$_3$, 3H), 2.17-2.36 (m, C*H*$_2$O, 2H), 3.07-3.13 (m, 6H, C*H*$_2$N), 3.69-3.70 (m, C*H*O, 1H), 3.76-3.78 (m, C*H*$_2$O, 2H), 4.04-4.05 (m, C*H*O, 1H), 4.71-4.73 (m, C*H*O, 1H), 6.19-6.22 (m, C*H*O, 1H), 7.76-7.77 (m, Ar*H*, 1H). ESI MS calcd for $C_{26}H_{46}N_2O_8P$,545.63 ($M-Et_3NH^+$), found 542.29.

Compound 5b

Yield 95%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.87-0.89 (t, *J* = 7 Hz, C*H*$_3$, 3H), 1.28-1.32 (m, C*H*$_2$, 12H), 1.43-1.49 (m, C*H*$_2$, 2H), 1.79-1.84 (m, C*H*$_2$, 2H), 2.74 (b, O*H*, 1H), 4.05-4.08 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 4.98 (s, ArC*H*$_2$OH, 2H), 6.86-6.88 (dd, J_1 = 3 Hz, J_2 = 9 Hz, Ar*H*, 1H), 7.19-7.20 (d, *J* = 3 Hz, Ar*H*, 1H),8.15-8.17 (d, *J* = 9 Hz, Ar*H*, 1H); ESI MS calcd for $C_{17}H_{28}NO_4$, 310.40 ($M+H^+$), found 310.20; $C_{17}H_{27}NO_4Na$, 332.39 ($M+Na^+$), found 332.19; $C_{17}H_{27}NO_4K$, 348.50 ($M+K^+$), found 348.16;

Compound 6b

Yield 93%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.87-0.89 (t, *J* = 7 Hz, C*H*$_3$, 3H), 1.27-1.32 (m, C*H*$_2$, 16H), 1.43-1.48 (m, C*H*$_2$, 2H), 1.79-1.84 (m, C*H*$_2$, 2H), 2.74 (b, O*H*, 1H), 4.05-4.08 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 4.98 (s, ArC*H*$_2$OH, 2H), 6.86-6.88 (dd, J_1 = 3 Hz, J_2 = 9 Hz, Ar*H*, 1H), 7.19-7.20 (d, *J* = 3 Hz, Ar*H*, 1H),8.15-8.17 (d, *J* = 9 Hz, Ar*H*, 1H); ESI MS calcd for $C_{19}H_{30}NO_4$, 338.45 ($M+H^+$), found 338.2; $C_{19}H_{29}NO_4Na$ 360.44 ($M+Na^+$), found 360.21.

Compound 7b

Yield 94%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.87-0.89 (t, *J* = 7 Hz, C*H*$_3$, 3H), 1.26-1.31 (m, C*H*$_2$, 20H), 1.43-1.48 (m, C*H*$_2$, 2H), 1.79-1.84 (m, C*H*$_2$, 2H), 2.74 (b, O*H*, 1H), 4.05-4.08 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 4.98 (s, ArC*H*$_2$OH, 2H), 6.86-6.88 (dd, J_1 = 3 Hz, J_2 = 9 Hz, Ar*H*, 1H), 7.19-7.20 (d, *J* = 3 Hz, Ar*H*, 1H),8.15-8.17 (d, *J* = 9 Hz, Ar*H*, 1H); ESI MS calcd for $C_{21}H_{34}NO_4$, 366.51 ($M+H^+$), found 366.26; $C_{21}H_{33}NO_4Na$, 388.50 ($M+Na^+$), found 388.24.

Compound 8b

Yield 83%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.87-0.89 (t, *J* = 7 Hz, C*H*$_3$, 3H), 1.26-1.31 (m, C*H*$_2$, 24H), 1.43-1.48 (m, C*H*$_2$, 2H), 1.79-1.84 (m, C*H*$_2$, 2H), 2.74 (b, O*H*, 1H), 4.05-4.08 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 4.98 (s, ArC*H*$_2$OH, 2H), 6.86-6.88 (dd, J_1 = 3 Hz, J_2 = 9 Hz, Ar*H*, 1H), 7.19-7.20 (d, *J* = 3 Hz, Ar*H*, 1H),8.16-8.18 (d, *J* = 9 Hz, Ar*H*, 1H); ESI MS calcd for $C_{23}H_{38}NO_4$, 394.56 ($M+H^+$), found 394.26; $C_{23}H_{37}NO_4Na$, 416.55 ($M+Na^+$), found 416.28.

Compound 5c

Yield 70%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.86-0.89 (m, C*H*$_3$, 3H),

1.27-1.35 (m, CH_2, 12H), 1.44-1.46 (m, CH_2, 2H), 1.80-1.85 (m, CH_2, 2H), 1.88 (s, CH_3, 3H), 2.46-2.52 (m, CH_2O, 2H), 2.85-2.86 (m, CH_2CN, 2H), 3.29 (b, O*H*, 1H), 3.89-3.91 (m, CH_2O, 2H), 4.07-4.09 (t, J = 6 Hz, CH_2OAr, 2H), 4.24-4.27 (m, CH_2O, 2H), 4.35-4.40 (m, CH_2O, 2H), 5.26 (s, C*H*O, 1H), 5.55-5.56 (d, J = 6.5 Hz, ArCH_2O, 2H), 6.23-6.24 (d, J = 4 Hz, C*H*O, 1H), 6.92-6.94 (d, J_1 = 2.5 Hz, J_2= 9 Hz, Ar*H*, 1H), 7.17-7.18 (d, J= 2.5 Hz, Ar*H*, 1H), 7.55-7.57 (d, J = 9 Hz, Ar*H*, 1H),8.18-8.20 (d, J = 9 Hz, Ar*H*, 1H); ESI MS calcd for $C_{30}H_{44}N_4O_{11}P$,667.66 ($M+H^+$), found 667.27; $C_{30}H_{47}N_5O_{11}P$, 68.69 ($M+NH_4^+$), found 684.29; $C_{30}H_{43}NaN_4O_{11}P$,689.65 ($M+Na^+$), found 689.25; $C_{30}H_{43}KN_4O_{11}P$,705.75 ($M+K^+$), found 705.23.

Compound 6c

Yield 72%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.85-0.89 (m, CH_3, 3H), 1.27-1.35 (m, CH_2, 14H), 1.44-1.47 (m, CH_2, 2H), 1.79-1.86 (m, CH_2, 2H), 1.88 (s, CH_3, 3H), 2.47-2.52 (m, CH_2O, 2H), 2.85-2.86 (m, CH_2CN, 2H), 3.29 (b, O*H*, 1H), 3.89-3.91 (m, CH_2O, 2H), 4.07-4.09 (t, J = 6.5 Hz, CH_2OAr, 2H), 4.24-4.26 (m, CH_2O, 2H), 4.35-4.40 (m, CH_2O, 2H), 5.26 (s, C*H*O, 1H), 5.54-5.56 (d, J = 6.5 Hz, ArCH_2O, 2H), 6.21-6.22 (d, J = 4 Hz, C*H*O, 1H), 6.90-6.93(d, J_1 = 2.5 Hz, J_2= 9 Hz, Ar*H*, 1H), 7.17-7.18 (d, J= 2.5 Hz, Ar*H*, 1H), 7.55-7.58 (d, J = 9 Hz, Ar*H*, 1H),8.18-8.20 (d, J = 9 Hz, Ar*H*, 1H); ESI MS calcd for $C_{32}H_{48}N_4O_{11}P$,695.71 ($M+H^+$), found 695.30; $C_{32}H_{51}N_5O_{11}P$, 712.75 ($M+NH_4^+$), found 712.33.

Compound 7c

Yield 62%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.86-0.89 (m, CH_3, 3H), 1.26-1.35 (m, CH_2, 18H), 1.43-1.47 (m, CH_2, 2H), 1.79-1.84 (m, CH_2, 2H), 1.88 (s, CH_3, 3H), 2.49-2.58 (m, CH_2O, 2H), 2.84-2.87 (m, CH_2CN, 2H), 3.29 (b, O*H*, 1H), 3.88-3.90 (m, CH_2O, 2H), 4.06-4.09 (t, J = 6.5 Hz, CH_2OAr, 2H), 4.24-4.27 (m, CH_2O, 2H), 4.35-4.40 (m, CH_2O, 2H), 5.26 (s, C*H*O, 1H), 5.54-5.56 (d, J = 6.5 Hz, ArCH_2O, 2H), 6.21-6.26 (m, C*H*O, 1H), 6.91-6.93 (d, J_1 = 2.5 Hz, J_2= 9 Hz, Ar*H*, 1H), 7.18-7.19 (d, J= 2.5 Hz, Ar*H*, 1H), 7.55-7.58 (d, J = 9 Hz, Ar*H*, 1H),8.17-8.20 (d, J = 9 Hz, Ar*H*, 1H); ESI MS calcd for $C_{34}H_{52}N_4O_{11}P$,723.77($M+H^+$), found 723.34, $C_{34}H_{55}N_5O_{11}P$, 740.82 ($M+NH_4^+$), found 740.36.

Compound 8c

Yield 66%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.86-0.89 (m, C*H*$_3$, 3H), 1.26-1.35 (m, C*H*$_2$, 22H), 1.43-1.47 (m, C*H*$_2$, 2H), 1.80-1.83 (m, C*H*$_2$, 2H), 1.87 (s, C*H*$_3$, 3H), 2.49-2.58 (m, 2H, C*H*$_2$O), 2.85-2.86 (m, C*H*$_2$CN, 2H), 3.29 (b, O*H*, 1H), 3.88 (m, C*H*$_2$O, 2H), 4.06-4.09 (t, *J* = 6.5 Hz, C*H*$_2$OAr, 2H), 4.24-4.27 (m, C*H*$_2$O, 2H), 4.35-4.40 (m, C*H*$_2$O, 2H), 5.26 (s, C*H*O, 1H), 5.55-5.56 (d, *J* = 6.5 Hz, ArC*H*$_2$O, 2H), 6.21-6.26 (m, C*H*O, 1H), 6.91-6.93 (d, J_1 = 2.5 Hz, J_2= 9 Hz, Ar*H*, 1H), 7.18-7.19 (d, *J*= 2.5 Hz, Ar*H*, 1H), 7.55-7.58 (d, *J* = 9 Hz, Ar*H*, 1H),8.17-8.20 (d, *J* = 9 Hz, Ar*H*, 1H); ESI MS calcd for $C_{36}H_{56}N_4O_{11}P$,751.82 ($M+H^+$), found 751.37; $C_{36}H_{59}N_5O_{11}P$, 768.85 ($M+NH_4^+$), found 768.39.

Compound 5d

Yield 61%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.86-0.89 (m C*H*$_3$, 3H), 1.26-1.33 (m, C*H*$_2$+ C*H*$_3$, 21H), 1.40-1.43 (m, C*H*$_2$, 2H), 1.74-1.77 (m, C*H*$_2$, 2H), 1.85 (s, C*H*$_3$, 3H), 2.27-2.29 (m, C*H*$_2$, 2H), 3.07-3.11 (q, *J* = 7.5 Hz, C*H*$_2$, 6H), 3.79-3.80 (m, C*H*O, 2H), 4.00-4.03 (m, C*H*, 2H), 4.13 (s, C*H*, 1H), 5.06 (s, C*H*O, 1H),5.31-5.32 (s, ArC*H*$_2$O, 2H), 6.16-6.21 (m, C*H*, 1H), 6.77-6.79 (m, 1H, Ar*H*), 7.30-7.33 (m, 1H, Ar*H*), 7.64-7.65 (m, 1H, Ar*H*), 8.05-8.07 (m, 1H, Ar*H*).ESI MS calcd for $C_{27}H_{40}N_3O_{11}P$,613.59(M-Et_3N+H^+), found 614.25; $C_{27}H_{44}N_4O_{11}P$,631.63 (M-Et_3N+NH_4^+), found 631.28.

Compound 6d

Yield 57%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.86-0.89 (m C*H*$_3$, 3H), 1.26-1.33 (m, C*H*$_2$+ C*H*$_3$, 25H), 1.40-1.43 (m, C*H*$_2$, 2H), 1.74-1.77 (m, C*H*$_2$, 2H), 1.85 (s, C*H*$_3$, 3H), 2.27-2.29 (m, C*H*$_2$, 2H), 3.07-3.11 (q, *J* = 7.5 Hz, C*H*$_2$, 6H), 3.79-3.89 (m, C*H*O, 2H), 4.00-4.03 (m, C*H*, 2H), 4.13 (s, C*H*, 1H), 5.06 (s, C*H*O, 1H),5.31-5.32 (s, ArC*H*$_2$O, 2H), 6.20-6.22 (m, C*H*, 1H), 6.77-6.79 (m, 1H, Ar*H*), 7.30-7.33 (m, 1H, Ar*H*), 7.64-7.65 (m, 1H, Ar*H*), 8.05-8.07 (m, 1H, Ar*H*).ESI MS calcd for $C_{29}H_{45}N_3O_{11}P$,642.65 (M-Et_3N+H^+), found 642.28; $C_{29}H_{47}N_4O_{11}P$,658.67 (M-Et_3N+NH_4^+), found 659.31.

Compound 7d

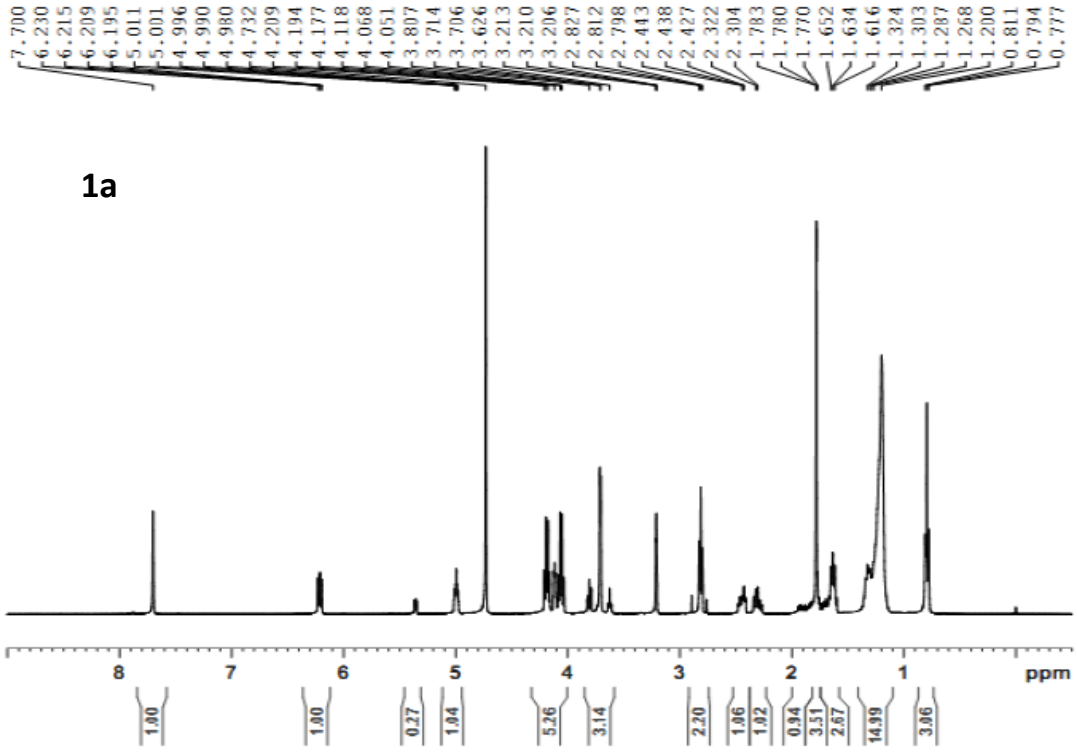
Yield 52%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.86-0.89 (m C*H*$_3$, 3H), 1.26-1.33 (m, C*H*$_2$+ C*H*$_3$, 27H), 1.39-1.45 (m, C*H*$_2$, 2H), 1.74-1.79 (m, C*H*$_2$, 2H),

1.85 (s, CH_3, 3H), 2.27-2.49 (m, CH_2, 2H), 3.09-3.13 (q, J = 7.5 Hz, CH_2, 6H), 3.79-3.93 (m, CHO, 2H), 4.02-4.16 (m, CH, 2H), 4.13 (s, CH, 1H), 5.10 (s, CHO, 1H),5.31-5.32 (s, ArCH_2O, 2H), 6.22-6.25 (m, CH, 1H), 6.77-6.79 (m, 1H, ArH), 7.35-7.37 (m, 1H, ArH), 7.70-7.71 (m, 1H, ArH), 8.05-8.07 (m, 1H, ArH). ESI MS calcd for $C_{31}H_{49}N_3O_{11}P$,670.71(M-Et_3N+H^+), found 670.31; $C_{31}H_{51}N_4O_{11}P$ 686.73 (M-Et_3N+NH_4^+), found 687.34.

