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Oxanorbornane-based Amphiphilic Systems: Design, Synthesis and Material Properties

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General experimental information:

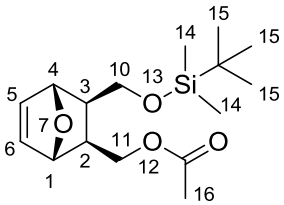
Experiments which required anhydrous conditions were carried out under nitrogen atmosphere using dry solvents. Thin-layer chromatography (TLC) was performed on 0.25 mm silica gel plates (60 F_{254} grade) from Merck, and were analyzed using a 254 nm UV light. The chromatographic separation was carried out on 100-200 mesh silica gel. Melting points were obtained on electro-thermal apparatus and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker Avance 400 MHz instrument, and the chemical shifts are reported in parts per million (ppm) relative to tetramethylsilane, with *J* values in Hertz. The splitting patterns in ^{1}H NMR spectra are reported as follows: s = singlet; d = doublet; t = triplet; q = quartet; dd = doublet of doublet; quin = quintet; ddd = doublet of doublet of doublet; m = multiplet. ^{13}C NMR data are reported with the solvent peak ($CDCl_3$ = 77.0 ppm) as the internal standard. High-resolution mass spectra (HRMS) were recorded on a Waters Q-Tof *micro*™ spectrometer with lock spray source. Infrared spectra were recorded using a Nicolet 6700 FT-IR spectrophotometer. The samples for SEM imaging were coated with Au-Pd (Gatan precision etching coating system (model No. 682) operating at 5 KeV) and analysed by FEI Quanta FEG 200 High Resolution Scanning Electron Microscope operating at 10-30 kV.

The intensity data collection during X-ray crystallographic analysis was carried out on a Bruker AXS (kappa apex II) diffractometer[1] equipped with graphite monochromated Mo (K_α) radiation. The data were collected for θ up to 25^{o} for Mo (K_α) radiation. ω and ϕ scans were employed to collect the data. The frame width for ω was set to 0.5 deg for data collection. The frames were integrated and data were reduced for Lorentz and polarization correction using SAINT- Plus. The multi-scan absorption correction[2] was applied to the data.

[1] Bruker (2004). APEX-II and SAINT-Plus (Version 7.06a), Bruker AXS Inc., Madison, Wisconsin, USA.
[2] Bruker (1999). SADABS, Bruker AXS Inc., Madison, Wisconsin, USA.

All structures were solved using SIR-92 and refined using SHELXL-97[3]. The molecular and packing diagrams were drawn using ORTEP-3[4] and Mercury 1.4.2. The non-hydrogen atoms were refined with anisotropic displacement parameter. All hydrogen atoms could be located in the difference Fourier map. However, the hydrogen atoms bonded to carbons were fixed at chemically meaningful positions and were allowed to ride with parent atom during the refinement.

((1S,2S,3R,4R)-3-(((tert-butyldimethylsilyl)oxy)methyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methyl acetate (+)-4

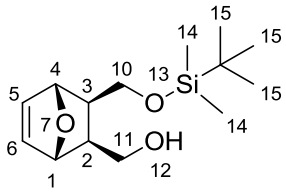


The meso diol **1** (Scheme 1, manuscript) was stereoselectively desymmetrized using *Pseudomonas Amano Lipase* (PS-Amano) through trans-acetylation with vinyl acetate using Bloch's protocol.[5] To a stirred solution of the resulting monoacylated alcohol (0.2 g, 1.01 mmol) in dry CH_2Cl_2 (6 mL) was added imidazole (0.171 g, 2.53 mmol), DMAP (12.3 mg, 0.10 mmol), followed by TBDMSCl (0.15 g, 1.81 mmol) at 0 ^oC. The mixture was allowed to slowly warm to room temperature and stirred overnight. After completion of the reaction, the solvents were evaporated, the residue dissolved in EtOAc (35 mL), washed with water (65 mL), dried (Na_2SO_4) and concentrated under reduced pressure to get a residue which was purified by column chromatography on silica gel using 10-20% EtOAc/Hexane; $[\alpha]_D^{23}$ +11.28 (*c* 1.0, MeOH); Gummy liquid; Yield = 98%; R_f (10% EtOAc/Hexane), 0.30; ^1H NMR ($CDCl_3$, 400 MHz): δ 6.32 (d, 1H, *J* = 6.4 Hz, 6-**H**), 6.28 (d, 1H, *J* = 6.4 Hz, 5-**H**), 4.78 (s, 1H, 1-**H**), 4.73 (s, 1H, 4-**H**), 4.25 (dd, 1H, *J* = 4.8, 10.6 Hz, 11-$\mathbf{H_a}$), 3.86 (t, 1H, *J* = 10.4 Hz, 11-$\mathbf{H_b}$), 3.66 (dd, 1H, *J* = 5.7, 9.8 Hz, 10-$\mathbf{H_a}$), 3.48 (t, 1H, *J* = 9.6 Hz, 10-$\mathbf{H_b}$), 2.01 (s, 3H, 16-**H**), 1.86-1.78 (m, 2H, 2-**H**, 3-**H**), 0.84 (s, 9H, 15-**H**), 0.00 (s, 6H, 14-**H**); ^{13}C NMR ($CDCl_3$,

[3] Sheldrick, G. M. (1997). SHELX-97 and SHELXL-97. University of Göttingen, Germany.
[4] ORTEP-3 for windows Farrugia, L. J. *J. Appl. Crystallogr.* **1997**, *30*, 565-566.
[5] C. Cinquin, I. Schaper, G. Mandville, R. Bloch, *Synlett* **1995**, 339-340.

100 MHz): δ 170.9, 136.0, 135.0, 80.4, 80.3, 64.1, 62.1, 42.6, 38.9, 25.9 (3C), 21.0, 18.2, -5.4 (2C): IR (neat): 2964, 2925, 2854, 1731, 1456, 1402, 1238, 1188, 1026, 899, 775 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{16}H_{28}O_4SiNa$ $(M+Na)^+$ 335.1655, found $(M+Na)^+$ 335.1642.

((1S,2S,3R,4R)-3-(((tert-butyldimethylsilyl)oxy)methyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methanol



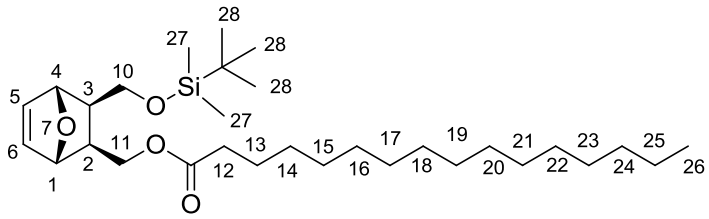
To a stirred solution of **4 (**0.6 g, 1.92 mmol**)** in dry MeOH (7 mL) was added NaOMe (0.156 g, 2.88 mmol) at 0 ^oC and the reaction mixture was allowed to stir at room temperature for 3 h. After completion of the reaction, saturated brine (20 mL) was added to the mixture, extracted with ethyl acetate, dried (Na_2SO_4) and concentrated under reduced pressure to get a crude product was directly used for the next step without purification; $[\alpha]_D^{25}$ +7.28 (*c* 1.0, MeOH); Gummy liquid; Yield = 74%; R_f (40% EtOAc/Hexane), 0.31; ^1H NMR ($CDCl_3$, 400 MHz): δ 6.32-6.25 (m, 2H, 5-**H**, 6-**H**), 4.60 (s, 1H, 4-**H**), 4.56 (s, 1H, 1-**H**), 3.80-3.64 (m, 3H, 10-**H**, 11-**H**), 3.64-3.55 (m, 1H, 11-**H**), 3.34 (bs, 1H, 12-**H**), 1.89-1.79 (m, 2H, 2-**H**, 3-**H**), 0.80 (s, 9H, 15-**H**), 0.00 (s, 6H, 14-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.0, 135.5, 81.4, 81.0, 63.9, 62.7, 42.9, 42.8, 25.9 (3C), 18.2, -5.4, -5.5: IR (neat): 3459, 2933, 2885, 2858, 1469, 1389, 1255, 1114, 1067, 837, 777, 689 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{14}H_{27}O_3Si$ $(M+H)^+$ 271.1729, found $(M+H)^+$ 271.1739.

General procedure for the preparation of ((+)-5a-e):

To a stirred solution of the crude alcohol **(**1.0 eq**)** and Et_3N (3.0 eq) in dry dichloromethane (4 mL of CH_2Cl_2 for 300 mg of starting material) was added appropriate acid chloride (1.2 eq) at 0 ^oC under N_2 atmosphere. The reaction mixture was warmed to room temperature and allowed to stir for 45 min. After completion of the reaction, the mixture was washed with water and extracted with dichloromethane. The organic layer was

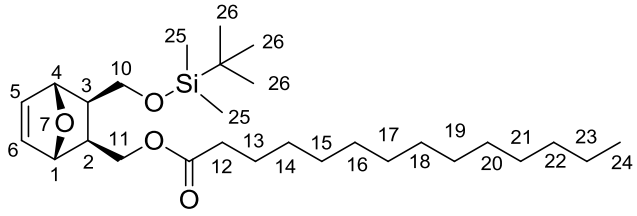
dried using sodium sulfate and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel using 10-20% EtOAc/Hexane. Yields and spectroscopic details of various compounds synthesized are given below.

((1S,2S,3R,4R)-3-(((tert-butyldimethylsilyl)oxy)methyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methyl palmitate ((+)-5a)



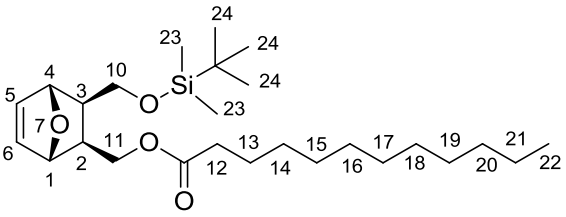
Gummy liquid; Yield = 74%; R_f (5% EtOAc/Hexane), 0.34; $[\alpha]_D^{25}$ +5.56 (*c* 1.0, MeOH); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.32 (dd, 1H, *J* = 1.6, 6.0 Hz, 6-**H**), 6.28 (dd, 1H, *J* = 1.2, 5.2 Hz, 5-**H**), 4.79 (s, 1H, 1-**H**), 4.72 (s, 1H, 4-**H**), 4.24 (dd, 1H, *J* = 4.8, 10.4 Hz, 11-$\mathbf{H_a}$), 3.87 (t, 1H, *J* = 10.4 Hz, 11-$\mathbf{H_b}$), 3.68 (dd, 1H, *J* = 5.2, 9.6 Hz, 10-$\mathbf{H_a}$), 3.48 (t, 1H, *J* = 9.6 Hz, 10-$\mathbf{H_b}$), 2.26 (t, 2H, *J* = 7.6 Hz, 12-**H**), 1.88-1.74 (m, 2H, 2-**H**, 3-**H**), 1.60-1.52 (m, 2H, 13-**H**), 1.19 (bs, 24H, 14-25-**H**), 0.84 (s, 9H, 28-**H**), 0.82 (t, 3H, 26-**H,** merged with 28-**H**), 0.00 (s, 6H, 27-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 135.9, 135.0, 80.4, 80.3, 63.8, 62.1, 42.5, 39.0, 34.4, 31.9, 29.7 (3C), 29.67 (2C), 29. 64, 29.5, 29.34, 29.26, 29.16, 25.9 (3C), 25.0, 22.7, 18.2, 14.1, -5.4 (2C): IR (neat): 2926, 2855, 1739, 1466, 1254, 1168, 1113, 1092, 839, 777, 688 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{30}H_{57}O_4Si$ $(M+H)^+$ 509.4026, found $(M+H)^+$ 509.4009.

((1S,2S,3R,4R)-3-(((tert-butyldimethylsilyl)oxy)methyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methyl tetradecanoate ((+)-5b)



Gummy liquid; Yield = 86%; R_f (5% EtOAc/Hexane), 0.34; $[\alpha]_D^{25}$ +5.77 (*c* 1.0, MeOH); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.32 (d, 1H, *J* = 5.6 Hz, 6-**H**), 6.28 (d, 1H, *J* = 5.6 Hz, 5-**H**), 4.79 (s, 1H, 1-**H**), 4.72 (s, 1H, 4-**H**), 4.24 (dd, 1H, *J* = 4.8, 10.4 Hz, 11-$\mathbf{H_a}$), 3.87 (t, 1H, *J* = 10.4 Hz, 11-$\mathbf{H_b}$), 3.68 (dd, 1H, *J* = 5.6, 9.6, 10-$\mathbf{H_a}$), 3.48 (t, 1H, *J* = 9.6 Hz, 10-$\mathbf{H_b}$), 2.26 (t, 2H, *J* = 7.2 Hz, 12-**H**), 1.88-1.76 (m, 2H, 2-**H**, 3-**H**), 1.58 -1.53 (m, 2H, 13-**H**), 1.26-1.19 (m, 20H, 14-23-**H**), 0.84 (s, 9H, 26-**H**), 0.82 (t, 3H, 24-**H**, merged with 26-**H**), 0.00 (s, 6H, 25-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 135.9, 135.0, 80.4, 80.2, 63.8, 62.1, 42.5, 39.0, 34.4, 31.9, 29.62 (3C), 29.58, 29.45, 29.3, 29.25, 29.15, 25.9 (3C), 25.0, 22.7, 18.2, 14.1, -5.4 (2C): IR (neat): 2926, 2855, 1739, 1465, 1254, 1168, 1113, 1093, 837, 775 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{28}H_{53}O_4Si$ $(M+H)^+$ 481.3713, found $(M+H)^+$ 481.3701.

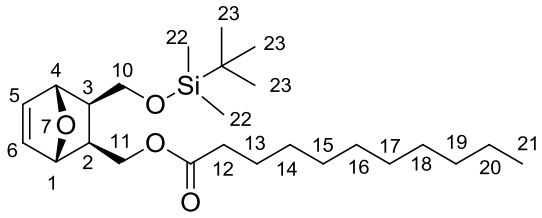
((1S,2S,3R,4R)-3-((tert-butyldimethylsilyloxy)methyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methyl dodecanoate ((+)-5c)



Gummy liquid; Yield = 83%; R_f (5% EtOAc/Hexane), 0.34; $[\alpha]_D^{26}$ +6.12 (*c* 1.0, MeOH); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.32 (dd, 1H, *J* = 1.2, 5.6 Hz, 6-**H**), 6.27 (dd, 1H, *J* = 1.6, 6.0 Hz, 5-**H**), 4.78 (s, 1H, 1-**H**), 4.71 (s, 1H, 4-**H**), 4.23 (dd, 1H, *J* = 5.2, 10.8 Hz, 11-$\mathbf{H_a}$), 3.87 (t, 1H, *J* = 10.0 Hz, 11-$\mathbf{H_b}$), 3.67 (dd, 1H, *J* = 6.0, 10.0 Hz, 10-$\mathbf{H_a}$), 3.48 (t, 1H, *J* = 9.6 Hz, 10-$\mathbf{H_b}$), 2.25 (t, 2H, *J* = 7.2 Hz, 12-**H**), 1.87-1.75 (m, 2H, 2-**H**, 3-**H**), 1.56 (quin, 2H, *J* = 7.2 Hz, 13-**H**), 1.26-1.19 (m, 16H, 14-21-**H**), 0.83 (s, 9H, 24-**H**), 0.81 (t, 3H, *J* = 6.8 Hz, 22-**H**), 0.00 (s, 6H, 23-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.6, 135.9, 135.0, 80.4, 80.2, 63.8, 62.1, 42.5, 38.9, 34.3, 31.9, 29.6 (2C), 29.4, 2

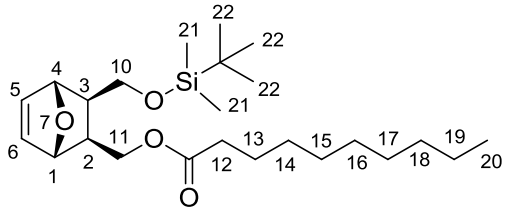
9.3, 29.2, 29.1, 25.9 (3C), 24.9, 22.6, 18.2, 14.1, -5.4 (2C): IR (neat): 2925, 2854, 1738, 1465, 1253, 1165, 1091, 1071, 835, 775, 687 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{26}H_{49}O_4Si$ $(M+H)^+$ 453.3400, found $(M+H)^+$ 453.3389.

((1S,2S,3R,4R)-3-(((tert-butyldimethylsilyl)oxy)methyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methyl undecanoate ((+)-5d)



Gummy liquid; Yield = 84%; R_f (5% EtOAc/Hexane), 0.34; $[\alpha]_D^{25}$ +5.12 (*c* 1.0, MeOH); ^1H NMR ($CDCl_3$, 500 MHz): δ 6.37 (dd, 1H, *J* = 1.5, 6.0 Hz, 6-**H**), 6.33 (dd, 1H, *J* = 1.5, 6.0 Hz, 5-**H**), 4.85 (s, 1H, 1-**H**), 4.78 (s, 1H, 4-**H**), 4.30 (dd, 1H, *J* = 5.0, 10.5 Hz, 11-$\mathbf{H_a}$), 3.93 (t, 1H, *J* = 10.5 Hz, 11-$\mathbf{H_b}$), 3.73 (dd, 1H, *J* = 6.0, 10.0 Hz, 10-$\mathbf{H_a}$), 3.48 (t, 1H, *J* = 9.6 Hz, 10-$\mathbf{H_b}$), 2.31 (t, 2H, *J* = 7.5 Hz, 12-**H**), 1.93-1.88 (m, 1H, 3-**H**), 1.87-1.82 (m, 1H, 2-**H**), 1.64-1.61 (m, 2H, 13-**H**), 1.33-1.23 (m, 14H, 14-20-**H**), 0.89 (s, 9H, 23-**H**), 0.82 (t, 3H, 21-**H,** merged with 23-**H**), 0.00 (s, 6H, 22-**H**); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 173.6, 135.9, 135.0, 80.4, 80.3, 63.8, 62.1, 42.6, 39.0, 34.4, 31.9, 29.5 (2C), 29.3 (2C), 29.2 4, 25.9 (3C), 25.0, 22.7, 18.2, 14.1, -5.4 (2C): IR (neat): 2954, 2927, 2856, 1739, 1465, 1255, 1112, 1073, 912, 838, 776, 737 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{25}H_{46}O_4SiNa$ $(M+Na)^+$ 461.3063, found $(M+Na)^+$ 461.3083.

((1S,2S,3R,4R)-3-(((tert-butyldimethylsilyl)oxy)methyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methyl decanoate ((+)-5e)

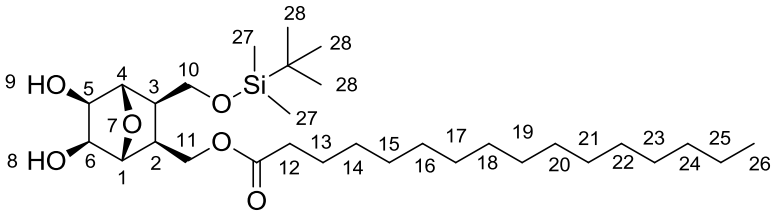


Gummy liquid; Yield = 96%; R_f (5% EtOAc/Hexane), 0.34; $[\alpha]_D^{26}$ +6.33 (*c* 1.0, MeOH); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.39-6.33 (m, 2H, 5-**H**, 6-**H**), 4.85 (s, 1H, 1-**H**), 4.78 (s, 1H, 4-**H**), 4.30 (dd, 1H, *J* = 4.8, 10.4 Hz, 11-$\mathbf{H_a}$), 3.93 (t, 1H, *J* = 10.4 Hz, 11-$\mathbf{H_b}$), 3.73 (dd, 1H, *J* = 6.0, 9.6 Hz, 10-$\mathbf{H_a}$), 3.54 (t, 1H, *J* = 9.6 Hz, 10-$\mathbf{H_b}$), 2.32 (t, 2H, *J* = 7.2 Hz, 12-**H**), 1.94-1.82 (m, 2H, 2-**H**, 3-**H**), 1.65-1.59 (m, 2H, 13-**H**), 1.29-1.26 (m, 12H, 14-19-**H**), 0.9 (s, 9H, 22-**H**), 0.88 (t, 3H, merged with 22-**H**, 20-**H**), 0.06 (s, 6H, 21-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 135.9, 135.1, 80.4, 80.3, 63.8, 62.1, 42.5, 39.0, 34.4, 31.8, 29.4, 29.2 (2C), 29 .1, 25.9 (3C), 25.0, 22.6, 18.2, 14.1, -5.4 (2C): IR (neat): 2931, 2861, 2403, 1728, 1522, 1425, 1216, 767, 674 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{24}H_{45}O_4Si$ $(M+H)^+$ 425.3087, found $(M+H)^+$ 425.3079.

General procedure for dihydroxylation of 5a-e:

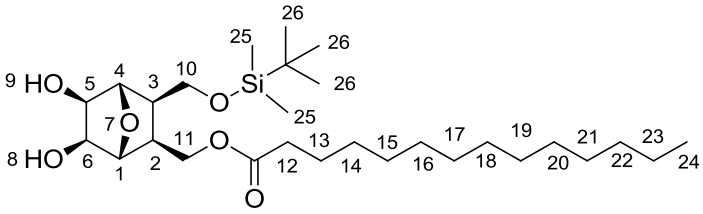
To a stirred solution containing a mixture of **5a-e** (1.0 eq), N-methyl morpholine N-Oxide (2.4 eq) and pyridine (30 uL for 100mg of the alkene) in ^{t}BuOH-H_2O (3:1) was added osmium tetraoxide (0.02M solution in ^{t}BuOH, 0.01 eq). The reaction mixture was heated at 80 ^{o}C for 5-6 h. After completion of the reaction, the mixture was cooled to room temperature, treated with 15% aq. Na_2SO_3 solution (4 mL), allowed to stir for 5-10 min and water (8 mL) was added to it. All the solvents were removed under reduced pressure and the resulting residue was extracted with ethyl acetate. The organic layer was dried using sodium sulfate and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel using Hexane/EtOAc in a gradient mode. Yields and spectroscopic details of various compounds synthesized are given below.

((1R,2S,3R,4S,5R,6S)-3-(((tert-butyldimethylsilyl)oxy)methyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptan-2-yl)methyl palmitate



Colorless solid; Yield = 82%; R_f (40% EtOAc/Hexane), 0.36; $[\alpha]_D^{25}$ +13.76 (*c* 1.0, MeOH); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.19 (s, 1H, 1-**H**), 4.15 (s, 1H, 4-**H**), 4.15-4.12 (m, 1H, 11-$\mathbf{H_a}$), 3.87-3.80 (m, 3H, 5-**H**, 6-**H**, 11-$\mathbf{H_b}$), 3.54 (dd, 1H, *J* = 6.0, 9.6 Hz, 10-$\mathbf{H_a}$), 3.44 (t, 1H, *J* = 9.6 Hz, 10-$\mathbf{H_b}$), 3.05 (bs, -OHs), 2.24 (t, 2H, *J* = 7.2 Hz, 12-**H**), 2.00 (ddd, 1H, *J* = 5.6, 9.6, 9.6 Hz, 3-**H**), 1.94-1.88 (m, 1H, 2-**H**), 1.61-1.51 (m, 2H, 13-**H**), 1.21 (bs, 24H, 14-25-**H**), 0.84 (s, 9H, 28-**H**), 0.83 (t, 3H, merged with 28-**H**, 26-**H**), 0.00 (s, 6H, 27-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.6, 84.6, 84.3, 74.3 (2C), 62.0, 60.6, 43.4, 39.9, 34.3, 31.9, 29.7 (5C), 29.6, 29.5, 29.3, 29.24, 29.16, 25.9 (3C), 24.9, 22.7, 18.2, 14.1, -5.4 (2C): IR (neat): 3404, 2926, 2855, 1739, 1466, 1171, 1090, 838, 778 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{30}H_{59}O_6Si$ $(M+H)^+$ 543.4081, found $(M+H)^+$ 543.4097.

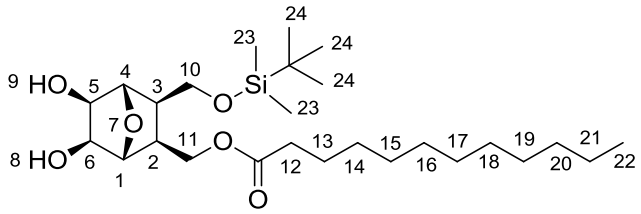
((1R,2S,3R,4S,5R,6S)-3-(((tert-butyldimethylsilyl)oxy)methyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptan-2-yl)methyl tetradecanoate



Colorless solid; Yield = 89%; R_f (40% EtOAc/Hexane), 0.36; $[\alpha]_D^{26}$ +13.45 (*c* 1.0, MeOH); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.19 (s, 1H, 1-**H**), 4.15 (s, 1H, 4-**H**), 4.14 (dd, 1H, 11-$\mathbf{H_a}$, merged with 4-**H**), 3.87-3.80 (m, 3H, 11-$\mathbf{H_b}$, 5-**H**, 6-**H**), 3.54 (dd, 1H, *J* = 6.0, 9.6 Hz, 10-$\mathbf{H_a}$), 3.44 (t, 1H, *J* = 9.6 Hz, 10-$\mathbf{H_b}$), 3.36 (bs, 1H, -OH), 2.25 (t, 2H, *J* = 7.6 Hz, 12-**H**), 2.03-1.86 (m, 2H, 3-**H**, 2-**H**), 1.82 (bs, 1H, -OH), 1.60-1.55 (m, 2H, 13-**H**), 1.21 (bs, 20H, 14-23-

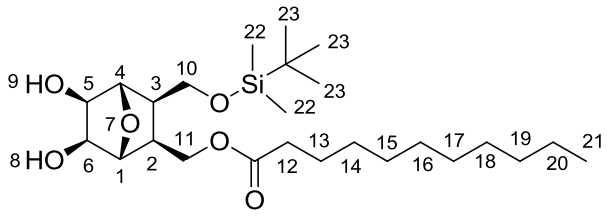
H), 0.84 (bs, 12H, 26-**H**, 24-**H**), 0.00 (s, 6H, 25-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 84.5, 84.3, 74.3 (2C), 61.9, 60.5, 43.4, 39.9, 34.3, 31.9, 29.62 (3C), 29.58, 29 .4, 29.3, 29.2, 29.1, 25.8 (3C), 24.9, 22.7, 18.2, 14.1, -5.4 (2C): IR (neat): 3369, 2927, 2856, 1740, 1462, 1254, 1218, 1092, 1064, 839, 774 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{28}H_{54}O_6NaSi$ $(M+Na)^+$ 537.3587, found $(M+Na)^+$ 537.3575.

((1R,2S,3R,4S,5R,6S)-3-(((tert-butyldimethylsilyl)oxy)methyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptan-2-yl)methyl dodecanoate



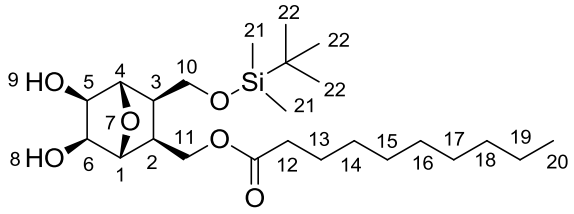
Colorless solid; Yield = 91%; R_f (40% EtOAc/Hexane), 0.36; $[\alpha]_D^{25}$ +13.88 (*c* 1.0, MeOH); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.18 (s, 1H, 1-**H**), 4.15 (s, 1H, 4-**H**), 4.14 (dd, 1H, 11-**H$_a$**, merged with 4-**H**), 3.85-3.80 (m, 4H, -OH, 11-**H$_b$**, 5-**H**, 6-**H**), 3.53 (dd, 1H, *J* = 6.4, 9.6 Hz, 10-**H$_a$**), 3.44 (t, 1H, *J* = 9.6 Hz, 10-**H$_b$**), 2.24 (t, 2H, *J* = 7.6 Hz, 12-**H**), 2.00 (ddd, 1H, *J* = 5.6, 9.6, 9.6 Hz, 3-**H**), 1.94-1.88 (m, 1H, 2-**H**), 1.56 (quin, 2H, *J* = 7.2 Hz, 13-**H**), 1.23 (bs, 16H, 14-21-**H**), 0.84 (bs, 12H, 24-**H**, 22-**H**), 0.00 (s, 6H, 23-**H**); -OH proton did not appear; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.6, 84.5, 84.2, 74.3 (2C), 61.9, 60.6, 43.3, 39.8, 34.2, 31.8, 29.5 (2C), 29.4, 29.2 5, 29.17, 29.09, 25.8 (3C), 24.9, 22.6, 18.1, 14.0, -5.5 (2C): IR (neat): 3409, 2927, 2856, 1739, 1465, 1395, 1254, 1172, 1088, 1065, 839, 778 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{26}H_{51}O_6Si$ $(M+H)^+$ 487.3455, found $(M+H)^+$ 487.3465.

((1R,2S,3R,4S,5R,6S)-3-(((tert-butyldimethylsilyl)oxy)methyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptan-2-yl)methyl undecanoate



Colorless solid; Yield = 73%; R_f (40% EtOAc/Hexane), 0.36; $[\alpha]_D^{26}$ +11.41 (*c* 1.0, MeOH); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.19 (s, 1H, 1-**H**), 4.15 (s, 1H, 4-**H**), 4.15-4.12 (m, 1H, 11-$\mathbf{H_a}$), 3.87-3.80 (m, 3H, 5-**H**, 6-**H**, 11-$\mathbf{H_b}$), 3.54 (dd, 1H, *J* = 6.0, 9.6 Hz, 10-$\mathbf{H_a}$), 3.44 (t, 1H, *J* = 9.6 Hz, 10-$\mathbf{H_b}$), 3.08 (bs, 2H, OHs), 2.24 (t, 2H, *J* = 7.2 Hz, 12-**H**), 2.00 (ddd, 1H, *J* = 5.6, 9.6, 9.6 Hz, 3-**H**), 1.94-1.88 (m, 1H, 2-**H**), 1.61-1.51 (m, 2H, 13-**H**), 1.21 (bs, 14H, 14-20-**H**), 0.84 (s, 9H, 23-**H**), 0.83 (t, 3H, merged with 23-**H**, 21-**H**), 0.00 (s, 6H, 22-**H**); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 173.6, 84.6, 84.3, 74.3 (2C), 62.0, 60.5, 43.4, 39.9, 34.3, 31.9, 29.5, 29.4, 29.3, 29.23, 29.15, 25.9 (3C), 24.9, 22.7, 18.2, 14.1, -5.4 (2C): IR (neat): 3401, 2954, 2927, 2856, 1737, 1466, 1255, 1167, 1134, 1093, 1063, 838, 759 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{25}H_{48}O_6SiNa$ $(M+Na)^+$ 495.3118, found $(M+Na)^+$ 495.3137.

((1R,2S,3R,4S,5R,6S)-3-(((tert-butyldimethylsilyl)oxy)methyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptan-2-yl)methyl decanoate



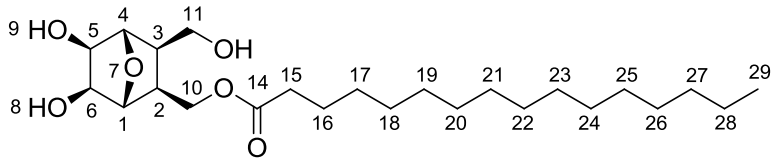
Colorless solid; Yield = 93%; R_f (40% EtOAc/Hexane), 0.36; $[\alpha]_D^{23}$ +13.37 (*c* 1.0, MeOH); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.23 (s, 1H, 1-**H**), 4.19 (s, 1H, 4-**H**), 4.18 (dd, 1H, merged with 4-**H**, 11-$\mathbf{H_a}$), 3.91-3.84 (m, 3H, 5-**H**, 6-**H**, 11-$\mathbf{H_b}$), 3.59 (dd, 1H, *J* = 6.4, 9.6 Hz, 10-$\mathbf{H_a}$), 3.49 (t, 1H, *J* = 9.2 Hz, 10-$\mathbf{H_b}$), 3.13 (bs, 2H, -OH), 2.29 (t, 2H, *J* = 7.2 Hz, 12-**H**), 2.05 (ddd, 1H, *J* = 5.6, 9.6, 9.6 Hz, 3-**H**), 1.96 (ddd, 1H, *J* = 6.0, 8.8, 8.8 Hz, 2-**H**), 1.61 (quin,

2H, *J* = 7.2 Hz, 13-**H**), 1.29-1.26 (m, 12H, 14-19-**H**), 0.88 (bs, 12H, 22-**H**, 20-**H**), 0.04 (s, 6H, 21-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.6, 84.6, 84.3, 74.3 (2C), 62.0, 60.6, 43.4, 39.9, 34.3, 31.8, 29.4 (2C), 29.2, 29.1, 25.9 (3C), 24.9, 22.6, 18.2, 14.1, -5.4 (2C): IR (neat): 3426, 2929, 2857, 1739, 1467, 1254, 1062, 838, 778 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{24}H_{47}O_6Si$ $(M+H)^+$ 459.3142, found $(M+H)^+$ 459.3148.

General procedure for deprotection of TBDMS group from the above intermediates

To a stirred solution of the above silyl protected precursors (1.0 eq) in THF (6 mL) was added *n*-Bu_4NF (2.0 eq) and allowed to stir at room temperature for 3 h. After completion of the reaction, the mixture was concentrated *in vacuo* and the residue was purified by column chromatography on silica gel using ethyl acetate-MeOH system in a gradient mode. Yields and spectroscopic details of various compounds synthesized are given below.

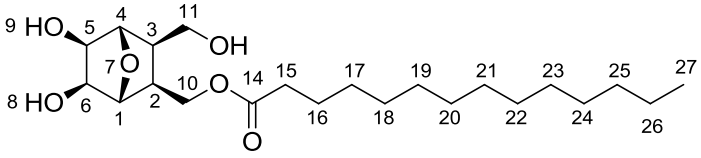
((1R,2S,3R,4S,5R,6S)-5,6-dihydroxy-3-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptan-2-yl)methyl palmitate ((+)-6a)



Colorless solid; Yield = 90%; R_f (EtOAc), 0.36; $[\alpha]_D^{27}$ +8.68 (*c* 1.0, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.30 (s, 1H, 4-**H**), 4.22 (s, 1H, 1-**H**), 4.20 (dd, 1H, 10-$\mathbf{H_a}$, merged with 1-**H**), 3.97 (t, 1H, *J* = 10.4 Hz, 10-$\mathbf{H_b}$), 3.95-3.92 (m, 2H, 5-**H**, 6-**H**), 3.71 (dd, 1H, *J* = 6.4, 10.0 Hz, 11-$\mathbf{H_a}$), 3.60 (t, 1H, *J* = 9.6 Hz, 11-$\mathbf{H_b}$), 2.70 (bs, 1H, -O**H**), 2.63 (bs, 1H, -O**H**), 2.30 (t, 2H, *J* = 7.6 Hz, 15-**H**), 2.08 (ddd, 1H, *J* = 6.0, 9.2, 9.2 Hz, 2-**H**), 2.05-1.99 (m, 1H, 3-**H**), 1.73 (bs, 1H, -O**H**), 1.65-1.58 (m, 2H, 16-**H**), 1.26 (bs, 24H, 17-28-**H**), 0.88 (t, 3H, *J* = 6.4 Hz, 29-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 84.9, 84.6, 74.4, 74.2, 62.4, 60.4, 43.3, 40.3, 34.4, 31.9, 29.7 (6C), 29.5, 29.3, 29.3, 2

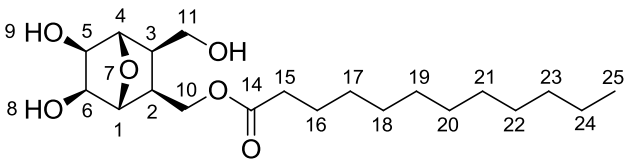
9.2, 25.0, 22.7, 13.9; IR (neat): 3394, 2919, 2851, 1732, 1467, 1264, 1177, 1127, 1074, 968, 915, 748 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{24}H_{45}O_6$ $(M+H)^+$ 429.3216, found $(M+H)^+$ 429.3206.

((1R,2S,3R,4S,5R,6S)-5,6-dihydroxy-3-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptan-2-yl)methyl tetradecanoate ((+)-6b)



Colorless solid; Yield = 96%; R_f (EtOAc), 0.36; $[\alpha]_D^{27}$ +8.56 (*c* 1.0, $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 4.31 (s, 1H, 4-**H**), 4.22 (s, 1H, 1-**H**), 4.19 (dd, 1H, *J* = 6.0, 11.2 Hz, 10-$\mathbf{H_a}$), 3.99-3.93 (m, 3H, 10-$\mathbf{H_b}$, 5-**H**, 6-**H**), 3.72 (dd, 1H, *J* = 6.0, 10.0 Hz, 11-$\mathbf{H_a}$), 3.60 (t, 1H, *J* = 8.8 Hz, 11-$\mathbf{H_b}$), 2.81 (bs, 1H, -O**H**), 2.30 (t, 2H, *J* = 7.6 Hz, 15-**H**), 2.15-2.09 (m, 1H, 2-**H**), 2.05-2.02 (m, 1H, 3-**H**), 1.69 (bs, 2H, -OH), 1.62-1.61 (m, 2H, 16-**H**), 1.26 (bs, 20H, 17-26-**H**), 0.88 (t, 3H, *J* = 6.8 Hz, 27-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.9, 84.6, 84.4, 74.1, 73.9, 62.2, 59.8, 42.8, 39.8, 34.2, 31.9, 29.6 (4C), 29.5, 29.34, 29.29, 29.17, 24.9, 22.7, 14.1; IR (neat): 3391, 2919, 2853, 1731, 1408, 1051, 1015, 908, 710, 632 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{22}H_{40}O_6Na$ $(M+Na)^+$ 423.2723, found $(M+Na)^+$ 423.2711.

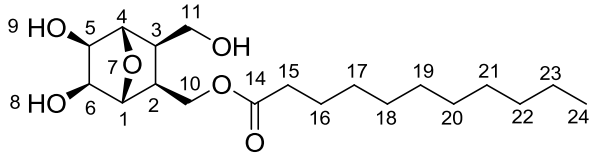
((1R,2S,3R,4S,5R,6S)-5,6-dihydroxy-3-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptan-2-yl)methyl dodecanoate ((+)-6c)



Colorless solid; Yield = 93%; R_f (10% MeOH/EtOAc), 0.58; $[\alpha]_D^{27}$ +8.39 (*c* 1.0, $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 4.34 (s, 1H, 4-**H**), 4.20 (s, 1H, 1-**H**), 4.12 (dd, 1H, *J* = 5.6, 10.8

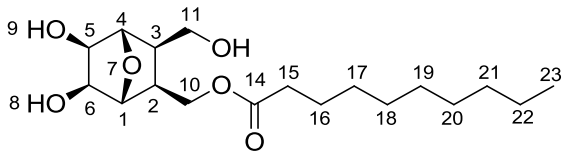
Hz, 10-**H$_a$**), 4.00-3.87 (m, 4H, -O**H**, 5-**H**, 6-**H**, 10-**H$_b$**), 3.67 (dd, 2H, *J* = 5.2, 10.4 Hz, -O**H**, 11-**H$_a$**), 3.54 (t, 1H, *J* = 9.6 Hz, 11-**H$_b$**), 2.92 (bs, 1H, -O**H**), 2.30 (t, 2H, *J* = 7.2 Hz, 15-**H**), 2.10 (ddd, 1H, *J* = 6.4, 9.2, 9.2 Hz, 2-**H**), 2.04-1.98 (m, 1H, 3-**H**), 1.61 (quin, 2H, *J* = 6.8 Hz, 16-**H**), 1.40-1.20 (bs, 16H, 17-24-**H**), 0.88 (t, 3H, *J* = 6.4 Hz, 25-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.8, 84.7, 84.4, 74.2, 74.0, 62.2, 60.1, 42.9, 39.9, 34.3, 31.9, 29.6 (2C), 29.5, 29.3 0, 29.25, 29.15, 24.9, 22.7, 14.1: IR (neat): 3411, 2919, 2850, 1714, 1458, 1297, 1176, 1048, 959, 911, 827, 654, 529 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{20}H_{36}O_6Na$ $(M+Na)^+$ 395.2410, found $(M+Na)^+$ 395.2419.

((1R,2S,3R,4S,5R,6S)-5,6-dihydroxy-3-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptan-2-yl)methyl undecanoate ((+)-6d)



Colorless solid; Yield = 83%; R_f (10% MeOH/EtOAc), 0.58; $[\alpha]_D^{27}$ +7.74 (*c* 1.0, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.34 (s, 1H, 4-**H**), 4.19 (s, 1H, 1-**H**), 4.13-4.09 (m, 1H, 10-**H$_a$**), 3.96-3.94 (m, 3H, 5-**H**, 6-**H**, 10-**H$_b$**), 3.82 (bs, 1H, -**OH**), 3.72 (dd, 1H, *J* = 5.0, 10.5 Hz, 11-**H$_a$**), 3.59 (t, 1H, *J* = 9.5 Hz, 11-**H$_b$**), 3.19 (bs, 1H, -**OH**), 2.30 (t, 2H, *J* = 7.5 Hz, 15-**H**), 2.11-2.08 (m, 1H, 2-**H**), 2.02 (ddd, 1H, *J* = 5.5, 9.5, 9.5 Hz, 3-**H**), 1.60 (qn, 2H, *J* = 7.0 Hz, -16-**H**), 1.33-1.26 (m, 14H, 17-23-**H**), 0.88 (t, 3H, *J* = 7.0 Hz, 24-**H**) one -OH proton exchanged; ^{13}C NMR ($CDCl_3$, 125 MHz): δ 173.9, 84.6, 84.4, 74.2, 74.0, 62.1, 59.9, 42.9, 39.9, 34.3, 31.9, 29.54, 29.46, 29.28, 29.24, 29.1, 24.9, 22.7, 14.1: IR (neat): 3366, 2921, 2854, 1719, 1465, 1407, 1177, 1017, 963 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{19}H_{34}O_6Na$ $(M+Na)^+$ 381.2253, found $(M+Na)^+$ 381.2258.

((1R,2S,3R,4S,5R,6S)-5,6-dihydroxy-3-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptan-2-yl)methyl decanoate ((+)-6e)

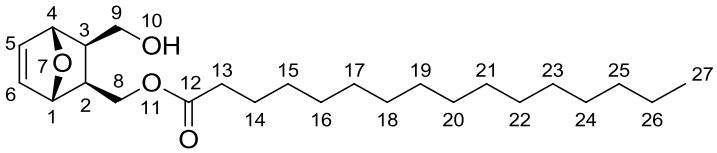


Colorless solid; Yield = 93%; R_f (10% MeOH/EtOAc), 0.58; $[\alpha]_D^{27}$ +8.99 (*c* 1.0, $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 4.30 (s, 1H, 4-**H**), 4.22 (s, 1H, 1-**H**), 4.18 (dd, 1H, *J* = 6.0, 11.2 Hz, 10-$\mathbf{H_a}$), 4.00-3.90 (m, 3H, 5-**H**, 6-**H**, 10-$\mathbf{H_b}$), 3.72 (dd, 1H, *J* = 6.0, 10.0 Hz, 11-$\mathbf{H_a}$), 3.59 (t, 1H, *J* = 8.8 Hz, 11-$\mathbf{H_b}$), 2.82 (d, 1H, *J* = 5.2 Hz, -O**H**), 2.78 (bs, 1H, -O**H**), 2.30 (t, 2H, *J* = 7.6 Hz, 15-**H**), 2.11 (ddd, 1H, *J* = 5.6, 8.8, 8.8 Hz, 2-**H**), 2.02 (ddd, 1H, *J* = 6.0, 8.4, 8.4 Hz, 3-**H**), 1.83 (bs, 1H, -O**H**), 1.65-1.59 (bs, 2H, 16-**H**, merged with water peak), 1.35-1.21 (m, 12H, 17-22-**H**), 0.88 (t, 3H, *J* = 6.8 Hz, 23-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.9, 84.7, 84.4, 74.2, 74.0, 62.2, 59.9, 42.9, 39.9, 34.3, 31.8, 29.4 (2C), 29.2, 29.1 , 24.9, 22.6, 14.1: IR (neat): 3472, 2954, 2920, 2850, 1714, 1459, 1214, 1177, 1128, 1048, 825, 725, 652, 504 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{18}H_{32}O_6Na$ $(M+Na)^+$ 367.2097, found $(M+Na)^+$ 367.2100.

General procedure for the preparation of (±)-2a-e:

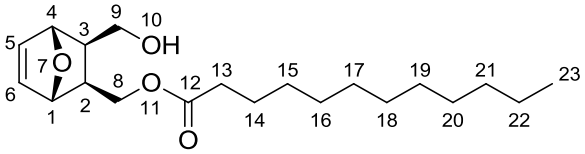
To a stirred solution of **1** (1.0 eq; Scheme 1, manuscript) and Et_3N (1.2 eq) in dry dichloromethane was added the appropriate acid chloride (1.0 eq) at 0 ^oC under N_2 atmosphere. The reaction mixture was warmed to room temperature and allowed to stir for 45mins. After completion of the reaction, the mixture was washed with water and extracted with dichloromethane. The organic layer was dried using sodium sulfate and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel using 10-20% EtOAc/Hexane in a gradient mode. Yields and spectroscopic details of various compounds synthesized are given below.

(3-(hydroxymethyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methyl palmitate ((±)-2a)



Colorless solid; Yield = 31%; R_f (50% EtOAc/Hexane), 0.40; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.44-6.38 (m, 2H, 5-**H**, 6-**H**), 4.90 (s, 1H, 4-**H**), 4.80 (s, 1H, 1-**H**), 4.31 (dd, 1H, *J* = 5.6, 10.8 Hz, 8-$\mathbf{H_a}$), 4.04 (t, 1H, *J* = 9.6 Hz, 8-$\mathbf{H_b}$), 3.84 (dd, 1H, *J* = 5.6, 10.4 Hz, 9-$\mathbf{H_a}$), 3.66 (t, 1H, *J* = 8.8 Hz, 9-$\mathbf{H_b}$), 2.33 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.05 (bs, 1H, -O**H**), 2.00-1.88 (m, 2H, 2-**H**, 3-**H**), 1.63 (quin, 2H, *J* = 6.8 Hz, 14-**H**), 1.28-1.21 (bs, 24H, 15-26-**H**), 0.88 (t, 3H, *J* = 6.8 Hz, 27-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 135.7, 135.4, 80.6, 80.5, 63.9, 61.9, 42.0, 39.2, 34.3, 31.9, 29.6 (6C), 29.4, 2 9.3, 29.2, 29.1, 24.9, 22.7, 14.1; IR (neat): 2920, 2853, 1728, 1465, 1162, 1106, 1024, 891, 848, 694, 575 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{24}H_{42}O_4Na$ $(M+Na)^+$ 417.2981 found $(M+Na)^+$ 417. 2990.

