Supporting Information

Synthesis of sulfonamides via I_2-mediated reaction of sodium sulfinates with amines in aqueous medium at room temperature

Xiaojun Pan, Jian Gao, Juan Liu, Junyi Lai, Huanfeng Jiang and Gaoqing Yuan*

School of Chemistry and Chemical Engineering, South China University of Technology, Guangzhou 510640, China
E-mail: gqyuan@scut.edu.cn

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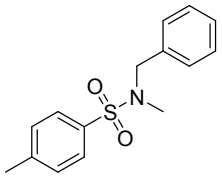
General Information

The ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Advance 400 spectrometer in $CDCl_3$ at 400 MHz and 100 MHz, respectively. The chemical shifts were referenced to signals at 7.26 and 77.0 ppm, respectively. Proton coupling patterns were described as singlet (s), doublet (d), triplet (t), quartet (q) and multiplet (m). Mass spectra were recorded on a Shimadzu GCMS-QP5050A spectrometer at an ionization voltage of 70 eV equipped with a DB-WAX capillary column (internal diameter: 0.25 mm, length: 30 m). GC-MS was obtained using electron ionization. High resolution mass spectra (HRMS) were recorded on a MAT95XP high resolution mass spectrometer. TLC was performed using commercially prepared 100-400 mesh silica gel plates (GF254), and visualization was effected at 254 nm. All substrates were purchased commercially without further purification.

Typical procedure for the synthesis of sulfonamides

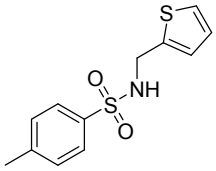
The mixture of iodine (0.25 mmol), sodium sulfinates (0.6 mmol), water (2 mL) and amines (0.5 mmol) were added to a 10 mL tube successively. Then, the reaction was performed at room temperature under magnetic stirring for 3 h. When it was finished, some ethyl acetate was added to the tube to extract the aqueous solution. After extracting, the obtained organic layer was dried with anhydrous $MgSO_4$. The solvent was removed under vacuum, and the crude residue was purified by silica gel column chromatography to afford the desired pure product.

Analytical Data of the prepared compounds



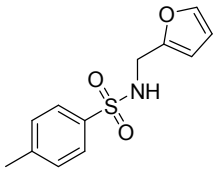
N-Benzyl-N,4-dimethylbenzenesulfonamide (3a)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, *J* =7.7Hz, 2H), 7.36-7.27 (m, 7H), 4.12 (s, 2H), 2.58 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.4, 135.7, 134.3, 129.7, 128.6, 128.3, 127.8, 127.5, 54.1, 34.3, 21.5; MS (EI) *m/z*: 275, 198, 184, 155, 120, 91.



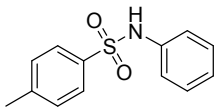
4-Methyl-N-(thiophen-2-ylmethyl)benzenesulfonamide (3b)[2]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, *J* =7.5 Hz, 2H), 7.32 (d, *J* =7.7 Hz, 2H), 7.20 (d, *J* =4.6 Hz, 1H), 6.88 (d, *J* =5.5 Hz, 2H), 5.18 (t, *J* =5.4 Hz, 1H), 4.34 (d, *J* =6.0 Hz, 2H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.5, 139.0, 136.7, 129.6, 127.1, 126.8, 126.4, 125.6, 41.9, 21.4; MS (EI) *m/z*: 267, 207, 139, 112, 91.



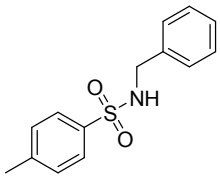
N-(Furan-2-ylmethyl)-4-methylbenzenesulfonamide (3c)[3]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* =7.6 Hz, 2H), 7.25 (d, *J* =7.8 Hz, 2H), 7.21 (s, 1H), 6.19 (s, 1H), 6.08 (s, 1H), 5.18 (br, 1H), 4.14 (d, *J* =5.8 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.6, 143.3, 142.3, 136.8, 129.5, 127.0, 110.3, 108.1, 40.0, 21.4; MS (EI) *m/z*: 251, 186, 155, 139, 96.



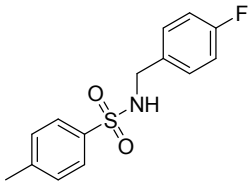
4-Methyl-N-phenylbenzenesulfonamide (3d)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* =8.2 Hz, 2H), 7.59 (br, 1H), 7.21-7.03 (m, 7H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.8, 136.6, 135.9, 129.6, 129.2, 127.2, 125.0, 121.2, 21.4; MS (EI) *m/z*: 247, 182, 168, 155, 91.



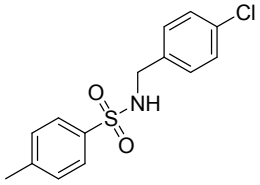
N-Benzyl-4-methylbenzenesulfonamide ($3e_1$)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* =7.7 Hz, 2H), 7.31-7.18 (m, 7H), 4.79 (br, 1H), 4.11 (d, *J* =5.9 Hz, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.4, 136.8, 136.3, 129.6, 128.6, 127.8, 127.7, 127.1, 47.1, 21.4; MS (EI) *m/z*: 261, 196, 155, 106, 91.



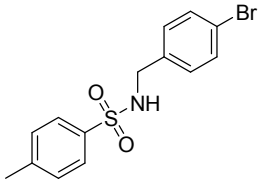
N-(4-Fluorobenzyl)-4-methylbenzenesulfonamide ($3e_2$)[4]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* =7.6 Hz, 2H), 7.27 (d, *J* =7.6 Hz, 2H), 7.16-7.13 (m, 2H), 6.93-6.89(m, 2H), 5.24 (t, *J* =5.6 Hz, 1H), 4.06 (d, *J* =6.1 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.4, 161.0, 143.5, 136.8, 132.2, 129.7, 129.6, 129.5, 127.0, 115.5, 115.3, 46.4, 21.4; MS (EI) *m/z*: 279, 214, 155, 124, 91.



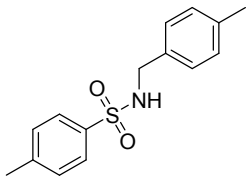
N-(4-Chlorobenzyl)-4-methylbenzenesulfonamide ($3e_3$)[4]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, *J* =7.5 Hz, 2H), 7.25 (d, *J* =7.8 Hz, 2H), 7.18 (d, *J* =7.4 Hz, 2H), 7.10 (d, *J* =7.8 Hz, 2H), 5.42 (br, 1H), 4.04 (d, *J* =6.3 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.5, 136.7, 134.9, 133.4, 129.6, 129.2, 128.6, 127.0, 46.4, 21.4; MS (EI) *m/z*: 295, 230, 140, 125, 91.



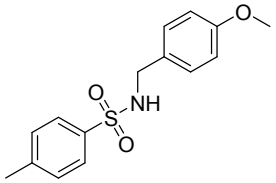
N-(4-Bromobenzyl)-4-methylbenzenesulfonamide ($3e_4$)[4]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, *J* =8.0 Hz, 2H), 7.33 (d, *J* =8.1 Hz, 2H), 7.25 (d, *J* = 7.7 Hz, 2H), 7.05 (d, *J* =8.0 Hz, 2H), 5.37 (br, 1H), 4.03 (d, *J* =4.2 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.6, 136.7, 135.5, 131.6, 129.7, 129.5, 127.0, 121.6, 46.4, 21.4; MS (EI) *m/z*: 339, 207, 184, 157, 91.



