Efficient *N*-acylation of sulfonamides using Cesium salt of Wells–Dawson heteropolyacid as catalyst: Synthesis of new *N*-acyl sulfonamides and cyclic imides

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Supporting Information

Table of contents:

Preparation of *N*-acyl sulfonamides (2a-j), (3a-m), (5a-f) and cyclic imides (3n-r)

Under nitrogen atmosphere, a mixture of sulfonamide (1 mmol), acylating agent (2 mmol) and $Cs_5HP_2W_{18}O_{62}$ catalyst (5 mmol %) in water (2 mL), was stirred at room temperature to obtain (2a-j), (3a-m), (5a-f) and under reflux for (3n-r). Reaction was monitored by TLC. After completion of the reaction, the catalyst was removed by filtration. The filtrate was washed by water (10 mL) and extracted with EtOAc (3x15 mL). The combined organic layers were dried over anhydrous Na_2SO_4, then the solvent was evaporated in vacuum, and the crude compound was purified by flash chromatography (Merck silica gel 60 H, CH_2Cl_2/MeOH, 9:1) to afford the corresponding products.

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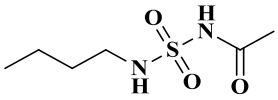
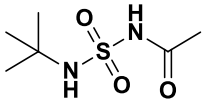
NMR data of *N*-acyl sulfonamide derivatives (2a-j)



2a

***N*-(*N*-butylsulfamoyl)acetamide (2a)**

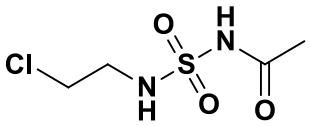
Yield: 91%, mp 131°C, Rf= 0.49(CH_2Cl_2/MeOH, 9/1). **^{1}HNMR** ($CDCl_3$,δppm): 0.92 (t, 3H, ***J***=5.02 Hz, C**H**$_3$), 1.37 (m, 2H, C**H**$_2$), 1.54 (m, 2H, C**H**$_2$), 2.02 (s, 3H, CO-C**H**$_3$), 3.21 (m, 2H, C**H**$_2$-N), 5.15 (t, *J*=6.86 Hz, 1H, N**H**-CO).
^{13}C NMR ($CDCl_3$, δ ppm): 13.5, 19.8, 29.6, 21.7, 30.9, 43.5, 169.8. **Ms** (ESI^+ 70 eVm/s): 151.04 (14%), 212.10 ($[M+NH_4]^+$, 100%).
Elemental anal. (%), calculated: C, 37.10; H, 7.26; N, 14.42; found: C, 37.14; H, 7.24; N, 14.45.



2b

***N*-(*N*-tert-butylsulfamoyl)acetamide (2b)**

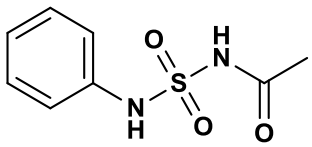
Yield: 92%, mp 135°C, Rf= 0.53(CH_2Cl_2/MeOH, 9/1). **^{1}HNMR** ($CDCl_3$,δppm): 1.43(s, 9H, 3(C**H**$_3$), 2.02 (s, 3H, CO-C**H**$_3$). **^{13}CNMR** (CDl_3, δppm): 19.7, 29.7, 44.6, 171.2, **Ms** (ESI^+ 70 eVm/s): 72.05 (14%), 212.10 ($[M+NH_4]^+$, 100%). **Elemental anal.** (%), calculated: C, 37.10; H, 7.26; N, 14.42; found: C, 37.08; H, 7.27; N, 14.4O.



2c

N-(N-(2-chloroethyl)sulfamoyl)acetamide (2c)

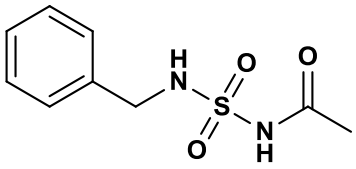
Yield: 79%, mp 131°C, Rf= 0.37(CH_2Cl_2/MeOH, 9/1). **^{1}HNMR** ($CDCl_3$,δppm): 1.97 (s, 3H, CO-C**H**$_3$), 3.35 (m, 2H, N-C**H**$_2$), 3.60 (t, *J*=6.24Hz, 2H, Cl-C**H**$_2$), 7.60(s, 1H, N**H**).8.25(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 21.8, 40.9, 43.2, 171.6. **Ms** (ESI$^+$ 70 eVm/s): 201.19([M+1]$^+$, 100%). **Elemental anal**. (%), calculated: C, 23.94; H, 4.52; Cl, 17.67; N, 13.96; found: C, 23.96; H, 4.50; N, 13.91.



2d

***N*-(*N*-phenylsulfamoyl) acetamide (2d)**

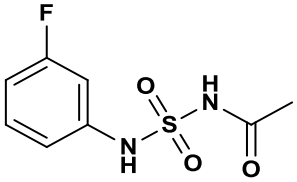
Yield: 92%, mp 140 °C, Rf = 0.28(CH_2Cl_2/MeOH, 9/1), **^{1}H NMR** ($CDCl_3$, δ ppm): 2.18 (s, 3H, CO-C**H**$_3$), 6.79(d, 2H, Ar-**H),** 7.00(t, 1H, Ar-**H),** 7.60(t, 2H, Ar-**H),** 8.00(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 21.8, 119,9, 122.8, 129.8, 140.4, 172.5, **MS** (ESI$^+$ 70 eV m/z): 214.04 ([M]$^+$, 100%). **Elemental anal**. (%), calculated: C, 44.85; H, 4.70; N, 13.08; found: C, 44.81; H, 4.73; N, 13.09**.**



2e

***N*- (*N*-benzylsulfamoyl) acetamide (2e)**

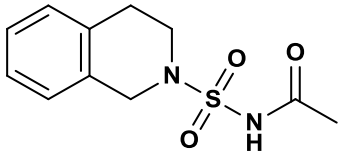
Yield: 85%, mp 125 °C, Rf = 0.50(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 1.92 (s, 3H, CO-C**H**$_3$), 4.32(s, 2H, C**H**$_2$-Ph), 6.05(t, *J*=7.46 Hz, 1H, **NH**), 7.18(m, 3H, **H**-Ar), 7.26(m, 2H, **H**-Ar), 8.10(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 23.1, 44.2, 126.9, 127.2, 128.6, 139.3, 170.0. **MS** (ESI$^+$ 70 eV m/z): 106.05 (4%), 229.09 ([M+1]$^+$, 13%), 246.11 ([M+NH_4]$^+$, 100%). **Elemental anal.** (%), calculated: CC, 47.35; H, 5.30; N, 12.27; found: C, 47.38; H, 5.34; N, 12.23.



2f

***N*- (*N*-3-fluorophenylsulfamoyl) Acetamide (2f)**

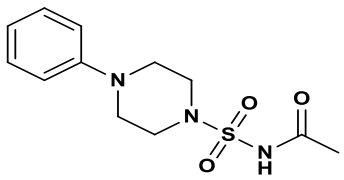
Yield: 76%, mp 147°C, Rf = 0.45 (CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 1.95 (s, 3H, CO-C**H**$_3$), 6.90 (m, 1H, **H**-Ar), 7.06 (m, 2H, **H**-Ar), 7.30 (m, 1H, **H**-Ar), 7.85(s, 1H, N**H**$_{amide}$). **^{13}C NMR** (DMSO-d_6, δ ppm): 22.8, 105.6, 110.1, 114.6, 130.7, 139.4, 163.2, 178.6. **MS** (ESI$^+$ 70 eV m/z): 111.04 (11%), 190.06 (5%), 250.10 ($[M+NH_4]^+$, 100%). **Elemental anal**. (%), calculated: C, 41.37; H, 3.91; N, 12.06; found: C, 41.39; H, 3.89; N, 12.05.



2g

***N*-acetyl-1,2,3,4-tetrahydroisoquinoline-2-sulfonamide (2g)**

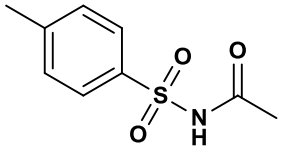
Yield: 88%, mp 140 °C, Rf = 0.58(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 1.95(s, 3H, CO-C**H**$_3$), 2.90(t, 2H, J=5.8Hz, C**H**$_2$-Ar), 3.59(t, 2H, J=5.7Hz, C**H**$_2$N), 4.50(s, 2H, Ar-C**H**$_2$), 7.13-7.25(m, 4H, Ar-**H**), 8.07(s, 1H, N**H**$_{amide}$). **^{13}C NMR** (DMSO-d_6, δ ppm): 22.8, 28.2, 28.6, 43.9, 47.0, 126.0, 126.2, 126.4, 128.6, 131.9, 133.3, 169.5. **MS** (ESI$^+$ 70 eV m/z): 130.99 (12%), 254.99 ($[M+1]^+$, 42%), 272.02 ($[M+NH_4]^+$, 100%). **Elemental anal**. (%), calculated: C, 51.95; H, 5.55; N, 11.02; found: C, 51.98; H, 5.57; N, 11.00.



2h

4-oxo-4-(4-phenylpiperazine-1-sulfonamido)butanoic acid (2h)

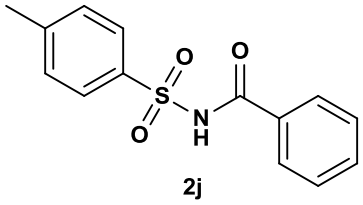
Yield: 85 %, mp 147 °C, Rf = 0.45(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.09 (s, 3H, CO-C**H**$_3$), 2.82(t, *J*= 6.8, 4H, C**H**$_2$-N), 3.25(t, *J*= 8.1, 4H, C**H**$_2$-N), 7.03(m, 3H, Ar-**H**), 7.30(t, 2H, Ar-**H**), 7.95(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 22.8, 44.9, 52.9, 115.3, 121.9 130.2, 150.3, 169.7. **MS** (ESI$^+$ 70 eV m/z): 240.15 (50%), 282.17 ([M-1], 100%), 301.18 ($[M+NH_4]^+$, 43%). **Elemental anal**. (%), calculated: C, 50.87; H, 6.05; N, 14.83; found: C, 50.90; H, 6.02; N, 14.85.



2i

***N*-tosylacetamide (2i)**

Yield: 92 %, mp 169 °C, Rf = 0.33(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.02 (s, 3H, CO-C**H**$_3$), 2.39 (s, 3H, C**H**$_3$-Ph), 7.26 (d, *J*= 8.1Hz, 2H, Ar-**H**), 7.71 (d, *J*= 8.2Hz, 2H, Ar-**H**), 8.08(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 21.6 , 21.8, 128.5, 129.4, 136.8, 137.1, 170.6. **MS** (ESI^+ 70 eV m/z): 214.02($[M+1]^+$, 70%). **Elemental anal**. (%), calculated: C, 50.69; H, 5.20; N, 6.57; found: C, 50.67; H, 5.23; N, 6.52.

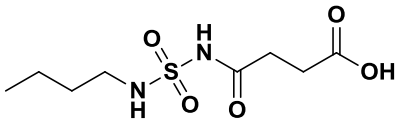


2j

***N*-tosylabenzamide (2j)**

Yield: 92 %, mp 169 °C, Rf = 0.33(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.34 (s, 3H, C**H**$_3$-Ph), 7.29 (t, *J*= 7.9Hz, 1H, Ar-**H**), 7.36 (t, *J*= 7.4Hz, 2H, Ar-**H**), 7.50 (d, *J*= 8.1Hz, 1H, Ar-**H**), 7.75 (d, *J*= 8.2Hz, 2H, Ar-**H**), 7.98 (d, *J*= 8.2Hz, 2H, Ar-**H**), 9.25(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 21.8, 126.7, 127.6, 128.5, 130.2, 134.1, 136.1, 142.2, 143.3, 172.6. **MS** (ESI^+ 70 eV m/z): 276.06($[M+1]^+$, 78%). **Elemental anal**. (%), calculated: C, 61.07; H, 4.76; N, 5.09; found: C, 61.09; H, 4.73; N, 5.10.

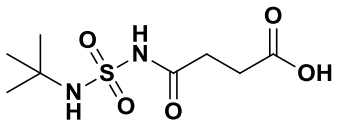
NMR data of *N*-acyl sulfonamide derivatives containing carboxylic acid moiety (3a-m)



3a

4-oxo-4-((*N*-butylsulfamoyl)amino)butanoic acid (3a)

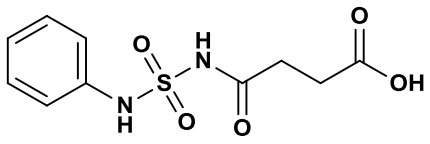
Yield: 90%, mp 131°C, Rf= 0.37(CH_2Cl_2/MeOH, 9/1). **^{1}HNMR** ($CDCl_3$,δppm): 0.92 (t, 3H, ***J***=5.02 Hz, C**H**$_3$), 1.37 (m, 2H, C**H**$_2$), 1.54 (m, 2H, C**H**$_2$), 2.38(t, 2H, *J*= 7.1Hz, C**H**$_2$-CO_{amide}), 2.59(t, 2H, *J*= 7.4Hz, C**H**$_2$-CO_2H), 3.21 (m, 2H, C**H**$_2$-N), 11.02(s, 1H, CO_2**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 13.5, 19.8, 29.6, 29.7, 30.9, 43.5, 173.6, 175.1. **Ms** (ESI^+ 70 eVm/s): 253.13($[M+1]^+$, 100%). **Elemental anal**. (%), calculated: C, 38.09; H, 6.39; N, 11.10; found: C, 38.19; H, 6.35; N, 11.18.



3b

4-oxo-4-((*N*-tert-butylsulfamoyl)amino)butanoic acid (3b)

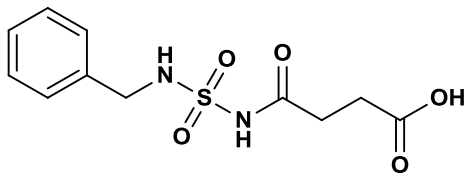
Yield: 92%, mp 129°C, Rf= 0.33(CH_2Cl_2/MeOH, 9/1). **^{1}HNMR** ($CDCl_3$,δppm): 1.43(s, 9H, 3(C**H**$_3$), 2.38(t, 2H, *J*= 7.1Hz, C**H**$_2$-CO_{amide}), 2.98(t, 2H, *J*= 7.4Hz, C**H**$_2$-CO_2H), 7.74-7.79 (m, 2H, **NH**), 11.10(s, 1H, CO_2**H**). **^{13}CNMR** (CDl_3, δppm): 29.4, 29.7, 44.6, 172.6, 173.9. **Ms** (ESI^+ 70 eVm/s): 101.94(3%), 136.93(7%), 207.02(14%), 253.13($[M+1]^+$, 100%). **Elemental anal.** (%), calculated: C, 38.09; H, 6.35; N, 11.11; found: C, 38.13; H, 6.39; N, 11.08.



3d

4-oxo-4-((*N*-phenylsulfamoyl)amino) butanoic acid (3d)

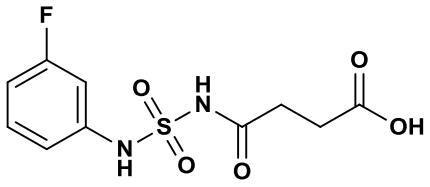
Yield: 89%, mp 140 °C, Rf = 0.28(CH_2Cl_2/MeOH, 9/1), **^{1}H NMR** ($CDCl_3$, δ ppm): 2.61(t, *J*= 7.3Hz, 2H, C**H**$_2$-CO_{amide}), 2.89(t, *J*= 7.7, 2H, C**H**$_2$-CO_2H), 4.05(s, 1H, N**H**), 6.79(m, 5H, Ar-**H),** 7.74(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 29.8, 29.9, 121.2, 124.3, 126.1, 140.4, 173.5, 175.8. **MS** (ESI^+ 70 eV m/z): 272 ($[M]^+$, 100%). **Elemental anal**. (%), calculated: C, 44.12; H, 4.41; N, 10.29; found: C, 44.09; H, 4.46; N, 10.32**.**



3e

4-oxo-4-((*N*-benzylsulfamoyl)amino)butanoic acid (3e)

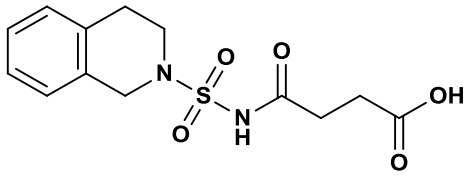
Yield: 85%, mp 135 °C, Rf = 0.31(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.54(t, 2H, *J*= 7.2Hz, C**H**$_2$-CO_{amide}); 2.75(t, 2H, *J*=7.4, C**H**$_2$-CO $_2$H), 4.10(s, 2H, C**H**$_2$-Ph), 7.12(m, 2H, **H**-Ar), 7.18(m, 3H, **H**-Ar),8.10(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 29.7, 30.8, 40.5, 122.6, 122.9, 123.8, 133.5, 173.2, 175.2. **MS** (ESI^+ 70 eV m/z): 105.98 (8%), 186.01 (5%), 287.01 ($[M+1]^+$, 15%), 304.02 ($[M+NH_4]^+$, 94%). **Elemental anal.** (%), calculated: C, 46.15; H, 4.89; N, 9.79; found: C, 46.12; H, 4.92; N, 9.81.



3f

4-oxo-4-((*N*-3-fluorophenylsulfamoyl)amino) butanoic acid (3f)

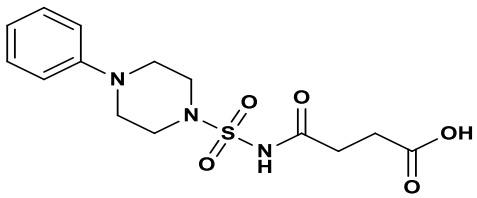
Yield: 77%, mp 148°C, Rf = 0.28 (CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.62 (t, 2H, *J*= 7.2Hz, C**H**$_2$-NH$_{amide}$), 2.90(t, 2H, *J*= 7.5Hz, C**H**$_2$CO$_2$H), 6.90 (m, 2H, C**H**ar), 6.96 (m, 1H, C**H**ar), 7.26 (m, 1H, C**H**ar), 7.89(s, 1H, N**H**$_{amide}$), 10.90(s, 1H, COO**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 29.8, 29.9, 105.6, 110.1, 114.6, 130.7, 139.4, 163.2, 173.3, 175.6. **MS** (ESI$^+$ 70 eV m/z): 111.04 (12%), 1906.0 (6%), 290.10([M+1]$^+$, 3%), 308.25 ([M+NH$_4$]$^+$, 100%). **Elemental anal**. (%), calculated: C, 41.38; H, 3.79; N, 9.65; found: C, 41.39; H, 3.76; N, 9.69.



3g

4-oxo-4-(1,2,3,4-tetrahydroisoquinoline-2 -sulfonamido)]butanoic acid (3g)

Yield: 94%, mp 140 °C, Rf = 0.30(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.56(t, 2H, *J*= 7.2Hz, C**H**$_2$-CO amide), 2.75(t, 2H, *J*= 7.5,C**H**$_2$-CO$_2$H), 2.88(t, 2H, J=5.8Hz, C**H**$_2$-Ar), 3.60(t, 2H, J=5.7Hz, C**H**$_2$N), 4.51(s, 2H, Ar-C**H**$_2$N), 7.05(m, 1H, Ar-**H**), 7.09(m, 1H, Ar-**H**), 7.17(m, 2H, Ar-**H**), 8.07(s, 1H, N**H**$_{amide}$), . **^{13}C NMR** ($CDCl_3$, δ ppm): 28.5, 28.6, 29.9, 44.6, 47.5, 126.2, 126.6, 127.2, 128.9, 131.1, 133.1, 172.5, 175.1. **MS** (ESI$^+$ 70 eV m/z): 131.93 (17%), 210.99 (4%), 257.19(4%), 313.07([M+1]$^+$, 8%), 330.01 ([M+NH$_4$]$^+$, 100%). **Elemental anal**. (%), calculated: C, 50.00; H, 5.13; N, 8.97; found: C, 50.07; H, 5.10; N, 8.99.

