Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

S1. CD spectrum of $(dC)_3$ in HEPES buffer

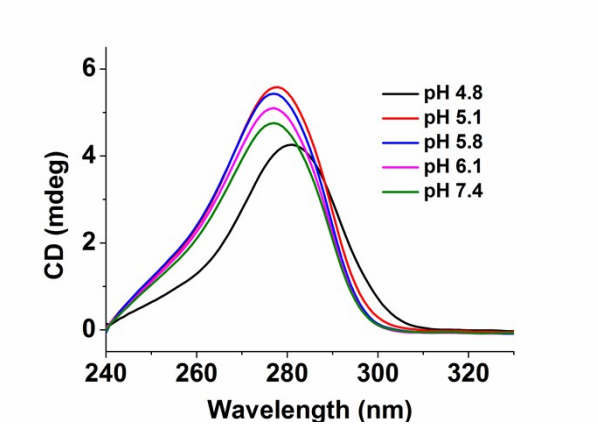


Figure S1. CD spectrum of $(dC)_3$ in HEPES buffer

S2. The Packing parameters of $C_n(dC)_3$ and DLS data of nucleolipids.

$$\Gamma_{max} = -\frac{1}{2.303nRT}\left(\frac{\partial\gamma}{\partial logC}\right)_T$$

$$A_{min} = \left(N_A\Gamma\right)^{-1} \times 10^{16}$$

$$P = \frac{V_c}{A_{min} \times l_c} = \frac{(27.4 + 26.9 \times n) \times 10^{-3}\, nm^3}{A_{min} \times (0.15 + 0.1265 \times n) nm}$$

Table S1. The Packing parameters of $C_n(dC)_3$ at different pH

		$10^{10}\Gamma_{max}$ (mol/m^2)	A_{min} (nm^2/molecule)	P
pH 4.8	$C_{10}(dC)_3$	1.63	1.0	0.21
	$C_{12}(dC)_3$	1.06	1.56	0.13
	$C_{14}(dC)_3$	1.10	1.51	0.14
	$C_{16}(dC)_3$	2.37	0.74	0.29
pH 7.4	$C_{10}(dC)_3$	1.37	1.21	0.17
	$C_{12}(dC)_3$	1.86	0.91	0.28
	$C_{14}(dC)_3$	2.01	0.83	0.25
	$C_{16}(dC)_3$	1.02	1.66	0.13

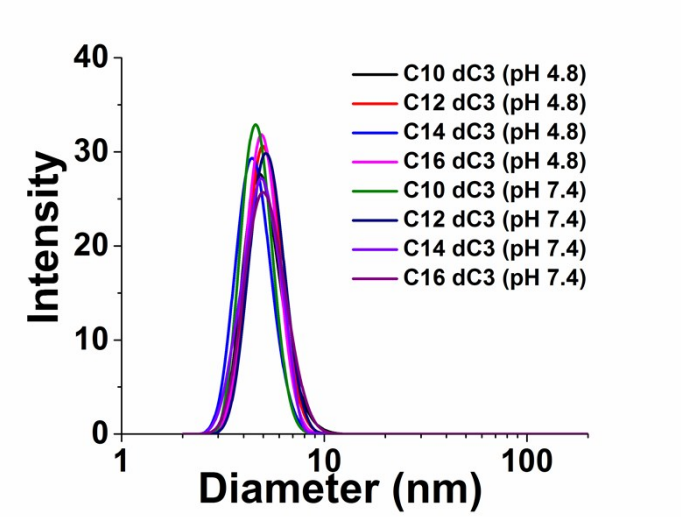


Figure S2. DLS of $C_n(dC)_3$ in HEPES buffer (concentration was set at 100 μM)

S3. NMR and MS data of compound 1-24

Compound 1

^{1}H NMR ($CDCl_3$, 400 MHz): δ = 0.88 (t, C*H*$_3$, 3H), 1.70 (m, C*H*$_2$, 16H), 2.27 (m, 2-*H*, 2H), 2.78-2.81 (m, C*H*$_2$CN+2-*H*, 3H), 3.97-3.98 (m, 5-*H*, 2H), 4.09-4.11 (m, OCH$_2$C*H*$_2$CN, 2H), 4.27 (m, C*H*$_2$O, 2H), 4.35 (m, 3-*H*, 1H), 8.29-8.31 (d, *J* = 8 Hz, N*H*, 1H), 7.92-7.94 (d, *J* = 8 Hz, Ar*H*, 2H), 7.52-7.53, 7.62 (d, *J* = 4 Hz, Ar*H*, 5H), 6.24 (t, 1-*H*, 1H), 5.18 (m, 4-*H*, 1H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 168.30, 163.46, 150.84, 140.69, 131.63, 128.88, 127.87, 125.34, 118.68, 102.55, 85.83, 84.38, 78.75, 68.29, 62.85, 61.39, 31.72, 30.87, 30.05, 29.36, 28.93, 25.30, 22.53, 19.57, 14.40. High Resolution MS calcd for $C_{29}H_{41}N_4O_8P$, 604.2662; found 605.2727 ($M+H^+$).Yield 82%.

Compound 2

^{1}H NMR ($CDCl_3$, 400 MHz): δ = 0.88 (t, C*H*$_3$, 3H), 1.70 (m, C*H*$_2$, 20H), 2.27 (m, 2-*H*, 1H), 2.77-2.80 (m, C*H*$_2$CN+2-*H*, 3H), 3.95-3.97 (m, *5*-*H*, 2H), 4.09-4.11 (m, OC*H*$_2$CH$_2$CN, 2H), 4.27-4.28 (m, C*H*$_2$O, 2H), 4.34-4.35 (m, 3-*H*, 1H), 5.18 (m, 4-*H*, 1H), 6.23-6.26 (t, 1-*H*, 1H), 7.51-7.53, 7.61 (d, J = 8 Hz, Ar*H*, 4H), 7.90-7.92 (d, *J* = 8 Hz, Ar*H*, 2H), 8.27-8.29 (d, *J* = 8 Hz, Ar*H*, 1H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz):168.29, 163.45, 150.83, 140.69, 134.69, 131.63, 128.62, 127.87, 118.68, 102.55, 84.41, 68.29, 62.80, 61.39, 31.74, 30.87, 30.05, 29.48, 29.35, 28.93, 25.30, 22.53, 19.57, 14.40. High Resolution MS calcd for $C_{31}H_{45}N_4O_8P$, 632.2975; found 633.3073 ($M + H^+$);

655.2886 (M + Na^+).Yield 84%.

Compound 3

^{1}H NMR ($CDCl_3$, 400 MHz): δ =0.88 (t, C*H*$_3$, 3H), 1.70 (m, C*H*$_2$, 24H),2.50-2.52 (m, 2-*H*, 1H),2.77-2.80 (m, C*H*$_2$CN+2-H, 3H), 3.95-3.96 (m, 5-*H*, 2H), 4.08-4.11 (m, OC*H*$_2$CH$_2$CN, 2H), 4.34-4.35 (m, 3-*H*+C*H*$_2$O, 4H), 5.20-5.21 (m, 4-*H*, 1H), 6.25 (t, 1-*H*, 1H), 7.50-7.52, 7.60 (d, *J* = 8 Hz, Ar*H*, 4H), 7.90-7.92 (d, *J* = 8 Hz, Ar*H*, 2H),8.31-8.33 (d, *J* = 8 Hz, Ar*H*, 1H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 168.31, 163.46, 150.84, 140.68, 134.69, 131.61, 128.60, 127.87, 118.65, 102.55, 84.42, 78.77, 68.29, 62.81, 61.39, 31.74, 30.85, 30.05, 29.48, 29.36, 28.95, 25.30, 22.54, 19.57, 14.38. High Resolution MS calcd for $C_{33}H_{49}N_4O_8P$, 660.3288; found 661.3390 (M + H^+); 683.3217 (M + Na^+). Yield 78%.

Compound 4

^{1}H NMR ($CDCl_3$, 400 MHz): δ = 0.88 (t, C*H*$_3$, 3H), 1.70 (m, C*H*$_2$, 28H), 2.50-2.52 (m, 2-*H*, 1H),2.77-2.80 (m, C*H*$_2$CN+2-H, 3H),3.95-3.96 (m, 5-*H*, 2H), 4.08-4.11 (m, OC*H*$_2$CH$_2$CN, 2H), 4.34-4.3 5(m, 3-*H*+C*H*$_2$O, 4H), 5.20-5.21 (m, 4-*H*, 1H), 6.25 (t, 1-*H*, 1H), 7.50-7.52, 7.60 (d, J = 8 Hz, Ar*H*, 4H), 7.90-7.92 (d, *J* = 8 Hz, Ar*H*, 2H), 8.31-8.33 (d, *J* = 8 Hz, Ar*H*, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): 162.65, 155.19, 145.34, 133.14, 128.88, 127.77, 125.48, 116.52, 98.39, 97.07, 87.85, 86.39, 69.10, 67.35, 62.14, 61.50, 39.81, 33.24, 31.90, 30.18, 29.57, 29.11, 25.35, 23.42, 19.73, 14.10. High Resolution MS calcd for $C_{35}H_{53}N_4O_8P$, 688.3601; found 689.3650 (M + H^+); 711.3454 (M + Na^+ - H^+).Yield 80%.

Compound 5

^{1}H-NMR ($CDCl_3$, 400 MHz): δ = 0.87 (s, C*H*$_3$, 3H), 1.26-1.34 (m, C*H*$_2$, 14H), 1.70 (m, *CH*$_2$, 2H), 2.82 (m, *CH*$_2$CN, 4H), 3.70-3.73 (m, O*CH*$_2$, 2H), 3.90-3.93 (m, O*CH*$_2$, 2H), 4.10-4.44 (m, O*CH*$_2$+O*CH*, 8H), 5.21-5.28 (m, ArC*H*$_2$O, 2H), 6.22-6.23 (m, Ar*H*, 2H), 7.47-7.58 (m, Ar*H*, 8H), 7.88-7.90 (m, Ar*H*, 4H), 8.04-8.06 (m, Ar*H*, 1H), 8.39-8.40 (m, Ar*H*, 1H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 168.30, 163.46, 150.84, 140.69, 134.69, 131.63, 128.62, 127.87, 125.34, 118.68, 102.55, 85.81, 84.38, 78.75, 68.29, 62.85, 61.39, 45.95, 31.72, 30.00, 29.36, 29.12, 28.93, 25.30, 22.66, 22.59, 19.65, 14.40. High Resolution MS calcd for $C_{48}H_{60}N_8O_{15}P_2$, 1050.3653; found 526.2936 (M+2*e*), 1051.3707 (M+H^+).Yield 67%.

Compound 6

^{1}H-NMR ($CDCl_3$, 400 MHz): δ = 0.86-0.89 (t, C*H*$_3$, 3H), 1.21-1.30 (m, C*H*$_2$, 18H), 1.71 (m, *CH*$_2$,

2H), 2.82-2.83 (m, CH_2CN, 4H), 3.89-3.90 (m, OCH_2, 2H), 4.34-4.44 (m, OCH_2+OCH, 8H), 5.28-5.29 (m, ArCH_2O, 2H), 6.22-6.23 (m, ArH, 2H), 7.47-7.54 (m, ArH, 8H), 7.88-7.91 (m, ArH, 4H), 8.04-8.06 (m, ArH, 1H), 8.39-8.40 (m, ArH, 1H). ^{13}C NMR (d^6-DMSO, 100 MHz): 169.54, 163.93, 163.62, 150.61, 150.37, 141.01, 140.77, 133.28, 132.97, 131.98, 128.83, 128.25, 127.91, 116.91, 102.88, 97.27, 86.47, 86.08, 82.97, 69.26, 66.76, 62.83, 62.43, 61.53, 31.88, 30.18, 29.49, 25.34, 22.66, 19.72, 14.11. High Resolution MS calcd for $C_{50}H_{64}N_8O_{15}P_2$, 1078.3966; found 1079.3976 (M + H^+), 1098.8718 (M + Na^+).Yield 58%.

Compound 7

^{1}H-NMR ($CDCl_3$, 400 MHz): δ = 0.86-0.89 (t, CH_3, 3H), 1.21-1.30 (m, CH_2, 22H), 1.70 (m, CH_2, 2H), 2.82-2.83 (m, CH_2CN, 4H), 3.92-3.93 (m, OCH_2, 2H), 4.09-4.11 (m, OCH_2, 2H), 4.30-4.44 (m, OCH_2+OCH, 8H), 5.21-5.29 (m, ArCH_2O, 2H), 6.19-6.23 (m, ArH, 2H), 7.47-7.58 (m, ArH, 8H), 7.87-7.90 (m, ArH, 4H), 8.02-8.04 (m, ArH, 1H), 8.33-8.36 (m, ArH, 1H). ^{13}C NMR (d^6-DMSO, 100 MHz): 168.29, 163.45, 150.83, 140.69, 139.62, 134.70, 131.63, 128.62, 127.87, 125.34, 118.67, 102.55, 85.81, 84.33, 68.29, 62.80, 61.39, 31.74, 30.05, 29.45, 29.15, 28.93, 25.30, 22.54, 19.57, 14.40. High Resolution MS calcd for $C_{52}H_{68}N_8O_{15}P_2$, 1106.4279; found 554.2199 (M+ H^++2e), 1107.4282(M + H^+).Yield 61%.

Compound 8

^{1}H-NMR ($CDCl_3$, 400 MHz): δ = 0.86-0.89 (t, CH_3, 3H), 1.21-1.30 (m, CH_2, 26H), 1.71 (m, CH_2, 2H), 2.84-2.86 (m, CH_2CN, 4H), 3.91-3.95 (m, OCH_2, 2H), 4.10-4.12 (m, OCH_2, 2H), 4.30-4.43 (m, OCH_2+OCH, 8H), 5.24-5.30 (m, ArCH_2O, 2H), 6.16-6.23 (m, ArH, 2H), 7.49-7.60 (m, ArH, 8H), 7.92-7.94 (m, ArH, 4H), 8.02-8.04 (m, ArH, 1H), 8.42-8.44 (m, ArH, 1H). ^{13}C NMR (d^6-DMSO, 100 MHz): 168.31, 163.46, 150.84, 140.68, 134.69, 131.61, 128.60, 127.87, 118.65, 85.83, 84.42, 78.77, 68.34, 62.85, 61.39, 31.74, 30.85, 30.01, 29.50, 29.36, 29.16, 28.94, 25.30, 22.54, 19.57, 14.38. High Resolution MS calcd for $C_{54}H_{72}N_8O_{15}P_2$, 1134.4592; found 1135.4586 (M+H^+).Yield 52%.

Compound 9

^{1}H-NMR ($CDCl_3$, 400 MHz): δ = 0.87 (t, CH_3, 3H), 1.19-1.35 (m, CH_2, 14H), 1.41-1.45 (m, CH_2, 2H), 1.69-1.70 (m, CH_2, 2H), 2.82-2.83 (m, CH_2CN, 6H), 3.88-3.92 (m, OCH_2, 2H), 4.10-4.34 (m, OCH_2, 2H), 4.20-4.47 (m, CH, 10H), 5.28 (m, ArCH_2O, 2H), 6.18-6.22 (m, ArH, 3H), 7.44-7.48 (m, ArH, 10H), 7.55-7.57 (m, ArH, 5H), 8.19-8.20 (m, ArH, 2H), 8.32-8.34 (m, ArH, 1H). ^{13}C

NMR ($CDCl_3$, 100 MHz): 169.54, 163.93, 163.62, 150.61, 150.37, 140.77, 140.62, 133.28, 133.09, 132.97, 131.98, 128.83, 128.59, 127.91, 127.36, 69.26, 66.99, 66.71, 62.83, 62.43, 38.04, 31.88, 30.18, 29.61, 29.49, 29.32, 29.12, 25.34, 22.66, 19.72, 14.11. High Resolution MS calcd for $C_{67}H_{79}N_{12}O_{22}P_3$, 1496.4645; found 749.2460 (M+2*e*+H^+).Yield 62%.

Compound 10

^{1}H-NMR ($CDCl_3$, 400 MHz): δ = 0.87 (t, C*H*$_3$, 3H), 1.19-1.35 (m, C*H*$_2$, 18H), 1.41-1.45 (m, C*H*$_2$, 2H), 1.69-1.70 (m, *CH*$_2$, 2H), 2.81-2.87 (m, *CH*$_2$CN, 6H), 3.88-3.92 (m, O*CH*$_2$, 2H), 4.10-4.34 (m, O*CH*$_2$, 2H), 4.31-4.47 (m, *CH*, 10H), 5.26-5.30 (m, ArC*H*$_2$O, 2H), 6.19-6.23 (m, Ar*H*, 3H), 7.44-7.48 (m, Ar*H*, 10H), 7.45-7.57 (m, Ar*H*, 5H), 8.01-8.03 (m, Ar*H*, 2H), 8.36-8.39 (m, Ar*H*, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): 169.57, 167.48, 167.31, 163.01, 162.76, 155.10, 154.82, 150.44, 150.33, 145.33, 145.21, 145.04, 140.80, 140.54, 133.30, 133.08, 132.86, 131.96, 128.72, 128.57, 128.03, 127.37, 125.49, 117.18, 116.95, 102.78, 97.20, 69.18, 67.06, 66.91, 63.01, 62.44, 31.88, 30.30, 30.18, 29.61, 29.49, 29.32, 25.34, 22.66, 19.71, 14.11. High Resolution MS calcd for $C_{69}H_{83}N_{12}O_{22}P_3$, 1524.4958; found 1525.5089 (M+H^+).Yield 57%.

Compound 11

^{1}H-NMR ($CDCl_3$, 400 MHz): δ = 0.87 (t, C*H*$_3$, 3H), 1.19-1.35 (m, C*H*$_2$, 22H), 1.41-1.45 (m, C*H*$_2$, 2H), 1.69-1.70 (m, *CH*$_2$, 2H), 2.81-2.87 (m, *CH*$_2$CN, 6H), 3.88-3.92 (m, O*CH*$_2$, 2H), 4.10-4.34 (m, O*CH*$_2$, 2H), 4.34-4.47 (m, *CH*, 10H), 5.26-5.30 (m, ArC*H*$_2$O, 2H), 6.18-6.22 (m, Ar*H*, 3H), 7.44-7.48 (m, Ar*H*, 10H), 7.46-7.58 (m, Ar*H*, 5H), 8.01-8.03 (m, Ar*H*, 2H), 8.36-8.39 (m, Ar*H*, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): 166.92, 166.78, 162.86, 162.59, 155.03, 154.84, 145.39, 144.94, 133.09, 132.80, 128.78, 128.54, 128.27, 127.91, 127.38, 116.99, 97.15, 96.93, 88.24, 88.09, 87.98, 97.55, 87.46, 86.45, 83.79, 83.62, 78.78, 69.19, 67.10, 66.92, 66.65, 62.97, 62.81, 62.42, 61.32, 39.75, 39.17, 31.89, 30.18, 29.64, 29.50, 39.33, 29.14, 25.34, 22.66, 19.72, 14.10. High Resolution MS calcd for $C_{71}H_{87}N_{12}O_{22}P_3$, 1552.5271; found 777.2736(M +2*e* + H^+).Yield 62%.

Compound 12

^{1}H-NMR ($CDCl_3$, 400 MHz): 0.87 (t, C*H*$_3$, 3H), 1.19-1.35 (m, C*H*$_2$, 26H), 1.41-1.45 (m, C*H*$_2$, 2H), 1.69-1.70 (m, *CH*$_2$, 2H), 2.81-2.87 (m, *CH*$_2$CN, 6H), 3.88-3.92 (m, O*CH*$_2$, 2H), 4.10-4.34 (m, O*CH*$_2$, 2H), 4.34-4.47 (m, *CH*, 10H), 5.26-5.30 (m, ArC*H*$_2$O, 2H), 6.18-6.22 (m, Ar*H*, 3H), 7.44-7.48 (m, Ar*H*, 10H), 7.46-7.58 (m, Ar*H*, 5H), 8.01-8.03 (m, Ar*H*, 2H), 8.36-8.39 (m, Ar*H*, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): 169.50, 167.26, 167.08, 166.74, 164.10, 163.97, 162.86, 155.16,

154.97, 150.48, 150.30, 145.25, 144.74, 140.92, 140.74, 133.29, 133.01, 132.85, 131.98, 128.58, 128.32, 128.00, 127.36, 117.21, 117.06, 102.76, 102.68, 97.35, 97.16, 86.25, 86.07, 83.70, 82.90, 69.19, 67.04, 66.86, 62.99, 62.44, 31.90, 30.30, 30.20, 29.69, 29.34, 29.16, 25.35, 22.67, 19.71, 14.11. High Resolution MS calcd for $C_{73}H_{91}N_{12}O_{22}P_3$, 1580.5584; found 791.2968 (M + 2*e* + H^+), 1601.5576 (M + Na).Yield 46%.

Compound 13

^{1}H NMR (MeOD, 400 MHz): δ = 0.91 (t, C*H*$_3$, 3H), 1.26 (m, C*H*$_2$, 16H), 2.57-2.59 (m, 2-*H*, 2H), 3.83 (m, 5-*H*, 2H), 3.88-3.89 (m, C*H*$_2$O, 2H), 4.11-4.13 (m, 3-*H*, 1H), 4.89 (m, 4-*H*, 1H), 5.98-6.00 (t, 1-*H*, 1H), 6.28 (d, Ar*H*, 1H), 8.21-8.23 (d, *J* = 8 Hz, Ar*H*, 1H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 164.77, 155.10, 141.98, 116.41, 94.76, 86.72, 86.16, 75.02, 65.40, 61.30, 39.59, 35.39, 31.68, 30.51, 29.42, 29.38, 29.09, 25.56, 22.35, 15.37, 13.11. High Resolution MS calcd for $C_{19}H_{37}N_4O_7P$, 464.2400; found 446.2050 (M - NH_4^+).Yield 88%.

Compound 14

^{1}H NMR (MeOD, 400 MHz): δ = 0.90-0.93 (t, C*H*$_3$, 3H), 1.63-1.65 (m, C*H*$_2$, 20H),2.23, 2.57-2.59 (m, 2-*H*, 2H),3.90-3.91 (m, C*H*$_2$O + 5-*H*, 4H), 4.18-4.19 (m, 3-*H*, 1H), 4.89 (m, 4-*H*, 1H), 5.96-5.98 (t, 1-*H*, 1H), 6.29 (d, Ar*H*, 1H),8.19-8.20 (d, *J* = 4 Hz, Ar*H*, 1H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 162.94, 152.49, 142.97, 94.44, 86.93, 86.24, 74.93, 65.42, 61.16, 39.61, 31.68, 30.50, 29.42, 29.38, 29.36, 29.09, 25.56, 22.34, 13.09. High Resolution MS calcd for $C_{21}H_{41}N_4O_7P$, 492.2713; found 474.2369 (M - NH_4^+).Yield 84%.

Compound 15

^{1}H NMR (MeOD, 400 MHz): δ = 0.90-0.94 (t, C*H*$_3$, 3H), 1.65 (m, C*H*$_2$, 16H),2.28-2.29 (m, 2-*H*, 2H),3.82-3.83 (m, 5-*H*, 2H), 3.88-3.89 (m, C*H*$_2$O, 2H), 4.19-4.20 (m, 3-*H*, 1H), 4.80 (m, 4-*H*, 1H), 5.99-6.01 (t, 1-*H*, 1H),6.28 (d, Ar*H*, 1H), 8.22-8.24 (d, *J* = 8 Hz, Ar*H*, 1H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 164.79, 155.11, 141.98, 94.72, 86.72, 86.16, 74.99, 65.39, 61.29, 39.59, 35.38, 31.67, 30.51, 29.41, 29.37, 29.08, 25.56, 22.34, 13.10. High Resolution MS calcd for $C_{23}H_{45}N_4O_7P$, 520.3026; found 502.2682 (M - NH_4^+).Yield 82%.

Compound 16

^{1}H NMR (MeOD, 400 MHz): δ = 0.92 (t, C*H*$_3$, 3H), 1.63-1.65 (m, C*H*$_2$, 16H), 2.28-2.29 (m, 2-*H*, 2H), 3.83 (m, 5-*H*, 2H), 3.88-3.89 (m, C*H*$_2$O, 2H), 4.19-4.20 (m, 3-*H*, 1H), 4.89 (m, 4-*H*, 1H),5.98-6.00 (t, 1-*H*, 1H),6.28 (d, Ar*H*, 1H), 8.20-8.22 (d, *J* = 8 Hz, Ar*H*, 1H). ^{13}C NMR (*d*^{6}-

DMSO, 100 MHz): 164.79, 155.11, 141.98, 94.74, 86.72, 86.16, 75.00, 65.40, 61.29, 39.56, 35.38, 31.67, 30.51, 29.41, 29.36, 29.08, 25.56, 22.34, 15.37, 13.10. High Resolution MS calcd for $C_{25}H_{49}N_4O_7P$, 548.3339; found 530.2996 (M - NH_4^+).Yield 88%.