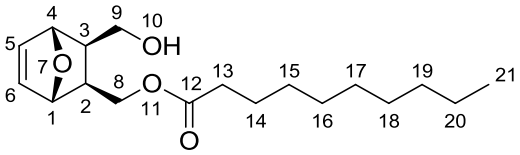
(3-(hydroxymethyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methyl dodecanoate ((±)-2b)



Colorless solid; Yield = 28%; R_f (40% EtOAc/Hexane), 0.40; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.42-6.38 (m, 2H, 5-**H**, 6-**H**), 4.90 (s, 1H, 4-**H**), 4.80 (s, 1H, 1-**H**), 4.31 (dd, 1H, *J* = 5.2, 10.8 Hz, 8-$\mathbf{H_a}$), 4.04 (t, 1H, *J* = 9.6 Hz, 8-$\mathbf{H_b}$), 3.84 (dd, 1H, *J* = 5.6, 10.4 Hz, 9-$\mathbf{H_a}$), 3.66 (t, 1H, *J* = 8.4 Hz, 9-$\mathbf{H_b}$), 2.33 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.00-1.89 (m, 2H, 2-**H**, 3-**H**), 1.65-1.54 (m, 2H, 14-**H**), 1.35-1.26 (bs, 16H, 15-22-**H**), 0.88 (t, 3H, *J* = 6.8 Hz, 24-**H**); -OH proton did not appear; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 135.7, 135.4, 80.6, 80.5, 63.9, 61.9, 42.0, 39.3, 34.3, 31.9, 29.6 (2C), 29.4, 2 9.3, 29.2, 29.1, 24.9, 22.6, 14.1; IR (neat): 3446, 2922, 2853, 1734, 1466, 1168, 1108, 1022,

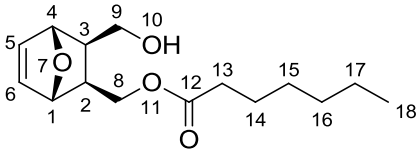
899, 689, 628 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{20}H_{34}O_4Na$ $(M+Na)^+$ 361.2355, found $(M+Na)^+$ 361.2347.

(3-(hydroxymethyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methyl decanoate ((±)-2c)



Colorless solid; Yield = 31%; R_f (50% EtOAc/Hexane), 0.40; ^1H NMR ($CDCl_3$, 400 MHz): δ 6.40-6.36 (m, 2H, 5-**H**, 6-**H**), 4.90 (s, 1H, 4-**H**), 4.79 (s, 1H, 1-**H**), 4.31 (dd, 1H, *J* = 5.2, 10.4 Hz, 8-$\mathbf{H_a}$), 4.04 (t, 1H, *J* = 9.6 Hz, 8-$\mathbf{H_b}$), 3.84 (dd, 1H, *J* = 5.6, 10.4 Hz, 9-$\mathbf{H_a}$), 3.65 (t, 1H, *J* = 8.4 Hz, 9-$\mathbf{H_b}$), 2.32 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.00-1.88 (m, 2H, 2-**H**, 3-**H**), 1.63 (quin, 2H, *J* = 7.2 Hz, 14-**H**), 1.35-1.21 (bs, 12H, 15-20-**H**), 0.88 (t, 3H, *J* = 6.8 Hz, 21-**H**); -OH proton did not appear; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 135.7, 135.4, 80.6, 80.5, 63.9, 61.9, 42.0, 39.3, 34.3, 31.8, 29.4, 29.2 (2C), 2 9.1, 24.9, 22.6, 14.1: IR (neat): 3445, 2924, 2854, 1734, 1466, 1311, 1164, 1107, 1025, 898, 688, 627 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{18}H_{30}O_4Na$ $(M+Na)^+$ 333.2042, found $(M+Na)^+$ 333.2037.

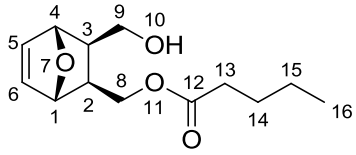
(3-(hydroxymethyl)-7-oxabicyclo[2.2.1]hept-5-en-2-yl)methyl heptanoate ((±)-2d)



Gummy liquid; Yield = 39%; R_f (50% EtOAc/Hexane), 0.36; ^1H NMR ($CDCl_3$, 400 MHz): δ 6.40 (dd, 1H, *J* = 1.2, 5.6 Hz, 6-**H**), 6.37 (dd, 1H, *J* = 1.6, 6.0 Hz, 5-**H**), 4.90 (s, 1H, 4-**H**), 4.80 (s, 1H, 1-**H**), 4.31 (dd, 1H, *J* = 5.6, 10.8 Hz, 8-$\mathbf{H_a}$), 4.03 (t, 1H, *J* = 10.0 Hz, 8-$\mathbf{H_b}$), 3.84 (dd, 1H, *J* = 5.6, 10.4 Hz, 9-$\mathbf{H_a}$), 3.66 (dd, 1H, *J* = 8.4, 10.0 Hz, 9-$\mathbf{H_b}$), 2.33 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.01-1.88 (m, 2H, 2-**H**, 3-**H**), 1.63 (quin, 2H, *J* = 7.2 Hz, 14-**H**), 1.40-1.26 (m, 6H, 15-17-**H**), 0.89 (t, 3H, *J* = 6.8 Hz, 18-**H**); -OH proton did not appear; ^{13}C NMR

($CDCl_3$, 100 MHz): δ 173.7, 135.7, 135.3, 80.55, 80.46, 63.9, 61.8, 41.9, 39.2, 34.3, 31.4, 28.8, 24.9, 22.4, 13.9: IR (neat): 3429, 2930, 2858, 1733, 1467, 1105, 1025, 972, 897, 689, 628 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{15}H_{24}O_4Na$ $(M+Na)^+$ 291.1572, found $(M+Na)^+$ 291.1563.

(3-(hydroxymethyl)-7-oxabicyclo [2.2.1]hept-5-en-2-yl)methyl pentanoate ((±)-2e)



Gummy liquid; Yield = 38%; R_f (20% EtOAc/Hexane), 0.43; ^1H NMR ($CDCl_3$, 400 MHz): δ 6.46-6.36 (m, 2H, 5-**H**, 6-**H**), 4.90 (s, 1H, 4-**H**), 4.80 (s, 1H, 1-**H**), 4.31 (dd, 1H, *J* = 5.6, 10.8 Hz, 8-$\mathbf{H_a}$), 4.04 (t, 1H, *J* = 10.0 Hz, 8-$\mathbf{H_b}$), 3.84 (dd, 1H, *J* = 5.6, 10.4 Hz, 9-$\mathbf{H_a}$), 3.66 (t, 1H, *J* = 8.4 Hz, 9-$\mathbf{H_b}$), 2.34 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.01-1.88 (m, 2H, 2-**H**, 3-**H**), 1.79 (bs, 1H, 10-**H**), 1.62 (quin, 2H, *J* = 7.2 Hz, 14-**H**), 1.35 (sext, 2H, *J* = 7.2 Hz, 15-**H**), 0.93 (t, 3H, *J* = 7.6 Hz, 16-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 135.7, 135.4, 80.6, 80.5, 63.9, 61.8, 42.0, 39.2, 34.0, 27.0, 22.2, 13.7: IR (neat): 3446, 2958, 2934, 2873, 1731, 1466, 1311, 1259, 1171, 1107, 1024, 897, 689, 628 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{13}H_{20}O_4Na$ $(M+Na)^+$ 263.1259, found $(M+Na)^+$ 263.1263.

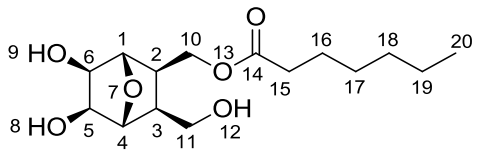
Preparation of Compounds (±)-3a-e

To a stirred solution containing a mixture of **2a-e** (1.0 eq), N-methyl morpholine N-Oxide (2.4 eq) and pyridine (30 uL for 100 mg of the alkene) in ^{t}BuOH-H_2O (3:1) was added osmium tetraoxide (0.02M solution in ^{t}BuOH, 0.01 eq). The reaction mixture was heated at 80 ^oC for 5-6 h. After completion of the reaction, the mixture was cooled to room temperature, treated with 15% aq. Na_2SO_3 solution (4 mL), allowed to stir for 5-10 min and water (8 mL) was added to it. All the solvents were then removed under reduced pressure and

the residue was extracted with ethyl acetate. The organic layer was dried using sodium sulfate, the solvent evaporated under reduced pressure and the residue was purified by column chromatography on silica gel using EtOAc-Hexane-MeOH solvent system in a gradient mode. Yields and spectroscopic details of various compounds synthesized are given below.

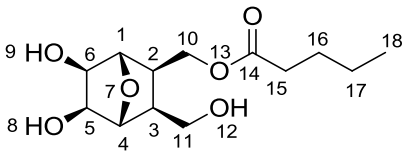
Since the compounds (±)-**3a, 3b** and **3c** respectively are racemic forms of (+)-**6a, 6b** & **6d**, they showed the same spectral data. The yields of these compounds through the racemic route (Scheme 1, manuscript) are: **3a** (87%), **3b** (74%) and **3c** (75%). The spectral data of the remaining compounds **3d** and **3e** are given below.

(5,6-dihydroxy-3-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptan-2-yl)methyl heptanoate (±)-3d



Colorless solid; Yield = 69%; R_f (10% MeOH/EtOAc), 0.52; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.35 (s, 1H, 4-**H**), 4.19 (s, 1H, 1-**H)**, 4.09 (dd, 1H, *J* = 6.8, 12.0 Hz, 10-$\mathbf{H_a}$), 3.95-3.89 (m, 3H, 5-**H**, 6-**H**, 10-$\mathbf{H_b}$), 3.65 (dd, 1H, *J* = 5.2, 10.4, 11-$\mathbf{H_a}$), 3.49 (t, 1H, *J* = 10.0 Hz, 11-$\mathbf{H_b}$), 2.31 (t, 2H, *J* = 7.2 Hz, 15-**H**), 2.13-1.98 (m, 2H, 2-**H**, 3-**H**), 1.61 (quin, 2H, *J* = 6.8 Hz, 16-**H**), 1.27 (bs, 6H, 17-19-**H**), 0.89 (t, 3H, *J* = 6.4 Hz, 20-**H**); Three -OH protons did not appear; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.9, 84.6, 84.3, 74.2, 73.9, 62.1, 59.8, 42.9, 39.8, 34.2, 31.4, 28.7, 24.8, 22.4, 13.9: IR (neat): 3386, 2929, 2858, 1731, 1457, 1233, 1168, 1121, 1063, 1027, 912, 820, 785, 640 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{15}H_{26}O_6Na$ $(M+Na)^+$ 325.1627, found $(M+Na)^+$ 325.1627.

(5,6-dihydroxy-3-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptan-2-yl)methyl pentanoate (±)-3e

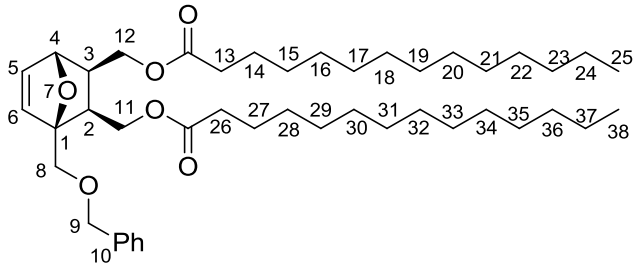


Colorless solid; Yield = 75%; R_f (10% MeOH/EtOAc), 0.52; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.57 (bs, 1H, -**OH**), 4.35 (s, 1H, 4-**H**), 4.19 (s, 2H, -OH, 1-**H**), 4.08 (dd, 1H, *J* = 5.6, 10.8 Hz, 10-$\mathbf{H_a}$), 4.00-3.90 (m, 3H, 5-**H**, 6-**H**, 10-$\mathbf{H_b}$), 3.65 (dd, 1H, *J* = 5.2, 10.8 Hz, 11-$\mathbf{H_a}$), 3.49 (t, 1H, *J* = 10.0 Hz, 11-$\mathbf{H_b}$), 2.32 (t, 2H, *J* = 7.2 Hz, 15-**H**), 2.14-1.98 (m, 2H, 2-**H**, 3-**H**), 1.59 (quin, 2H, *J* = 7.6 Hz, 16-**H**), 1.34 (sext, 2H, *J* = 7.2 Hz, 17-**H**), 0.92 (t, 3H, *J* = 7.2 Hz, 18-**H**); one -OH proton did not appear; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.9, 84.6, 84.3, 74.2, 73.9, 62.1, 59.8, 42.9, 39.8, 33.9, 26.9, 22.2, 13.6: IR (neat): 3446, 2956, 2930, 2858, 1735, 1262, 1170, 1032, 764, 750, 698 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{13}H_{22}O_6Na$ $(M+Na)^+$ 297.1314, found $(M+Na)^+$ 297.1305.

General procedure for the preparation of compounds (±)-11a-f:

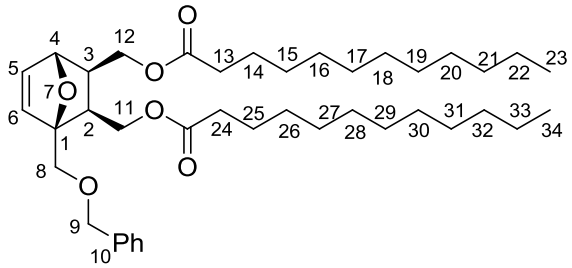
To a stirred solution of the alcohol **10** (1.0 eq) and Et_3N (3.0 eq) in dry dichloromethane was added the appropriate acid chloride (2.2 eq) at 0 ^oC under N_2 atmosphere. The reaction mixture was warmed to room temperature and allowed to stir for 45mins. After completion of the reaction, the mixture was washed with water and extracted with dichloromethane. The organic layer was dried using sodium sulfate and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel using 10-20% EtOAc/Hexane. Yields and spectroscopic details of various compounds synthesized are given below.

(1-((benzyloxy)methyl)-7-oxabicyclo[2.2.1]hept-5-ene-2,3-diyl)bis(methylene) ditetradecanoate ((±)-11a)



Colorless solid; Yield = 49%; R_f (20% EtOAc/Hexane), 0.50; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.28 (m, 5H, 10-**H**), 6.41-6.36 (m, 2H, 5-**H**, 6-**H**), 4.82 (d, 1H, *J* = 1.2 Hz, 4-**H**), 4.69 (d, 1H, *J* = 12.4 Hz, 9-$\mathbf{H_a}$), 4.55 (d, 1H, *J* = 12.4 Hz, 9-$\mathbf{H_b}$), 4.42-4.37 (m, 1H, 11-$\mathbf{H_a}$), 4.17 (dd, 1H, *J* = 7.2, 11.2 Hz, 12-$\mathbf{H_a}$), 4.12 (dd, 1H, *J* = 6.4, 11.2 Hz, 12-$\mathbf{H_b}$), 4.00-3.94 (m, 1H, 11-$\mathbf{H_b}$), 3.92 (d, 1H *J* = 10.8 Hz, 8-$\mathbf{H_a}$), 3.75 (d, 1H, *J* = 10.8 Hz, 8-$\mathbf{H_b}$), 2.33 (t, 2H, *J* = 7.6 Hz, 26-**H**), 2.24 (t, 2H, *J* = 7.2 Hz, 13-**H**), 2.14-2.06 (m, 2H, 2-**H**, 3-**H**), 1.64-1.55 (m, 4H, 14-**H**, 27-**H**), 1.26 (bs, 40H, 15-24-**H**, 28-37-**H**), 0.88 (t, 6H, *J* = 6.4 Hz, 25-**H**, 38-**H**); ^{13}C NMR ($CDCl_3$, 100 Hz): δ 173.6, 173.4, 137.8, 137.3, 135.5, 128.4 (2), 127.9, 127.8 (2), 89.9, 80.4, 73.7, 67.7, 63 .6, 62.0, 41.3, 40.7, 34.3, 31.9 (2C), 29.6 (9C), 29.5 (2C), 29.34 (2C), 29.25 (2C), 29.1 (2C), 24.9, 24.8, 22.7 (2C), 14.1 (2C): IR (neat): 2958, 2932, 2872, 1733, 1454, 1255, 1167, 1092, 736, 697 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{44}H_{72}O_6Na$ $(M+Na)^+$ 719.5227, found $(M+Na)^+$ 719.5203.

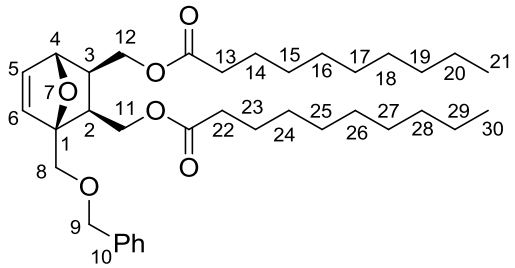
(1-(benzyloxymethyl)-7-oxabicyclo[2.2.1]hept-5-ene-2,3-diyl)bis(methylene) didodecanoate ((±)-11b)



Colorless solid; Yield = 61%; R_f (20% EtOAc/Hexane), 0.50; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.27 (m, 5H, 10-**H**), 6.40-6.36 (m, 2H, 5-**H**, 6-**H**), 4.82 (s, 1H, 4-**H**), 4.69 (d,

1H, *J* = 12.0 Hz, 9-**H$_a$**), 4.55 (d, 1H, *J* = 12.0 Hz, 9-**H$_b$**), 4.41-4.37 (m, 1H, 11-**H$_a$**), 4.20-4.08 (m, 1H, 12-**H$_a$**), 4.13-4.08 (m, 1H, 12-**H$_b$**), 3.98 (t, 1H, *J* = 10.0 Hz, 11-**H$_b$**), 3.92 (d, 1H *J* = 10.8 Hz, 8-**H$_a$**), 3.75 (d, 1H, *J* = 10.8 Hz, 8-**H$_b$**), 2.33 (t, 2H, *J* = 7.2 Hz, 24-**H**), 2.24 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.15-2.07 (m, 2H, 2-**H**, 3-**H**), 1.68-1.54 (m, 4H, 14-**H**, 25-**H**), 1.26 (bs, 32H, 15-22-**H**, 26-33-**H**), 0.88 (t, 6H, *J* = 6.4 Hz, 23-**H**, 34-**H**); ^{13}C NMR ($CDCl_3$, 100 Hz): δ 173.5, 173.3, 137.8, 137.4, 135.5, 128.4 (2C), 128.1, 127.7 (2), 89.9, 80.4, 73.7, 67.7, 63.6, 61.9, 41.3, 40.7, 36.6, 34.3, 31.9 (2C), 29.6 (4C), 29.4 (2C), 29.3 (2C), 29.2 (2C), 29.1 (2C), 24.9, 24.8, 22.6 (2C), 14.1 (2C): IR (neat): 2925, 2857, 1737, 1713, 1460, 1168, 1111, 731 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{40}H_{65}O_6$ $(M+H)^+$ 641.4781, found $(M+H)^+$ 641.4794.

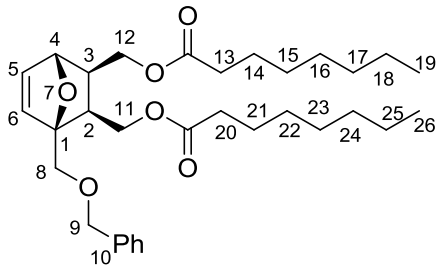
(1-(benzyloxymethyl)-7-oxabicyclo[2.2.1]hept-5-ene-2,3-diyl)bis(methylene) bis(decanoate) ((±)-11c)



Colorless solid; Yield = 92%; R_f (20% EtOAc/Hexane), 0.50; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.27 (m, 5H, 10-**H**), 6.40-6.36 (m, 2H, 5-**H**, 6-**H**), 4.82 (s, 1H, 4-**H**), 4.69 (d, 1H, *J* = 12.0 Hz, 9-**H$_a$**), 4.55 (d, 1H, *J* = 12.0 Hz, 9-**H$_b$**), 4.39 (dd, 1H, *J* = 4.0, 10.4 Hz, 11-**H$_a$**), 4.18 (dd, 1H, *J* = 6.8, 11.2 Hz, 12-**H$_a$**), 4.11 (dd, 1H, *J* = 6.4, 11.6 Hz, 12-**H$_b$**), 3.98 (t, 1H, *J* = 10.4 Hz, 11-**H$_b$**), 3.92 (d, 1H, *J* = 10.8 Hz, 8-**H$_a$**), 3.75 (d, 1H, *J* = 10.8 Hz, 8-**H$_b$**), 2.33 (t, 2H, *J* = 7.2 Hz, 22-**H**), 2.24 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.13-2.08 (m, 2H, 2-**H**, 3-**H**), 1.66-1.54 (m, 4H, 14-**H**, 23-**H**), 1.26 (bs, 24H, 15-20-**H**, 24-29-**H**), 0.88 (t, 6H, *J* = 6.4 Hz, 21-**H**, 30-**H**); ^{13}C NMR ($CDCl_3$, 100 Hz): δ 173.6, 173.4, 137.8, 137.4, 135.5, 128.4 (2C), 127.9 (2C), 127.8, 89.9, 80.4, 73.7, 67.8, 63.

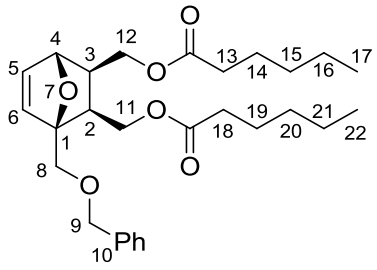
6, 61.9, 41.4, 40.7, 34.3 (2C), 31.8 (2C), 29.4 (2C), 29.2 (3C), 29.1 (2C), 25.0, 24.8, 24.7, 22.6 (2C), 14.1 (2C): IR (neat): 2927, 2861, 1731, 1516, 1458, 1368, 1170, 988, 714 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{36}H_{56}O_6Na$ $(M+Na)^+$ 607.3975, found $(M+Na)^+$ 607.3984.

(1-(benzyloxymethyl)-7-oxabicyclo[2.2.1]hept-5-ene-2,3-diyl)bis(methylene) dioctanoate ((±)-11d)



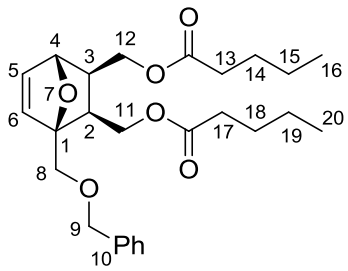
Gummy liquid; Yield = 78%; R_f (20% EtOAc/Hexane), 0.46; ^1H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.28 (m, 5H, 10-**H**), 6.40-6.36 (m, 2H, 5-**H**, 6-**H**), 4.82 (s, 1H, 4-**H**), 4.69 (d, 1H, *J* = 12.0 Hz, 9-$\mathbf{H_a}$), 4.55 (d, 1H, *J* = 12.4 Hz, 9-$\mathbf{H_b}$), 4.39 (dd, 1H, *J* = 4.0, 10.4 Hz, 11-$\mathbf{H_a}$), 4.18 (dd, 1H, *J* = 7.2, 11.6 Hz, 12-$\mathbf{H_a}$), 4.13-4.08 (m, 1H, 12-$\mathbf{H_b}$), 3.98 (t, 1H, *J* = 10.4, 11-$\mathbf{H_b}$), 3.92 (d, 1H, *J* = 10.8 Hz, 8-$\mathbf{H_a}$), 3.75 (d, 1H, *J* = 10.8 Hz, 8-$\mathbf{H_b}$), 2.33 (t, 2H, *J* = 7.6 Hz, 20-**H**), 2.24 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.13-2.08 (m, 2H, 2-**H**, 3-**H**), 1.66-1.55 (m, 4H, 14-**H**, 21-**H**), 1.30-1.27 (m, 16H, 15-18-**H**, 22-25-**H**), 0.88 (t, 6H, *J* = 6.4 Hz, 19-**H**, 26-**H**); ^{13}C NMR ($CDCl_3$, 100 Hz): δ 173.6, 173.3, 137.8, 137.4, 135.5, 128.4 (2C), 127.8 (2C), 127.7, 89.9, 80.4, 73.7, 67.7, 63.6, 61.9, 41.3, 40.7, 34.3 (2C), 31.6 (2C), 29.1 (2C), 28.9 (2C), 24.9, 24.8, 22.6 (2C), 14.0 (2C): IR (neat): 2928, 2861, 1731, 1459, 1277, 1168, 1109, 1026, 713 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{32}H_{48}O_6Na$ $(M+Na)^+$ 551.3349, found $(M+Na)^+$ 551.3345.

(1-(benzyloxymethyl)-7-oxabicyclo[2.2.1]hept-5-ene-2,3-diyl)bis(methylene) dihexanoate ((±)-11e)



Gummy liquid; Yield = 85%; R_f (20% EtOAc/Hexane), 0.46; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.27 (m, 5H, 10-**H**), 6.40-6.36 (m, 2H, 5-**H**, 6-**H**), 4.82 (s, 1H, 4-**H**), 4.70 (d, 1H, *J* = 12.0 Hz, 9-$\mathbf{H_a}$), 4.55 (d, 1H, *J* = 12.4 Hz, 9-$\mathbf{H_b}$), 4.39 (dd, 1H, *J* = 4.0, 10.8 Hz, 11-$\mathbf{H_a}$), 4.19 (dd, 1H, *J* = 6.8, 11.2 Hz, 12-$\mathbf{H_a}$), 4.11 (dd, 1H, *J* = 6.0, 11.2 Hz, 12-$\mathbf{H_b}$), 4.01-3.96 (m, 1H, 11-$\mathbf{H_b}$), 3.92 (d, 1H, *J* = 10.8 Hz, 8-$\mathbf{H_a}$), 3.75 (d, 1H, *J* = 10.8 Hz, 8-$\mathbf{H_b}$), 2.33 (t, 2H, *J* = 7.6 Hz, 18-**H**), 2.24 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.13-2.09 (m, 2H, 2-**H**, 3-**H**), 1.67-1.55 (m, 4H, 14-**H**, 19-**H**), 1.34-1.26 (m, 8H, 15-**H**, 16-**H**, 20-**H**, 21-**H**), 0.92-0.87 (m, 6H, 17-**H**, 22-**H**); ^{13}C NMR ($CDCl_3$, 100 Hz): δ 173.6, 173.4, 137.8, 137.4, 135.5, 128.4, 127.8, 127.7, 89.9, 80.4, 73.7, 67.7, 63.6, 61.9, 41.3, 40.7, 34.25, 34.21, 33.9, 31.22, 31.16, 24.6, 24.5, 24.3, 22.2 (2C), 13.8 (2C): IR (neat): 2930, 2864, 1732, 1459, 1171, 1107, 1031, 709 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{28}H_{41}O_6$ $(M+H)^+$ 473.2903, found $(M+H)^+$ 473.2896.

(1-(benzyloxymethyl)-7-oxabicyclo[2.2.1]hept-5-ene-2,3-diyl)bis(methylene) dipentanoate ((±)-11f)



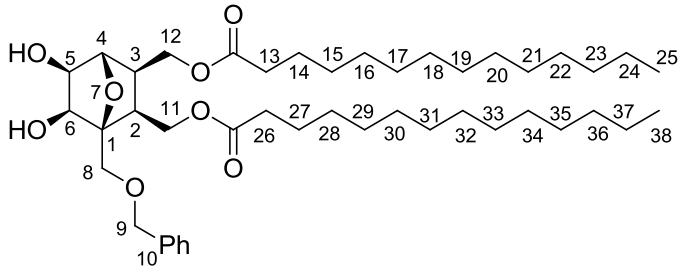
Gummy liquid; Yield = 89%; R_f (20% EtOAc/Hexane), 0.46; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.28 (m, 5H, 10-**H**), 6.40-6.36 (m, 2H, 5-**H**, 6-**H**), 4.82 (d, 1H, *J* = 1.20 Hz, 4-**H**), 4.69 (d, 1H, *J* = 12.4 Hz, 9-$\mathbf{H_a}$), 4.56 (d, 1H, *J* = 12.4 Hz, 9-$\mathbf{H_b}$), 4.41-4.38 (m, 1H, 11-

H_a), 4.18 (dd, 1H, *J* = 7.2, 11.2 Hz, 12-**H_a**), 4.11 (dd, 1H, *J* = 6.4, 11.2 Hz, 12-**H_b**), 4.00-3.95 (m, 1H, 11-**H_b**), 3.92 (d, 1H, *J* = 10.8 Hz, 8-**H_a**), 3.75 (d, 1H, *J* = 10.8 Hz, 8-**H_b**), 2.34 (t, 2H, *J* = 7.6 Hz, 17-**H**), 2.25 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.14-2.07 (m, 2H, 2-**H**, 3-**H**), 1.66-1.53 (m, 4H, 14-**H**, 18-**H**), 1.40-1.26 (m, 4H, 15-**H**, 19-**H**), 0.94-0.88 (m, 6H, 16-**H**, 20-**H**); ^{13}C NMR ($CDCl_3$, 100 Hz): δ 173.6, 173.4, 137.9, 137.4, 135.6, 128.5 (2C), 127.9 (2C), 127.8, 89.9, 80.5, 73.8, 67.8, 63.7, 62.0, 41.4, 40.8, 34.1, 34.0, 27.1, 26.9, 22.3 (2C), 13.7 (2C): IR (neat): 2955, 2928, 2858, 1735, 1455, 1362, 1237, 1162, 1099, 984, 735 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{26}H_{36}O_6Na$ $(M+Na)^+$ 467.2410, found $(M+Na)^+$ 467.2387.

General procedure for the dihydroxylation of (±)-11a-g:

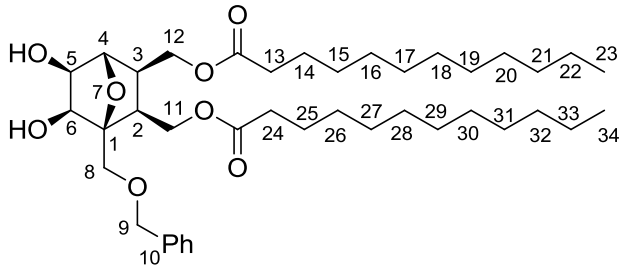
To a stirred solution containing a mixture of the alkene ((±)-**11a-g**; 1.0 eq), N-Methyl morpholine N-Oxide (2.4 eq) and pyridine (30 uL for 100 mg of alkene) in ^{t}BuOH-H_2O (3:1) was added osmium tetraoxide (0.01eq, 0.02M solution) and it was heated at 80 ^{o}C for 5-6 h. After completion of the reaction, the mixture was cooled to room temperature, treated with 15% aq. Na_2SO_3 solution (4 mL), stirred for 5-10 minutes and water (8 mL) was added to it. All the volatiles were removed under reduced pressure and the resulting residue was extracted with ethyl acetate. The organic layer was dried using sodium sulfate and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel using Hexane-EtOAc solvent system. Yields and spectroscopic details of various compounds synthesized are given below.

(1-(benzyloxymethyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) ditetradecanoate ((±)-12a)



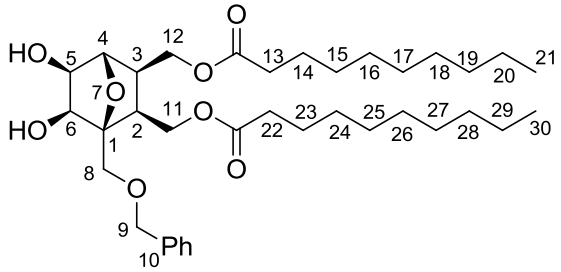
Colorless solid; Yield = 71%; R_f (40% EtOAc-Hexane), 0.34; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.39-7.30 (m, 5H, 10-**H**), 4.66 (d, 1H, *J* = 11.6 Hz, 9-$\mathbf{H_a}$), 4.58 (d, 1H, *J* = 12.0 Hz, 9-$\mathbf{H_b}$), 4.26 (s, 1H, 4-**H**), 4.25 (dd, 1H, 11-$\mathbf{H_a}$, merged with 4-**H**), 4.18 (dd, 1H, *J* = 6.8, 11.6 Hz, 12-$\mathbf{H_a}$), 4.05-4.00 (m, 2H, -**OH**, 12-$\mathbf{H_b}$), 3.97 (m, 1H, 11-$\mathbf{H_b}$), 3.92 (s, 2H, 5-**H**, 6-**H**), 3.94-3.88 (m, 2H, 8-**H**), 3.35 (d, 1H, *J* = 5.6 Hz, -O**H**), 2.30 (t, 2H, *J* = 7.6 Hz, 26-**H**), 2.25 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.23-2.17 (m, 1H, 3-**H**), 2.14-2.08 (m, 1H, 2-**H**), 1.63-1.55 (m, 4H, 14-**H**, 27-**H**, merged with water peak), 1.26 (bs, 40H, 15-24-**H**, 28-37-**H**), 0.88 (t, 6H, *J* = 6.4 Hz, 25-**H**, 38-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.5, 173.2, 136.9, 128.6 (2C), 128.1, 127.9 (2C), 86.7, 84.1, 75.6, 74.2, 73.9, 66.7 , 61.9, 60.7, 41.9, 40.9, 34.2 (2C), 31.9 (2C), 29.60 (4C), 29.57 (2C), 29.50 (2C), 29.43 (2C), 29.3 (2C), 29.2 (2C), 29.1 (2C), 24.9, 24.8, 22.6 (2C), 14.1 (2C): IR (neat): 3434, 2923, 2856, 1731, 1461, 1361, 1247, 1163, 1113, 736 cm^{-1}. HRMS (ESI) exact mass calcd. for $C_{44}H_{74}O_8Na$ $(M+Na)^+$ 753.5281, found $(M+Na)^+$ 753.5288.

(1-(benzyloxymethyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) didodecanoate ((±)-12b)



Colorless solid; Yield = 73%; R_f (40% EtOAc/Hexane), 0.34; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.40-7.32 (m, 5H, 10-**H**), 4.67 (d, 1H, *J* = 11.6 Hz, 9-$\mathbf{H_a}$), 4.57 (d, 1H, *J* = 11.6 Hz, 9-$\mathbf{H_b}$), 4.26 (s, 1H, 4-**H**), 4.24 (dd, 1H, 11-$\mathbf{H_a}$, merged with 4-**H**), 4.17 (dd, 1H, *J* = 7.2, 11.6 Hz, 12-$\mathbf{H_a}$), 4.09 (d, 1H, *J* = 4.8 Hz, -**OH**), 4.03-4.00 (m, 1H, 11-$\mathbf{H_b}$), 3.99-3.96 (m, 1H, 12-$\mathbf{H_b}$), 3.93-3.92 (m, 2H, 5-**H**, 6-**H**), 3.91-3.87 (m, 2H, 8-**H**), 3.32 (d, 1H, *J* = 5.6 Hz, -O**H**), 2.30 (t, 2H, *J* = 7.2 Hz, 24-**H**), 2.25 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.22-2.17 (m, 1H, 3-**H**), 2.15-2.09 (m, 1H, 2-**H**), 1.65-1.55 (bs, 4H, 14-**H**, 25-**H**, merged with water peak), 1.26 (bs, 32H, 15-22-**H**, 26-33-**H**), 0.88 (t, 6H, *J* = 6.4 Hz, 23-**H**, 34-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.6, 173.3, 136.9, 128.6 (2C), 128.2, 128.0 (2C), 86.6, 84.1, 75.8, 74.3, 73.9, 66.9 , 61.9, 60.6, 42.1, 40.9, 34.3 (2C), 31.9 (2C), 29.6 (4C), 29.5 (2C), 29.3 (2C), 29.2 (2C), 29.1 (2C), 24.9, 24.8, 22.7 (2C), 14.1 (2C): IR (neat): 3441, 3387, 2956, 2921, 2851, 1735, 1725, 1469, 1367, 1249, 1169, 1111, 1074, 1012, 742 cm^{-1}**.** HRMS (ESI) exact mass calcd. for $C_{40}H_{67}O_8$ $(M+H)^+$ 675.4836, found $(M+H)^+$ 675.4852.

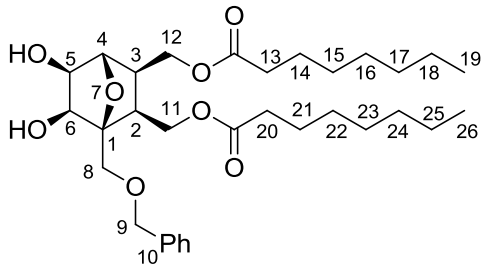
(1-(benzyloxymethyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) bis(decanoate) ((±)-12c)



Colorless solid; Yield = 76%; R_f (40% EtOAc/Hexane), 0.35; NMR ($CDCl_3$, 400 MHz): δ 7.39-7.32 (m, 5H, 10-**H**), 4.65 (d, 1H, *J* = 11.6 Hz, 9-$\mathbf{H_a}$), 4.57 (d, 1H, *J* = 11.6 Hz, 9-$\mathbf{H_b}$), 4.26 (s, 1H, 4-**H**), 4.25 (dd, 1H, 11-$\mathbf{H_a}$, merged with 4-**H**), 4.19 (dd, 1H, *J* = 6.8, 11.2 Hz, 12-$\mathbf{H_a}$), 4.06 (d, 1H, *J* = 4.4 Hz, -**OH**), 4.03-4.01 (m, 1H, 12-$\mathbf{H_b}$), 3.98-3.95 (m, 1H, 11-$\mathbf{H_b}$), 3.94-3.89 (m, 2H, 8-**H**), 3.91 (s, 2H, 5-**H**, 6-**H**), 3.38 (bs, 1H, -**OH**), 2.30 (t, 2H, *J* = 7.6 Hz, 22-**H**), 2.25 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.22 (ddd, 1H, 3**H** - merged with 13-**H**), 2.15-2.07

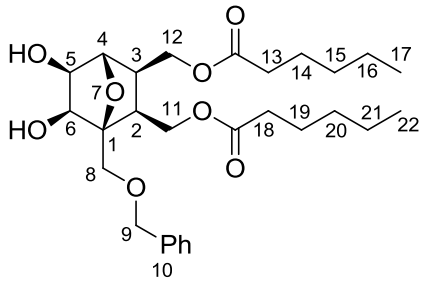
(m, 1H, 2-**H**), 1.65-1.58 (m, 4H, 14-**H**, 23-**H** - merged with water), 1.28 (bs, 24H, 15-20-**H**, 24-29-**H**), 0.88 (t, 6H, *J* = 6.4 Hz, 21-**H**, 30-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.6, 173.3, 136.8, 128.6 (2C), 128.2, 128.0 (2C), 86.6, 84.1, 75.8, 74.3, 73.9, 66.9 , 61.9, 60.6, 42.1, 40.9, 34.2 (2C), 31.8 (2C), 29.4 (2C), 29.2 (4C), 29.1 (2C), 24.9, 24.8, 22.6 (2C), 14.1 (2C): IR (neat): 3435, 2924, 2856, 1731, 1461, 1247, 1164, 1113, 832, 743 cm^{-1}. HRMS (ESI) exact mass calcd. for $C_{36}H_{59}O_8$ $(M+H)^+$ 619.4210, found $(M+H)^+$ 619.4211.

(1-(benzyloxymethyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) dioctanoate ((±)-12d)



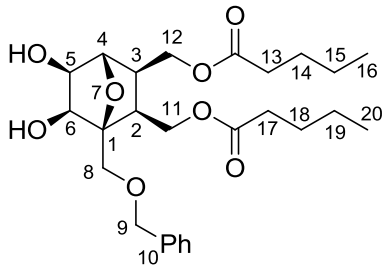
Colorless solid; Yield = 86%; R_f (40% EtOAc/Hexane), 0.34; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.38-7.29 (m, 5H, 10-**H**), 4.64 (d, 1H, *J* = 12.0 Hz, 9-$\mathbf{H_a}$), 4.57 (d, 1H, *J* = 12.0 Hz, 9-$\mathbf{H_b}$), 4.25 (s, 1H, 4-**H**), 4.27-4.21 (m, 2H, 11-$\mathbf{H_a}$, 12-$\mathbf{H_a}$), 4.00 (dd, 1H, *J* = 6.4, 9.2 Hz, 12-$\mathbf{H_b}$), 3.93 (m, 1H, 11-$\mathbf{H_b}$ - merged with 5H), 3.91 (s, 2H, 5-**H**, 6-**H**), 3.89-3.86 (m, 2H, 8-**H**), 3.46 (bs, 1H, -**OH**; the other -OH did not appear), 2.30 (t, 2H, *J* = 7.5 Hz, 20-**H**), 2.25 (t, 2H, *J* = 7.5 Hz, 13-**H**), 2.19 (ddd, 1H, *J* = 5.0, 7.2, 7.2 Hz, 3-**H**), 2.13-2.08 (m, 1H, 2-**H**), 1.60 (quin, 4H, *J* = 6.8 Hz, 14-**H**, 21-**H**), 1.31-1.28 (m, 16H, 15-18-**H**, 22-25-**H**), 0.89 (t, 6H, *J* = 7.0 Hz, 19-**H**, 26-**H**); ^{13}C NMR (CDCl3, 125 MHz): δ 173.5, 173.2, 137.0, 128.6 (2C), 128.1, 128.0 (2C), 86.8, 84.1, 75.6, 74.3, 74.0, 66.7, 62.0, 60.7, 42.0, 41.0, 34.2 (2C), 31.6 (2C), 29.1 (2C), 28.8 (2C), 24.9, 24.8, 22.5 (2C), 14.0 (2C); IR (neat): 3412, 2928, 2857, 1739, 1456, 1364, 1166, 1116, 1072, 738, 699 cm^{-1}. HRMS (ESI) exact mass calcd. for $C_{32}H_{50}O_8Na$ $(M+Na)^+$ 585.3403, found $(M+Na)^+$ 585.3408.

(1-((benzyloxy)methyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) dihexanoate ((±)-12e)



White crystalline solid; Yield = 85%; R_f (40% EtOAc/Hexane), 0.34; Mp. 88-89°C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.39-7.32 (m, 5H, 10-**H**), 4.66 (d, 1H, *J* = 11.6 Hz, 9-$\mathbf{H_a}$), 4.58 (d, 1H, *J* = 11.6 Hz, 9-$\mathbf{H_b}$), 4.26 (s, 1H, 4-**H**), 4.27-4.23 (m, 1H, 11-$\mathbf{H_a}$ - merged with 4-**H**), 4.19 (dd, 1H, *J* = 6.8, 11.2 Hz, 12-$\mathbf{H_a}$), 4.04-3.99 (m, 2H, -**OH**, 12-$\mathbf{H_b}$), 3.97-3.94 (m, 1H, 11-$\mathbf{H_b}$), 3.92 (s, 2H, 5-**H**, 6-**H**), 3.91-3.88 (m, 2H, 8-**H**), 3.27 (d, 1H, *J* = 5.7 Hz, -OH), 2.30 (t, 2H, *J* = 7.2 Hz, 18-**H**), 2.25 (t, 2H, *J* = 7.6 Hz, 13-**H**), 2.23-2.16 (m, 1H, 3-**H** - merged with 13-**H**), 2.14-2.08 (m, 1H, 2-**H**), 1.66-1.56 (m, 4H, 14-**H**, 19-**H**), 1.34-1.24 (m, 8H, 15-**H**, 16-**H**, 20-**H**, 21-**H**), 0.91-0.87 (m, 6H, 17-**H**, 22-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.5, 173.2, 136.7, 128.6 (2C), 128.2, 128.0 (2C), 86.5, 84.1, 75.8, 74.3, 73.9, 66.9 , 61.9, 60.6, 42.1, 40.8, 34.2 (2C), 31.2 (2C), 24.6, 24.5, 22.3 (2C), 13.9 (2C): IR (neat): 3423, 2959, 2933, 2872, 1735, 1266, 1172, 1100, 742, 704 cm^{-1}. HRMS (ESI) exact mass calcd. for $C_{28}H_{42}O_8Na$ $(M+Na)^+$ 529.2777, found $(M+Na)^+$ 529.2797.

(1-(benzyloxymethyl)-5,6-dihydroxy-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) dipentanoate: ((±)-12f)

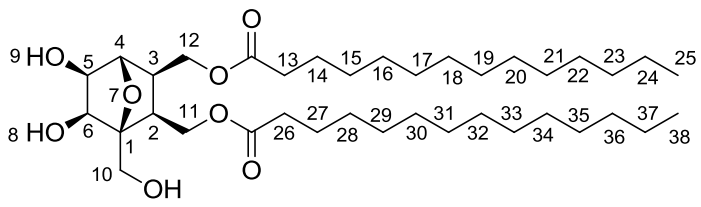


Colorless solid; Yield = 84%; R_f (40% EtOAc/Hexane), 0.33; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.39-7.30 (m, 5H, 10-**H**), 4.66 (d, 1H, *J* = 11.6 Hz, 9-$\mathbf{H_a}$), 4.57 (d, 1H, *J* = 11.6 Hz, 9-$\mathbf{H_b}$), 4.26 (s, 1H, 4-**H**), 4.25 (dd, 1H, 11-$\mathbf{H_a}$ - merged with 4-**H**), 4.20 (dd, 1H, *J* = 6.8, 11.6 Hz, 12-$\mathbf{H_a}$), 4.03-3.98 (m, 1H, 12-$\mathbf{H_b}$), 3.97-3.93 (m, 1H, 11- $\mathbf{H_b}$), 3.92-3.89 (m, 5H, -**OH**, 5-**H**, 6-**H**, 8-**H**), 3.42 (bs, 1H, -**OH**), 2.31 (t, 2H, *J* = 7.6 Hz, 17-**H**), 2.26 (t, 2H, *J* = 7.2 Hz, 13-**H**), 2.24-2.17 (m, 1H, 3-**H**), 2.15-2.08 (m, 1H, 2-**H**), 1.64-1.53 (m, 4H, 14-**H**, 18-**H** - merged with water peak), 1.39-1.27 (m, 4H, 15-**H**, 19-**H**), 0.94-0.89 (m, 6H, 16-**H**, 20-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.5, 173.2, 136.9, 128.5 (2C), 128.0,127.9 (2C), 86.8, 84.0, 75.4, 74.2, 73.9, 66.5, 61.9, 60.8, 41.8, 40.9, 33.9 (2C), 26.9, 26.8, 22.1 (2C), 13.6 (2C): IR (neat): 3404, 2960, 2871, 1734, 1460, 1266, 1173, 910, 742 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{26}H_{38}O_8Na$ $(M+Na)^+$ 501.2464, found $(M+Na)^+$ 501.2466.