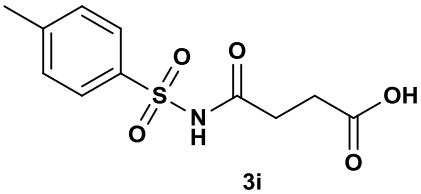


3h

4-oxo-4-(4-phenylpiperazine-1- sulfonamido)butanoic acid (3h)

Yield: 91 %, mp 147 °C, Rf = 0.35(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.65(t, *J*= 7.3Hz, 2H, C**H**$_2$-CO$_{amide}$), 2.82(t, *J*= 7.6, 2H, C**H**$_2$-CO$_2$H), 3.22(t, *J*= 6.8, 4H, C**H**$_2$-N), 3.58(t, *J*= 8.1, 4H, C**H**$_2$-N), 6.87(m, 3H, Ar-**H**), 7.29(m, 2H, Ar-**H**), 7.59(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 29.6, 31.1, 46.5, 49.1, 117.1, 121.1 129.5, 150.6, 172.6, 175.1. **MS** (ESI$^+$ 70 eV m/z): 161.20 (6%), 216.00 (6%), 241.99 (7%), 342.10 ([M+H]$^+$, 100%). **Elemental anal**. (%), calculated: C, 49.26; H, 5.57;

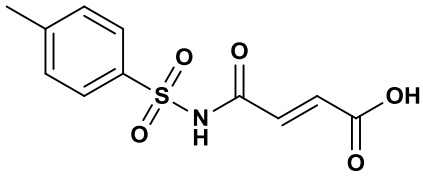
N, 12.32; found: C, 49.30; H, 5.60; N, 12.37.



3i

4-oxo- 4-(4-methylphenylsulfonamido)- butanoic acid (3i)

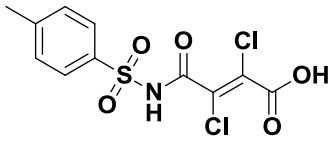
Yield: 90 %, mp 169 °C, Rf = 0.33(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.39 (s, 3H, C**H**$_3$-Ph), 2.70(t, *J*= 7.1Hz, 2H, C**H**$_2$-CO_{amide}), 2.85(t, *J*= 7.5, 2H, C**H**$_2$-CO_2H), 7.26 (d, *J*= 8.1Hz, 2H, C**H**$_{arom}$), 7.71 (d, *J*= 8.2Hz, 2H, C**H**$_{arom}$), 8.08(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 21.6 , 29.5, 29.9, 127.1, 130.0, 136.9, 143.9, 172.6, 173.9. **MS** (ESI$^+$ 70 eV m/z): 90.97 (3%), 155.04 (100%), 170.12(6%), 272.02([M+1]$^+$, 70%). **Elemental anal**. (%), calculated: C, 48.70; H, 4.79; N, 5.16; found: C, 48.72; H, 4.82; N, 5.19.



3j

(E)-4-oxo- 4-(4-methylphenylsulfonamido)- but-2-enoic acid (3j)

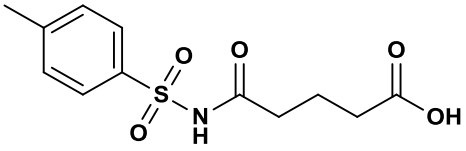
Yield: 79 %, mp 166 °C, Rf = 0.35(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.37 (s, 3H, C**H**$_3$), 6.20 (d, 2H, C**H**= C**H**), 7.26(d, 2H, Ar-**H**), 7.76(d, 2H, Ar-**H**), 9.40(s, 1H, N**H**), 12.23 (s, 1H, C**OOH**). **^{13}C NMR** ($CDCl_3$, δ ppm): 23.2, 127.3, 129.5, 135.8, 136.5, 141.3, 166.4. **Elemental anal**. (%), calculated: C, 49.06; H, 4.12; N, 5.20; found: C, 49.17; H, 4.18; N, 5.17.



3k

(E)-2,3-dichloro-4-oxo- 4-(4-methylphenylsulfonamido)- but-2-enoic acid (3k)

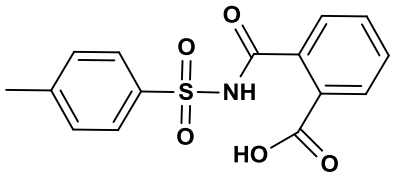
Yield: 75 %, mp 169 °C, Rf = 0.29(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.46 (s, 3H, C**H**$_3$), 7.35(d, 2H, Ar-**H**), 7.85(d, 2H, Ar-**H**), 8.03(s, 1H, N**H**), 11.70(s, 1H, COO**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 24.3, 127.2, 129.6, 136.4, 137.5, 141.3, 167.1. **Elemental anal.** (%), calculated: C, 39.07; H, 2.68; N, 4.14; found: C, 39.13; H, 2.69; N, 4.19.



3l

5-oxo- 5-(4-methylphenylsulfonamido)-pentanoic acid (3l)

Yield: 80 %, mp 170 °C, Rf = 0.31(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 1.25 (m, 2H, CH_2-C**H**$_2$-CH_2, 2.85(t, 2H, *J*= 7.6, 2H, C**H**$_2$-CO_{amide}), 2.45(t, *J*= 7.5, 2H, C**H**$_2$-CO_2H), 2.46 (s, 3H, C**H**$_3$), 7.28 (d, *J*= 8.1Hz, 2H, Ar-**H**), 7.67 (d, *J*= 8.2Hz, 2H, Ar-**H**), 7.95(s, 1H, N**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 20.9, 21.6 , 32.5, 34.9, 127.1, 129.2, 137.3, 142.9, 172.7, 173.7. **Elemental anal**. (%), calculated: C, 50.52; H, 5.30; N, 4.91; found: C, 50.56; H, 5.280; N, 4.93.

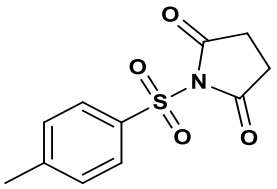


3m

2-(tosylcarbamoyl)benzoic acid (3m)

Yield: 55 %, mp 172 °C, Rf = 0.27(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.45 (s, 3H, C**H**$_3$-Ph), 7.37 (d, *J*= 8.1Hz, 2H, Ar-**H**), 7.83 (d, *J*= 8.2Hz, 2H, Ar-**H**), 7.91 (d, 2H, Ar-**H**), 8.09 (d, 2H, Ar-**H**), 8.26(s, 1H, N**H**), 11.58(s, 1H, COO**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 21.6 ,123.8, 127.7, 128.3, 130.0, 131.4, 132.1, 134.5, 137.2, 137.9, 172.9, 173.7. **MS** (ESI$^+$ 70 eV m/z): 320.17 ([M+1]$^+$, 100. **Elemental anal**. (%), calculated: C, 56.42; H, 4.10; N, 4.39; found: C, 56.49; H, 4.18; N, 4.29.

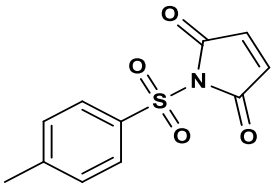
NMR data of cyclic imides (3n-r)



3n

1-tosylpyrrolidine-2,5-dione (3n)

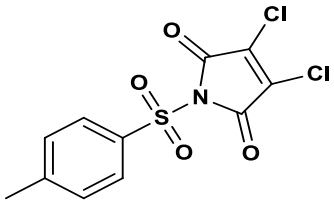
Yield: 82%, mp 126 °C, Rf = 0.88(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.45 (s, 3H, C**H**$_3$-Ph), 2.65(s, 4H, (C**H**$_2$)-CO_{amide}), 7.40 (d, *J*= 8.1Hz, 2H, Ar-**H**), 7.75 (d, *J*= 8.2Hz, 2H, Ar-**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 21.5, 26.9, 128.5, 129.3, 137.6, 137.9, 175.8. **MS** (ESI$^+$ 70 eV m/z): 254.09 ([M+1]$^+$, 40%), 271.02 ([M+NH_4]$^+$, 100%). **Elemental anal**. (%), calculated: C, 52.16; H, 4.38; N, 5.53; found: C, 52.19; H, 4.29; N, 5.59**.**



3o

1-tosyl-1H-pyrrole-2,5-dione (3o)

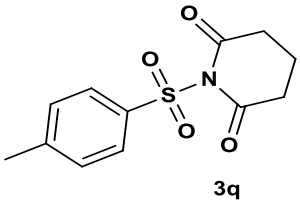
Yield: 78%, mp 135 °C, Rf = 0.84(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.35 (s, 3H, C**H**$_3$-Ph), 6.65(s, 2H, C**H**=C**H**), 7.30 (d, *J*= 8.1Hz, 2H, Ar-**H**), 7.85 (d, *J*= 8.2Hz, 2H, Ar-**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 21.5, 128.5, 129.3, 134.3, 136.3, 137.9, 175.8. **MS** (ESI$^+$ 70 eV m/z): 252.09 ([M+1]$^+$, 40%), 269.02 ([M+NH_4]$^+$, 100%). **Elemental anal**. (%), calculated: C, 52.58; H, 3.61; N, 5.57; found: C, 52.59; H, 3.58; N, 5.59.



3p

3,4-dichloro-1-tosyl-1H-pyrrole-2,5-dione (3p)

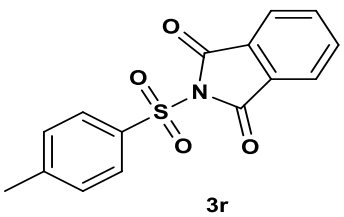
Yield: 73%, mp 136 °C, Rf = 0.80(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.45 (s, 3H, C**H**$_3$-Ph), 7.35 (d, *J*= 8.1Hz, 2H, Ar-**H**), 7.80 (d, *J*= 8.2Hz, 2H, Ar-**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 21.5, 128.5, 129.3, 136.8, 137.6, 137.9, 175.8. **MS** (ESI$^+$ 70 eV m/z): 321.09 ([M+1]$^+$, 40%), 338.02 ([M+NH_4]$^+$, 100%). **Elemental anal**. (%), calculated: C, 41.27; H, 2.20; N, 4.38; found: C, 41.30; H, 2.22; N, 4.35.



3q

1-tosylpiperidine-2,6-dione (3q)

Yield: 77%, mp 129 °C, Rf = 0.86(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.26 (m, 2H, C**H**$_2$-(CH_2)$_2$, 2.35(t, 2H, *J*= 7.6, 2H, C**H**$_2$-CO), 2.35 (s, 3H, C**H**$_3$), 7.40 (d, *J*= 8.1Hz, 2H, Ar-**H**), 7.77 (d, *J*= 8.2Hz, 2H, Ar-**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 19.2, 21.6 , 32.5, 127.1, 129.2, 133.5, 136.7, 172.7, 173.7. **MS** (ESI$^+$ 70 eV m/z): 268.04 ([M+1]$^+$, 100%). **Elemental anal**. (%), calculated: C, 53.92; H, 4.90; N, 5.24; found: C, 53.90; H, 4.93; N, 5.21.

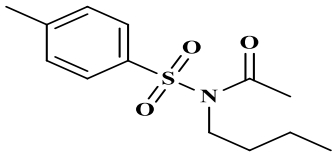


3r

2-tosylisoindoline-1,3-dione (3r)

Yield: 60%, mp 138 °C, Rf = 0.78(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.45 (s, 3H, C**H**$_3$), 7.35 (d, *J*= 8.1Hz, 2H, Ar-**H**), 7.80 (d, *J*= 8.2Hz, 2H, Ar-**H**), 7.90 (d, *J*= 8.5Hz, 2H, Ar-**H**), 8.20 (d, *J*= 8.7Hz, 2H, Ar-**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 21.5, 124.6, 127.5, 130.0, 131.1, 135.2, 145.1, 164.6. **MS** (ESI^+ 70 eV m/z): 302.12 ($[M+1]^+$, 38%), 319.02 ($[M+NH_4]^+$, 100%). **Elemental anal**. (%), calculated: C, 59.79; H, 3.68; N, 4.65; found: C, 59.76; H, 3.70; N, 4.64**.**

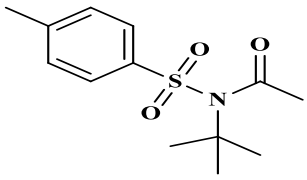
NMR data of *N*-acyl sulfonamide derivatives (5a-f)



5a

***N*-butyl-*N*-tosylacetamide (5a)**

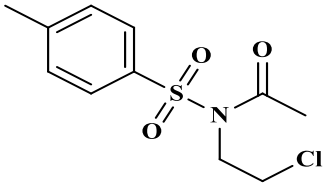
Yield: 85 %, mp: 140 °C, Rf = 0.48(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 0.76 (t, *J*=7.3Hz, 3H, CH_2-C**H**$_3$), 1.25 (m, 2H, C**H**$_2$-CH_3), 1.35 (m, 2H, C**H**$_2$-CH_2), 2.20 (s, 3H, CO-C**H**$_3$), 2.37 (s, 3H, Ph-C**H**$_3$), 2.80 (t, *J*=7.5Hz, 2H, C**H**$_2$-N), 7.28 (d, *J*=7.8Hz, 2H, Ar-**H**), 7.73 (d, *J*=7.8 Hz, 2H, Ar-**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 12.5, 18.6, 19.9. 20.4, 30.5, 42.3, 126.0, 128.8, 136.1, 142.2, 169.8. **MS** (ESI^+ 70 eV m/z): 73.98 (100%), 91.01 (10%), 190.00 (12%), 287.11 ($[M+NH_4]^+$, 55%). **Elemental anal**. (%), calculated: C, 57.97; H, 7.11; N, 5.20; found: C, 57.95; H, 7.14; N, 5.18.



5b

***N*-*tert*-butyl-*N*-tosylacetamide (5b)**

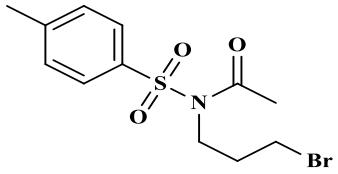
Yield: 88 %, mp 136 °C, Rf = 0.47(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 1.45 (s, 9H, 3(C**H**$_3$)), 2.10 (s, 3H, C**H**$_3$), 2.45 (s, 3H, C**H**$_3$), 7.25(d, 2H, Ar-**H**), 7.75(d, 2H, Ar-**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 19.8, 20.5, 29.1, 40.2, 126.5, 129.0, 136.1, 142.4, 169.8. **MS (ESI^+ 70 eV m/z):** 155.04 (100%), 227.16 (33%), 269.98 ($[M+1]^+$, 4%), 287.19 ($[M+NH_4]^+$, 10%). **Elemental anal**. (%), calculated: C, 57.97; H, 7.11; N, 5.20; found: C, 57.99; H, 7.10; N, 5.22.



5c

***N*-(2-chloroéthyl)-N-tosylacetamide (5c)**

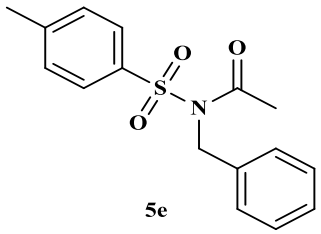
Yield: 81%, mp 146 °C, Rf = 0.42(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.37 (s, 3H, CO-C**H**$_3$), 2.47 (s, 3H, Ph-C**H**$_3$), 3.50 (t, 6.2Hz, 2H, N-C**H**$_2$), 3.70 (t, *J*=5.54 Hz, 2H, Cl-C**H**$_2$), 7.26 (d, *J*=8.1Hz, 2H, Ar-**H**), 7.71 (d, *J*=8.1Hz, 2H, Ar-**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 20.9, 21.6, 43.9, 44.9, 127.1, 130.0, 136.9, 143.9, 169.9. **MS (ESI$^+$ 70 eV m/z):** 91.08 (55%), 155.04 (100%), 276.08 ([M+1]$^+$, 54%). **Elemental anal**. (%), calculated: C, 47.91; H, 5.12; N, 5.08; found: C, 47.94; H, 5.10; N, 5.11.



5d

***N*-(3-bromopropyl)-*N*-tosylacetamide (5d)**

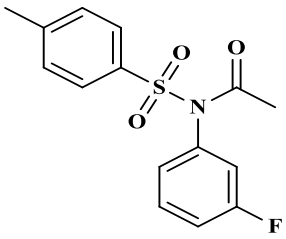
Yield: 80 %, mp 149°C, Rf = 0.40(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.02 (m, 2H, C**H**$_2$-CH_2), 2.24 (s, 3H, CO-C**H**$_3$), 2.38 (s, 3H, Ph-C**H**$_3$), 3.15 (m, 2H, C**H**$_2$-CH_2), 3.40 (t, *J*=6.24Hz, 2H, CH_2-C**H**$_3$), 7.28 (d, *J*= 8.1Hz, 2H, Ar-**H**), 7.72 (d, *J*= 8.2Hz, 2H, Ar-**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 19.8, 20.4, 29.1, 31.2, 40.3, 125.9, 128.7, 135.5, 142.**7**, 170.1. **MS (ESI$^+$ 70 eV m/z):** 335.04 ([M+1]$^+$, 78%). **Elemental anal.** (%), calculated: C, 43.12; H, 4.83; N, 4.19; found: C, 43.09; H, 4.84; N, 4.17.



5e

***N*-benzyl-*N*-tosylacetamide (5e)**

Yield: 79 %, mp 146 °C, Rf = 0.41(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 2.42 (s, 3H, CO-C**H**$_3$), 2.49 (s, 3H, C**H**$_3$-Ph), 4.10 (s, 2H, C**H**$_2$), 7.20 (d, *J*= 8.1Hz, 2H, Ar-**H**), 7.29 (d, *J*= 8.2Hz, 2H, Ar-**H**), 7.40 (m, 1H, Ar-**H**), 7.75 (m, 2H, Ar-**H**), 7.92 (d, *J*=7.5Hz, 2H, Ar-**H**). **^{13}C NMR** ($CDCl_3$, δ ppm): 19.9, 20.6, 44.3, 125.8, 126.2, 127.9, 128.3, 129.3, 137.6, 141.6, 142.3, 169.9. **MS** (ESI$^+$ 70 eV m/z): 43.98 (100%), 91.01 (14%), 304.02 ([M+H]$^+$, 12%), 321.11 ([M+NH_4]$^+$, 55%). **Elemental anal.** (%), calculated: C, 63.34; H, 5.65; N, 4.62; found: C, 63.37; H, 5.64; N, 4.60.



5f

***N*-(3-fluorophenyl)-*N*-tosylacetamide (5f)**

Yield: 75 %, mp 150 °C, Rf = 0.42(CH_2Cl_2/MeOH, 9/1). **^{1}H NMR** ($CDCl_3$, δ ppm): 1.98 (s, 3H, CO-C**H**$_3$), 2.35 (s, 3H, C**H**$_3$-Ph), 6.52 (d, *J*= 8.1Hz, 1H, Ar-**H**), 6.95(m, 2H, Ar-**H**), 7.10 (m, 1H, Ar-**H**), 7.15 (d, *J*= 8.2Hz, 2H, Ar-**H**), 7.80 (d, 2H, Ar-**H**), **^{13}C NMR** ($CDCl_3$, δ ppm): 19.7, 20.6, 104.9, 110.8, 116.5, 127.2, 128.3, 131.9, 137.3, 138.2, 139.7, 162.3, 171.4. **MS** (ESI^+ 70 eV m/z): 308.08 ($[M+H]^+$, 100%). **Elemental anal.** (%), calculated: C, 58.62; H, 4.59; N, 4.56; found: C, 58.60; H, 4.62; N, 4.52.