Compound 17

^{1}H-NMR (*d*^{6}-DMSO, 400 MHz): δ = 0.83-0.87 (t, C*H*$_3$, 3H), 1.15-1.23 (m, C*H*$_2$, 14H), 1.48-1.52 (m, C*H*$_2$, 2H), 1.95-2.13 (m, C*H*, 2H), 2.22-2.31 (m, C*H*, 2H), 2.87-2.88 (m, *CH*, 2H), 3.07-3.09 (m, *CH*$_2$, 4H), 3.58-3.69 (m, O*CH*$_2$, 2H), 3.87-4.11 (m, O*CH*$_2$, 2H), 4.70 (m, *CH*, 1H), 5.75-5.77 (m, ArC*H*$_2$O, 2H), 6.14 (m, Ar*H*, 2H), 7.13-7.25 (m, Ar*H*, 4H), 7.72 - 7.84 (m, Ar*H*, 2H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 165.72, 155.28, 141.39, 94.84, 94.66, 86.66, 85.25, 85.16, 84.89, 75.2, 74.52, 64.68, 61.86, 31.73, 30.85, 29.51, 29.15, 26.86, 25.86, 22.53, 15.82, 15.76, 14.40. High Resolution MS calcd for $C_{28}H_{48}N_8O_{13}P_2$, 770.3129; found 737.2650 (M-2NH_3+H^+).Yield 75%.

Compound 18

^{1}H-NMR (*d*^{6}-DMSO, 400 MHz): δ = 0.84-0.85 (t, C*H*$_3$, 3H), 1.15-1.23 (m, C*H*$_2$, 18H), 1.48-1.50(m, C*H*$_2$, 2H), 1.95-2.13 (m, C*H*, 2H), 2.26-2.34 (m, C*H*, 2H), 2.89-2.91 (m, *CH*, 2H), 3.09-3.11 (m, *CH*$_2$, 4H), 3.58-3.69 (m, O*CH*$_2$, 2H), 3.87-4.11 (m, O*CH*$_2$, 2H), 4.70 (m, *CH*, 1H), 5.82-5.84 (m, ArC*H*$_2$O, 2H), 6.15-6.17 (m, Ar*H*, 2H), 7.45-7.47 (m, Ar*H*, 4H), 7.87-7.89 (m, Ar*H*, 2H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 162.94, 152.49, 142.97, 94.44, 86.93, 86.24, 74.93, 65.42, 61.16, 39.61, 31.68, 30.50, 29.42, 29.38, 29.36, 29.06, 25.56, 22.34, 13.09. High Resolution MS calcd for $C_{30}H_{56}N_8O_{13}P_2$, 798.3442; found 765.3068 (M-2NH_3+H^+).Yield 70%.

Compound 19

^{1}H-NMR (*d*^{6}-DMSO, 400 MHz): δ = 0.83-0.87 (t, C*H*$_3$, 3H), 1.15-1.23 (m, C*H*$_2$, 22H), 1.47-1.49 (m, C*H*$_2$, 2H), 1.97-2.14 (m, C*H*, 2H), 2.26-2.34 (m, C*H*, 2H), 2.86-2.90 (m, *CH*, 2H), 3.06-3.10 (m, *CH*$_2$, 4H), 3.58-3.69 (m, O*CH*$_2$, 2H), 3.87-4.11 (m, O*CH*$_2$, 2H), 4.68 (m, *CH*, 1H), 5.75-5.76 (m, ArC*H*$_2$O, 2H), 6.15-6.17 (m, Ar*H*, 2H), 7.45-7.47 (m, Ar*H*, 4H), 7.72-7.83 (m, Ar*H*, 2H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 164.79, 155.11, 141.98, 94.72, 86.72, 86.16, 74.99, 65.39, 61.29, 39.56, 35.38, 31.67, 30.45, 29.37, 29.36, 25.56, 22.34, 15.36, 13.10. High Resolution MS calcd for $C_{32}H_{60}N_8O_{13}P_2$, 826.3755; found 793.2963(M-2NH_3+H^+).Yield 66%.

Compound 20

^{1}H-NMR (*d*^{6}-DMSO, 400 MHz): δ = 0.83-0.87 (t, C*H*$_3$, 3H), 1.15-1.23 (m, C*H*$_2$, 26H), 1.47-1.49

(m, CH_2, 2H), 1.97-2.14 (m, C*H*, 2H), 2.24-2.31 (m, C*H*, 2H), 2.84-2.88 (m, *CH*, 2H), 3.06-3.10 (m, *CH*$_2$, 4H), 3.58-3.69 (m, O*CH*$_2$, 2H), 3.87-4.11 (m, O*CH*$_2$, 2H), 4.68-4.72 (m, *CH*, 1H), 5.74-5.76 (m, ArCH_2O, 2H), 6.14-6.17 (m, Ar*H*, 2H), 7.45-7.47 (m, Ar*H*, 4H), 7.72-7.83 (m, Ar*H*, 2H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 165.72, 155.28, 141.39, 141.28, 94.84, 94.66, 86.66, 86.25, 75.20, 74.52, 64.68, 61.86, 31.37, 30.85, 30.80, 29.51, 29.43, 29.27, 29.15, 25.86, 22.53, 14.40. High Resolution MS calcd for $C_{34}H_{64}N_8O_{13}P_2$, 854.4068; found 819.3381 (M-2NH_4^+).Yield 62%.

Compound 21

^{1}H-NMR (*d*^{6}-DMSO, 400 MHz): δ = 0.83-0.87 (t, CH_3, 3H), 1.15-1.23 (m, CH_2, 14H), 1.48-1.52 (m, CH_2, 2H), 1.95-2.13 (m, C*H*, 3H), 2.22-2.31 (m, C*H*, 3H), 2.84-2.88 (m, *CH*$_2$, 4H), 3.09-3.13 (m, *CH*$_2$, 4H), 3.66-3.70 (m, O*CH*$_2$, 6H), 3.87-3.99 (m, O*CH*$_2$, 6H), 4.06-4.10 (m, *CH*$_2$, 2H), 4.70 (m, *CH*, 3H), 5.79-5.80 (m, ArCH_2O, 2H), 5.96-6.18 (m, *CH*$_2$, 4H), 6.14 (m, Ar*H*, 2H), 7.34-7.53 (m, Ar*H*, 6H), 7.86-7.96 (m, Ar*H*, 3H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 165.72, 155.28, 141.39, 94.84, 94.66, 86.66, 86.25, 85.16, 84.89, 75.20, 75.16, 74.52, 64.68, 64.64, 61.86, 31.73, 30.85, 29.55, 29.43, 29.27, 26.86, 25.86, 22.53, 15.82, 15.76, 14.40. High Resolution MS calcd for $C_{37}H_{67}N_{12}O_{19}P_3$, 1076.3858; found 1026.3140 (M-3NH_3+H^+).Yield 86%.

Compound 22

^{1}H-NMR (*d*^{6}-DMSO, 400 MHz): δ = 0.84-0.86 (t, CH_3, 3H), 1.15-1.23 (m, CH_2, 18H), 1.49-1.51 (m, CH_2, 2H), 1.95-2.13 (m, C*H*, 3H), 2.22-2.31 (m, C*H*, 3H), 2.84-2.88 (m, *CH*$_2$, 4H), 3.09-3.13 (m, *CH*$_2$, 4H), 3.66-3.70 (m, O*CH*$_2$, 6H), 3.87-3.99 (m, O*CH*$_2$, 6H), 4.06-4.10 (m, *CH*$_2$, 2H), 4.70 (m, *CH*, 3H), 5.79-5.80 (m, ArCH_2O, 2H), 5.96-6.18 (m, *CH*$_2$, 4H), 6.14 (m, Ar*H*, 2H), 7.45-7.51 (m, Ar*H*, 6H), 7.86-7.96 (m, Ar*H*, 3H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 165.74, 155.35, 141.28, 94.84, 94.66, 86.64, 86.25, 85.16, 84.89, 75.20, 74.52, 64.64, 61.86, 31.73, 30.85, 29.51, 29.43, 29.27, 25.86, 22.53, 14.40. High Resolution MS calcd for $C_{39}H_{71}N_{12}O_{19}P_3$, 1104.4171; found 1053.3453 (M-3NH_3+H^+).Yield 89%.

Compound 23

^{1}H-NMR (*d*^{6}-DMSO, 400 MHz): δ =δ = 0.83-0.86 (t, CH_3, 3H), 1.15-1.23 (m, CH_2, 22H), 1.48-1.52 (m, CH_2, 2H), 1.94-2.09 (m, C*H*, 3H), 2.23-2.27 (m, C*H*, 3H), 2.87-2.91 (m, *CH*$_2$, 4H), 3.09-3.13 (m, *CH*$_2$, 4H), 3.59-3.68 (m, O*CH*$_2$, 6H), 3.86-4.09 (m, O*CH*$_2$, 6H), 4.06-4.10 (m, *CH*$_2$, 2H), 4.68-4.72 (m, *CH*, 3H), 5.76-5.80 (m, ArCH_2O, 2H), 5.96-6.19 (m, *CH*$_2$, 4H), 6.14 (m, Ar*H*, 2H), 7.34-7.53 (m, Ar*H*, 6H), 7.86-7.96 (m, Ar*H*, 3H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 165.74,

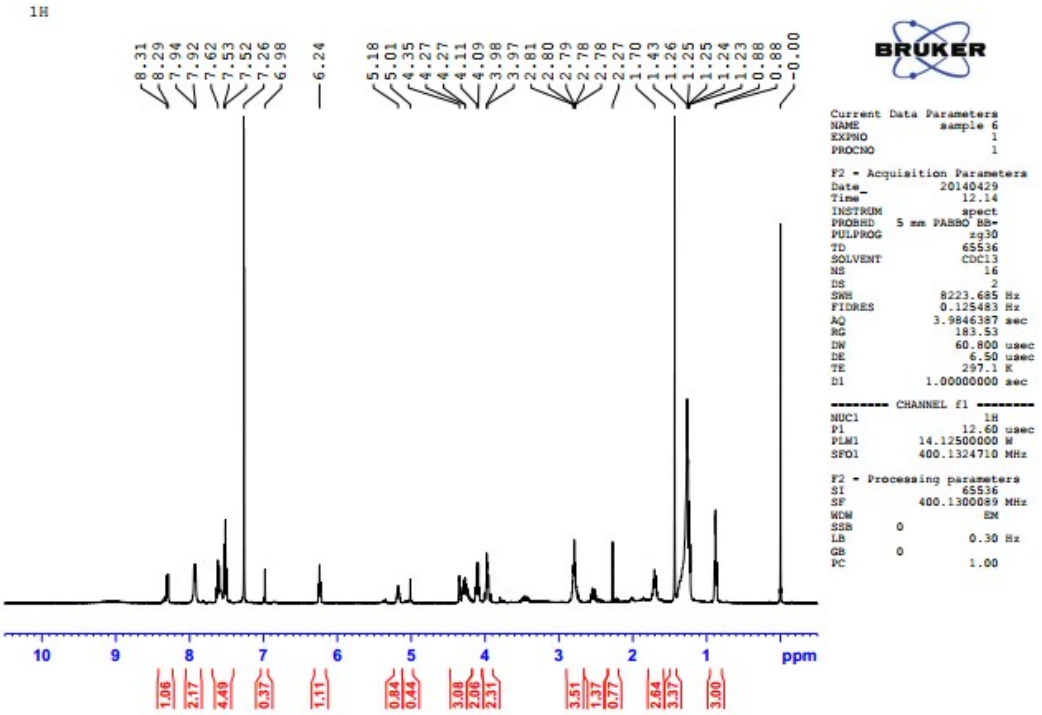
165.72, 155.35, 155.28, 141.39, 141.28, 94.84, 94.66, 86.66, 85.25, 85.16, 84.89, 75.20, 75.16, 74.52, 64.64, 61.86, 31.73, 30.85, 30.80, 29.51, 29.43, 29.27, 26.86, 25.86, 22.53. High Resolution MS calcd for $C_{41}H_{75}N_{12}O_{19}P_3$, 1132.4484; found 1104.3773(M-3NH_3+H^+).Yield 91%.

Compound 24

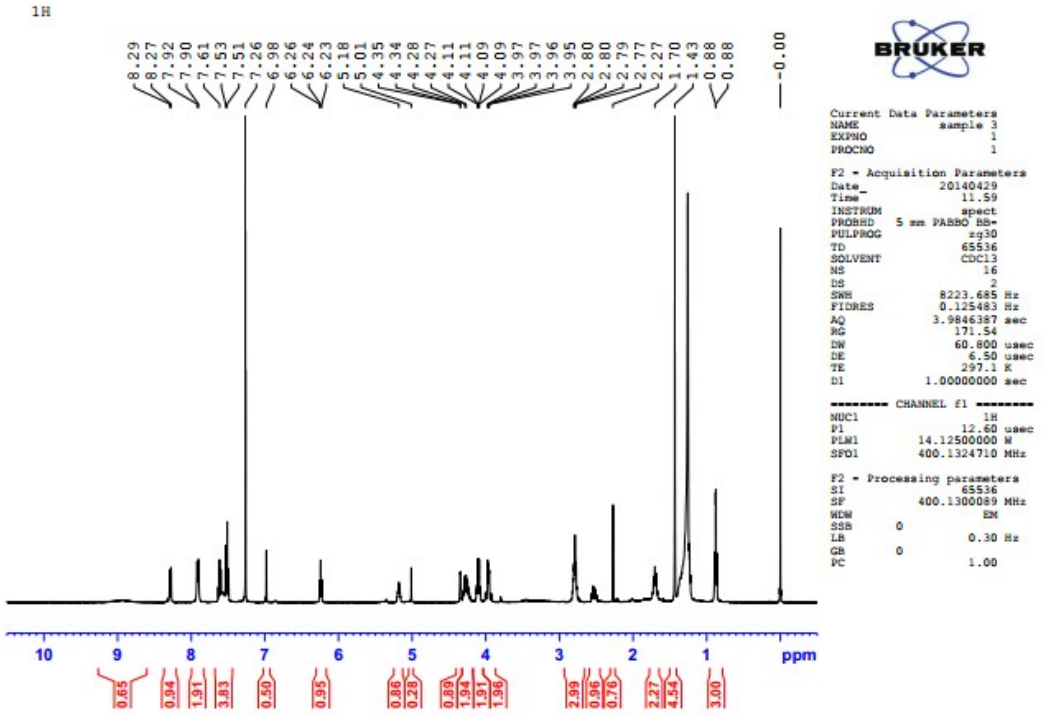
^{1}H-NMR (*d*^{6}-DMSO, 400 MHz): δ = 0.83-0.87 (t, C*H*$_3$, 3H), 1.15-1.23 (m, C*H*$_2$, 26H), 1.48-1.52 (m, C*H*$_2$, 2H), 1.95-2.13 (m, C*H*, 3H), 2.22-2.31 (m, C*H*, 3H), 2.84-2.88 (m, *CH*$_2$, 4H), 3.09-3.13 (m, *CH*$_2$, 4H), 3.66-3.70 (m, O*CH*$_2$, 6H), 3.87-3.99 (m, O*CH*$_2$, 6H), 4.06-4.10 (m, *CH*$_2$, 2H), 4.68-4.71 (m, *CH*, 3H), 5.73-5.75 (m, ArC*H*$_2$O, 2H), 5.96-6.18 (m, *CH*$_2$, 4H), 6.14 (m, Ar*H*, 2H), 7.34-7.53 (m, Ar*H*, 6H), 7.86-7.96 (m, Ar*H*, 3H). ^{13}C NMR (*d*^{6}-DMSO, 100 MHz): 166.08, 155.75, 145.15, 95.33, 95.09, 94.62, 85.18, 79.71, 75.20, 74.97, 74.55, 64.92, 64.62, 64.55, 31.82, 30.98, 29.61, 29.57, 29.44, 29.23, 26.96, 26.03, 22.63, 15.94, 14.50. High Resolution MS calcd for $C_{43}H_{79}N_{12}O_{19}P_3$, 1160.4797; found 1108.7005 (M-NH_4^+-2NH_3), 1111.1070 (M-3NH_3+2H^+).Yield 85%.