Compound 8d

Yield 54%, yellow solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.86-0.89 (m, CH_3, 3H), 1.26-1.33 (m, CH_2+ CH_3, 31H), 1.39-1.45 (m, CH_2, 2H), 1.73-1.79 (m, CH_2, 2H), 1.86 (s, CH_3, 3H), 2.27-2.49 (m, CH_2, 2H), 3.07-3.11 (q, J = 7.5 Hz, CH_2, 6H), 3.79-3.93 (m, CHO, 2H), 4.00-4.13 (m, CH, 2H), 4.13 (s, CH, 1H), 5.07 (s, CHO, 1H),5.31-5.32 (s, ArCH_2O, 2H), 6.20-6.23 (m, CH, 1H), 6.77-6.79 (m, 1H, ArH), 7.30-7.34 (m, 1H, ArH), 7.65-7.66 (m, 1H, ArH), 8.06-8.08 (m, 1H, ArH). ESI MS calcd for $C_{33}H_{53}N_3O_{11}P$,698.76(M-Et_3N+H^+), found 698.34, $C_{33}H_{55}N_4O_{11}P$,714.78 (M-Et_3N+NH_4^+), found 715.37.

7.1. NMR spectra

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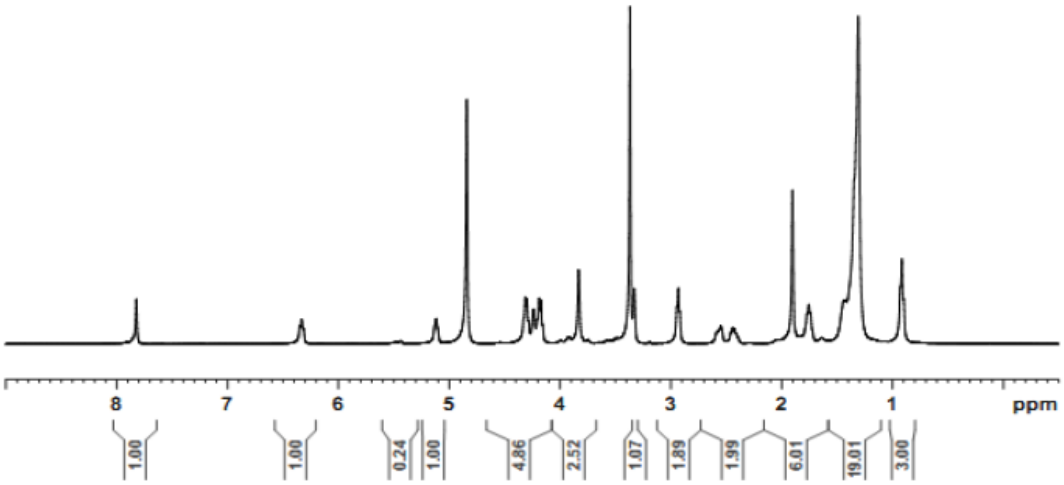