Selected spectroscopic data for new compounds (2a-j)

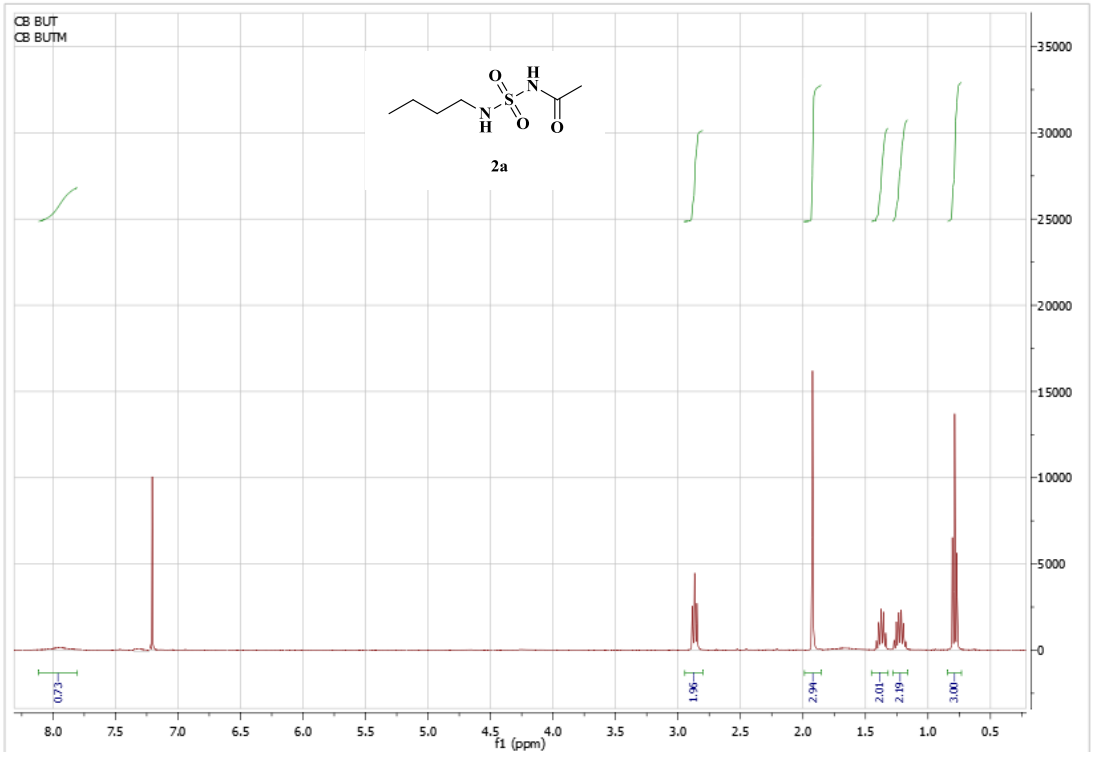


Figure S1: ^{1}H NMR of 2a

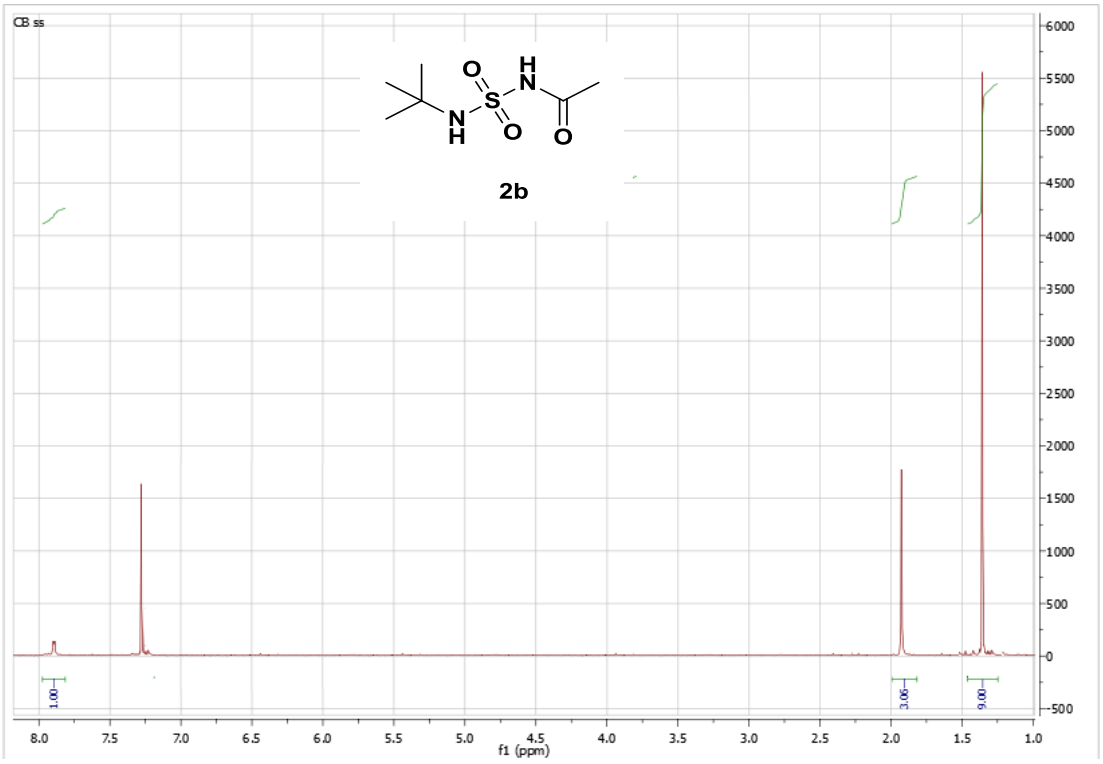


Figure S2: ^{1}H NMR of 2b

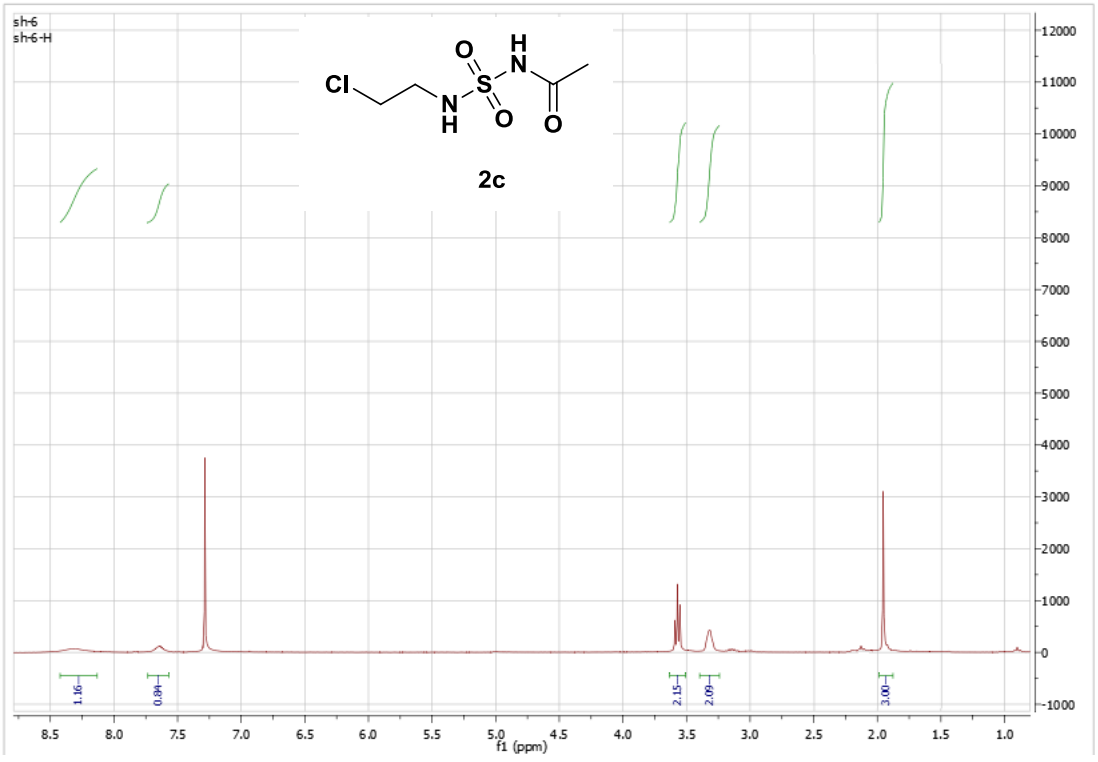


Figure S3: ^{1}H NMR of 2c

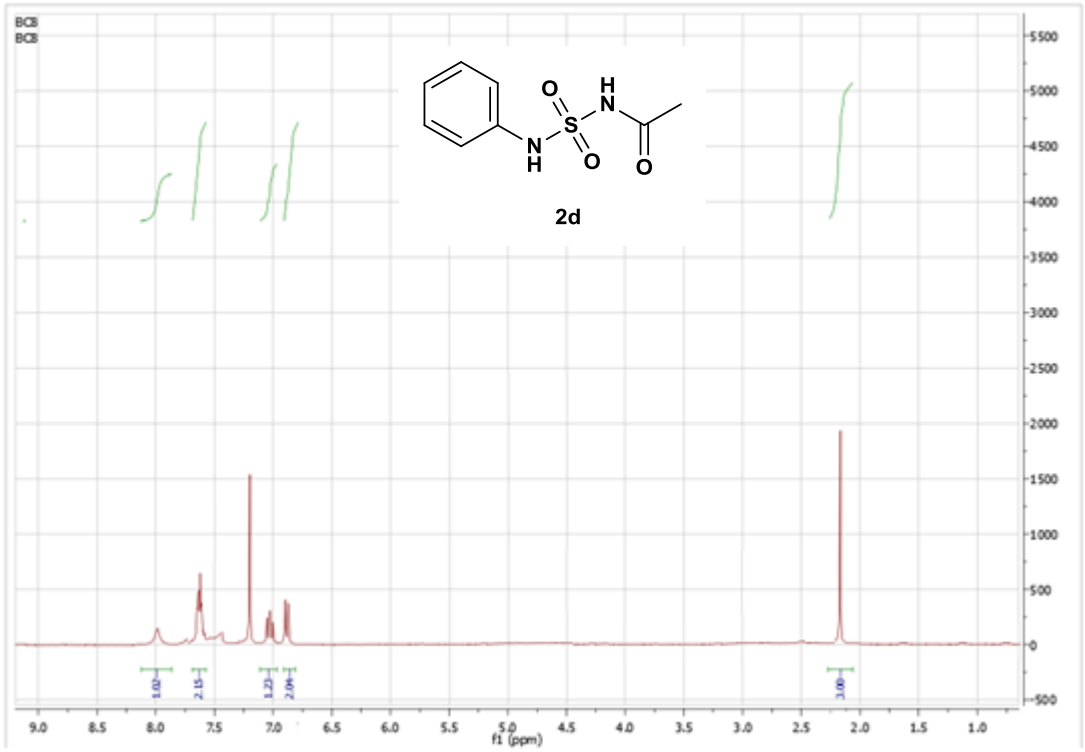


Figure S4: ^{1}H NMR of 2d

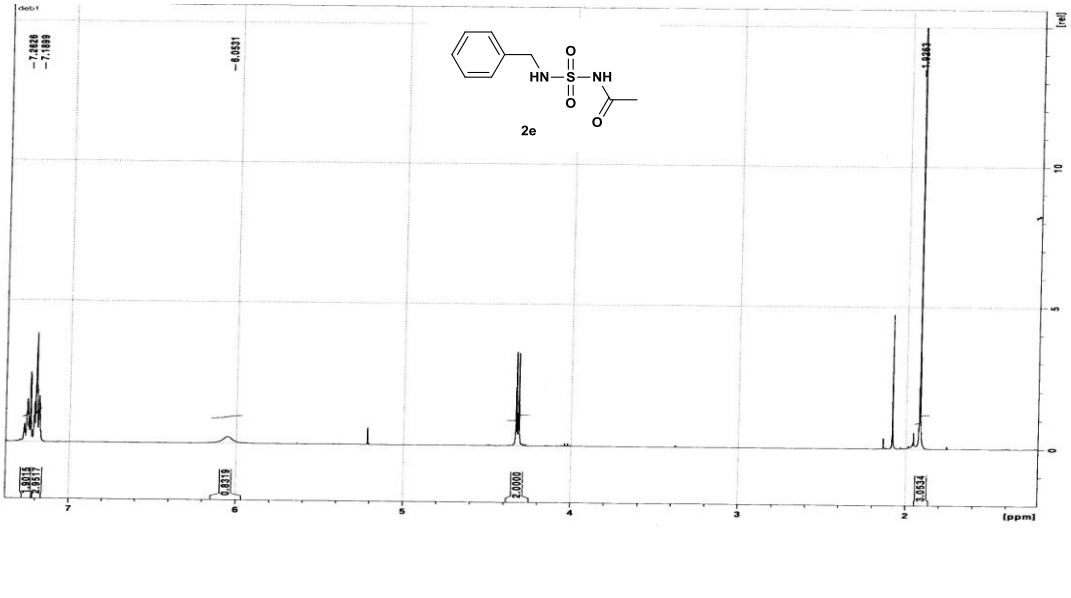


Figure S5: ^{1}H NMR of 2e

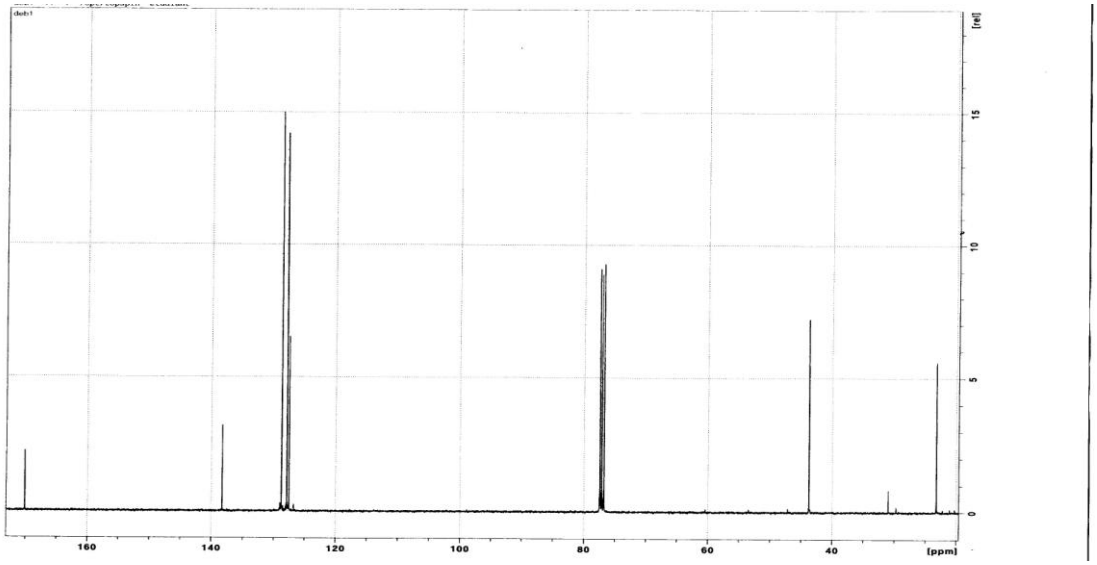


Figure S6: ^{13}C NMR of 2e

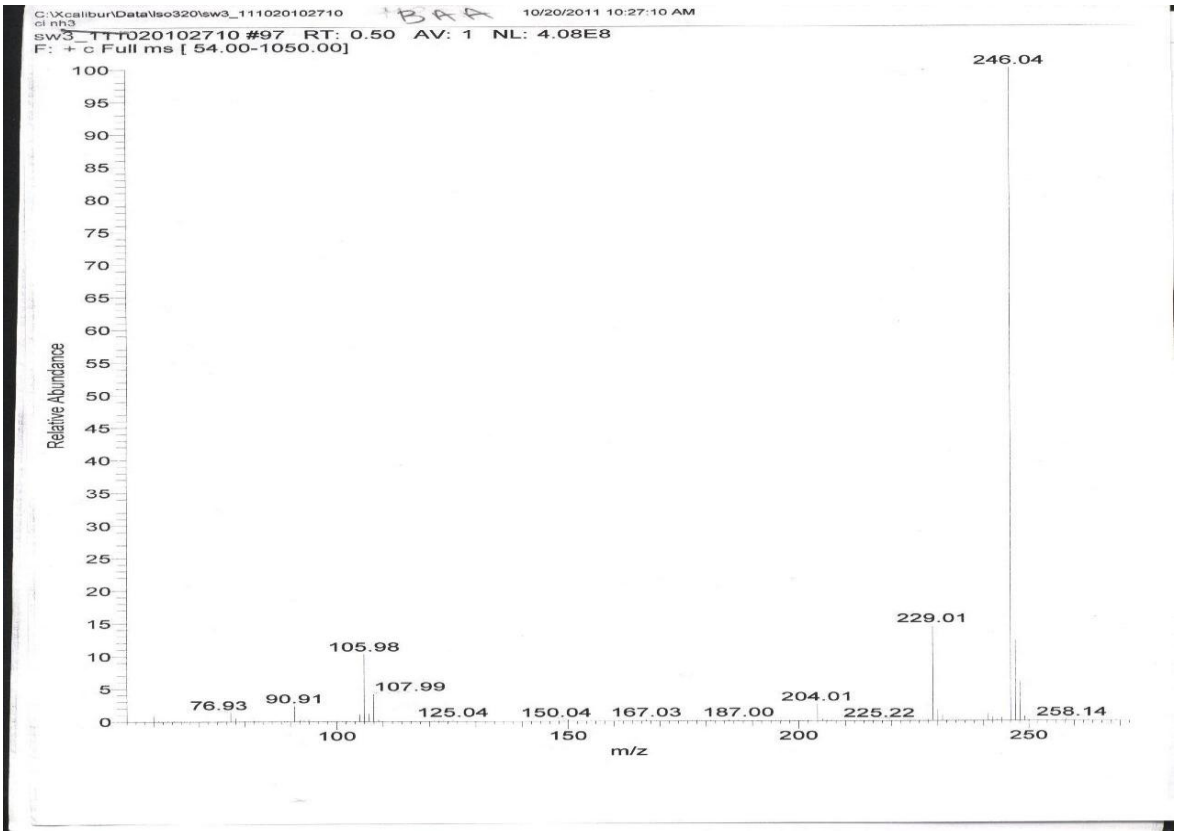


Figure S7: MS of 2e

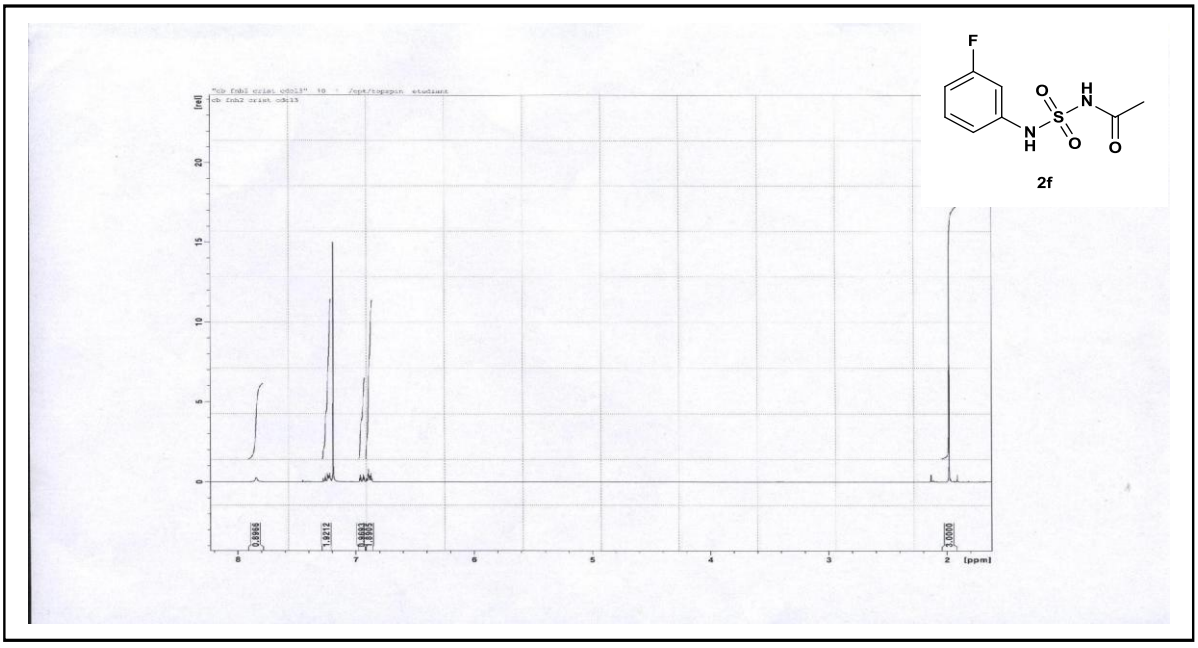


Figure S8: ^{1}H NMR of 2f

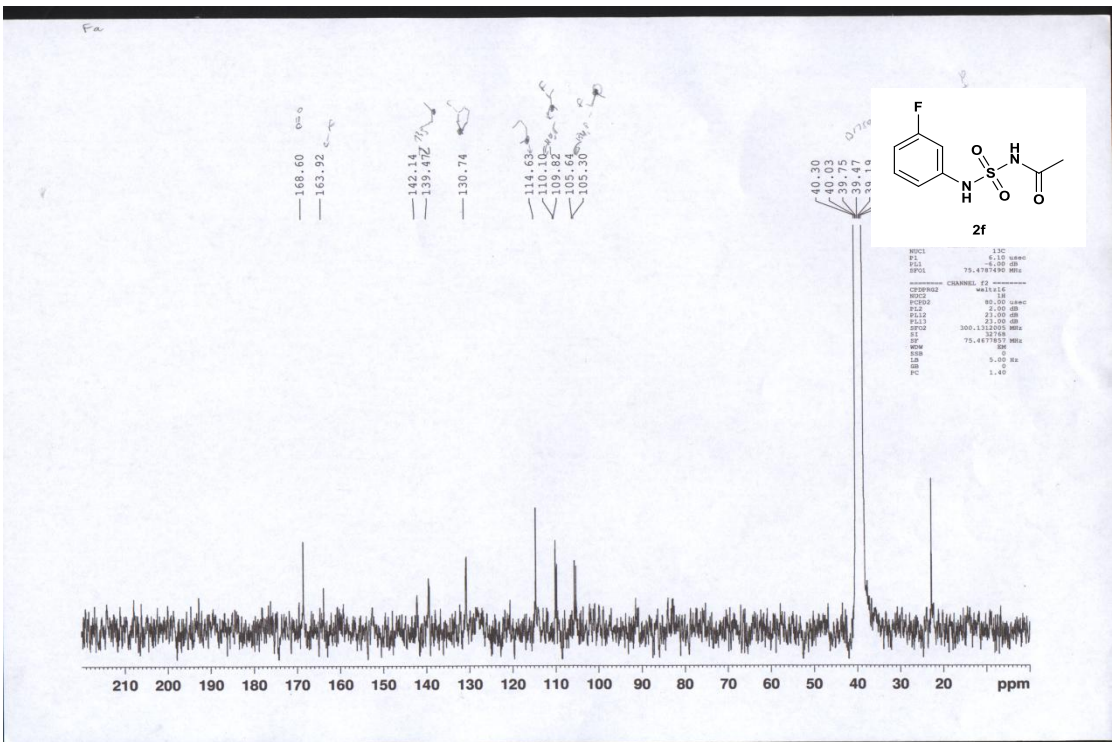


