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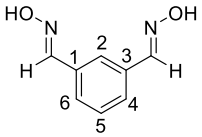
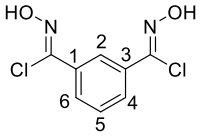
1.0 Extra experimental data.

1.1 (1*E*,3*E*)-Isophthalaldehyde dioxime[1]



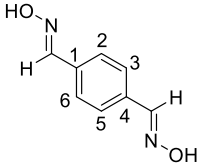
Sodium acetate (13.4 g, 164 mmol, 2.2 eq.) and hydroxylamine hydrochloride (11.4 g, 164 mmol, 2.2 eq.) were dissolved in water (50 ml) and added to isophthalaldehyde (10.0 g, 75 mmol, 1 eq.) dispersed in ethanol (50 ml). A white precipitate began to form within 30 minutes. After 4 hours the solid precipitate was recovered by Buchner filtration and washed with cold ethanol, purification of the precipitate by recrystallisation from EtOAc gave a cream powder. (8.7 g, 71 %); m.p. 186 – 188 °C (lit. 181.2 – 182.0 °C); ν_{max}/cm^{-1} 3203 (OH), 2920 (C-H), 2790 (C-H), 1642 (C=N), 1490 (N-O), 945 (C-H); ^{1}H NMR (400MHz, d6-acetone) δ 10.46 (s, 2H, OH), 8.17 (s, 2H, HC=N), 7.88 (s, 1H, H^{2}), 7.62 (dd, *J* = 7.5, 1.5 Hz, 2H, $H^{4,6}$), 7.42 (t, *J* = 7.5 Hz 1H, H^{5}); ^{13}C (101 MHz, d6-Acetone) δ 148.97 (C=N), 134.74 ($C^{1,3}$), 129.86 (C^{5}), 128.25 ($C^{6,4}$), 125.47 (C^{2}).

1.2 (*1Z*,3*Z*)-N'^{1},N'^{3}-Dihydroxyisophthalimidoyl dichloride 1a[1]



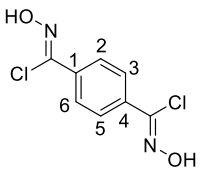
Isophthalaldehyde dioxime (7.4 g, 45.1 mmol, 1eq.) was dissolved in DMF (50 ml) at RT. *N*-Chlorosuccinimide (13.2 g, 99.2 mmol, 2.2 eq.) was introduced in 5 parts over the course of an hour. Two hours after the last portion was added the reaction mixture was poured into cold water (150 ml) and extracted with ethyl acetate (150 ml). The organic layer was washed with water (5 × 50 ml) and saturated sodium thiosulfate solution (1 × 50 ml), then dried over $MgSO_4$. Solvent removal *in vacuo* gave a crude yellow solid, which was purified by recrystallization from toluene to give a cream solid, (8.8 g, 84 %); m.p. 159 – 161 °C (lit. 155.7 – 156.0 °C) ν_{max}/cm^{-1} 3265 (OH), 1624 (C=N), 1483 (N-O), 990 (C-H), 802 (C-Cl); ^{1}H NMR (500MHz, Acetone) 11.58 (s, 2H, OH), 8.35 (s, 1H, H^{2}), 7.96 (dd, *J* = 8.0, 1.5 Hz, 2H, $H^{4,6}$), 7.57 (t, *J* = 8.0 Hz, 1H, H^{5}); ^{13}C NMR (126 MHz, Acetone) δ 136.90 (C=N), 134.45 ($C^{1,3}$), 129.93 (C^{5}), 129.37 ($C^{4,6}$), 125.82 (C^{2}).

1.3 (1*E*,4*E*)-Terephthalaldehyde dioxime[1]



Sodium acetate (13.4 g, 164 mmol, 2.2 eq.) and hydroxylamine hydrochloride (11.4 g, 164 mmol, 2.2 eq.) were dissolved in water (50 ml) and added to isophthalaldehyde (10.0 g, 75 mol, 1 eq.) dispersed in ethanol (50 ml). A white precipitate began to form within 30 minutes. After 4 hours the solid precipitate was recovered by Buchner filtration and washed with cold ethanol, purification of the precipitate by recrystallisation from EtOAc gave a pure white solid, (9.3 g, 76 %); m.p. 176 – 178 °C (lit. 181.2 – 182.0 °C); ν_{max}/cm^{-1} 3137 (OH), 2983 (C-H), 1624 (C=N); ^{1}H NMR (400MHz, d6-acetone) δ 10.44 (s, 2H, OH), 8.15 (s, 2H, HC=N), 7.65 (s, 4H, $H^{2,3,5,6}$); ^{13}C (101 MHz, d6-Acetone) δ 148.98 (C=N), 135.14 ($C^{1,3}$), 127.71 ($C^{2,3,5,6}$).

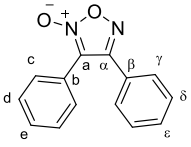
1.4 (*1Z*,3*Z*)-N'^{1},N'^{3}-Dihydroxyterephthalimidoyl dichloride 1b[1]



Terephthalaldehyde dioxime (8.7 g, 53 mmol, 1 eq.) was dissolved in DMF (50 ml) at RT. *N*-Chlorosuccinimide (14.2 g, 106 mmol, 2.0 eq.) was introduced in 5 parts over the course of an hour. Two hours after the last portion was added the reaction mixture was poured into cold water (150 ml) and extracted with ethyl acetate (150

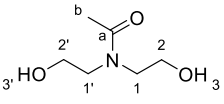
ml). The organic layer was washed with water (5 × 50 ml) and saturated sodium thiosulfate solution (1 × 50 ml), then dried over $MgSO_4$. Solvent removal *in vacuo* gave a crude yellow solid, which was purified by recrystallization from toluene to give a white solid. (9.1 g, 74 %); m.p. 206 –208 °C; ν_{max}/cm^{-1} 3260 (OH), 3053 (C-H), 1675 (C=N), 802(C-Cl); ^{1}H NMR (500MHz, d6-acetone) 11.62 (s, 2H, OH), 7.94 (s, 4H, H^{2,3,5,6}); ^{13}C NMR (126 MHz, d6-acetone) δ 136.99 (C=N), 135.65 (C^{1,4}), 127.83 (C^{2,3,5,6}).

1.5 3,4-Diphenyl-1,2,5-oxadiazol-2-oxide 2[2]



A solution of saturated sodium carbonate (5 ml) was added to a solution of benzaldehyde oximoyl chloride (0.26 g, 1.67 mmol) in diethyl ether (10 ml). The mixture was stirred rapidly for 24 h and then transferred to a separating funnel. The organic layer was removed, and the aqueous layer extracted with diethyl ether (2 x 10 ml). The combined organic layers were washed with water (2 x 5 ml) and dried with anhydrous magnesium sulphate. Evaporation of the solvent yielded crude 3,4-diphenyl-1,2,5-oxadiazol-2-oxide 9a (0.13 g, 65 %). Recrystallisation from ethanol gave white needles (0.06 g, 30 %); m.p. 110 – 112 °C; ν_{max} / cm^{-1}: 1591 (C=N), 1572 (C=N), 1504, 1441, 1419, 1327; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 – 7.51 (m, 5H, H$^{c\gamma\varepsilon}$), 7.47 – 7.42 (m, 5H, H$^{d\delta e}$); ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.34 (C$^{\alpha}$), 131.10 (C$^{\varepsilon}$), 130.66 (C^{e}), 129.13 (C$^{d/\delta}$), 129.05 (C$^{d/\delta}$), 128.78 (C^{c}), 128.39 (C$^{\gamma}$), 126.75 (C$^{\beta}$), 122.96 (C^{b}), 114.38(C^{a}); *m/z*: (ES$^+$) calcd. ($C_{14}H_{10}N_2O_2$ + Na$^+$): 261.0634, found: 261.0636 (M + Na$^+$).

1.6 *N*,*N*-Bis(2-hydroxyethyl)acetamide 4a[3]



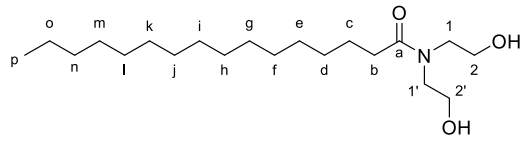
Diethanolamine (20.0 g, 19.0 mmol, 1 eq.) was dissolved in THF (150 ml) and cooled to 0 °C in an ice bath. Acetic anhydride (23.3 g, 22.8 mmol, 1.2 eq.) was added dropwise (1 drop every two seconds) over the course of 4 hours. The solution was left to come to room temperature overnight. A sample was taken in the morning and analysed with NMR and MS, revealing a mixture of products. The solution was put under vacuum at 65 °C to remove acetic acid and any residual acetic anhydride. MS/NMR continued to show multiple products, particularly double acetylated impurities. The crude product was taken back into methanol (100 ml) along with K_2CO_3 (7.00 g, 50.6 mmol) and refluxed for 5 hours. Water was then added until the solution was clear, following this it was neutralised with aqueous HCl (2 M, 80 ml). Methanol and water were removed in vacuo, to leave a yellow oil and solid. The oil was taken into acetone and filtered to remove the solid. The filtrate was then reduced *in vacuo* to leave a yellow oil and some white solid. Under N_2 the yellow oil was taken into dry acetone and dried with 4 Å MS for 1 hour, the solution was then filtered to remove the residual white solid and sieves. The filtrate was once again reduced *in vacuo* to leave a yellow oil. (1.9 g, 69 %); ν_{max} / cm^{-1}: 3353 (OH), 2934 (C-H), 1605 (C=O), 1036 (C-O); ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.81 (brs, 2H, OH), 3.76 – 3.67 (m, 4H, H$^{2/2'}$), 3.51 – 3.38 (m, 4H, H$^{1/1'}$), 2.09 (s, 3H, CH_3); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.15 (C=O), 60.84 (C$^{1/1'}$), 60.46 (C$^{1/1'}$), 53.15 (C$^{2/2'}$), 50.34 (C$^{2/2'}$), 22.07 (CH_3); *m/z*: (ES$^+$) 169.5 [MNa]$^+$.

1.7 General procedure for the synthesis of diols 4b-f

N-methyl morpholine (1.1 eq.) was added to the appropriate fatty acid (1 eq., 0.5 M) in dry Et_2O for saturated fatty acids or dry THF for unsaturated fatty acids). Ethyl chloroformate (1.1 eq.) was added dropwise and the reaction was stirred for 30 minutes. The precipitate was removed by filtration and the filtrate was added dropwise to diethanolamine (1.1 eq., 0.5M) and triethylamine (1.1 eq., 0.5 M)

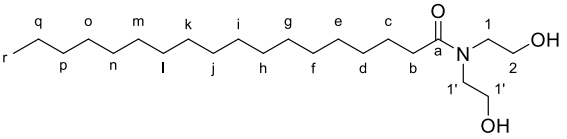
in dry DMF at room temperature under N_2. After 3 hrs the reaction was quenched with 2 M HCl (20 mL) and extracted with Et_2O (200 mL). The organic layer washed with water (2 x 50 mL) and brine (50 mL), before drying over $MgSO_4$. The solvent was removed *in vacuo* to leave a crude product. Unsaturated amides **4d-f** were purified by column chromatography through a silica plug (EtOAc, R_f = 0.2). Saturated amides **4b-c** were purified by recrystallisation from Et_2O.

1.7.1 *N,N*-Bis(2-hydroxyethyl)palmitamide 4b



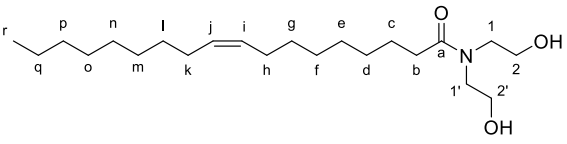
General procedure for the synthesis of diols was followed using palmitic acid (12.8 g, 49.9 mmol, 1 eq.), NMM (5.55 g, 54.9 mmol, 1.1 eq.), ethyl chloroformate (5.96 g, 54.9 mmol, 1.1 eq.), diethanolamine (5.8 g, 54.9 mmol, 1.1 eq.), and TEA (5.6 g, 54.9 mmol, 1.1 eq.) to give a white solid. (9.4 g, 55%); R_f = 0.2 in 100 % EtOAc; m.p. 60 – 62 °C; ν_{max}/cm^{-1} 3317 (O-H), 2916 (C-H), 2849 (C-H), 1611 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.90 (t, J = 5.0 Hz, 2H, $H^{1/1'}$), 3.79 (t, J = 5.0 Hz, 2H, $H^{1/1'}$), 3.57 (t, J = 5.0 Hz, 2H, $H^{2/2'}$), 3.49 (t, J = 5.0 Hz, 2H, $H^{2/2'}$), 3.24 (brs, 2H, H^{OH}), 2.39 (t, J = 7.5 Hz, 2H, H^b), 1.73 – 1.50 (m, 2H, H^c) 1.37 – 1.15 (m, 24H, $H^{d\text{-}o}$), 0.88 (t, J = 7.0 Hz, 3H, H^p); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.85 (C^a), 61.52 ($C^{2/2'}$), 61.12 ($C^{2/2'}$), 52.42 ($C^{1/1'}$), 50.77 ($C^{1/1'}$), 33.89 (C^b), 31.98 (C^n), 29.87 – 29.23 ($C^{d\text{-}m}$), 25.14 (C^c), 22.74 (C^o), 14.13 (C^p); *m/z*: (ES^+) calcd. ($C_{20}H_{41}NO_3$ + Na^+): 366.3, found: 366.1 (M + Na^+).

1.7.2 *N,N*-Bis(2-hydroxyethyl)stearamide 4c[4]



General procedure for the synthesis of diols was followed using stearic acid (30.0 g, 105.4 mmol, 1 eq.), NMM (11.7 g, 116.0 mmol, 1.1 eq.), ethyl chloroformate (12.6 g, 116.0 mmol, 1.1 eq.), diethanolamine (12.2 g, 116.0 mmol, 1.1 eq.), and TEA (11.7 g, 116.0 mmol, 1.1 eq.) to give a white solid. (25.0, 64 %); R_f = 0.2 in 100 % EtOAc; m.p. 72 – 75 °C; ν_{max}/cm^{-1} 3409 (O-H) 2918 (C-H), 2849 (C-H), 1616 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.86 (t, J = 5.0 Hz, 2H, $H^{2/2'}$), 3.80 (t, J = 5.0 Hz, 2H, $H^{2/2'}$), 3.57 (t, J = 5.0, 2H, $H^{1/1'}$), 3.52 (t, J = 5.0 Hz, 2H, $H^{1/1'}$), 2.40 (t, J = 7.5 Hz, 2H, H^b), 1.71 – 1.58 (m, 2H, H^c), 1.40 – 1.19 (m, 28H, $H^{d\text{-}q}$), 0.90 (t, J = 7.0 Hz, 3H, H^r); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.92 (C^a), 61.87 ($C^{2/2'}$), 61.04 ($C^{2/2'}$), 52.35 ($C^{1/1'}$), 50.68 ($C^{1/1}$), 33.78 (C^b), 32.06 (C^p), 20.95 – 29.40 ($C^{d\text{-}q}$), 25.43 (C^c), 22.83 (C^q), 14.13 (C^r); *m/z*: (ES^+) calcd. ($C_{22}H_{45}NO_3$ + Na^+): 394.3, found: 394.1 (M + Na^+).

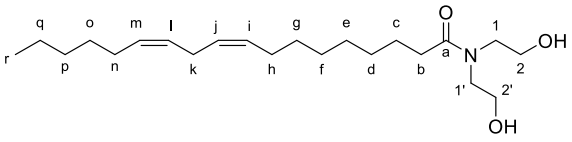
1.7.3 *N,N*-Bis(2-hydroxyethyl)oleamide 4d[5]



General procedure for the synthesis of diols was followed using oleic acid (30 g, 106.2 mmol, 1 eq.), NMM (11.8 g, 116.8 mmol, 1.1 eq.), ethyl chloroformate (12.7 g, 116.8 mmol, 1.1 eq.), diethanolamine (12.3 g, 116.8 mmol, 1.1 eq.), and TEA (11.4 g, 116.8 mmol, 1.1 eq.) to give a pale yellow oil. (33.7 g, 86 %); R_f = 0.2 in 100 % EtOAc; ν_{max}/cm^{-1} 3309 (O-H), 3005(=C-H), 2922 (C-H), 2853 (C-H), 1610 (C=O), 1463 (C-H), 1419 (C-N), 756 (=C-H); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.42 – 5.24 (m, 2H, $H^{i/j}$), 4.51 (brs, 2H, H^{OH}), 3.79 (t, J = 5.0 Hz, 2H, $H^{2/2'}$), 3.75 (t, J = 5.0 Hz, 2H, $H^{2/2'}$), 3.52 (t, J = 5.0 Hz, 2H, $H^{1/1'}$), 3.48 (t, J = 5.0 Hz, 2H, $H^{1/1'}$), 2.77 (t, J = 6.5 Hz, $H^{linoleic}$), 2.48 (t, *J* = 7.5 Hz, 2H, H^b), 2.07 – 1.94 (m, 4H, $H^{h/k}$), 1.67 – 1.54 (m, 2H, H^c), 1.38 – 1.21 (m, 20H, $H^{d\text{-}g/l\text{-}q}$), 0.88 (t, J = 7.0 Hz, 3H, H^r); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.63 (C^a), 130.21 ($C^{linoleic}$), 130.02 (C^i), 129.78 (C^j), 127.92 ($C^{linoleic}$), 61.22 ($C^{2/2'}$), 60.73

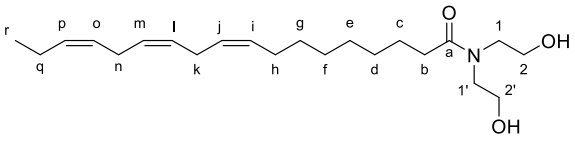
($C^{2/2'}$), 52.31 ($C^{1/1'}$), 50.63 ($C^{1/1'}$), 33.66 (C^{b}), 31.95 (C^{p}), 30.87 – 29.20 ($C^{d\text{-}g/l\text{-}o}$), 27.27 ($C^{h}$), 27.25 ($C^{k}$), 25.63 ($C^{linoleic}$), 25.37 ($C^{c}$), 22.72 ($C^{q}$), 14.16 ($C^{r}$); *m/z*: ($ES^+$) calcd. ($C_{22}H_{43}NO_3$ + Na^+): 392.3, found: 392.4 (M + Na^+).

1.7.4 (9*Z*,12*Z*)-*N*,*N*-Bis(2-hydroxyethyl)octadeca-9,12-dienamide 4e[5]



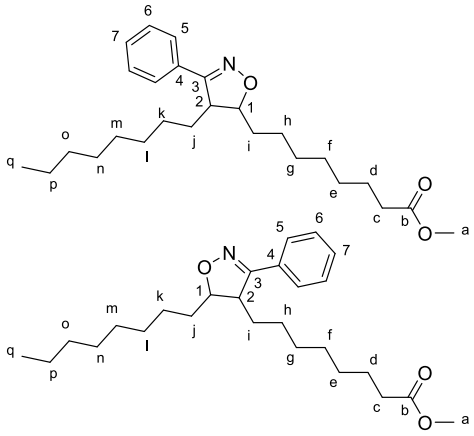
General procedure for the synthesis of diols was followed using linoleic acid (30 g, 107.0 mmol, 1 eq.), NMM (11.9 g, 117.7 mmol, 1.1 eq.), ethyl chloroformate (12.8 g, 117.7 mmol, 1.1 eq.), diethanolamine (12.4 g, 117.7 mmol, 1.1 eq.), TEA (11.9 g, 117.7 mmol, 1.1 eq.) to give a pale yellow oil, (33.7 g, 78 %, R_f = 0.2 in 100% EtOAc). ν_{max}/cm^{-1}: 3339 (O-H), 3008 (=C-H), 2922 (C-H), 2853 (C-H), 1616 (C=O), 1464 (C-H), 1420 (C-N), 1050 (C-OH), 723 (=C-H); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.55 – 5.17 (m, 4H, $H^{i/j/l/m}$), 4.87 (brs, 2H, H^{OH}), 3.79 (t, J = 5.0 Hz, 2H, $H^{2/2'}$), 3.76 (t, J = 5.0 Hz, 2H, $H^{2/2'}$), 3.53 (t, J = 4.5 Hz, 2H, $H^{1/1'}$), 3.49 (t, J = 5.0 Hz, 2H, $H^{1/1'}$), 2.77 (t, J = 6.5 Hz, 2H, H^{k}), 2.39 (t, J = 7.5 Hz, 2H, H^{b}), 2.07 – 2.02 (m, 4H, $H^{h/n}$), 1.70 – 1.53 (m, 2H, H^{d}), 1.41 – 1.21 (m, 14H, $H^{c\text{-}g/o\text{-}q}$), 0.89 (t, J = 7.0 Hz, 3H, H^{r}); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.79 (C^{a}), 130.20 (C^{m}), 130.01 (C^{i}), 128.02 (C^{j}), 127.90 (C^{l}), 61.05 ($C^{2/2'}$), 60.63 ($C^{2/2'}$), 52.31 ($C^{1/1'}$), 50.54 ($C^{1/1'}$), 33.61 (C^{b}), 31.52 (C^{p}), 29.66 (C^{g}), 29.50 (C^{o}), 29.45 (C^{d}), 29.43 (C^{e}), 29.24 (C^{f}), 27.23 (C^{n}), 27.19 (C^{h}), 25.63 (C^{k}), 25.33 (C^{c}), 22.57 (C^{q}), 14.08 (C^{r}); *m/z*: (ES^+) calcd. ($C_{22}H_{41}NO_3$ + Na^+): 390.3, found: 390.4 (M + Na^+).

1.8 (9*Z*,12*Z*,15*Z*)-*N*,*N*-Bis(2-hydroxyethyl)octadeca-9,12,15-trienamide 4f[5]



General procedure for the synthesis of diols was followed using linolenic acid, 70 % (9 g, 32.3 mmol, 1.1 eq.), NMM (3.6 g, 35.6 mmol, 1.1 eq.), ethyl chloroformate (3.9 g, 35.6 mmol, 1.1 eq.), diethanolamine (3.7 g, 35.6 mmol, 1.1 eq.) and TEA (3.6 g, 35.6 mmol, 1.1 eq.) to give a pale yellow oil. (7.0 g, 59 %); R_f = 0.2 in 100 % EtOAc; ν_{max}/cm^{-1} 3345(O-H) 2920 (C-H), 2857 (C-H), 1624 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.54 – 5.17 (m, 6H, $H^{i/j/l/m/o/p}$), 4.13 (brs, 2H, H^{OH}), 3.79 (t, J = 5.0 Hz, 2H, $H^{2/2'}$), 3.74 (t, J = 5.0 Hz, 2H, $H^{2/2'}$), 3.51 (t, J = 5.0 Hz, 2H, $H^{1/1'}$), 3.47 (t, J = 5.0 Hz, 2H, $H^{1/1'}$), 2.87 – 2.71 (m, 4H, H^{kn}), 2.36 (t, J = 7.5 Hz, 2H, H^{b}), 2.14 – 1.94 (m, 4H, H^{hq}), 1.75 – 1.49 (m, 2H, H^{c}), 1.37 – 1.22 (m, 8H, $H^{d\text{-}g}$), 0.95 (t, J = 7.5 Hz, 3H, H^{r}); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.74 (C^{a}), 132.04 ($C^{i/j/l/m/o/p}$), 130.35 ($C^{i/j/l/m/o/p}$), 128.37 ($C^{i/j/l/m/o/p}$), 128.33 ($C^{i/j/l/m/o/p}$), 127.80 ($C^{i/j/l/m/o/p}$), 127.20 ($C^{i/j/l/m/o/p}$), 61.46 ($C^{2/2'}$), 60.84 ($C^{2/2'}$), 52.35 ($C^{1/1'}$), 50.68 ($C^{1/1'}$), 33.70 (C^{b}), 29.70 (C^{g}), 29.51 (C^{d}), 29.48 (C^{e}), 29.28 (C^{f}), 27.31 (C^{h}), , 25.70 (C^{k}), 25.61 (C^{n}), 25.38 (C^{c}), 20.63 (C^{q}), 14.36 (C^{r}); *m/z*: (ES^+) calcd. ($C_{22}H_{39}NO_3$ + Na^+): 388.3, found: 388.1 (M + Na^+).

1.9 Methyl 8-(4-octyl-3-phenyl-4,5-dihydroisoxazol-5-yl)octanoate and regioisomers 8a-d

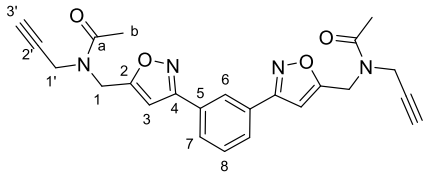


Methyl oleate (0.38 g, 1.3 mmol, 1 eq.) and **1a** (0.20 g, 1.3 mmol, 1 eq.) were dissolved in DMF (0.5 M) at RT, 4Å MS were added and the mixture was stirred for 30 minutes. It was then heated to 80 °C for 24 hours. The solution was diluted with $CHCl_3$ (100 ml) and the sieves were removed by filtration. The organic filtrate was washed with water (6 × 100 ml) and brine (100 ml), then dried with $MgSO_4$ and filtered. Solvent was removed *in vacuo* to leave a crude product. Purification by column chromatography through a silica plug resulted in a clear oil. (43 mg, 8 %); R_f = 0.2 in 1: 4 EtOAc: 40-60° petroleum ether; ν_{max}/cm^{-1} 2925 (C-H), 2854 (C-H), 1737 (C=O), 1196 (C-O), 1108 (C-O), 765, 692; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.70 – 7.69 (m, 2H, H^5), 7.42– 7.41 (m, 3H, $H^{6/7}$), 4.50 – 4.45 (m, 1H, H^1), 4.45 – 4.40 (m, 1H, H^1), 3.69 – 3.63 (m, 3H, H^a), 3.43 – 3.38 (m, 1H, H^2), 3.27 – 3.23 (m, 1H, H^2), 2.35 – 2.21 (m, 2H, H^c), 1.89 – 1.59 (m, 2H, H^d), 1.58 – 1.10 (m, 24H, $H^{e\text{-}i,j\text{-}p}$), 0.98 – 0.72 (m, 3H, H^q); ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.45 (C^b), 174.38 (C^b), 162.29 (C^3), 162.23 (C^3), 159.36 (C^3), 159.28 (C^3), 130.04 (C^4), 129.95 (C^7), 129.84 (C^7), 129.52 (C^4), 128.89 (C^6), 128.86 (C^6), 127.12 (C^5), 127.02 (C^5), 86.65 (C^1), 86.60 (C^1), 85.52 (C^1), 85.46 (C^1), 52.69 (C^2), 52.63 (C^2), 51.61 (C^a), 48.47 (C^2), 48.39 (C^2), 35.55 (C^c), 34.29 – 22.57 ($C^{d\text{-}i/j\text{-}o}$), 14.28 – 14.21 ($C^q$); *m/z:* ($ES^+$) calcd. ($C_{26}H_{41}NO_3$ + Na^+): 438.2979, found: 438.2977 (M + Na^+).

1.10 General procedure for the synthesis of oligomers $O^1$3, $O^1$5a, $O^2$5a and $O^1$5c(S) Stearic

Compound **1a** (1 eq.) was dissolved in DMF (0.5 M) followed by dipolarophile (8 eq.) and 4 Å MS (1600 g / mol of **1a**), the mixture was then raised to 80 °C for 24 hours. The solution was diluted with $CHCl_3$ (100 ml) and the sieves were removed by filtration. The organic filtrate was washed with water (6 × 100 ml) and brine (100 ml), then dried with $MgSO_4$ and solvent removed *in vacuo*, to leave a crude mixture of repeat units and dipolarophile. The crude product was separated by column chromatography, initially running in 100 % EtOAc to recover residual dipolarophile followed by 20 % acetone in EtOAc then 50 % acetone in EtOAc to recover the oligomers separately.

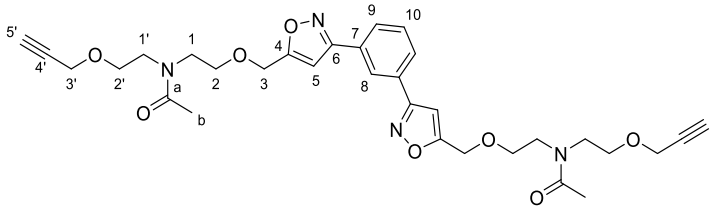
1.10.1 $O^1$3-ABA oligomer (3-1a-3)



Orange waxy oil, (53 %); Rf = 0.55 in 100 % EtOAc; ν_{max} / cm^{-1}: 3289 (C≡C−H), 3241 (C≡C−H), 2925 (C-H), 2854 (C-H), 2118 (C≡C), 1647 (C=O), 1407 (C-N), 912 (C-H); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 (m, 1H, H^6), 7.91 – 7.86 (m, 2H, H^7), 7.59 – 7.50 (m, 1H, H^8), 6.61 (s, 2H, H^3-min), 6.60 (s, 2H, H^3-maj), 4.82 (s, 4H, H^1), 4.35 (d, *J* = 2.0 Hz, 4H, $H^{1'}$-min), 4.18 (s, 4H, $H^{1'}$-maj), 2.36 (t, *J* = 2.0 Hz, 2H, $H^{3'}$-maj), 2.29 (t, *J* = 2.0 Hz, 2H, $H^{3'}$-maj), 2.27 (s, 6H, H^b-min), 2.25 (s, 6H, H^b-maj); ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.62 (C^a-maj), 170.22 (C^a-min), 169.31 (C^2-min), 169.22 (C^2-maj), 168.59 (C^2-min), 168.47 (C^2-maj), 162.19 (C^4-maj), 162.11 (C^4-min), 129.93 (C^5-min), 129.87 (C^8-maj), 129.79 (C^5-maj), 129.76 (C^8-min), 129.54 (C^5-min), 129.40 (C^5-maj), 128.70 (C^7-min), 128.41 (C^7-maj), 125.36 (C^6-min), 125.33 (C^6-maj), 101.47 (C^3-maj), 101.17 (C^3-min), 101.14 (C^3-min), 78.25 ($C^{2'}$-min), 77.54 ($C^{2'}$-min), 73.70 ($C^{3'}$-maj), 73.25 ($C^{3'}$-min),

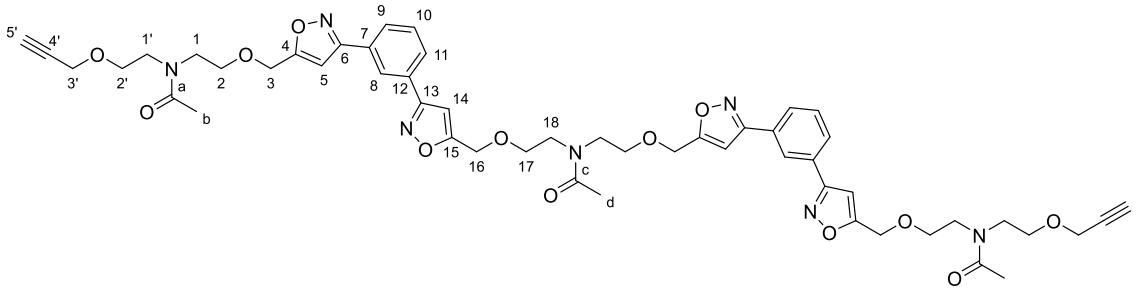
43.37 (C^{1}-min), 40.80 (C^{1}-maj), 38.85 ($C^{1'}$-maj), 38.81 ($C^{1'}$-maj), 34.62 ($C^{1'}$-min), 21.72 (C^{b}-min), 21.59 (C^{b}-maj); *m/z*: (ES^+) calcd. ($C_{24}H_{22}N_4O_4$ + Na^+): 453.1533, found: 453.1531 (M + Na^+).

1.10.2 O^{1}5a-ABA oligomer (5a-1a-5a)



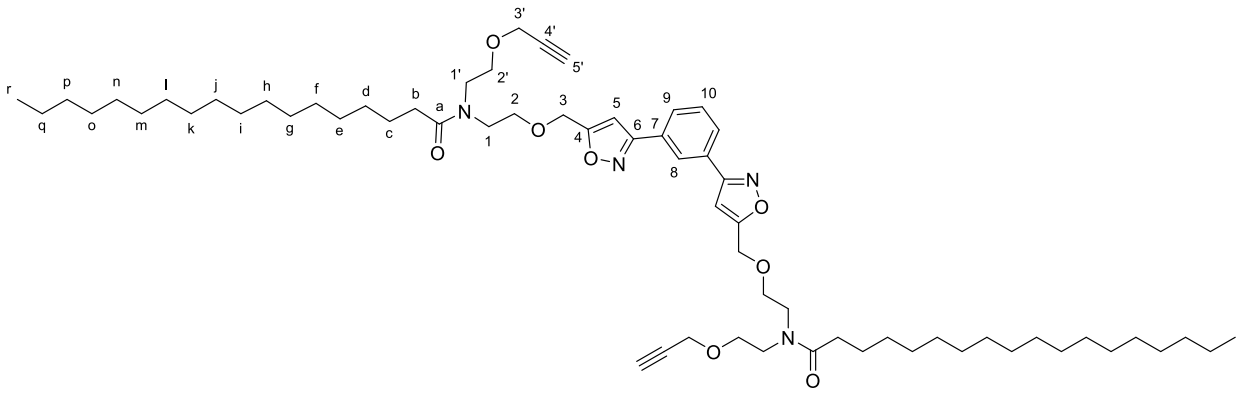
Oil (75 %); Rf = 0.20 in 100 % EtOAc; ν_{max} / cm^{-1}: 3286 (C≡C–H), 2922 (C-H), 2854 (C-H), 2114 (C≡C), 1630 (C=O), 1435 (C-N), 1098 (C-O), 910 (C-H); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.49 (d, *J* = 3.5 Hz, $H_{4,5\text{-isoxazole}}$), 8.22 (s, 1H, H_8), 7.93 – 7.87 (m, 2H, H_9), 7.59 – 7.53 (m, 1H, H_{10}), 6.64 (s, 2H, H_5), 6.63 (s, 2H, H_5), 6.62 (s, 2H, H_5), 6.61 (s, 2H, H_5), 4.67 (s, 4H, H_3-min), 4.64 (s, 4H, H_3-maj), 4.14 (d, *J* = 2.5 Hz, 4H, $H_{3'}$-maj), 4.12 (d, *J* = 2.5 Hz, 4H, $H_{3'}$-min), 3.74 (dt, *J* = 10.5, 5.5 Hz, 4H, H_2), 3.67 (dt, *J* = 14.5, 4.5 Hz, 8H, $H_{2'}$), 3.64 – 3.56 (m, 8H, $H_{1/1'}$), 2.43 (t, *J* = 2.5 Hz, $H_{5'}$-maj), 2.41 (t, *J* = 2.5 Hz, $H_{5'}$-min), 2.16 (s, H_b-min), 2.13 (s, H_b-maj); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.47 (C_a-maj), 171.43 (C_a), 170.02 (C_3-maj), 169.92 (C_3), 162.04 (C_6), 161.95 (C_6-maj), 129.88 (C_7), 129.85 (C_7), 129.84 (C_{10}-min), 129.81 (C_{10}-maj), 129.77 (C_{10}-min), 129.75 (C_7), 128.49 (C_9), 128.45 (C_9), 125.37 (C_8), 101.33 (C_5), 101.30 (C_5), 101.23 (C_5), 101.21 (C_5), 79.69 ($C_{4'}$-min), 79.37 ($C_{4'}$-maj), 75.01 ($C_{5'}$-maj), 74.68 ($C_{5'}$-min), 69.87 (C_2-maj), 69.44 (C_2-min), 68.90 ($C_{2'}$-min), 68.07 ($C_{2'}$-maj), 64.30 (C_3-min), 64.00 (C_3-maj), 58.63 ($C_{3'}$-maj), 58.44 ($C_{3'}$-min), 49.95 ($C_{1/1'}$-maj), 49.91 ($C_{1/1'}$-min), 46.45 ($C_{1/1'}$-min), 46.43 ($C_{1/1'}$-maj), 21.98 (C_b-min), 21.93 (C_b-maj); *m/z*: (ES^+) calcd. ($C_{32}H_{38}N_4O_8$ + Na^+): 629.2582, found: 629.2578 (M + Na^+).

1.10.3 O^{2}5a-ABABA oligomer (5a-1a-5a-1a-5a)



Cream solid, (3 %); Rf = 0.1 in 20 % Ac in EtOAc; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.49 (d, *J* = 3.5 Hz, $H_{4,5\text{-isoxazoline}}$), 8.21 (s, 2H, H^{8}), 7.93 – 7.84 (m, 4H, $H^{9/11}$), 7.54 (t, *J* = 7.5 Hz, 2H, H^{10}), 6.66 – 6.58 (m, 4H, $H^{5/14}$), 4.72 – 4.58 (m, 8H, $H^{3/16}$), 4.14 (d, *J* = 2.5 Hz, 4H, $H^{3'}$-maj), 4.11 (d, *J* = 2.5 Hz, 4H, $H^{3'}$-min), 3.79 – 3.70 (m, 8H, $H^{2/17}$), 3.70 – 3.55 (m, 16H, $H^{1/18/1'/2'}$), 2.43 (t, *J* = 2.5 Hz, 2H, $H^{5'}$-maj), 2.41 (t, *J* = 2.5 Hz, 2H, $H^{5'}$-min), 2.16 (s, 9H, $H^{b/d}$-maj), 2.12 (s, 9H, $H^{b/d}$-min); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.52 ($C^{a/c}$), 171.49 ($C^{a/c}$), 171.45 ($C^{a/c}$), 170.02 ($C^{5/14}$), 169.92 ($C^{5/14}$), 169.89 ($C^{5/14}$), 169.82 ($C^{5/14}$), 162.01 ($C^{6/13}$), 161.95 ($C^{6/13}$), 161.93 ($C^{6/13}$), 129.87 ($C^{7/12}$), 129.85 ($C^{7/12}$), 129.83 (C^{10}), 129.80 (C^{10}), 129.75 ($C^{7/12}$), 129.72 ($C^{7/12}$), 128.47 ($C^{9/11}$), 128.43 ($C^{9/11}$), 128.34 ($C^{9/11}$), 125.34 (C^{8}), 101.41 ($C^{5/14}$), 101.39 ($C^{5/14}$), 101.33 ($C^{5/14}$), 101.31 ($C^{5/14}$), 101.23 ($C^{5/14}$), 101.20 ($C^{5/14}$), 79.70 ($C^{4'}$), 79.38 ($C^{4'}$), 75.02 ($C^{5'}$-maj), 74.69 ($C^{5'}$-min), 69.95 ($C^{2/17/2'}$), 69.86 ($C^{2/17/2'}$), 69.46 ($C^{2/17/2'}$), 68.88 ($C^{2/17/2'}$), 68.06 ($C^{2/17/2'}$), 64.29 ($C^{3/16}$), 64.23 ($C^{3/16}$), 63.99 ($C^{3/16}$), 63.95 ($C^{3/16}$), 58.63 ($C^{3'}$), 58.44 ($C^{3'}$), 50.06 ($C^{1/18/1'}$), 49.95 ($C^{1/18/1'}$), 49.91 ($C^{1/18/1'}$), 46.65 ($C^{1/18/1'}$), 46.44 ($C^{1/18/1'}$), 46.42 ($C^{1/18/1'}$), 21.98 ($C^{b/d}$-maj), 21.93 ($C^{b/d}$-maj) ; MS (ESI) m/z calcd. ($C_{52}H_{59}N_7O_{13}$ + Na^+): 1012.4063, found: 1012.4067 (M + Na^+).