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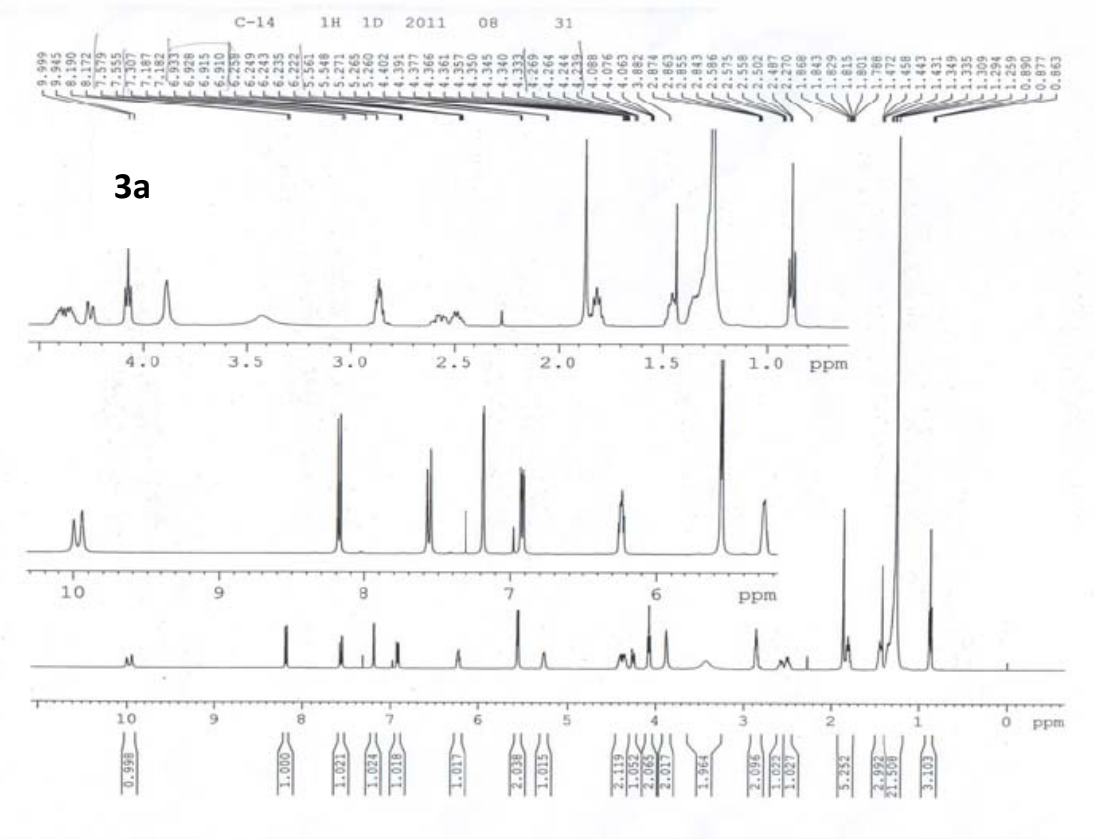
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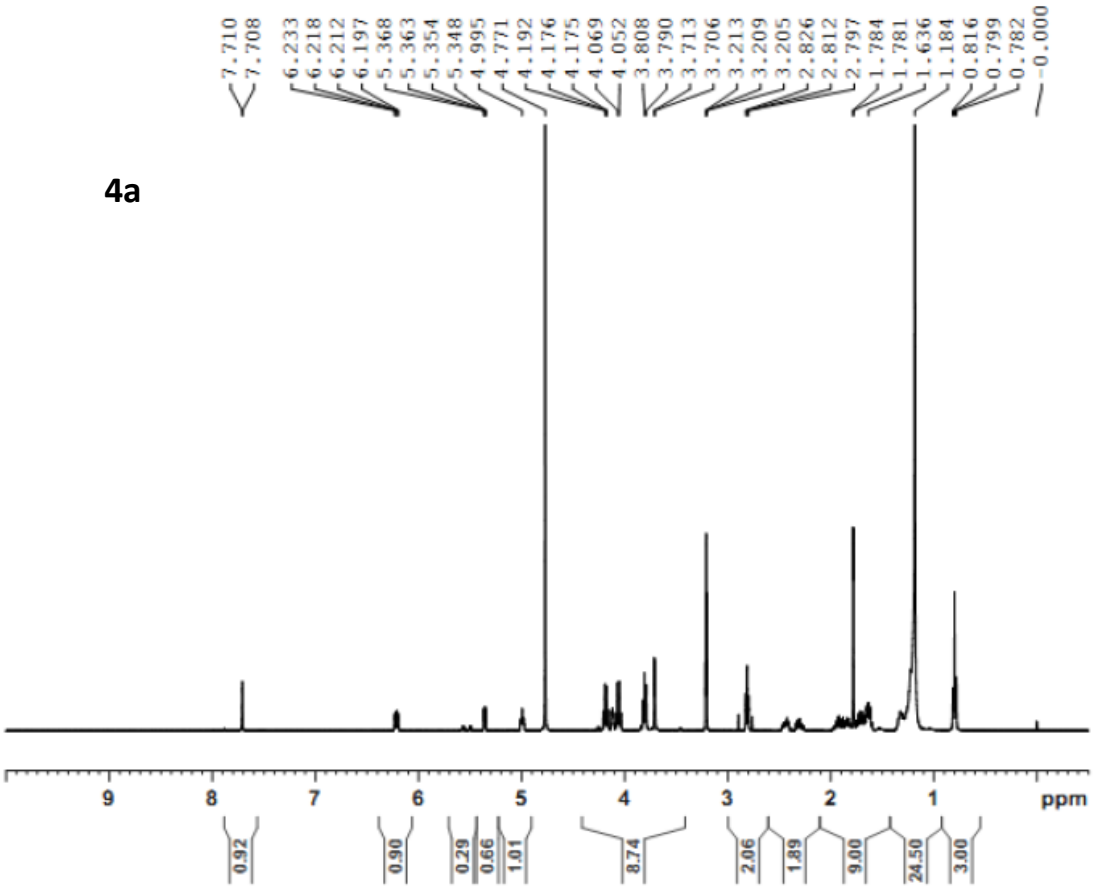
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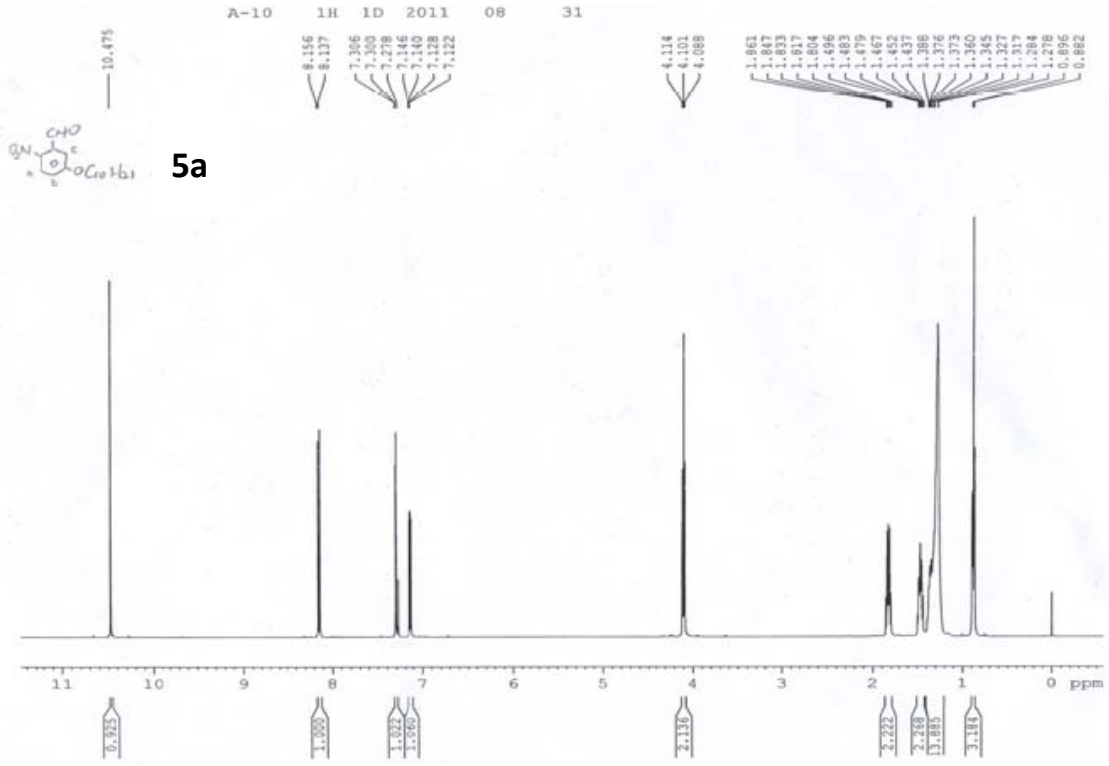
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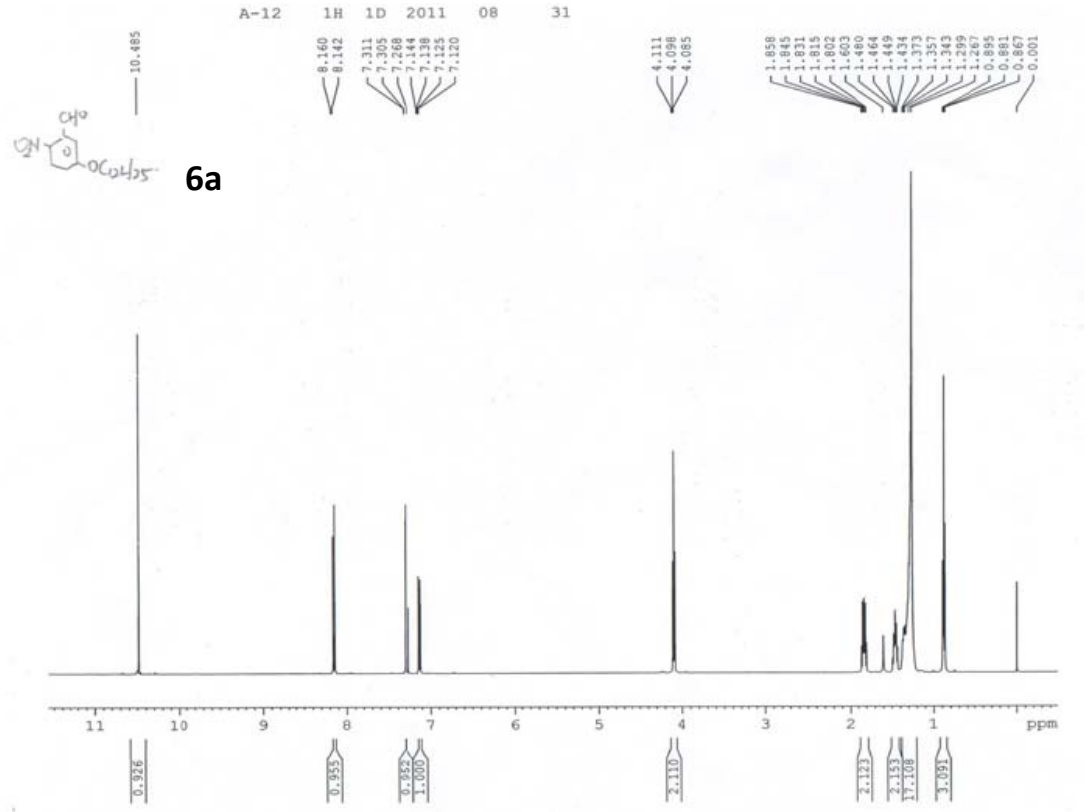
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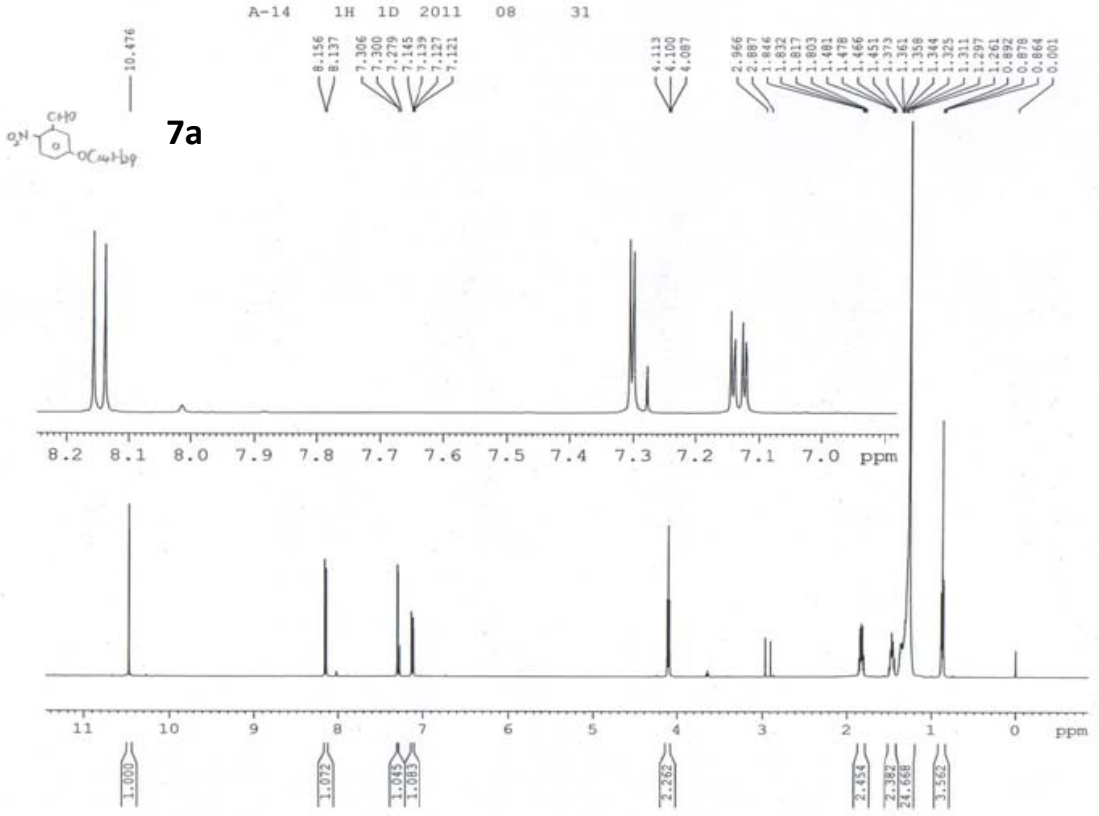
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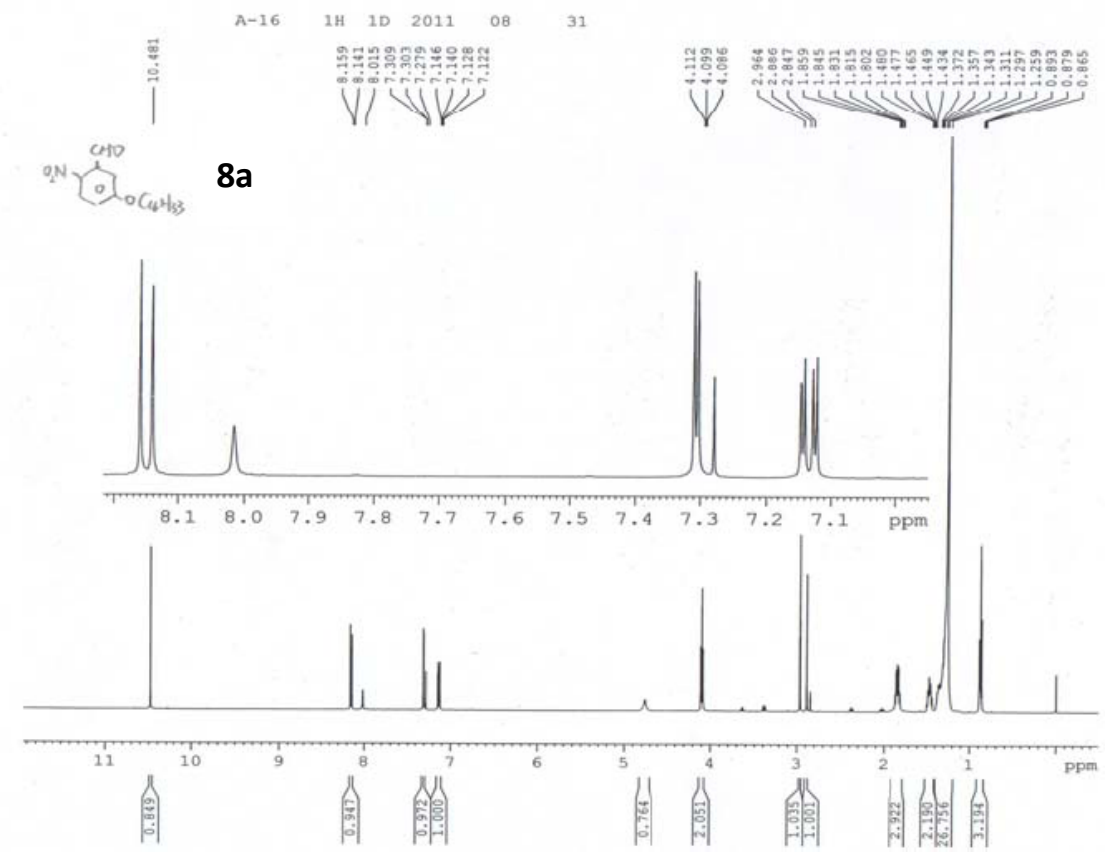
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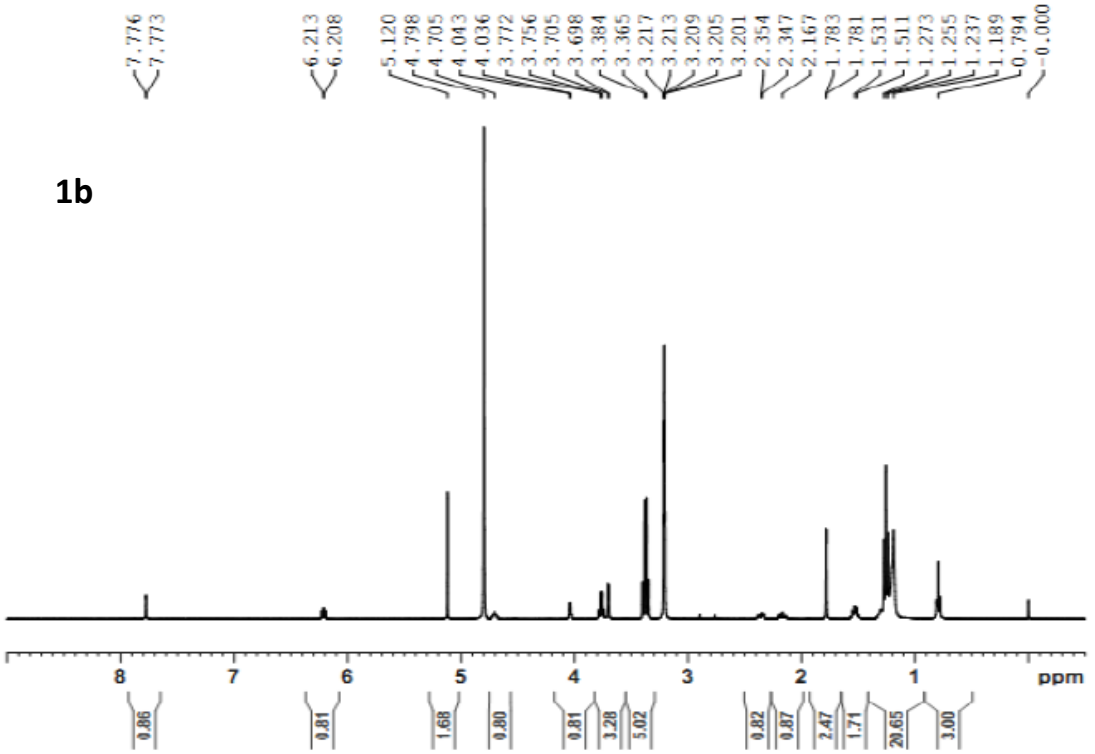
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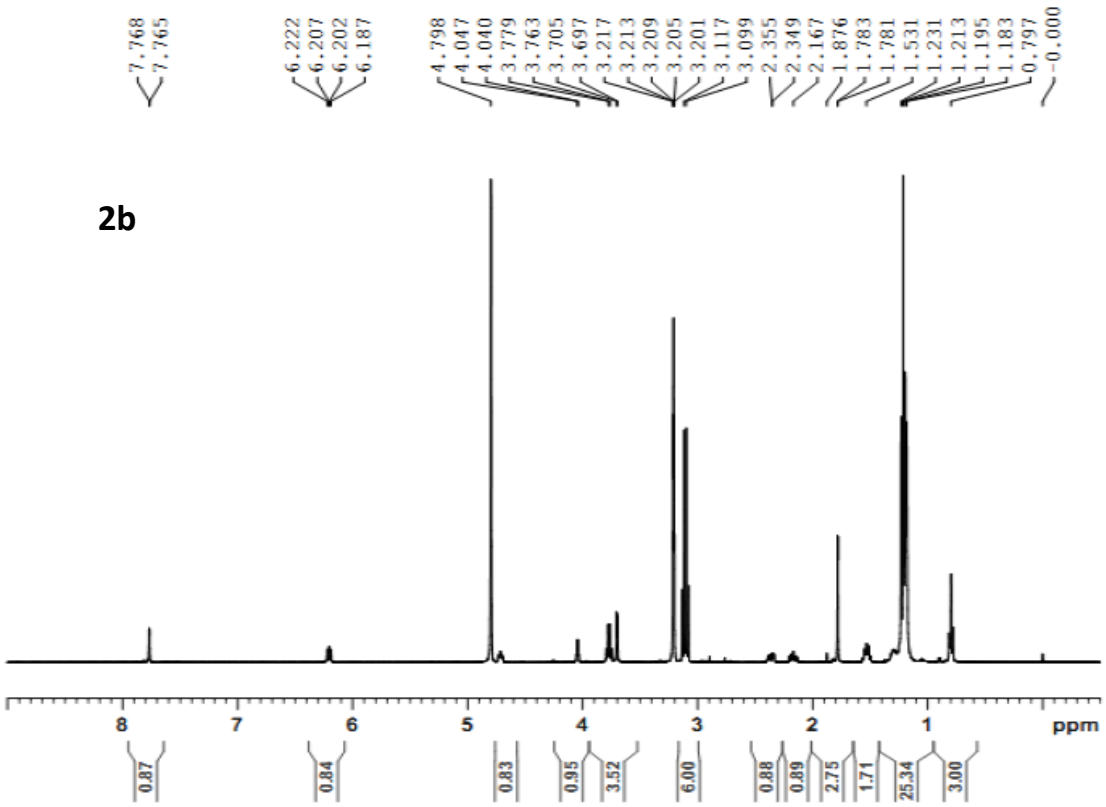