Figure S9: ^{13}C NMR of 2f

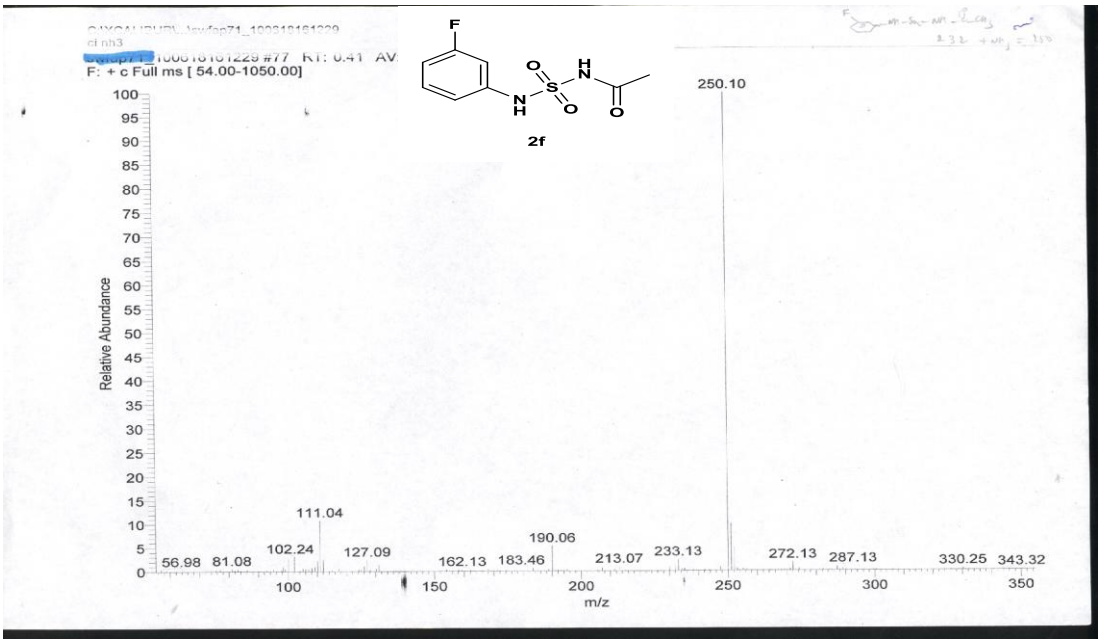


Figure S10: MS of 2f

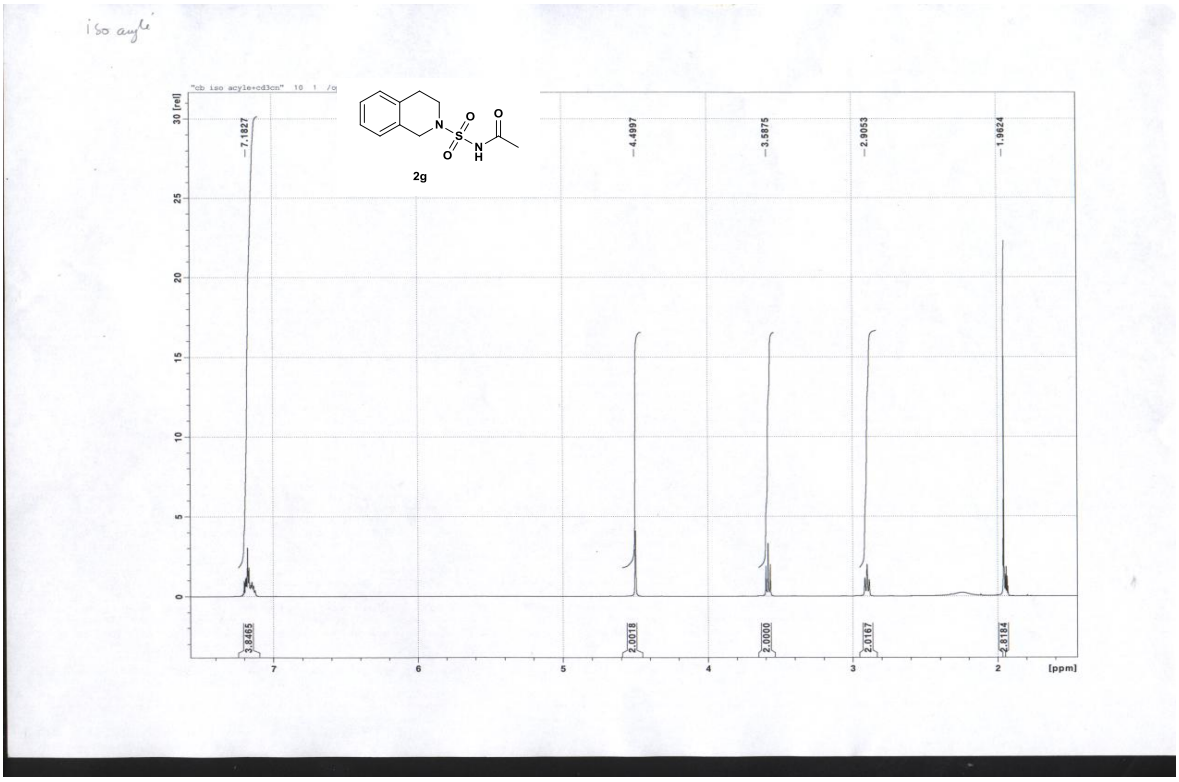


Figure S11: ^{1}H NMR of 2g

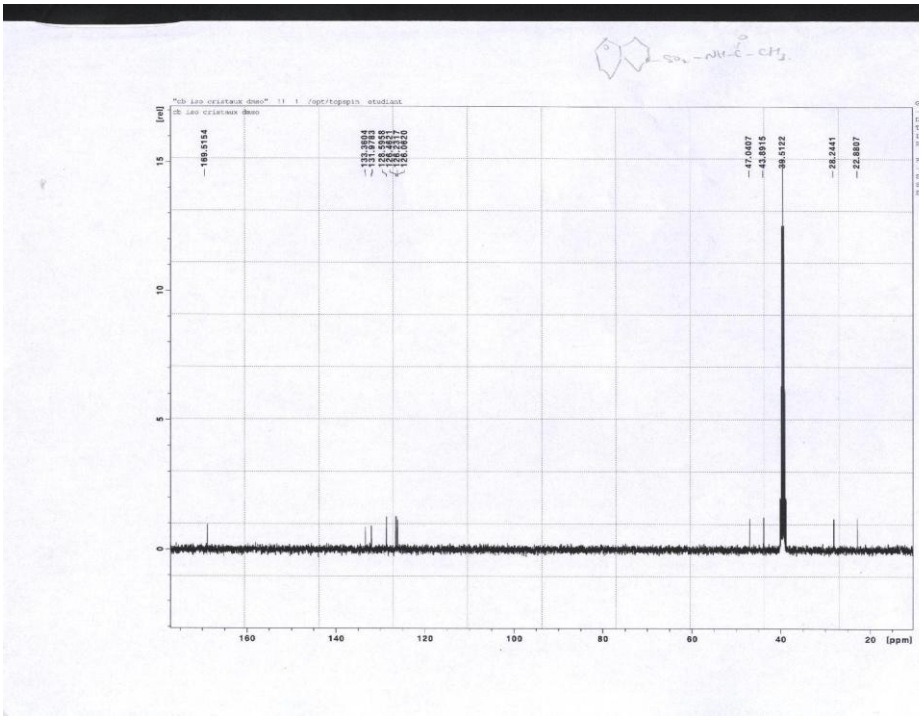


Figure S12: ^{13}C NMR of 2g

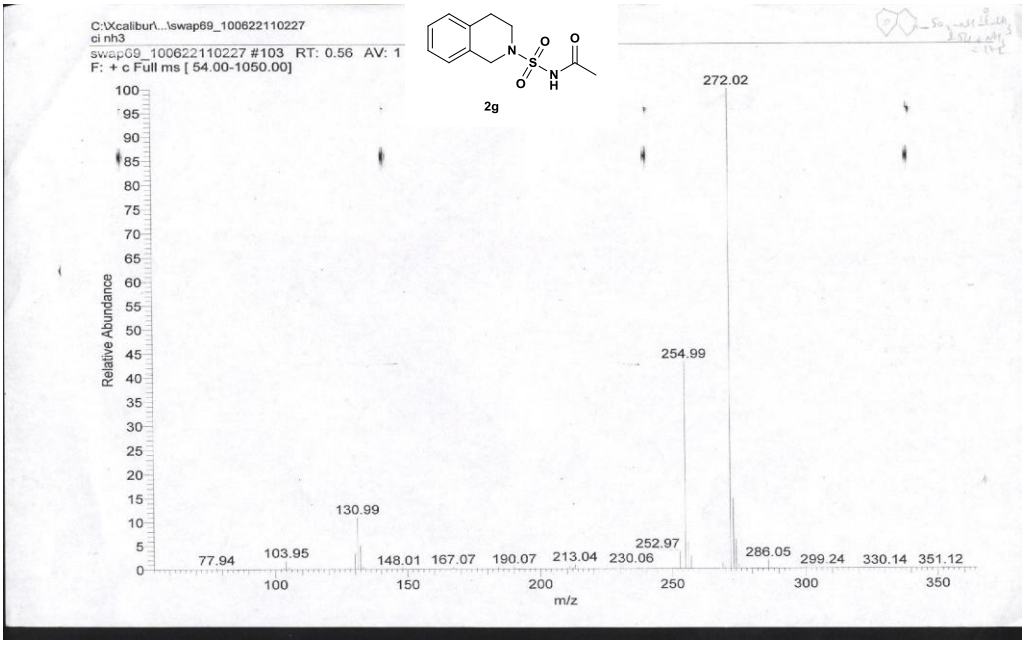


Figure S13: MS of 2g

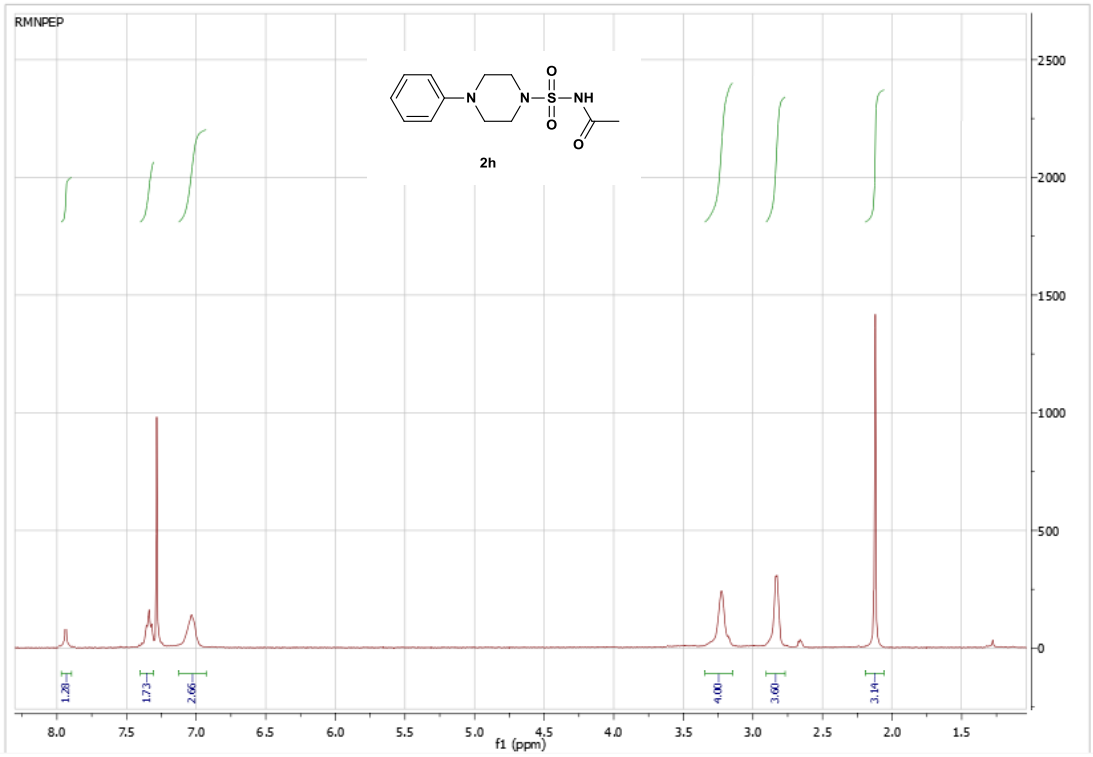


Figure S14: ^{1}H NMR of 2h

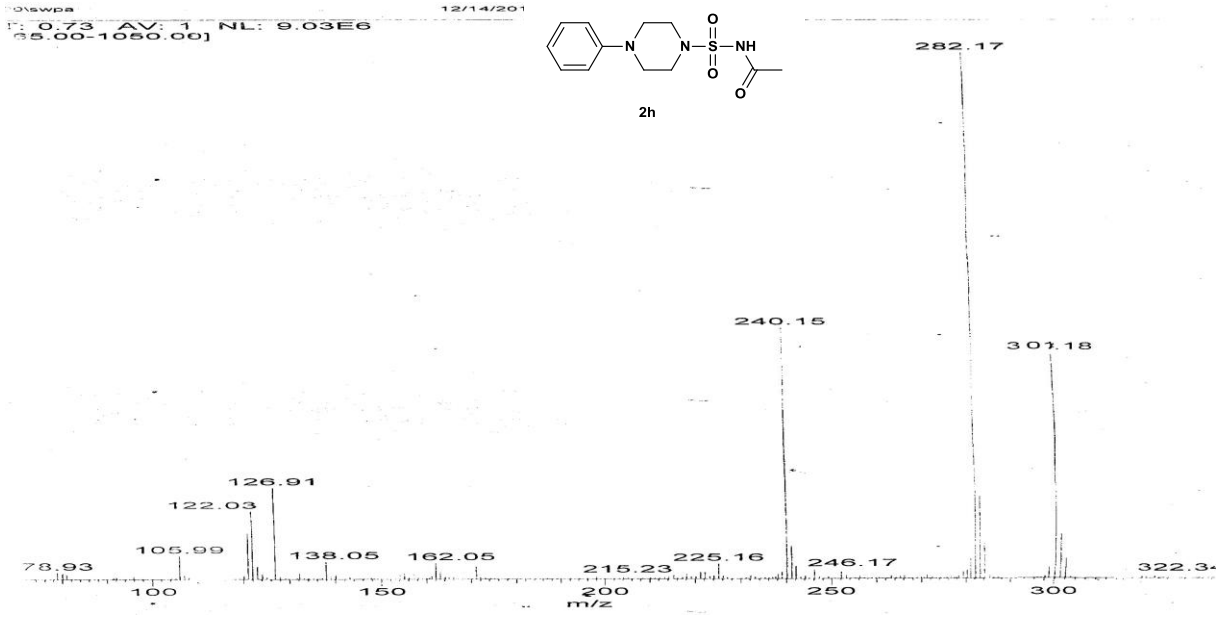


Figure S15: MS of 2h

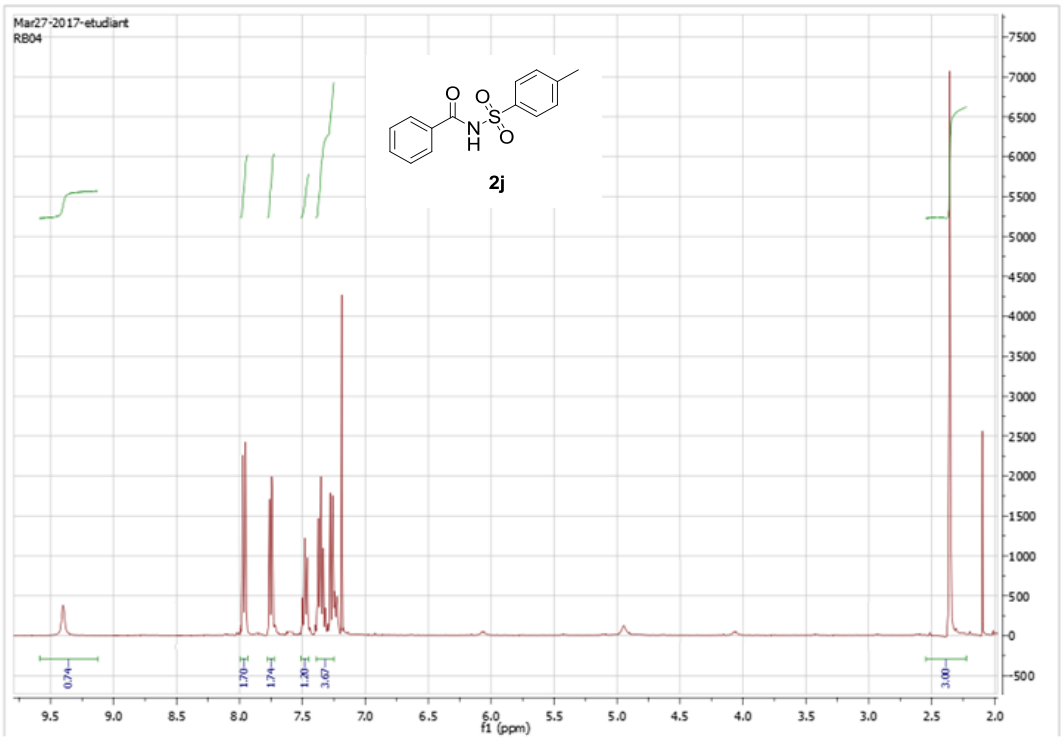


Figure S16: ^{1}H NMR of 2j

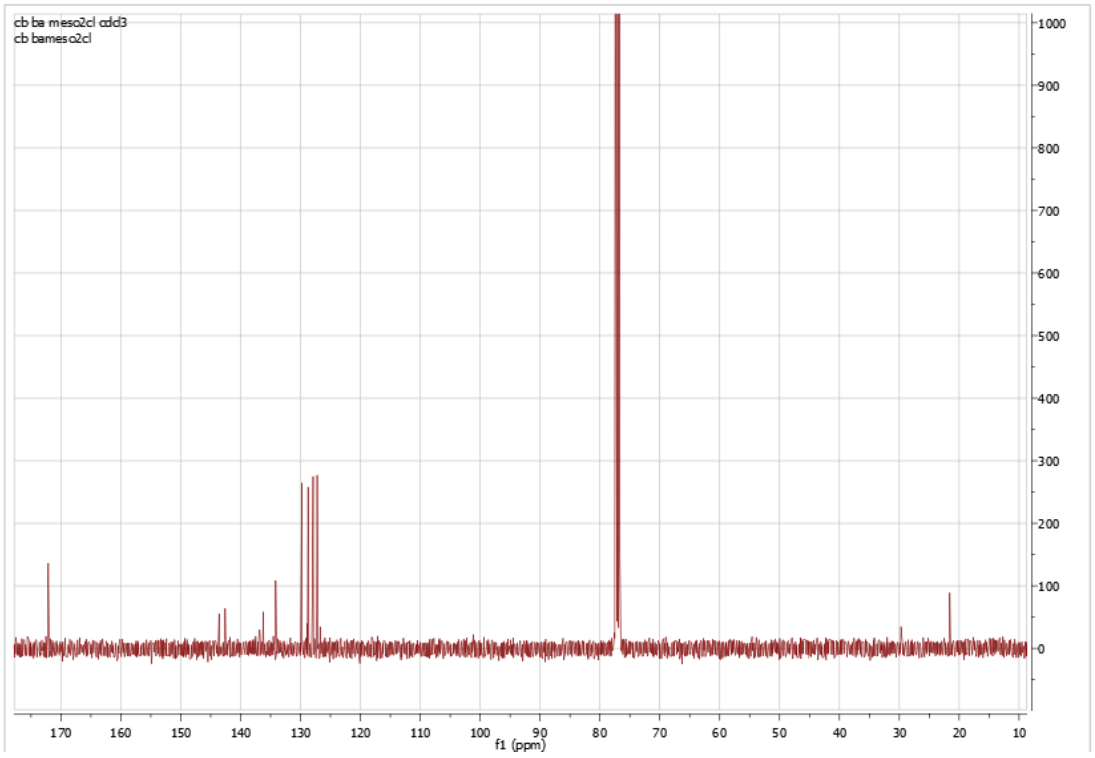


Figure S17: ^{13}C NMR of 2j

Selected spectroscopic data for new compounds (3a-m)