S4. ^{1}H-NMR spectrum of compound 1-24

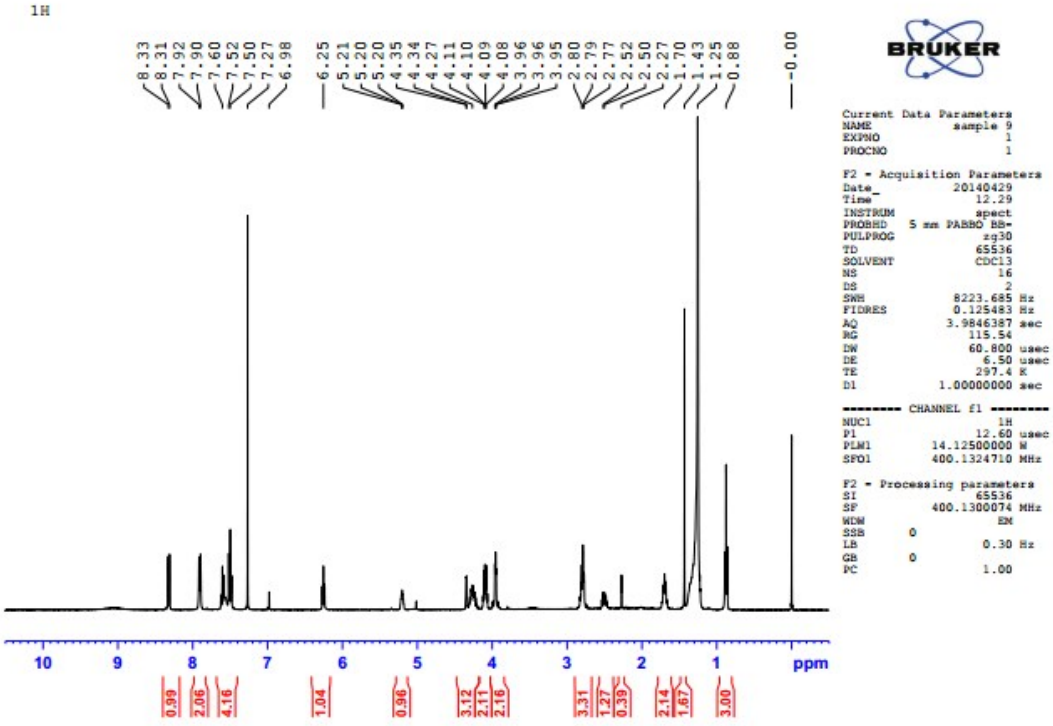
Compound 1



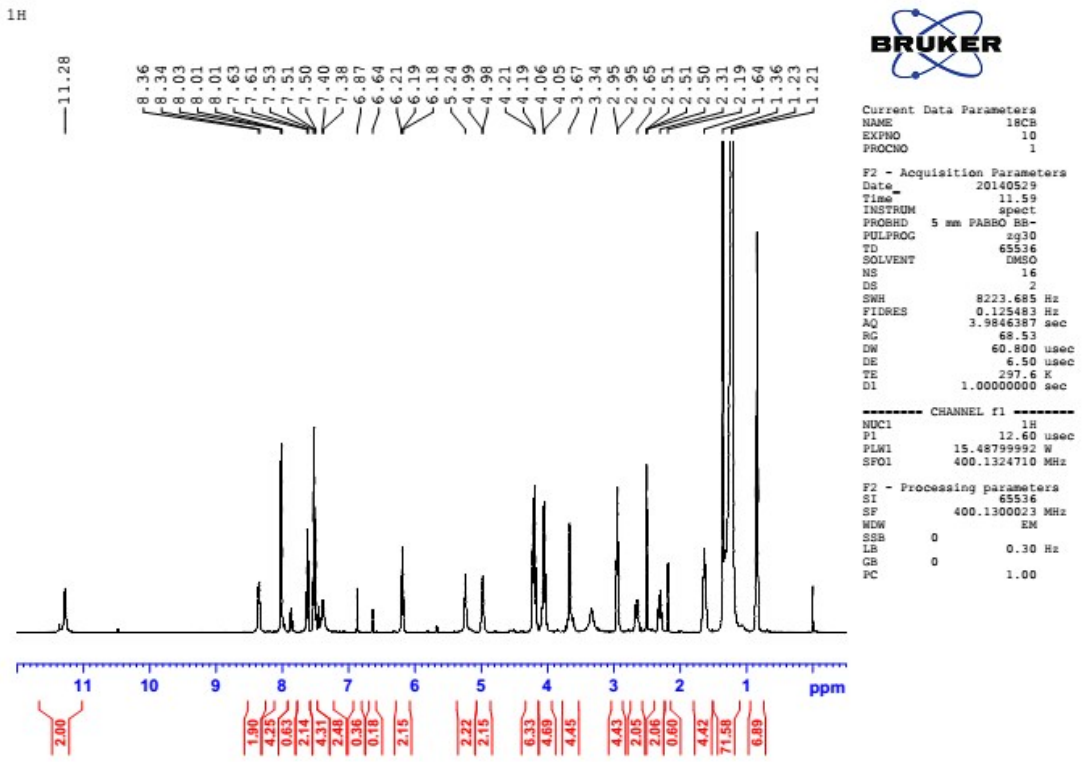
Compound 2



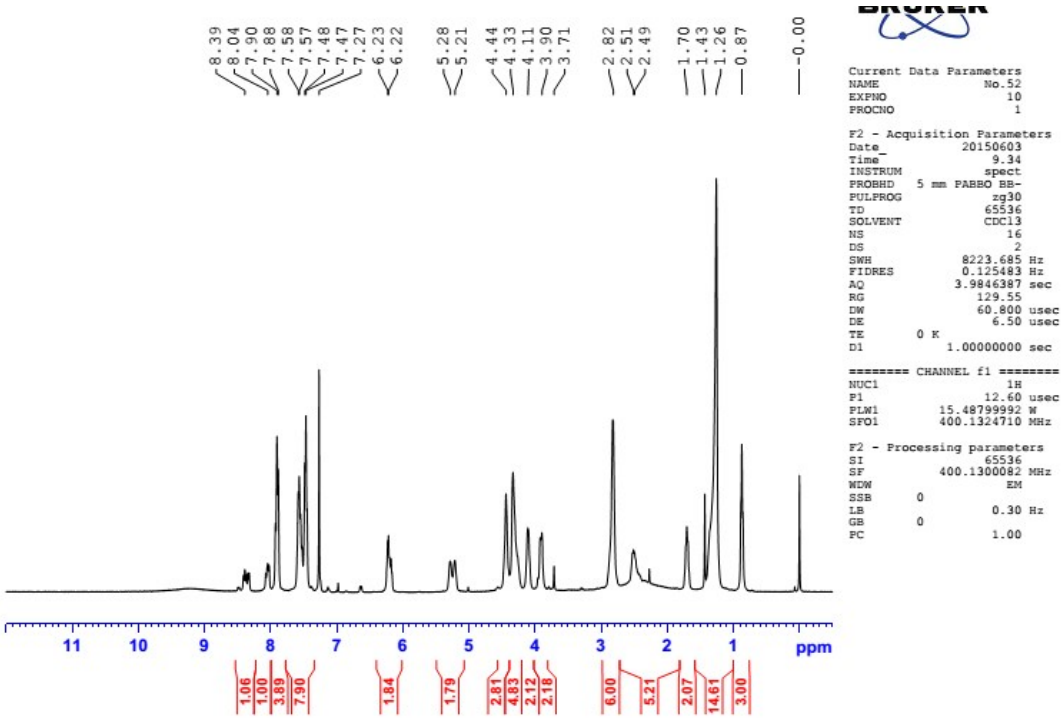
Compound 3



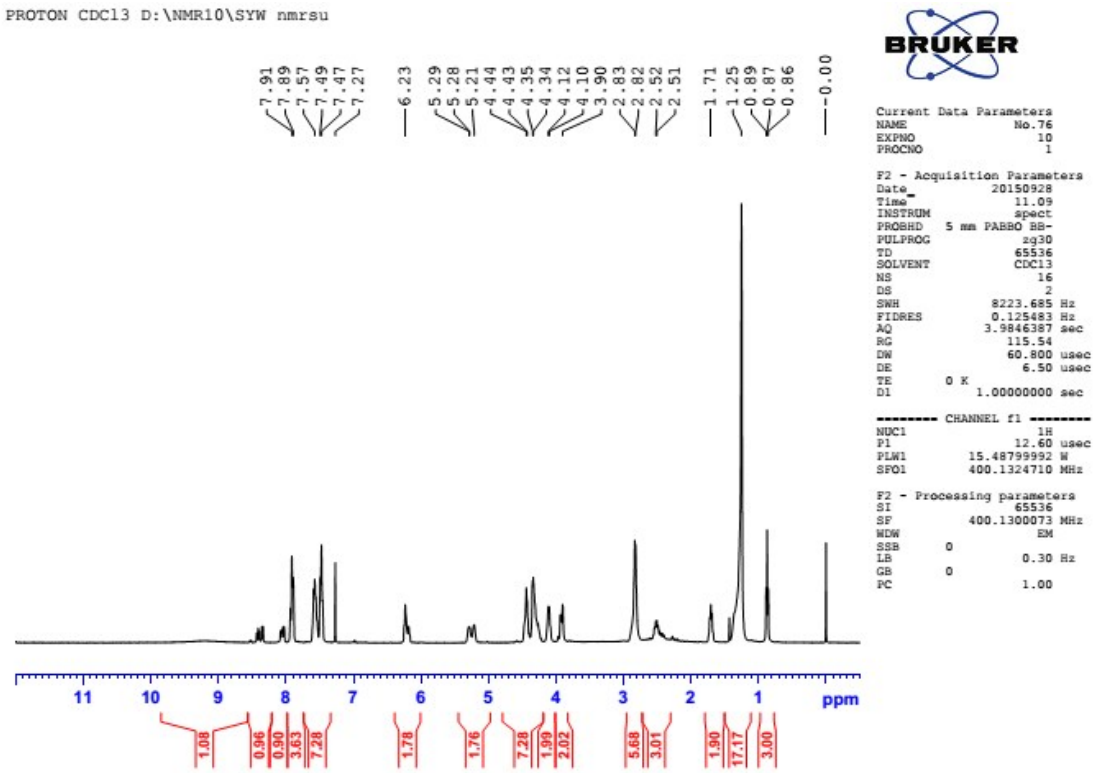
Compound 4



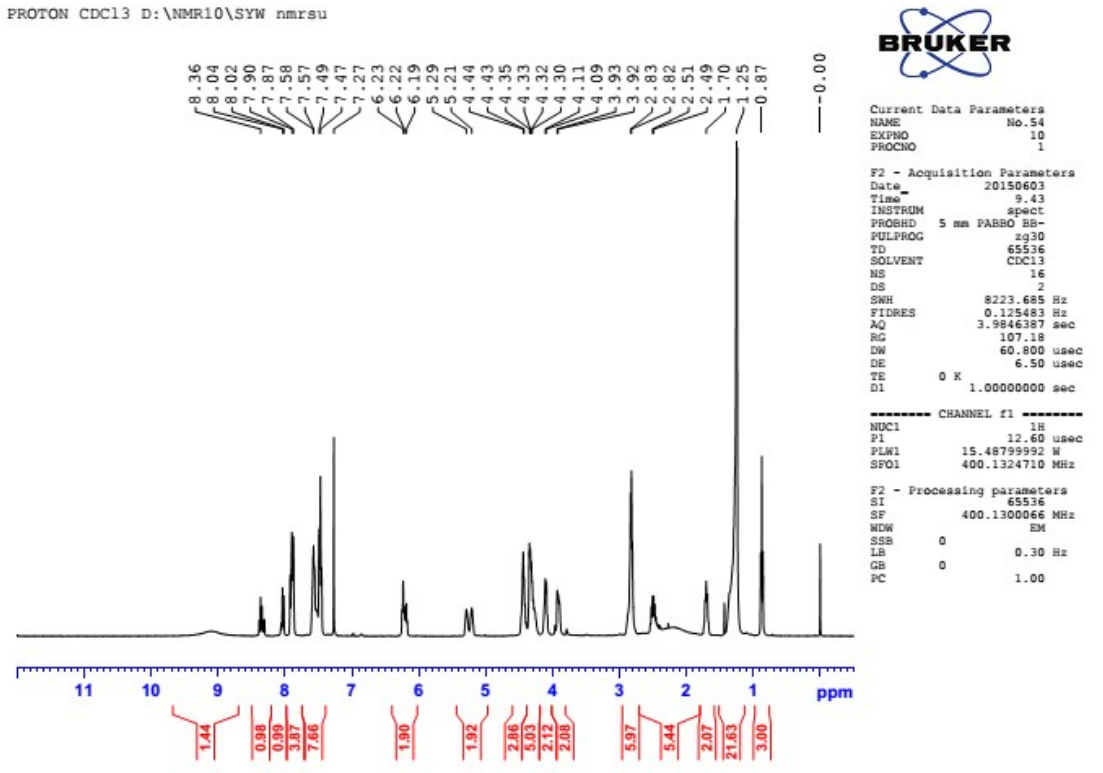
Compound 5



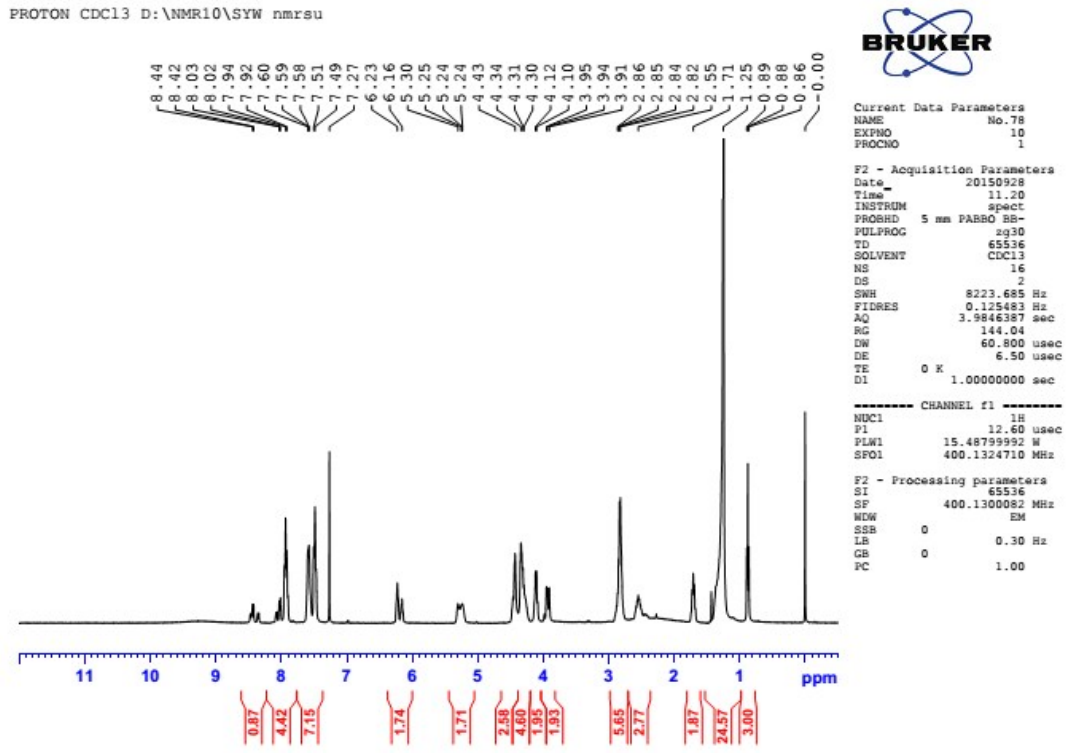
Compound 6



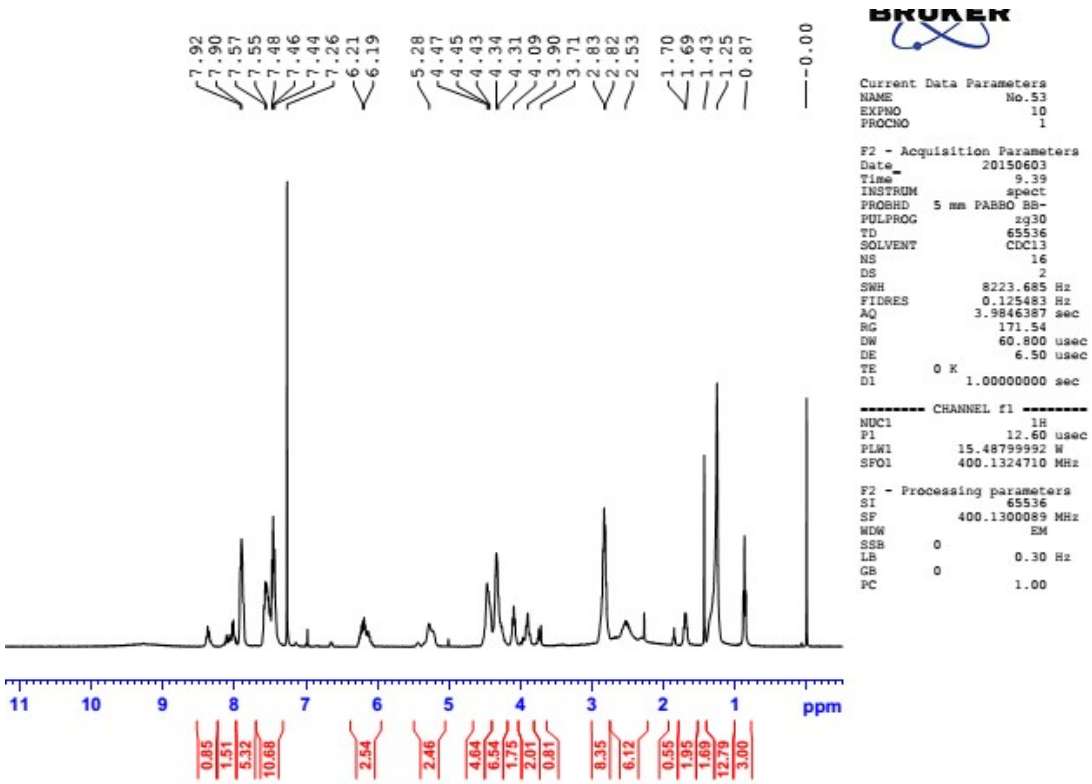
Compound 7



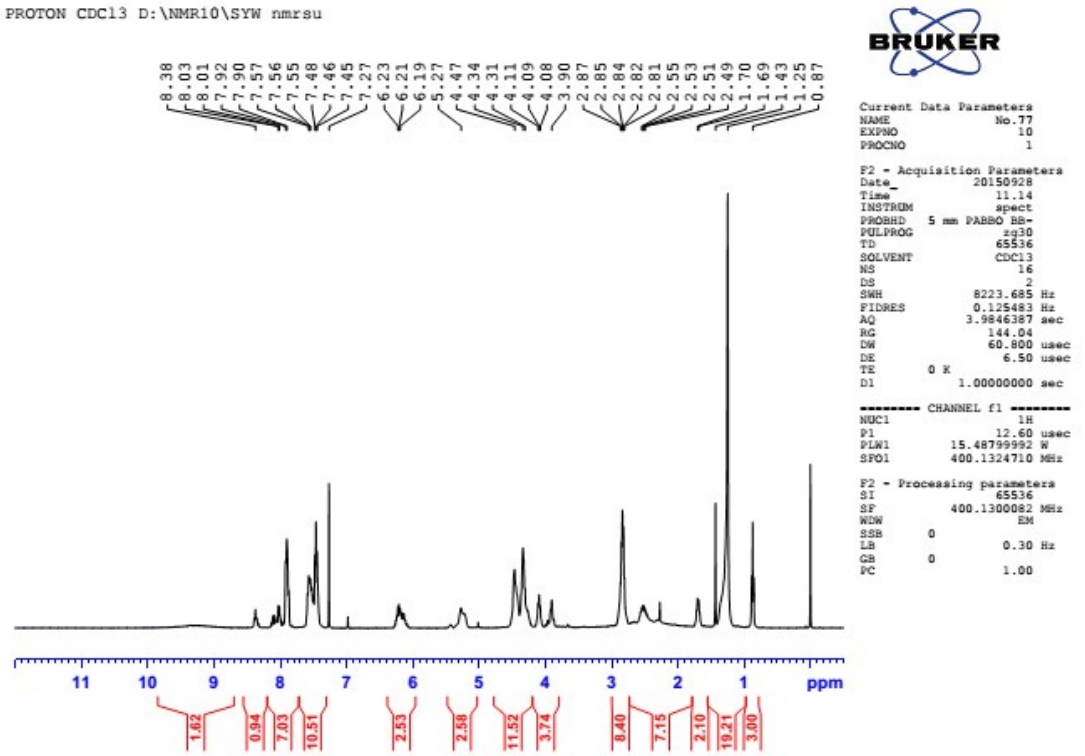
Compound 8



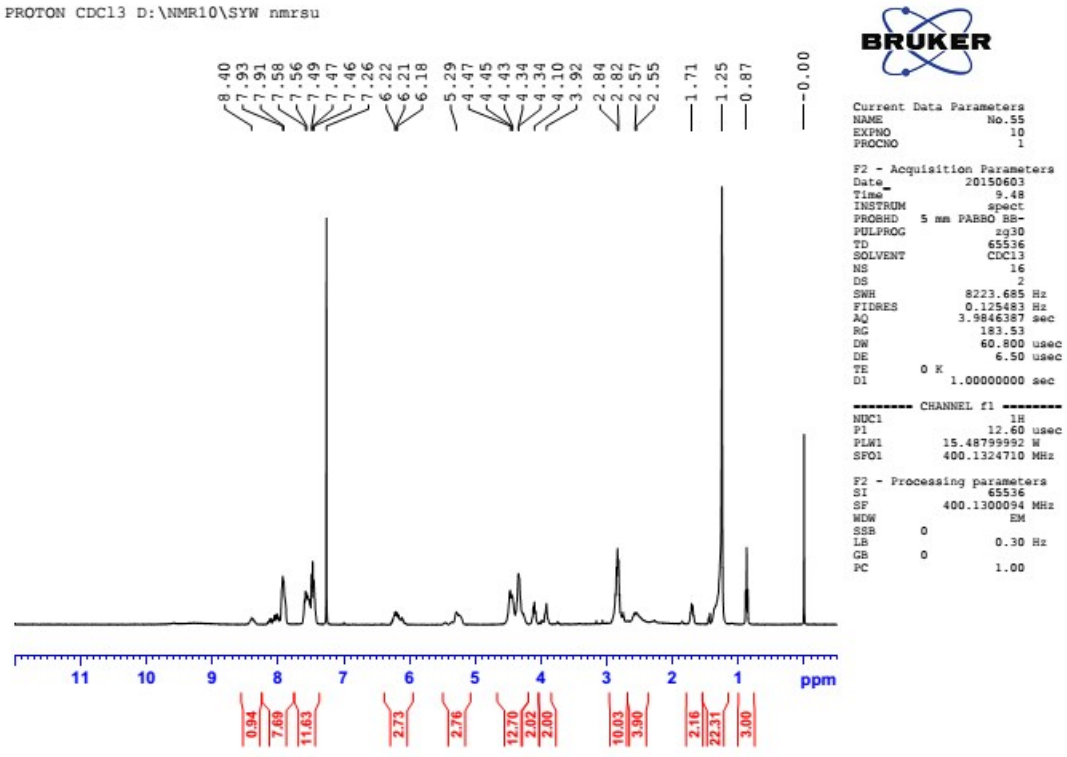
Compound 9



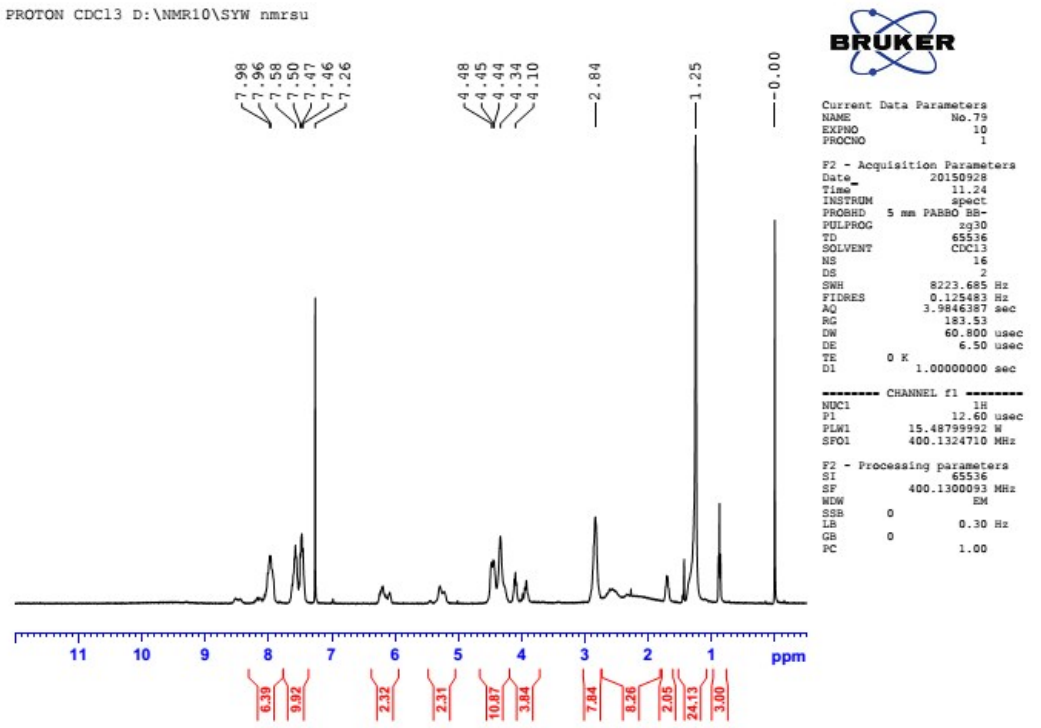
Compound 10



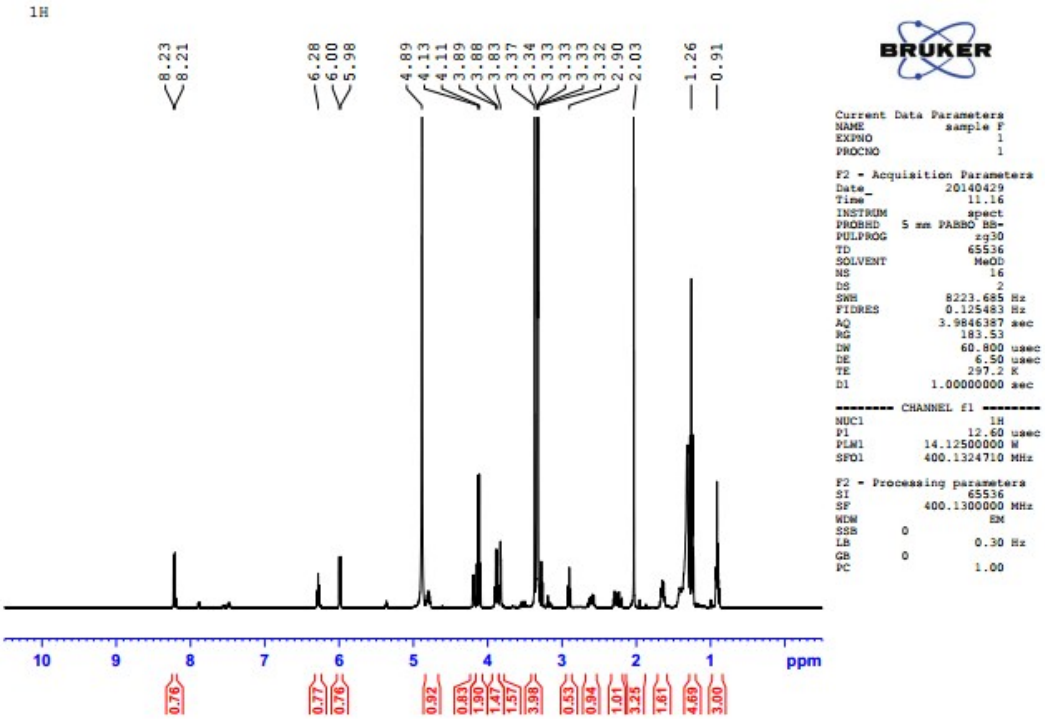