General procedure the deprotection of Benzyl groups from (±)-12a-f:

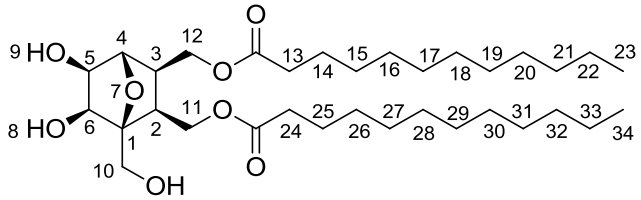
A mixture of the benzyl ether (**12a-f**) and 10% Pd/c (20% w/w) in $CHCl_3$ was stirred under hydrogen atmosphere (H_2 balloon) at room temperature for 5 h. After completion of the reaction, the resulting mixture was filtered through a celite bed and the solvent was evaporated under reduced pressure. The resulting solid was purified by column chromatography using EtOAc-Hexanes as solvent system in a gradient mode. Yields and spectroscopic details of various compounds synthesized are given below.

(5,6-dihydroxy-1-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) ditetradecanoate ((±)-13a)



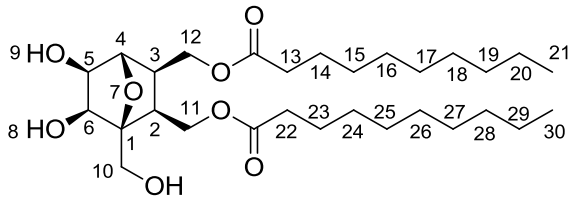
Colorless solid; Yield = 86%; R_f (EtOAc), 0.60; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.28 (d, 1H, *J* = 4.8 Hz, -O**H**), 4.24 (s, 1H, 4-**H**), 4.21 (dd, 1H, *J* = 5.6, 11.2 Hz, 12-$\mathbf{H_a}$), 4.30-3.98 (m, 4H, 10-**H**, 11-**H**), 3.96-3.91 (m, 3H, 5-**H**, 6-**H**, 12-$\mathbf{H_b}$), 3.40 (d, 1H, *J* = 5.6 Hz, -**OH**), 2.96 (bs, 1H, -**OH**), 2.34-2.28 (m, 4H, 13-**H**, 26-**H**), 2.24 (ddd, 1H, *J* = 5.6, 9.6, 9.6 Hz, 3-**H**), 2.15-2.09 (m, 1H, 2-**H**), 1.64-1.58 (m, 4H, 14-**H**, 27-**H**), 1.30-1.24 (m, 40H, 15-24-**H**, 28-37-**H**), 0.88 (t, 6H, *J* = 6.4 Hz, 25-**H**, 38-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.6, 173.2, 87.7, 83.6, 76.3, 74.0, 61.8, 60.6, 60.1, 41.9, 41.0, 34.3 (2C), 31.9 (2C), 29.7 (8C), 29.5 (2C), 29.4 (2C), 29.3 (2C), 29.2 (2C), 24.92, 24.85, 22.7 (2C), 14.1 (2C); IR (neat): 3354, 2921, 2852, 1727, 1264, 1112, 1087, 1020, 743 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{37}H_{68}O_8Na$ $(M+Na)^+$ 663.4812, found $(M+Na)^+$ 663.4830.

(5,6-dihydroxy-1-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) didodecanoate ((±)-13b)



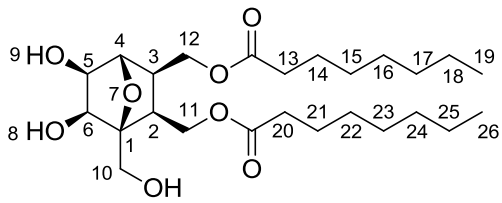
Colorless solid; Yield = 72%; R_f (EtOAc), 0.60; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.24 (s, 1H, 4-**H**), 4.21 (dd, 1H, *J* = 5.6, 10.8 Hz, 12-$\mathbf{H_a}$), 4.17-4.08 (m, 5H, 11-**H**, 10-**H, -OH**), 4.03-3.90 (m, 3H, 12-$\mathbf{H_b}$, 5-**H**, 6-**H**), 3.22 (bs, 1H, -**OH**), 2.72 (bs, 1H, -**OH**), 2.33-2.29 (m, 4H, 13-**H**, 24-**H**), 2.24 (ddd, 1H, *J* = 6.4, 10.0, 10.0 Hz, 3-**H**), 2.16-2.10 (m, 1H, 2-**H**), 1.65-1.59 (m, 4H, 14-**H**, 25-**H** - merged with water peak), 1.26 (bs, 32H, 15-22-**H**, 26-33-**H**), 0.88 (t, 6H, *J* = 6.8 Hz, 23-**H**, 34-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.8, 173.4, 87.9, 83.7, 76.2, 74.1, 62.0, 60.8, 59.8, 41.9, 41.1, 34.4 (2C), 32.0 (2C), 29.7 (4C), 29.6 (2C), 29.5 (2C), 29.4 (2C), 29.3 (2C), 25.0, 24.9, 22.8 (2C), 14.2 (2C); IR (neat): 3366, 2921, 2854, 1729, 1465, 1407, 1267, 1177, 1017, 826, 721 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{33}H_{60}O_8Na$ $(M+Na)^+$ 607.4186, found $(M+Na)^+$ 607.4182.

(5,6-dihydroxy-1-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) bis(decanoate) ((±)-13c)



Colorless solid; Yield = 95%; R_f (EtOAc), 0.60; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.46 (bs, 1H, -**OH**), 4.24 (s, 1H, 4-**H**), 4.21 (dd, 1H, *J* = 5.6, 10.8 Hz, 12-$\mathbf{H_a}$), 4.17-4.06 (m, 4H, 10-**H**, 11-**H**), 4.04-3.91 (m, 3H, 5-**H**, 6-**H**, 12-$\mathbf{H_b}$), 3.66 (bs, 1H, -**OH**), 3.27 (bs, 1H, -**OH**), 2.35-2.28 (m, 4H, 13-**H**, 22-**H**), 2.24 (ddd, 1H, *J* = 5.6, 9.6, 9.6 Hz, 3-**H**), 2.15-2.09 (m, 1H, 2-**H**), 1.68-1.56 (m, 4H, 14-**H**, 23-**H**), 1.27 (bs, 24H, 15-20-**H**, 24-29-**H**), 0.88 (t, 6H, *J* = 6.8 Hz, 21-**H**, 30-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.6, 173.2, 87.7, 83.5, 76.2, 73.9, 61.8, 60.5, 59.9, 41.8, 40.9, 34.2 (2C), 31.8 (2 C), 29.4 (2C), 29.2 (4C), 29.1 (2C), 24.9, 24.8, 22.6 (2C), 14.1 (2C): IR (neat): 3384, 2923, 2856, 1729, 1462, 1261, 1179, 1021, 827, 730 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{29}H_{53}O_8$ $(M+H)^+$ 529.3740, found $(M+H)^+$ 529.3740.

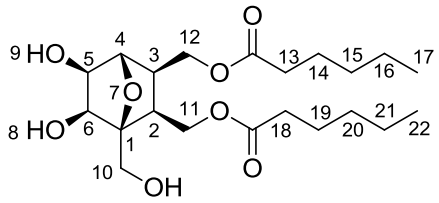
(5,6-dihydroxy-1-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) dioctanoate ((±)-13d)



Colorless solid; Yield = 95%; R_f (EtOAc), 0.58; ^{1}H NMR ($CDCl_3$, 400 MHz): 4.57 (bs, 1H, -**OH**), 4.23 (s, 1H, 4-**H**), 4.22-4.16 (m, 2H, 12-$\mathbf{H_a}$, -**OH**), 4.12-4.00 (m, 4H, 11-**H**, 10-**H**), 3.99-3.90 (m, 3H, 5-**H**, 6-**H**, 12-$\mathbf{H_b}$), 3.57 (bs, 1H, -**OH**), 2.34-2.28 (m, 4H, 13-**H**, 20-**H**), 2.23 (ddd, 1H, *J* = 6.0, 9.6, 9.6 Hz, 3-**H**), 2.16-2.10 (m, 1H, 2-**H**), 1.66-1.58 (m, 4H, 14-**H**, 21-**H**), 1.29 (bs, 16H, 15-18-**H**, 22-25-**H**), 0.89 (t, 6H, *J* = 6.0 Hz, 19-**H**, 26-**H**); ^{13}C NMR

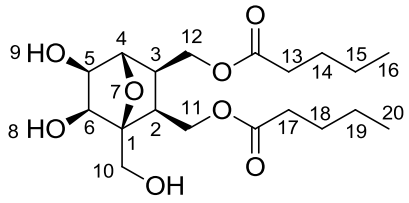
($CDCl_3$, 100 MHz): δ 173.6, 173.3, 87.8, 83.5, 76.0, 73.9, 61.8, 60.7, 59.6, 41.8, 40.9, 34.2 (2C), 31.6 (2C), 29.0 (2C), 28.8 (2C), 24.83, 24.76, 22.5 (2C), 13.9 (2C); IR (neat): 3367, 2951, 2864, 1729, 1464, 1408, 1276, 1180, 1013, 962, 827 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{25}H_{45}O_8$ $(M+H)^+$ 473.3114, found $(M+H)^+$ 473.3109.

(5,6-dihydroxy-1-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) dihexanoate ((±)-13e)



Colorless solid; Yield = 93%; R_f (EtOAc), 0.58; ^1H NMR ($CDCl_3$, 400 MHz): δ 4.40 (bs, 1H, -**OH**), 4.24 (s, 1H, 4-**H**), 4.20 (dd, 1H, *J* = 5.2, 10.8 Hz, 12-$\mathbf{H_a}$), 4.12-4.04 (m, 4H, 10-**H**, 11-**H**), 4.01-3.90 (m, 3H, 5-**H**, 6-**H**, 12-$\mathbf{H_b}$), 3.58 (bs, 1H, -**OH**), 3.15 (bs, 1H, -O**H**), 2.33-2.28 (m, 4H, 13-**H**, 18-**H**) 2.27-2.21 (m, 1H, 3-**H**), 2.15-2.09 (m, 1H, 2-**H**), 1.65-1.58 (m, 4H, 14-**H**, 19-**H**), 1.33-1.23 (m, 8H, 15-16-**H**, 20-21-**H**), 0.89 (t, 6H, *J* = 6.4 Hz, 17-**H**, 22-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 173.4, 87.8, 83.5, 75.8, 73.9, 61.9, 60.7, 59.4, 41.6, 40.9, 33.9 (2C), 26.9 (2C), 26.8 (2C), 22.2 (2C), 13.6 (2C). IR (neat): 3446, 3056, 2956, 2929, 2866, 1736, 1463, 1170, 1113, 1024, 744, 613 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{21}H_{36}O_8Na$ $(M+Na)^+$ 439.2308, found $(M+Na)^+$ 439.2307.

(5,6-dihydroxy-1-(hydroxymethyl)-7-oxabicyclo[2.2.1]heptane-2,3-diyl)bis(methylene) dipentanoate ((±)-13f)



Colorless solid; Yield = 91%; R_f (EtOAc), 0.58; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.69 (bs, 1H, -**OH**), 4.23 (s, 1H, 4-**H**), 4.22 (dd, 1H, 12-$\mathbf{H_a}$ - merged with 4**H**), 4.16-4.00 (m, 4H, 10-**H**, 11-**H**), 3.98-3.93 (m, 3H, 5-**H**, 6-**H** & 12-$\mathbf{H_b}$), 3.81(bs, 1H, -**OH**), 2.58 (bs, 1H, -**OH**), 2.35-2.29 (m, 4H, 13-**H**, 17-**H**), 2.23 (ddd, 1H, *J* = 5.2, 9.2, 9.2 Hz, 3-**H**), 2.12-2.11 (m, 1H, 2-**H**), 1.65-1.56 (m, 4H, 14-**H**, 18-**H**), 1.41-1.29 (m, 4H, 15-**H**, 19-**H**), 0.94-0.89 (m, 6H, 16-**H**, 20-**H**); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 173.7, 173.4, 87.8, 83.5, 75.8, 73.9, 61.9, 60.7, 59.4, 41.6, 40.9, 33.9 (2C), 26.85, 26.77, 22.2 (2C), 13.6 (2C): IR (neat): 3403, 2960, 2871, 1734, 1266, 1173, 910, 742 cm^{-1}; HRMS (ESI) exact mass calcd. for $C_{19}H_{32}O_8K$ $(M+K)^+$ 427.1734, found $(M+K)^+$ 427.1726.

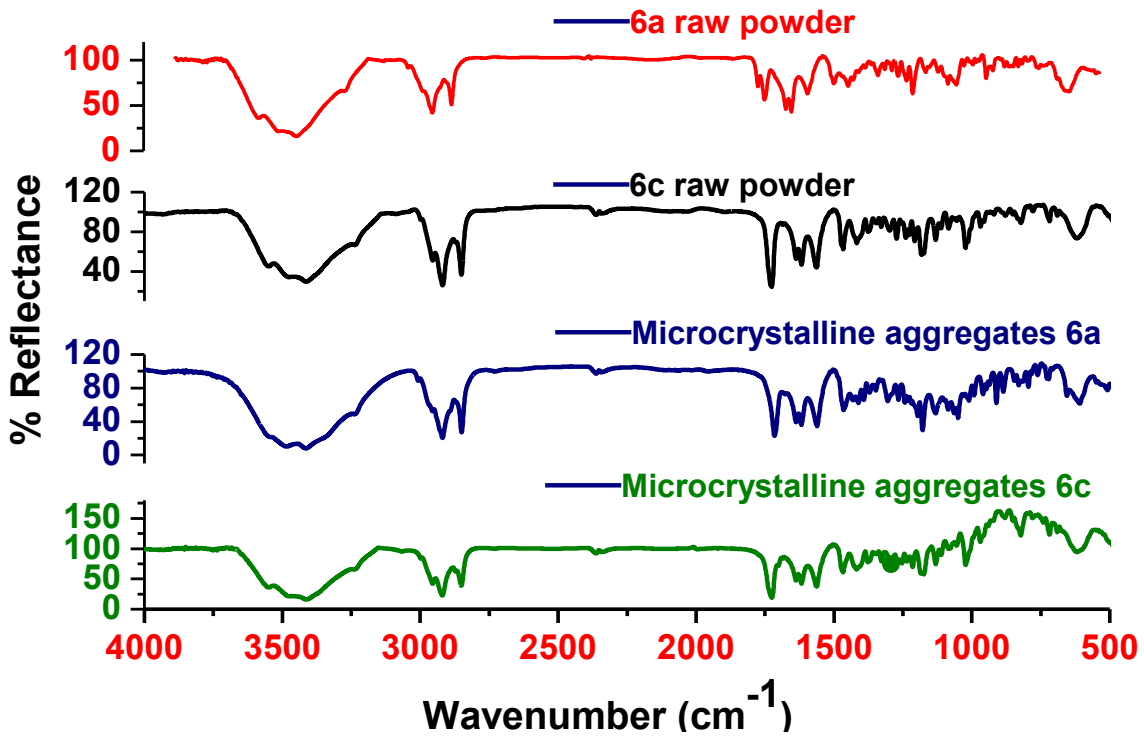


Figure 1. IR Spectra comparison of authentic samples of **6a** and **6c** with the microcrystalline aggregates of these samples prepared by adding their acetonitrile solution (200 uL; 1 mg mL^{-1}) to a solution of P123 in water (5g L^{-1}, 1 mL)

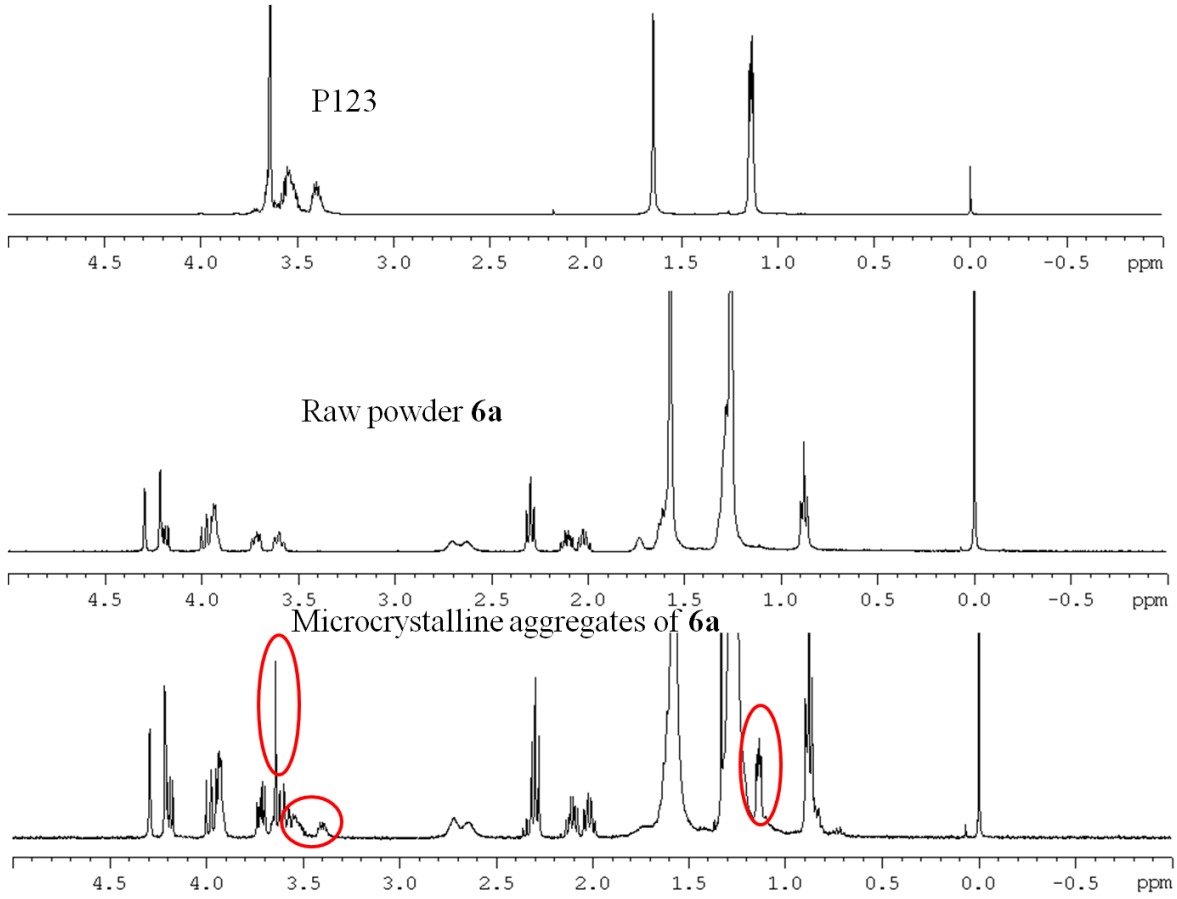


Figure 2. ^{1}H-NMR (400 MHz, $CDCl_3$) Spectra comparison of authentic sample of **6a** with the microcrystalline aggregates. Red colored circle indicates presence of trace amount of P123 in microcrystalline aggregates **6a**.

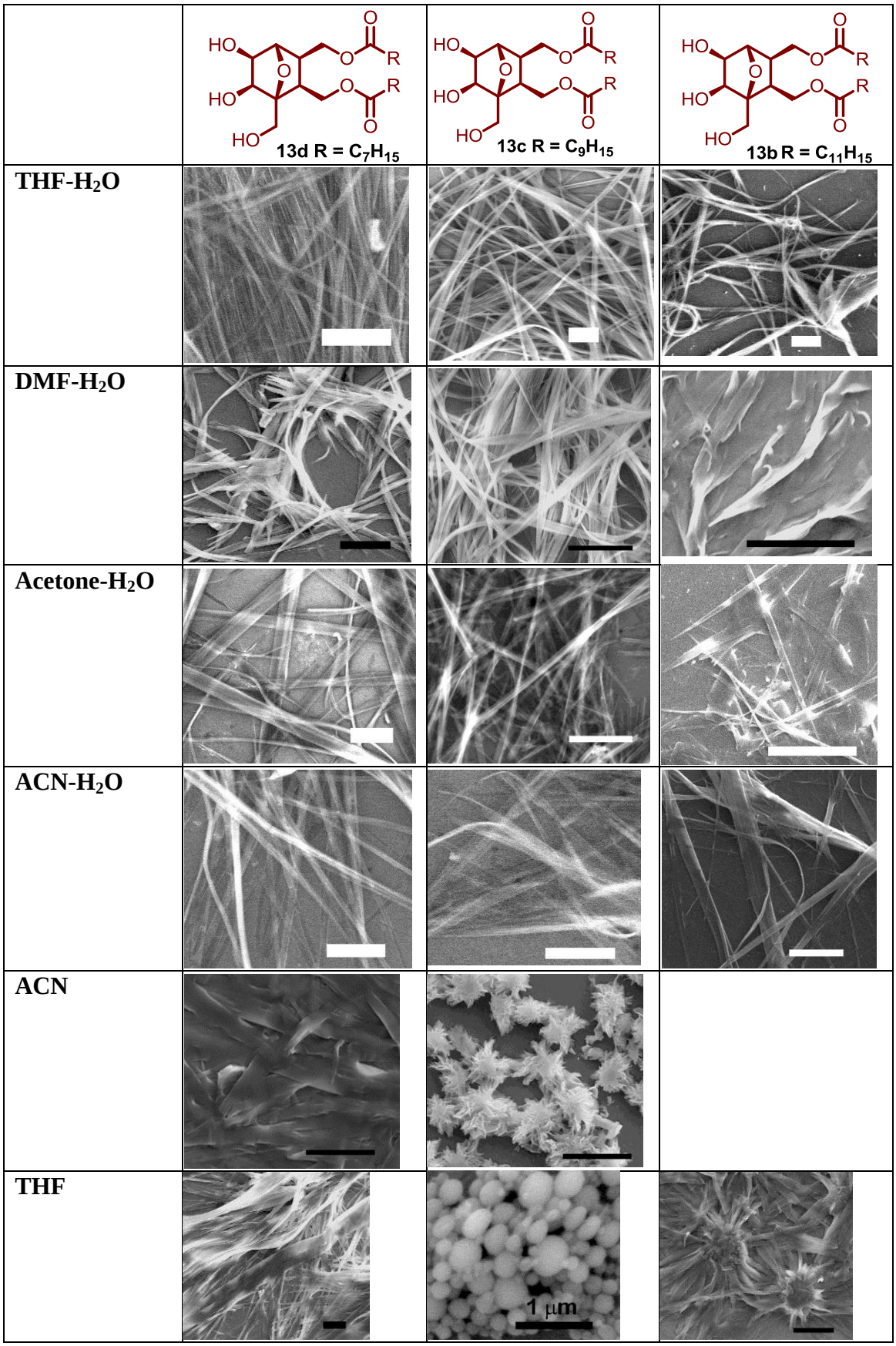
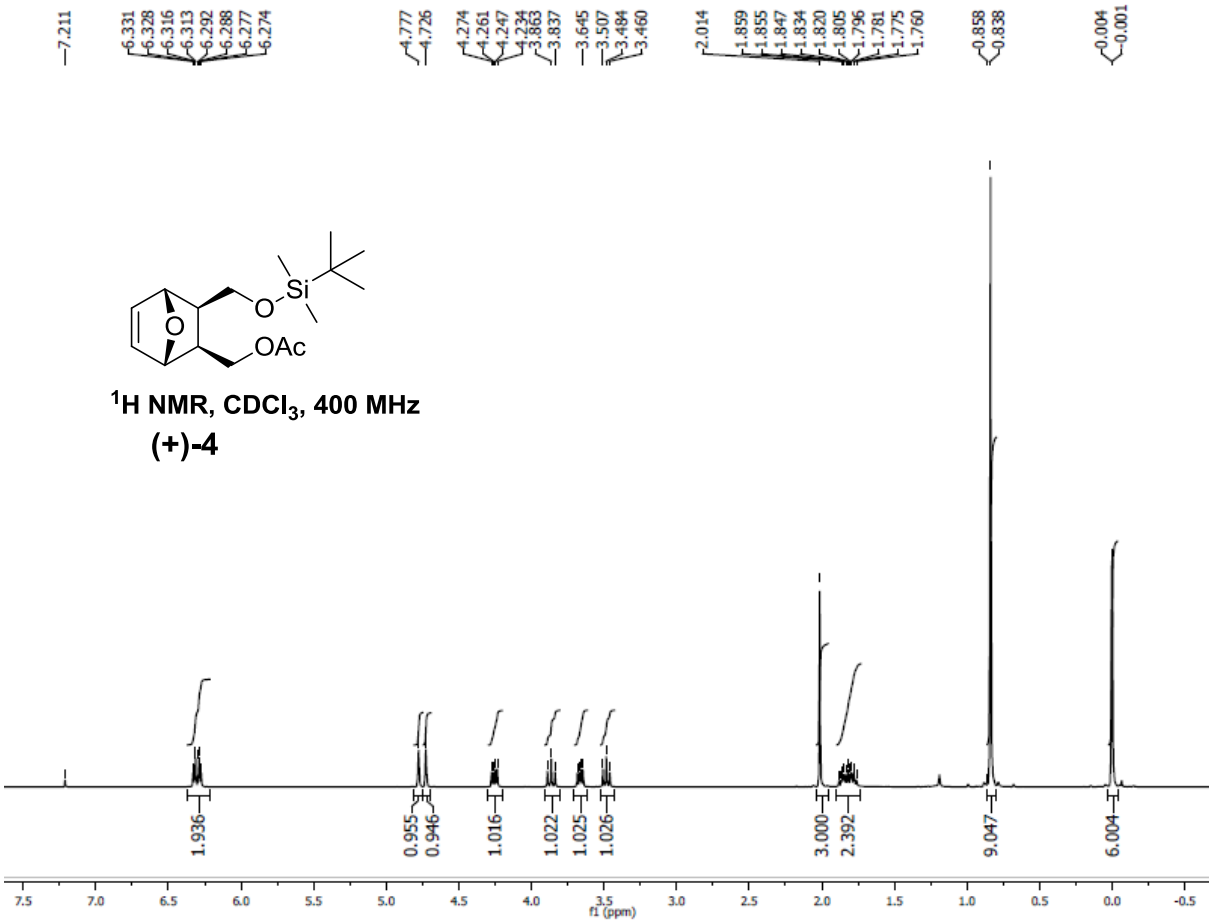


Figure 3. SEM images of the samples prepared under various conditions; Scale bar 10 μM.

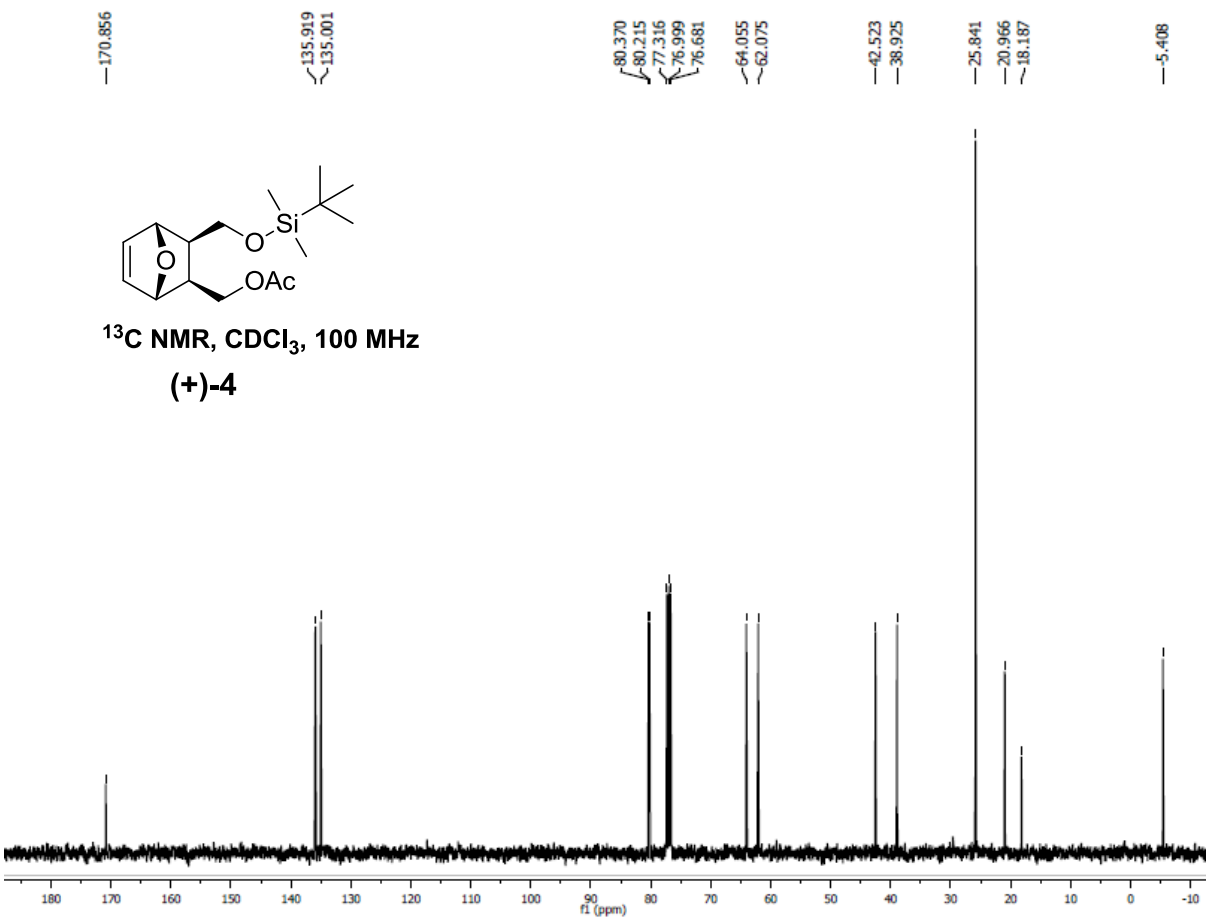
Crystallographic information of compounds 3c & 12e

	3c	**12e**
CCDC number	846065	812794
Empirical formula	$C_{18}\ H_{32}\ O_6$	$C_{28}\ H_{42}\ O_8$
M_r	344.44	506.62
Crystal system	Monoclinic	Triclinic
Space group	P2(1)	P-1
a [Å]	5.5386(5)	5.474(3)
b [Å]	7.0291(7)	12.149(7)
c [Å]	23.255(2)	21.524(11)
β [°]	93.015(5)	86.90(3)
V [Å^3]	904.10(15)	1418.2(13)
Z	2	2
ρ_{calcd} [mg m^{-3}]	1.265	1.186
μ [mm^{-1}]	0.093	0.086
F(000)	376	548
Crystal size [mm]	0.35 x 0.22 x 0.15	0.30 x 0.20 x 0.20
θ range [°]	0.88 to 30.65	0.95 to 24.18
Reflections collected	7136	22200
Independent reflections	4128 [R(int) = 0.0287]	4498 [R(int) = 0.0297]
Data/restraints/parameters	4128 / 1 / 231	4498 / 72 / 343
Goodness-of-fit on F^2	1.033	1.074
Final R indices [I>2σ(I)]	R1 = 0.0496, wR2 = 0.0990	R1 = 0.0704, wR2 = 0.1861