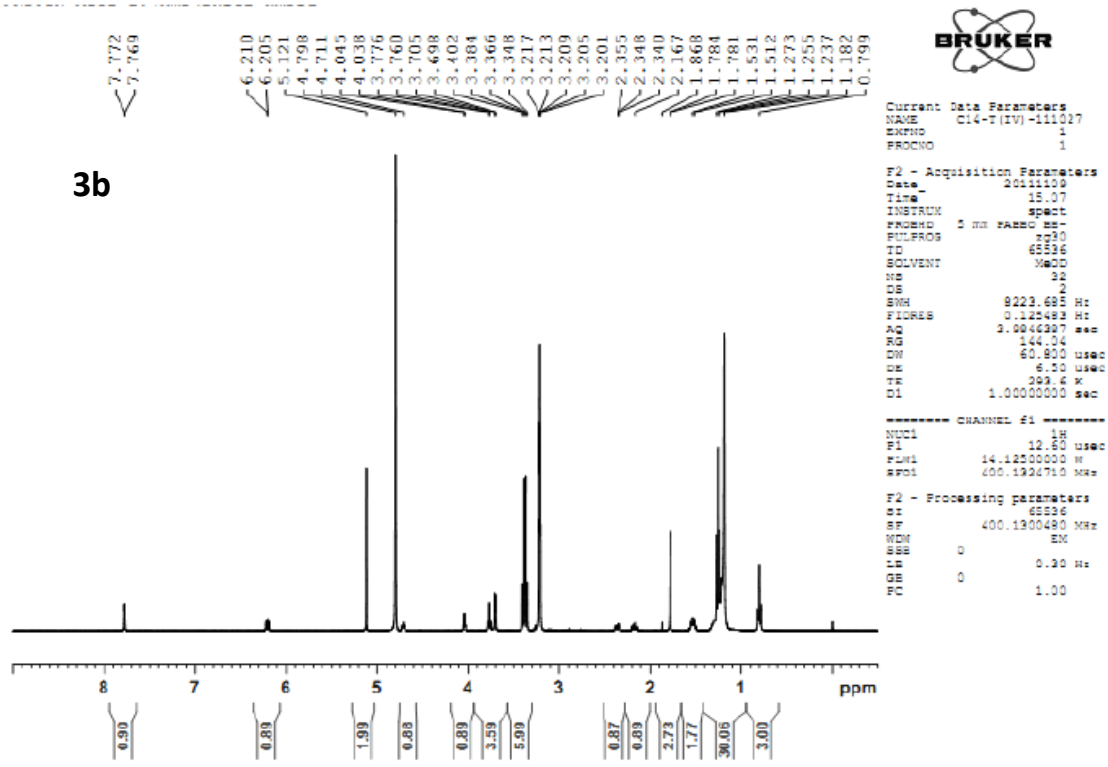

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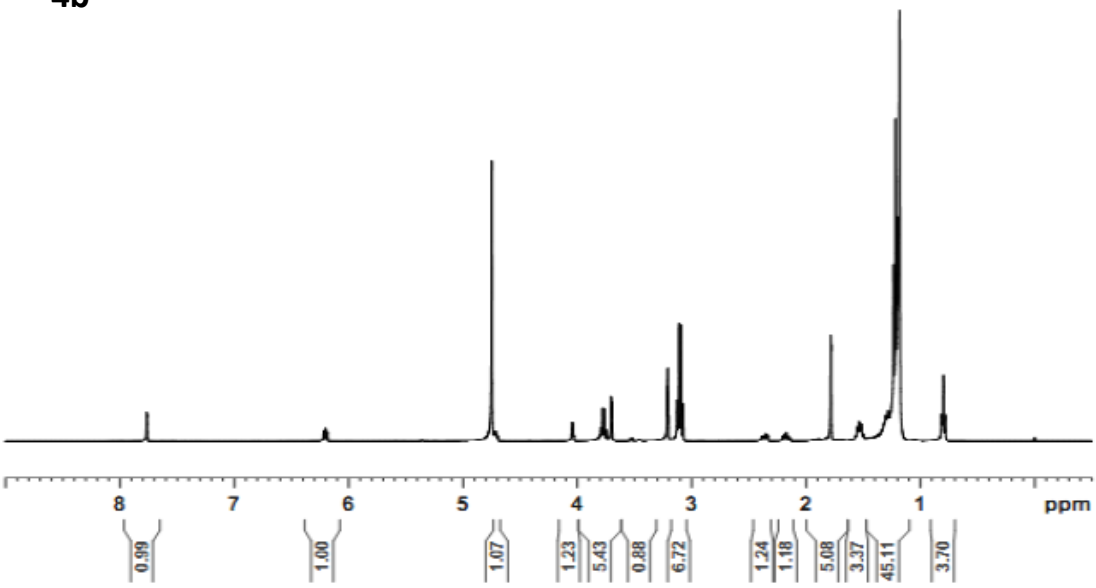
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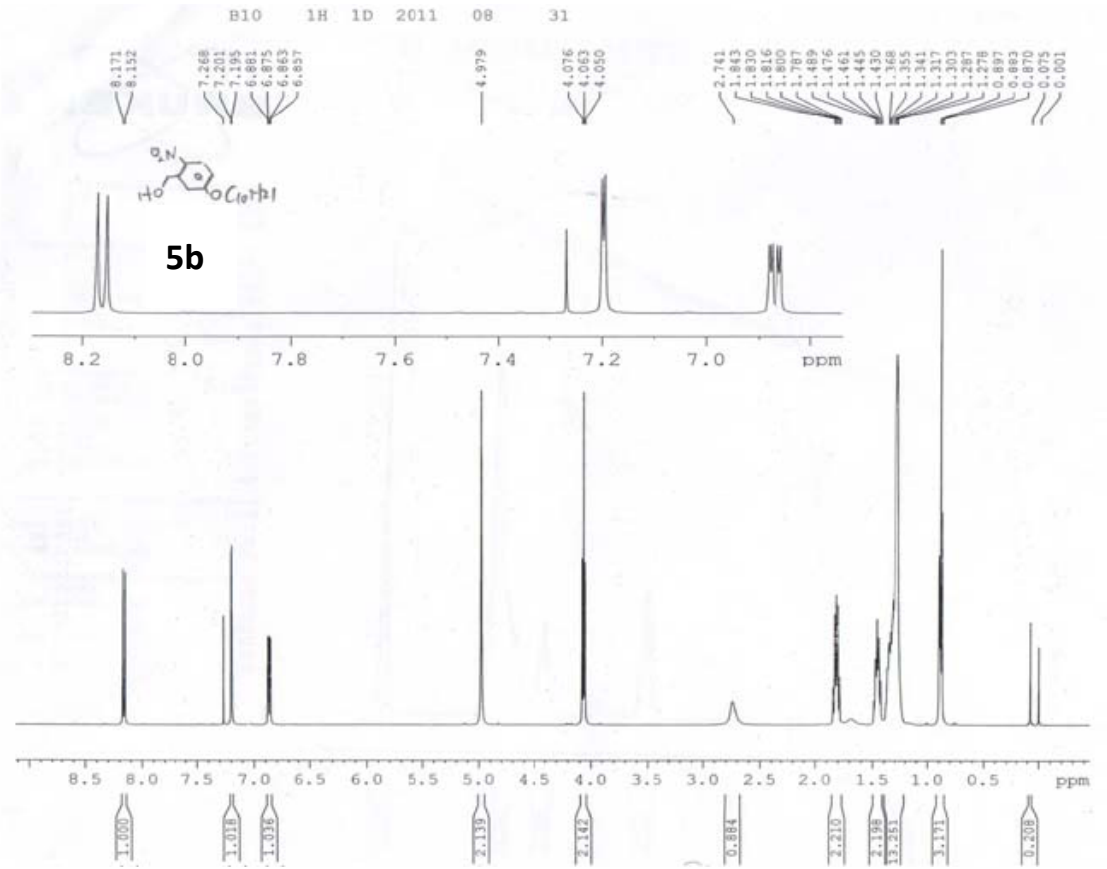
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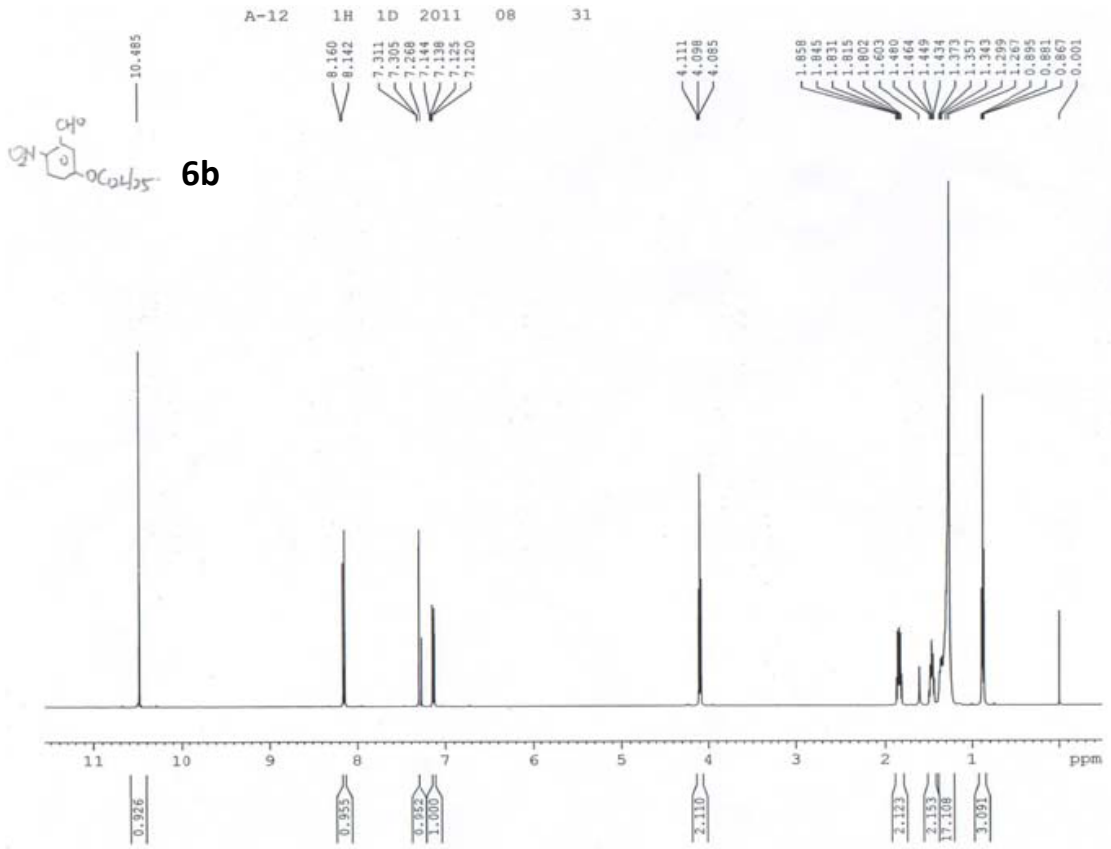
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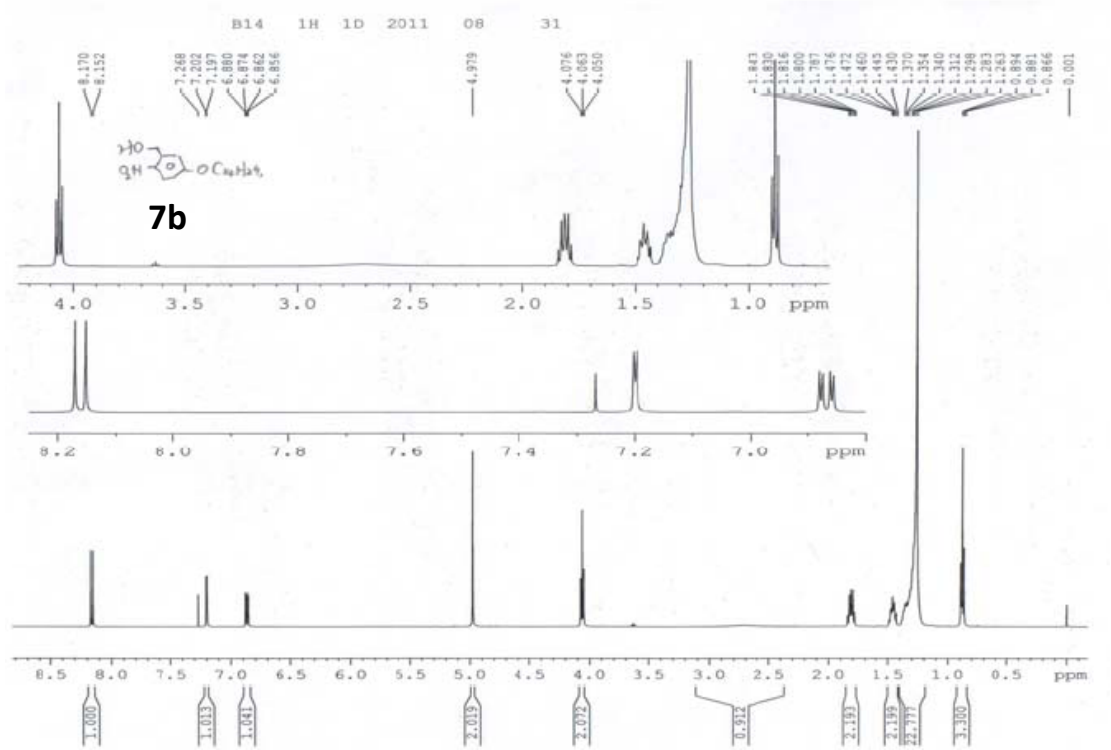
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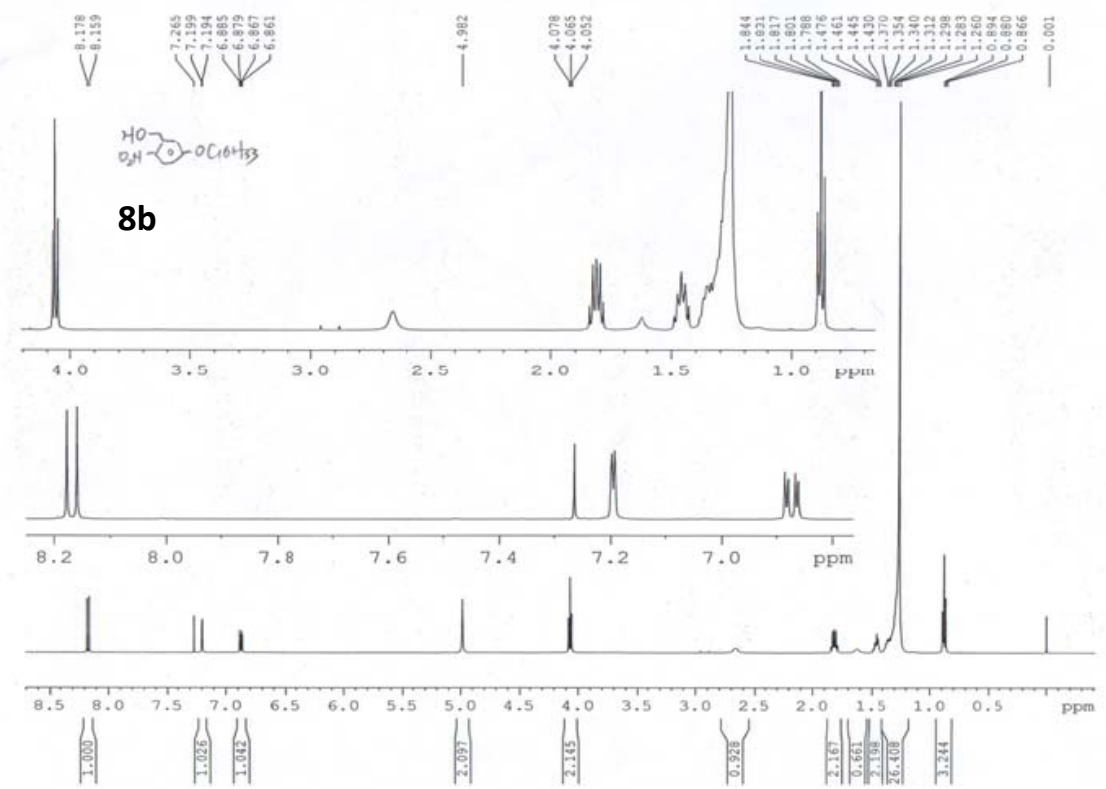
