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Copper-Catalyzed Electrophilic Amination of Sodium Sulfinates at Room Temperature

Haibo Zhu,[a] Yajing Shen,[b] Qinyue Deng[c] and Tao Tu*[a,b]

[a] *H. Zhu,Y. Shen, Q. Deng and Prof. T. Tu*
Department of Chemistry, Fudan University 220 Handan Road, Shanghai, 200433 (China)
E-mail: taotu@fudan.edu.cn
[b] *Prof. T. Tu*
State Key Laboratory of Elemento-organic Chemistry,
Nankai University, Tianjin, 300071 (China)

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1. General

All commercial reagents were used directly without further purification, unless otherwise stated. Dry dimethylsulfoxide (DMSO) was purchased from J & K chemical, stored over 4 Å molecular sieves and handled under N_2. Anhydrous methanol (MeOH) was distilled from anhydrous calcium chloride, Dioxane, Tetrahydrofuran (THF), toluene and *m*-Xylene were distilled from sodium/benzophenone, 1,2-Dimethoxyethane (DME) and 1,2-Dichloroethane (DCE) were distilled from calcium hydride prior to use. *t*-BuONa was purchased from J & K chemical. All schlenk tubes and sealed vessels (50 mL) were purchased from Beijing Synthware Glass. $CDCl_3$ was purchased from Cambridge Isotope Laboratories.^{1}H NMR and ^{13}C NMR spectra were recorded on Jeol ECA-400 and Bruker 400 DRX spectrometers. ^{13}C NMR spectra were referenced to the carbon signal of $CDCl_3$ (77.0 ppm). GC-MS spectra were recorded on Agilent Technologies 1890A GC system and 5975C inert MSD with Triple-Axis Detector.

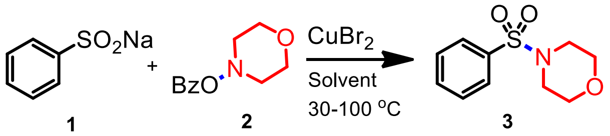
2. Experimental sections

2.1 General procedure for sulfonamides from *O*-benzoyl hydroxylamines and sodium sulfinates.

To a 50 mL schlenk tube containing sodium sulfinate (1.0 mmol) and *O*-benzoyl hydroxylamine (0.5 mmol) were added and the tube was purged with N_2 for 3 times. Then DCE (4.0 mL), subsequently (Note, if *O*-benzoyl hydroxylamine was a liquid, it was introduced to the tube after addition of DCE). The resulted mixture was allowed to stir for 12 h at room temperature under atmosphere of N_2, then $CuBr_2$ (2 mol%) was added and stirred another 12 h. After the completion of the reaction, the resulting mixture was concentrated under the vacuum and directly purified by flash chromatography to give the desired product.

2.2 Optimization of reaction conditions

Table S1. Catalyst and solvent effects (excluded data in the Table 1) [a]

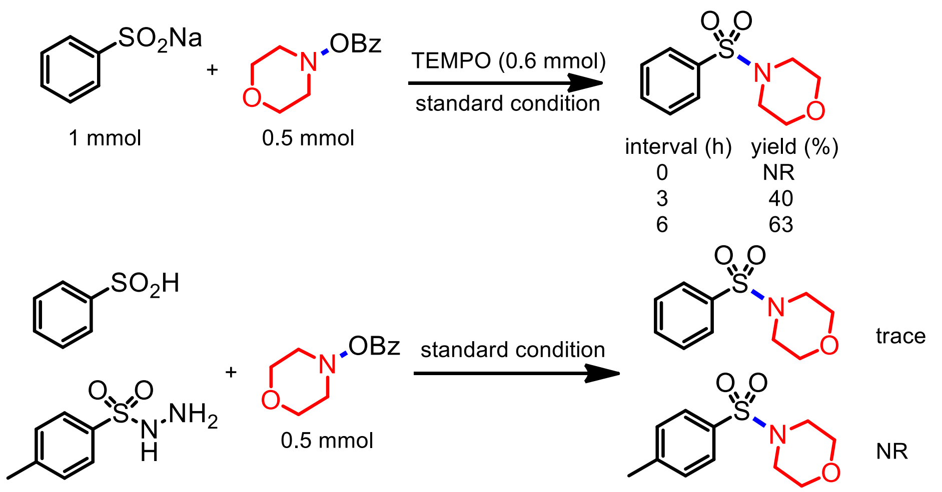


Entry	Solvent	Cat.	Temp. (°C)	Yield (%)[b]
1	EtOH/ H_2O (2:1)	$CuBr_2$	100	77
2	EtOH/ H_2O (1:1)	$CuBr_2$	100	56
3	EtOH/ H_2O (1:2)	$CuBr_2$	100	43
4	DCE	$Cu(NO_3)_2·3H_2O$	100	71
5	DCE	$CuSO_4·5H_2O$	100	67
6	DCE	$Cu(OAc)_2·H_2O$	100	85
7	DCE	$CuCl_2·2H_2O$	100	88
8	DCE	$FeBr_2$	100	41
9	DCE	$NiBr_2$	100	56
10	DCE	CuCl	100	45

[a] Reaction was carried out with 0.5 mmol scale: 2 equiv. **1** and 1equiv. **2** were dissolved in 4 mL solvent and stirred in 12 h under atmosphere of N_2, then the catalyst was added and stirred for additional 12 h.

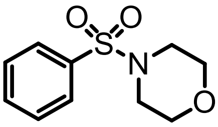
[b] Isolate yield based on **2.**

2.3 Control experiments of reaction conditions

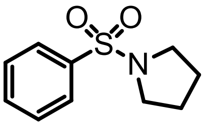


Scheme S1 Control experiments.

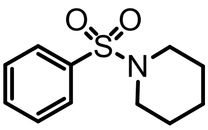
3. Data for the amination products

3[S1]: 

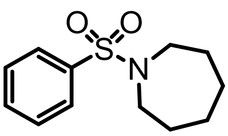
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.77-7.75 (m, 2H, Ar*H*), 7.65-7.61 (m, 1H, Ar*H*), 7.58-7.54 (m, 2H, Ar*H*), 3.74 (t, *J* = 5.0 Hz, 4H, OCH_2), 3.00 (t, *J* = 5.0 Hz, 4H, NCH_2); ^{13}C NMR (100 MHz, $CDCl_3$, , 298 K, ppm) δ 134.97 (Ar*C*), 133.06 (Ar*C*), 129.12 (Ar*C*), 127.82 (Ar*C*), 66.08 (O*C*H_2), 45.96 (N*C*H_2); Yield: >99%, white solid, mp: 115-117 ^{o}C; R_f = 0.50 (Petrol/EtOAc 4:1).

5a[S1]: 

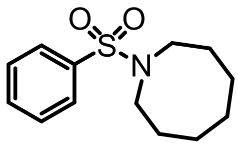
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.85-7.83 (m, 2H, Ar*H*), 7.62-7.51 (m, 3H, Ar*H*), 3.27-3.23 (m, 4H, NCH_2), 1.80-1.72 (m, 4H, CH_2); ^{13}C NMR: (100 MHz, $CDCl_3$, 298 K, ppm) δ 136.79 (Ar*C*), 132.48 (Ar*C*), 128.90 (Ar*C*), 127.33 (Ar*C*), 47.82 (N*C*H_2), 25.10 (*C*H_2); Yield: >99%, white solid, mp: 156-157 ^{o}C; R_f = 0.60 (Petrol/EtOAc 8:1).

5b[S1]**:** 

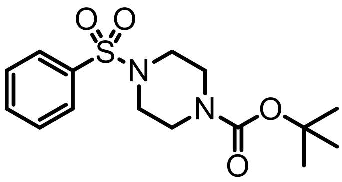
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.77-7.75 (m, 2H, Ar*H*), 7.62-7.57 (m, 1H, Ar*H*), 7.55-7.51 (m, 2H, Ar*H*), 2.99 (t, *J* = 5.6 Hz, 4H, NCH_2), 1.67-1.61 (m, 4H, CH_2), 1.44-1.39 (m, 2H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ = 136.36 (Ar*C*), 132.51 (Ar*C*), 128.88 (Ar*C*), 127.61 (Ar*C*), 46.90 (N*C*H_2), 25.14 (*C*H_2), 23.47 (*C*H_2); Yield: >99%, white solid, mp: 90-92 ^{o}C; R_f = 0.60 (Petrol/EtOAc 8:1).

5c[S2]**:** 

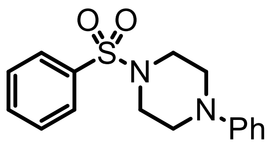
^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.79-7.77 (m, 2H, Ar*H*), 7.56-7.47 (m, 3H, Ar*H*), 3.26 (t, *J* = 5.6 Hz, 4H, NC*H*$_2$), 1.70-1.57 (m, 4H, C*H*$_2$), 1.56-1.55 (m, 4H, C*H*$_2$); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ = 139.50 (Ar*C*), 132.13 (Ar*C*), 128.90 (Ar*C*), 126.81 (Ar*C*), 48.17 (N*C*H$_2$), 29.06 (*C*H$_2$), 26.80 (*C*H$_2$); Yield: 88%, colourless liquid, R_f = 0.70 (Petrol/EtOAc 8:1).

5d[S3]: 

^{1}H NMR ($CDCl_3$, 400 MHz, 298 K): δ = 7.80-7.78 (m, 2H, Ar*H*), 7.58-7.48 (m, 3H, Ar*H*), 3.14 (t, *J* = 5.6 Hz, 4H, NC*H*$_2$), 1.73-1.64 (m, 10H, C*H*$_2$); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 142.72 (Ar*C*), 132.19 (Ar*C*), 128.92 (Ar*C*), 127.06 (Ar*C*), 48.70 (N*C*H$_2$), 29.68 (*C*H$_2$), 27.75 (*C*H$_2$), 26.61 (*C*H$_2$), 25.04 (*C*H$_2$); Yield: 79%, colourless liquid, R_f = 0.75 (Petrol/EtOAc 8:1).

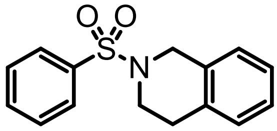
6a[S4]: 

^{1}H NMR (400 MHz, $CDCl_3$, 298 K) δ = 7.76-7.74 (m, 2H, Ar*H*), 7.642-7.61 (m, 1H, Ar*H*), 7.57-7.53 (m, 2H, Ar*H*), 3.51 (t, *J* = 4.8 Hz, 4H, NC*H*$_2$), 2.98 (t, *J* = 4.8 Hz, 4H, NC*H*$_2$), 1.40 (s, 9H, C*H*$_3$); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ = 154.07 (*C*=O), 132.95 (Ar*C*), 129.07 (Ar*C*), 127.59 (Ar*C*), 80.32 (*C*-O), 45.75 (N*C*H$_2$), 28.16 (*C*H$_3$); Yield: >99%, yellow solid, mp: 127-128 $^{\circ}$C; R_f = 0.60 (Petrol/EtOAc 4:1).

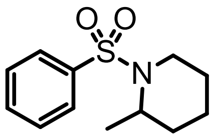
6b[S5]: 

^{1}H NMR (400 MHz, $CDCl_3$, 298 K) δ = 7.81-7.79 (m, 2H, Ar*H*), 7.64-7.61 (m, 1H, Ar*H*), 7.58-7.54 (m, 2H, Ar*H*), 7.27-7.23 (m, 2H, Ar*H*), 6.91-6.86 (m, 3H, Ar*H*), 3.26-3.23 (m, 4H, NC*H*$_2$), 3.19-3.17 (m, 4H, NC*H*$_2$); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ = 135.48 (Ar*C*), 132.98 (Ar*C*), 129.24 (Ar*C*), 129.11 (Ar*C*), 127.84 (Ar*C*), 122.73 (Ar*C*), 120.86 (Ar*C*), 116.90 (Ar*C*), 49.18 (N*C*H$_2$), 46.08 (N*C*H$_2$); Yield: >99%, white solid, mp: 133-135 $^{\circ}$C; R_f = 0.50

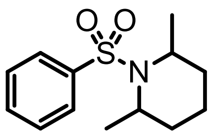
(Petrol/EtOAc 4:1).

7[S6]: 

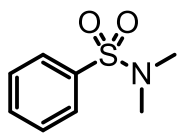
^{1}H NMR (400 MHz, $CDCl_3$, 298 K) δ = 7.86-7.84 (m, 2H, Ar*H*), 7.62-7.52 (m, 3H, Ar*H*), 7.17-7.12 (m, 2H, Ar*H*), 7.09-7.02 (m, 2H, Ar*H*), 4.27 (s, 2H, NCH_2), 3.38 (t, *J* = 5.6 Hz, 2H, NCH_2), 2.93 (t, *J* = 6.0 Hz, 2H, NCH_2CH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ = 136.30 (Ar*C*), 133.40 (Ar*C*), 132.97 (Ar*C*), 132.81 (Ar*C*), 131.15 (Ar*C*), 129.05 (Ar*C*), 128.78 (Ar*C*), 127.62 (Ar*C*), 126.73 (Ar*C*), 126.31 (Ar*C*), 47.47 (NCH_2), 43.68 (NCH_2), 28.77 (NCH_2CH_2); Yield: 61%, white solid, mp: 155-157 ^{o}C; R_f = 0.65 (Petrol/EtOAc 8:1).

8[S7]: 

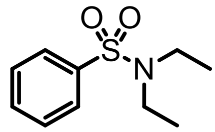
^{1}H NMR (400 MHz, $CDCl_3$, 298 K) δ = 7.79 (d, *J* = 6.8 Hz, 2H, Ar*H*), 7.53-7.44 (m, 3H, Ar*H*), 4.23-4.20 (m, 1H, NCH_2), 3.71-3.68 (m, 1H, NCH_2), 2.96 (td, *J* = 12.8 Hz, *J* = 2.0 Hz, 1H, NC*H*), 1.56-1.31 (m, 6H, $CH_2CH_2CH_2$), 1.03 (d, *J* = 6.8 Hz, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ = 141.20 (Ar*C*), 132.04 (Ar*C*), 128.87 (Ar*C*), 126.77 (Ar*C*), 48.40 (NCH_2), 40.20 (N*C*H), 30.22 (CH_2), 25.09 (CH_2), 18.02 (CH_2), 15.22 (CH_3); Yield: 74%, yellow oil, R_f = 0.67 (Petrol/EtOAc 8:1).

9[S8]: 

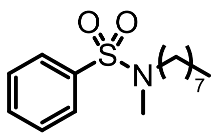
^{1}H NMR (400 MHz, $CDCl_3$, 298 K) δ = 7.83-7.80 (m, 2H, Ar*H*), 7.54-7.45 (m, 3H, Ar*H*), 4.22-4.15 (m, 2H, NC*H*), 1.46-1.33 (m, 12H, CH_2, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$, 298 K, ppm) δ = 141.88 (Ar*C*), 131.91 (Ar*C*), 128.89 (Ar*C*), 126.52 (Ar*C*), 47.98 (N*C*H), 29.57 (CH_2), 22.31 (CH_2), 13.28 (CH_3); Yield: 41%, white solid, mp: 99-102 ^{o}C; R_f = 0.65 (Petrol/EtOAc 8:1).

10[S9]: 

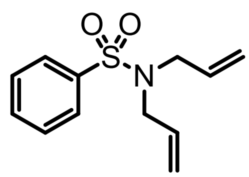
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.78-7.75 (m, 2H, Ar*H*), 7.62-7.58 (m, 1H, Ar*H*), 7.55-7.51 (m, 2H, Ar*H*), 2.69 (s, 6H, Me); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 135.41 (Ar*C*), 132.64 (Ar*C*), 128.95 (Ar*C*), 127.63 (Ar*C*), 37.85 (Me); Yield: 93%, white solid, mp: 45-46 ^{o}C; R_f = 0.45 (Petrol/EtOAc 8:1).

11[S1]: 

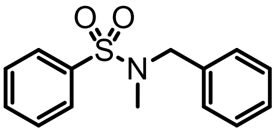
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.81-7.79 (m, 2H, Ar*H*), 7.57-7.52 (m, 1H, Ar*H*), 7.50-7.46 (m, 2H, Ar*H*), 3.26-3.21 (m, 4H, NCH_2), 1.11 (t, *J* = 7.2 Hz, 6H, CH_2CH_3); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 140.38 (Ar*C*), 132.19 (Ar*C*), 128.95 (Ar*C*), 126.93 (Ar*C*), 41.98 (NCH_2), 14.08 (CH_2CH_3); Yield: 85%, yellow solid, mp: 35-37 ^{o}C; R_f = 0.50 (Petrol/EtOAc 8:1).

12[S10]: 

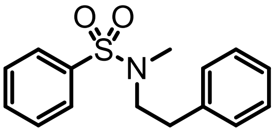
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.76-7.74 (m, 2H, Ar*H*), 7.57-7.47 (m, 3H, Ar*H*), 2.97 (t, *J* = 7.2 Hz, 2H, NCH_2), 2.69 (s, 3H, NCH_3), 1.50-1.45 (m, 2H, NCH_2CH_2), 1.25-1.23 (m, 10H), 0.85 (t, *J* = 6.4 Hz, 3H, CH_2CH_3); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 137.50 (Ar*C*), 132.33 (Ar*C*), 128.88 (Ar*C*), 127.18 (Ar*C*), 50.00 (NCH_2), 34.41 (NCH_3), 31.63 (NCH_2CH_2), 29.04, 27.45, 26.37, 22.50, 13.96; Yield: 89%, yellow oil, R_f = 0.70 (Petrol/EtOAc 8:1).

13[S1]: 

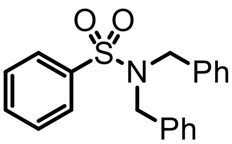
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.84-7.82 (m, 2H, Ar*H*), 7.59-7.49 (m, 3H, Ar*H*), 5.65-5.55 (m, 2H, CH_2=C*H*), 5.16-5.12 (m, 4H, NC*H*$_2$), 3.82 (d, *J* = 6.0 Hz, 4H, C*H*$_2$=CH); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 140.41 (Ar*C*), 132.49 (Ar*C*), 129.04 (Ar*C*), 127.07 (CH_2=*C*H), 119.01 (N*C*H$_2$), 49.28 (*C*H$_2$=CH); Yield: 50%, yellow oil, R_f = 0.55 (Petrol/EtOAc 8:1).

14[S11]: 

^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.87-7.84 (m, 2H, Ar*H*), 7.65-7.55 (m, 3H, Ar*H*), 7.36-7.28 (m, 5H, Ar*H*), 4.15 (s, 2H, NC*H*$_2$), 2.61 (s, 3H, Me); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 137.43 (Ar*C*), 135.55 (Ar*C*), 132.65 (Ar*C*), 129.12 (Ar*C*), 128.62 (Ar*C*), 128.33 (Ar*C*), 127.90 (Ar*C*), 127.42 (Ar*C*), 54.10 (N*C*H$_2$), 34.30 (Me); Yield: 89%, white solid, mp: 86-89 ^{o}C; R_f = 0.55 (Petrol/EtOAc 8:1).

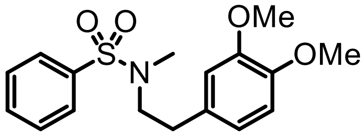
15[S12]: 

^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.78-7.75 (m, 2H, Ar*H*), 7.58-7.55 (m, 1H, Ar*H*), 7.52-7.49 (m, 2H, Ar*H*), 7.31-7.28 (m, 2H, Ar*H*), 7.24-7.17 (m, 3H, Ar*H*), 3.29-3.25 (m, 2H, NC*H*$_2$), 2.88-2.84 (m, 2H, NCH$_2$C*H*$_2$), 2.76 (s, 3H, Me); ^{13}CNMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 138.10 (Ar*C*), 132.42 (Ar*C*), 129.99 (Ar*C*), 128.93 (Ar*C*), 128.64 (Ar*C*), 127.11 (Ar*C*), 126.42 (Ar*C*), 51.64 (N*C*H$_2$), 35.00 (NCH$_2$*C*H$_2$), 34.64 (Me); Yield: 96%, colourless oil, R_f = 0.62 (Petrol/EtOAc 4:1).

16[S13]: 

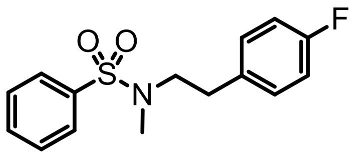
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.86-7.84 (m, 2H, Ar*H*), 7.61-7.58 (m, 1H, Ar*H*), 7.53-7.49 (m, 2H, Ar*H*), 7.22-7.21 (m, 6H, Ar*H*), 7.05-7.03 (m, 4H, Ar*H*), 4.34 (s, 4H, NC*H*$_2$); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 135.52 (Ar*C*), 132.48 (Ar*C*), 129.10 (Ar*C*), 128.54 (Ar*C*), 128.42 (Ar*C*), 127.66 (Ar*C*), 127.14 (Ar*C*), 50.42 (N*C*H$_2$); Yield: 54%, white solid, mp:

72-74 ^{o}C; R_f = 0.70 (Petrol/EtOAc 4:1).



17[S14]**:**

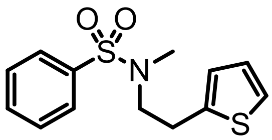
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.74-7.72 (m, 2H, Ar*H*), 7.56-7.52 (m, 1H, Ar*H*), 7.48-7.41 (m, 2H, Ar*H*), 6.77-6.68 (m, 3H, Ar*H*), 3.82 (d, *J* = 4.8 Hz, 6H, OCH_3), 3.24-3.21 (m, 2H, NCH_2), 2.78 (t, *J* = 8.0 Hz, 2H, NCH_2CH_2), 2.72 (s, 3H, NCH_3); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 148.75 (Ar*C*), 147.51 (Ar*C*), 137.56 (Ar*C*), 132.35 (Ar*C*), 130.60 (Ar*C*), 128.87 (Ar*C*), 127.05 (Ar*C*), 120.56 (Ar*C*), 111.85 (Ar*C*), 111.16 (Ar*C*), 55.69 (OCH_3), 51.69 (NCH_2), 34.95 (NCH_2CH_2), 34.19 (NCH_3); Yield: 83%, yellow oil, R_f = 0.60 (Petrol/EtOAc 2:1).



18 [S14]**:**

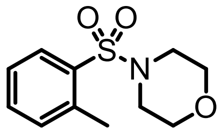
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.75 (d, *J* = 7.6 Hz, 2H, Ar*H*), 7.58-7.55 (m, 1H, Ar*H*), 7.51-7.48 (m, 2H, Ar*H*), 7.15-7.12 (m, 2H, Ar*H*), 6.96 (t, *J* = 8.4 Hz, 2H, Ar*H*), 3.23 (t, *J* = 11.6 Hz, 2H, NCH_2); 2.82 (t, *J* = 8.0 Hz, 2H, NCH_2CH_2); 2.74 (s, 3H, Me); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 162.76 (Ar*C*), 160.33 (Ar*C*), 137.62 (Ar*C*), 133.83 (Ar*C*), 132.50 (Ar*C*), 130.18 (Ar*C*), 130.10 (Ar*C*), 128.99 (Ar*C*), 127.13 (Ar*C*), 115.35 (Ar*C*), 115.14 (Ar*C*), 51.64 (NCH_2), 35.06 (NCH_2CH_2), 33.84 (Me);

^{19}F NMR ($CDCl_3$, 376 MHz, 298 K): δ = -116.43; Yield: 95%, yellow oil; R_f = 0.65 (Petrol/EtOAc 4:1).

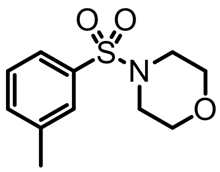


19[S15]**:**

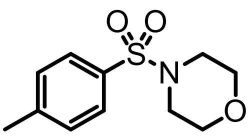
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.78-7.76 (m, 2H, Ar*H*), 7.60-7.56 (m, 1H, Ar*H*), 7.53-7.49 (m, 2H, Ar*H*), 7.14 (dd, *J* = 4.8 Hz, *J* = 0.8 Hz, 1H, Ar*H*), 6.93-6.91 (m, 1H, Ar*H*), 6.84 (d, *J* = 3.6 Hz, 1H, Ar*H*), 3.30 (t, *J* = 7.2 Hz, 2H, NCH_2), 3.07 (t, *J* = 8.0 Hz, 2H, NCH_2CH_2), 2.76 (s, 3H, Me); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 140.13 (Ar*C*), 137.53 (Ar*C*), 132.50 (Ar*C*), 128.98 (Ar*C*), 127.11 (Ar*C*), 126.85 (Ar*C*), 125.37 (Ar*C*), 123.82 (Ar*C*), 51.66 (N*C*H_2), 35.15 (NCH_2*C*H_2), 28.80 (Me); Yield: 95%, yellow oil, R_f = 0.55 (Petrol/EtOAc 4:1).

20a[S1]**:** 

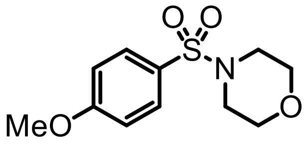
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.91-7.88 (m, 1H, Ar*H*), 7.50-7.46 (m, 1H, Ar*H*), 7.35-7.32 (m, 2H, Ar*H*), 3.72 (t, *J* = 4.4 Hz, 4H, OC*H_2*), 3.15 (t, *J* = 4.8 Hz, 4H, NC*H_2*), 2.65 (s, 3H, Me); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 138.05 (Ar*C*), 134.88 (Ar*C*), 133.00 (Ar*C*), 132.83 (Ar*C*), 130.33 (Ar*C*), 126.08 (Ar*C*), 66.21 (O*C*H_2), 45.22 (N*C*H_2), 20.74 (Me); Yield: 98%, white solid, mp: 89-91 ^{o}C; R_f = 0.54 (Petrol/EtOAc 4:1).

20b[S1]**:** 

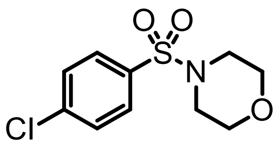
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.56-7.55 (m, 2H, Ar*H*), 7.44-7.43 (m, 2H, Ar*H*), 3.75 (t, *J* = 4.4 Hz, 4H, OC*H_2*), 3.00 (t, *J* = 4.8 Hz, 4H, NC*H_2*), 2.45 (s, 3H, Me); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 139.26 (Ar*C*), 134.77 (Ar*C*), 133.78 (Ar*C*), 128.87 (Ar*C*), 127.99 (Ar*C*), 124.91 (Ar*C*), 65.97 (O*C*H_2), 45.90 (N*C*H_2), 21.27 (Me); Yield: >99%, white solid, mp: 121-123 ^{o}C; R_f = 0.54 (Petrol/EtOAc 4:1).

20c[S1]**:** 

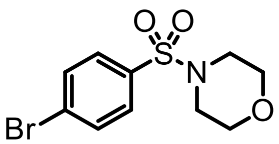
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.64 (d, *J* = 8.0 Hz, 2H, Ar*H*), 7.35 (d, *J* = 8.0 Hz, 2H, Ar*H*), 3.74 (t, *J* = 4.4 Hz, 4H, OC*H_2*), 2.98 (t, *J* = 4.4 Hz, 4H, NC*H_2*), 2.45 (s, 3H, Me); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 143.86 (Ar*C*), 132.00 (Ar*C*), 129.67 (Ar*C*), 127.81 (Ar*C*), 66.01 (O*C*H_2), 45.92 (N*C*H_2), 21.45 (Me); Yield: >99%, white solid, mp: 149-151 ^{o}C; R_f = 0.55 (Petrol/EtOAc 4:1).

21[S1]: 

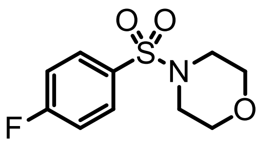
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.70-7.68 (m, 2H, Ar*H*), 7.02-7.00 (m, 2H, Ar*H*), 3.88 (s, 3H, OMe), 3.74 (t, *J* = 4.8 Hz, 4H, OCH_2), 2.98 (t, *J* = 4.4 Hz, 4H, NCH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 163.15 (Ar*C*), 129.88 (Ar*C*), 126.53 (Ar*C*), 114.21 (Ar*C*), 65.97 (O*C*H_2), 55.56 (O*C*H_3), 45.92 (N*C*H_2); Yield: 97%, white solid, mp: 112-114 ^{o}C; R_f = 0.65 (Petrol/EtOAc 2:1).

22a[S1]: 

^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.71-7.69 (m, 2H, Ar*H*), 7.55-7.53 (m, 2H, Ar*H*), 3.75 (t, *J* = 4.8 Hz, 4H, OCH_2), 3.00 (t, *J* = 4.8 Hz, 4H, NCH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 139.63 (Ar*C*), 133.67 (Ar*C*), 129.41 (Ar*C*), 129.15 (Ar*C*), 65.95 (O*C*H_2), 45.86 (N*C*H_2); Yield: 99%, white solid, mp: 149-151 ^{o}C; R_f = 0.45 (Petrol/EtOAc 4:1).