Compound 11



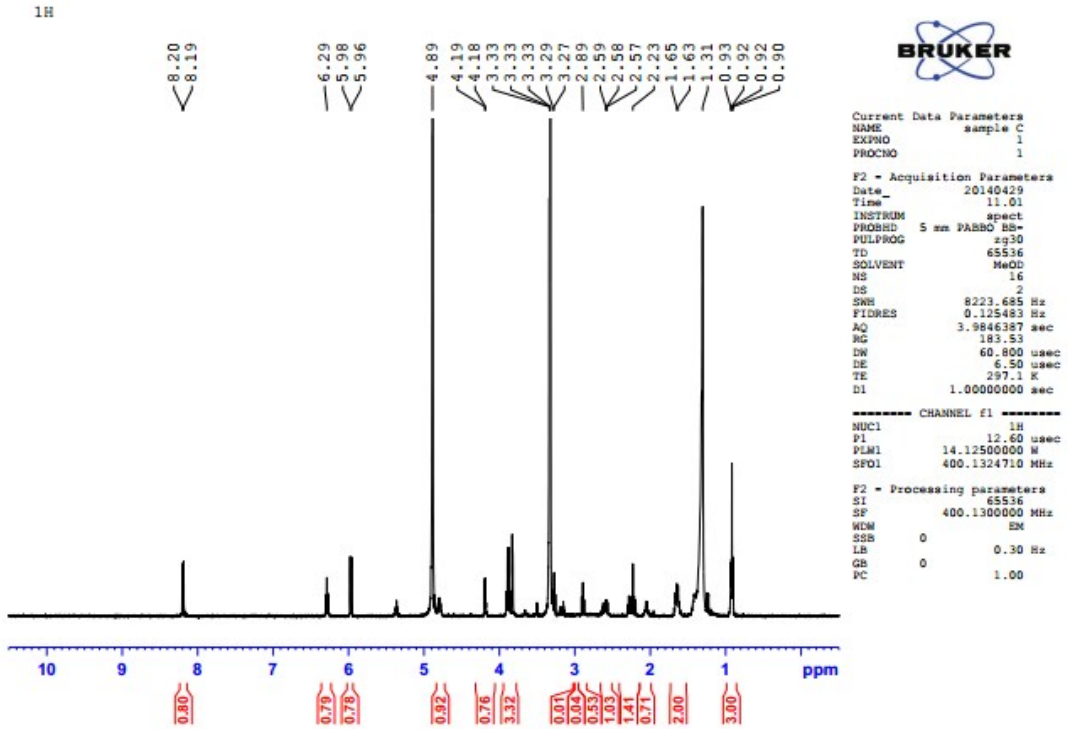
Compound 12



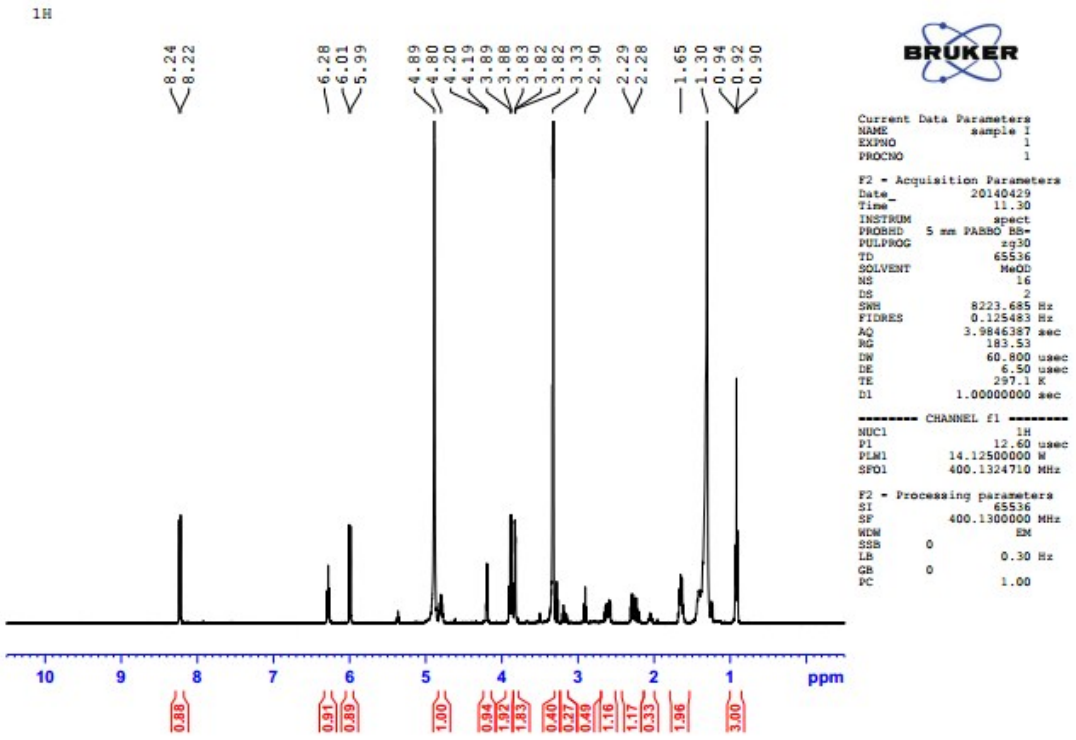
Compound 13



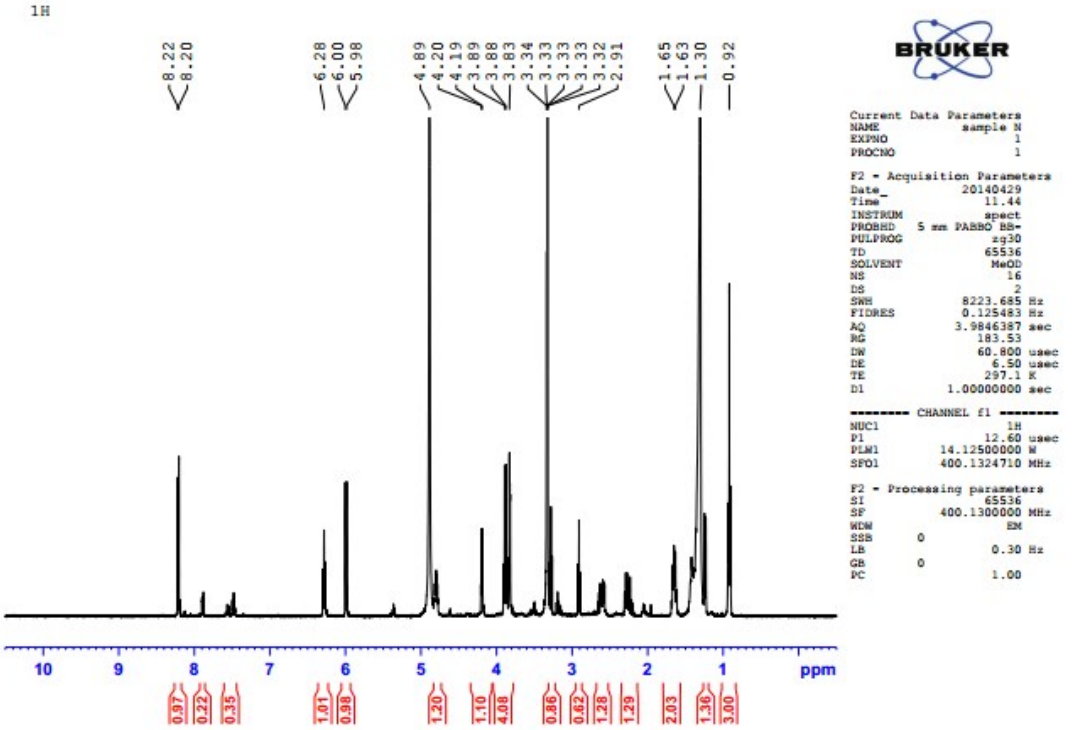
Compound 14



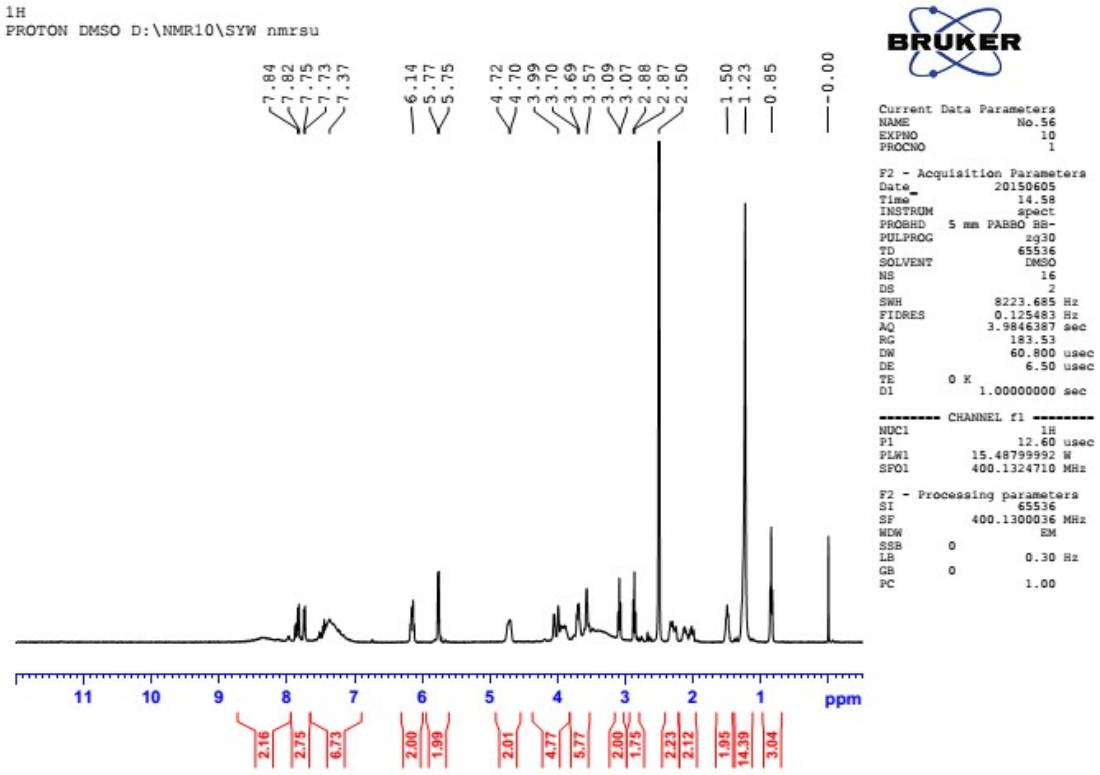
Compound 15



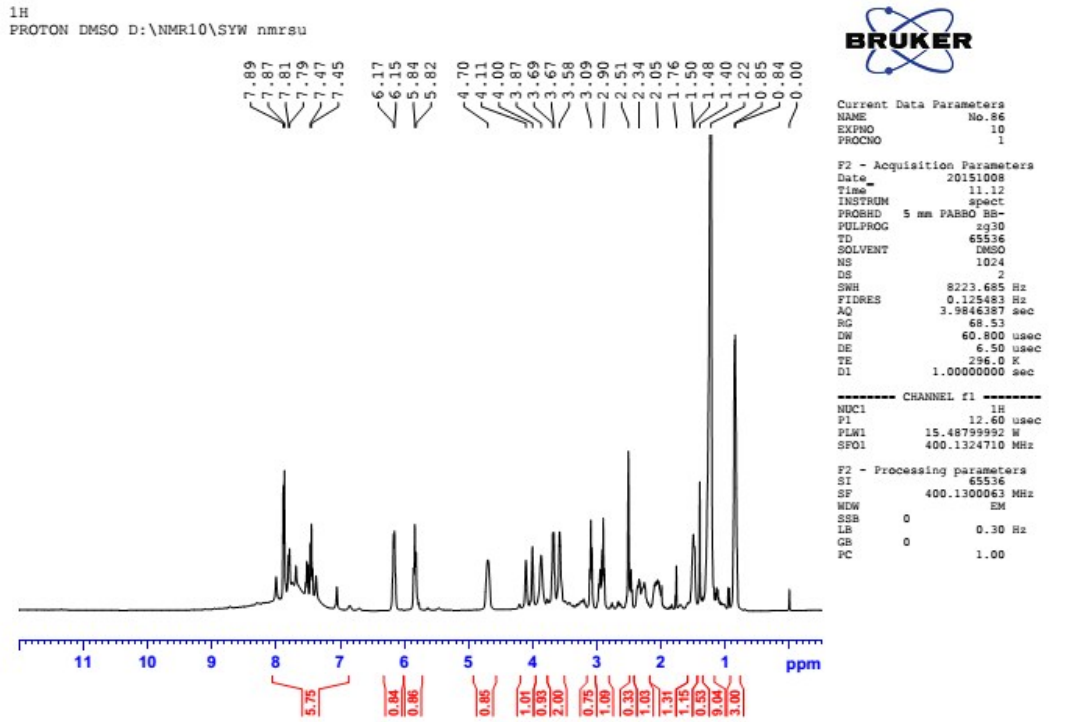
Compound 16



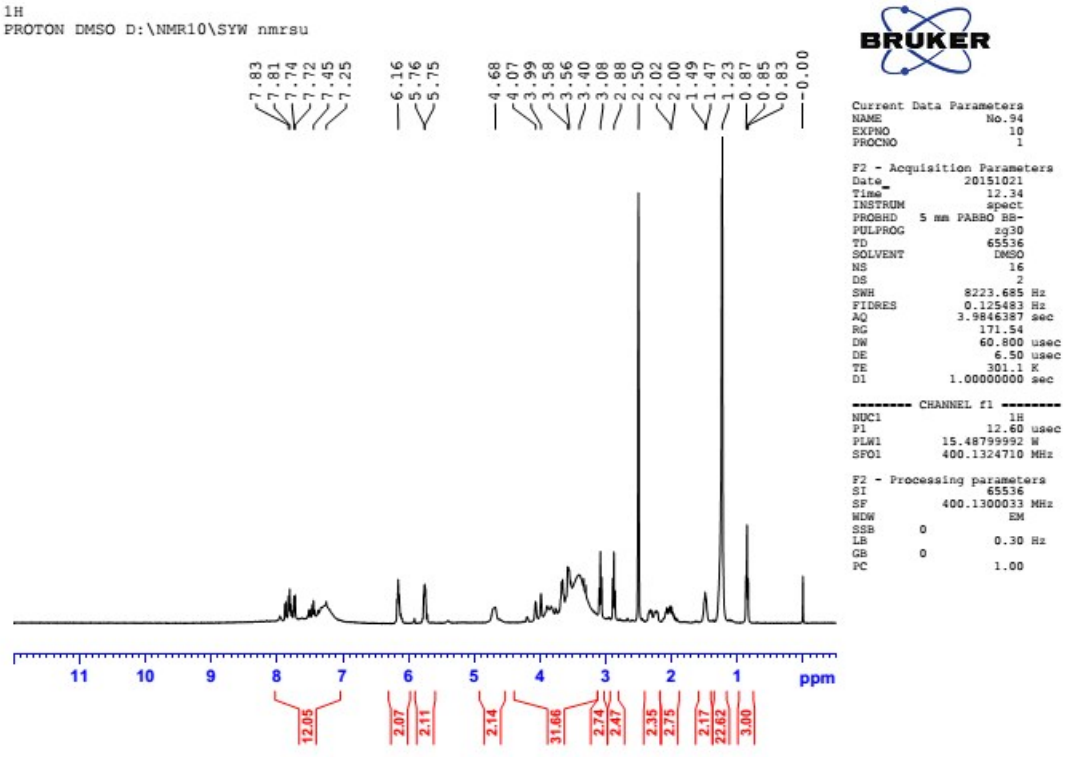
Compound 17



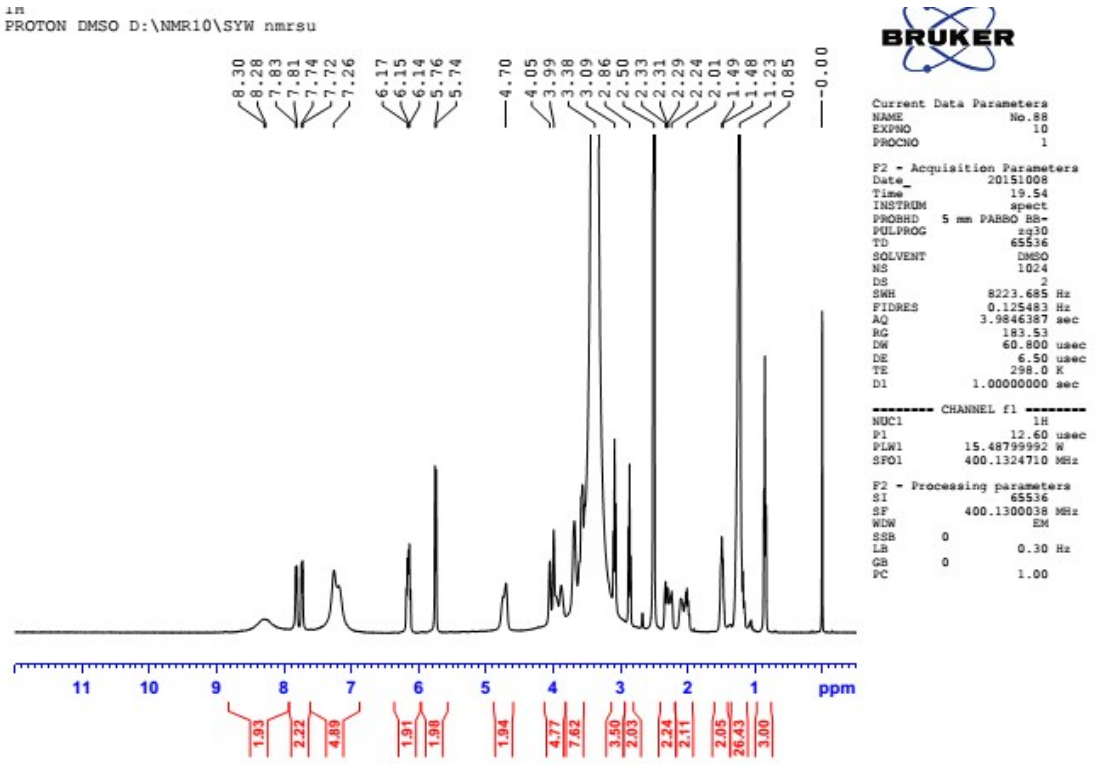
Compound 18



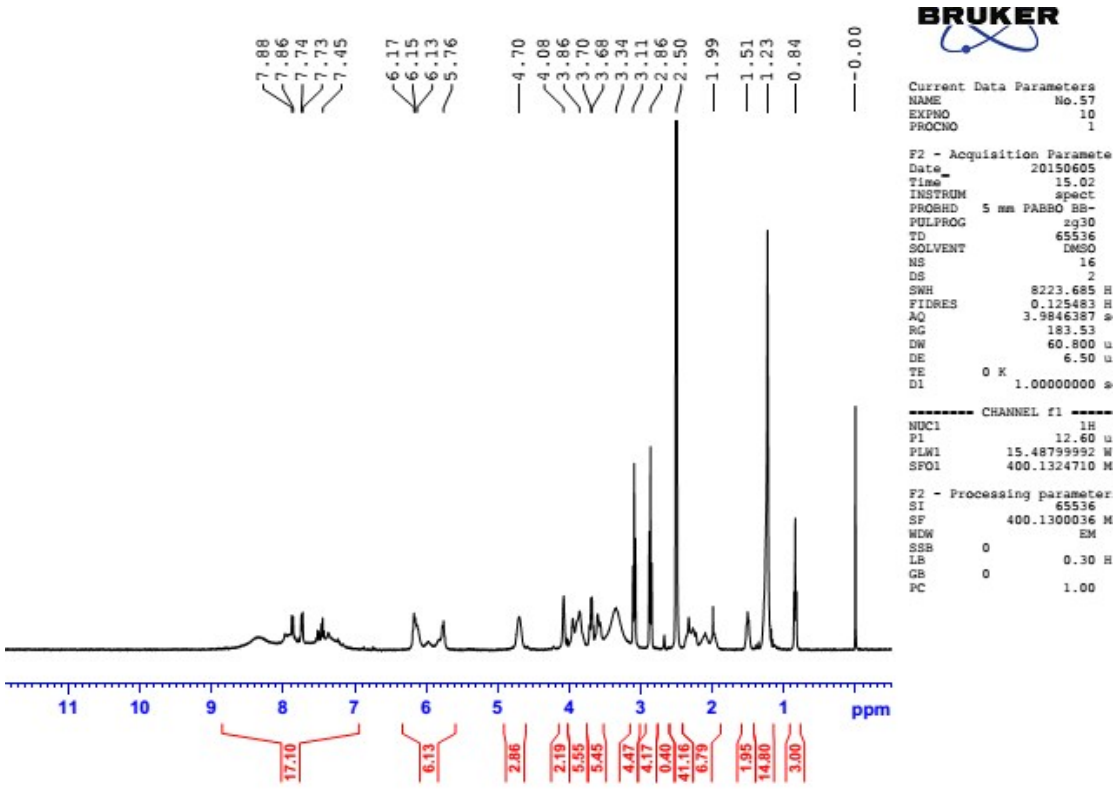
Compound 19



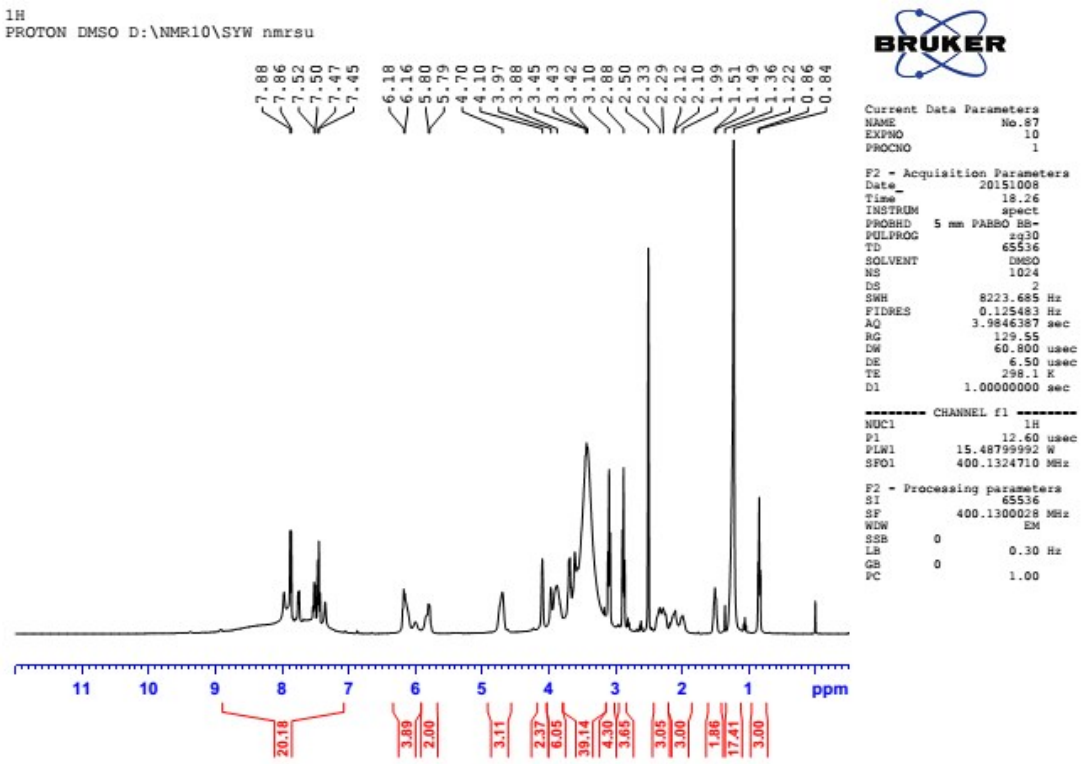
Compound 20



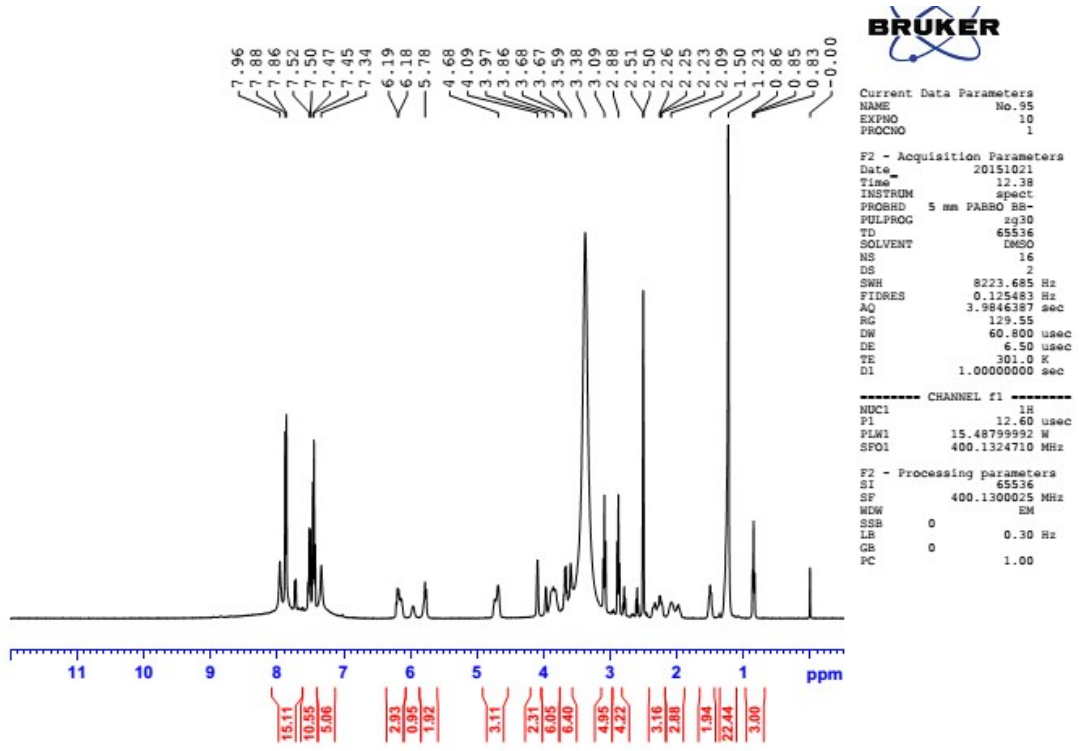
Compound 21



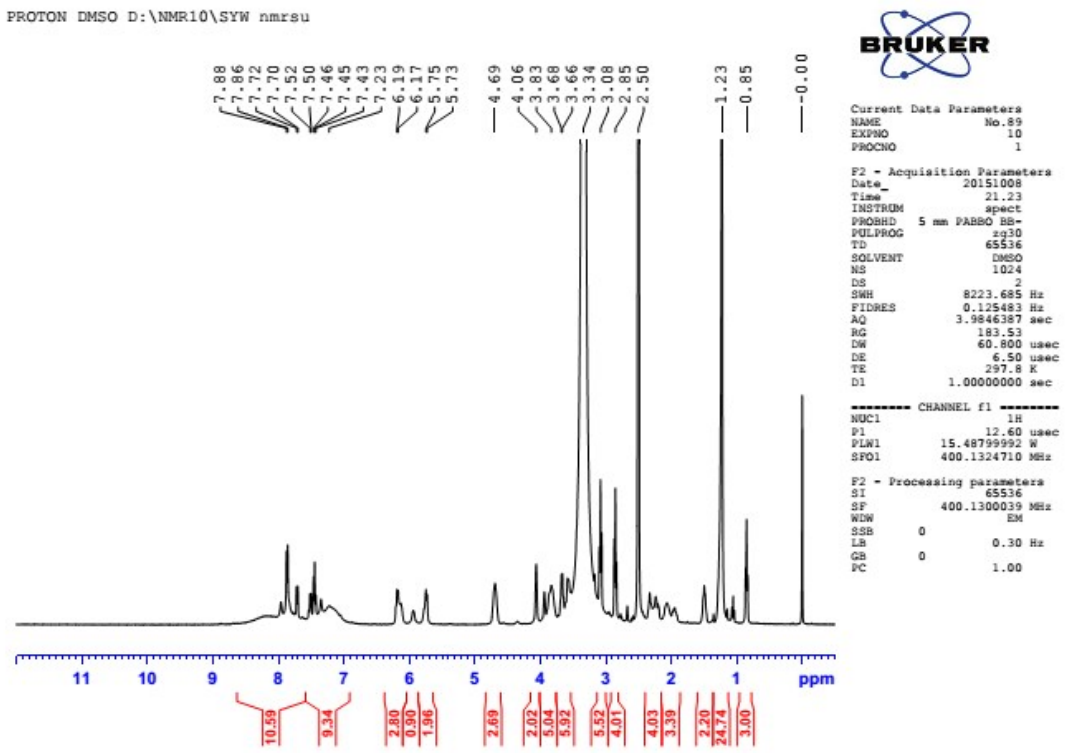
Compound 22