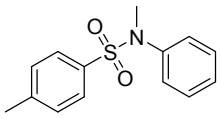
4-Methyl-N-(4-methylbenzyl)benzenesulfonamide ($3e_5$)[4]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* =7.7 Hz, 2H), 7.24 (d, *J* =7.7Hz, 2H), 7.06-7.01 (m, 4H), 5.18 (br, 1H), 4.01 (d, *J* =6.0 Hz, 2H), 2.39 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$): δ 143.2, 137.3, 136.8, 133.2, 129.5, 129.1, 127.7, 127.0, 46.8, 21.3, 20.9; MS (EI) *m/z*: 275, 210, 139, 120, 91.



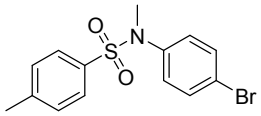
N-(4-Methoxybenzyl)-4-methylbenzenesulfonamide (3e$_6$)[5]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* =7.4 Hz, 2H), 7.25 (d, *J* =7.3 Hz, 2H), 7.08 (d, *J* =7.4 Hz, 2H), 6.75 (d, *J* =7.4 Hz, 2H), 5.22 (br, 1H), 4.00 (d, *J* =3.8 Hz, 2H), 3.73 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.0, 143.2, 136.8, 129.5, 129.1, 128.3, 127.0, 113.8, 55.1, 46.5, 21.3; MS (EI) *m/z*: 291, 155, 135, 121, 91.



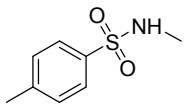
N,4-Dimethyl-N-phenylbenzenesulfonamide (3f$_1$)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, *J* =7.8 Hz, 2H), 7.31-7.22 (m, 5H), 7.10 (d, *J* =7.6 Hz, 2H), 3.16 (s, 3H), 2.41(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.5, 141.6, 133.6, 129.3, 128.8, 127.9, 127.2, 126.6, 38.1, 21.5; MS (EI) *m/z*: 261, 197, 155, 106, 91, 77.



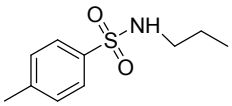
N-(4-Bromophenyl)-N,4-dimethylbenzenesulfonamide (3f$_2$)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 (d, *J* =8.5 Hz, 4H), 7.24 (d, *J* =7.7 Hz, 2H), 6.97 (d, *J* =7.3 Hz, 2H), 3.12 (s, 3H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.8, 140.6, 132.9, 131.8, 129.4, 128.0, 127.7, 120.8, 37.8, 21.4; MS (EI) *m/z*: 340, 196, 155, 105, 91; HRMS (ESI) *m/z*: calcd for $C_{14}H_{14}BrNNaO_2S$ [M+Na]+, 361.9827; found 361.9821.



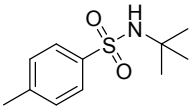
N,4-Dimethylbenzenesulfonamide (3g$_1$)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, *J* =8.1Hz, 2H), 7.33 (d, *J* =8.0 Hz, 2H), 4.65 (br, 1H), 2.65 (d, *J* =5.0 Hz, 3H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.5, 135.8, 129.7, 127.2, 29.3, 21.5; MS (EI) *m/z*: 185, 155, 121, 91.



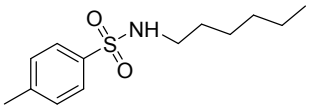
4-Methyl-N-propylbenzenesulfonamide (3g$_2$)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, *J* =7.7 Hz, 2H), 7.33(d, *J* =7.7 Hz, 2H), 4.76 (br, 1H), 2.94-2.89 (m, 2H), 2.45 (s, 3H), 1.54-1.45 (m, 2H), 0.88 (t, *J* =7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.3, 137.0, 129.6, 127.0, 44.9, 22.9, 21.4, 11.0; MS (EI) *m/z*: 213, 184, 155, 91.



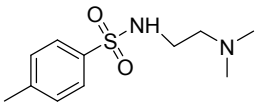
N-Tert-butyl-4-methylbenzenesulfonamide (3g$_3$)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, *J* =8.2 Hz, 2H), 7.27 (d, *J* =8.3 Hz, 2H), 4.63 (br, 1H), 2.42 (s, 3H), 1.22 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.8, 140.5, 129.4, 127.0, 54.6, 30.1, 21.4; MS (EI) *m/z*: 227, 212, 155, 91.



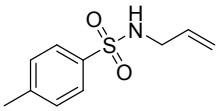
N-Hexyl-4-methylbenzenesulfonamide (3g$_4$)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* =8.1 Hz, 2H), 7.30 (d, *J* =8.0 Hz, 2H), 4.57 (br, 1H), 2.95-2.90 (m, 2H), 2.43 (s, 3H), 1.47-1.40 (m, 2H), 1.26-1.21 (m, 6H), 0.84 (t, *J* =6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.3, 137.0, 129.6, 127.1, 43.2, 31.2, 29.5, 26.1, 22.4, 21.5, 13.9; MS (EI) *m/z*: 255, 184, 155, 100, 91.



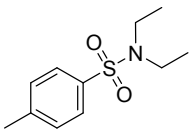
N-(2-(Dimethylamino)ethyl)-4-methylbenzenesulfonamide (3h)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, *J* =7.7 Hz, 2H), 7.31 (d, *J* =7.6 Hz, 2H), 2.98-2.96 (m, 2H), 2.43 (s, 3H), 2.35-2.32 (m, 2H), 2.09 (s, 6H), 1.26 (br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.3, 136.7, 129.6, 127.1, 57.0, 44.7, 40.0, 21.5; MS (EI) *m/z*: 242, 213, 155, 148, 91; HRMS (ESI) *m/z*: calcd for $C_{11}H_{18}N_2NaO_2S$ [M+Na]+, 265.0980; found 265.0981.



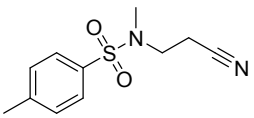
N-Allyl-4-methylbenzenesulfonamide (3i)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, *J* =8.3 Hz, 2H), 7.33 (d, *J* =8.0 Hz, 2H), 5.79-5.70 (m, 1H), 5.21-5.10 (m, 2H), 4.55 (br, 1H), 3.61 (t, *J* = 6.0 Hz, 2H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.5, 137.0, 133.0, 129.7, 127.1, 117.7, 45.8, 21.5; MS (EI) *m/z*: 211, 184, 155, 91.



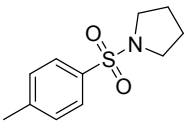
N,N-Diethyl-4-methylbenzenesulfonamide (3j)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* =7.6 Hz, 2H), 7.30 (d, *J* =8.0 Hz, 2H), 3.24 (q, *J* =7.1 Hz, 4H), 2.43 (s, 3H), 1.14 (t, *J* =7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.9, 137.4, 129.5, 127.0, 42.0, 21.4, 14.1; MS (EI) *m/z*: 227, 184, 155, 91.