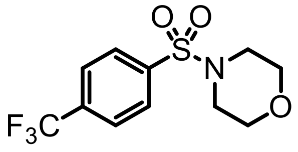
22b[S1]: 

^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.70 (d, *J* = 8.8 Hz, 2H, Ar*H*), 7.62 (d, *J* = 8.8 Hz, 2H, Ar*H*), 3.75 (t, *J* = 4.8 Hz, 4H, OCH_2), 3.00 (t, *J* = 4.8 Hz, 4H, NCH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 134.20 (Ar*C*), 132.41 (Ar*C*), 129.24 (Ar*C*), 128.15 (Ar*C*), 65.97 (O*C*H_2), 45.87 (N*C*H_2); Yield: 99%, white solid, mp: 154-157 ^{o}C; R_f = 0.42 (Petrol/EtOAc 4:1).

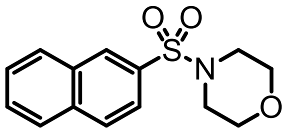
22c[S1]: 

^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.80-7.76 (m, 2H, Ar*H*), 7.24-7.22 (m, 2H, Ar*H*), 3.75 (t, *J* = 4.8 Hz, 4H, OCH_2), 3.00 (t, *J* = 4.8 Hz, 4H, NCH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 166.60, 164.05 (Ar*C*), 131.23 (Ar*C*), 130.52 (Ar*C*), 130.43 (Ar*C*), 116.51 (Ar*C*), 116.28 (Ar*C*), 65.99 (O*C*H_2), 45.91 (N*C*H_2); ^{19}F NMR ($CDCl_3$, 376 MHz, 298 K): δ = -104.51; Yield:

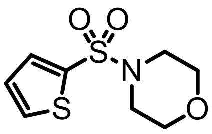
93%, white solid, mp: 104-105 °C; R_f = 0.45 (Petrol/EtOAc 4:1).

23[S1]: 

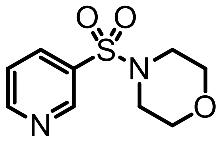
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.89 (d, J = 8.4 Hz, 2H, ArH), 7.83 (d, J = 8.4 Hz, 2H, ArH), 3.76 (t, J = 4.8 Hz, 4H, OCH_2), 3.04 (t, J = 4.4 Hz, 4H, NCH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 139.00 (ArC), 134.92 (ArC), 134.59 (ArC), 128.28 (ArC), 126.31 (ArC), 126.28 (ArC), 66.01 (OCH$_2$), 45.89 (NCH$_2$); ^{19}F NMR ($CDCl_3$, 376 MHz, 298 K): δ = -63.15; Yield: 88%, white solid, mp: 131-133 °C; R_f = 0.44 (Petrol/EtOAc 4:1).

24[S1]: 

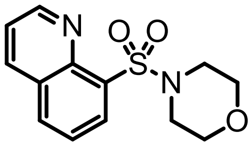
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 8.34 (s, 1H, ArH), 8.01-7.99 (m, 2H, ArH), 7.95-7.93 (m, 1H, ArH), 7.76-7.74 (m, 1H, ArH), 7.70-7.62 (m, 2H, ArH), 3.75 (t, J = 4.8 Hz, 4H, OCH_2), 3.07 (t, J = 4.8 Hz, 4H, NCH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 134.97 (ArC), 132.34 (ArC), 132.29 (ArC), 132.18 (ArC), 129.30 (ArC), 129.23 (ArC), 128.97 (ArC), 127.94 (ArC), 127.67 (ArC), 122.95 (ArC), 66.11 (OCH$_2$), 46.06 (NCH$_2$); Yield: 93%, white solid, mp: 159-161 °C; R_f = 0.62 (Petrol/EtOAc 4:1).

25[S16]: 

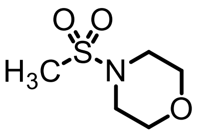
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 7.65 (dd, J = 5.2 Hz, J = 1.2 Hz, 1H, ArH), 7.55 (dd, J = 3.6 Hz, J = 1.2 Hz, 1H, ArH), 7.19-7.17 (m, 1H, ArH), 3.78 (t, J = 4.8 Hz, 4H, OCH_2), 3.06 (t, J = 4.8 Hz, 4H, NCH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 135.27 (ArC), 132.75 (ArC), 132.46 (ArC), 127.76 (ArC), 65.95 (OCH$_2$), 45.95 (NCH$_2$); Yield: 98%, white solid, mp: 106-107 °C; R_f = 0.52 (Petrol/EtOAc 4:1).

26[S17]**:** 

^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 8.99 (d, *J* = 2.0 Hz, 1H, Ar*H*), 8.86 (dd, *J* = 4.8 Hz, *J* = 1.6 Hz, 1H, Ar*H*), 8.06-8.04 (m, 1H, Ar*H*), 7.54-7.50 (m, 1H, Ar*H*), 3.77 (t, *J* = 4.8 Hz, 4H, OCH_2), 3.05 (t, *J* = 4.8 Hz, 4H, NCH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 153.58 (Ar*C*), 148.42 (Ar*C*), 135.34 (Ar*C*), 131.94 (Ar*C*), 123.71 (Ar*C*), 65.86 (OCH_2), 45.73 (NCH_2); Yield: 83%, white solid, mp: 115-117 ^{o}C; R_f = 0.48 (Petrol/EtOAc 1:1).

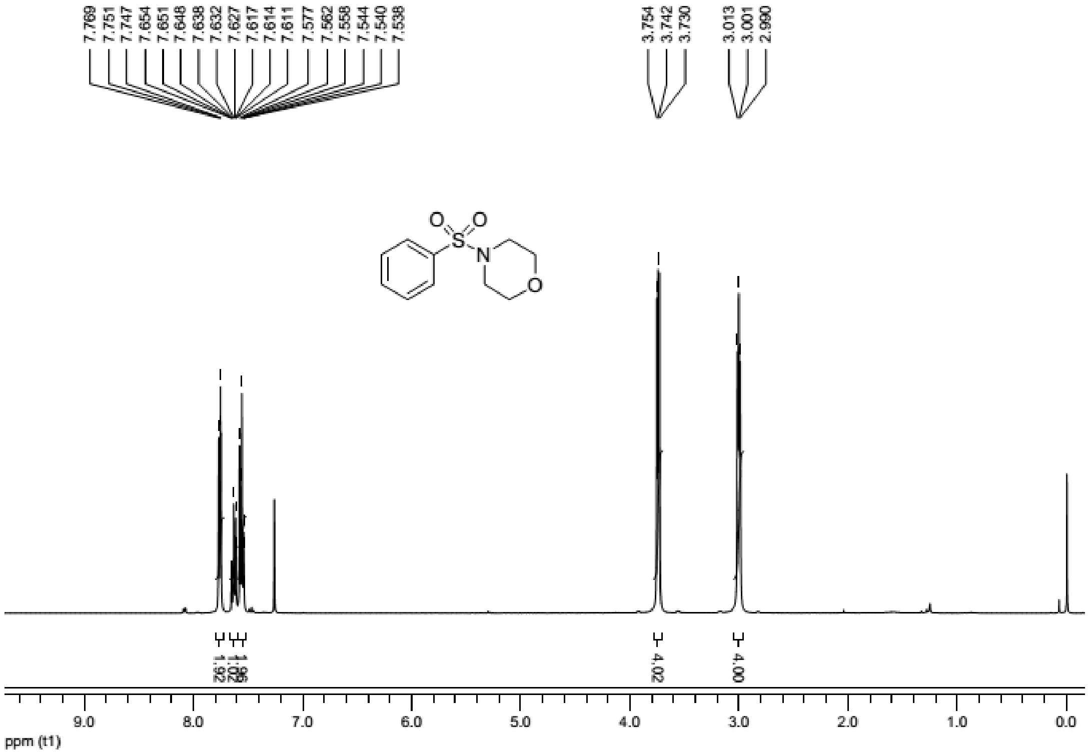
27[S1]**:** 

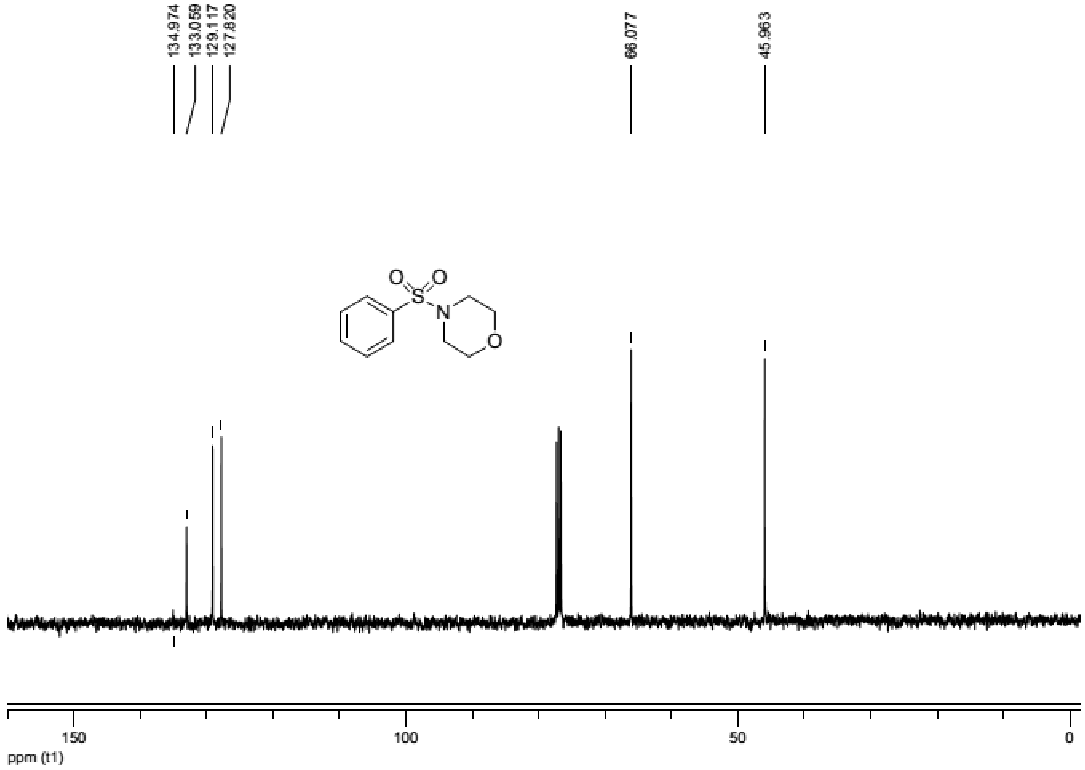
^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 9.06 (dd, *J* = 4.0 Hz, *J* = 1.6 Hz, 1H, Ar*H*), 8.48 (dd, *J* = 7.6 Hz, *J* = 1.2 Hz, 1H, Ar*H*), 8.25 (dd, *J* = 8.4 Hz, *J* = 1.6 Hz, 1H, Ar*H*), 8.05 (dd, *J* = 8.4 Hz, *J* = 1.2 Hz, 1H, Ar*H*), 7.62 (t, *J* = 7.6Hz, 1H, Ar*H*), 7.55-7.52 (m, 1H, Ar*H*), 3.71 (t, *J* = 4.4 Hz, 4H, OCH_2), 3.45 (t, *J* = 4.8 Hz, 4H, NCH_2); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 151.18 (Ar*C*), 144.16 (Ar*C*), 136.43 (Ar*C*), 133.60 (Ar*C*), 133.30 (Ar*C*), 128.98 (Ar*C*), 125.48 (Ar*C*), 122.06 (Ar*C*), 66.87 (OCH_2), 46.38 (NCH_2); Yield: 72%, white solid, mp: 183-187 ^{o}C; R_f = 0.55 (Petrol/EtOAc 1:1).

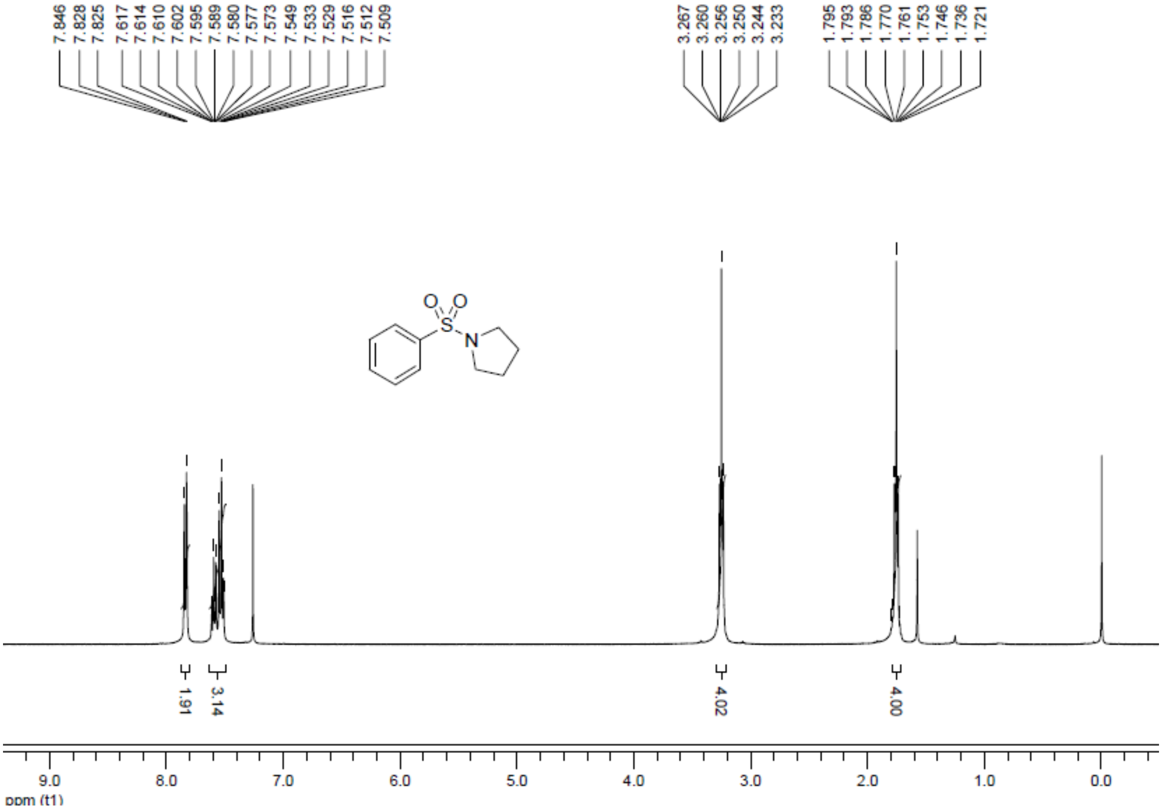
28a[S18]**:** 

^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ = 3.77 (t, *J* = 4.8 Hz, 4H, OCH_2), 3.20 (t, *J* = 4.8 Hz, 4H, NCH_2), 2.78 (s, 3H, Me); ^{13}C NMR (100 MHz, $CDCl_3$, 298K, ppm) δ = 66.25 (OCH_2), 45.80 (NCH_2), 33.93 (Me); white solid, Yield: 32%, mp: 92-93 ^{o}C; R_f = 0.35 (Petrol/EtOAc 4:1).

4. ^{1}H NMR, ^{13}C NMR and MS spectra for important compounds



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7.533
7.529
7.516
7.512
7.509
3.267
3.260
3.256
3.250
3.244
3.233
1.795
1.793
1.786
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1.746
1.736
1.721
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3.14
4.02
4.00
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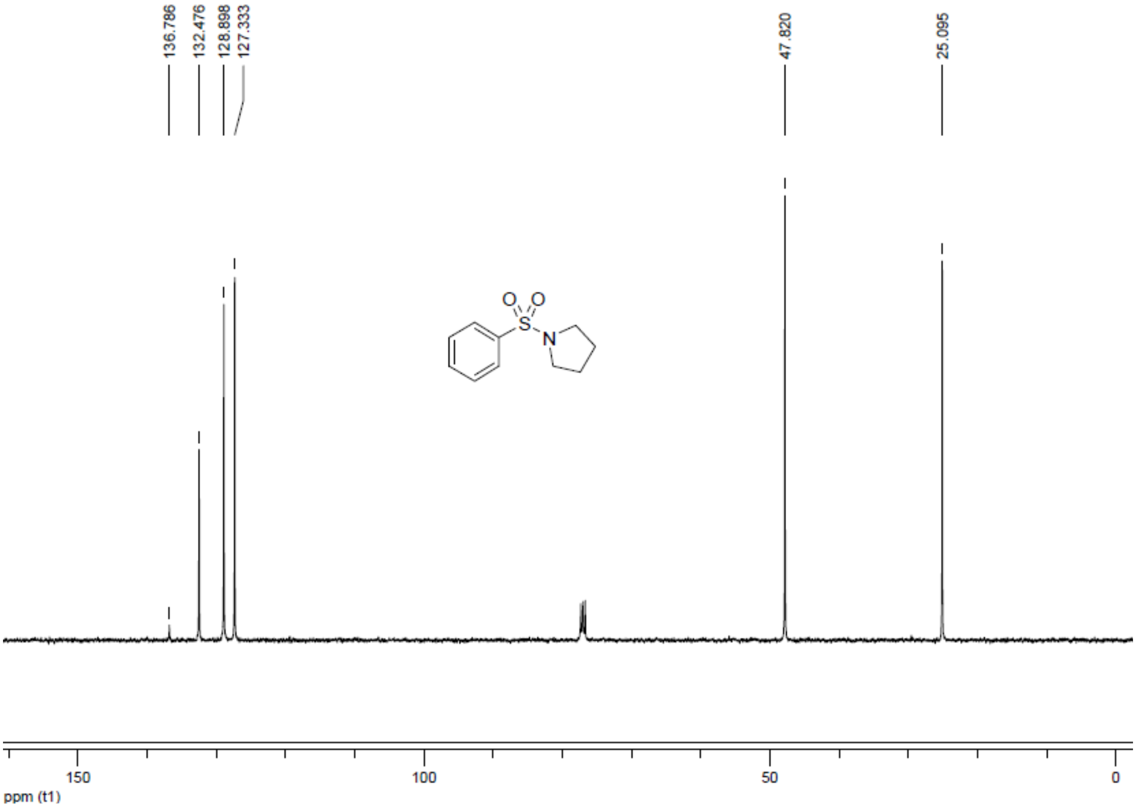
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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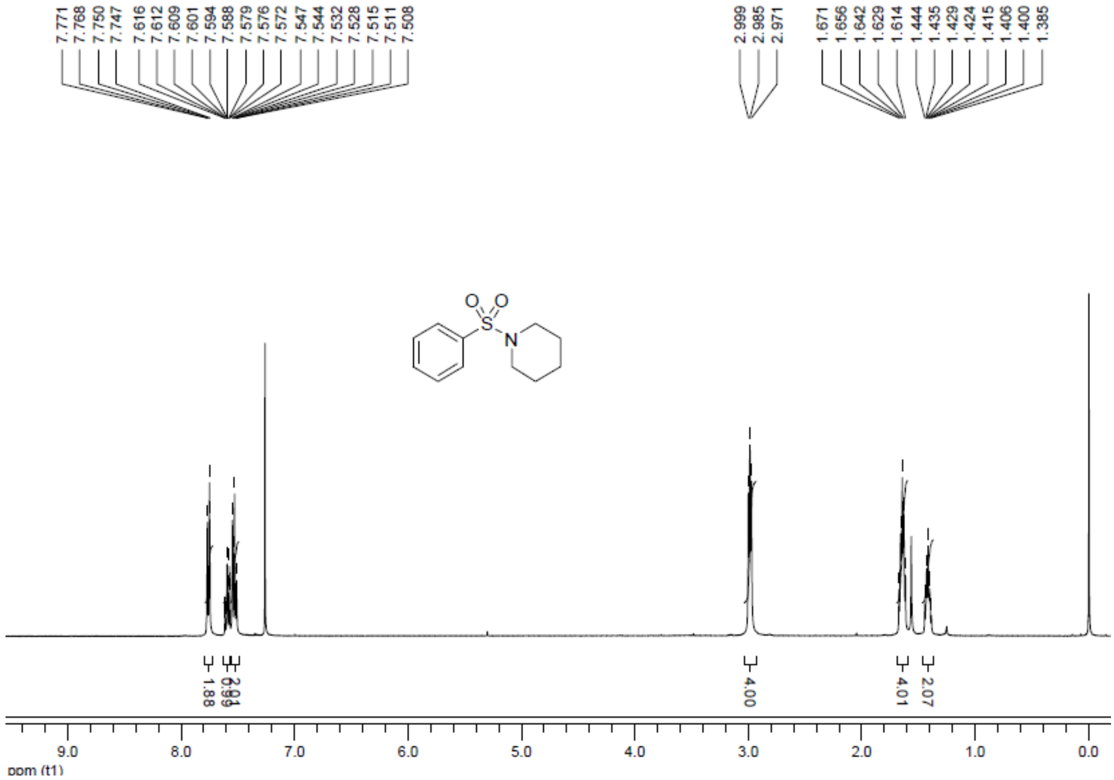
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7.511
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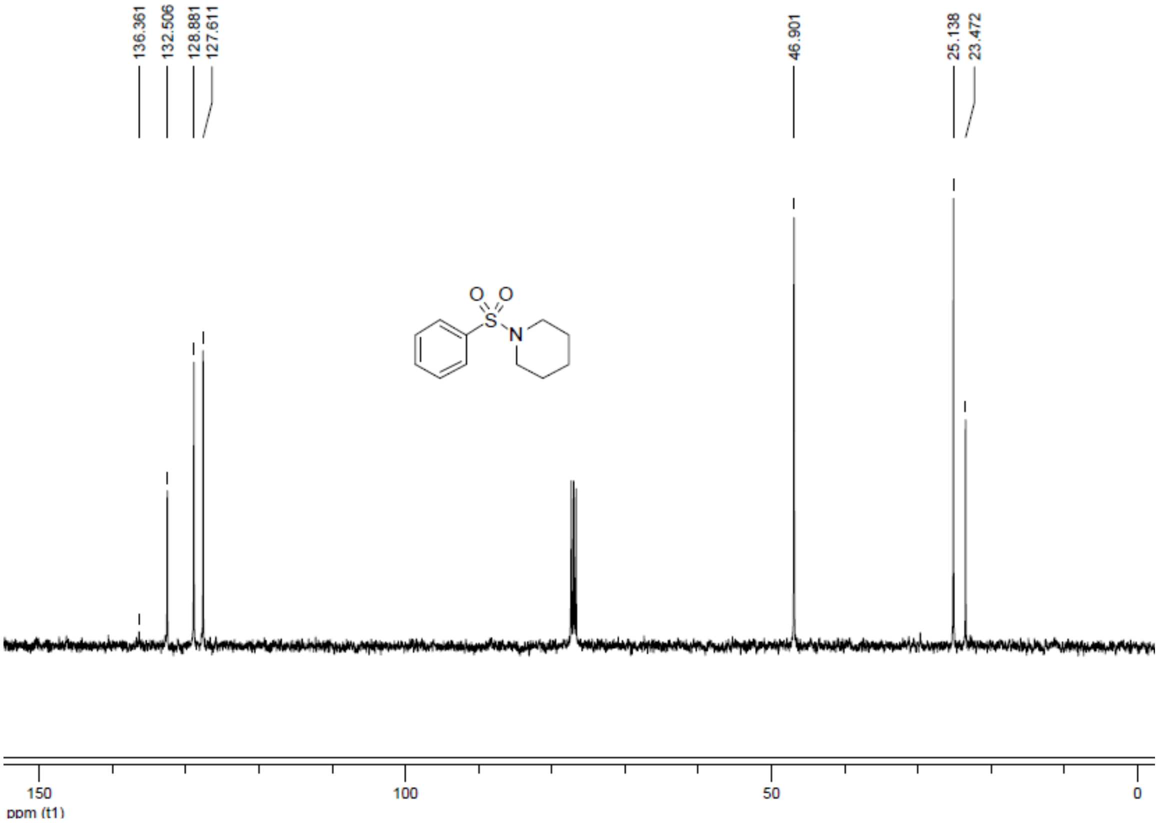
0.0
ppm (t1)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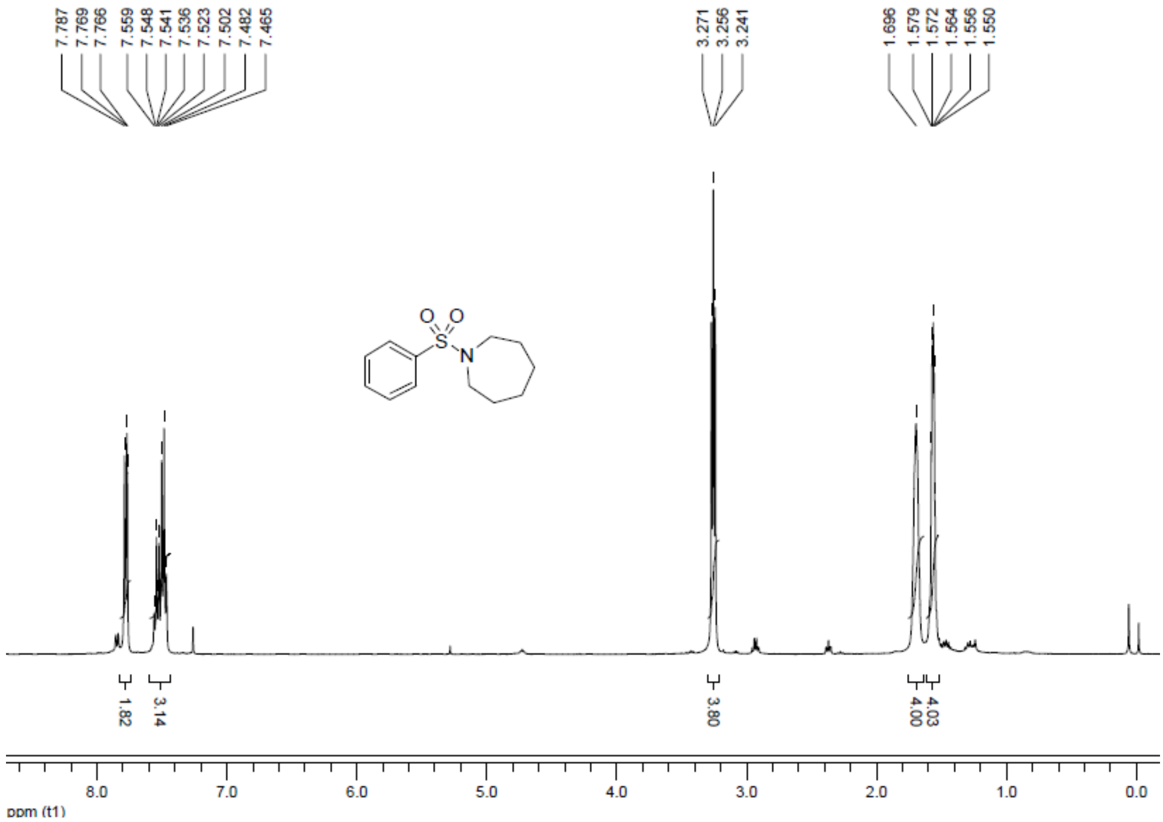
ppm (t1)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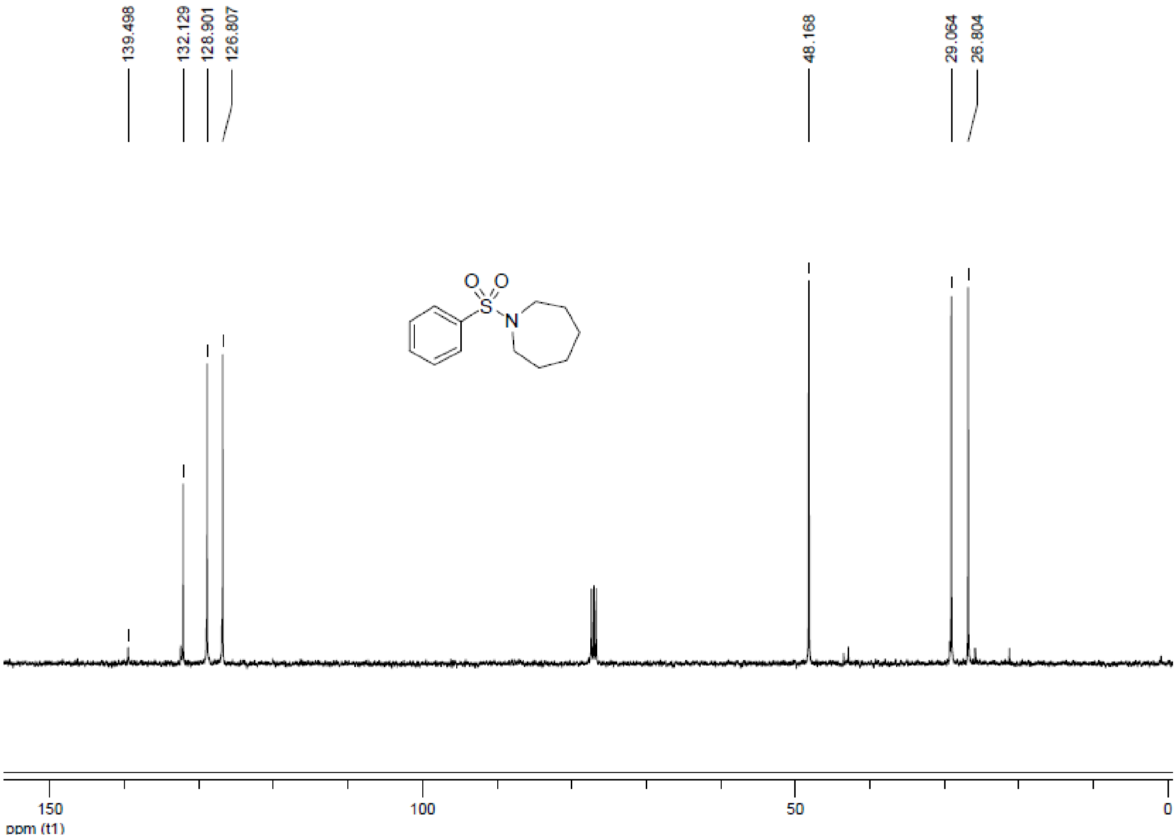
ppm (t1)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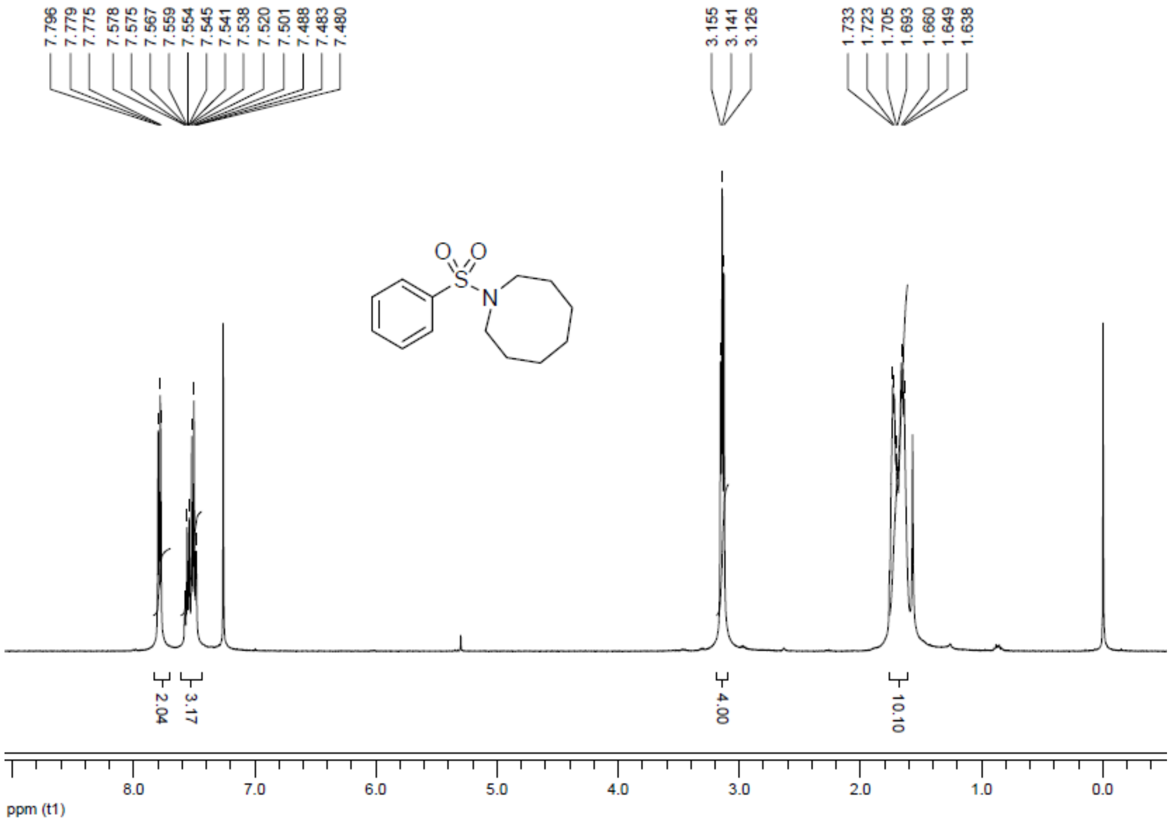
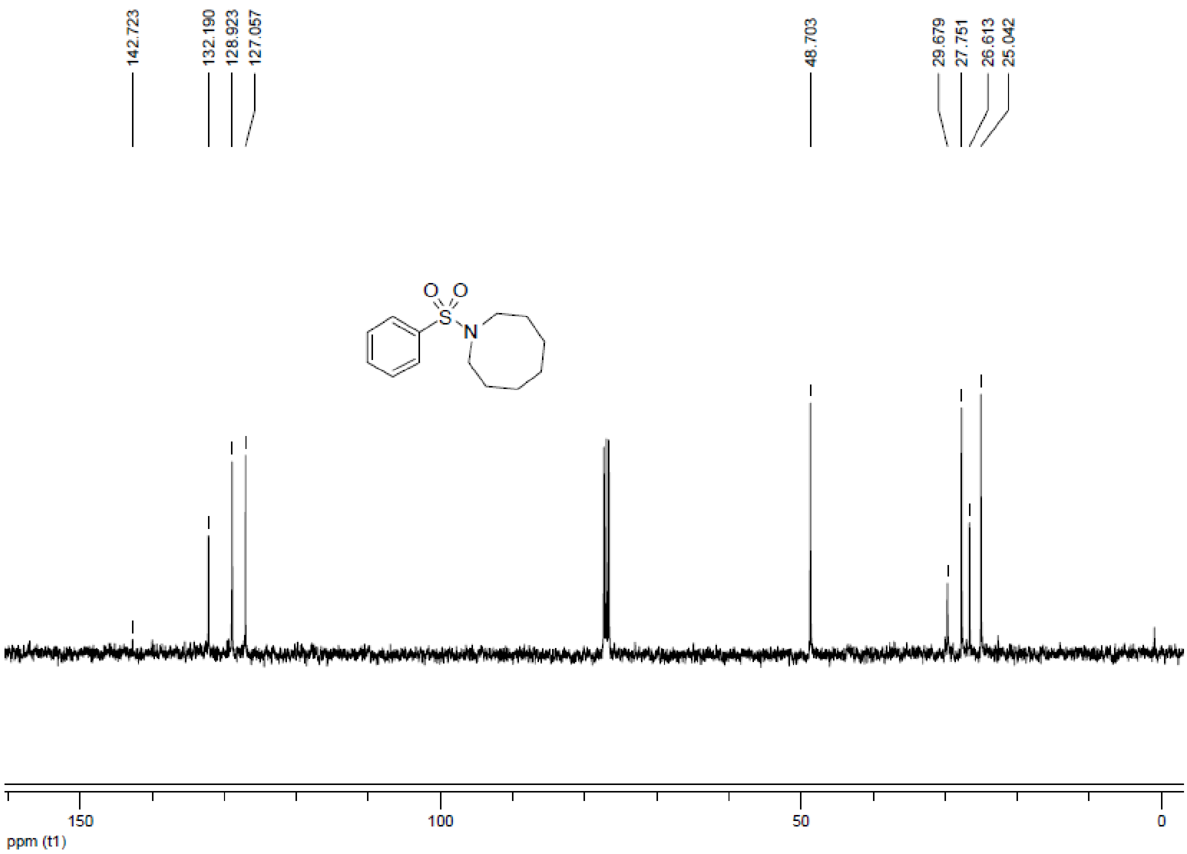
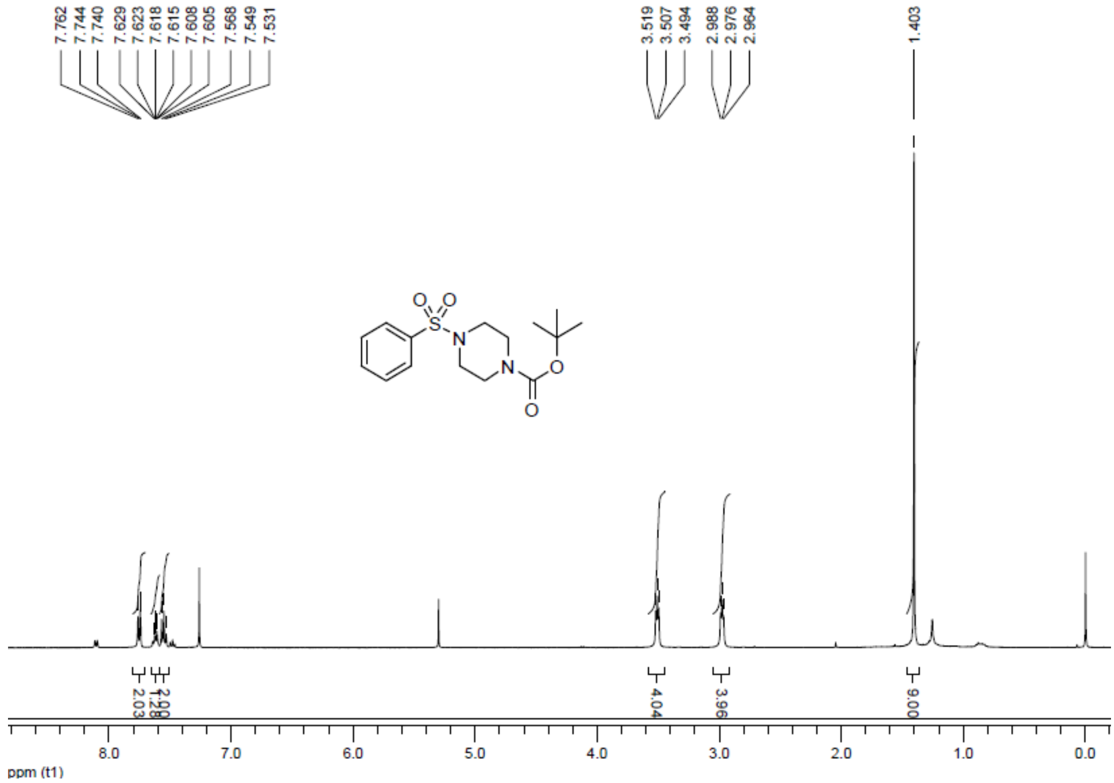
ppm (t1)