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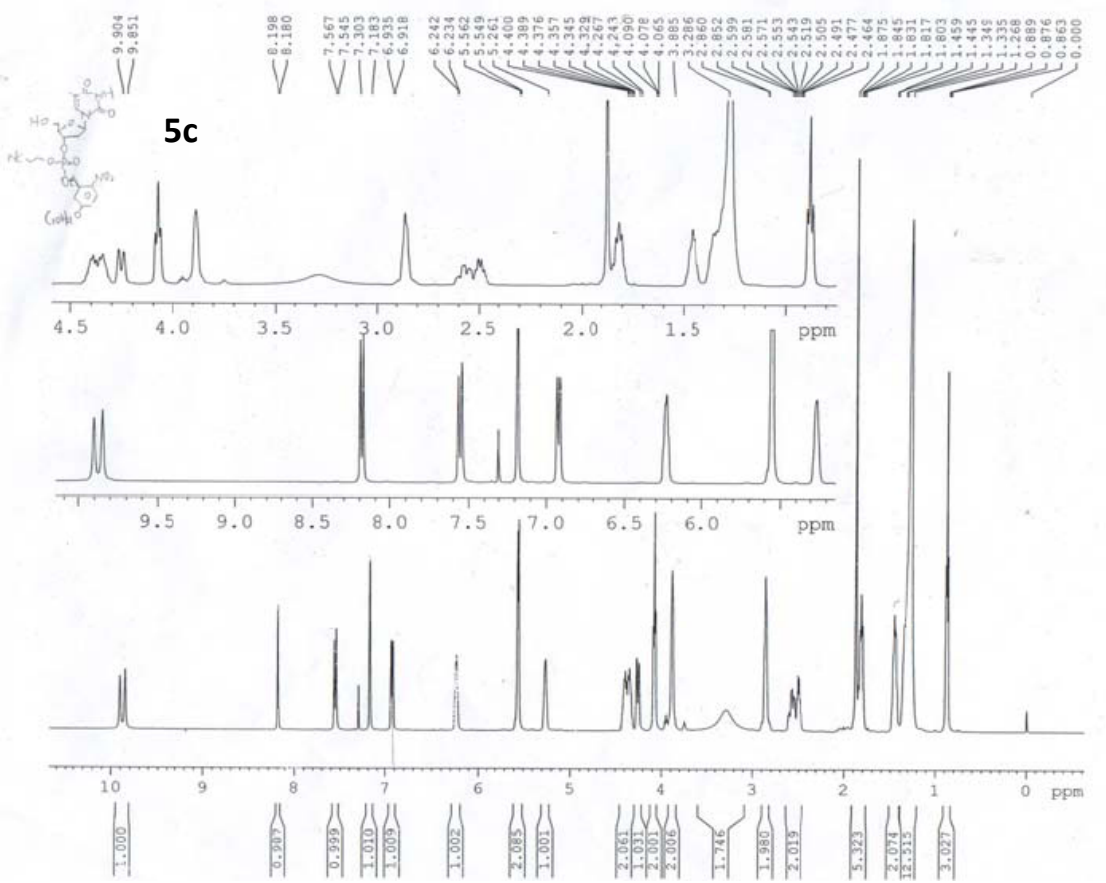
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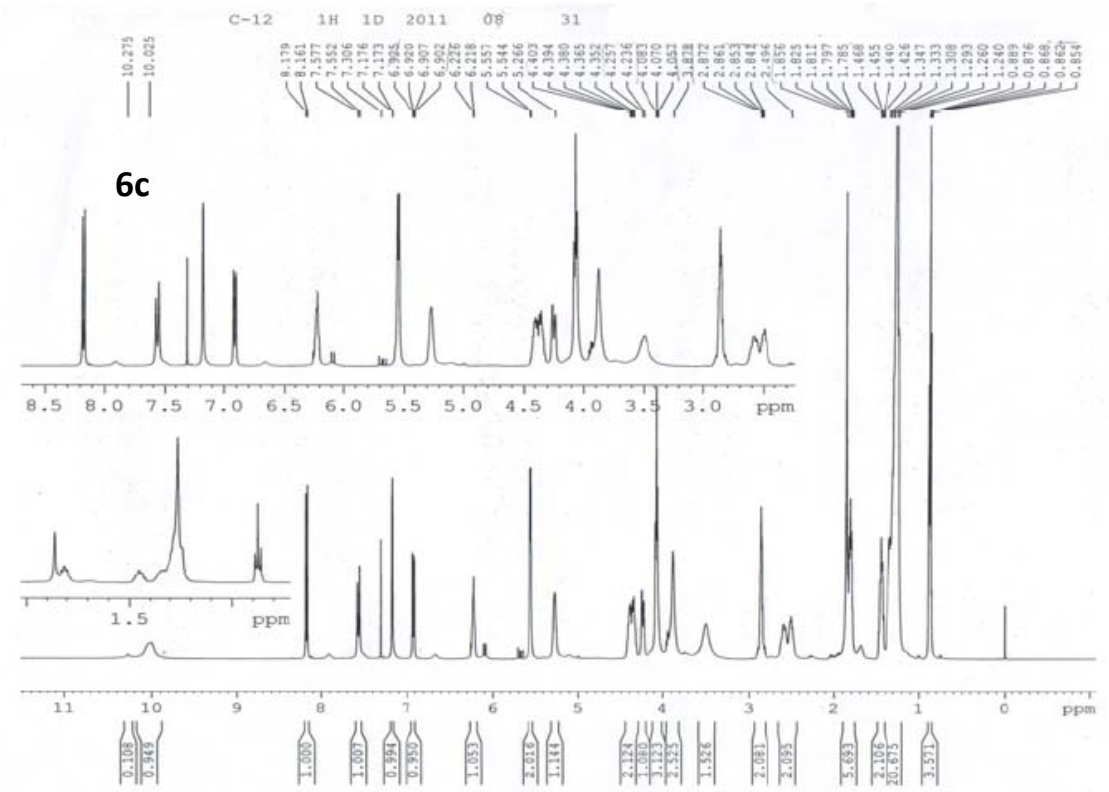
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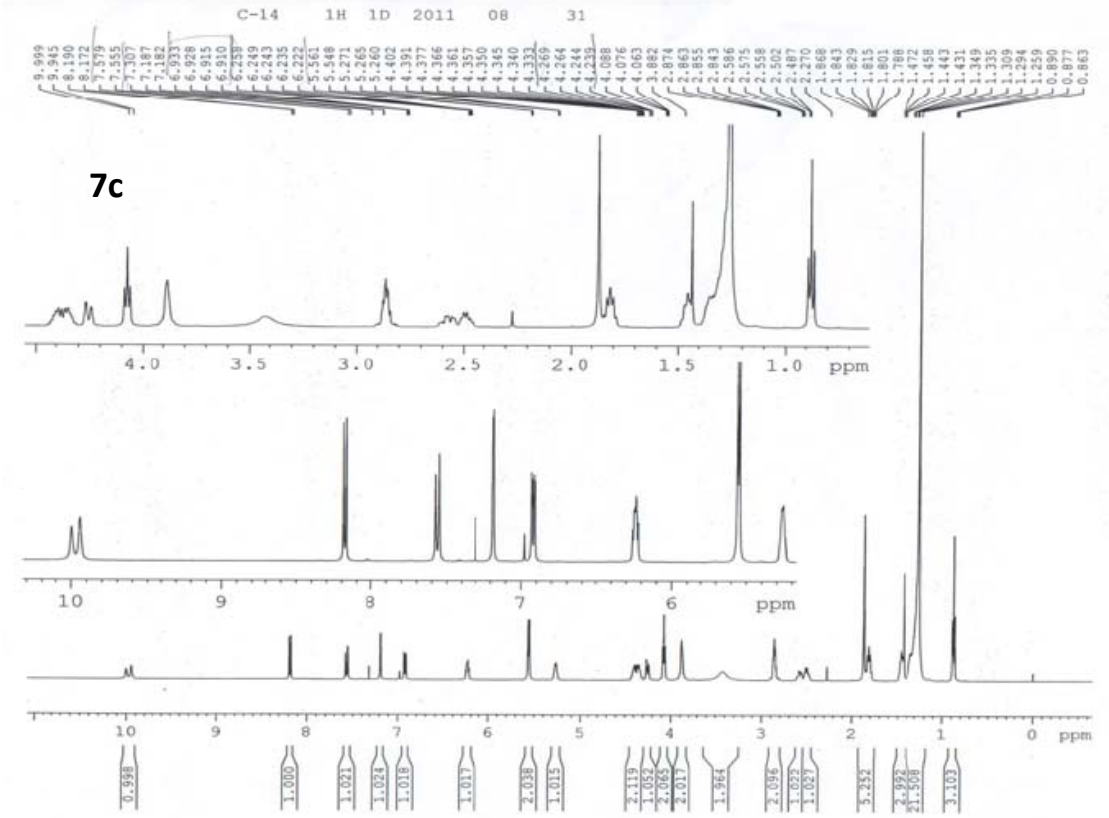
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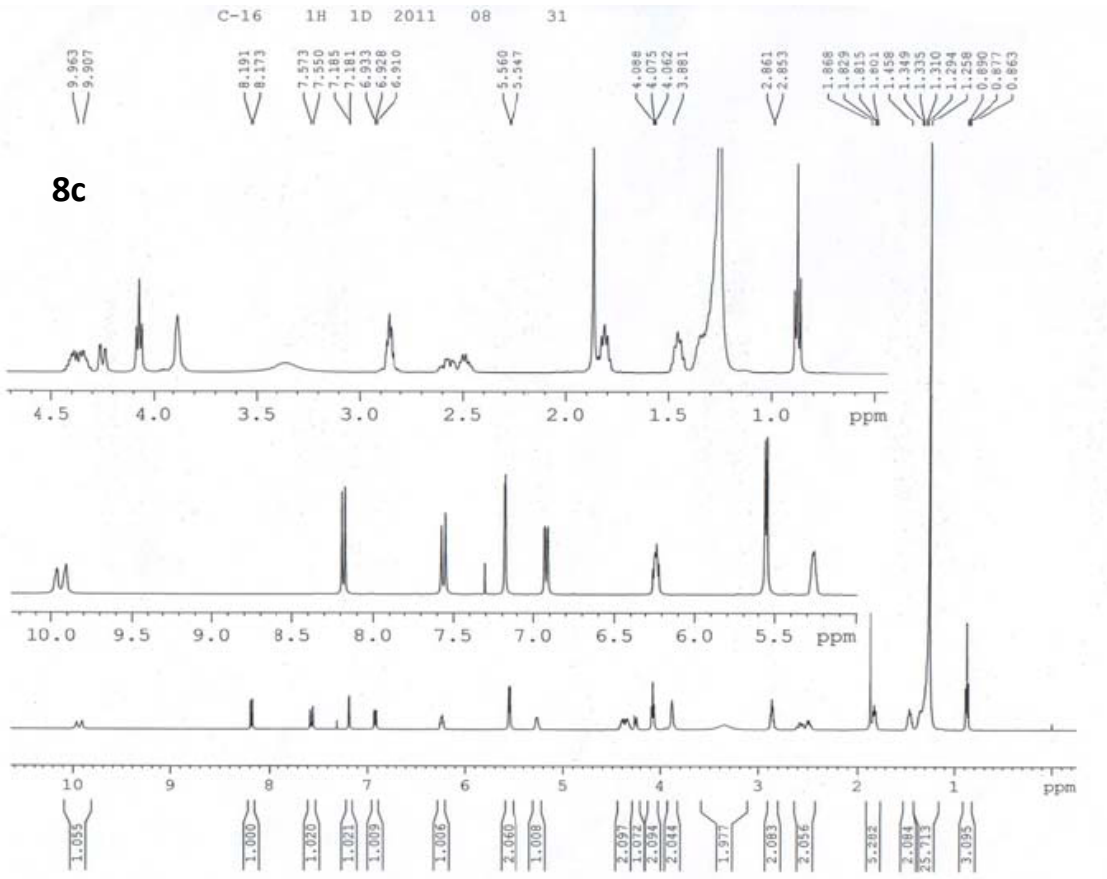
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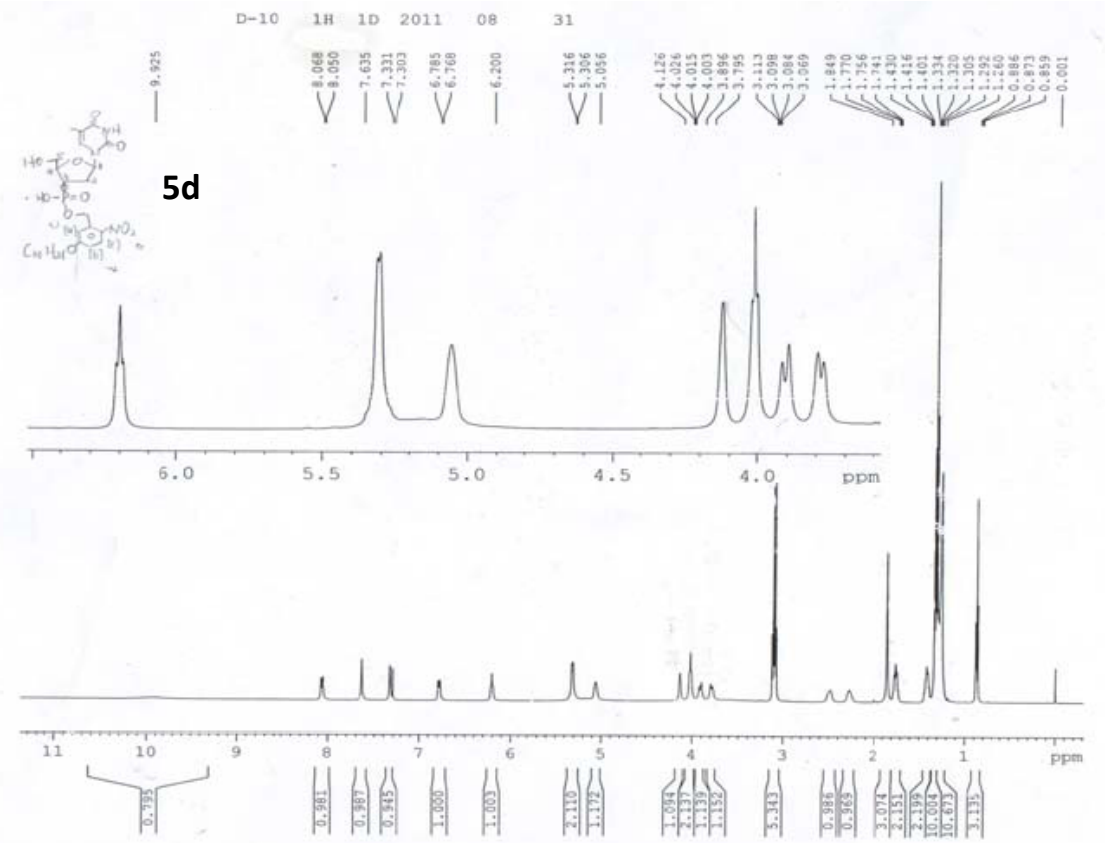
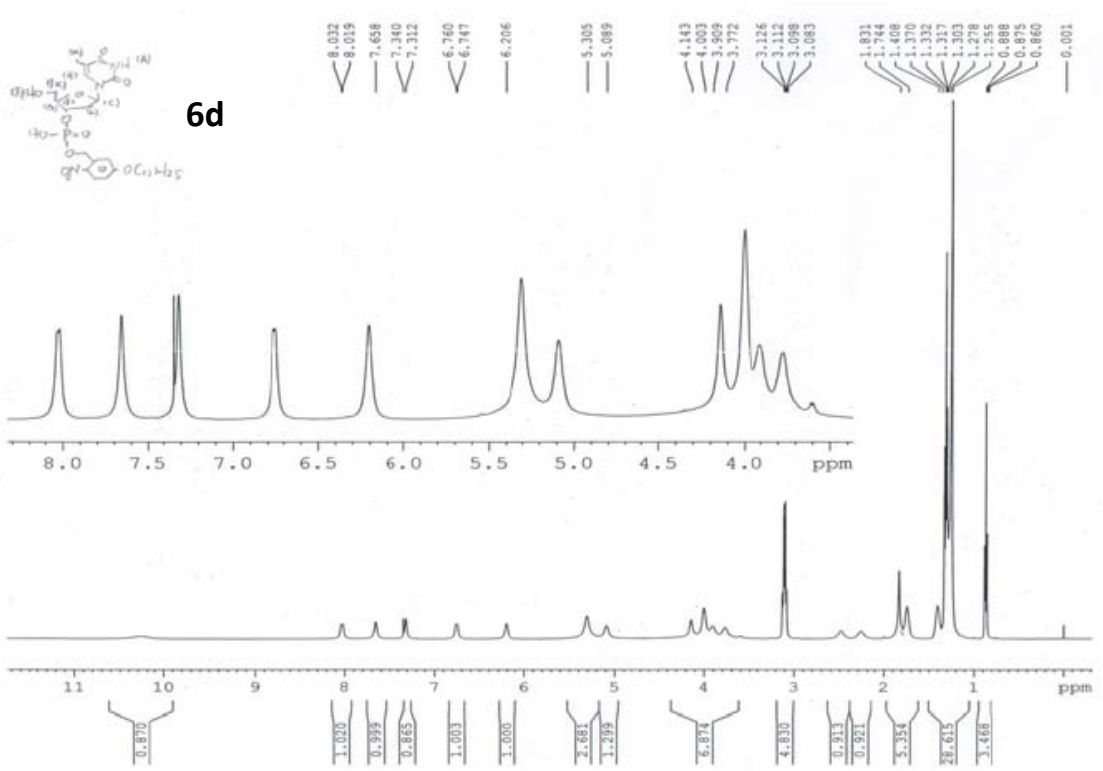
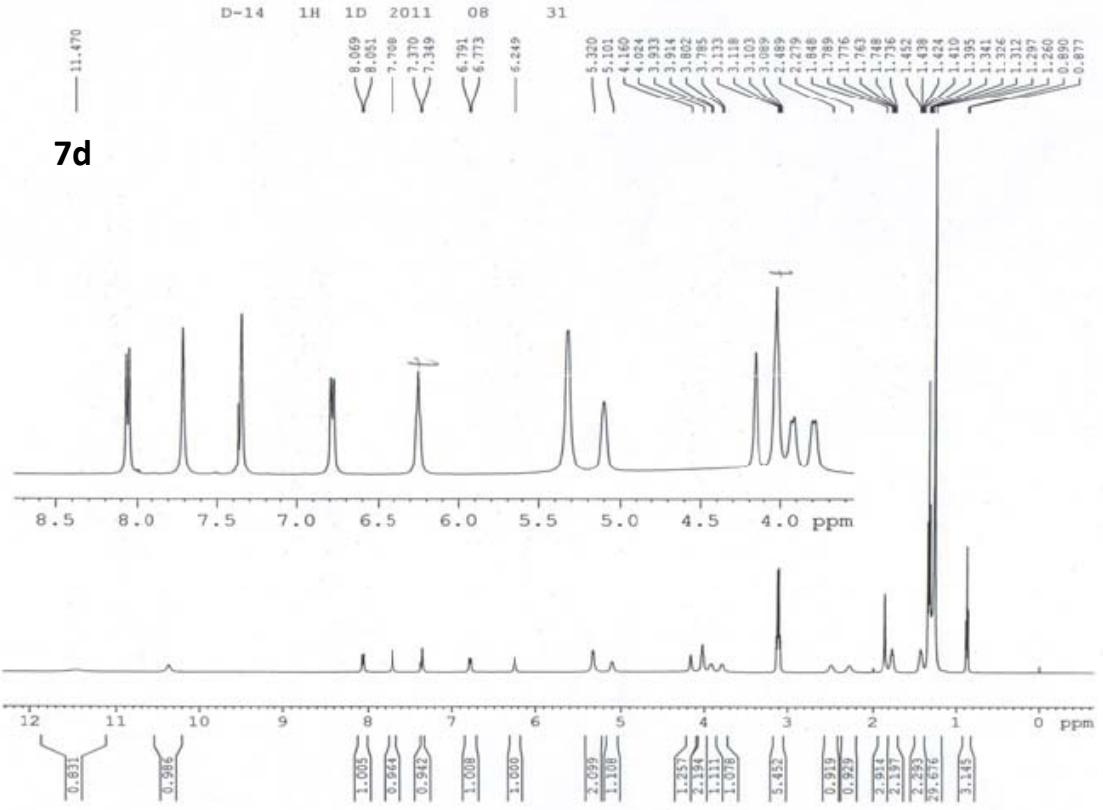
8c
ppm