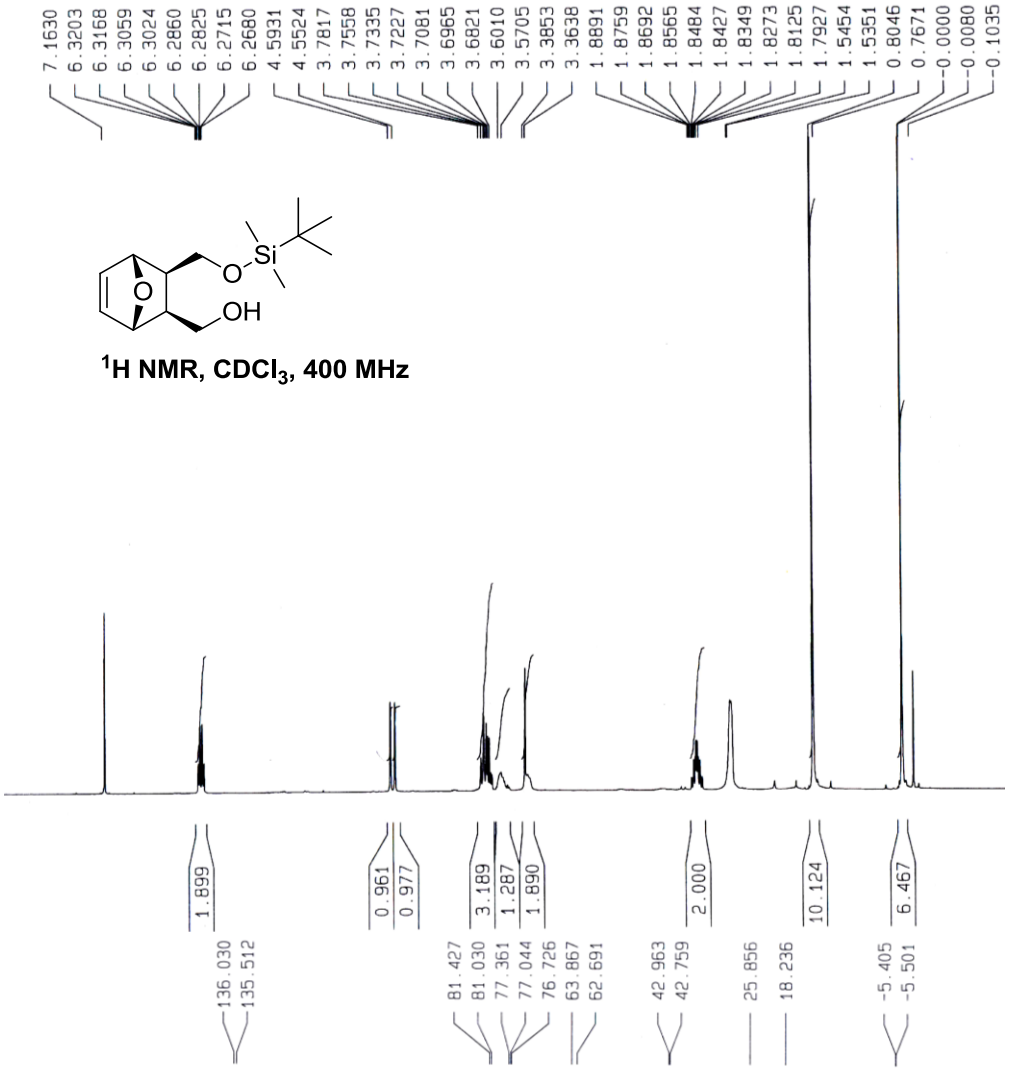
^{1}H NMR, $CDCl_3$, 400 MHz

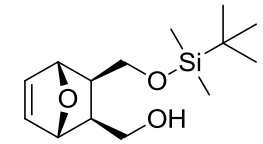
(+)-4



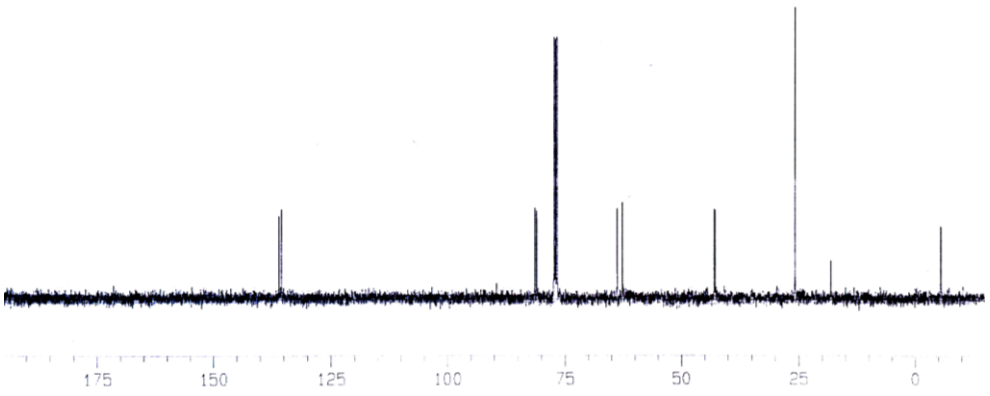
^{13}C NMR, $CDCl_3$, 100 MHz

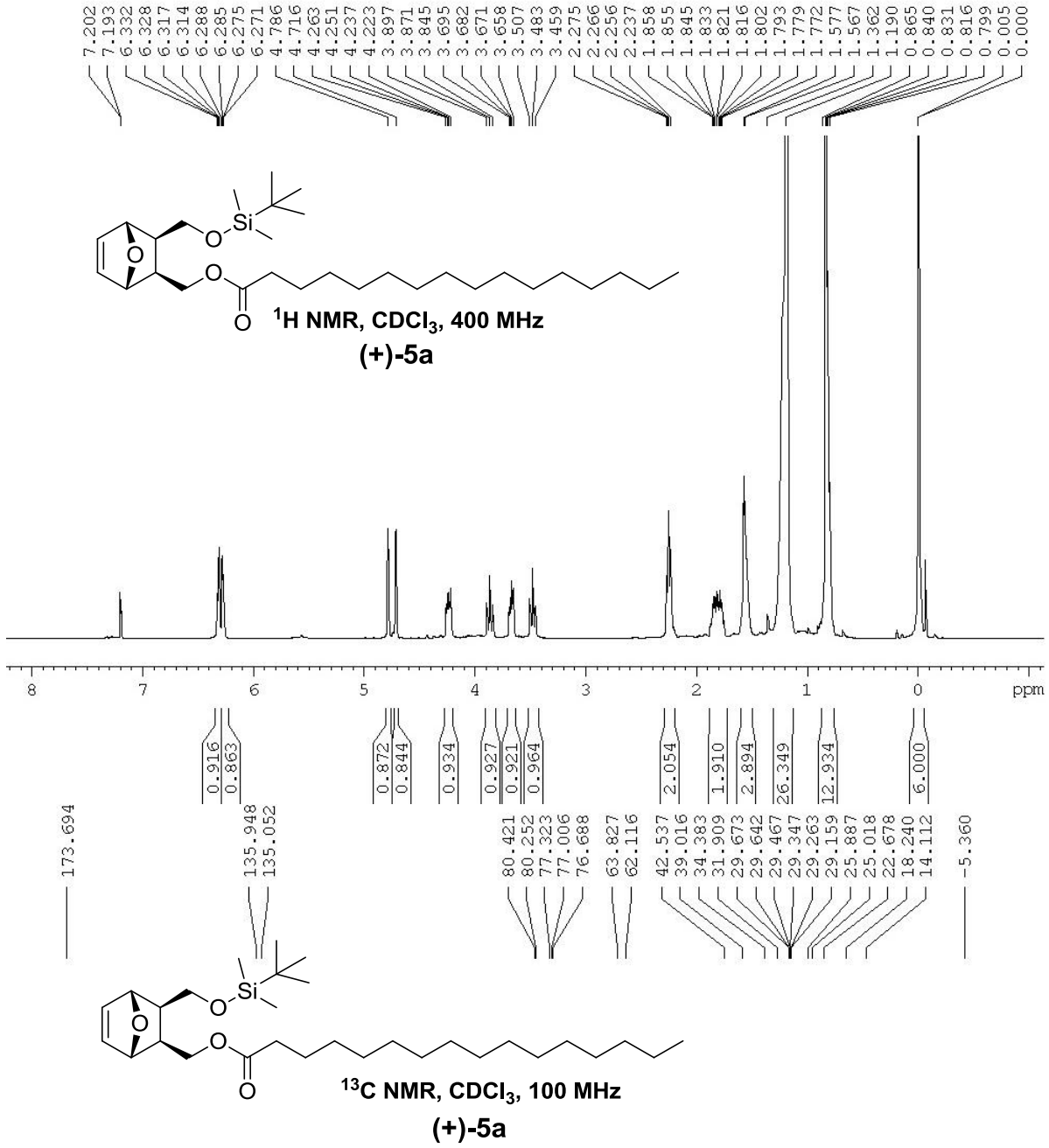
(+)-4

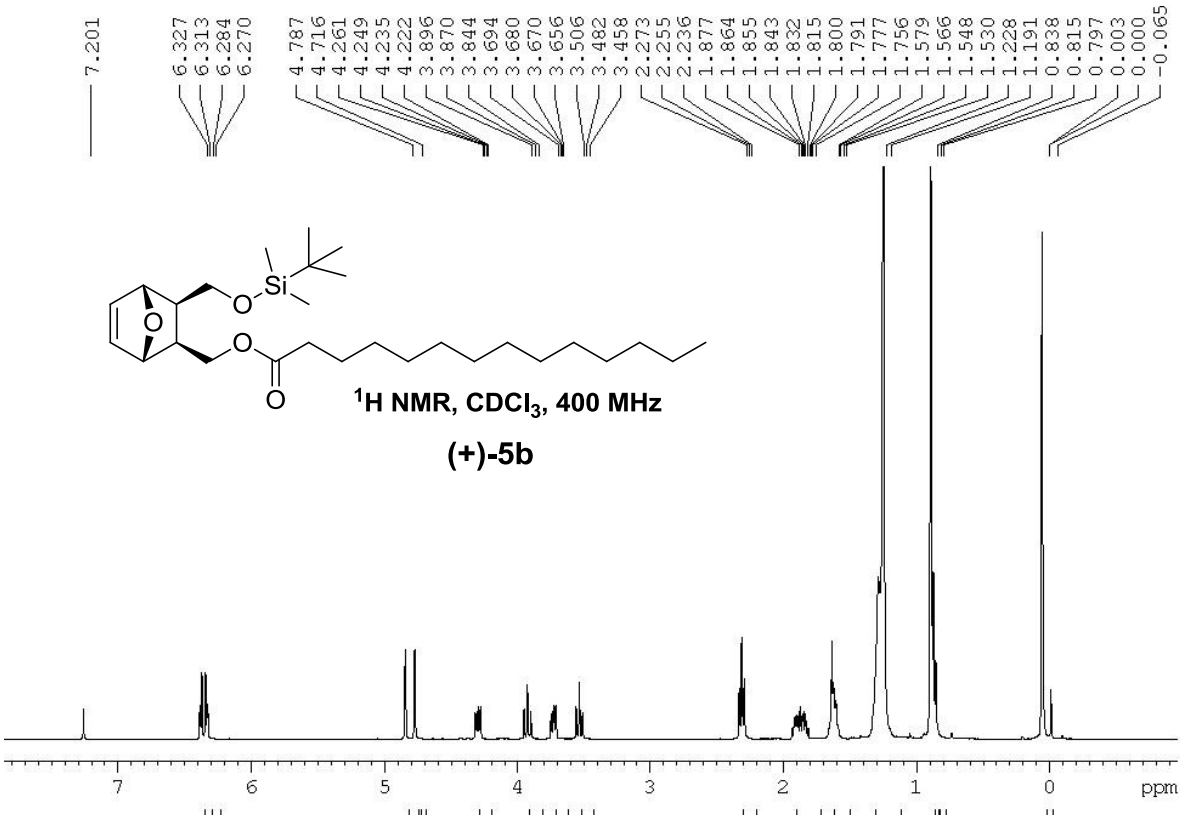




^{13}C NMR, $CDCl_3$, 100 MHz

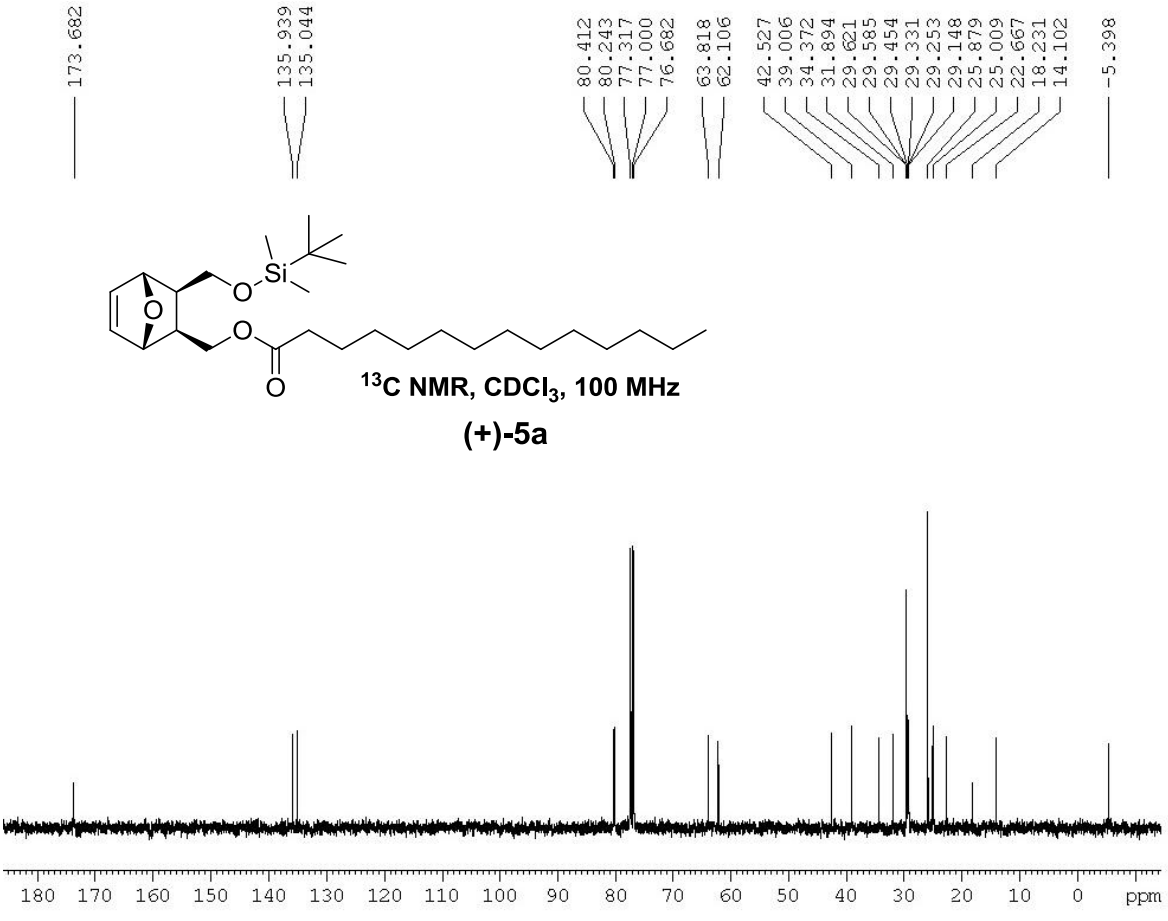






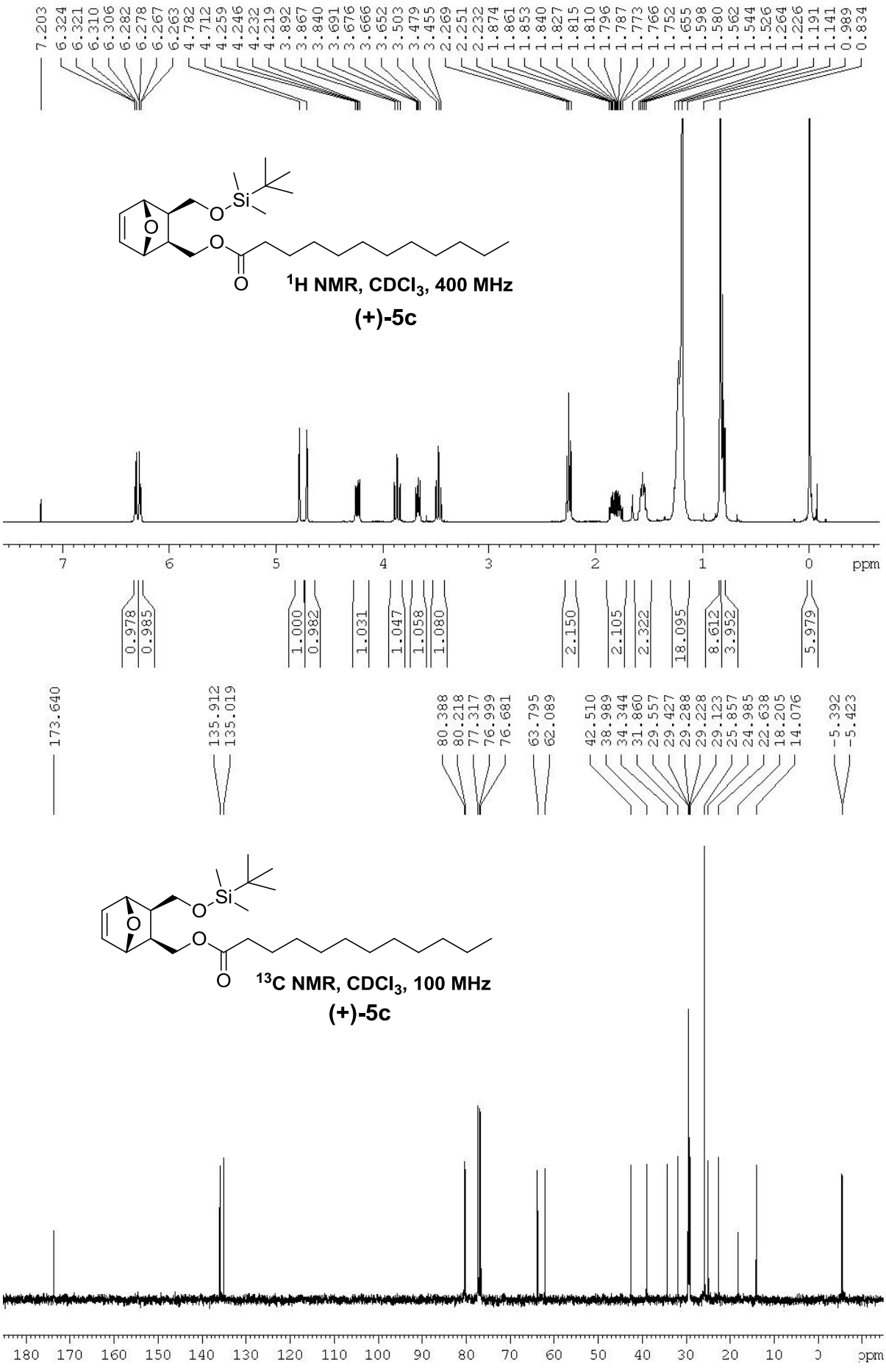
^{1}H NMR, $CDCl_3$, 400 MHz

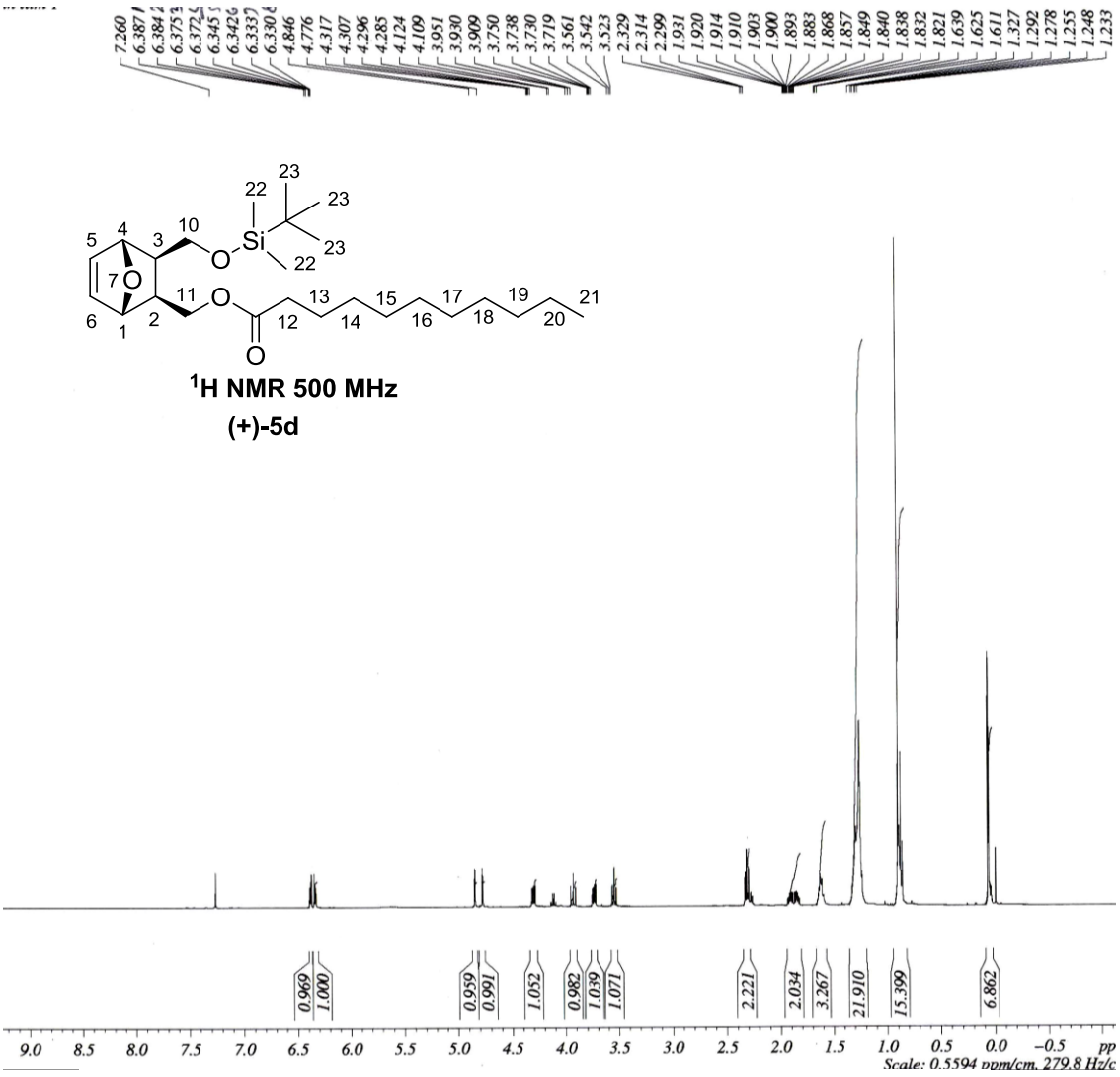
(+)-5b



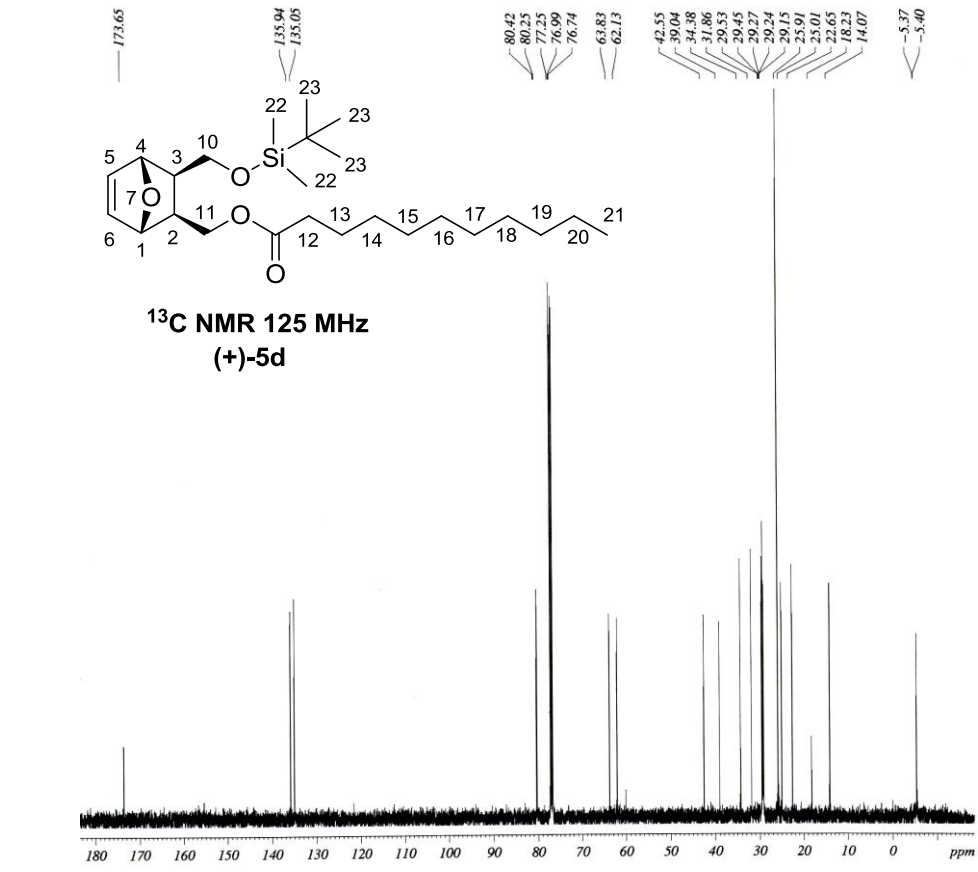
^{13}C NMR, $CDCl_3$, 100 MHz

(+)-5a

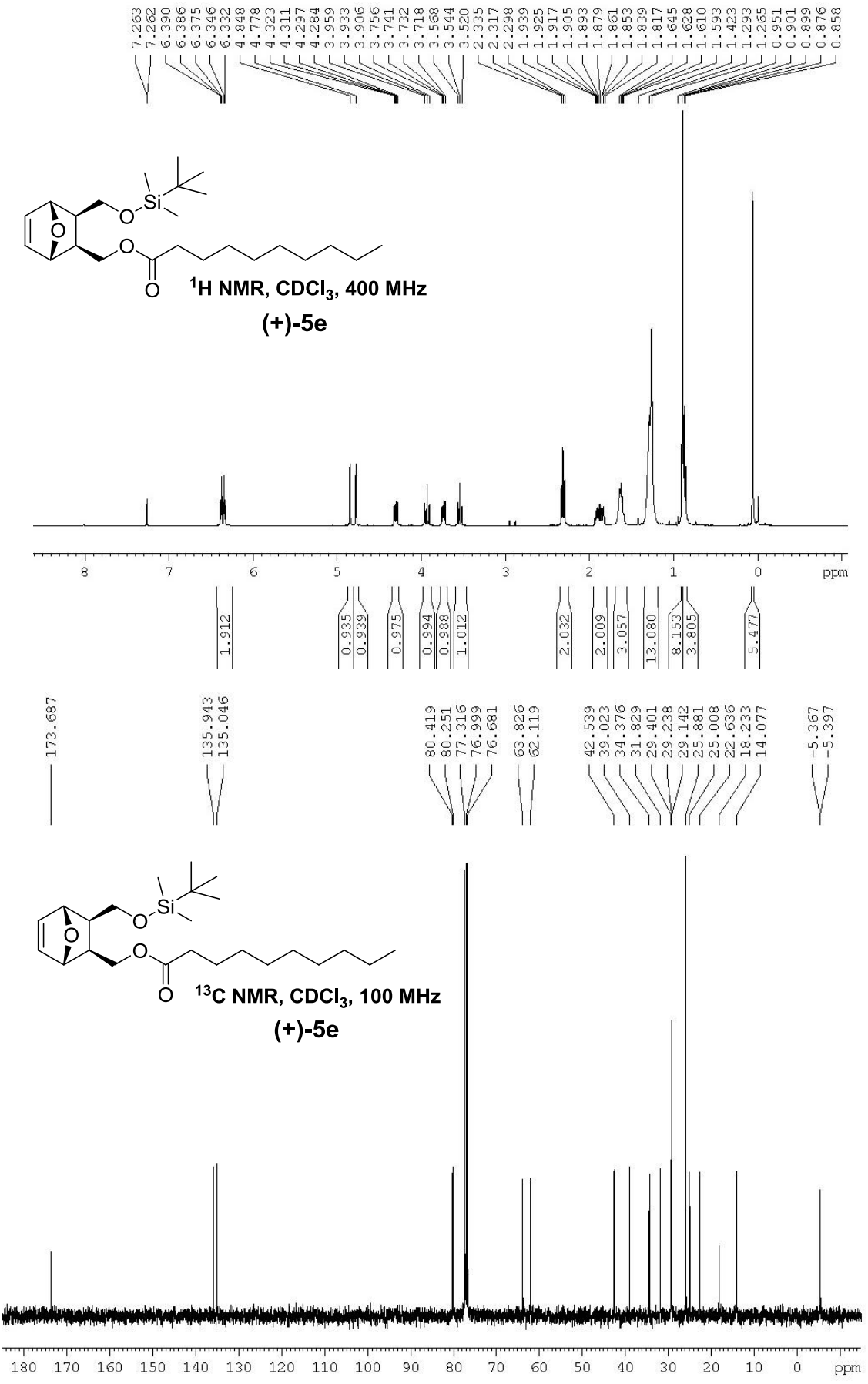

7.203
6.324
6.321
6.310
6.306
6.282
6.278
6.267
6.263
4.782
4.712
4.259
4.246
4.232
4.219
3.892
3.867
3.840
3.691
3.676
3.666
3.652
3.503
3.479
3.455
2.269
2.251
2.232
1.874
1.861
1.853
1.840
1.827
1.815
1.810
1.796
1.787
1.773
1.766
1.752
1.655
1.598
1.580
1.562
1.544
1.526
1.264
1.226
1.191
1.141
0.989
0.834
Si
O
O
O
1H NMR, CDCl3, 400 MHz
(+)-5c
7
6
5
4
3
2
1
0
ppm
0.978
0.985
1.000
0.982
1.031
1.047
1.058
1.080
2.150
2.105
2.322
18.095
8.612
3.952
5.979
173.640
135.912
135.019
80.388
80.218
77.317
76.999
76.681
63.795
62.089
42.510
38.989
34.344
31.860
29.557
29.427
29.288
29.228
29.123
25.857
24.985
22.638
18.205
14.076
-5.392
-5.423
Si
O
O
O
13C NMR, CDCl3, 100 MHz
(+)-5c
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



^{1}H NMR 500 MHz
(+)-5d



^{13}C NMR 125 MHz
(+)-5d


7.263
7.262
6.390
6.386
6.375
6.346
6.332
4.848
4.778
4.323
4.311
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3.959
3.933
3.906
3.756
3.741
3.732
3.718
3.568
3.544
3.520
2.335
2.317
2.298
1.939
1.925
1.917
1.905
1.893
1.879
1.861
1.853
1.839
1.817
1.645
1.628
1.610
1.593
1.423
1.293
1.265
0.951
0.901
0.899
0.876
0.858
Si
O
O
O
1H NMR, CDCl3, 400 MHz
(+)-5e
8
7
6
5
4
3
2
1
0
ppm
1.912
0.935
0.939
0.975
0.994
0.988
1.012
2.032
2.009
3.057
13.080
8.153
3.805
5.477
173.687
135.943
135.046
80.419
80.251
77.316
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76.681
63.826
62.119
42.539
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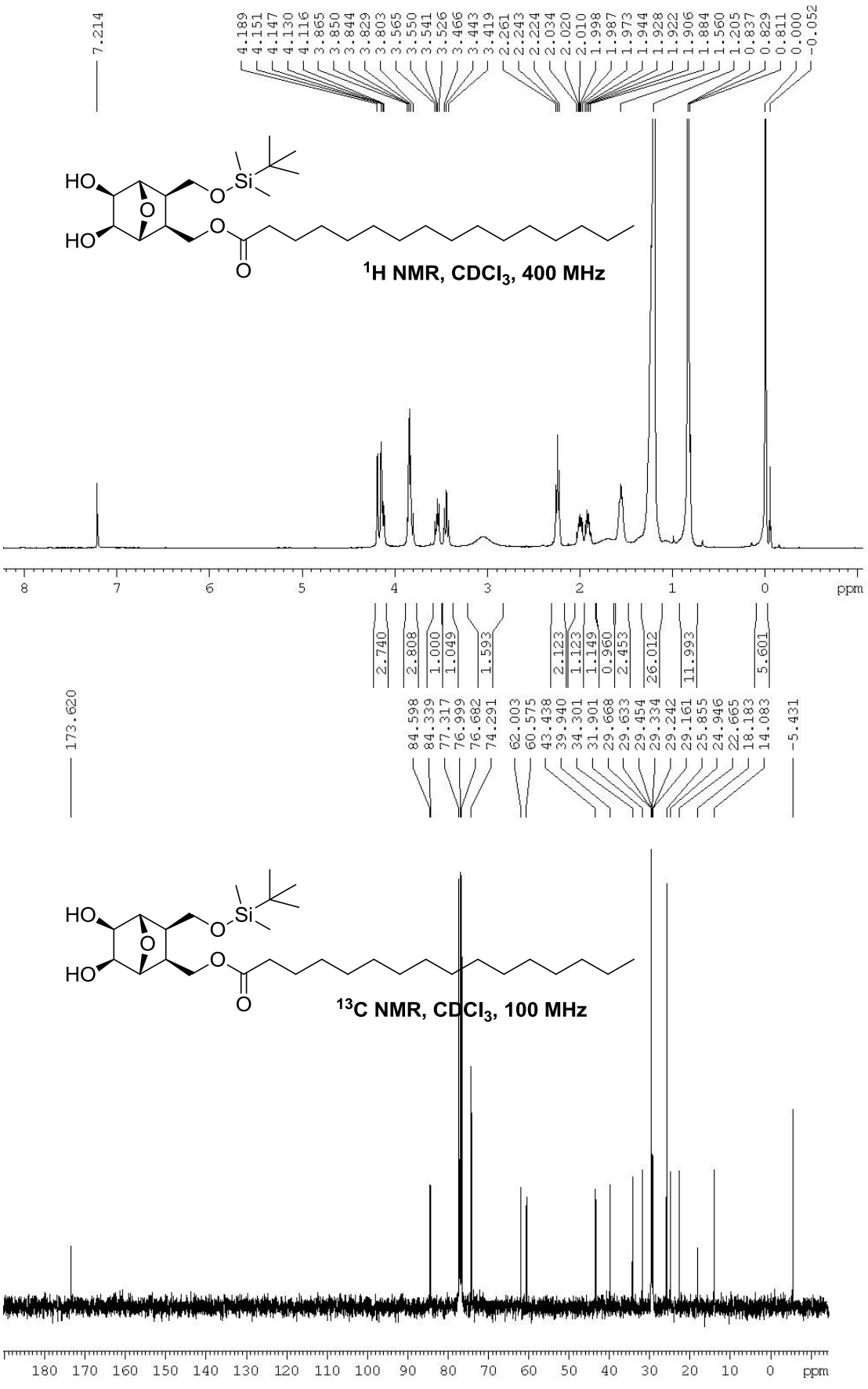
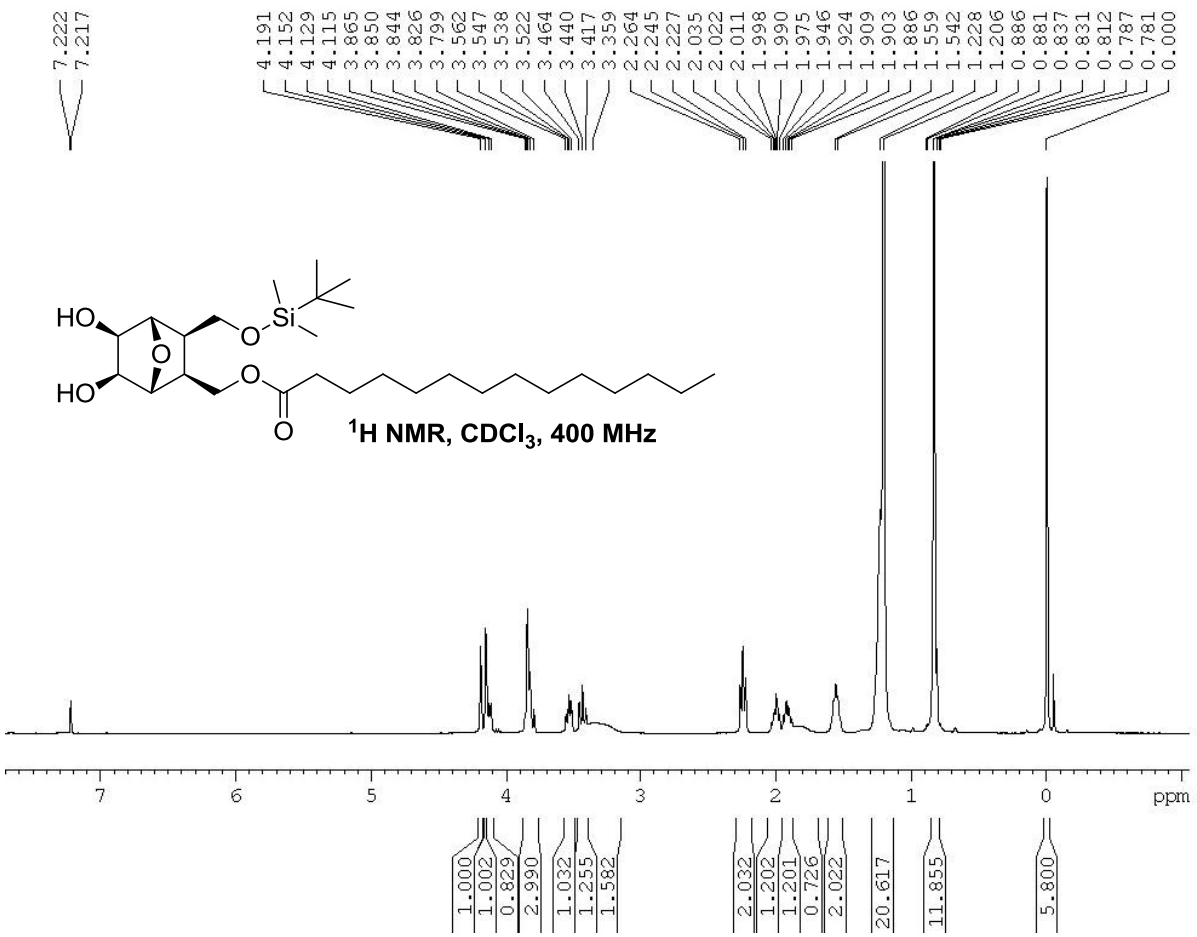
34.376
31.829
29.401
29.238
29.142
25.881
25.008
22.636
18.233
14.077
-5.367
-5.397
Si
O
O
O
13C NMR, CDCl3, 100 MHz
(+)-5e
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

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4.115
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3.850
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3.799
3.562
3.547
3.538
3.522
3.464
3.440
3.417
3.359
2.264
2.245
2.227
2.035
2.022
2.011
1.998
1.990
1.975
1.946
1.924
1.909
1.903
1.886
1.559
1.542
1.228
1.206
0.886
0.881
0.837
0.831
0.812
0.787
0.781
0.000
HO
O
Si
O
HO
O
O
1H NMR, CDCl3, 400 MHz
7
6
5
4
3
2
1
0
ppm
1.000
1.002
0.829
2.990
1.032
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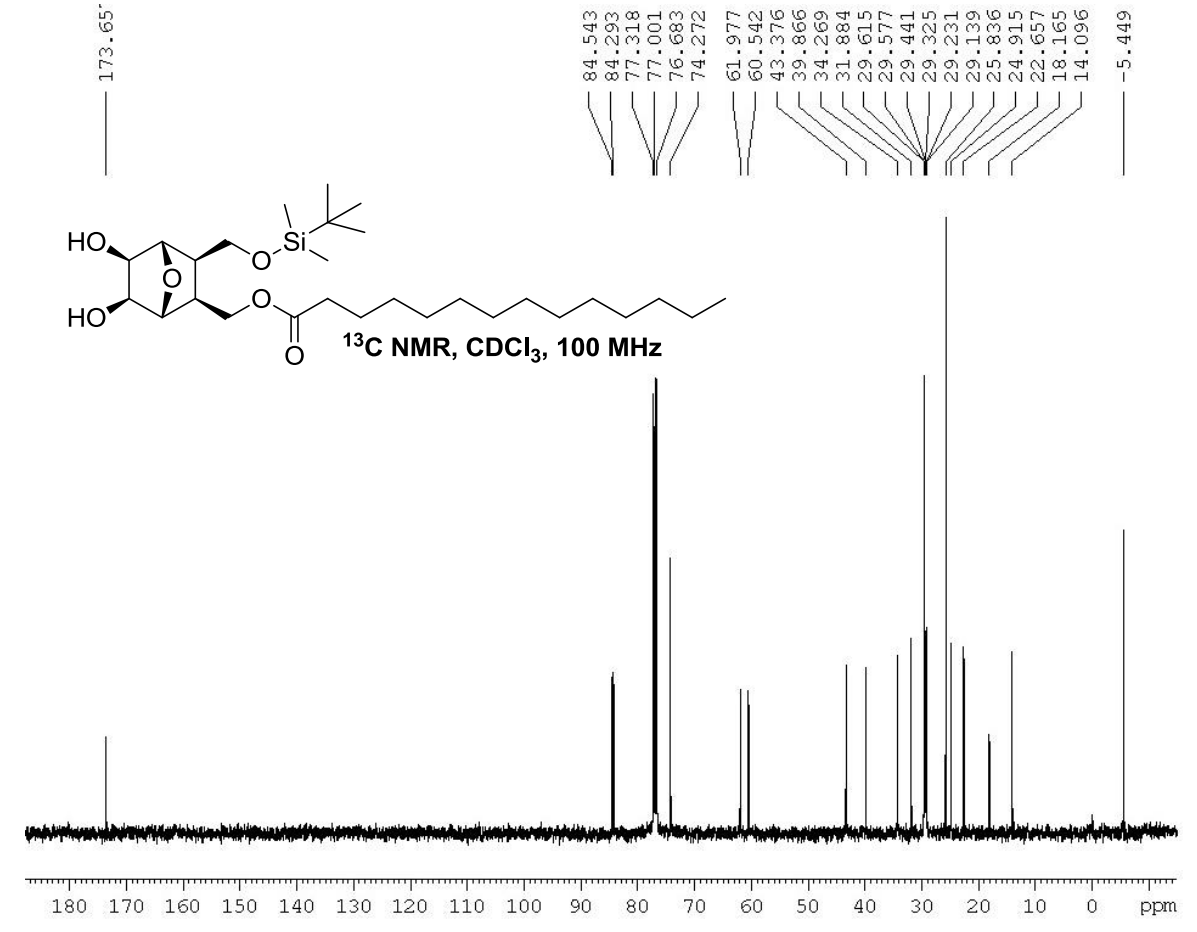
1.582
2.032
1.202
1.201
0.726
2.022
20.617
11.855
5.800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61.977
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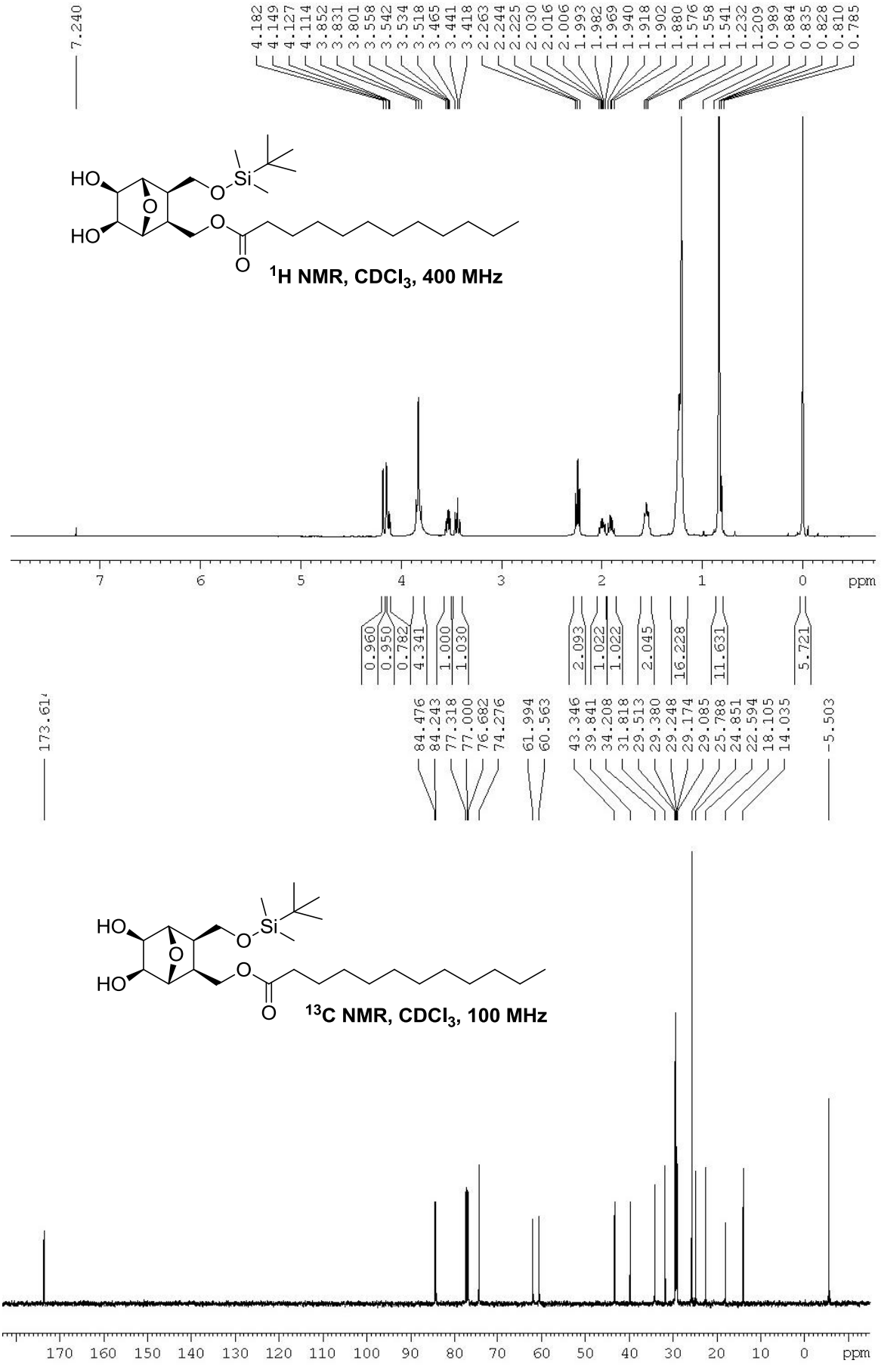
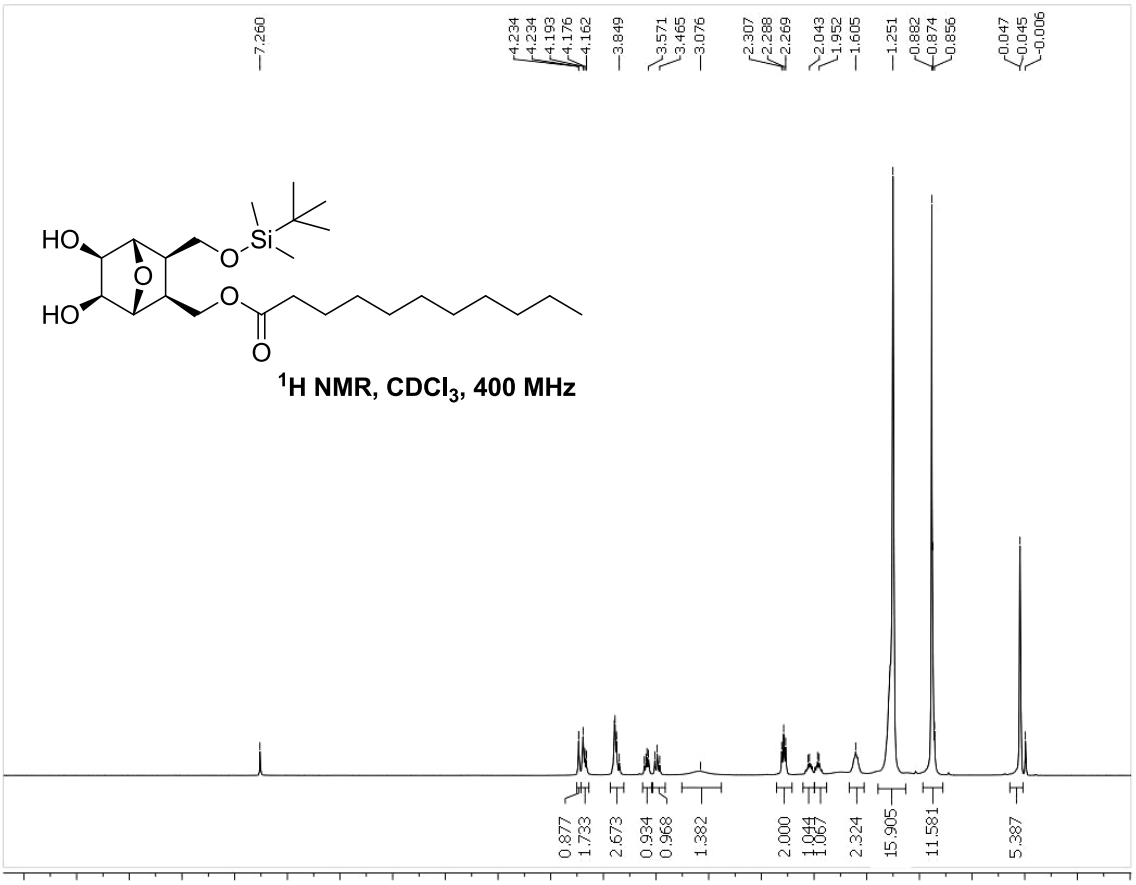
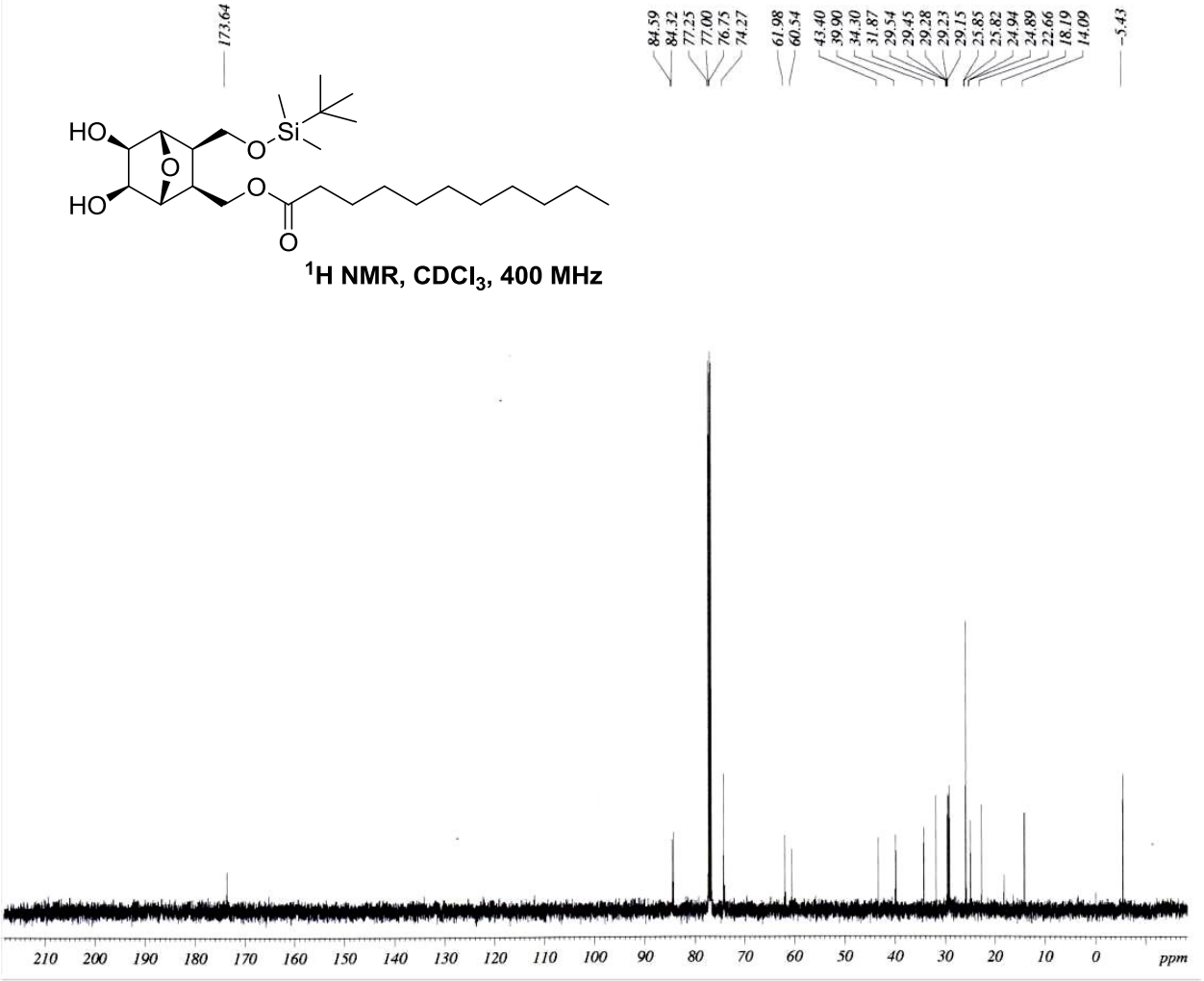
31.884
29.615
29.577
29.441
29.325
29.231
29.139
25.836
24.915
22.657
18.165
14.096
-5.449
HO
O
Si
O
HO
O
O
13C NMR, CDCl3, 100 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

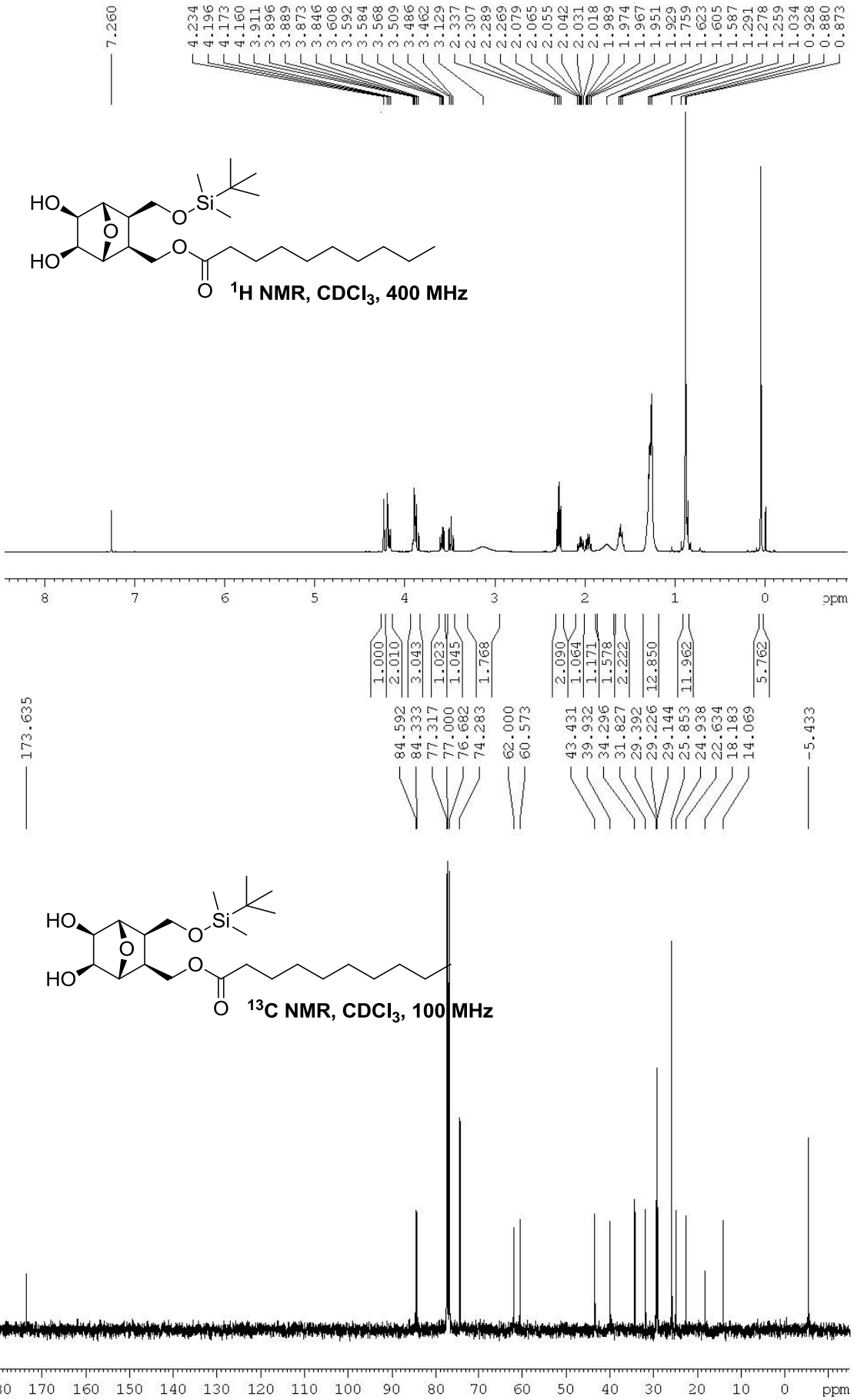




^{1}H NMR, $CDCl_3$, 400 MHz



^{1}H NMR, $CDCl_3$, 400 MHz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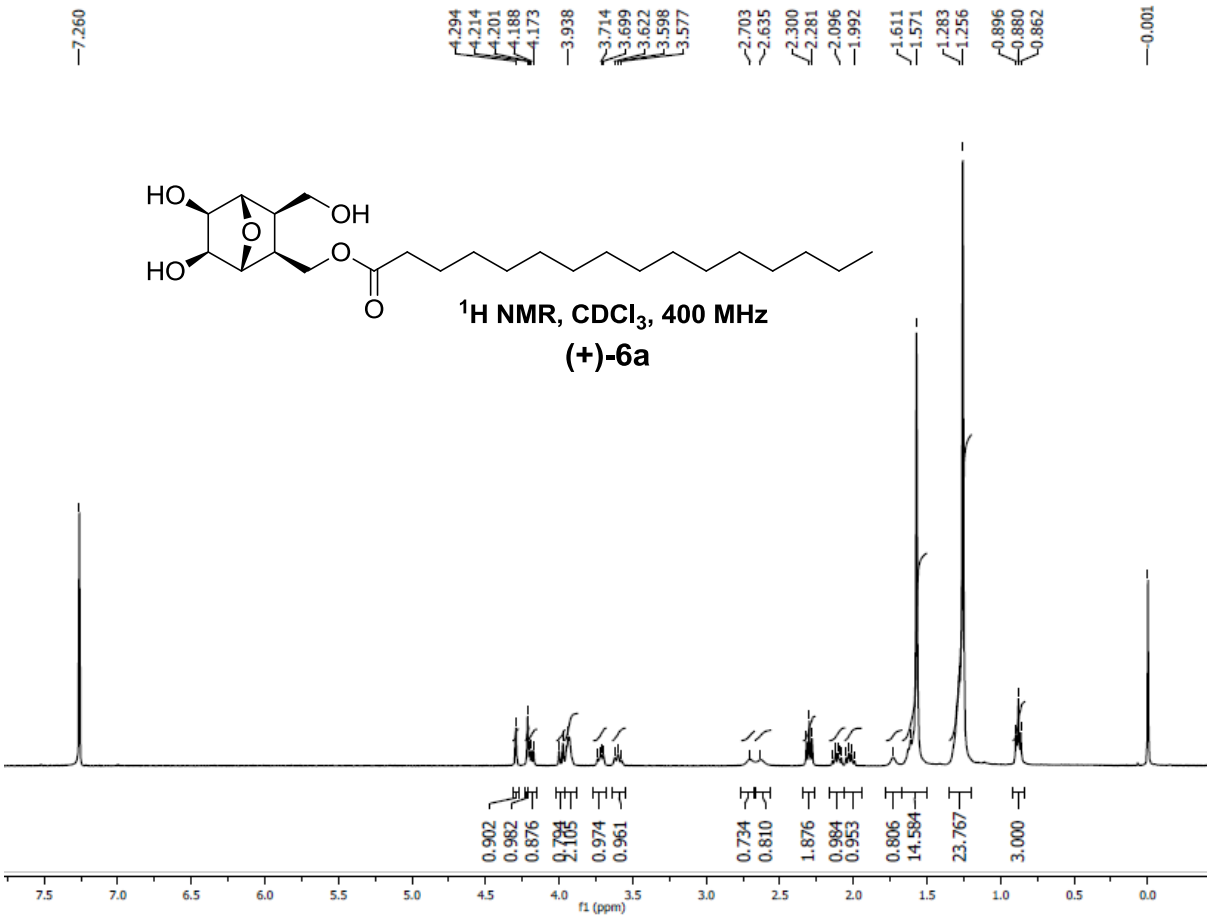
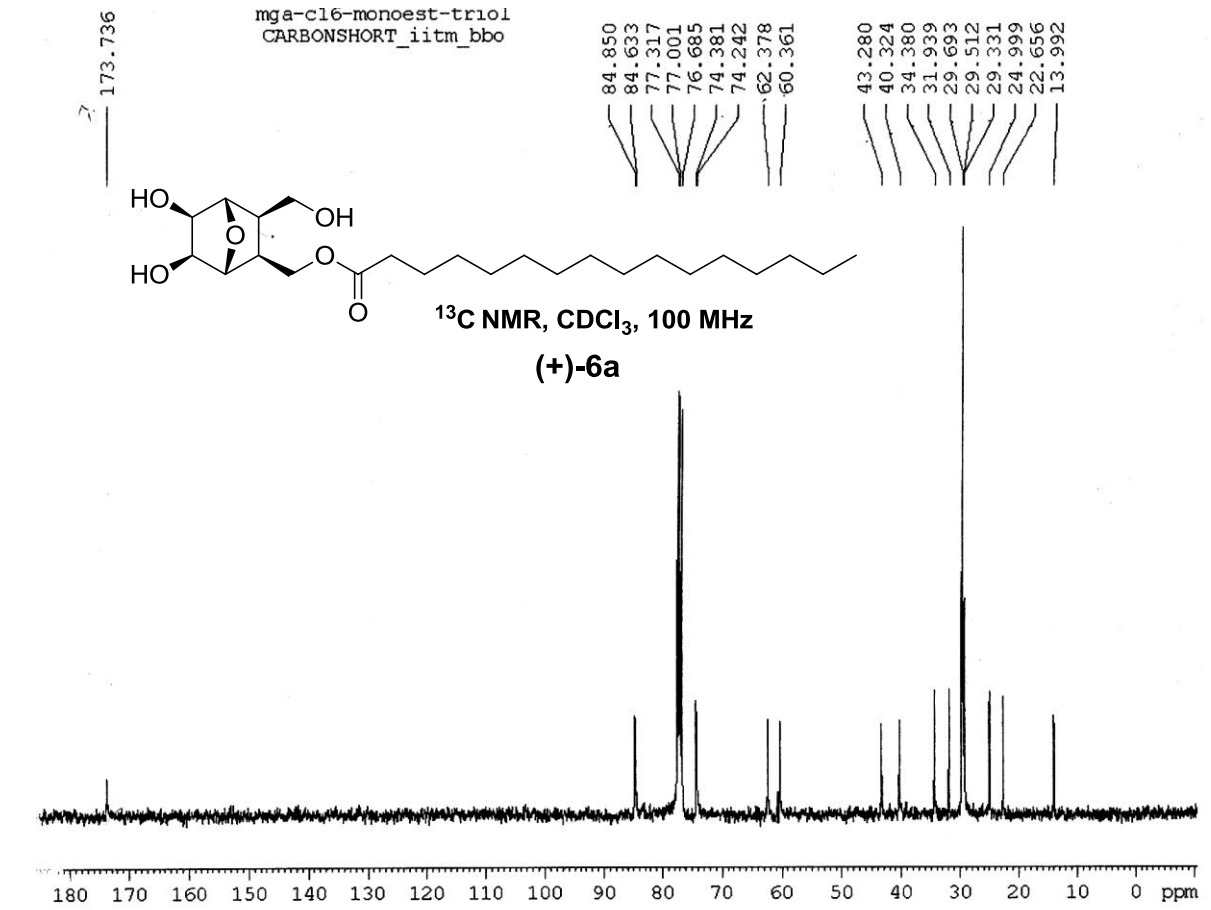
4.234
4.196
4.173
4.160
3.911
3.896
3.889
3.873
3.846
3.608
3.592
3.584
3.568
3.509
3.486
3.462
3.129
2.337
2.307
2.289
2.269
2.079
2.065
2.055
2.042
2.031
2.018
1.989
1.974
1.967
1.951
1.929
1.759
1.623
1.605
1.587
1.291
1.278
1.259
1.034
0.928
0.880
0.873
HO
HO
O
Si
O
O
O
1H NMR, CDCl3, 400 MHz
8
7
6
5
4
3
2
1
0
ppm
1.000
2.010
3.043
1.023
1.045
1.768
2.090
1.064
1.171
1.578
2.222
12.850
11.962
5.762
173.635
84.592
84.333
77.317
77.000
76.682
74.283
62.000
60.573
43.431
39.932
34.296
31.827
29.392
29.226
29.144
25.853
24.938
22.634
18.183
14.069
-5.433
HO
HO
O
Si
O
O
O
13C NMR, CDCl3, 100 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