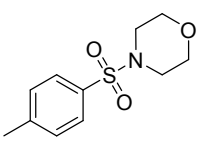
N-(2-cyanoethyl)-N,4-dimethylbenzenesulfonamide (3k)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, *J* =7.7 Hz, 2H), 7.35 (d, *J* =7.8 Hz, 2H), 3.33 (t, *J* =6.8 Hz, 2H), 2.86 (s, 3H), 2.67 (t, *J* =6.8 Hz, 2H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.0, 134.0, 129.9, 127.2, 117.4, 46.3, 36.0, 21.4, 18.1; MS (EI) *m/z*: 238, 198, 155, 91; HRMS (ESI) *m/z*: calcd for $C_{11}H_{14}N_2NaO_2S$ [M+Na]+, 261.0674; found 261.0668.



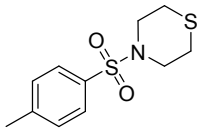
1-Tosylpyrrolidine (3l)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 (d, *J* =8.0 Hz, 2H), 7.32 (d, *J* =8.0 Hz, 2H), 3.23 (t, *J* =6.6 Hz, 4H), 2.43 (s, 3H), 1.78-1.72(m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.2, 133.9, 129.5, 127.5, 47.8, 25.1, 21.4; MS (EI) *m/z*: 225, 155, 91.



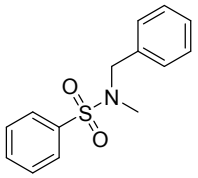
4-Tosylmorpholine (3m)[1]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, *J* =8.1 Hz, 2H), 7.35 (d, *J* =8.0 Hz, 2H), 3.74(t, *J* =4.8 Hz, 4H), 2.98 (t, *J* =4.8 Hz, 4H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.9, 132.0, 129.7, 127.8, 66.0, 45.9, 21.5; MS (EI) *m/z*: 241, 198, 155, 106, 91, 86.



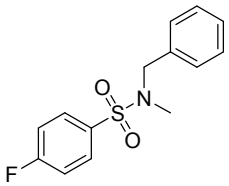
4-Tosylthiomorpholine (3n)[6]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (d, *J* =7.7 Hz, 2H), 7.35 (d, *J* =7.8 Hz, 2H), 3.34 (s, 4H), 2.72 (s, 4H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.7, 133.7, 129.8, 127.4, 47.8, 27.3, 21.5; MS (EI) *m/z*: 257, 242, 193, 155, 102, 91.



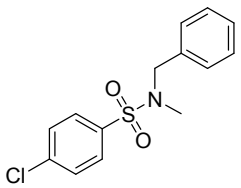
N-Benzyl-N-methylbenzenesulfonamide ($3o_1$)[7]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* =7.6 Hz, 2H), 7.64-7.54 (m, 3H), 7.35-7.27 (m, 5H), 4.15 (s, 2H), 2.61 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.4, 135.5, 132.6, 129.1, 128.6, 128.3, 127.9, 127.4, 54.1, 34.3; MS (EI) *m/z*: 261, 184, 141, 118, 91.



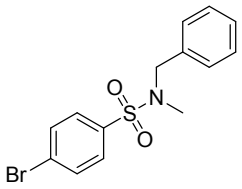
N-Benzyl-4-fluoro-N-methylbenzenesulfonamide ($3o_2$)[7]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87-7.84 (m, 2H), 7.33-7.21 (m, 7H), 4.15 (s, 2H), 2.61 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.3, 163.8, 135.3, 133.6, 130.1, 130.0, 128.6, 128.3, 127.9, 116.4, 116.2, 54.0, 34.2; MS (EI) *m/z*: 279, 202, 120, 91.



N-Benzyl-4-chloro-N-methylbenzenesulfonamide ($3o_3$)[7]

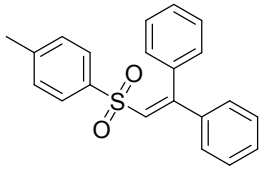
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, *J* =7.9 Hz, 2H), 7.53 (d, *J* =8.0 Hz, 2H), 7.35-7.26 (m, 5H), 4.15 (s, 2H), 2.61 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.2, 136.0, 135.2, 129.4, 128.8, 128.6, 128.3, 128.0, 54.0, 34.2; MS (EI) *m/z*: 295, 218, 175, 120, 91.



N-Benzyl-4-bromo-N-methylbenzenesulfonamide (3o$_4$)[7]

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (s, 4H), 7.35-7.26 (m, 5H), 4.15 (s, 2H), 2.61 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.5, 135.2, 132.4, 128.9, 128.7, 128.3, 128.0, 127.6, 54.0, 34.3; MS (EI) *m/z:* 339, 262, 155, 120, 91.

2-Tosylethene-1,1-diyldibenzene (4a)[8]



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J* =8.2 Hz, 2H), 7.38-7.33 (m, 2H), 7.29 (td, *J* =7.4, 1.6 Hz, 4H), 7.20-7.18 (m, 2H), 7.14 (d, *J* =8.1 Hz, 2H), 7.10-7.08 (m, 2H), 6.99 (s, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.6, 143.7, 139.2, 138.6, 135.5, 130.2, 129.7, 129.3, 128.9, 128.8, 128.5, 128.1, 127.7, 127.6, 21.5; MS (EI) *m/z:* 334, 269, 178, 167, 139, 91.

References

1. X. D. Tang, L. B. Huang, C. R. Qi, X. Wu, W. Q. Wu and H. F. Jiang, *Chem. Commun.* 2013, **49**, 6102.
2. F. Shi, M. K. Tse, X. J. Cui, D. Gördes, D. Michalik, K. Thurow, Y. Q. Deng and M. Beller, *Angew. Chem., Int. Ed.* 2009, **48**, 5912.
3. A. Kamal, J. S. Reddy, E. V. Bharathi and D. Dastagiri, *Tetrahedron Letters.* 2008, **49**, 348.
4. X. J. Cui, F. Shi, Y. Zhang and Y. Q. Deng, *Tetrahedron Letters.* 2010, **51**, 2048.
5. C. Z. Liu, S. H. Liao, Q. Li, S. L. Feng, Q. Sun, X. C. Yu and Q. Xu, *J. Org. Chem.* 2011, **76**, 5759.
6. K. Saruta, T. Ogiku and K. Fukase, *Tetrahedron Letters.* 2009, **50**, 4364.
7. D. A. Powell and H. Fan, *J. Org. Chem.* 2010, **75**, 2726.
8. D. C. Reeves, S. Rodriguez, H. Lee, N. Haddad, D. Krishnamurthy and C. H. Senanayake, *Tetrahedron Letters.* 2009, **50**, 2870.