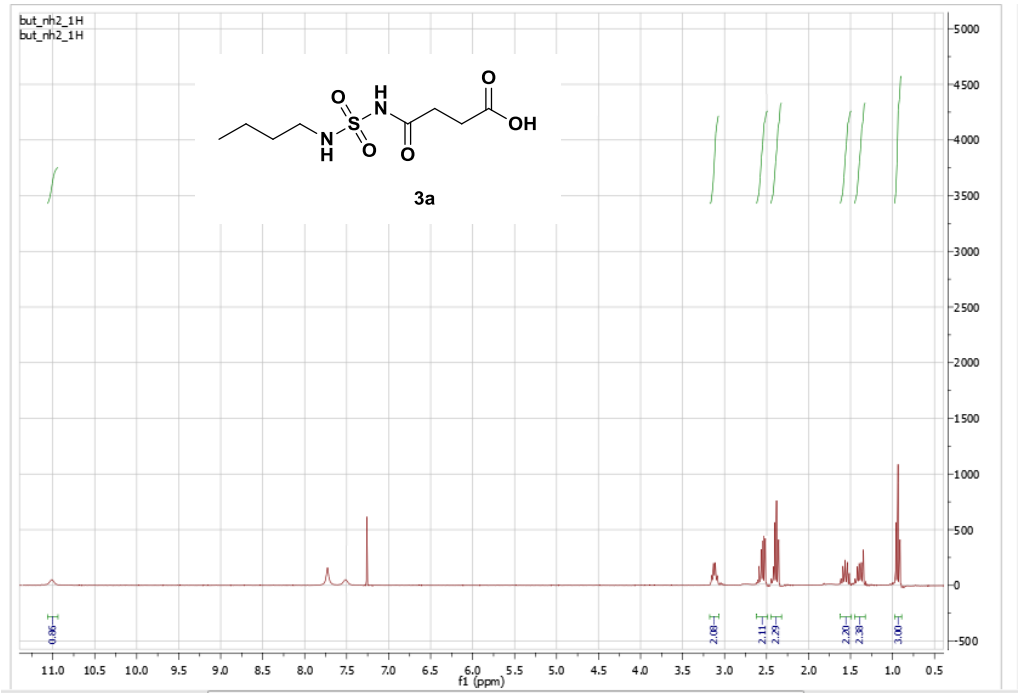


Figure S18: ^{1}H NMR of 3a

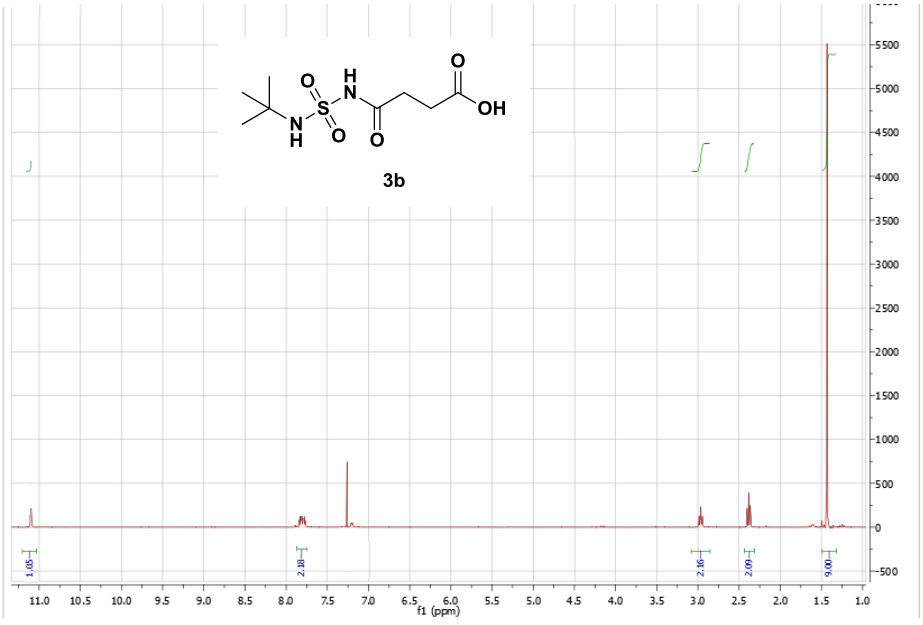


Figure S19: ^{1}H NMR of 3b

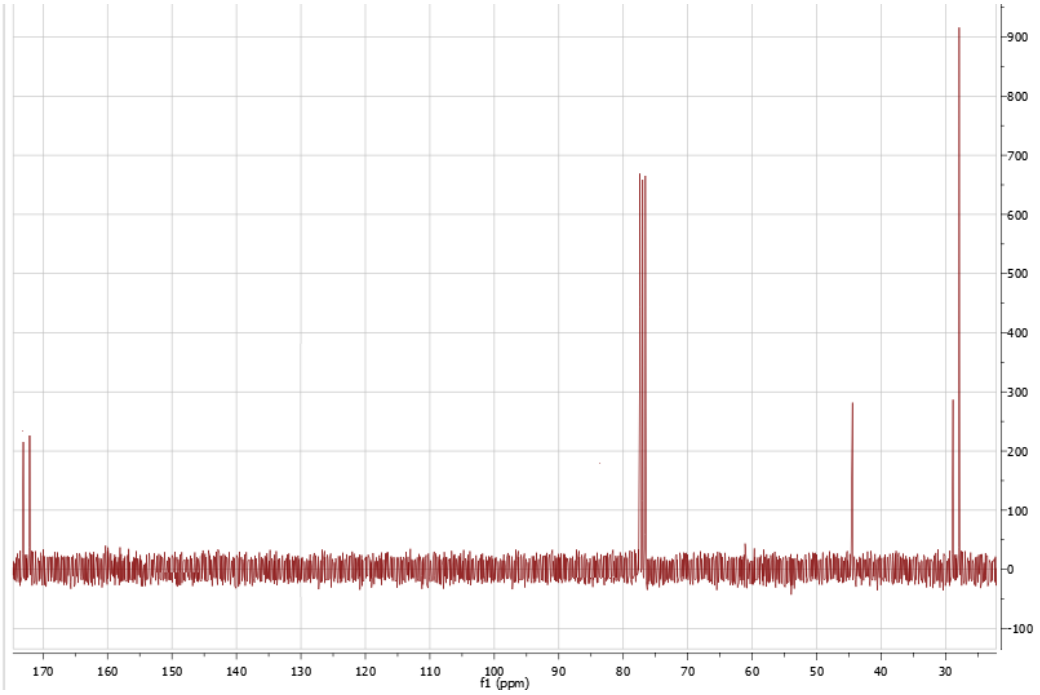


Figure S20: ^{13}C NMR of 3b

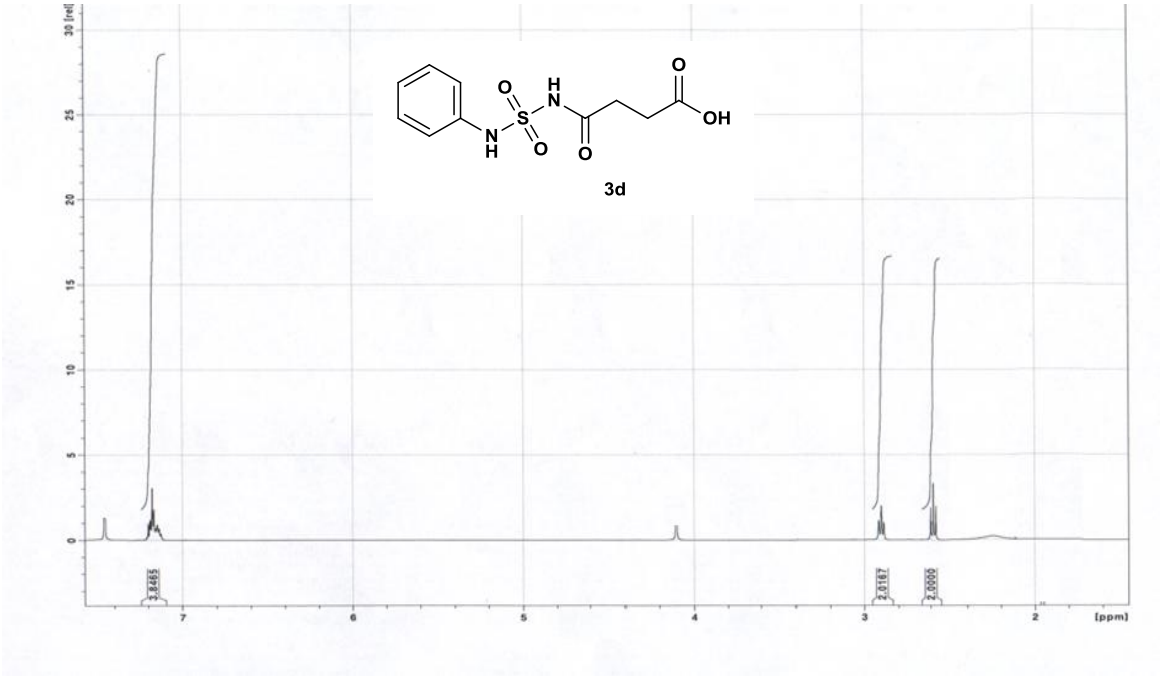


Figure S21: ^{1}H NMR of 3d

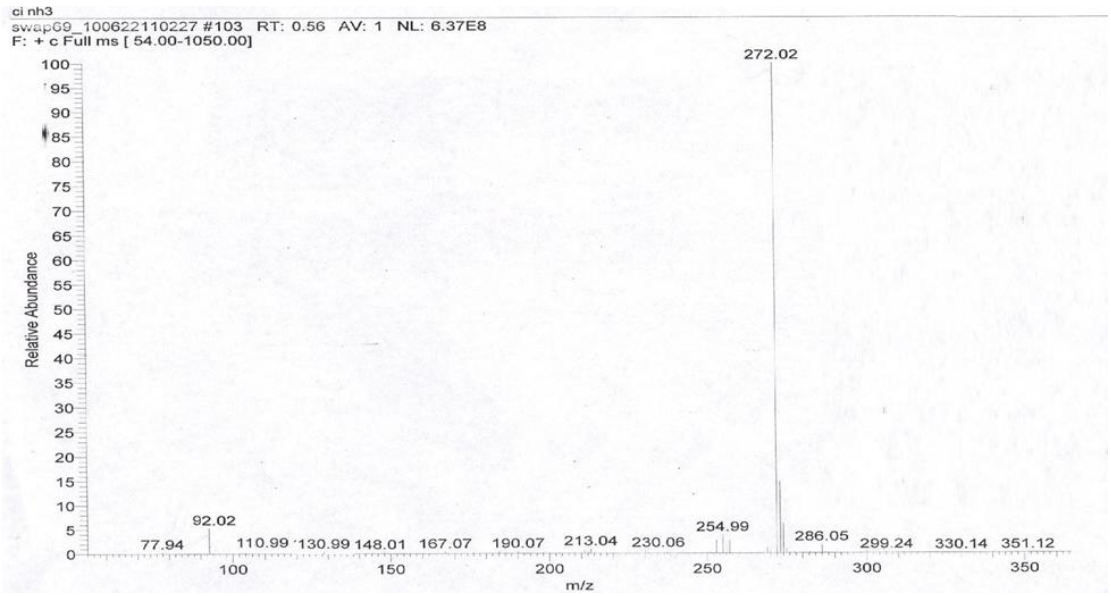


Figure S22: MS of 3d

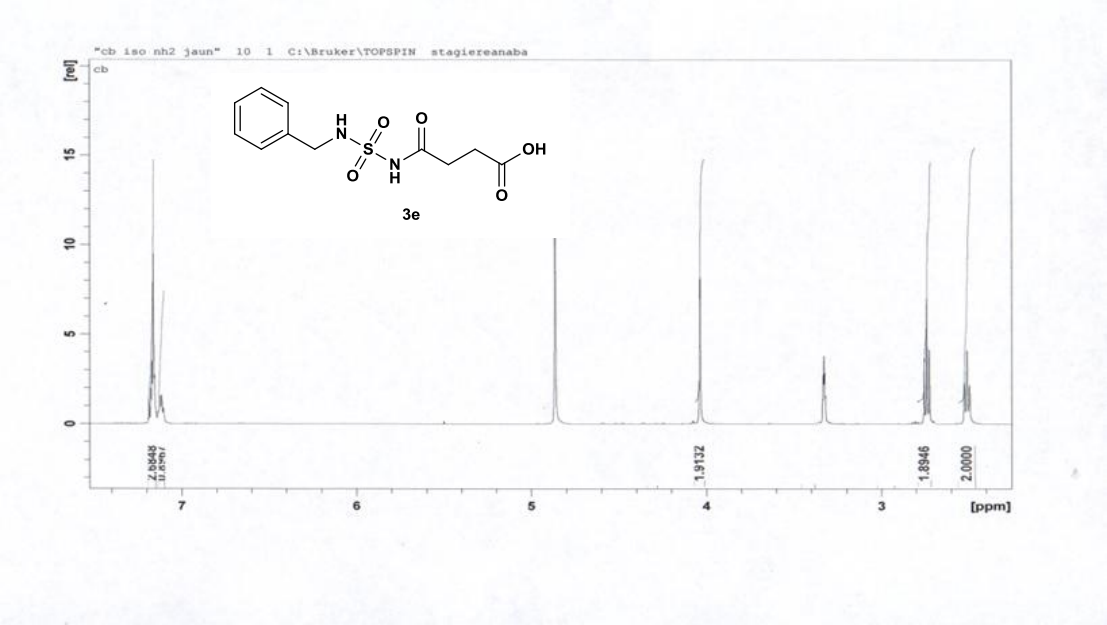


Figure S23: ^{1}H NMR of 3e

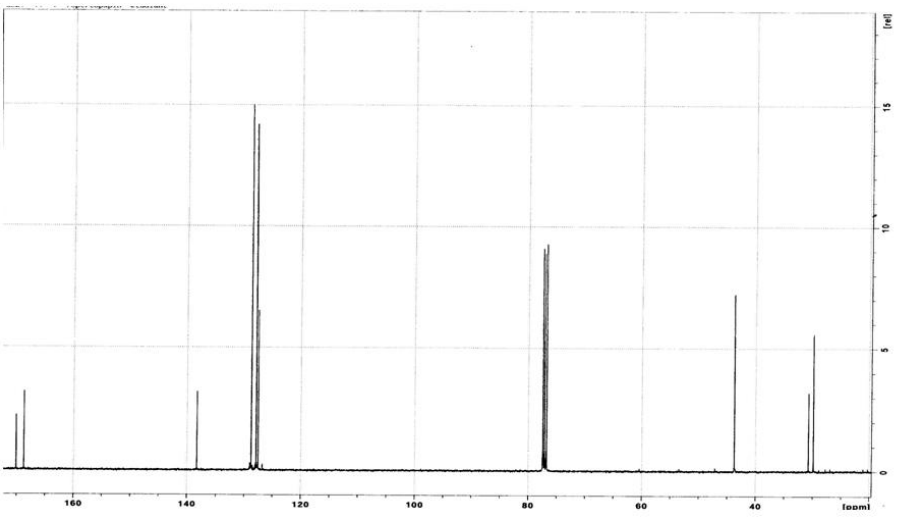


Figure S24: ^{13}C NMR of 3e

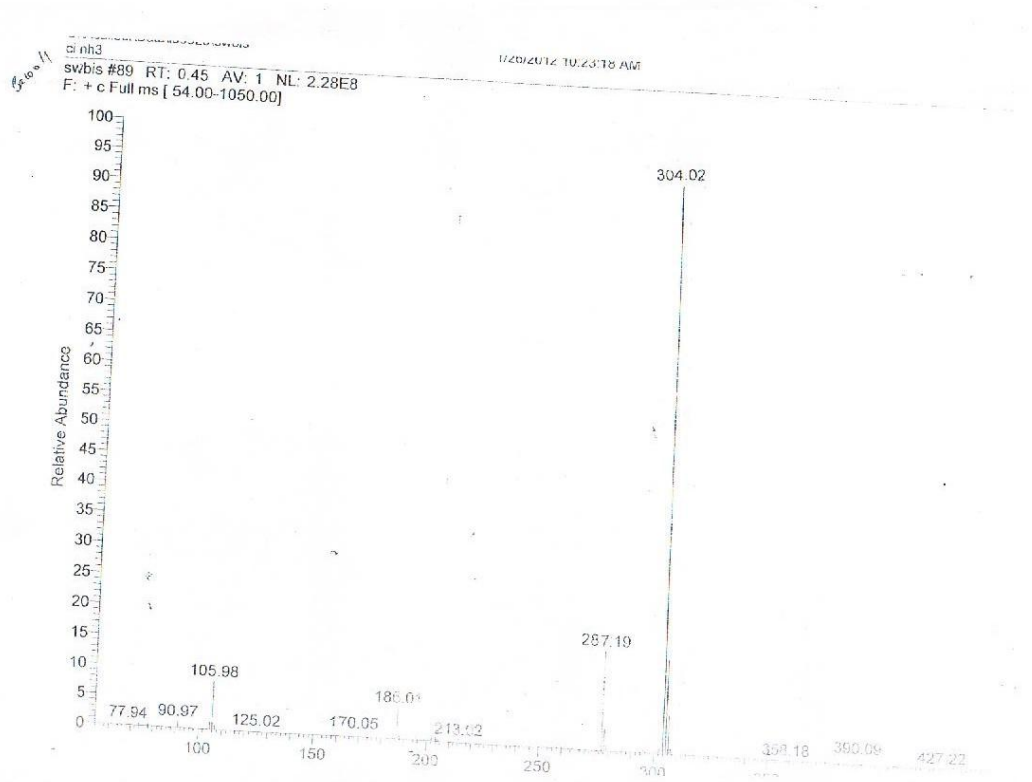


Figure S25: MS of 3e

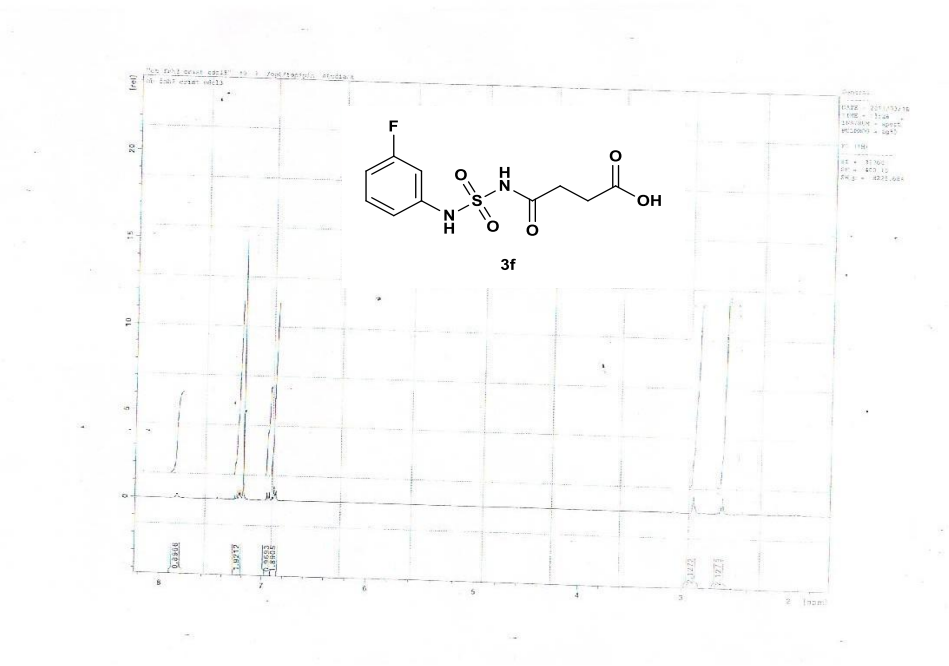


Figure S26: ^{1}H NMR of 3f

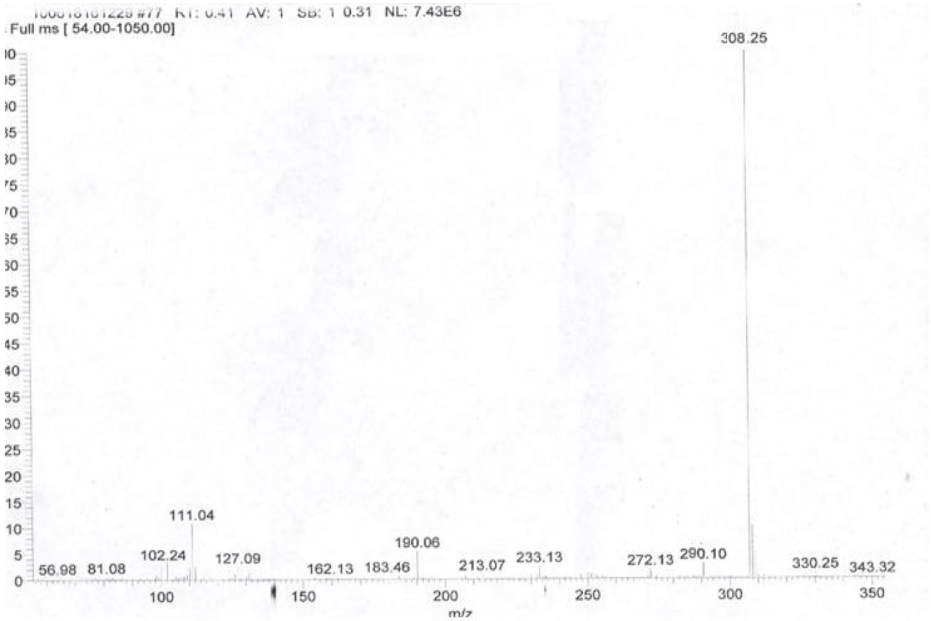


Figure S27: MS of 3f