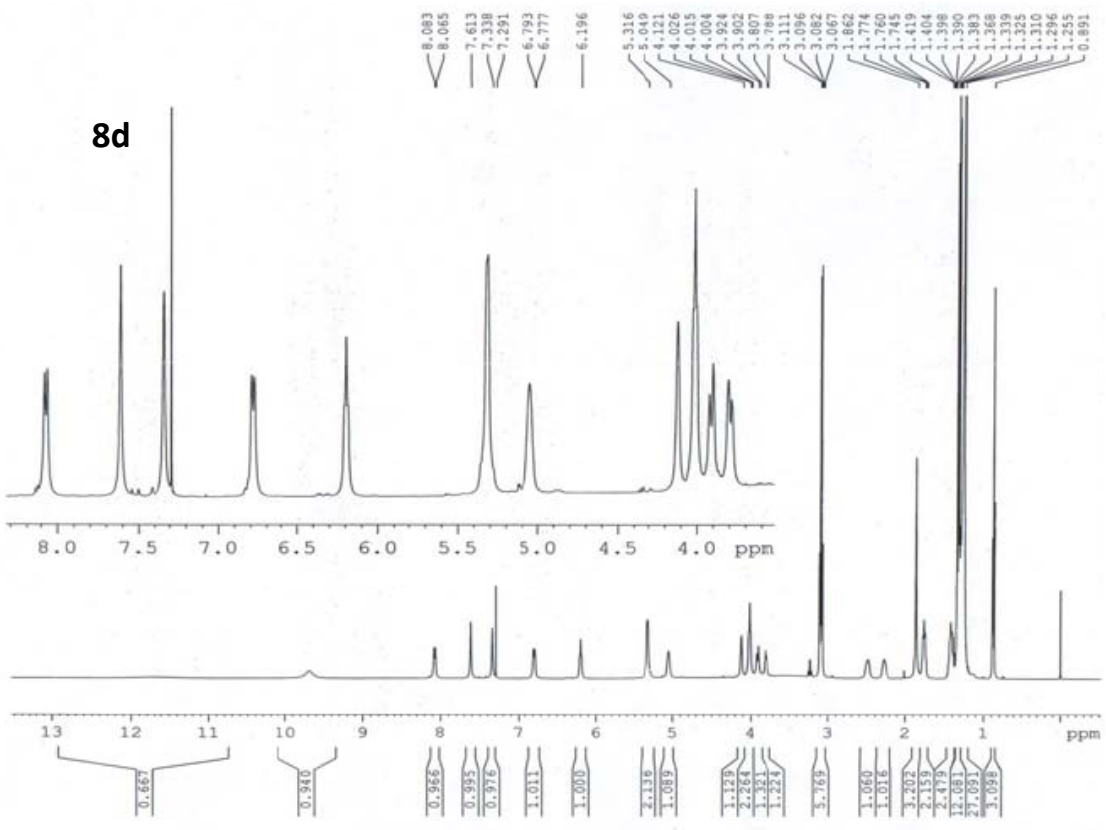

D-10 1H 1D 2011 08 31
5d
ppm



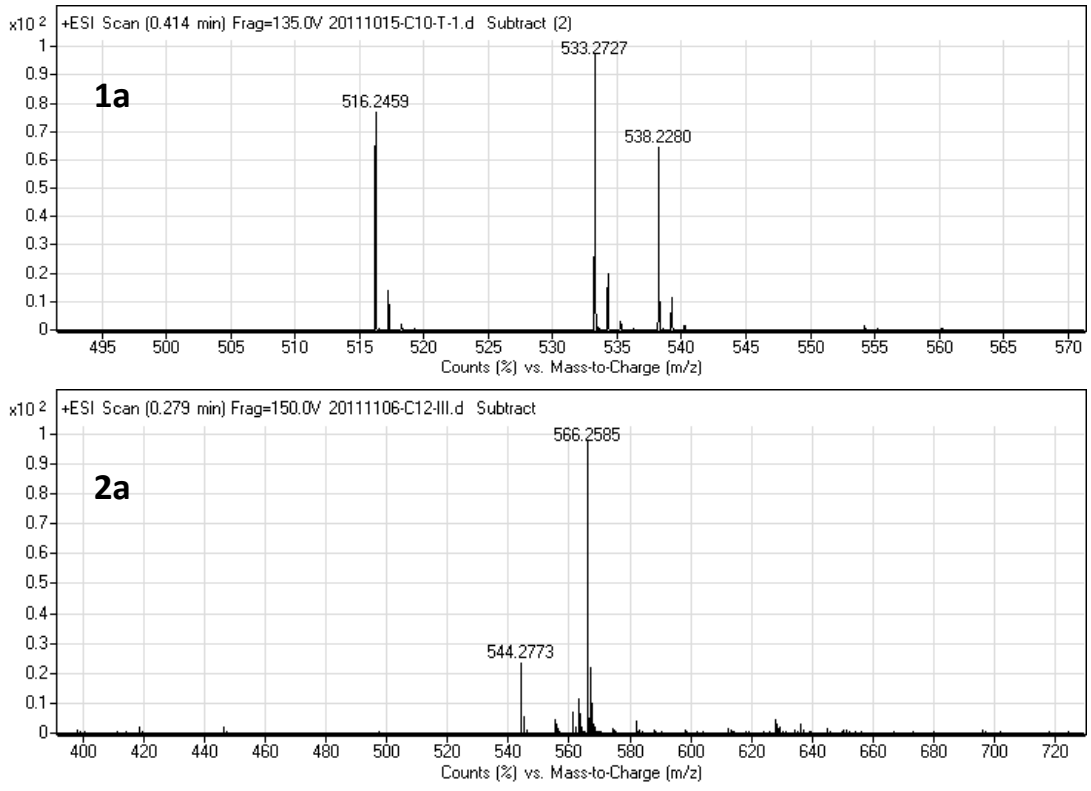
6d

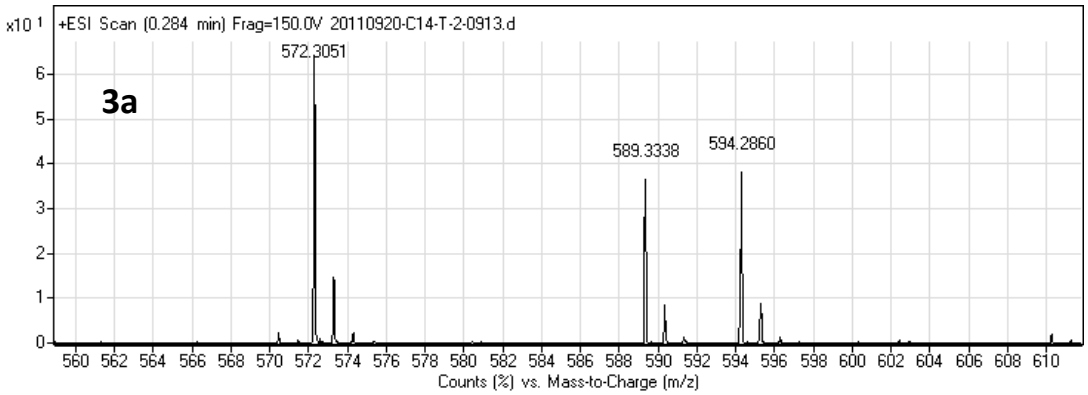


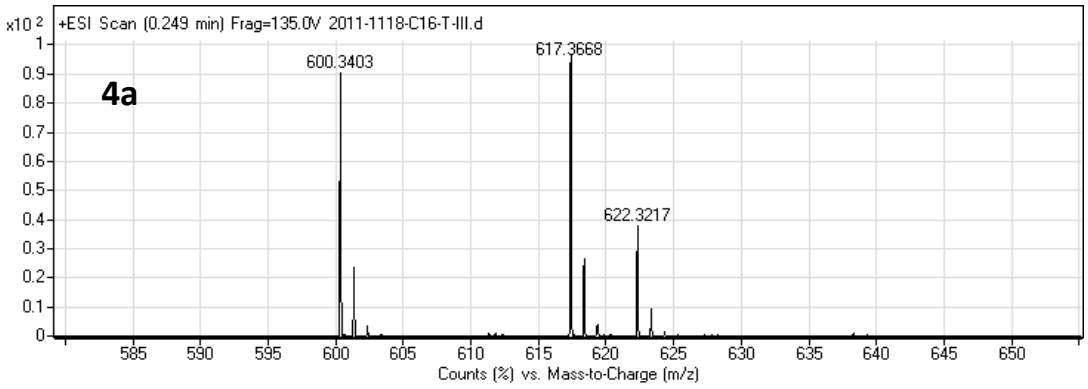
7d

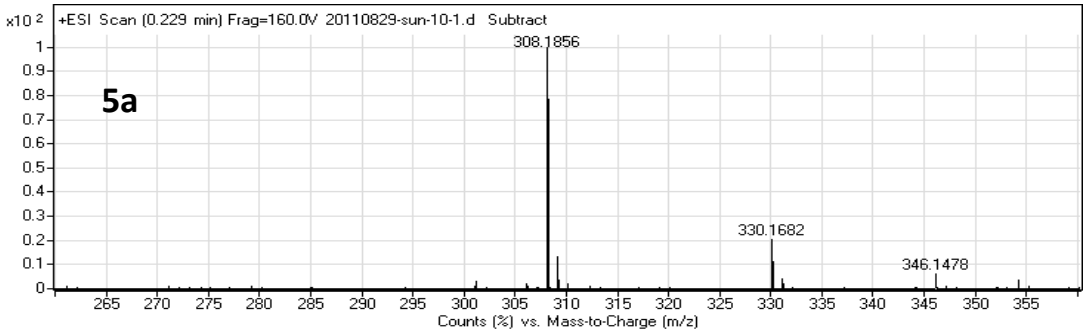


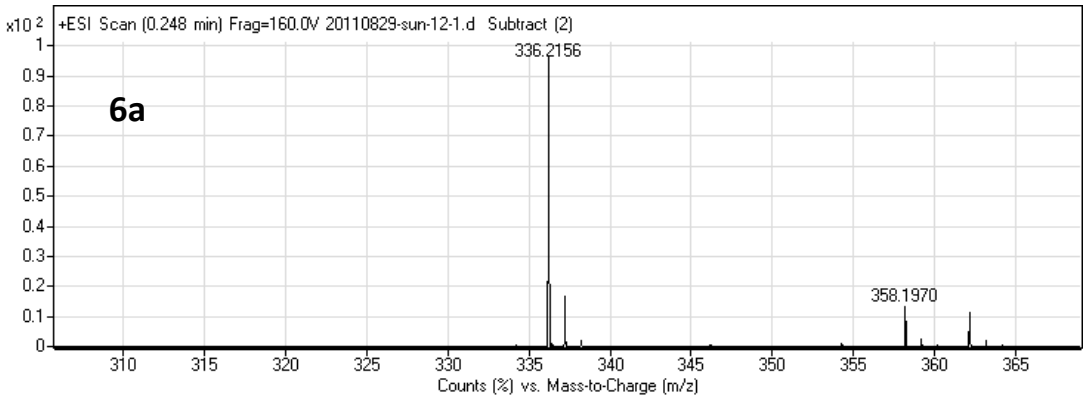
7.2. MS spectra

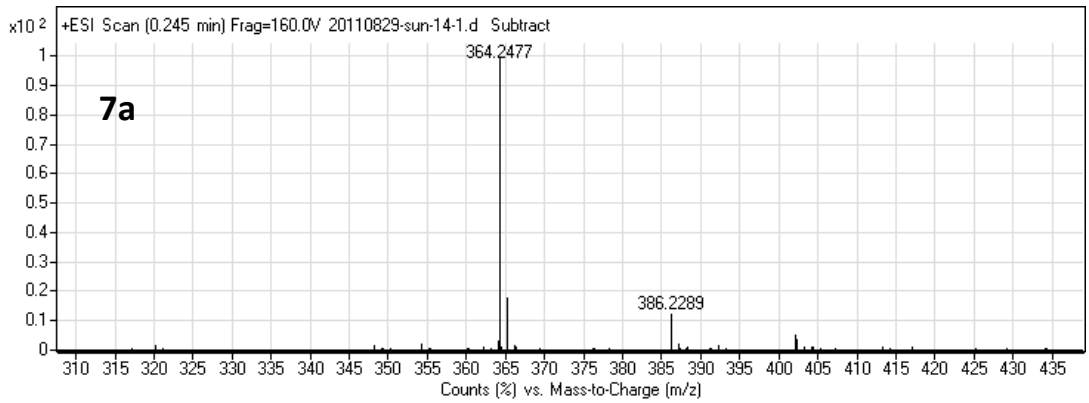


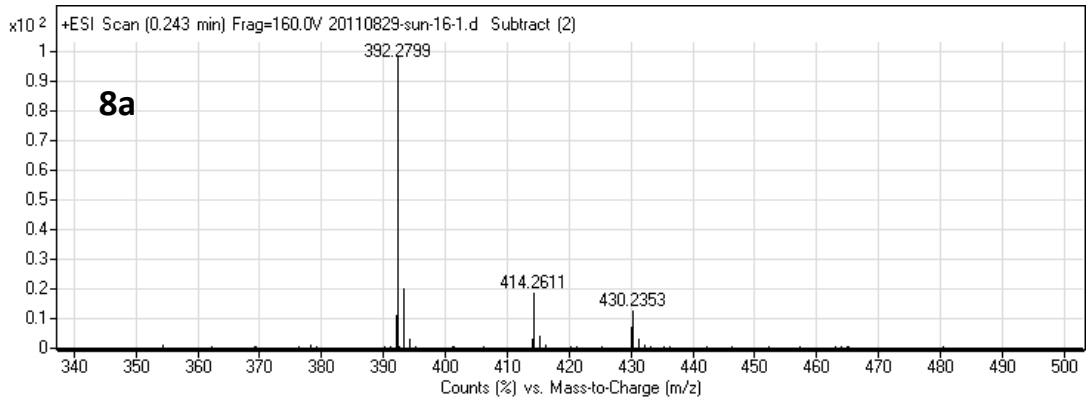

x10 1
+ESI Scan (0.284 min) Frag=150.0V 20110920-C14-T-2-0913.d
572.3051
3a
589.3338
594.2860
Counts (%) vs. Mass-to-Charge (m/z)

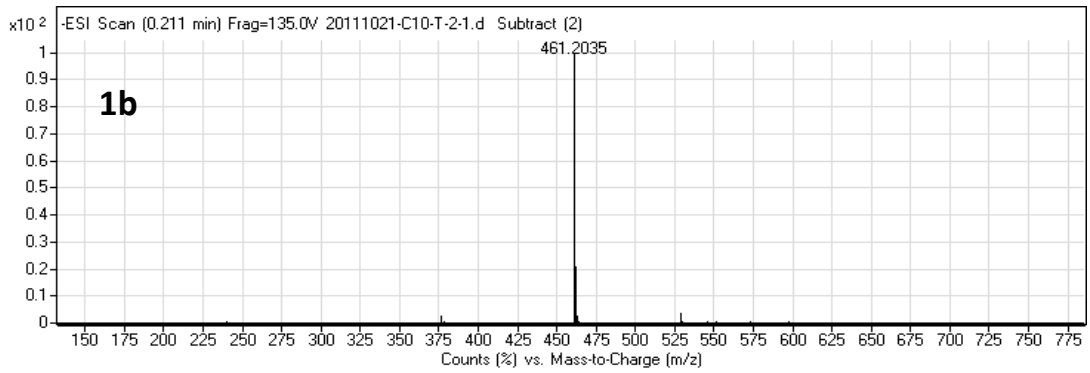

x10 2
+ESI Scan (0.249 min) Frag=135.0V 2011-1118-C16-T-III.d
600.3403
617.3668
4a
622.3217
Counts (%) vs. Mass-to-Charge (m/z)

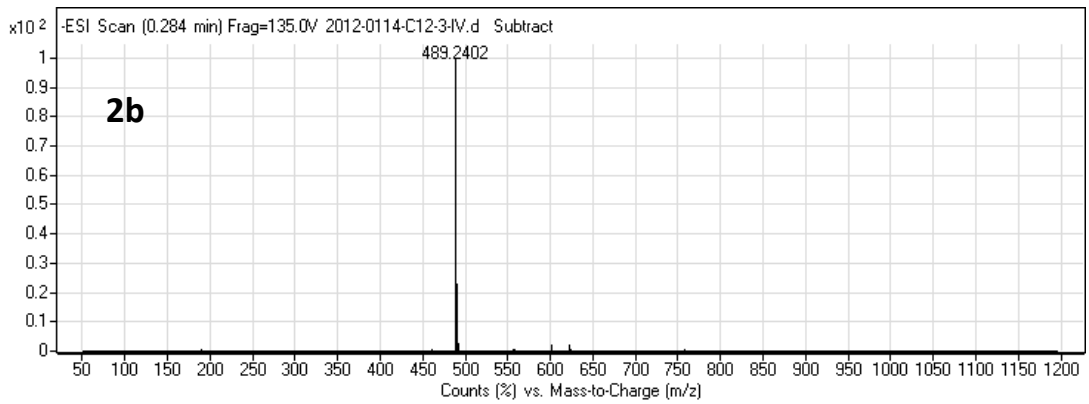

x10 2
+ESI Scan (0.229 min) Frag=160.0V 20110829-sun-10-1.d Subtract
308.1856
5a
330.1682
346.1478
Counts (%) vs. Mass-to-Charge (m/z)