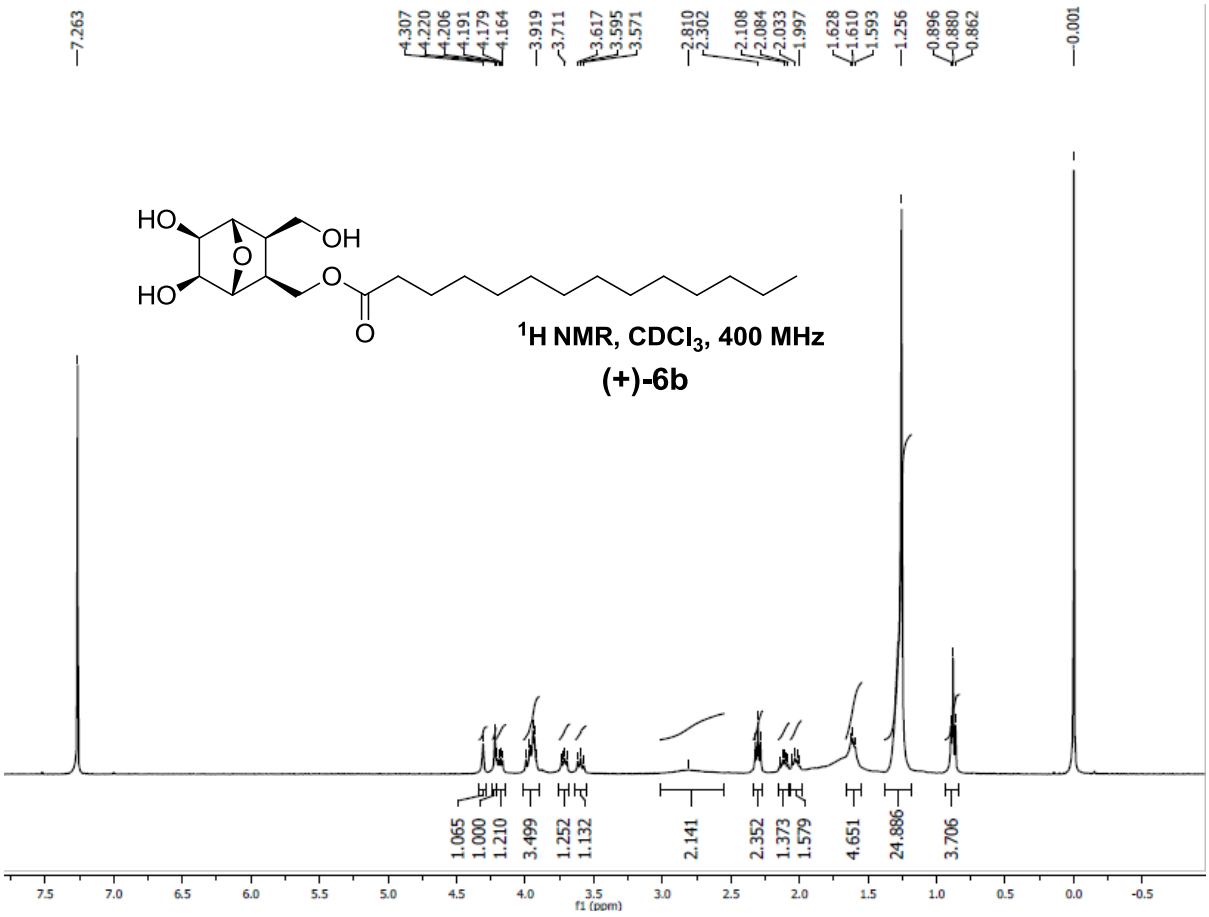
^{1}H NMR, $CDCl_3$, 400 MHz

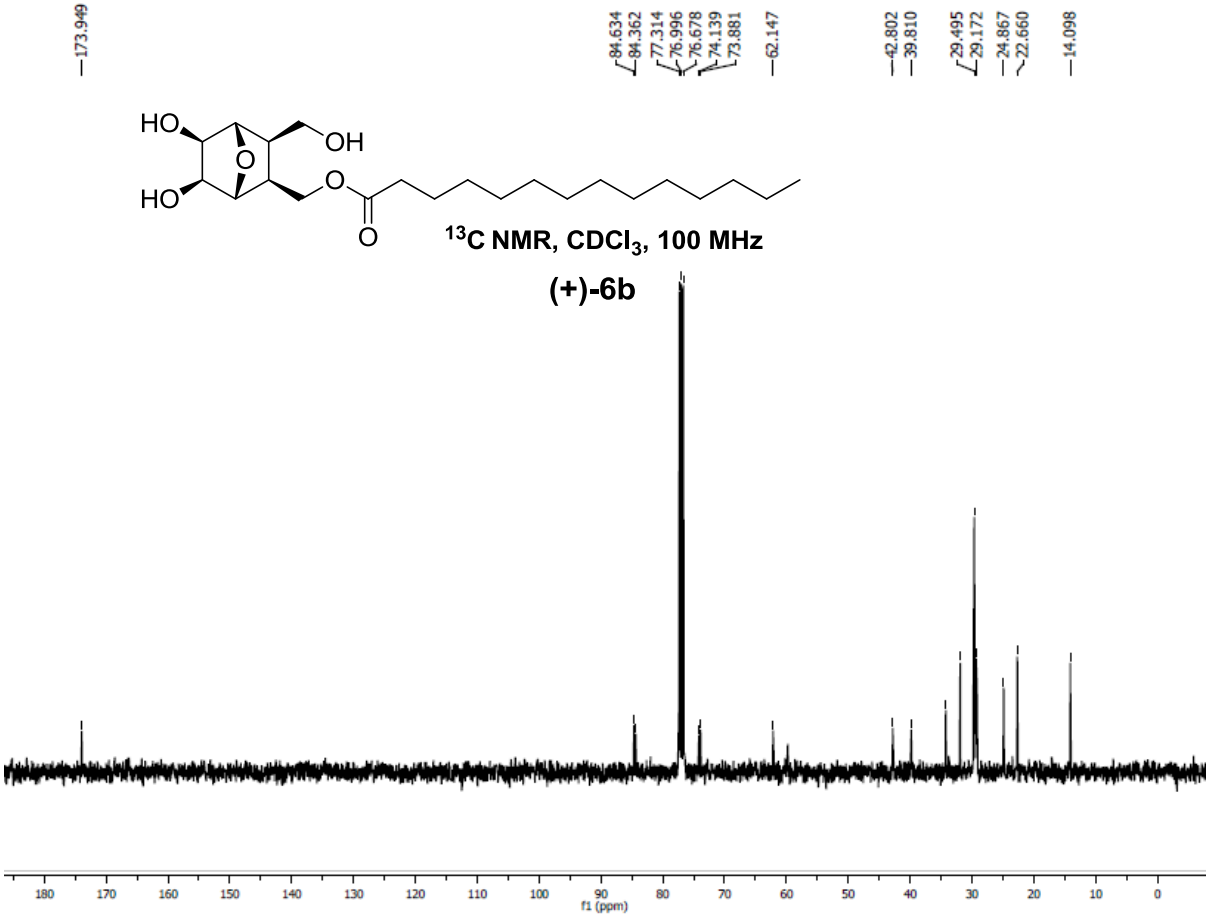
(+)-6a

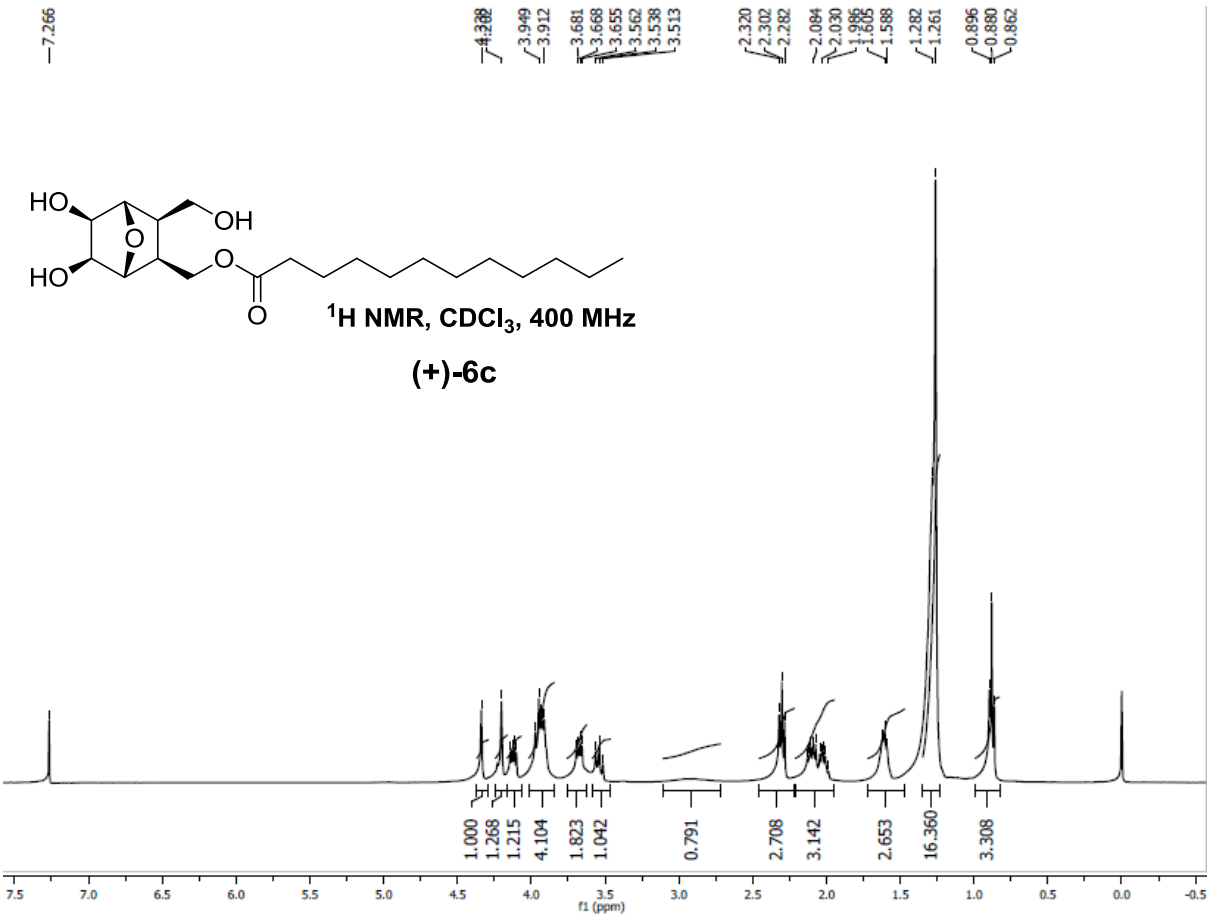


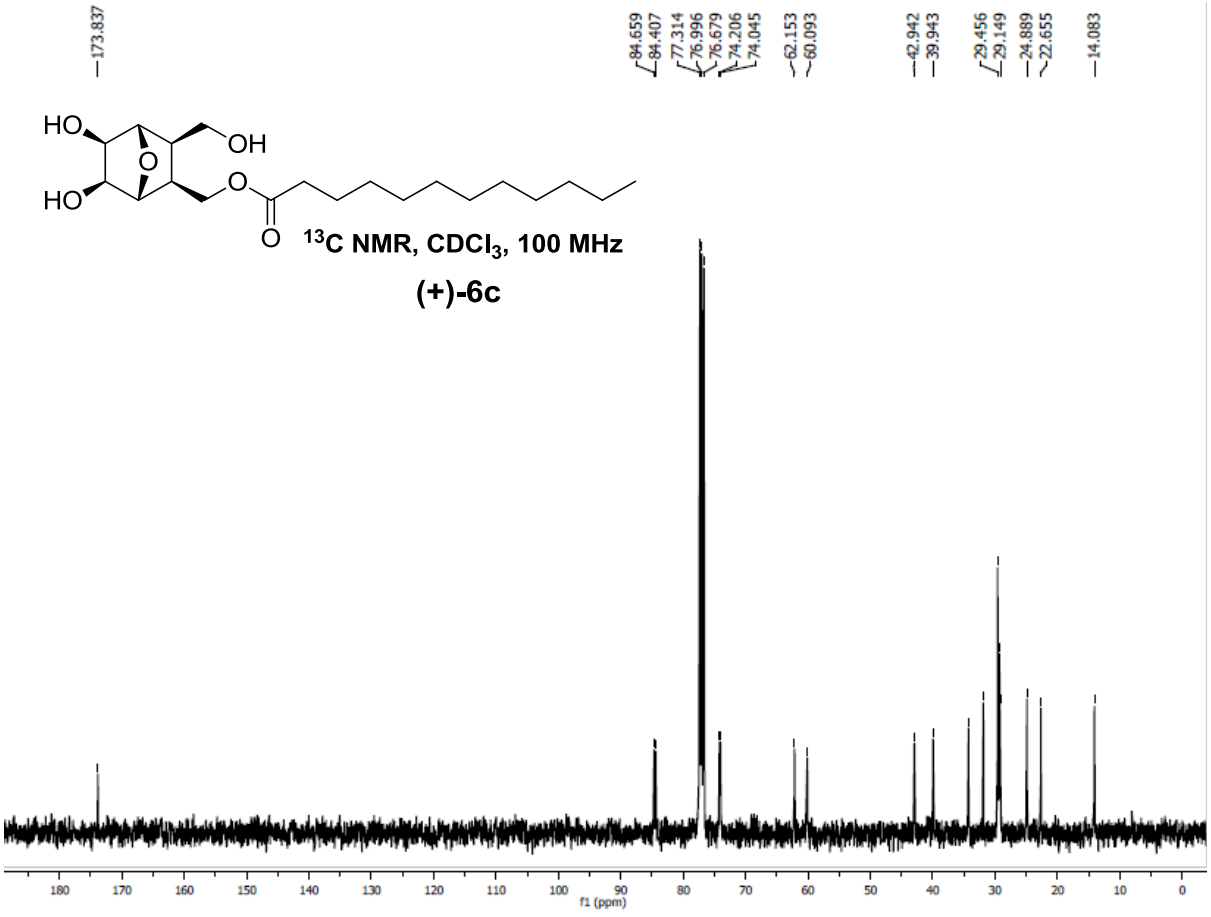
^{13}C NMR, $CDCl_3$, 100 MHz

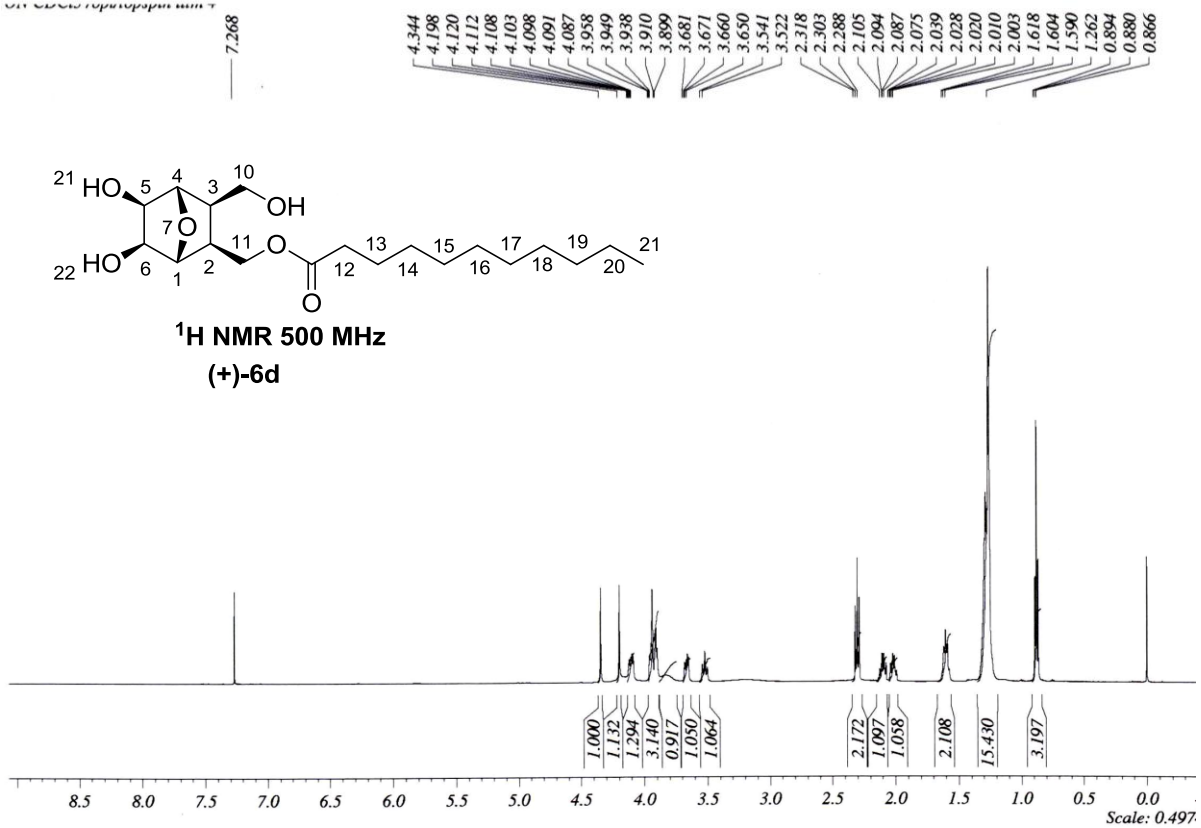
(+)-6a



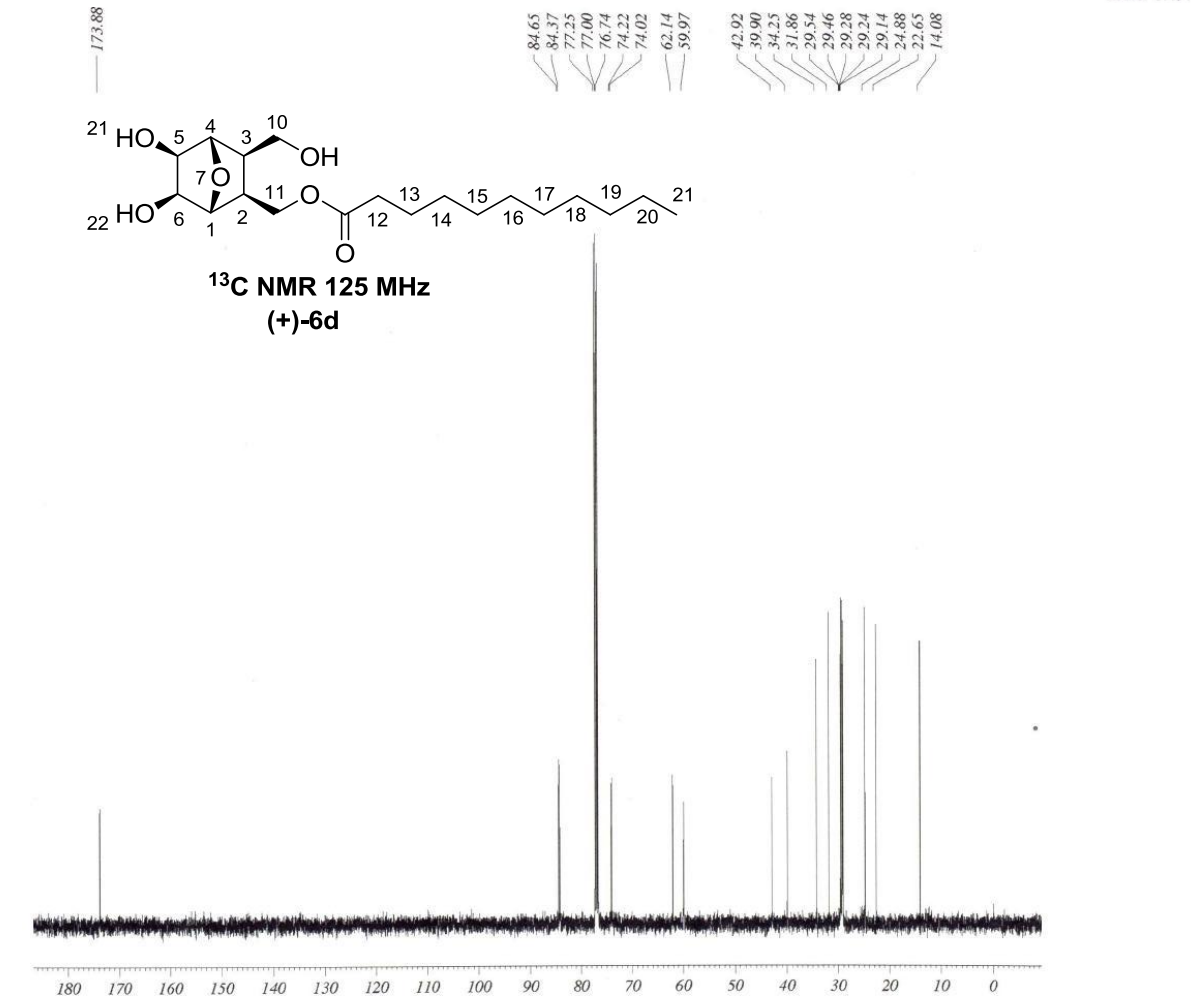




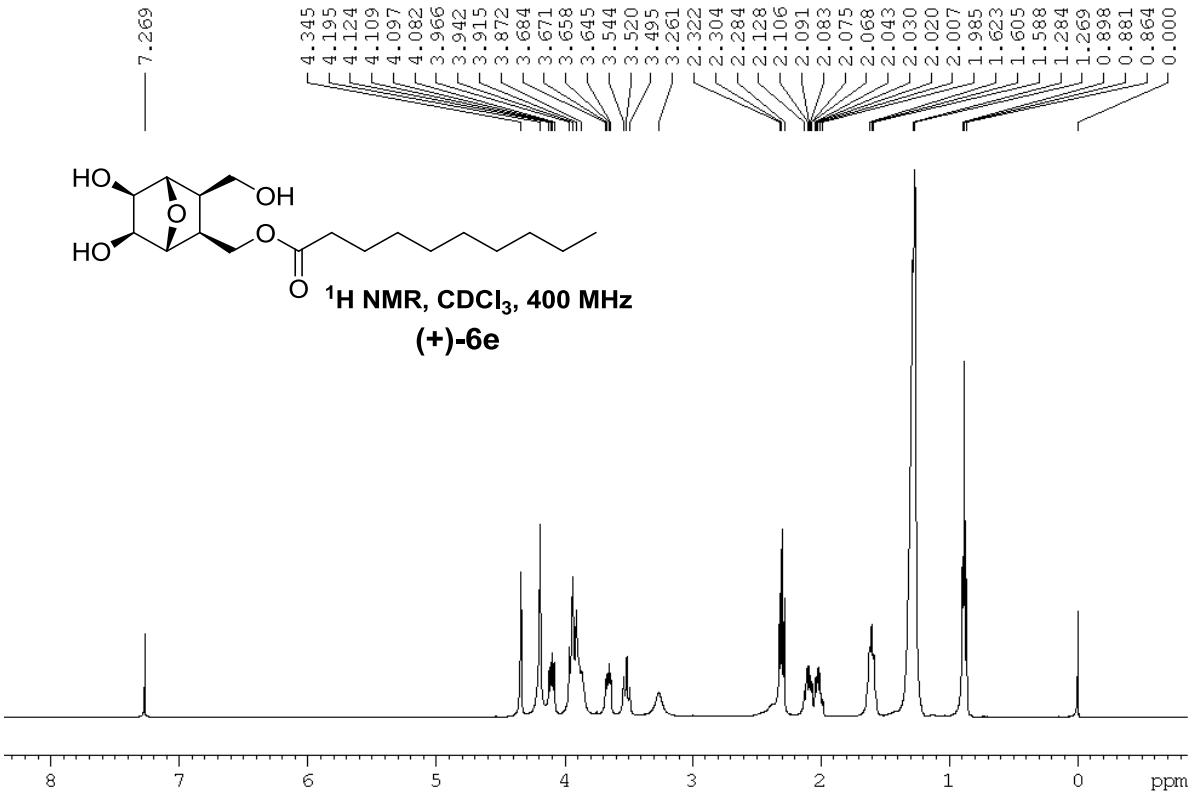


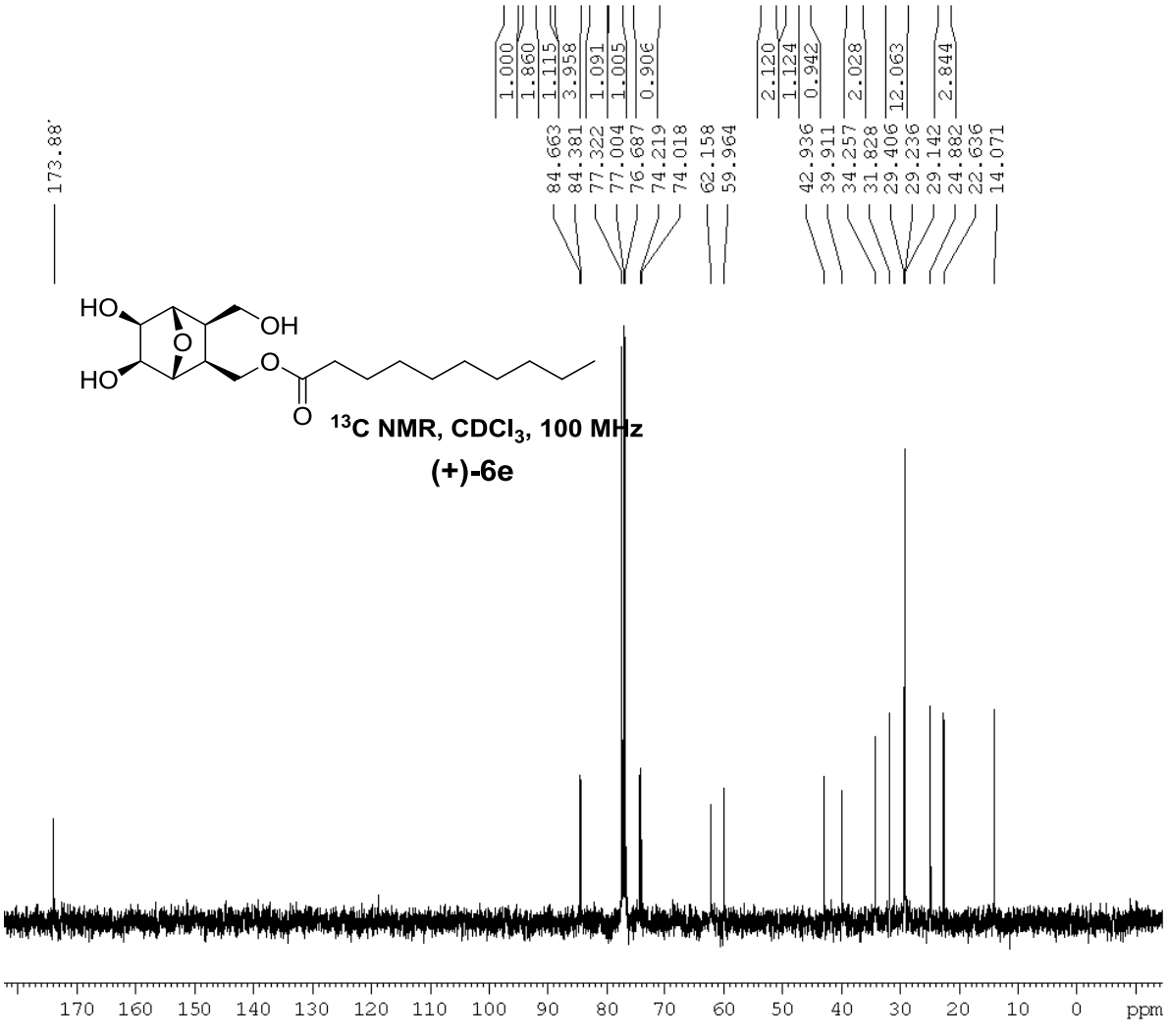


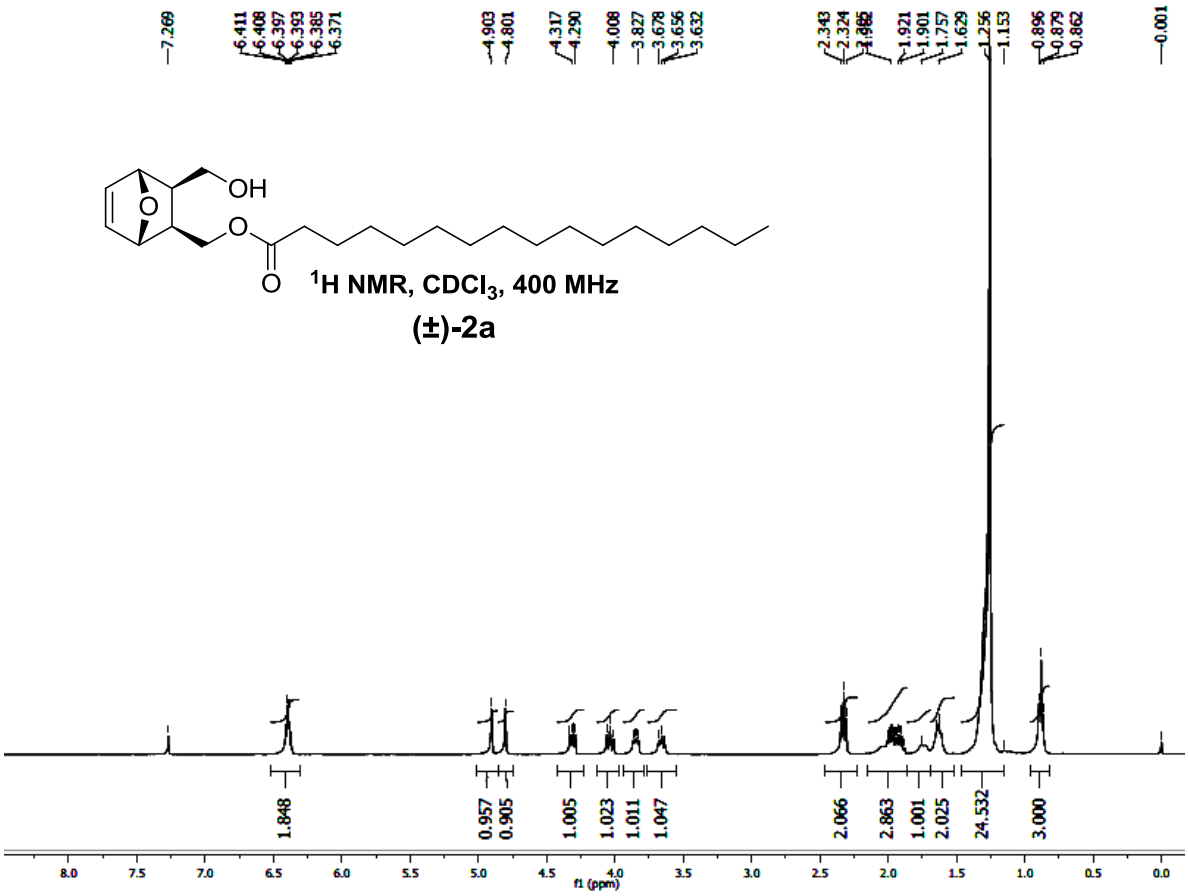
^{1}H NMR 500 MHz
(+)-6d

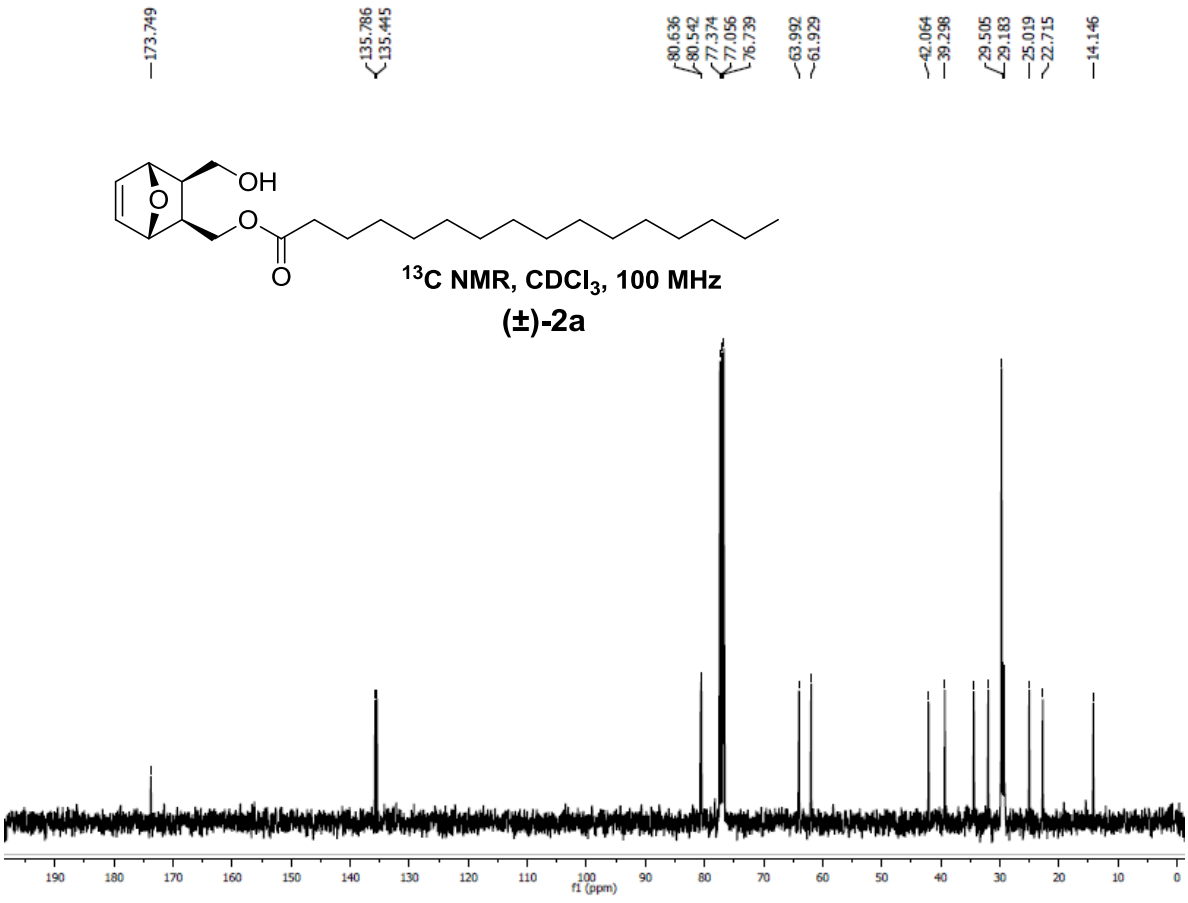


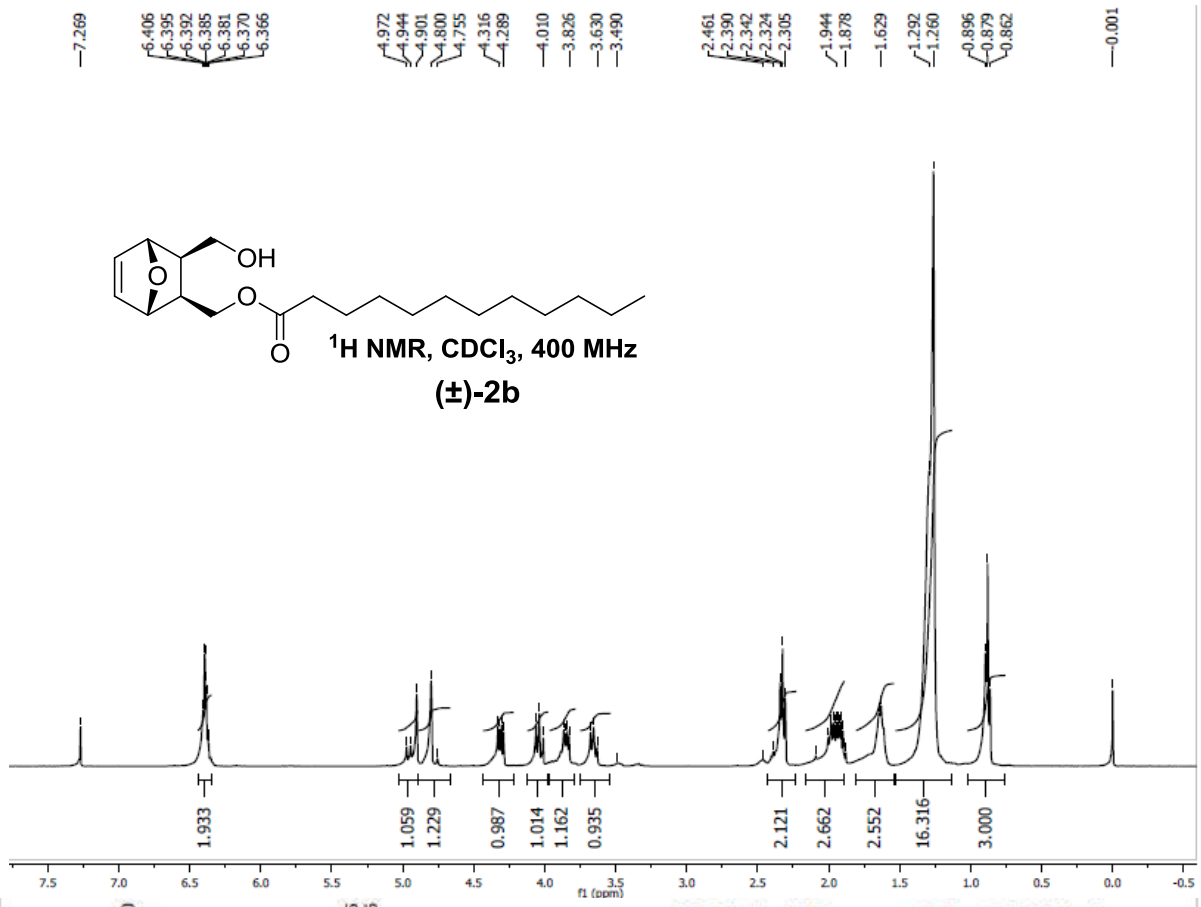
^{13}C NMR 125 MHz
(+)-6d

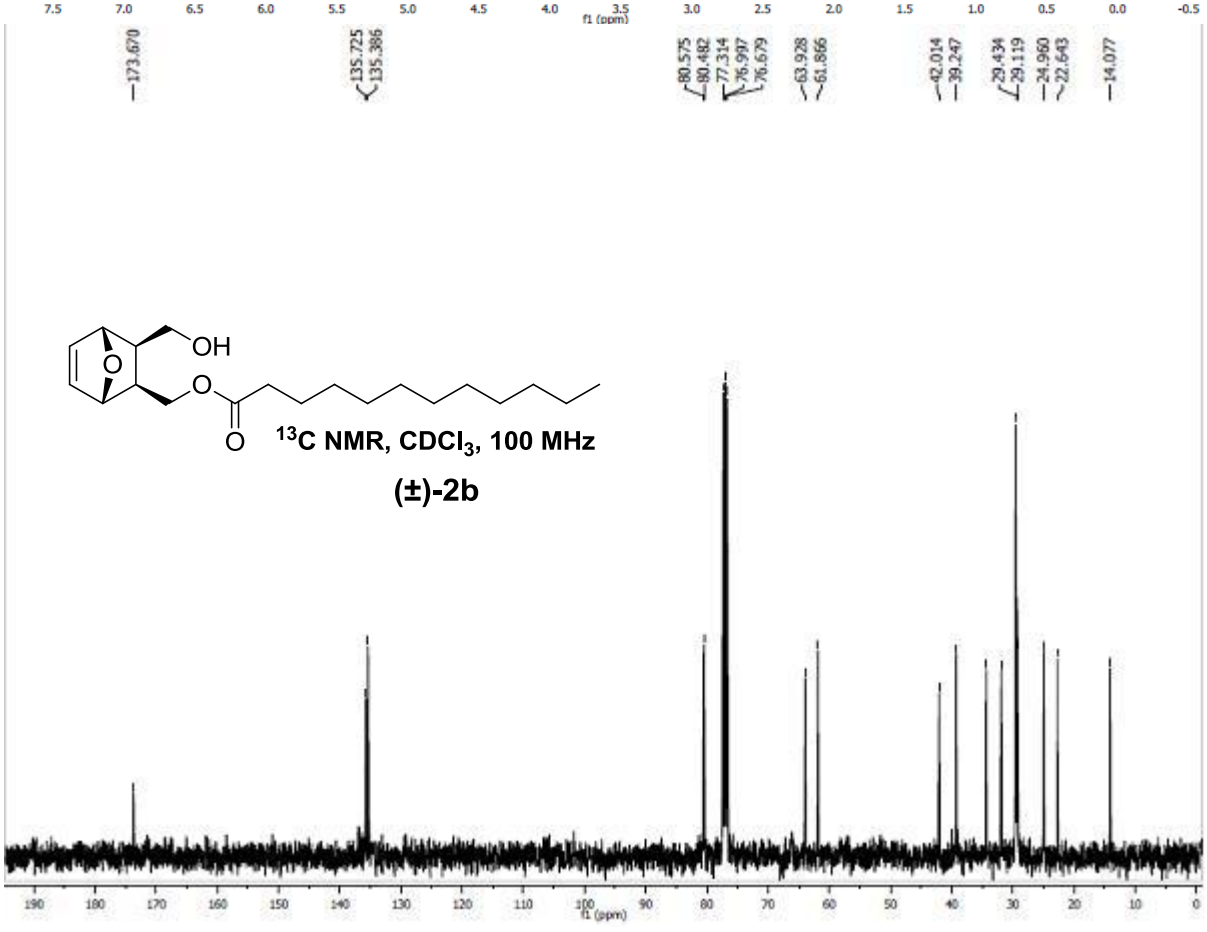


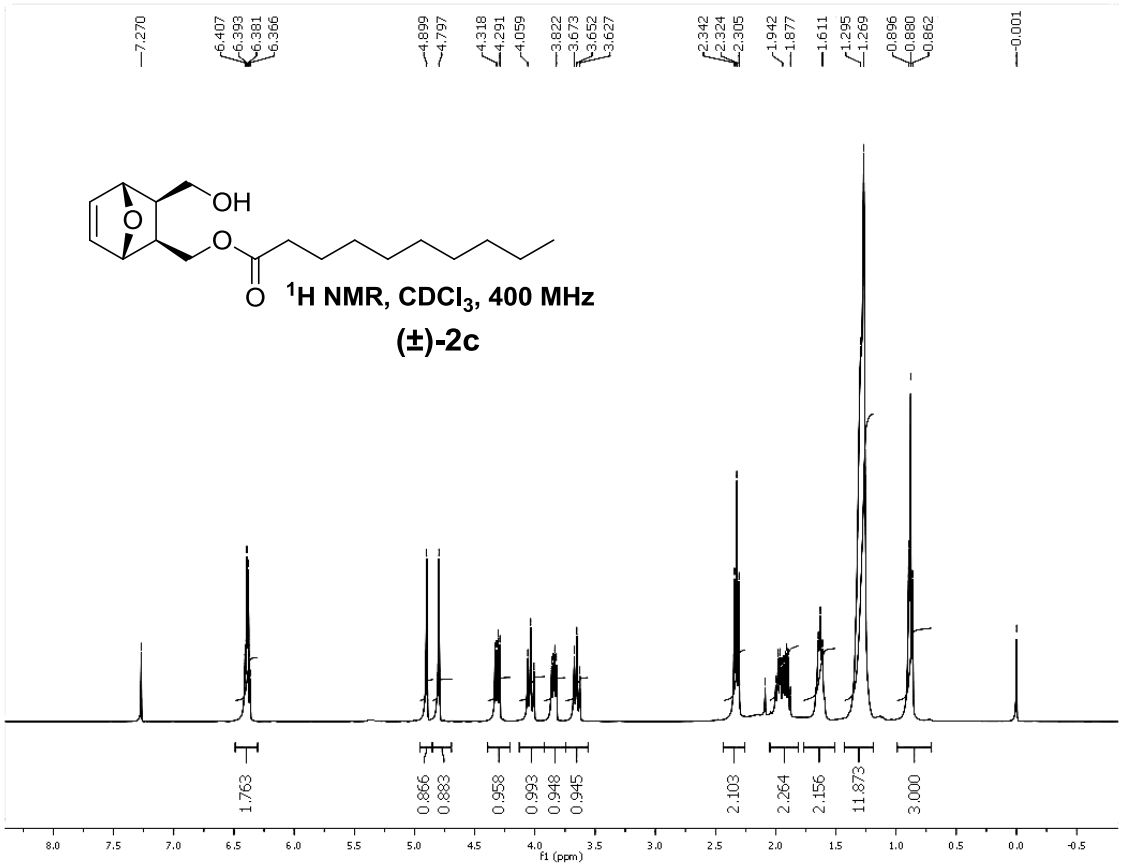


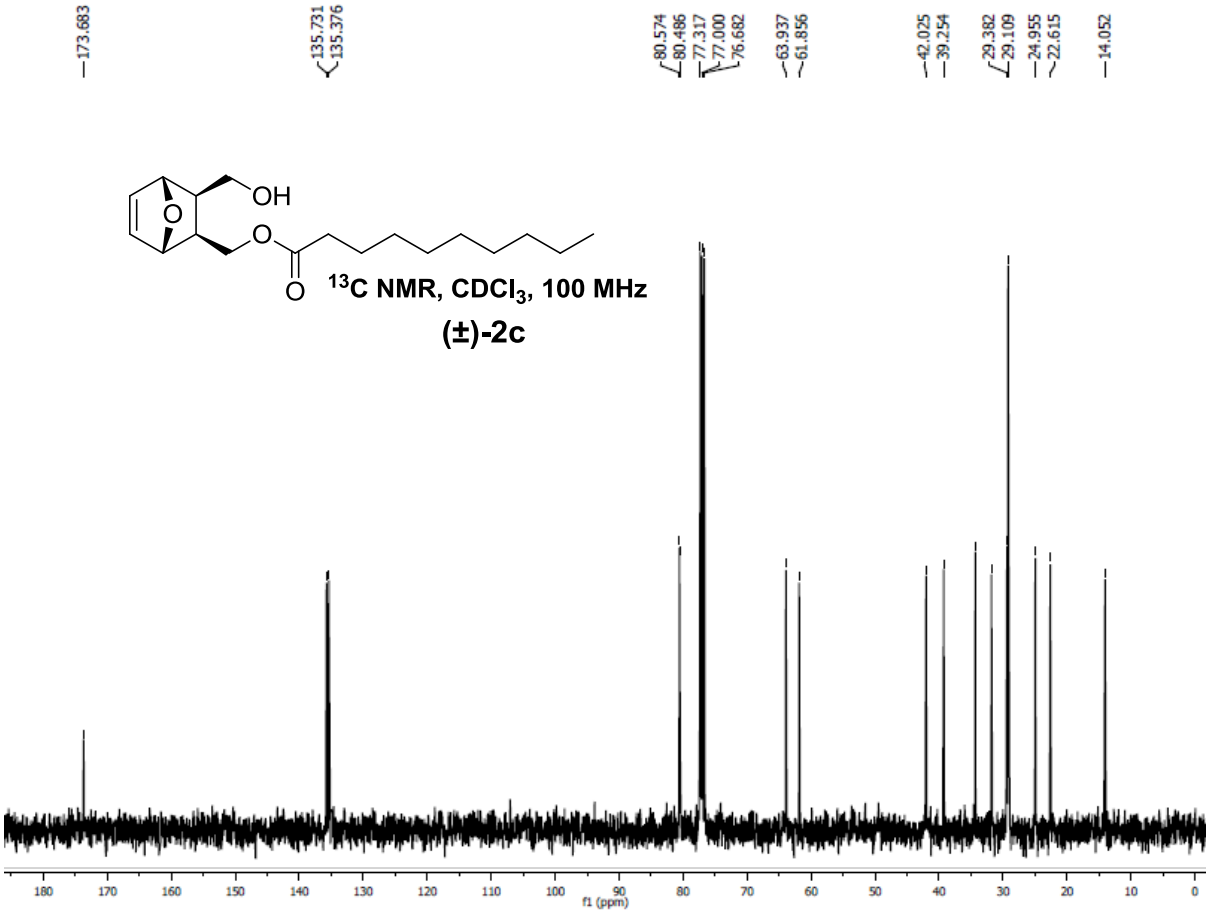


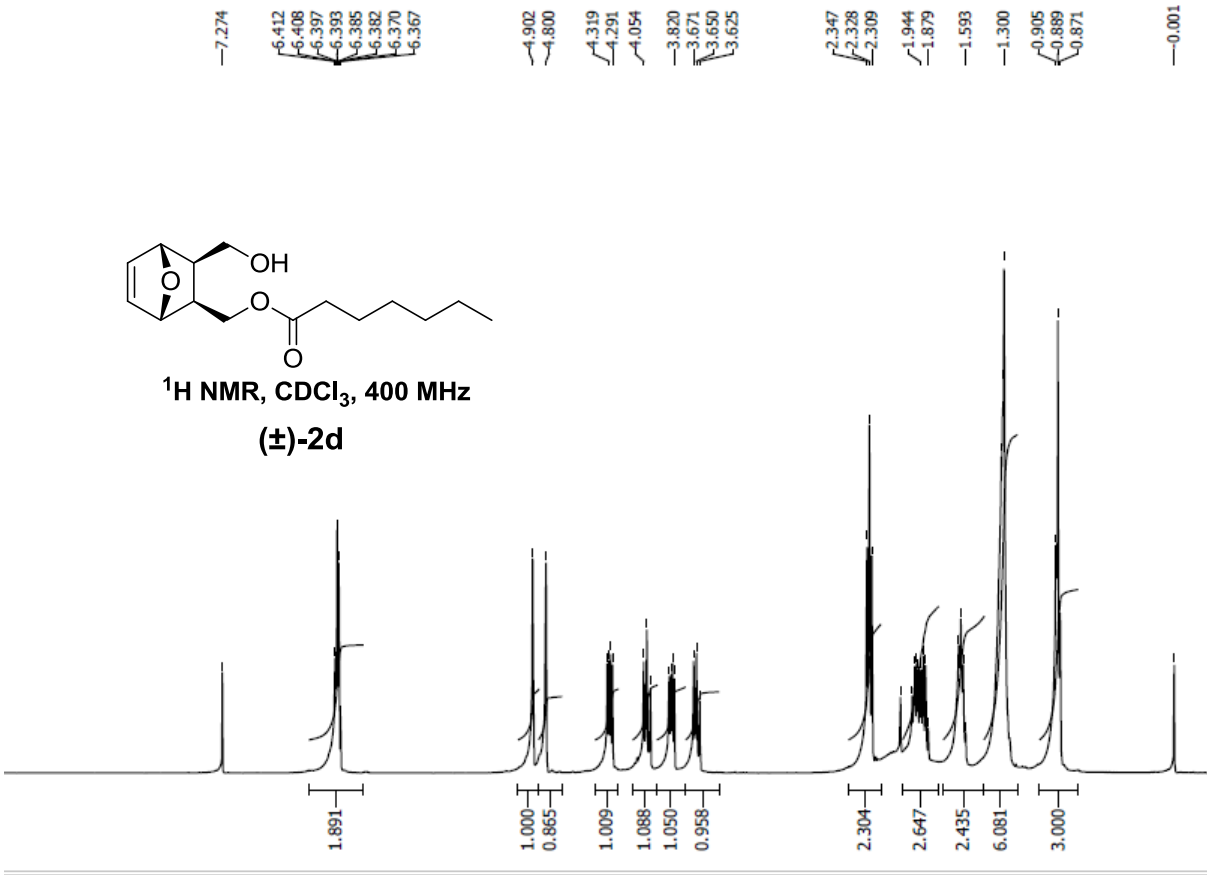


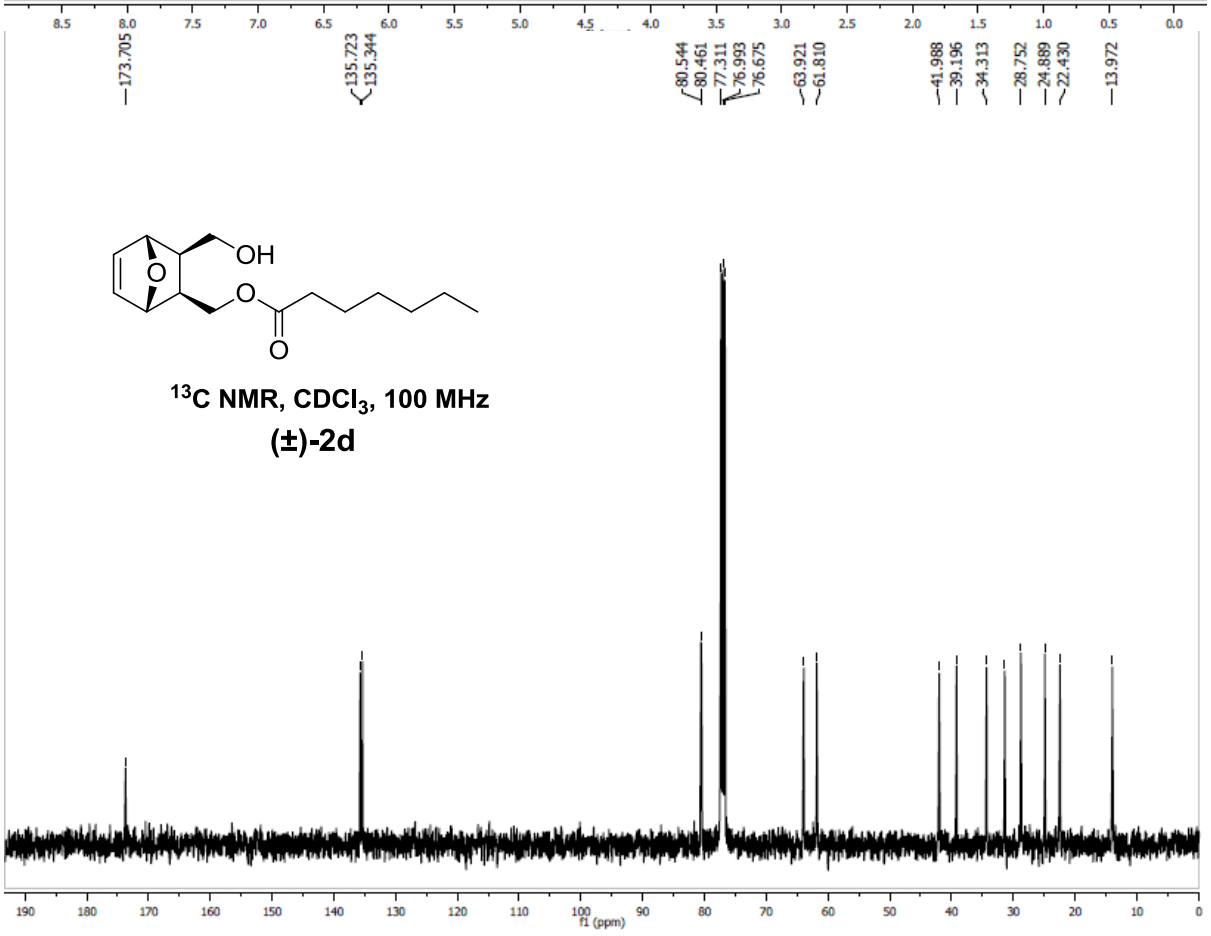


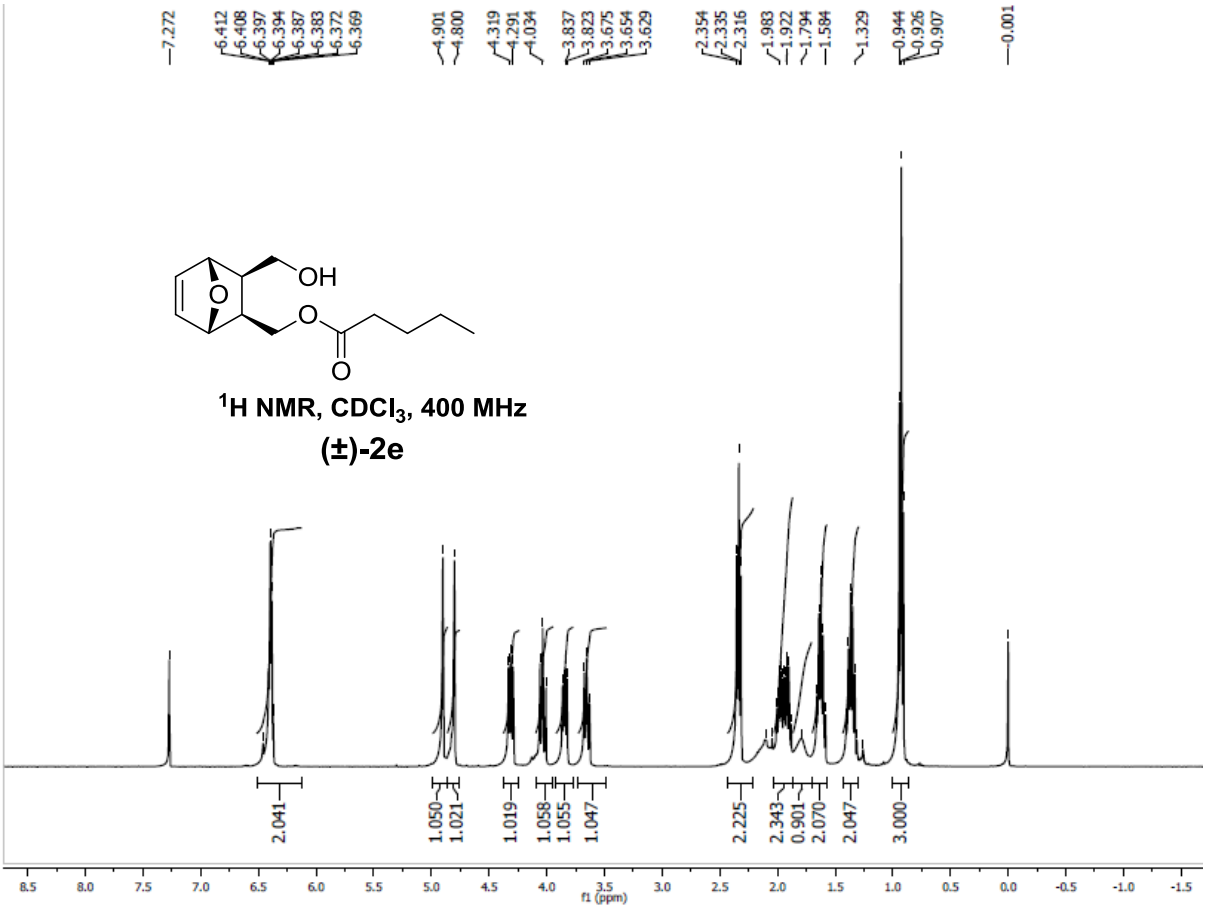






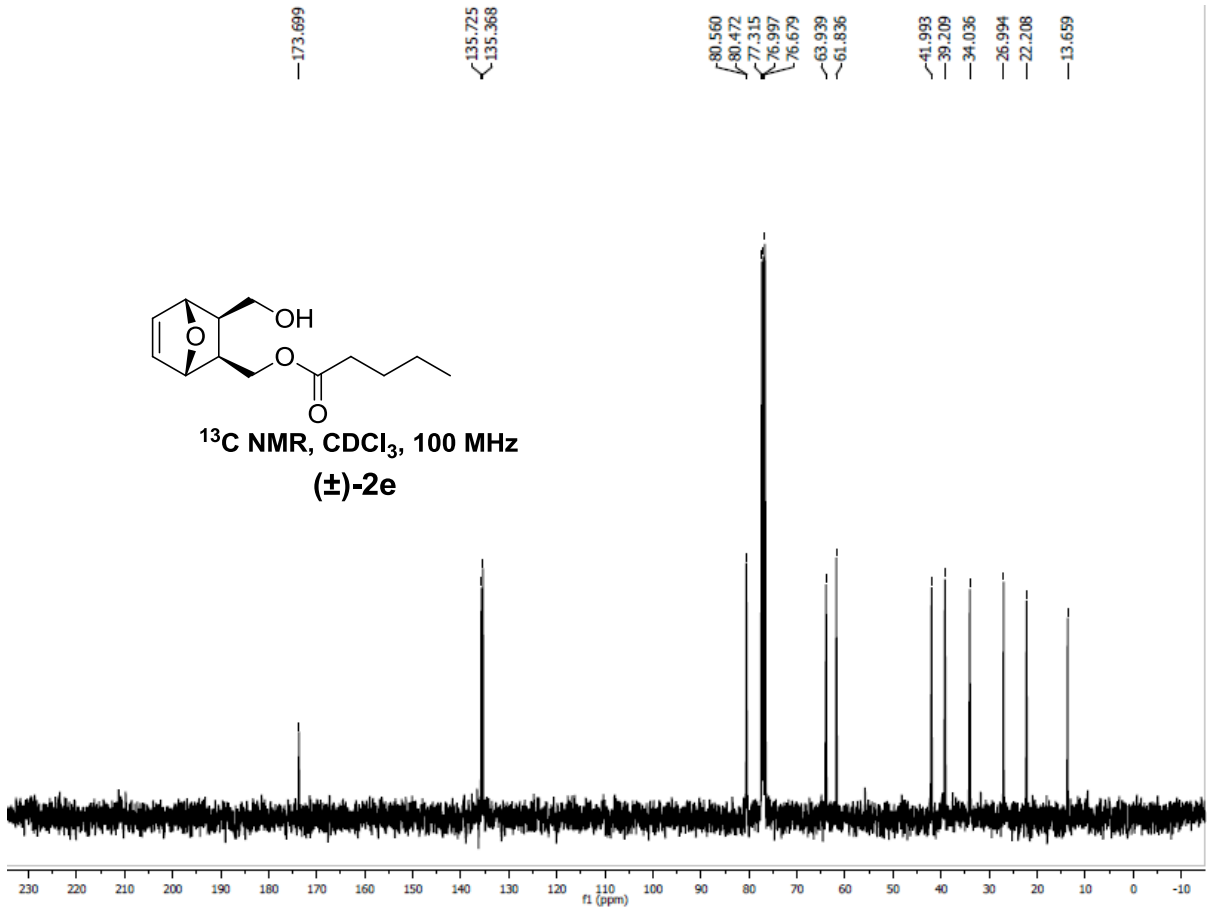






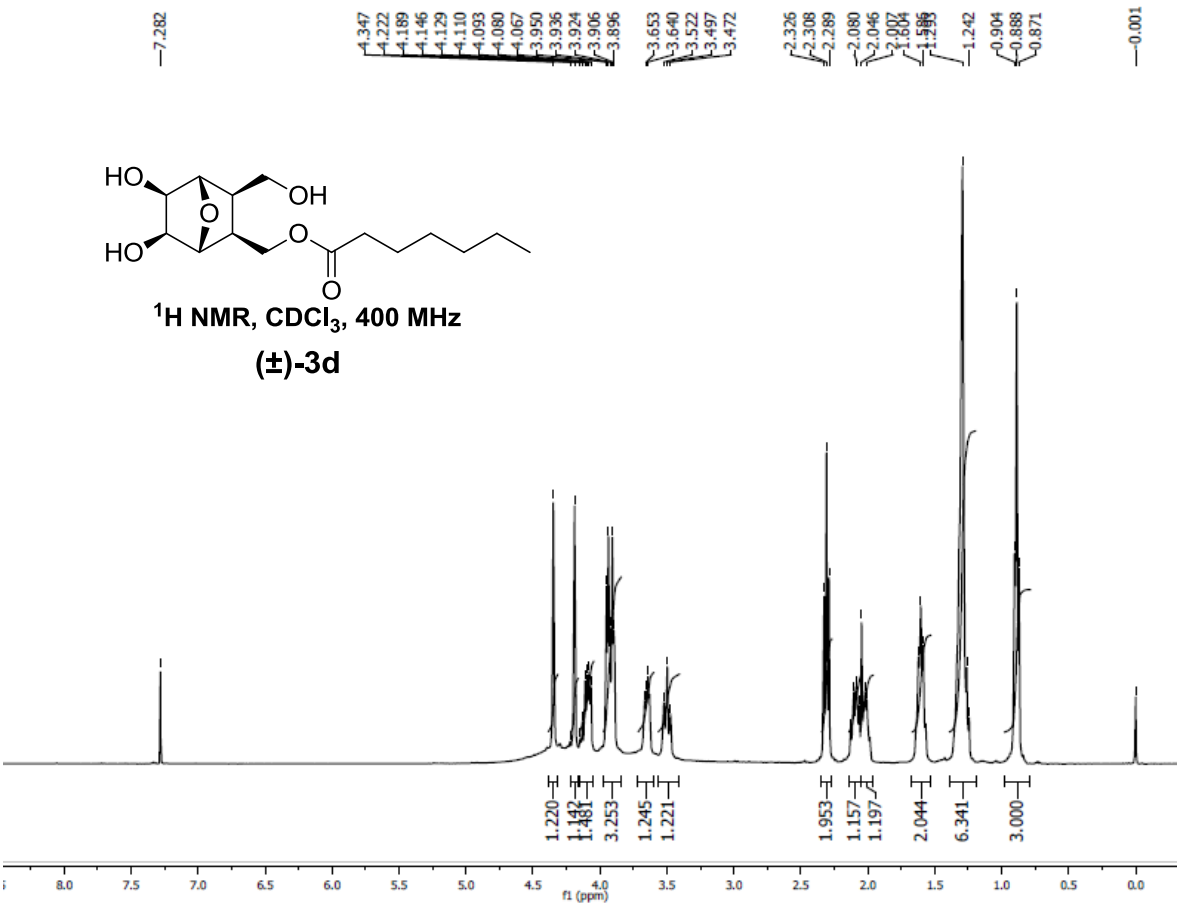
^{1}H NMR, $CDCl_3$, 400 MHz

(±)-2e



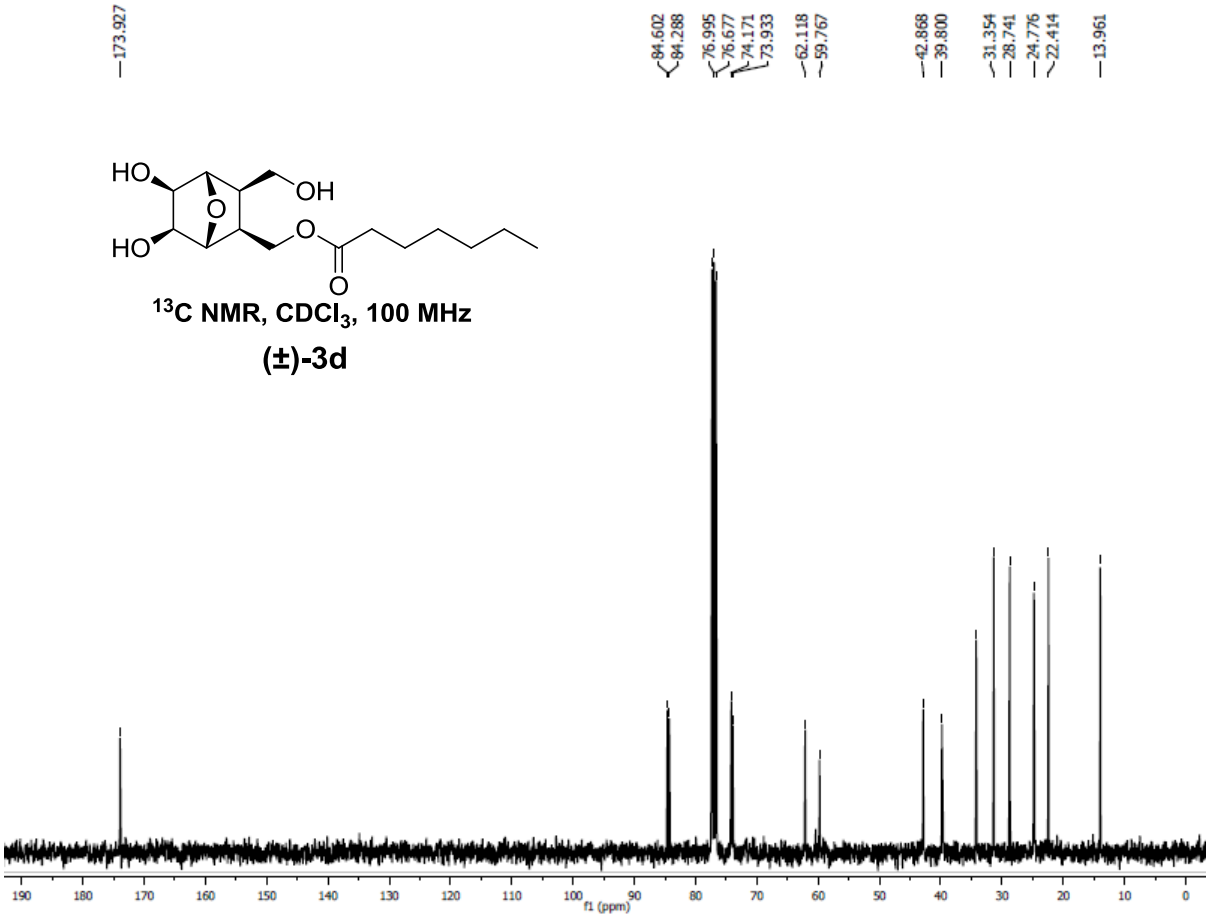
^{13}C NMR, $CDCl_3$, 100 MHz

(±)-2e



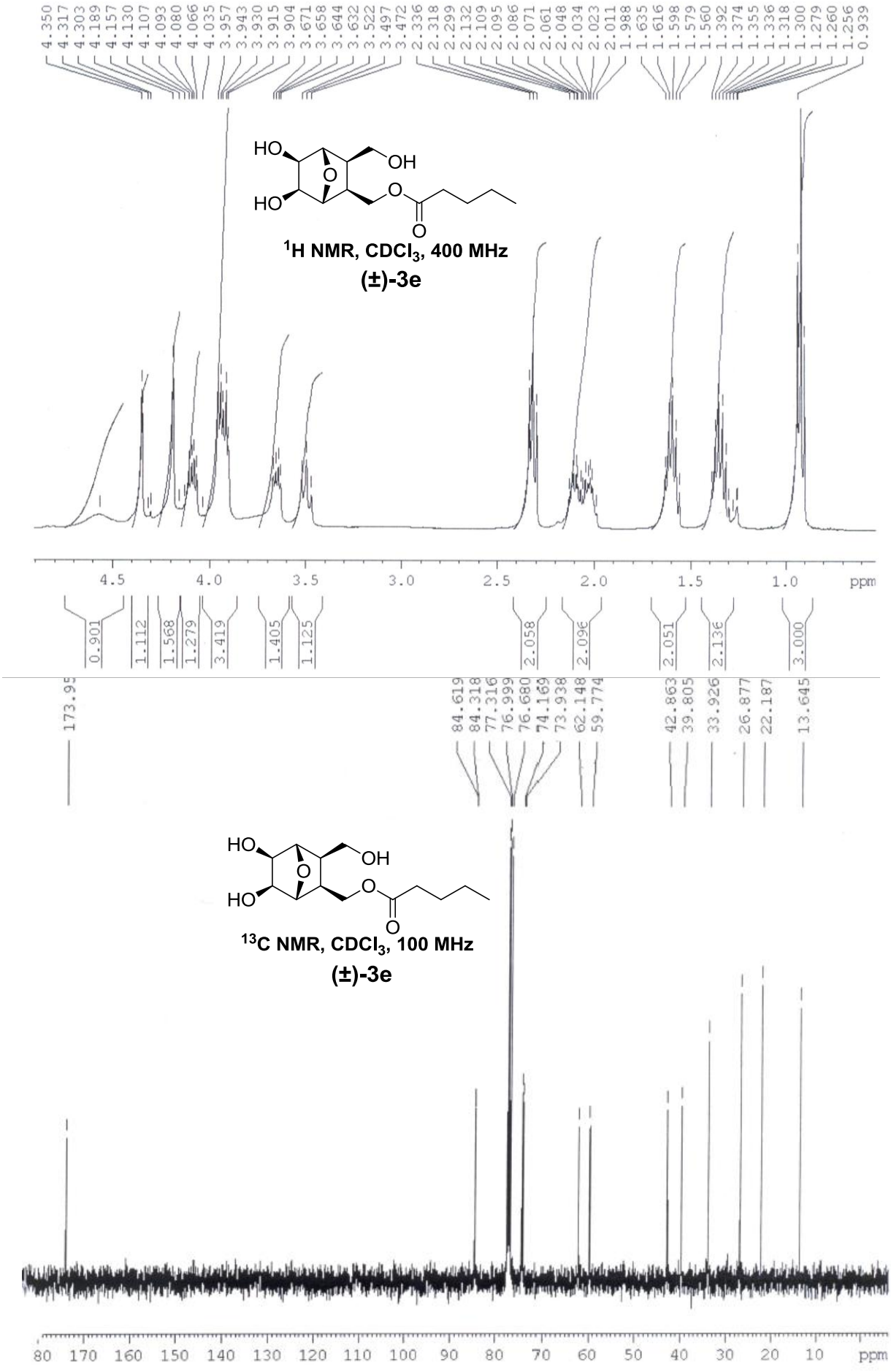