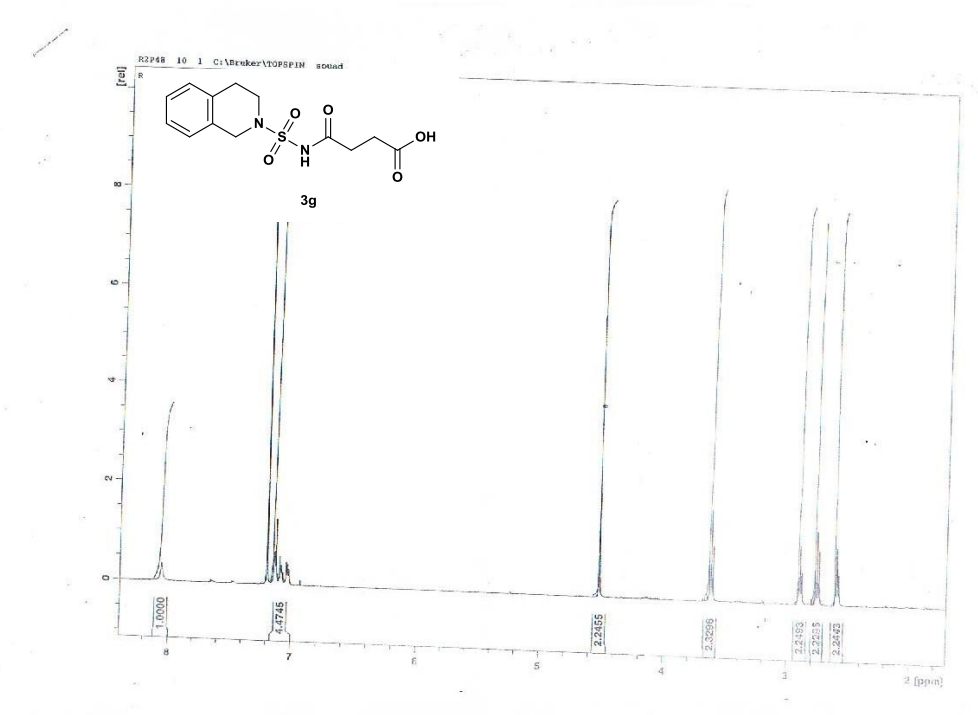


Figure S28: ^{1}H NMR of 3g

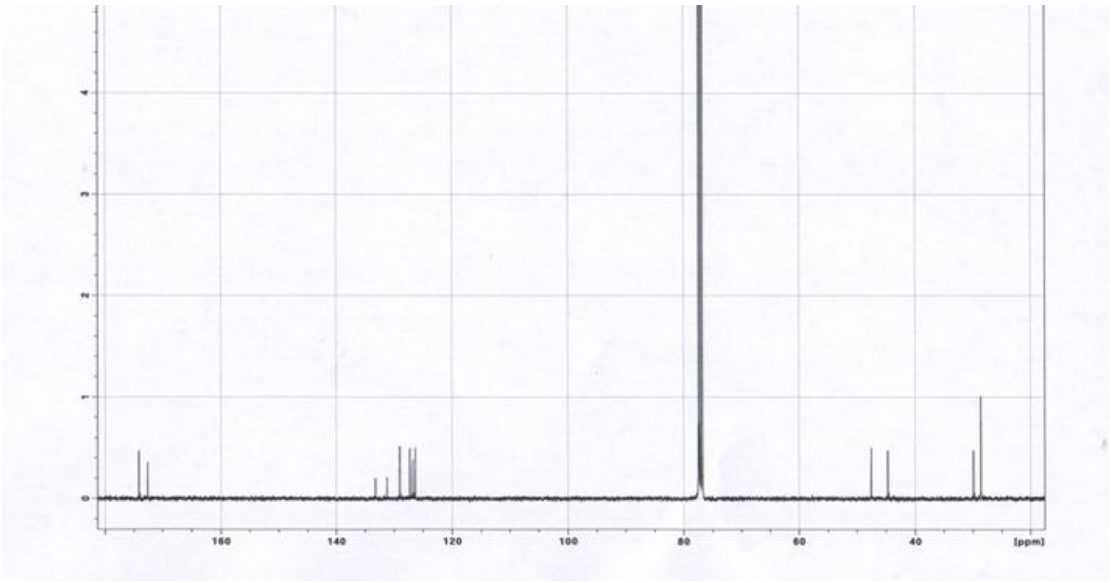


Figure S29: ^{13}C NMR of 3g

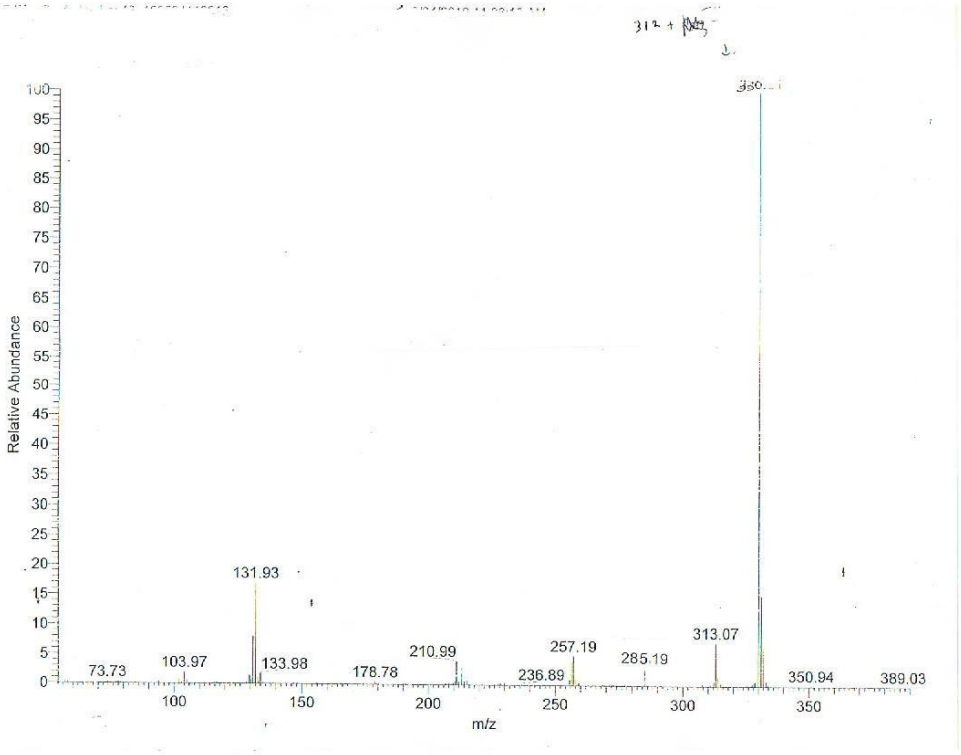


Figure S30: MS of 3g

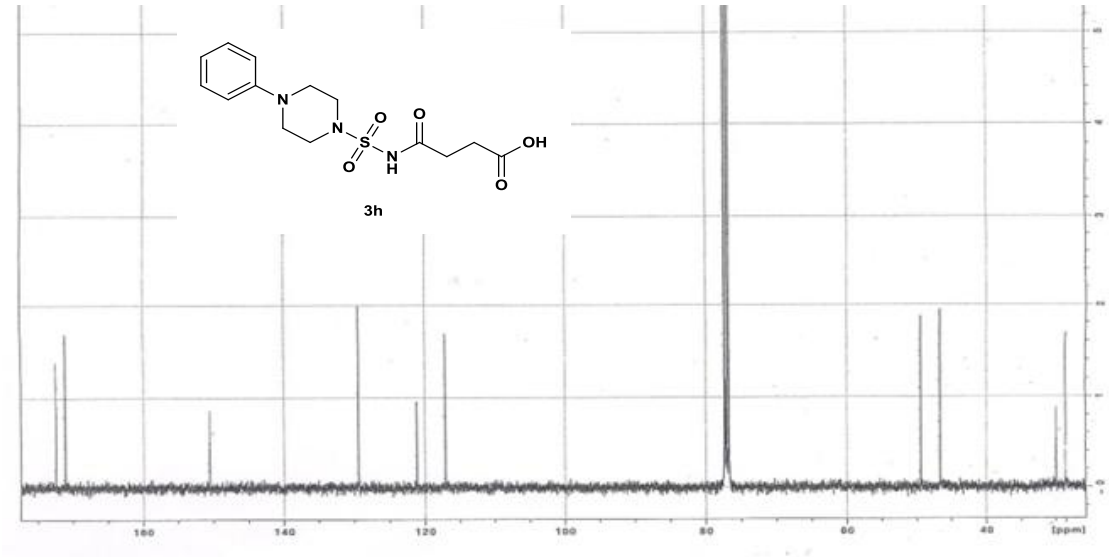


Figure S31: ^{13}C NMR of 3h

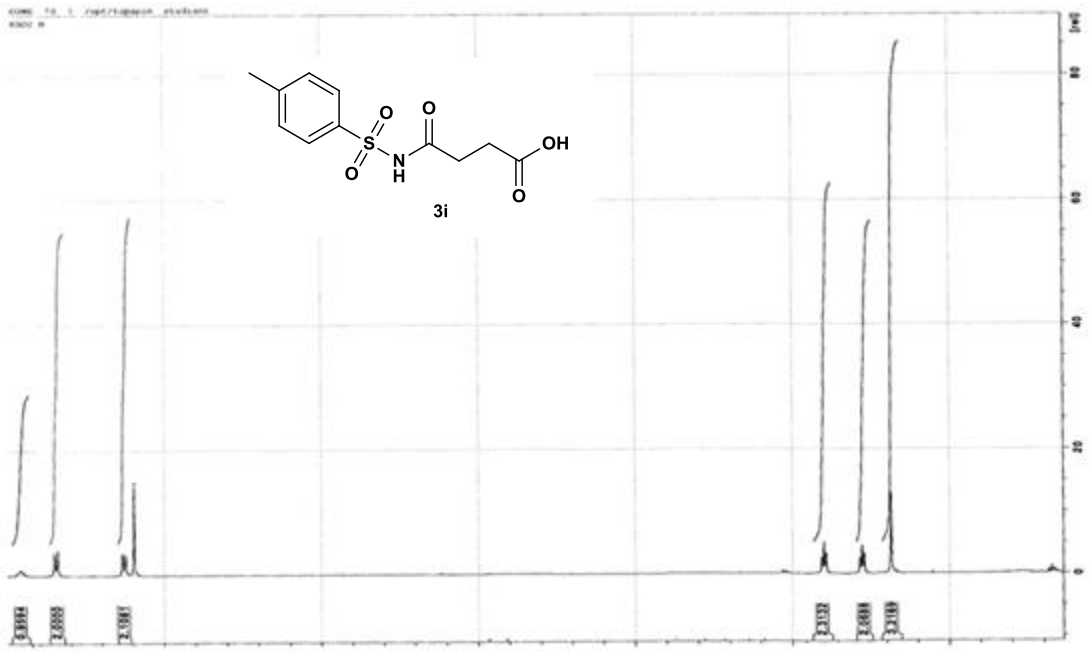


Figure S32: ^{1}H NMR of 3i

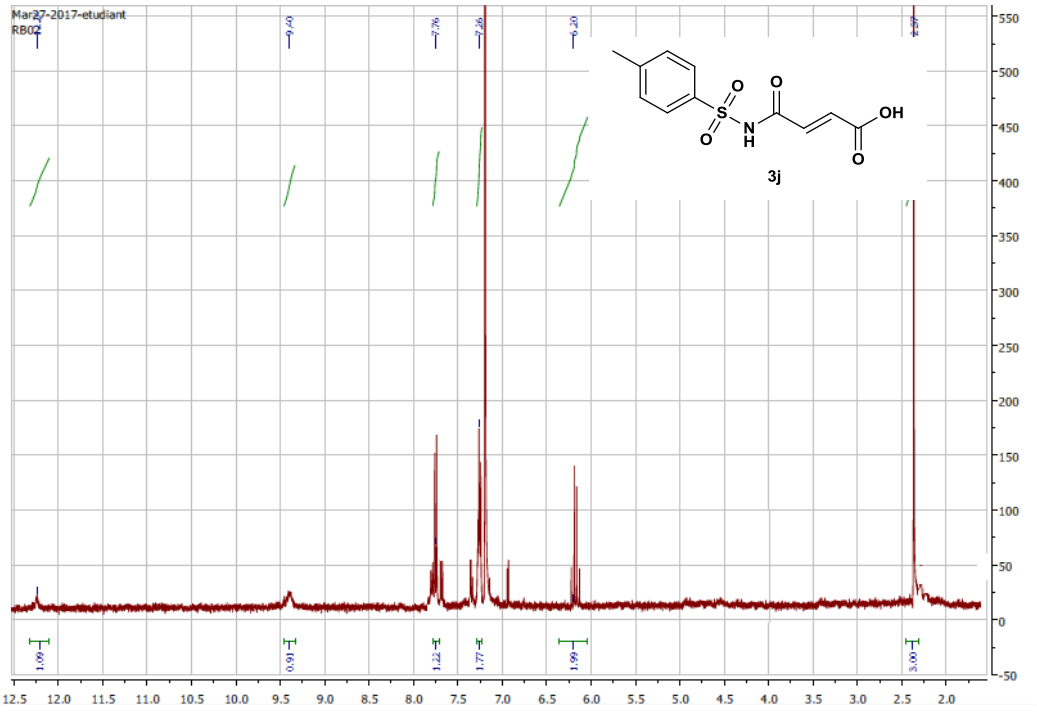


Figure S33: ^{1}H NMR of 3j

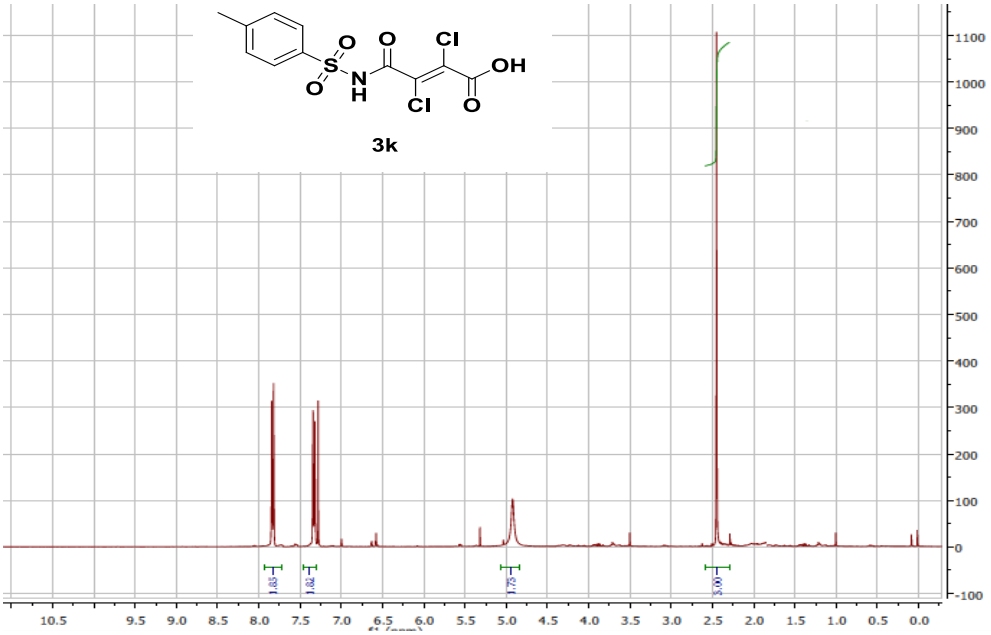


Figure S34: ^{1}H NMR of 3k

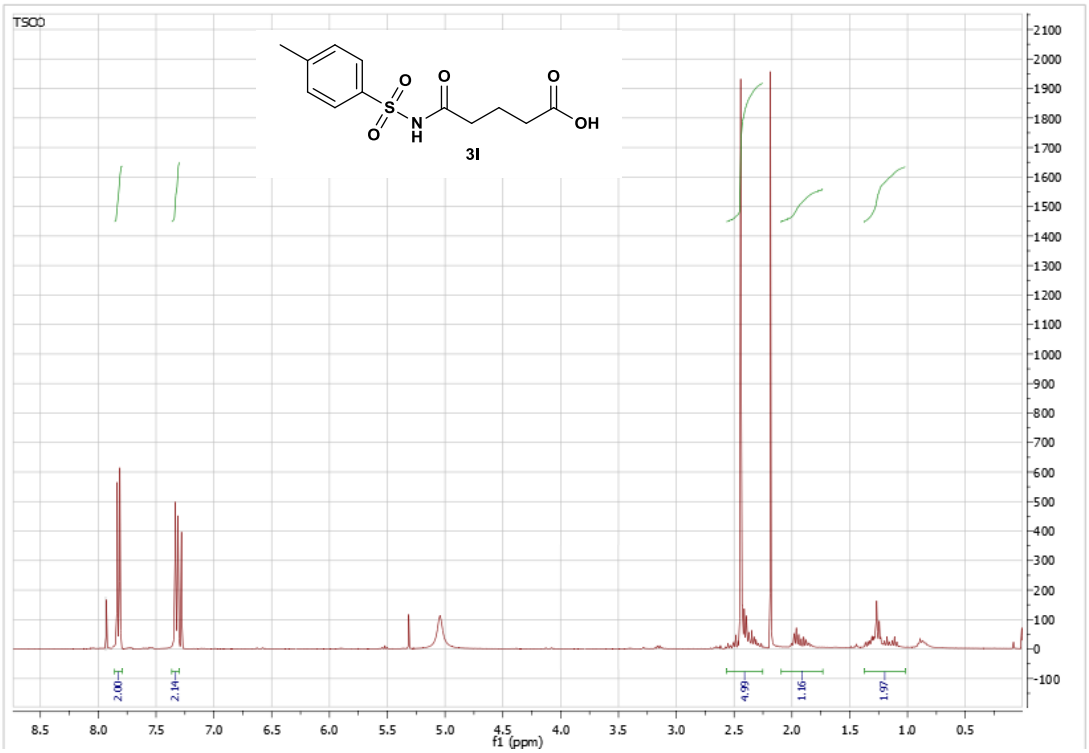


Figure S35: ^{1}H NMR of 3l

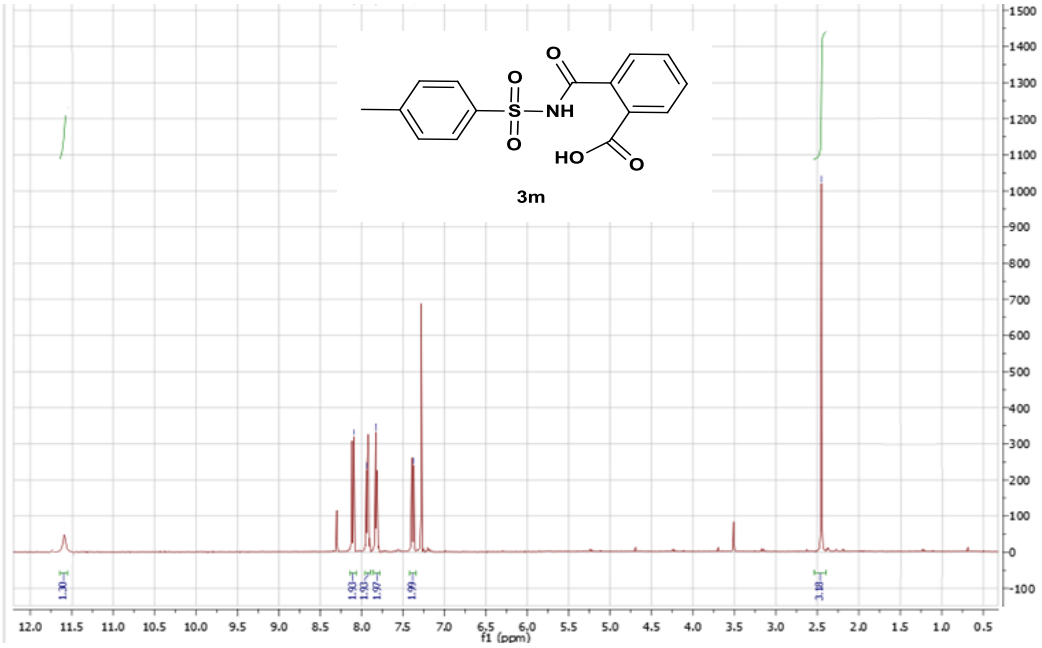


Figure S36: ^{1}H NMR of 3m

Selected spectroscopic data of compounds (3n-r)