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3.507
3.494
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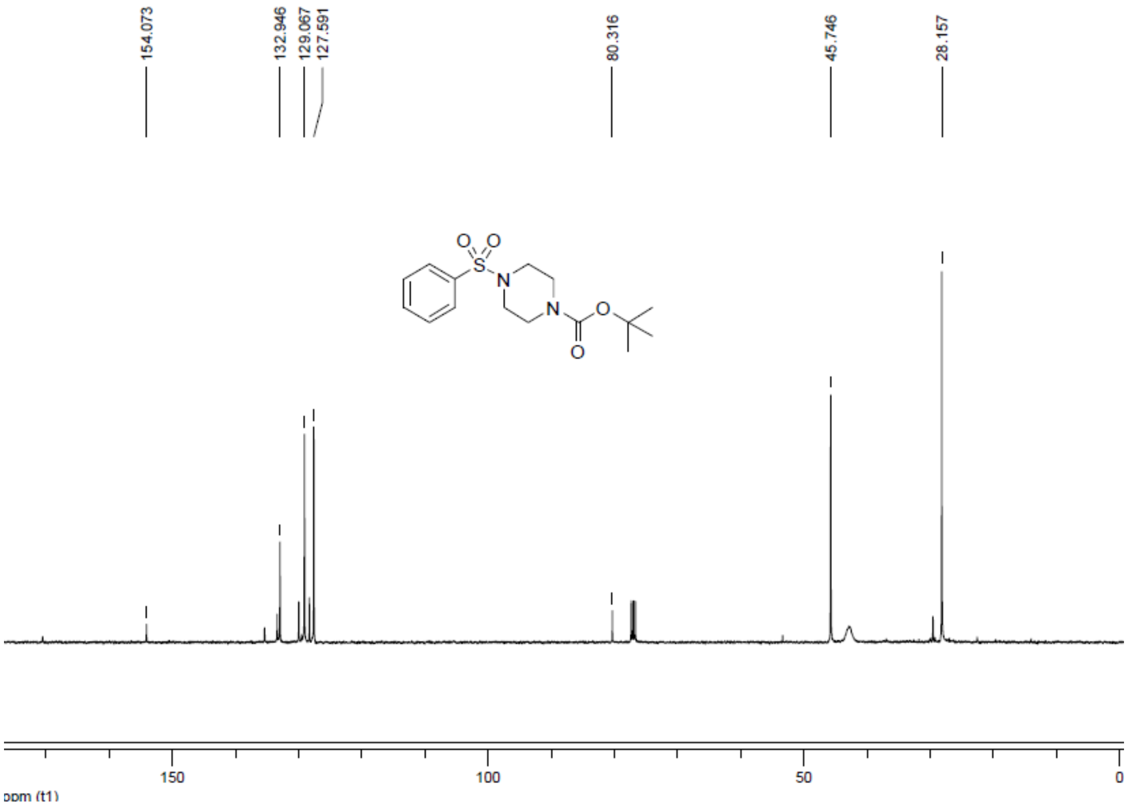
ppm (t1)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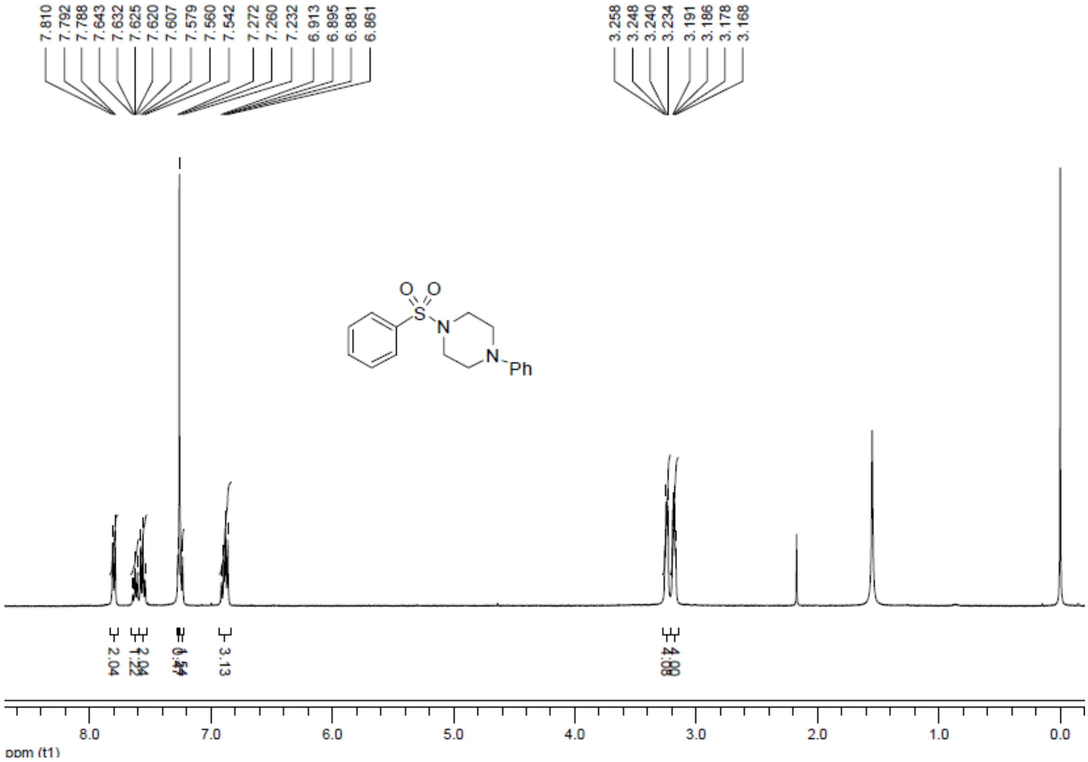
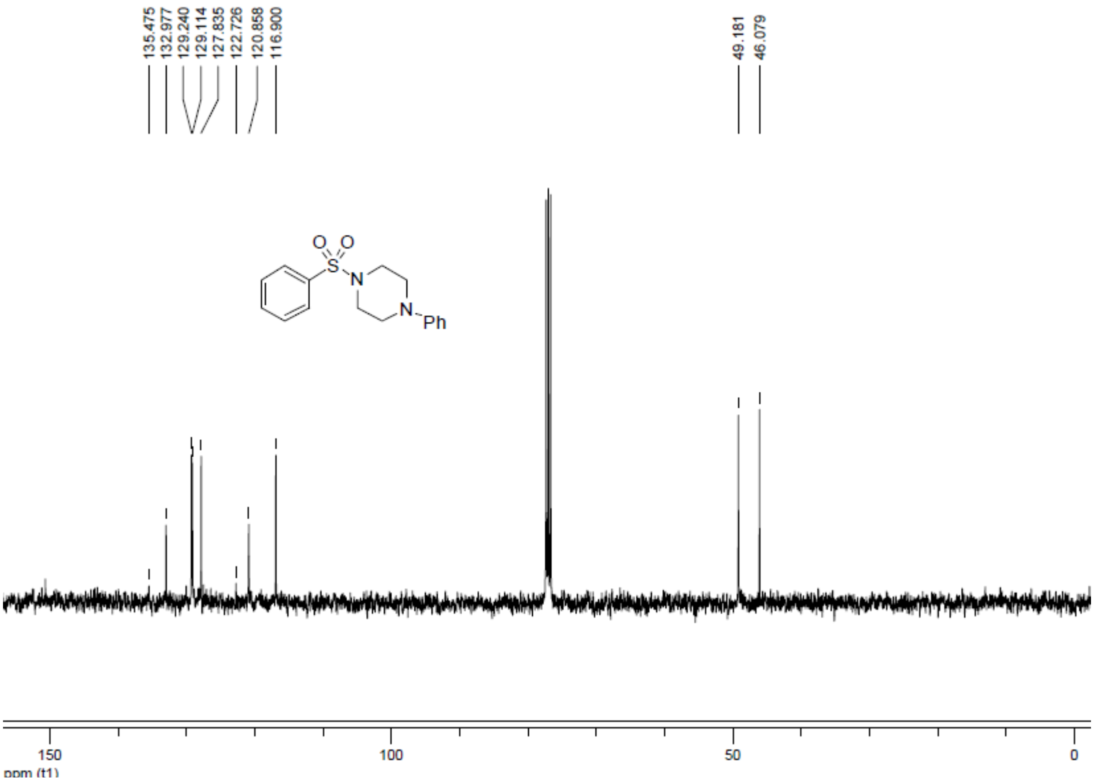
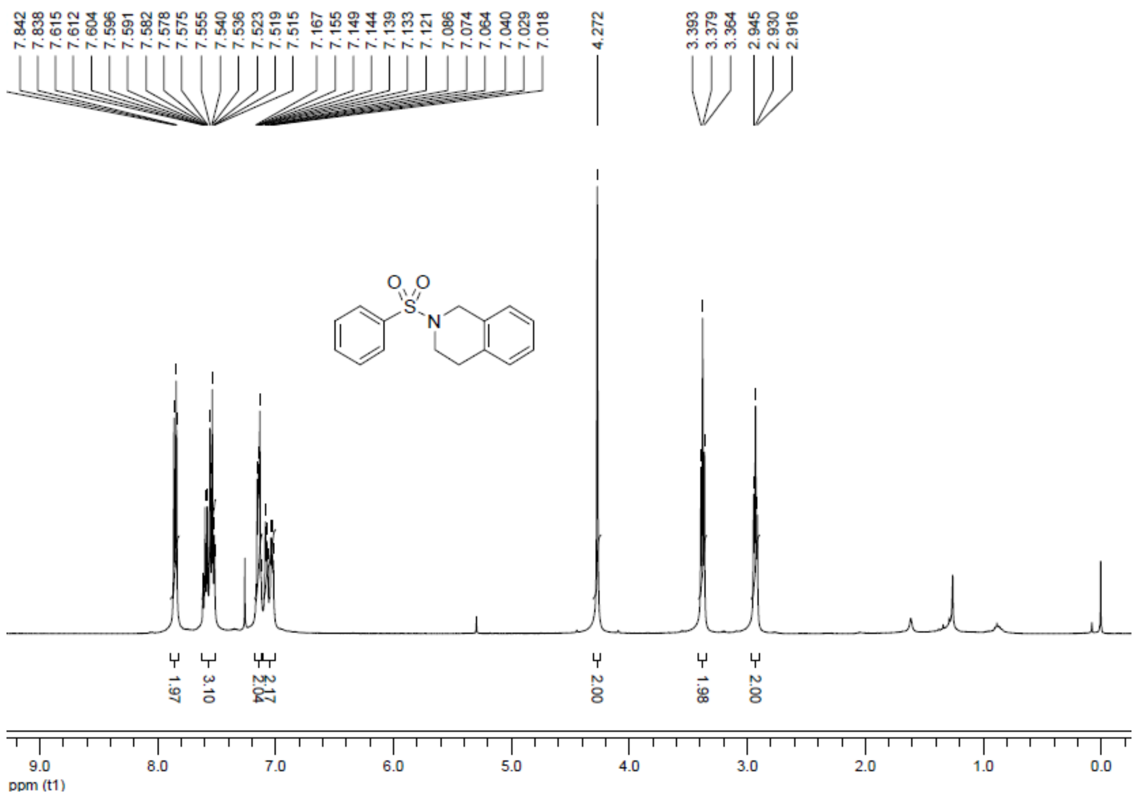
50
0
ppm (t1)







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7.582
7.578
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7.536
7.523
7.519
7.515
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7.155
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7.144
7.139
7.133
7.121
7.086
7.074
7.064
7.040
7.029
7.018
4.272
3.393
3.379
3.364
2.945
2.930
2.916
1.97
3.10
2.04
2.17
2.00
1.98
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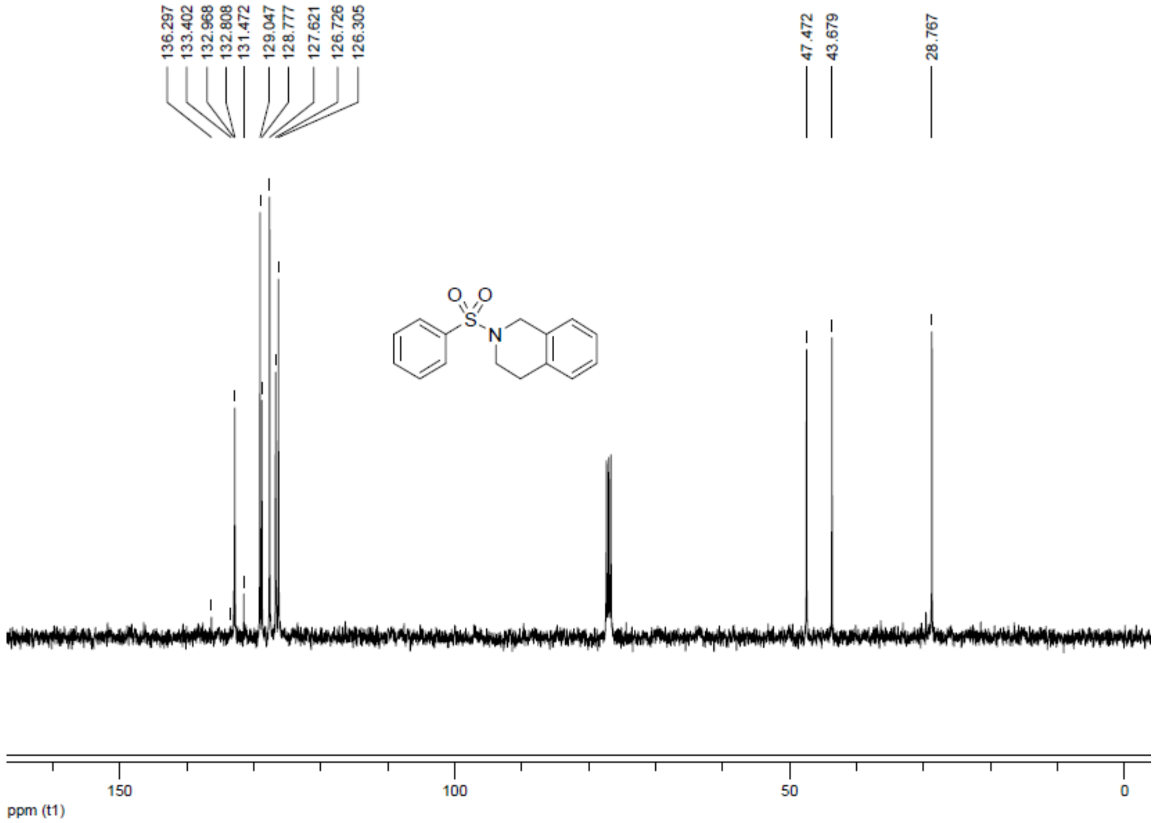