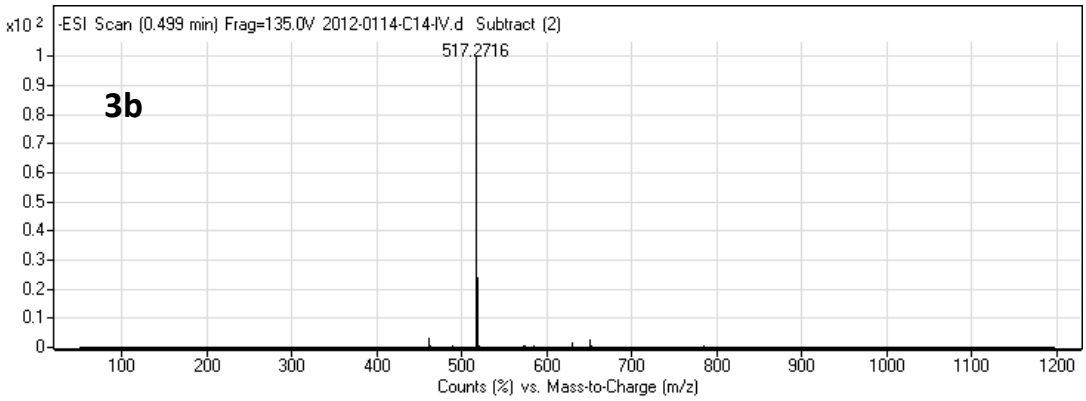

x10 2
+ESI Scan (0.248 min) Frag=160.0V 20110829-sun-12-1.d Subtract (2)
336.2156
6a
358.1970
Counts (%) vs. Mass-to-Charge (m/z)

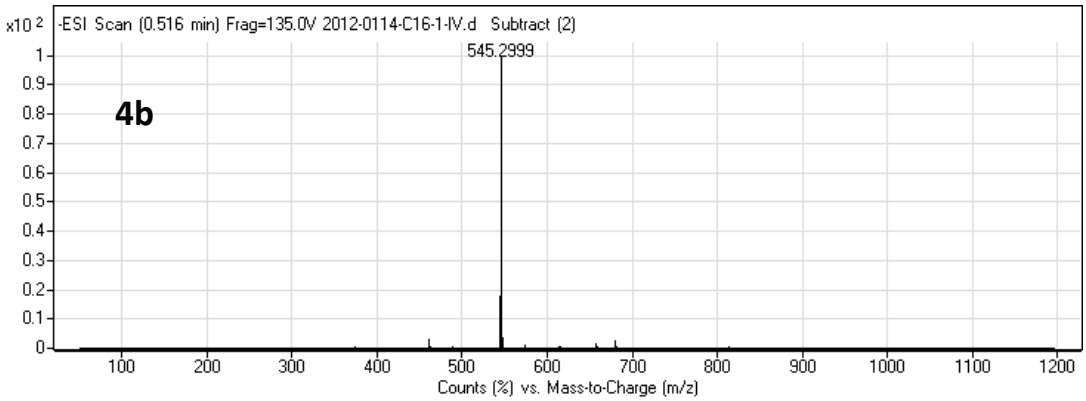

x10 2
+ESI Scan (0.245 min) Frag=160.0V 20110829-sun-14-1.d Subtract
364.2477
386.2289
7a
Counts (%) vs. Mass-to-Charge (m/z)

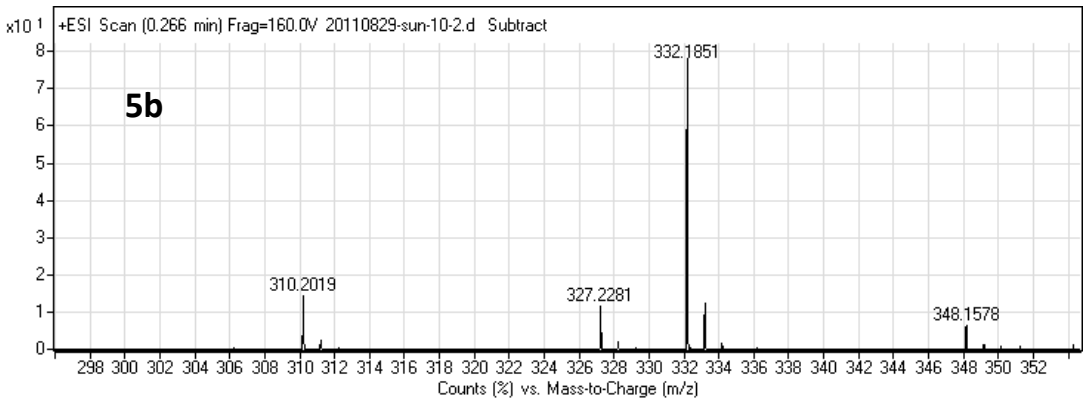

x10 2
+ESI Scan (0.243 min) Frag=160.0V 20110829-sun-16-1.d Subtract (2)
392.2799
414.2611
430.2353
8a
Counts (%) vs. Mass-to-Charge (m/z)

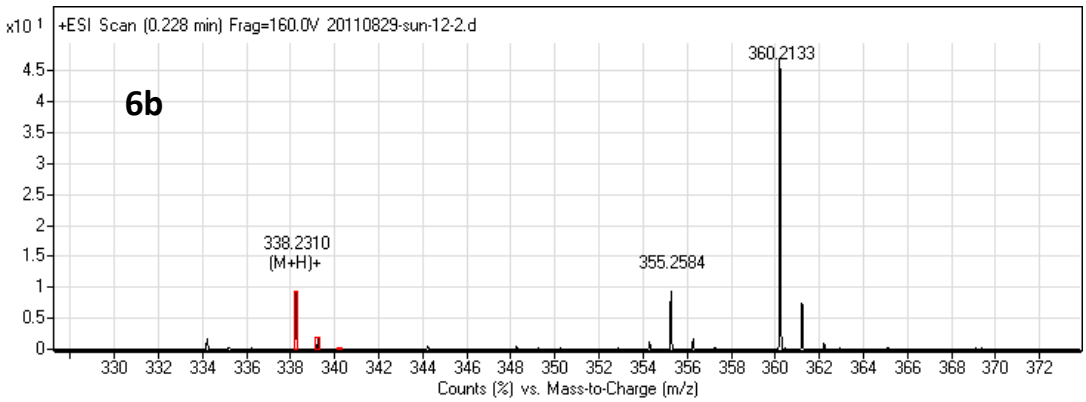

x10 2
-ESI Scan (0.211 min) Frag=135.0V 20111021-C10-T-2-1.d Subtract (2)
461.2035
1b
Counts (%) vs. Mass-to-Charge (m/z)

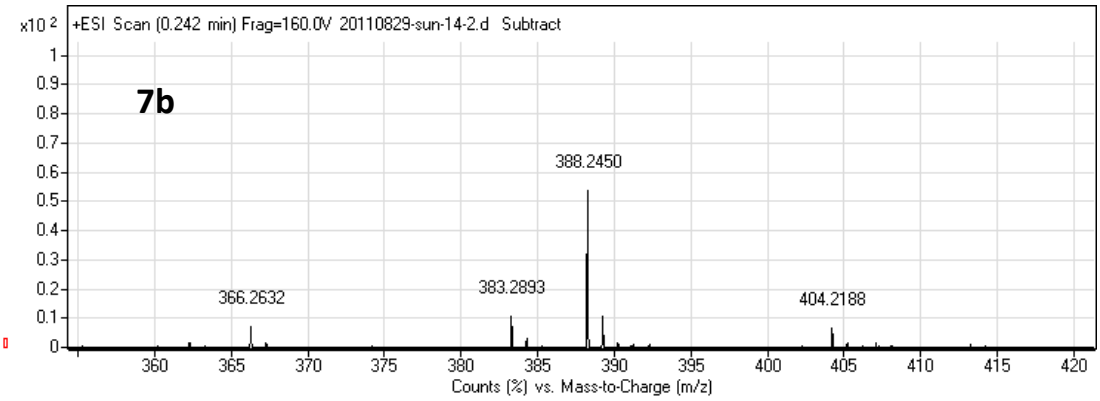

x10 2
-ESI Scan (0.284 min) Frag=135.0V 2012-0114-C12-3-IV.d Subtract
489.2402
2b
Counts (%) vs. Mass-to-Charge (m/z)