NMR spectra of the prepared compounds

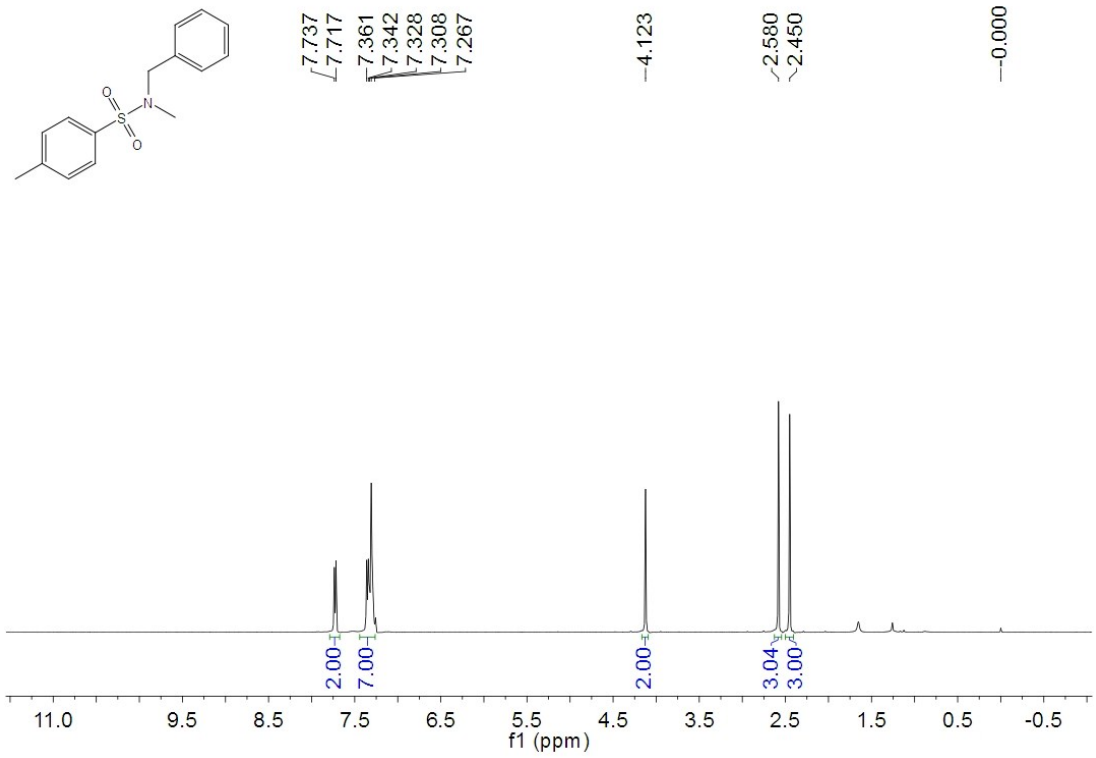


Fig. S-1. ^{1}H-NMR spectrum of **3a**.

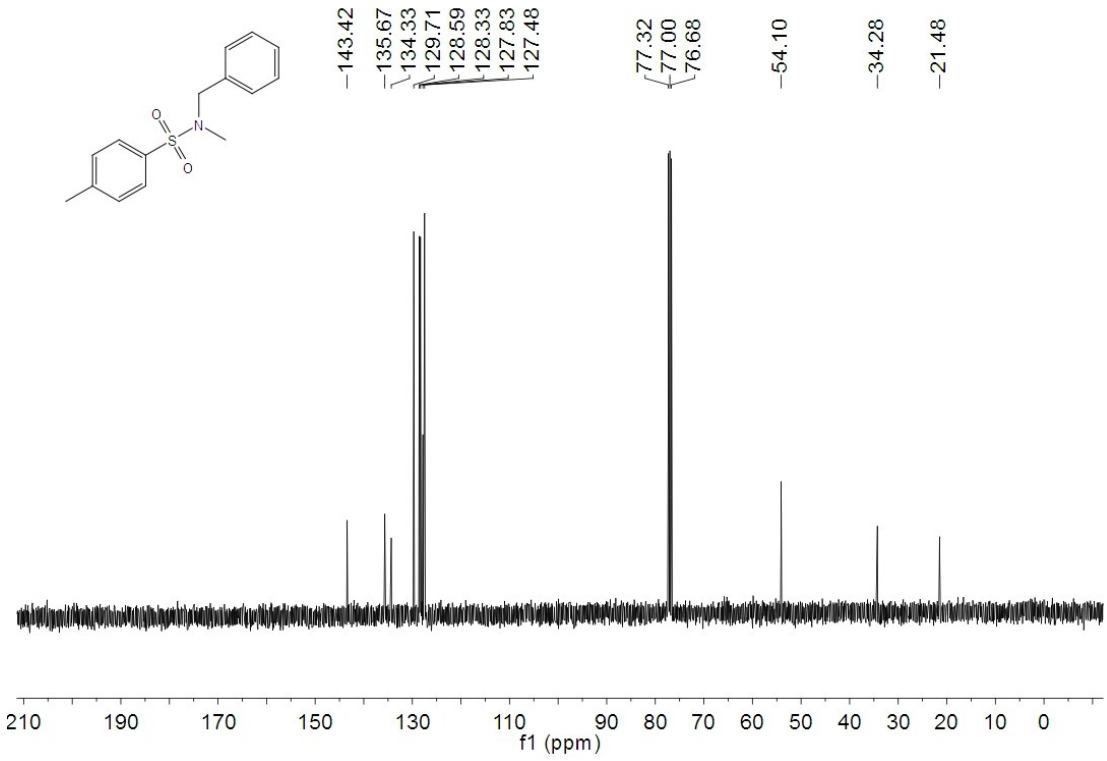


Fig. S-2. ^{13}C-NMR spectrum of **3a**.

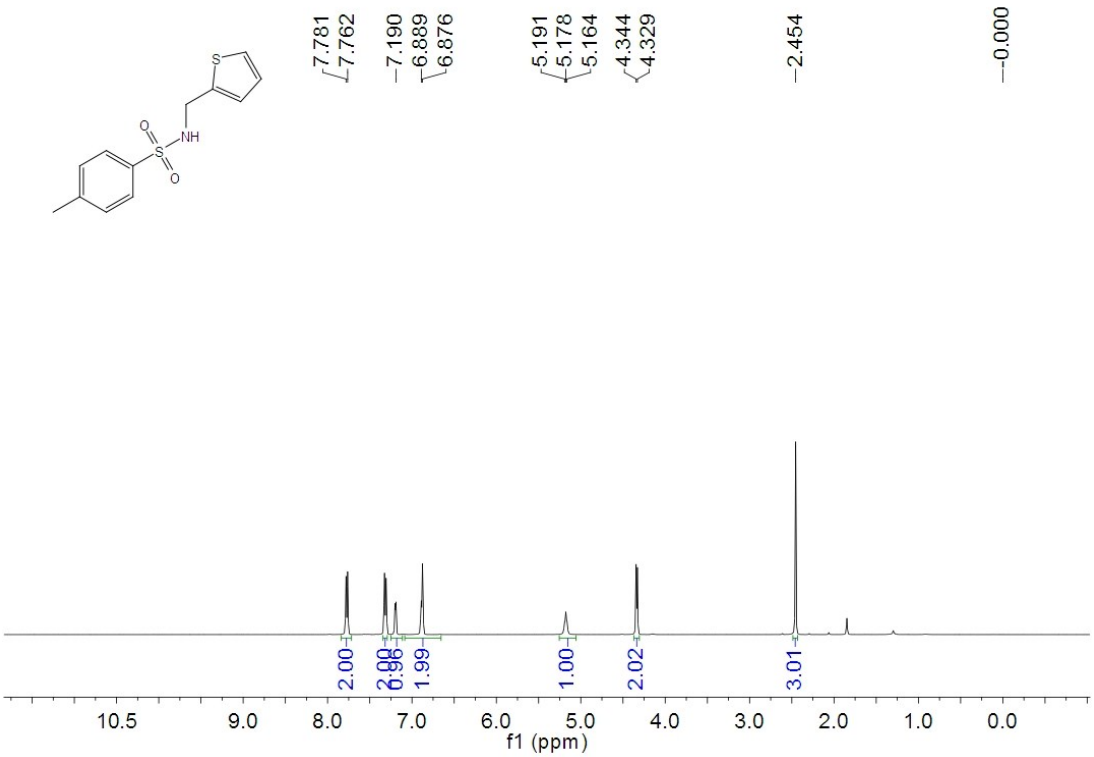


Fig. S-3. ^{1}H-NMR spectrum of **3b**.