Compound 23

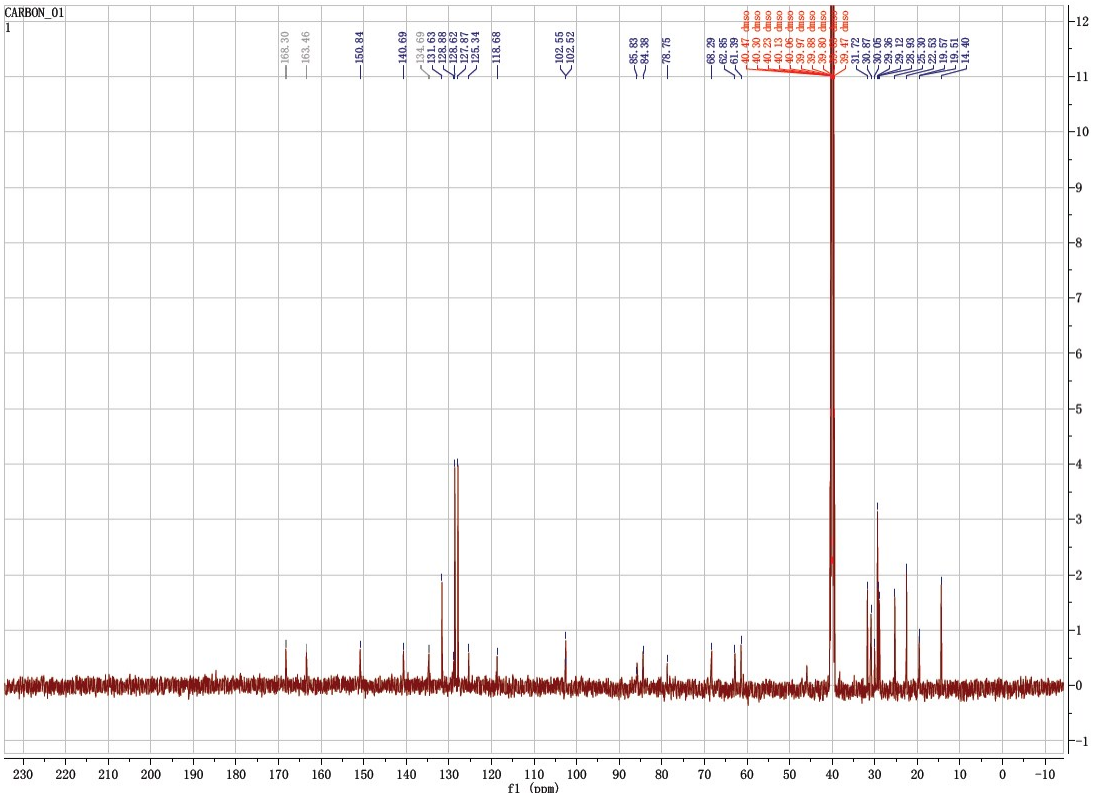


Compound 24

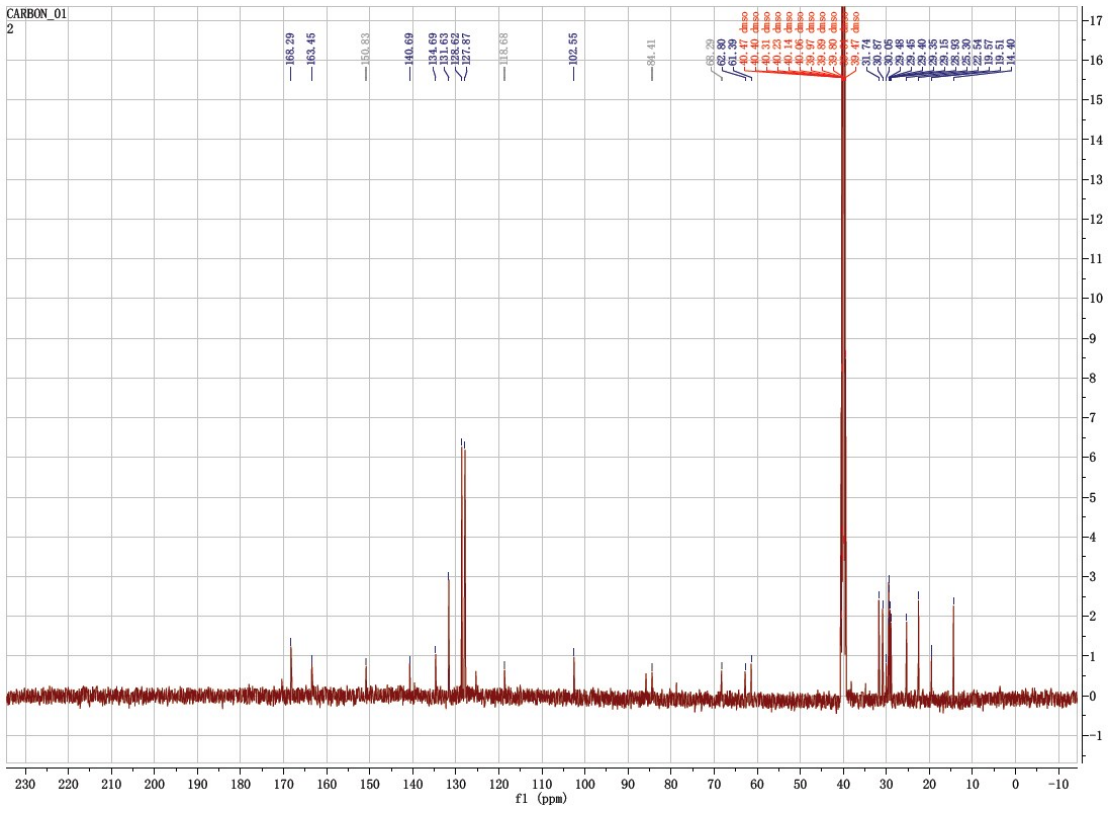


S5. ^{13}C-NMR spectrum of compound 1-24

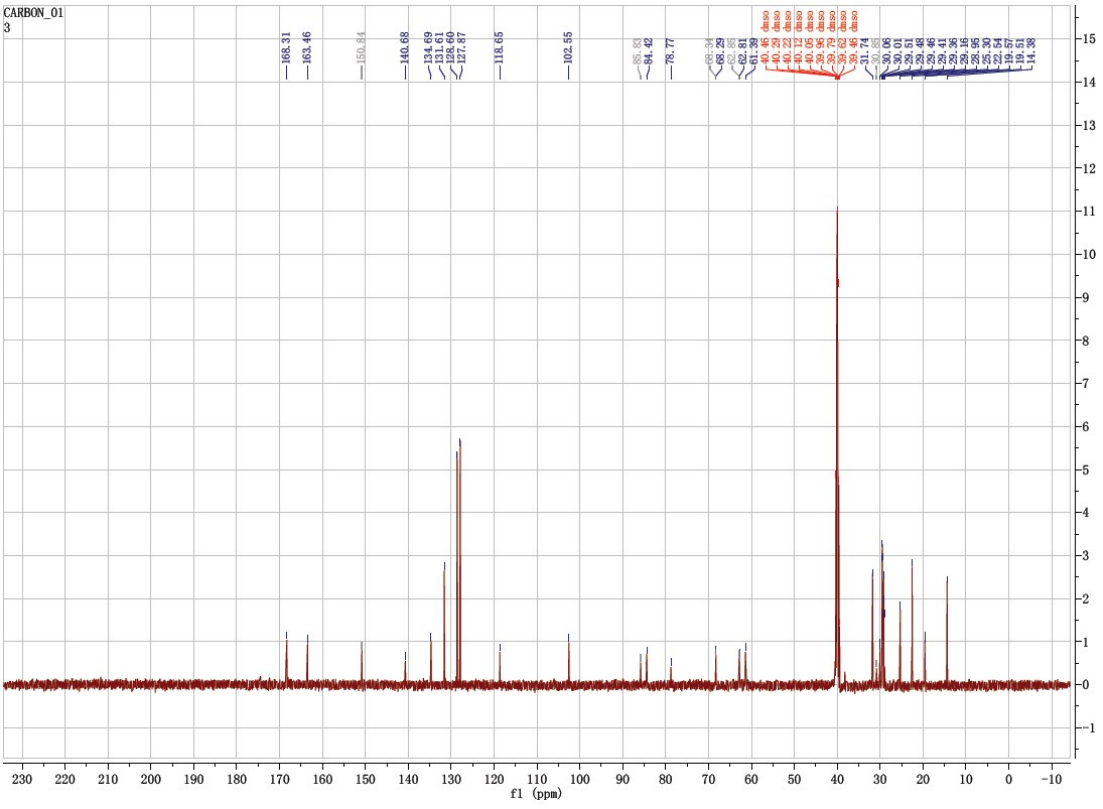
Compound 1



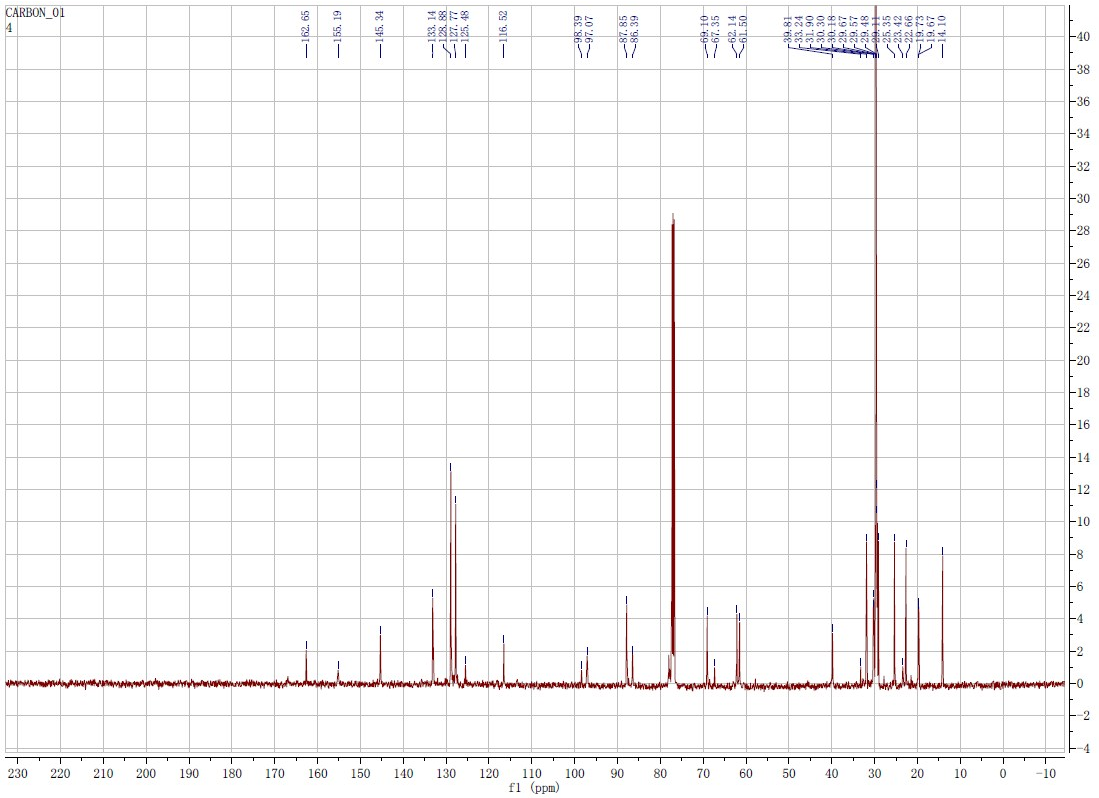
Compound 2



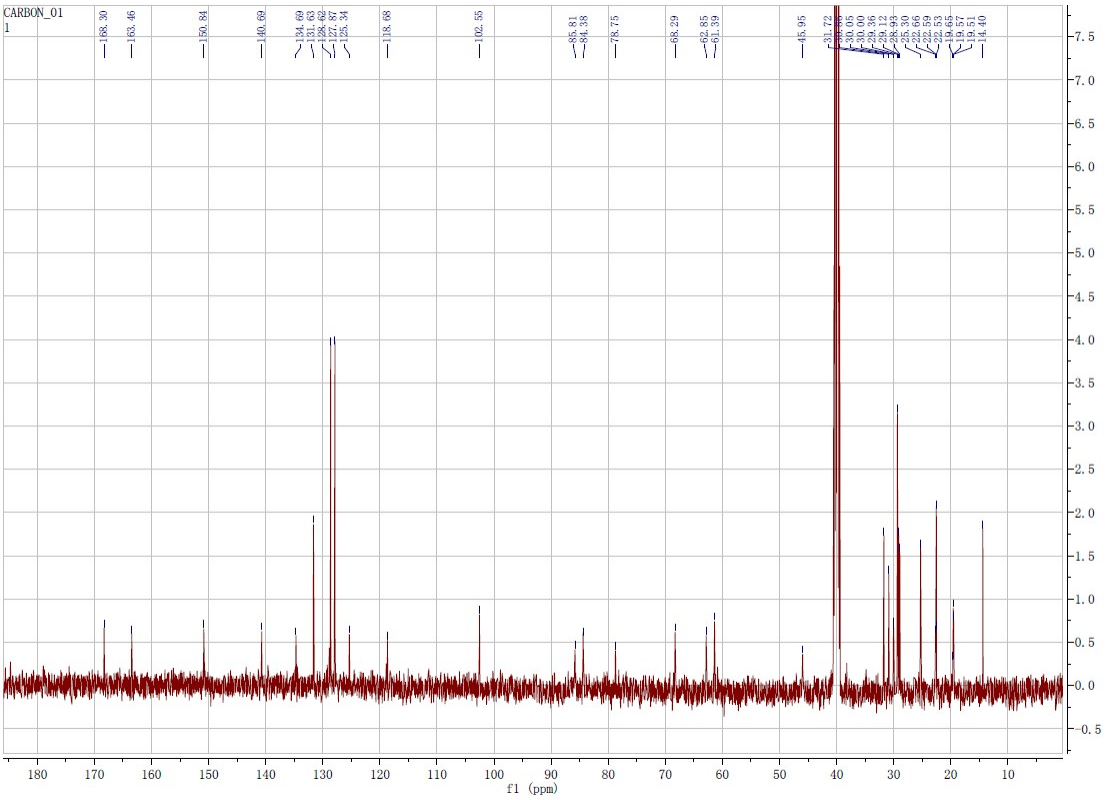
Compound 3



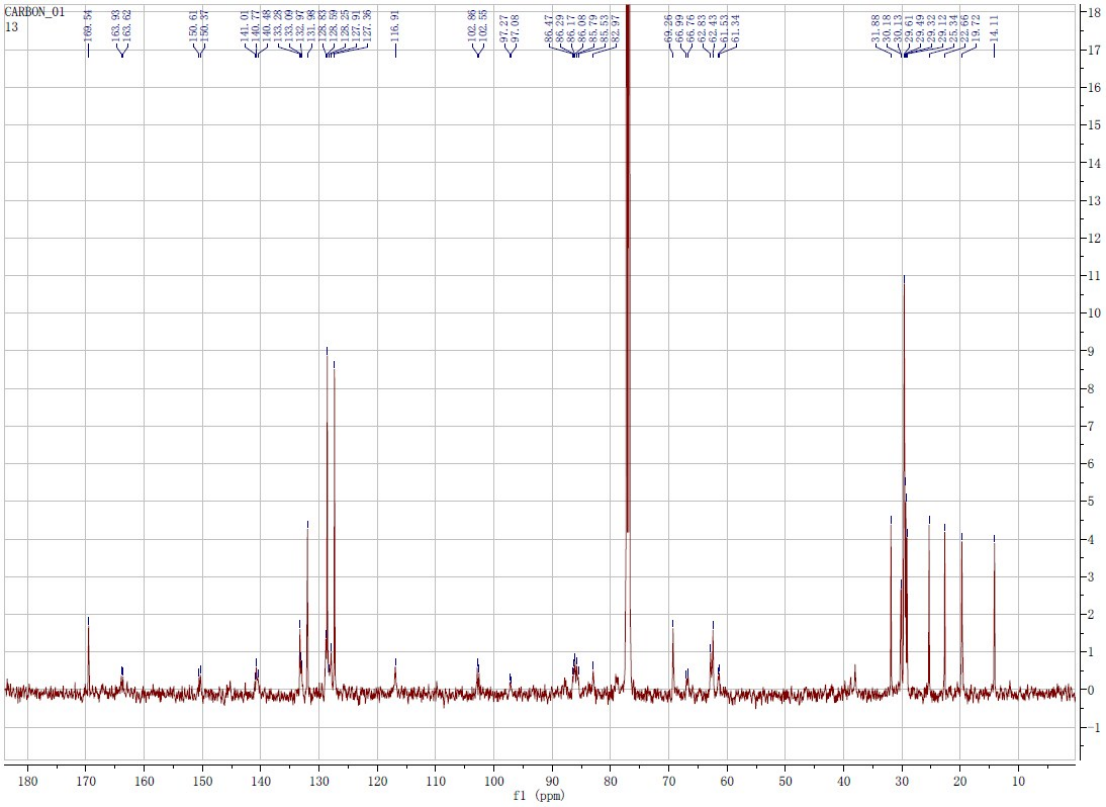
Compound 4



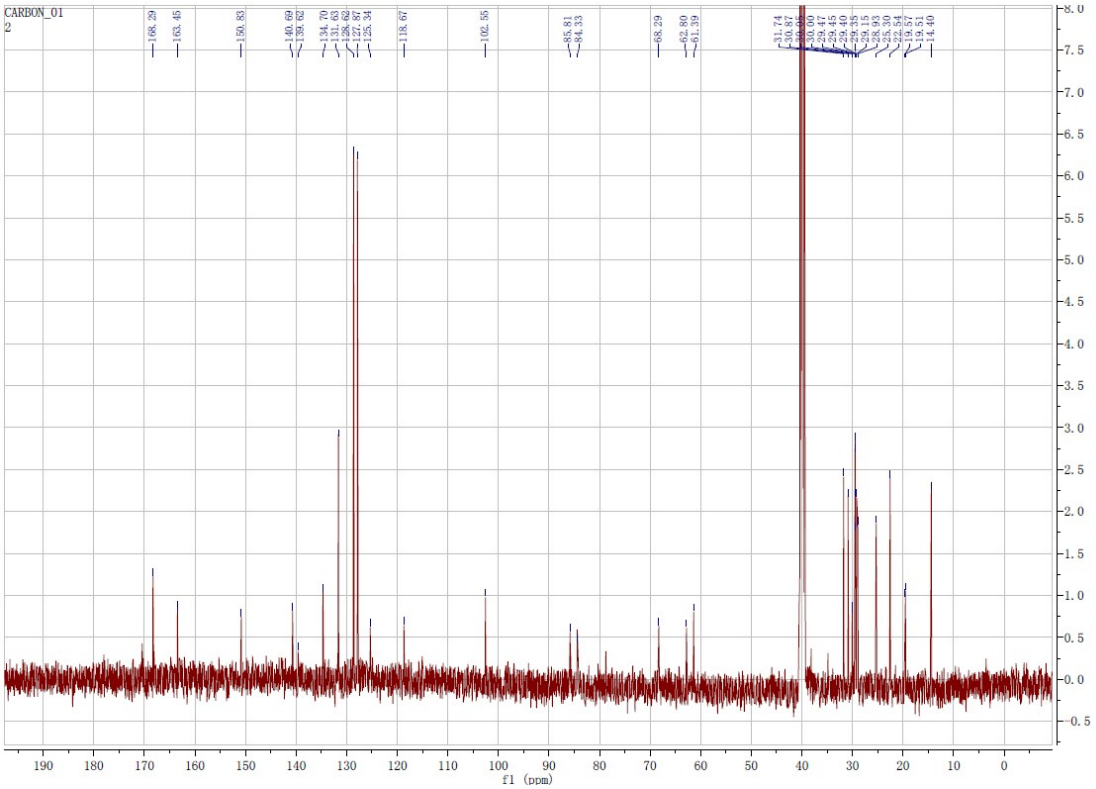
Compound 5



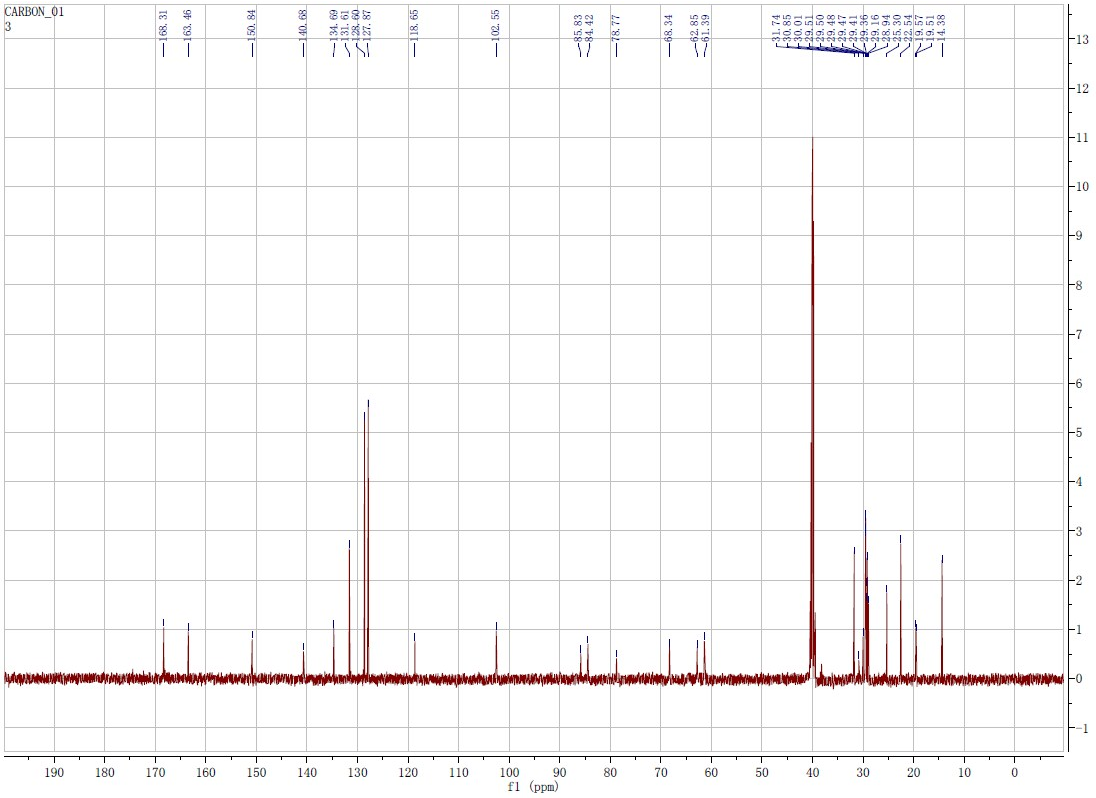
Compound 6



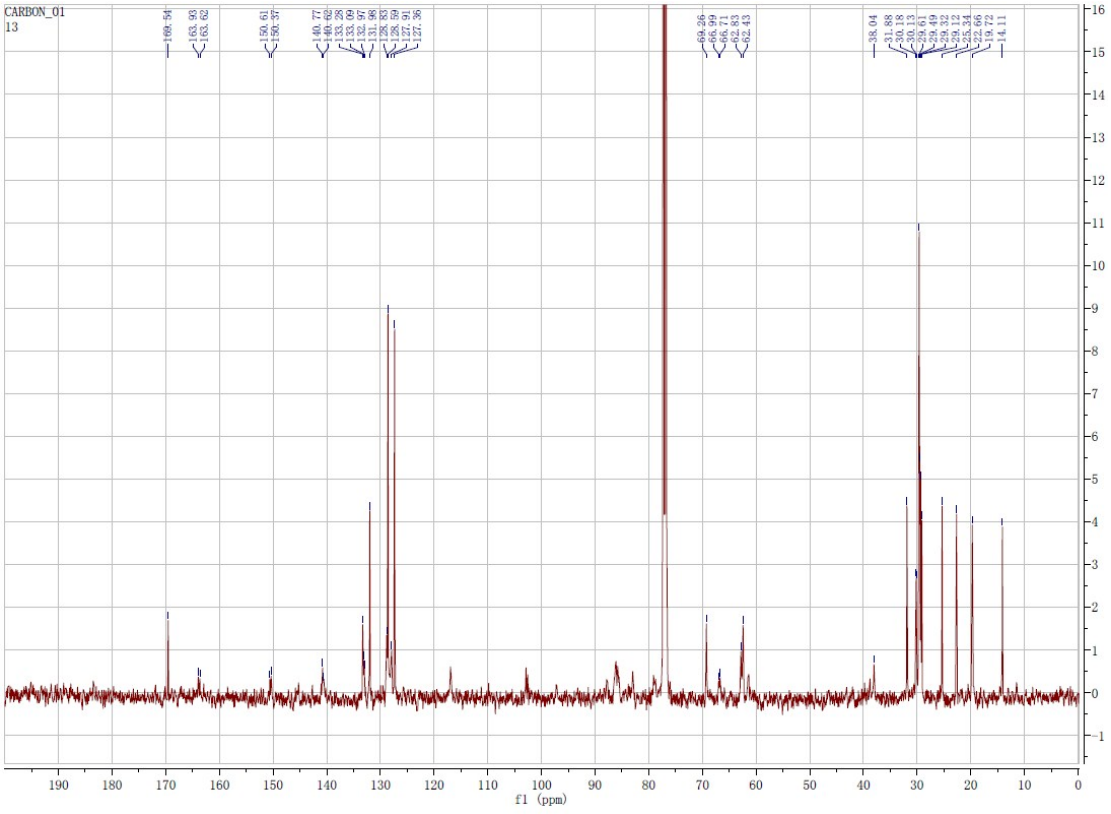
Compound 7



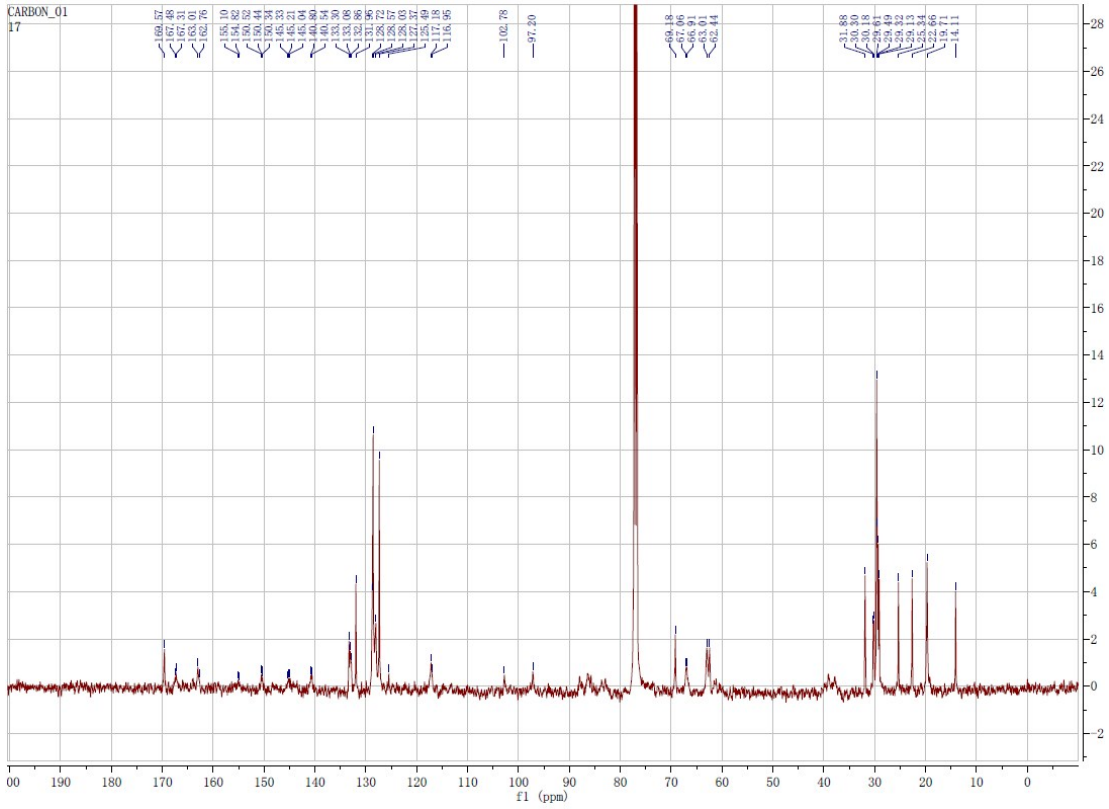
Compound 8



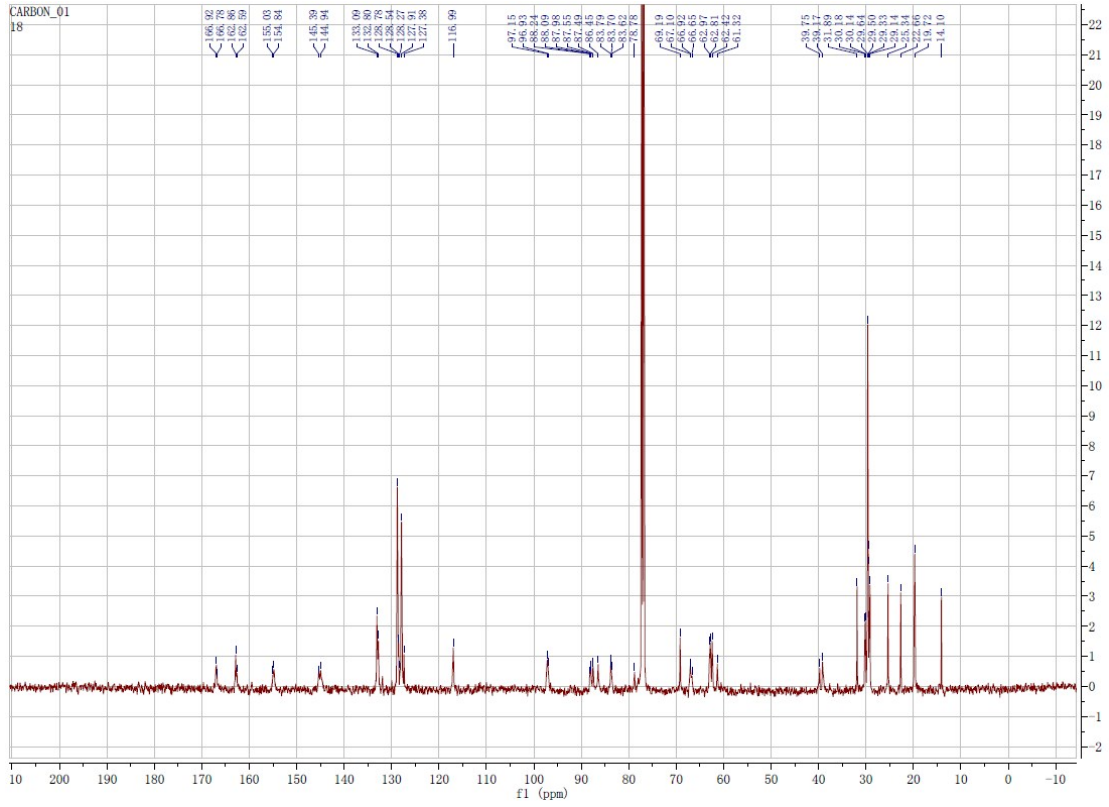