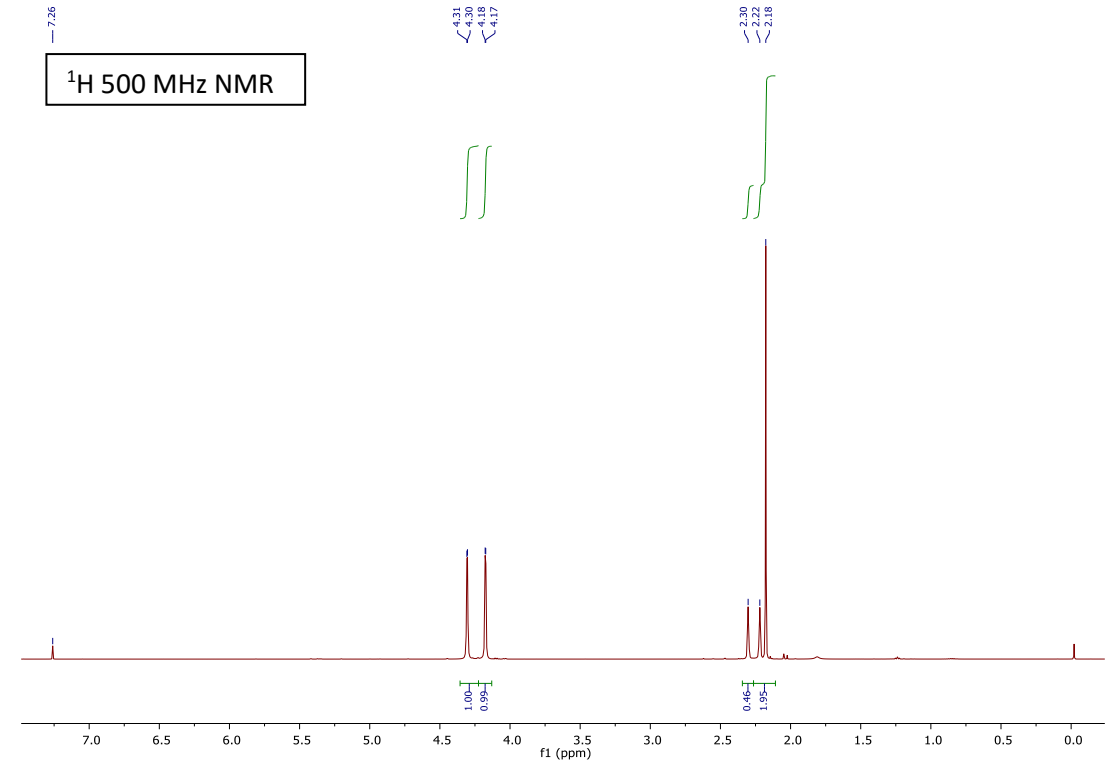
1.10.4 $O^1$5c(S)-ABA oligomer (5c(S)-1a-5c(S))

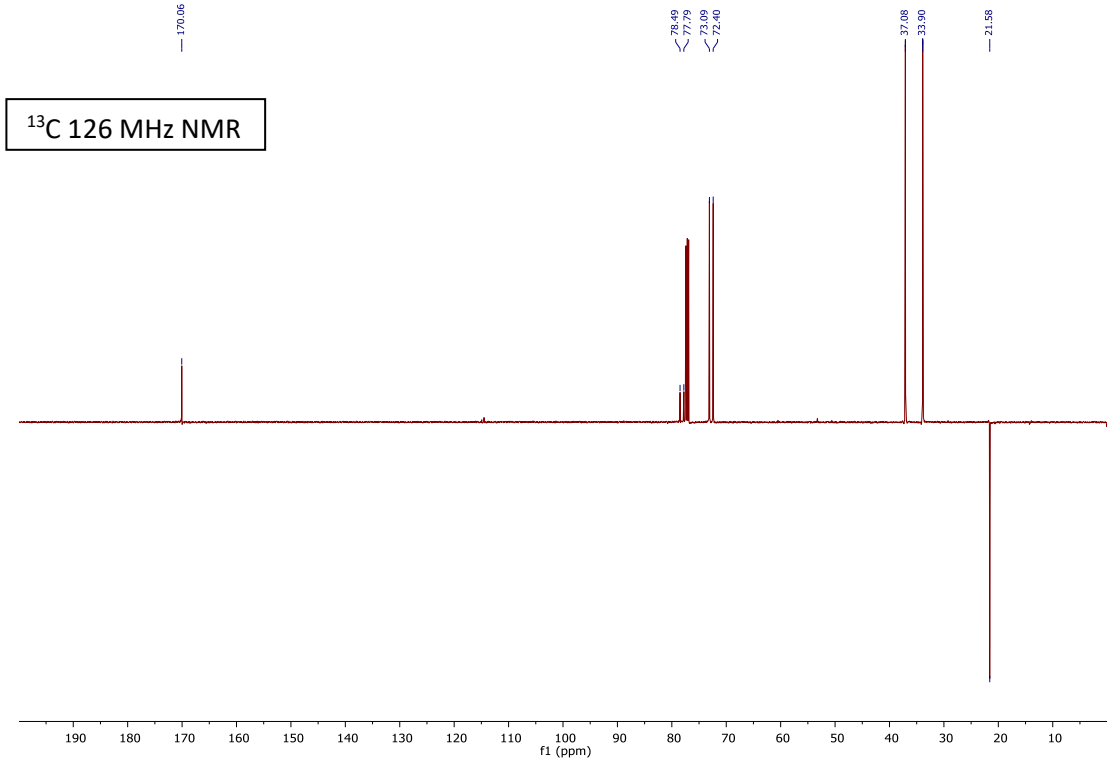


White waxy residue (48 %); Rf 0.1 in 50 % EtOAc; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.49 (s, $H^{3,4\text{-isoxazole}}$), 8.25 – 8.21 (m, 1H, H^8), 7.92 – 7.88 (m, 2H, H^9), 7.59 – 7.53 (m, 1H, H^{10}), 6.64 (s, 2H, H^5), 6.63 (s, 2H, H^5) 6.62 (s, 2H, H^5), 6.62 (s, 2H, H^5) 4.67 (s, 2H, H^3-min), 4.64 (s, 2H, H^3-maj), 4.14 (d, *J* = 2.5 Hz, 2H, $H^{3'}$-maj), 4.12 (d, *J* = 2.5 Hz, 2H, $H^{3'}$-min), 3.77 – 3.57 (m, 8H, $H^{1/1'/2/2'}$), 2.43 (t, *J* = 2.5 Hz, 2H, $H^{5'}$), 2.41 (t, *J* = 2.5 Hz, 2H, $H^{5'}$), 2.40 – 2.33 (m, 2H, H^b), 1.66 – 1.57 (m, 2H, H^c), 1.34 – 1.18 (m, 56H, $H^{d\text{-}q}$), 0.88 (t, *J* = 6.9 Hz, 6H, H^r); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.98 (C^a-maj), 173.93 (C^a-min) 170.10 (C^4-maj), 169.94 (C^4-min), 162.02 (C^6), 161.94 (C^6), 129.93 (C^7), 129.89 (C^7), 129.83 (C^{10}), 129.79 ($C^{10)}$, 129.76 (C^{10}), 128.49 (C^9), 128.45 (C^9), 125.35 (C^8), 101.27 (C^5), 101.25 (C^5), 101.23 (C^5), 79.76 ($C^{4'}$-maj), 79.40 ($C^{4'}$-min), 75.00 ($C^{5'}$-maj), 74.62 ($C^{5'}$-min), 69.94 (C^2-maj), 69.62 (C^2-min), 68.97 ($C^{2'}$-min), 68.24 ($C^{2'}$-maj), 64.32 (C^3-min), 64.02 (C^3-maj), 58.64 ($C^{3'}$-maj), 58.44 ($C^{3'}$-min), 49.02 ($C^{1/1'}$-maj), 48.99 ($C^{1/1'}$-min), 46.69 ($C^{1/1'}$-min), 46.44 ($C^{1/1'}$-maj), 33.32 (C^b), 33.28 (C^b), 32.07 (C^p), 29.89 – 29.51 ($C^{d\text{-}o}$), 25.49 (C^c), 25.44 (C^c), 22.84 (C^q), 14.27 (C^r); *m/z:* (ES^+) calcd. ($C_{64}H_{102}N_4O_8$ + Na^+): 1077.7590, found: 1077.7590 (M + Na^+).

2.0 NMR and VT NMR data for new compounds

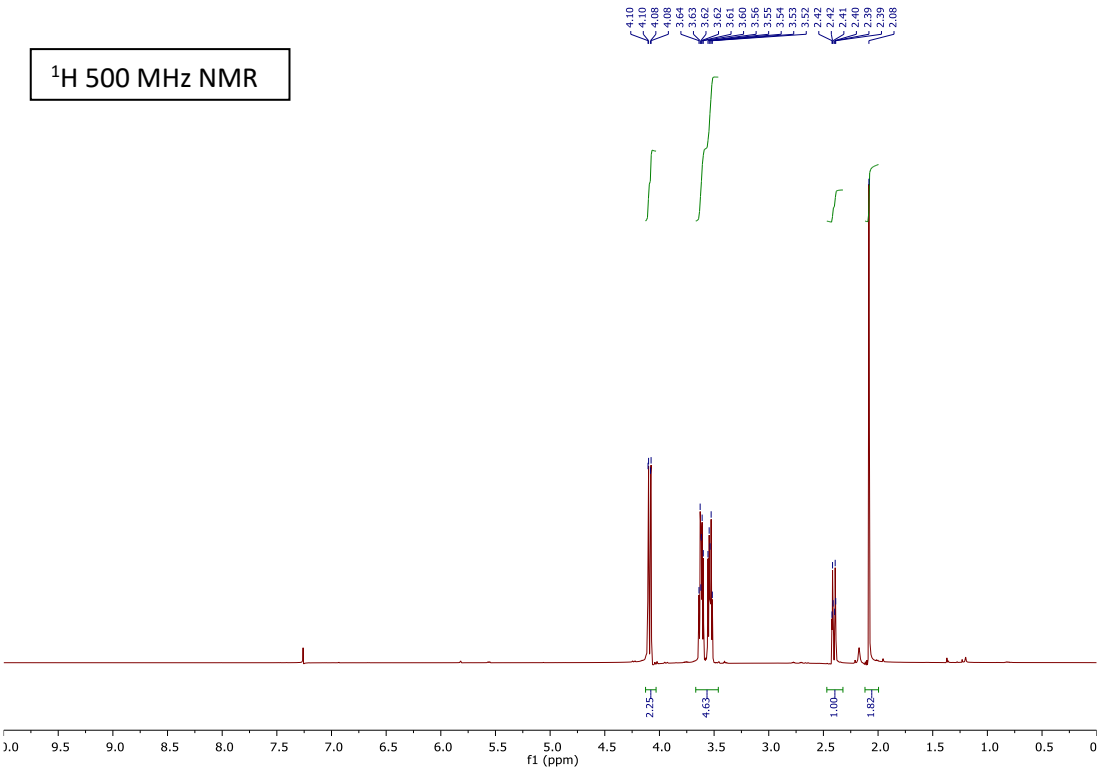
2.1 *N*,*N*-di(prop-2-yn-1-yl)acetamide 3



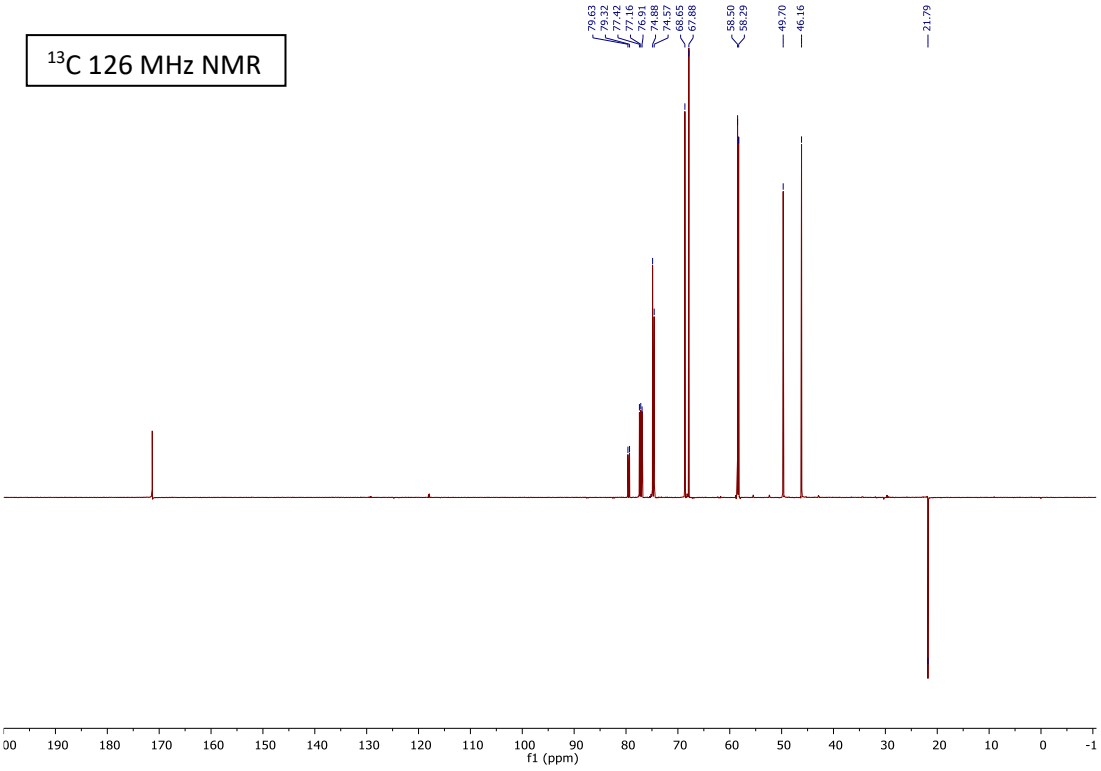


2.2 *N,N*-bis(2-(prop-2-yn-1-yloxy)ethyl)acetamide 5a(Me)

^{1}H 500 MHz NMR

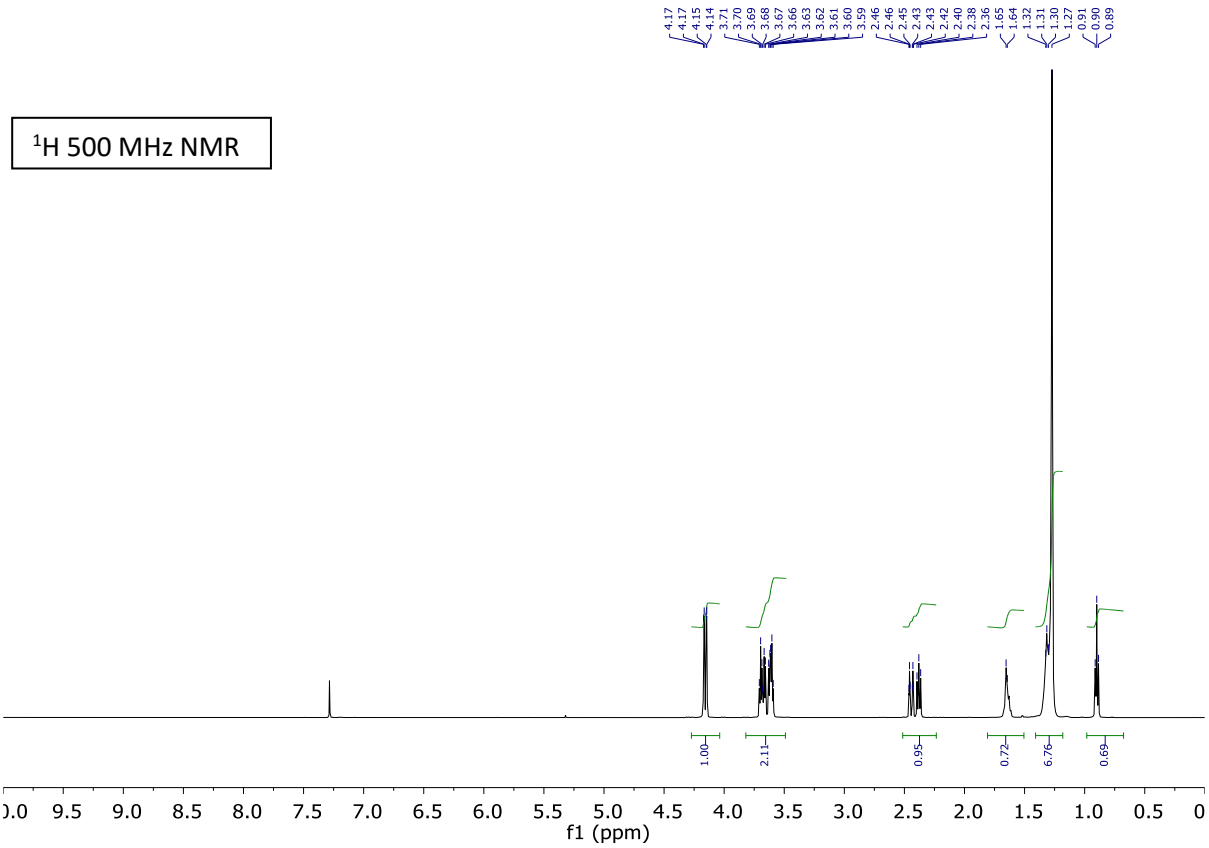


^{13}C 126 MHz NMR

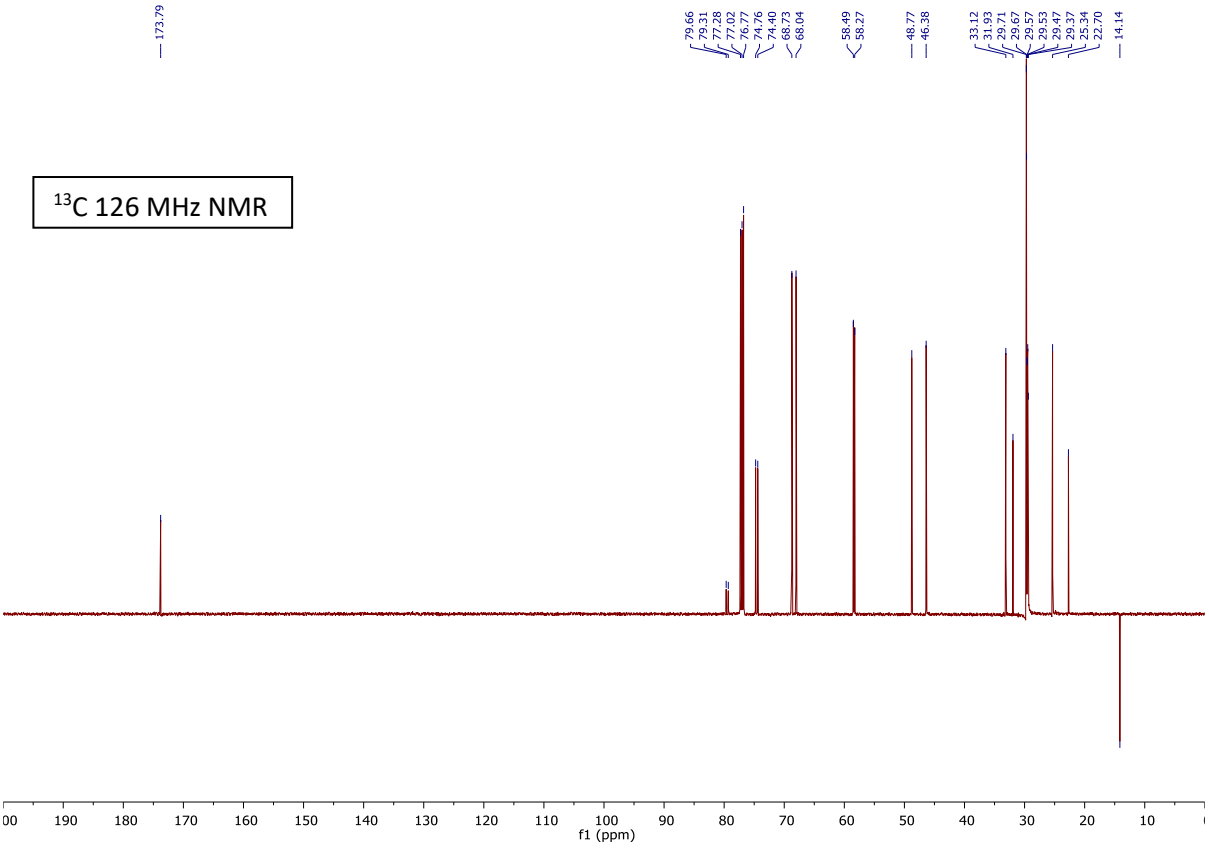


2.3 *N,N*-bis(2-(prop-2-yn-1-yloxy)ethyl)palmitamide 5b(P)

^{1}H 500 MHz NMR

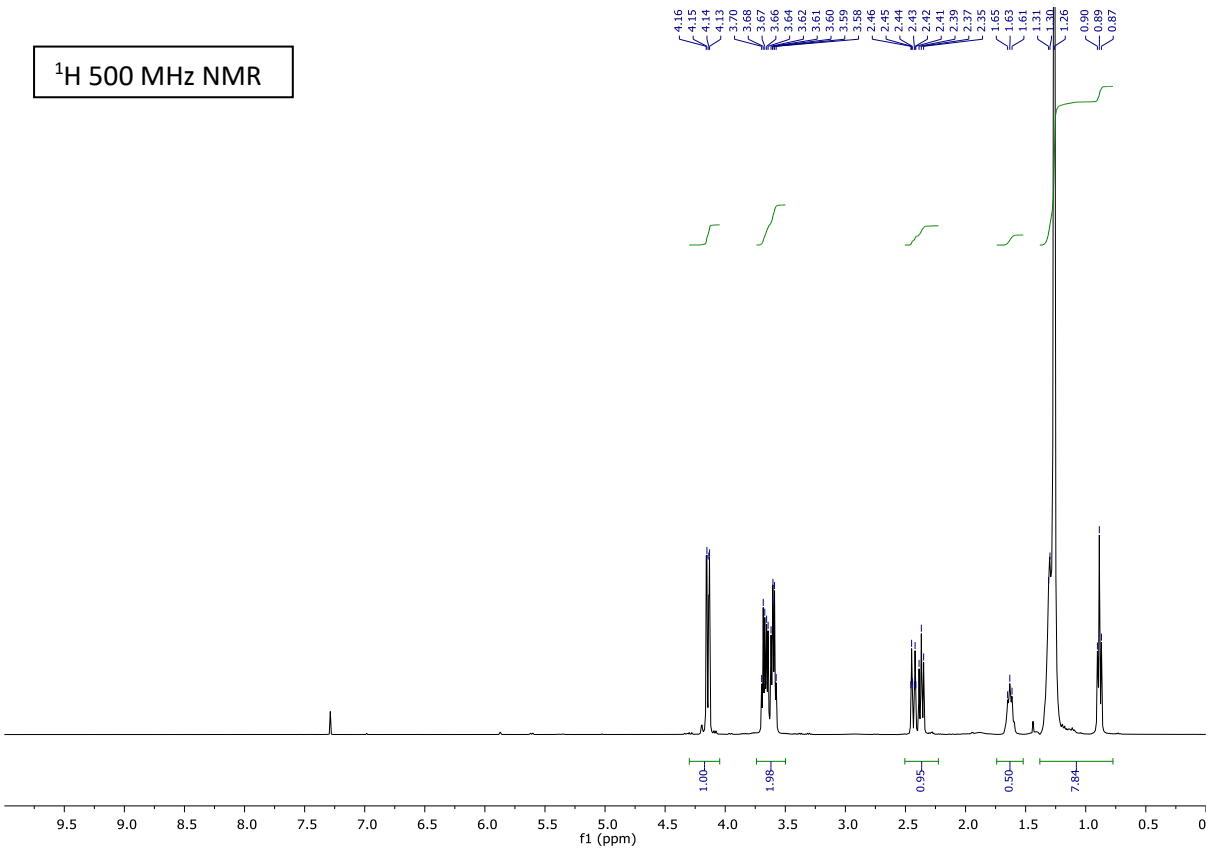


^{13}C 126 MHz NMR

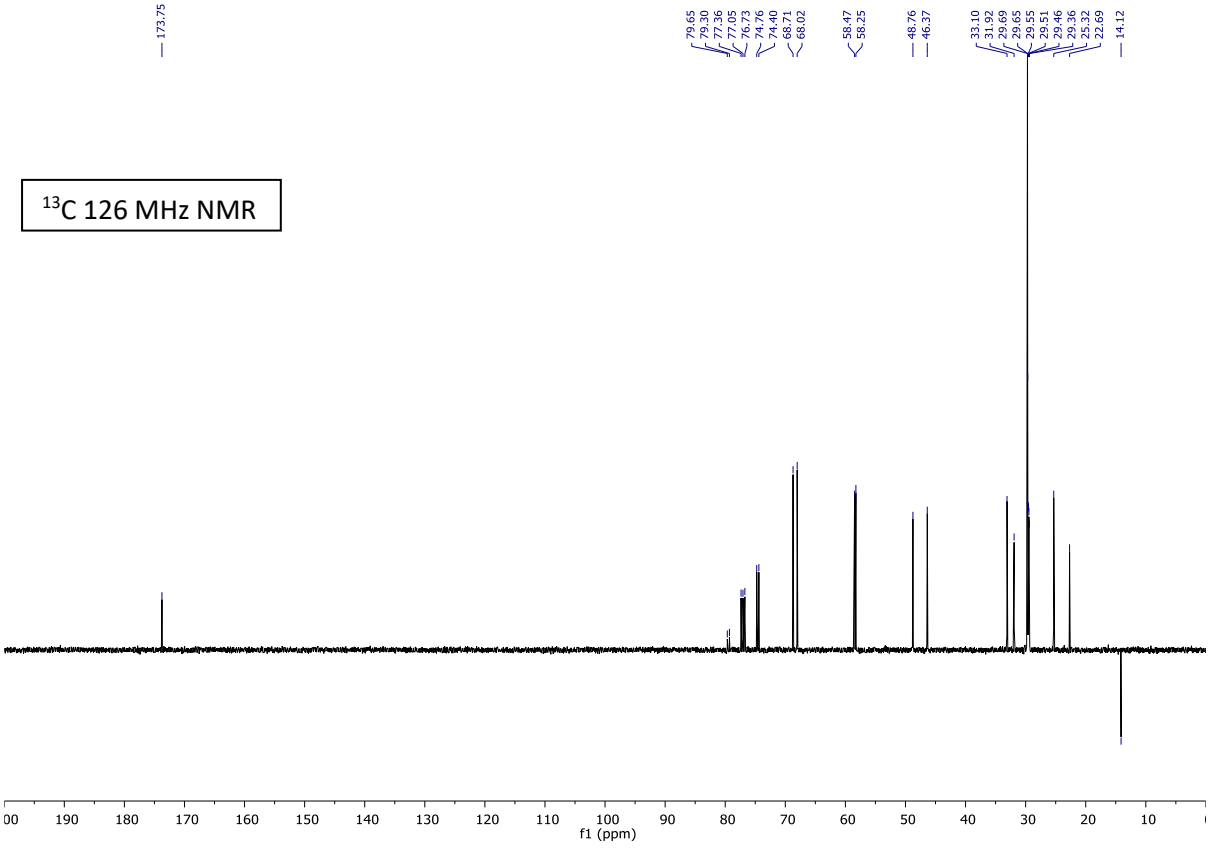


2.4 *N,N*-bis(2-(prop-2-yn-1-yloxy)ethyl)stearamide 5c(S)

^{1}H 500 MHz NMR

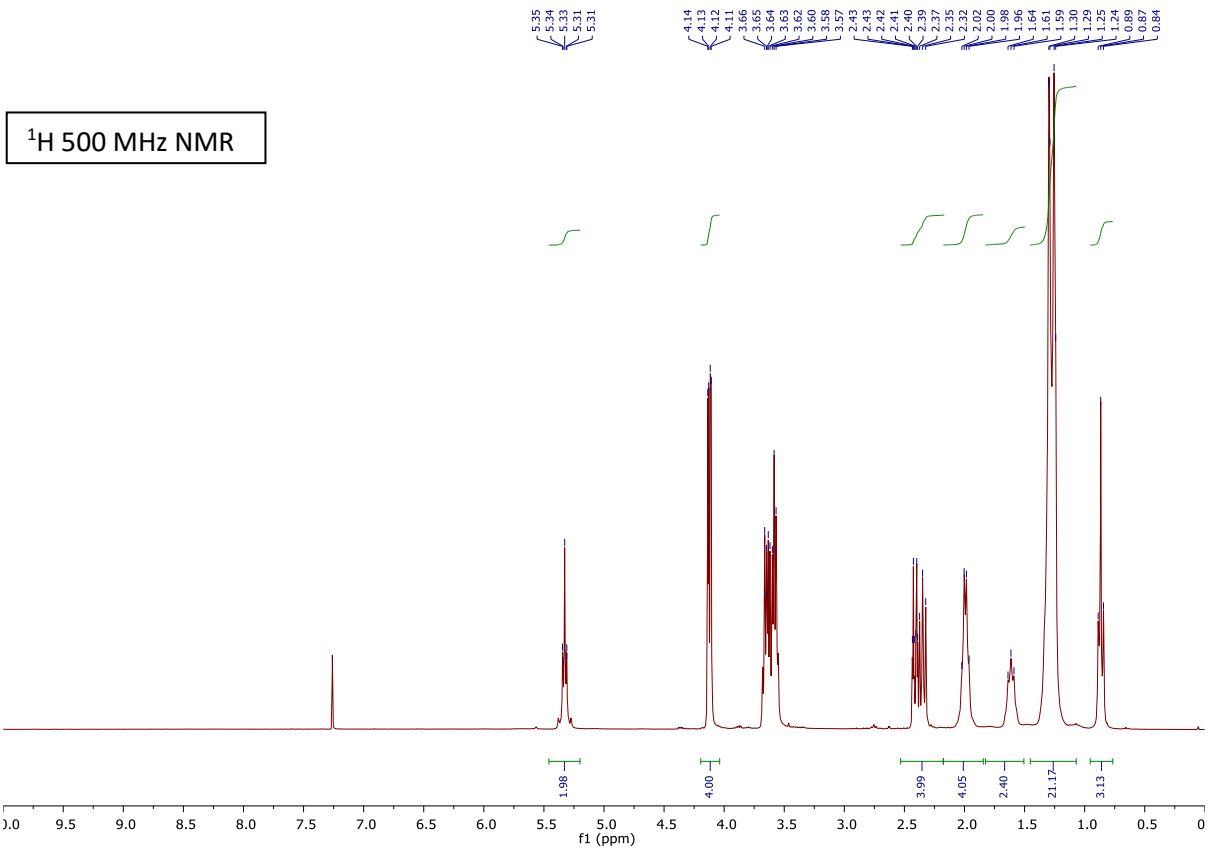


^{13}C 126 MHz NMR

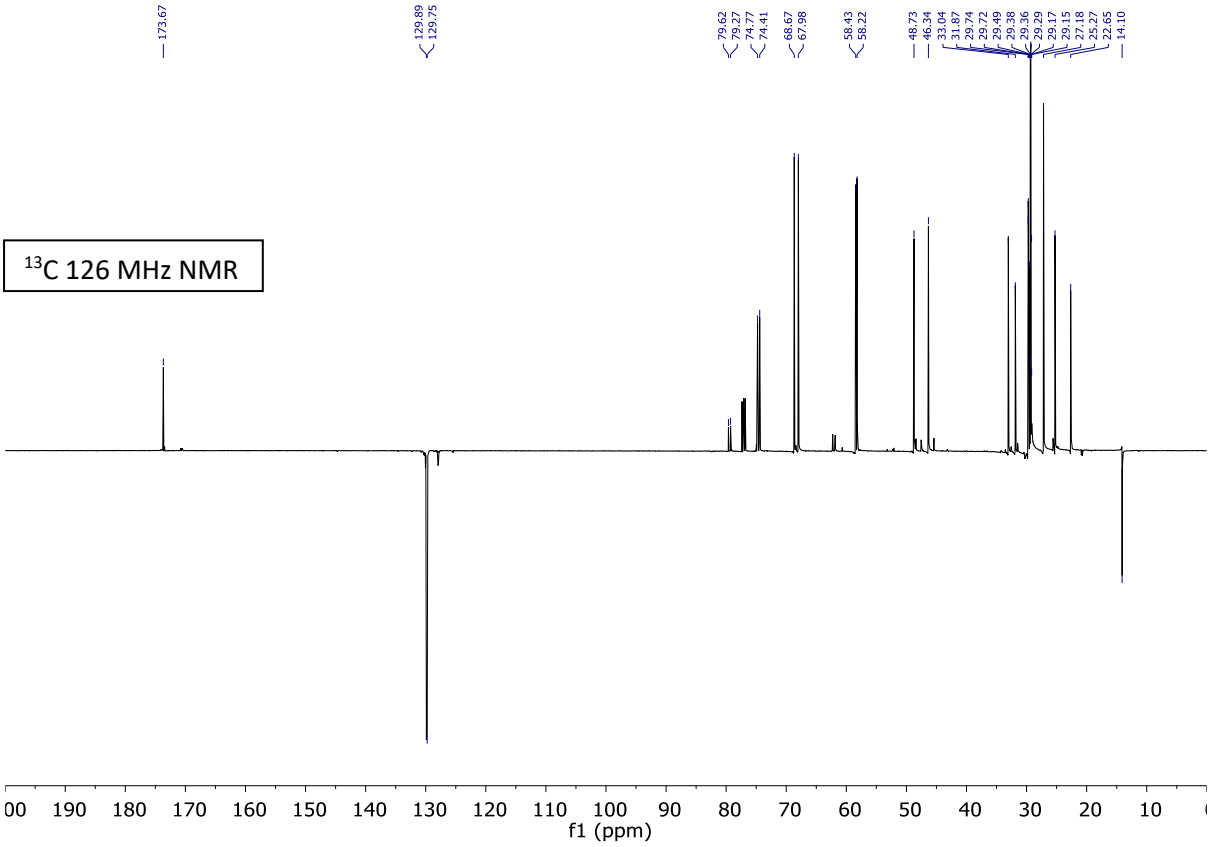


2.5 *N,N*-bis(2-(prop-2-yn-1-yloxy)ethyl)oleamide 5d(O)

^{1}H 500 MHz NMR

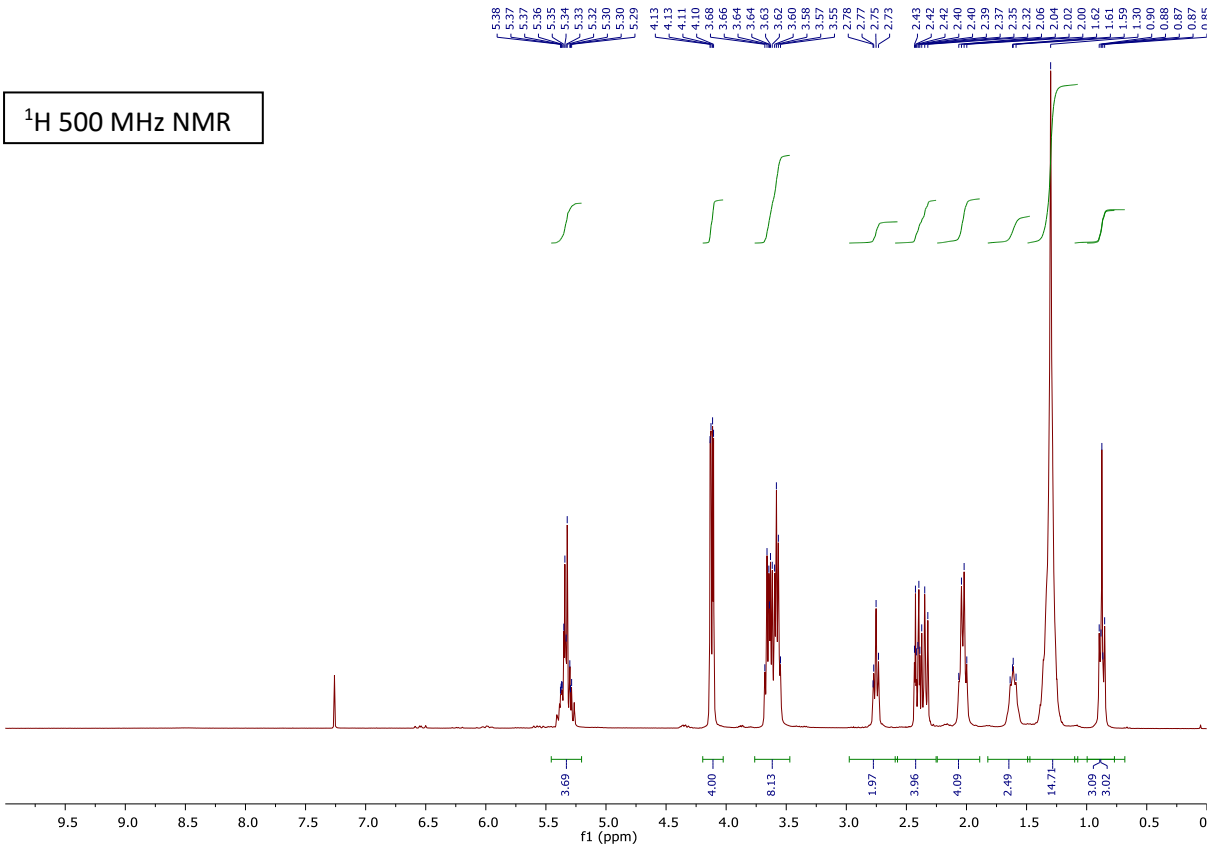


^{13}C 126 MHz NMR

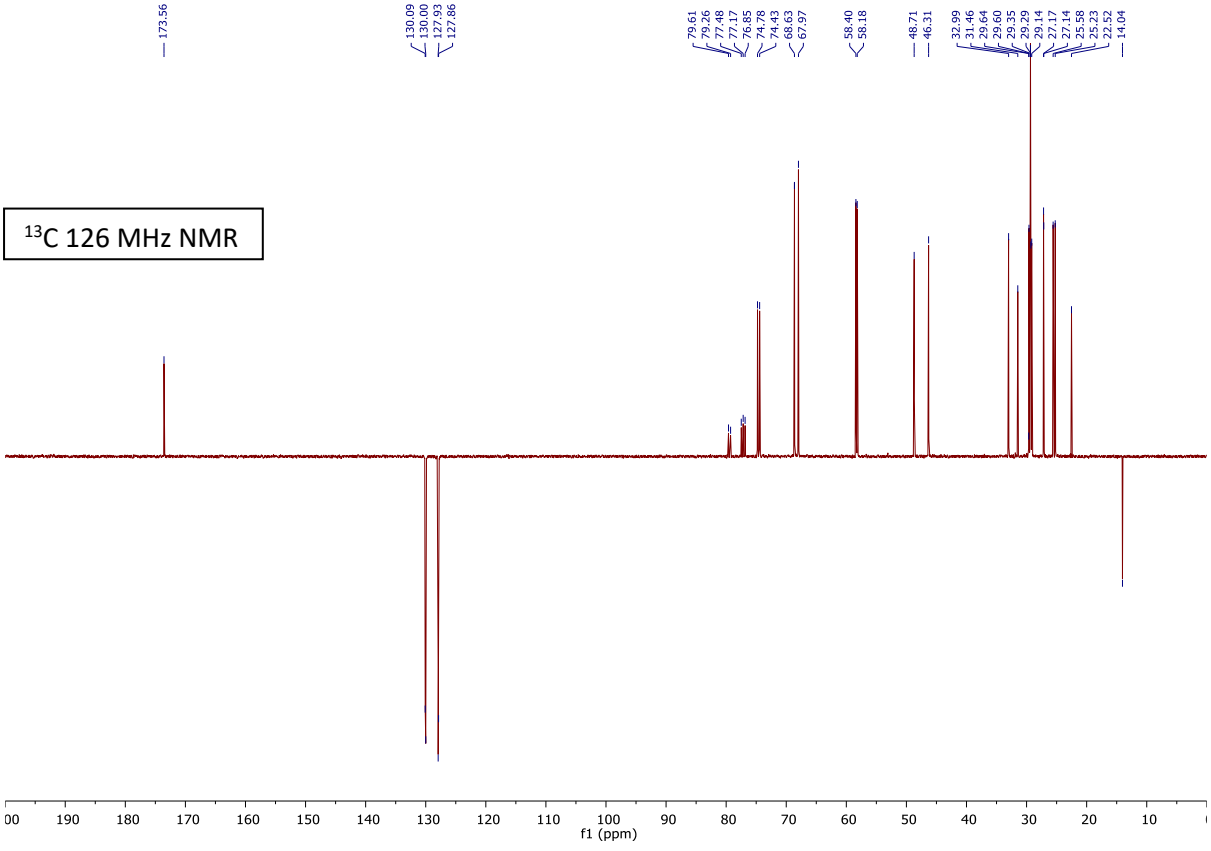


2.6 (9*Z*,12*Z*)-*N*,*N*-bis(2-(prop-2-yn-1-yloxy)ethyl)octadeca-9,12-dienamide 5e(L)