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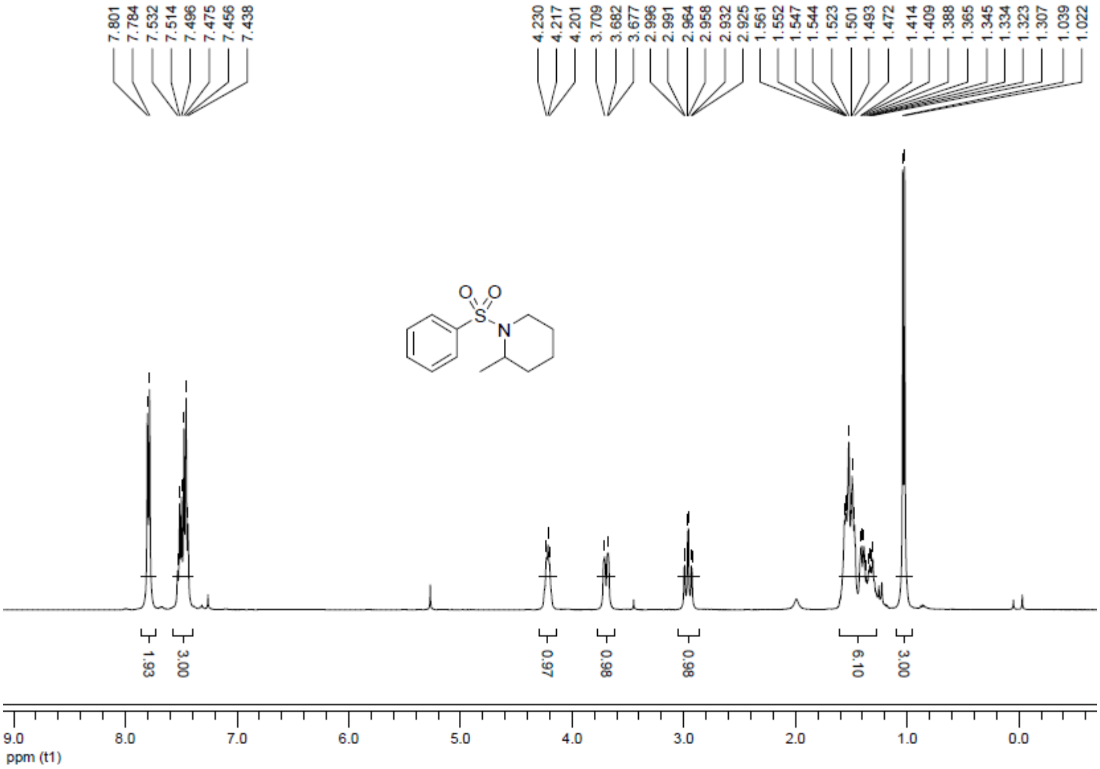
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7.438
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4.201
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3.682
3.677
2.996
2.991
2.964
2.958
2.932
2.925
1.561
1.552
1.547
1.544
1.523
1.501
1.493
1.472
1.414
1.409
1.388
1.365
1.345
1.334
1.323
1.307
1.039
1.022
O
O
S
N
1.93
3.00
0.97
0.98
0.98
6.10
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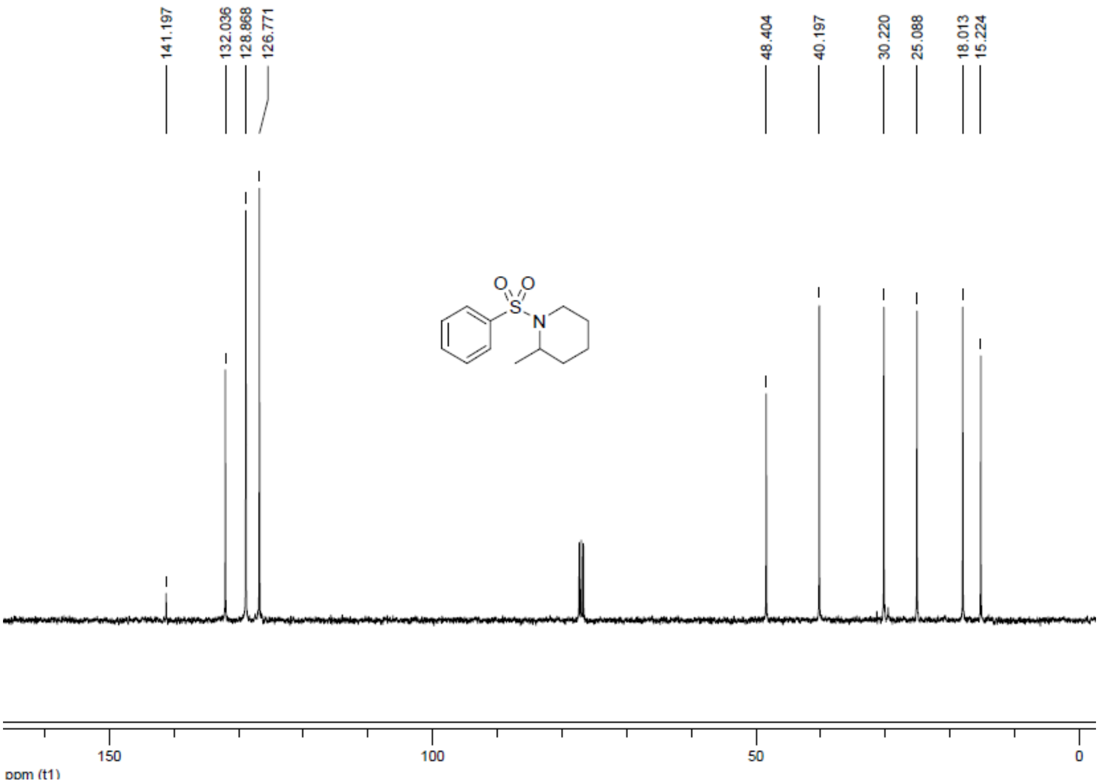
ppm (t1)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128.868
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25.088
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N
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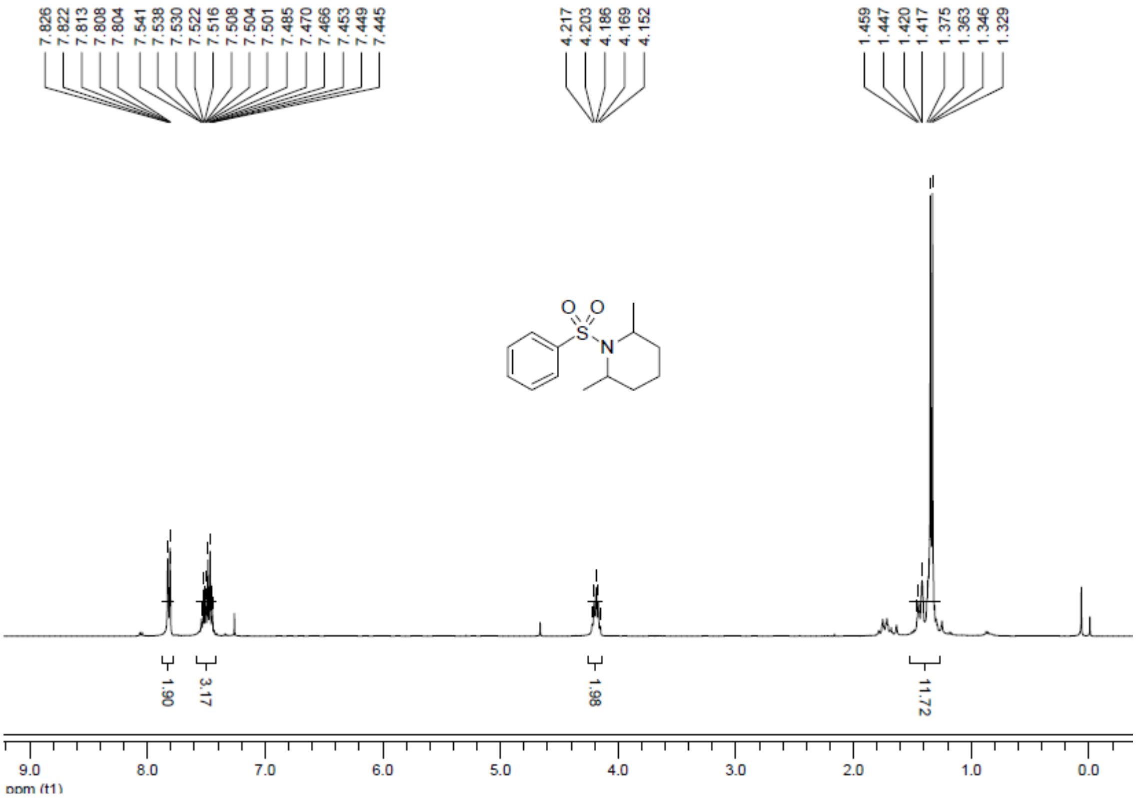
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7.530
7.522
7.516
7.508
7.504
7.501
7.485
7.470
7.466
7.453
7.449
7.445
4.217
4.203
4.186
4.169
4.152
1.459
1.447
1.420
1.417
1.375
1.363
1.346
1.329
1.90
3.17
1.98
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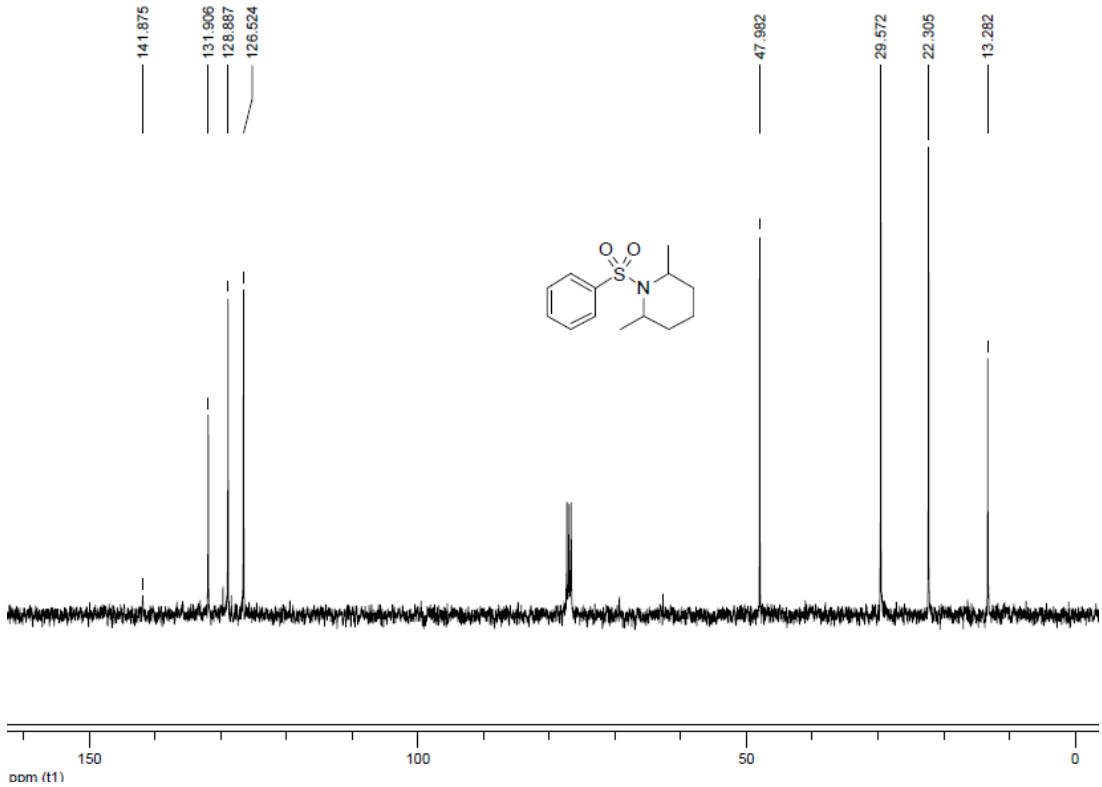
ppm (t1)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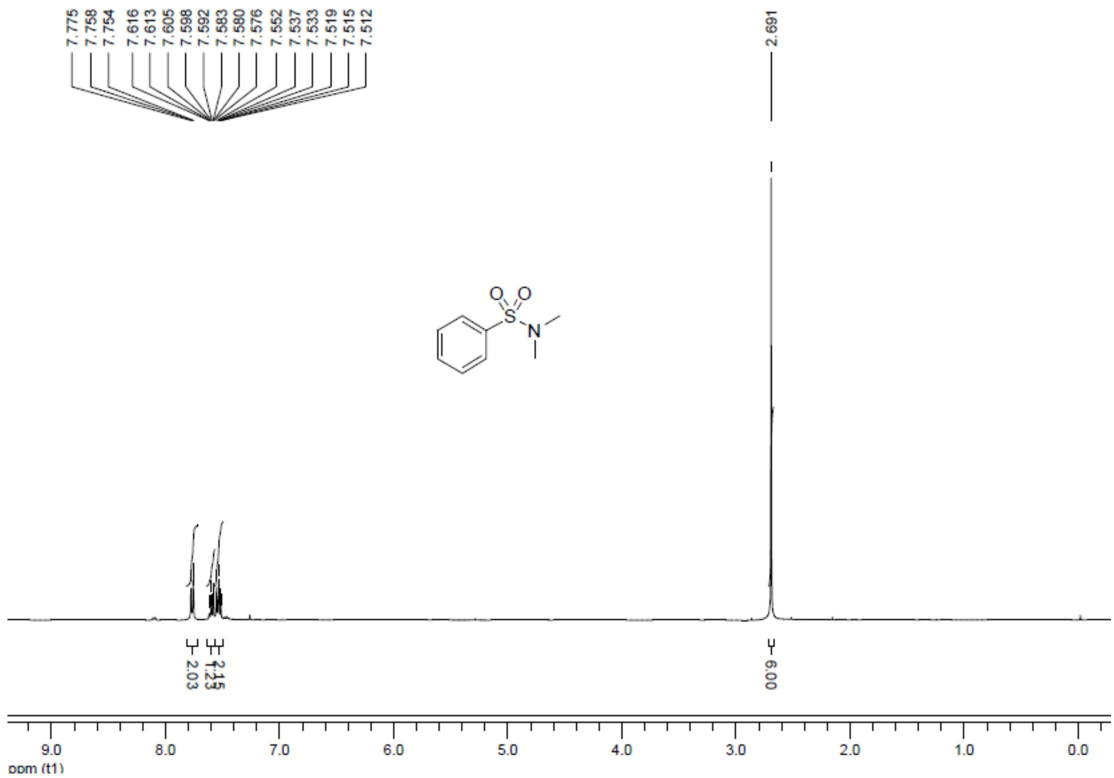
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7.592
7.583
7.580
7.576
7.552
7.537
7.533
7.519
7.515
7.512
2.691
2.03
1.25
2.15
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6.0
5.0
4.0
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2.0
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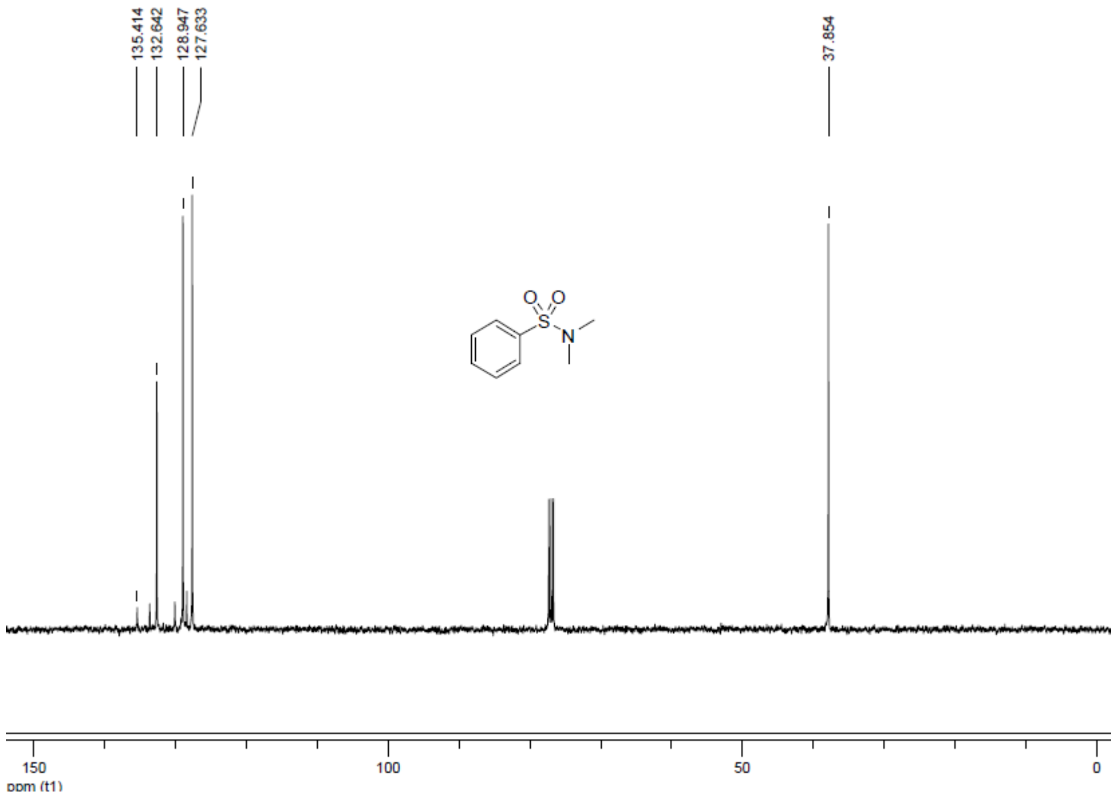
ppm (t1)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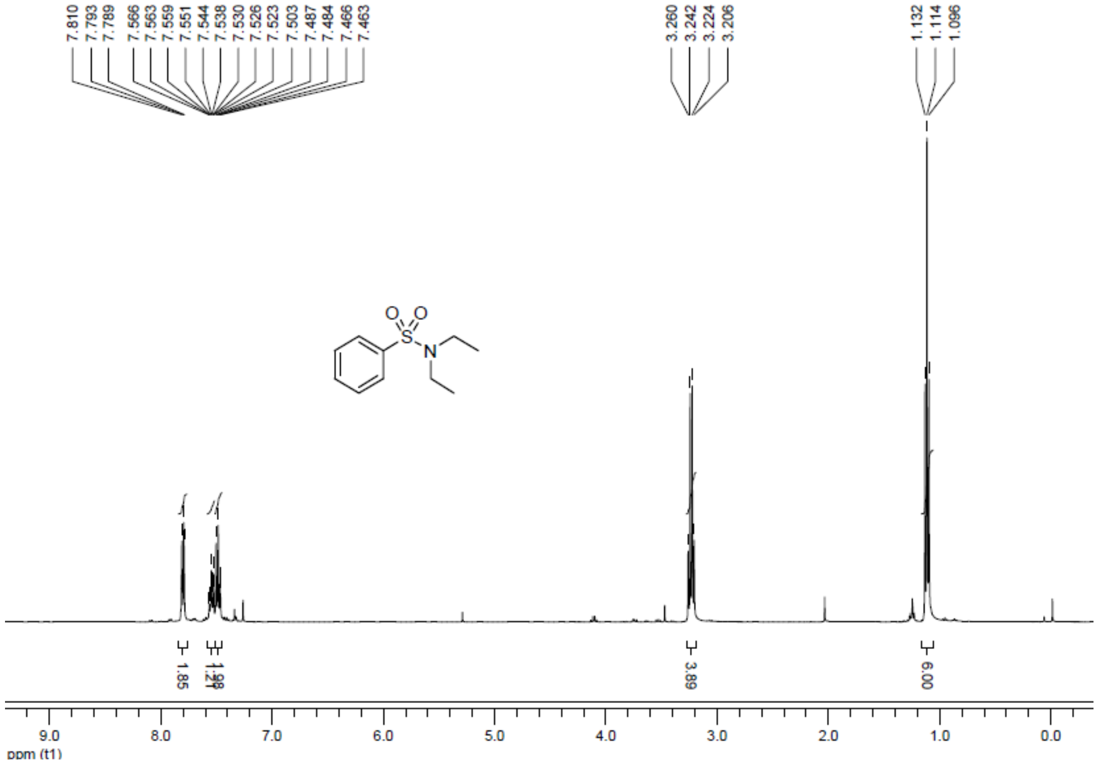
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7.563
7.559
7.551
7.544
7.538
7.530
7.526
7.523
7.503
7.487
7.484
7.466
7.463
3.260
3.242
3.224
3.206
1.132
1.114
1.096
1.85
3.89
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6.0
5.0
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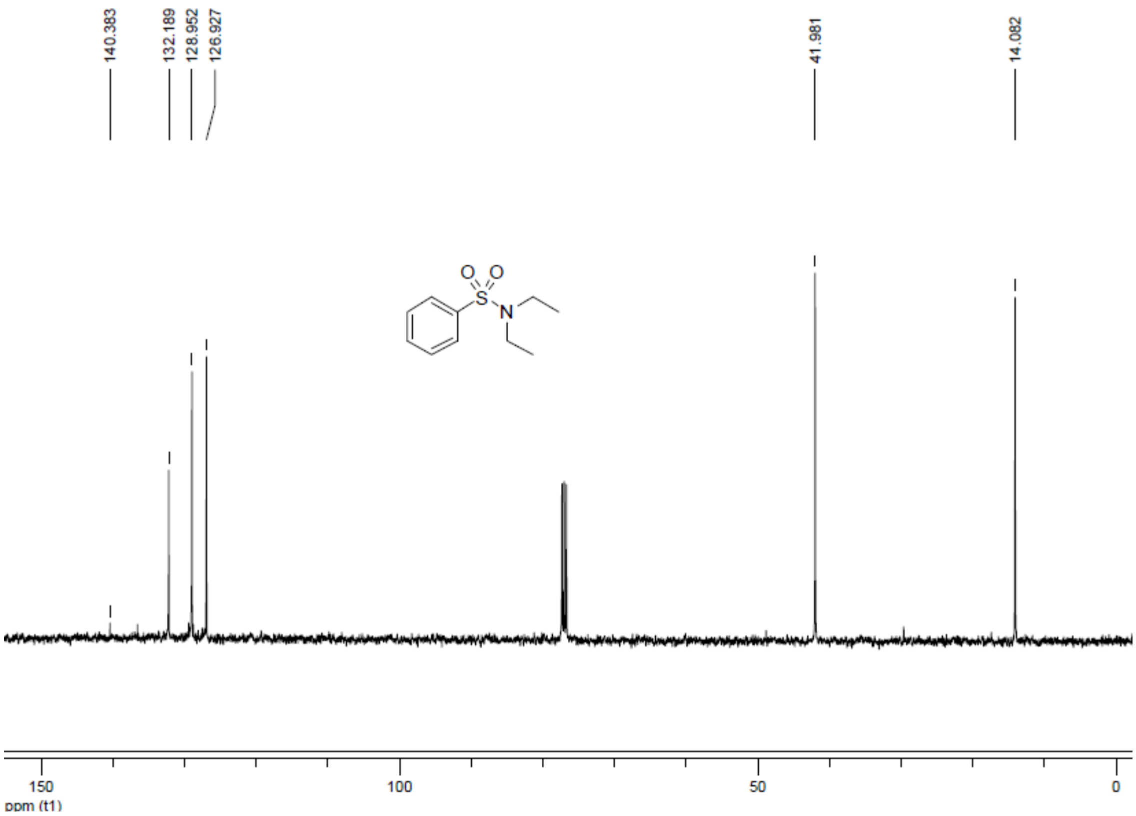
ppm (t1)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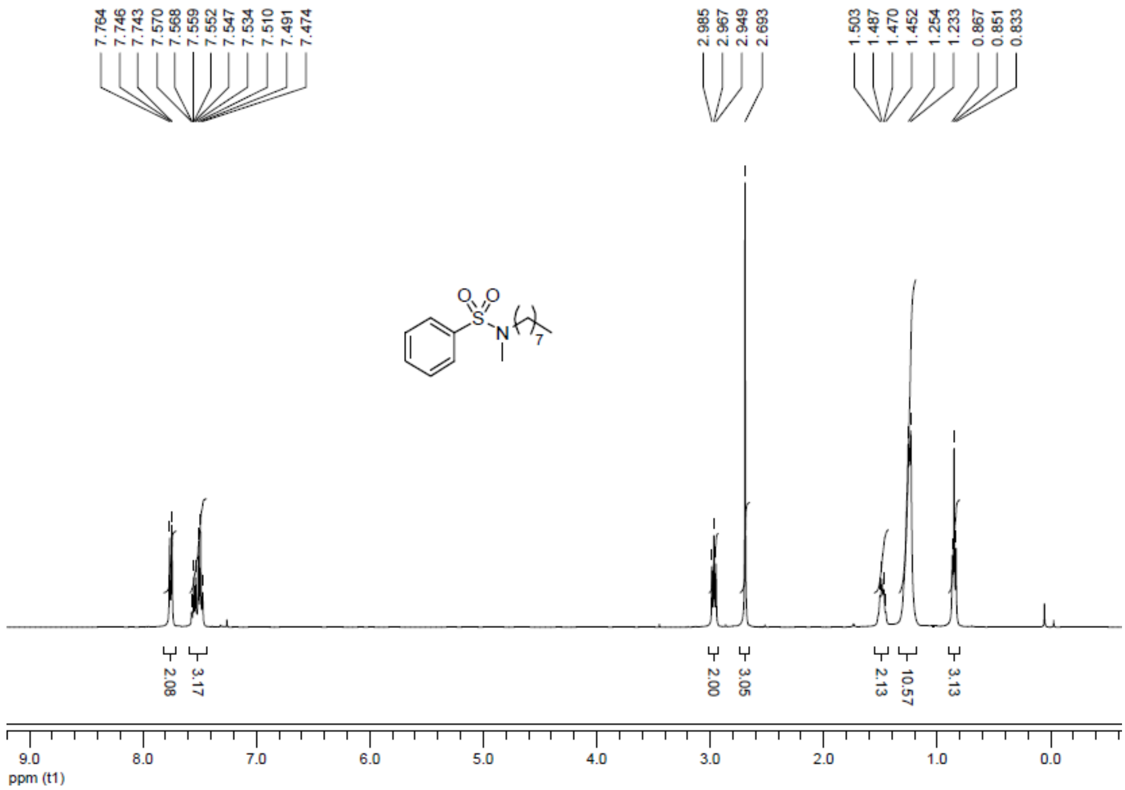
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7.743
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7.568
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7.552
7.547
7.534
7.510
7.491
7.474
2.985
2.967
2.949
2.693
1.503
1.487
1.470
1.452
1.254
1.233
0.867
0.851
0.833
2.08
3.17
2.00
3.05
2.13
10.57
3.13
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7.0
6.0
5.0
4.0
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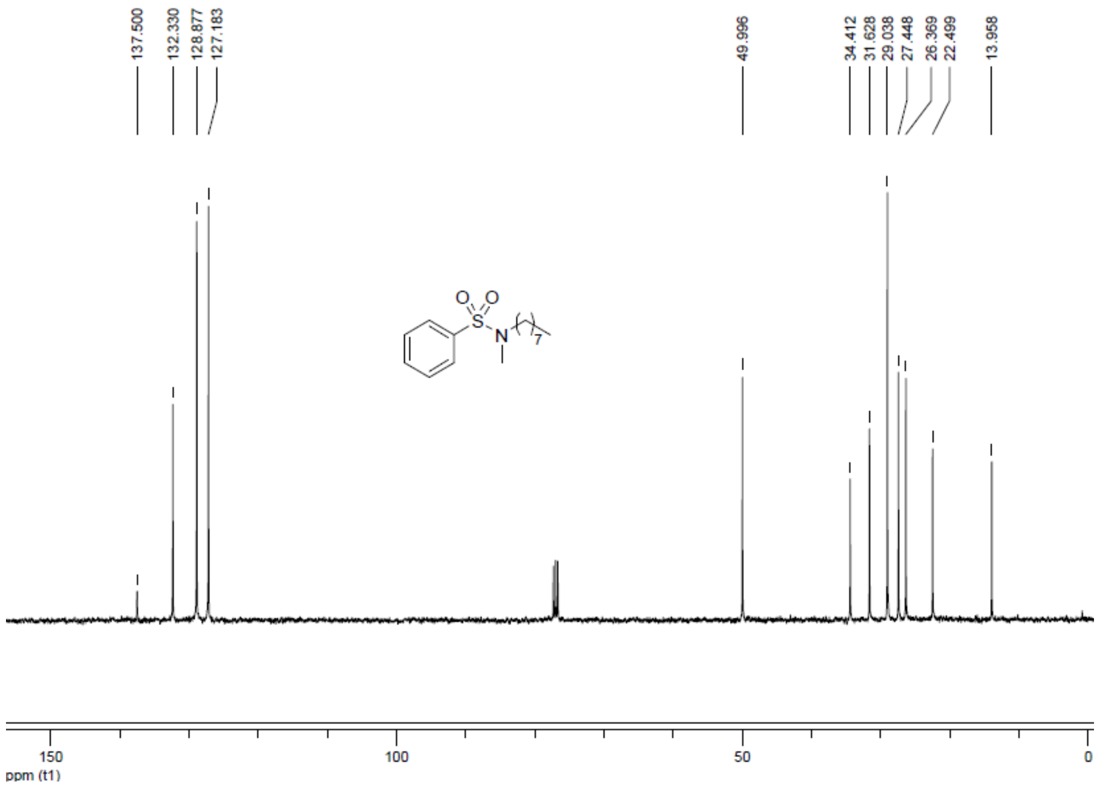
2.0
1.0
0.0
ppm (t1)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29.038
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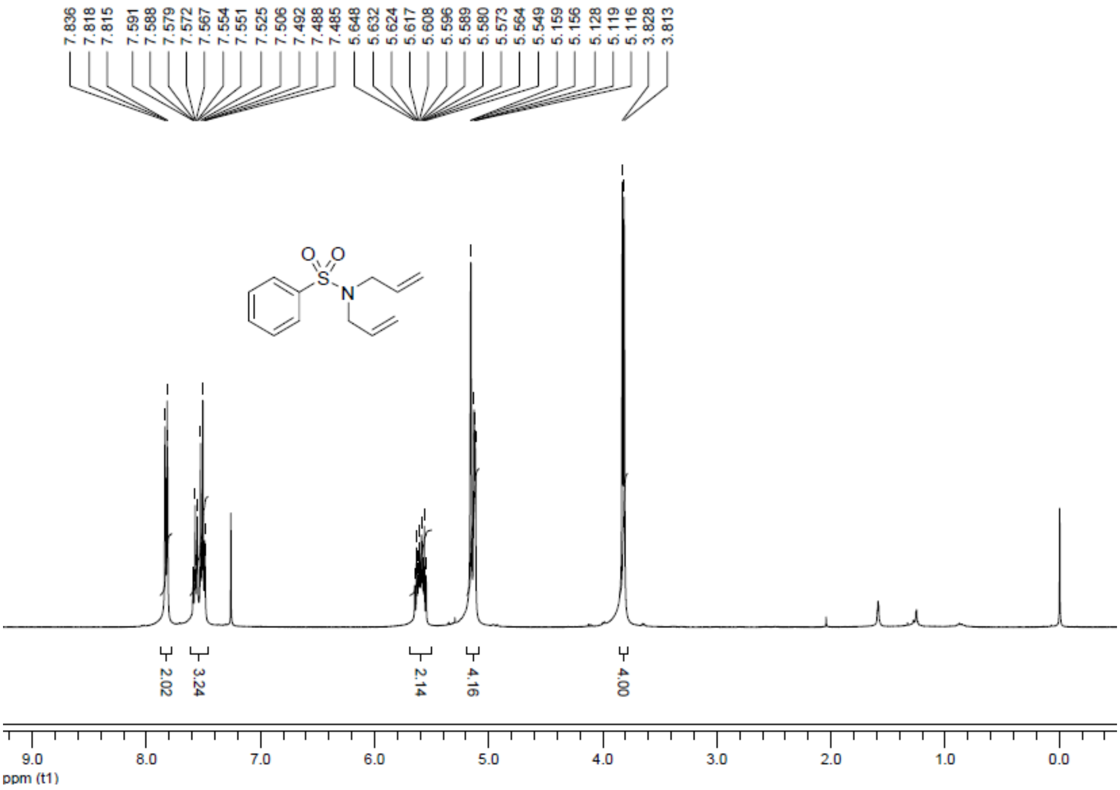
ppm (t1)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7.551
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7.506
7.492
7.488
7.485
5.648
5.632
5.624
5.617
5.608
5.596
5.589
5.580
5.573
5.564
5.549
5.159
5.156
5.128
5.119
5.116
3.828
3.813
O
O
S
N
2.02
3.24
2.14
4.16
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5.0
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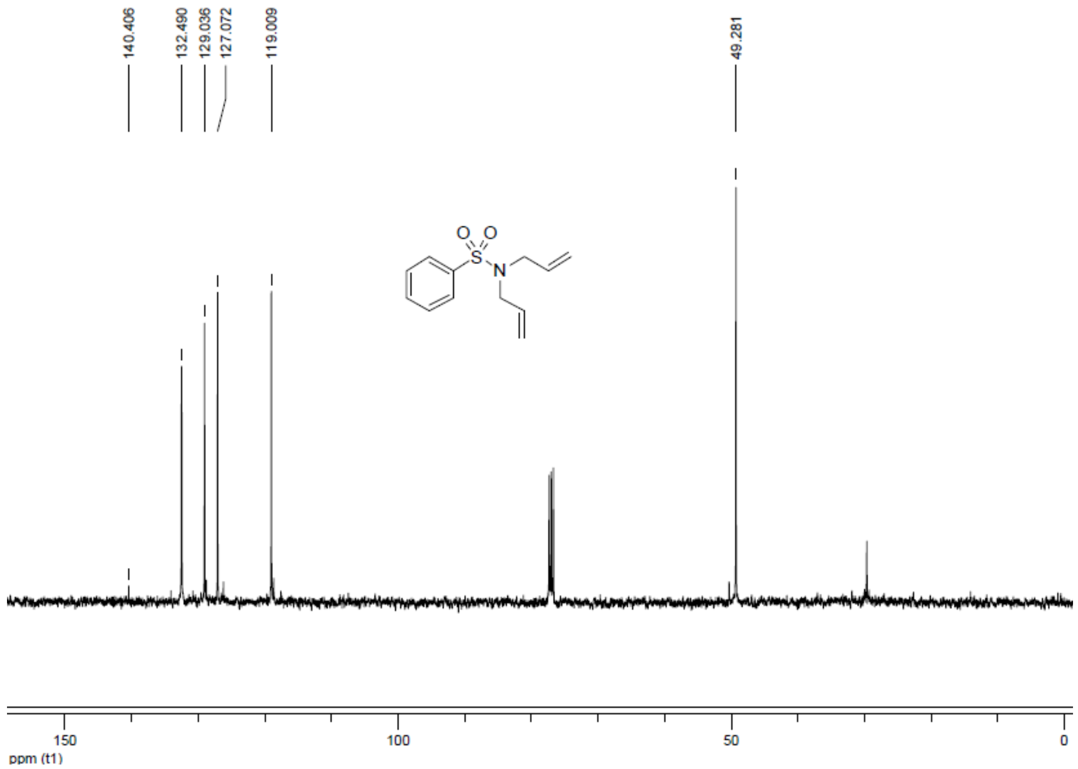
ppm (t1)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S
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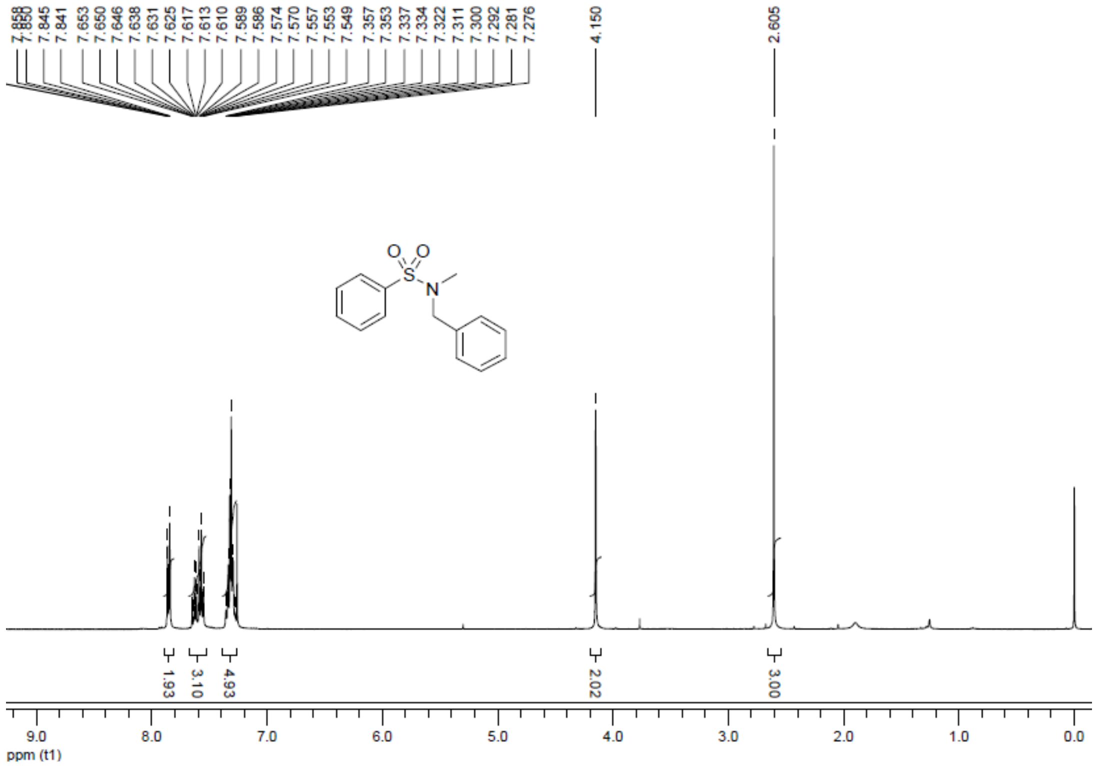