^{1}H NMR, $CDCl_3$, 400 MHz

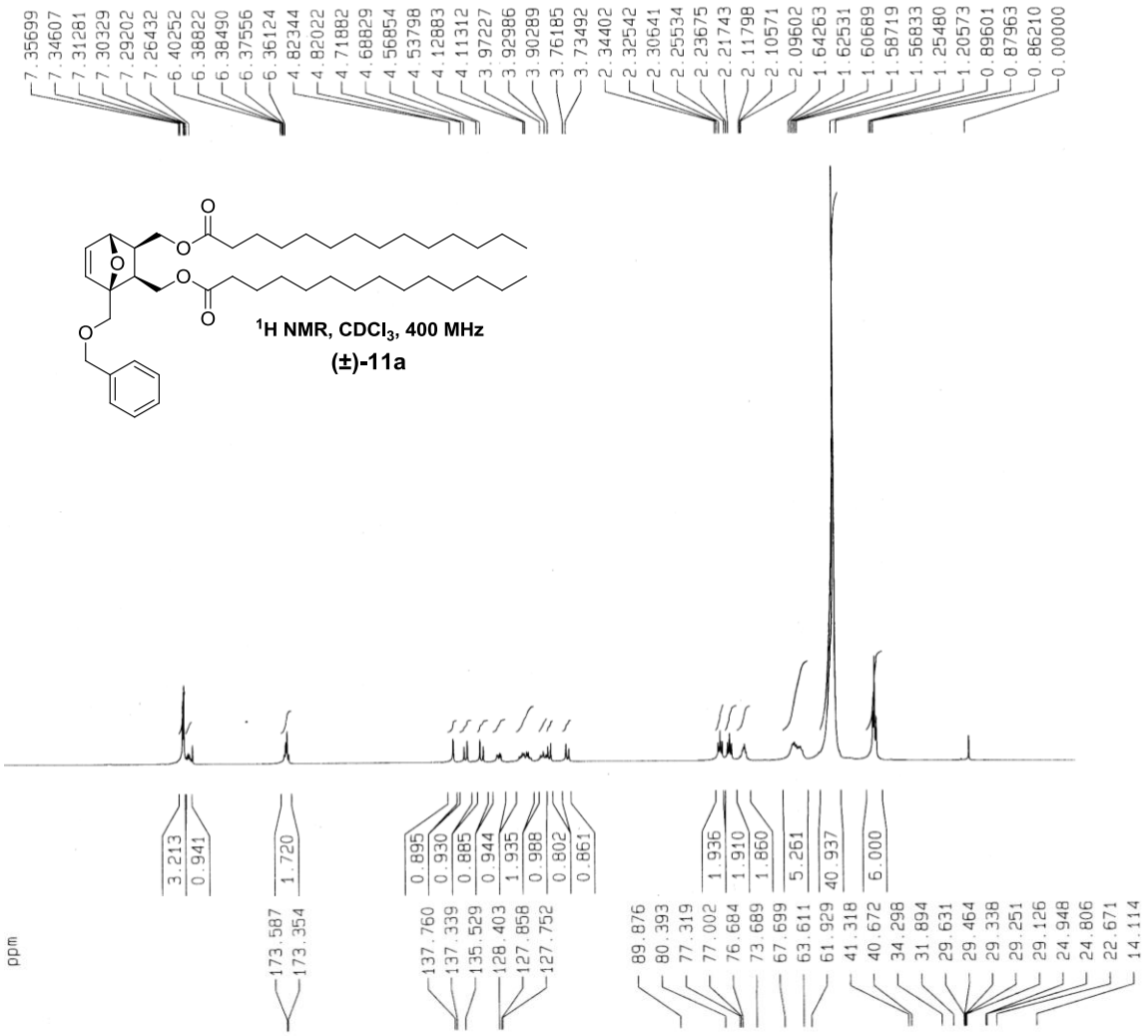
(±)-3d



^{13}C NMR, $CDCl_3$, 100 MHz

(±)-3d

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7.31281
7.30329
7.29202
7.26432
6.40252
6.38822
6.38490
6.37556
6.36124
4.82344
4.82022
4.71882
4.68829
4.56854
4.53798
4.12883
4.11312
3.97227
3.92986
3.90289
3.76185
3.73492
2.34402
2.32542
2.30641
2.25534
2.23675
2.21743
2.11798
2.10571
2.09602
1.64263
1.62531
1.60689
1.58719
1.56833
1.25480
1.20573
0.89601
0.87963
0.86210
0.00000
O
O
O
O
O
1H NMR, CDCl3, 400 MHz
(±)-11a
3.213
0.941
1.720
0.895
0.930
0.885
0.944
1.935
0.988
0.802
0.861
1.936
1.910
1.860
5.261
40.937
6.000
ppm
173.587
173.354
137.760
137.339
135.529
128.403
127.858
127.752
89.876
80.393
77.319
77.002
76.684
73.689
67.699
63.611
61.929
41.318
40.672
34.298
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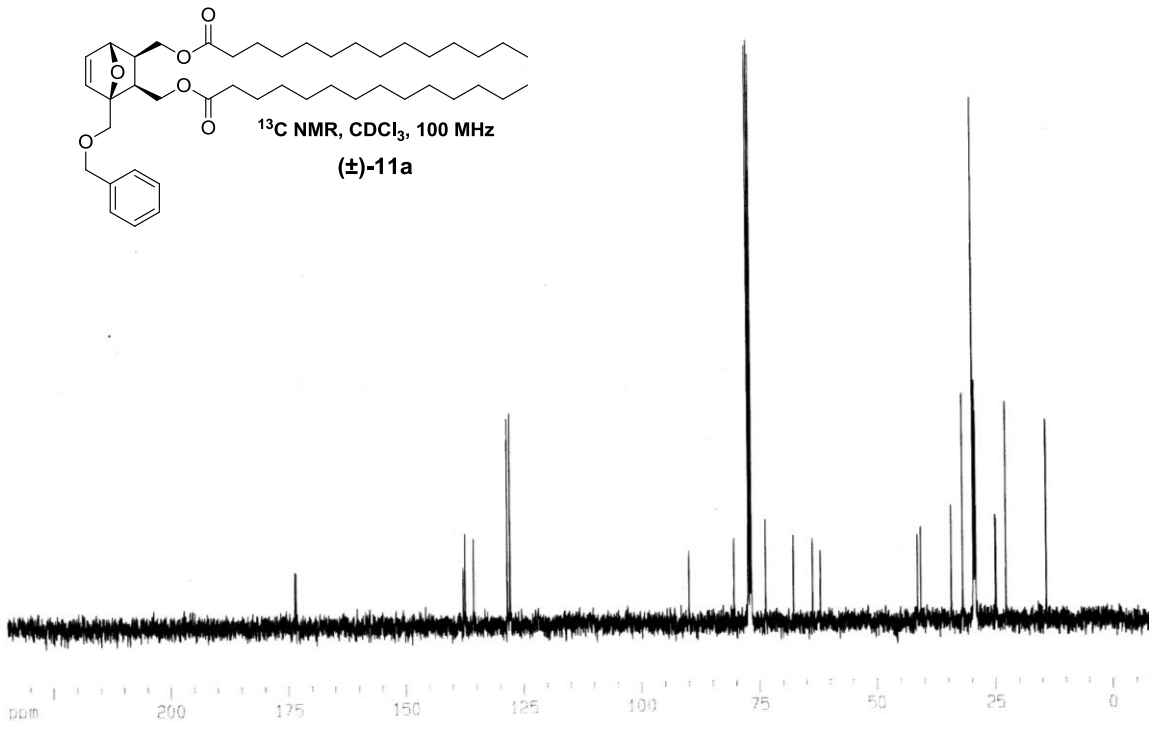
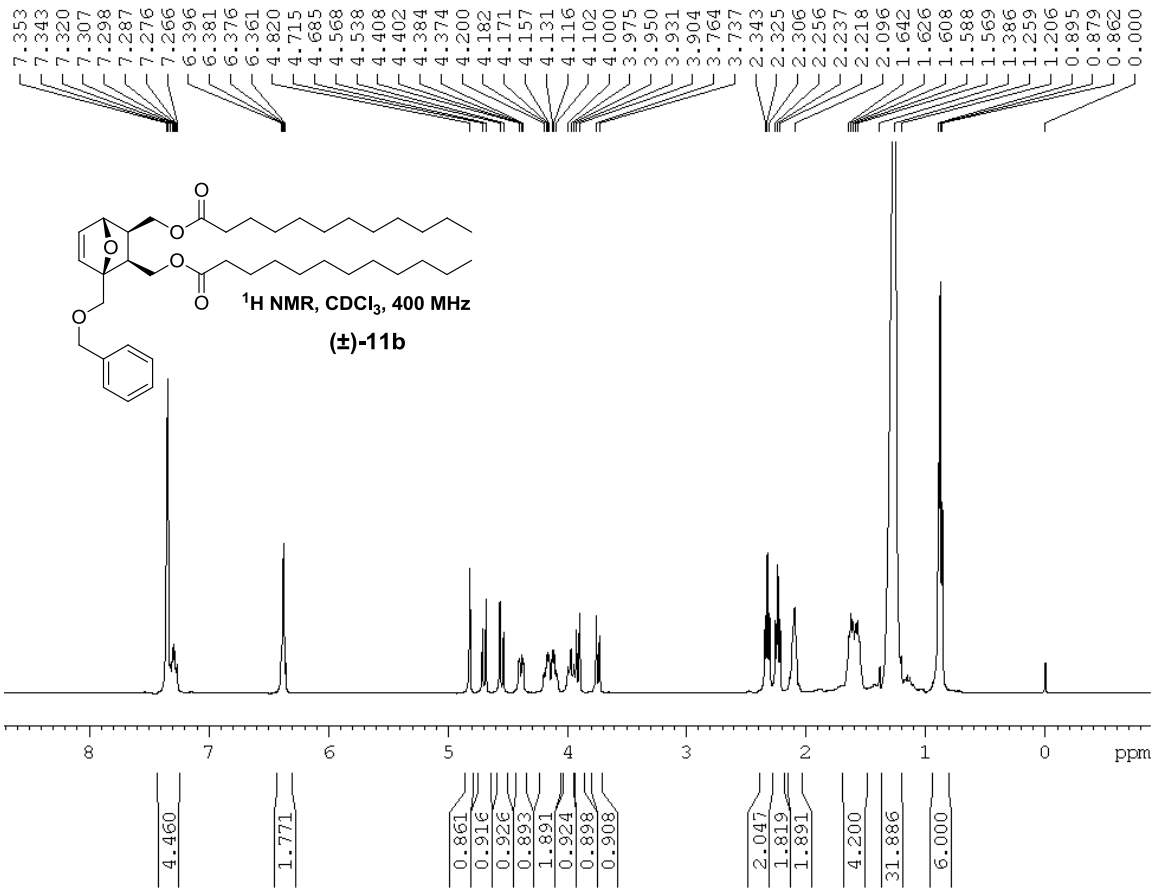
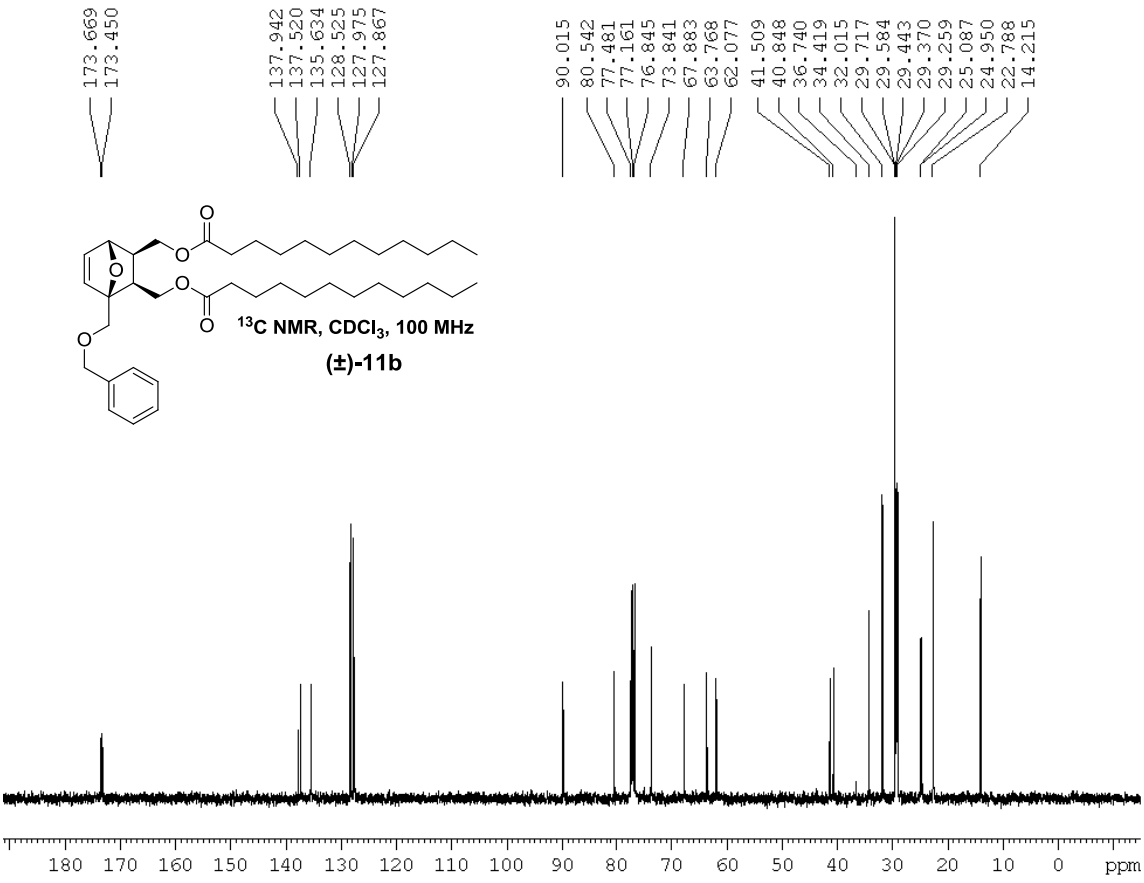
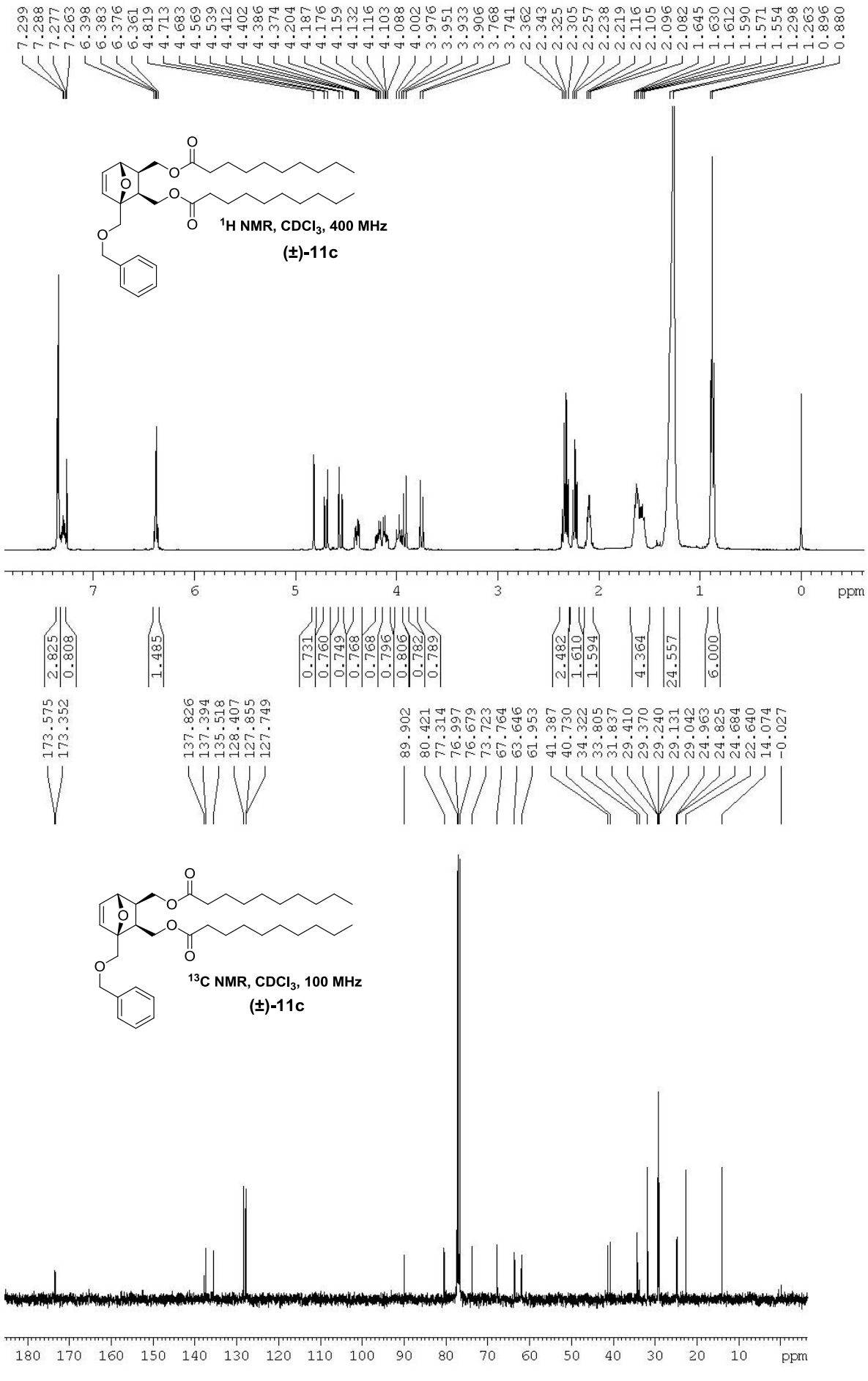
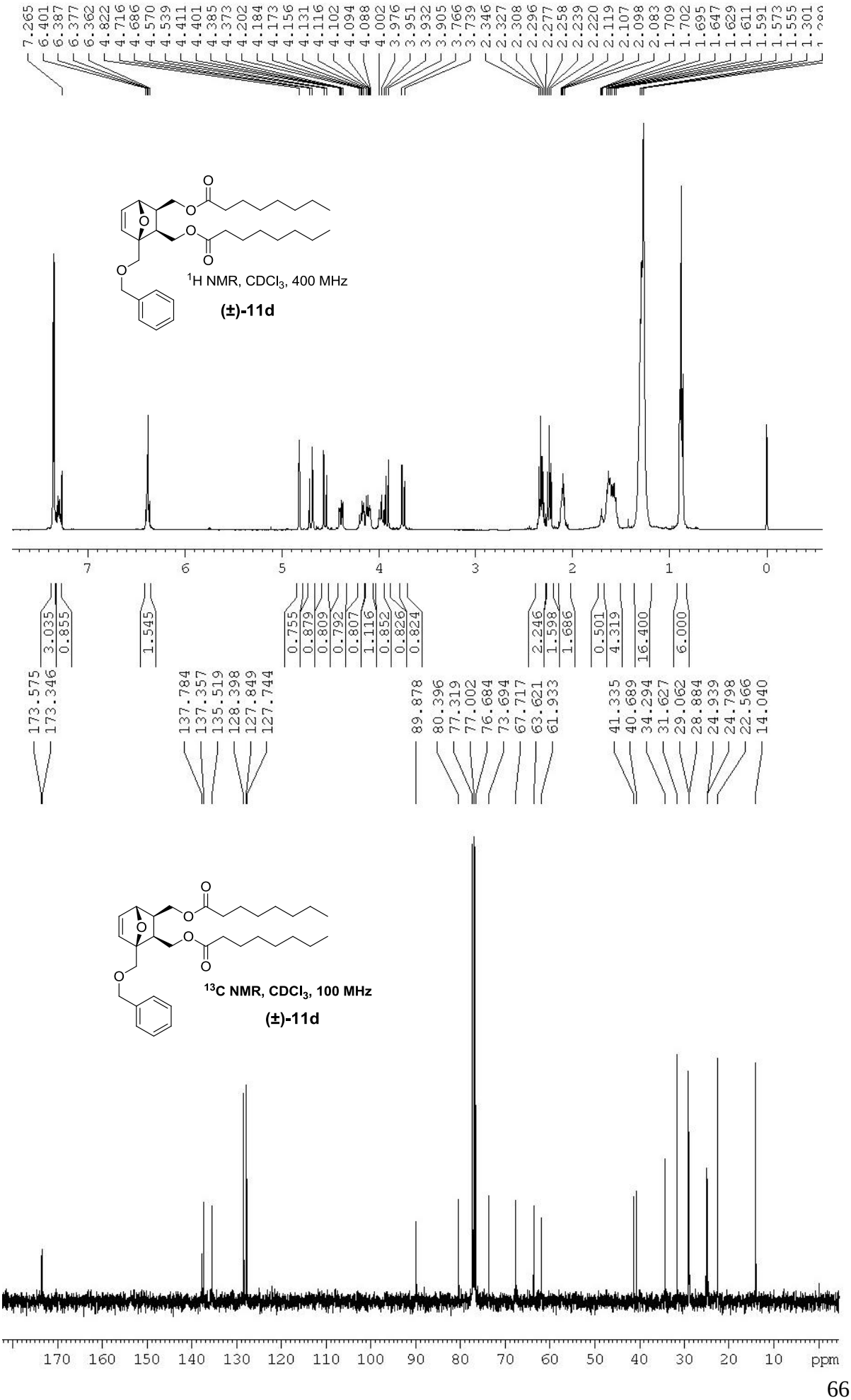
29.631
29.464
29.338
29.251
29.126
24.948
24.806
22.671
14.114