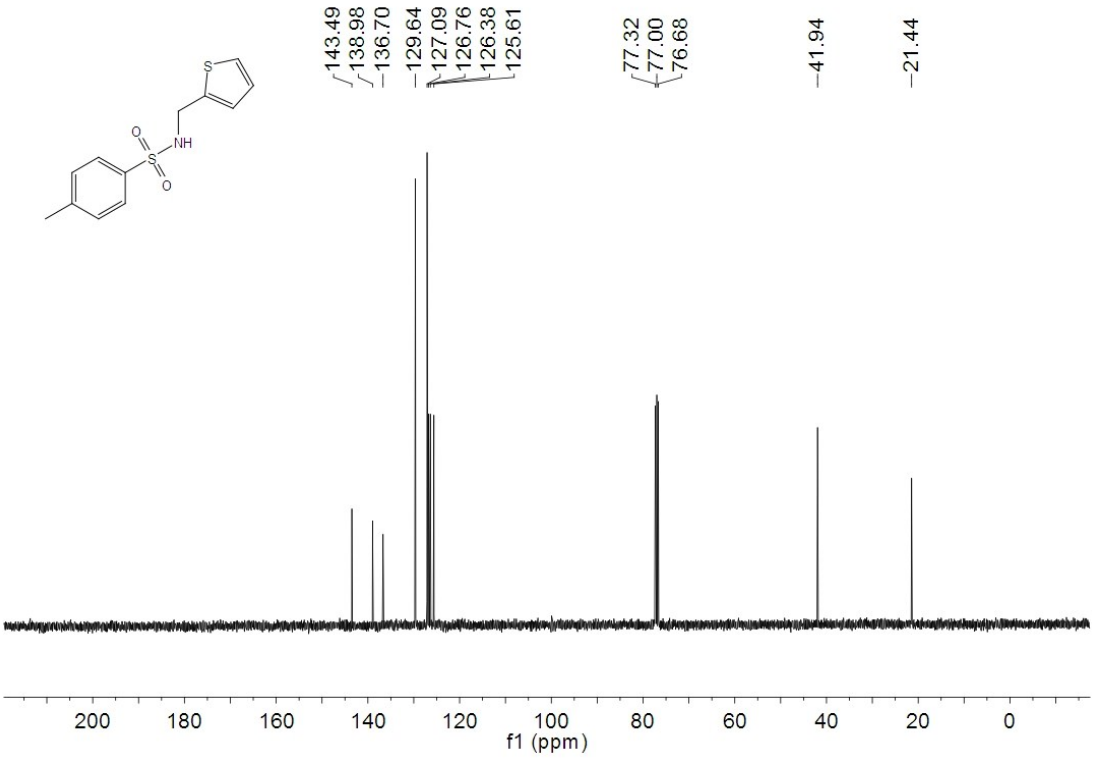


Fig. S-4. ^{13}C-NMR spectrum of **3b**.

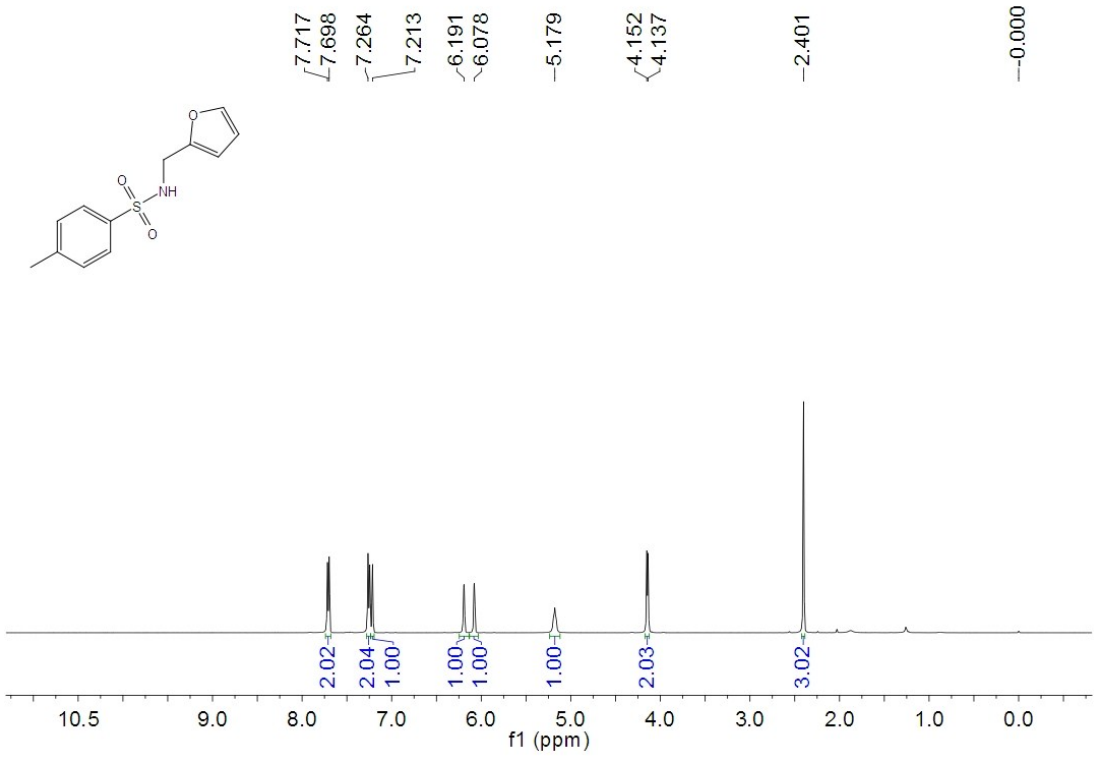


Fig. S-5. ^{1}H-NMR spectrum of **3c**.