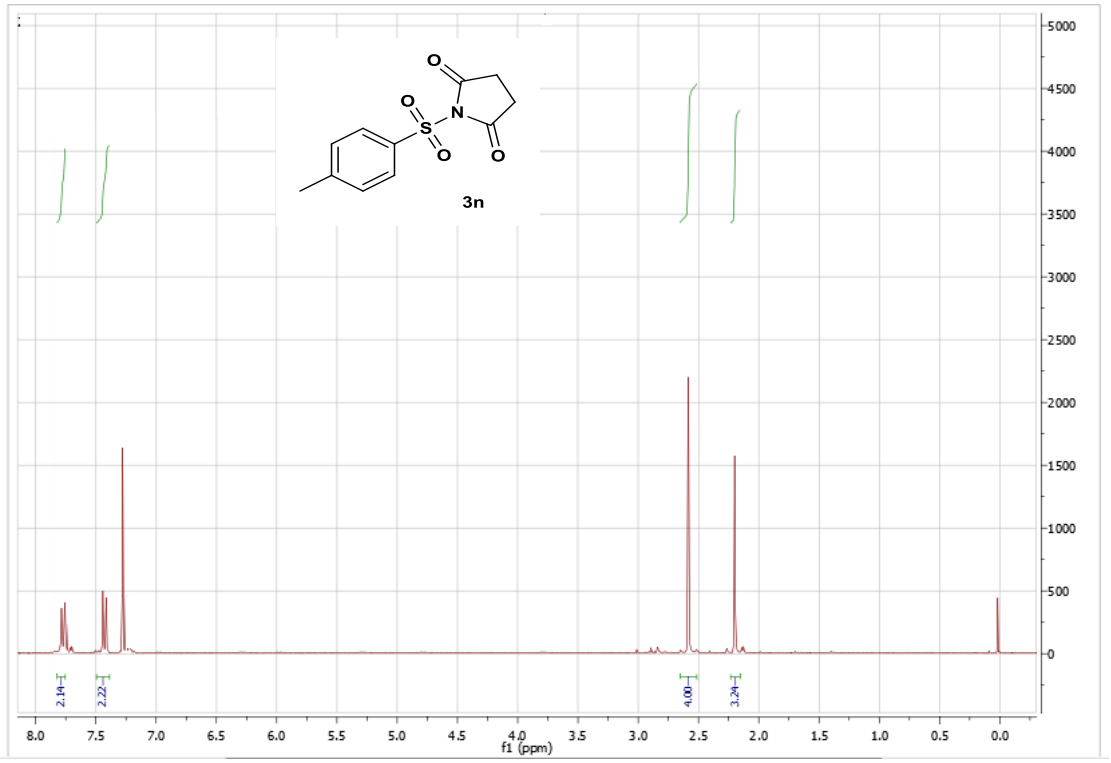


Figure S37: ^{1}H NMR of 3n

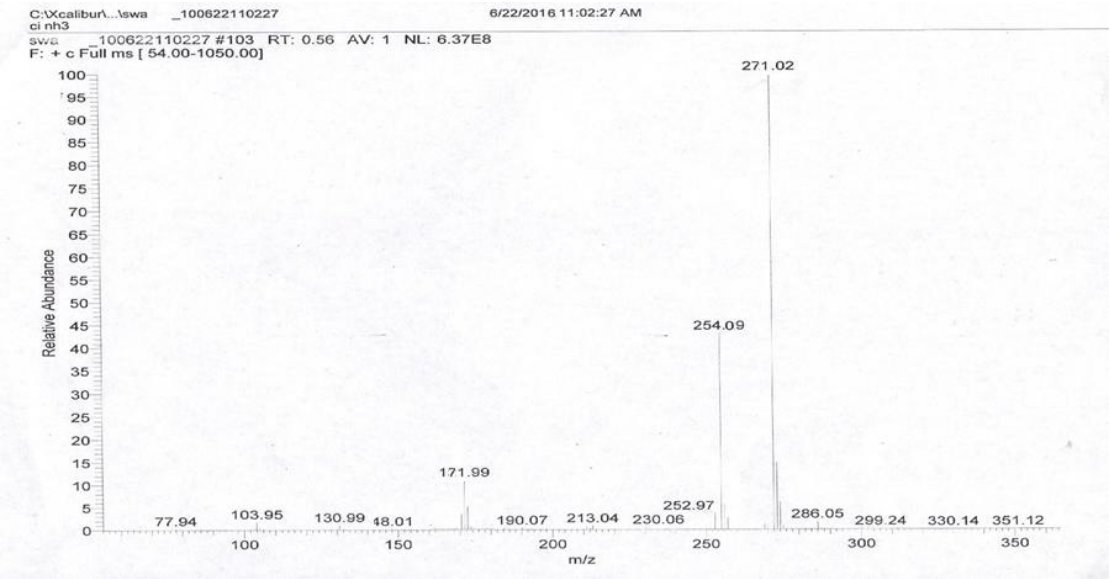


Figure S38: MS of 3n

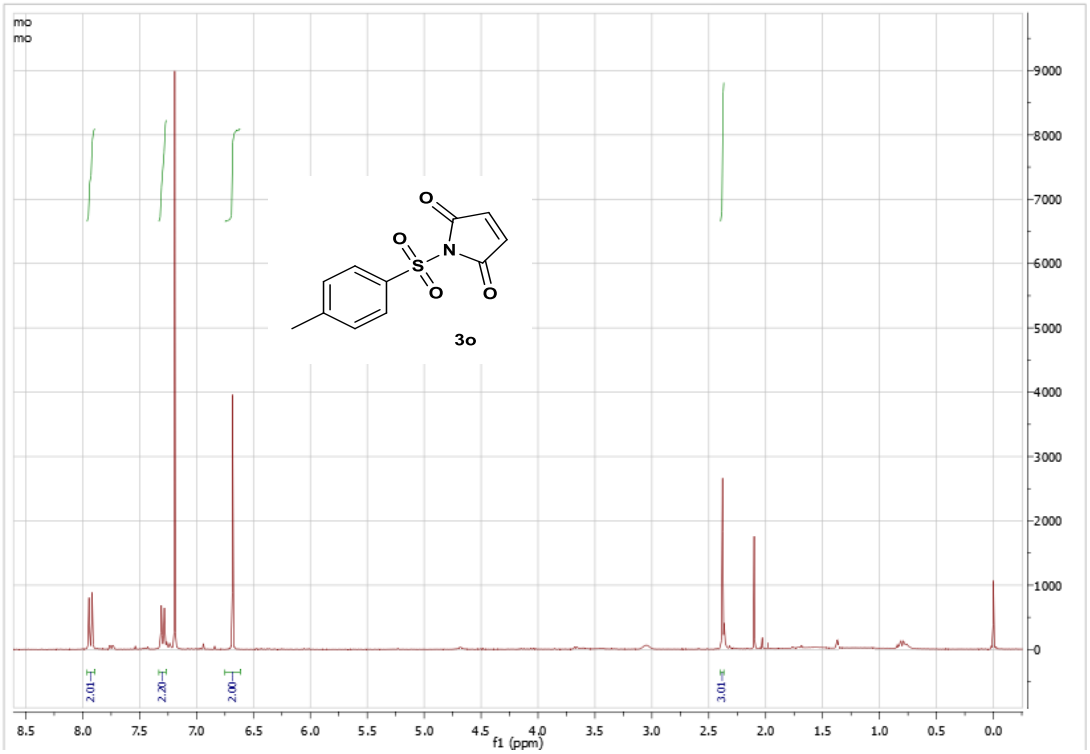


Figure S39: ^{1}H NMR of 3 o

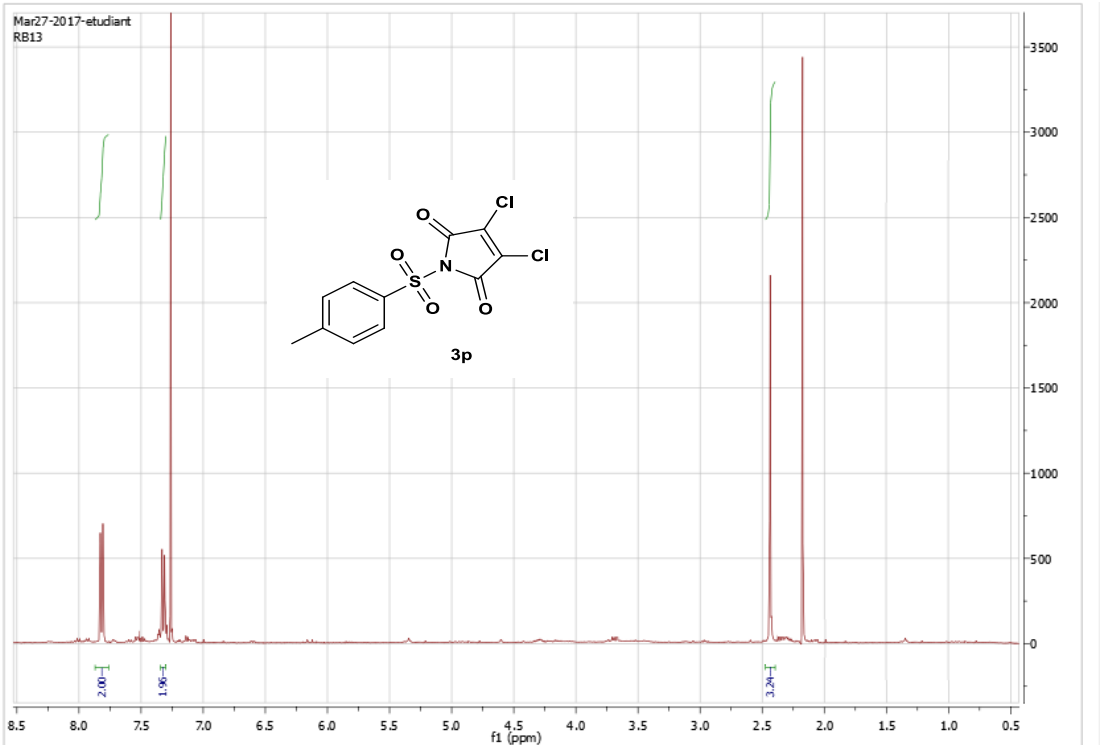


Figure S40: ^{1}H NMR of 3p

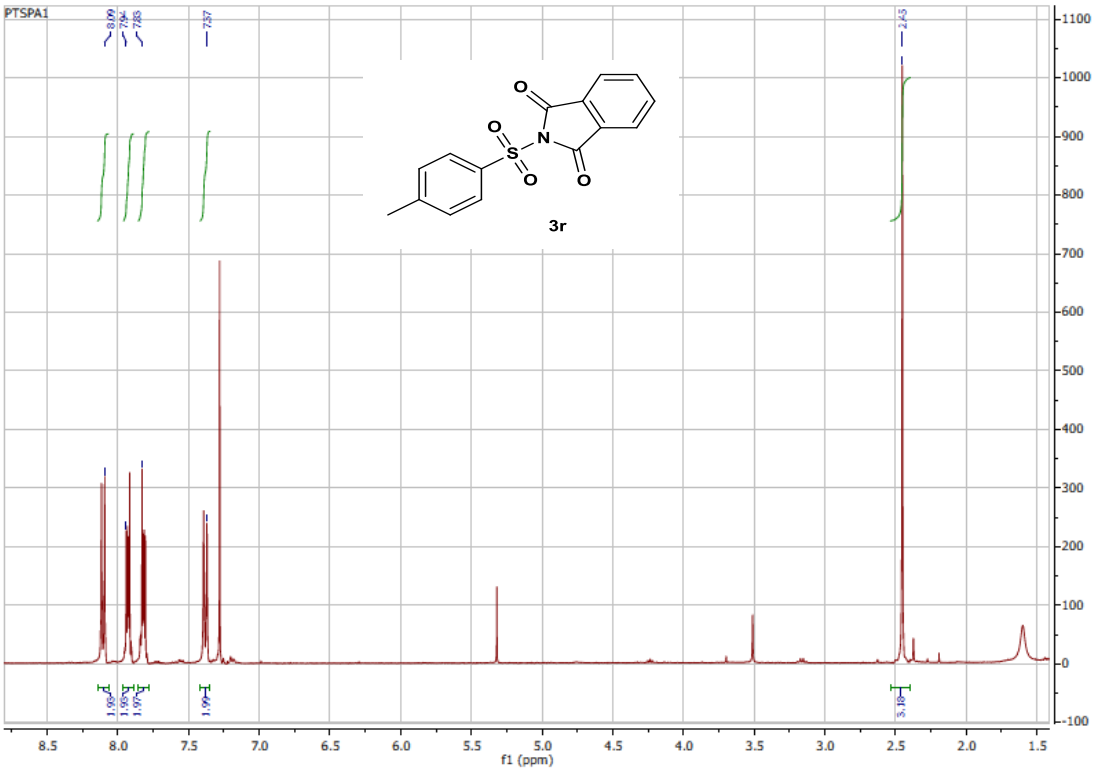


Figure S41: ^{1}H NMR of 3r

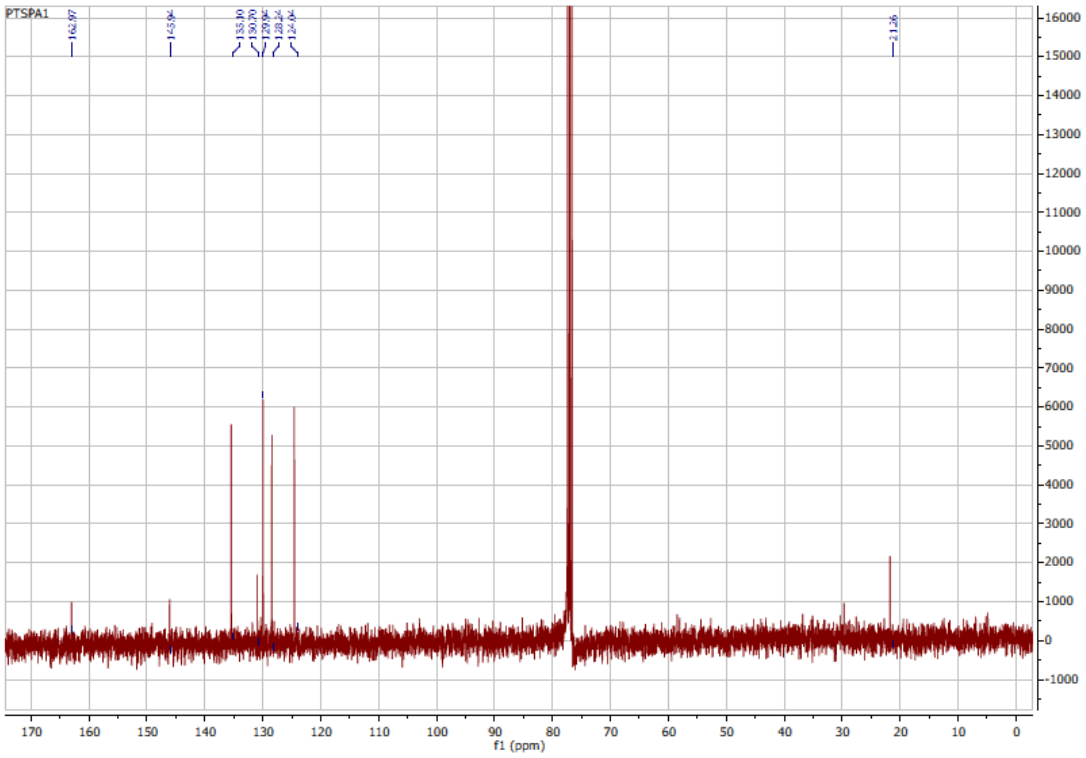


Figure S42: ^{13}C NMR of 3r