O
O
O
O
O
13C NMR, CDCl3, 100 MHz
(±)-11a
ppm
200
175
150
125
100
75
50
25
0





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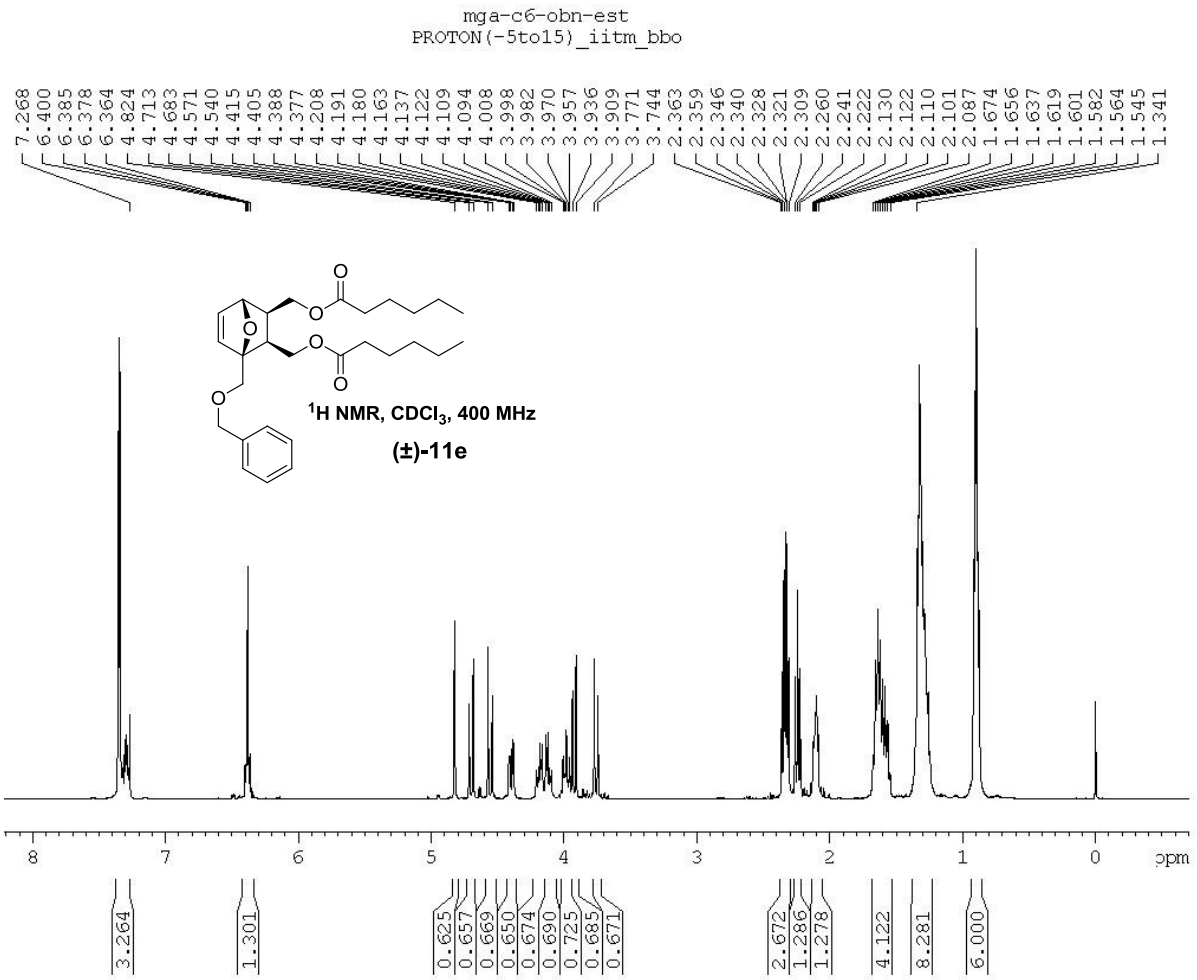
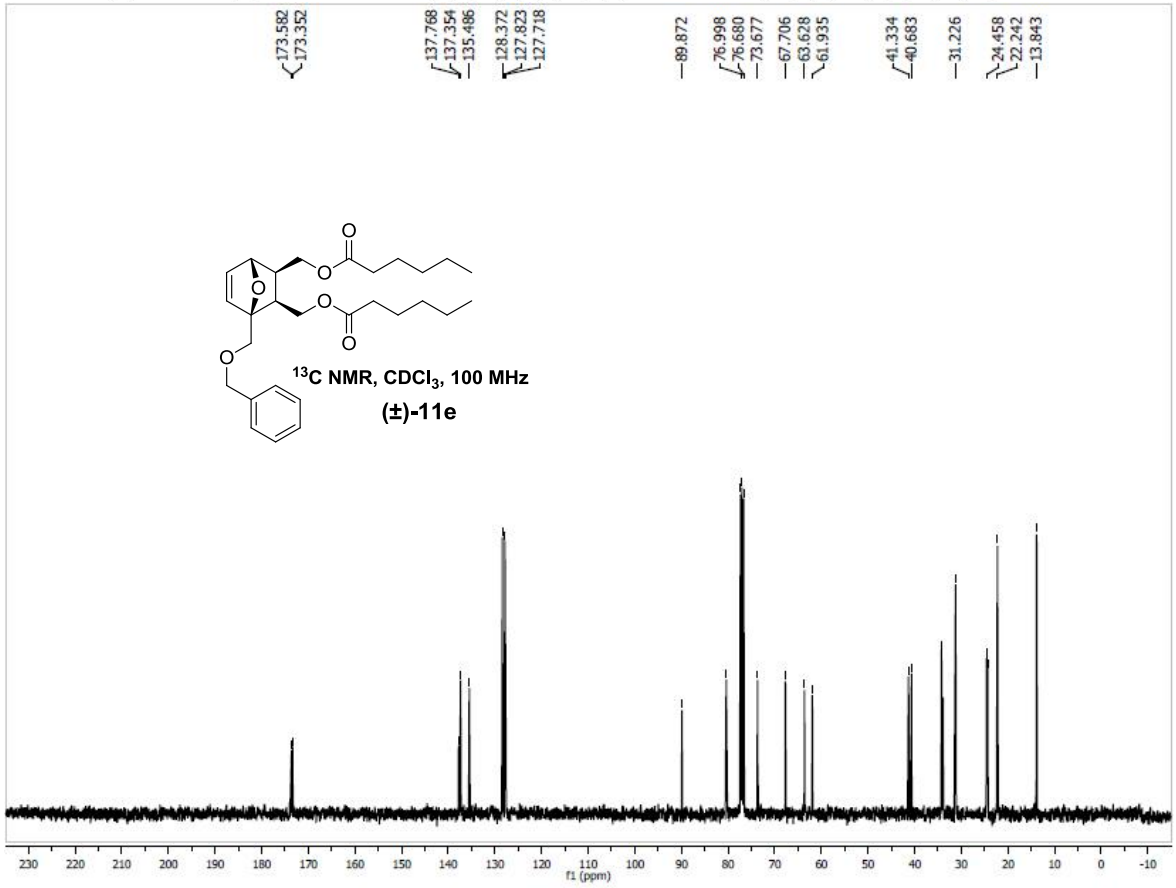
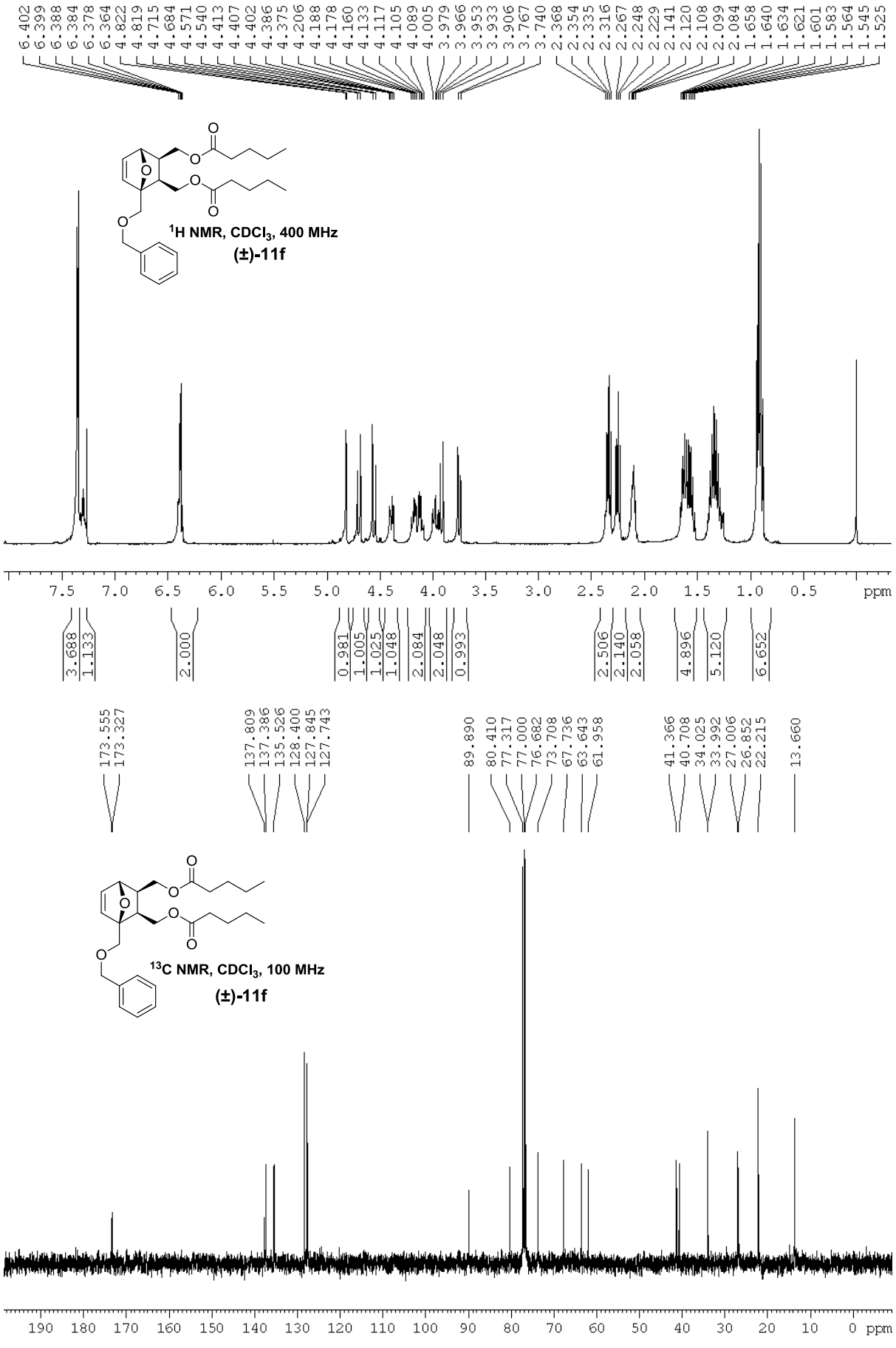
6.387
6.377
6.362
4.822
4.716
4.686
4.570
4.539
4.411
4.401
4.385
4.373
4.202
4.184
4.173
4.156
4.131
4.116
4.102
4.094
4.088
4.002
3.976
3.951
3.932
3.905
3.766
3.739
2.346
2.327
2.308
2.296
2.277
2.258
2.239
2.220
2.119
2.107
2.098
2.083
1.709
1.702
1.695
1.647
1.629
1.611
1.591
1.573
1.555
1.301
O
O
O
O
O
O
1H NMR, CDCl3, 400 MHz
(±)-11d
7
6
5
4
3
2
1
0
3.035
0.855
1.545
0.755
0.879
0.809
0.792
0.807
1.116
0.852
0.826
0.824
2.246
1.598
1.686
0.501
4.319
16.400
6.000
173.575
173.346
137.784
137.357
135.519
128.398
127.849
127.744
89.878
80.396
77.319
77.002
76.684
73.694
67.717
63.621
61.933
41.335
40.689
34.294
31.627
29.062
28.884
24.939
24.798
22.566
14.040
O
O
O
O
O
O
13C NMR, CDCl3, 100 MHz
(±)-11d
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


mga-c6-obn-est
PROTON(-5to15)_iitm_bbo
^{1}H NMR, CDCl$_3$, 400 MHz
(±)-11e


^{13}C NMR, CDCl$_3$, 100 MHz
(±)-11e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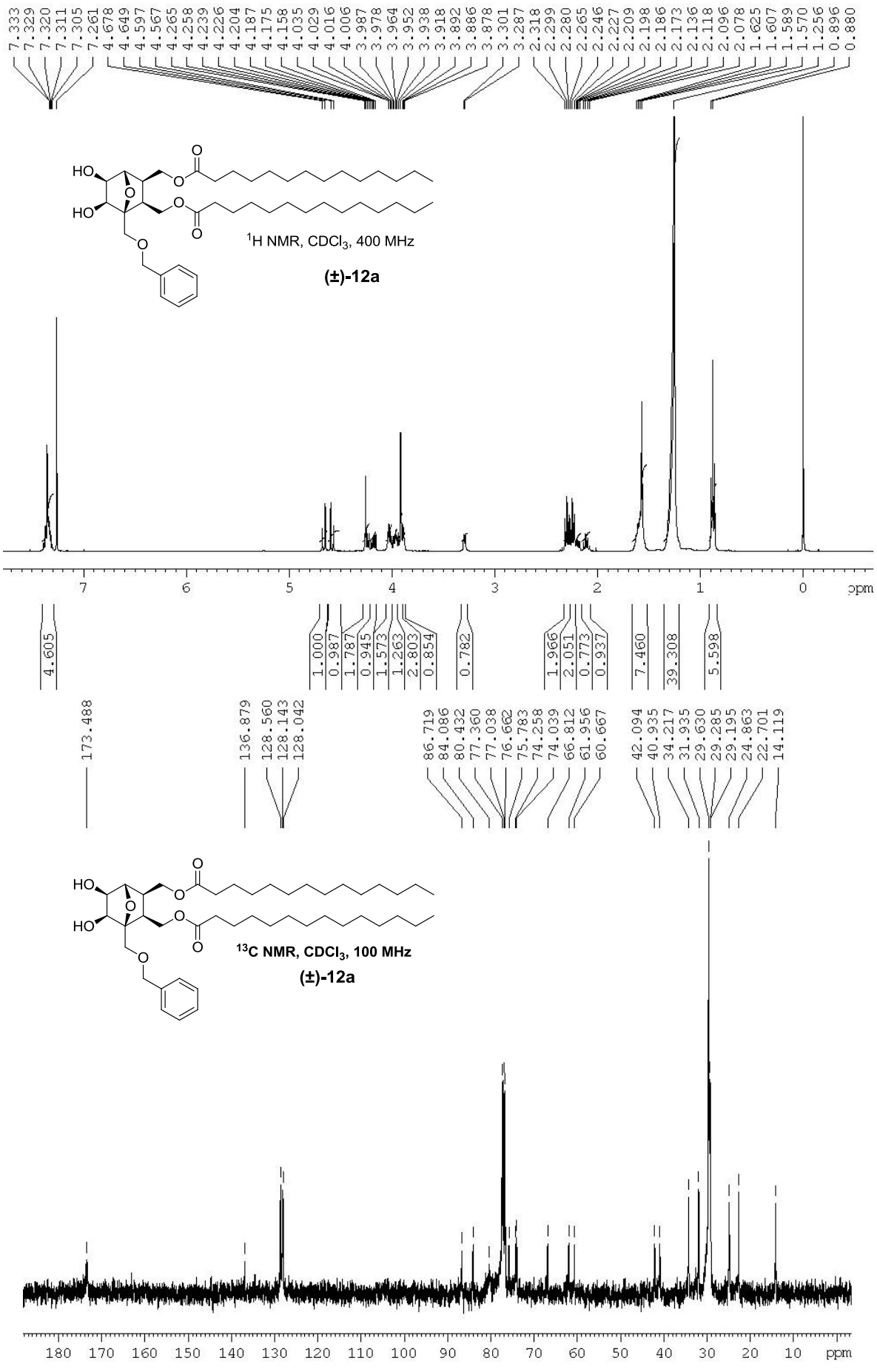
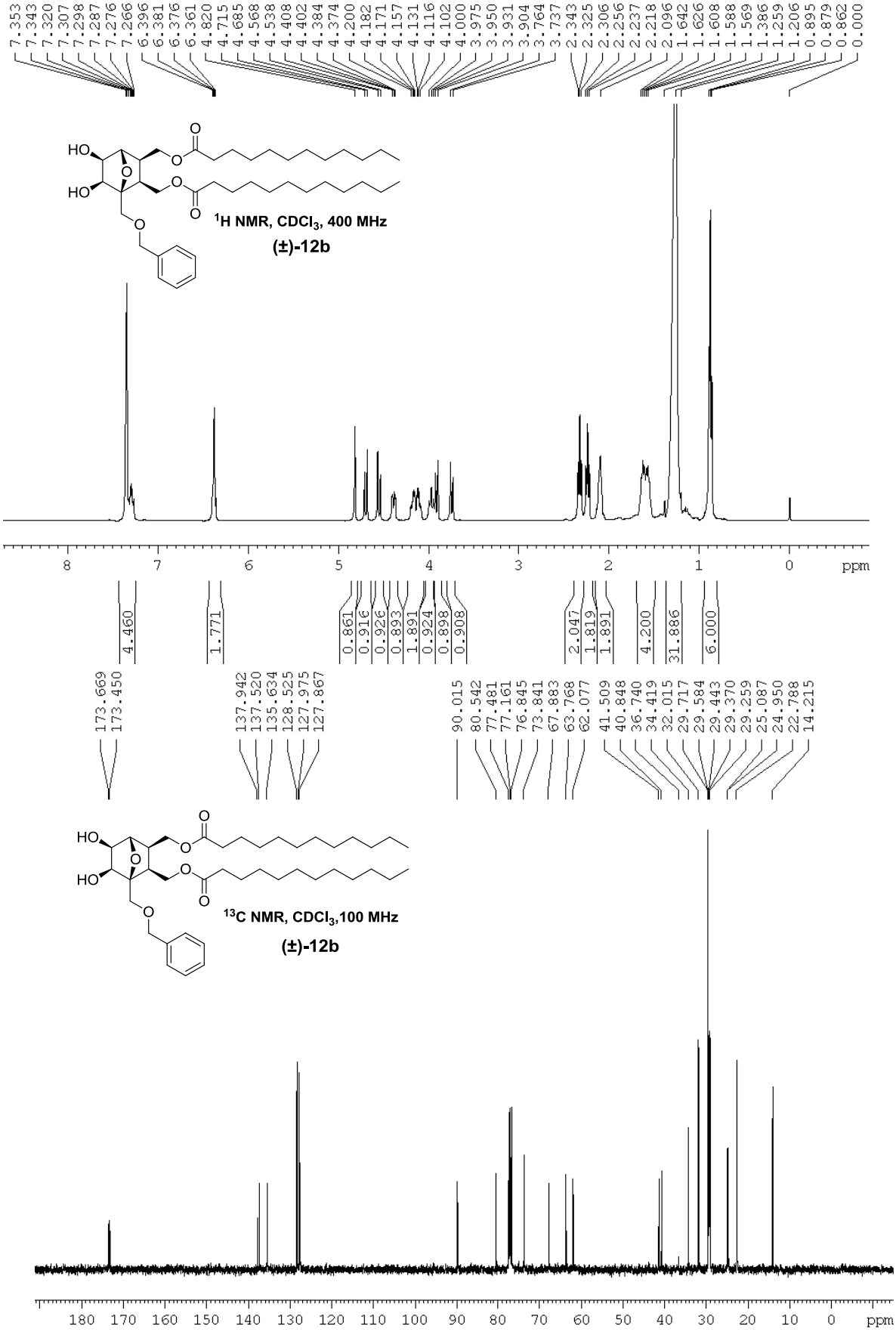
6.399
6.388
6.384
6.378
6.364
4.822
4.819
4.715
4.684
4.571
4.540
4.413
4.407
4.402
4.386
4.375
4.206
4.188
4.178
4.160
4.133
4.117
4.105
4.089
4.005
3.979
3.966
3.953
3.933
3.906
3.767
3.740
2.368
2.354
2.335
2.316
2.267
2.248
2.229
2.141
2.120
2.108
2.099
2.084
1.658
1.640
1.634
1.621
1.601
1.583
1.564
1.545
1.525
1H NMR, CDCl3, 400 MHz
(±)-11f
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
3.688
1.133
2.000
0.981
1.005
1.025
1.048
2.084
2.048
0.993
2.506
2.140
2.058
4.896
5.120
6.652
173.555
173.327
137.809
137.386
135.526
128.400
127.845
127.743
89.890
80.410
77.317
77.000
76.682
73.708
67.736
63.643
61.958
41.366
40.708
34.025
33.992
27.006
26.852
22.215
13.660
13C NMR, CDCl3, 100 MHz
(±)-11f
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

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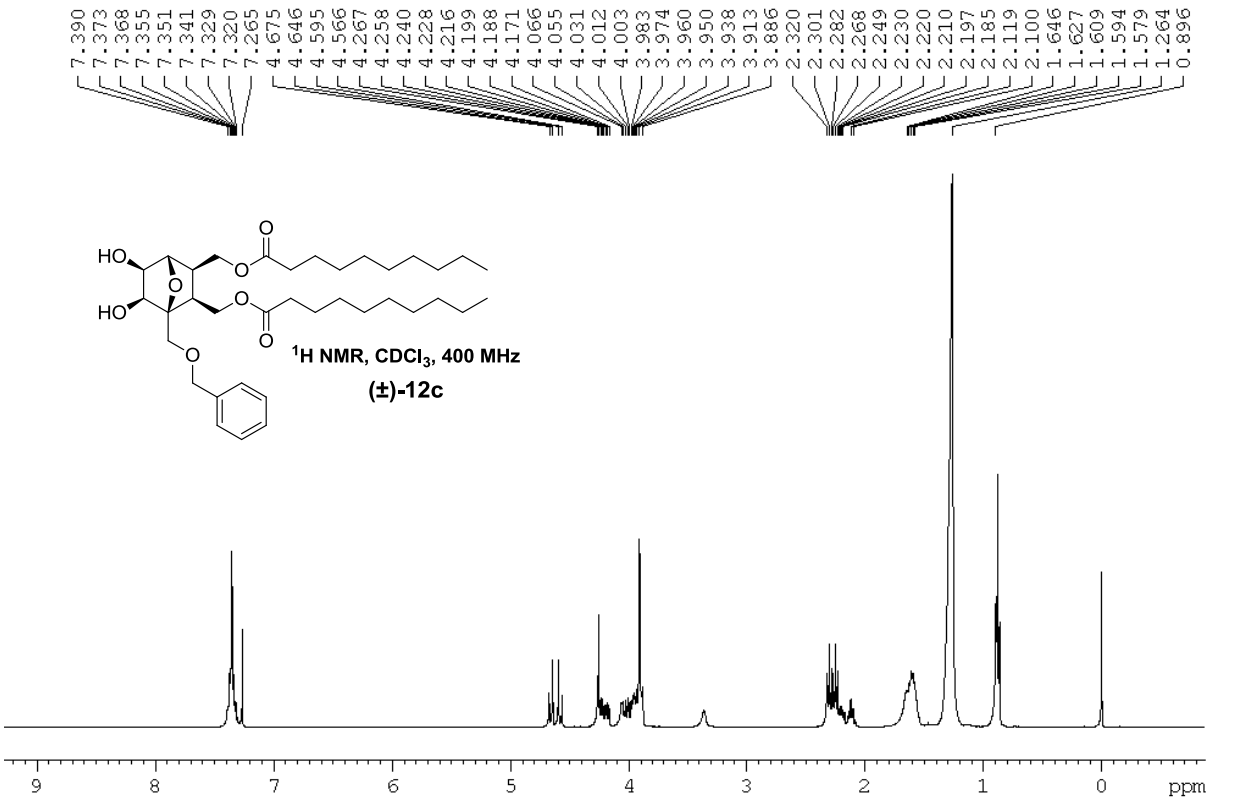
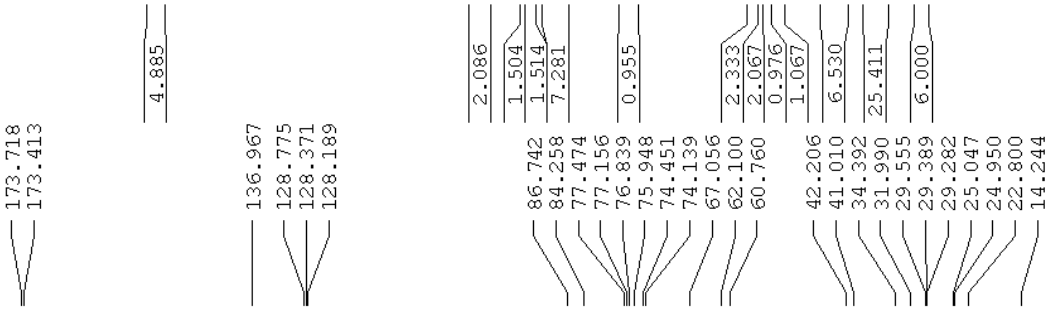
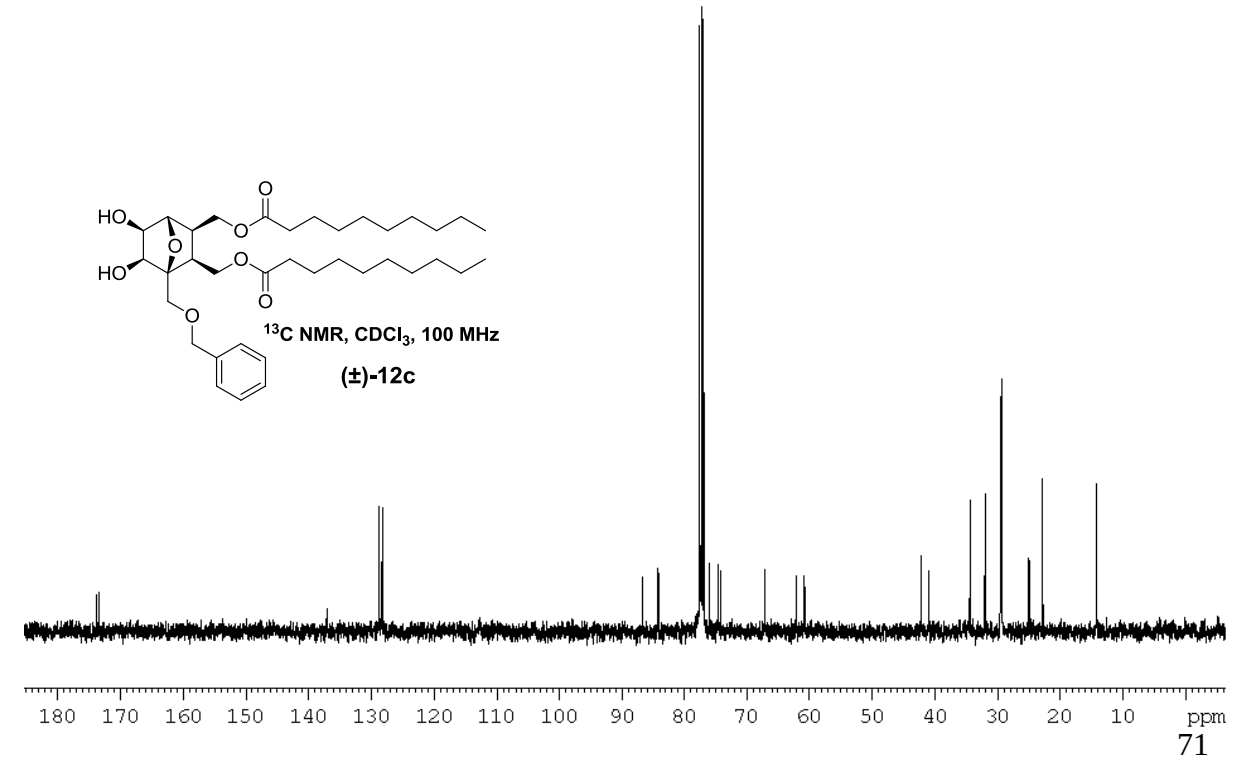
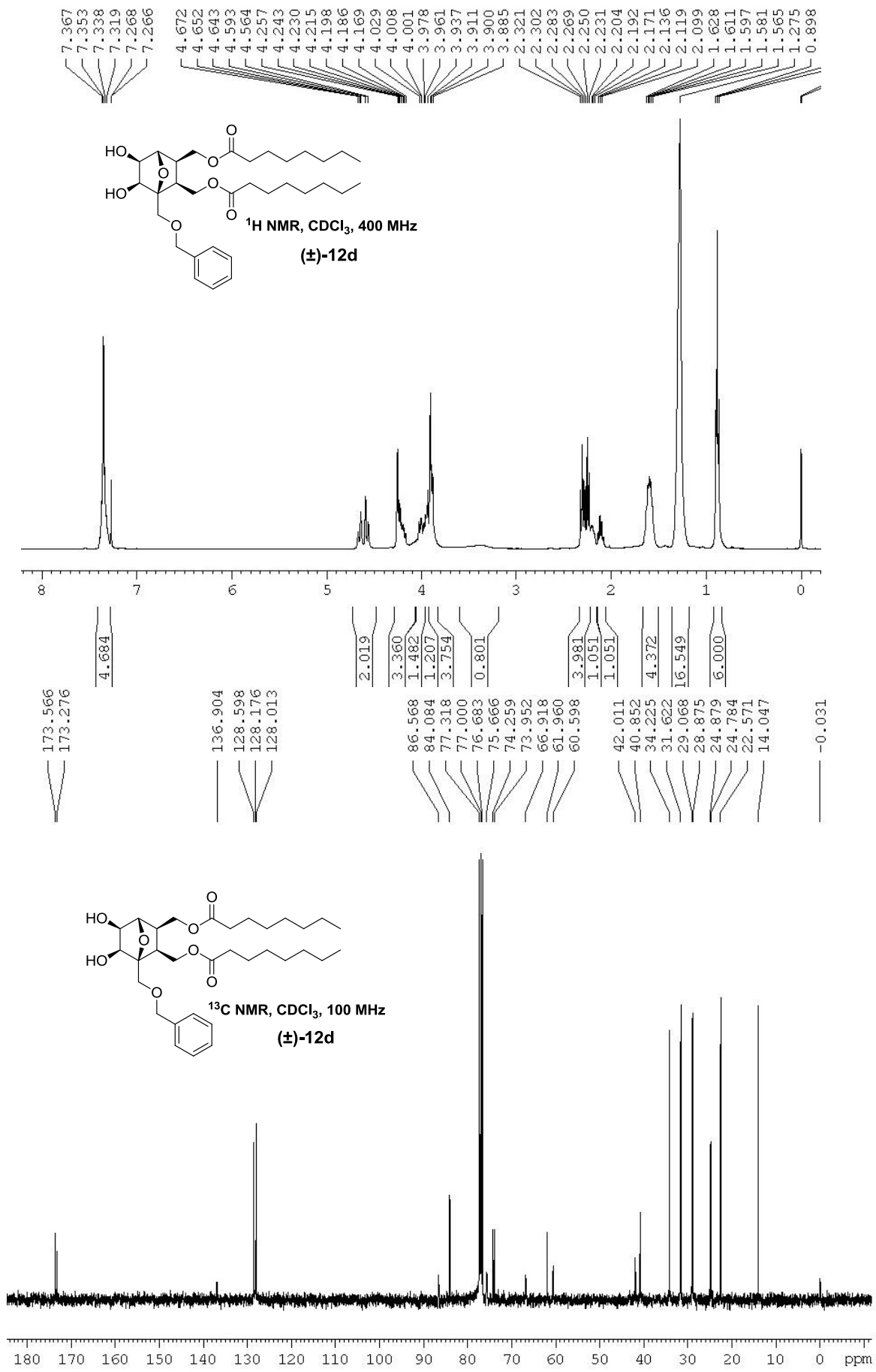
7.343
7.320
7.307
7.298
7.287
7.276
7.266
6.396
6.381
6.376
6.361
4.820
4.715
4.685
4.568
4.538
4.408
4.402
4.384
4.374
4.200
4.182
4.171
4.157
4.131
4.116
4.102
4.000
3.975
3.950
3.931
3.904
3.764
3.737
2.343
2.325
2.306
2.256
2.237
2.218
2.096
1.642
1.626
1.608
1.588
1.569
1.386
1.259
1.206
0.895
0.879
0.862
0.000
HO
HO
O
O
O
O
O
O
^{1}H NMR, CDCl$_3$, 400 MHz
(±)-12b
8
7
6
5
4
3
2
1
0
ppm
4.460
1.771
0.861
0.916
0.926
0.893
1.891
0.924
0.898
0.908
2.047
1.819
1.891
4.200
31.886
6.000
173.669
173.450
137.942
137.520
135.634
128.525
127.975
127.867
90.015
80.542
77.481
77.161
76.845
73.841
67.883
63.768
62.077
41.509
40.848
36.740
34.419
32.015
29.717
29.584
29.443
29.370
29.259
25.087
24.950
22.788
14.215
HO
HO
O
O
O
O
O
O
^{13}C NMR, CDCl$_3$,100 MHz
(±)-12b
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.390
7.373
7.368
7.355
7.351
7.341
7.329
7.320
7.265
4.675
4.646
4.595
4.566
4.267
4.258
4.240
4.228
4.216
4.199
4.188
4.171
4.066
4.055
4.031
4.012
4.003
3.983
3.974
3.960
3.950
3.938
3.913
3.886
2.320
2.301
2.282
2.268
2.249
2.230
2.220
2.210
2.197
2.185
2.119
2.100
1.646
1.627
1.609
1.594
1.579
1.264
0.896
HO
HO
O
O
O
O
O
O
^{1}H NMR, CDCl$_3$, 400 MHz
(±)-12c
9
8
7
6
5
4
3
2
1
0
ppm


4.885
2.086
1.504
1.514
7.281
0.955
2.333
2.067
0.976
1.067
6.530
25.411
6.000

173.718
173.413
136.967
128.775
128.371
128.189
86.742
84.258
77.474
77.156
76.839
75.948
74.451
74.139
67.056
62.100
60.760
42.206
41.010
34.392
31.990
29.555
29.389
29.282
25.047
24.950
22.800
14.244