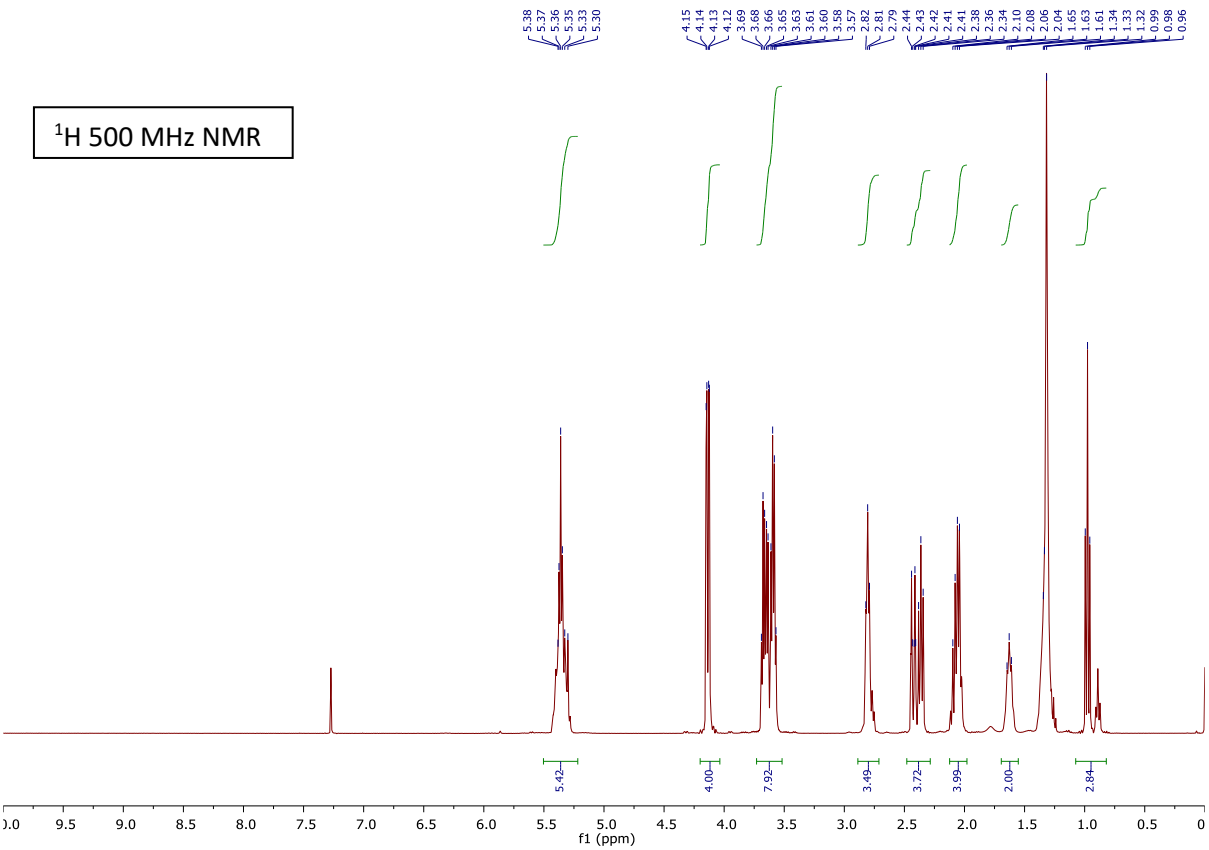
^{1}H 500 MHz NMR

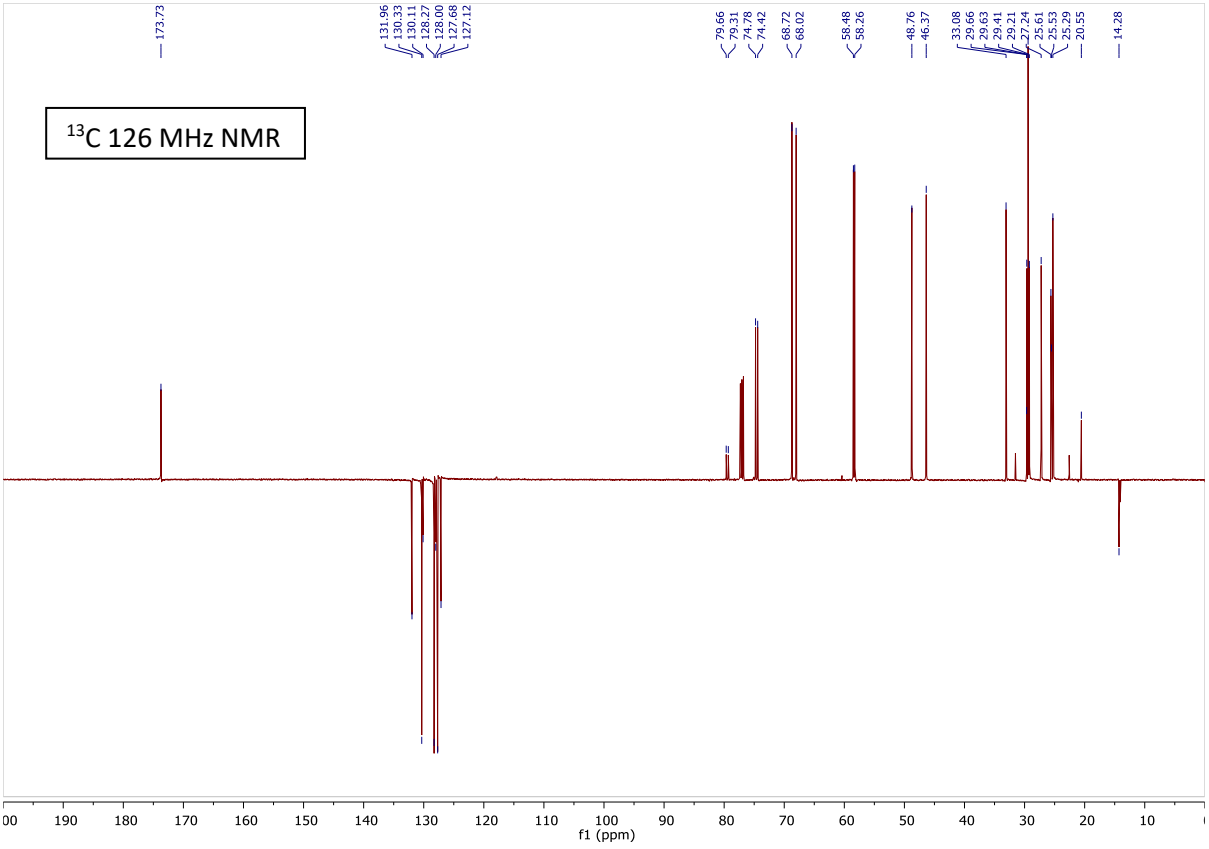


^{13}C 126 MHz NMR

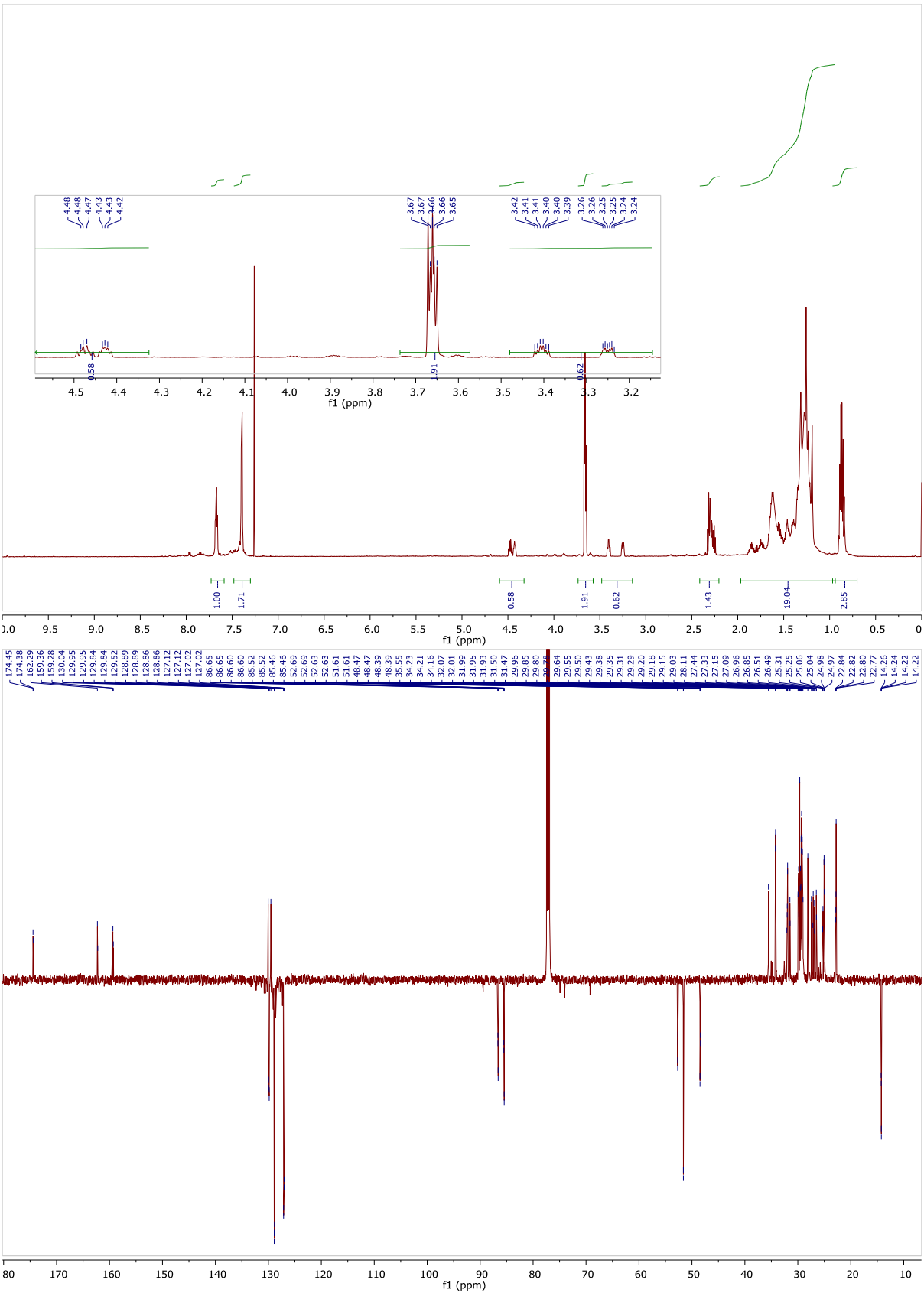


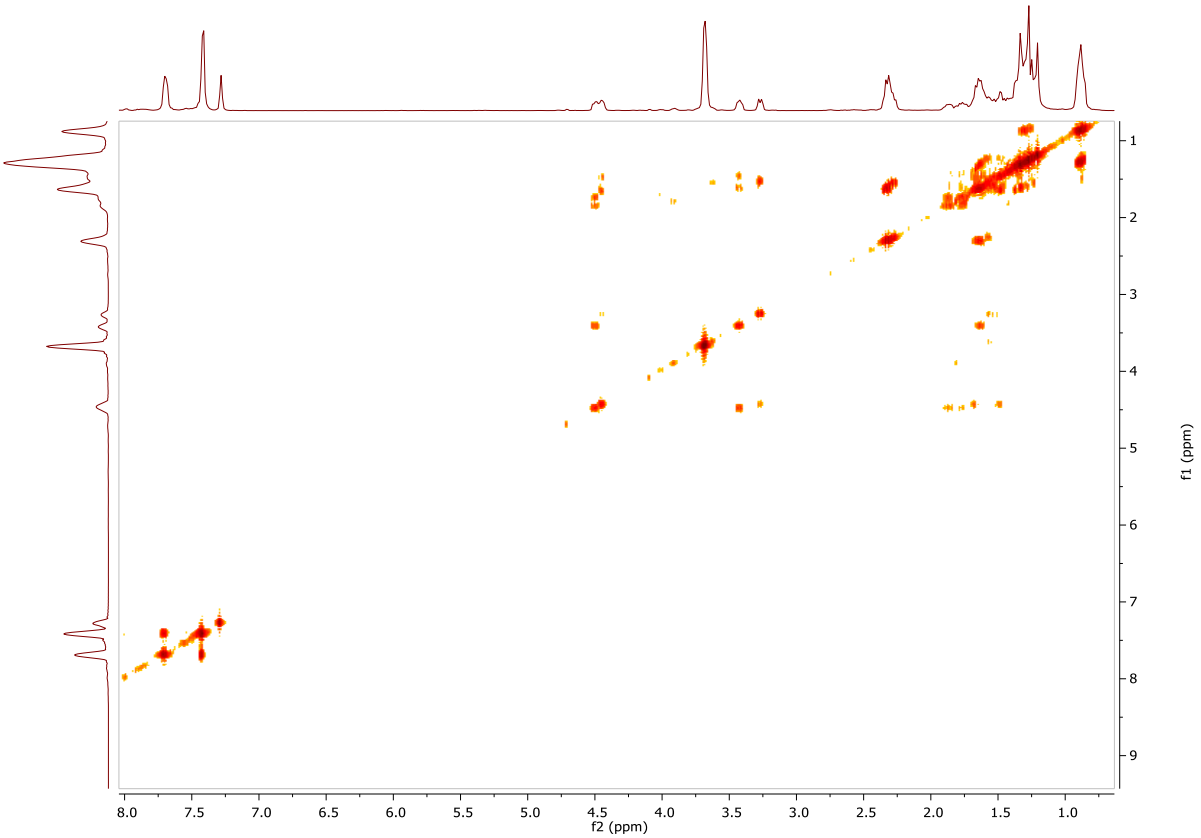
2.7 (9*Z*,12*Z*,15*Z*)-*N*,*N*-bis(2-(prop-2-yn-1-yloxy)ethyl)octadeca-9,12,15-trienamide 5f(Ln)

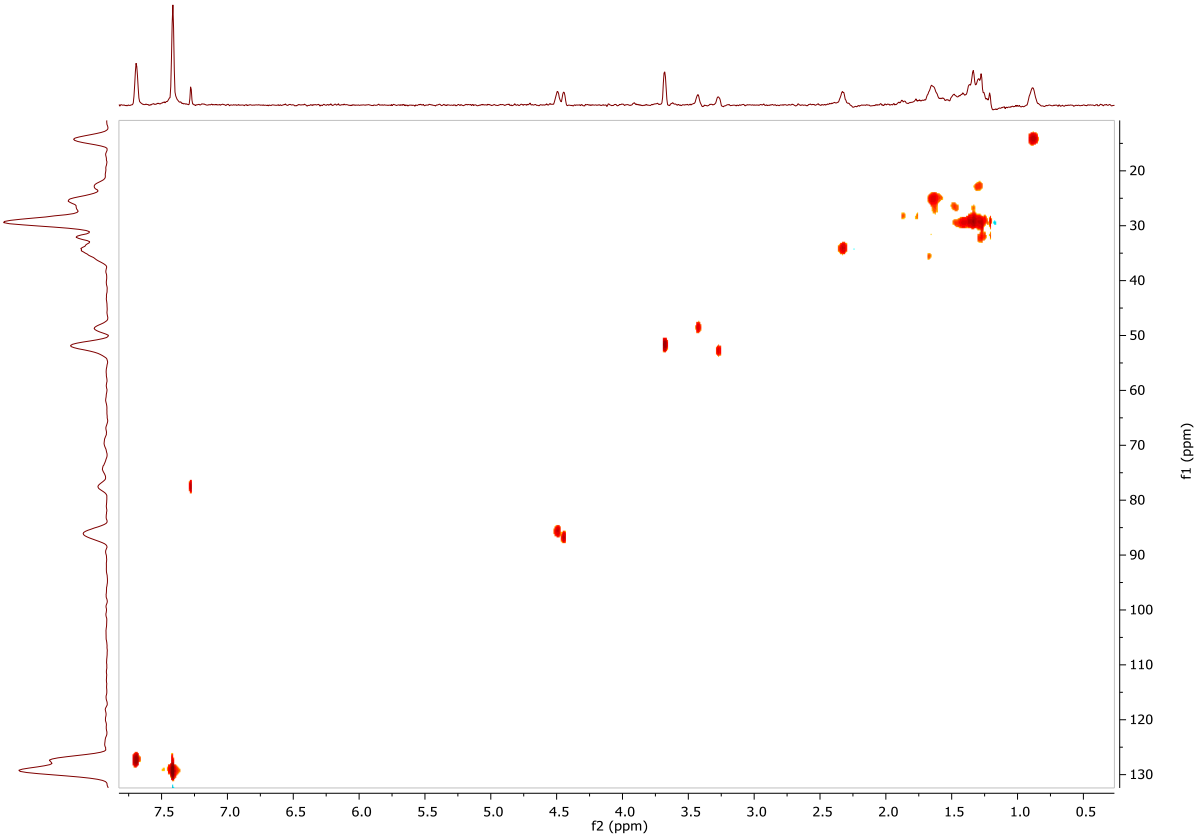




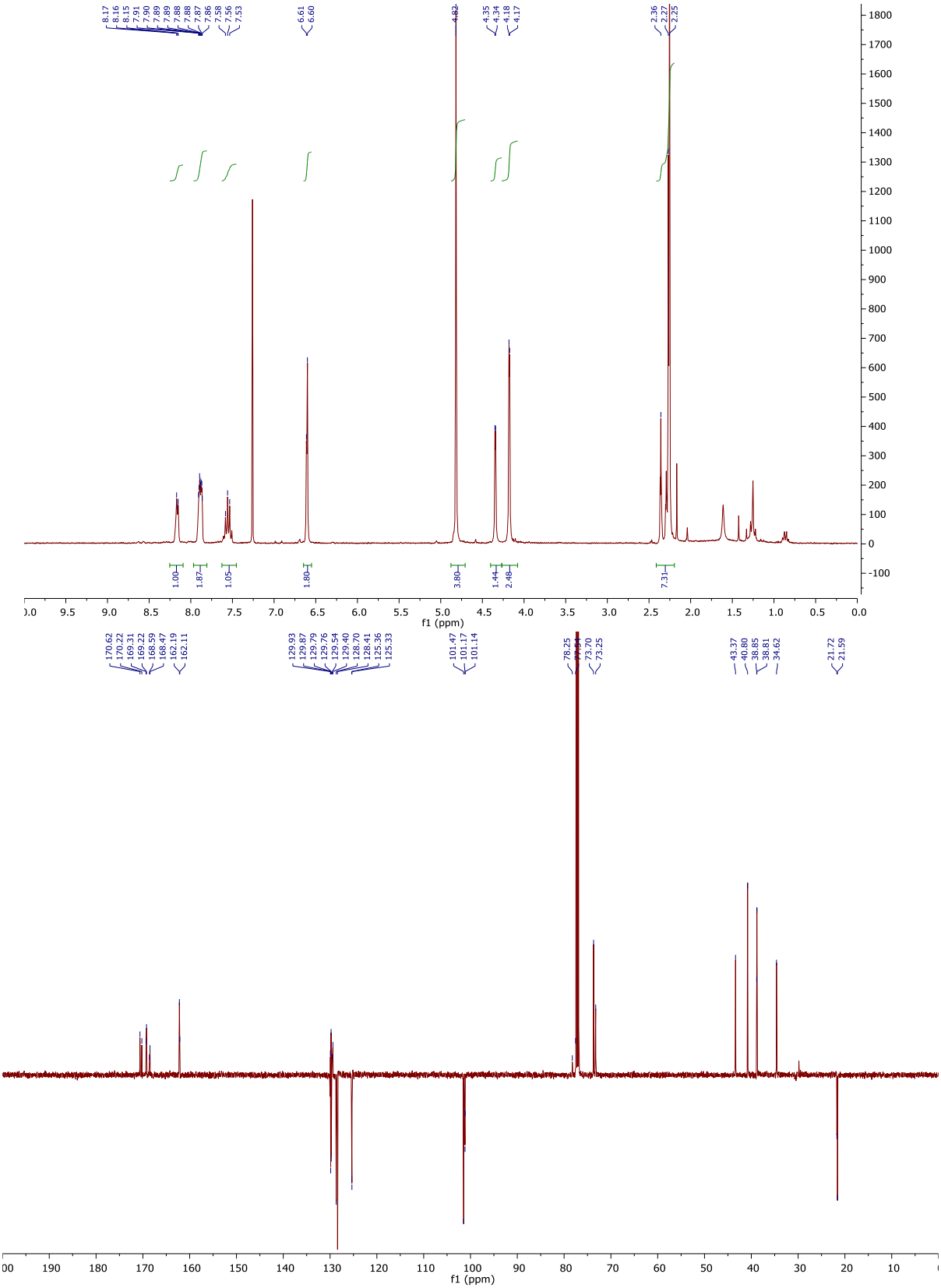
2.8 Methyl-8-(4-octyl-3-phenyl-4,5-dihydroisoxazol-5-yl)octanoate and regioisomer 8a-d



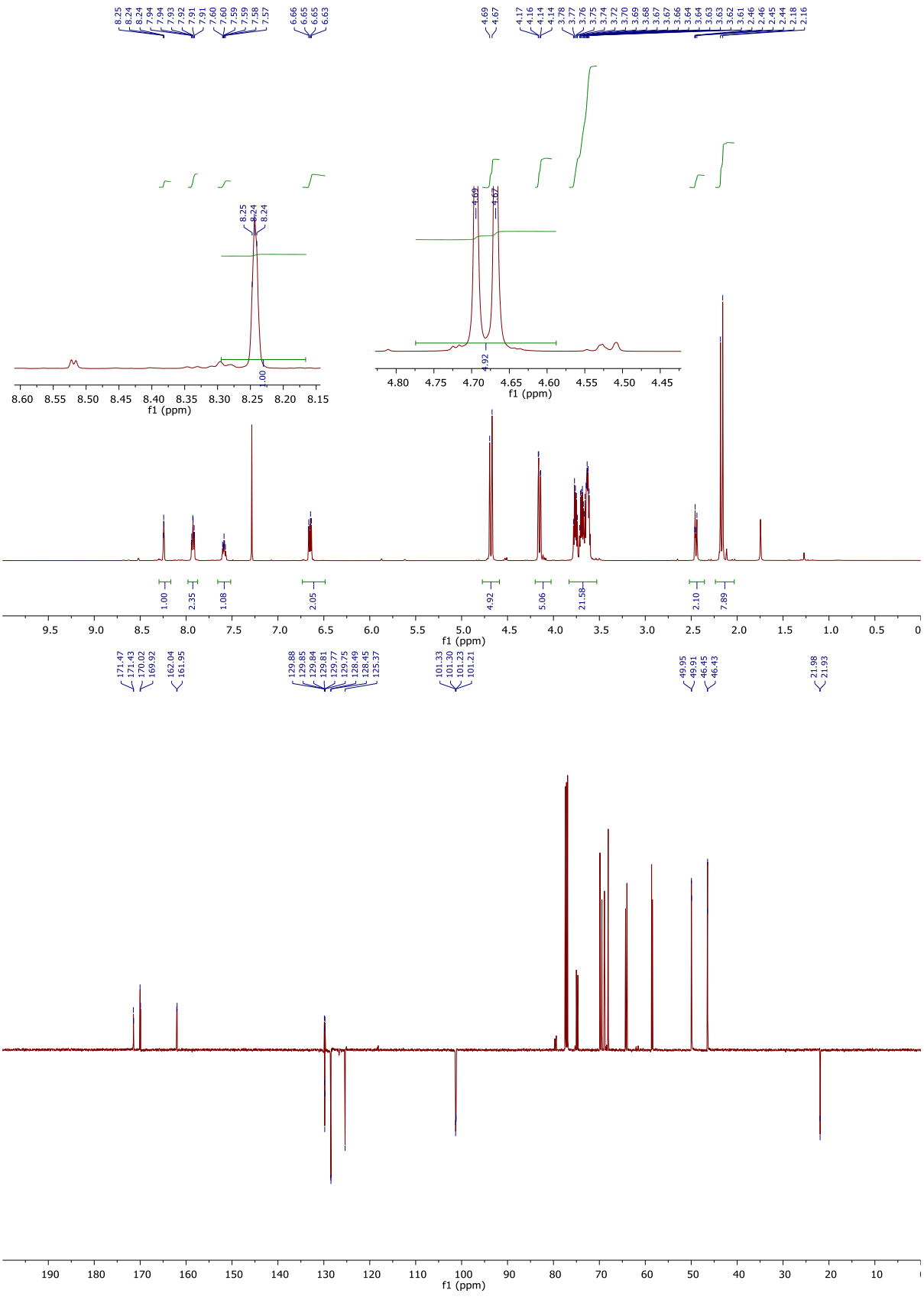

f2 (ppm)
f1 (ppm)


f2 (ppm)
f1 (ppm)

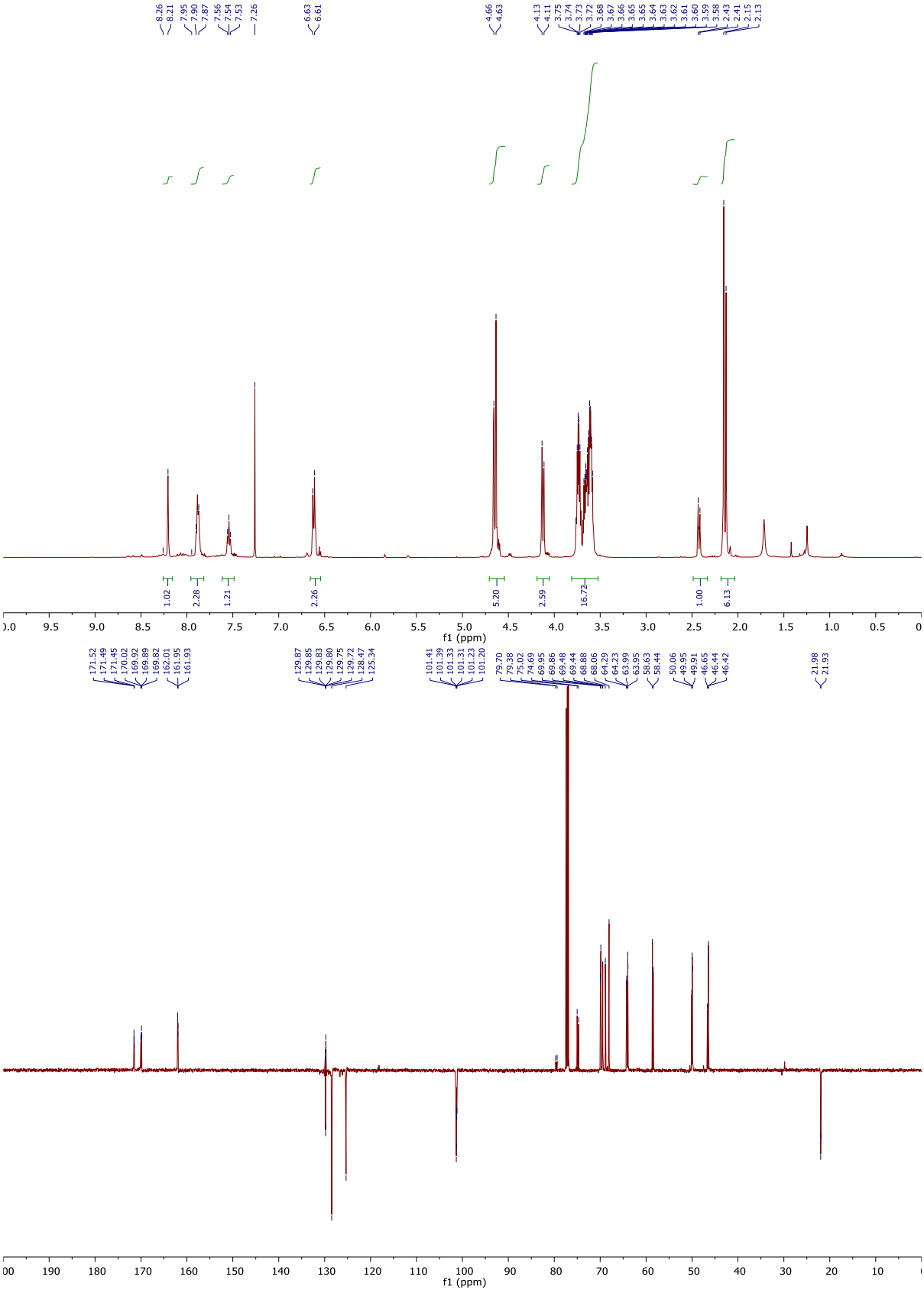
2.9 O[1]3-ABA


8.17
8.16
8.15
7.91
7.90
7.89
7.88
7.88
7.87
7.86
7.58
7.56
7.53
6.61
6.60
4.82
4.35
4.34
4.18
4.17
2.36
2.27
2.25
1.00
1.87
1.05
1.80
3.80
1.44
2.48
7.31
f1 (ppm)
170.62
170.22
169.31
169.22
168.59
168.47
162.19
162.11
129.93
129.87
129.79
129.76
129.54
129.40
128.70
128.41
125.36
125.33
101.47
101.17
101.14
78.25
77.84
73.70
73.25
43.37
40.80
38.85
38.81
34.62
21.72
21.59
f1 (ppm)

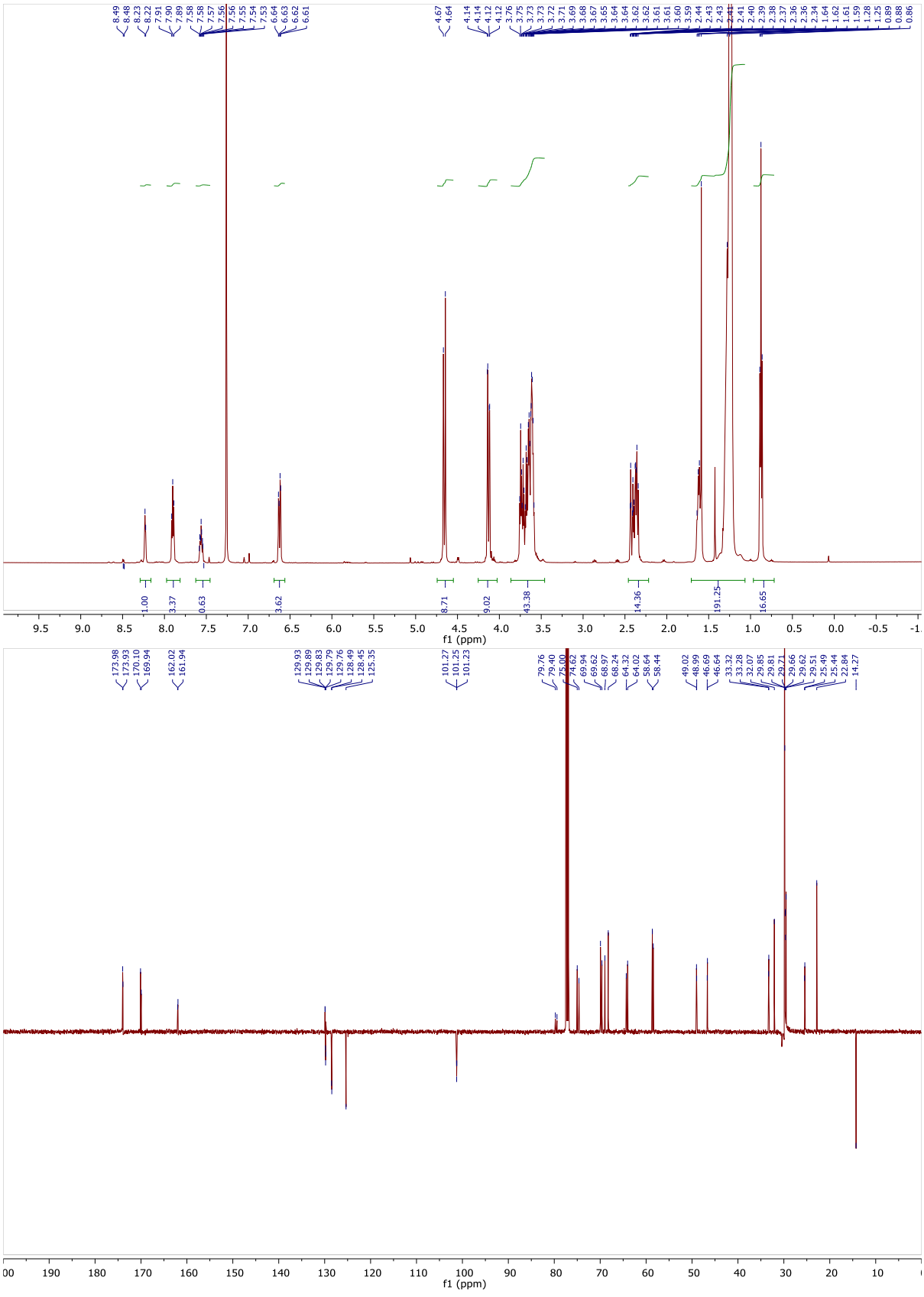
2.10 O^{1}5a(Me)-ABA



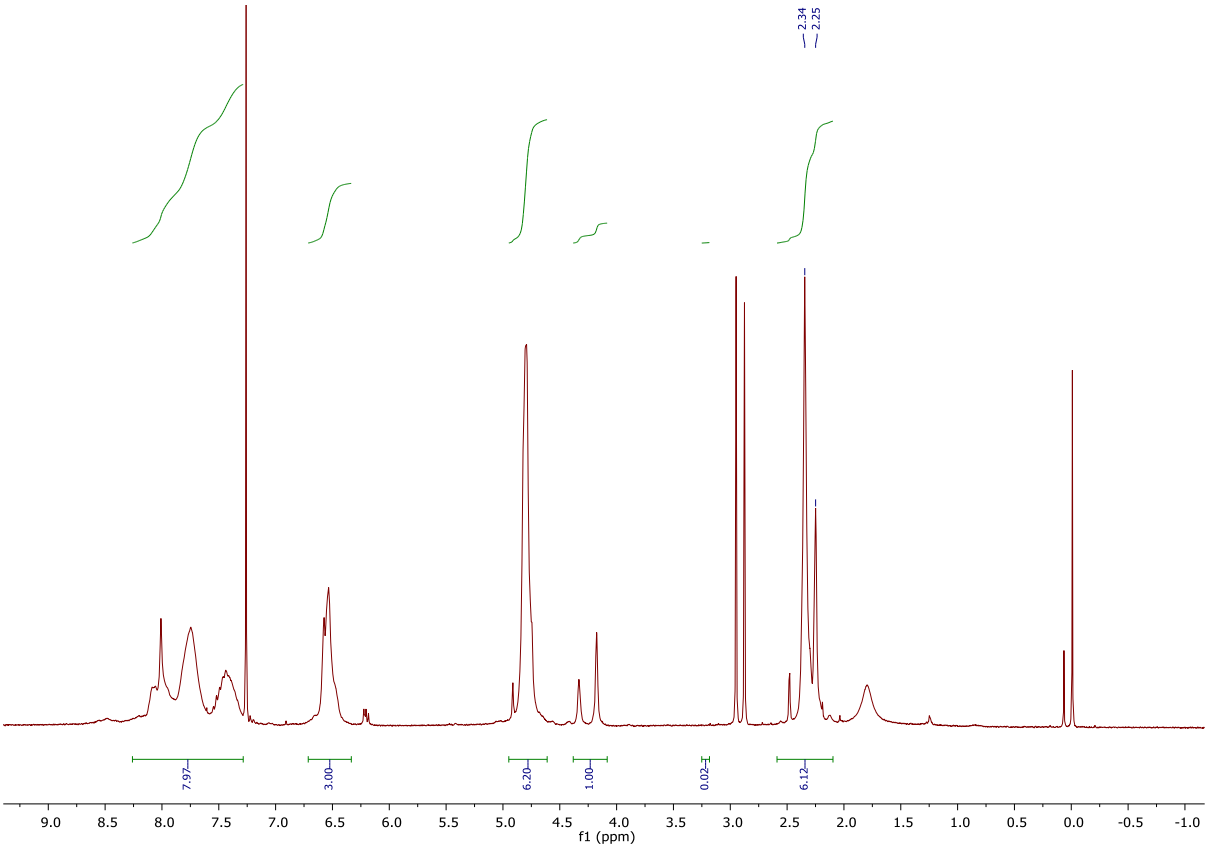
$O^2$5a(Me)-ABABA



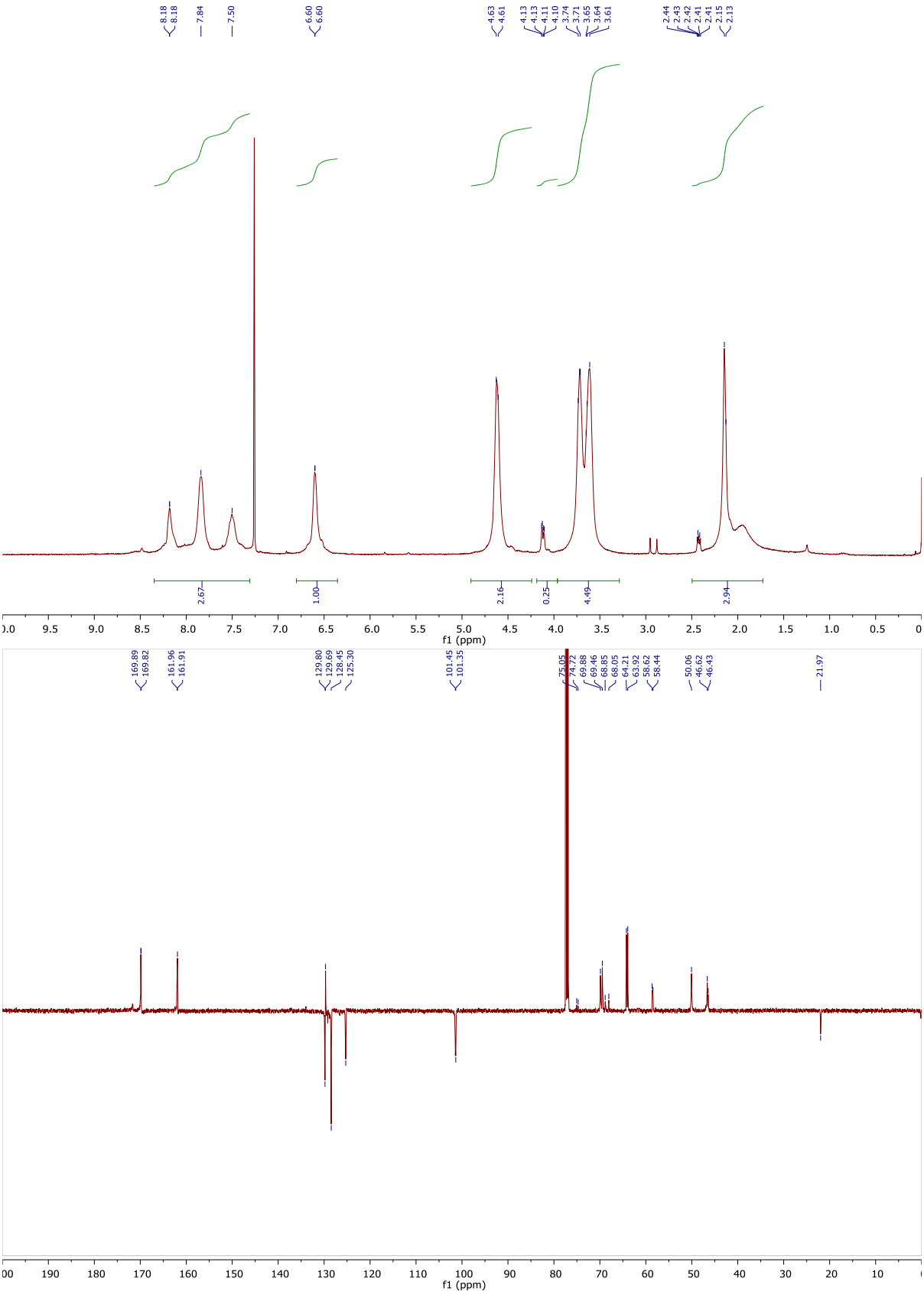
2.11 O[1]5c(S)-ABA stearic



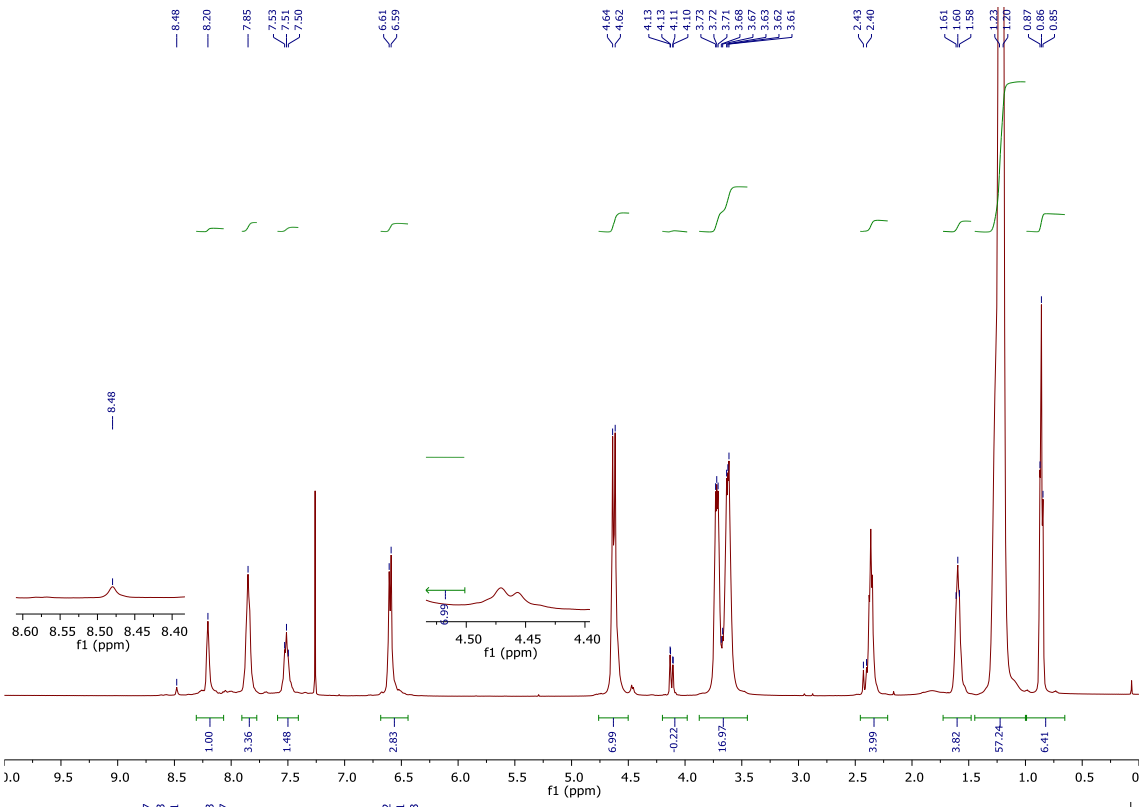
2.12 P(1a-co-3)

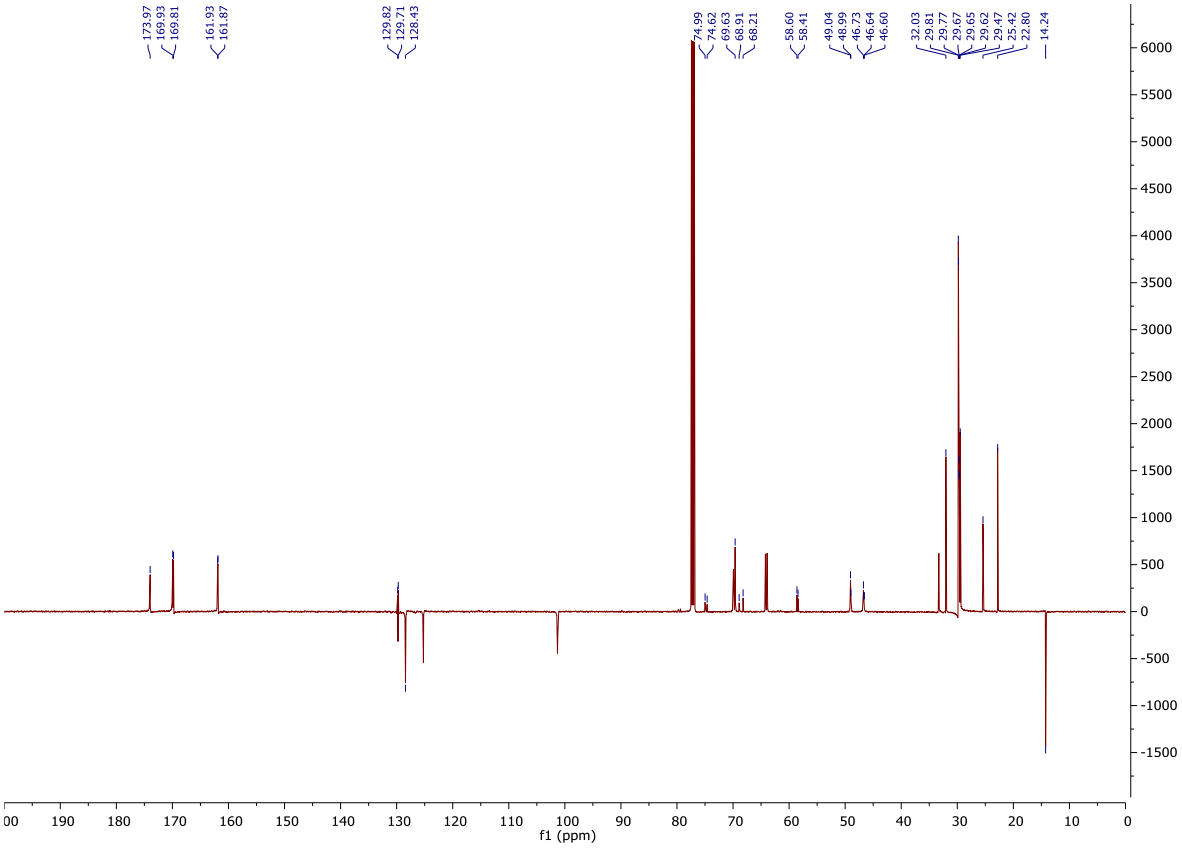

2.34
2.25
7.97
3.00
6.20
1.00
0.02
6.12
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

2.13 P(1a-co-5a(Me))

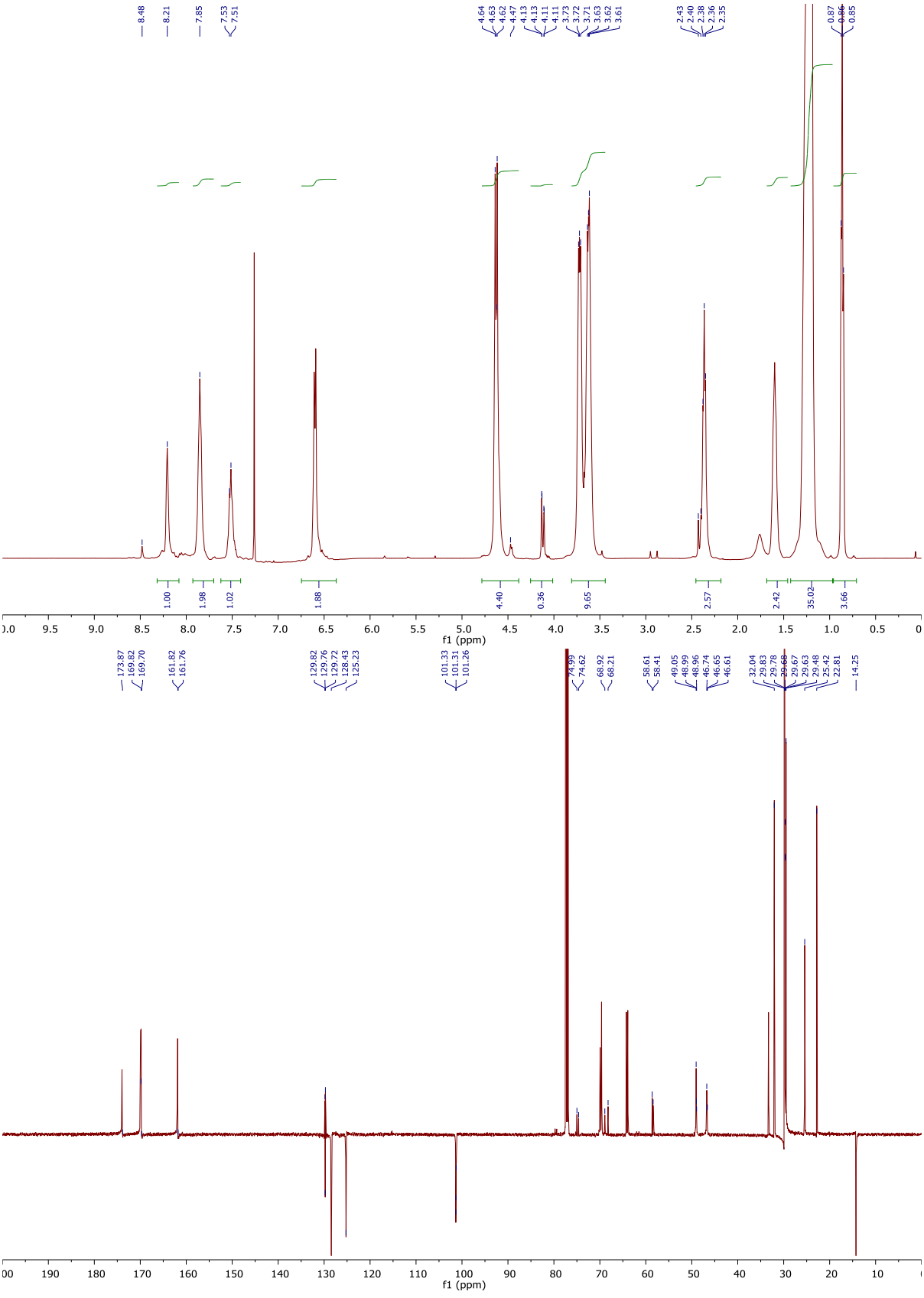


2.14 P(1a-co-5b(P)) Palimitic

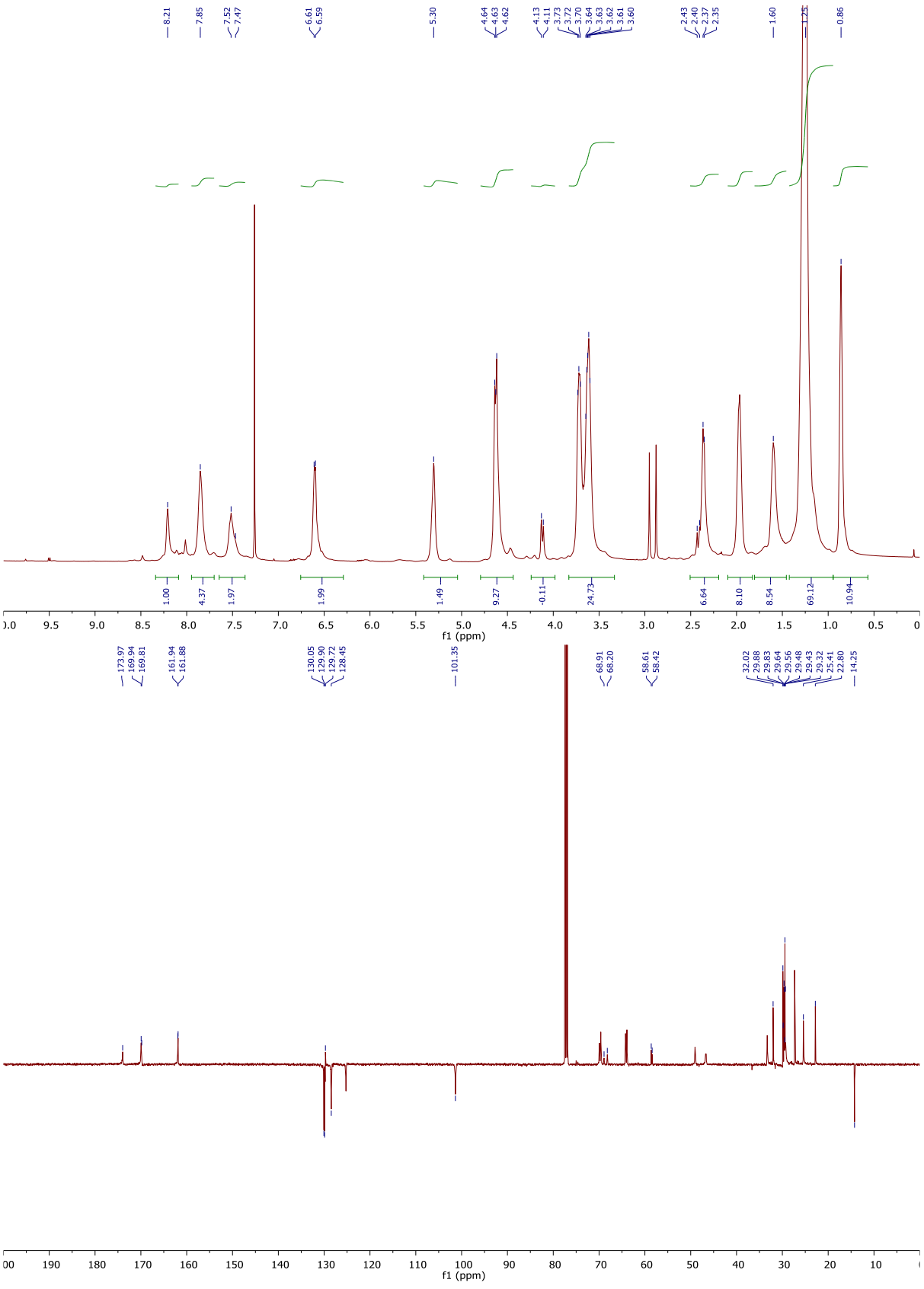


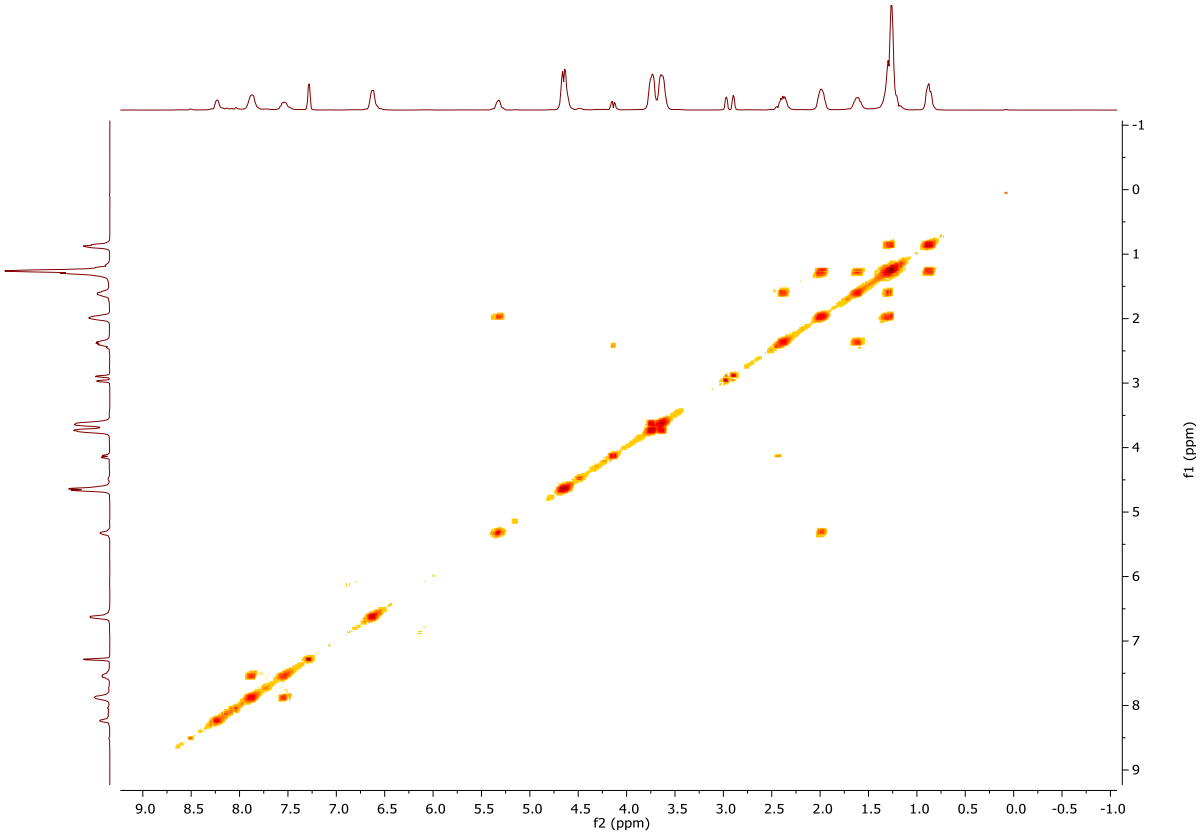


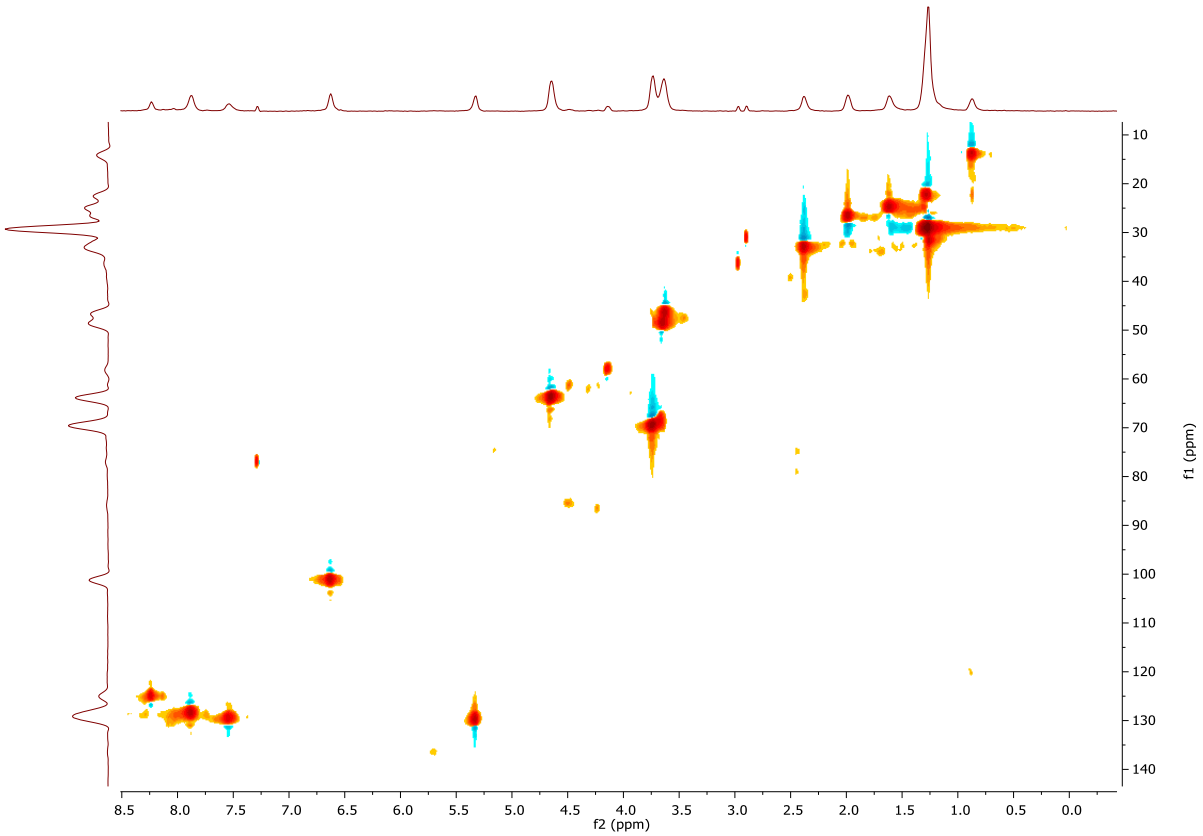
2.15 P(1a-co-5c(S)) Stearic

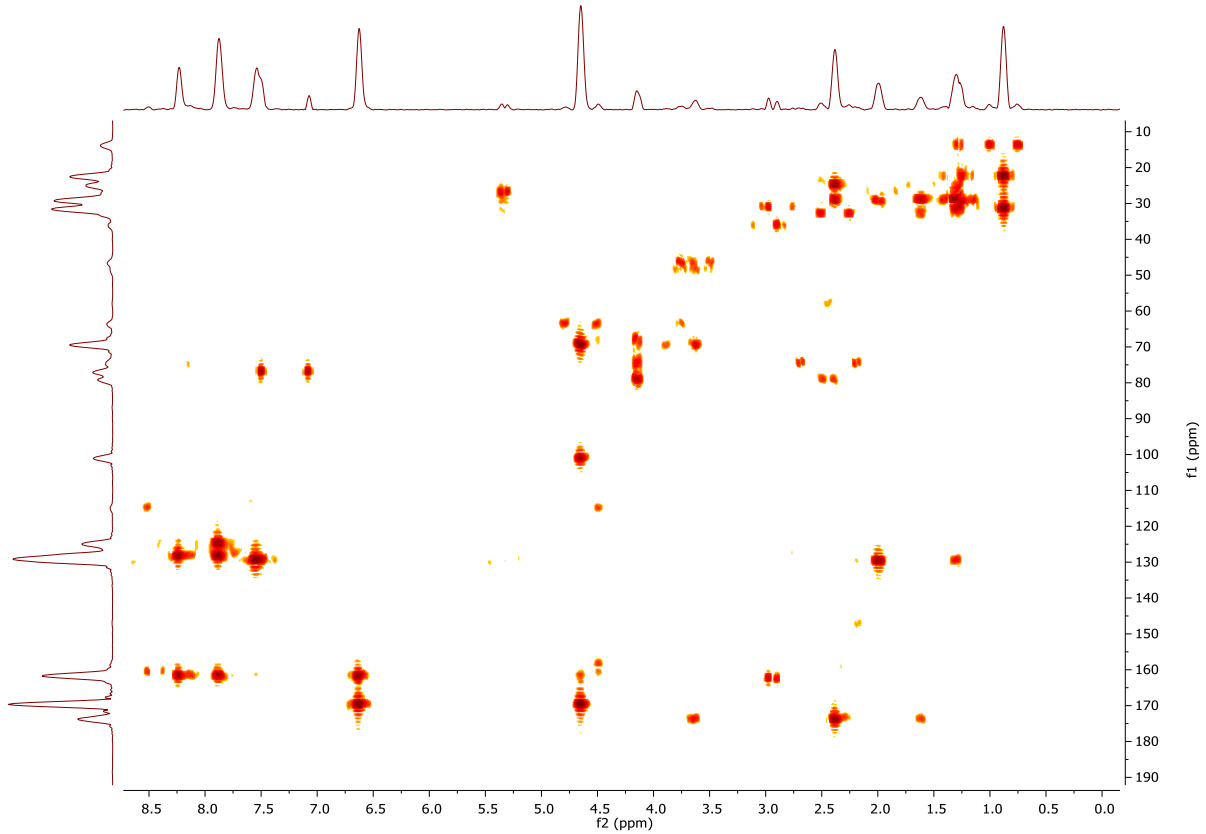


2.16 P(1a-co-5d(O)) Oleic

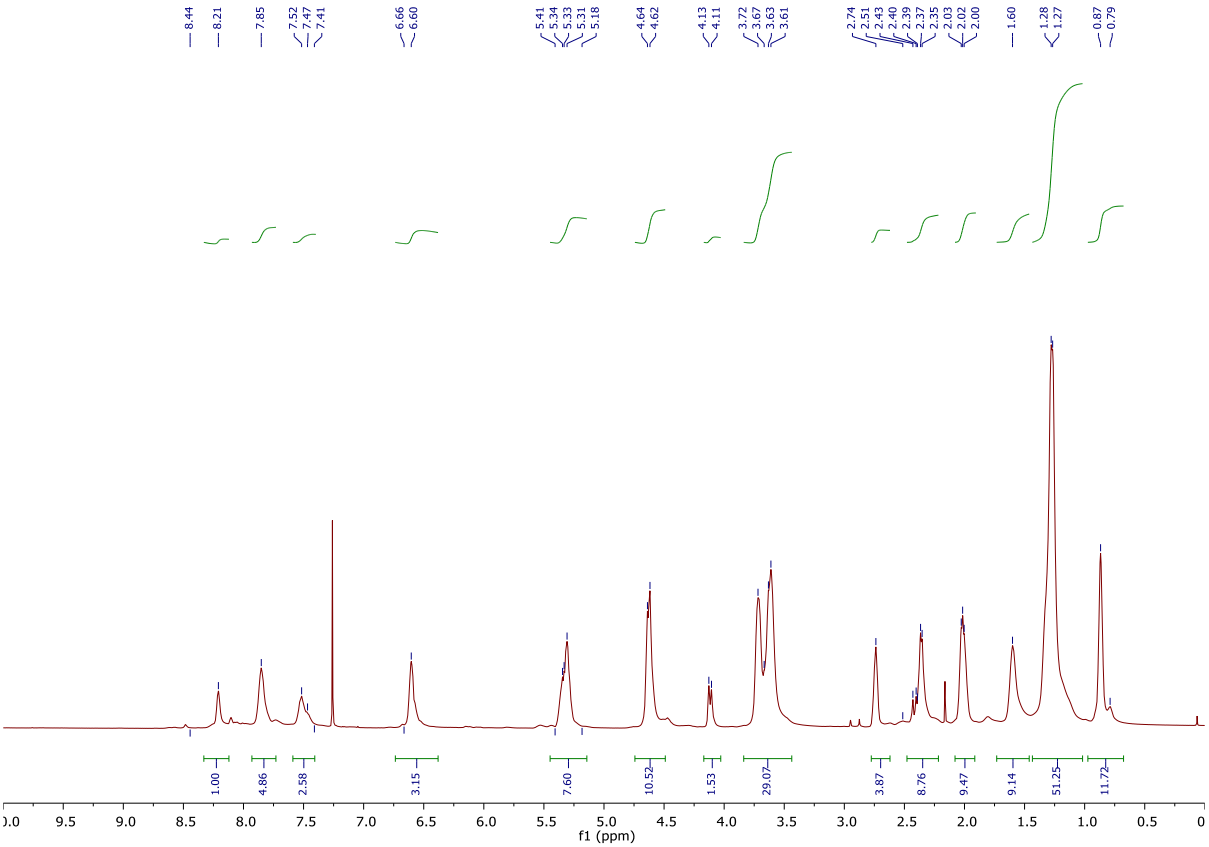


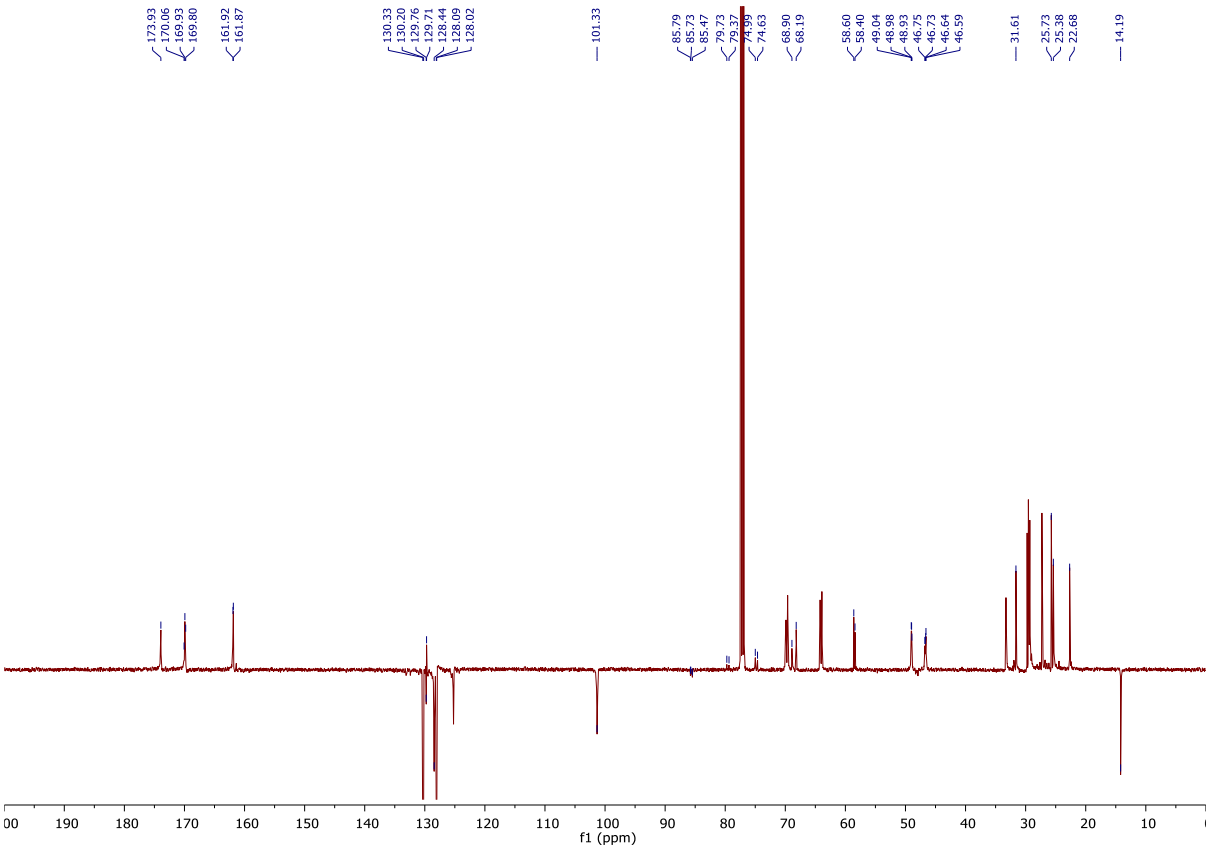

f2 (ppm)
f1 (ppm)

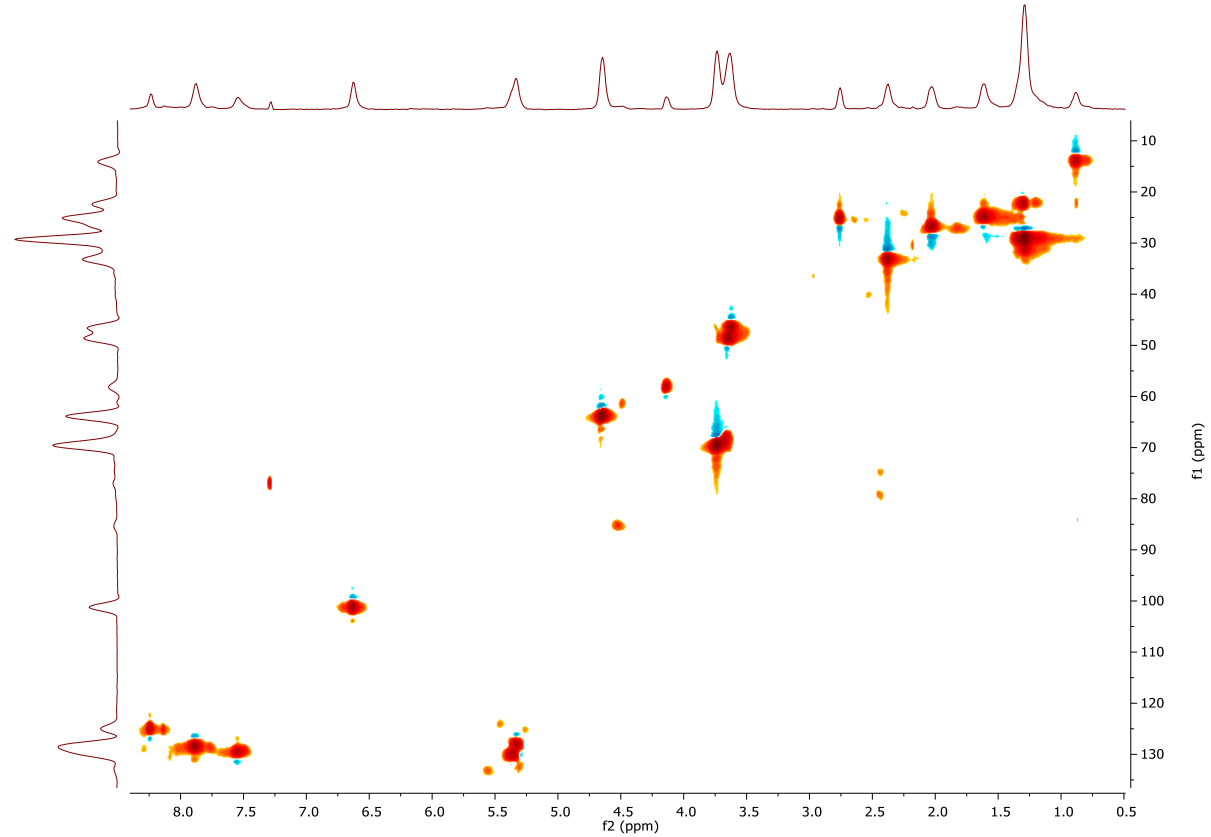

f2 (ppm)
f1 (ppm)