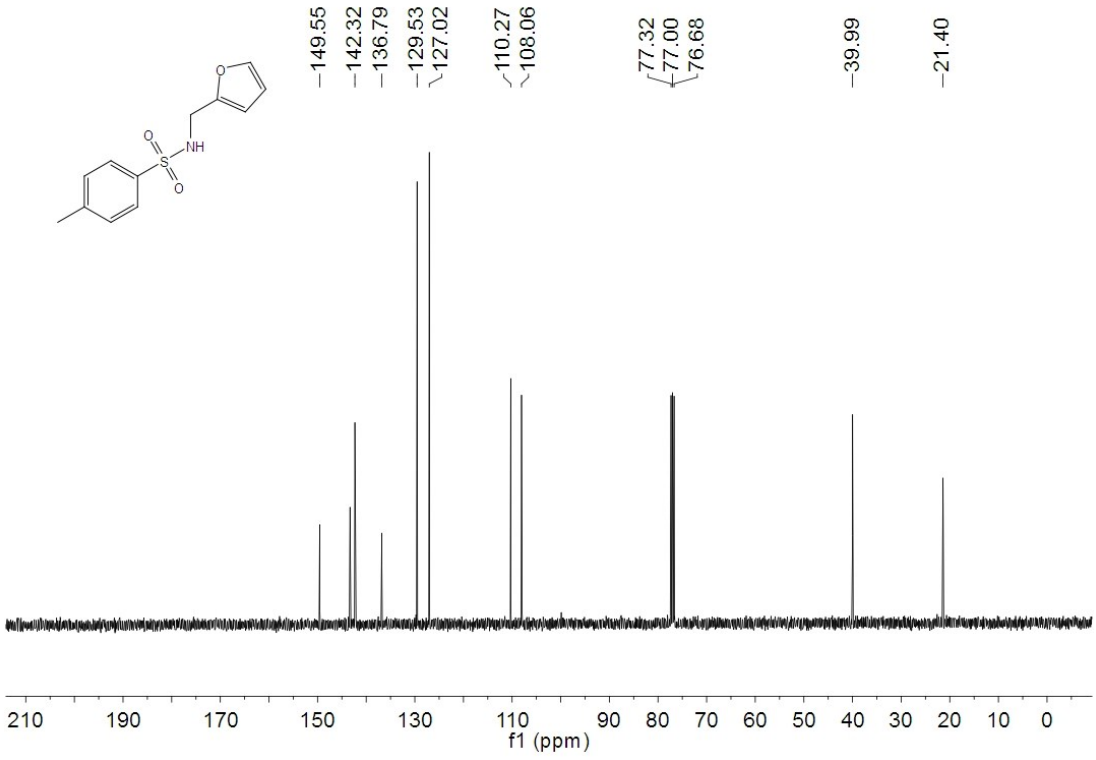


Fig. S-6. ^{13}C-NMR spectrum of **3c**.

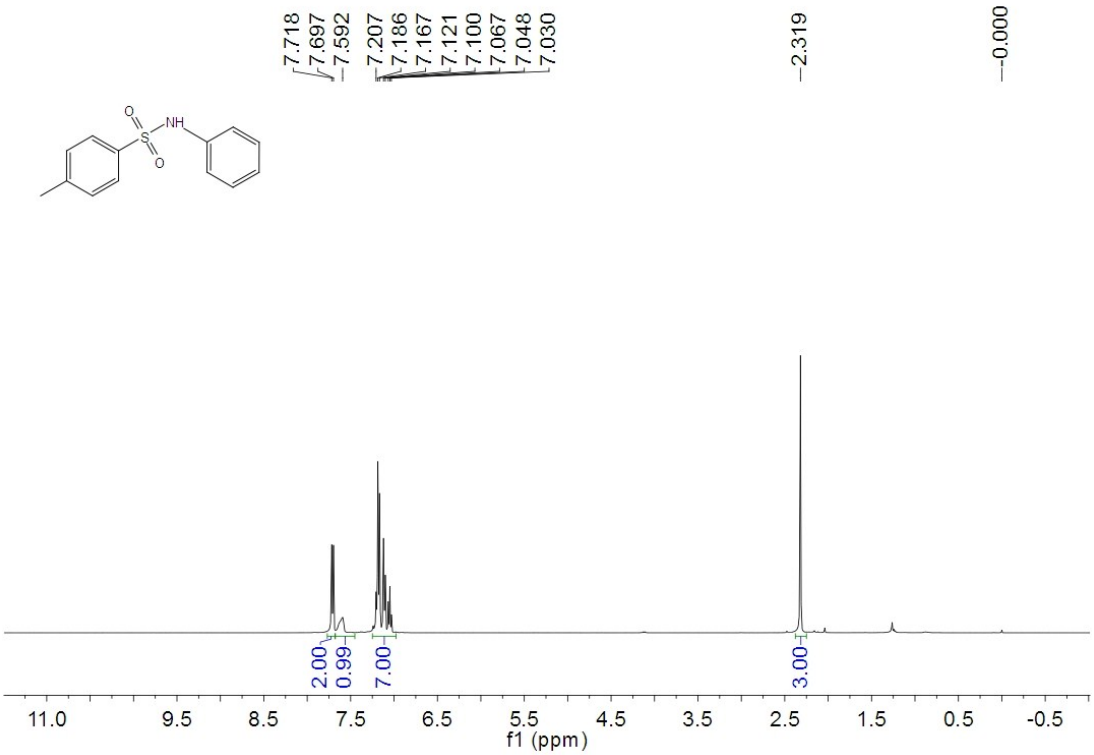


Fig. S-7. ^{1}H-NMR spectrum of **3d**.

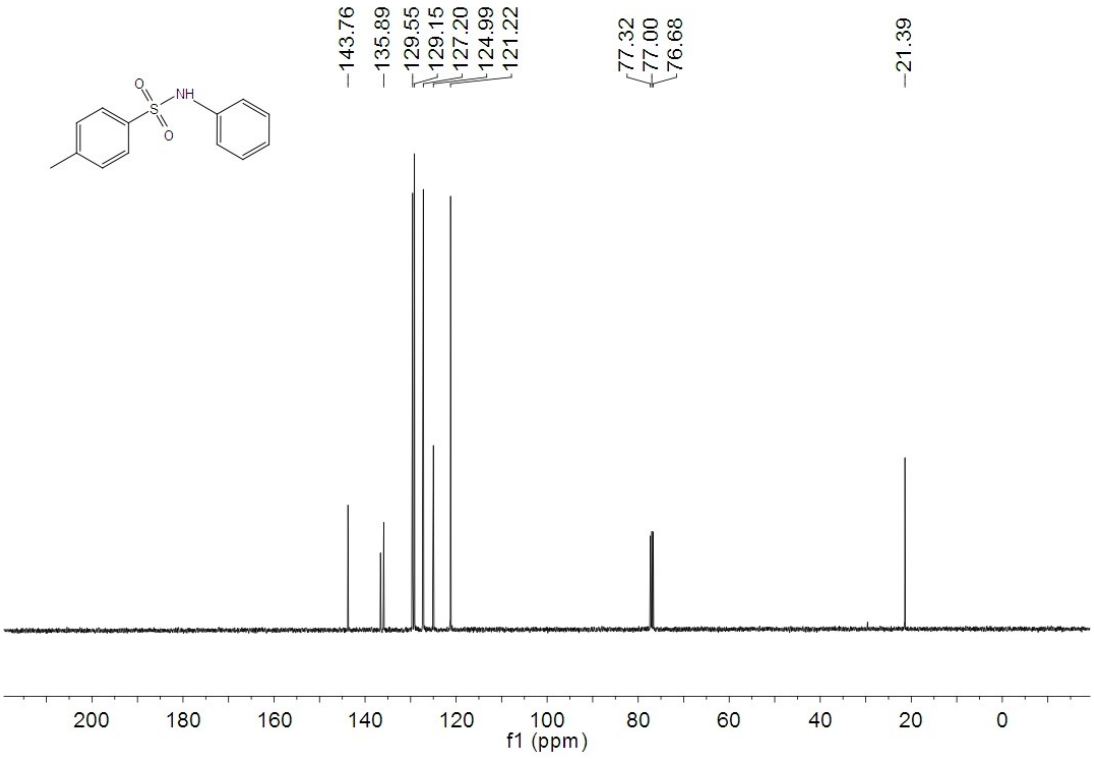


Fig. S-8. ^{13}C-NMR spectrum of **3d**.