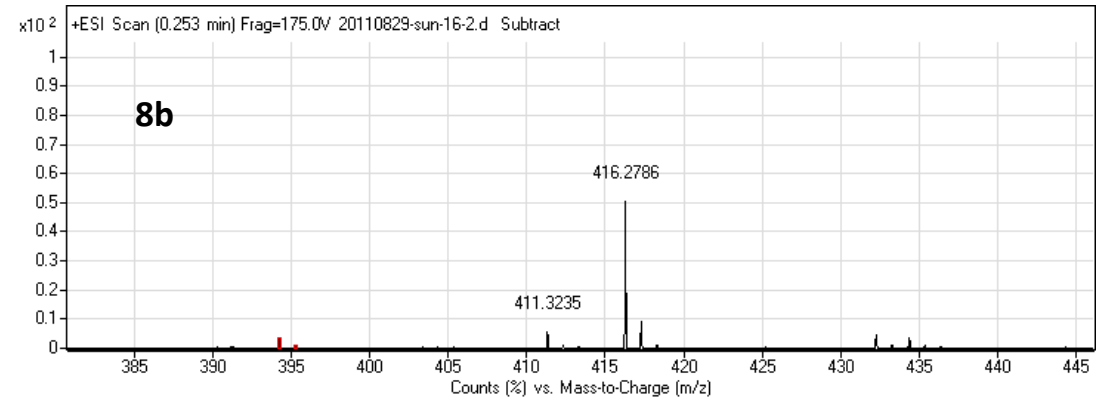

x10 2
-ESI Scan (0.499 min) Frag=135.0V 2012-0114-C14-IV.d Subtract (2)
517.2716
3b
Counts (%) vs. Mass-to-Charge (m/z)

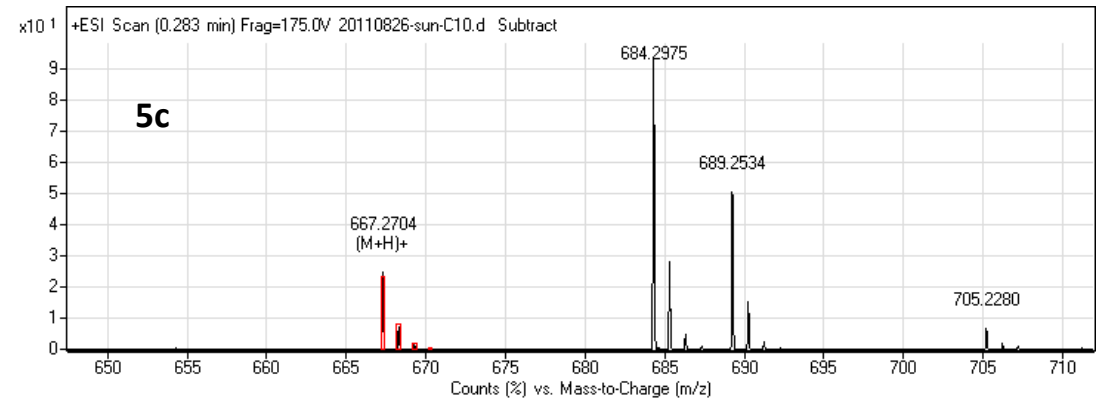

x10 2
-ESI Scan (0.516 min) Frag=135.0V 2012-0114-C16-1-IV.d Subtract (2)
545.2999
4b
Counts (%) vs. Mass-to-Charge (m/z)

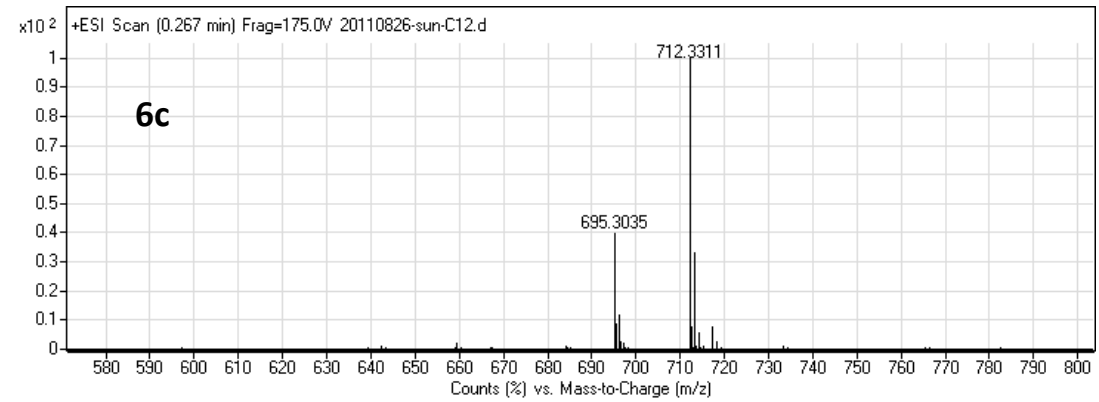

x10 1
+ESI Scan (0.266 min) Frag=160.0V 20110829-sun-10-2.d Subtract
332.1851
5b
310.2019
327.2281
348.1578
Counts (%) vs. Mass-to-Charge (m/z)

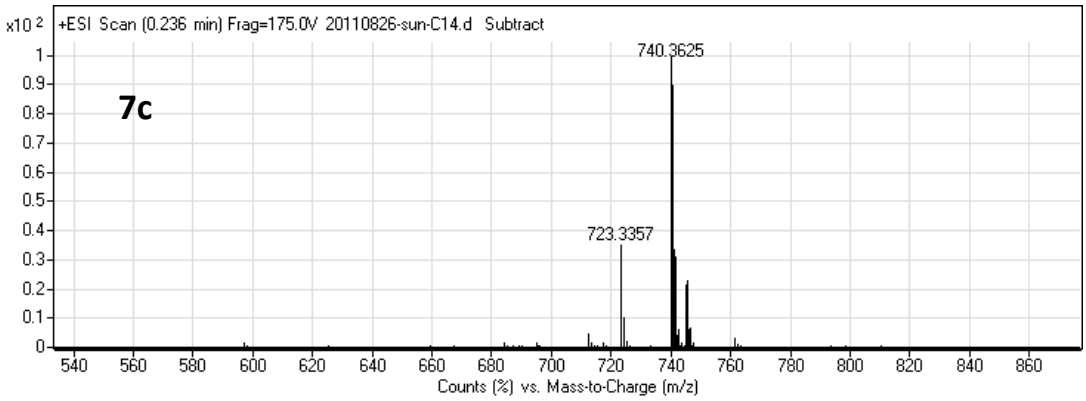

x10 1
+ESI Scan (0.228 min) Frag=160.0V 20110829-sun-12-2.d
360.2133
6b
338.2310
(M+H)+
355.2584
Counts (%) vs. Mass-to-Charge (m/z)

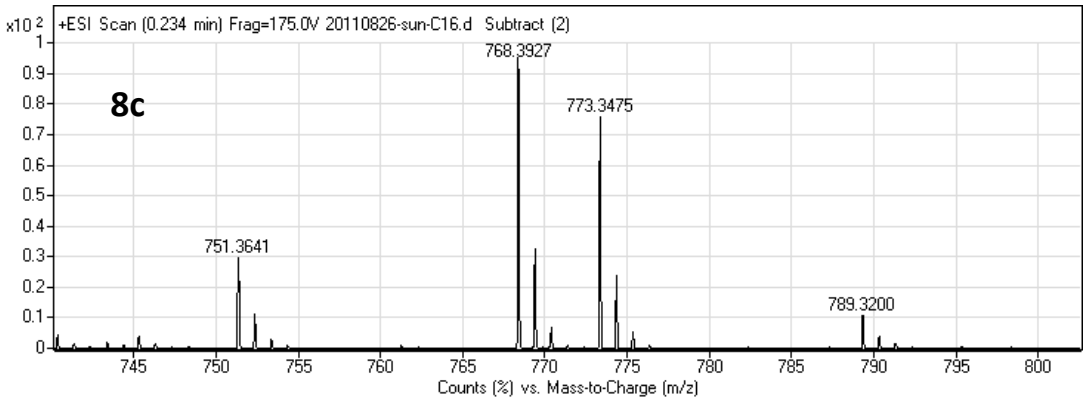

x10 2
+ESI Scan (0.242 min) Frag=160.0V 20110829-sun-14-2.d Subtract
7b
388.2450
366.2632
383.2893
404.2188
Counts (%) vs. Mass-to-Charge (m/z)

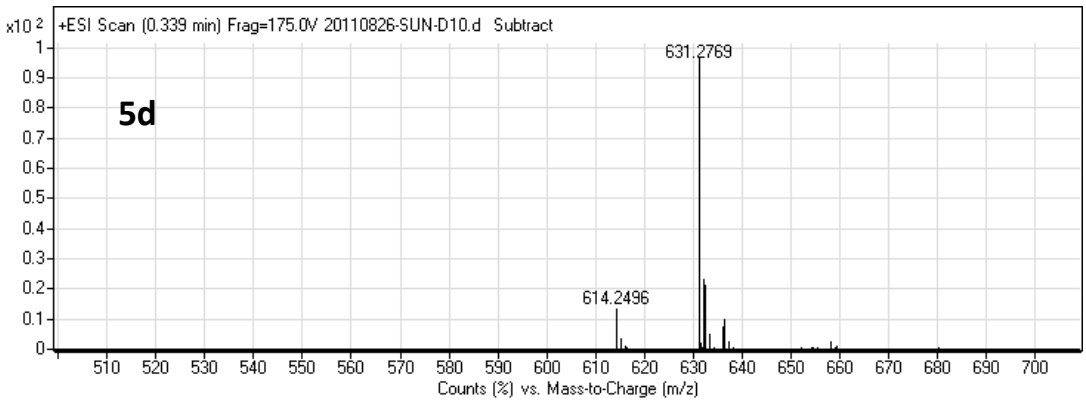

x10 2
+ESI Scan (0.253 min) Frag=175.0V 20110829-sun-16-2.d Subtract
8b
416.2786
411.3235
Counts (%) vs. Mass-to-Charge (m/z)

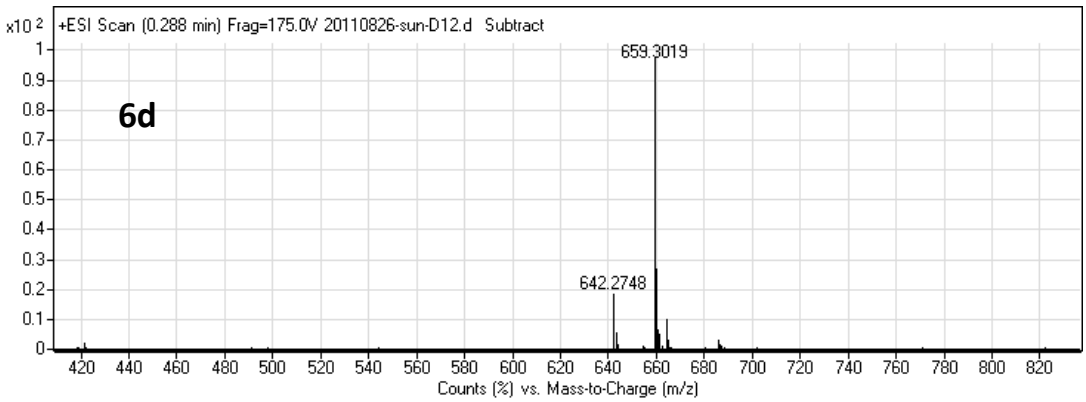

x10 1
+ESI Scan (0.283 min) Frag=175.0V 20110826-sun-C10.d Subtract
5c
684.2975
689.2534
667.2704
(M+H)+
705.2280
Counts (%) vs. Mass-to-Charge (m/z)

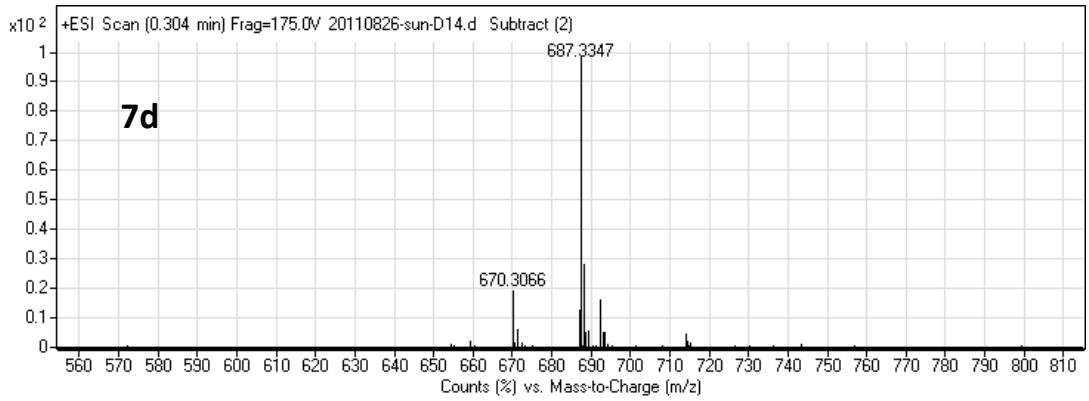

x10 2
+ESI Scan (0.267 min) Frag=175.0V 20110826-sun-C12.d
6c
712.3311
695.3035
Counts (%) vs. Mass-to-Charge (m/z)

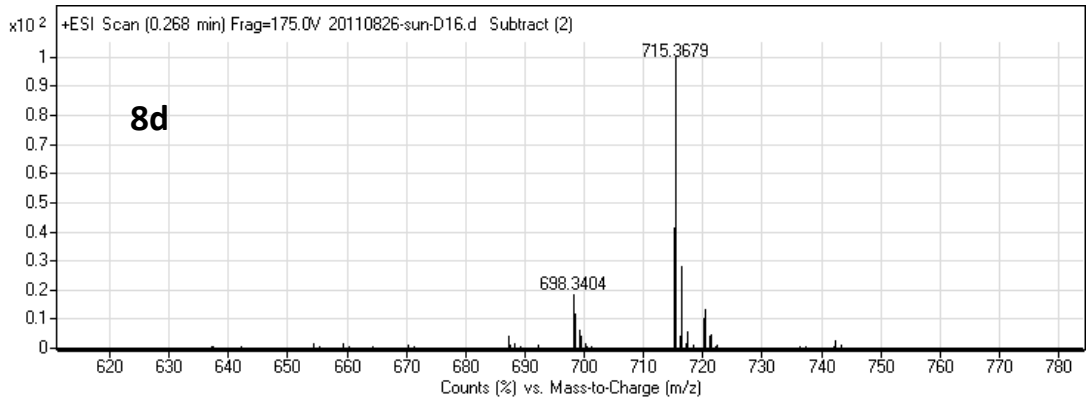

x10 2
+ESI Scan (0.236 min) Frag=175.0V 20110826-sun-C14.d Subtract
7c
740.3625
723.3357
Counts (%) vs. Mass-to-Charge (m/z)


x10 2
+ESI Scan (0.234 min) Frag=175.0V 20110826-sun-C16.d Subtract (2)
8c
768.3927
773.3475
751.3641
789.3200
Counts (%) vs. Mass-to-Charge (m/z)


x10 2
+ESI Scan (0.339 min) Frag=175.0V 20110826-SUN-D10.d Subtract
5d
631.2769
614.2496
Counts (%) vs. Mass-to-Charge (m/z)


x10 2
+ESI Scan (0.288 min) Frag=175.0V 20110826-sun-D12.d Subtract
6d
659.3019
642.2748
Counts (%) vs. Mass-to-Charge (m/z)


x10 2
+ESI Scan (0.304 min) Frag=175.0V 20110826-sun-D14.d Subtract (2)
687.3347
7d
670.3066
Counts (%) vs. Mass-to-Charge (m/z)


x10 2
+ESI Scan (0.268 min) Frag=175.0V 20110826-sun-D16.d Subtract (2)
715.3679
8d
698.3404
Counts (%) vs. Mass-to-Charge (m/z)