HO
HO
O
O
O
O
O
O
^{13}C NMR, CDCl$_3$, 100 MHz
(±)-12c
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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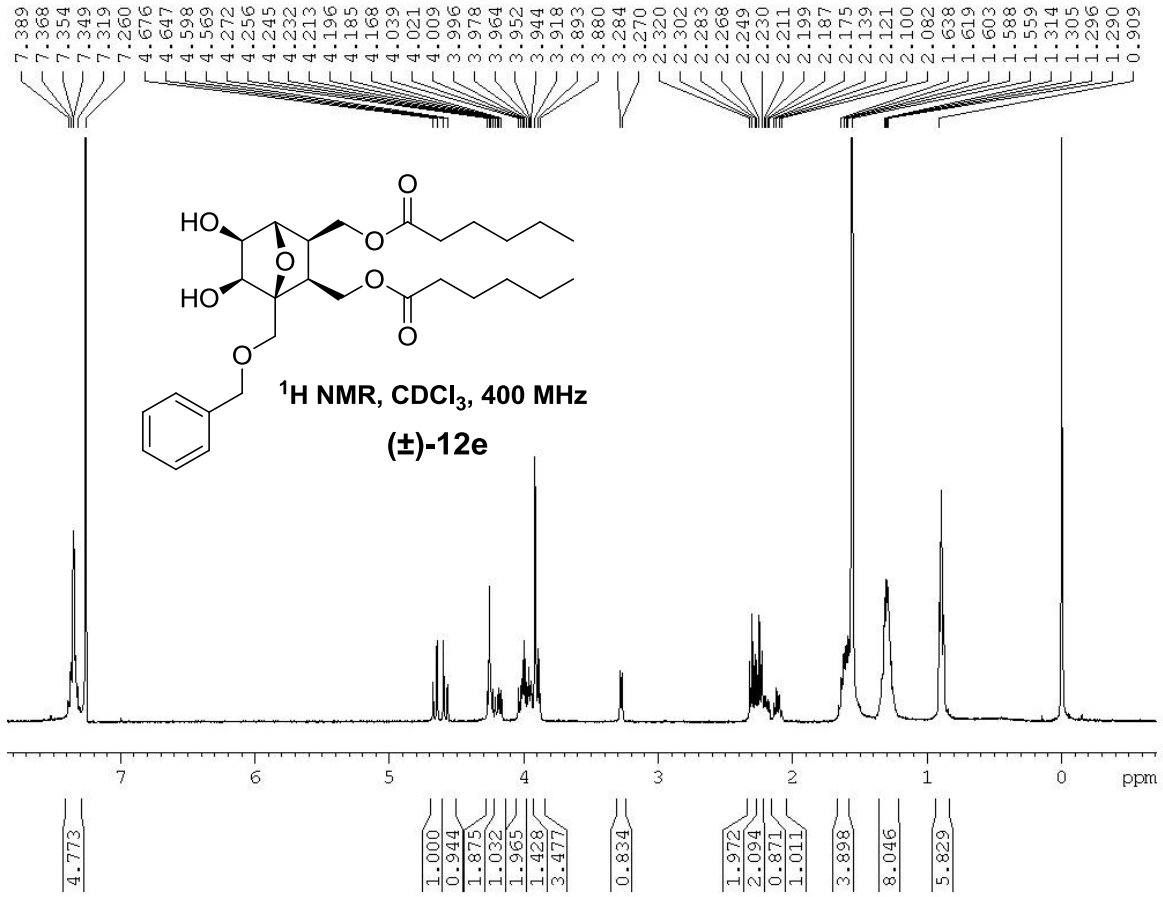
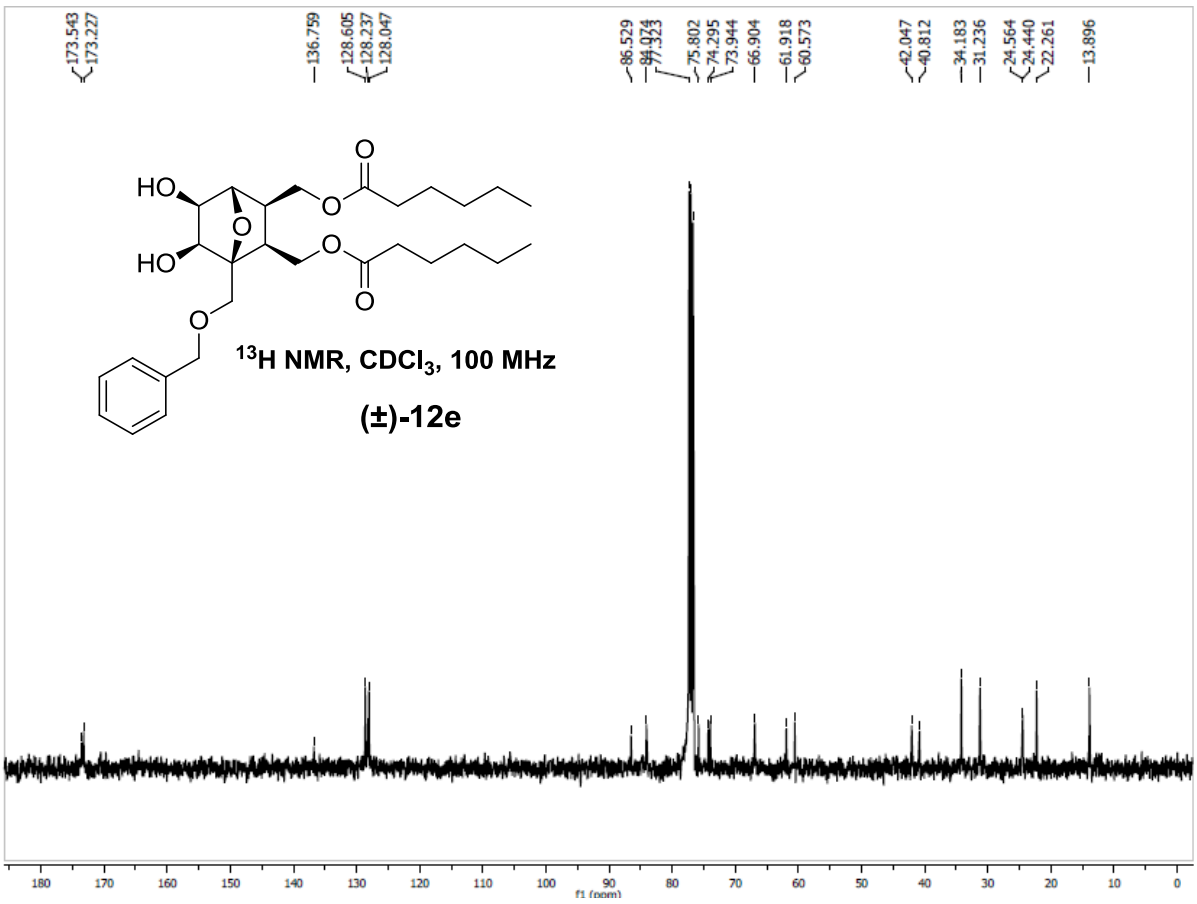
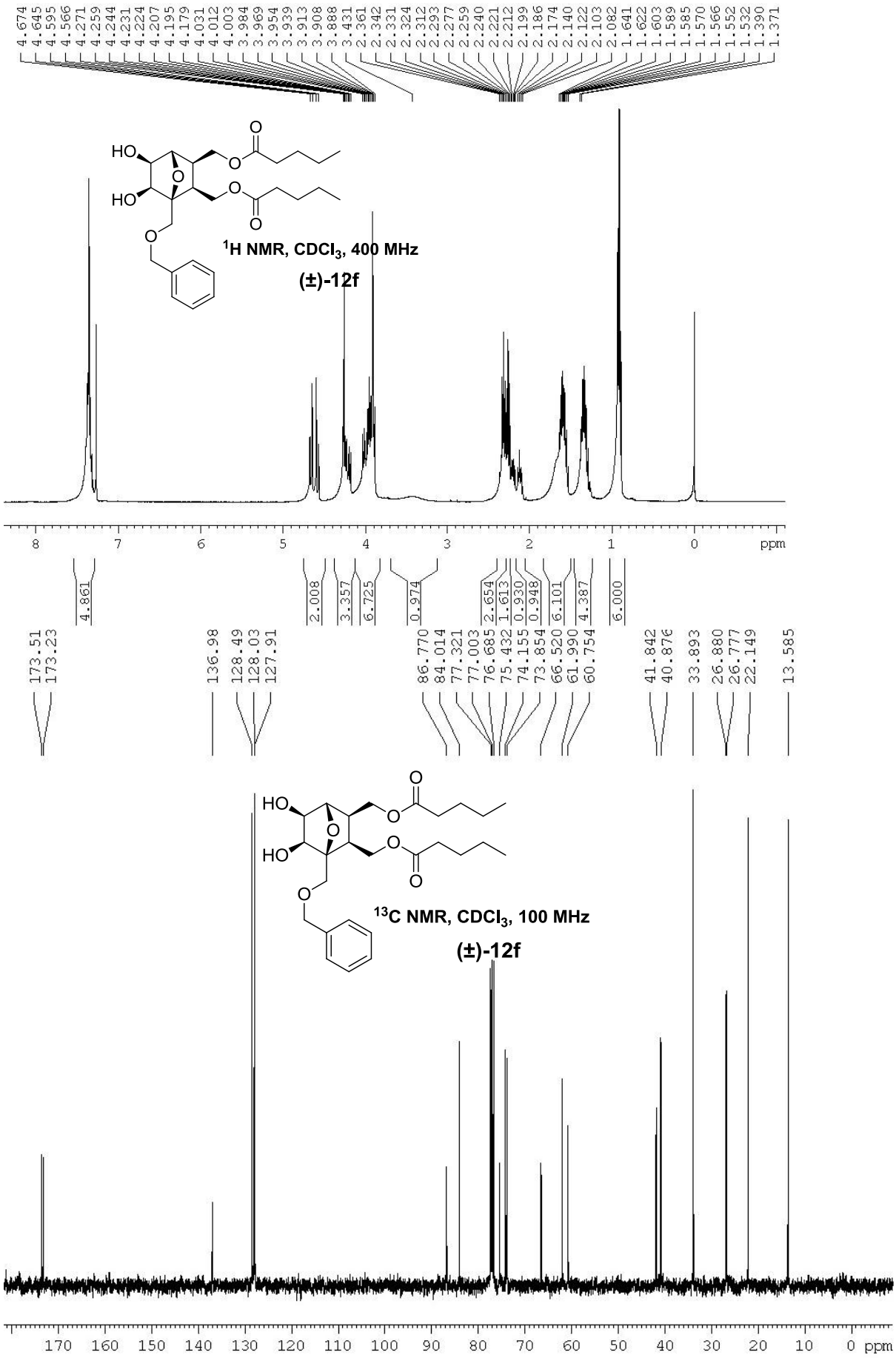
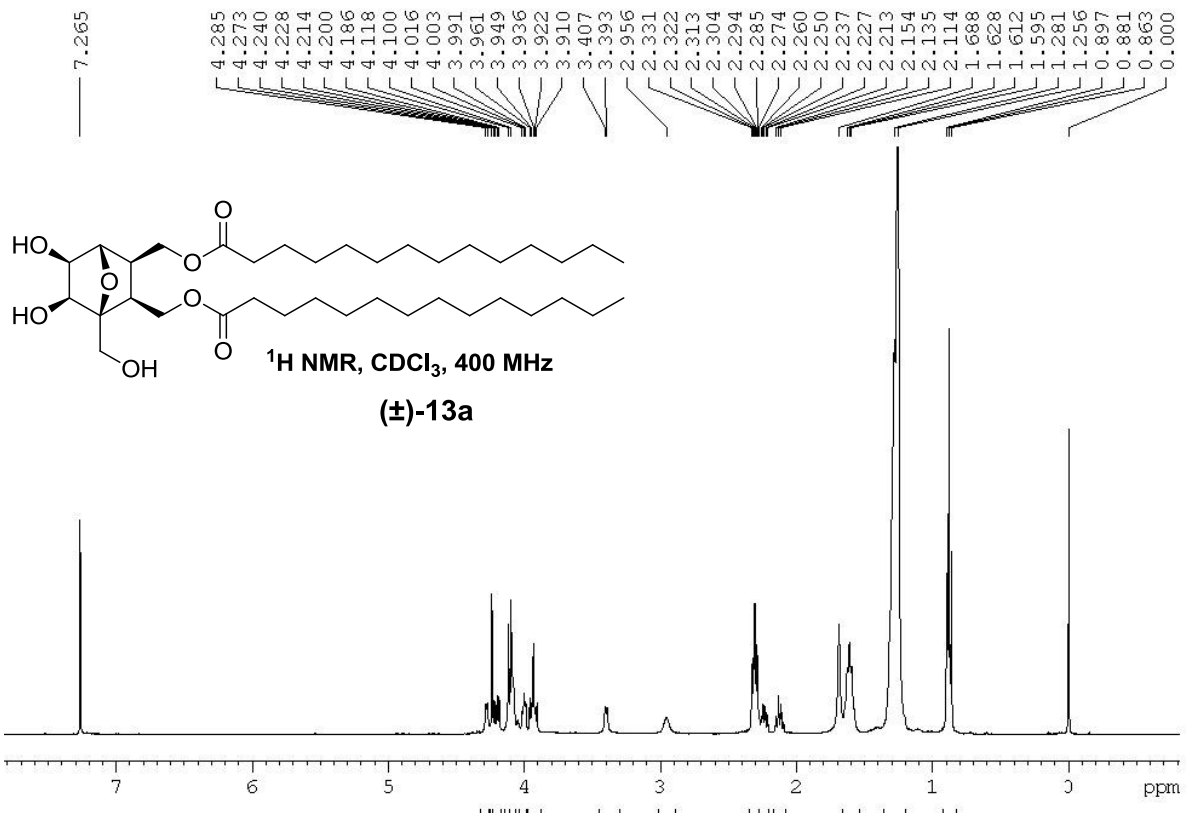
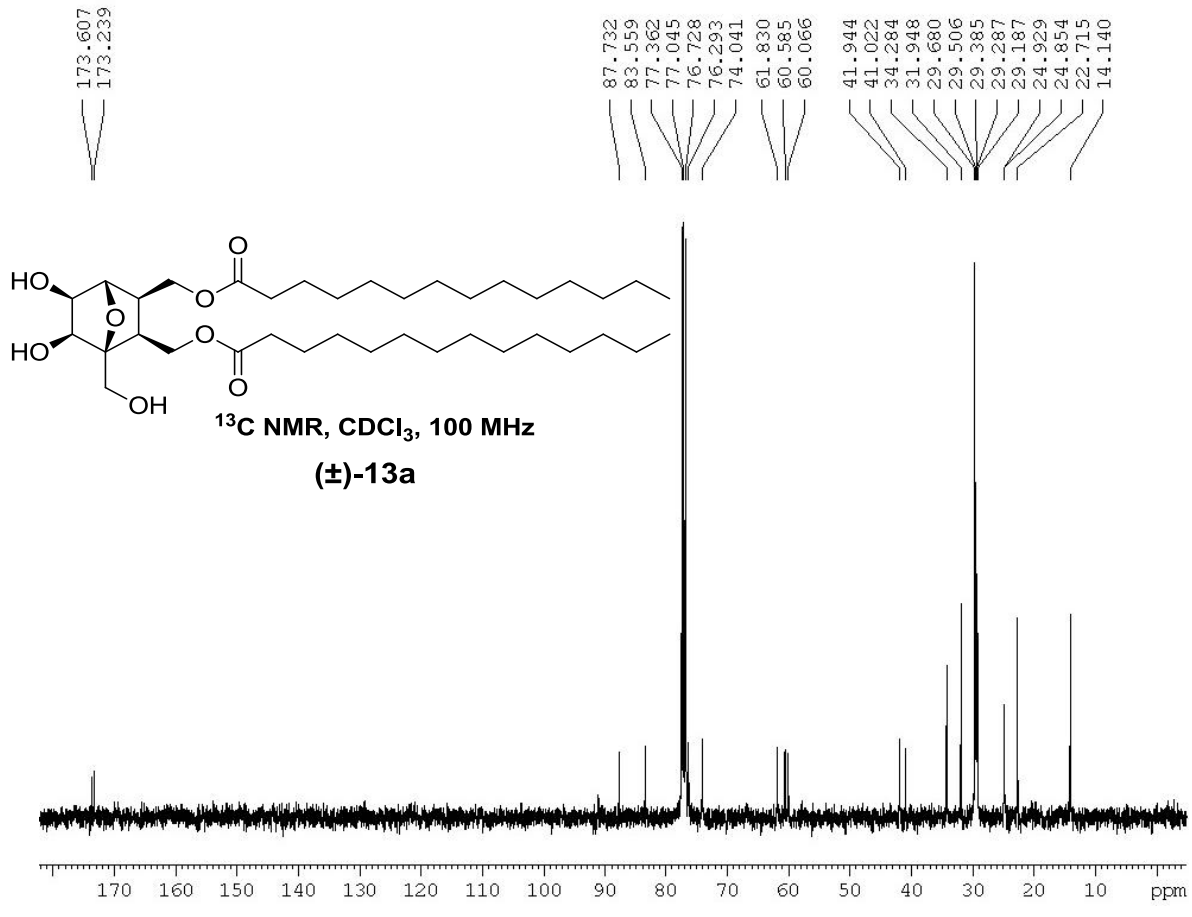
7.353
7.338
7.319
7.268
7.266
4.672
4.652
4.643
4.593
4.564
4.257
4.243
4.230
4.215
4.198
4.186
4.169
4.029
4.008
4.001
3.978
3.961
3.937
3.911
3.900
3.885
2.321
2.302
2.283
2.269
2.250
2.231
2.204
2.192
2.171
2.136
2.119
2.099
1.628
1.611
1.597
1.581
1.565
1.275
0.898
HO
HO
O
O
O
O
O
O
^{1}H NMR, CDCl$_3$, 400 MHz
(±)-12d
8
7
6
5
4
3
2
1
0
4.684
2.019
3.360
1.482
1.207
3.754
0.801
3.981
1.051
1.051
4.372
16.549
6.000
173.566
173.276
136.904
128.598
128.176
128.013
86.568
84.084
77.318
77.000
76.683
75.666
74.259
73.952
66.918
61.960
60.598
42.011
40.852
34.225
31.622
29.068
28.875
24.879
24.784
22.571
14.047
-0.031
HO
HO
O
O
O
O
O
O
^{13}C NMR, CDCl$_3$, 100 MHz
(±)-12d
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

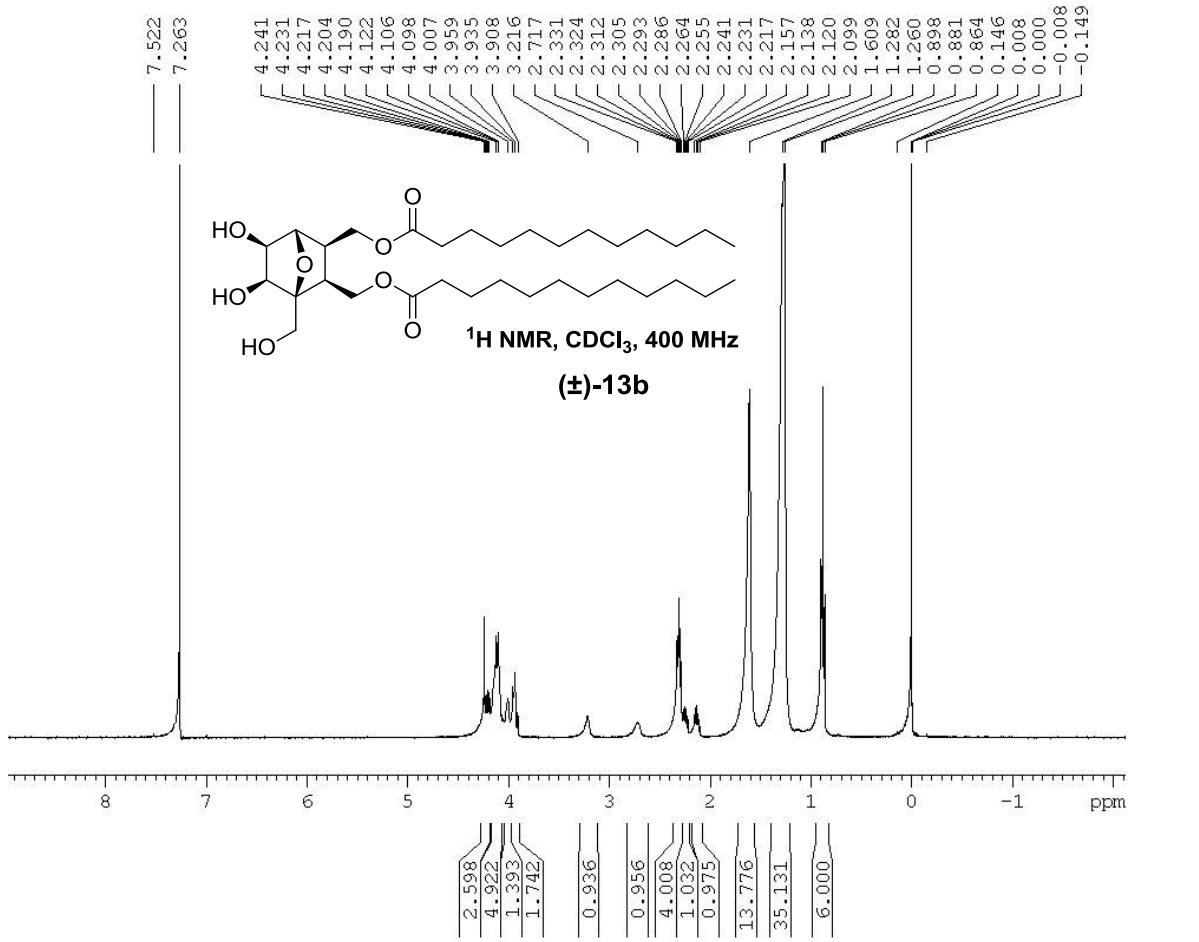

7.389
7.368
7.354
7.349
7.319
7.260
4.676
4.647
4.598
4.569
4.272
4.256
4.245
4.232
4.213
4.196
4.185
4.168
4.039
4.021
4.009
3.996
3.978
3.964
3.952
3.944
3.918
3.893
3.880
3.284
3.270
2.320
2.302
2.283
2.268
2.249
2.230
2.211
2.199
2.187
2.175
2.139
2.121
2.100
2.082
1.638
1.619
1.603
1.588
1.559
1.314
1.305
1.296
1.290
0.909
HO
HO
O
O
O
O
O
O
1H NMR, CDCl3, 400 MHz
(±)-12e
7
6
5
4
3
2
1
0
ppm
4.773
1.000
0.944
1.875
1.032
1.965
1.428
3.477
0.834
1.972
2.094
0.871
1.011
3.898
8.046
5.829

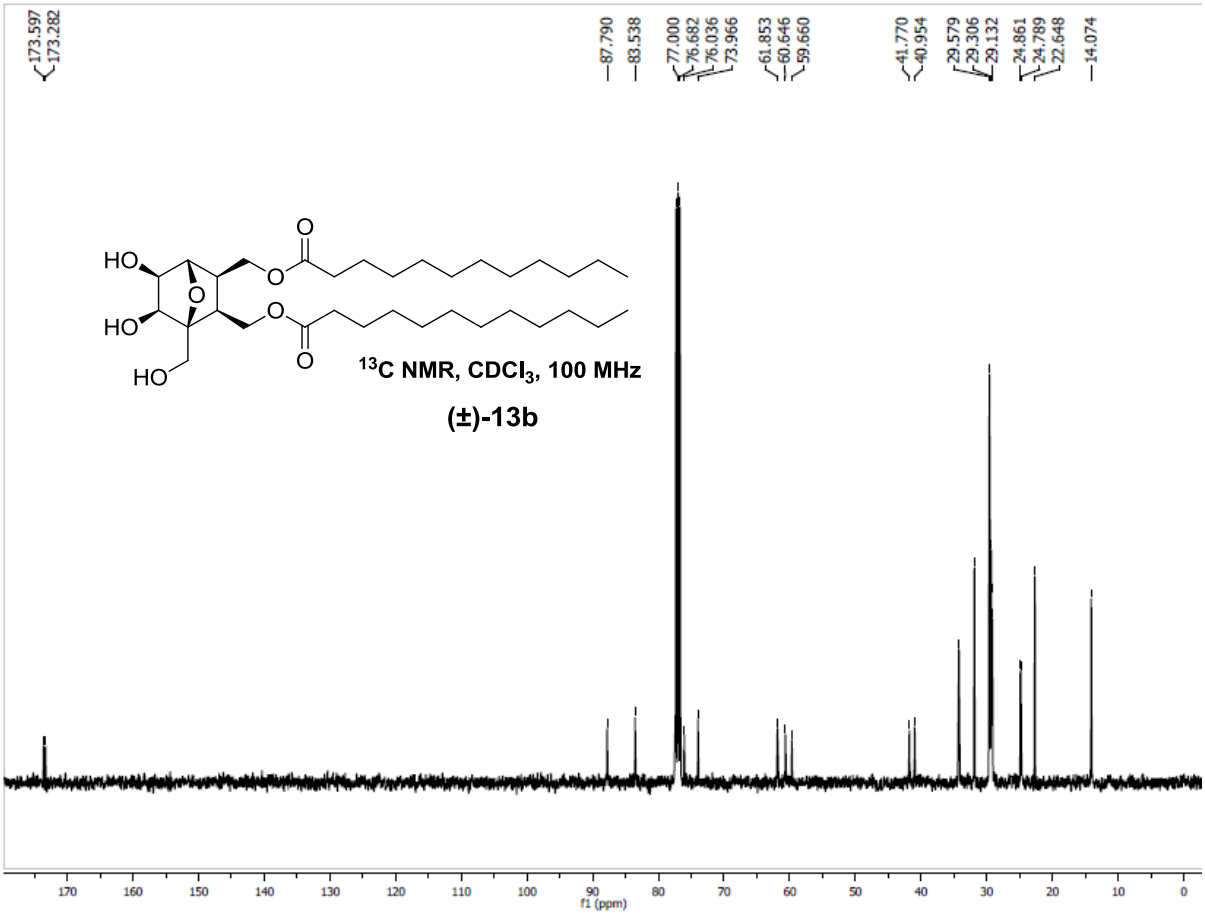

173.543
173.227
136.759
128.605
128.237
128.047
86.529
77.323
75.802
74.295
73.944
66.904
61.918
60.573
42.047
40.812
34.183
31.236
24.564
24.440
22.261
13.896
HO
HO
O
O
O
O
O
O
13H NMR, CDCl3, 100 MHz
(±)-12e
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

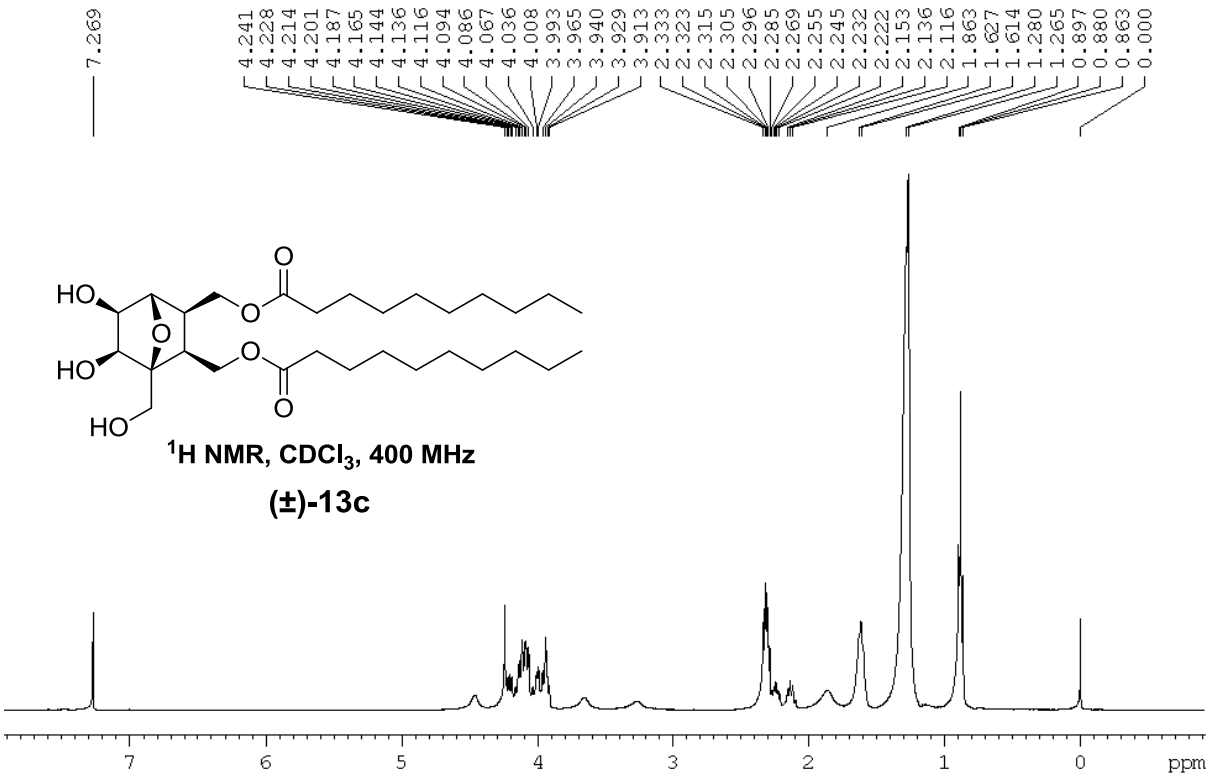

4.674 4.645 4.595 4.566 4.271 4.259 4.244 4.231 4.224 4.207 4.195 4.179 4.031 4.012 4.003 3.984 3.969 3.954 3.939 3.913 3.908 3.888 3.431 2.361 2.342 2.331 2.324 2.312 2.293 2.277 2.259 2.240 2.221 2.212 2.199 2.186 2.174 2.140 2.122 2.103 2.082 1.641 1.622 1.603 1.589 1.585 1.570 1.566 1.552 1.532 1.390 1.371
HO
HO
O
O
O
O
O
O
^{1}H NMR, CDCl$_3$, 400 MHz
(±)-12f
8 7 6 5 4 3 2 1 0 ppm
4.861 2.008 3.357 6.725 0.974 2.654 1.613 0.930 0.948 6.101 4.387 6.000
173.51 173.23 136.98 128.49 128.03 127.91 86.770 84.014 77.321 77.003 76.685 75.432 74.155 73.854 66.520 61.990 60.754 41.842 40.876 33.893 26.880 26.777 22.149 13.585
^{13}C NMR, CDCl$_3$, 100 MHz
(±)-12f
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

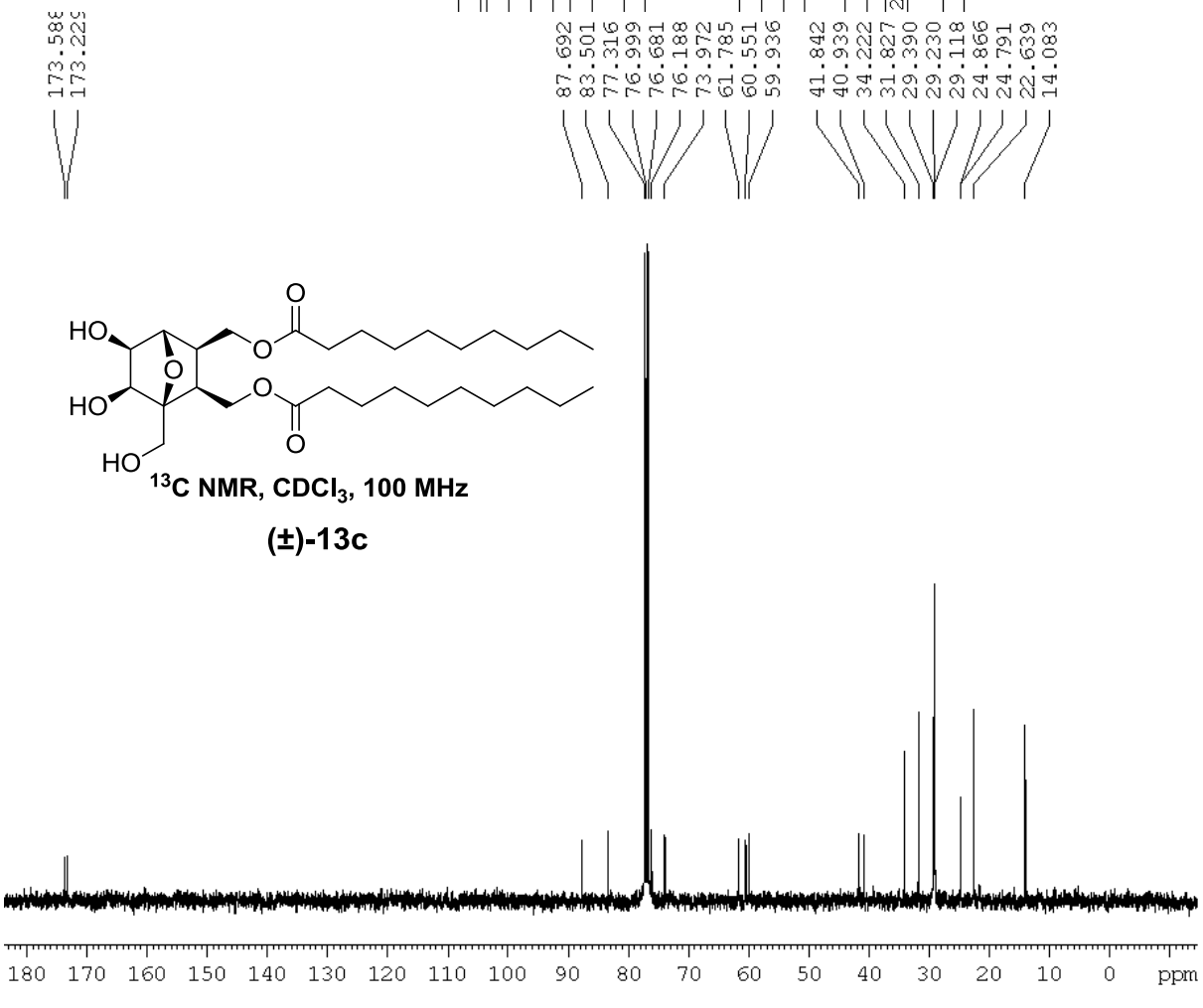


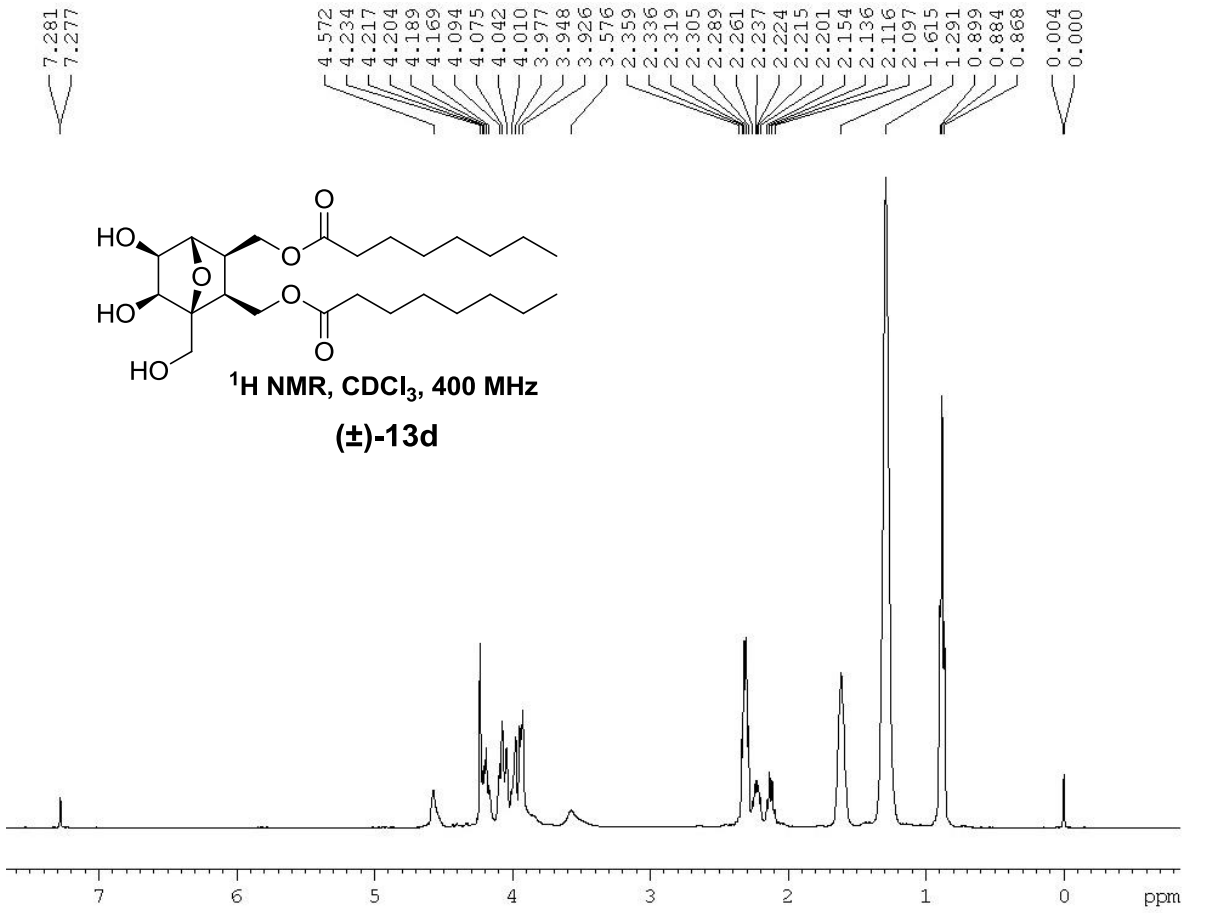


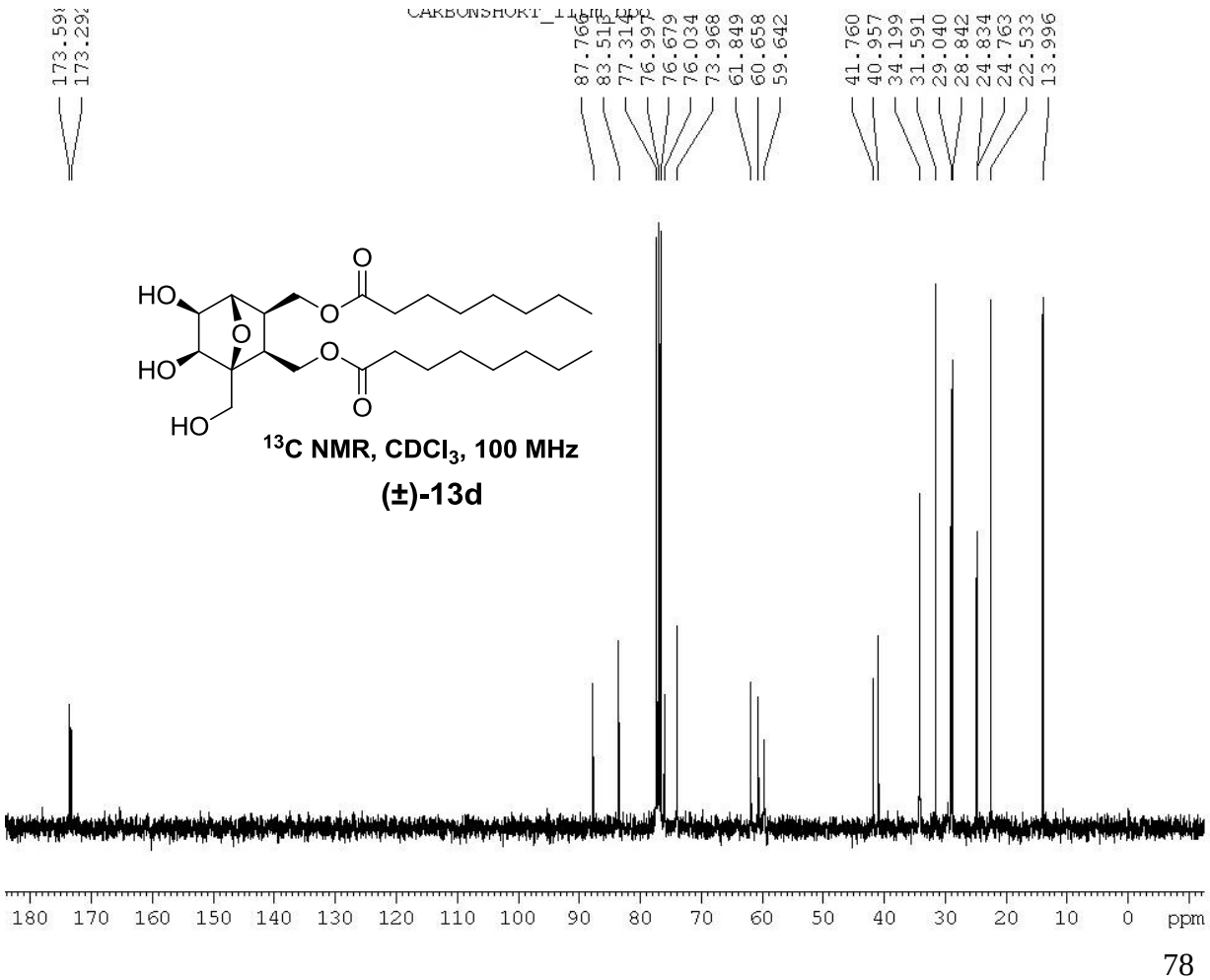

7.522
7.263
4.241
4.231
4.217
4.204
4.190
4.122
4.106
4.098
4.007
3.959
3.935
3.908
3.216
2.717
2.331
2.324
2.312
2.305
2.293
2.286
2.264
2.255
2.241
2.231
2.217
2.157
2.138
2.120
2.099
1.609
1.282
1.260
0.898
0.881
0.864
0.146
0.008
0.000
-0.008
-0.149
HO
HO
HO
O
O
O
O
O
^{1}H NMR, CDCl$_3$, 400 MHz
(±)-13b
8
7
6
5
4
3
2
1
0
−1
ppm
2.598
4.922
1.393
1.742
0.936
0.956
4.008
1.032
0.975
13.776
35.131
6.000

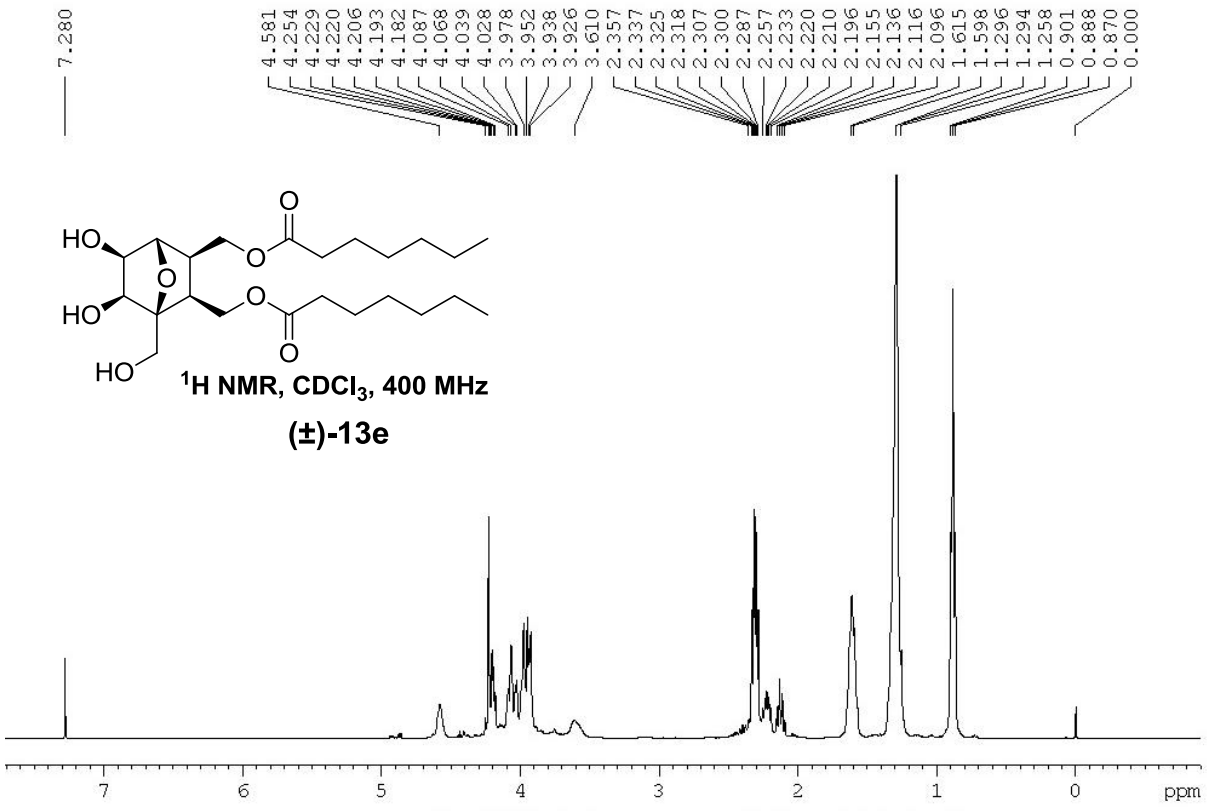

173.597
173.282
87.790
83.538
77.000
76.682
76.036
73.966
61.853
60.646
59.660
41.770
40.954
29.579
29.306
29.132
24.861
24.789
22.648
14.074
HO
HO
HO
O
O
O
O
O
^{13}C NMR, CDCl$_3$, 100 MHz
(±)-13b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)





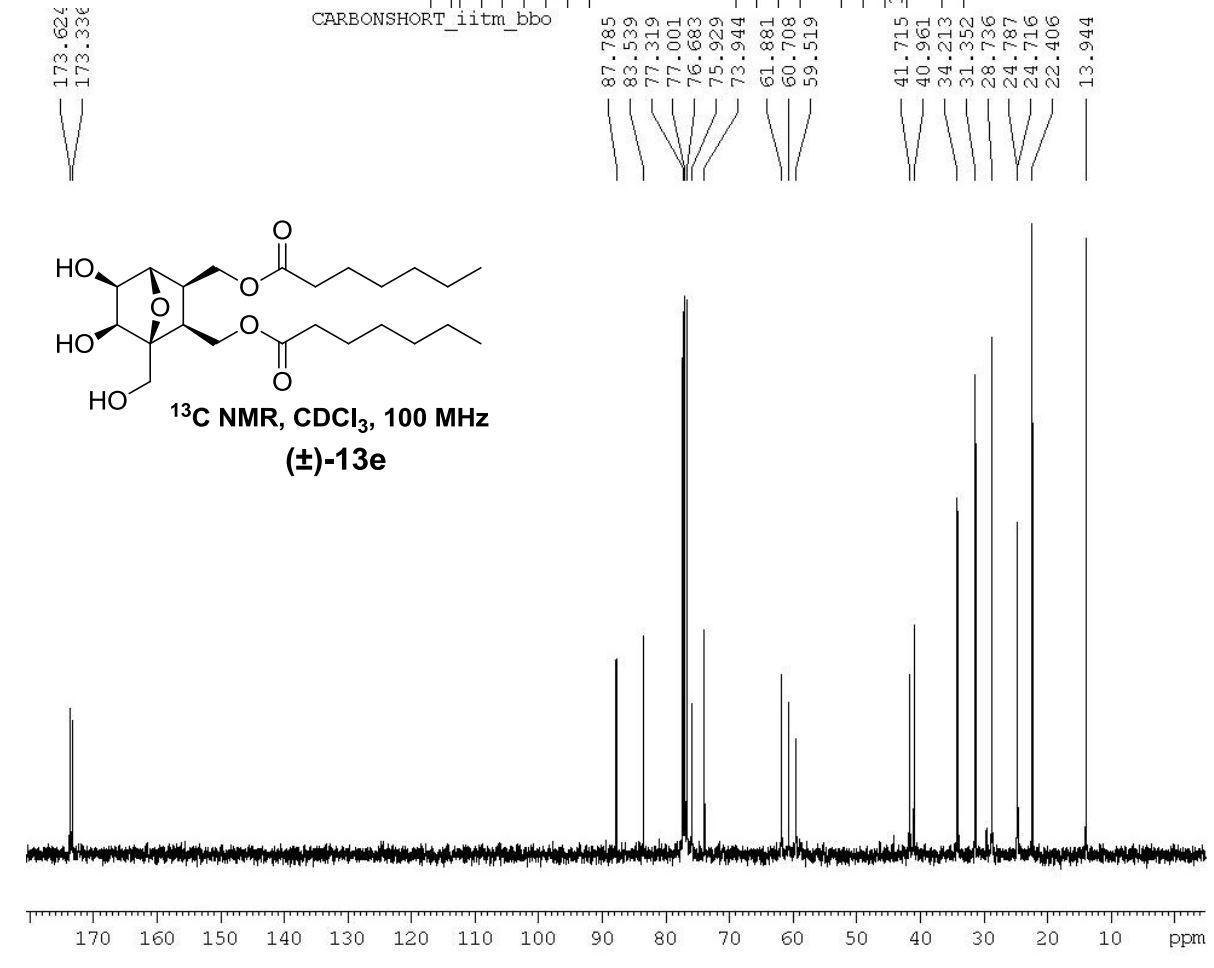

7.281
7.277
4.572
4.234
4.217
4.204
4.189
4.169
4.094
4.075
4.042
4.010
3.977
3.948
3.926
3.576
2.359
2.336
2.319
2.305
2.289
2.261
2.237
2.224
2.215
2.201
2.154
2.136
2.116
2.097
1.615
1.291
0.899
0.884
0.868
0.004
0.000
HO
HO
HO
O
O
O
O
O
^{1}H NMR, CDCl$_3$, 400 MHz
(±)-13d
7
6
5
4
3
2
1
0
ppm


173.59
173.29
87.766
83.515
77.314
76.997
76.679
76.034
73.968
61.849
60.658
59.642
41.760
40.957
34.199
31.591
29.040
28.842
24.834
24.763
22.533
13.996
HO
HO
HO
O
O
O
O
O
^{13}C NMR, CDCl$_3$, 100 MHz
(±)-13d
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



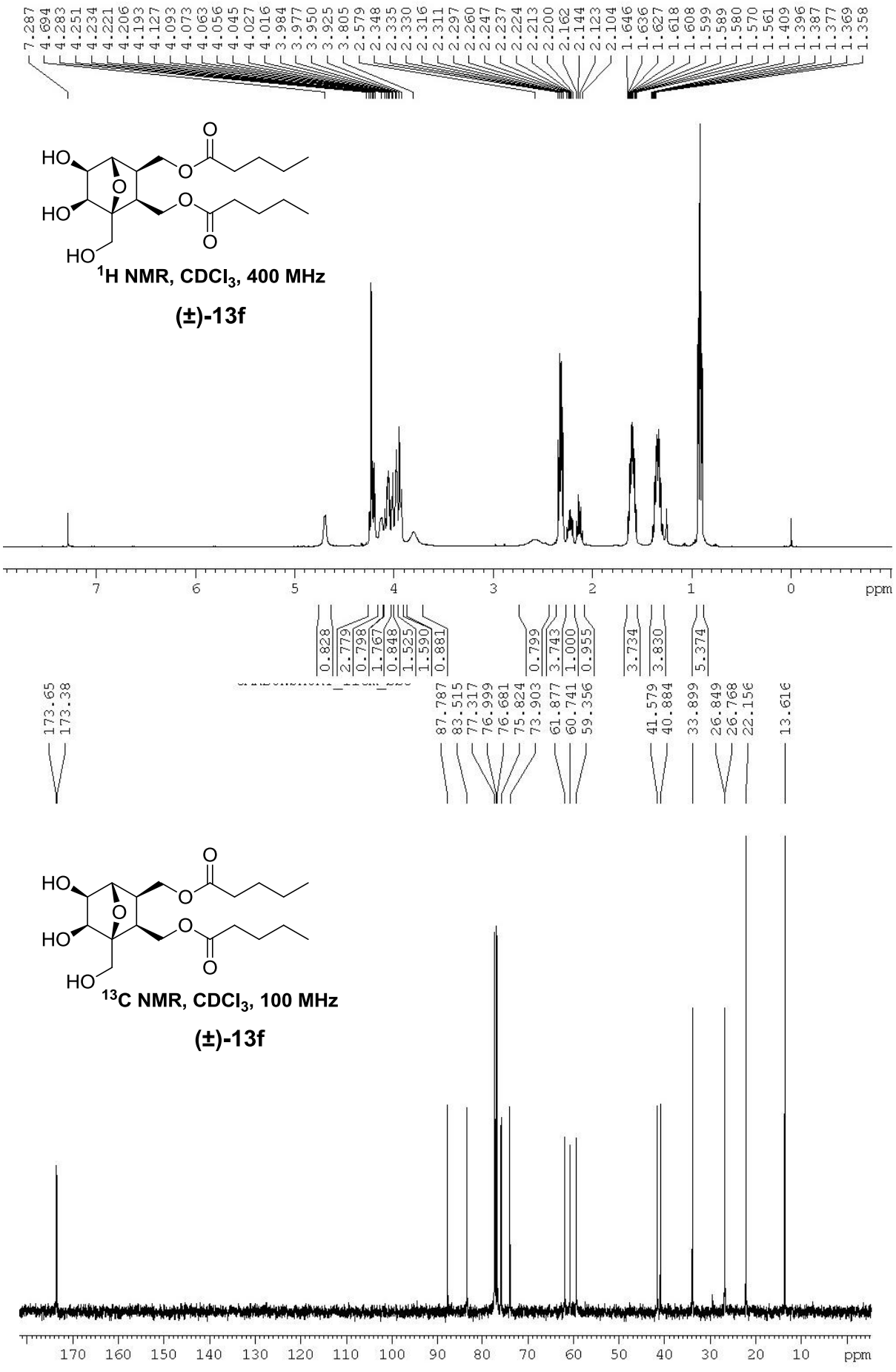
^{1}H NMR, $CDCl_3$, 400 MHz

(±)-13e



^{13}C NMR, $CDCl_3$, 100 MHz

(±)-13e


7.287
4.694
4.283
4.251
4.234
4.221
4.206
4.193
4.127
4.093
4.073
4.063
4.056
4.045
4.027
4.016
3.984
3.977
3.950
3.925
3.805
2.579
2.348
2.335
2.330
2.316
2.311
2.297
2.260
2.247
2.237
2.224
2.213
2.200
2.162
2.144
2.123
2.104
1.646
1.636
1.627
1.618
1.608
1.599
1.589
1.580
1.570
1.561
1.409
1.396
1.387
1.377
1.369
1.358
HO
O
HO
HO
O
O
O
1H NMR, CDCl3, 400 MHz
(±)-13f
7
6
5
4
3
2
1
0
ppm
0.828
2.779
0.798
1.767
0.848
1.525
1.590
0.881
0.799
3.743
1.000
0.955
3.734
3.830
5.374
173.65
173.38
87.787
83.515
77.317
76.999
76.681
75.824
73.903
61.877
60.741
59.356
41.579
40.884
33.899
26.849
26.768
22.156
13.616
13C NMR, CDCl3, 100 MHz
(±)-13f
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm