1. Analysis of target compounds

2-({2-*O*-[(butylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{4-[(3β)-cholest-5-en-3-yloxy]butyl}acetamide (5a): White solid (61% yield); δ_H (400 MHz; $CDCl_3$/MeOD; Me_4Si) 7.17 (1 H, t, *J* 5.6, N-H), 6.57 (1 H, t, *J* 5.3, N-H), 5.26 (1 H, m, H-6'), 4.91 (1 H, d, *J* 3.5, H-1), 4.45 (1 H, dd, *J* 3.5 and 10.1, H-2), 4.07 (1 H, d, *J* 15.4, H-7a), 3.90 (1 H, d, *J* 15.4, H-7b), 3.80-3.71 (2 H, m, H-3, H-6a), 3.64 (1 H, dd, *J* 5.5 and 12.3, H-6b), 3.50 (1 H, m, H-5), 3.46-3.40 (2 H, m, CH_2-12), 3.37 (1 H, t, *J* 9.6, H-4), 3.24-3.17 (2 H, m, CH_2-9), 3.13-2.97 (3 H, m, H-3', CH_2-14), 2.27 (1 H, ddd, *J* 13.1 4.5 and 2.0,H-4'a), 2.09 (1 H, m, H-4'b), 1.99-0.70 (49 H, m, H-Cholesterol, CH_2-10, CH_2-11, CH_2-15 to CH_2-17), 0.61 (1 H, s, CH_3-19'); δ_C (100 MHz; $CDCl_3$/MeOD; Me_4Si) 169.7, 156.3, 140.1, 121.4, 96.9, 79.0, 73.1, 72.4, 70.8, 69.8, 67.1, 66.1, 60.8, 56.4, 55.8, 49.8, 41.9, 40.2, 39.4, 39.1, 38.6, 38.4, 36.8, 36.4, 35.7, 35.4, 31.5, 31.4, 31.3, 27.9, 27.7, 27.5, 26.8, 25.8, 23.8, 23.3, 22.0, 21.8, 20.6, 19.4, 18.7, 18.0; $[\alpha]_D^{20}$ +11 (*c* 0.4 in $CHCl_3$/MeOH (1:1)); *m/z* (HRMS) 799.5446 ($M + Na^+$, $C_{44}H_{76}N_2O_9Na$ requires 799.5449).

2-({2-*O*-[(octylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{4-[(3β)-cholest-5-en-3-yloxy]butyl}acetamide (5b): White solid (65% yield); (Found: C, 68.72; H, 10.15; N, 3.23. $C_{48}H_{84}N_2O_9 \bullet 0.5H_2O$ requires C, 68.45; H, 10.17; N, 3.33); δ_H (300 MHz; $CDCl_3$/MeOD; Me_4Si) 7.16 (1 H, t, *J* 5.6, N-H), 6.57 (1 H, t, *J* 5.3, N-H), 5.26 (1 H, m, H-6'), 4.91 (1 H, d, *J* 3.6, H-1), 3.76 (1 H, dd, *J* 3.6 and 10.1, H-2), 4.06 (1 H, d, *J* 15.6, H-7a), 3.90 (1 H, d, *J* 15.6, H-7b), 3.79-3.71 (2 H, m, H-3, H-6a), 3.64 (1 H, dd, *J* 4.9 and 12.0, H-6b), 3.50 (1 H, ddd, *J* 2.3, 4.9, and 12.0, H-5), 3.42-3.34 (3 H, m, CH_2-12, H-4), 3.21-3.19 (2 H, m, CH_2-9), 3.12-2.98 (3 H, m, H-3', CH_2-12), 2.27 (1 H, m, H-4'a), 2.09 (1 H, m, H-4'b), 1.96-0.76 (57 H, m, H-Cholesterol, CH_2-10, CH_2-11, CH_2-15 to CH_2-20, CH_3-21), 0.60 (1 H, s, CH_3-19'); δ_C (75 MHz; $CDCl_3$/MeOD; Me_4Si) 176.4, 169.8, 140.5, 121.4, 97.3, 79.3, 73.4, 72.5, 71.2, 70.1, 67.5, 66.5, 61.2, 56.7, 56.1, 50.2,42.3, 41.0, 39.7, 39.5, 39.0, 38.8, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 29.7, 29.7, 29.6, 29.3, 28.3, 28.2, 27.9, 27.2, 26.8, 26.2, 24.2, 23.8, 22.6, 22.6, 22.4, 21.0, 19.1, 18.0, 13.4, 11.2; $[\alpha]_D^{20}$ +52 (*c* 0.5 in $CHCl_3$/MeOH (1:1)); *m/z* (ESI) 856.7 ($M + Na^+$).

2-({2-*O*-[(decylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{4-[(3β)-cholest-5-en-3-yloxy]butyl}acetamide (5c): White solid (53% yield) ; δ_H (400 MHz; $CDCl_3$/MeOD; Me_4Si) 7.03 (1 H, t, N-H), 6.26 (1 H, t, N-H), 5.25 (1 H, m, H-6'), 4.92 (1 H, d, *J* 3.7, H-1), 4.46 (1 H, dd, *J* 3.7 and 10.1, H-2), 4.06 (1 H, d, *J* 15.5, H-7a), 3.89 (1 H, d, *J* 15.6, H-7b), 3.80-3.70 (2 H, m, H-3, H-6a), 3.67 (1 H, m, H-6b), 3.50 (1 H, m, H-5), 3.46-3.36 (3 H, m, CH_2-12, H-4), 3.24-3.17 (2 H, m, CH_2-9), 3.13-2.97 (3 H, m, H-3', CH_2-14), 2.27 (1 H, m, H-4'a), 2.10 (1 H, m, H-4'b), 2.00-0.73 (61 H, m, H-Cholesterol, CH_2-10, CH_2-11, CH_2-15 to CH_2-22, CH_3-23), 0.61 (1 H, s, CH_3-19'); δ_C (100 MHz; $CDCl_3$/MeOD; Me_4Si) δ 169.8, 156.3, 140.4, 121.7, 97.2, 79.2, 73.3, 72.3, 71.1, 70.5, 67.4, 66.4, 61.1, 56.6, 56.0, 50.1, 42.2, 40.9, 39.6, 39.3, 38.9, 38.8, 38.7, 37.0, 36.7, 36.0, 35.6, 31.7, 29.6, 29.5, 29.4, 29.2, 29.2, 28.2, 28.0, 27.8, 27.1, 26.7, 26.1, 24.1, 23.6, 22.5, 22.2, 20.9, 19.1, 18.4, 13.8, 11.6; $[\alpha]_D^{20}$ +25 (*c* 0.5 in $CHCl_3$/MeOH (1:1)); *m/z* (HRMS) 883.6390 ($M + Na^+$, $C_{50}H_{88}N_2O_9Na$ requires 883.6388).

2-({2-*O*-[(dodecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{4-[(3β)-cholest-5-en-3-yloxy]butyl}acetamide (5d)[26]

2-({2-*O*-[(tetradecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{4-[(3β)-cholest-5-en-3-yloxy]butyl}acetamide (5e): White solid; (87% yield); δ_H (300 MHz; $CDCl_3$/MeOD; Me_4Si) 7.03 (1 H, t, N-H), 6.17 (1 H, t, N-H), 5.35 (1 H, m, H-6'), 4.98 (1 H, d, *J* 3.5, H-1), 4.54 (1 H, dd, *J* 3.5 and 10.1, H-2), 4.14 (1 H, d, *J* 15.6, H-7a), 3.96 (1 H, d, *J* 15.6, H-7b), 3.87-3.77 (3 H, m, H-3, H-6a, H-6b), 3.60-3.47 (4 H, m, CH_2-12, H-4, H-5), 3.28 (2 H, m, CH_2-9), 3.21-3.04 (3 H, m, H-3', CH_2-14), 2.34 (1 H, m, H-4'a), 2.17 (1 H, m, H-4'b), 2.03-0.80 (69 H, m, H-Cholesterol, CH_2-10, CH_2-11, CH_2-15 to CH_2-26, CH_3-27), 0.68 (1 H, s, CH_3-19'); δ_C (125 MHz; $CDCl_3$/MeOD; Me_4Si) 169.6, 156.4, 140.7, 121.9, 97.4, 79.4, 73.5, 72.3, 71.4, 70.3, 67.6, 66.7, 61.4, 56.8, 56.2, 50.2, 42.4, 41.2, 39.8, 39.6, 39.1, 38.8, 37.3, 36.9, 36.2, 35.8, 32.0, 31.9, 29.8-29.2 (10 x CH_2), 28.4, 28.3, 28.0, 27.3, 26.9, 26.3, 24.3, 23.9, 22.8, 22.7, 22.5, 21.1, 19.4, 18.7, 14.1, 11.9; $[\alpha]_D^{20}$ +29 (*c* 0.4 in $CHCl_3$/MeOH (5:1)); *m/z* (HRMS) 939.7013 ($M + Na^+$, $C_{54}H_{96}N_2O_9Na$ requires 939.7014).

2-({2-*O*-[(hexadecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{4-[(3β)-cholest-5-en-3-yloxy]butyl}acetamide (5f): White solid (75% yield); (Found: C, 71.42; H, 10.66; N, 3.13. $C_{56}H_{100}O_9N_2$ requires C, 71.14; H, 10.66; N, 2.96); δ_H (500 MHz; $CDCl_3$/MeOD; Me_4Si) 5.35 (1 H, m, H-6'), 4.92 (1 H, d, *J* 3.0, H-1), 4.54 (1 H, dd, *J* 3.0 and 9.9, H-2), 4.15 (1 H, d, *J* 15.8, H-7a), 3.95 (1 H, d, *J* 15.8, H-7b), 3.80-3.74 (3 H, m, H-3, H-6a, H-6b), 3.58-3.49 (4 H, m, CH_2-12, H-4, H-5), 3.28 (2 H, m, CH_2-9), 3.16-3.09 (3 H, m, H-3', CH_2-14), 2.36 (1 H, m, H-4'a), 2.18 (1 H, m, H-4'b), 2.05-0.80 (63 H, m, H-Cholesterol, CH_2-10, CH_2-11, CH_2-15 to CH_2-28, CH_3-29), 0.68 (1 H, s, CH_3-19'); δ_C (75 MHz; $CDCl_3$/MeOD; Me_4Si) 169.9, 156.5, 140. 7, 122.9, 97.4, 79.5, 73.5, 72.5, 71.4, 70.2, 67.7, 66.7, 61.3, 56.9, 56.3, 50.3, 42.4, 41.2, 39.9, 39.6, 39.2, 38.9, 37.3, 36.9, 36.3, 35.8, 32.0, 32.0, 29.9-29.5 (12 x CH_2), 28.5, 28.3, 28.1, 27.4, 27.0, 26.4, 24.4, 23.9, 22.9, 22.8, 22.6, 21.2, 19.4, 18.8, 14.2, 11.9; $[\alpha]_D^{20}$ +38 (*c* 0.4 in $CHCl_3$/MeOH (5:1)); *m/z* (HRMS) 967.7329 ($M + Na^+$, $C_{56}H_{100}O_9N_2Na$ requires 967.7327).

2-({2-*O*-[(octadecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{4-[(3β)-cholest-5-en-3-yloxy]butyl}acetamide (5g): White solid; (73% yield); δ_H (300 MHz; $CDCl_3$/MeOD; Me_4Si) 7.16 (1 H, t, N-H), 6.46 (1 H, t, N-H), 5.35 (1 H, m, H-6'), 5.01 (1 H, d, *J* 3.6, H-1), 4.54 (1 H, dd, *J* 3.6 and 10.1, H-2), 4.15 (1 H, d, *J* 15.6, H-7a), 3.97 (1 H, d, *J* 15.6, H-7b), 3.88-3.77 (3 H, m, H-3, H-6a, H-6b), 3.61-3.45 (4 H, m, CH_2-12, H-4, H-5), 3.29 (2 H, m, CH_2-9), 3.13 (3 H, m, H-3', CH_2-14), 2.36 (1 H, m, H-4'a), 2.20 (1 H, m, H-4'b), 2.05-0.85 (77 H, m, H-Cholesterol, CH_2-10, CH_2-11, CH_2-15 to CH_2-30, CH_3-31), 0.68 (1 H, s, CH_3-19'); δ_C (125 MHz; $CDCl_3$/MeOD; Me_4Si) 169.6, 156.3, 140. 7, 121.9, 97.4, 79.4, 73.4, 72.3, 71.4, 70.2, 67.6, 66.7, 61.4, 56.8, 56.2, 50.2, 42.4, 41.2, 39.8, 39.5, 39.1, 38.8, 37.2, 36.9, 36.2, 35.8, 32.0, 32.0, 29.8-29.4 (14 x CH_2), 28.4, 28.3, 28.0, 27.3, 27.3, 26.3, 24.3, 23.9, 22.8, 22.7, 22.5, 21.1, 19.4, 18.7, 14.1, 11.9; $[\alpha]_D^{20}$ +28 (*c* 1.0 in $CHCl_3$/MeOH (8:1)); *m/z* (HRMS) 995,7641 ($M + Na^+$, $C_{58}H_{104}O_9N_2Na$ requires 995,7640).

(3β)-3[(6-azidohexyl)oxy]cholest-5-ene (10): (63% yield) δ_H (300 MHz; $CDCl_3$; Me_4Si) 5.30 (1 H, m, H-6'), 3.45 (2 H, t, *J* 6.7, H-6), 3.26 (2 H, t, *J* 6.9, H-1), 3.14 (1 H, m, H-3'), 2.38-2.10 (2 H, m, H-4'), 2.00-0.80 (47 H, m, H-Cholesterol, CH_2-2 to CH_2-5), 0.64 (3 H, s, CH_3-18'); δ_C (75 MHz; $CDCl_3$; Me_4Si) 141.3, 121.6, 79.2, 68.0, 56.9, 56.3, 51.6, 50.4, 42.5, 40.0, 39.7, 39.4, 37.4, 37.1, 36.3, 35.9, 32.1, 32.0, 30.2, 29.0, 28.6, 28.4, 28.2, 26.7, 26.0, 24.4, 24.0, 23.0, 22.7, 21.2, 19.5, 18.9, 12.0; IR 2095 cm^{-1} (N_3); $[\alpha]_D^{20}$ -23 (*c* 0.9 in CH_2Cl_2); *m/z* (HRMS) 534.4398 ($M + Na^+$, $C_{33}H_{57}ON_3Na$ requires 534.4394).

(3β)-3[(8-azidooctyl)oxy]cholest-5-ene (11): (890 mg, 1.65 mmol, 80% yield); δ_H (300 MHz; $CDCl_3$; Me_4Si) 5.34 (1 H, m, H-6'), 3.45 (2 H, t, *J* 6.8, H-8), 3.25 (2 H, t, *J* 6.9, H-1), 3.12 (1 H, m, H-3'), 2.35-2.05 (2 H, m, H-4'), 2.00-0.75 (51 H, m, H-Cholesterol, CH_2-2 to CH_2-7), 0.61 (3 H, s, CH_3-18'); δ_C (75 MHz; $CDCl_3$; Me_4Si) 141.3, 121.6, 79.2, 68.2, 56.9, 56.3, 51.6, 50.4, 42.5, 40.0, 39.7, 39.4, 39.4, 37.4, 37.1, 36.3, 35.9, 32.1, 32.1, 32.1, 30.3, 29.5, 29.2, 29.0, 28.6, 28.4, 28.2, 26.7, 26.3, 24.4, 24.0, 22.7, 21.2, 19.5, 18.9, 12.0; IR 2095 cm^{-1} (N_3); $[\alpha]_D^{20}$ -24 (*c* 0.6 in CH_2Cl_2)); *m/z* (HRMS) 562.4704 ($M + Na^+$, $C_{35}H_{61}ON_3Na$ requires 562.4707).

(3β)-3[(10-azidodecyl)oxy]cholest-5-ene (12): (842 mg, 1.48 mmol, 72% yield); δ_H (300 MHz; $CDCl_3$; Me_4Si) 5.27 (1 H, m, H-6'), 3.37 (2 H, t, *J* 6.8, H-10), 3.27 (2 H, t, *J* 7.0, H-1), 3.05 (1 H, m, H-3'), 2.34-2.06 (2 H, m, H-4'), 2.2-0.(55 H, m, H-Cholesterol, CH_2-2 to CH_2-9), 0.61 (3 H, s, CH_3-18'); δ_C (75 MHz; $CDCl_3$; Me_4Si) 141.3, 121.5, 79.1, 68.2, 56.9, 56.3, 51.6, 50.4, 42.5, 40.0, 39.7, 39.4, 37.4, 37.1, 36.3, 35.9, 32.1, 32.0, 30.3, 29.6, 29.6, 29.5, 29.3, 29.0, 28.6, 28.1, 26.7, 26.3, 24.4, 24.0, 23.0, 22.7, 21.2, 19.5, 18.9, 12.0; IR 2095 cm^{-1} (N_3); $[\alpha]_D^{20}$ -25 (*c* 1.2 in CH_2Cl_2); *m/z* (HRMS) 590.5018 ($M + Na^+$, $C_{37}H_{65}ON_3Na$ requires 590.5020).

(3β)-3[(12-azidododecyl)oxy]cholest-5-ene (13): (923 mg, 1.55 mmol, 75% yield); δ_H (300 MHz; $CDCl_3$; Me_4Si) 5.27 (1 H, m, H-6'), 3.37 (2 H, t, *J* 6.8, H-12), 3.18 (2 H, t, *J* 7.0, H-1), 3.05 (1 H, m, H-3'), 2.33-2.05 (2 H, m, H-4'), 1.99-0.76 (59 H, m, H-Cholesterol, CH_2-2 to CH_2-11), 0.61 (3 H, s, CH_3-18'); δ_C (75 MHz; $CDCl_3$; Me_4Si) 141.3, 121.5, 79.0, 68.2, 56.9, 56.3, 51.6, 50.3, 42.4, 39.9, 39.6, 39.3, 37.4, 37.0, 36.3, 35.9, 32.1, 32.0, 30.3, 29.7, 29.6, 29.6, 29.6, 29.6, 29.2, 28.9, 28.6, 28.3, 28.1, 26.8, 26.3, 24.4, 23.9, 22.9, 22.7, 21.2, 19.5, 18.8, 12.0; IR 2094 cm^{-1} (N_3); $[\alpha]_D^{20}$ -27 (*c* 0.5 in CH_2Cl_2); *m/z* (HRMS) 618.5327 ($M + Na^+$, $C_{39}H_{69}N_3ONa$ requires 618.5333).

***N*-{6-[(3β)-cholest-5-en-3-yloxy]hexyl}-2-[(3,4,6-tri-*O*-acetyl-α-D-glucopyranosyl)oxy]acetamide (14a):** (72% yield) δ_H (300 MHz; $CDCl_3$; Me_4Si) 7.44 (1H, t, NH), 5.34 (1 H, m, H-6'), 5.29 (1H, t, *J* 9.8, H-3), 4.94 (1 H, t, *J* 9.8, H-4), 4.82 (1H, d, *J* 3.7, H-1)，4.26-3.93 (5 H, m, H-5, H-6a, H-6b, H-7a, H-7b), 3.74 (1 H, dd, *J* 9.5, 2.9, H-2)，3.44 (2 H, t, *J* 6.6, H-14), 3.40 (2 H, m, H-9), 3.01 (1 H, m, H-3'), 2.40-0.80 (58 H, m, H-Cholesterol, 3xAc, CH_2-10 to CH_2-13), 0.61 (3 H, s, CH_3-18'); δ_C (75 MHz; $CDCl_3$; Me_4Si) 171.2, 170.7, 169.7, 169.0, 140.8, 121.8, 114.7, 99.5, 79.0, 73.4, 70.1, 68.2, 68.1, 67.7, 67.6, 67.6, 62.0, 62.0, 56.8, 56.2, 50.3, 42.4, 39.9, 39.6, 39.2, 38.6, 37.2, 37.0, 36.3, 35.7, 32.0, 32.0, 32.0, 32.0, 29.9, 29.0, 28.5, 28.5, 28.3, 28.3, 28.1, 26.4, 25.6, 24.4, 23.9, 22.90, 22.6, 21.2, 21.0, 20.8, 20.7, 19.5, 18.8, 12.0; $[\alpha]_D^{20}$ +40 (*c* 0.8 in CH_2Cl_2); *m/z* (HRMS) 832.5540 ($M + H^+$, $C_{47}H_{78}NO_{11}$ requires 832.5569).

***N*-{8-[(3β)-cholest-5-en-3-yloxy]octyl}-2-[(3,4,6-tri-*O*-acetyl-α-D-glucopyranosyl)oxy]acetamide (15a):** (603 mg, 50% yield); δ_H (300 MHz; $CDCl_3$; Me_4Si) 7.14 (1 H, t, NH), 5.27 (1 H, m, H-6'), 5.21 (1 H, t, *J* 9.8, H-3), 4.95 (1 H, t, *J* 9.9, H-4), 4.83 (1 H, d, *J* 3.7, H-1)，4.26-3.92 (5 H, m, H-5, H-6a, H-6b, H-7a, H-7b), 3.75 (1 H, dd, *J* 9.5, 3.8, H-1)，3.38 (2 H, t, *J* 6.6, H-16), 3.20 (2 H, m, H-9), 3.06 (1 H, m, H-3'), 2.45-0.70 (62 H, m, H-Cholesterol, 3xAc, CH_2-10 to CH_2-15), 0.61 (3 H, s, CH_3-18'); δ_C (75MHz; $CDCl_3$; Me_4Si) 170.7, 169.7, 168.8, 141.1, 121.6, 99.2, 79.1, 73.6, 70.4, 68.2, 67.9, 67.4, 61.9, 56.8, 56.2, 50.3, 42.4, 39.9, 39.6, 39.2, 39.2, 37.3, 37.0, 36.3, 35.9, 32.0, 32.0, 30.1, 29.4, 29.3, 29.2, 28.5, 28.3, 28.1, 26.8, 26.1, 24.4, 23.9, 22.9, 22.6, 21.1,

21.0, 20.8, 20.7, 19.5, 18.8, 11.9; $[\alpha]_D^{20}$ +38 (*c* 0.8 in CH_2Cl_2); *m/z* (HRMS) 860.5852 ($M + H^+$, $C_{49}H_{82}NO_{11}$ requires 860.5882).

***N*-{10-[(3β)-cholest-5-en-3-yloxy]decyl}-2-[(3,4,6-tri-*O*-acetyl-α-D-glucopyranosyl)oxy]acetamide (16a):** (500 mg, 0.56 mmol, 35% yield); δ_H (300 MHz; $CDCl_3$; Me_4Si) 7.14 (1 H, t, NH), 5.28 (1 H, m, H-6'), 5.21 (1 H, t, *J* 9.7, H-3), 4.95 (1 H, t, *J* 9.8, H-4), 4.84 (1 H, d, *J* 3.5, H-1)，4.26-3.92 (5 H, m, H-5, H-6a, H-6b, H-7a, H-7b), 3.75 (1 H, dd, *J* 9.9, 3.7, H-2)，3.37 (2 H, t, *J* 6.7, H-18), 3.20 (2 H, m, H-9), 3.06 (1 H, m, H-3'), 2.34-0.74 (66 H, m, H-Cholesterol, 3xAc, CH_2-10 to CH_2-17), 0.61 (3 H, s, CH_3-18'); δ_C (75 MHz; $CDCl_3$; Me_4Si) 171.7, 170.7, 169.6, 169.5, 141.1, 121.5, 99.0, 79.0, 73.7, 70.6, 68.2, 68.2, 67.9, 61.9, 56.8, 56.2, 50.3, 42.4, 39.9, 39.6, 39.3, 37.4, 37.0, 35.9, 32.0, 32.0, 30.2, 29.5, 29.5, 29.3, 28.3, 28.1, 26.9, 26.2, 24.4, 23.9, 22.9, 22.6, 21.1, 21.0, 20.8, 20.7, 19.5, 18.8, 11.9; $[\alpha]_D^{20}$ +38 (*c* 0.8 in CH_2Cl_2); *m/z* (HRMS) 888.6162 ($M + H^+$, $C_{51}H_{86}NO_{11}$ requires 888.6195).

***N*-{12-[(3β)-cholest-5-en-3-yloxy]dodecyl}-2-[(3,4,6-tri-*O*-acetyl-α-D-glucopyranosyl)oxy]acetamide (17a): (**1,05 g, 1.15 mmol, 56% yield); δ_H (300 MHz; $CDCl_3$; Me_4Si) 7.16 (1H, t, NH), 5.34 (1 H, m, H-6'), 5.25 (1 H, t, *J* 9.7, H-3), 5.02 (1 H, t, *J* 9.9, H-4), 4.89 (1 H, d, *J* 3.7, H-1), 4.32-3.97 (5 H, m, H-5, H-6a, H-6b, H-7a, H-7b), 3.80 (1 H, dd, *J* 9.8, 3.8, H-2), 3.43 (2 H, t, *J* 6.8, H-20), 3.25 (2 H, m, H-9), 3.11 (1 H, m, H-3'), 2.38-0.74 (70 H, m, H-Cholesterol, 3xAc, CH_2-2 to CH_2-19), 0.66 (3 H, s, CH_3-18'); δ_C (75 MHz; $CDCl_3$; Me_4Si) 171.7, 170.7, 169.6, 168.7, 141.2, 121.5, 99.0, 79.0, 73.8, 70.6, 68.2, 68.2, 67.9, 67.3, 61.9, 56.9, 56.2, 50.3, 42.4, 39.9, 39.6, 39.3, 37.4, 37.0, 36.3, 35.9, 32.0, 32.0, 30.3, 29.6, 29.6, 29.5, 29.3, 28.6, 28.3, 28.1, 27.0, 26.3, 24.4, 23.9, 22.9, 22.6, 21.1, 21.0, 20.8, 20.7, 19.5, 18.8, 11.9; $[\alpha]_D^{20}$ +37 (*c* 1.5 in CH_2Cl_2); *m/z* (HRMS) 916.6475 ($M + H^+$, $C_{53}H_{90}NO_{11}$ requires 916.6508).

***N*-{6-[(3β)-cholest-5-en-3-yloxy]hexyl}-2-(α-D-glucopyranosyloxy) acetamide (14b):** (72% yield) δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.27 (1 H, m, H-6'), 4.72 (1 H, d, *J* 3.7, H-1), 4.08 (1 H, d, *J*15.9 Hz, H-7a), 3.91(1 H, d, *J* 15.9, H-7b), 3.73-3.60 (3 H, m, H-6a, H-6b, H-3), 3.57-3.32 (5 H, m, H-2, H-4, H-5, CH_2-14), 3.30 (1 H, m, NH), 3.22-3.01 (3 H, m, CH_2-9, H-3'), 2.32-2.03 (2 H, m, H4'), 1.96-0.77 (49 H, m, H-Cholesterol, CH_2-10 to CH_2-13), 0.60 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 170.5, 141.1, 122.0, 99.8, 79.5, 73.9, 72.6, 71.9, 70.3, 68.3, 67.1, 61.7, 57.1, 56.4, 50.5, 42.6, 40.1, 39.8, 39.3, 39.3, 37.5, 37.1, 36.5, 36.1, 32.2, 32.2, 30.2, 29.3, 28.7, 28.5, 28.3, 27.0, 26.1, 24.5, 24.1, 23.0, 22.8, 21.3, 19.6, 19.0, 12.1; $[\alpha]_D^{20}$ +33 (*c* 0.5 in CH_2Cl_2/MeOH, 9:1); *m/z* (HRMS) 706.5262 ($M + H^+$, $C_{41}H_{72}O_8N$ requires 706.5252).

***N*-{8-[(3β)-cholest-5-en-3-yloxy]octyl}-2-(α-D-glucopyranosyloxy) acetamide (15b):** (35mg, 0.05 mmol, 81% yield); δ_H (300 MHz; $CDCl_3$; Me_4Si) 5.28 (1 H, m, H-6'), 4.72 (1 H, d, *J* 3.7, H-1), 4.08 (1 H, d, *J* 15.9, H-7a), 3.91(1 H, d, *J* 15.9, H-7b), 3.74-3.60 (3 H, m, H-6a, H-6b, H-3), 3.57-3.32 (5 H, m, H-2, H-4, H-5, CH_2-16), 3.30 (1 H, m, NH), 3.22-3.01 (3 H, m, CH_2-9, H-3'), 2.32-2.03 (2 H, m, H4'), 1.96-0.77 (54 H, m, H-Cholesterol, CH_2-10 to CH_2-15), 0.60 (3 H, s, CH_3-18'); δ_C (75 MHz; $CDCl_3$; Me_4Si) 170.1, 140.8, 121.5, 99.4, 79.1, 73.6, 72.3, 71.6, 69.9, 68.1, 66.7, 61.4, 56.7, 56.1, 50.1, 42.3, 39.7, 39.5, 39.1, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 30.0, 29.3, 29.2, 29.1, 28.3, 28.2, 27.9, 26.8, 26.8, 26.0, 24.2, 23.8, 22.7, 22.4, 21.0, 19.3, 18.6, 11.8; $[\alpha]_D^{20}$ +34 (*c* 1.4 in CH_2Cl_2/MeOH, 9:1); *m/z* (HRMS) 734.5552 ($M + H^+$, $C_{43}H_{76}O_8N$ requires 734.5565).

***N*-{10-[(3β)-cholest-5-en-3-yloxy]decyl}-2-(α-D-glucopyranosyloxy) acetamide (16b):** (59 mg, 0.09 mmol, 91% yield); δ_H (300 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.28 (1 H, m, H-6'), 4.72 (1 H, d, *J* 3.7, H-1), 4.09 (1 H, d, *J* 15.9, H-7a), 3.91(1 H, d, *J* 15.9, H-7b), 3.74-3.60 (3 H, m, H-6a, H-6b, H-3), 3.57-3.32 (5 H, m, H-2, H-4, H-5, CH_2-18), 3.30 (1 H, m, NH), 3.22-3.01 (3 H, m, CH_2-9, H-3'), 2.32-2.03 (2 H, m, H4'), 1.96-0.77 (59 H, m, H-Cholesterol, CH_2-10 to CH_2-17), 0.60 (3 H, s, CH_3-18'); δ_C (75 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 170.0, 140.9, 121.5, 99.4, 79.1, 73.5, 72.3, 72.2, 71.6, 69.9, 68.2, 66.7, 61.4, 56.7, 56.1, 50.1, 42.3, 39.8, 39.7, 39.5, 39.5, 39.1, 39.0, 37.2, 36.8, 36.1, 31.9, 31.8, 29.5, 29.4, 29.2, 28.2, 27.9, 26.9, 26.1, 24.3, 24.2, 23.8, 22.7, 22.5, 21.0, 19.3, 18.6, 11.8; $[\alpha]_D^{20}$ +29 (*c* 0.7 in CH_2Cl_2/MeOH, 9/1); *m/z* (HRMS) 762.5870 ($M + H^+$, $C_{45}H_{80}NO_8$ requires 762.5878).

***N*-{12-[(3β)-cholest-5-en-3-yloxy]dodecyl}-2-(α-D-glucopyranosyloxy) acetamide (17b):** (63 mg, 0.08 mmol, 81% yield); δ_H (300 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.27 (1 H, m, H-6'), 4.72 (1 H, d, *J* 3.7, H-1), 4.09 (1 H, d, *J* 15.8, H-7a), 3.91(1 H, d, *J* 15.9, H-7b), 3.72-3.60 (3 H, m, H-6a, H-6b, H-3), 3.57-3.32 (5 H, m, H-2, H-4, H-5, CH_2-20), 3.30 (1 H, m, NH), 3.25 (2 H, t, *J* 7.3, CH_2-9), 3.22-3.01 (3 H, m, CH_2-9, H-3'), 2.32-2.03 (2 H, m, H4'), 1.96-0.77 (63 H, m, H-Cholesterol, CH_2-10 to CH_2-19), 0.60 (3 H, s, CH_3-18'); δ_C (75 MHz; $CDCl_3$; Me_4Si) 170.0, 140.9, 121.5, 99.4, 79.0, 73.5, 72.3, 71.6, 69.9, 68.2, 66.7, 66.7, 61.3, 56.7, 56.1, 50.1, 42.3, 39.7, 39.4, 39.1, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 30.0, 29.5, 29.5, 29.4, 29.3, 29.2, 28.3, 28.2, 27.9, 26.9, 26.1, 24.2, 23.8, 22.7, 22.4, 21.0, 19.3, 18.6, 11.8; $[\alpha]_D^{20}$ +30 (*c* 0.8 in CH_2Cl_2/MeOH, 9:1); *m/z* (HRMS) 790.6182 ($M + H^+$, $C_{47}H_{84}NO_8$ requires 790.6191).

2-({2-*O*-[(hexylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{6-[(3β)-cholest-5-en-3-yloxy]hexyl}acetamide (18a): (41 mg, 0.05 mmol, 50% yield); (Found: C 68.60, H 10.45, N 3.25. $C_{48}H_{84}N_2O_9 \bullet 0.5H_2O$ requires C 68.45, H 10.17, N 3.33); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.35 (1 H, m, H-6'), 5.01 (1 H, d, *J* 3.7, H-1), 4.55 (1 H, dd, *J* 3.6, 10.2, H-2), 4.14 (1 H, d, *J* 15.6 , H-7a), 4.00 (1 H, d, *J* 15.6, H-7b), 3.90-3.75 (3 H, m, H-6a, H-6b, H-3), 3.61-3.52 (4 H, m, H-5, H-4, CH_2-14), 3.36 (1 H, m, NH), 3.25 (2 H, t, *J* 7.3, CH_2-9), 3.19-3.04 (3 H, m, CH_2-16, H-3'), 2.40-2.10 (2 H, m, H-4'), 2.08-0.80 (57 H, m, H-Cholesterol, CH_2-10 to CH_2-13, CH_2-17 to CH_3-21), 0.68 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.5, 156.4, 140.9, 121.7, 97.3, 79.2, 73.5, 72.3, 71.3, 70.3, 70.3, 68.0, 66.6, 61.4, 56.8, 56.2, 50.3, 42.4, 41.2, 39.8, 39.6, 39.1, 39.1, 37.3, 36.9, 36.2, 35.8, 32.0, 31.9, 31.5, 30.0, 29.7, 29.5, 28.5, 28.3, 28.1, 26.8, 26.5, 25.9, 24.3, 23.9, 22.8, 22.6, 22.6, 21.1, 19.4, 18.7, 14.0, 11.9; $[\alpha]_D^{20}$ +36 (*c* 0.7 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 833.6 ($M+H^+$), 855.6 ($M+Na^+$).

2-({2-*O*-[(octylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{6-[(3β)-cholest-5-en-3-yloxy]hexyl}acetamide (18b): (52 mg, 0.06 mmol, 61% yield); (Found: C 69.12, H 10.56, N 3.07. $C_{50}H_{88}N_2O_9 \bullet 0.5H_2O$ requires C 69.01, H 10.31, N 3.22); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.35 (1 H, m, H-6'), 5.01 (1 H, d, *J* 3.7, H-1), 4.55 (1 H, dd, *J* 3.6, 10.1, H-2), 4.14 (1 H, d, *J* 15.6, H-7a), 3.98 (1 H, d, *J* 15.6, H-7b), 3.88-3.74 (3 H, m, H-6a, H-6b, H-3), 3.61-3.52 (4 H, m, H-5, H-4, CH_2-14), 3.36 (1 H, m, NH), 3.25 (2 H, t, *J* 7.3, CH_2-9), 3.19-3.04 (3 H, m, CH_2-16, H-3'), 2.40-2.10 (2 H, m, H-4'), 2.08-0.80 (61 H, m, H-Cholesterol, CH_2-10 to CH_2-13, CH_2-17 to CH_3-23), 0.68 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9/1; Me_4Si) 169.5, 156.3, 140.8, 121.6, 97.2, 79.1, 73.5, 72.3, 71.2, 70.1, 67.9, 66.5, 61.3, 56.7, 56.1, 50.2, 42.3, 41.1, 39.7, 39.5, 39.0, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 31.7, 29.9, 29.7, 29.3, 29.2, 29.2, 28.3, 28.2, 27.9, 26.8, 26.7, 25.8, 24.2, 23.8, 22.7, 22.6, 22.4, 21.0, 19.3, 18.6, 14.0, 11.8; $[\alpha]_D^{20}$ +33 (*c* 0.7 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 861.7 ($M+H^+$), 883.7 ($M+Na^+$).

2-({2-*O*-[(dodecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{6-[(3β)-cholest-5-en-3-yloxy]hexyl}acetamide (18c): (80 mg, 0.09 mmol, 87% yield); (Found: C 70.49, H 10.76, N 2.96. $C_{54}H_{96}N_2O_9$ requires C 70.70, H 10.55, N 3.05); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.35 (1 H, m, H-6'), 5.01 (1 H, d, *J* 3.6, H-1), 4.55 (1 H, dd, *J* 3.6, 10.0, H-2), 4.13 (1 H, d, *J* 15.6 , H-7a), 3.98 (1 H, d, *J* 15.6, H-7b), 3.88-3.74 (3 H, m, H-6a, H-6b, H-3), 3.61-3.44 (4 H, m, H-5, H-4, CH_2-14), 3.38 (1 H, m, NH), 3.25 (2 H, t, *J* 7.3, CH_2-9), 3.19-3.04 (3 H, m, CH_2-16, H-3'), 2.40-2.10 (2 H, m, H-4'), 2.08-0.80 (69 H, m, H-Cholesterol, CH_2-10 to CH_2-13, CH_2-17 to CH_3-27), 0.68 (3 H, s, $CH_3$18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.5, 156.3, 140.8, 121.6, 97.2, 79.1, 73.5, 72.3, 71.2, 70.2, 67.9, 66.5, 61.3, 56.7, 56.1, 50.2, 42.3, 41.1, 39.7, 39.5, 39.0, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 29.9, 29.7, 29.7, 29.6, 29.6, 29.6, 29.4, 29.3, 28.4, 28.2, 28.0, 26.8, 26.7, 25.8, 24.2, 23.8, 22.7, 22.6, 22.4, 21.0, 19.3, 18.6, 14.0, 11.8; $[\alpha]_D^{20}$ +34 (*c* 0.7 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 917.7 ($M+H^+$), 939.6 ($M+Na^+$).

2-({2-*O*-[(tetradecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{6-[(3β)-cholest-5-en-3-yloxy]hexyl}acetamide (18d): (49 mg, 0.05 mmol, 52% yield); (Found: C, 70.41; H, 10.59; N, 2.91. $C_{56}H_{100}N_2O_9 \bullet 0.5H_2O$ requires C, 70.47; H, 10.67; N, 2.94); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.21 (1 H, m, H-6'), 4.86 (1 H, d, *J* 3.5, H-1), 4.39 (1 H, dd, *J* 3.4, 10.0, H-2), 4.00 (1 H, d, *J* 15.5, H-7a), 3.83 (1 H, d, *J* 15.6, H-7b), 3.74-3.60 (3 H, m, H-6a, H-6b, H-3), 3.50-3.30 (4 H, m, H-5, H-4, CH_2-14), 3.23 (1 H, s, NH), 3.11 (2 H, t, *J* 7.3, CH_2-9), 3.08-2.92 (3 H, m, CH_2-16, H-3'), 2.30-2.00 (2 H, m, H4'), 1.96-0.77(73 H, m, H-cholesterol, CH_2-10 to CH_2-13, CH_2-17 to CH_3-29), 0.54 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9/1; Me_4Si) 169.5, 156.3, 140.7, 121.6, 97.2, 79.1, 73.4, 72.3, 71.2, 70.1, 67.9, 66.4, 61.2, 56.7, 56.1, 50.1, 42.3, 41.1, 39.7, 39.4, 39.0, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 29.9, 29.7, 29.6, 29.6, 29.6, 29.3, 28.3, 28.2, 27.9, 26.8, 26.7, 25.8, 24.2, 23.7, 22.7, 22.6, 22.4, 21.0, 19.3, 18.6, 14.0, 11.7; $[\alpha]_D^{20}$ +29 (*c* 0.9 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 946.5 ($M+H^+$), 968.5 ($M+Na^+$).

2-({2-*O*-[(hexadecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{6-[(3β)-cholest-5-en-3-yloxy]hexyl}acetamide (18e): (51 mg, 0.05 mmol, 53% yield); (Found: C, 69.05; H, 10.51; N, 2.65. $C_{58}H_{104}N_2O_9 \bullet 2H_2O$ requires C, 69.01; H, 10.78; N, 2.78); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.21 (1 H, m, H-6'), 4.86 (1 H, d, *J* 3.5, H-1), 4.39 (1 H, dd, *J* 3.4, 10.0, H-2), 4.04 (1 H, d, *J* 15.6, H-7a), 3.88 (1 H, d, *J* 15.6, H-7b), 3.74-3.60 (3 H, m, H-6a, H-6b, H-3), 3.50-3.30 (4 H, m, H-5, H-4, CH_2-14), 3.23 (1H, s, NH), 3.11 (2 H, t, *J* 7.3, CH_2-9), 3.04-2.92 (3 H, m, CH_2-16, H-3'), 2.30-2.00 (2 H, m, H-4'), 1.96-0.77 (77 H, m, H-Cholesterol, CH_2-10 to CH_2-13, CH_2-17 to CH_3-31), 0.54 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.5, 156.3, 140.7, 121.6, 97.1, 79.1, 73.4, 72.3, 71.2, 70.0, 67.9, 66.4, 61.2, 56.7, 56.1, 50.1, 42.2, 41.1, 39.7, 39.4, 39.0, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 29.9, 29.7, 29.7, 29.6, 29.6, 29.3, 28.3, 28.2, 27.9, 26.8, 26.7, 25.8, 24.2, 23.7, 22.7, 22.6, 22.4, 21.0, 19.3, 18.6, 14.0, 11.8; $[\alpha]_D^{20}$ +27 (*c* 1 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 973.8 ($M+H^+$), 995.8 ($M+Na^+$).

2-({2-*O*-[(octadecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{6-[(3β)-cholest-5-en-3-yloxy]hexyl}acetamide (18f): (50 mg, 0.05 mmol, 50% yield); (Found: C, 70.86; H, 10.73; N, 2.66. $C_{60}H_{108}N_2O_9 \bullet H_2O$ requires C, 70.68; H, 10.80; N, 2.75); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.23 (1 H, m, H-6'), 4.86 (1 H, d, *J* 3.5, H-1), 4.40 (1 H, dd, *J* 3.4, 10.1, H-2), 3.98 (1 H, d, *J* 15.6, H-7a), 3.82 (1 H, d, *J* 15.6, H-7b), 3.74-3.60 (3 H, m, H-6a, H-6b, H-3) 3.50-3.30 (4 H, m, H-5, H-4, CH_2-14), 3.23 (1 H, s, NH), 3.11 (2 H, t, *J* 7.3, CH_2-9), 3.05-2.90 (3 H, m, CH_2-16, H-3'), 2.30-2.00 (2 H, m, H-4'), 1.96-0.77 (81 H, m, H-Cholesterol, CH_2-10 to CH_2-13, CH_2-17 to CH_3-33), 0.53 (3 H, s, CH_3-18');

δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.5, 156.3, 140.7, 121.6, 97.2, 79.1, 73.4, 72.3, 71.2, 70.1, 67.9, 66.4, 61.2, 56.7, 56.1, 50.1, 42.3, 41.1, 39.7, 39.4, 39.0, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 29.9, 29.7, 29.7, 29.6, 29.6, 29.5, 29.5, 29.3, 29.3, 28.3, 28.2, 27.9, 26.8, 26.7, 25.8, 24.2, 23.8, 22.7, 22.6, 22.4, 21.0, 19.3, 18.6, 14.0, 11.8; $[\alpha]_D^{20}$ +30 (*c* 0.5 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 1001.8 ($M+H^+$), 1023.8 ($M + Na^+$).

2-({2-*O*-[(hexylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{8-[(3β)-cholest-5-en-3-yloxy]octyl}acetamide (19a): (53 mg, 0.06 mmol, 61% yield); (Found: C 68.27, H 10.47, N 3.03. $C_{50}H_{88}N_2O_9 \bullet H_2O$ requires C 68.30, H 10.32, N 3.19); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.35 (1 H, m, H-6'), 5.01 (1 H, d, *J* 3.6, H-1), 4.55 (1 H, dd, *J* 3.6, 10.2, H-2), 4.13 (1 H, d, *J* 15.6, H-7a), 3.91(1 H, d, *J* 15.6, H-7b), 3.87-3.74 (3 H, m, H-6a, H-6b, H-3), 3.61-3.52 (4 H, m, H-5, H-4, CH_2-16), 3.36 (1 H, m, NH), 3.25 (2 H, t, *J* 7.3, CH_2-9), 3.19-3.04 (3 H, m, CH_2-18, H-3'), 2.39-2.13 (2 H, m, H-4'), 2.08-0.80 (61 H, m, H-Cholesterol, CH_2-10 to CH_2-15, CH_2-19 to CH_3-23), 0.68 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.5, 156.3, 140.8, 121.6, 97.1, 79.1, 73.4, 72.3, 71.2, 70.0, 68.1, 66.4, 61.2, 56.7, 56.1, 50.1, 42.3, 41.0, 39.7, 39.4, 39.1, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 31.4, 30.0, 29.6, 29.4, 29.4, 29.2, 28.3, 28.2, 27.9, 26.8, 26.4, 26.0, 24.2, 23.7, 22.7, 22.5, 22.4, 21.0, 19.3, 18.6, 13.9, 11.8; $[\alpha]_D^{20}$ +34 (*c* 0.6 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 861.7 ($M+H^+$), 883.6 ($M+Na^+$).

2-({2-*O*-[(octylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{8-[(3β)-cholest-5-en-3-yloxy]octyl}acetamide (19b): (54 mg, 0.06 mmol, 61% yield); (Found: C 68.93, H 10.46, N 3.06. $C_{52}H_{92}N_2O_9 \bullet H_2O$ requires C 68.84, H 10.44, N 3.09); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.35 (1 H, m, H-6'), 5.01 (1 H, d, *J* 3.6, H-1), 4.53 (1 H, dd, *J* 3.4, 10.0, H-2), 4.13 (1 H, d, *J* 15.6, H-7a), 3.91(1 H, d, *J* 15.6, H-7b), 3.88-3.73 (3 H, m, H-6a, H-6b, H-3), 3.61-3.44 (4 H, m, H-5, H-4, CH_2-16), 3.38 (1 H, m, NH), 3.25 (2 H, t, *J* 7.2, CH_2-9), 3.19-3.04 (3 H, m, CH_2-18, H-3'), 2.39-2.13 (2 H, m, H-4'), 2.08-0.80 (65 H, m, H-Cholesterol, CH_2-10 to CH_2-15, CH_2-19 to CH_3-23), 0.68 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.4, 156.3, 140.9, 121.6, 97.2, 79.1, 73.5, 72.3, 71.2, 70.1, 68.1, 66.4, 61.3, 56.7, 56.1, 50.2, 42.3, 41.1, 39.7, 39.5, 39.1, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 31.7, 30.0, 29.7, 29.5, 29.4, 29.2, 29.2, 28.4, 28.2, 27.9, 26.8, 26.8, 26.1, 24.2, 23.8, 22.7, 22.6, 22.4, 21.0, 19.3, 18.6, 14.0, 11.8; $[\alpha]_D^{20}$ +36 (*c* 0.7 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 889.7 ($M+H^+$), 911.7 ($M+Na^+$).

2-({2-*O*-[(dodecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{8-[(3β)-cholest-5-en-3-yloxy]octyl}acetamide (19c): (63 mg, 0.07 mmol, 67% yield); (Found: C 71.14, H 10.66, N 2.96. $C_{56}H_{100}N_2O_9$ requires C 71.14, H 10.66, N 2.96); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.35 (1 H, m, H-6'), 5.01 (1 H, d, *J* 3.6, H-1), 4.55 (1 H, dd, *J* 3.6, 10.1, H-2), 4.13 (1 H, d, *J* 15.6, H-7a), 3.96 (1 H, d, *J* 15.6, H-7b), 3.87-3.74 (3 H, m, H-6a, H-6b, H-3), 3.61-3.52 (4 H, m, H-5, H-4, CH_2-16), 3.36 (1 H, m, NH), 3.25 (2 H, t, *J* 7.4, CH_2-9), 3.19-3.04 (3 H, m, CH_2-18, H-3'), 2.39-2.13 (2 H, m, H-4'), 2.08-0.80 (73 H, m, H-Cholesterol, CH_2-10 to CH_2-15, CH_2-19 to CH_3-27), 0.68 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.5, 156.3, 140.8, 121.6, 97.1, 79.1, 73.4, 72.3, 71.2, 70.0, 68.1, 66.4, 61.2, 56.7, 56.1, 50.1, 42.3, 41.0, 39.7, 39.4, 39.1, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 31.4, 30.0, 29.6, 29.4, 29.4, 29.2, 28.3, 28.2, 27.9, 26.8, 26.4, 26.0, 24.2, 23.7, 22.7, 22.5, 22.4, 21.0, 19.3, 18.6, 13.9, 11.8; $[\alpha]_D^{20}$ +35 (*c* 0.7 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 945.7 ($M+H^+$), 967.7 ($M+Na^+$).

2-({2-*O*-[(tetradecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{8-[(3β)-cholest-5-en-3-yloxy]octyl}acetamide (19d): (51 mg, 0.05 mmol, 52% yield); (Found: C, 70.66; H, 10.71; N, 2.71. $C_{58}H_{104}N_2O_9 \bullet H_2O$ requires C, 70.86; H, 10.78; N, 2.83); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.21 (1 H, m, H-6'), 4.86 (1 H, d, *J* 3.5, H-1), 4.40 (1 H, dd, *J* 3.4, 10.1, H-2), 4.00 (1 H, d, *J* 15.6, H-7a), 3.83 (1 H, d, *J* 15.6, H-7b), 3.74-3.60 (3 H, m, H-6a, H-6b, H-3), 3.50-3.30 (4 H, m, H-5, H-4, CH_2-16), 3.23 (1 H, s, NH), 3.11 (2 H, t, *J* 7.3, CH_2-9), 3.06-2.93 (3 H, m, CH_2-18, H-3'), 2.26-2.00 (2 H, m, H4'), 1.96-0.77 (77 H, m, H-Cholesterol, CH_2-10 to CH_2-15, CH_2-19 to CH_3-31), 0.54 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.5, 156.2, 140.8, 121.5, 97.1, 79.1, 73.4, 72.3, 71.2, 70.1, 68.1, 66.4, 61.2, 56.7, 56.1, 50.1, 42.3, 41.1, 39.7, 39.4, 39.1, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 30.0, 29.7, 29.6, 29.6, 29.6, 29.6, 29.5, 29.4, 29.4, 29.3, 29.2, 28.3, 28.2, 27.9, 26.8, 26.8, 26.0, 24.2, 23.7, 22.7, 22.6, 22.4, 21.0, 19.3, 18.6, 14.0, 11.8; $[\alpha]_D^{20}$ +30 (*c* 0.9 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 973.8 ($M+H^+$), 995.8 ($M+Na^+$).

2-({2-*O*-[(hexadecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{8-[(3β)-cholest-5-en-3-yloxy]octyl}acetamide (19e): (41 mg, 0.04 mmol, 41% yield); (Found: C, 70.42; H, 10.70; N, 2.68. $C_{60}H_{108}N_2O_9 \bullet H_2O$ requires C, 70.68; H, 10.88; N, 2.75); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.23 (1 H, m, H-6'), 4.86 (1 H, d, *J* 3.5, H-1), 4.40 (1 H, dd, *J* 3.4, 10.1, H-2), 3.98 (1 H, d, *J* 15.6, H-7a), 3.83 (1 H, d, *J* 15.6, H-7b), 3.74-3.60 (3 H, m, H-6a, H-6b, H-3), 3.50-3.30 (4 H, m, H-5, H-4, CH_2-16), 3.23 (1 H, s, NH), 3.11 (2 H, t, *J* 7.3, CH_2-9), 3.05-2.93 (3 H, m, CH_2-18, H-3'), 2.25-2.00 (2 H, m, H-4'), 1.96-0.67 (81 H, m, H-Cholesterol, CH_2-10 to CH_2-15, CH_2-19 to CH_3-33), 0.54 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.5, 156.2, 140.8, 121.6, 97.1, 79.1, 73.4, 72.3, 71.2, 70.1, 68.1, 66.4, 61.2, 56.7, 56.1, 53.4, 50.2, 42.3, 41.1, 39.7, 39.4, 39.1, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 30.0, 29.7, 29.7, 29.6, 29.6, 29.5, 29.5, 29.4, 29.4, 29.3, 29.2, 28.3, 28.2, 27.9, 26.8, 26.8, 26.0, 24.2, 23.7, 22.7, 22.6, 22.4, 22.3, 21.0, 19.3, 18.6, 14.0, 13.9, 11.8; $[\alpha]_D^{20}$ +26 (*c* 1 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 1001.8 ($M+H^+$), 1023.8 ($M + Na^+$).

2-({2-*O*-[(octadecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{8-[(3β)-cholest-5-en-3-yloxy]octyl}acetamide (19f): (54 mg, 0.05 mmol, 52% yield); (Found: C, 71.72; H, 10.87; N, 2.61. $C_{62}H_{112}N_2O_9 \bullet 0.5H_2O$ requires C, 71.70; H, 10.97; N, 2.70); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.23 (1 H, m, H-6'), 4.86 (1 H, d, *J* 3.6, H-1), 4.40 (1 H, dd, *J* 3.6, 10.1, H-2), 4.00 (1 H, d, *J* 15.6, H-7a), 3.83 (1 H, d, *J* 15.3, H-7b), 3.74-3.60 (3 H, m, H-6a, H-6b, H-3) 3.50-3.30 (4 H, m, H-5, H-4, CH_2-16), 3.23 (1 H, s, NH), 3.11 (2 H, t, *J* 7.3, CH_2-9), 3.05-2.90 (3 H, m, CH_2-18, H-3'), 2.30-2.00 (2 H, m, H-4'), 1.96-0.67 (85 H, m, H-Cholesterol, CH_2-10 to CH_2-15, CH_2-19 to CH_3-35), 0.53 (3 H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.5, 156.2, 140.8, 121.5, 97.1, 79.1, 73.5, 73.4, 72.3, 71.2, 70.1, 68.1, 66.4, 61.2, 56.7, 56.1, 50.2, 42.3, 41.1, 39.7, 39.4, 39.1, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 30.0, 29.7, 29.6, 29.6, 29.4, 29.4, 29.3, 29.2, 28.3, 28.2, 27.9, 26.8, 26.8, 26.1, 24.2, 23.7, 22.7, 22.6, 22.4, 21.0, 19.3, 18.6, 14.0, 11.8; $[\alpha]_D^{20}$ +29 (*c* 0.9 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 1029.8 ($M+H^+$), 1051.8 ($M + Na^+$).

2-({2-*O*-[(hexylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{10-[(3β)-cholest-5-en-3-yloxy]decyl}acetamide (20a): (0.075mmol, 75% yield); (Found: C 70.22, H 10.78, N 3.00. $C_{52}H_{92}N_2O_9$ requires C 70.23, H 10.43, N 3.15); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.35 (1H, m, H-6'), 5.02 (1H, d, *J* 3.6, H-1), 4.55 (1H, dd, *J* 3.5, 10.1, H-2), 4.15 (1H, d, *J* 15.6, H-7a), 3.98 (1H, d, *J* 15.6, H-7b), 3.89-3.75 (3H, m, H-6a, H-6b, H-3), 3.61-3.52 (4H, m, H-5, H-4, CH_2-18), 3.25 (2H, t, *J* 7.2, CH_2-9), 3.19-3.04 (3H, m, CH_2-20, H-3'), 2.40-2.10 (2H, m, H-4'), 2.08-0.80 (67H, m, H-Cholesterol, CH_2-10 to CH_2-17, CH_2-21 to CH_3-23), 0.69 (3H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.5, 156.3, 140.9, 121.5, 97.1, 79.0, 73.4, 72.3,

71.2, 70.0, 68.1, 66.4, 61.2, 56.7, 56.1, 50.2, 42.3, 41.0, 39.7, 39.4, 39.1, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 31.4, 30.0, 29.6, 29.5, 29.5, 29.5, 29.4, 29.3, 29.3, 28.3, 28.2, 27.9, 26.9, 26.4, 26.1, 24.2, 23.7, 22.7, 22.5, 22.4, 21.0, 19.3, 18.6, 13.9, 11.8; $[\alpha]_D^{20}$ +34 (*c* 0.7 in CH_2Cl_2/MeOH, 9:1); *m/z* (HRMS) 889.6868 ($M + H^+$, $C_{52}H_{93}N_2O_9$ requires 889.6876).

2-({2-*O*-[(octylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{10-[(3β)-cholest-5-en-3-yloxy]decyl}acetamide (20b): (0.066 mmol, 66% yield); (Found: C 69.48, H 10.65, N 2.95. $C_{54}H_{96}N_2O_9 \bullet H_2O$ requires C 69.34, H 10.56, N 2.99); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.35 (1H, m, H-6'), 5.01 (1H, d, *J* 3.5, H-1), 4.55 (1H, dd, *J* 3.3, 10.1, H-2), 4.13 (1H, d, *J* 15.7, H-7a), 3.97 (1H, d, *J* 15.6, H-7b), 3.89-3.71 (3H, m, H-6a, H-6b, H-3), 3.61-3.52 (4H, m, H-4, H-5, CH_2-18), 3.25 (2H, t, *J* 7.3, CH_2-9), 3.19-3.04 (3H, m, CH_2-20, H-3'), 2.40-2.10 (2H, m, H-4'), 2.08-0.80 (71H, m, H-Cholesterol, CH_2-10 to CH_2-17, CH_2-21 to CH_3-25), 0.68 (3H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.6, 156.4, 141.0, 121.6, 97.3, 79.2, 73.6, 72.4, 71.3, 70.3, 68.3, 66.6, 61.4, 56.9, 56.2, 50.3, 42.4, 41.2, 39.9, 39.6, 39.2, 39.2, 37.3, 37.0, 36.3, 35.9, 32.0, 32.0, 31.9, 30.1, 29.8, 29.6, 29.6, 29.6, 29.4, 29.3, 29.3, 28.5, 28.3, 28.1, 27.0, 26.9, 26.2, 24.4, 23.9, 22.8, 22.7, 22.6, 21.1, 19.4, 18.8, 14.1, 11.9; $[\alpha]_D^{20}$ +30 (*c* 0.8 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 917.7 ($M+H^+$), 939.7 ($M+Na^+$).

2-({2-*O*-[(dodecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{10-[(3β)-cholest-5-en-3-yloxy]decyl}acetamide (20c): (0.08mmol, 80% yield); (Found: C 71.37, H 10.96, N 2.71. $C_{58}H_{104}N_2O_9$ requires C 71.56, H 10.77, N 2.88); δ_H (400 MHz; $CDCl_3$/MeOD, 9/1; Me_4Si) 5.35 (1H, m, H-6'), 5.01 (1H, d, *J* 3.6, H-1), 4.55 (1H, dd, *J* 3.4, 10.0, H-2), 4.13 (1H, d, *J* 15.6, H-7a), 3.97 (1H, d, *J* 15.6, H-7b), 3.88-3.74 (3H, m, H-6a, H-6b, H-3), 3.61-3.52 (4H, m, H-4, H-5, CH_2-18), 3.25 (2H, t, *J* 7.4, CH_2-9), 3.19-3.04 (3H, m, CH_2-20, H-3'), 2.40-2.10 (2H, m, H-4'), 2.08-0.80 (79H, m, H-Cholesterol, CH_2-10 to CH_2-17, CH_2-21 to CH_3-29), 0.68 (3H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9/1; Me_4Si) 169.6, 156.4, 141.0, 121.7, 97.3, 79.2, 73.6, 72.4, 71.4, 70.3, 68.3, 66.6, 61.4, 56.9, 56.2, 50.3, 42.4, 41.2, 39.9, 39.6, 39.2, 39.2, 37.3, 37.0, 36.3, 35.9, 32.0, 32.0, 30.2, 29.8, 29.7, 29.7, 29.7, 29.6, 29.6, 29.6, 29.4, 28.5, 28.3, 28.1, 27.0, 26.9, 26.2, 24.4, 23.9, 22.8, 22.7, 22.6, 21.1, 19.4, 18.8, 14.1, 11.9; $[\alpha]_D^{20}$+31(*c* 0.8 in CH_2Cl_2/MeOH, 9/1); *m/z* (ESI) 973.8 ($M+H^+$), 995.8 ($M+Na^+$).

2-({2-*O*-[(hexylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{12-[(3β)-cholest-5-en-3-yloxy]dodecyl}acetamide (21a): (0.069mmol, 69% yield); (Found: C 70.67, H 10.73, N 2.88. $C_{54}H_{96}N_2O_9$ requires C 70.70, H 10.55, N 3.05); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.35 (1H, m, H-6'), 5.01 (1H, d, *J* 3.7, H-1), 4.54 (1H, dd, *J* 3.5, 10.1, H-2), 4.13 (1H, d, *J* 15.6, H-7a), 3.98 (1H, d, *J* 15.6, H-7b), 3.88-3.74 (3H, m, H-6a, H-6b, H-3), 3.61-3.52 (4H, m, H-5, H-4, CH_2-20), 3.25 (2H, t, *J* 7.5, CH_2-9), 3.19-3.08 (3H, m, CH_2-22, H-3'), 2.39-2.14 (2H, m, H-4'), 2.08-0.80 (71H, m, H-Cholesterol, CH_2-10 to CH_2-19, CH_2-23 to CH_3-27), 0.68 (3H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.6, 156.4, 141.0, 121.6, 97.3, 79.2, 73.6, 72.4, 71.4, 70.2, 68.3, 66.6, 61.4, 56.9, 56.2, 50.3, 42.4, 41.2, 39.9, 39.6, 39.2, 39.2, 37.3, 37.0, 36.3, 35.9, 32.0, 32.0, 31.9, 31.5, 30.1, 29.8, 29.8, 29.7, 29.6, 29.6, 29.4, 29.3, 29.3, 28.5, 28.3, 28.1, 27.0, 26.9, 26.5, 26.2, 24.4, 23.9, 22.8, 22.7, 22.6, 22.6, 21.1, 19.4, 18.8, 14.1, 14.0, 11.9; $[\alpha]_D^{20}$ +31 (*c* 0.7 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 917.7 ($M+H^+$), 939.7 ($M+Na^+$).

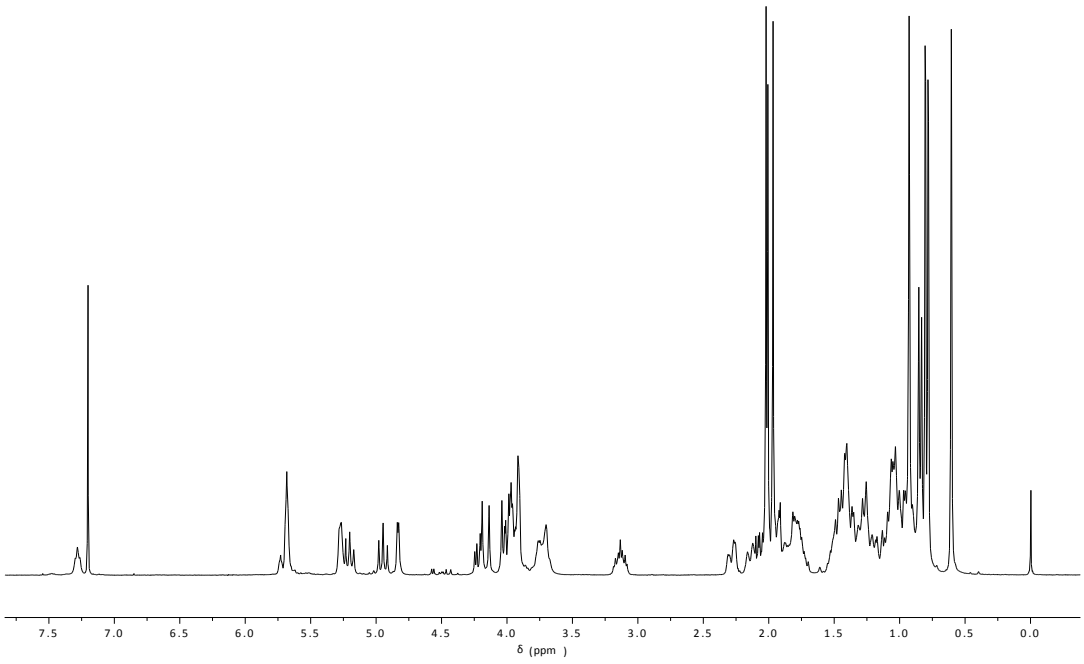
2-({2-*O*-[(octylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{12-[(3β)-cholest-5-en-3-yloxy]dodecyl}acetamide (21b): (0.070 mmol, 70% yield); (Found: C 69.84, H 10.85, N 2.83. $C_{56}H_{100}N_2O_9 \bullet H_2O$ requires C 69.81, H 10.67, N 2.91); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.34 (1H, m, H-6'), 5.00 (1H, d, *J* 3.6, H-1), 4.52 (1H, dd, *J* 3.6, 10.1, H-2), 4.12 (1H, d, *J* 15.6, H-7a), 3.96 (1H, d, *J* 15.6, H-7b), 3.87-3.73 (3H, m, H-6a, H-6b, H-3), 3.59-3.50 (4H, m, H-5, H-4, CH_2-20), 3.23 (2H, m, CH_2-9), 3.17-3.06 (3H, m, CH_2-22, H-3'), 2.37-2.12 (2H, m, H-4'), 2.06-0.78 (75H, m, H-Cholesterol, CH_2-10 to CH_2-19, CH_2-23 to CH_3-29), 0.66 (3H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.6, 156.3, 140.9, 121.5, 97.1, 79.0, 73.4, 72.3, 71.2, 70.1, 68.2, 66.4, 61.2, 56.7, 56.1, 50.2, 42.3, 41.1, 39.7, 39.4, 39.1, 39.0, 37.2, 36.8, 36.1, 35.7, 31.9, 31.8, 31.7, 30.0, 29.7, 29.6, 29.6, 29.5, 29.5, 29.4, 29.3, 29.2, 29.2, 28.3, 28.2, 27.9, 26.9, 26.8, 26.1, 24.2, 23.7, 22.7, 22.5, 22.4, 21.0, 19.3, 18.6, 13.9, 11.8; $[\alpha]_D^{20}$+31 (*c* 0.7 in CH_2Cl_2/MeOH, 9:1); *m/z* (HRMS) 967.7290 ($M + Na^+$, $C_{56}H_{100}N_2O_9Na$ requires 967.7321)

2-({2-*O*-[(dodecylamino)carbonyl]-α-D-glucopyranosyl}oxy)-*N*-{12-[(3β)-cholest-5-en-3-yloxy]dodecyl}acetamide (21c): (0.067mmol, 67% yield); (Found: C 71.15, H 11.12, N 2.64. $C_{60}H_{108}N_2O_9 \bullet 0.5H_2O$ requires C 71.31, H 10.87, N 2.77); δ_H (400 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 5.36 (1H, m, H-6'), 5.02 (1H, d, *J* 3.6, H-1), 4.55 (1H, dd, *J* 3.5, 10.1, H-2), 4.13 (1H, d, *J* 15.6, H-7a), 3.98 (1H, d, *J* 15.6, H-7b), 3.88-3.74 (3H, m, H-6a, H-6b, H-3), 3.61-3.52 (4H, m, H-5, H-4, CH_2-20), 3.25 (2H, t, *J* 7.5, CH_2-9), 3.19-3.08 (3H, m, CH_2-22, H-3'), 2.39-2.14 (2H, m, H-4'), 2.08-0.80 (83H, m, H-Cholesterol, CH_2-10 to CH_2-19, CH_2-23 to CH_3-33), 0.68 (3H, s, CH_3-18'); δ_C (100 MHz; $CDCl_3$/MeOD, 9:1; Me_4Si) 169.4, 156.2, 141.0, 121.5, 97.2, 79.1, 73.5, 72.2, 71.3, 70.2, 68.2, 66.5, 61.3, 56.8, 56.2, 50.2, 42.3, 41.1, 39.8, 39.5, 39.1, 37.3, 36.9, 36.2, 35.8, 31.9, 31.9, 30.1, 29.7, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 28.4, 28.2, 28.0, 26.9, 26.8, 26.1, 24.3, 23.8, 22.7, 22.6, 22.5, 21.0, 19.3, 18.7, 14.0, 11.8; $[\alpha]_D^{20}$ +25 (*c* 0.8 in CH_2Cl_2/MeOH, 9:1); *m/z* (ESI) 1001.8 ($M+H^+$).

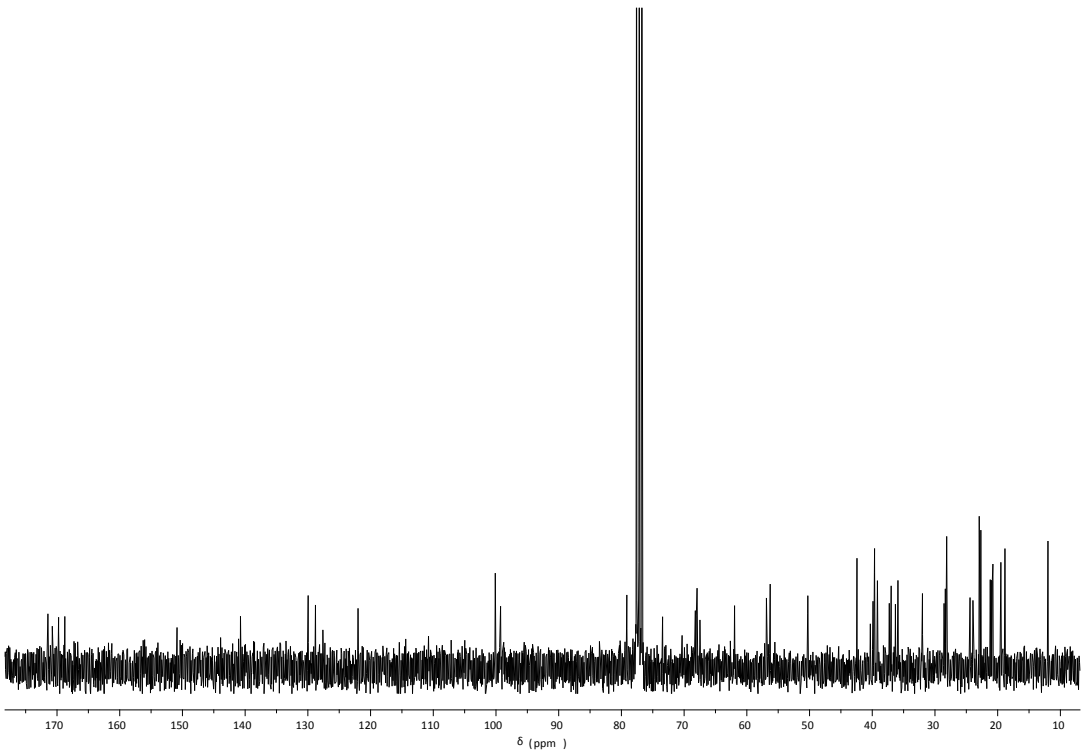
2. 1H and 13 C nmr spectra

Compound 3

^{1}H NMR

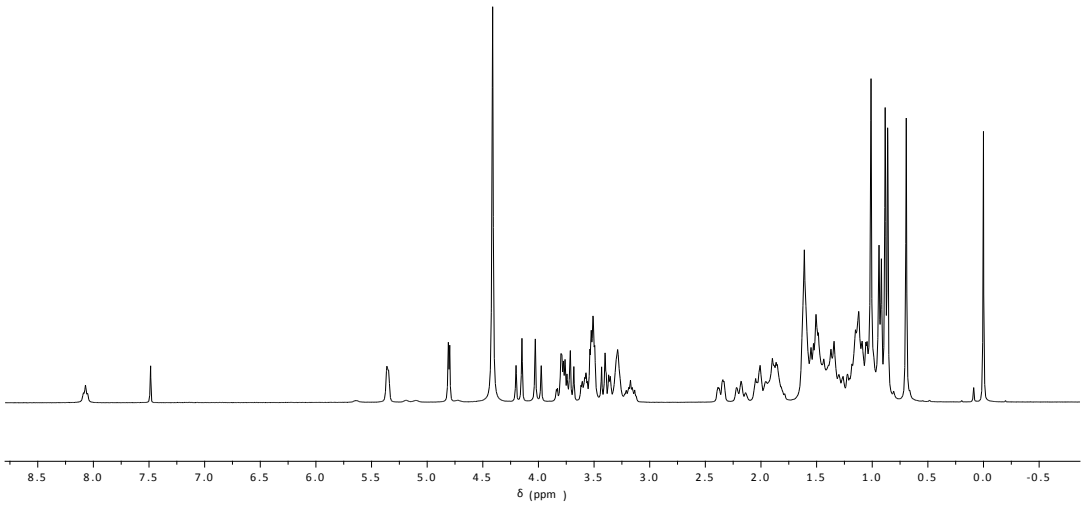


^{13}C NMR

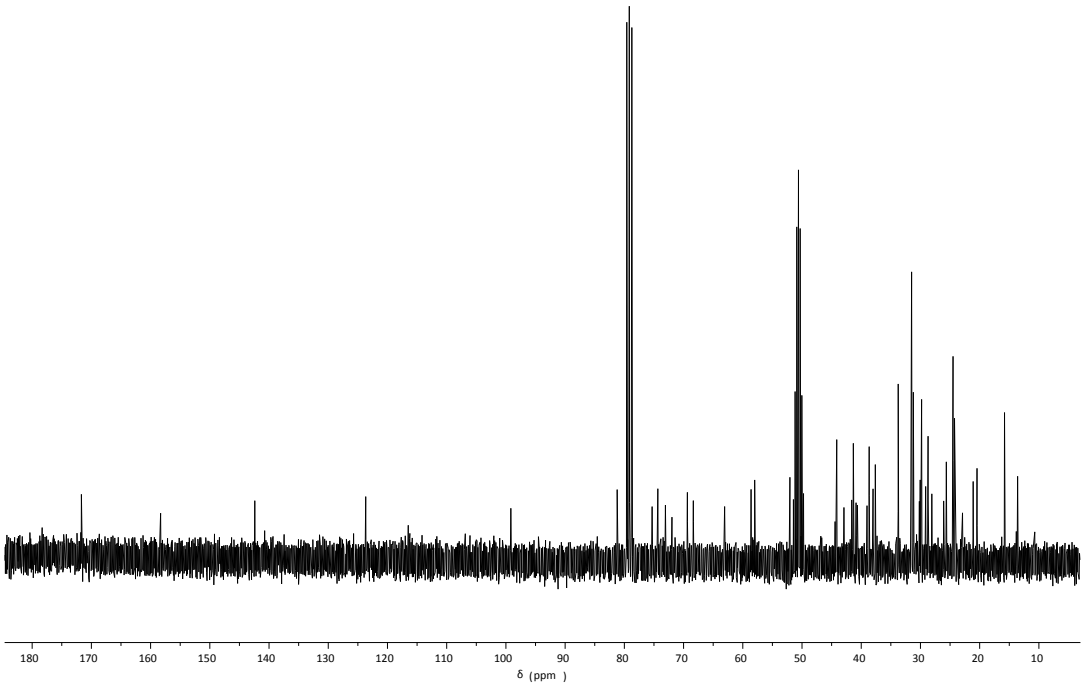


Compound 4b

^{1}H NMR

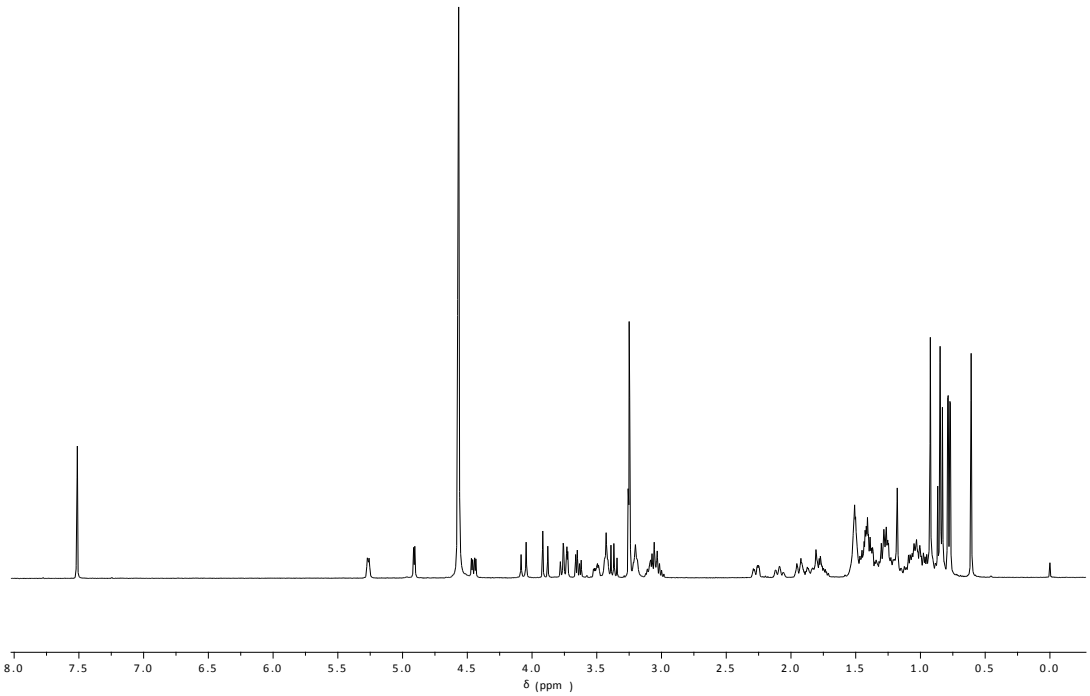


^{13}C NMR

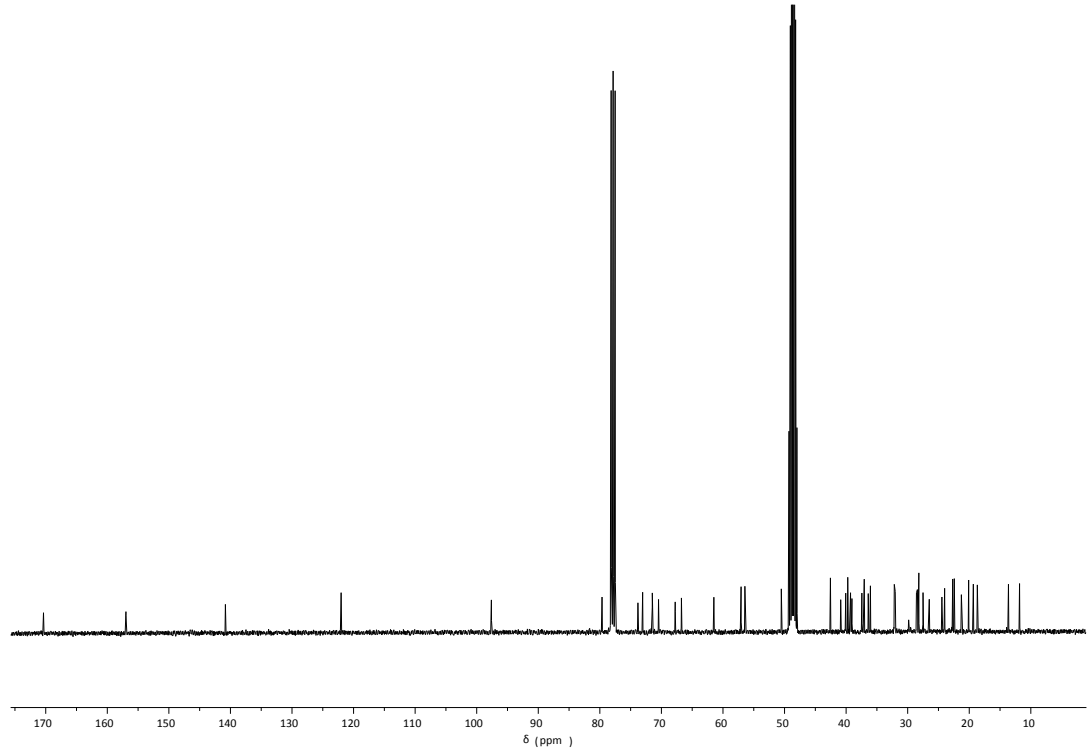


Compound 5a

^{1}H NMR

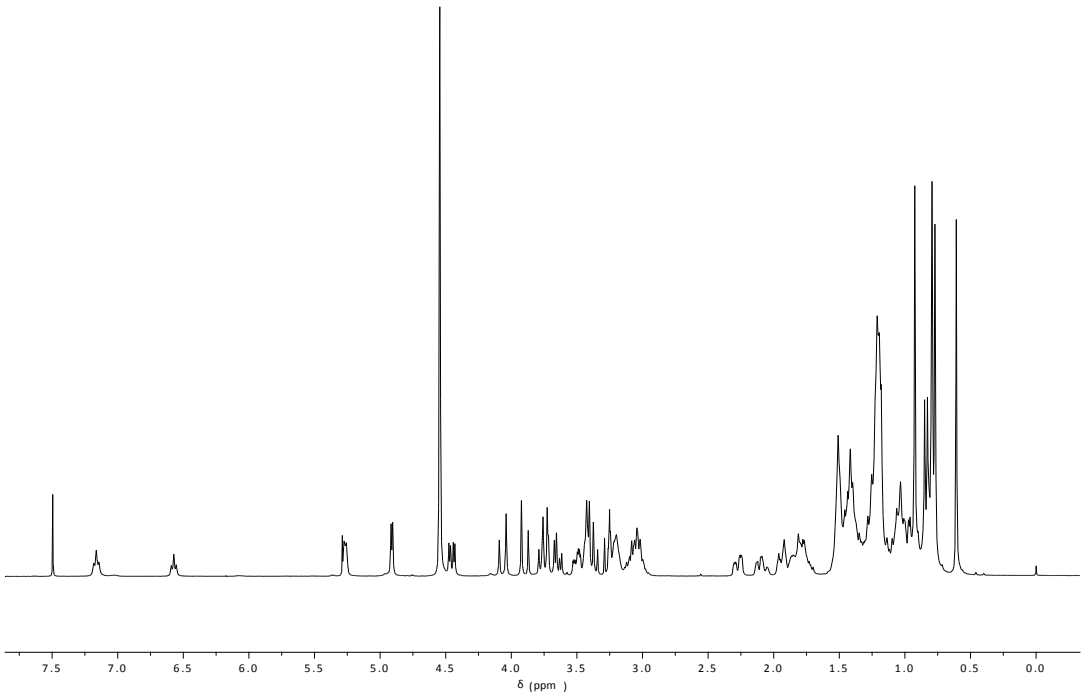


^{13}C NMR

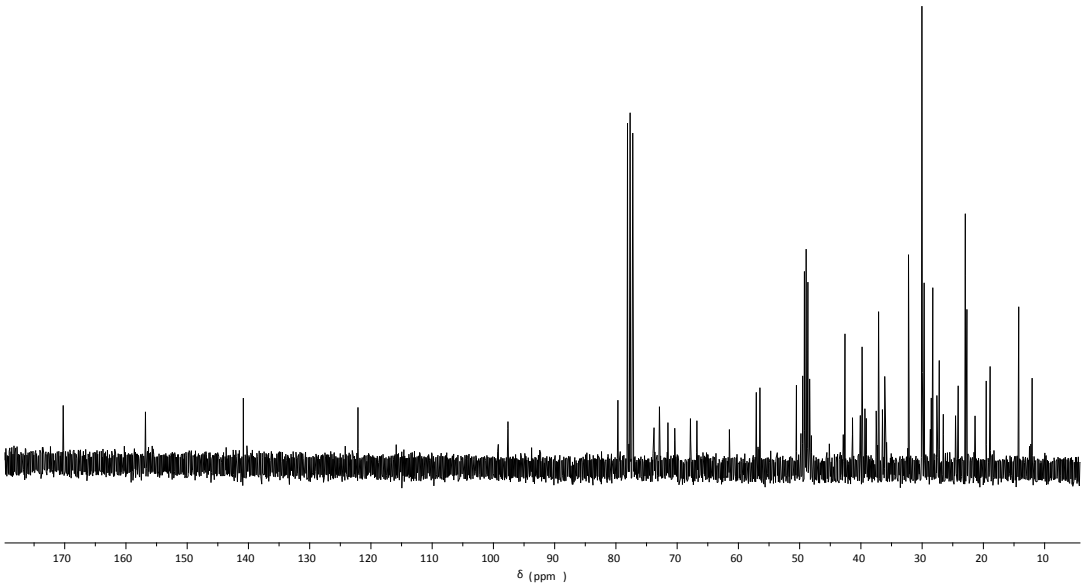


Compound 5b

^{1}H NMR

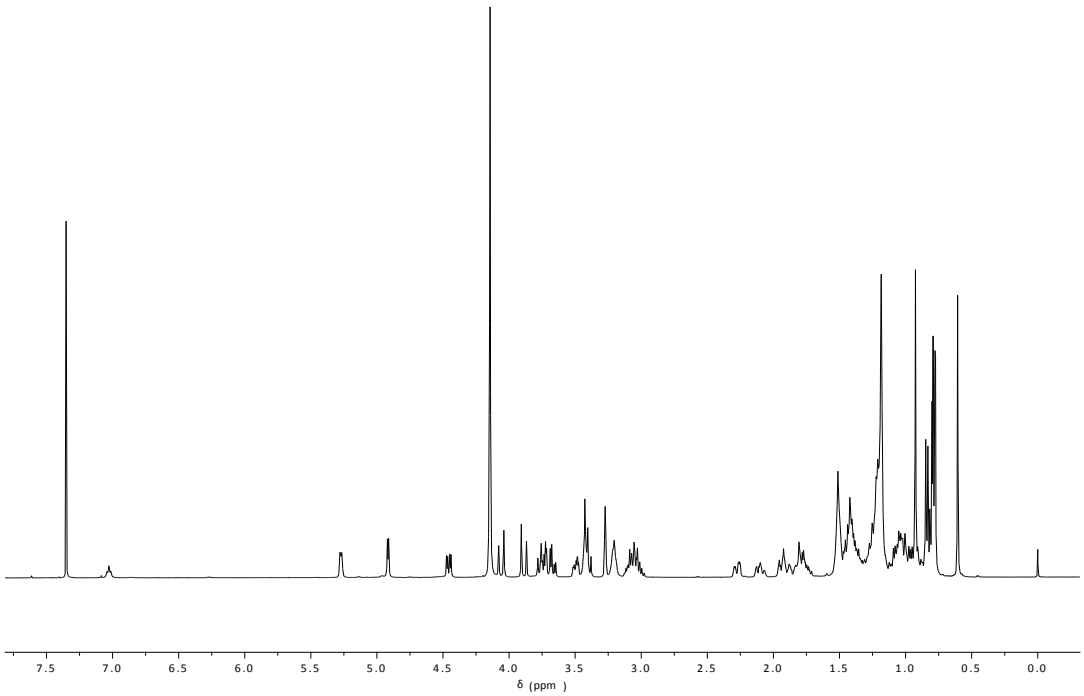


^{13}C NMR

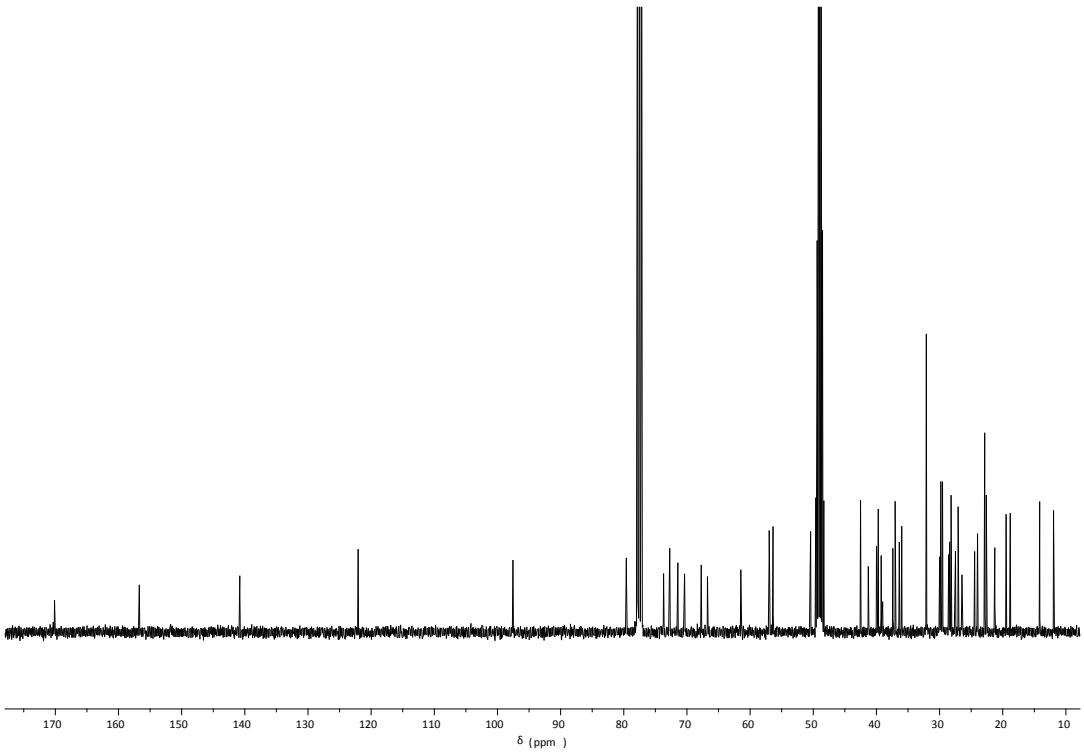


Compound 5c

^{1}H NMR

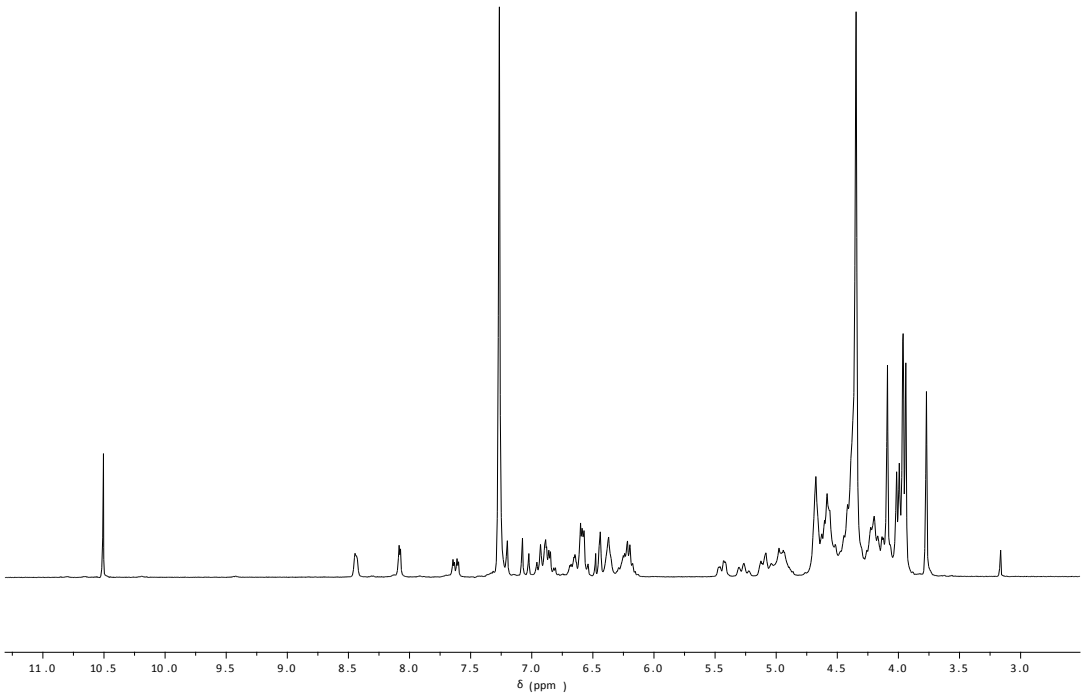


^{13}C NMR

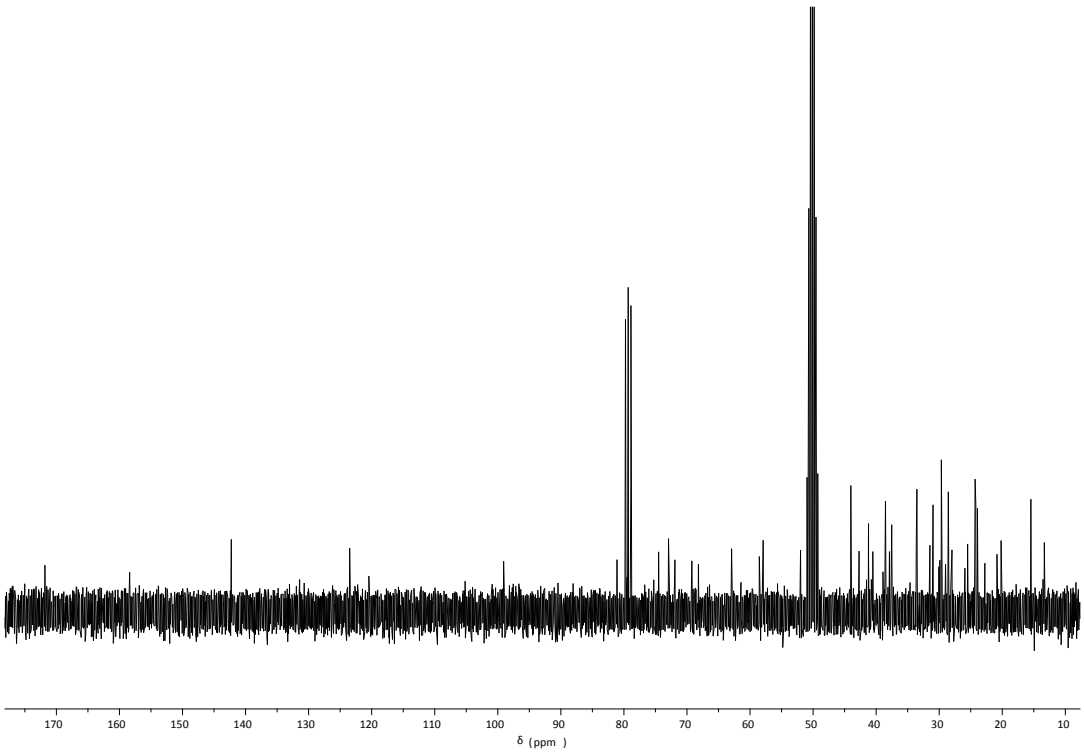


Compound 5d

^{1}H NMR

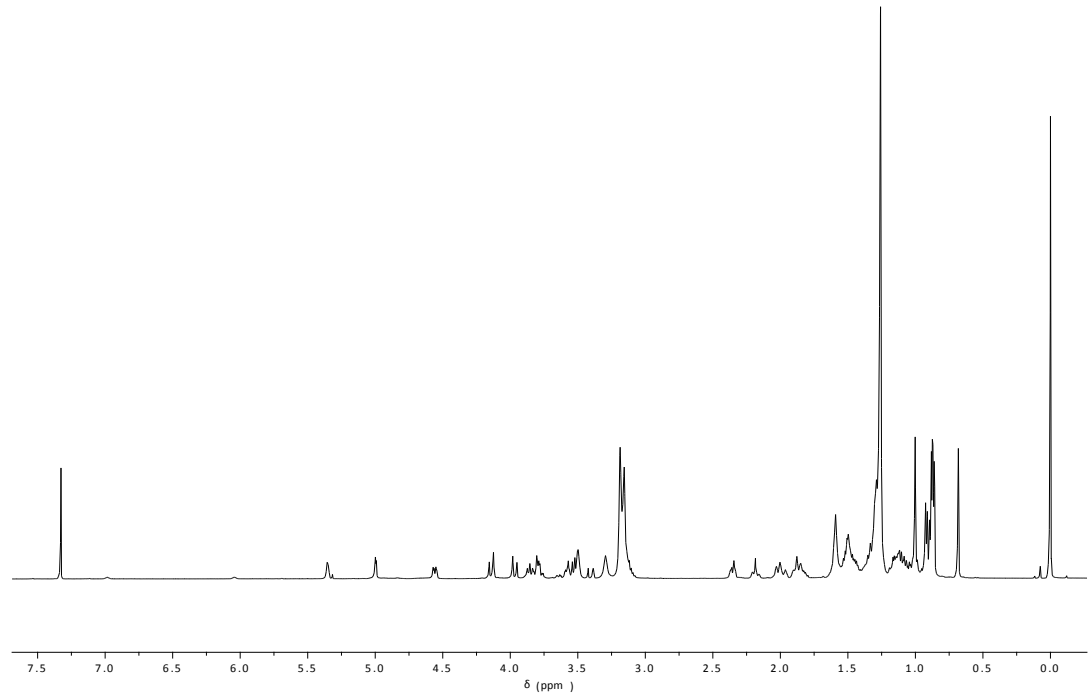


^{13}C NMR

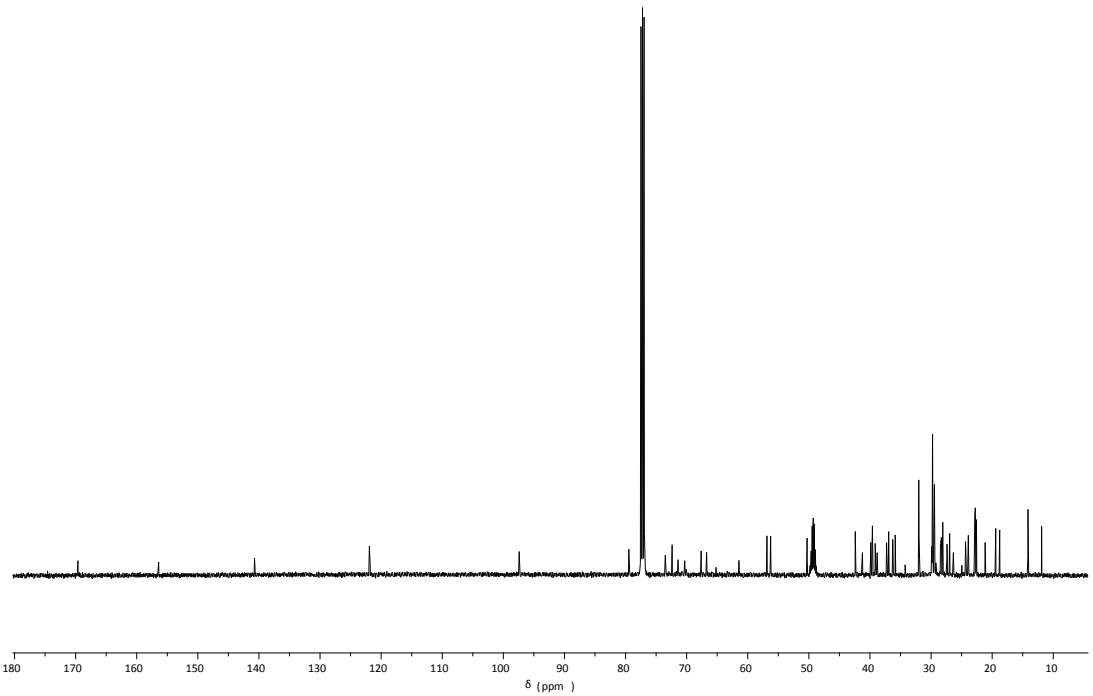


Compound 5e

^{1}H NMR

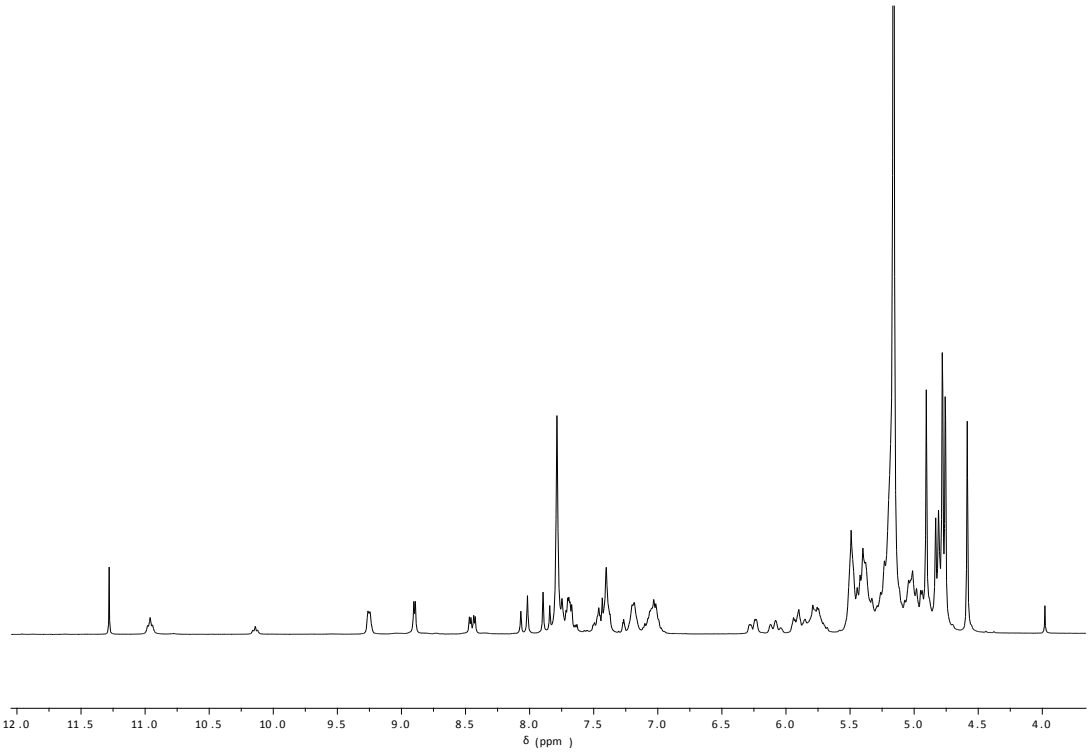


^{13}C NMR

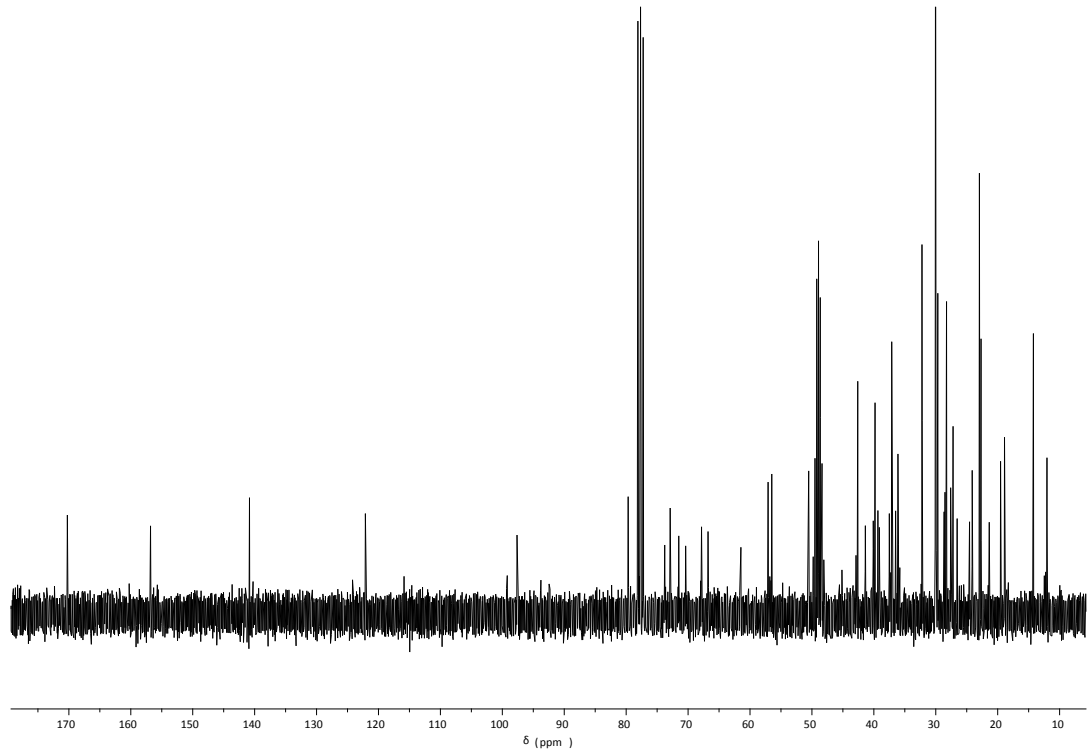


Compound 5f

^{1}H NMR

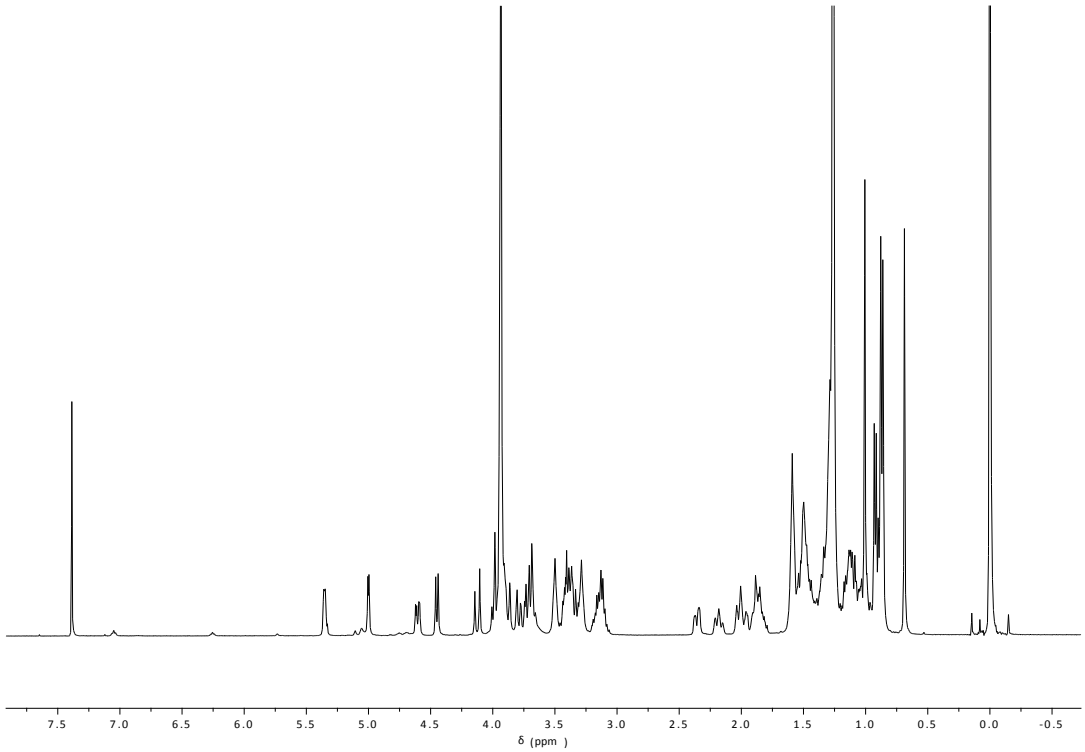


^{13}C NMR

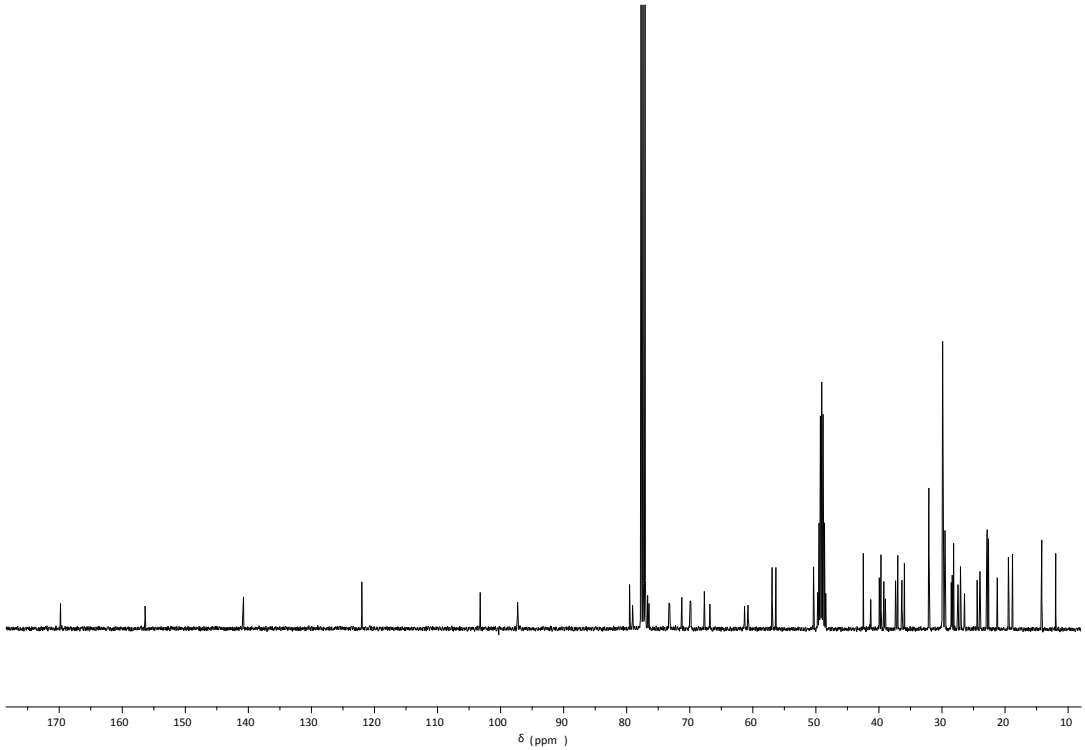


Compound 5g

^{1}H NMR

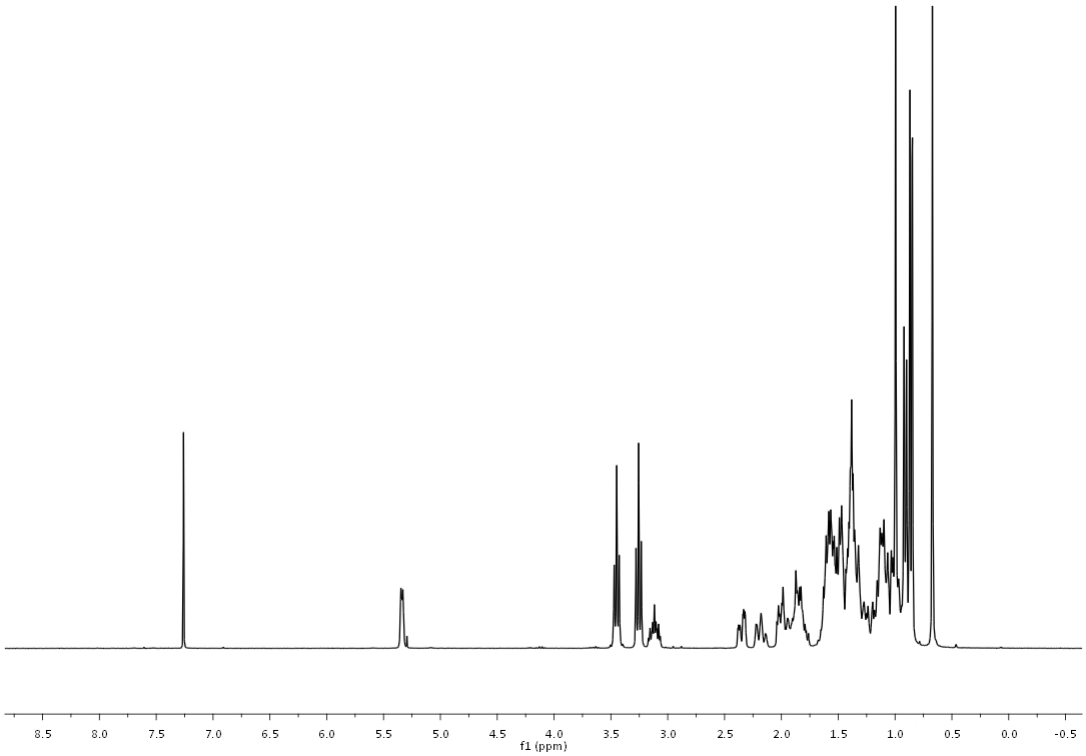


^{13}C NMR

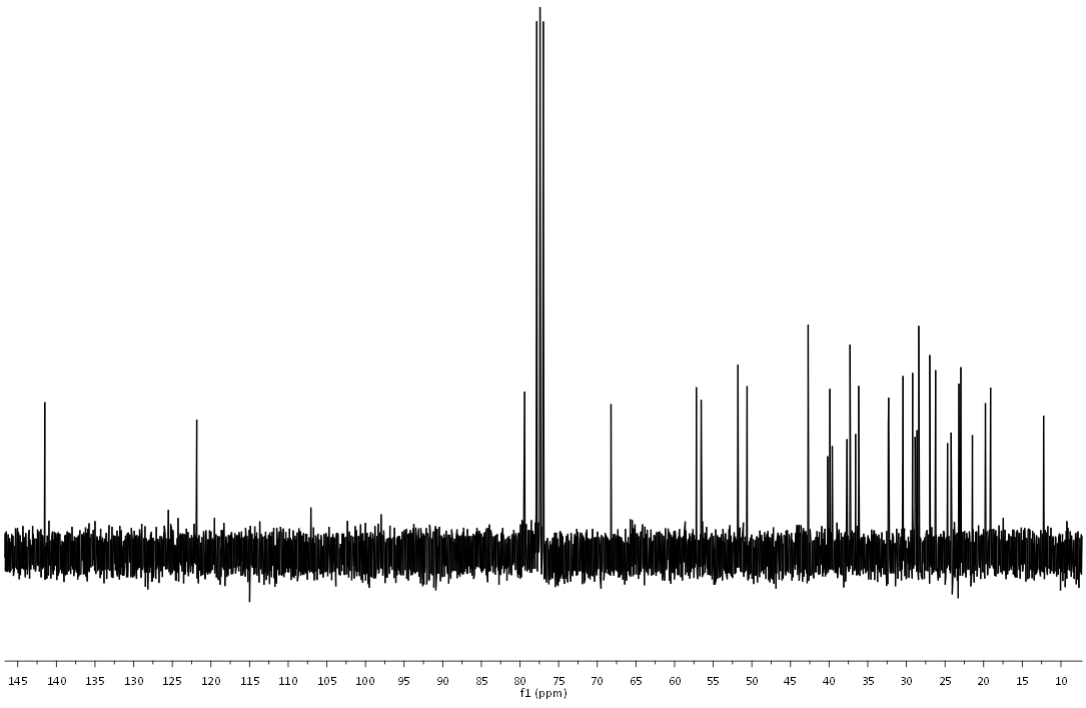


Compound **10**

^{1}H NMR

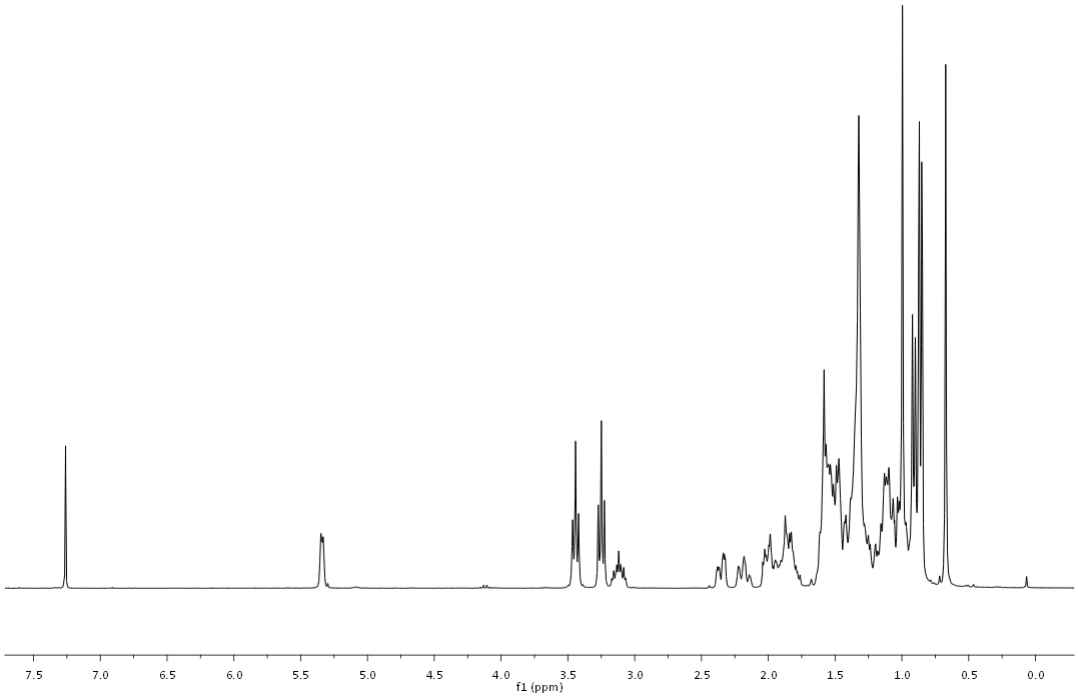


^{13}C NMR

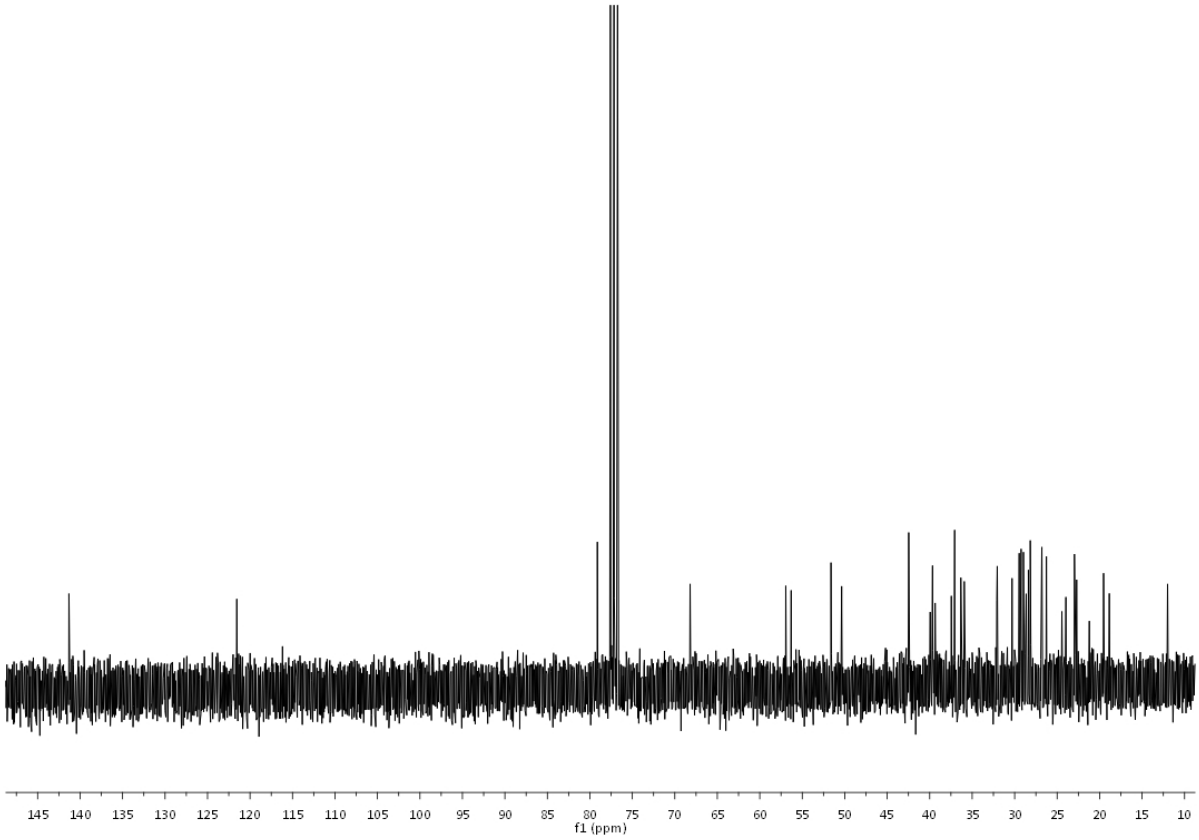


Compound **11**

^{1}H NMR

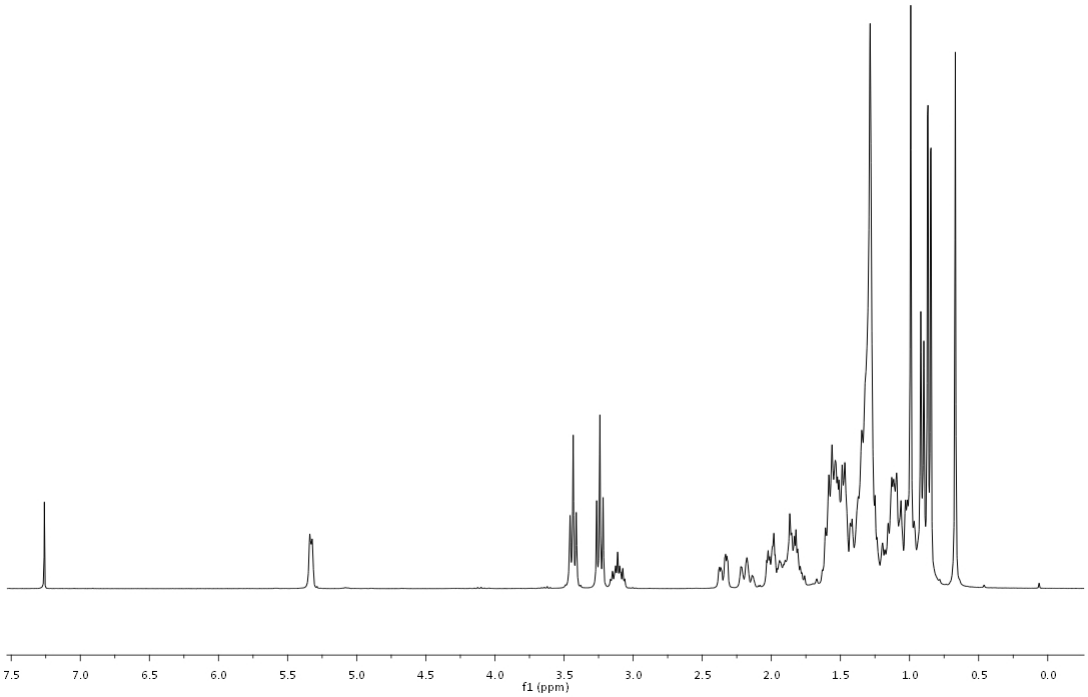


^{13}C NMR

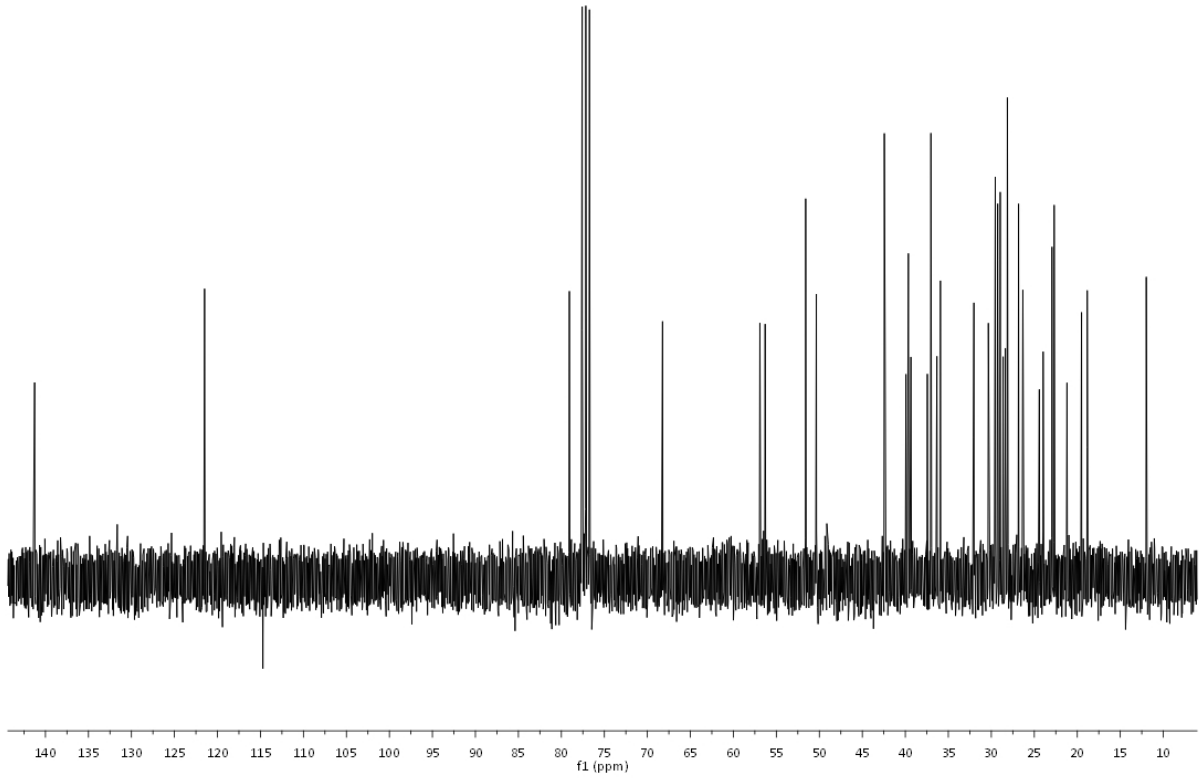


Compound **12**

^{1}H NMR

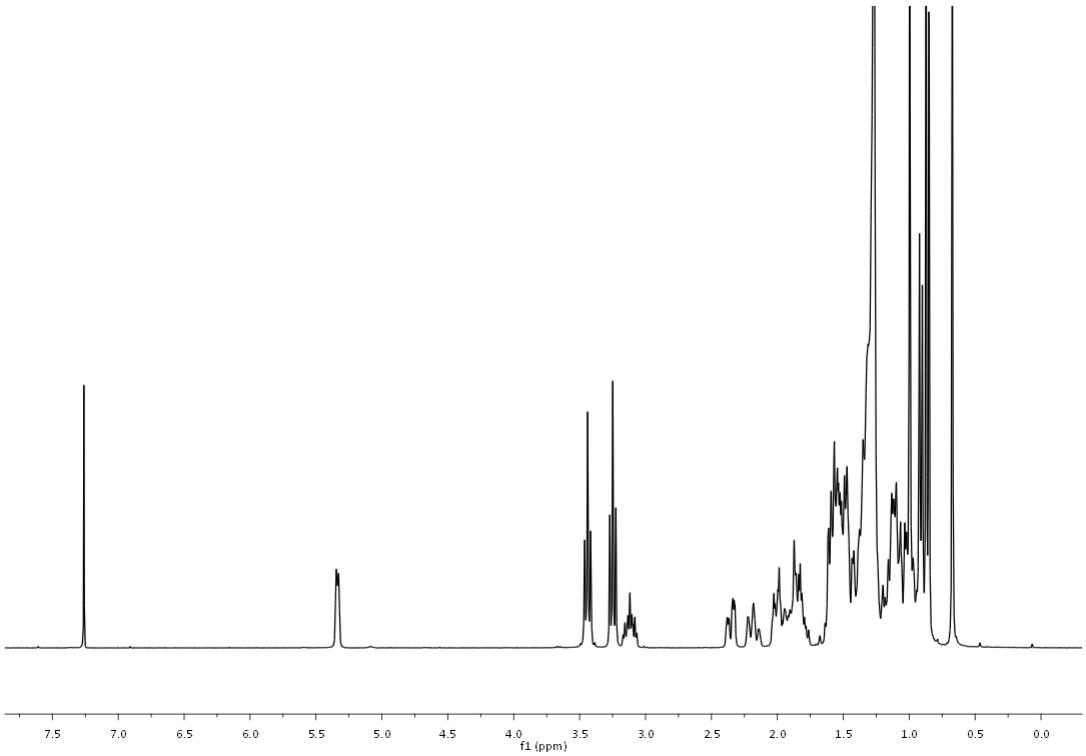


^{13}C NMR

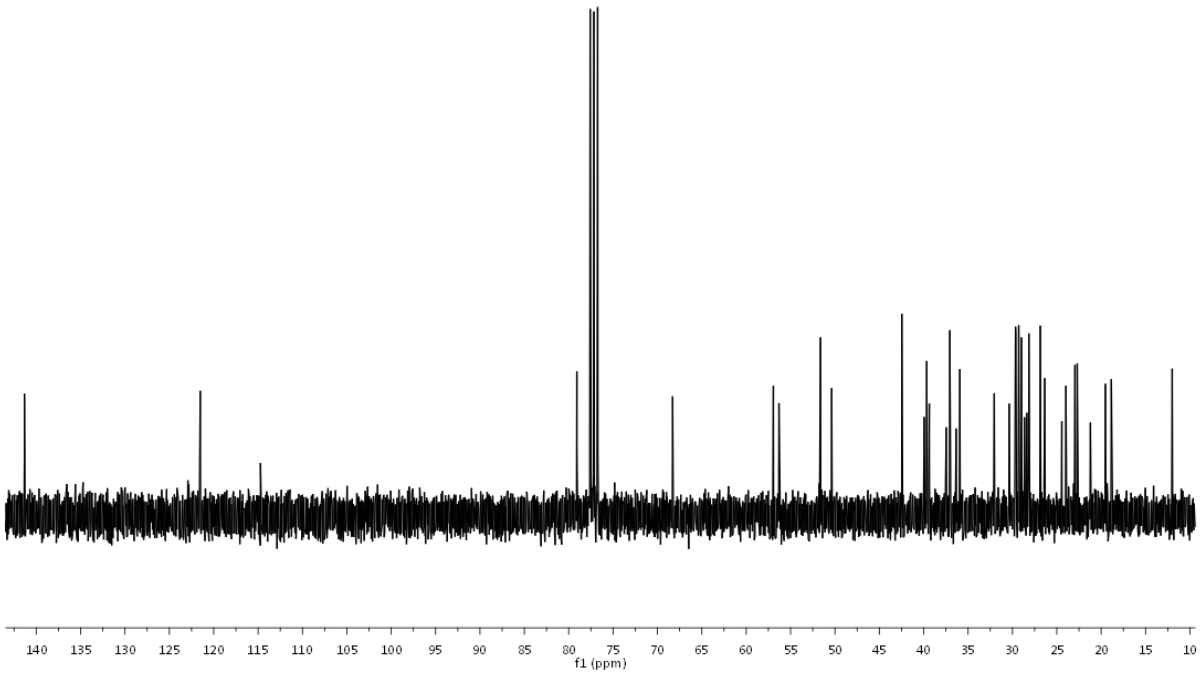


Compound **13**

^{1}H NMR

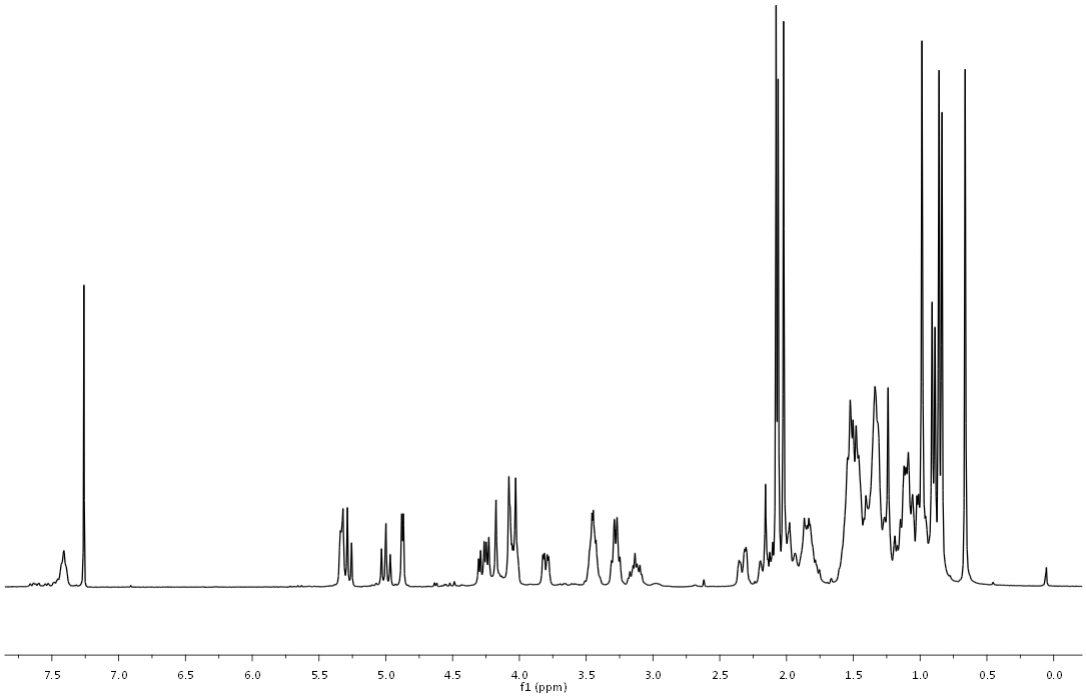


^{13}C NMR

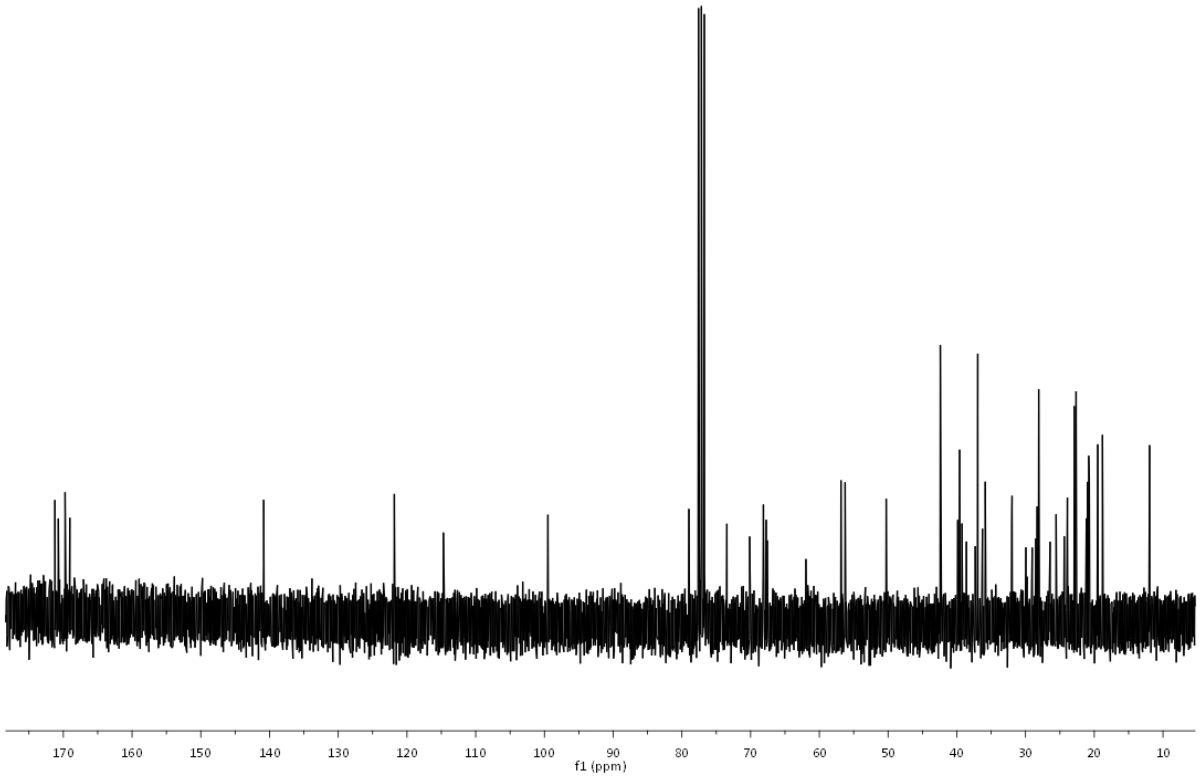


Compound **14a**

^{1}H NMR

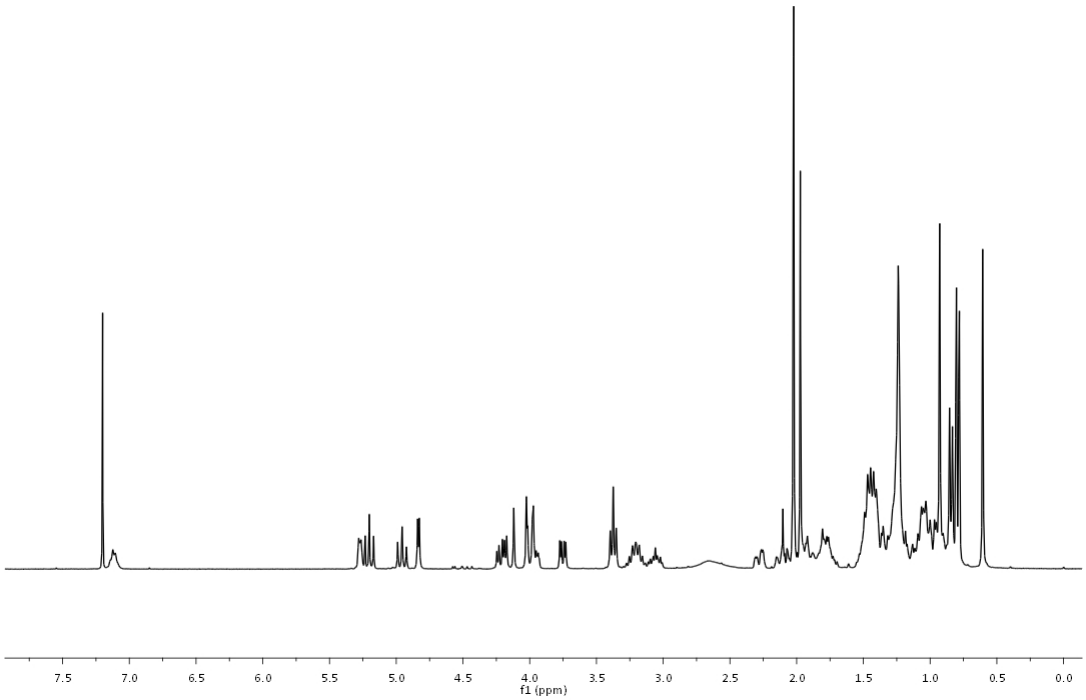


^{13}C NMR

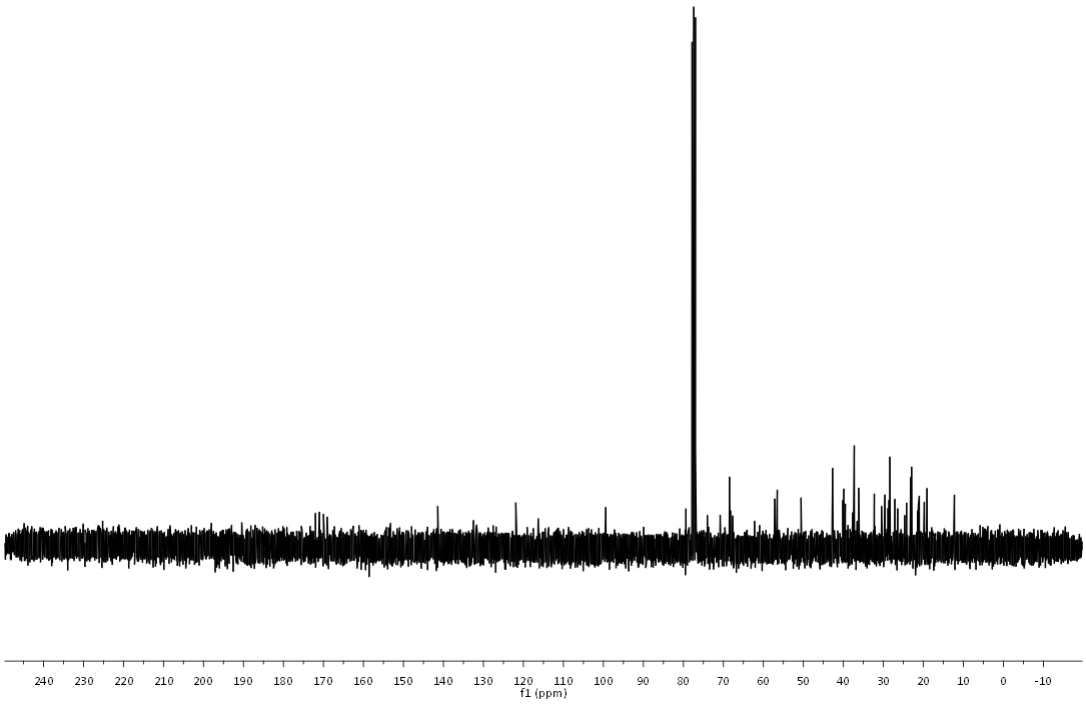


Compound **15a**

^{1}H NMR

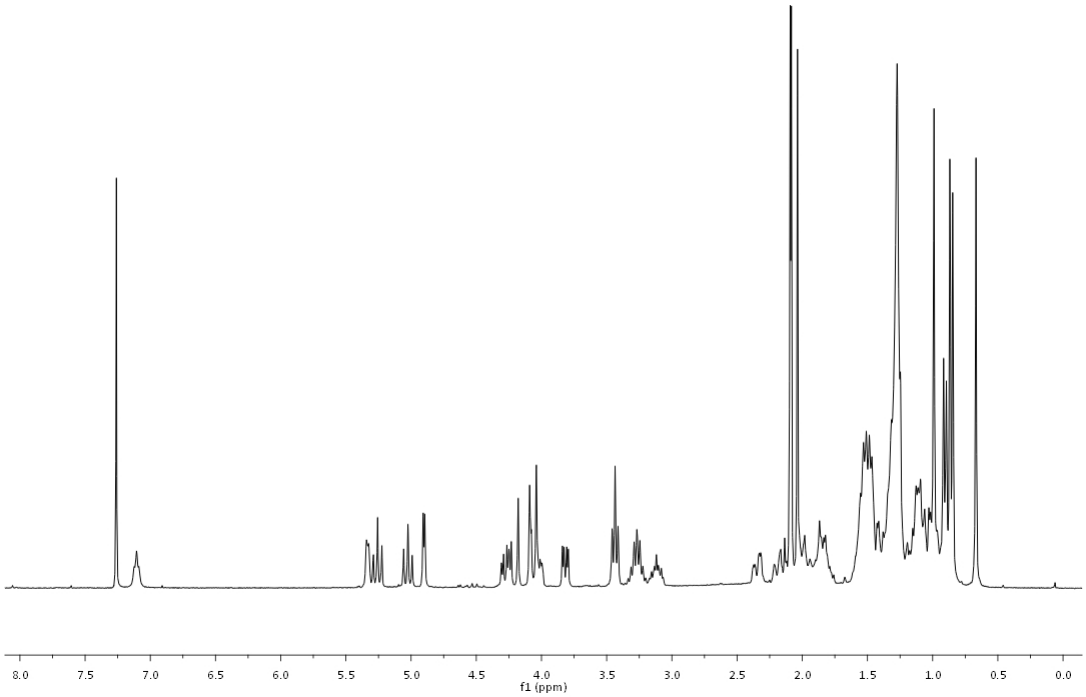


^{13}C NMR

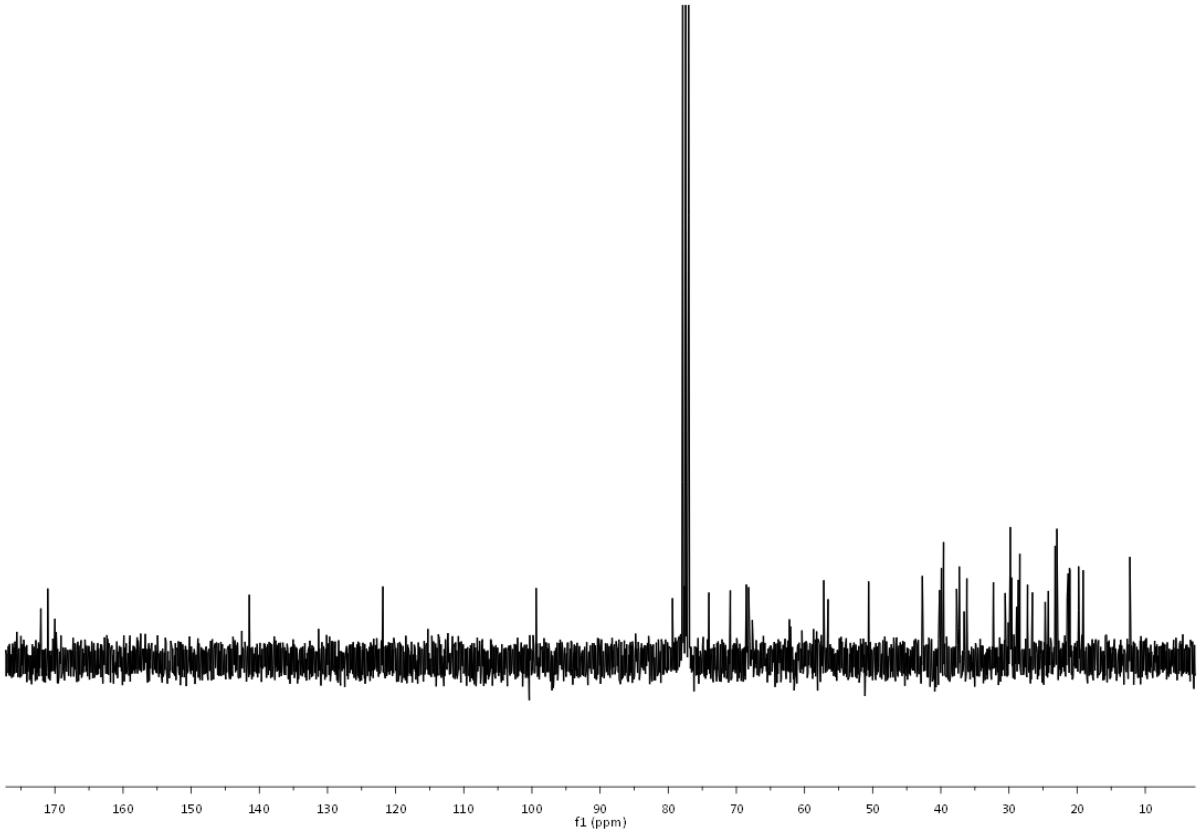


Compound **16a**

^{1}H NMR

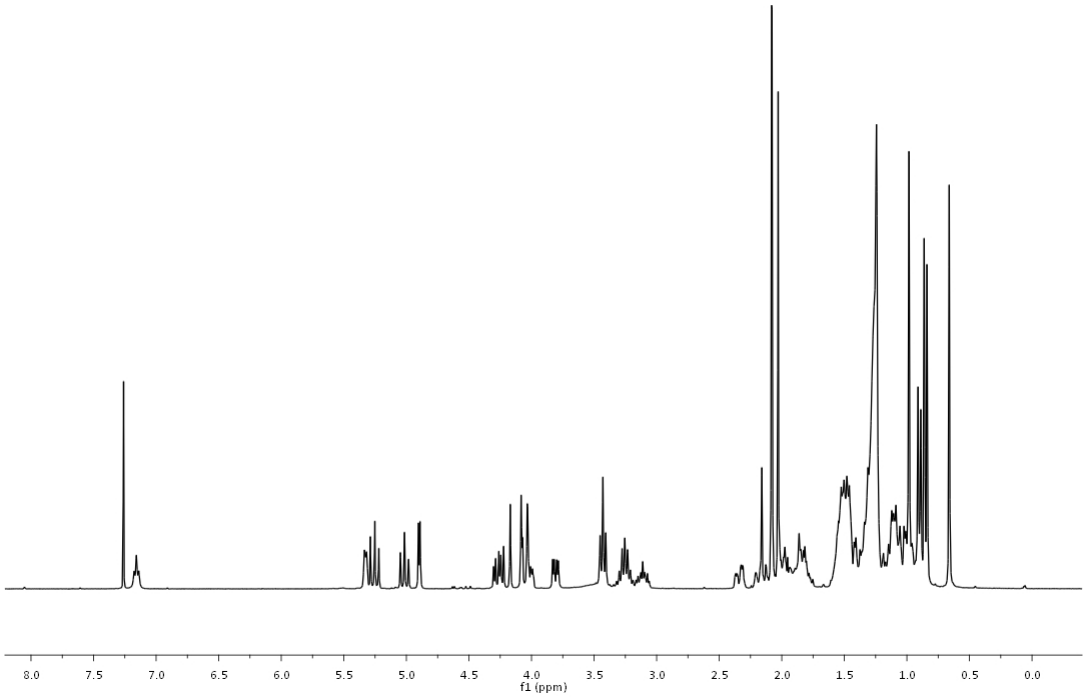


^{13}C NMR

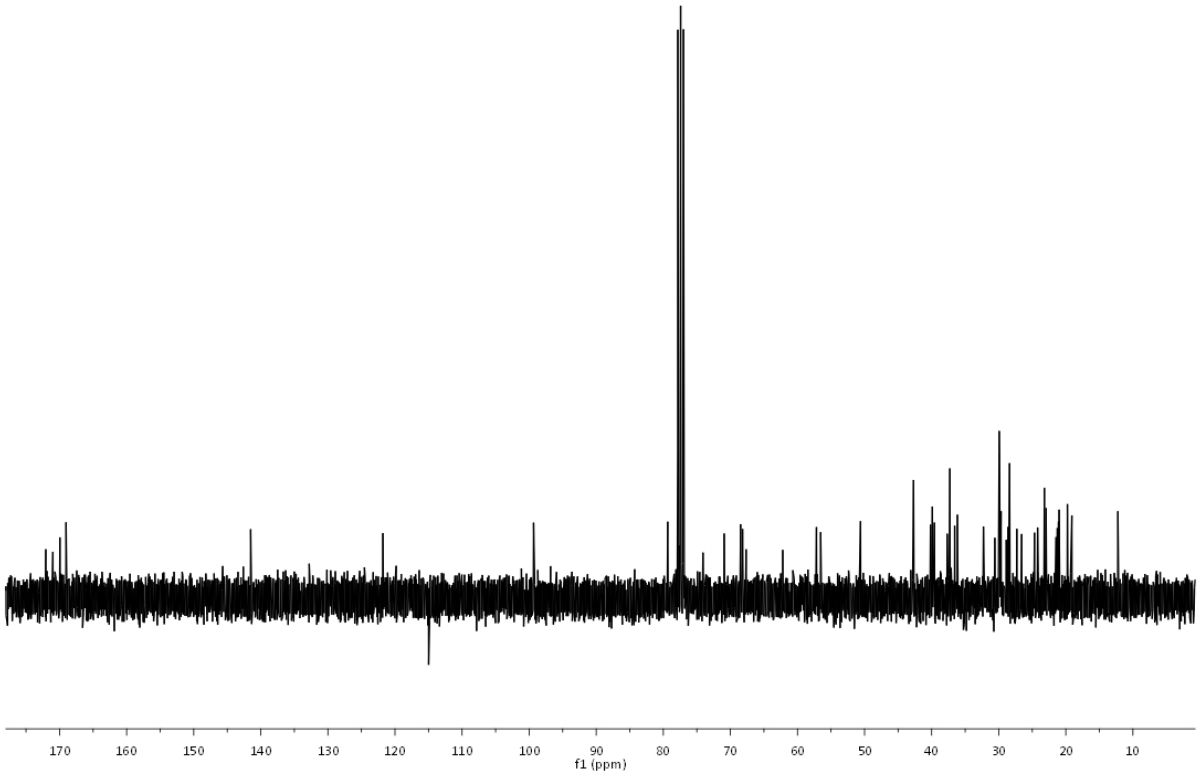


Compound **17a**

^{1}H NMR

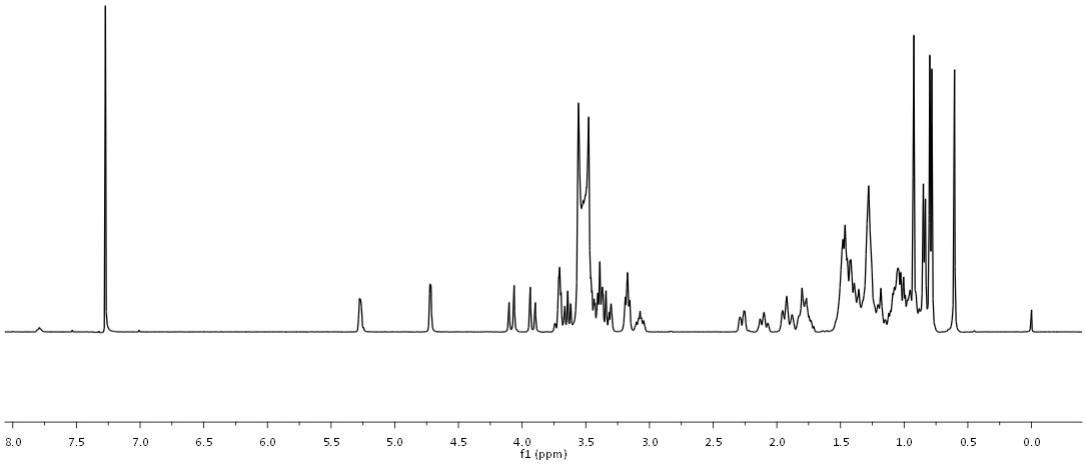


^{13}C NMR

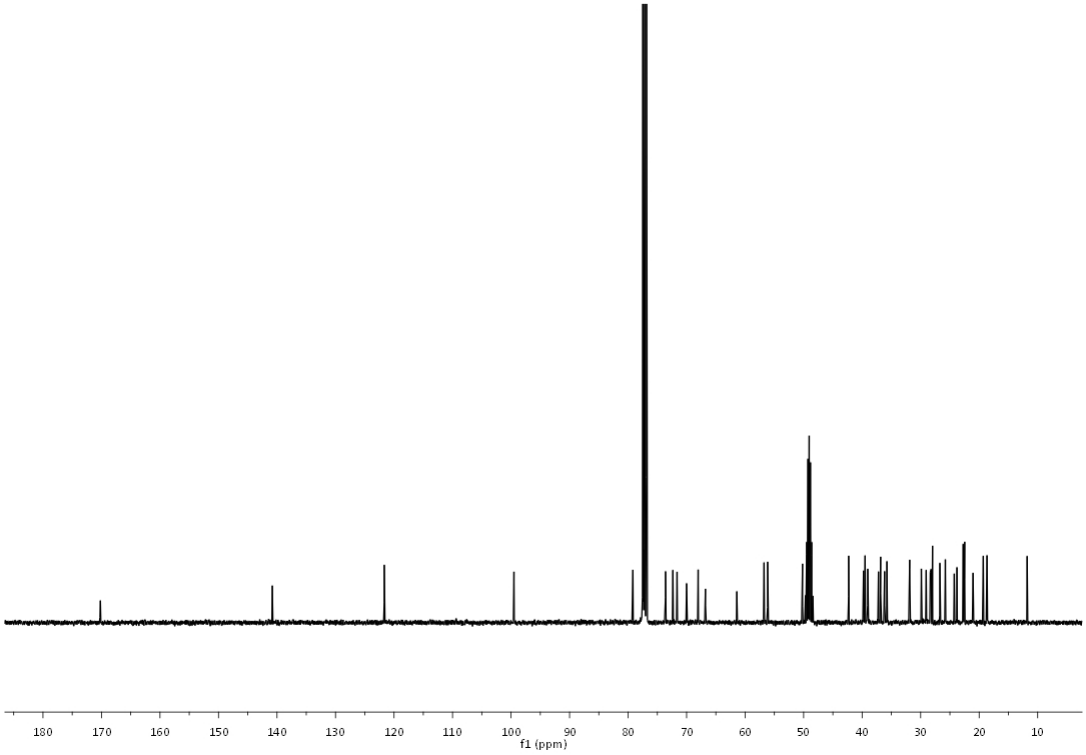


Compound **14b**

^{1}H NMR

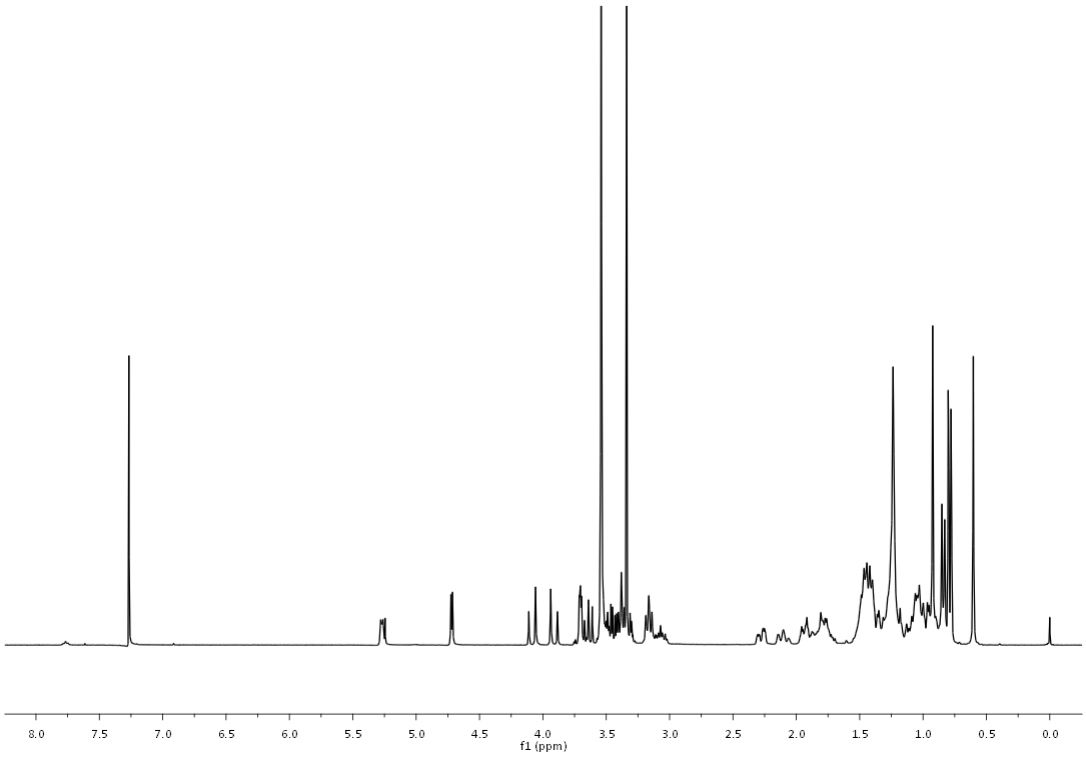


^{13}C NMR

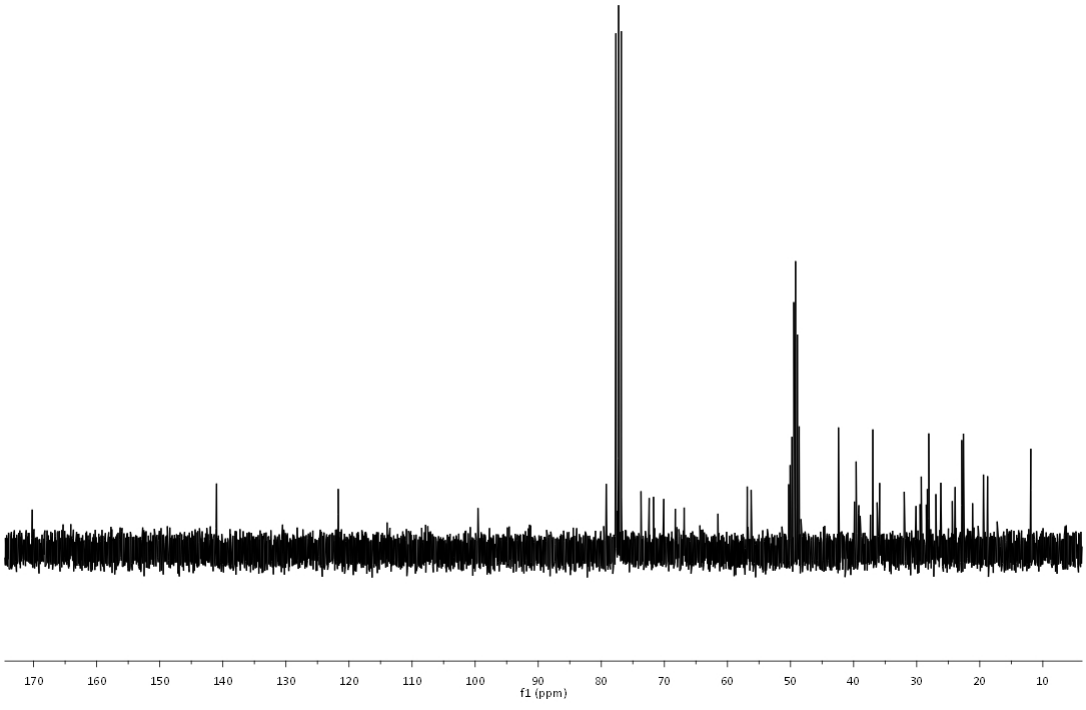


Compound **15b**

^{1}H NMR

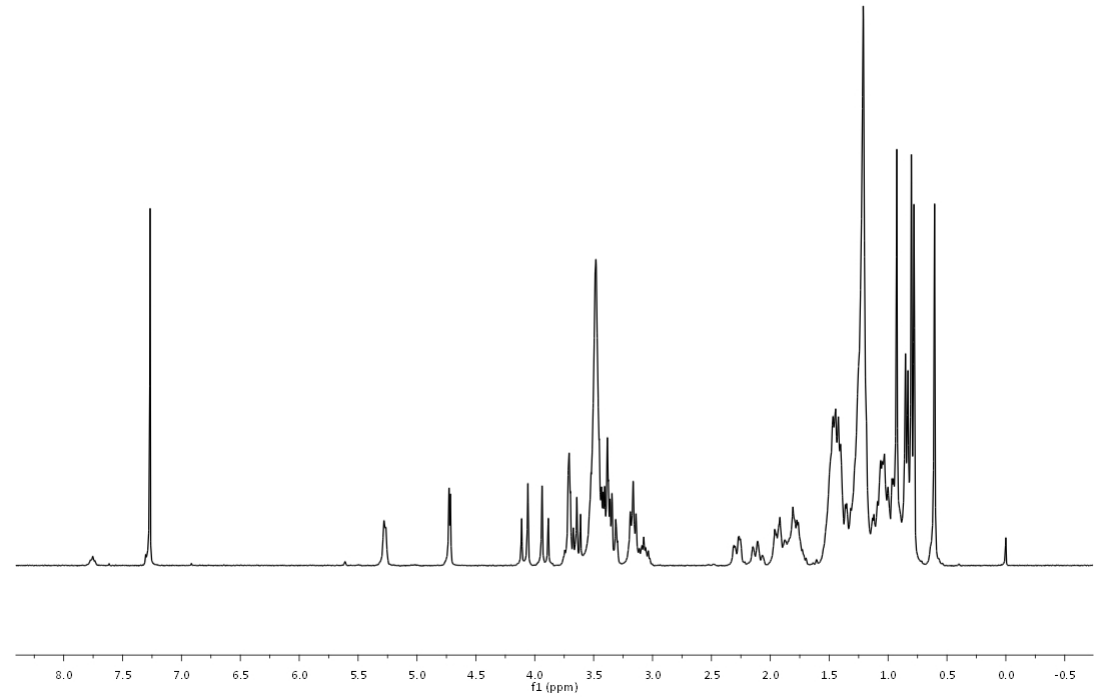


^{13}C NMR

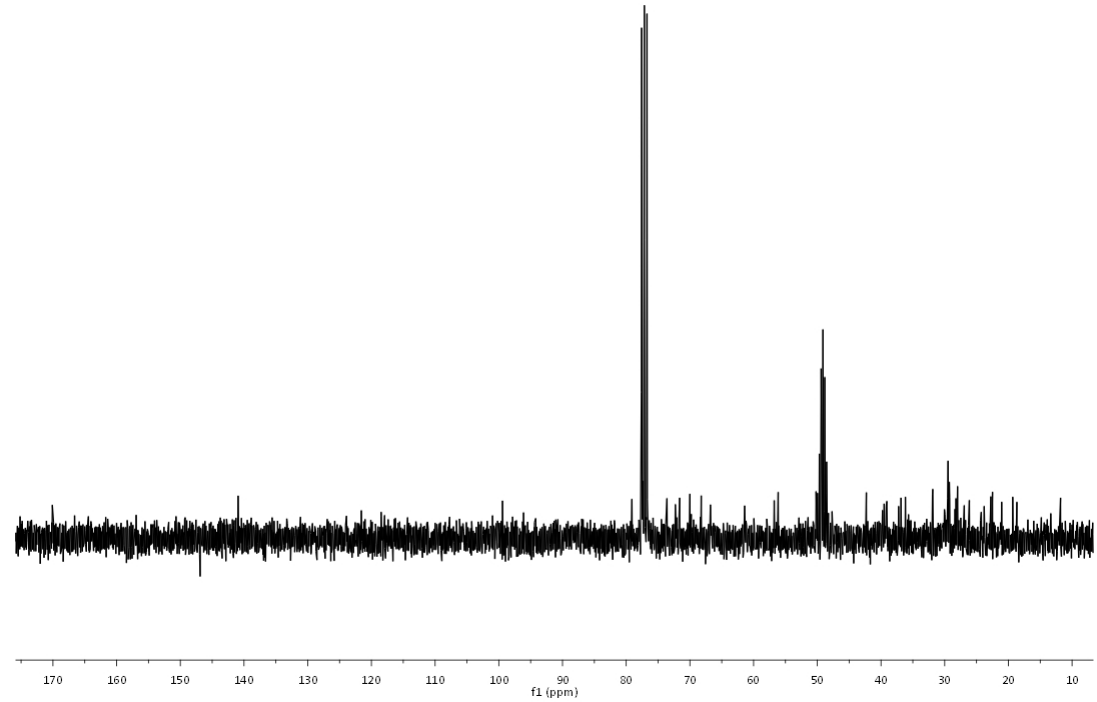


Compound **16b**

^{1}H NMR

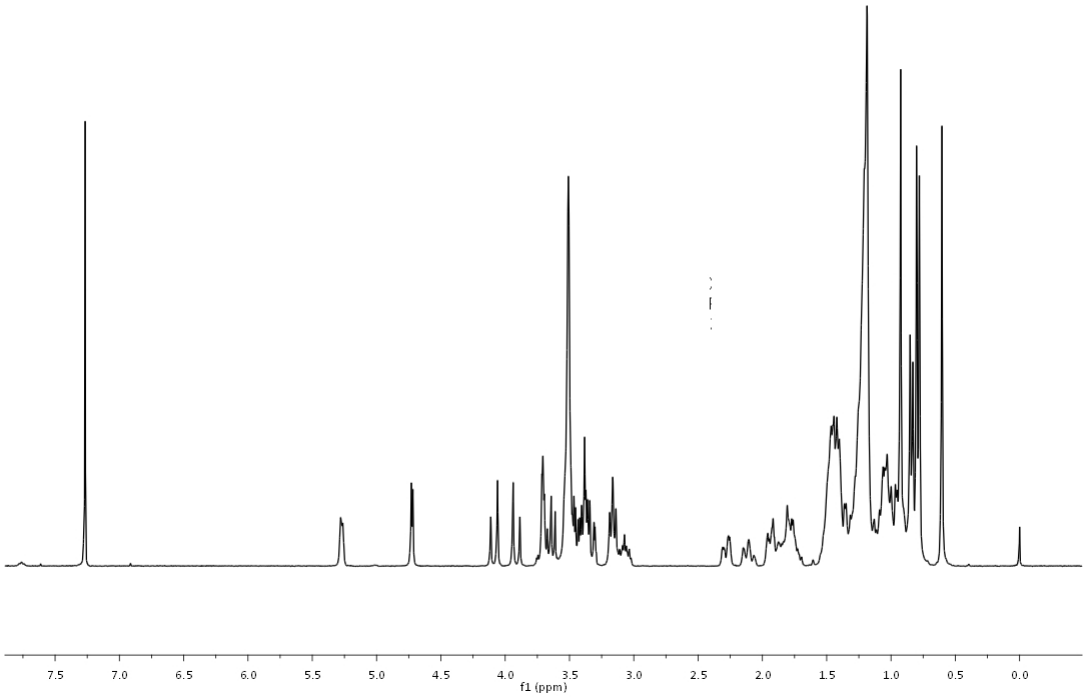


^{13}C NMR

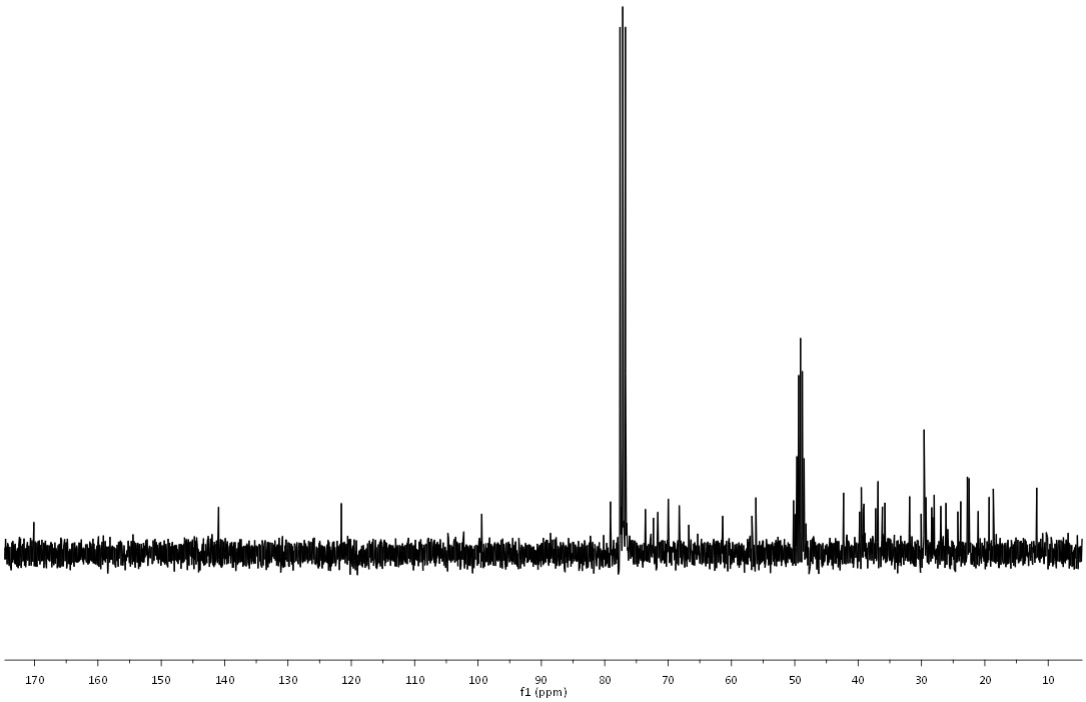


Compound **17b**

^{1}H NMR

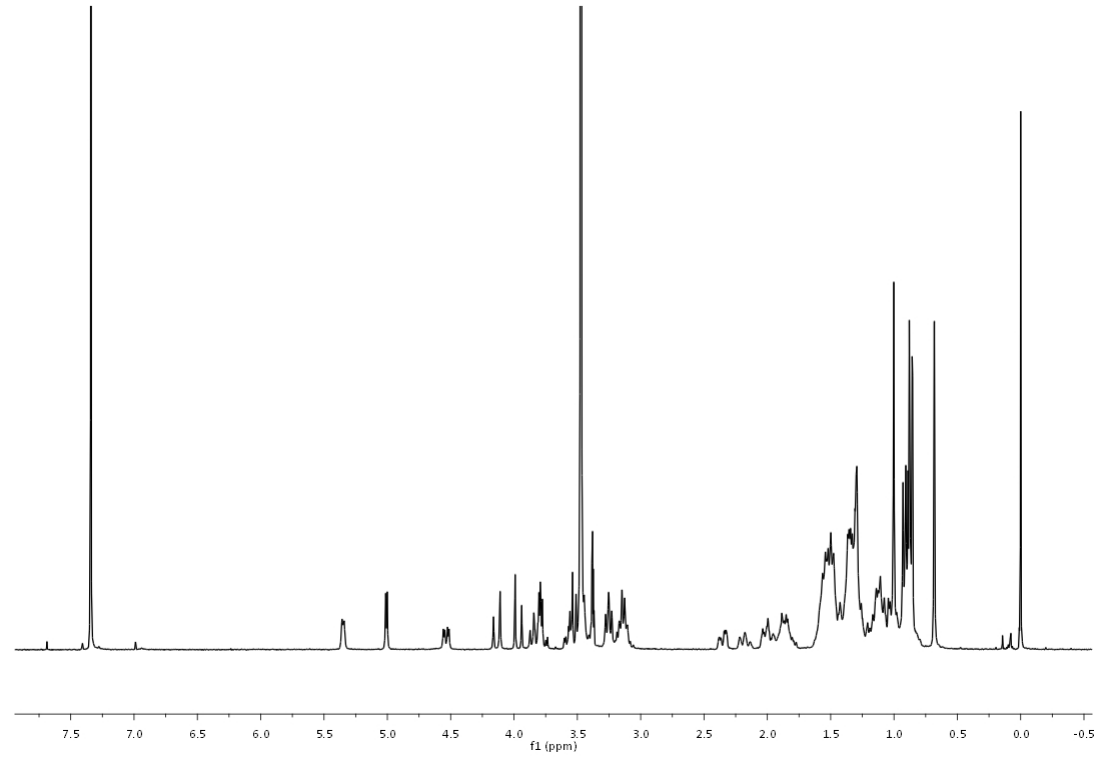


^{13}C NMR

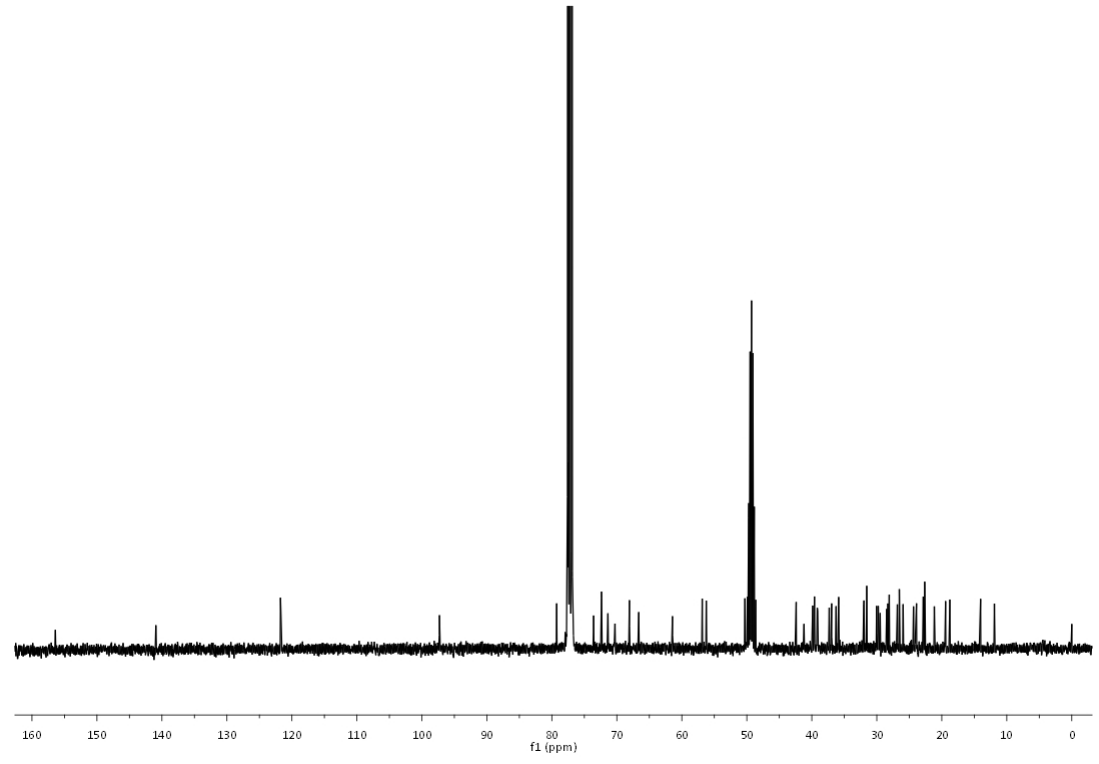


Compound **18a**

^{1}H NMR

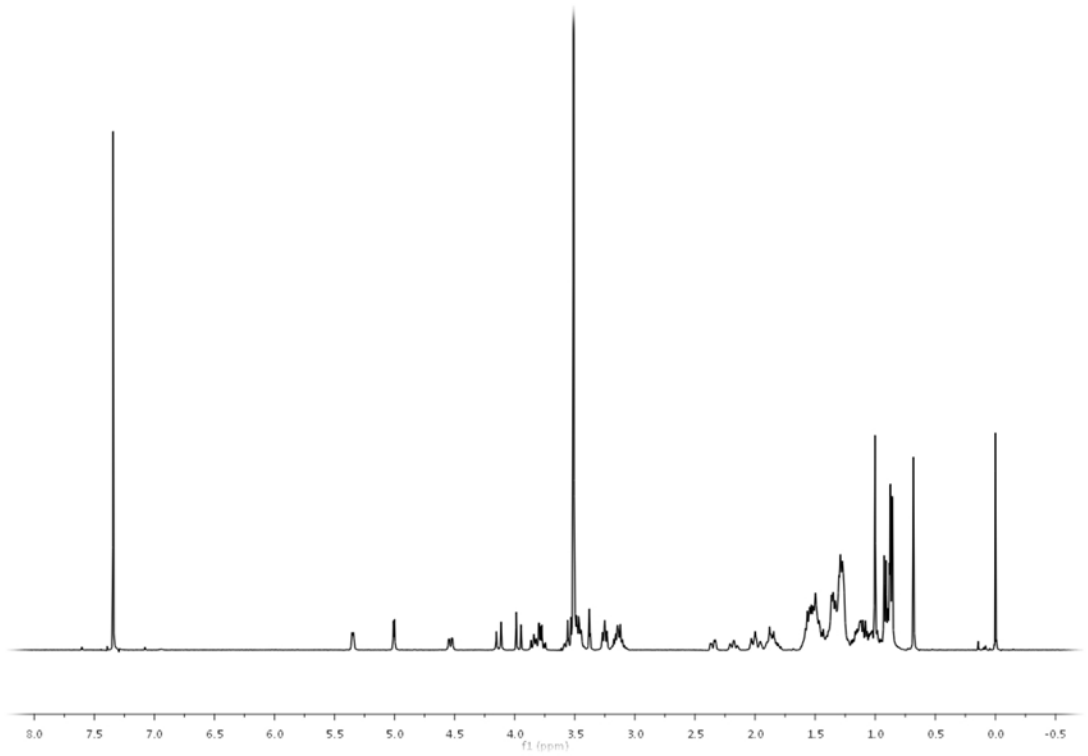


^{13}C NMR

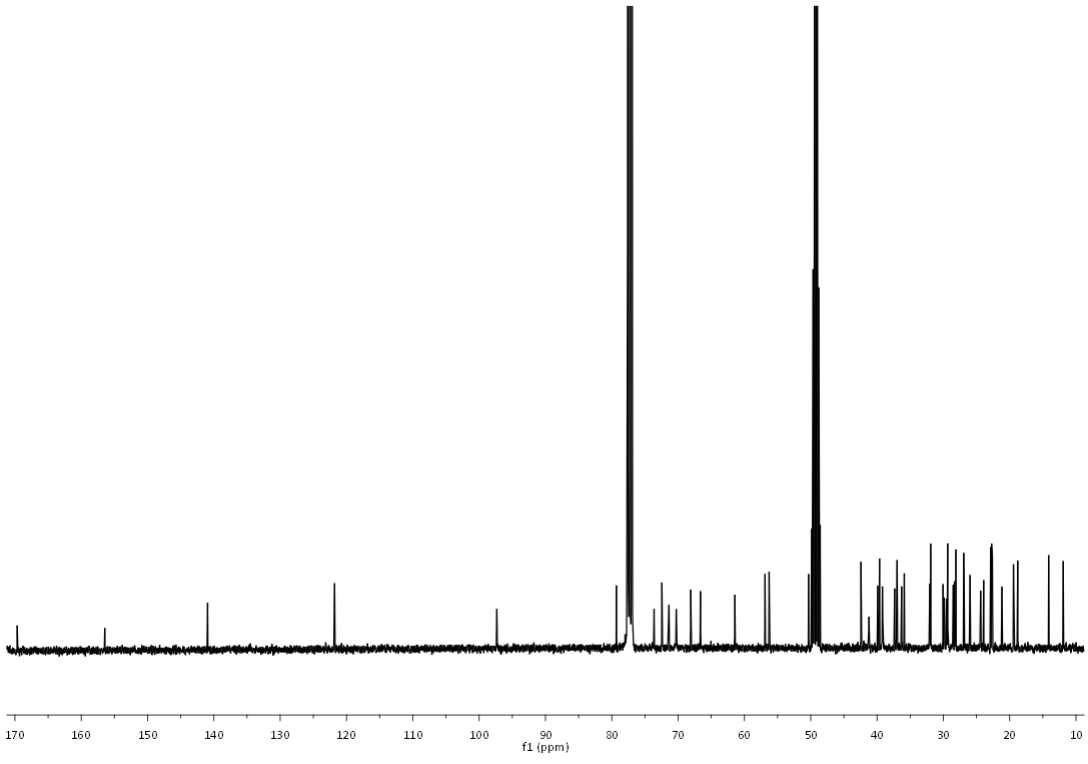


Compound **18b**

^{1}H NMR

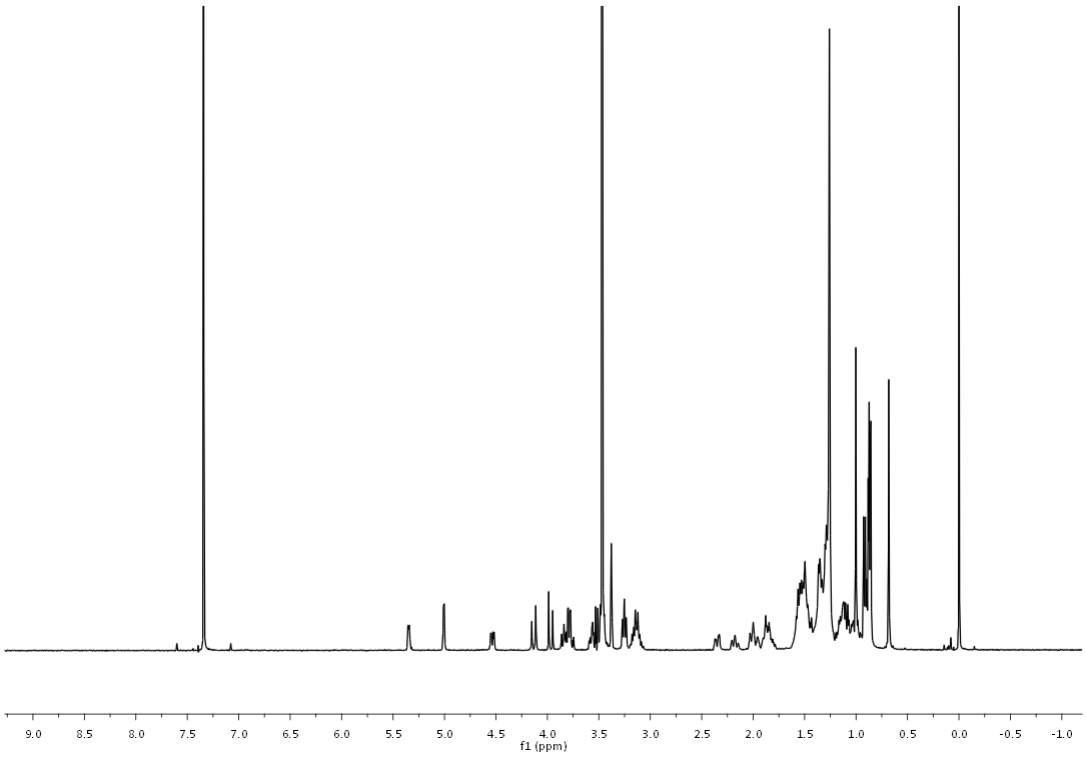


^{13}C NMR

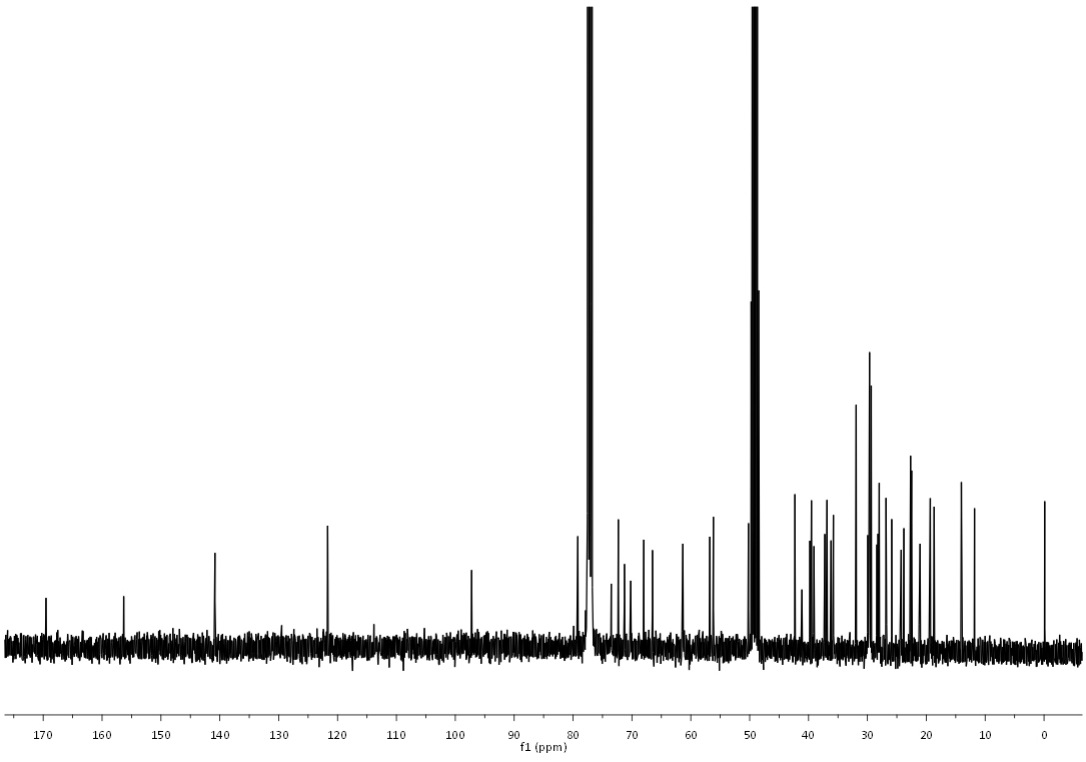


Compound **18c**

^{1}H NMR

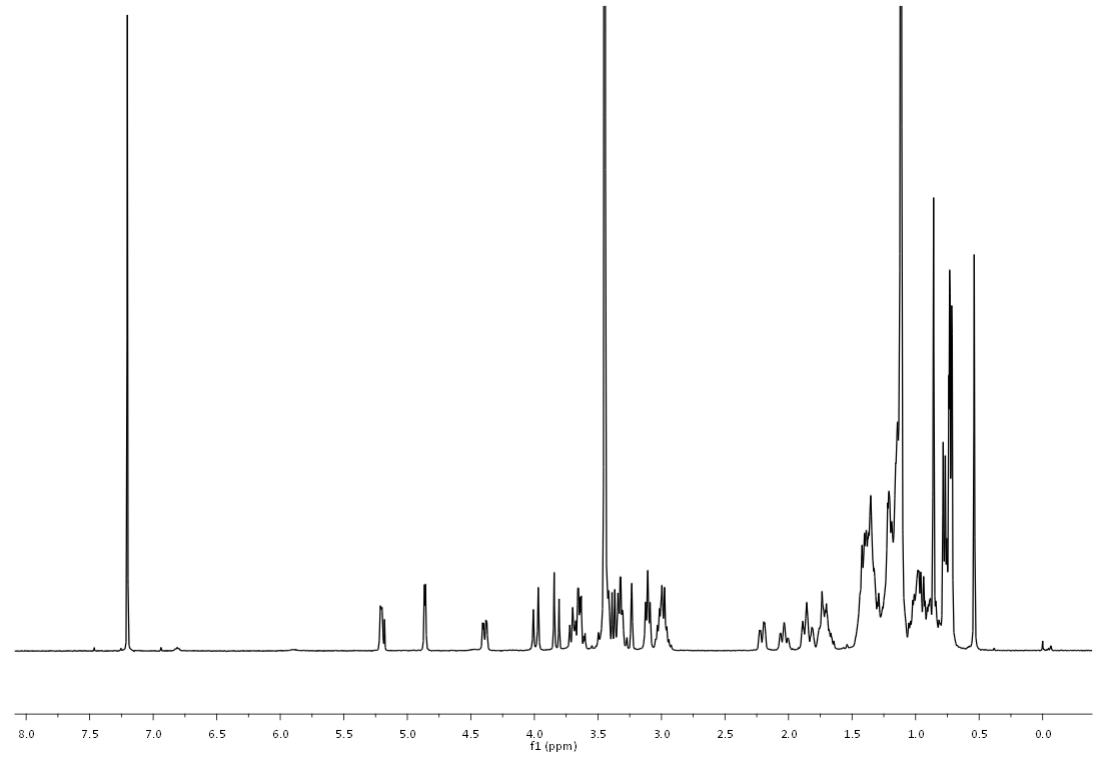


^{13}C NMR

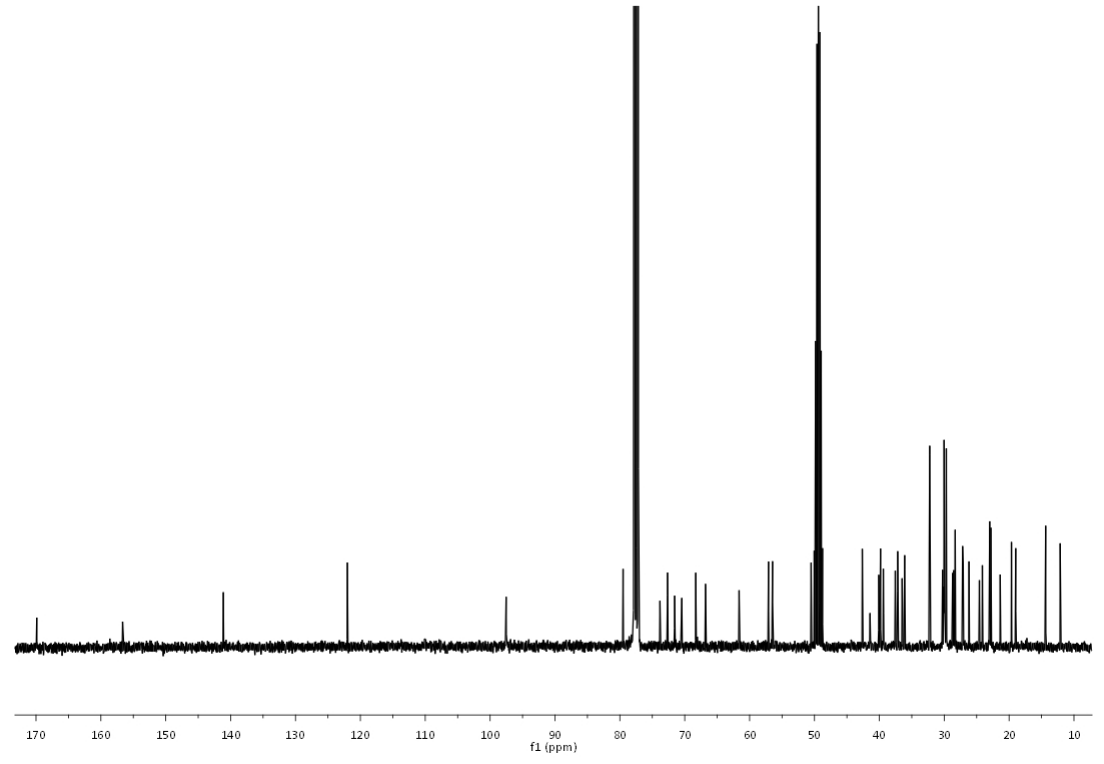


Compound **18d**

^{1}H NMR

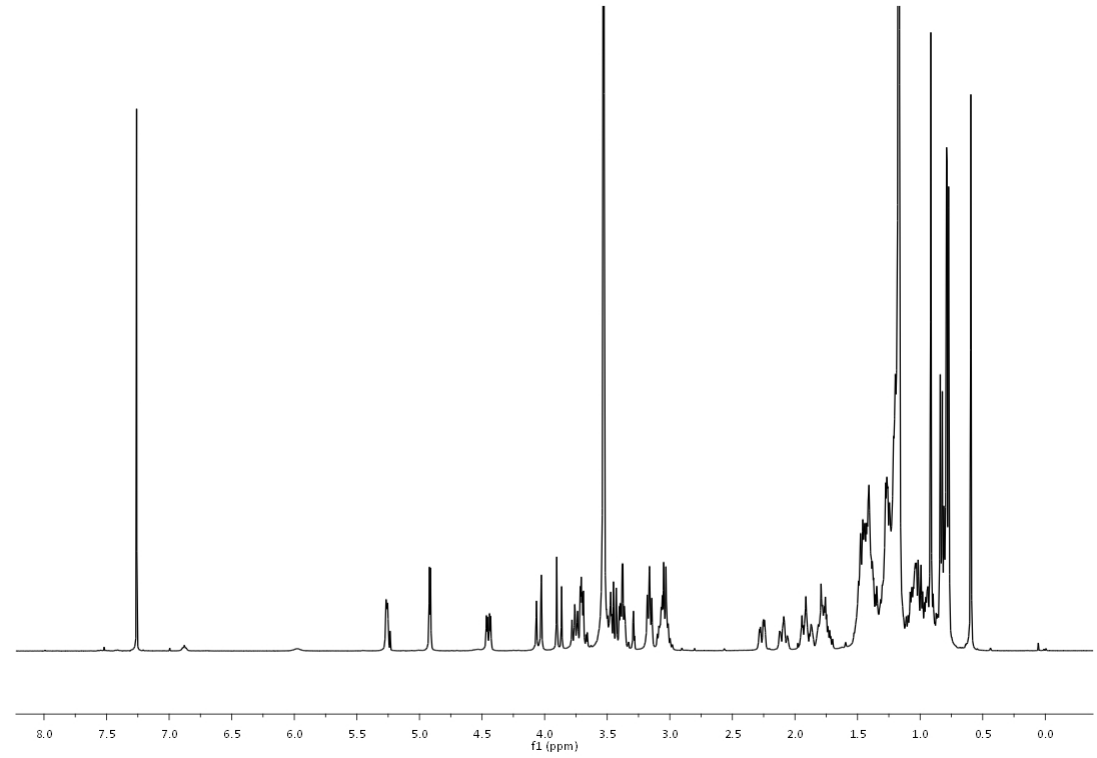


^{13}C NMR

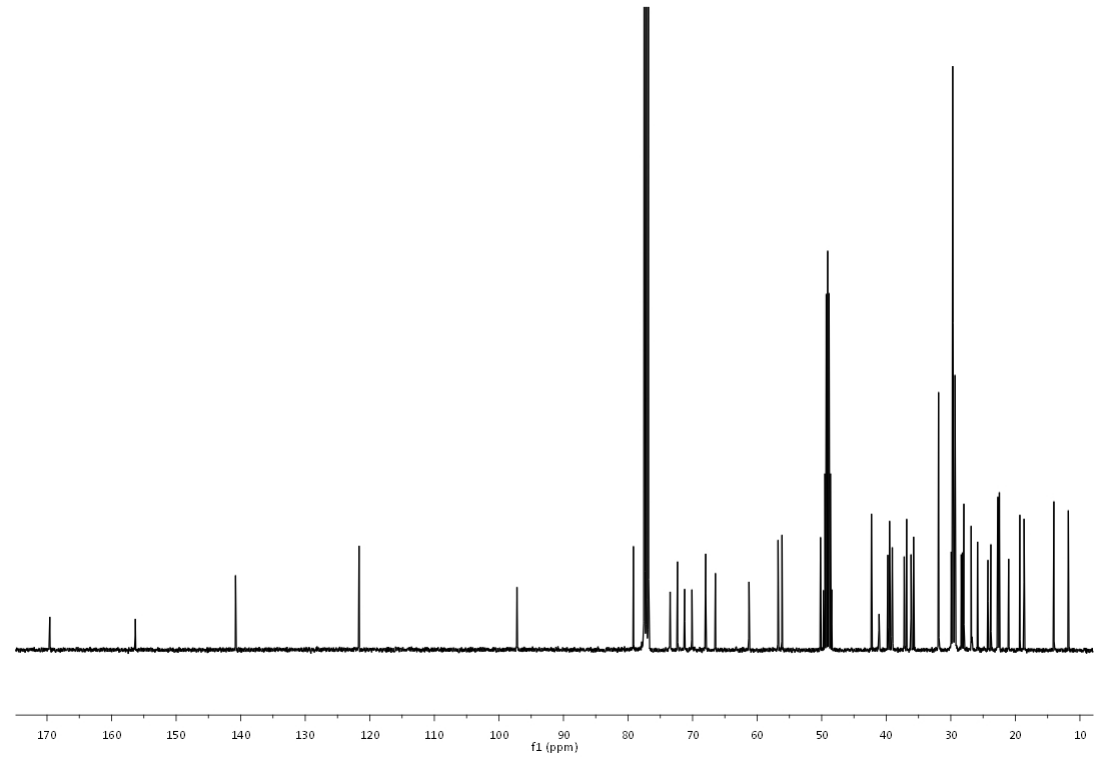


Compound **18e**

^{1}H NMR

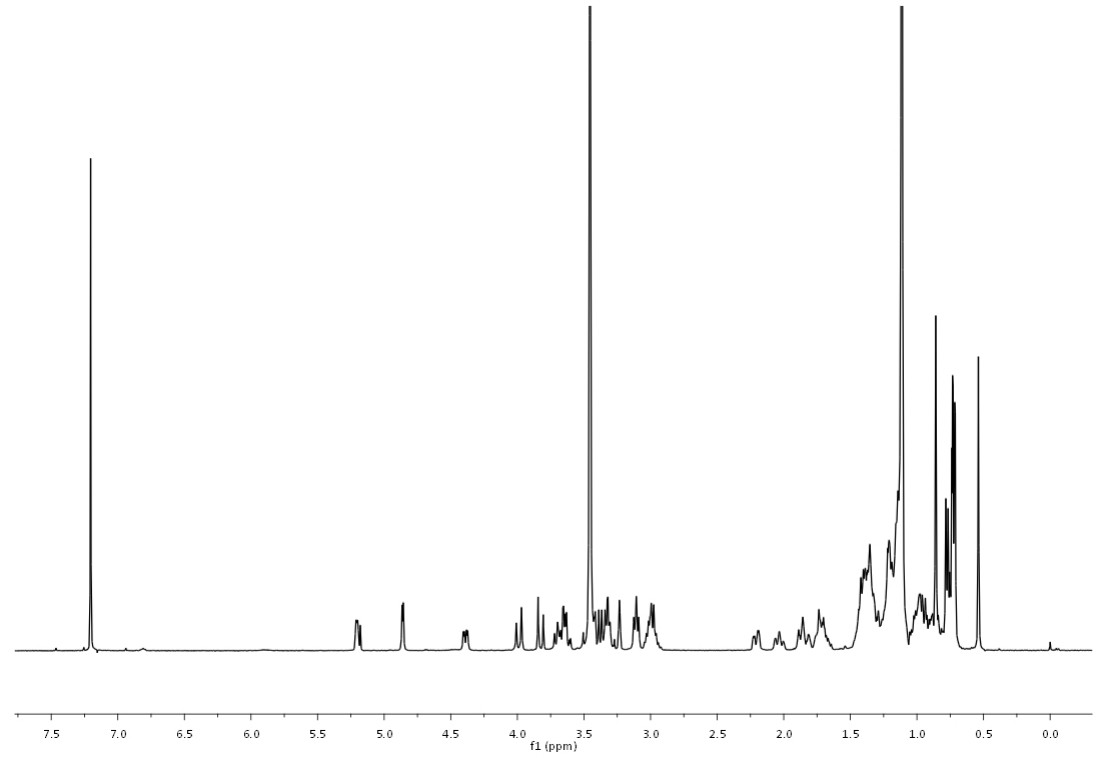


^{13}C NMR

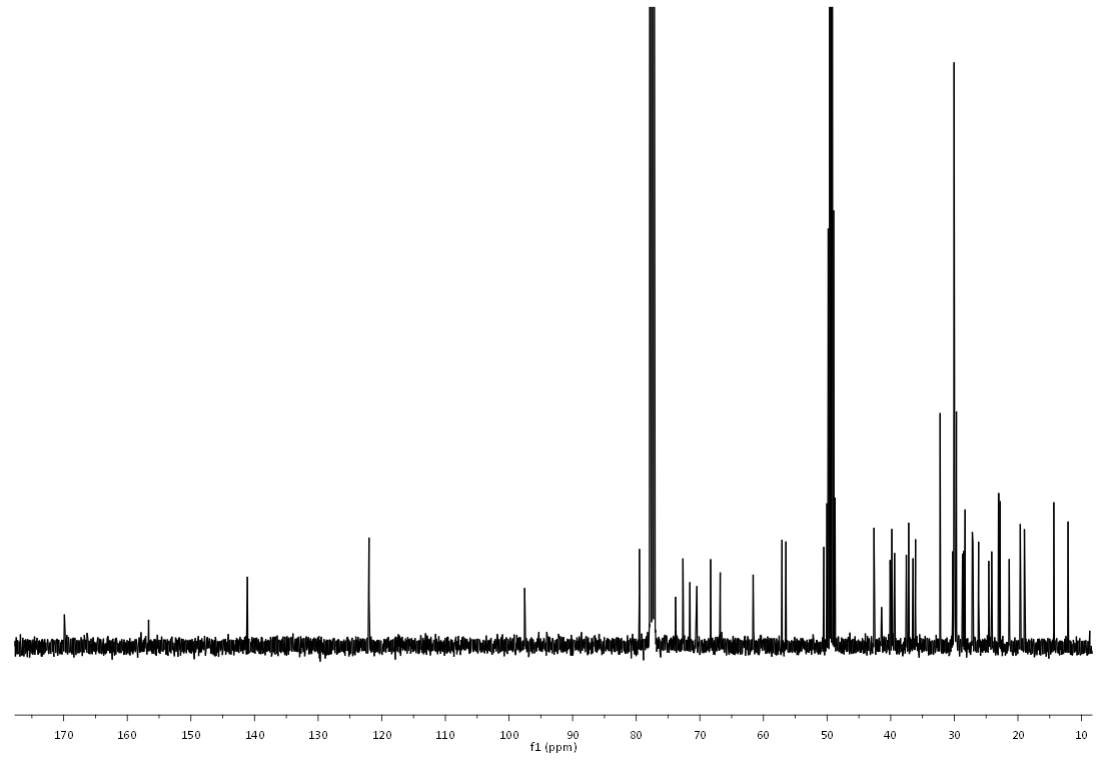


Compound **18f**

^{1}H NMR

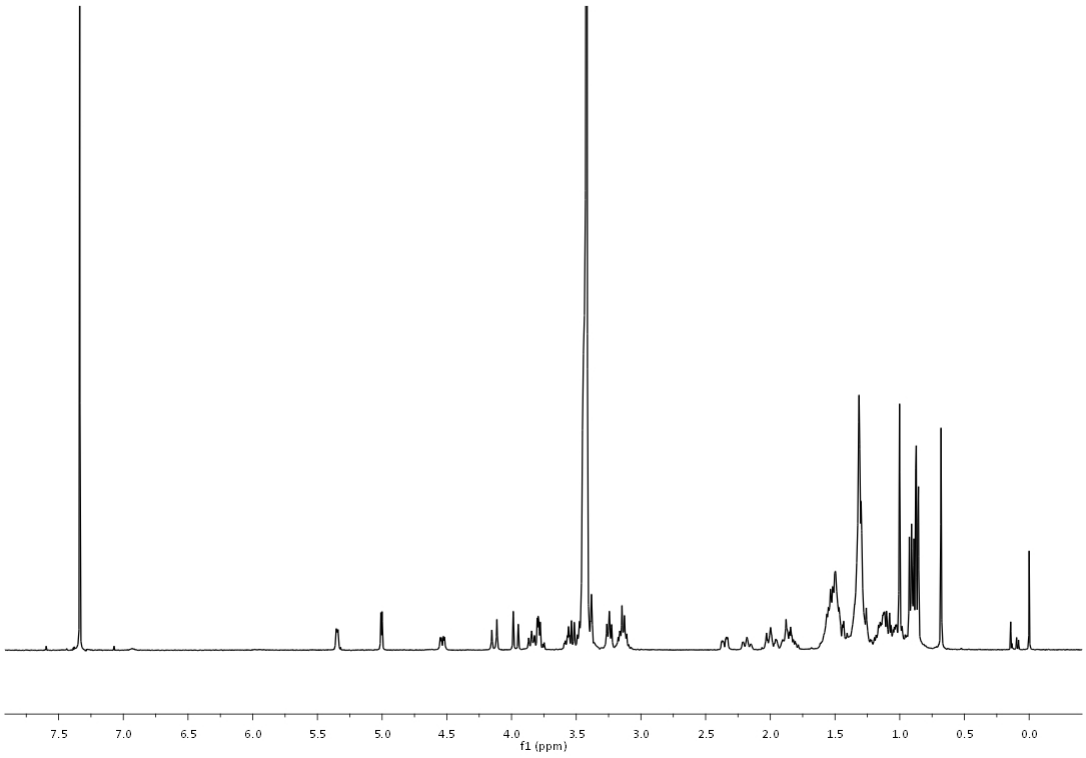


^{13}C NMR

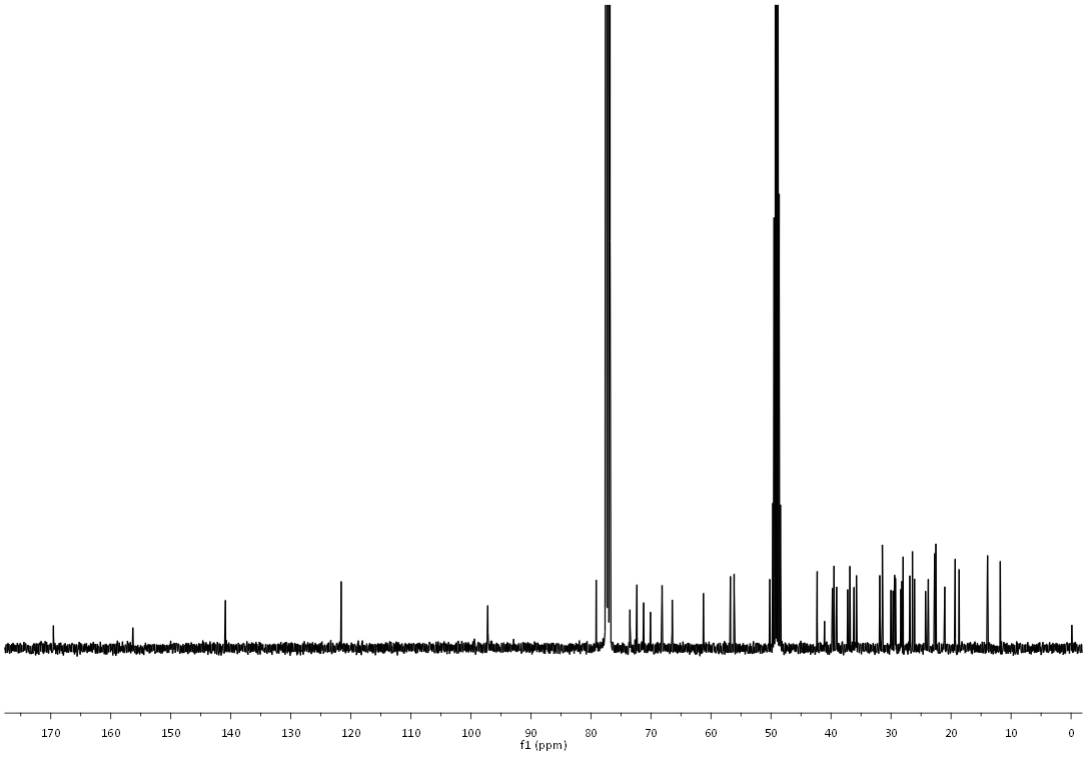


Compound **19a**

^{1}H NMR

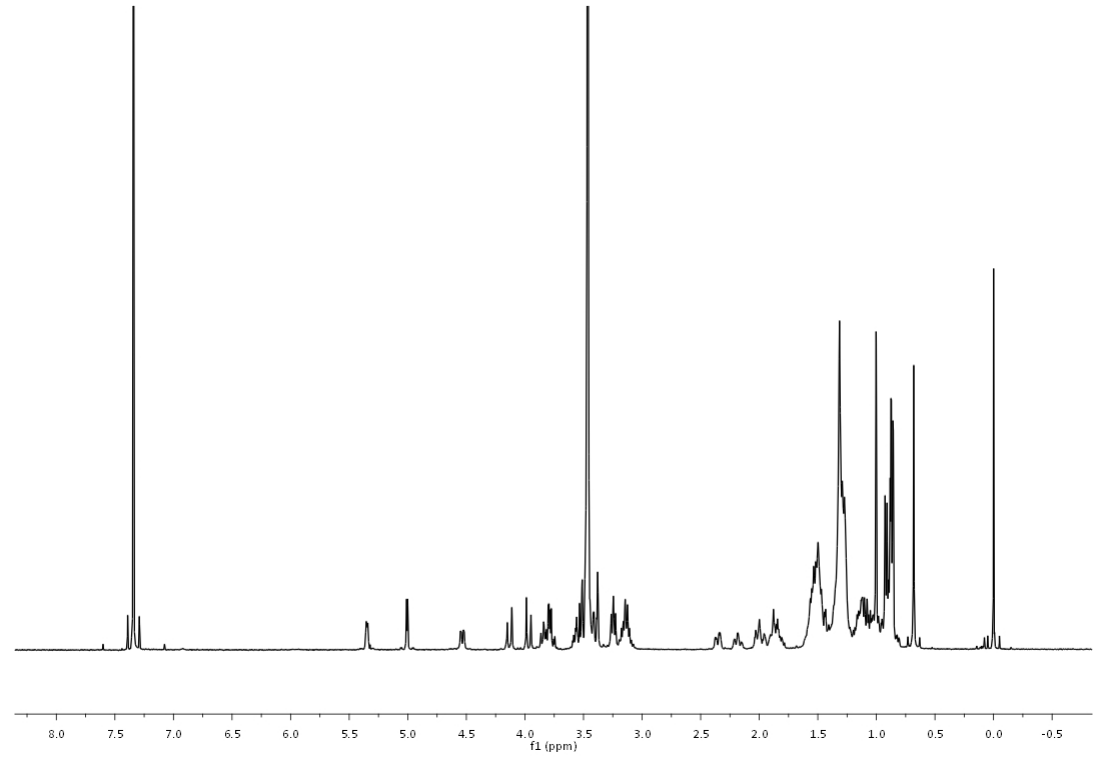


^{13}C NMR

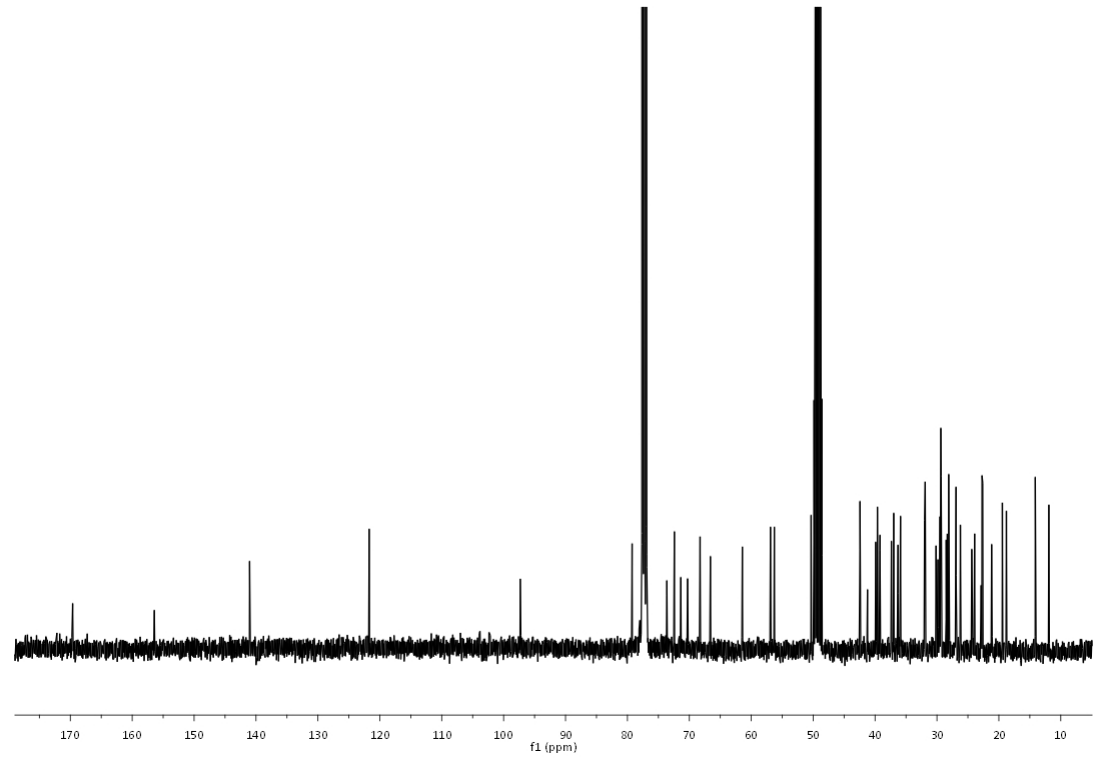


Compound **19b**

^{1}H NMR

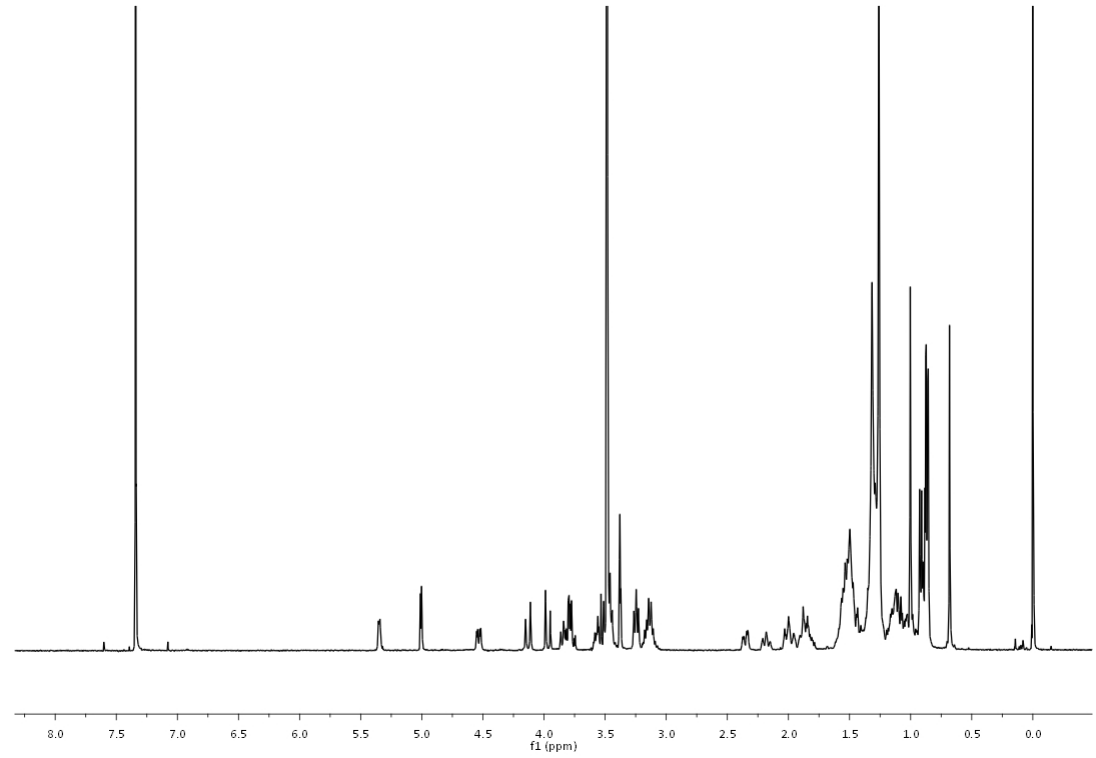


^{13}C NMR

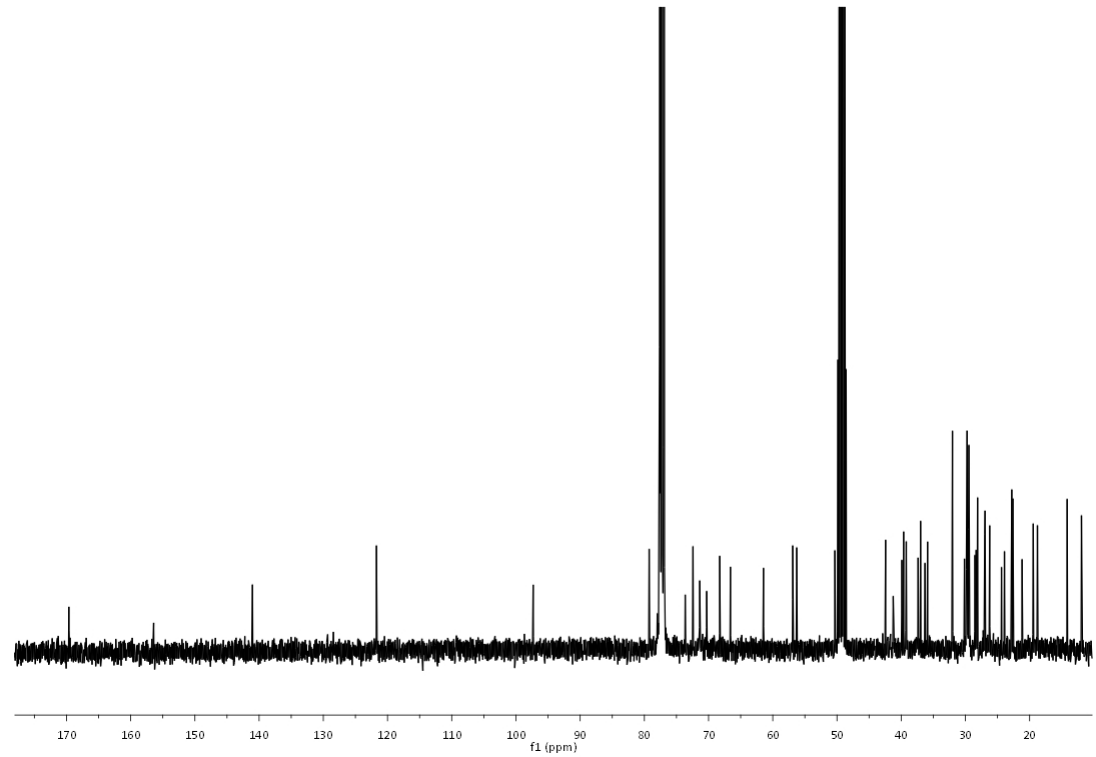


Compound **19c**

^{1}H NMR

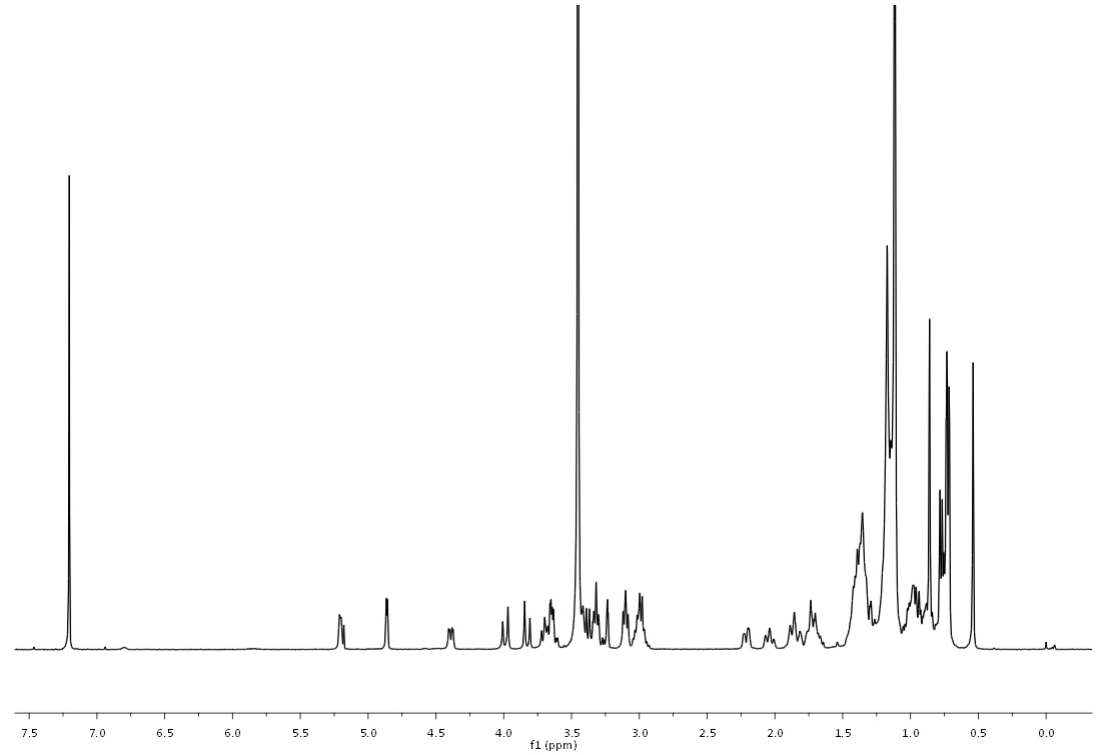


^{13}C NMR

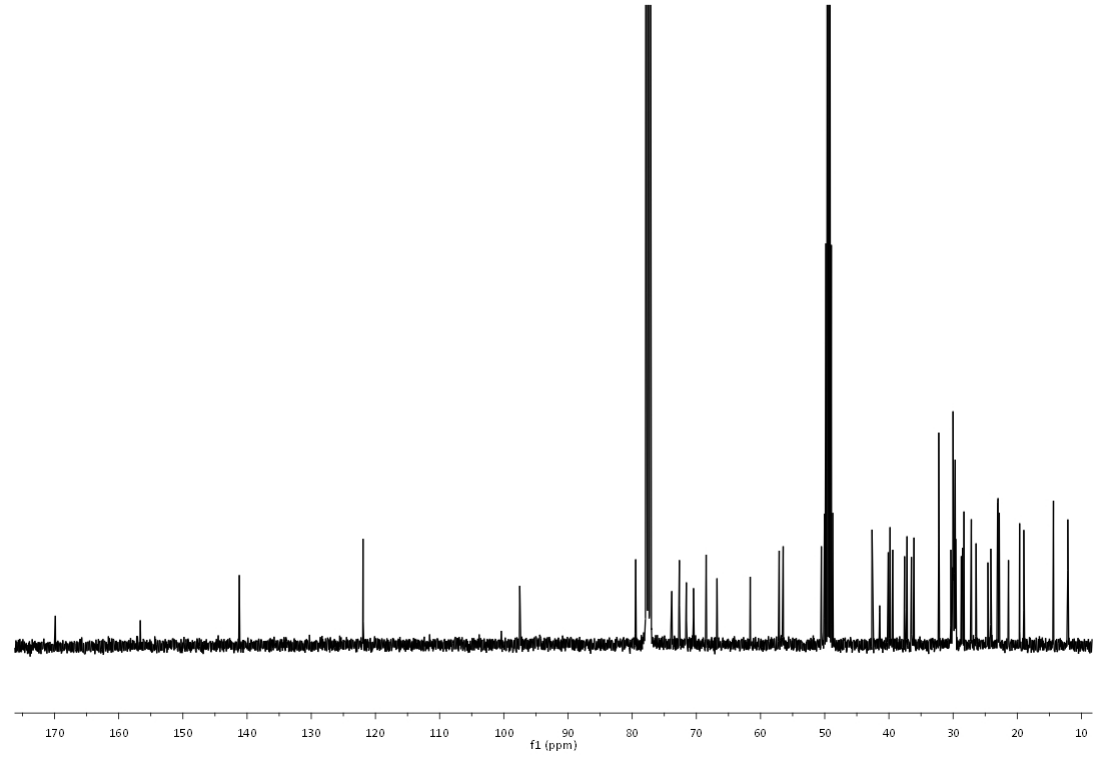


Compound **19d**

^{1}H NMR

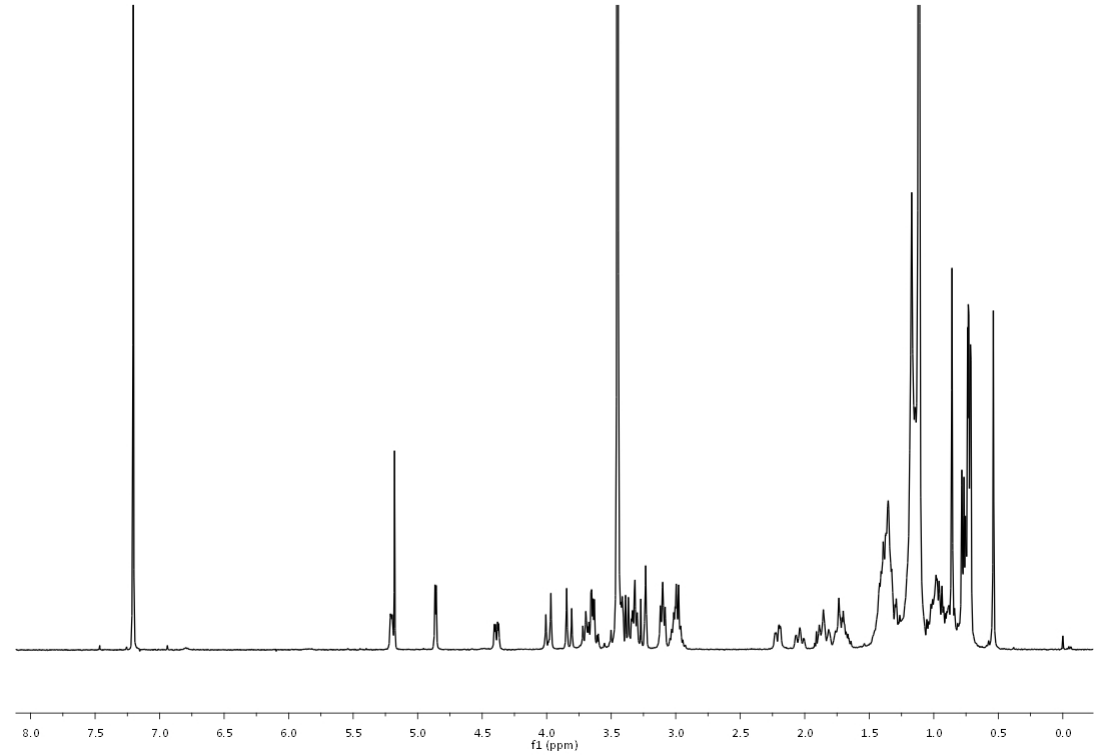


^{13}C NMR

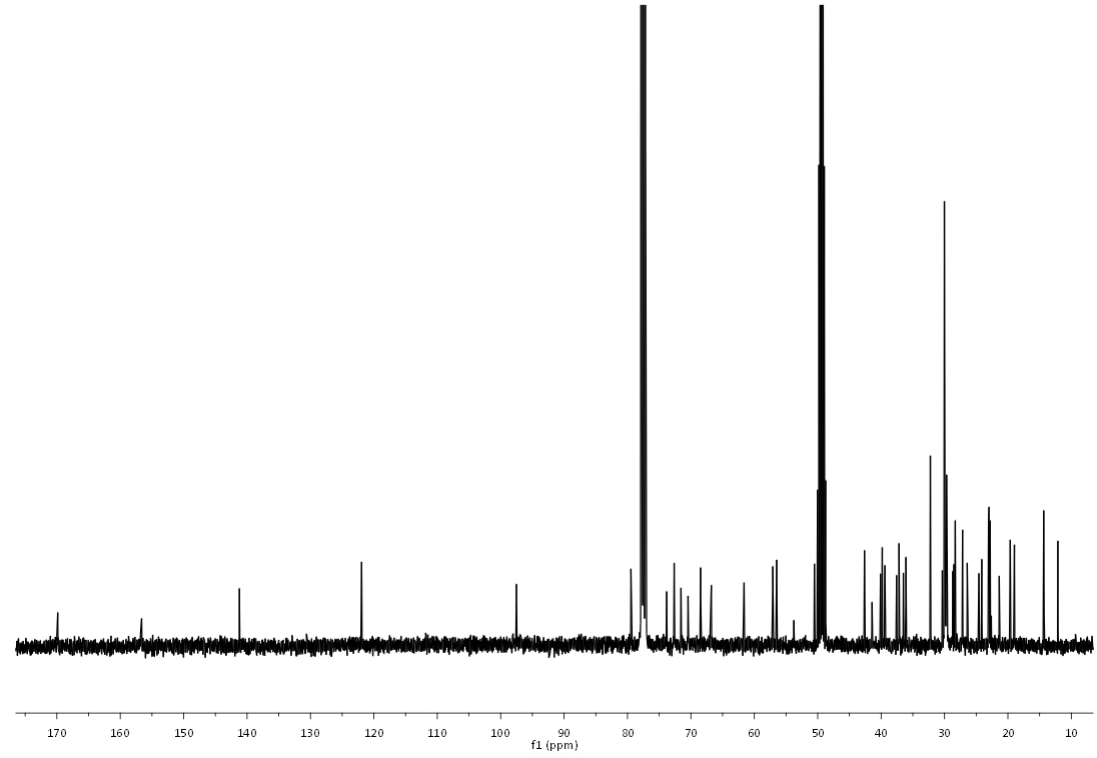


Compound **19e**

^{1}H NMR

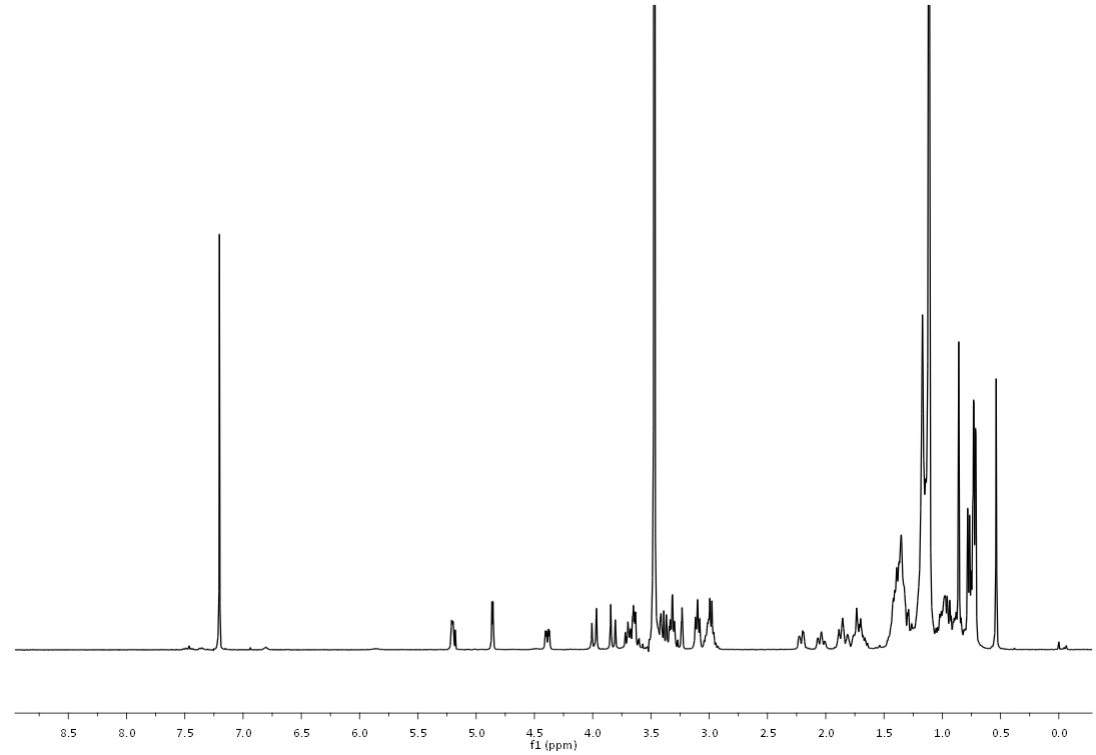


^{13}C NMR

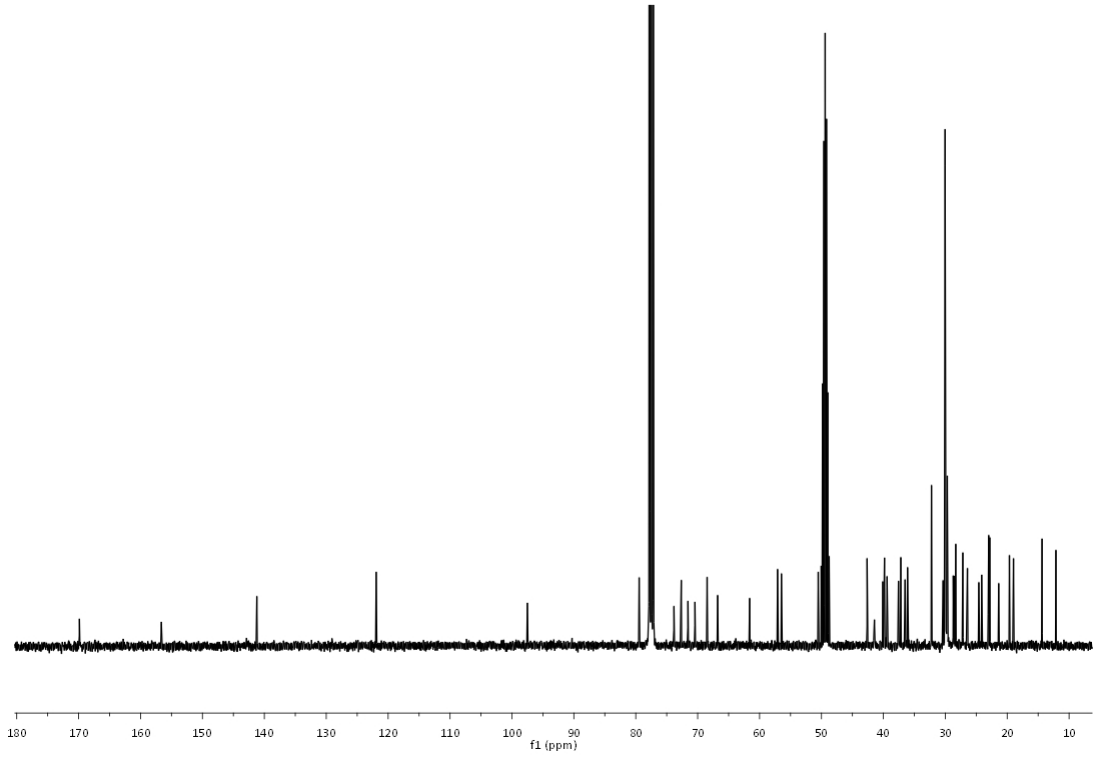


Compound **19f**

^{1}H NMR

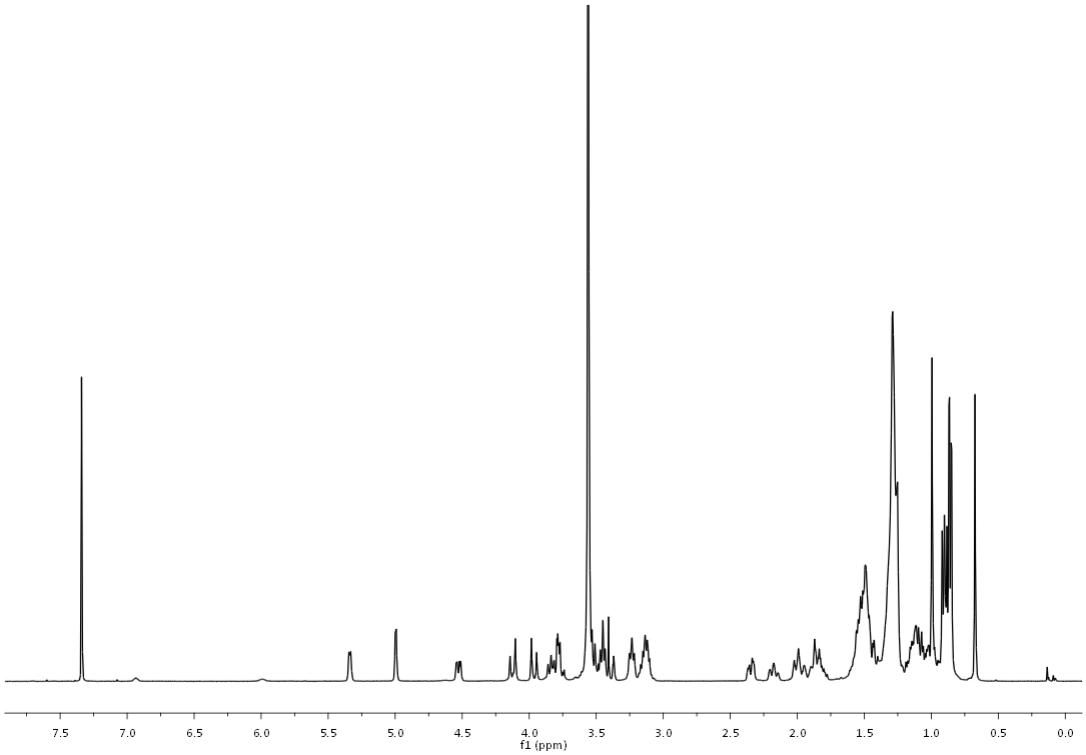


^{13}C NMR

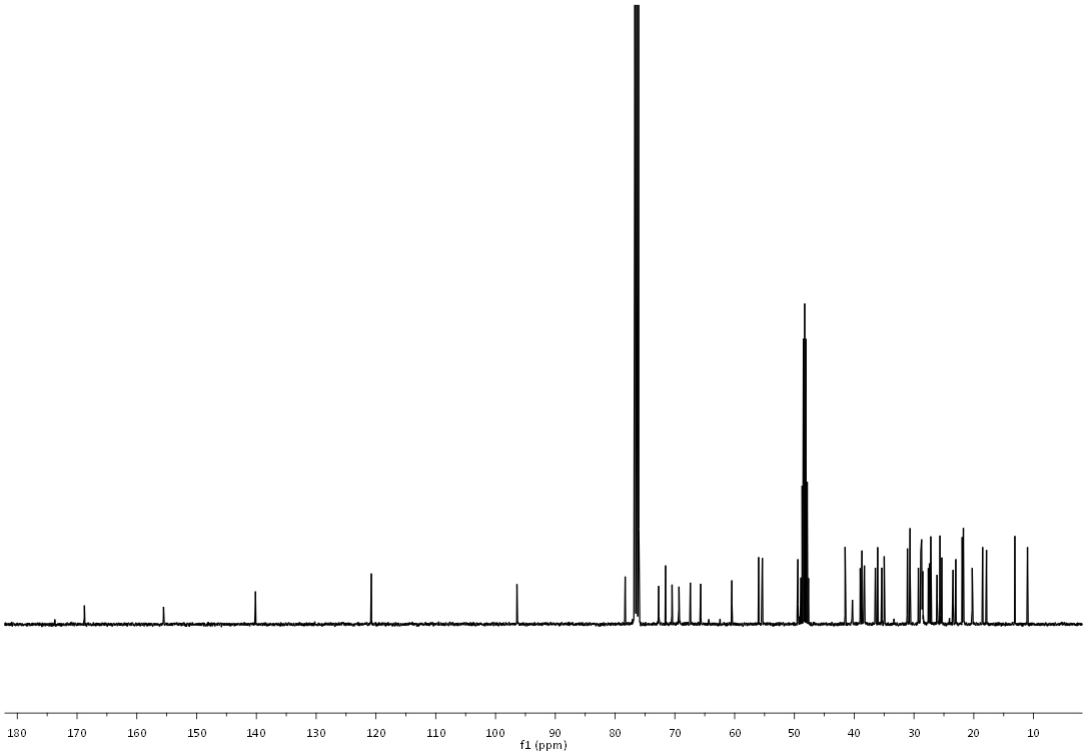


Compound **20a**

^{1}H NMR

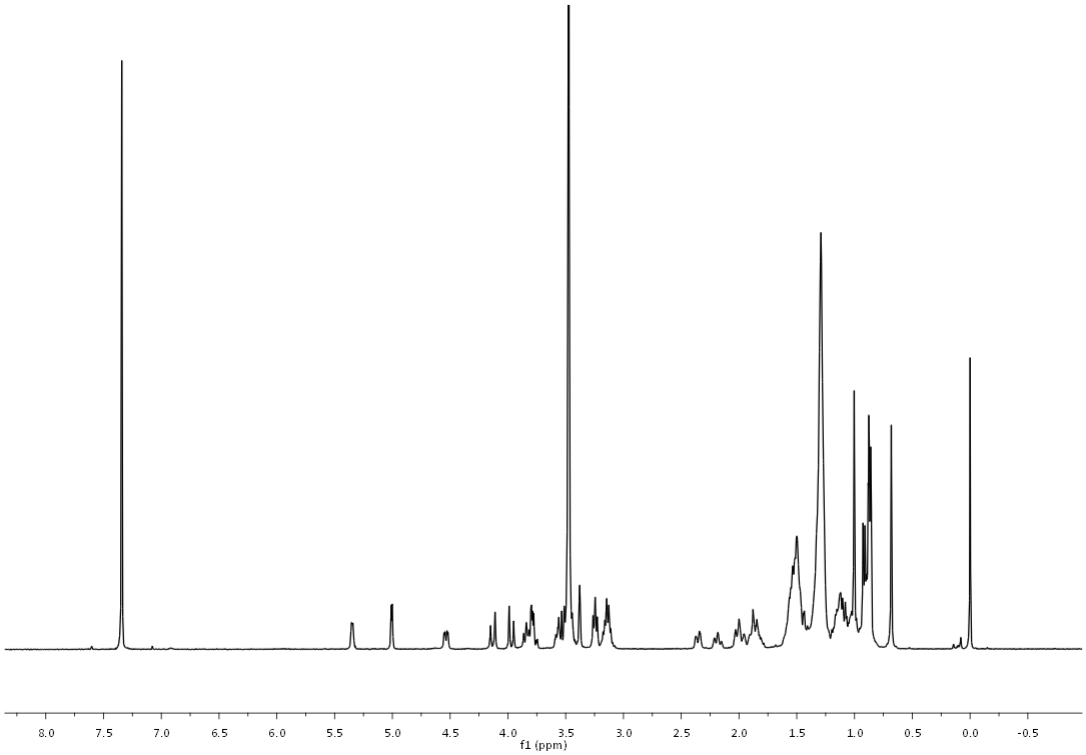


^{13}C NMR

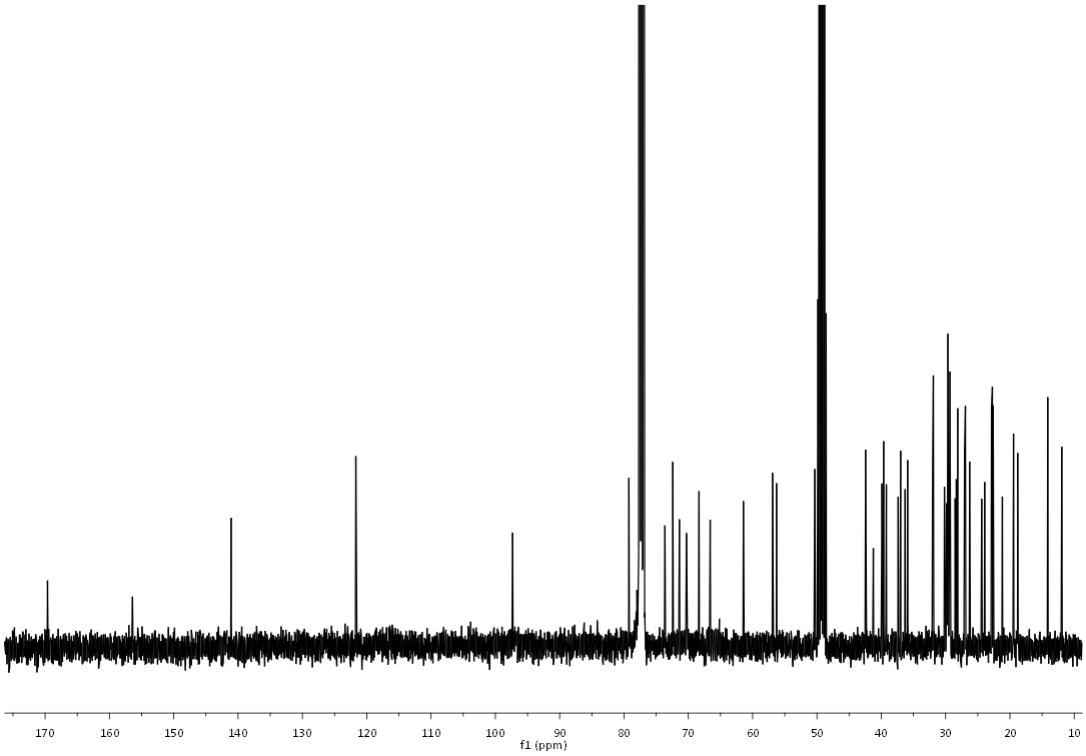


Compound **20b**

^{1}H NMR

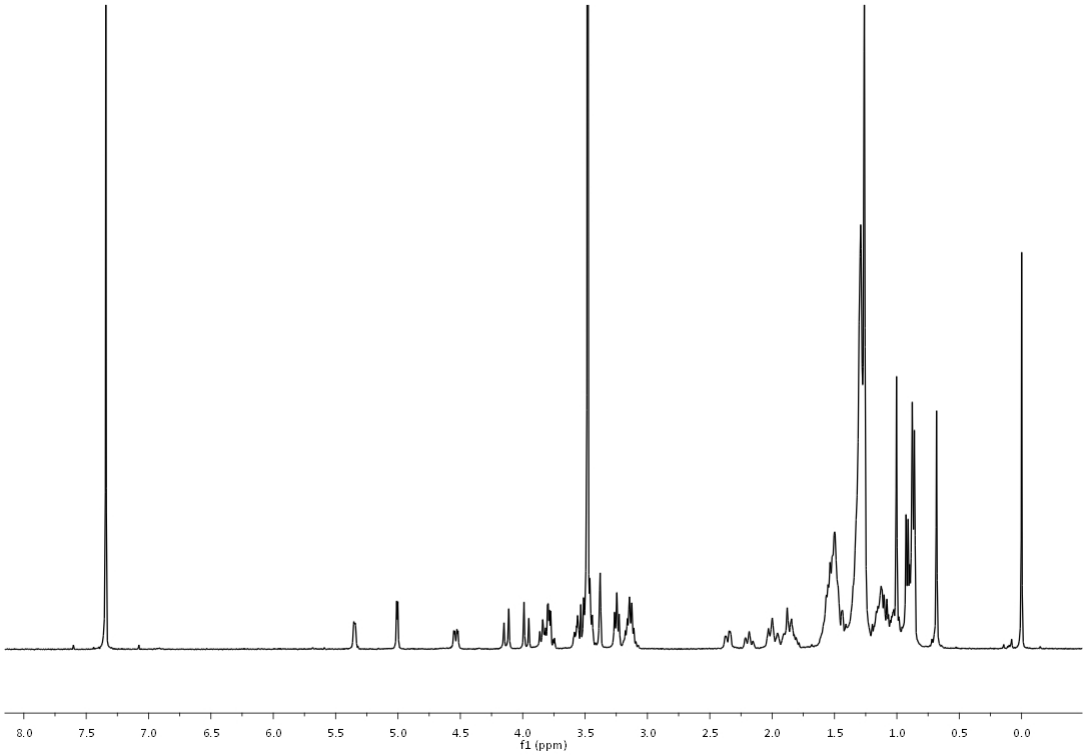


^{13}C NMR

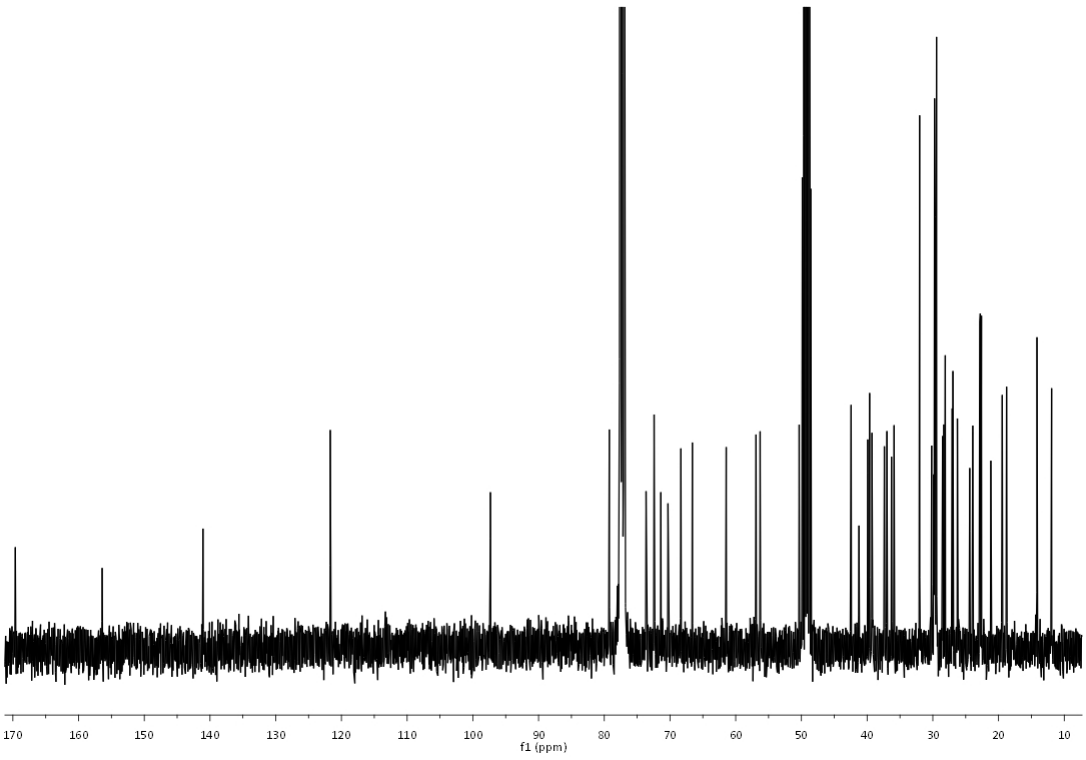


Compound **20c**

^{1}H NMR

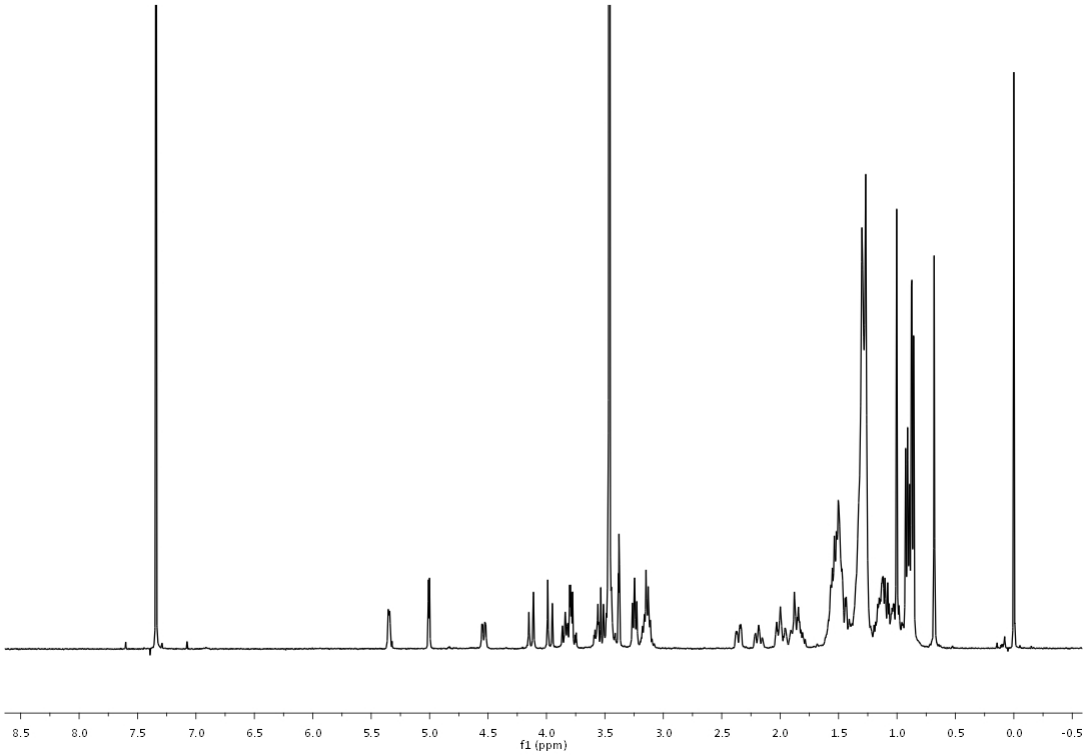


^{13}C NMR

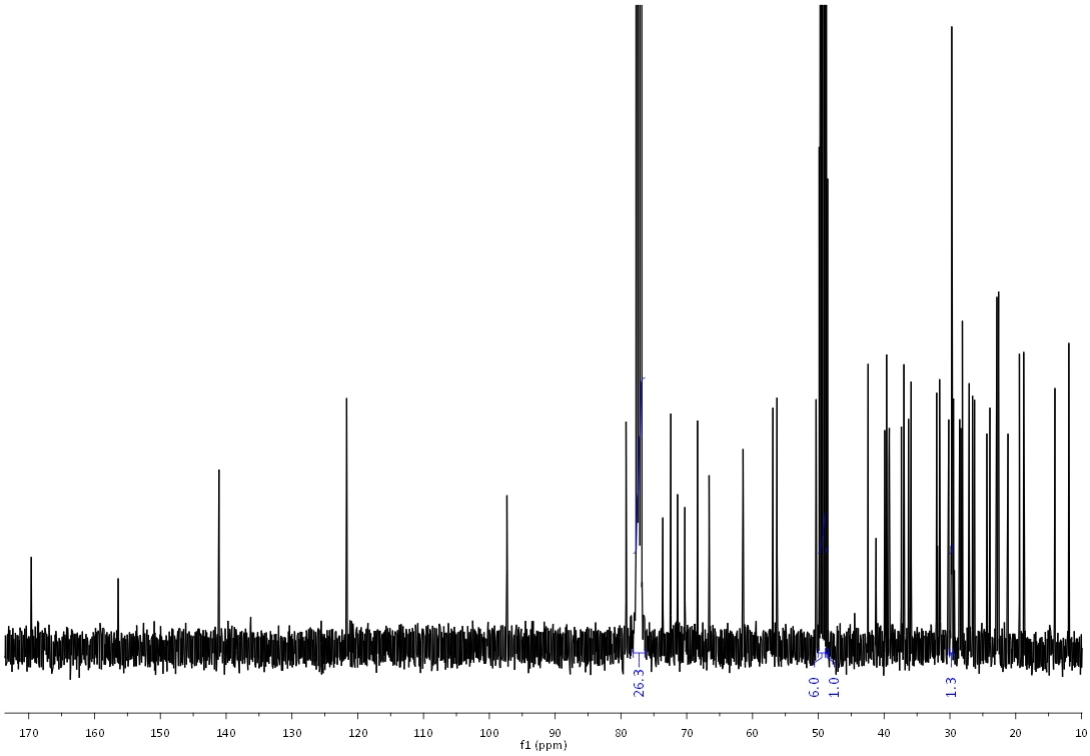


Compound **21a**

^{1}H NMR

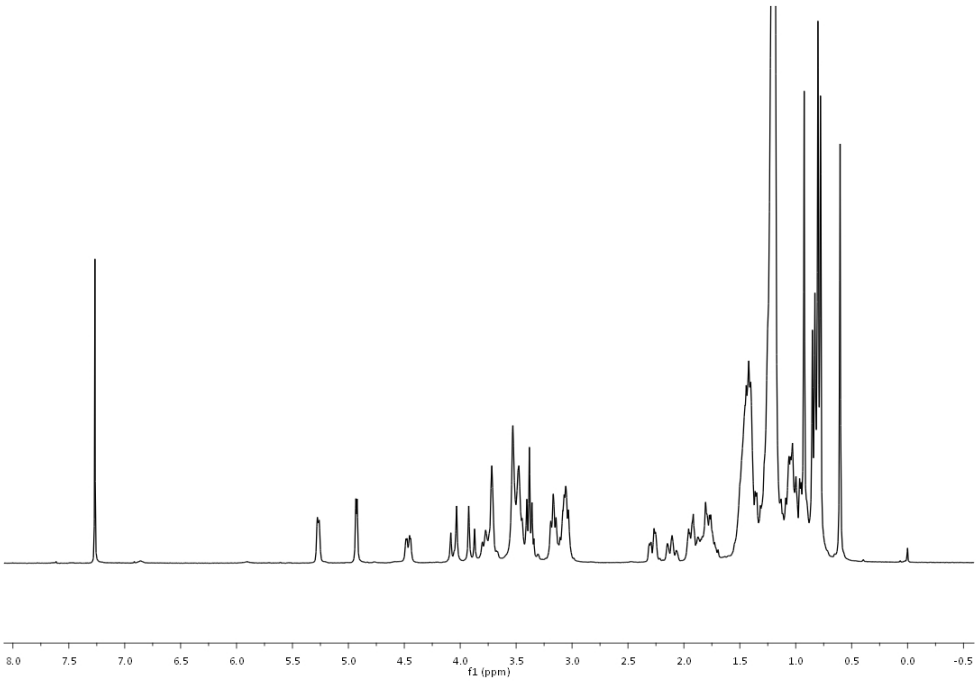


^{13}C NMR

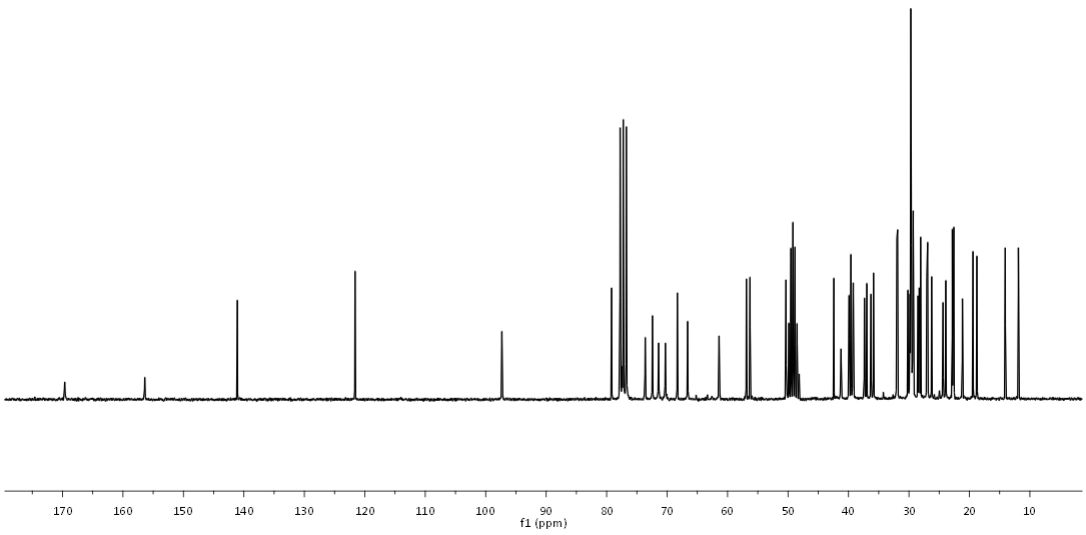


Compound **21b**

^{1}H NMR

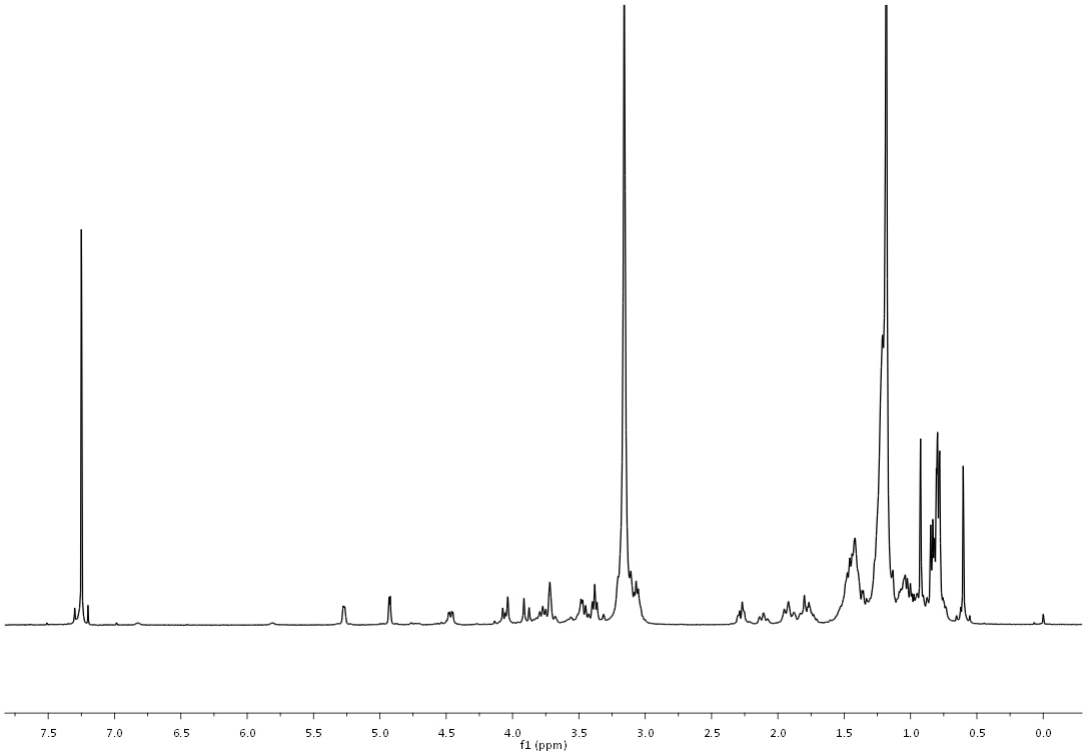


^{13}C NMR

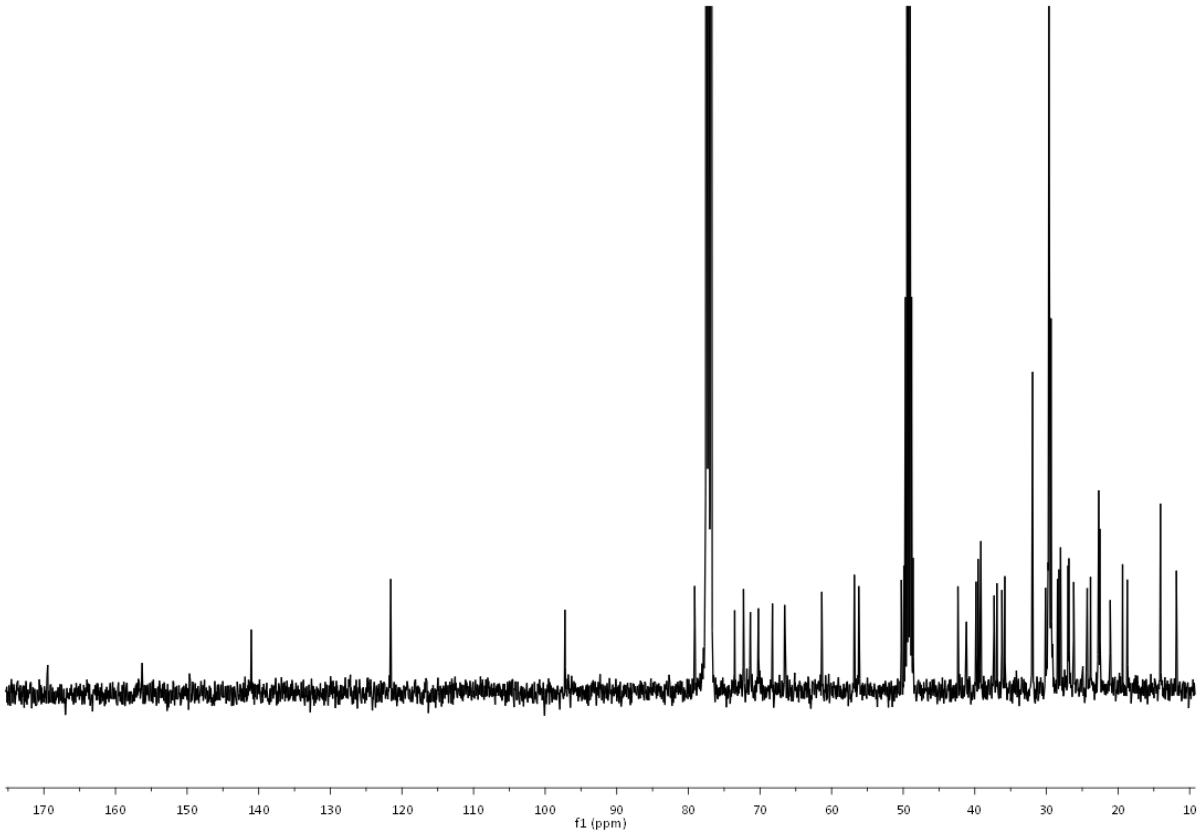


Compound **21c**

^{1}H NMR

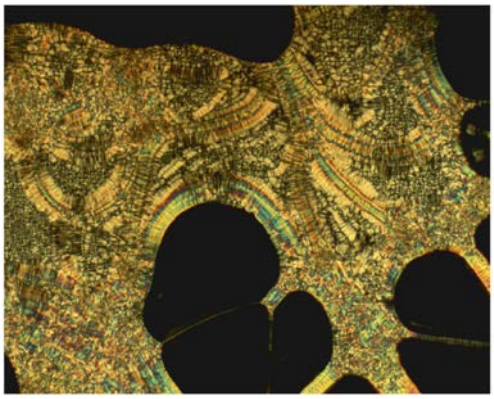


^{13}C NMR



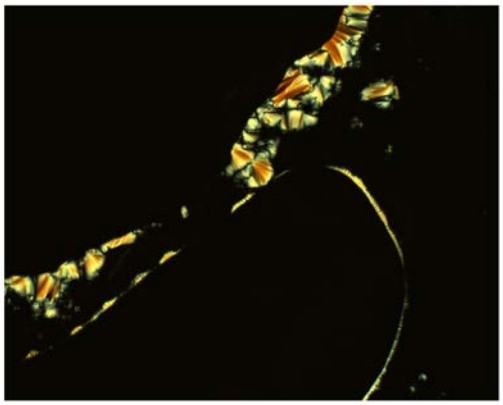
3. Microscopy of target compounds.

Compound **4b**



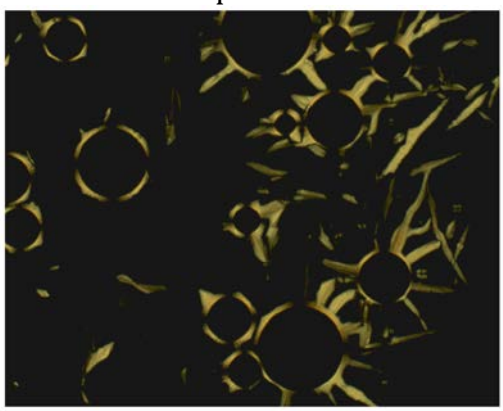
Smectic A phase at 250 °C (x100 magnification)

Compound **5a**



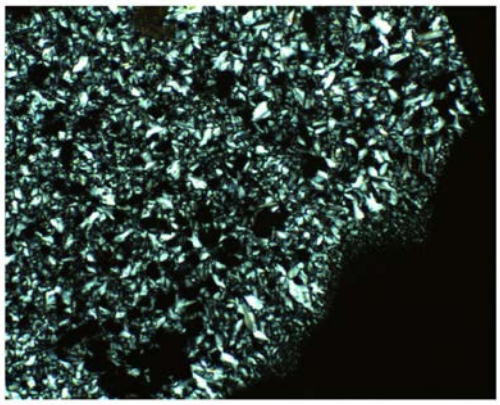
Smectic A phase at 220 °C (x100 magnification)

Compound **5b**



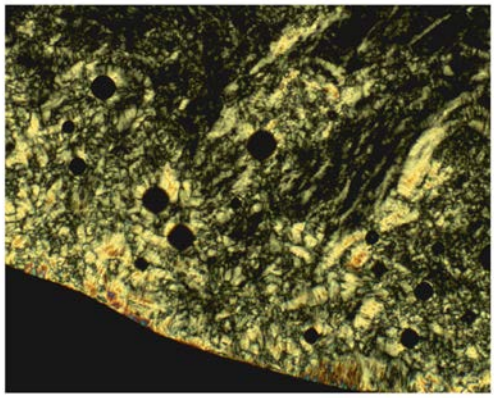
Smectic A phase at 170 °C (x100 magnification)

Compound **5c**



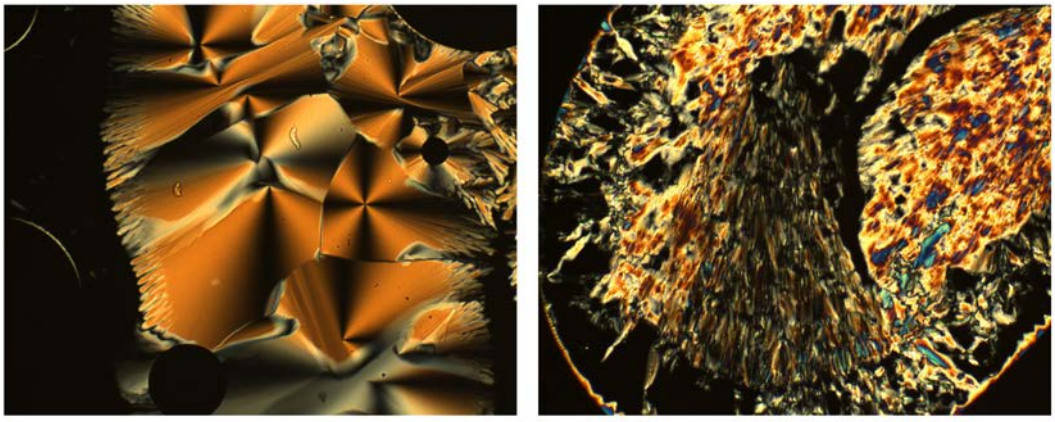
Smectic A phase at 150 °C (x100 magnification)

Compound **5d**



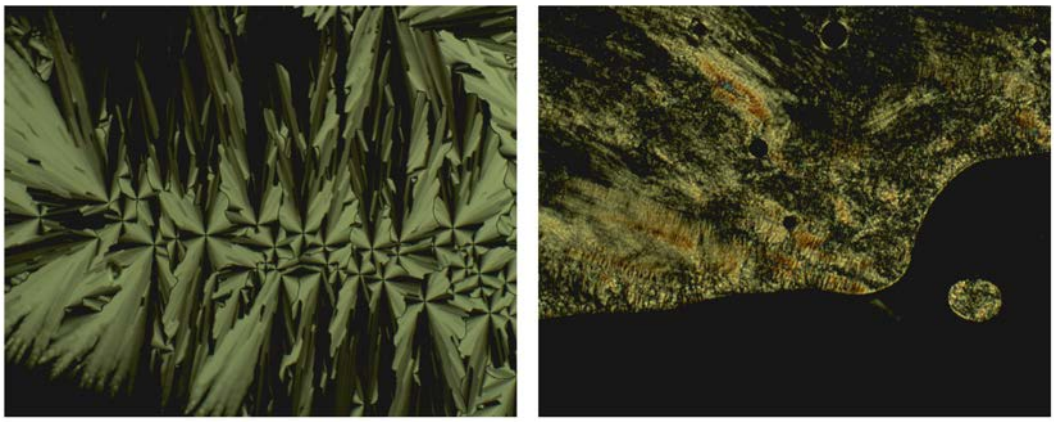
Smectic A phase at 142 °C (x100 magnification)

Compound **5e**



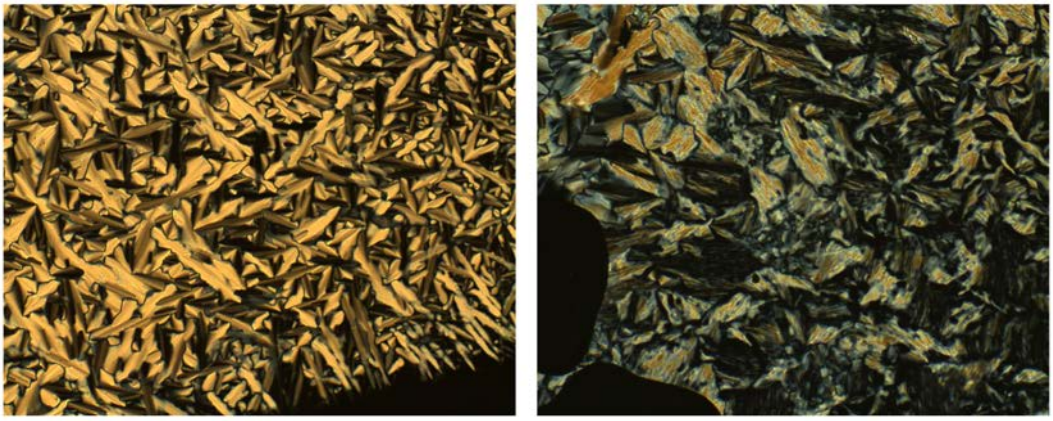
Columnar phase at 156 °C (left) and the Smectic A phase at 140 °C (right) (x100 magnification)

Compound **5f**



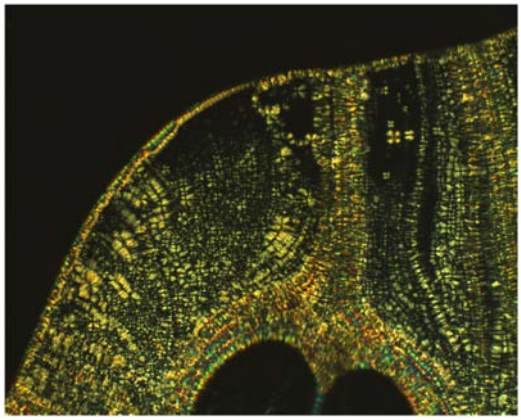
Columnar phase at 159 °C (left) and the Smectic A phase at 140 °C (right) (x100 magnification)

Compound **5g**



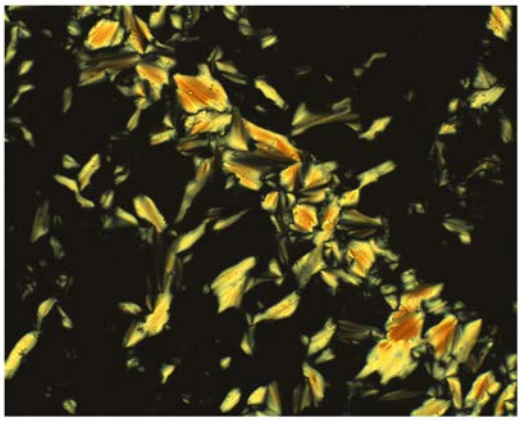
Columnar phase at 175 °C (left) and the transition to the Smectic A phase (right) at 156 °C (x100 magnifiaction)

Compound **14b**



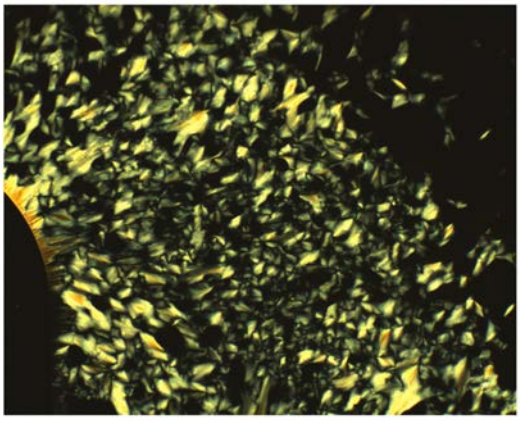
Smectic A phase at 250 °C (x100 magnifiaction)

Compound **18b**



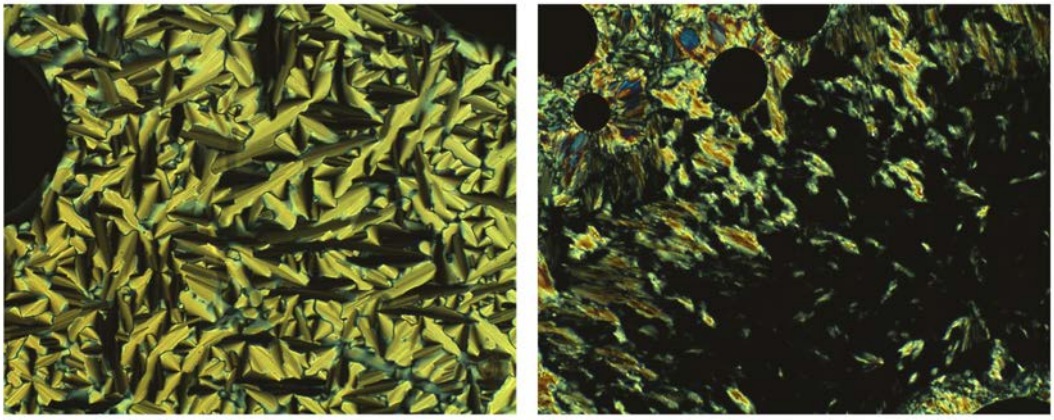
Smectic A phase at 180 °C (x100 magnification)

Compound **18c**



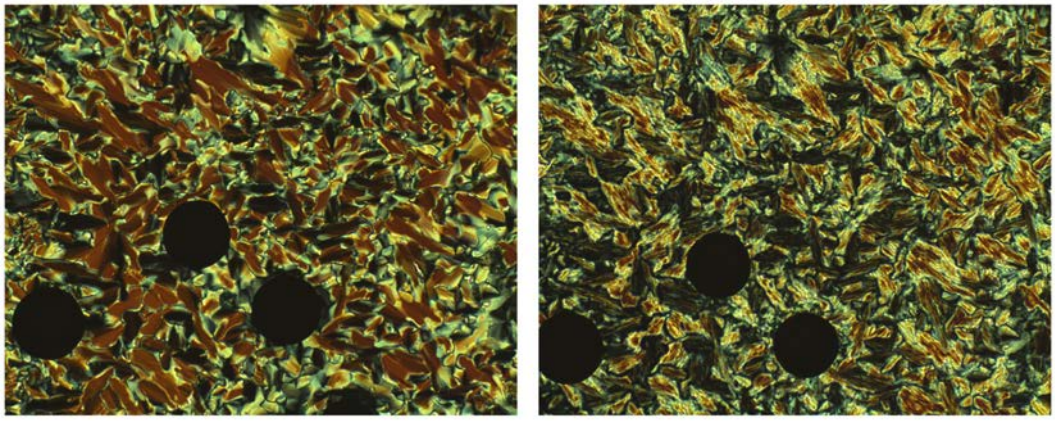
Smectic A phase at 142 °C (x100 magnification)

Compound **18d**



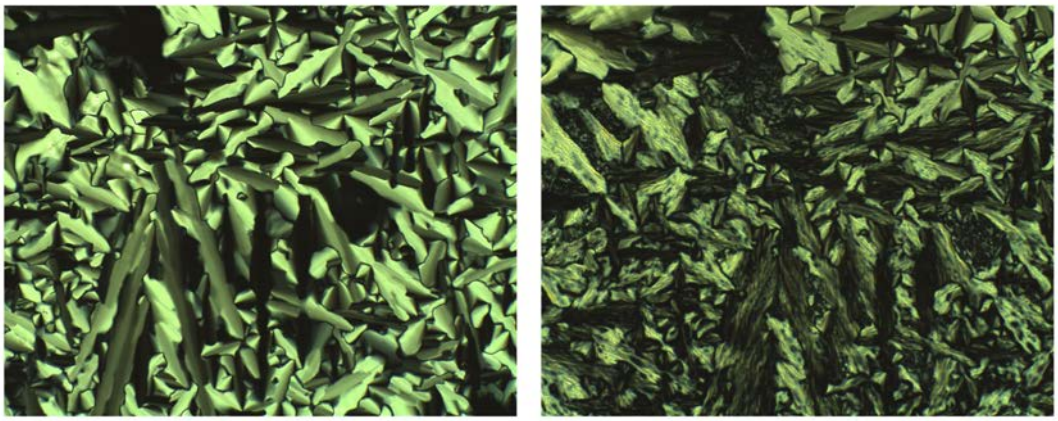
Columnar phase at 140 °C (left) and the Smectic A phase at 110 °C (right) (x100 magnification)

Compound **18e**



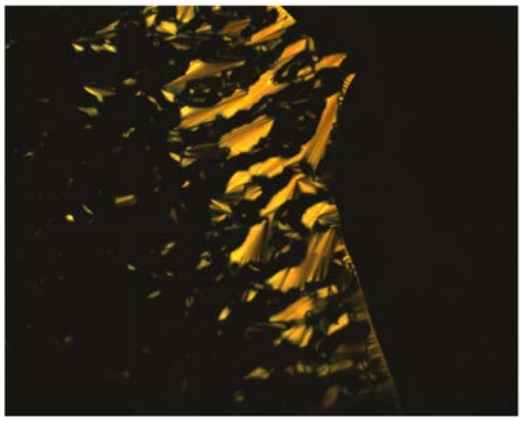
Columnar phase at 142 °C (left) and the transition into the Smectic A phase at 132.5 °C (right) (x100 magnification)

Compound **18f**



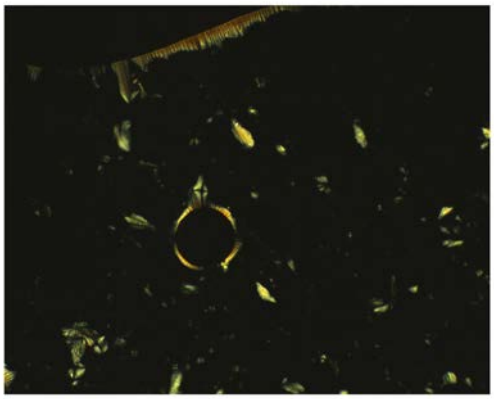
Columnar phase at 151 °C (left) and the Smectic A phase at 137 °C (right) (x100 magnification)

Compound **15b**



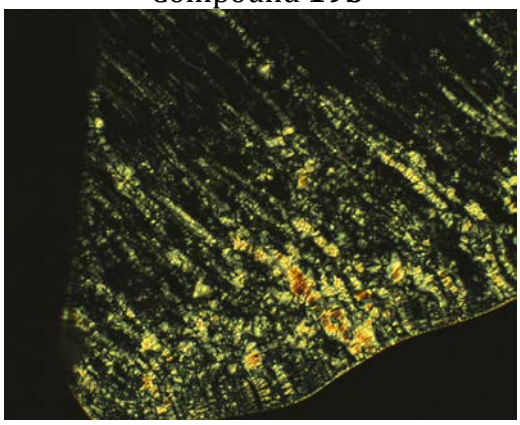
Smectic A phase at 190 °C (x100 magnifiaction)

Compound **19a**



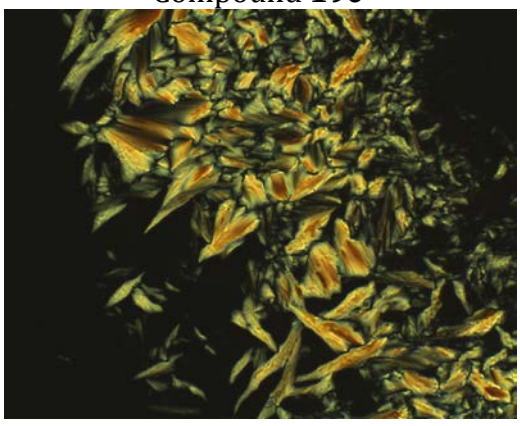
Smectic A phase at 180 °C (x100 magnifiaction)

Compound **19b**



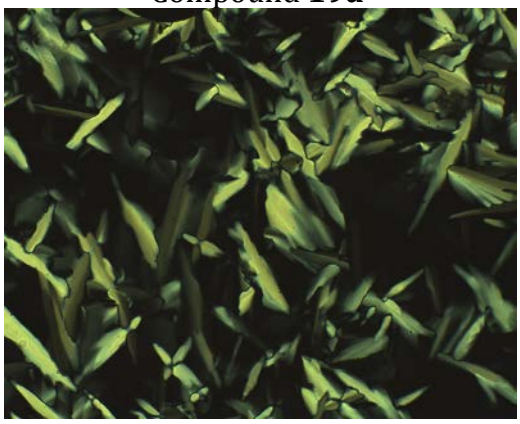
Smectic A phase at 160 °C (x100 magnification)

Compound **19c**



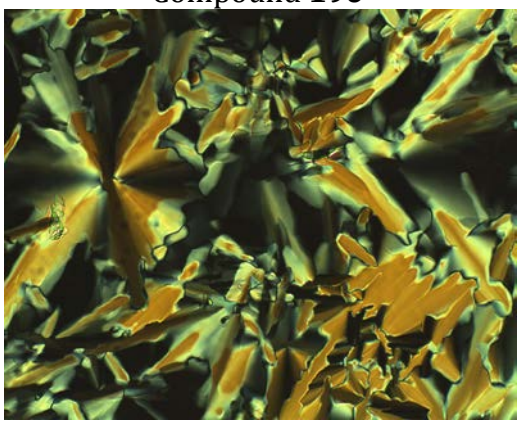
Smectic A phase at 152 °C (x100 magnication)

Compound **19d**



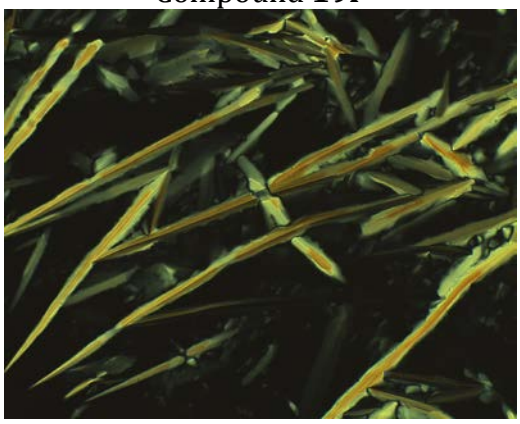
Columnar phase at 137.5 °C (x100 magnification)

Compound **19e**



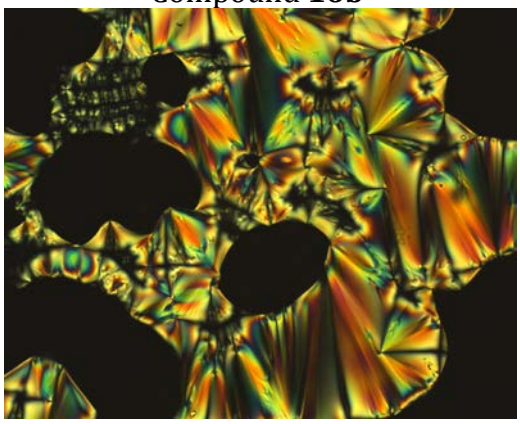
Columnar phase at 126 °C (x100 magnification)

Compound **19f**



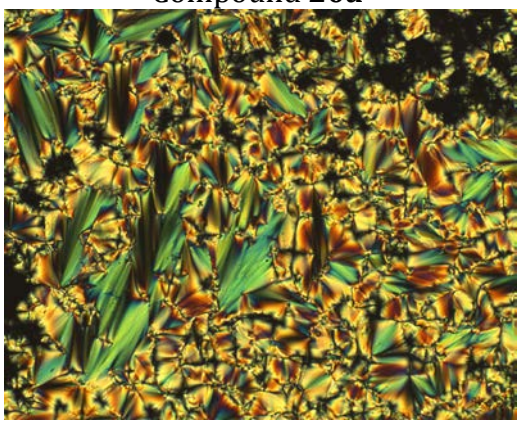
Columnar phase at 155 °C (x100 magnification)

Compound **16b**



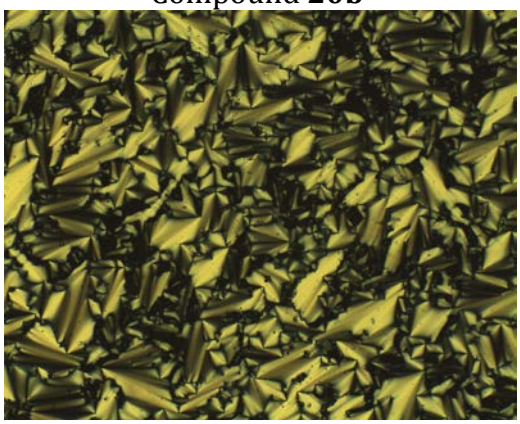
Smectic A phase at 225 °C (x100 magnification)

Compound **20a**



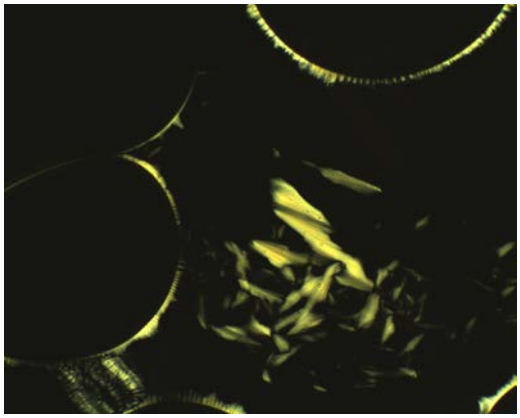
Smectic A phase at 150 °C (x100 magnification)

Compound **20b**



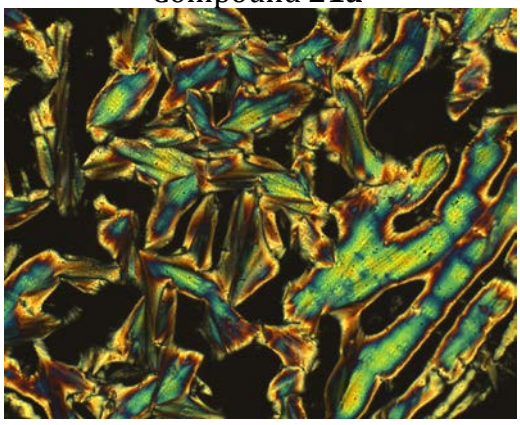
Smectic A phase at 150 °C (x100 magnification)

Compound **17b**



Smectic A phase at 220 °C (x100 magnification)

Compound **21a**



Smectic A phase at 178 °C (x100 magnification)