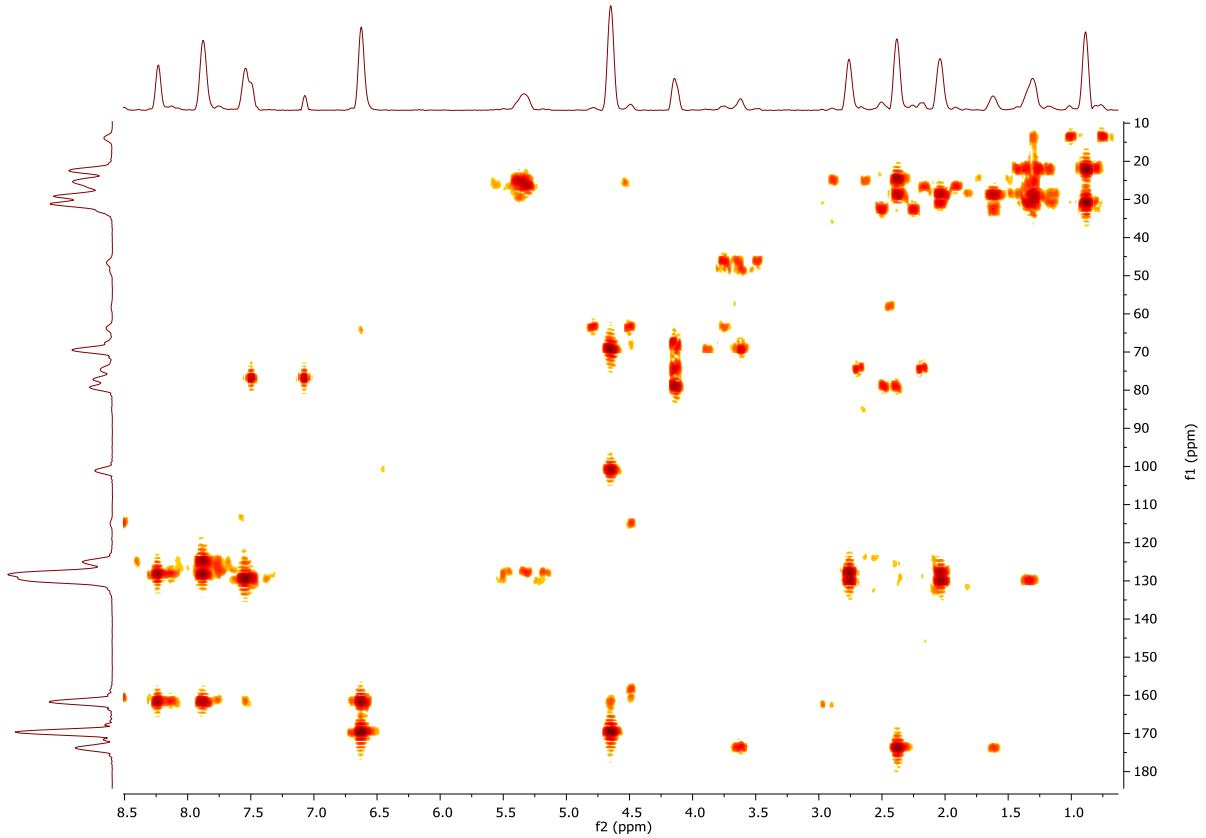


2.17 P(1a-co-5e(L)) Linoleic

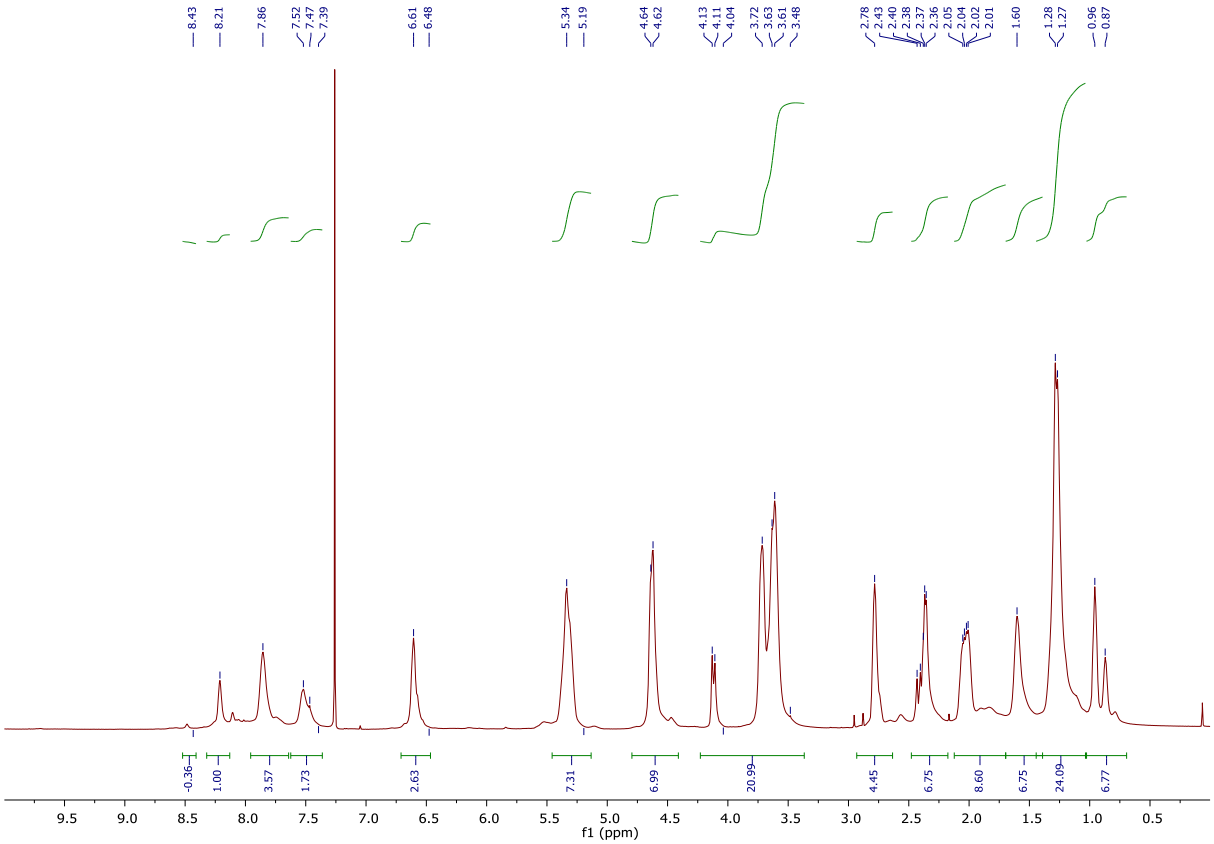


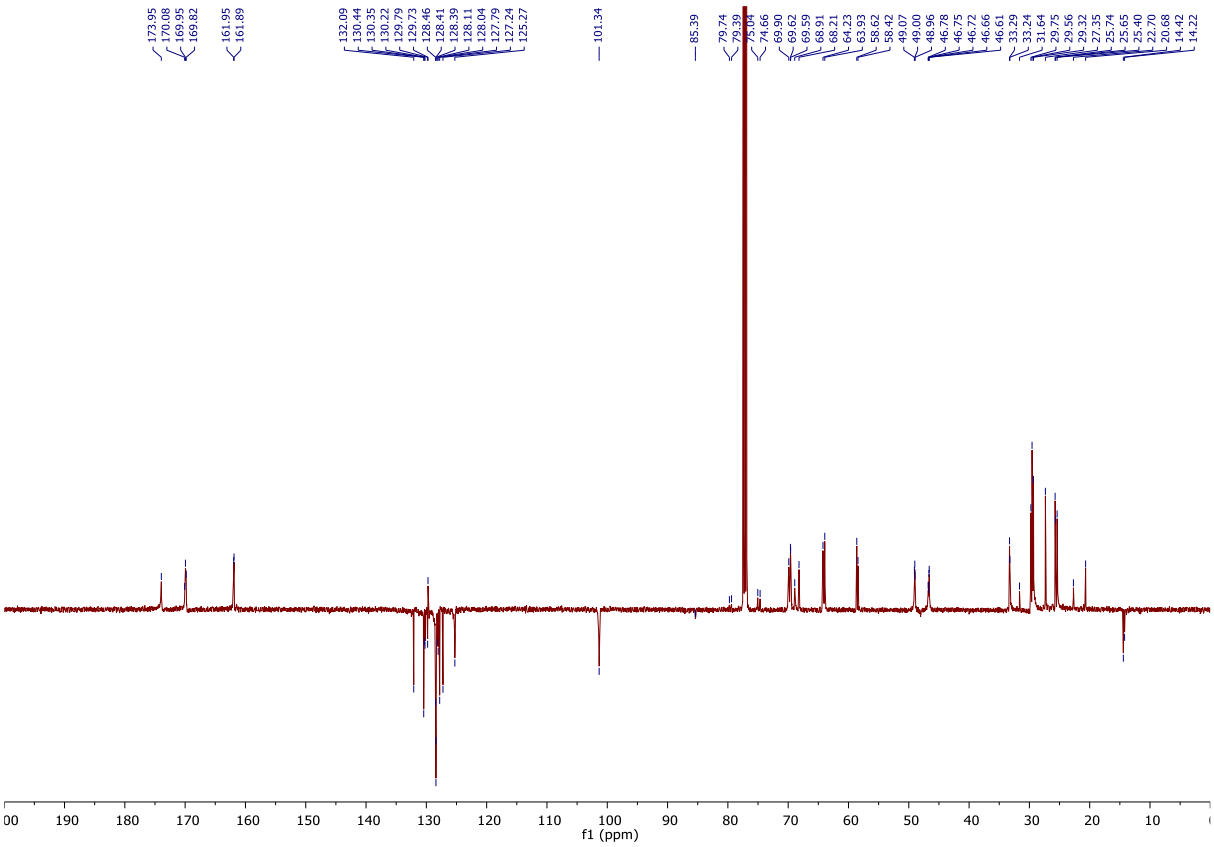

173.93
170.06
169.93
169.80
161.92
161.87
130.33
130.20
129.76
129.71
128.44
128.09
128.02
101.33
85.79
85.73
85.47
79.73
79.37
74.99
74.63
68.90
68.19
58.60
58.40
49.04
48.98
48.93
46.75
46.73
46.64
46.59
31.61
25.73
25.38
22.68
14.19
f1 (ppm)

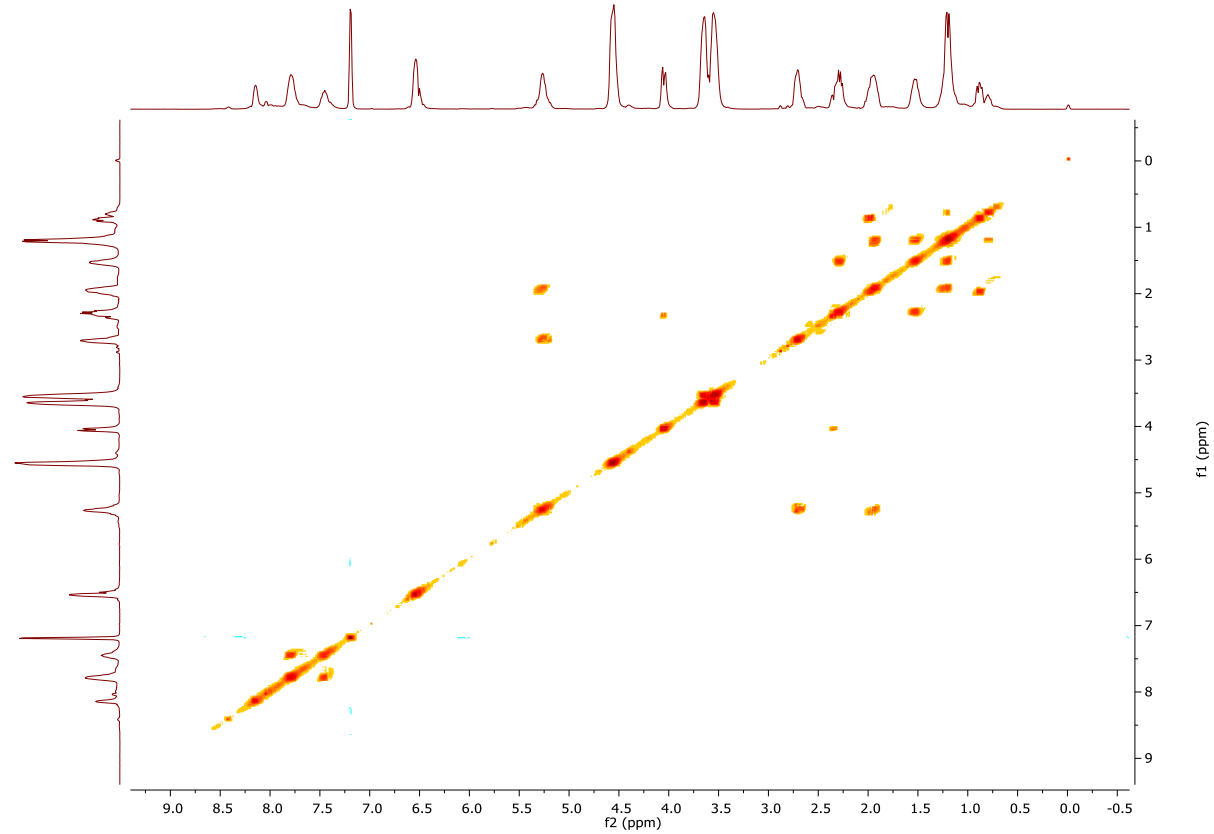

f2 (ppm)
f1 (ppm)

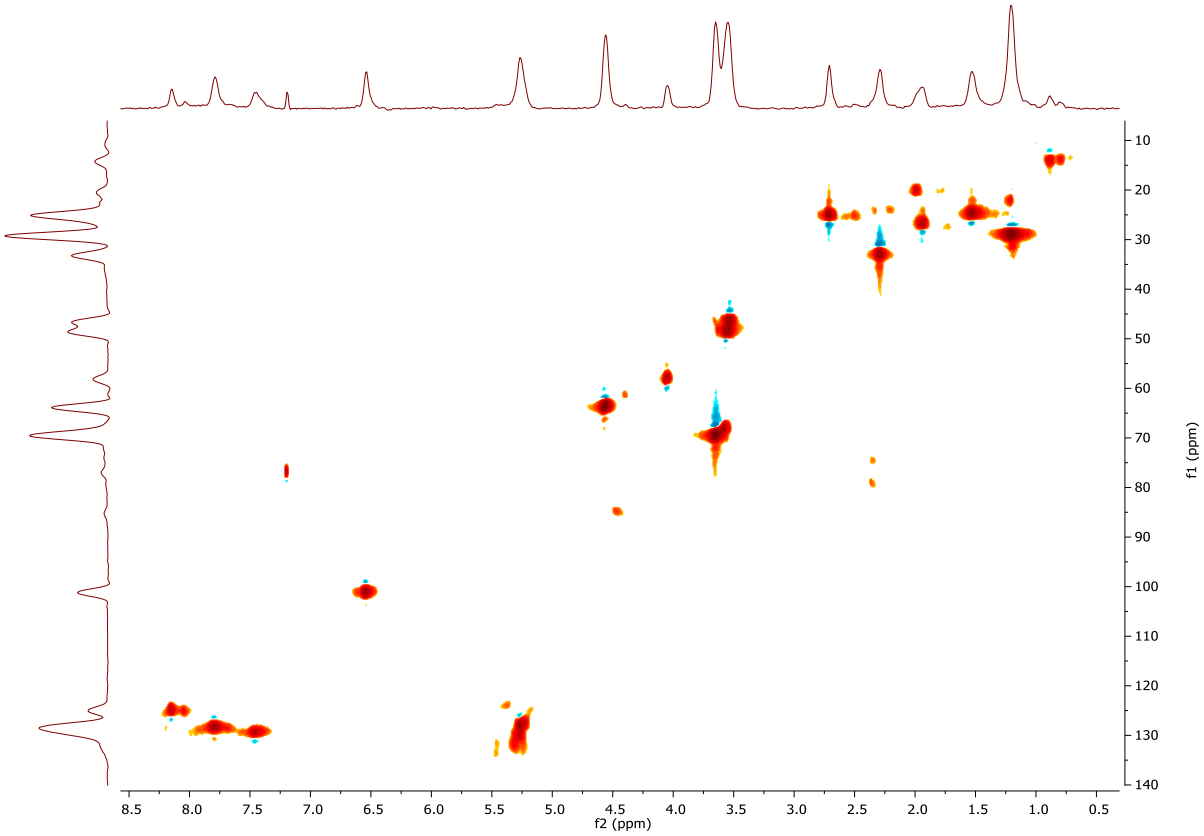


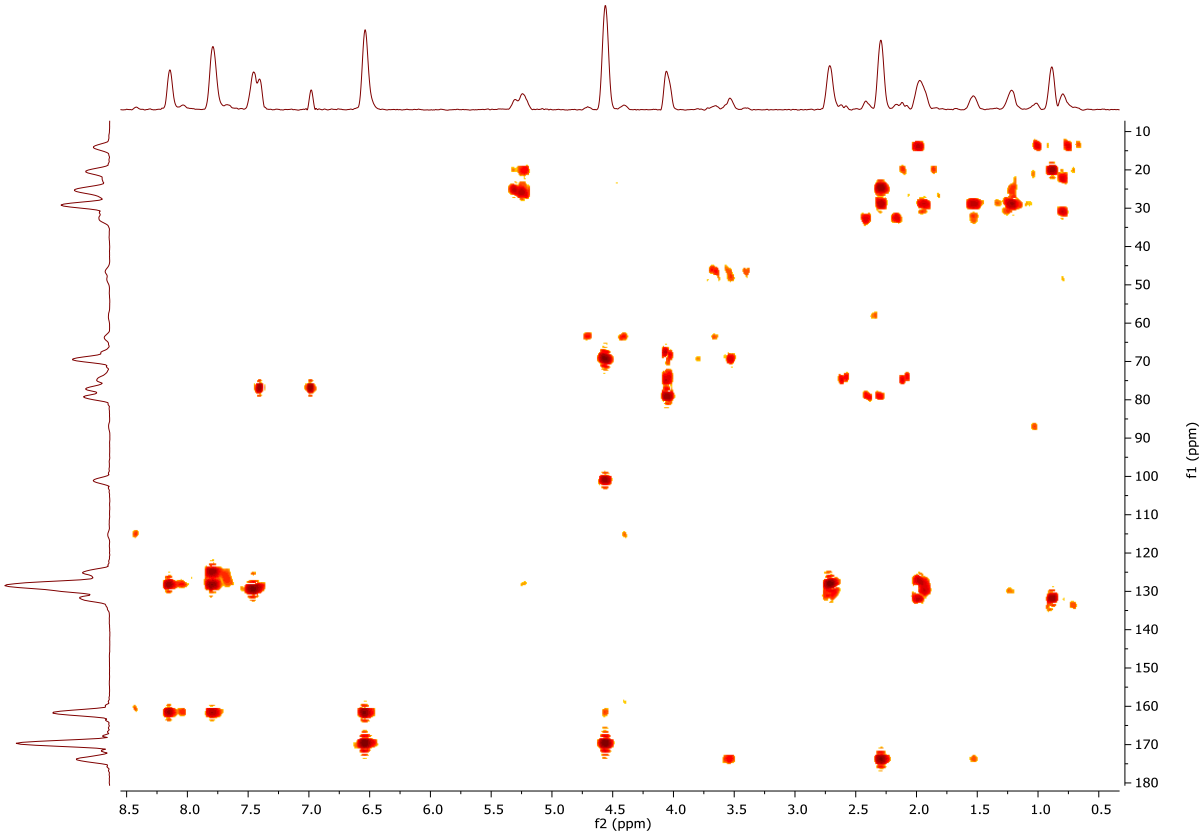
2.18 P(1a-co-5f(Ln)) Linolenic



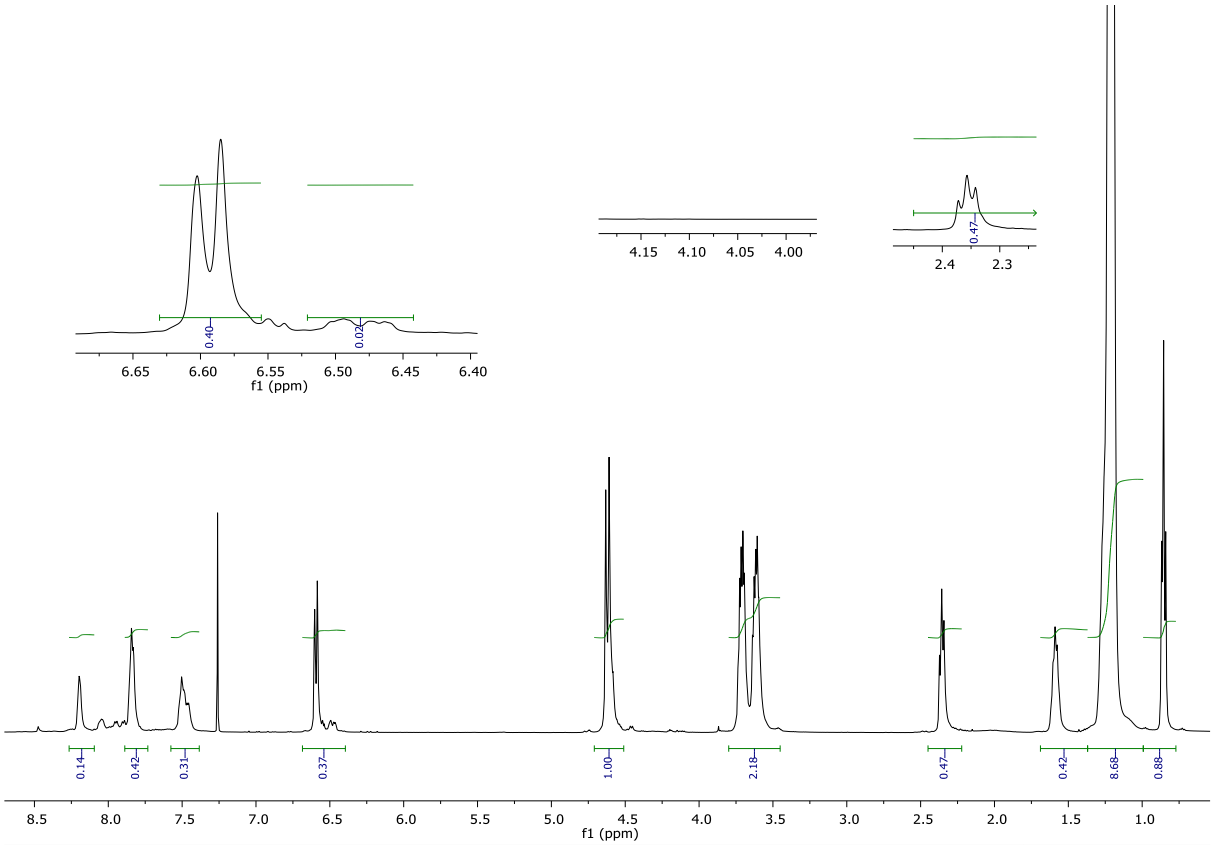

173.95
170.08
169.95
169.82
161.95
161.89
132.09
130.44
130.35
130.22
129.79
129.73
128.46
128.41
128.39
128.11
128.04
127.79
127.24
125.27
101.34
85.39
79.74
79.39
75.04
74.66
69.90
69.62
69.59
68.91
68.21
64.23
63.93
58.62
58.42
49.07
49.00
48.96
46.78
46.75
46.72
46.66
46.61
33.29
33.24
31.64
29.75
29.56
29.32
27.35
25.74
25.65
25.40
22.70
20.68
14.42
14.22
f1 (ppm)

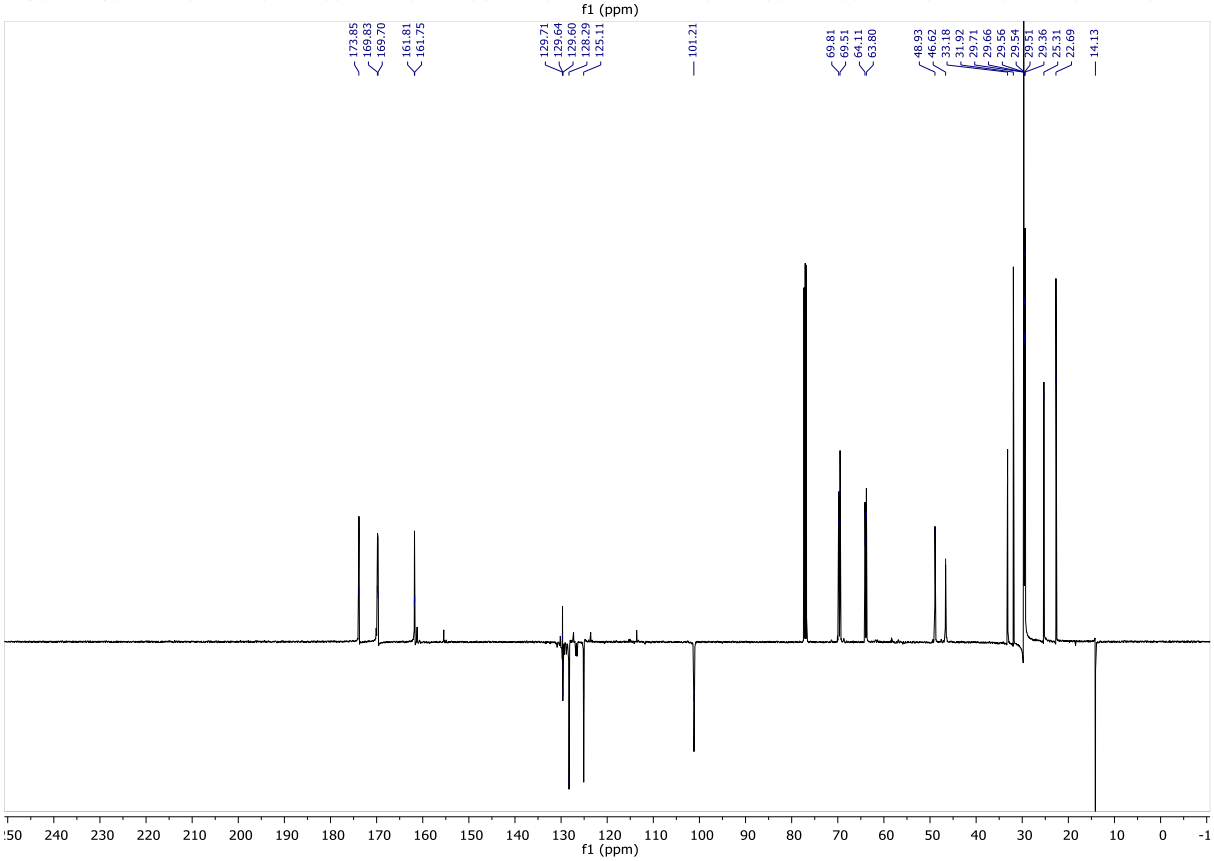

f2 (ppm)
f1 (ppm)

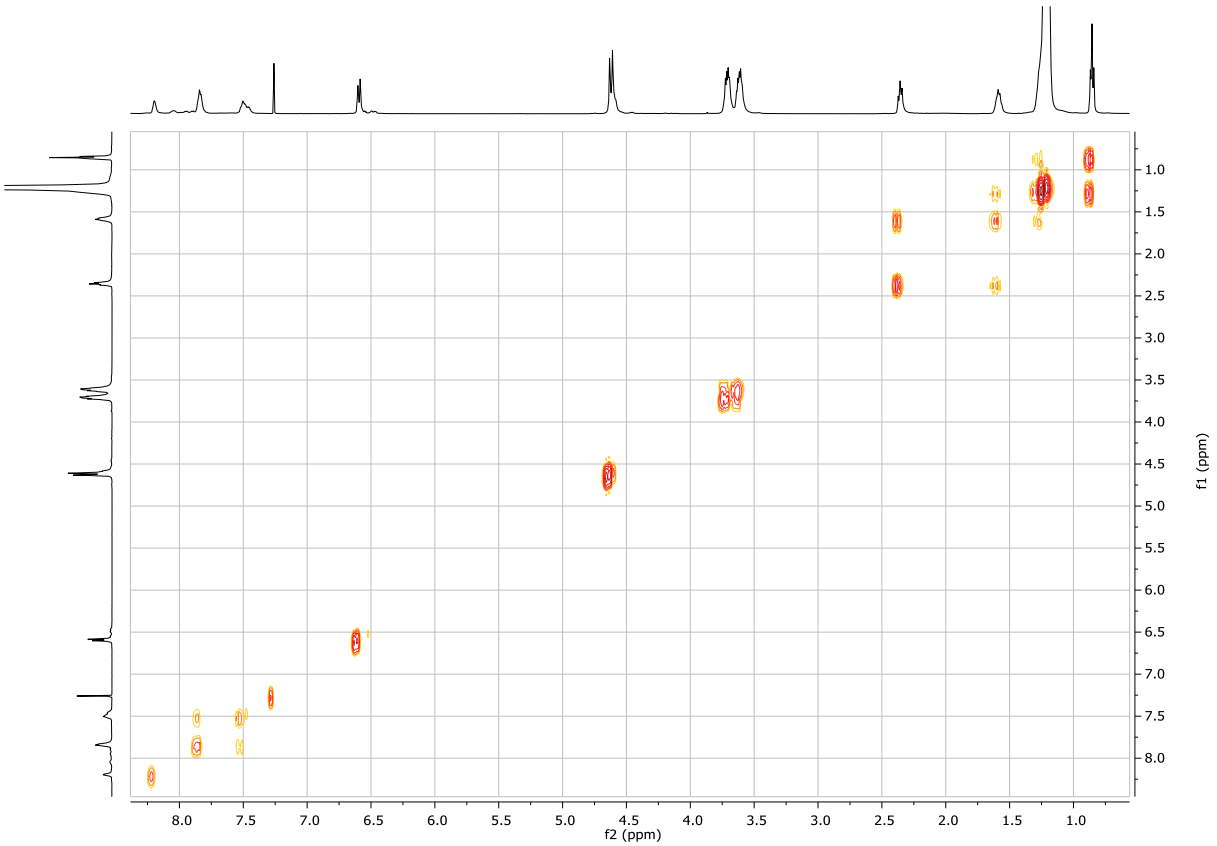

f2 (ppm)
f1 (ppm)

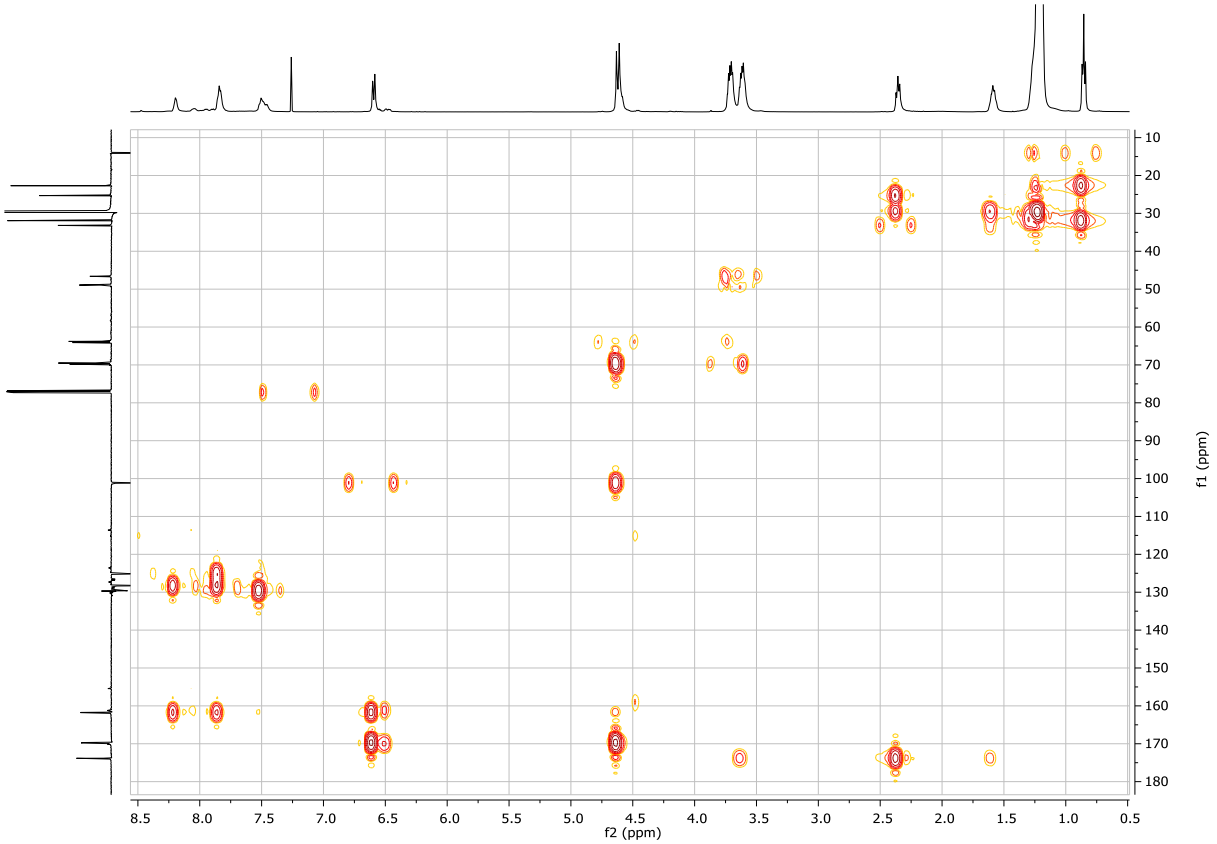

f2 (ppm)
f1 (ppm)

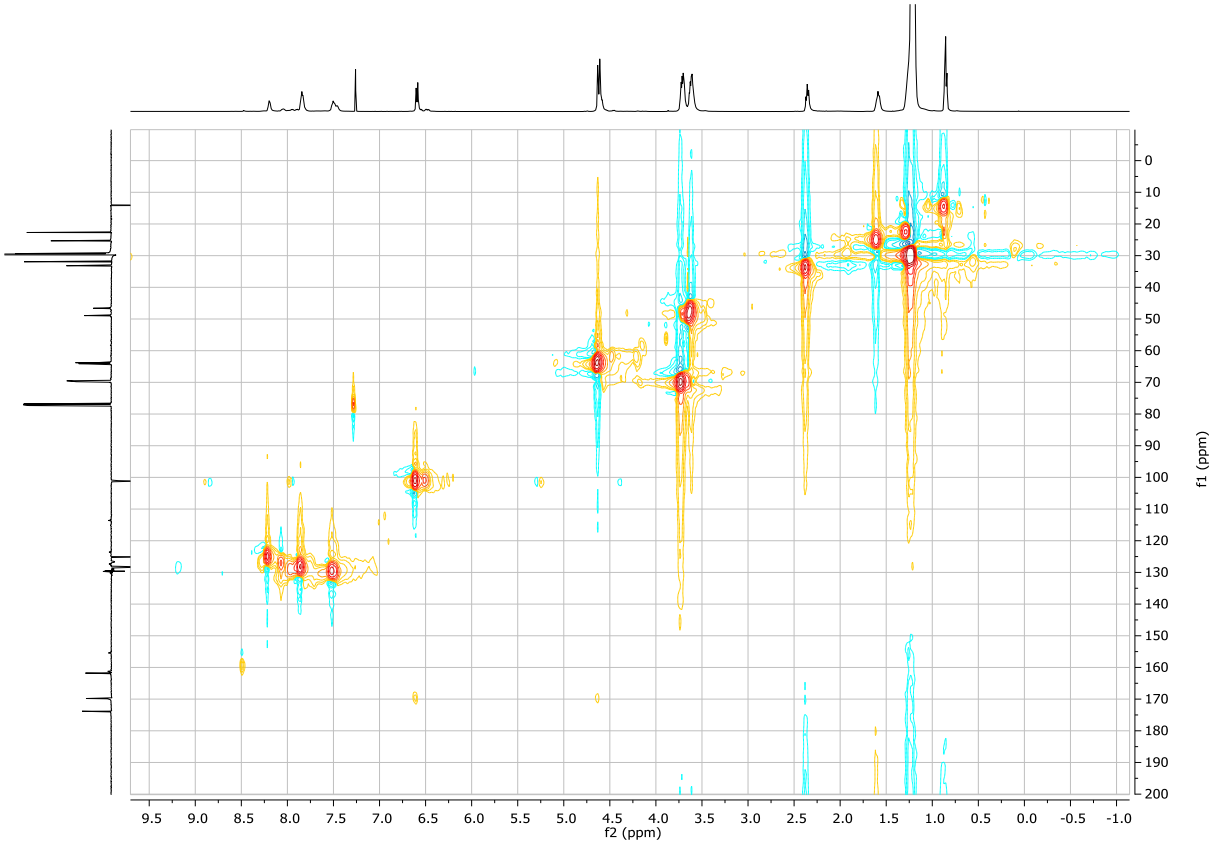
2.19 P_{base}(1a-co-5a(S)) Stearic



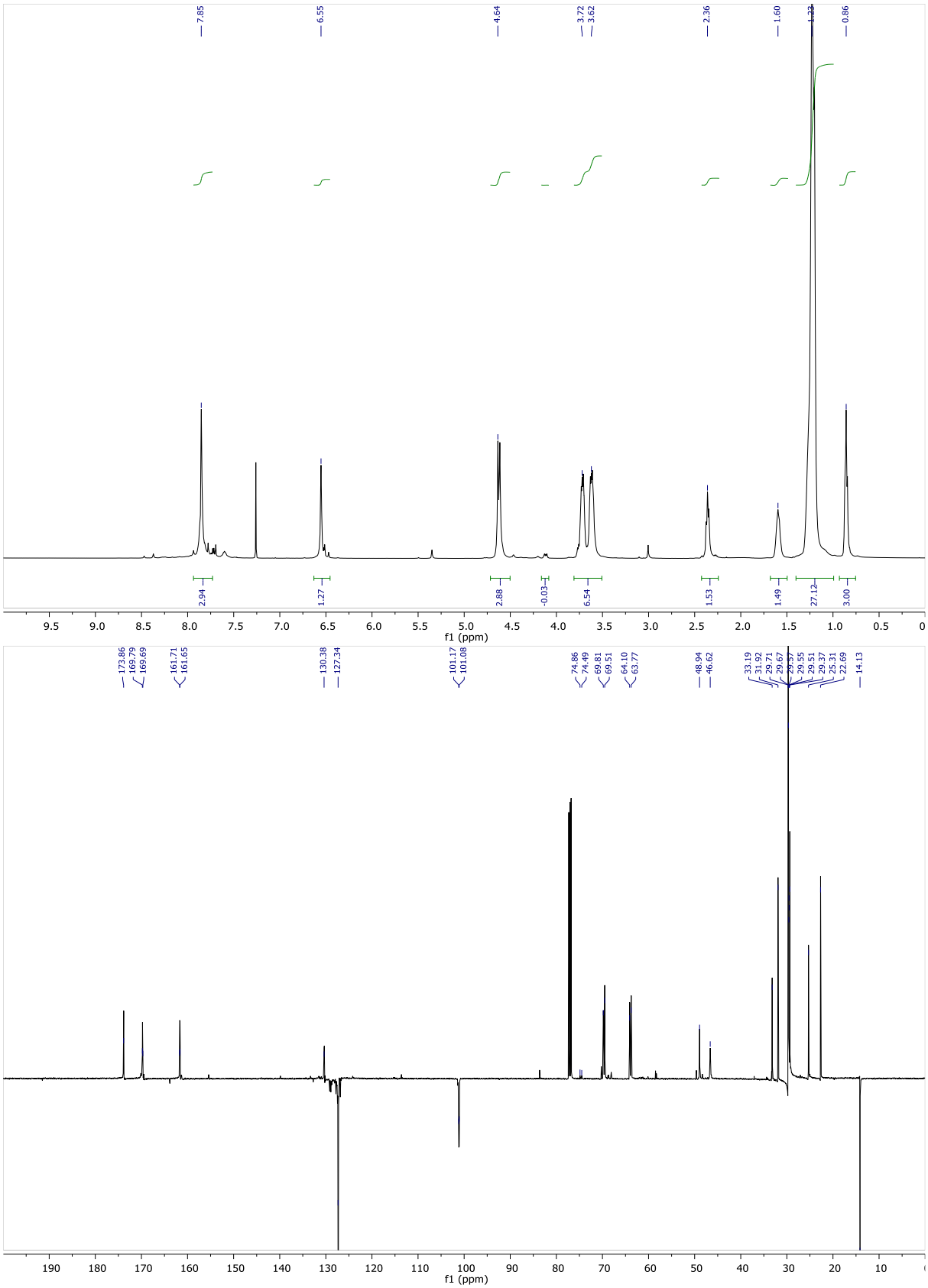


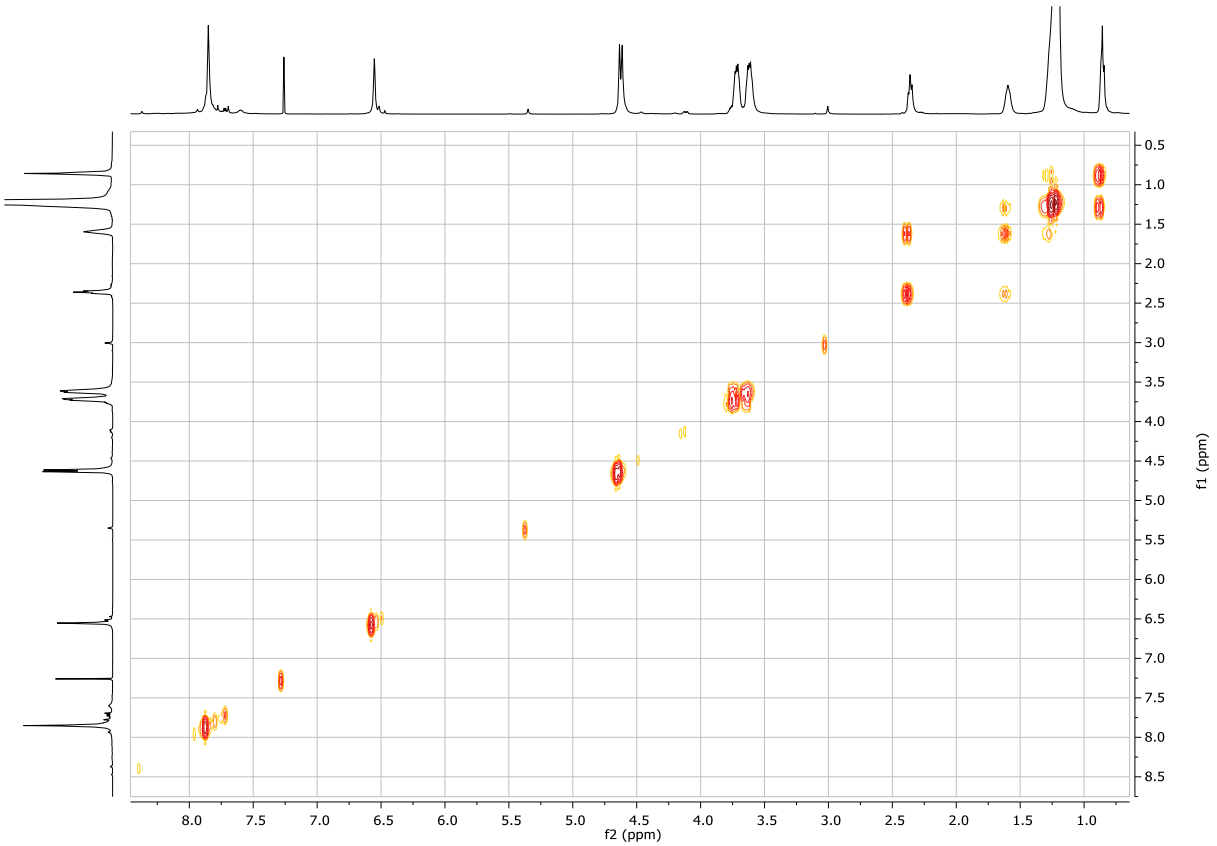

f2 (ppm)
f1 (ppm)

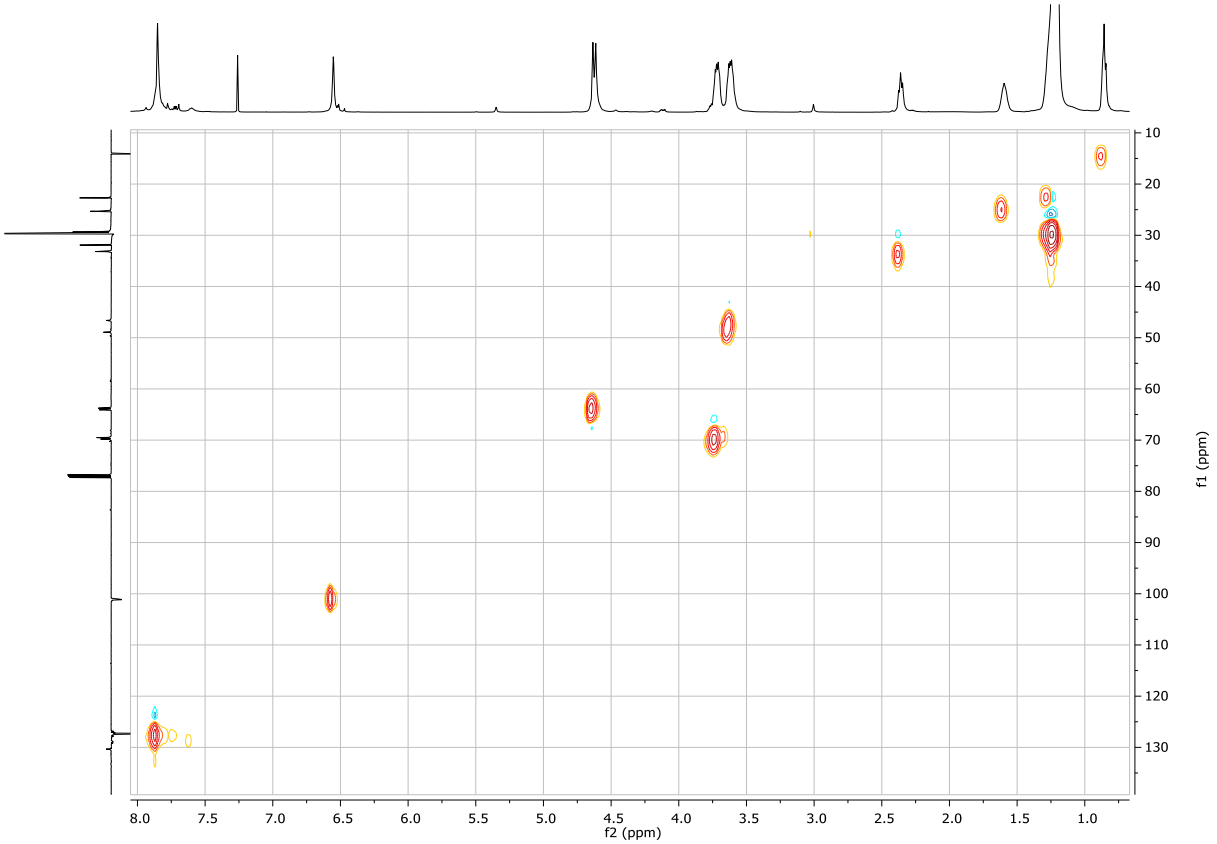

f2 (ppm)
f1 (ppm)

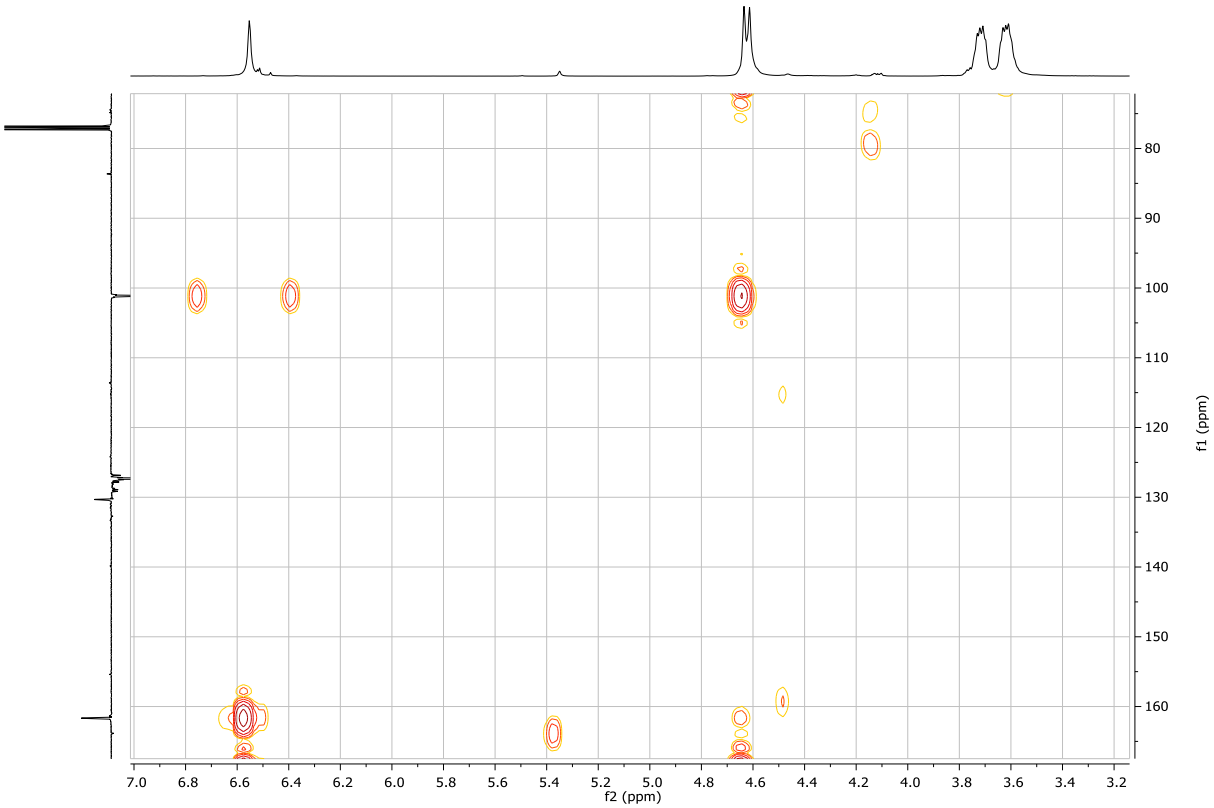

f2 (ppm)
f1 (ppm)

2.20 P_{base}(1b-co-5a(S)) Stearic

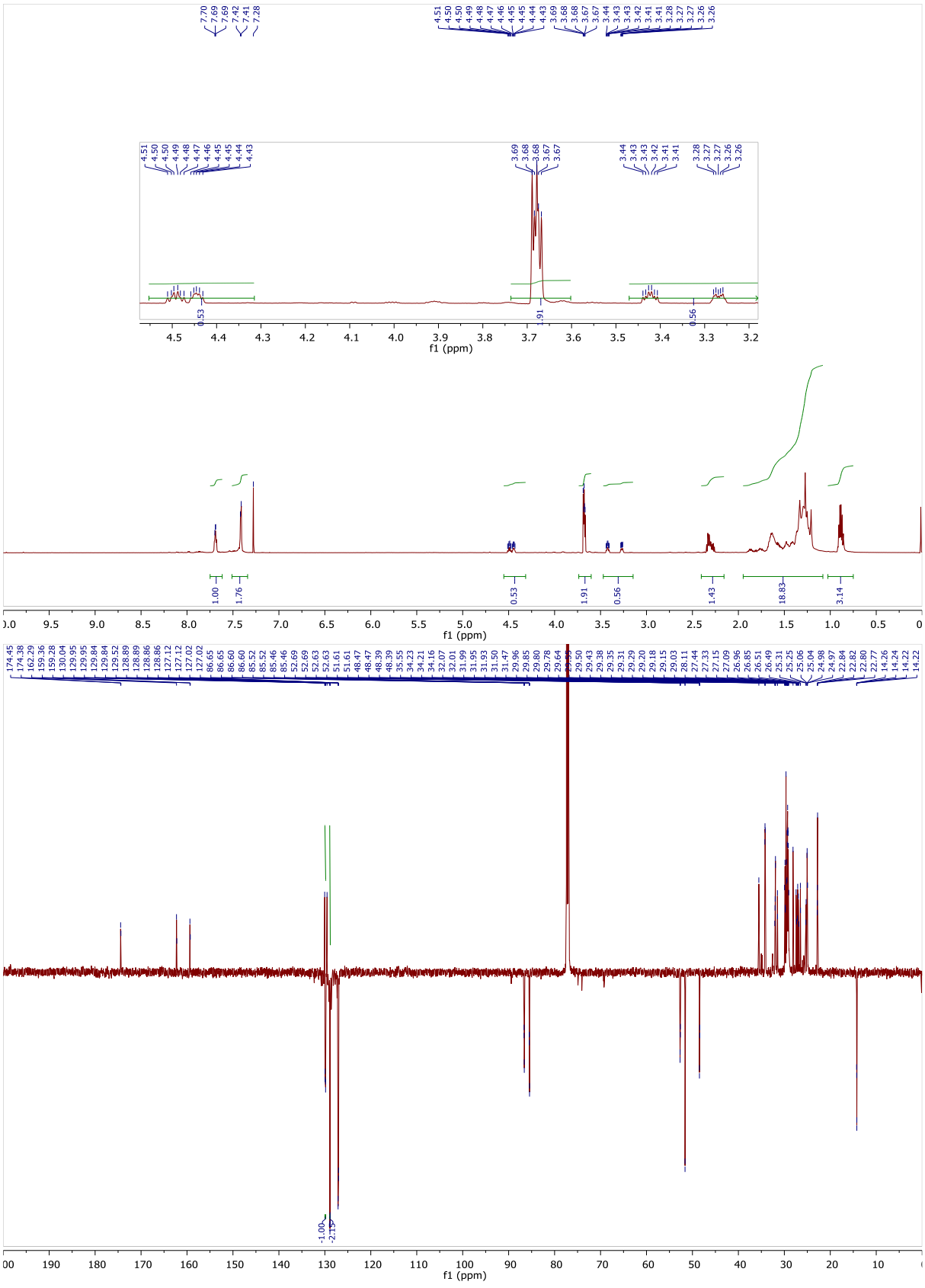


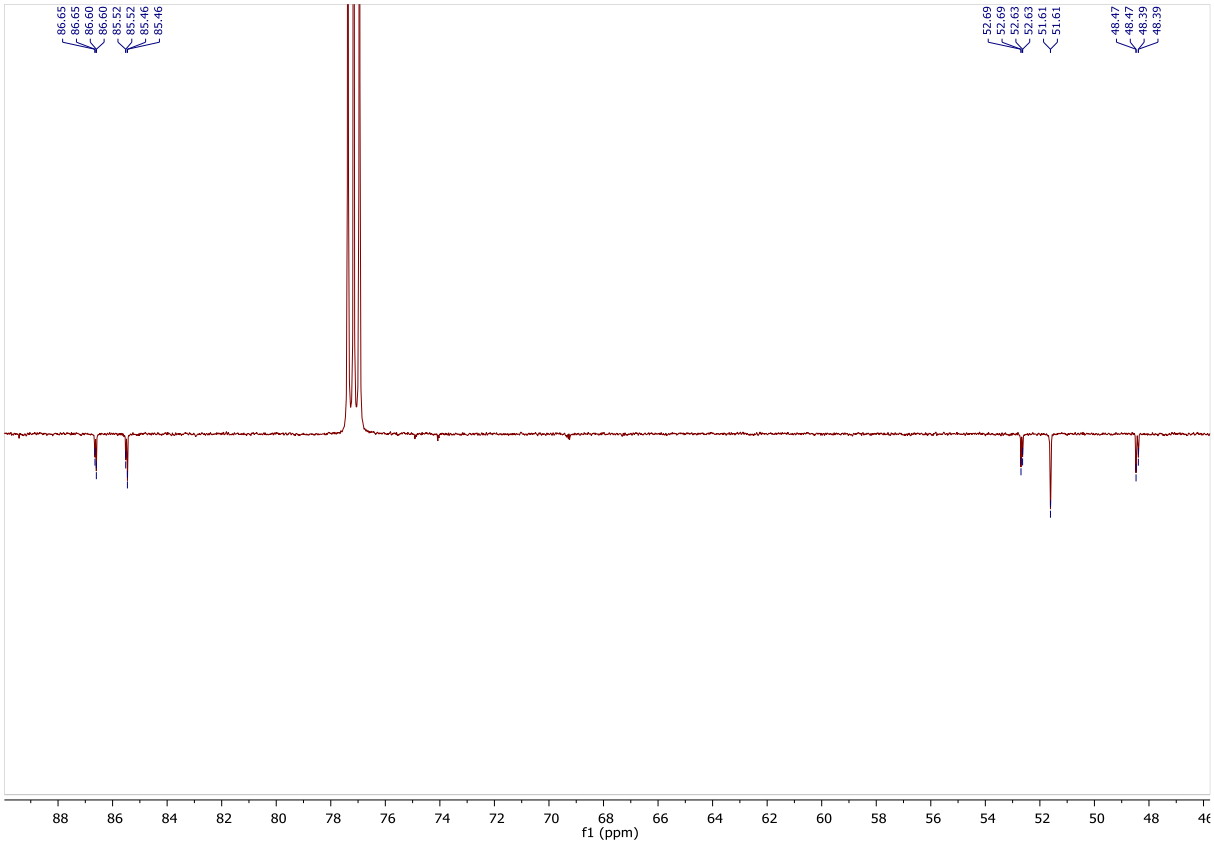

f2 (ppm)
f1 (ppm)

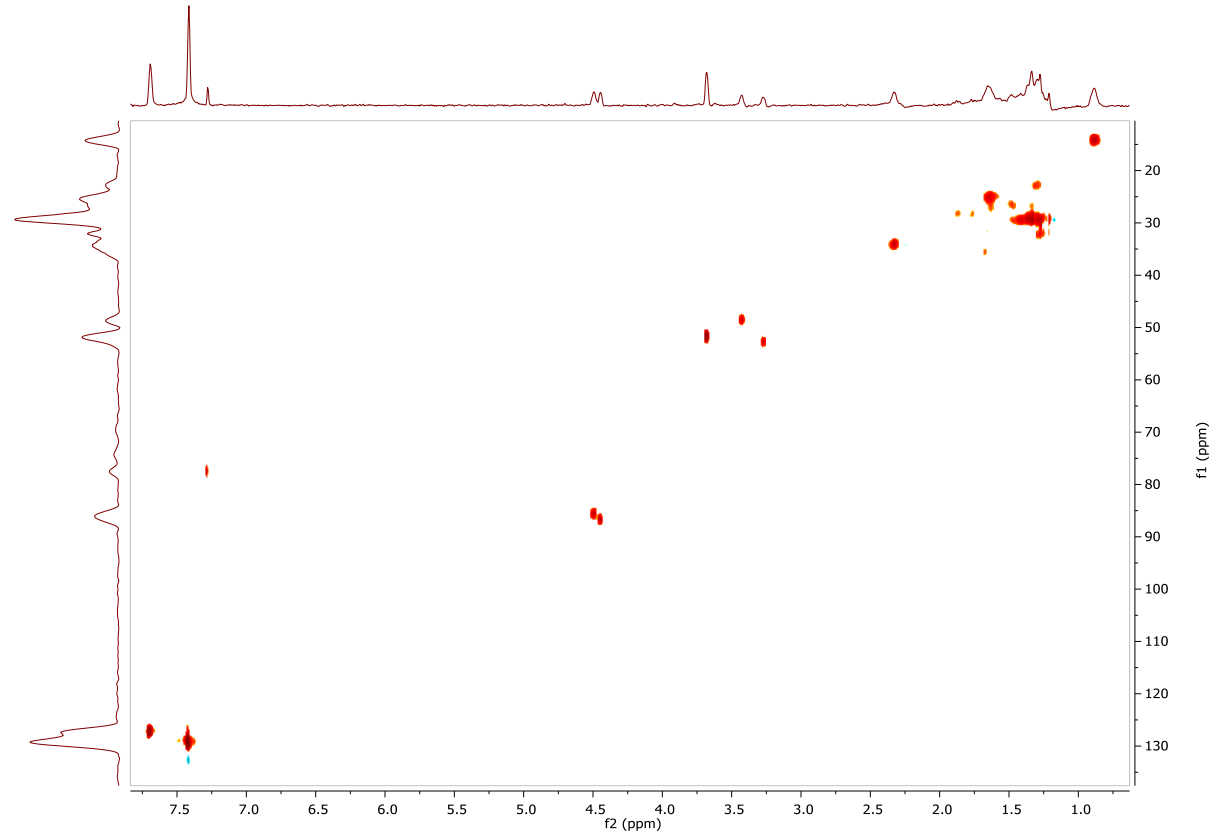

f2 (ppm)
f1 (ppm)

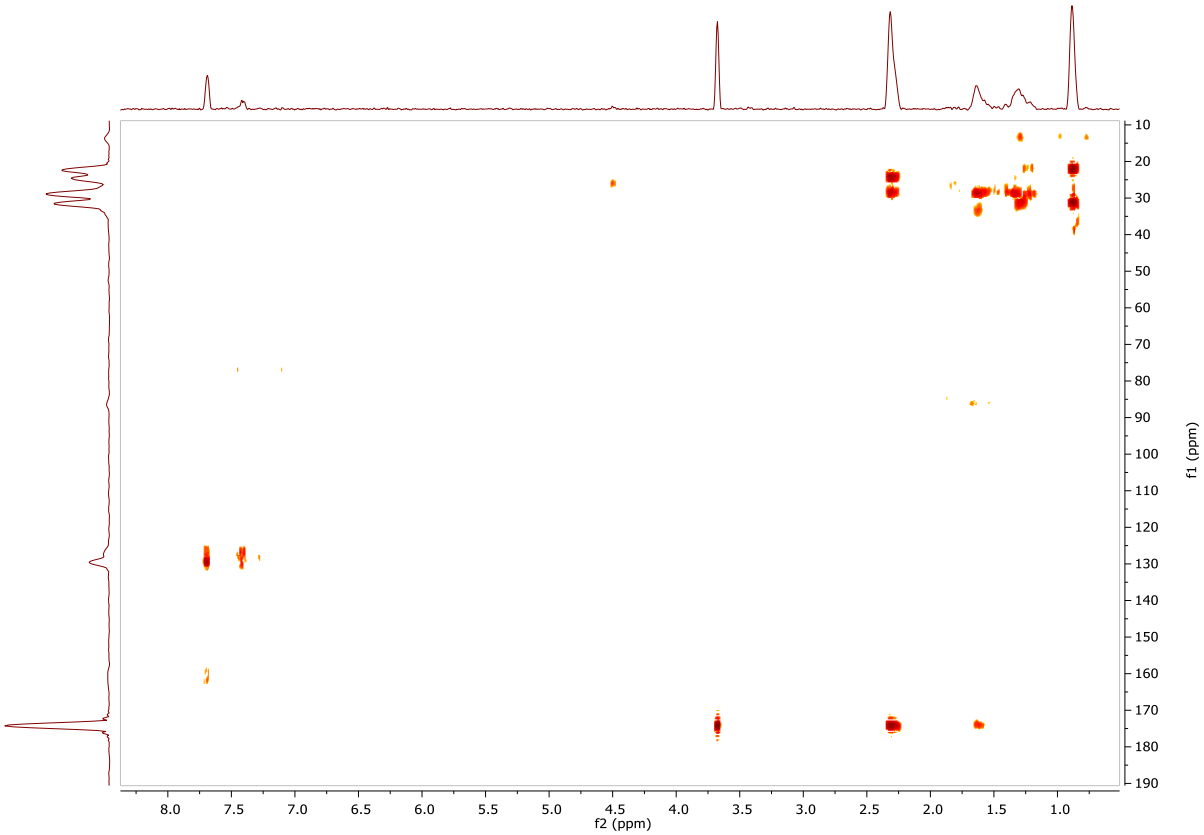

80
90
100
110
120
130
140
150
160
f1 (ppm)
7.0
6.8
6.6
6.4
6.2
6.0
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
f2 (ppm)

2.21 8a-d diastereomer and regioisomeric mixture

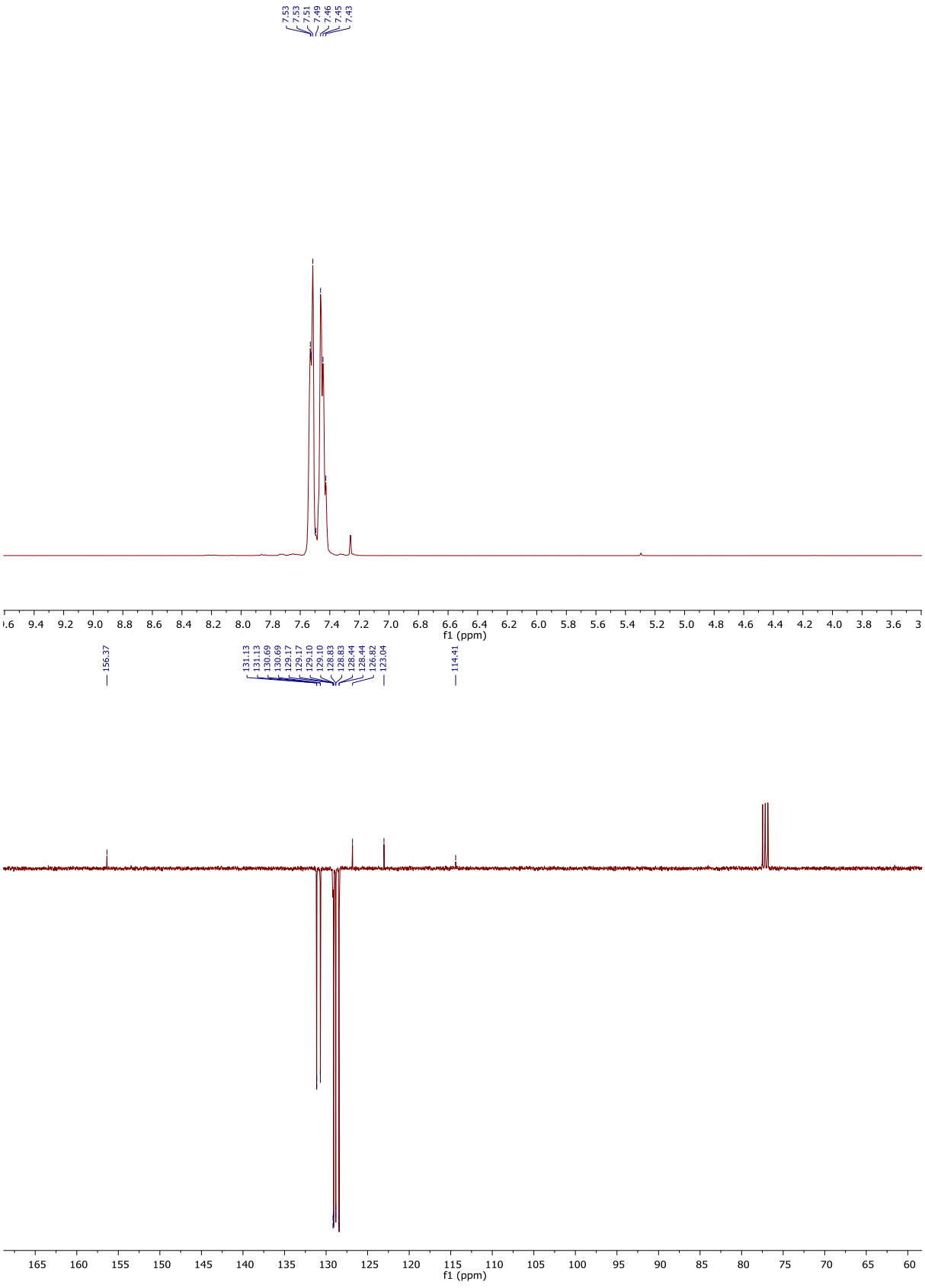



86.65
86.65
86.60
86.60
85.52
85.52
85.46
85.46
52.69
52.69
52.63
52.63
51.61
51.61
48.47
48.47
48.39
48.39
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
50
48
f1 (ppm)


20
30
40
50
60
70
80
90
100
110
120
130
f1 (ppm)
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
f2 (ppm)

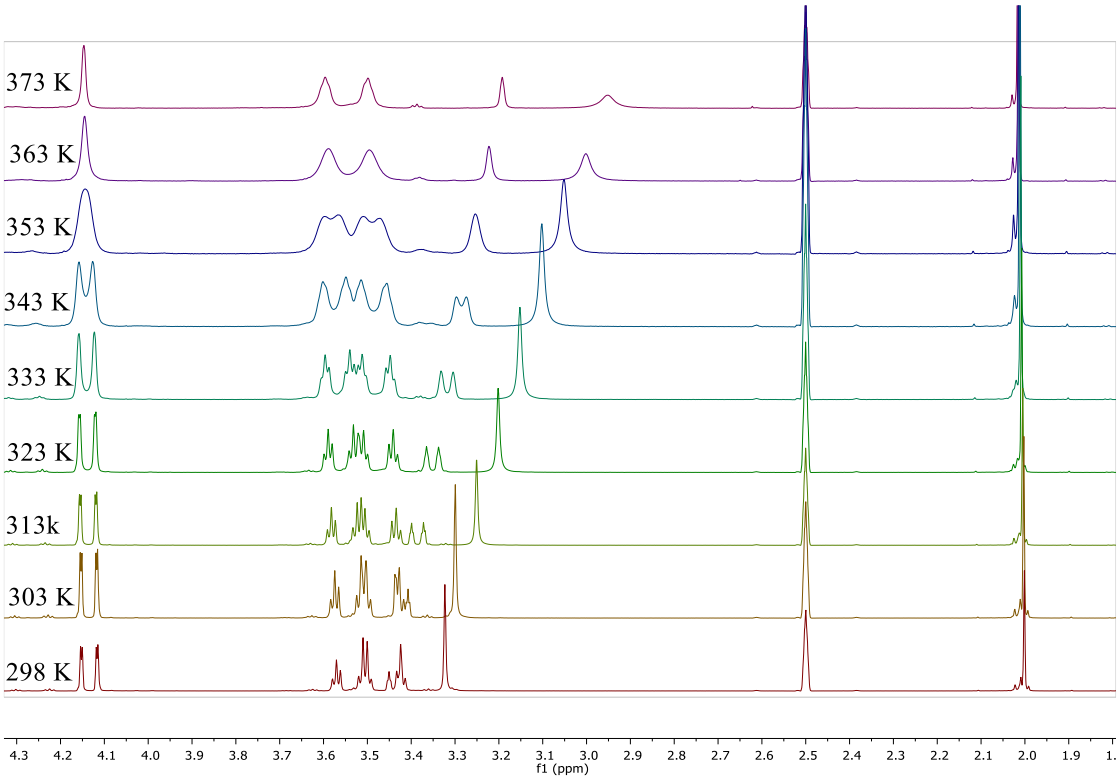

f2 (ppm)
f1 (ppm)

2.22 3,4-Diphenyl-1,2,5-oxadiazol-2-oxide 2

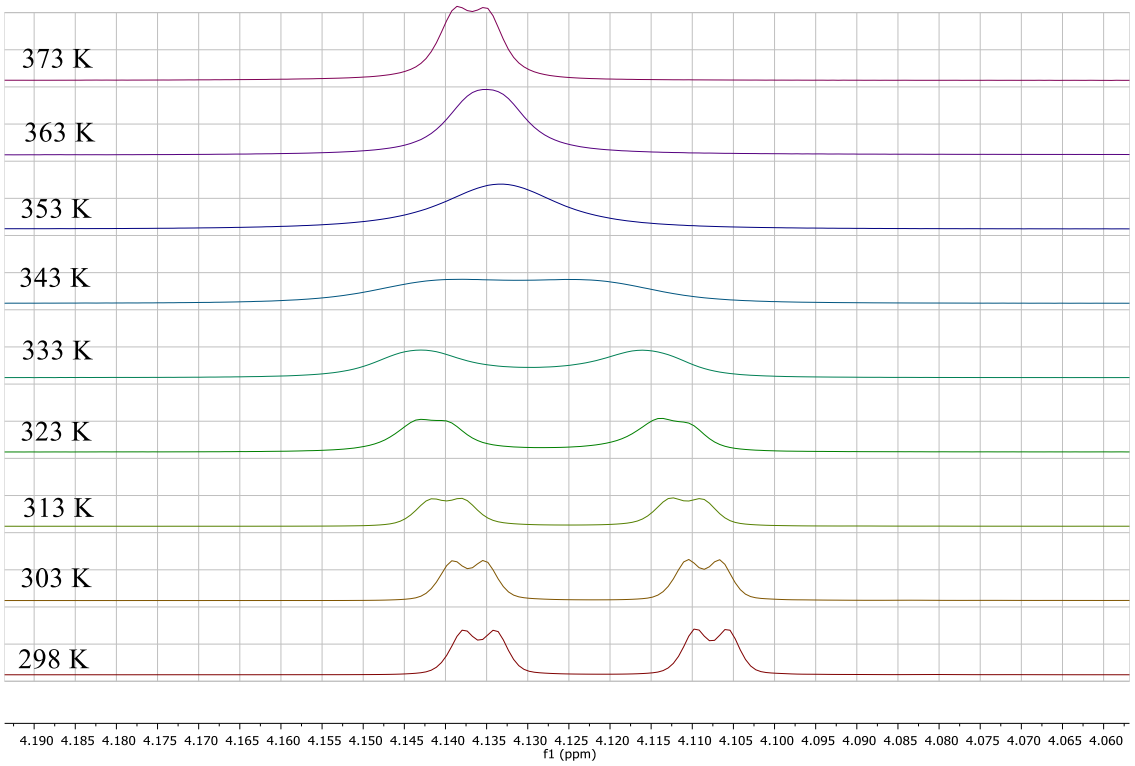


3.0 Variable Temperature NMR spectra

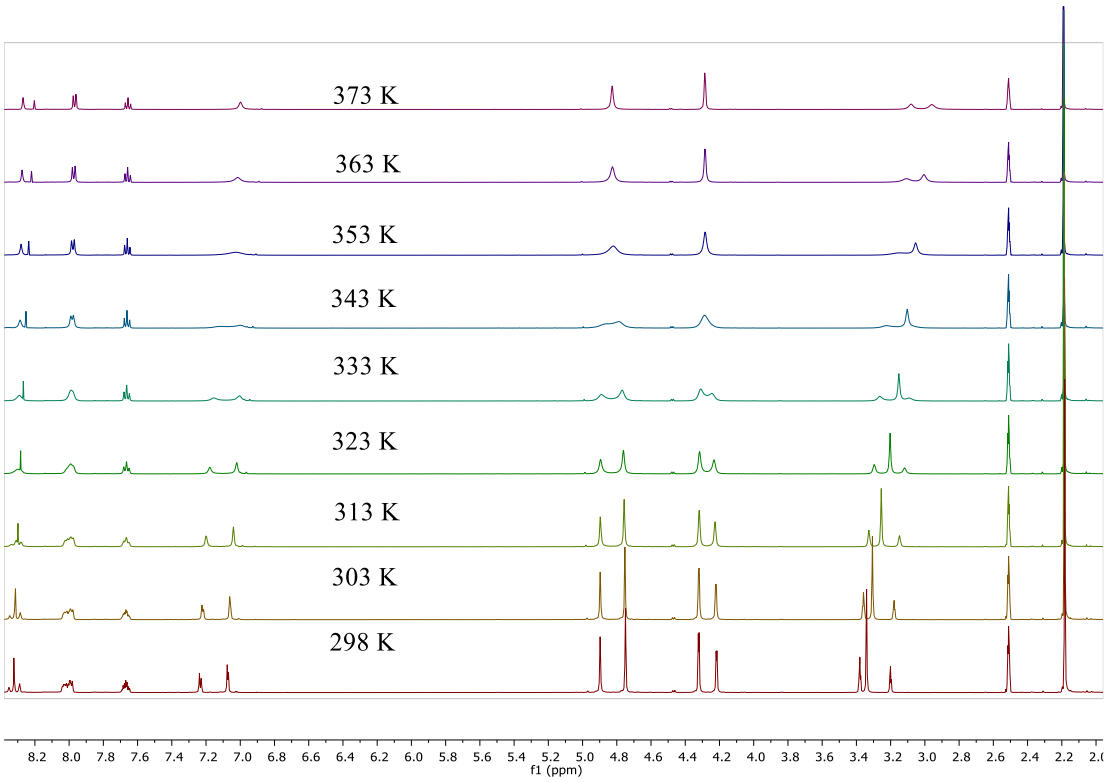
5a(Me)