Compound 9



Compound 10



Compound 11



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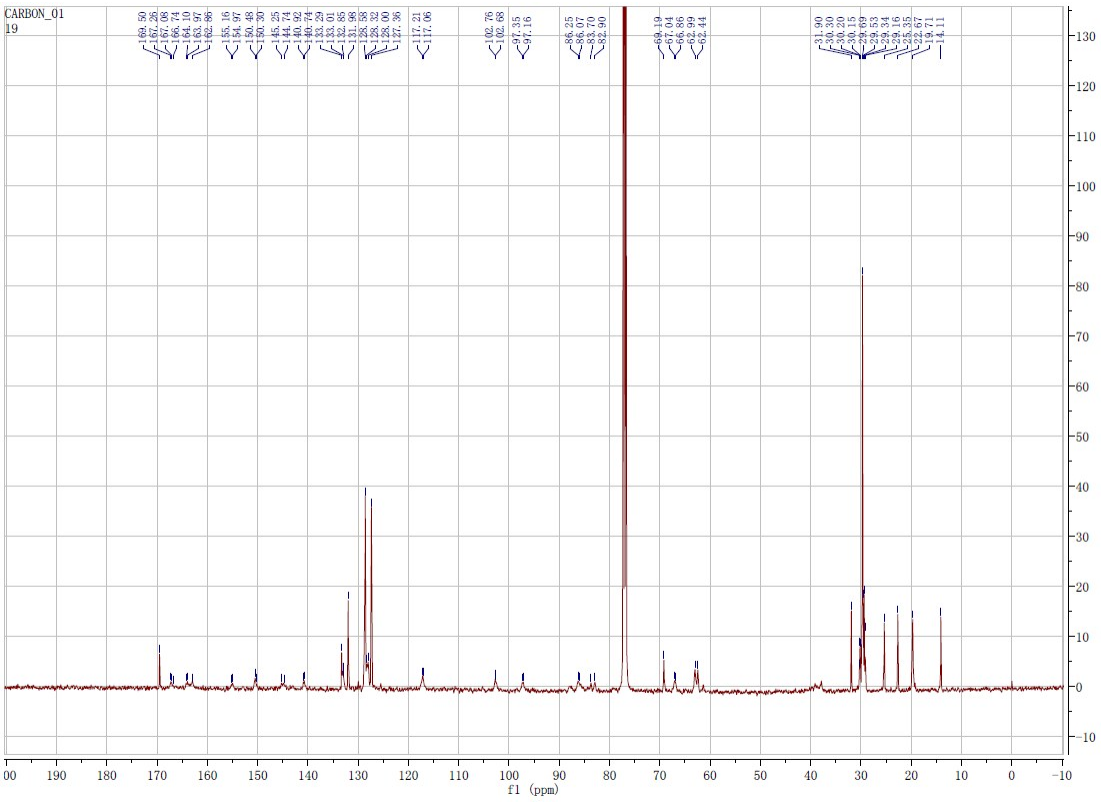
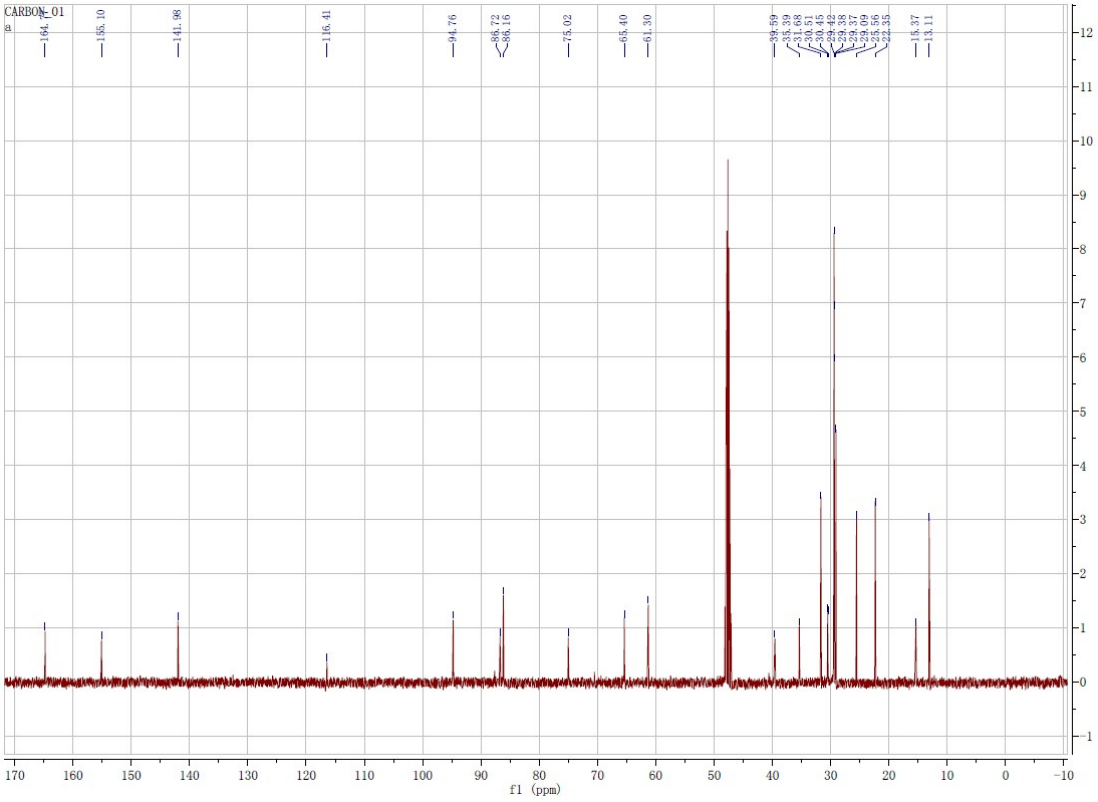
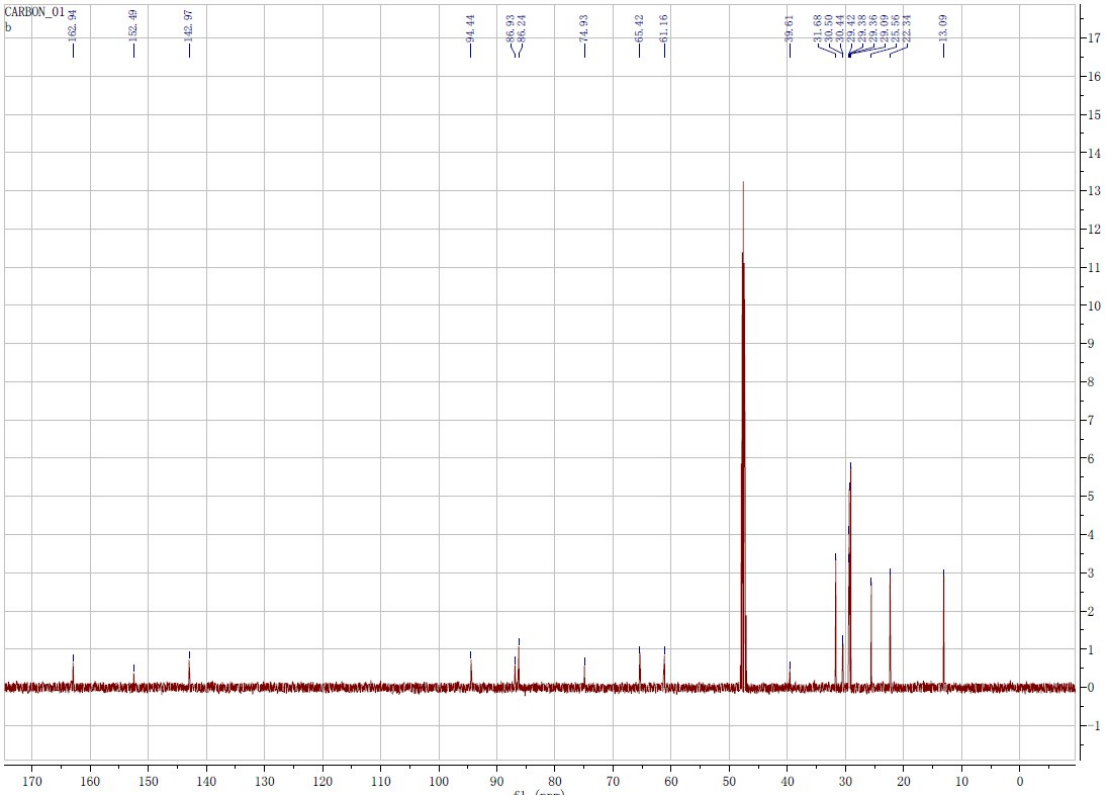


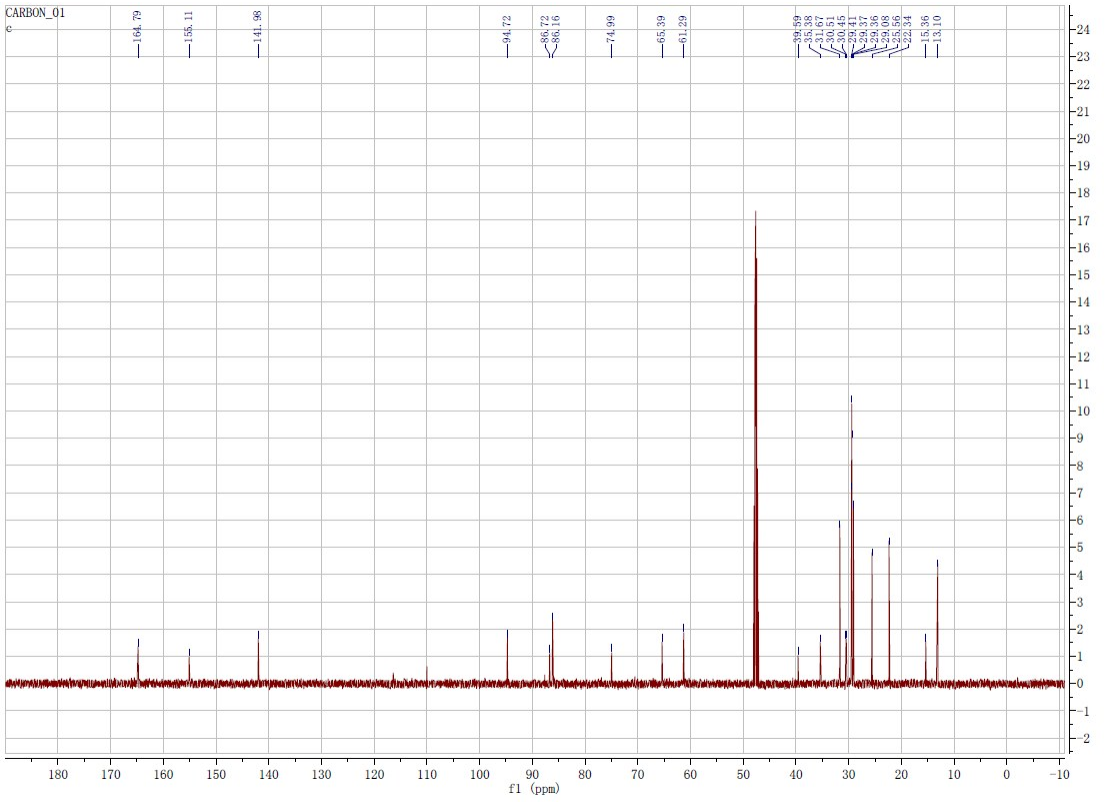
Compound 13



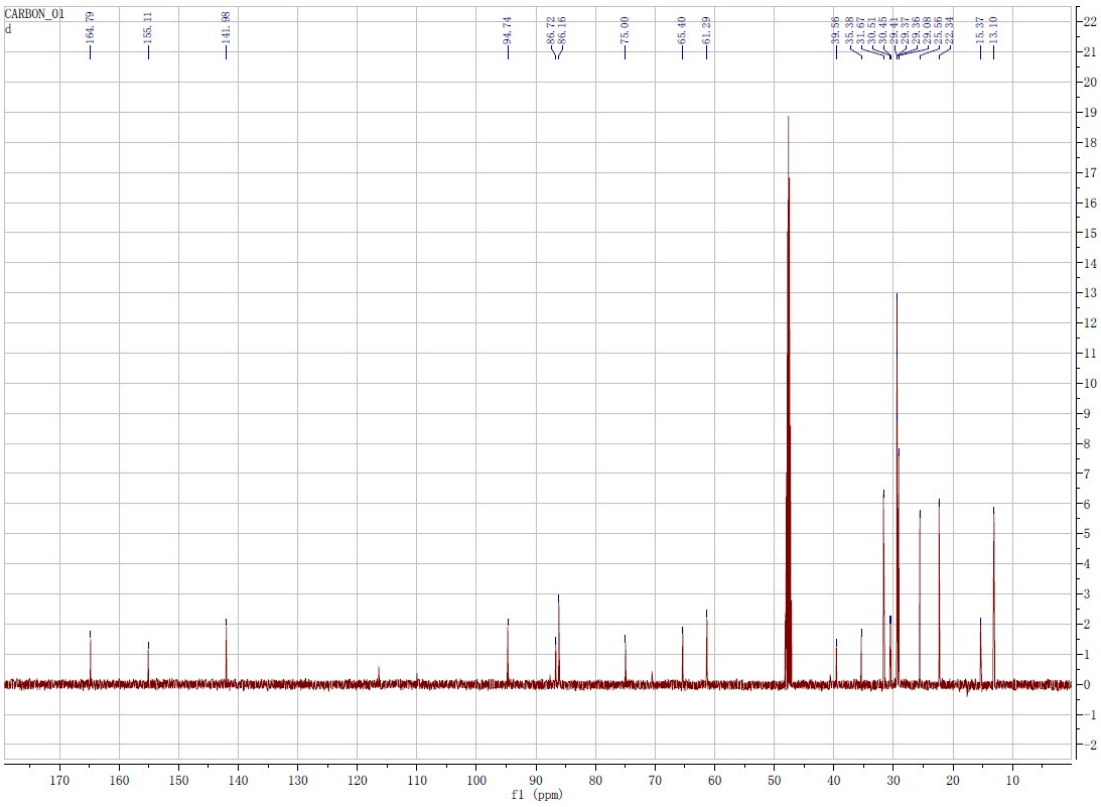
Compound 14



Compound 15



Compound 16



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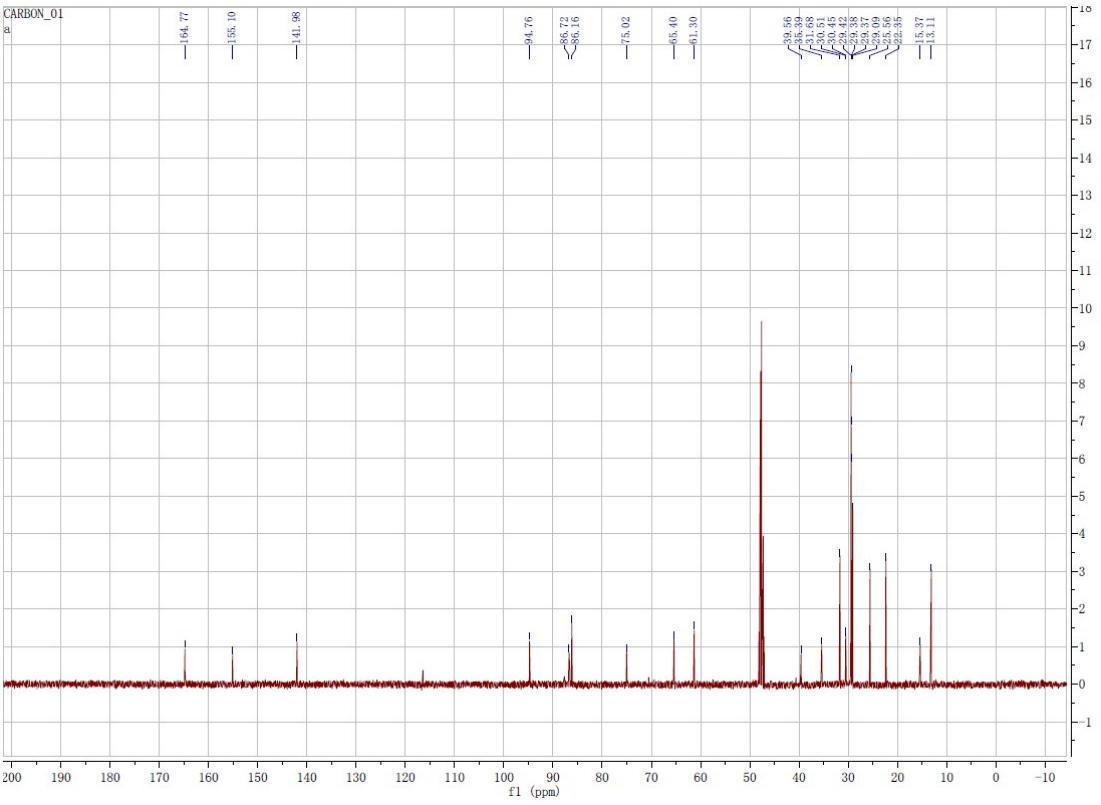

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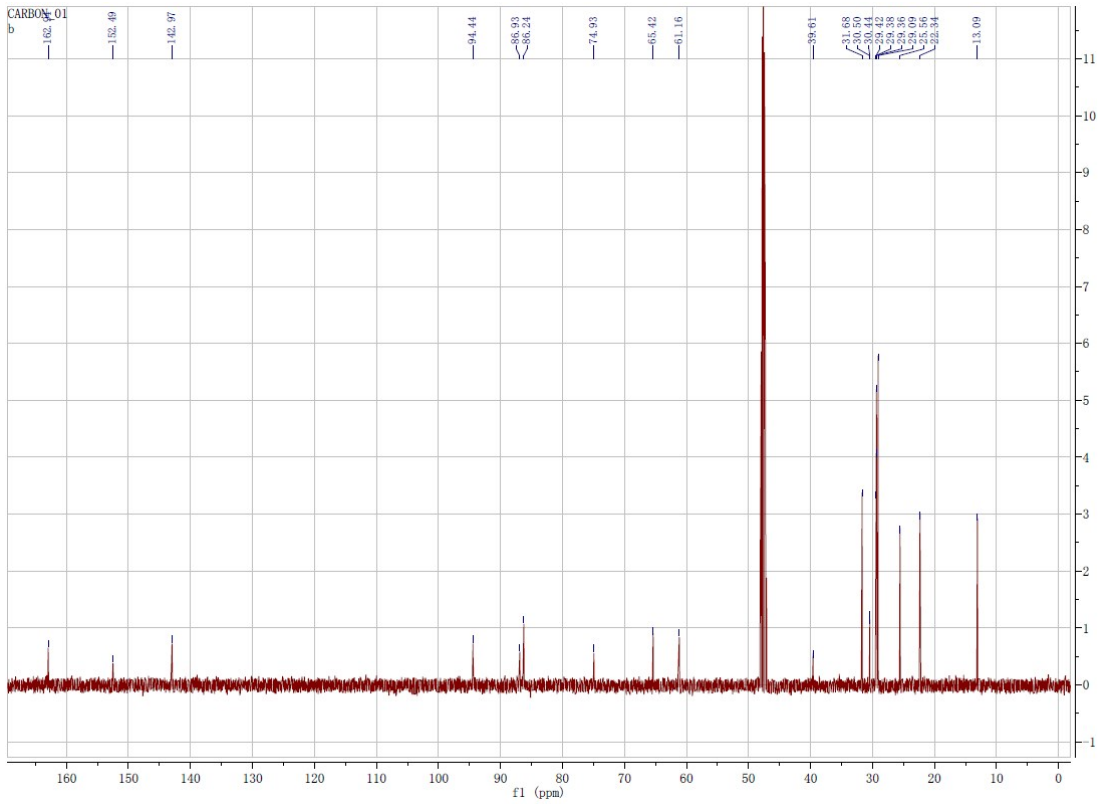
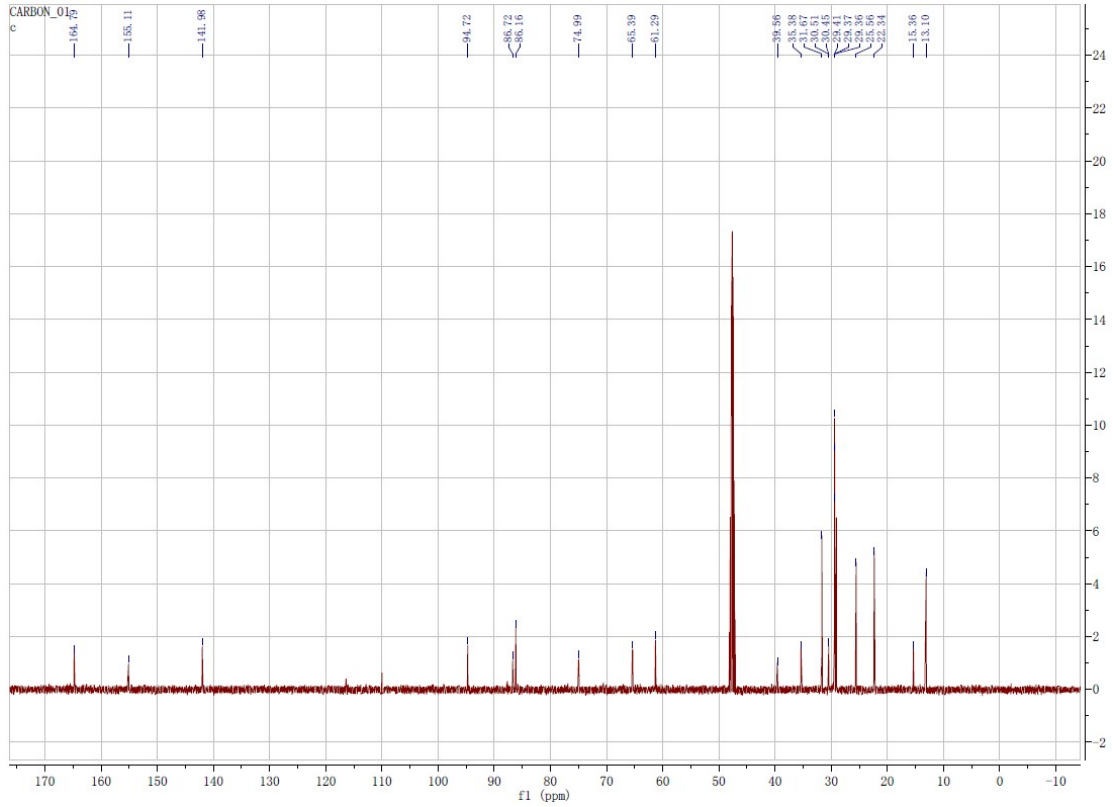
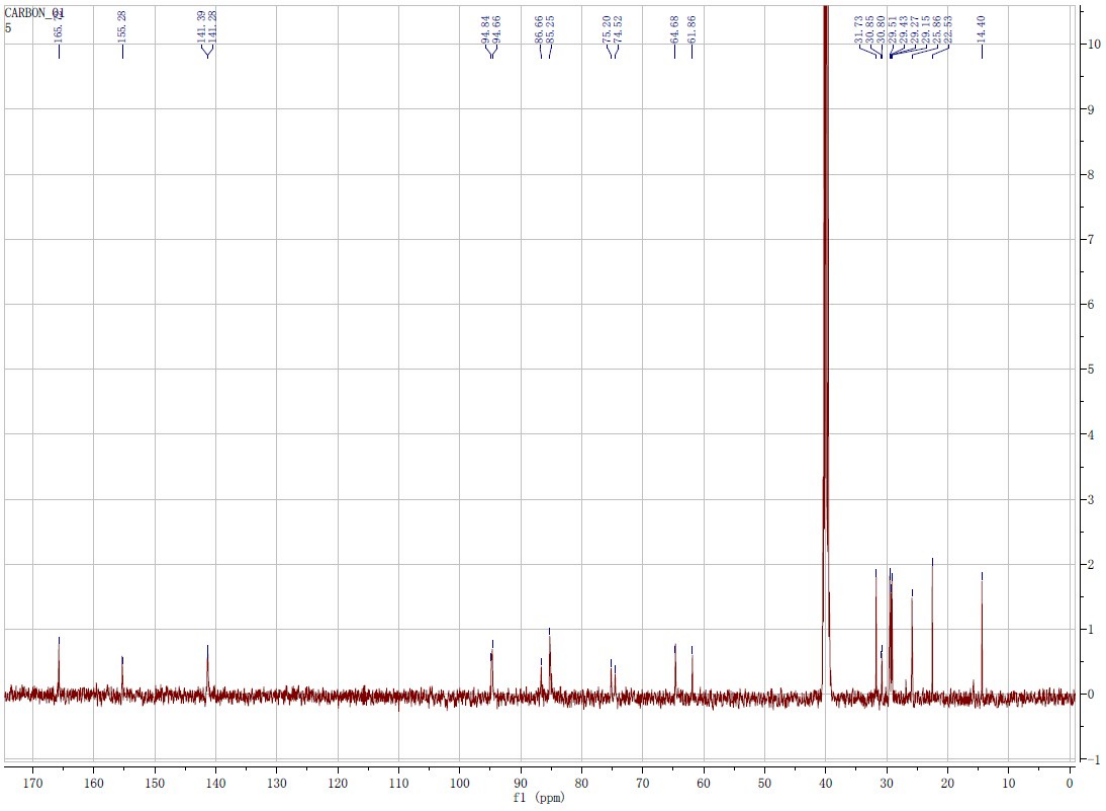