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7.841
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7.650
7.646
7.638
7.631
7.625
7.617
7.613
7.610
7.589
7.586
7.574
7.570
7.557
7.553
7.549
7.357
7.353
7.337
7.334
7.322
7.311
7.300
7.292
7.281
7.276
4.150
2.605
1.93
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4.93
2.02
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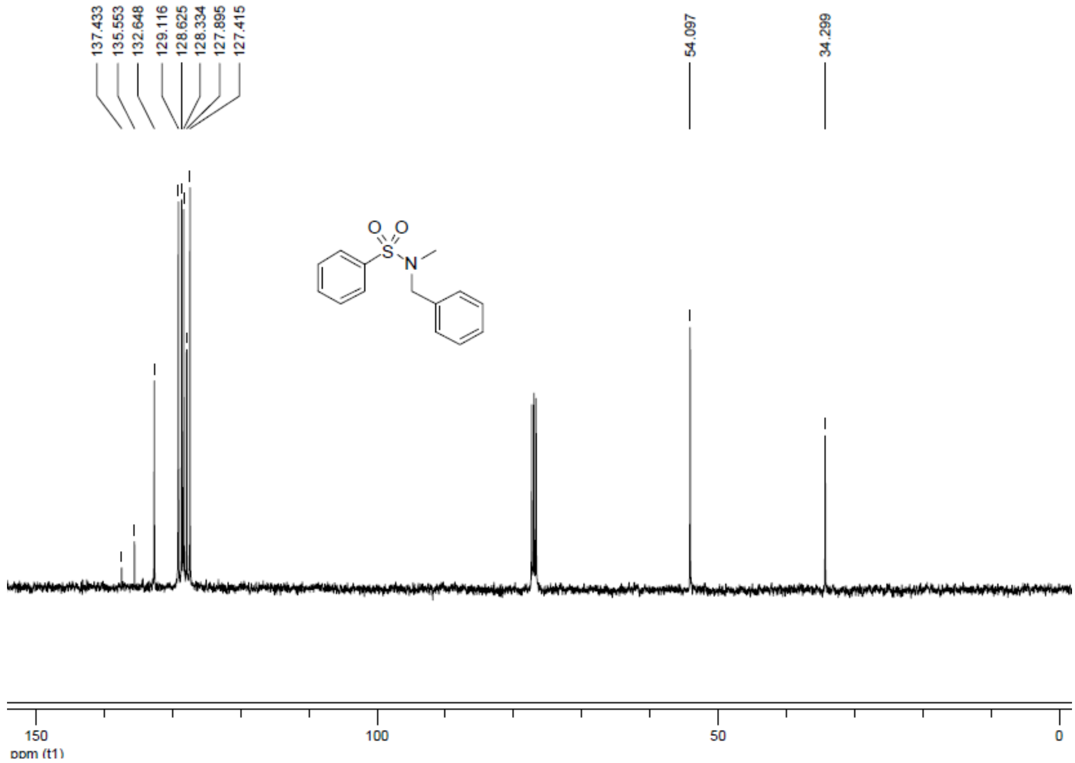
ppm (t1)



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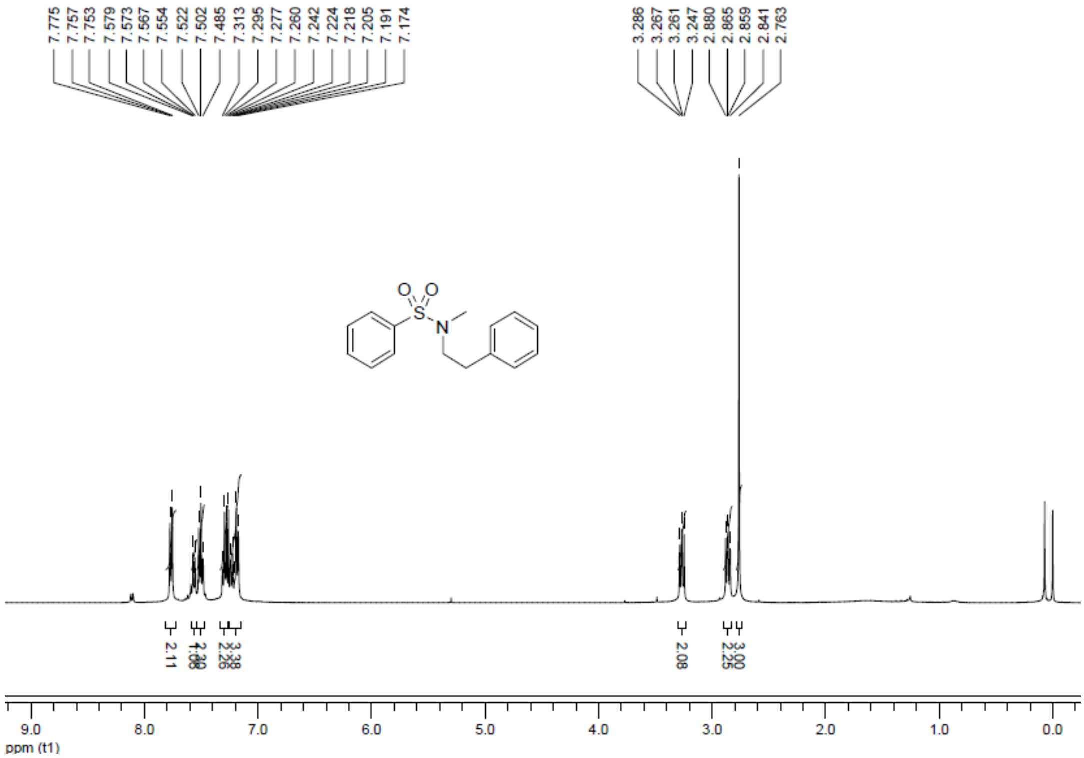
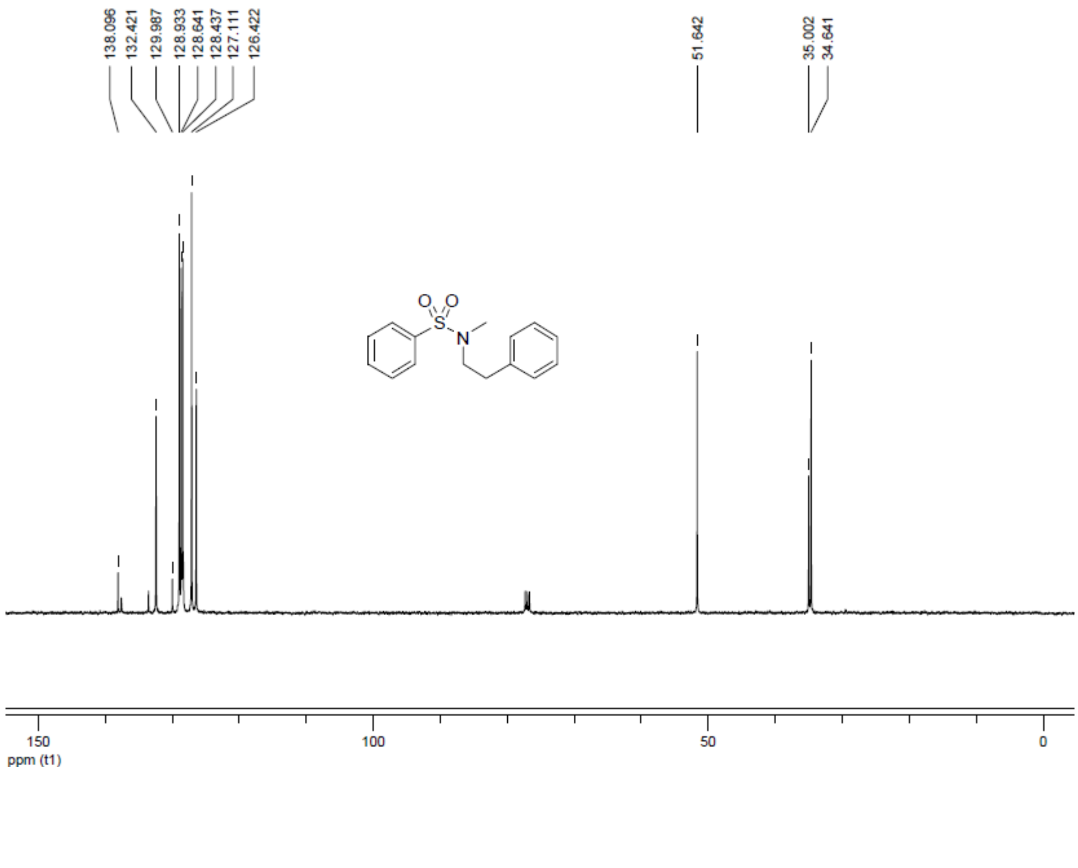
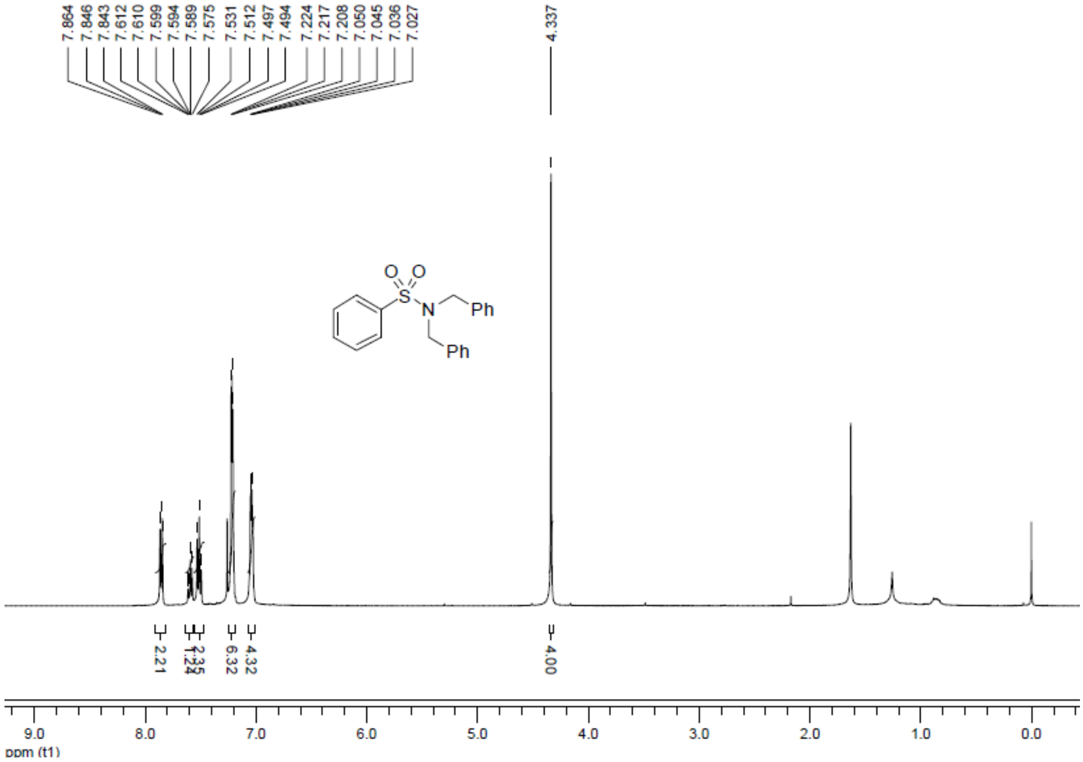
ppm (t1)