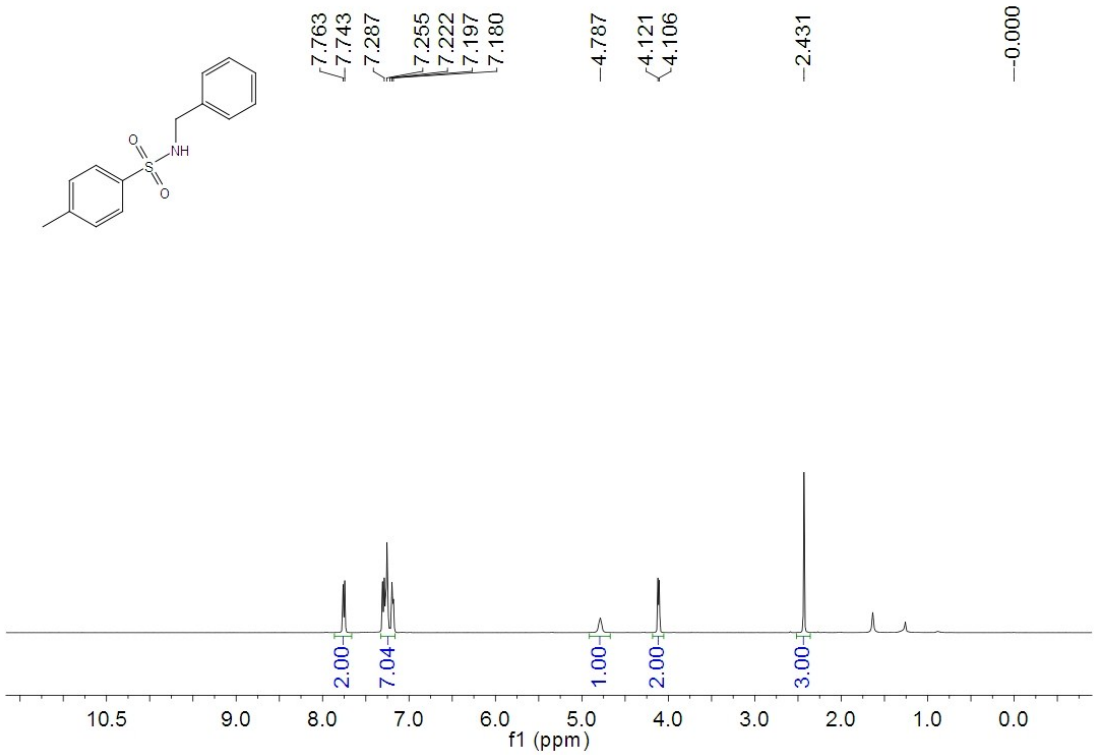


Fig. S-9. ^{1}H-NMR spectrum of **3e$_1$**.

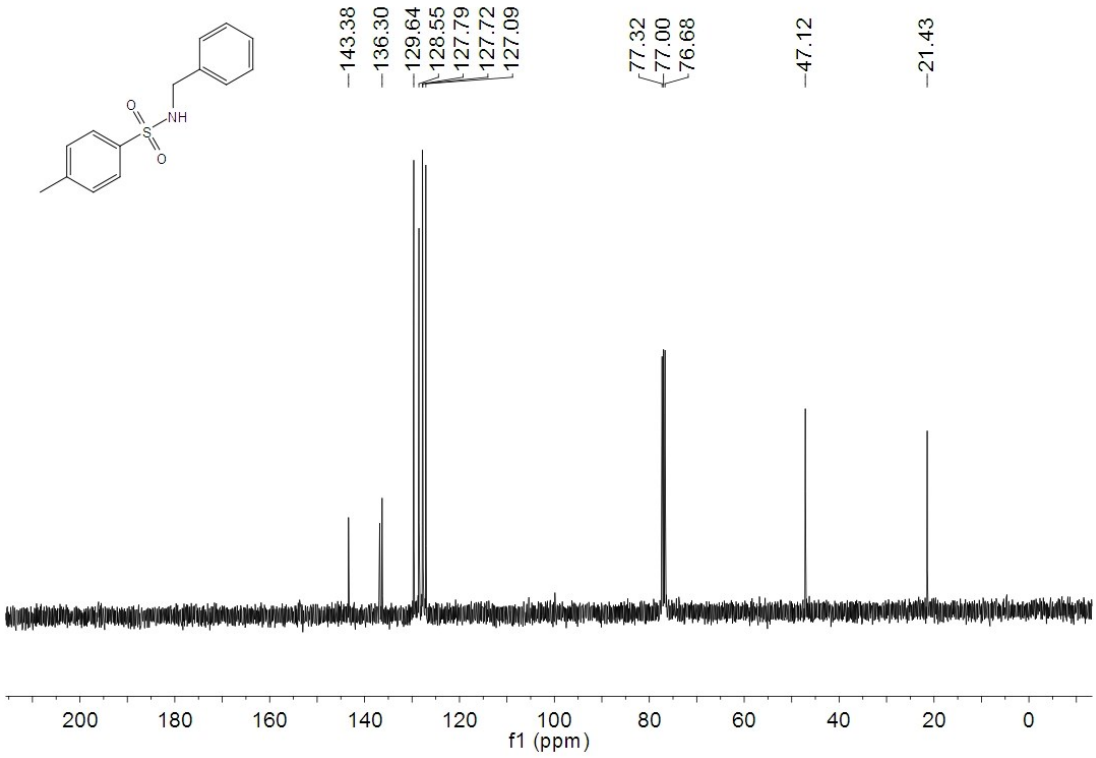


Fig. S-10. ^{13}C-NMR spectrum of **3e$_1$**.