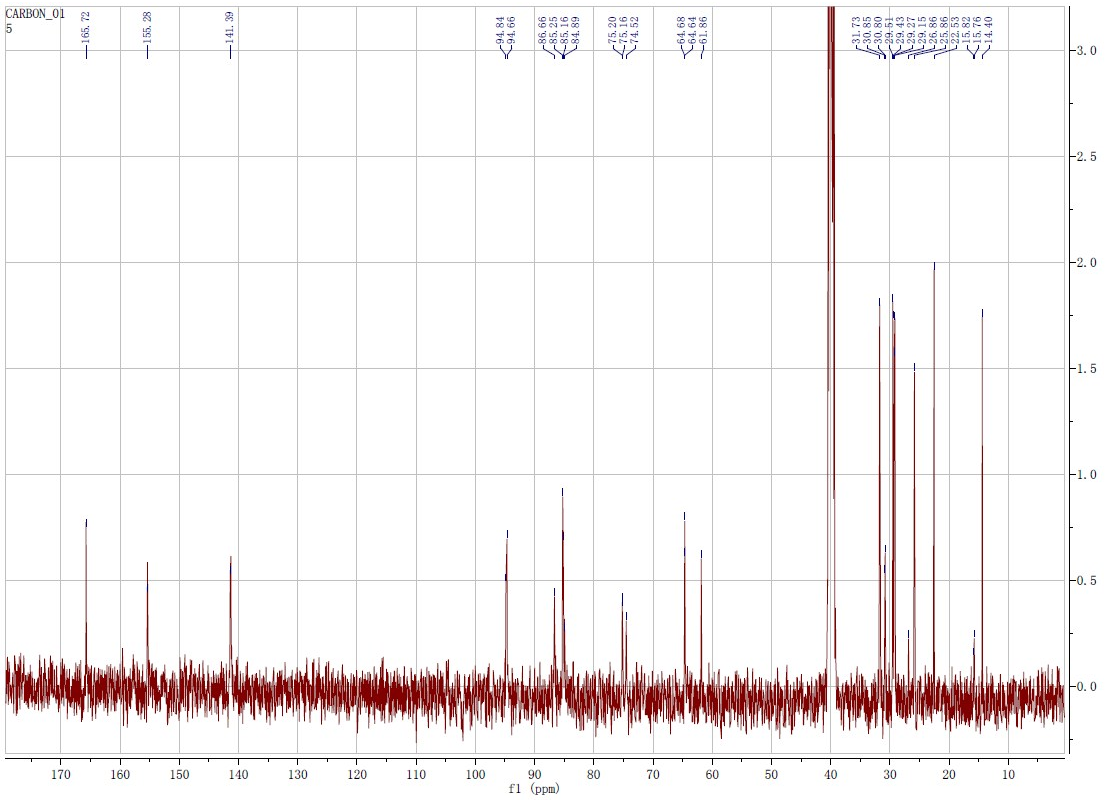
Compound 19



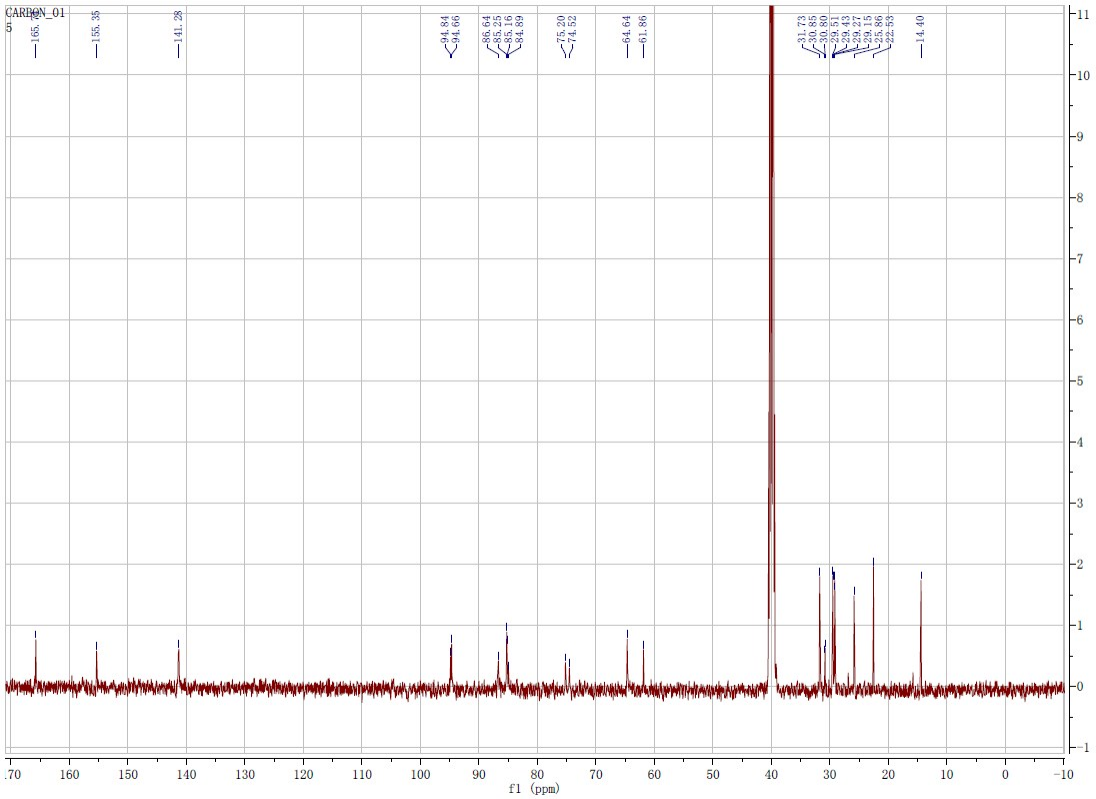
Compound 20



Compound 21



Compound 22



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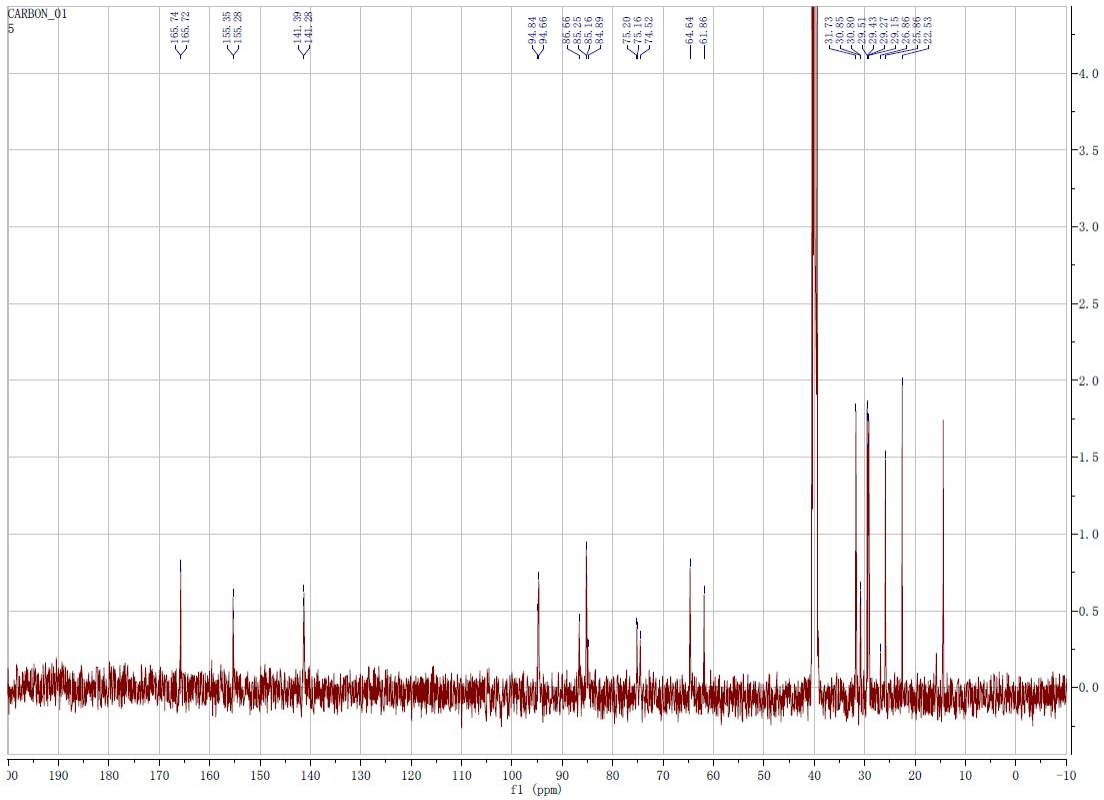
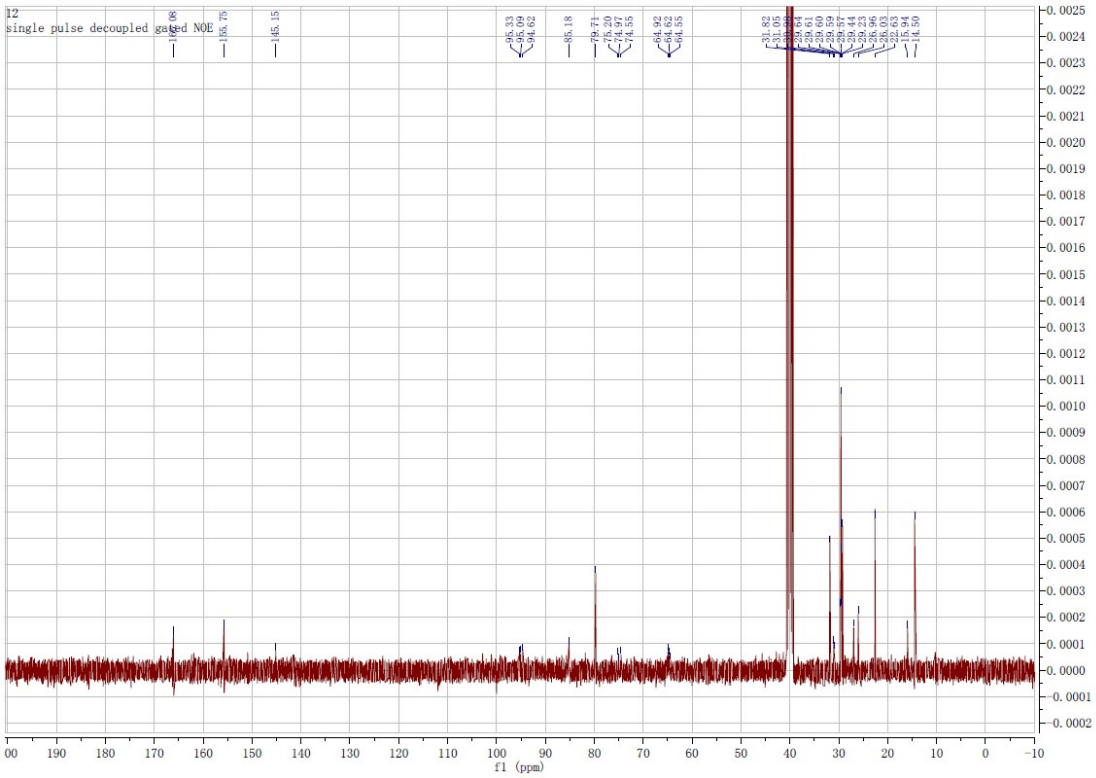
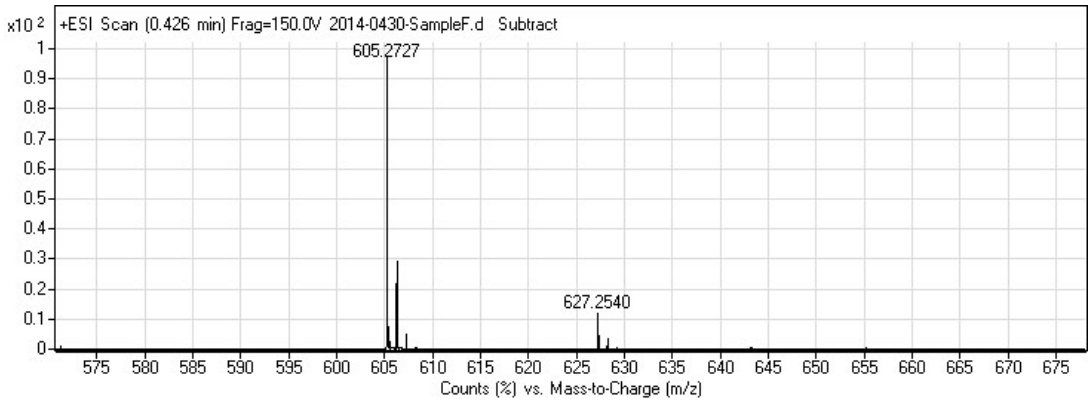