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7.589
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7.512
7.497
7.494
7.224
7.217
7.208
7.050
7.045
7.036
7.027
4.337
2.21
1.24
2.35
6.32
4.32
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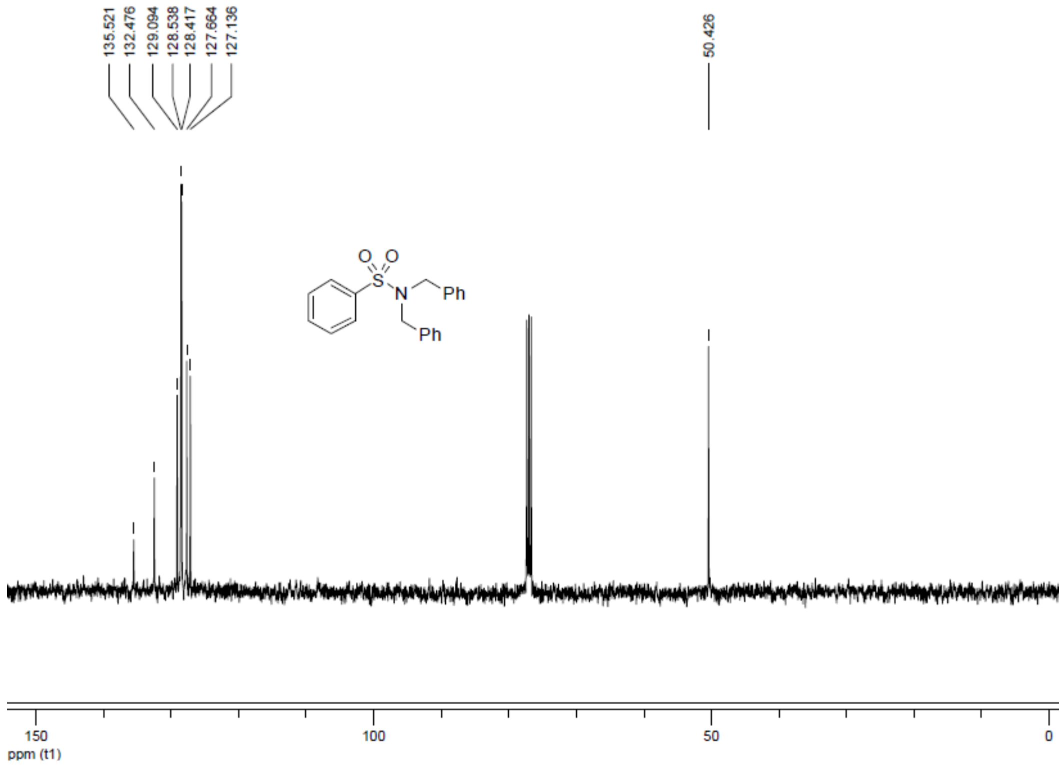
ppm (t1)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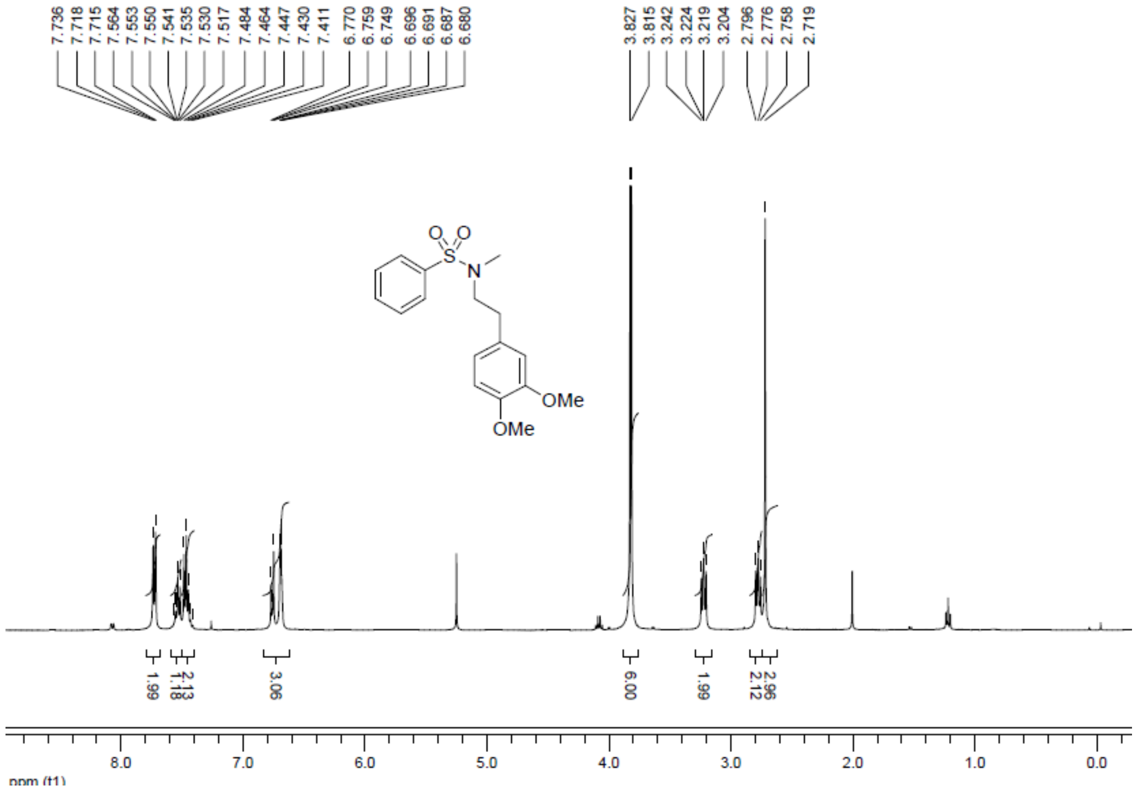
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7.535
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7.517
7.484
7.464
7.447
7.430
7.411
6.770
6.759
6.749
6.696
6.691
6.687
6.680
3.827
3.815
3.242
3.224
3.219
3.204
2.796
2.776
2.758
2.719
OMe
OMe
1.99
1.18
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6.00
1.99
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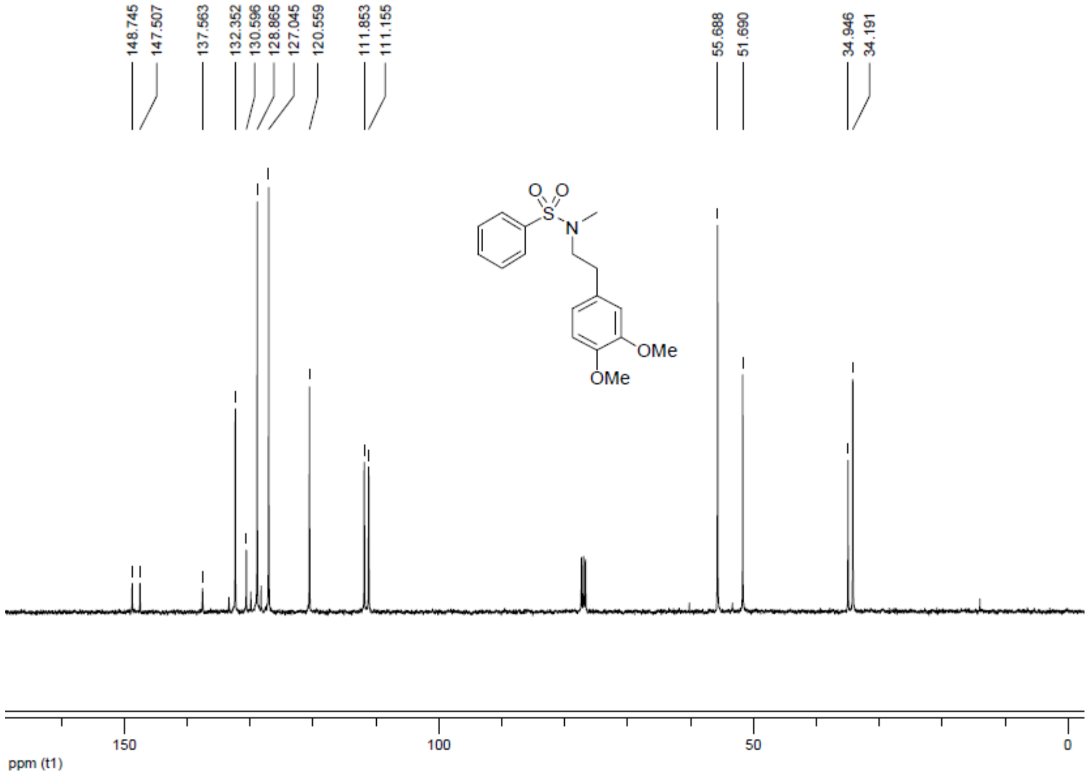
ppm (t1)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OMe
OMe
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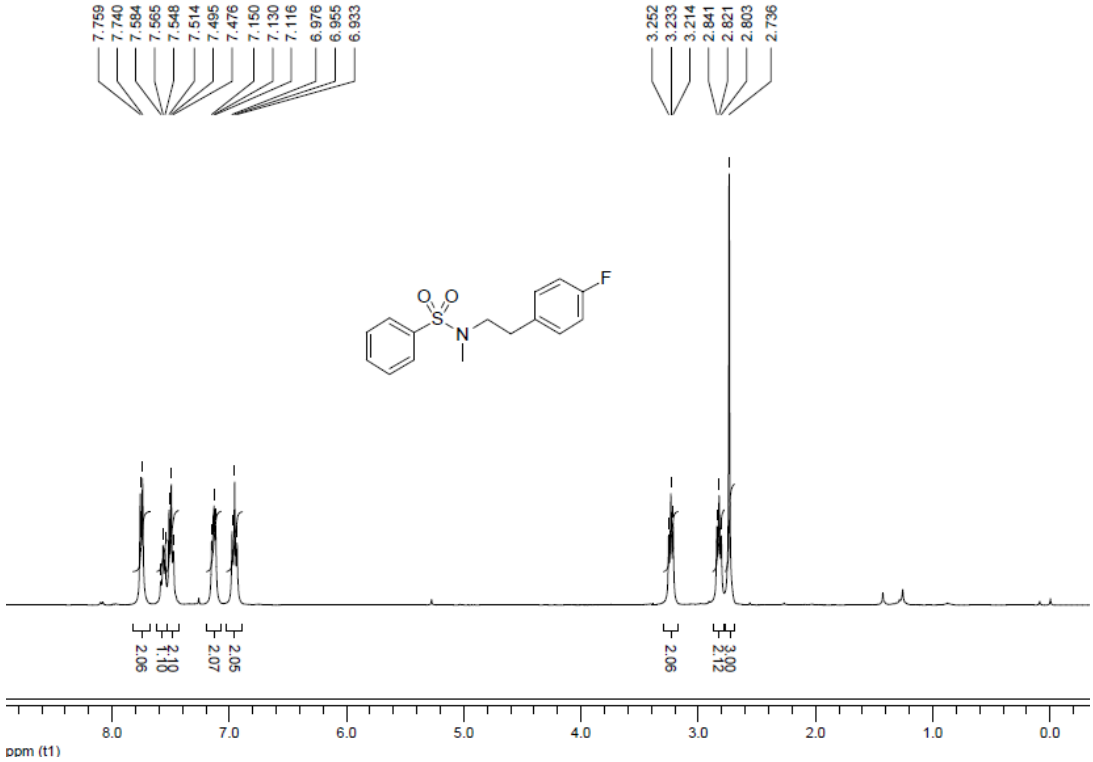
ppm (t1)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7.476
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7.130
7.116
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6.955
6.933
3.252
3.233
3.214
2.841
2.821
2.803
2.736
F
O
O
S
N
2.06
1.10
2.10
2.07
2.05
2.06
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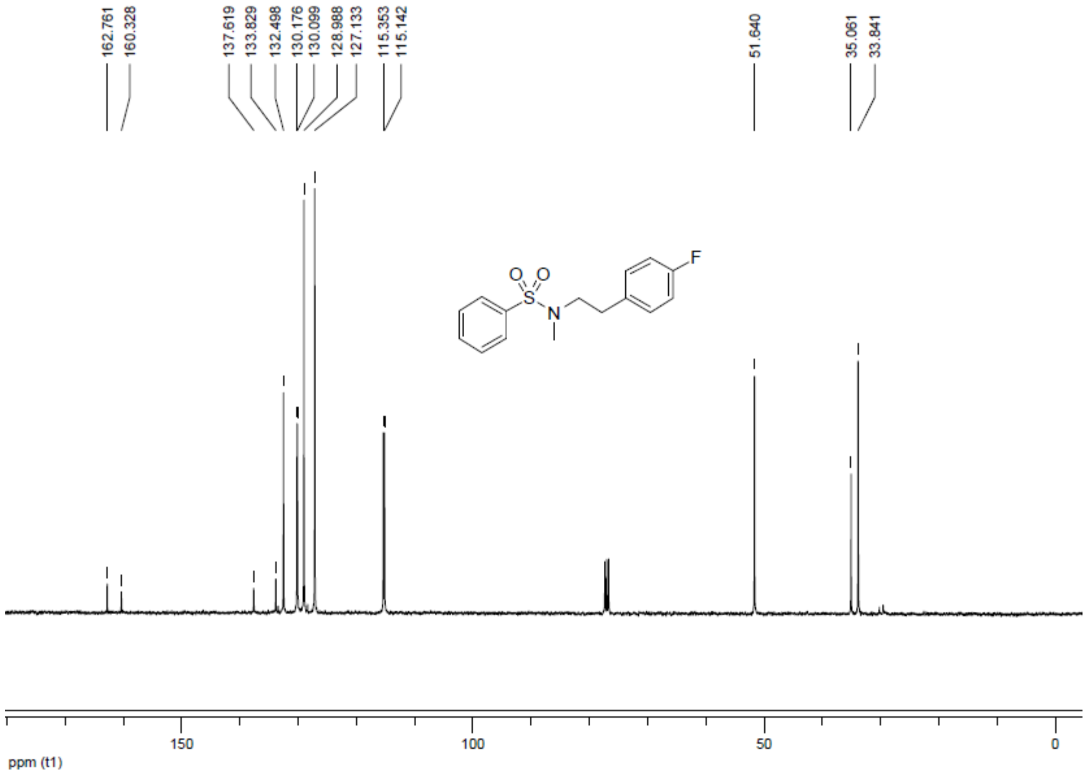
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35.061
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O
O
S
N
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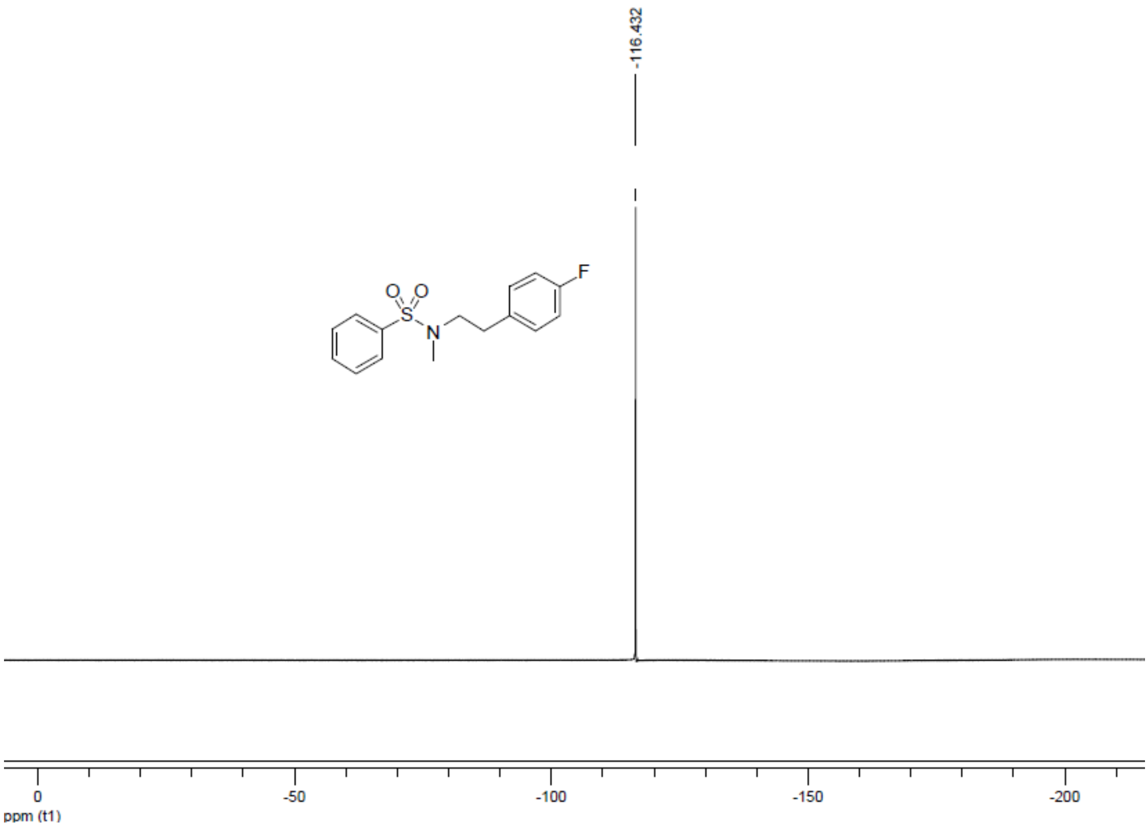
100
50
0
ppm (t1)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-200
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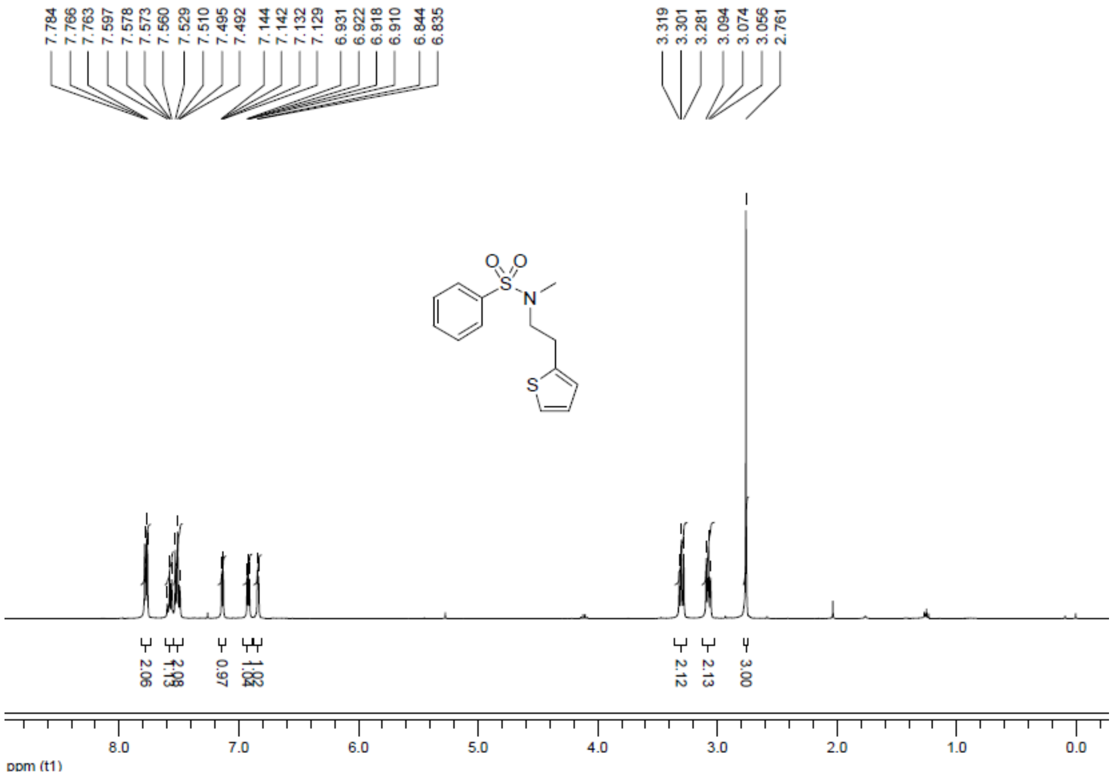
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7.560
7.529
7.510
7.495
7.492
7.144
7.142
7.132
7.129
6.931
6.922
6.918
6.910
6.844
6.835
3.319
3.301
3.281
3.094
3.074
3.056
2.761
O
O
S
N
S
2.06
0.97
2.12
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3.00
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5.0
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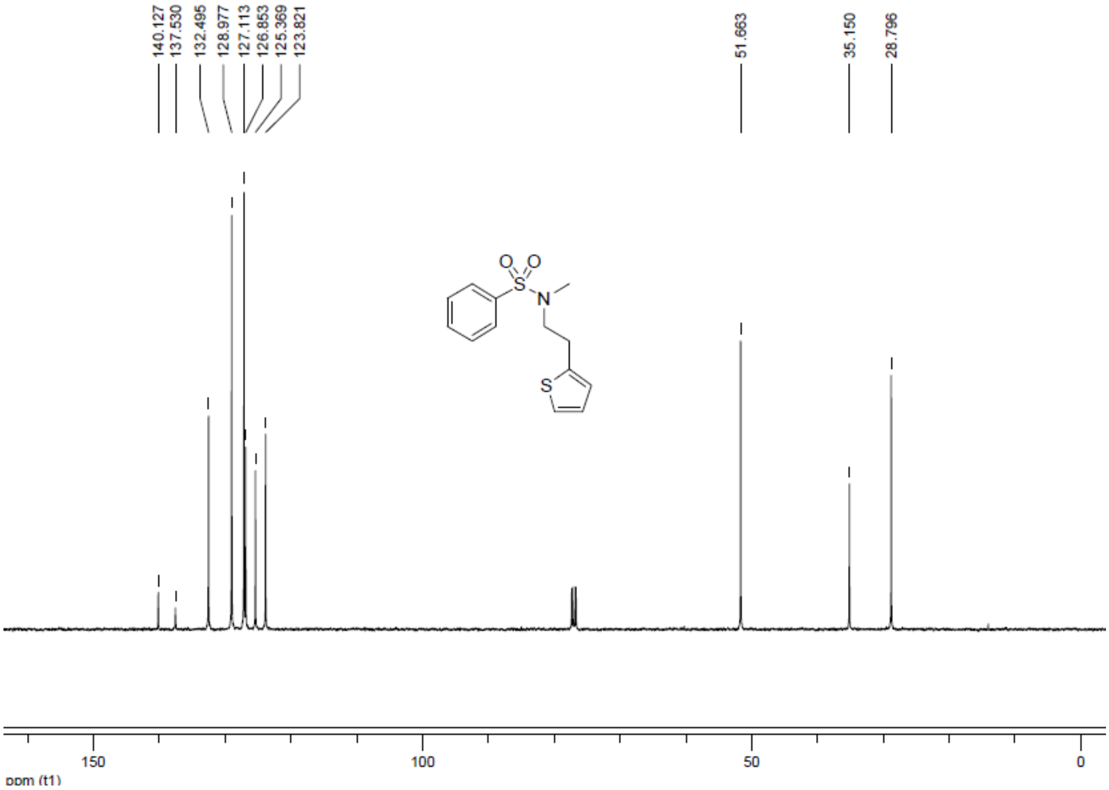
2.0
1.0
0.0
ppm (t1)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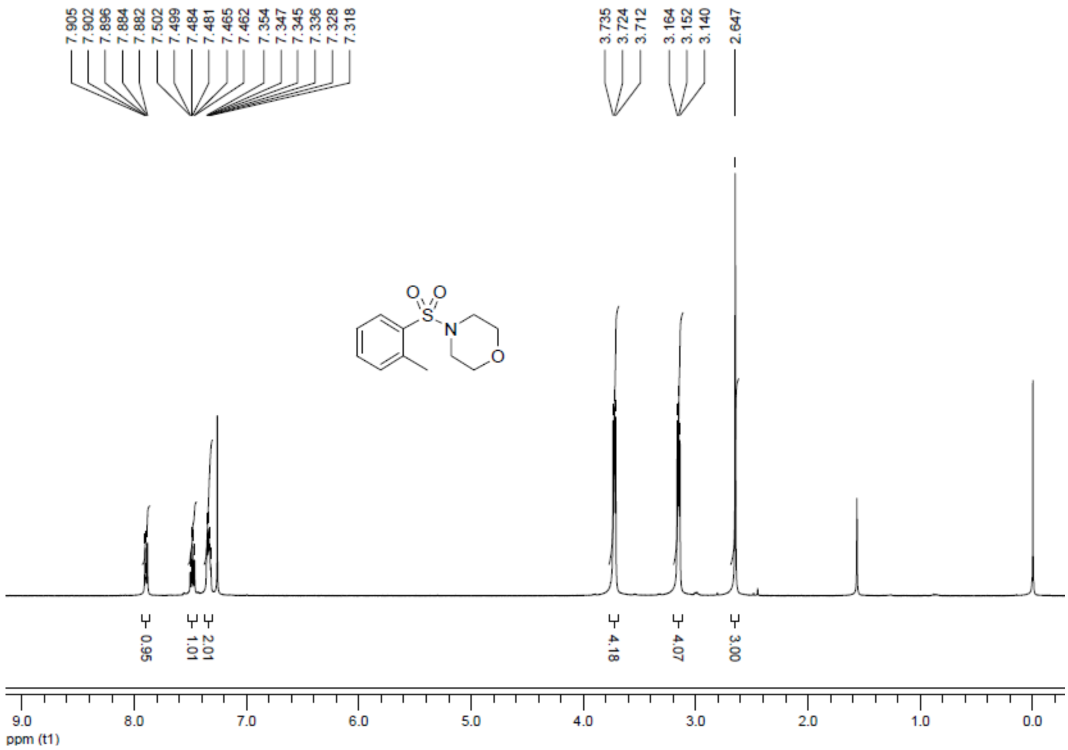
ppm (t1)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7.484
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7.462
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7.347
7.345
7.336
7.328
7.318
3.735
3.724
3.712
3.164
3.152
3.140
2.647
0.95
1.01
2.01
4.18
4.07
3.00
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5.0
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2.0
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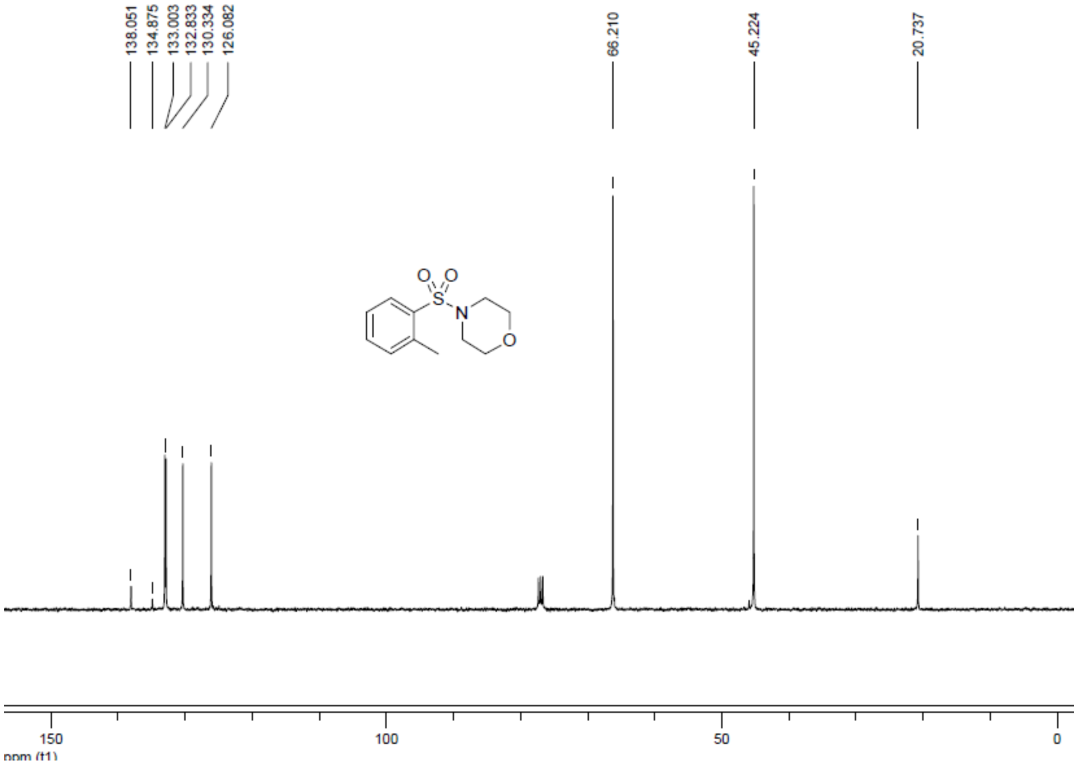
ppm (t1)


138.051
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133.003
132.833
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45.224
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N
O
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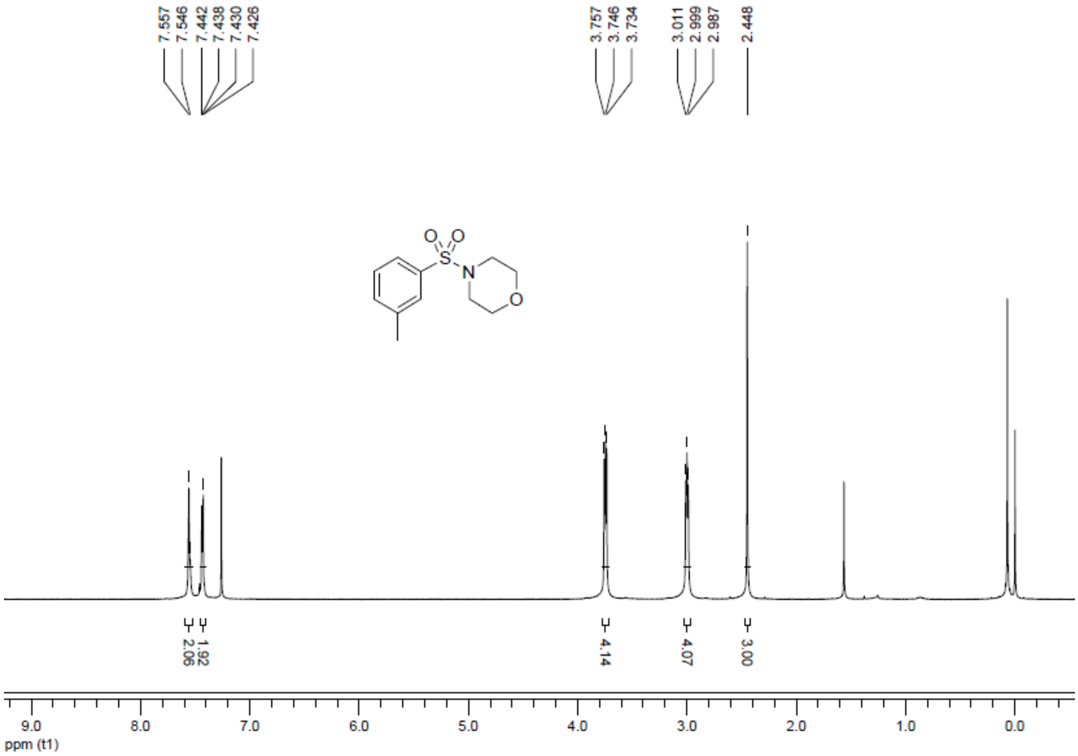
ppm (t1)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7.426
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3.746
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2.987
2.448
O
O
S
N
O
2.06
1.92
4.14
4.07
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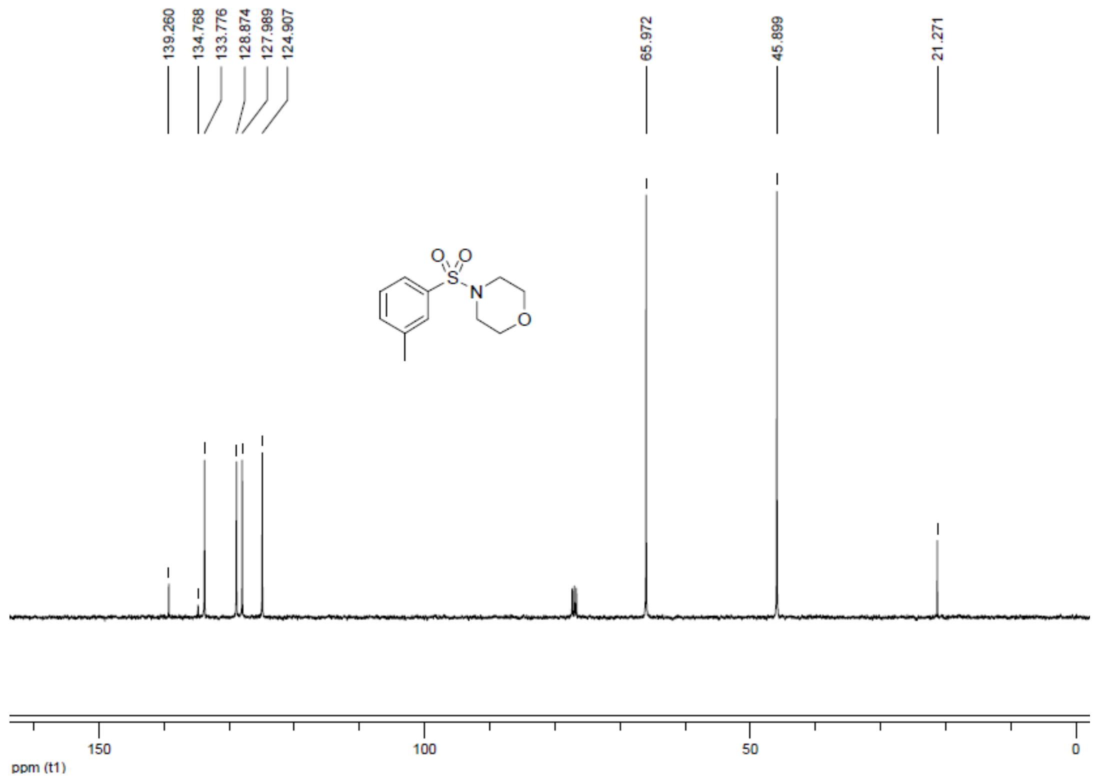
ppm (t1)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100
50
0
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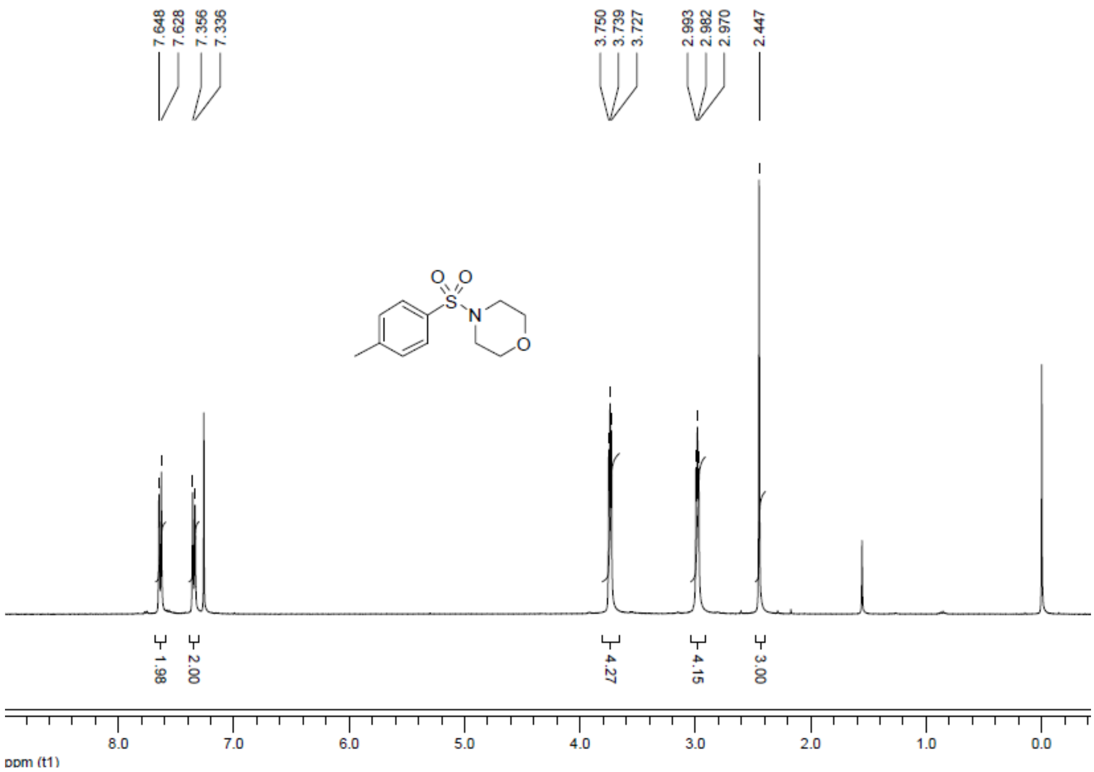
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3.727
2.993
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2.970
2.447
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2.00
4.27
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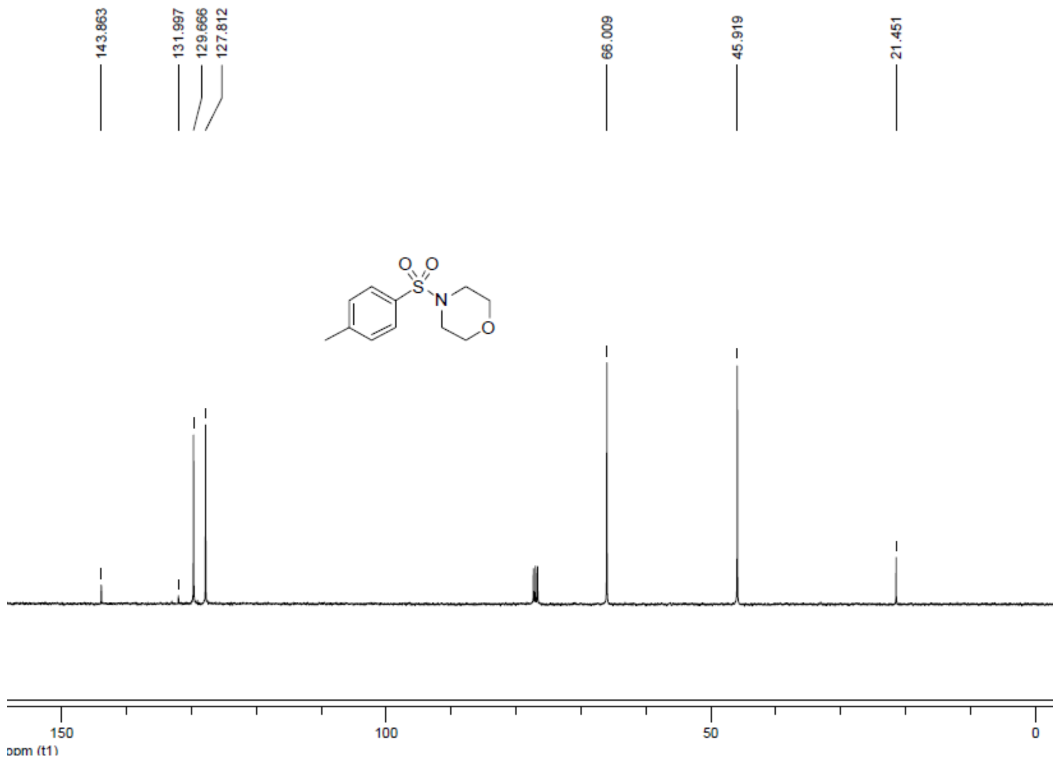
ppm (t1)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45.919
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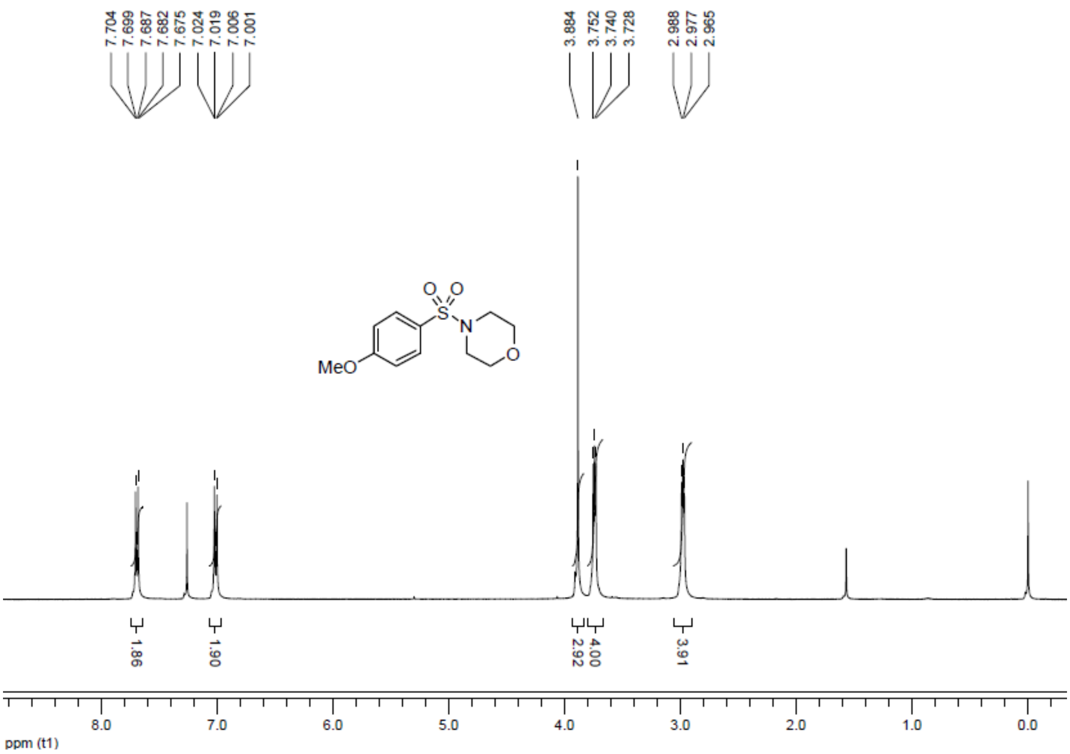
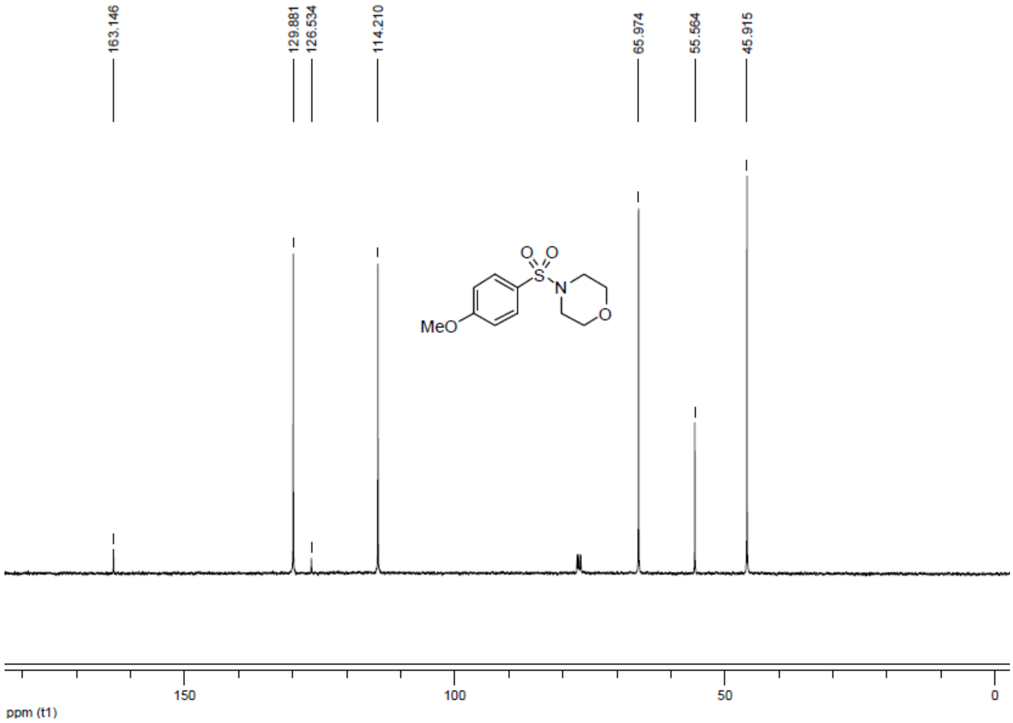
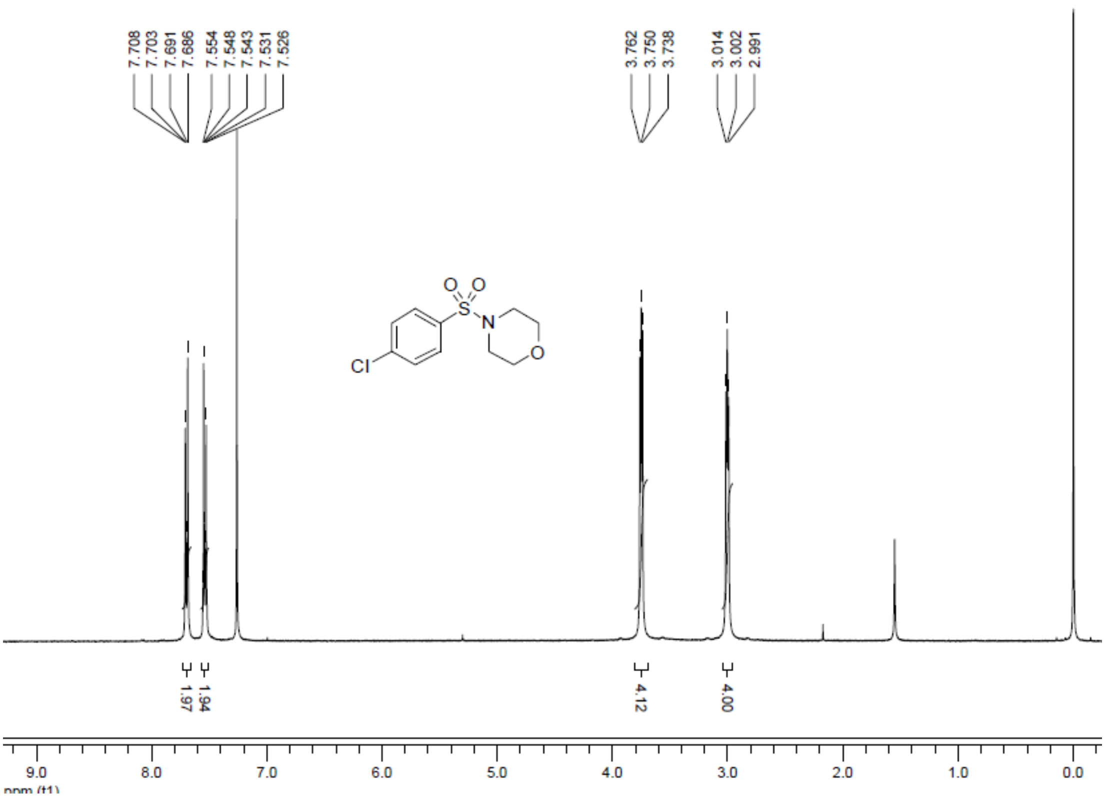
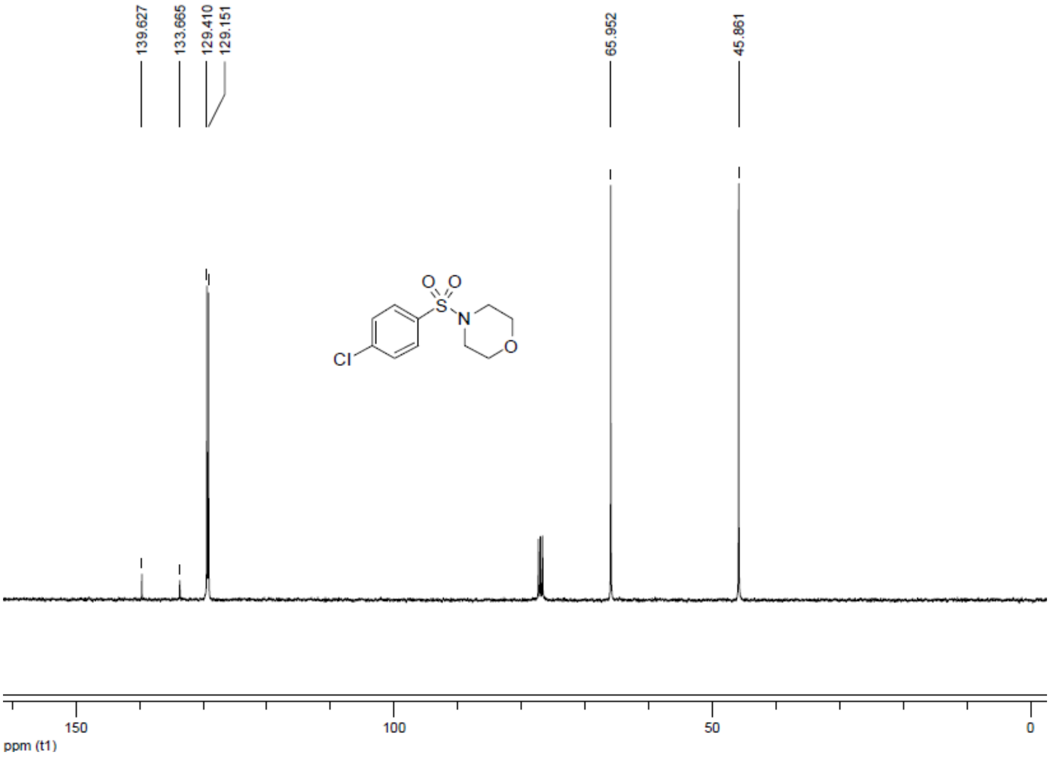
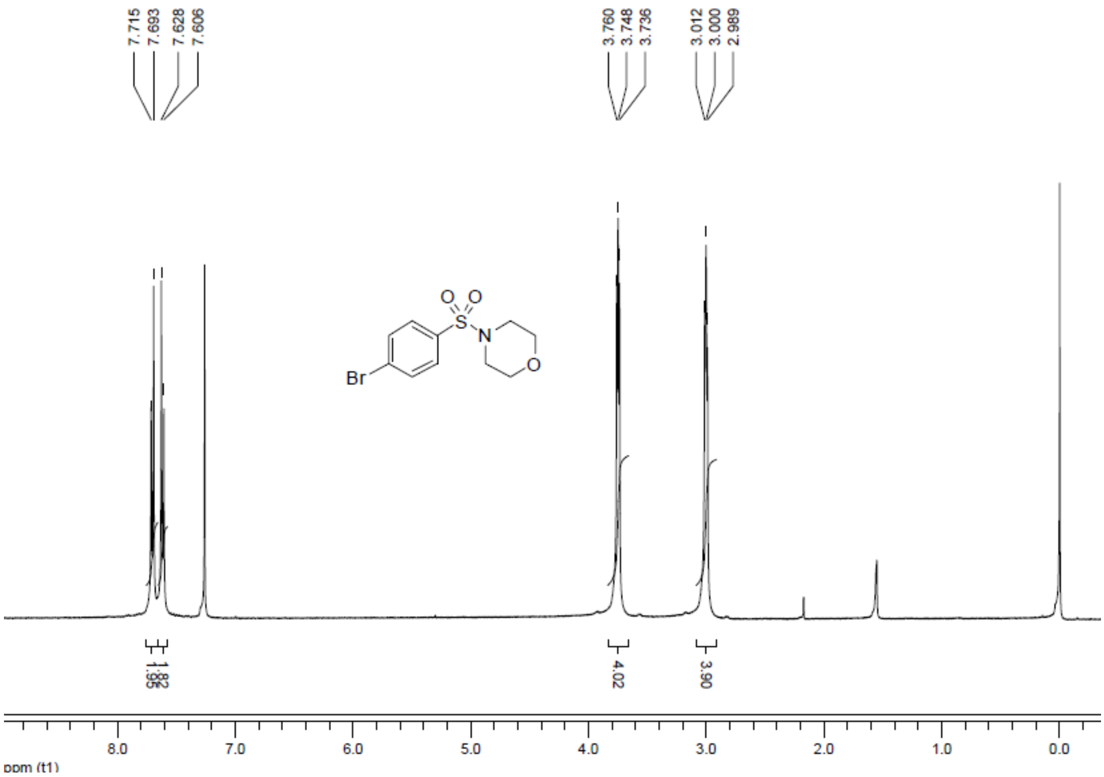
50
0
ppm (t1)