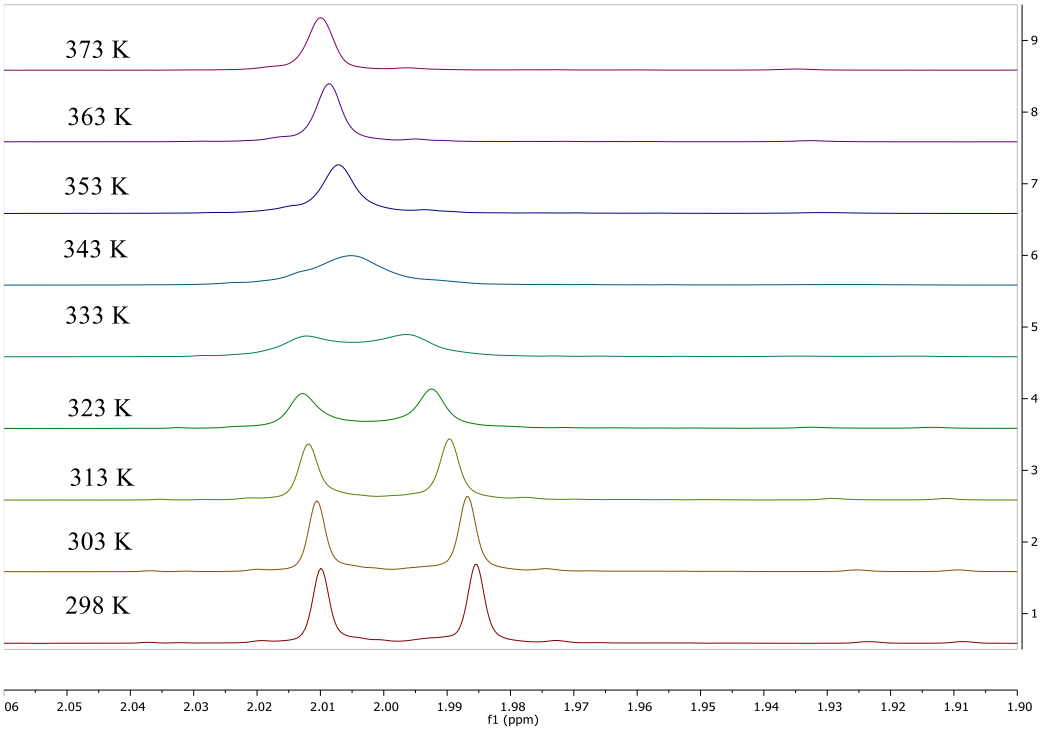
5c(O) Oleic

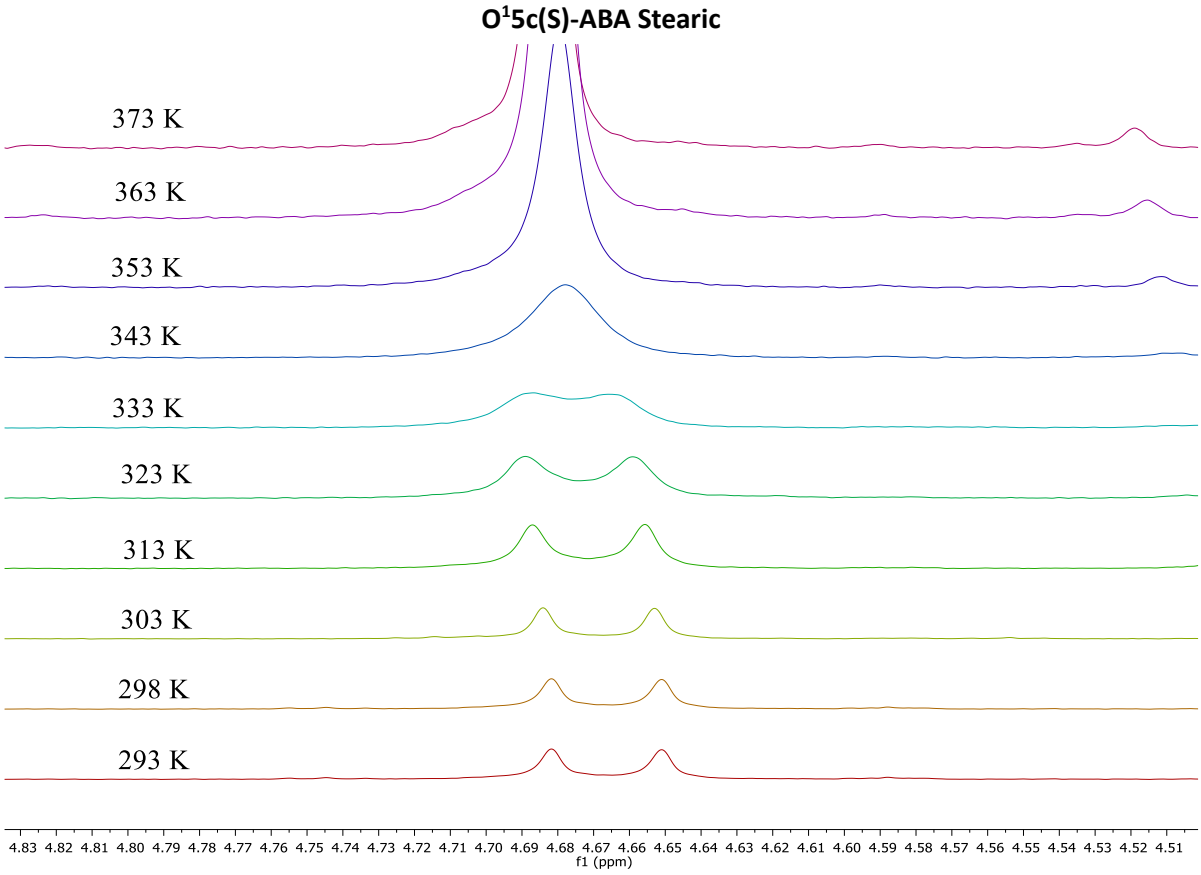


$O^1$3-ABA



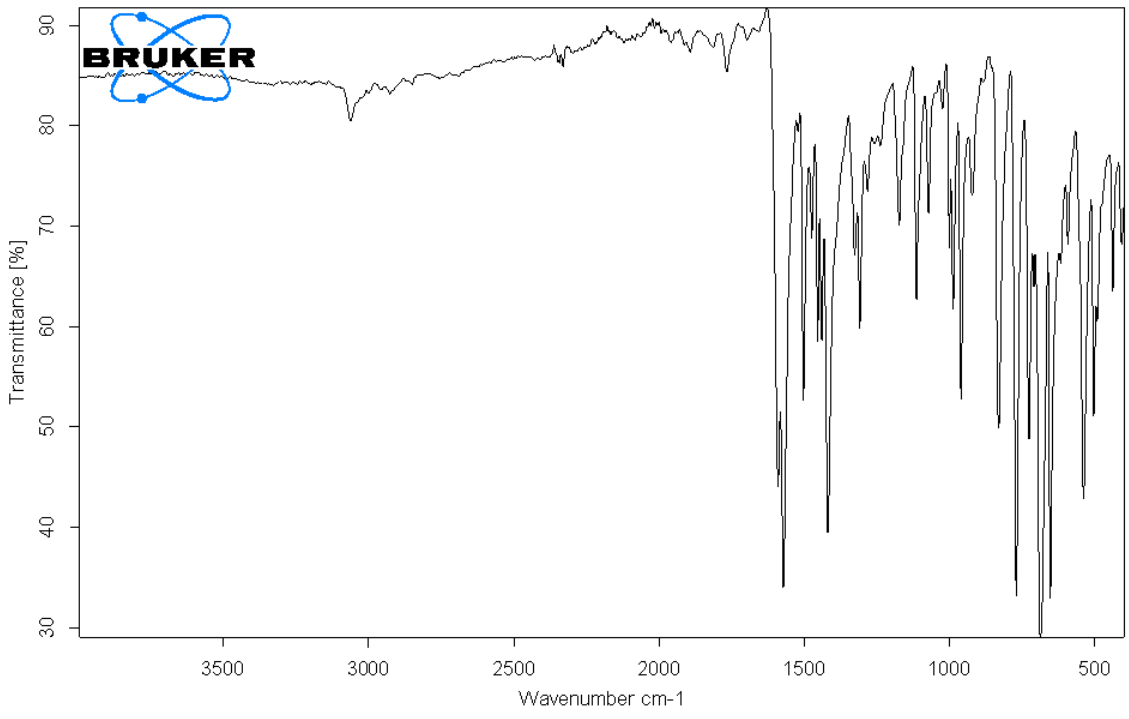
$O^1$5a(Me)-ABA



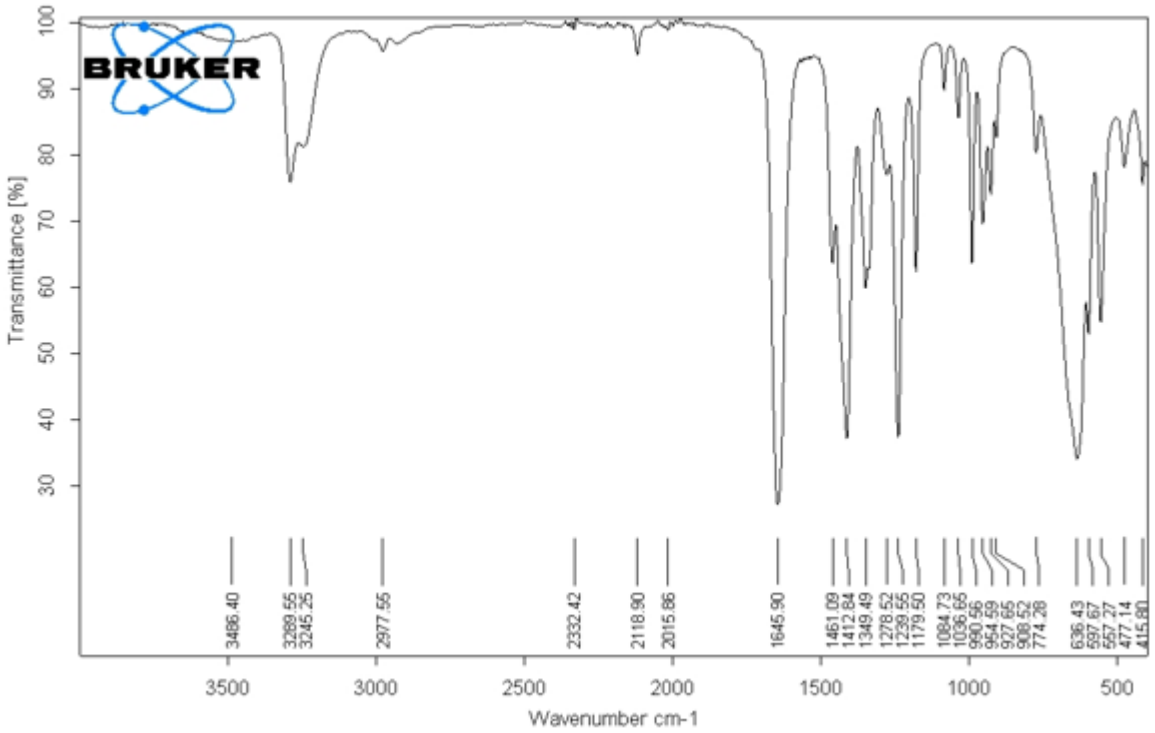


4.0 Infrared spectra

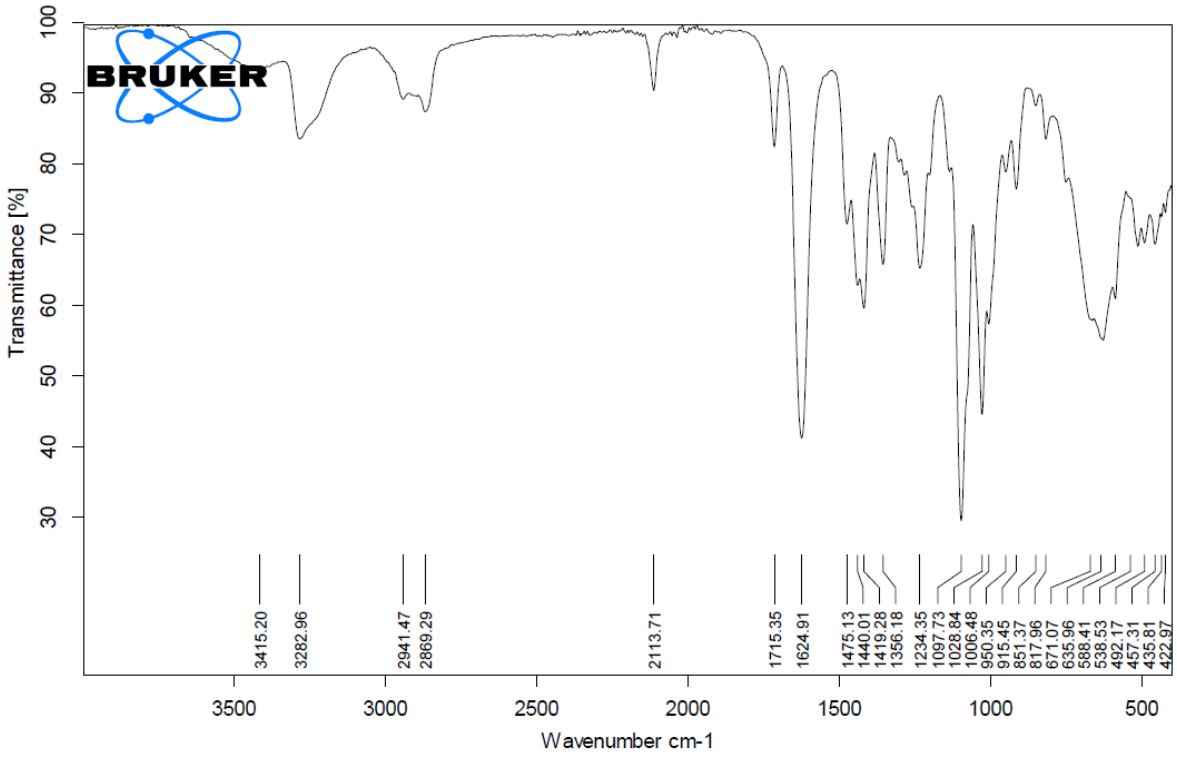
2



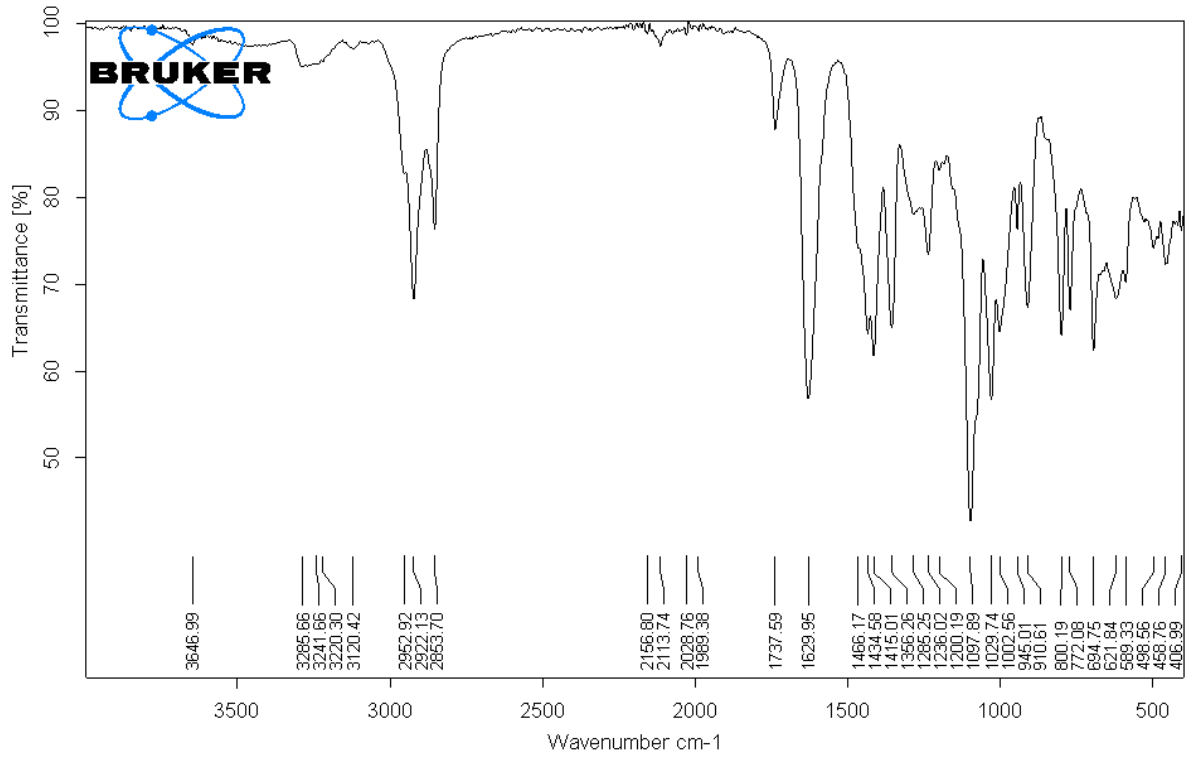
N,*N*-di(prop-2-yn-1-yl)acetamide **3**



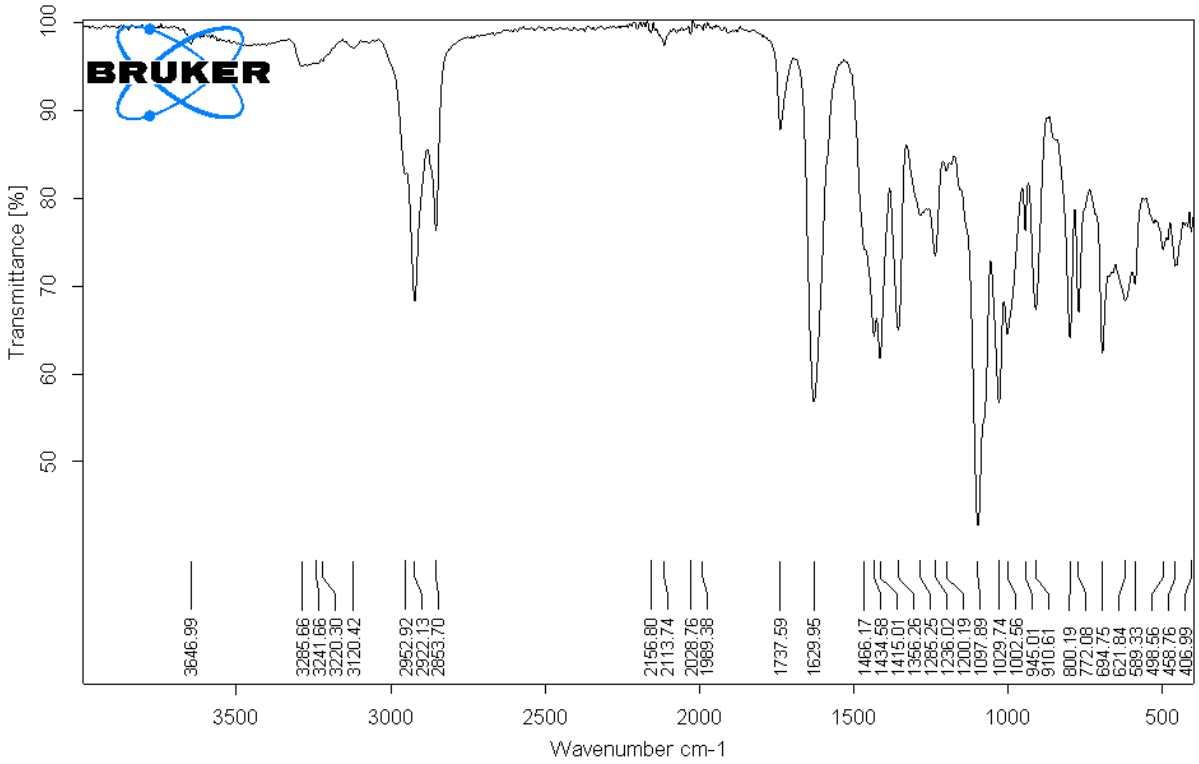
5a(Me)



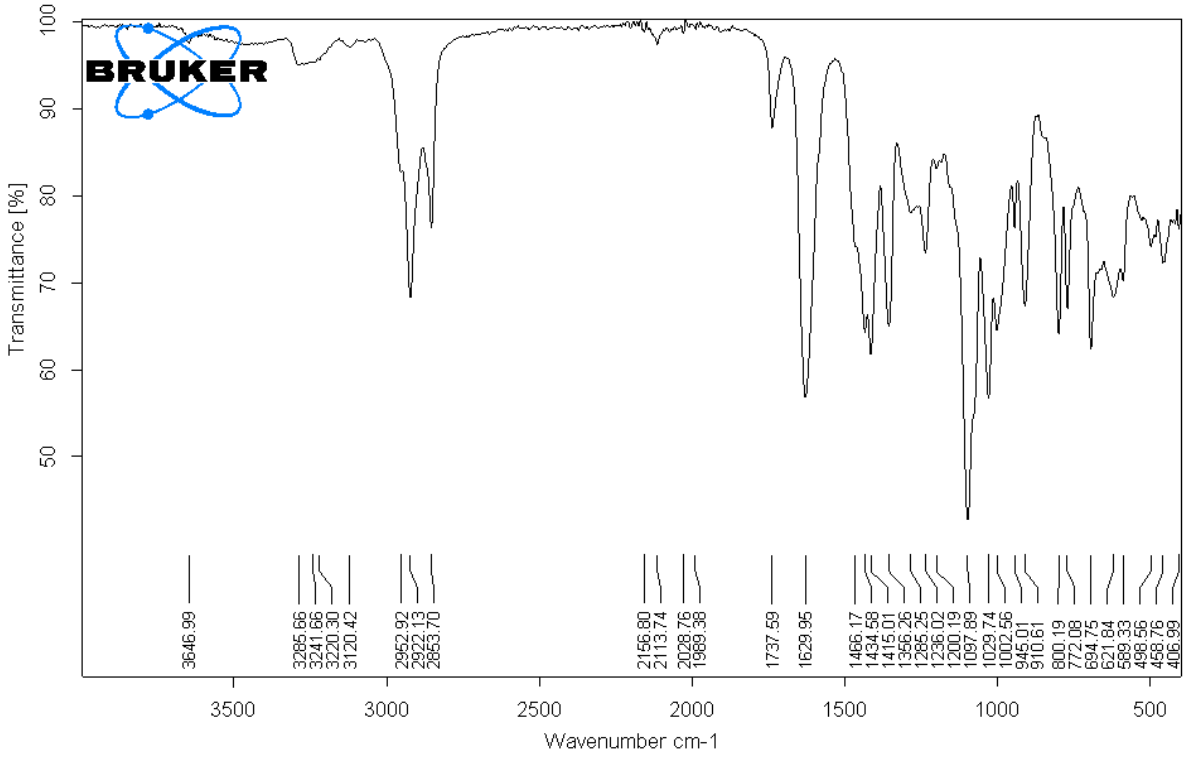
$O^1$3 oligomer ABA



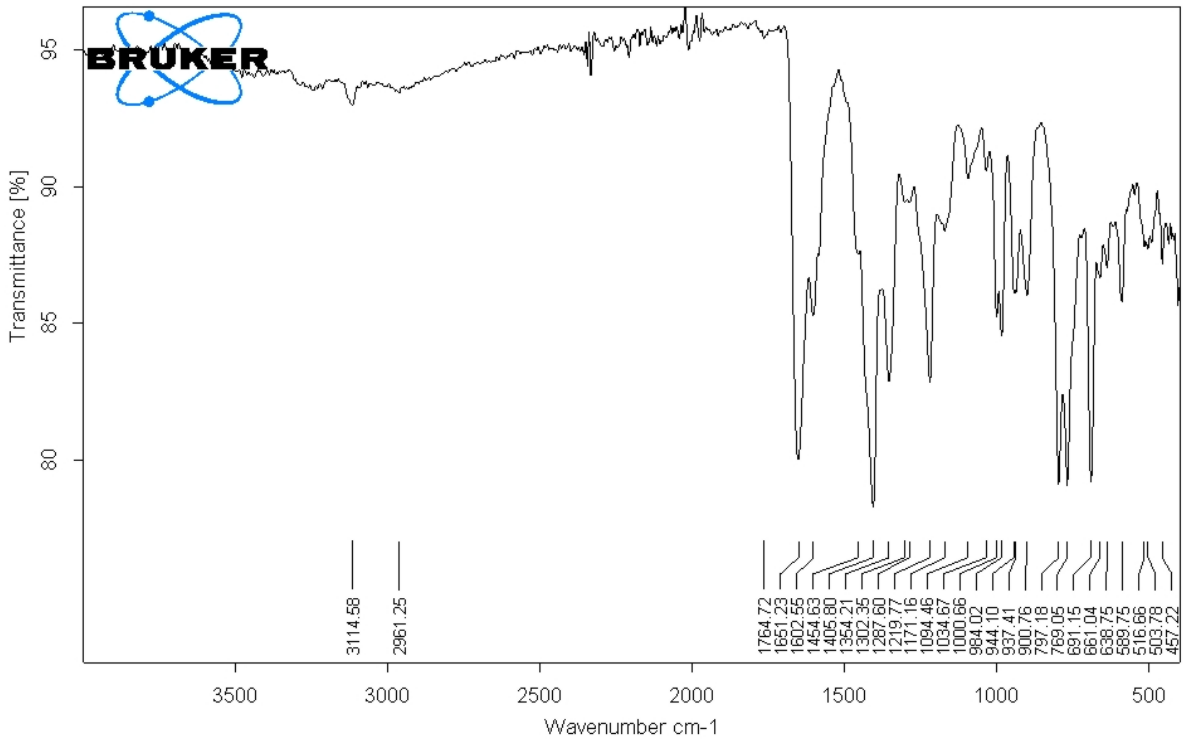
$O^1$5a(Me) oligomer ABA



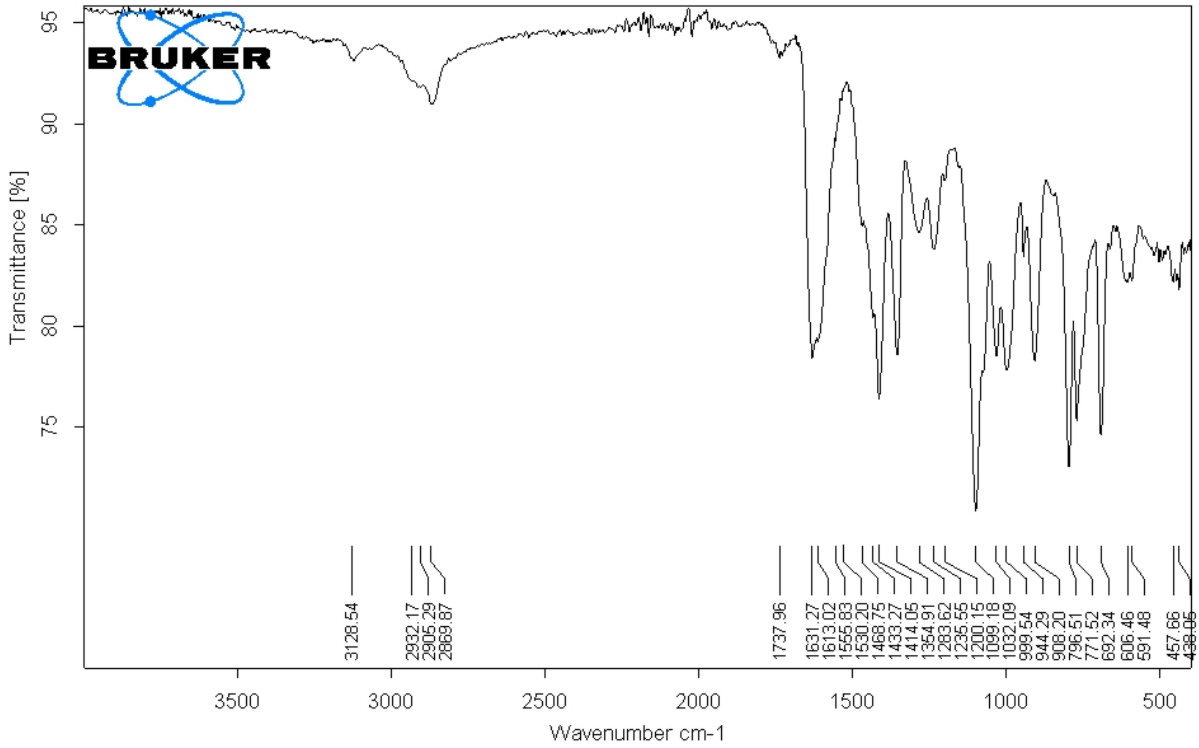
$O^2$5a(Me) oligomer $(AB)_2A$



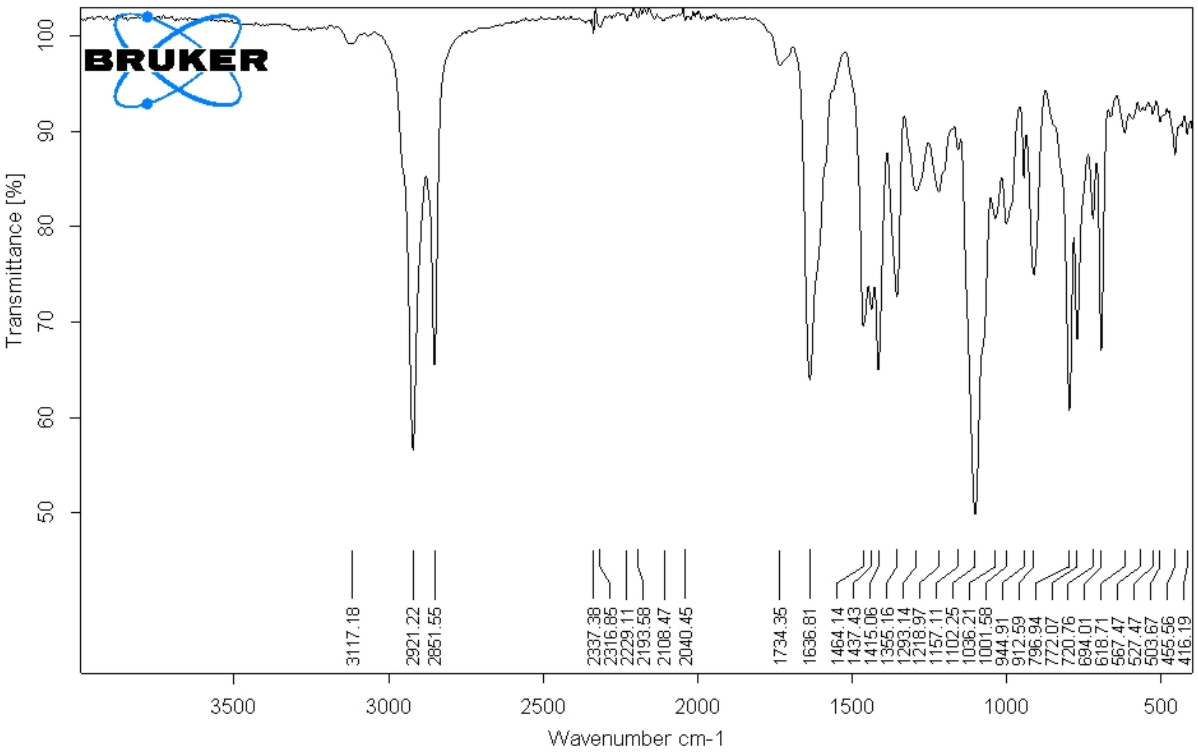
P(1a-co-3)



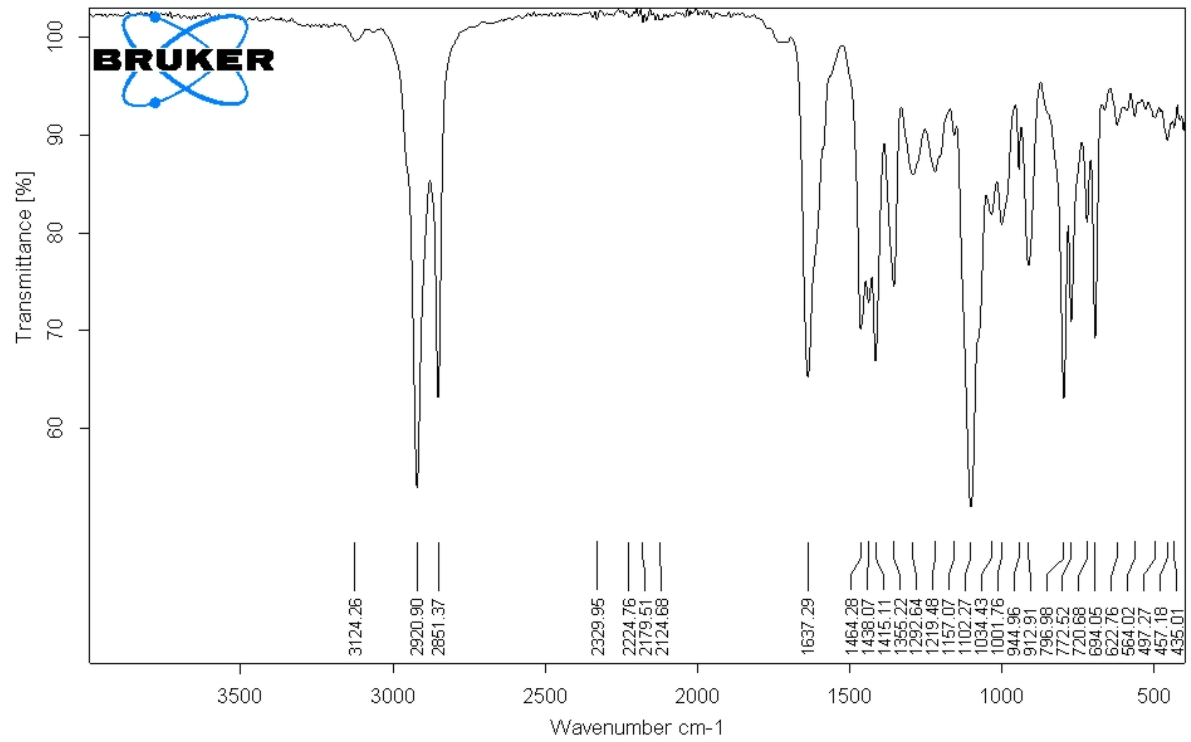
P(1a-co-5a)



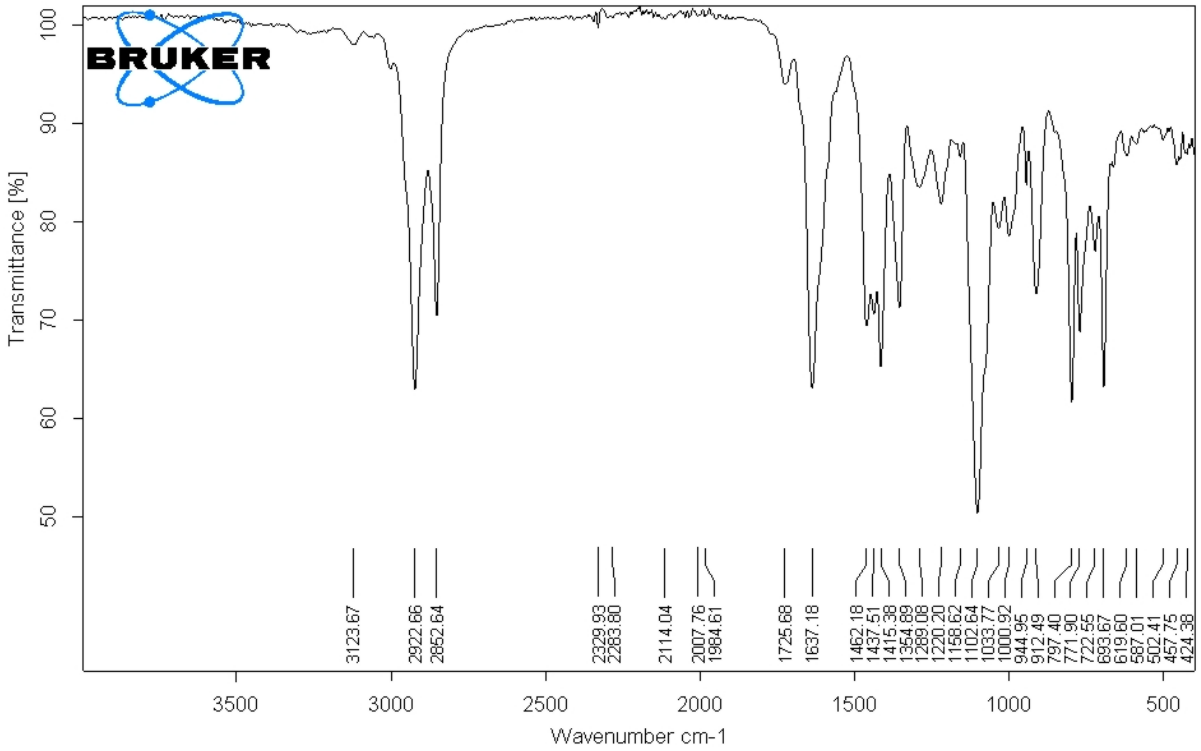
P(1a-co-5b(P)) Palmitic



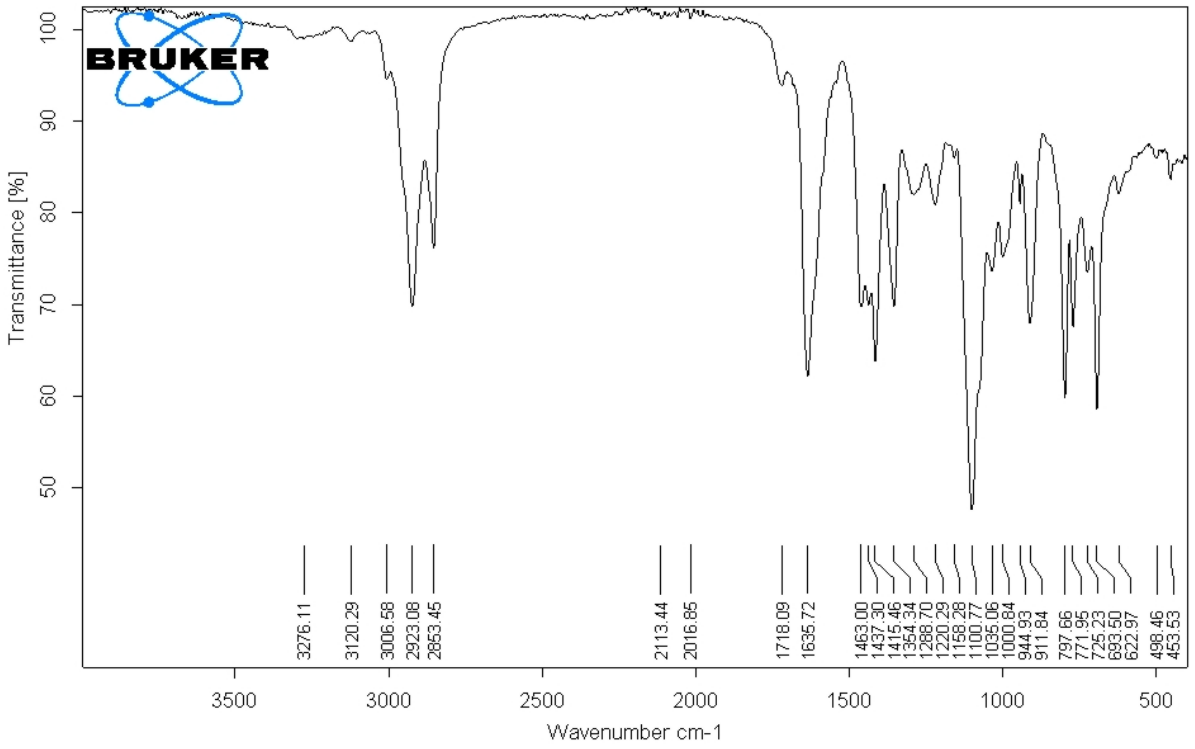
P(1a-co-5c(S)) Stearic



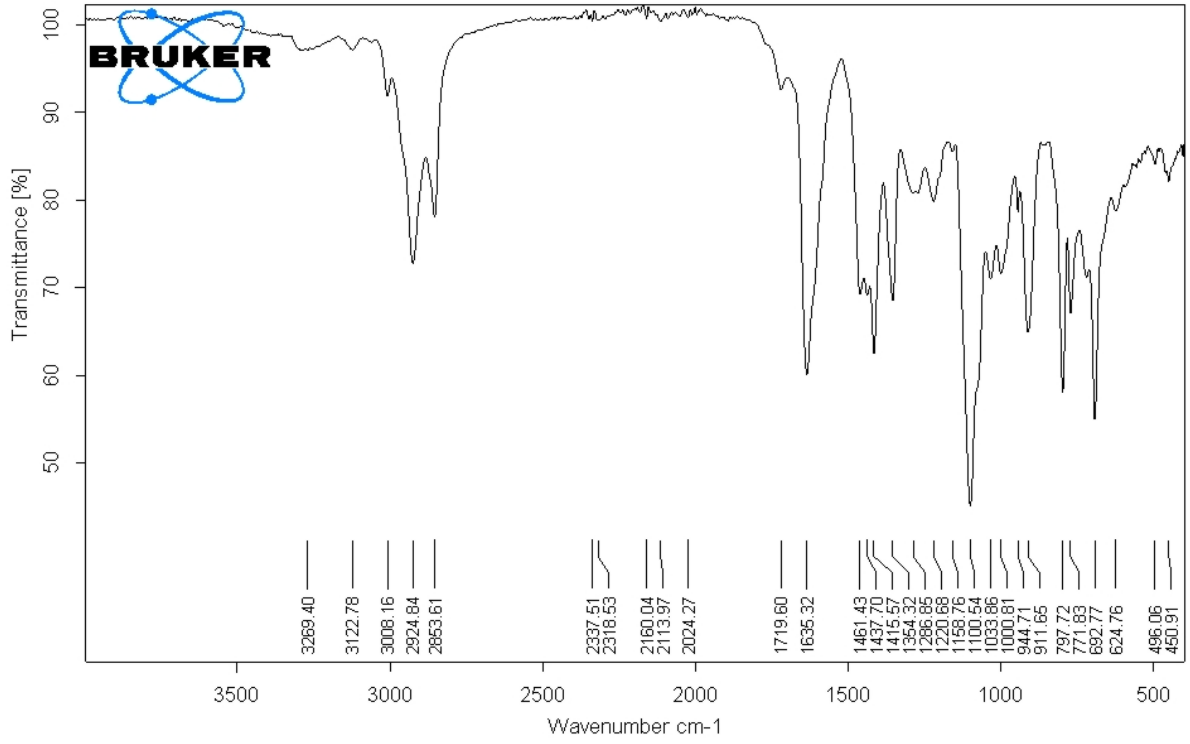
P(1a-co-5d(O)) Oleic



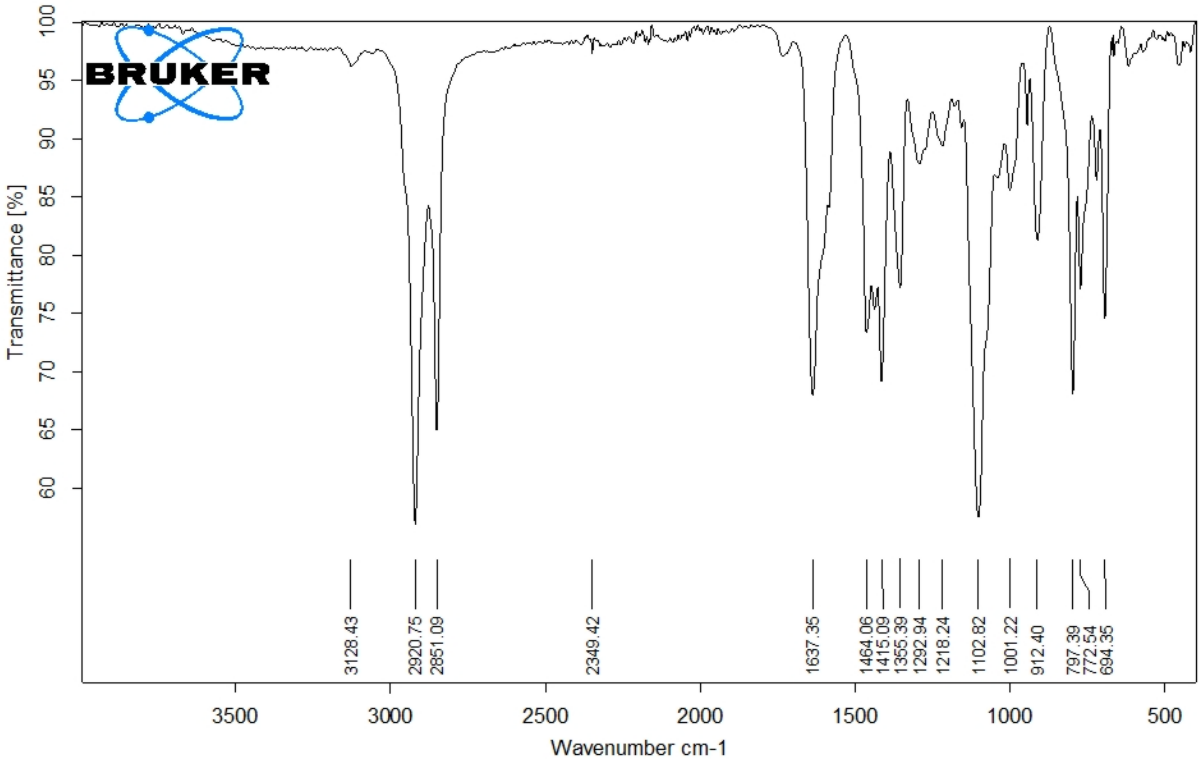
P(1a-co-5e(L)) Linoleic



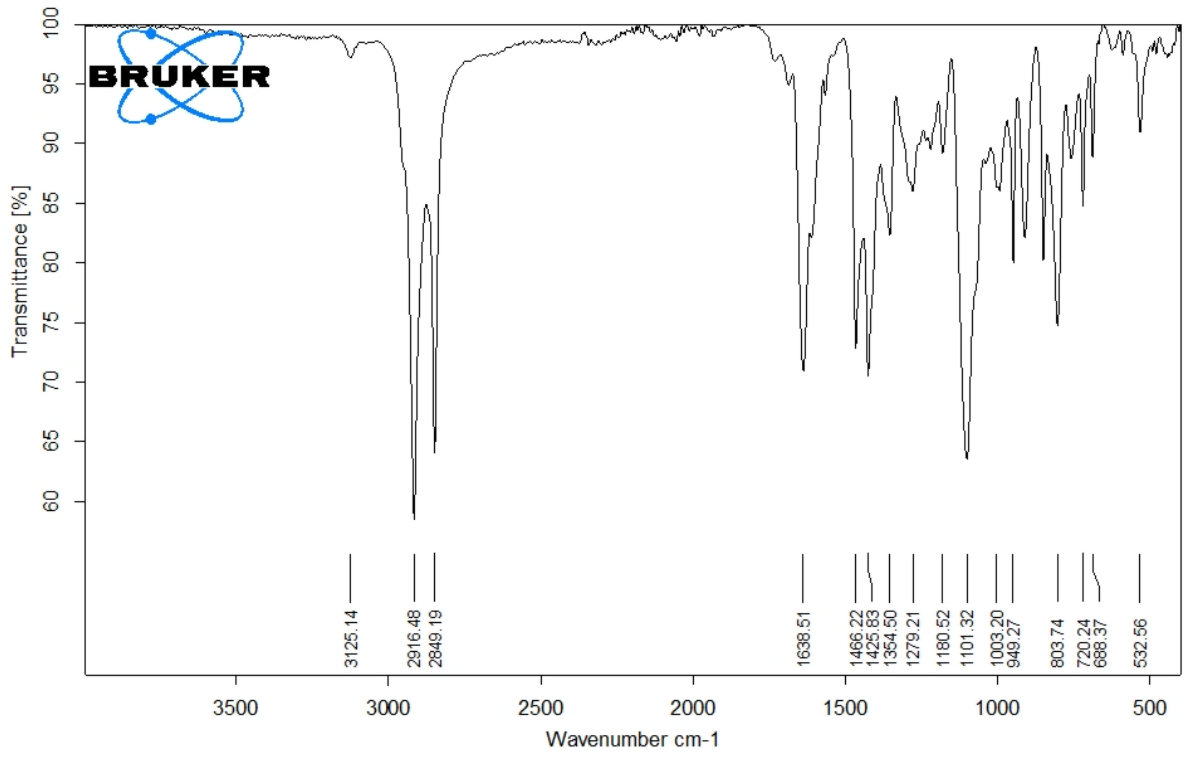
P(1a-co-5f(Ln)) Linolenic



P_{base}(1a-co-5c(S)) Stearic (*meta* nitrile-*N*-oxide)