Compound 24

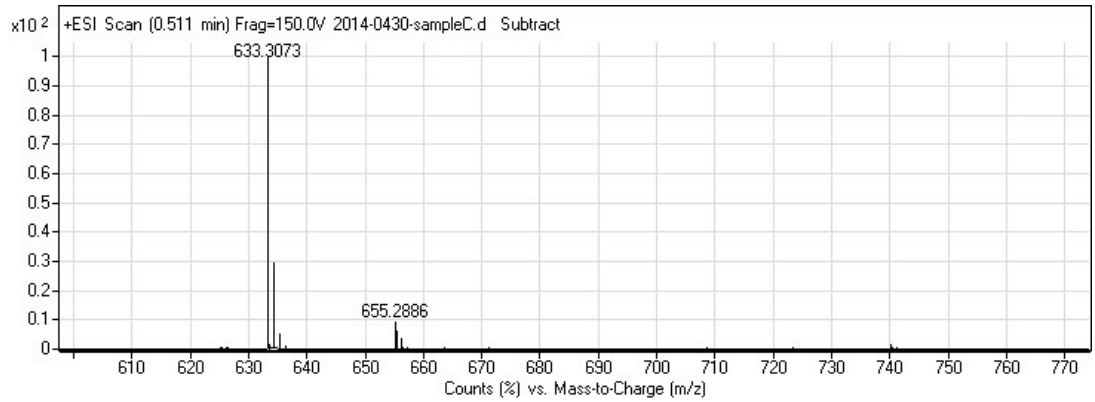


S6. High Resolution MS spectrum of compound 1-24

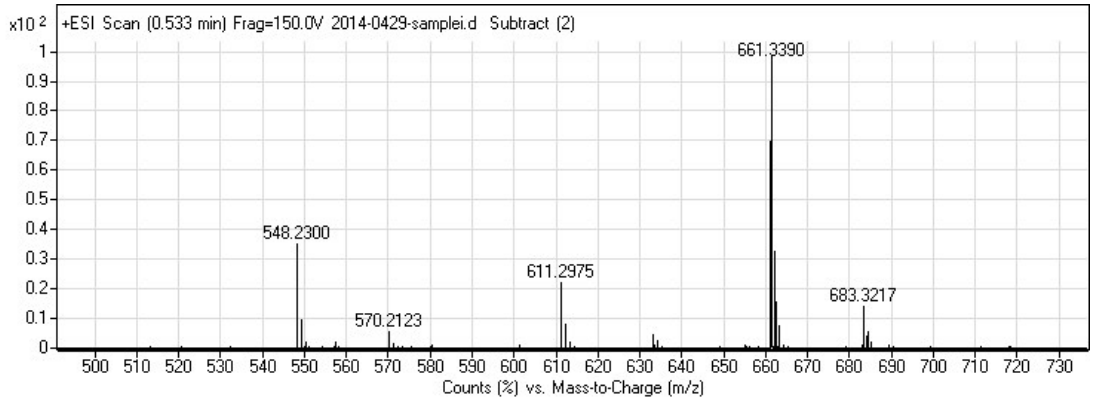
Compound 1



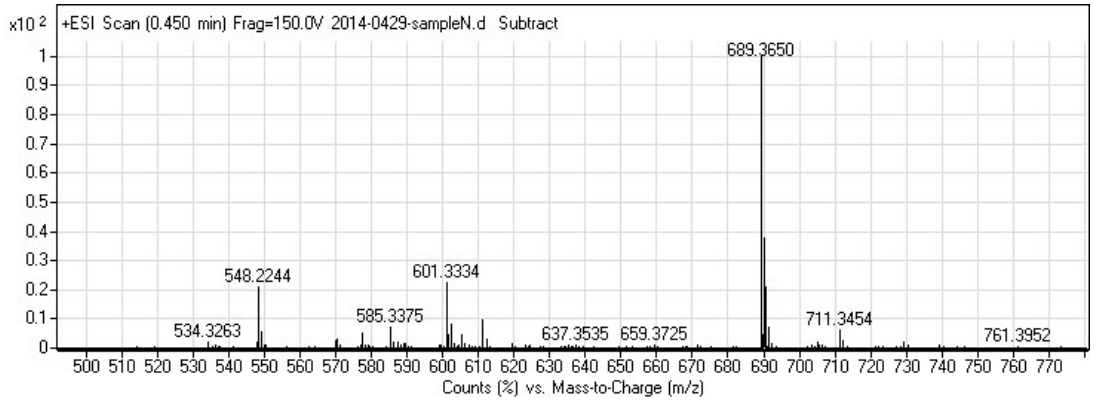
Compound 2



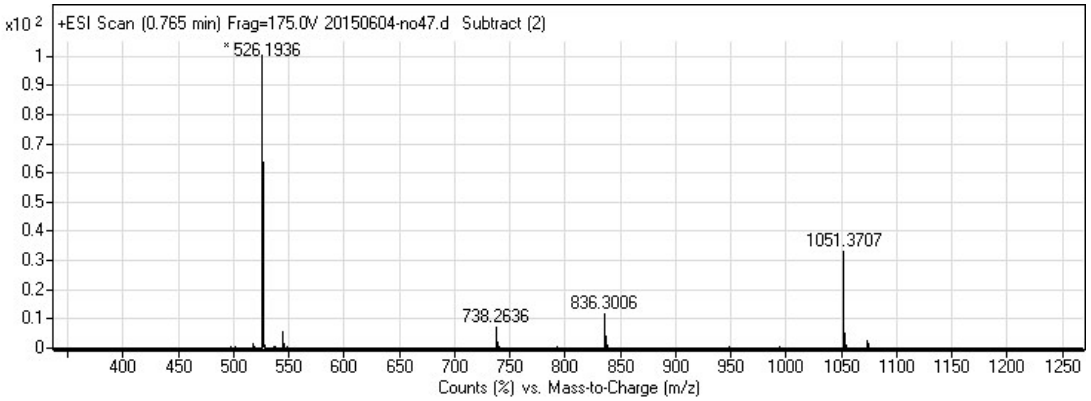
Compound 3



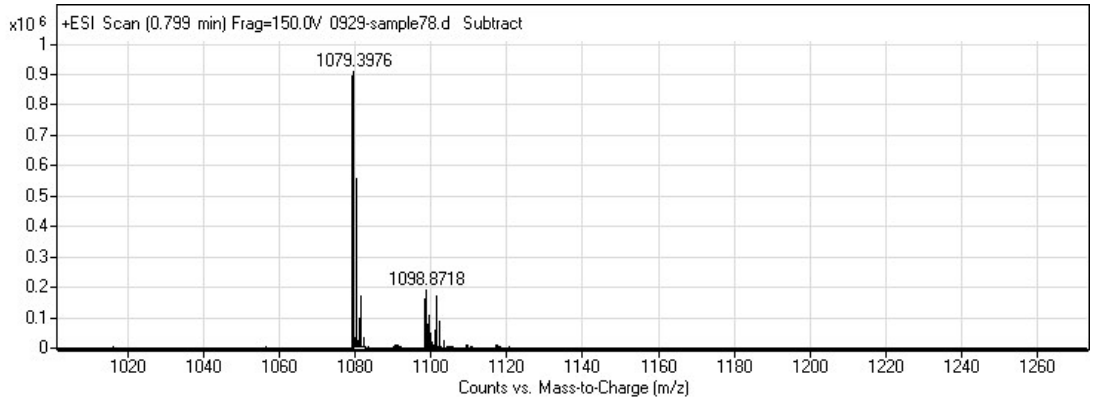
Compound 4



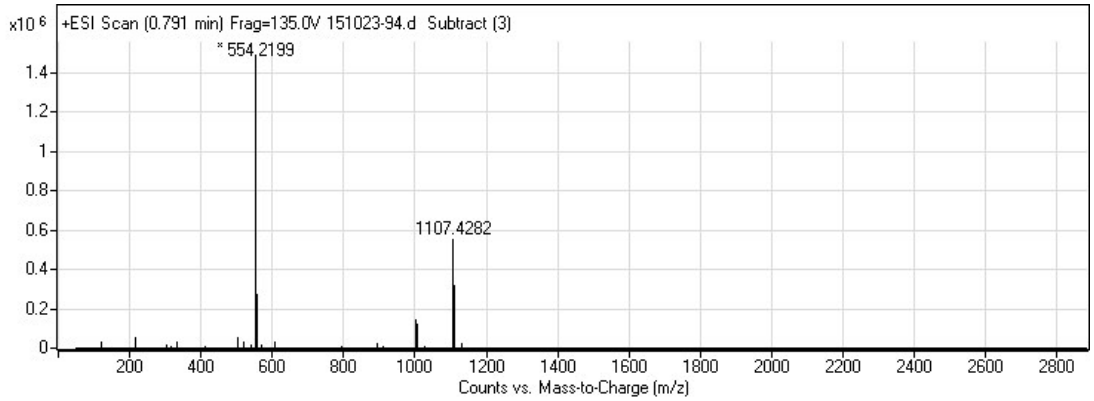
Compound 5



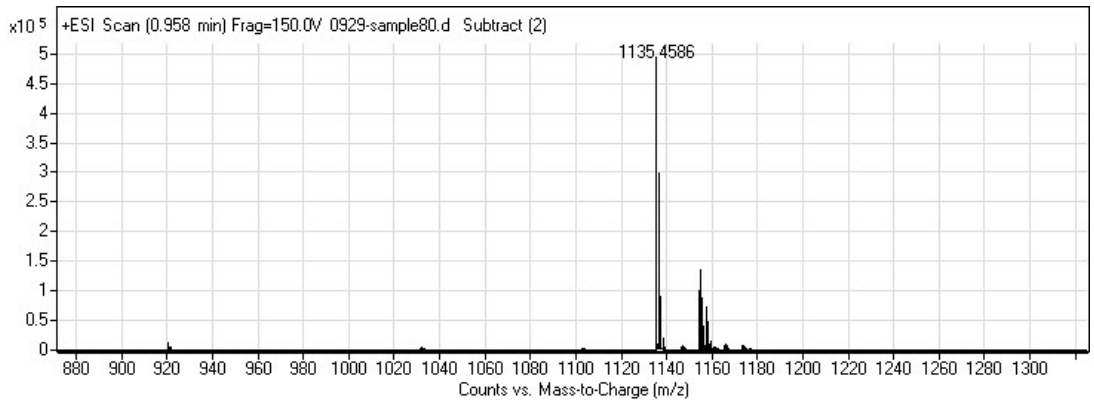
Compound 6



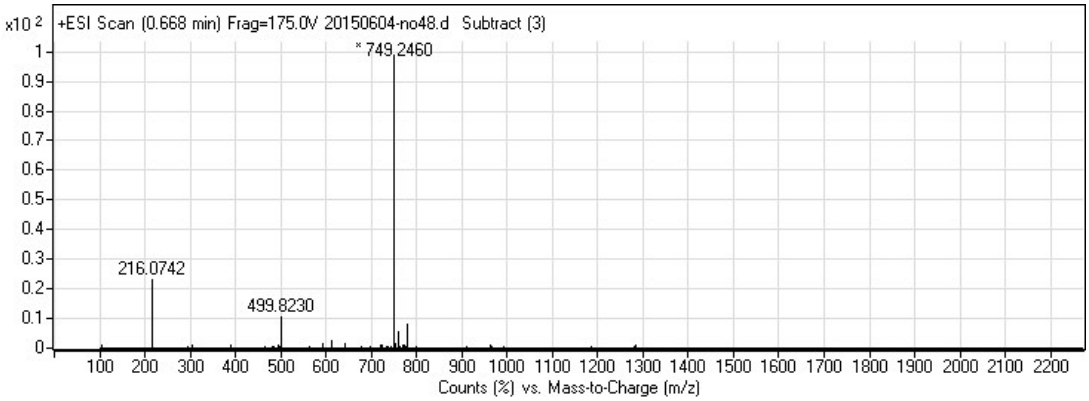
Compound 7



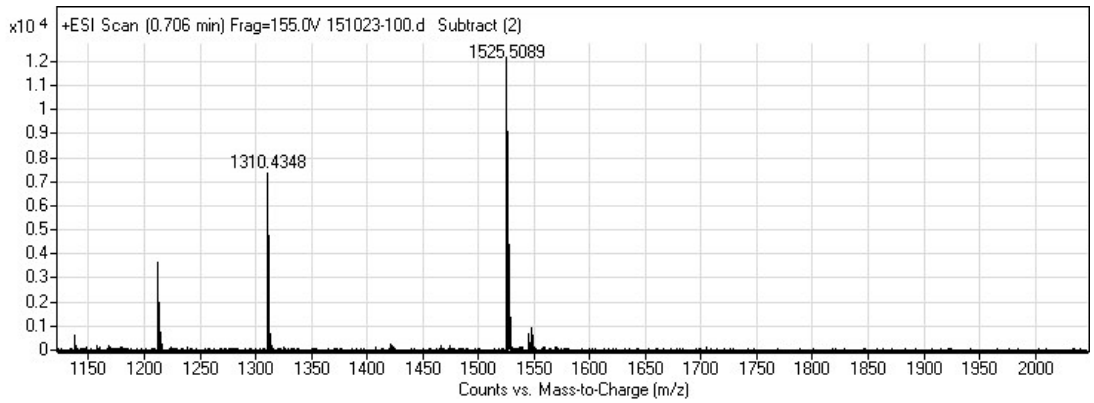
Compound 8



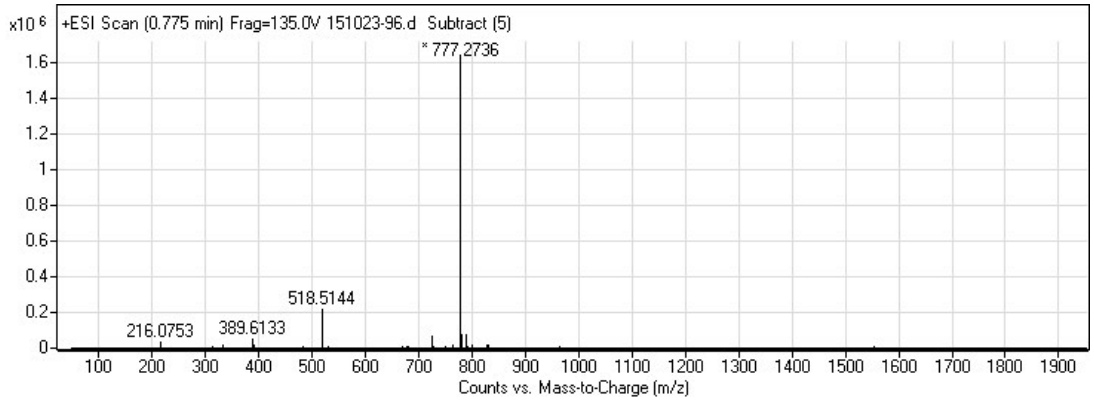
Compound 9



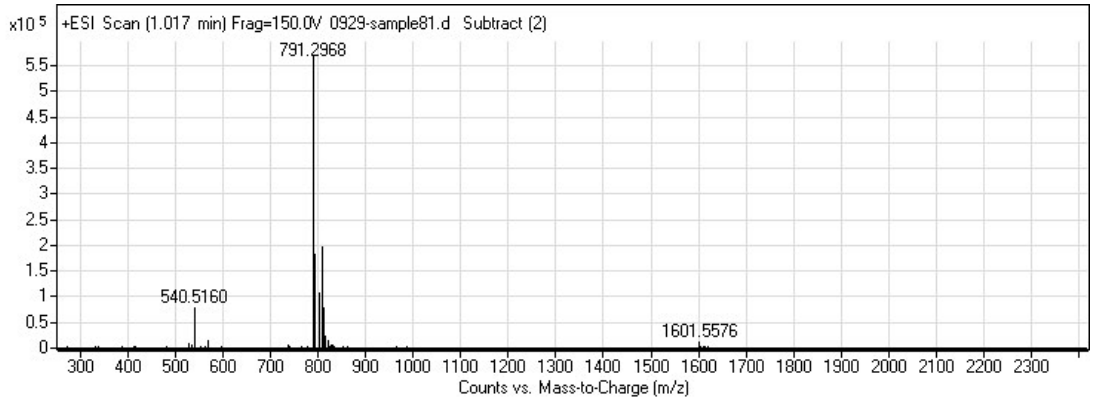
Compound 10



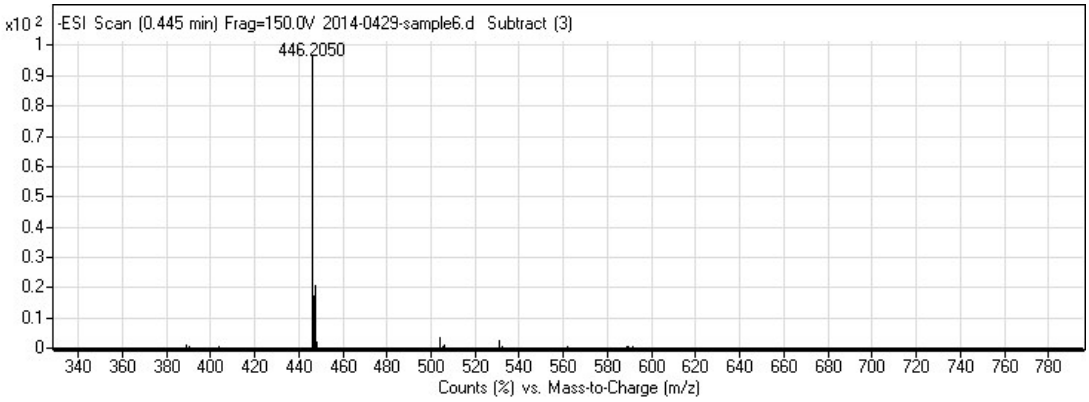
Compound 11



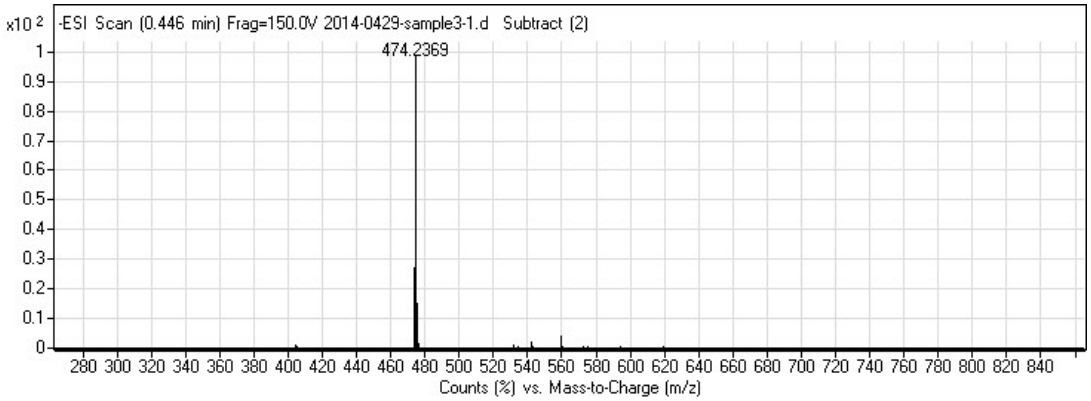
Compound 12



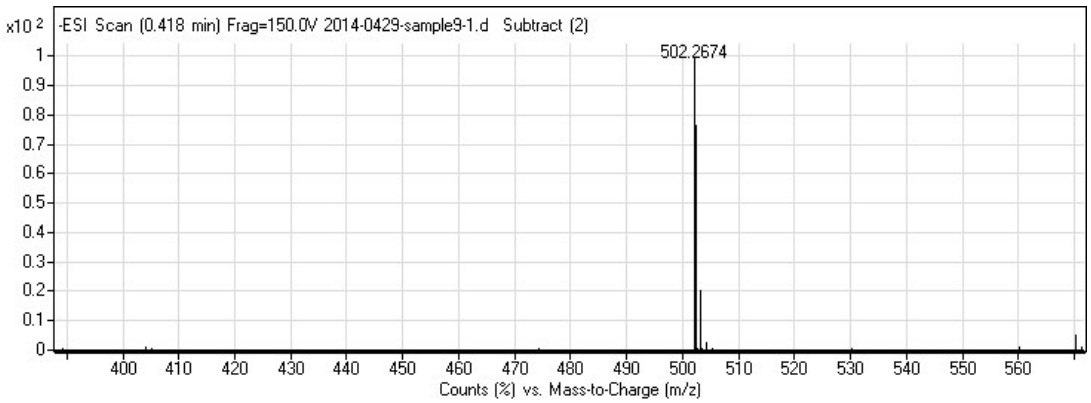
Compound 13



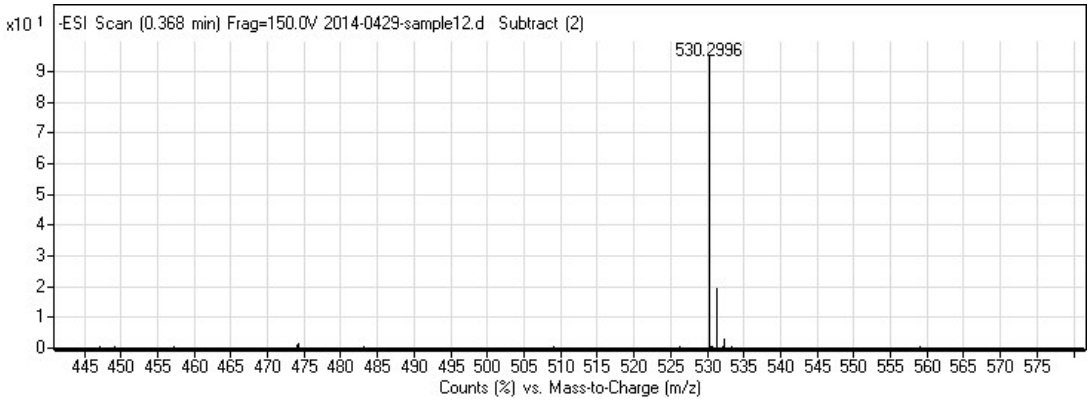
Compound 14



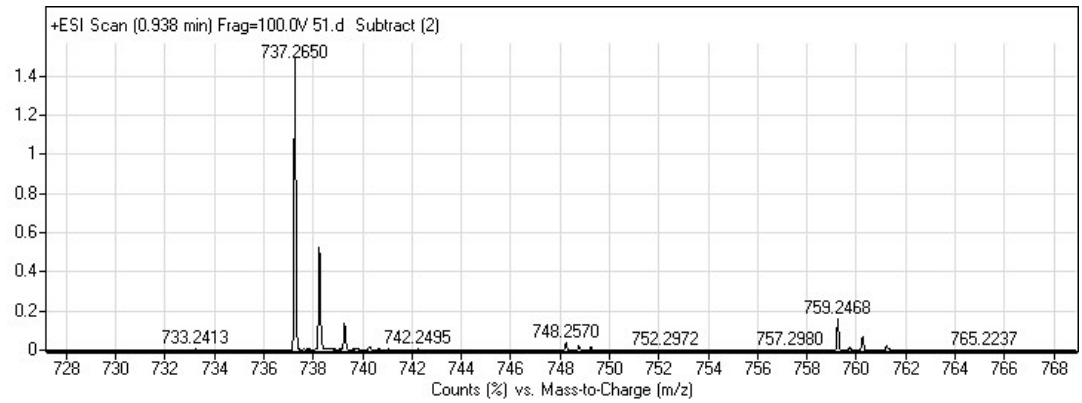
Compound 15



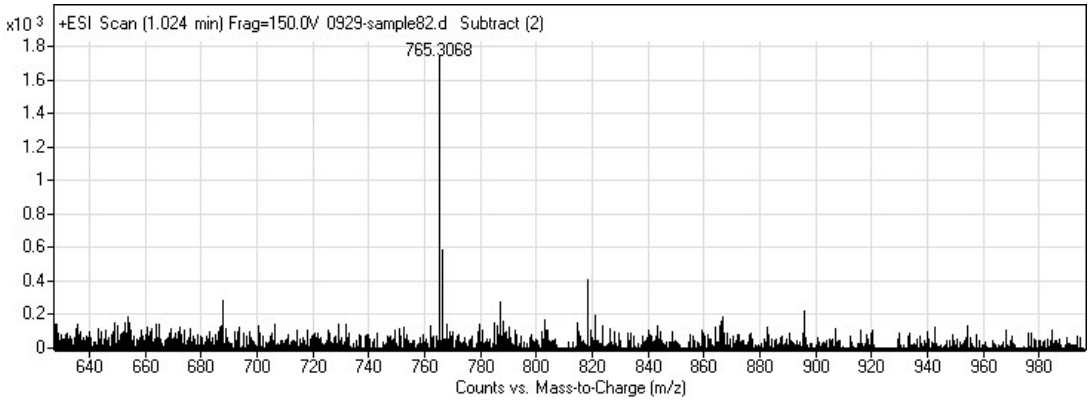
Compound 16



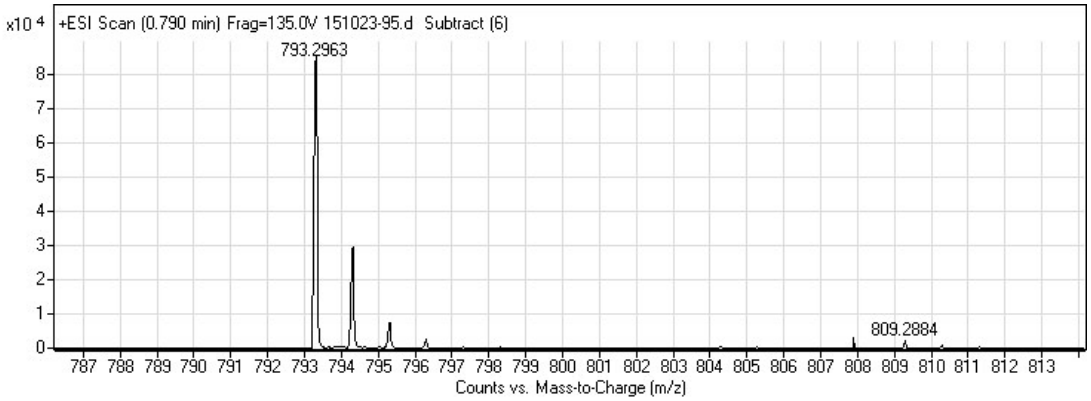
Compound 17



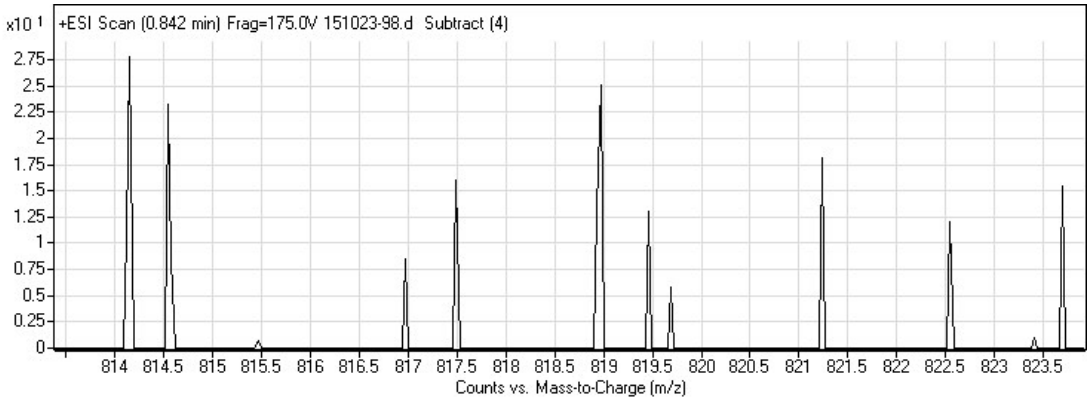
Compound 18



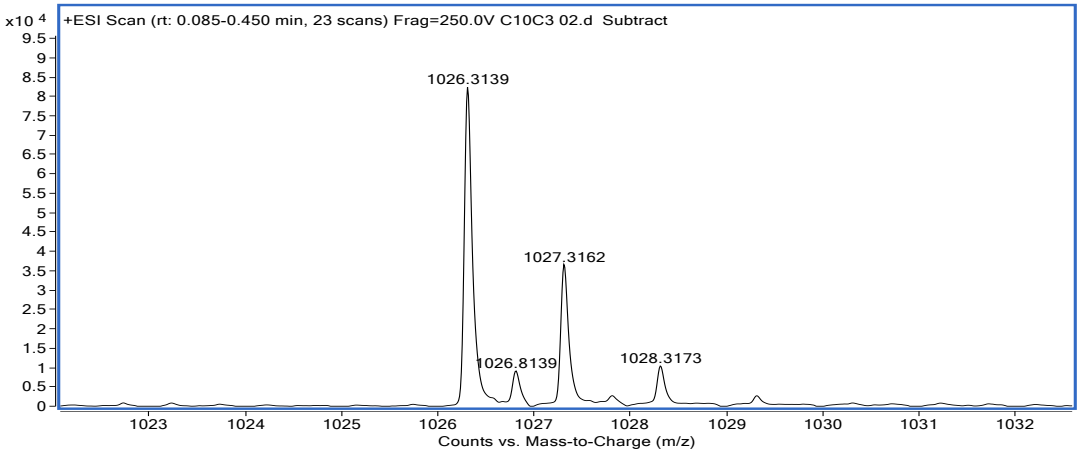
Compound 19



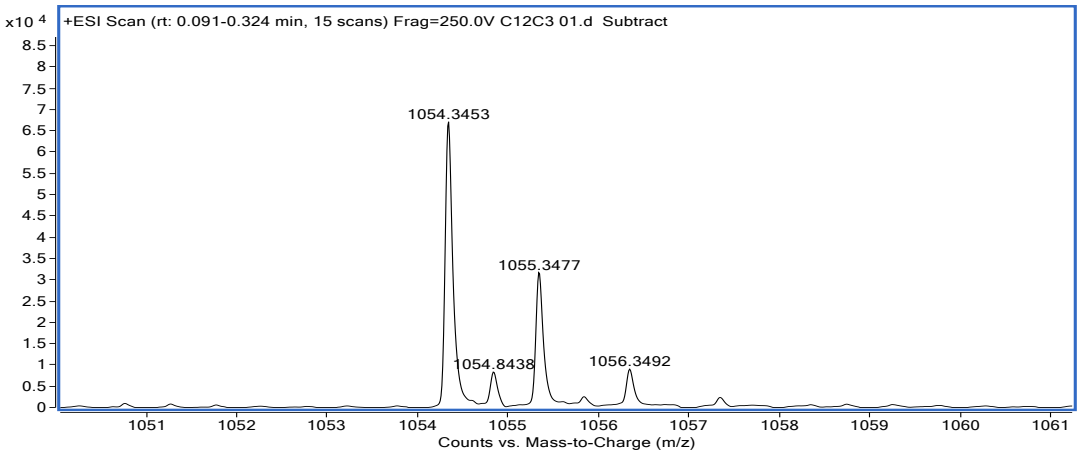
Compound 20



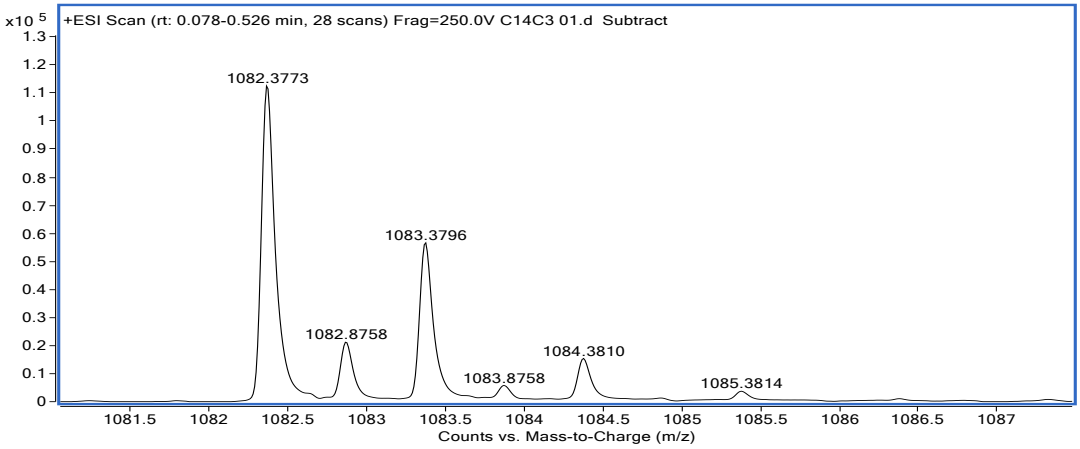
Compound 21



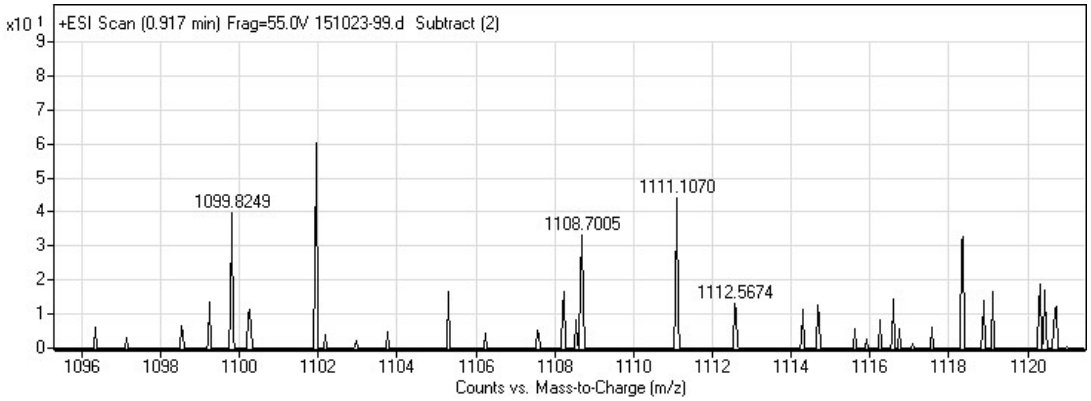
Compound 22



Compound 23



Compound 24


x10 1
+ESI Scan (0.917 min) Frag=55.0V 151023-99.d Subtract (2)
9
8
7
6
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3
2
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1099.8249
1108.7005
1111.1070
1112.5674
1096
1098
1100
1102
1104
1106
1108
1110
1112
1114
1116
1118
1120
Counts vs. Mass-to-Charge (m/z)