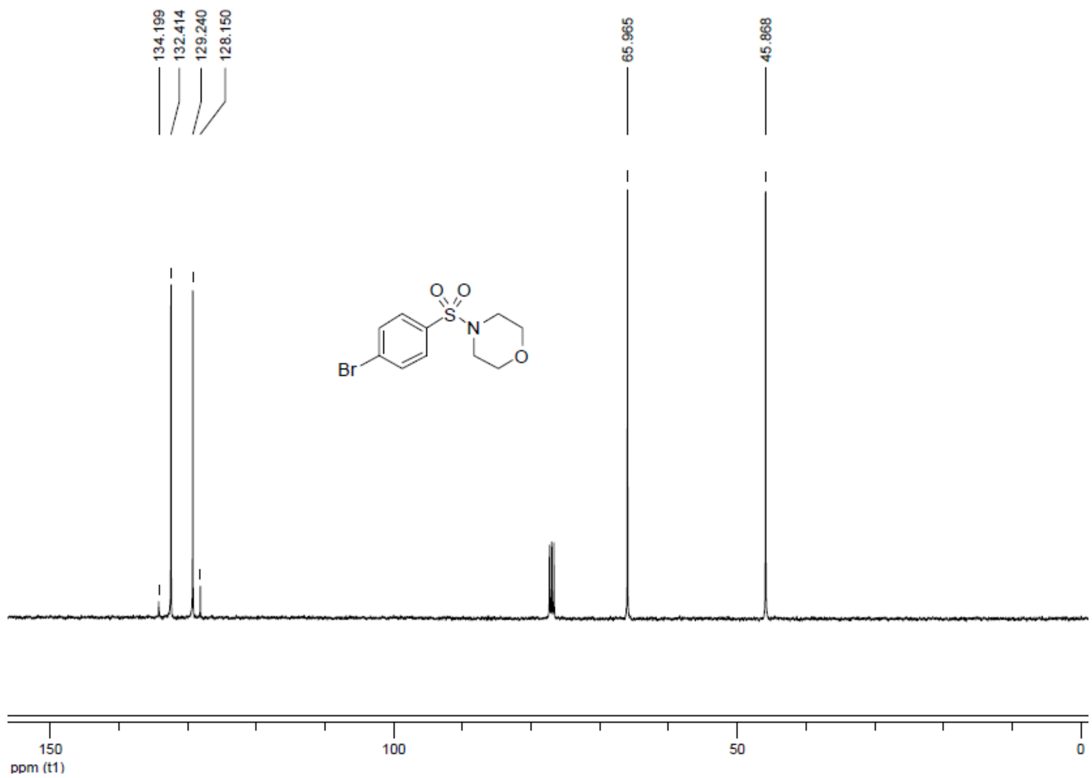

7.704
7.699
7.687
7.682
7.675
7.024
7.019
7.006
7.001
3.884
3.752
3.740
3.728
2.988
2.977
2.965
MeO
1.86
1.90
2.92
4.00
3.91
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

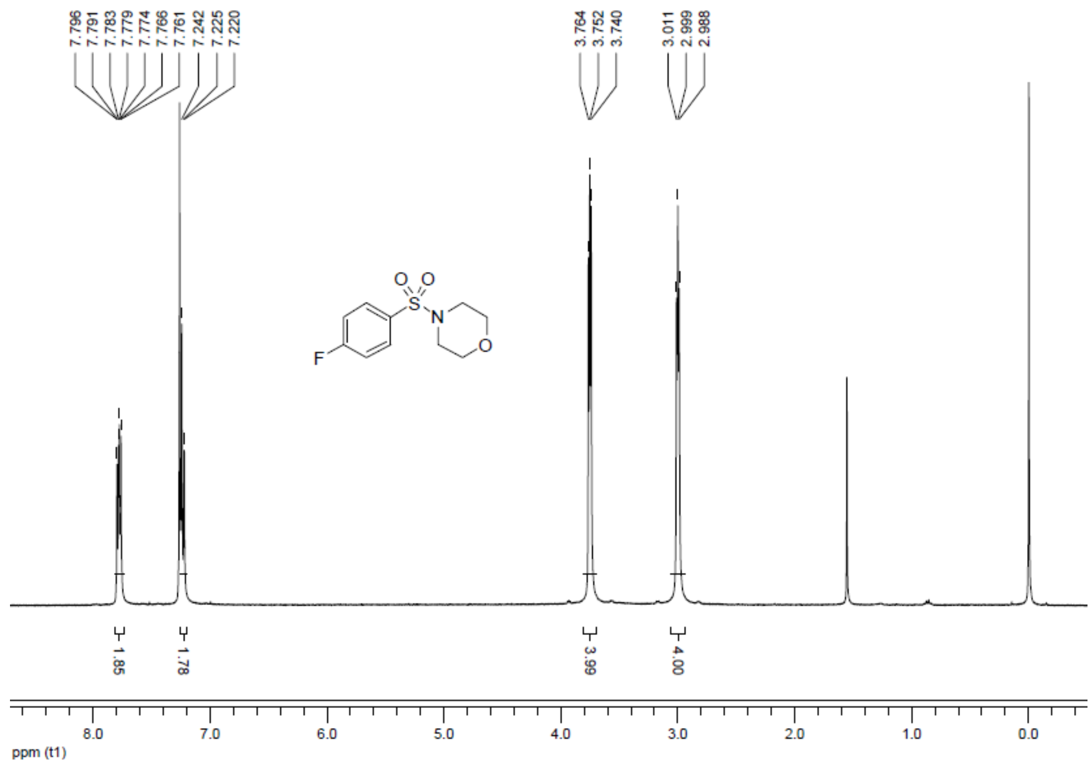

163.146
129.881
126.534
114.210
65.974
55.564
45.915
MeO
O
O
S
N
O
150
100
50
0
ppm (t1)

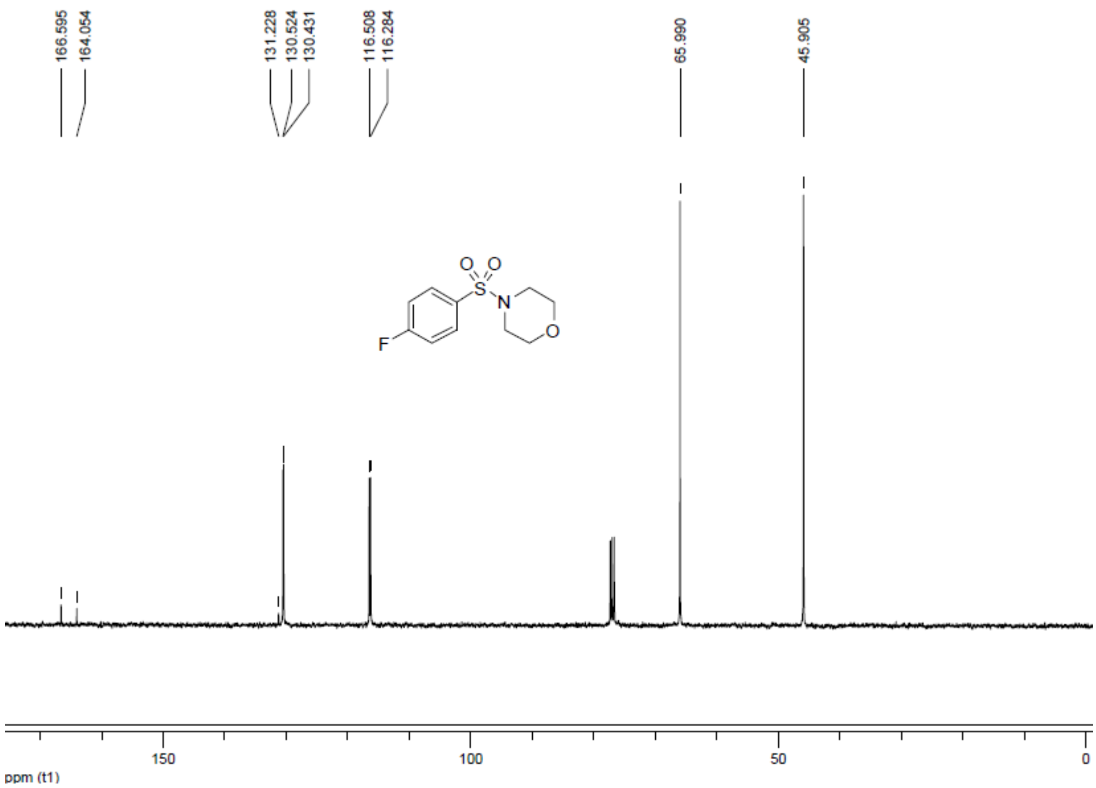

7.708
7.703
7.691
7.686
7.554
7.548
7.543
7.531
7.526
3.762
3.750
3.738
3.014
3.002
2.991
Cl
O
O
S
N
O
1.97
1.94
4.12
4.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

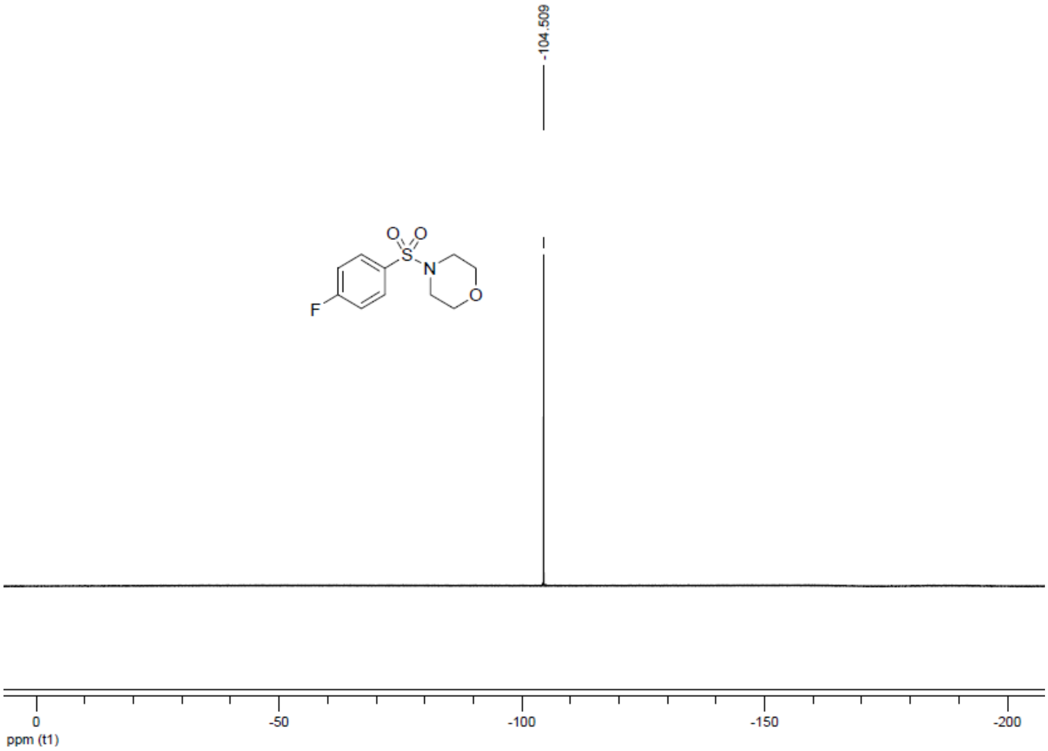

139.627
133.665
129.410
129.151
65.952
45.861
Cl
O
O
S
N
O
150
100
50
0
ppm (t1)

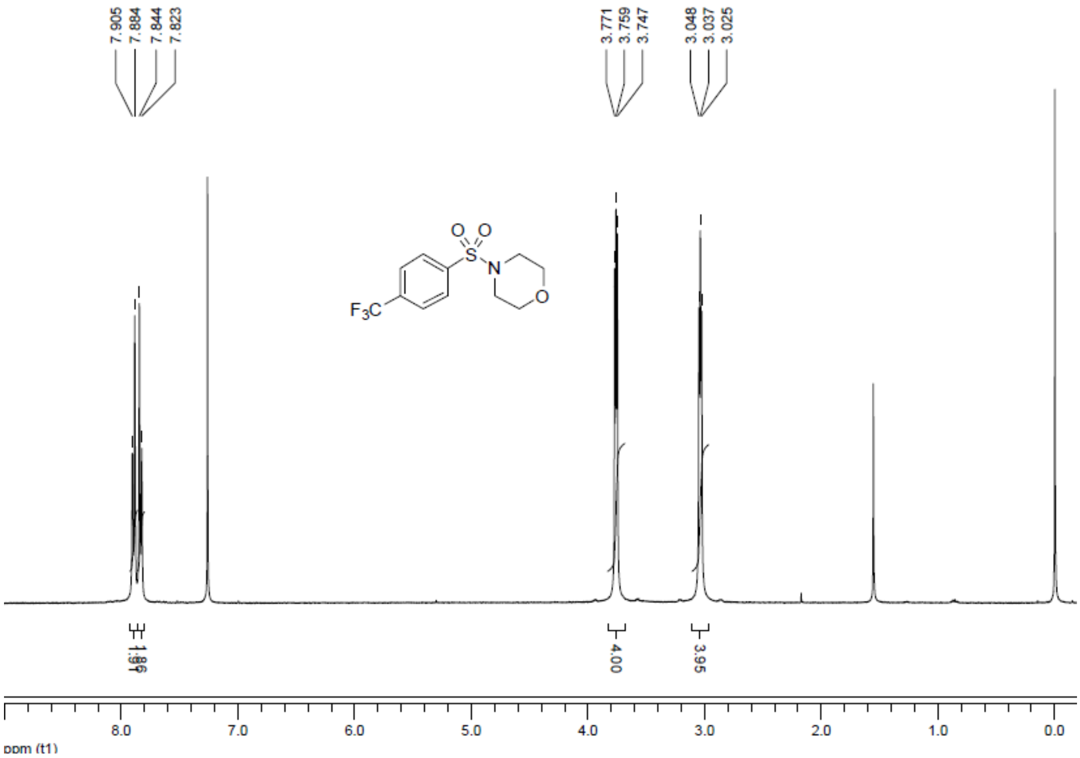

7.715
7.693
7.628
7.606
3.760
3.748
3.736
3.012
3.000
2.989
Br
O
O
S
N
O
4.02
3.90
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

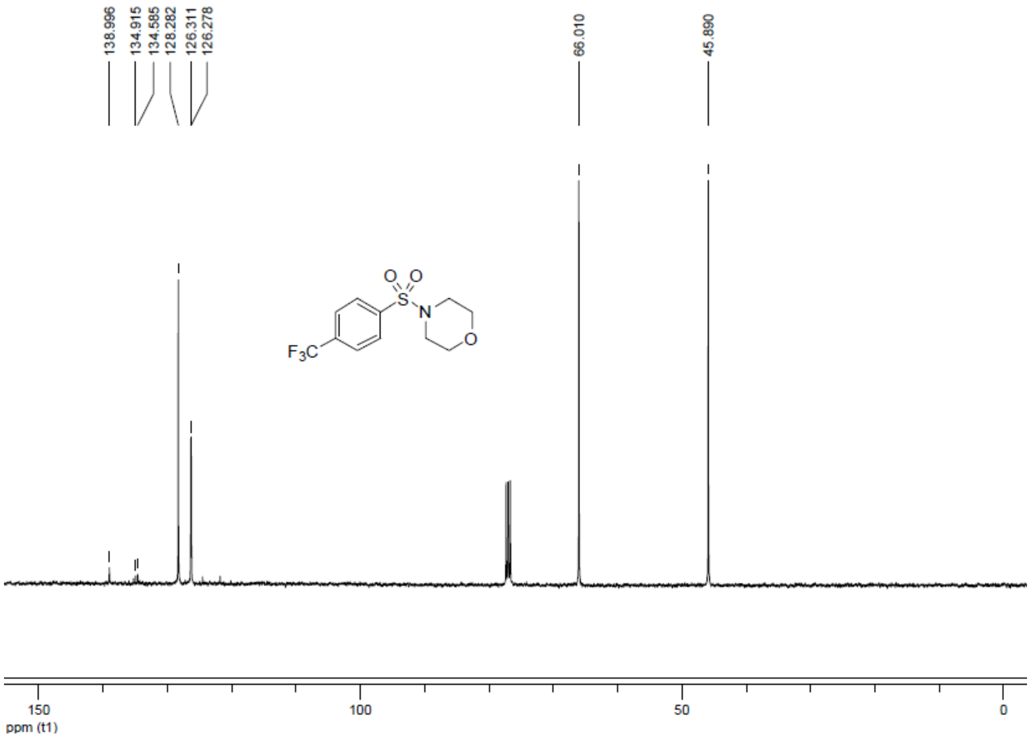

134.199
132.414
129.240
128.150
65.965
45.868
Br
150
100
50
0
ppm (t1)

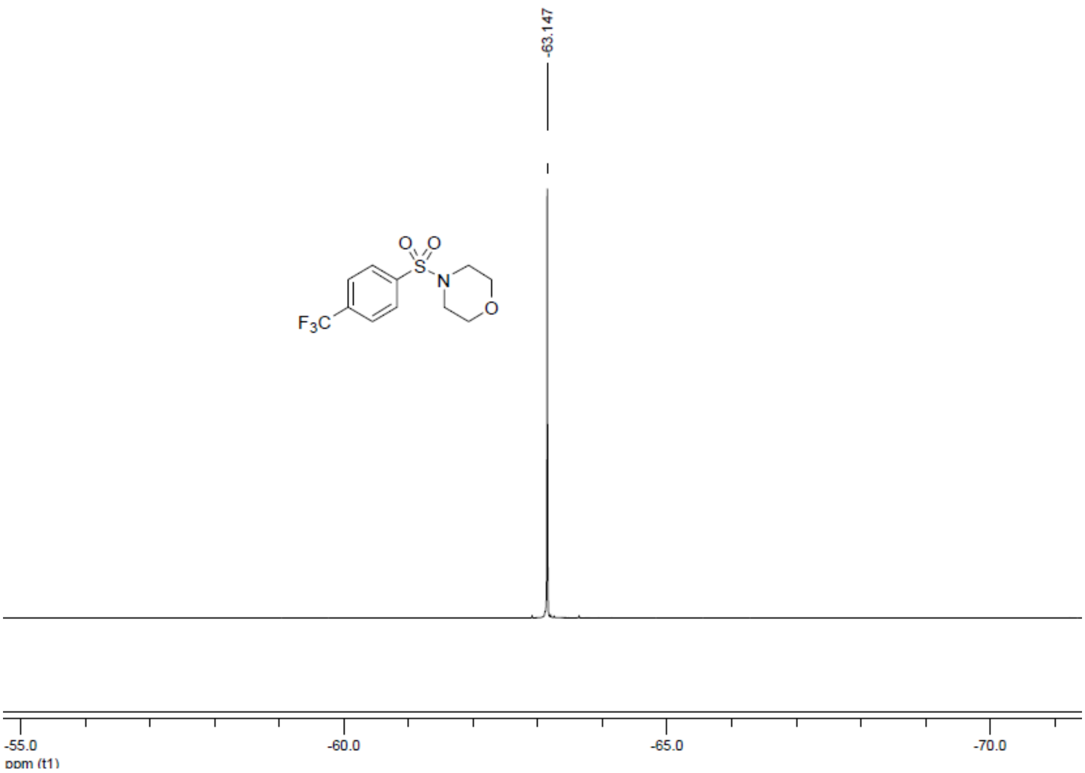

7.796
7.791
7.783
7.779
7.774
7.766
7.761
7.242
7.225
7.220
3.764
3.752
3.740
3.011
2.999
2.988
F
1.85
1.78
3.99
4.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