Selected spectroscopic data of compounds (5a-f)

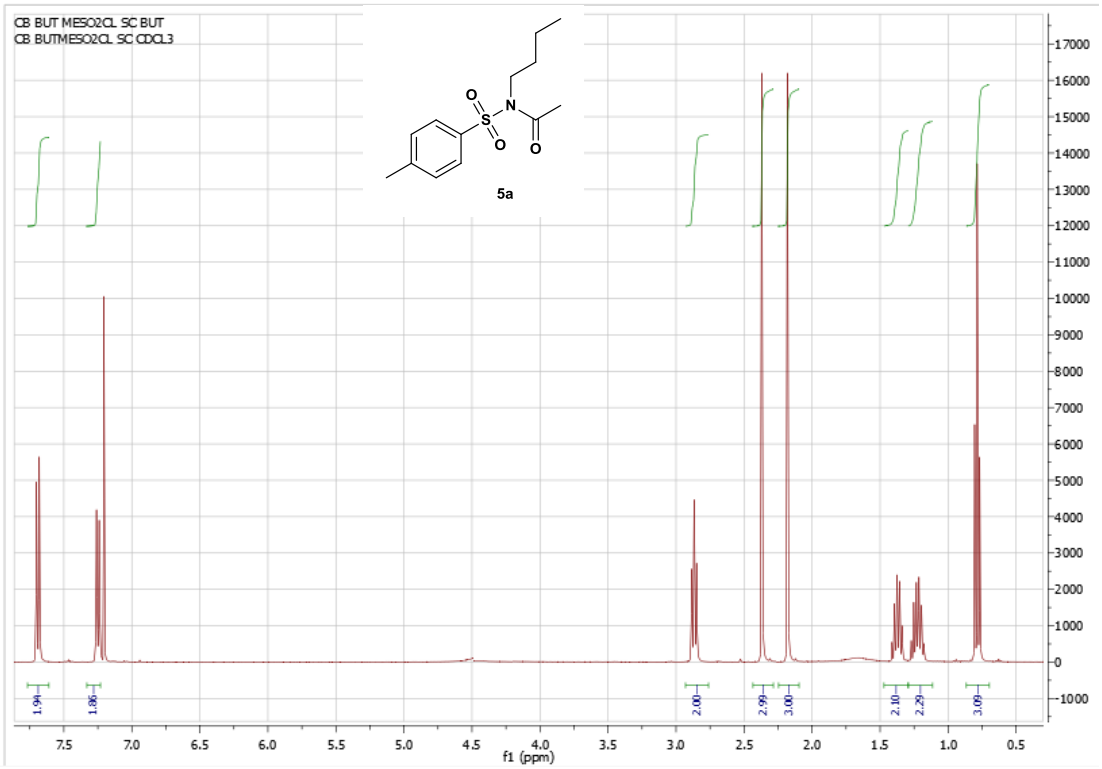


Figure S43: ^{1}H NMR of 5a

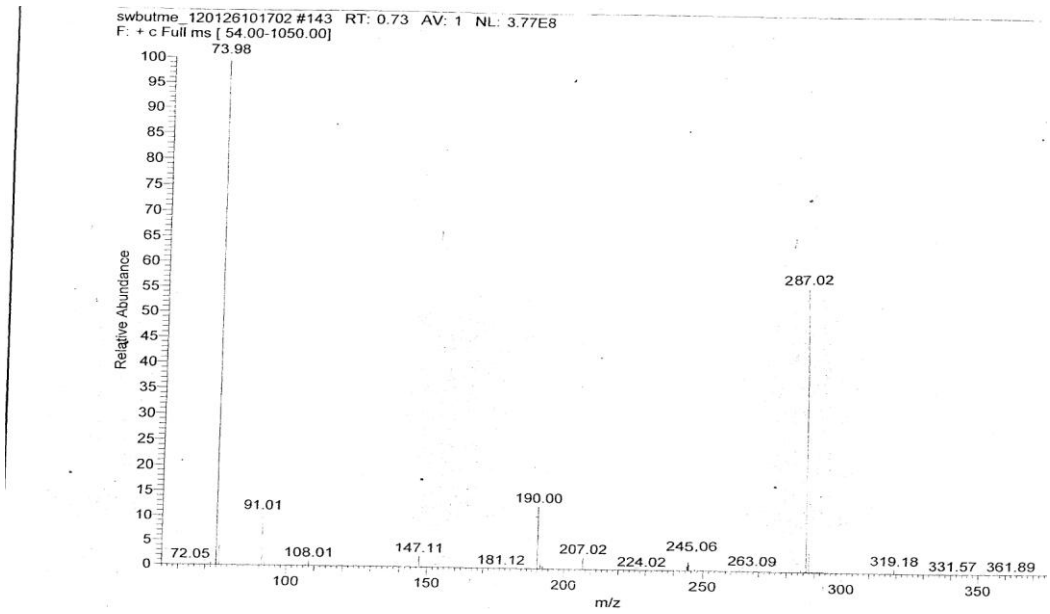


Figure S44: MS of 5a

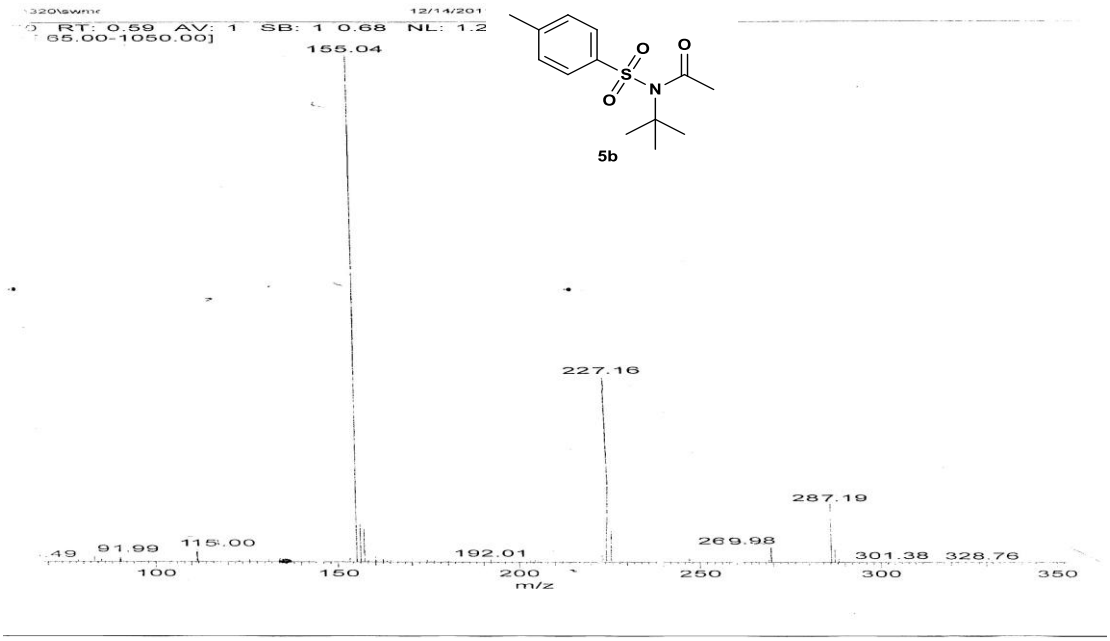


Figure S45: MS of 5b

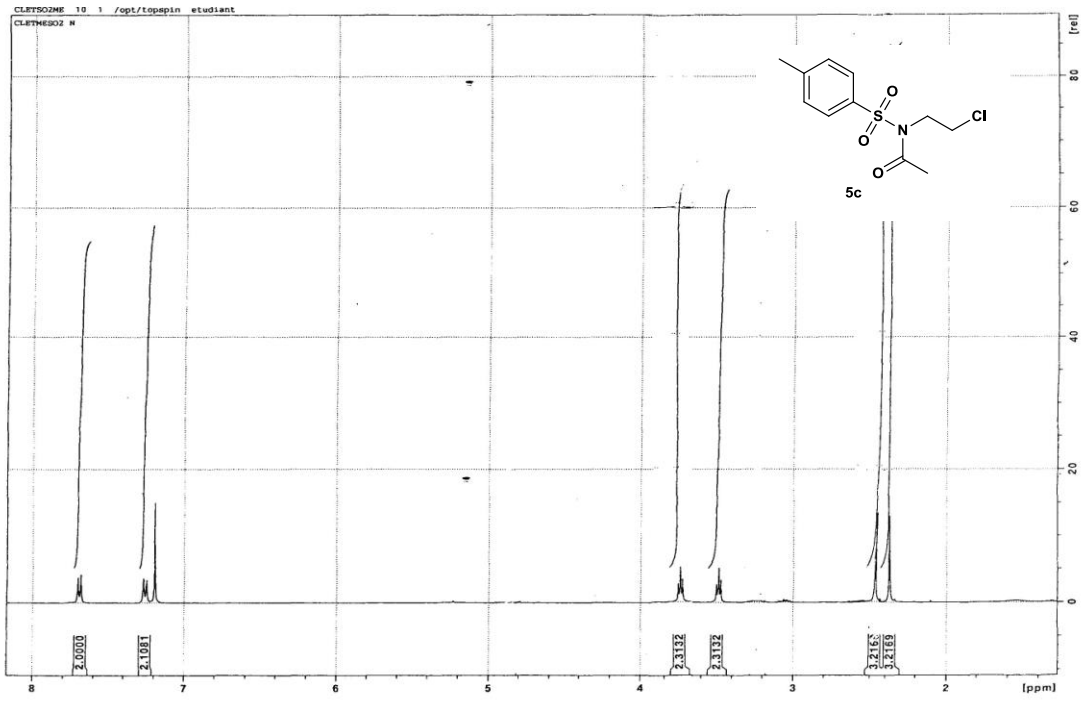


Figure S46: ^{1}H NMR of 5c

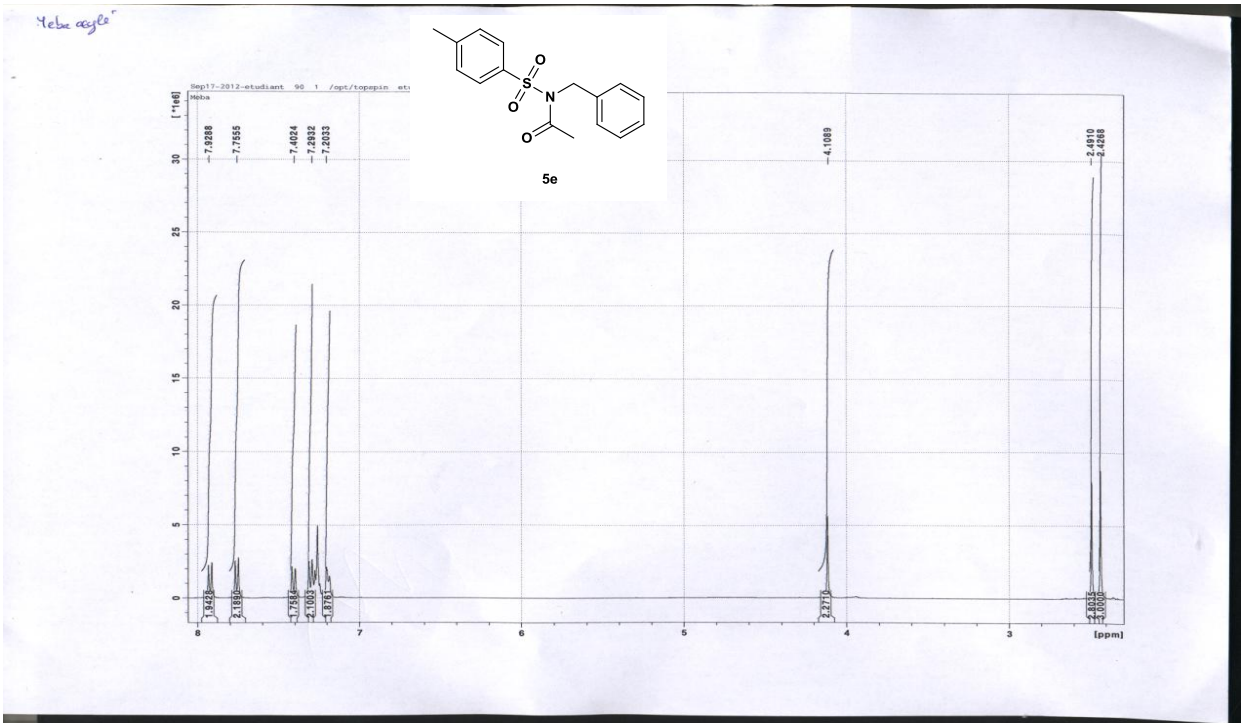


Figure S47: ^{1}H NMR of 5e

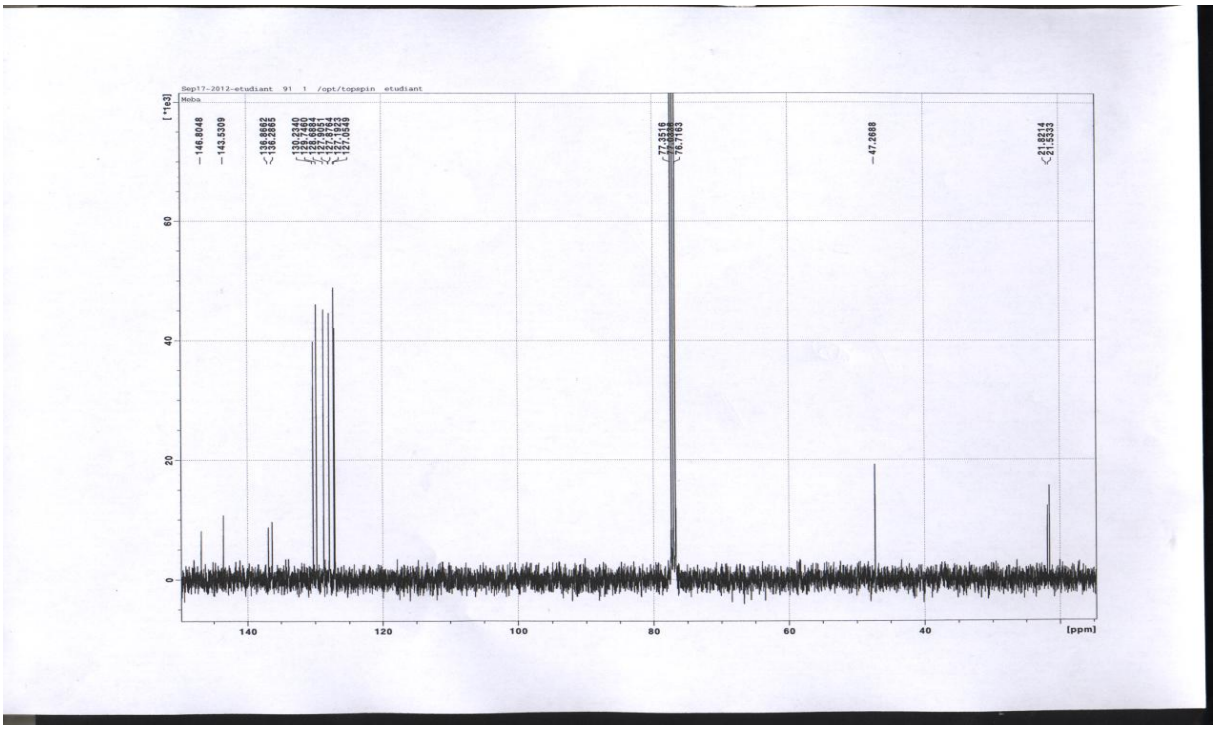


Figure S48: ^{13}C NMR of 5e