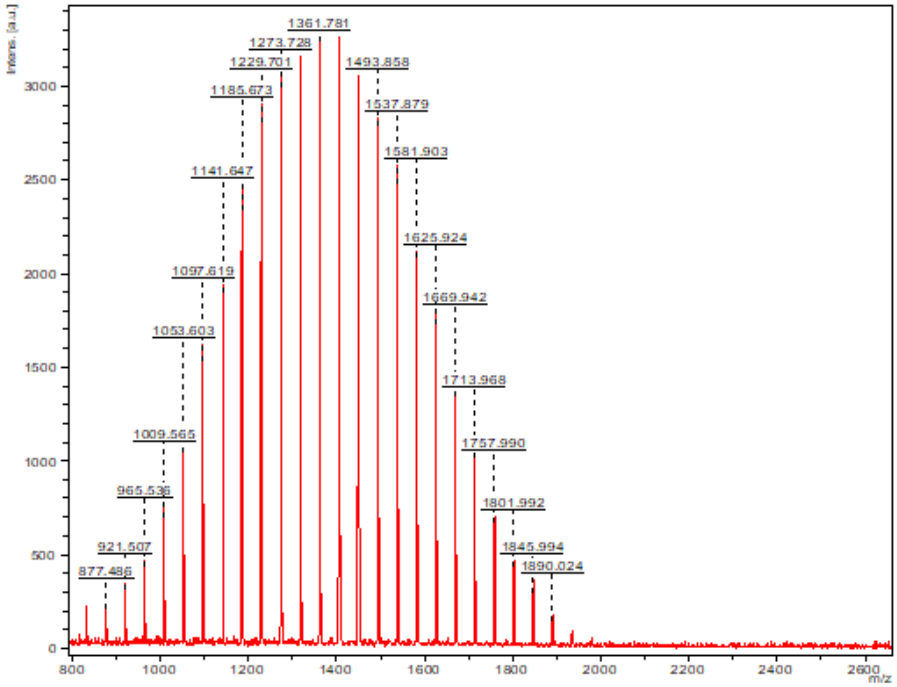


P_{base}(1b-co-5c(S)) Stearic (*para* nitrile-*N*-oxide)

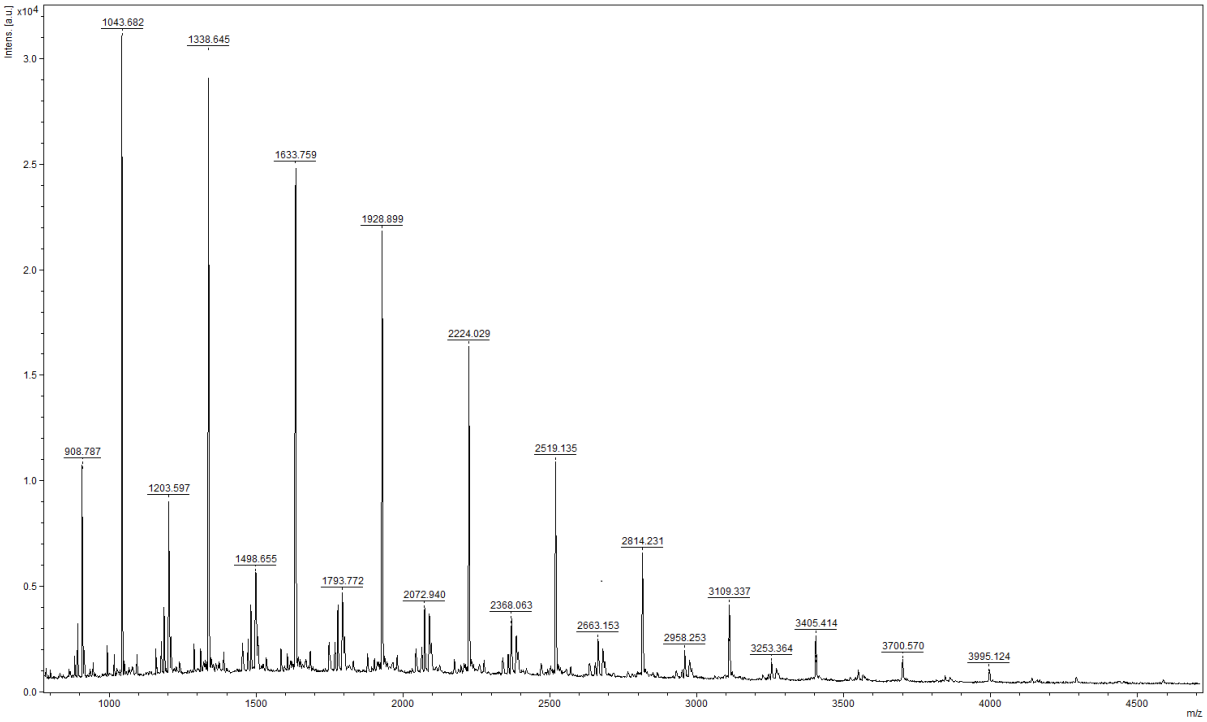


5.0 MALDI-TOF mass spectra for selected polymers

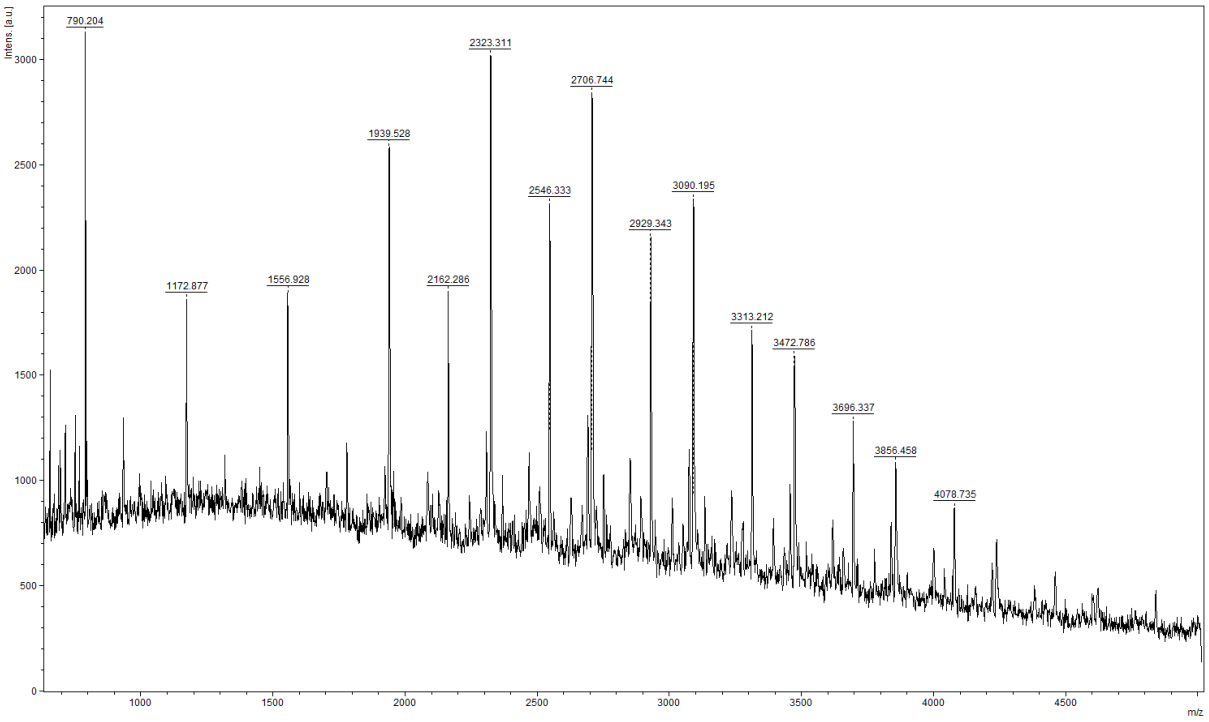
Calibration



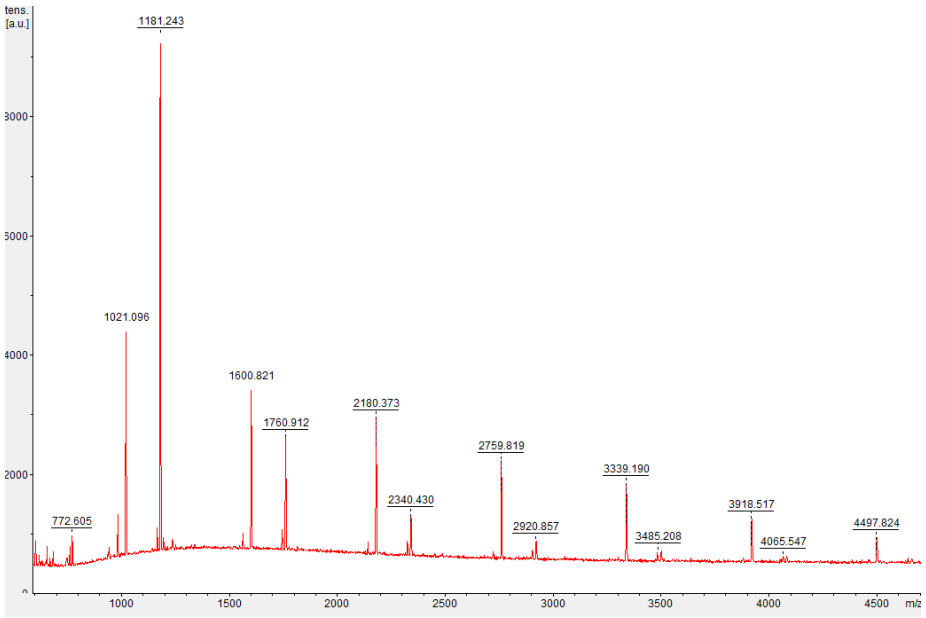
P(1a-co-3)

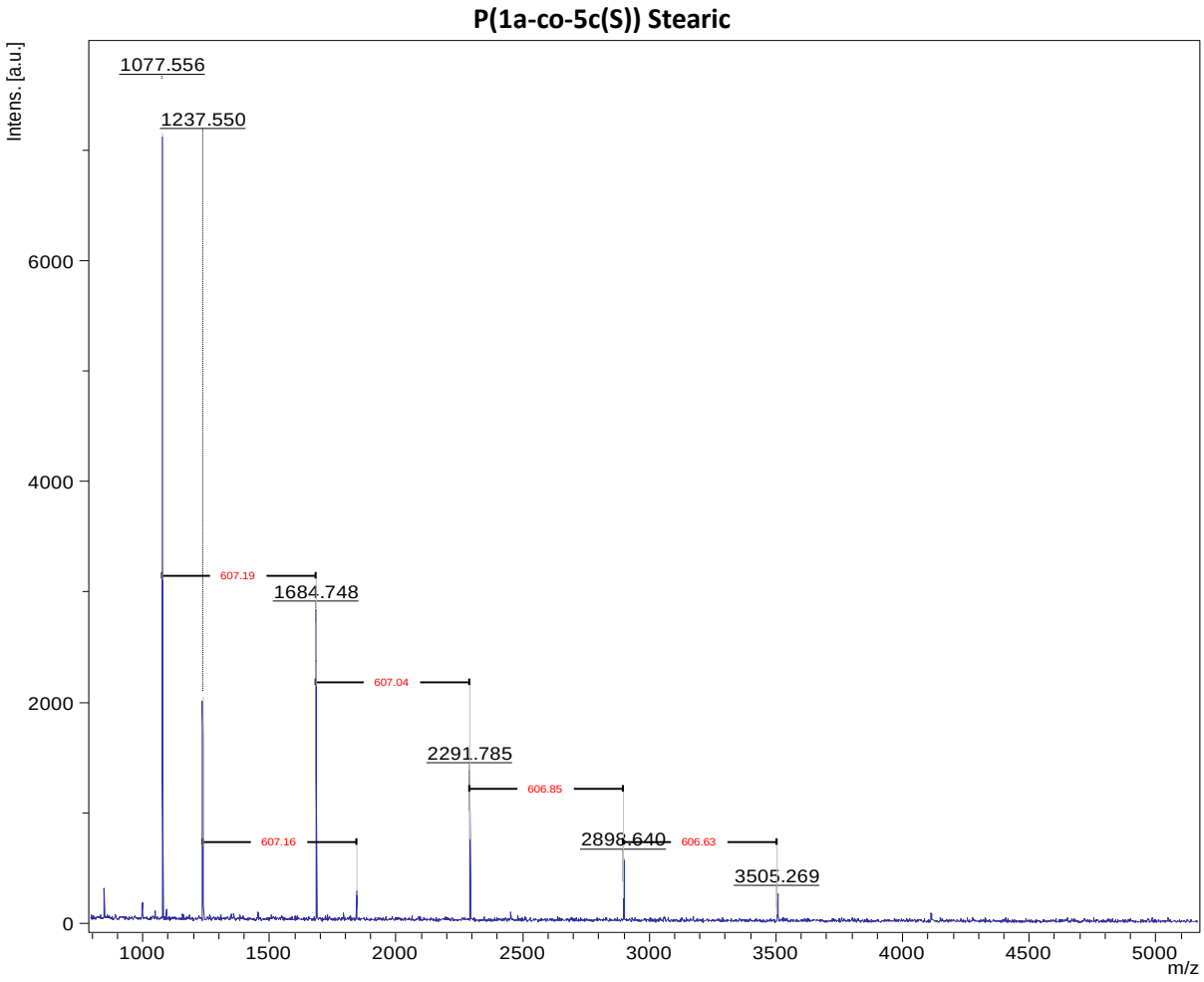


P(1a-co-5a(Me))

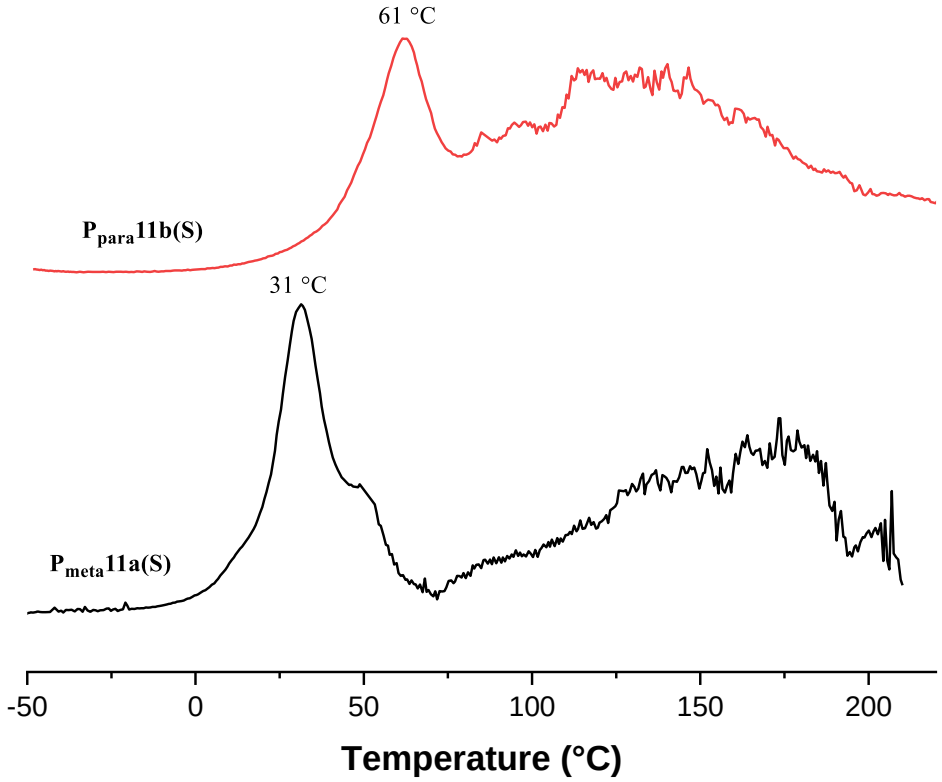


P(1a-co-5b(P)) Palmitic




P(1a-co-5c(S)) Stearic
Intens. [a.u.]
1077.556
1237.550
607.19
1684.748
607.04
2291.785
606.85
607.16
2898.640
606.63
3505.269
6000
4000
2000
0
1000
1500
2000
2500
3000
3500
4000
4500
5000
m/z

6.0 DMA data P_{base}(1a-co-5c(S)) and P_{base}(1b-co-5c(S)) Stearic.



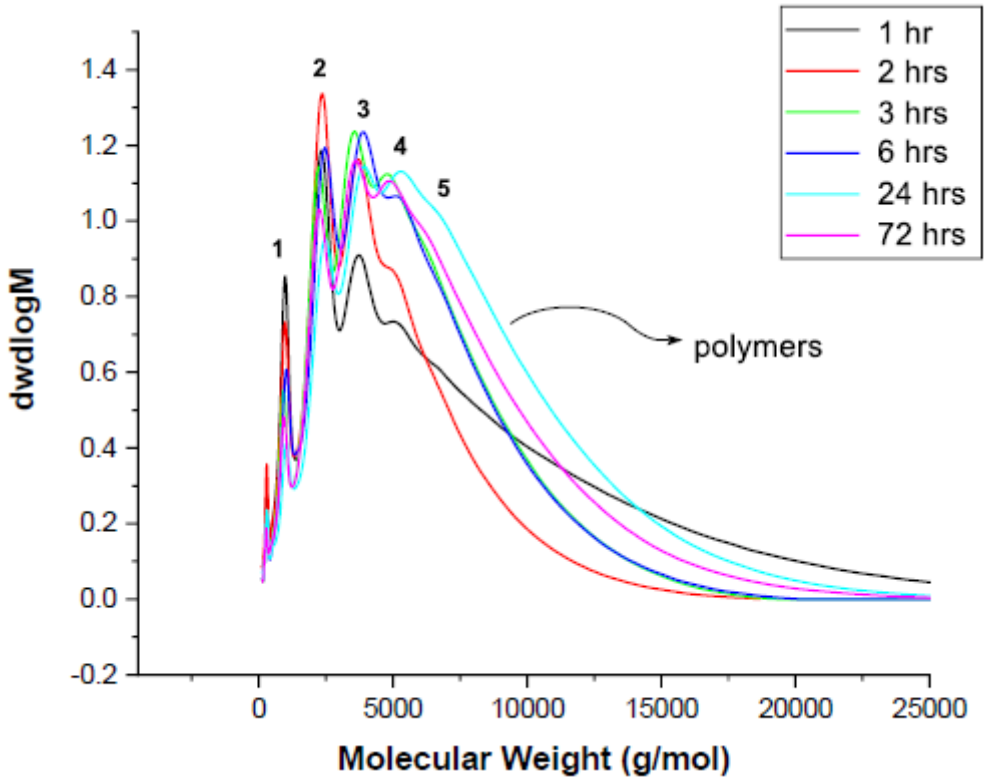
7.0 Experimental data for screening of base-mediated polymerisations.

All polymerisations were carried out at room temperature for 24 hours with 1:1 stoichiometry of **5c(S)** and **1a** unless highlighted by an asterisk in which case the stoichiometry was 1.0:1.1 **5c(S):1a**. All GPC's were carried out in THF as eluent and PMMA as a calibration standard.

Entry	Conditions	Solvent	Conversion	Mn	Mw	Đ
1	CuI, K_2CO_3	Et_2O	77%	5.4K	6.6K	1.2
2	CuI, K_2CO_3	DMF	57%	0.8K	1.9K	1.7
3	CuI, K_2CO_3	acetone	59%	1.0K	2.2K	1.8
4	CuI, K_2CO_3*	Et_2O	80%	3.5K	7.3K	2.1
5	CuI, K_2CO_3	$CHCl_3$	70%	1.9K	2.1K	1.1
6	CuI, Et_3N	$CHCl_3$	75%	3.4K	3.9K	1.1
7	$NaHCO_3$	EtOAc	77%	3.3K	3.4K	1.0
8	Na_2CO_3	DCM	85%	5.5K	7.7K	1.3
9	Na_2CO_3	DMF	55%	1.8K	2.0K	1.0
10	Na_2CO_3	acetone	70%	1.9K	2.0K	1.0
11	K_2CO_3	Et_2O	89%	6.7K	9.1K	1.8
12	K_2CO_3	acetone	66%	2.2K	3.0K	1.3
13	K_2CO_3	THF	86%	4.8K	6.8K	1.8
14	K_2CO_3	EtOH	94%	18.5K	35.5K	1.9
15	K_2CO_3*	EtOH	96%	19.0K	36.2K	1.9

8.0 GPC data of entries 1-15 from section 8.0

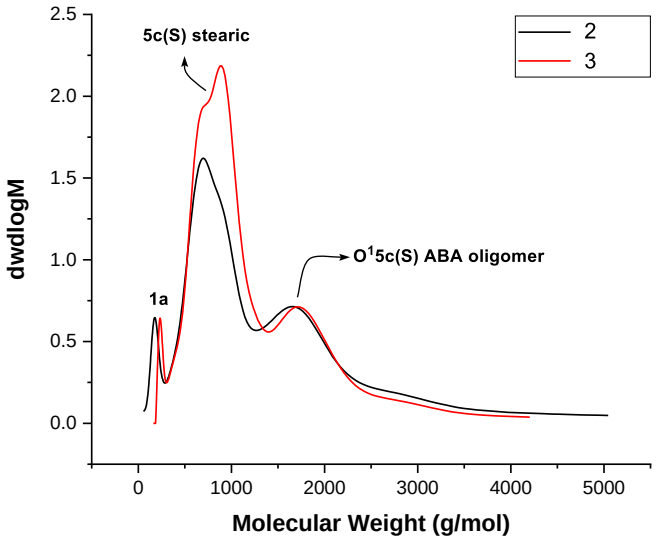
All GPC was carried out in THF using a PMMA calibration standard.



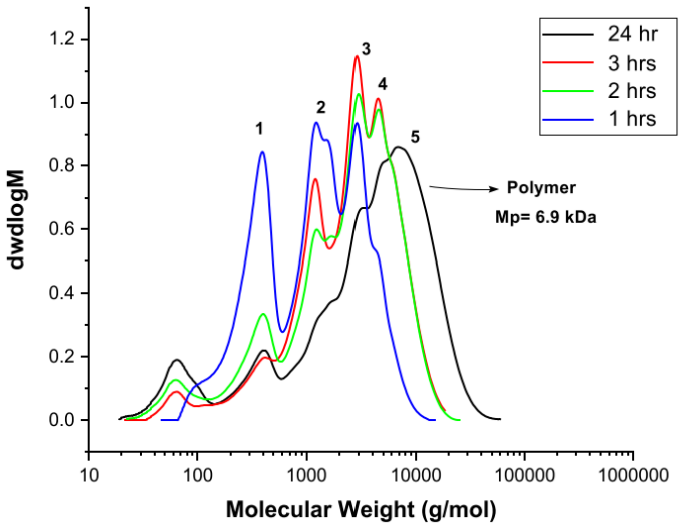
Polymer		Mp[a] (kDa)				
		Peak				
		1	2	3	4	5
Reaction time	1	0.3	1.0	2.3	3.7	4.5
	2	0.3	1.0	2.4	3.7	4.5
	3	0.3	1.0	2.3	3.6	4.8
	6	0.3	1.0	2.5	3.9	5.0
	24	0.3	1.0	2.5	3.9	5.2
	72	0.3	0.9	2.3	3.7	4.9

GPC data for polymers showing the peak molecular weight (Mp)

[a]Determined by GPC analysis in THF, calibrated against PMMA standards.



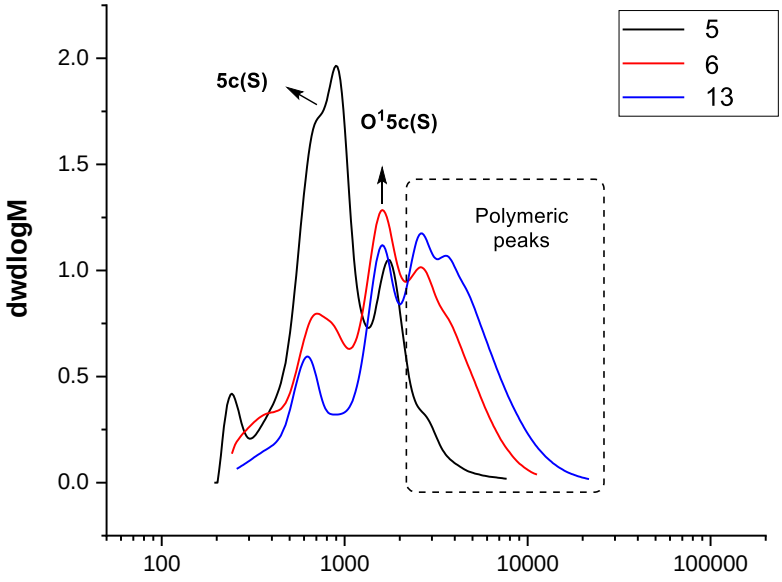
GPC data for entries 2 and 3



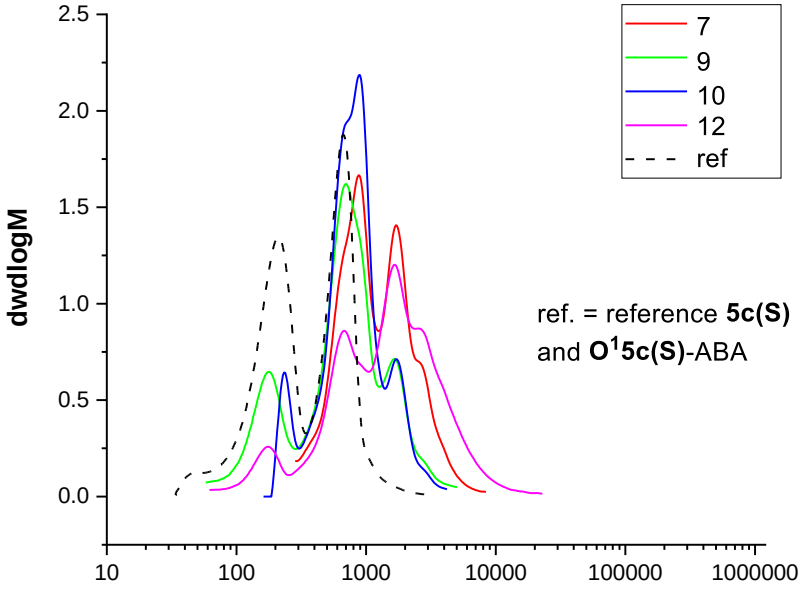
Polymer		Mp[a] (kDa)				
		Peak				
		1	2	3	4	5
Reaction	24	0.4	2.0	3.3	4.7	6.9
time	3	0.4	1.2	2.9	4.5	
	2	0.4	1.2	2.9	4.3	
	1	0.4	1.2	2.8	4.0	

GPC analysis of peak maximum (Mp) in polymerisation samples of entry 4.

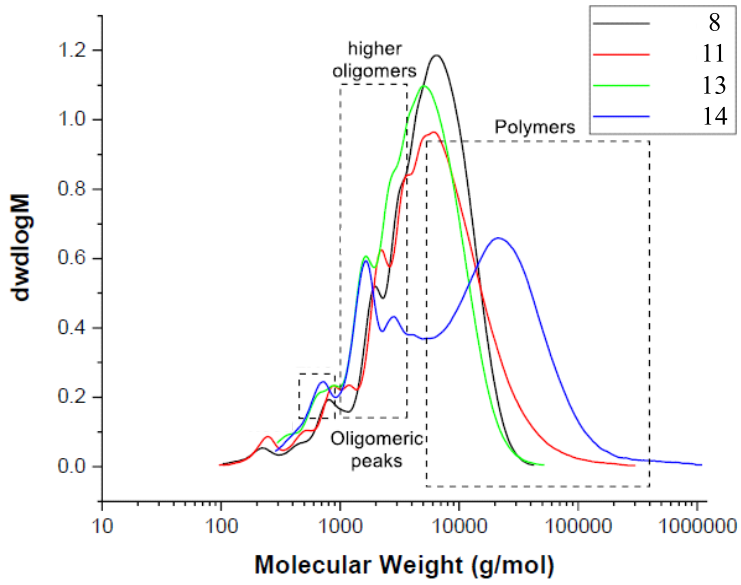
[a]GPC analysis was conducted in THF using PMMA as a calibration standard



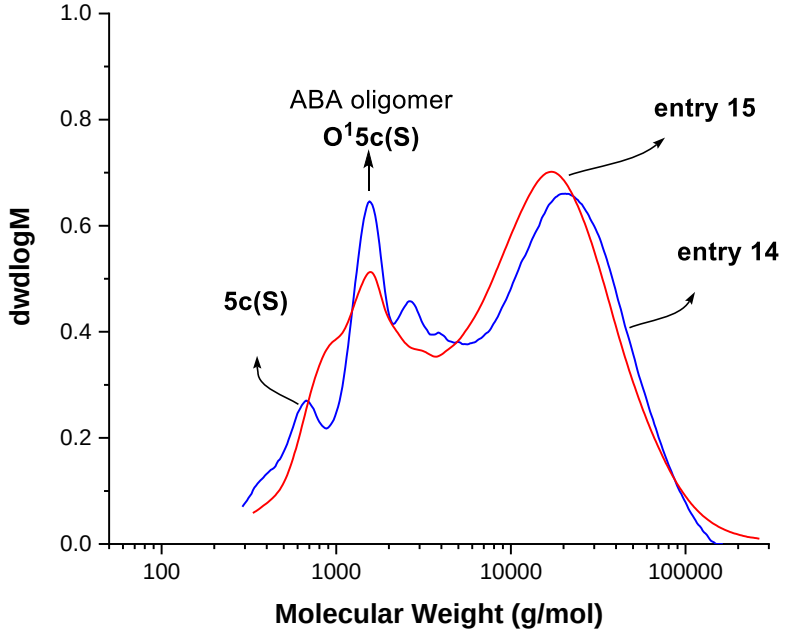
GPC data entries for 5, 6 and 13



GPC data for entries 7, 9, 10 and 12



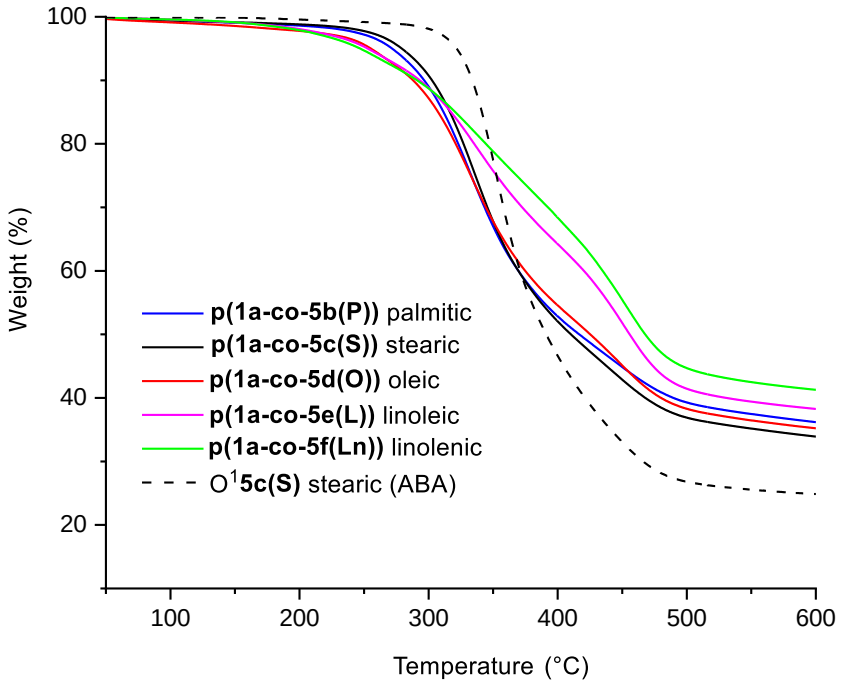
GPC data for entries 8, 11, 13, 14

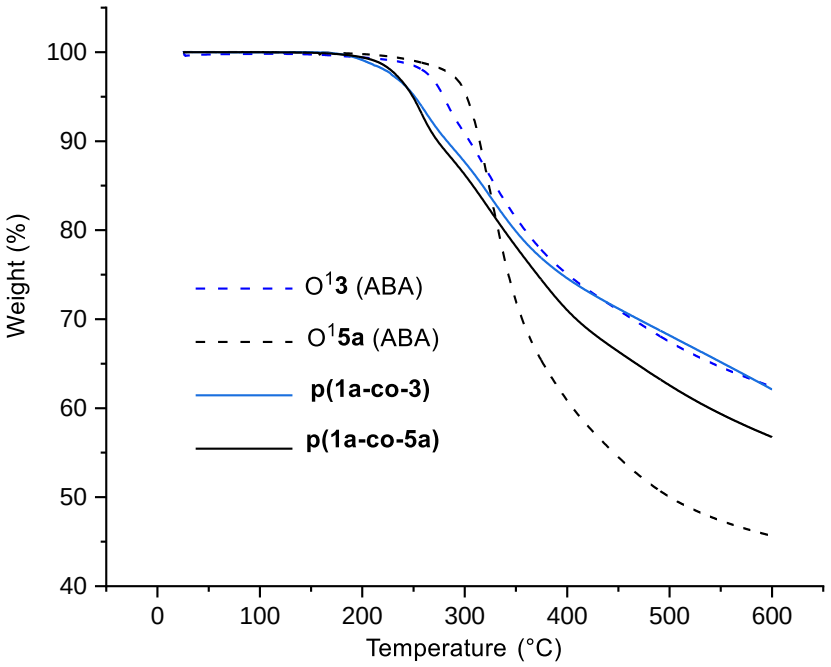


GPC data entries for 14 and 15

9.0 Thermal Gravimetric Analysis of all polymers and ABA type oligomers

TGA was achieved using a Mettler-Toledo TGA or a TA Instruments Discovery SDT 650 with autosampler. TGA samples, under nitrogen, were heated from 25 °C – 600 °C at 10 °C / min in 40 µl aluminium pans.





10.0 References

1. Koyama, Y.; Yonekawa, M.; Takata, T. *Chem. Lett.* **2008**, *37* (9), 918–919.
2. Kelly, D. R.; Baker, S. C.; King, D. S.; de Silva, D. S.; Lord, G.; Taylor, J. P. *Org. Biomol. Chem.* **2008**, *6* (4), 787–796.
3. Aitken, R. A.; Smith, M. H.; Wilson, H. S. *J. Mol. Struct.* **2016**, *1113*, 171–173.
4. Guan, L. P.; Sui, X.; Deng, X. Q.; Zhao, D. H.; Qu, Y. Le; Quan, Z. S. *Med. Chem. Res.* **2011**, *20* (5), 601–606.
5. Sagnella, S. M.; Conn, C. E.; Krodkiewska, I.; Drummond, C. J. *Phys. Chem. Chem. Phys.* **2011**, *13*, 13370–13381.