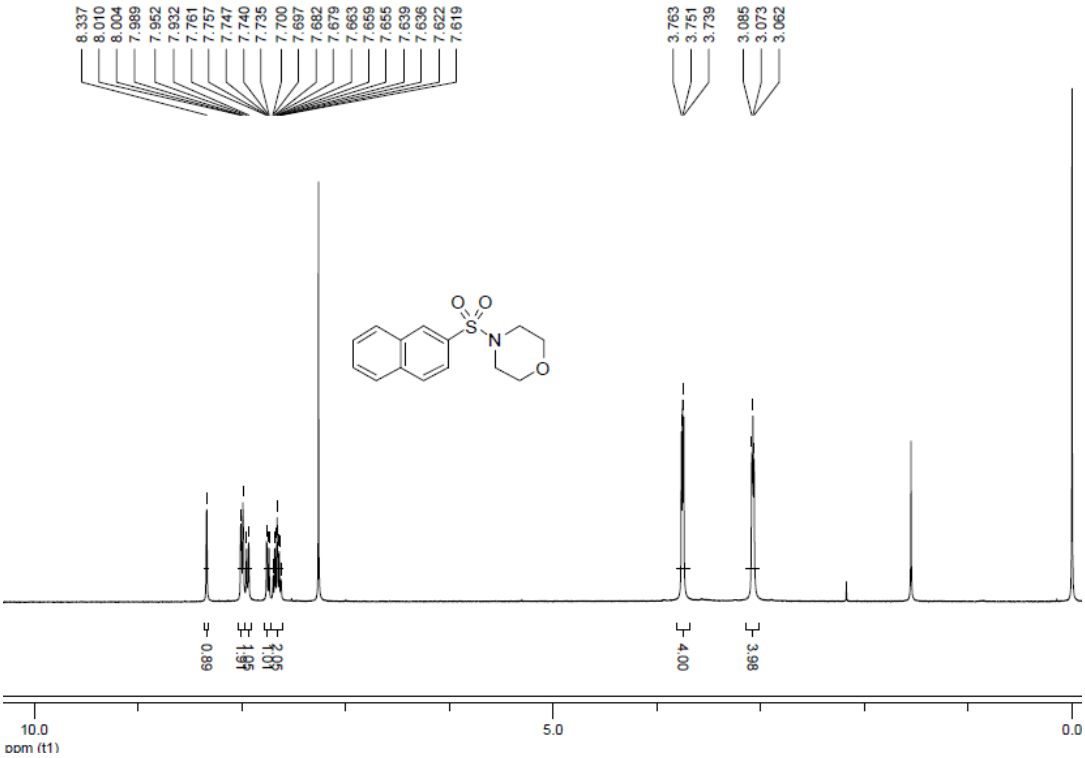

166.595
164.054
131.228
130.524
130.431
116.508
116.284
65.990
45.905
150
100
50
0
ppm (t1)
F
O
S
O
N
O

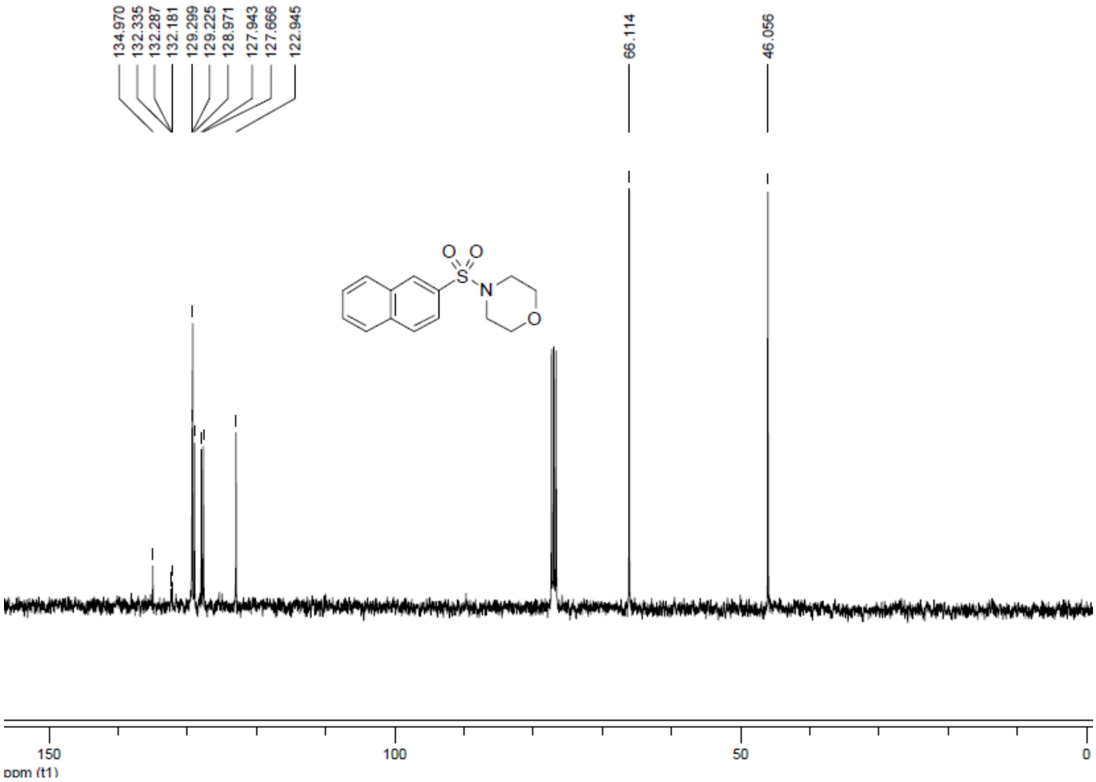

-104.509
0
-50
-100
-150
-200
ppm (t1)
F
O
S
O
N
O

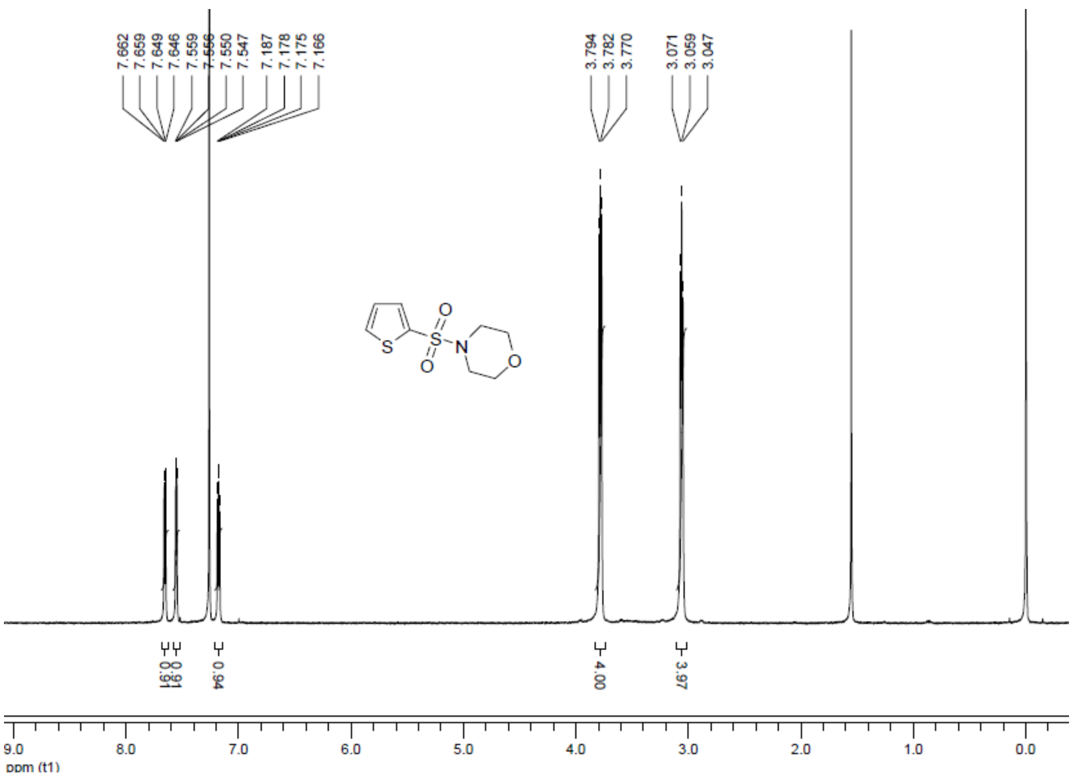

7.905
7.884
7.844
7.823
3.771
3.759
3.747
3.048
3.037
3.025
F3C
O
S
N
O
1.91
1.86
4.00
3.95
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

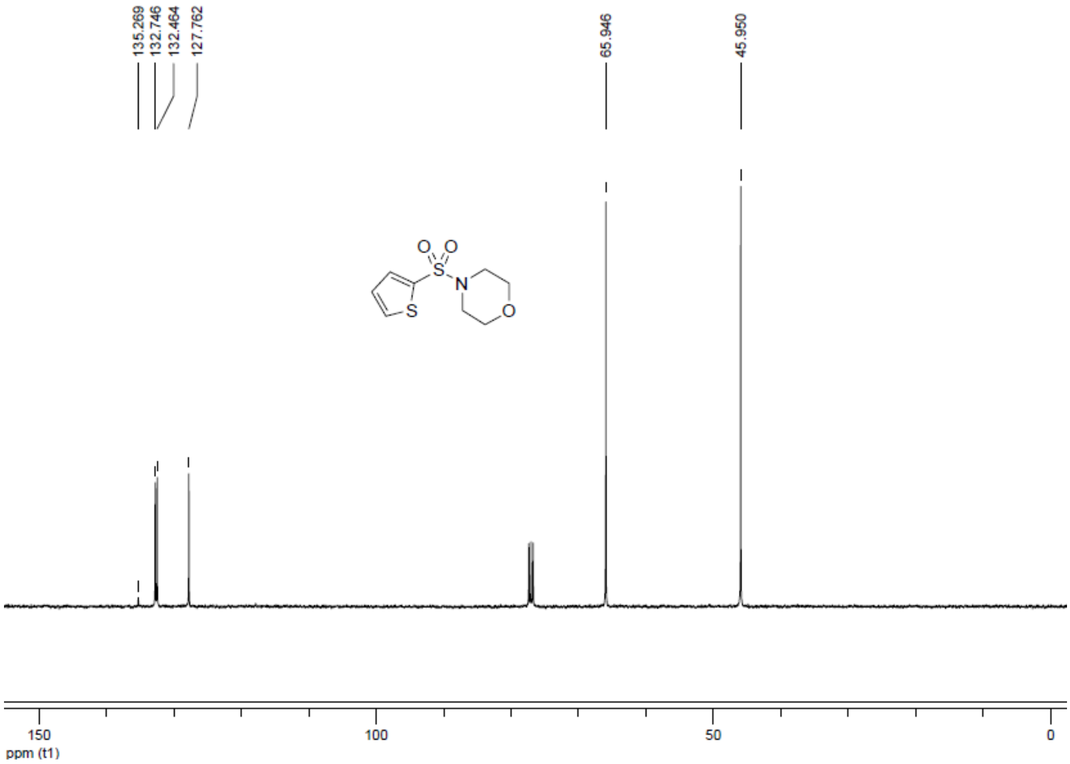

138.996
134.915
134.585
128.282
126.311
126.278
66.010
45.890
F3C
O
S
N
O
150
100
50
0
ppm (t1)

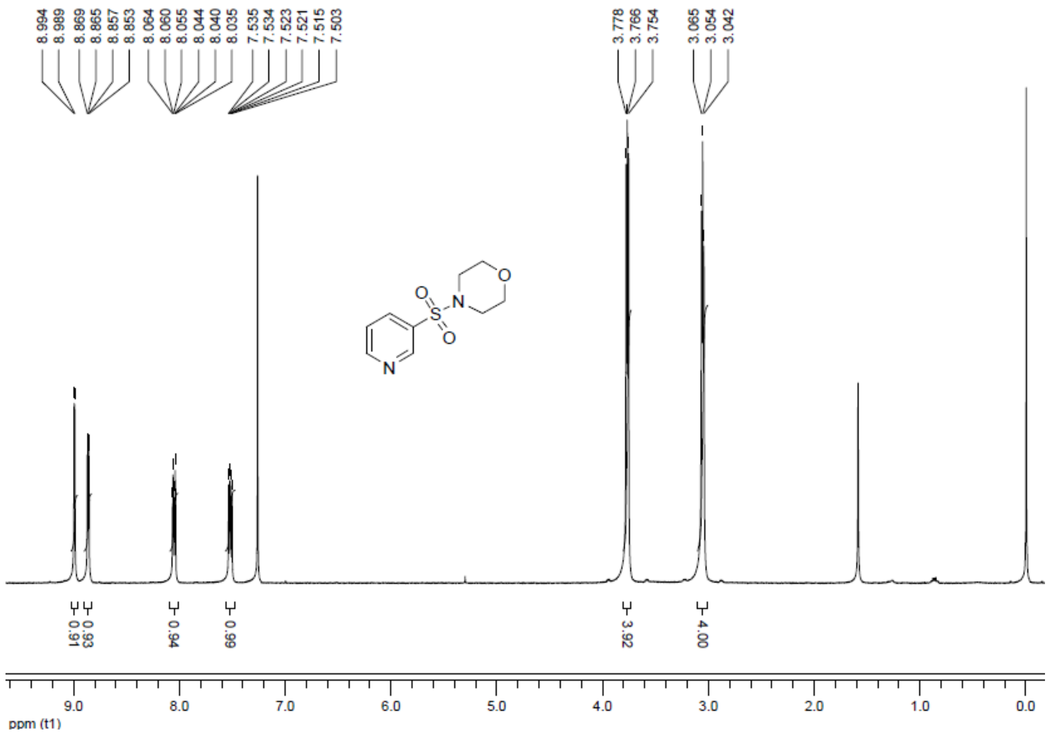

-63.147
F3C
-55.0
-60.0
-65.0
-70.0
ppm (t1)

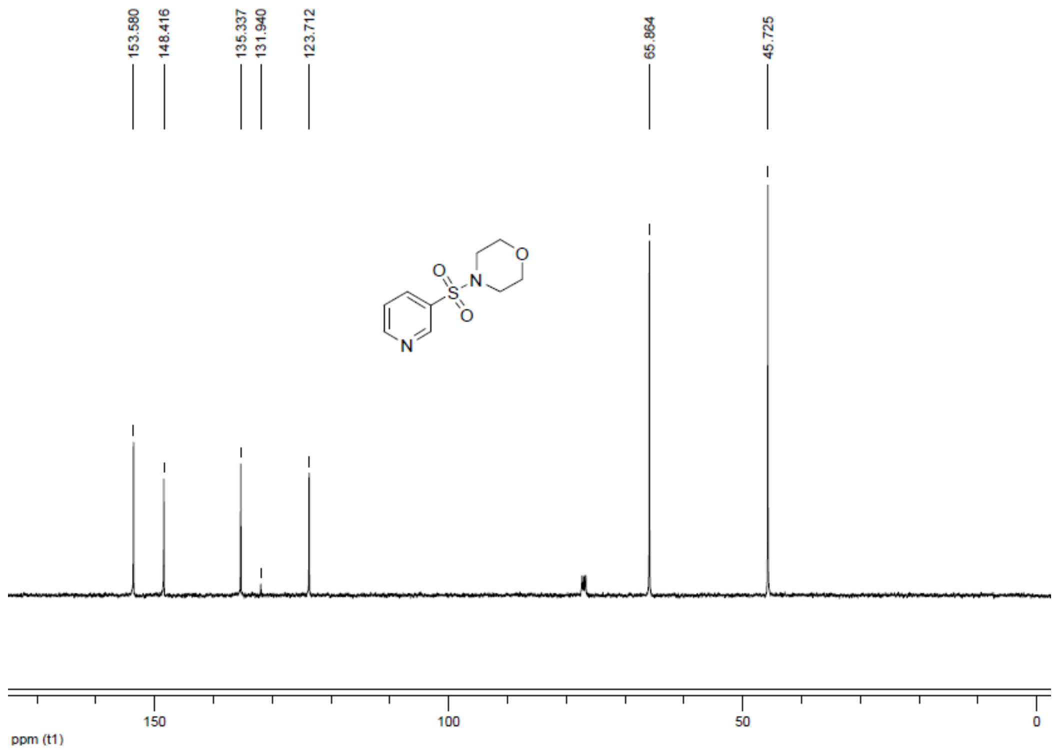

8.337
8.010
8.004
7.989
7.952
7.932
7.761
7.757
7.747
7.740
7.735
7.700
7.697
7.682
7.679
7.663
7.659
7.655
7.639
7.636
7.622
7.619
3.763
3.751
3.739
3.085
3.073
3.062
0.89
4.00
3.98
10.0
5.0
0.0
ppm (t1)

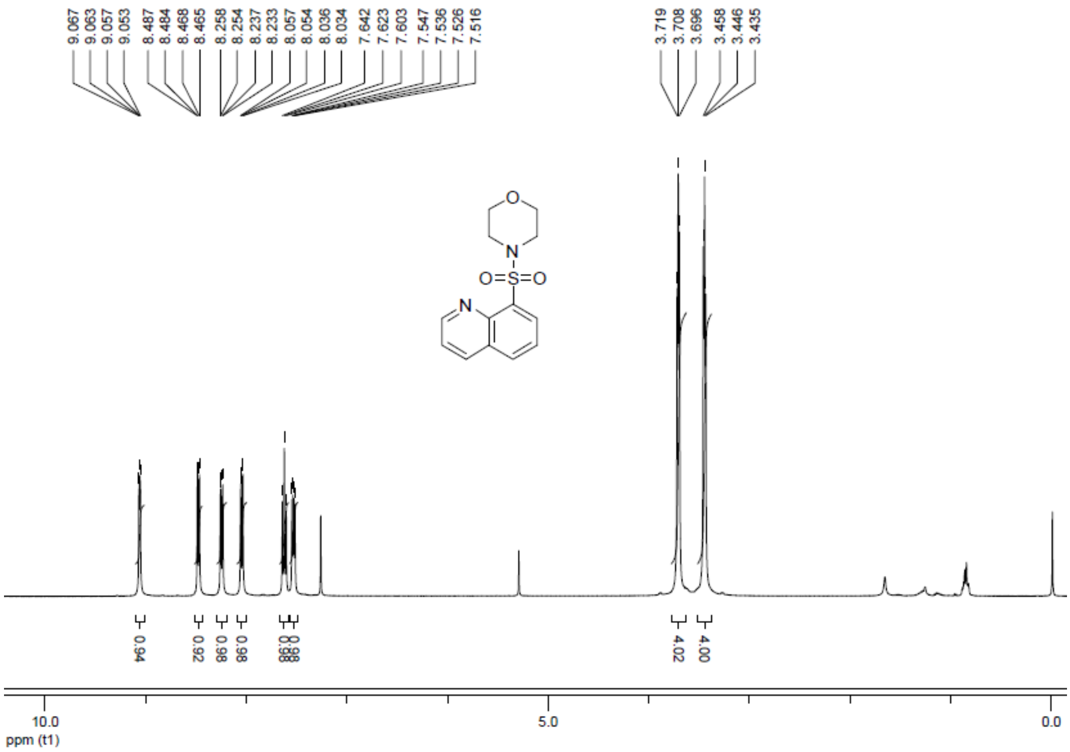

134.970
132.335
132.287
132.181
129.299
129.225
128.971
127.943
127.666
122.945
66.114
46.056
O
S
N
O
150
100
50
0
ppm (t1)

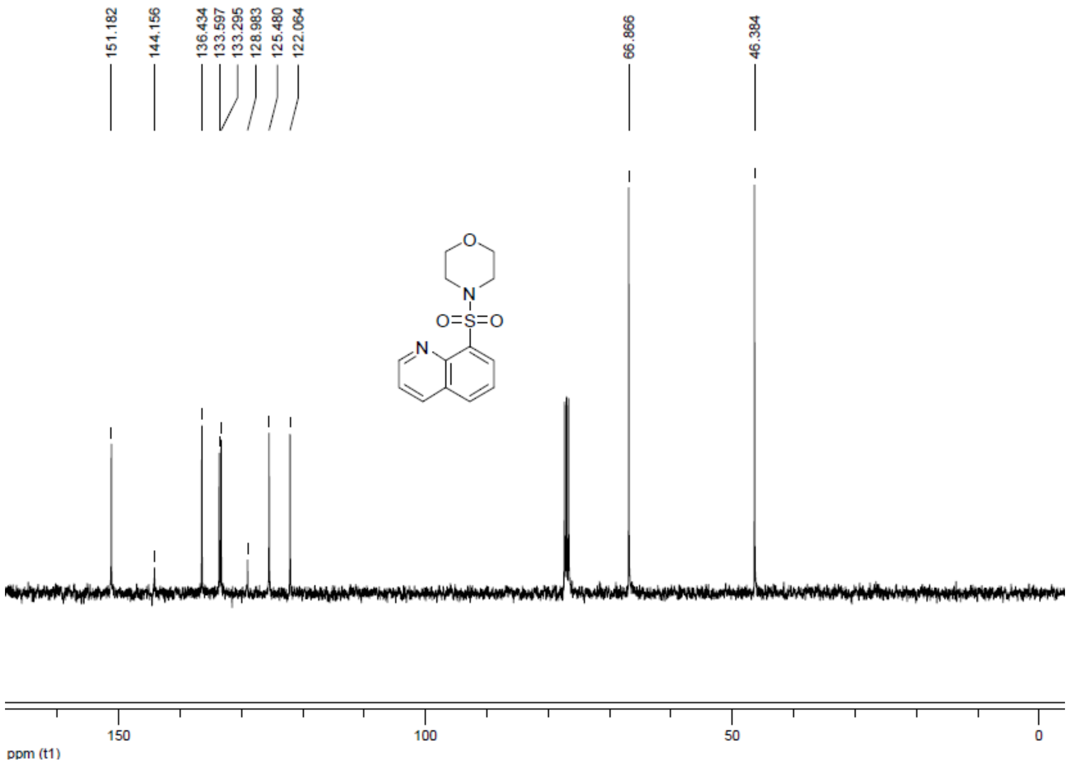

7.662
7.659
7.649
7.646
7.559
7.556
7.550
7.547
7.187
7.178
7.175
7.166
3.794
3.782
3.770
3.071
3.059
3.047
S
O
N
O
0.91
0.91
0.94
4.00
3.97
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

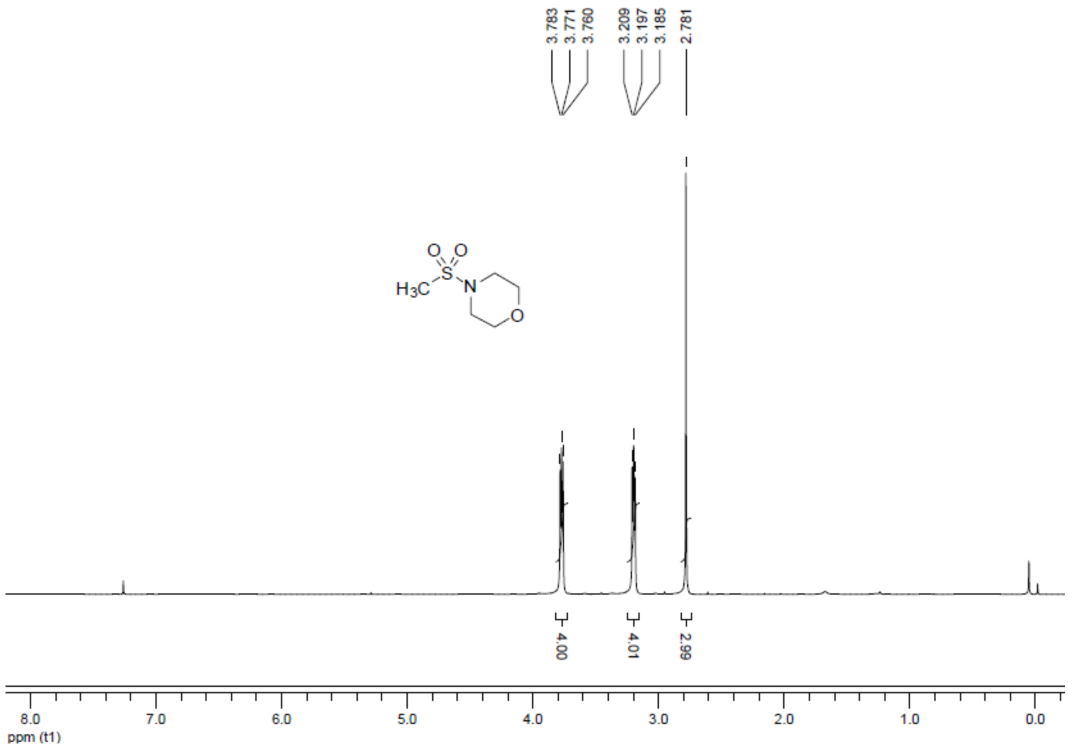

135.269
132.746
132.464
127.762
65.946
45.950
150
100
50
0
ppm (t1)

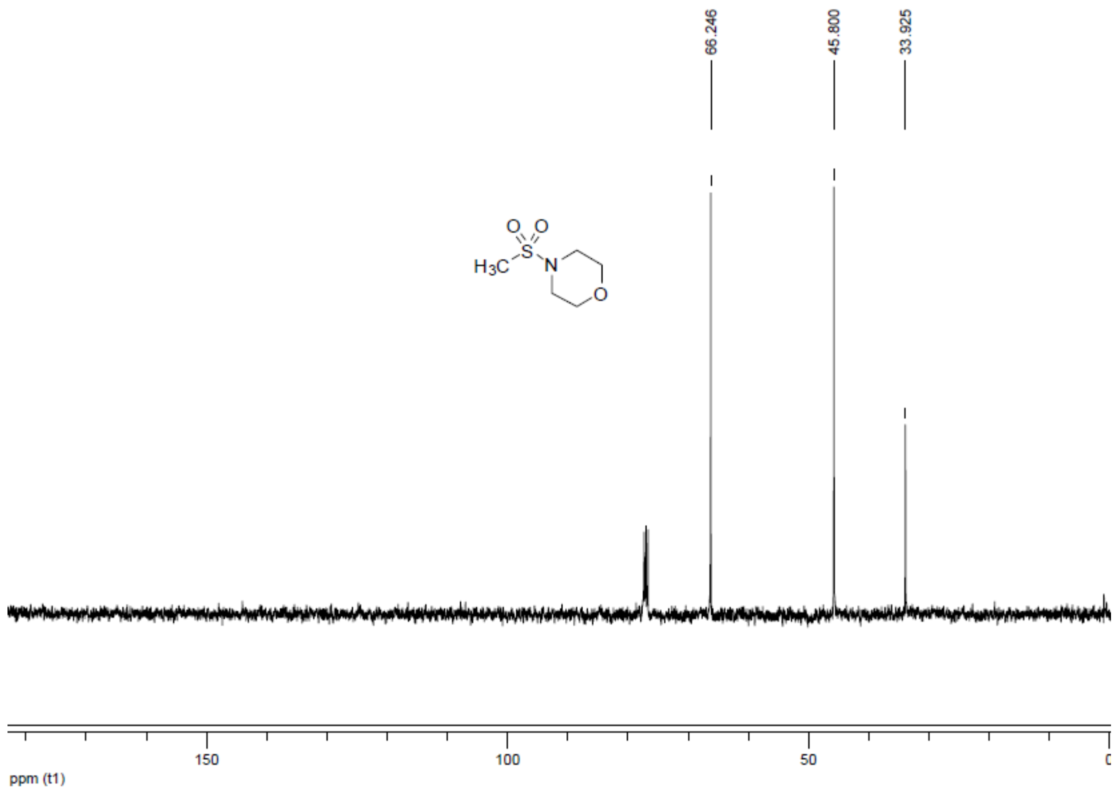

8.994
8.989
8.869
8.865
8.857
8.853
8.064
8.060
8.055
8.044
8.040
8.035
7.535
7.534
7.523
7.521
7.515
7.503
3.778
3.766
3.754
3.065
3.054
3.042
0.91
0.93
0.94
0.99
3.92
4.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)






151.182
144.156
136.434
133.597
133.295
128.983
125.480
122.064
66.866
46.384
N
O
O=S=O
150
100
50
0
ppm (t1)


3.783
3.771
3.760
3.209
3.197
3.185
2.781
O
O
S
H3C
N
O
4.00
4.01
2.99
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)



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