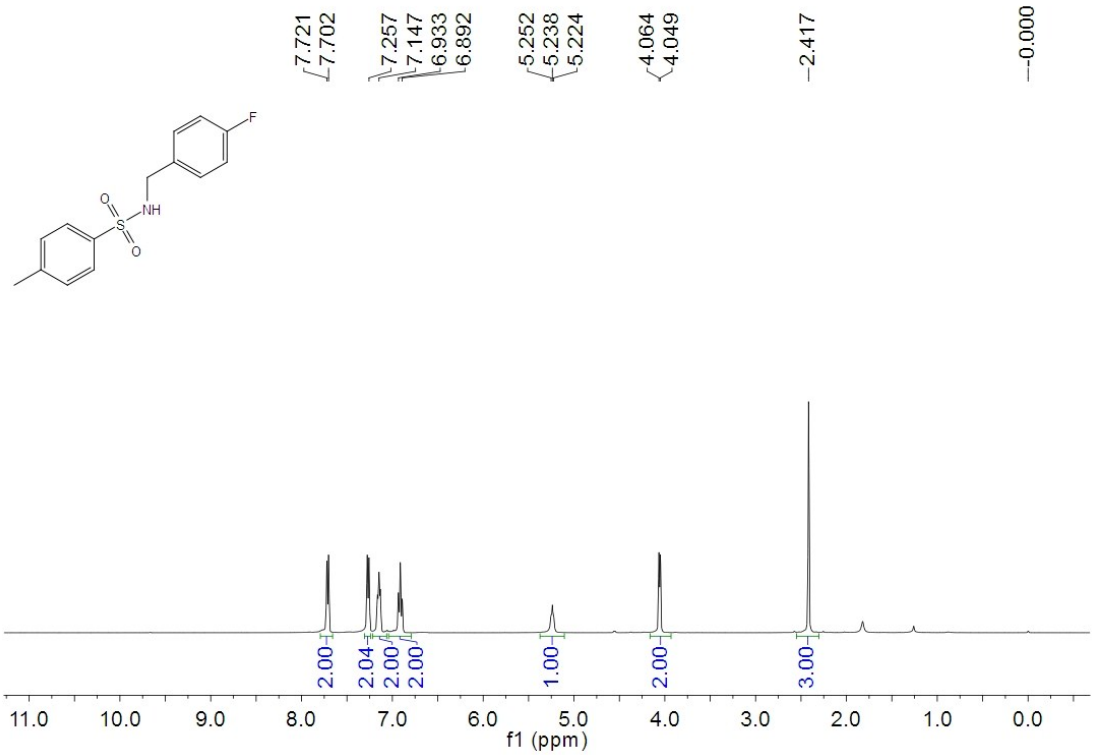


Fig. S-11. ^{1}H-NMR spectrum of **3e$_2$**.

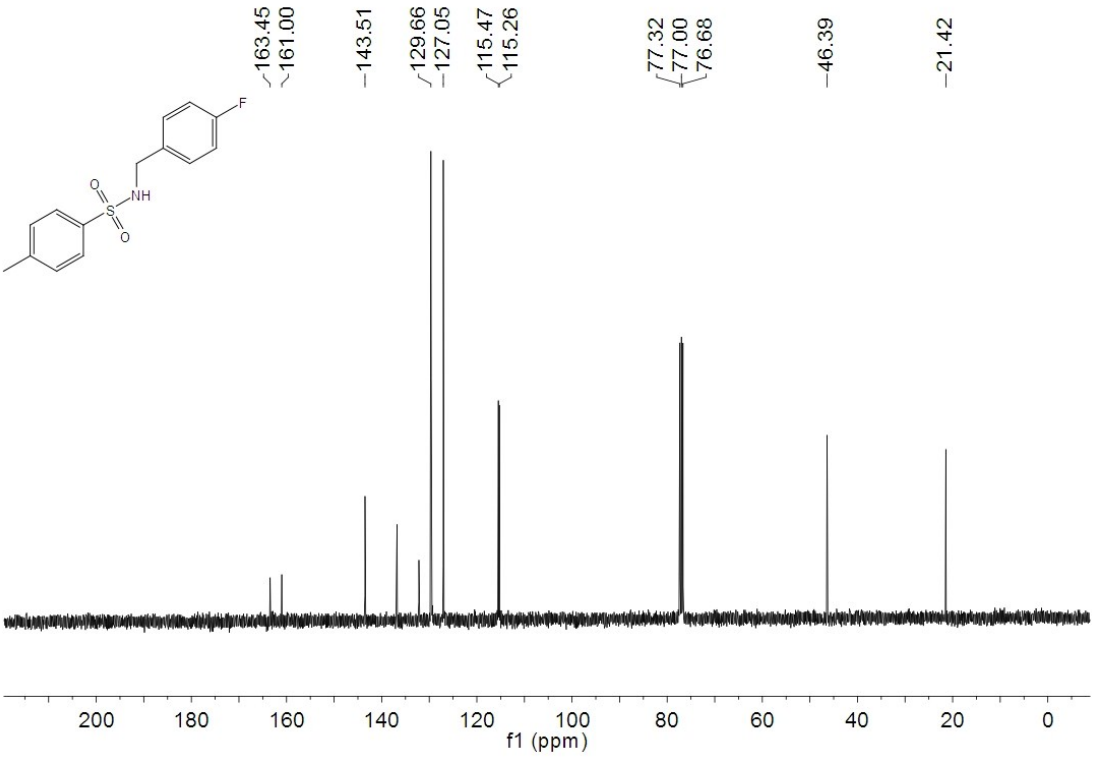


Fig. S-12. ^{13}C-NMR spectrum of **3e$_2$**.

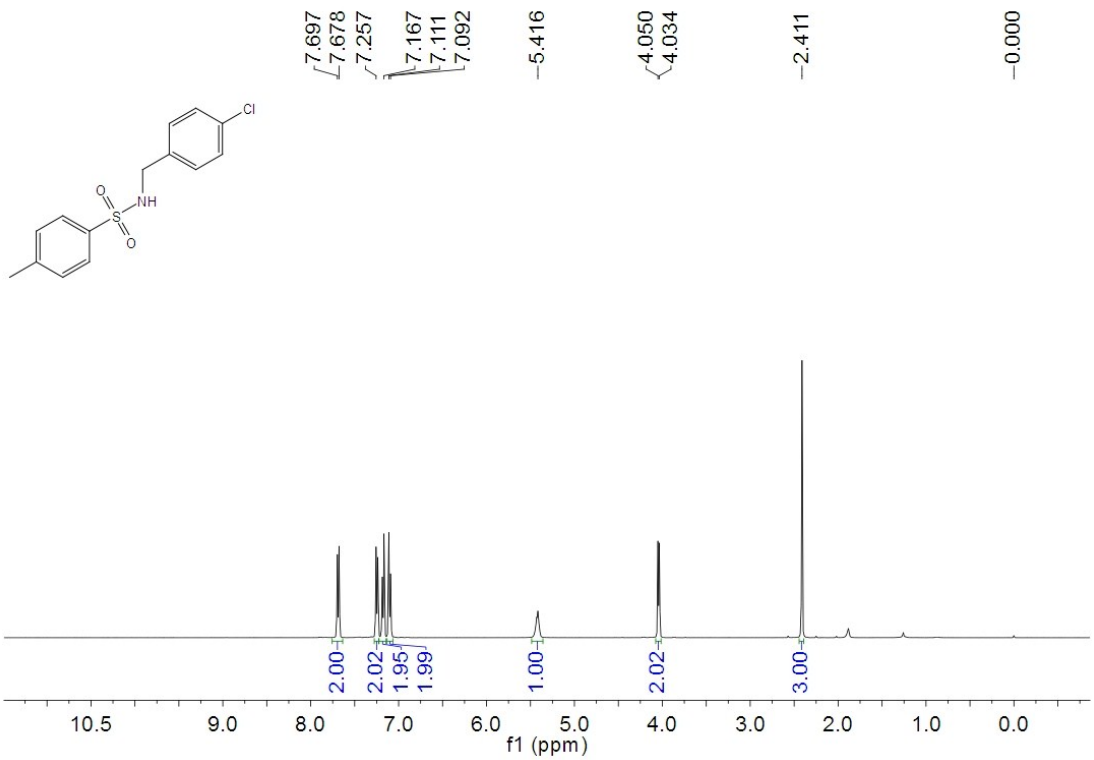


Fig. S-13. ^{1}H-NMR spectrum of **3e$_3$**.

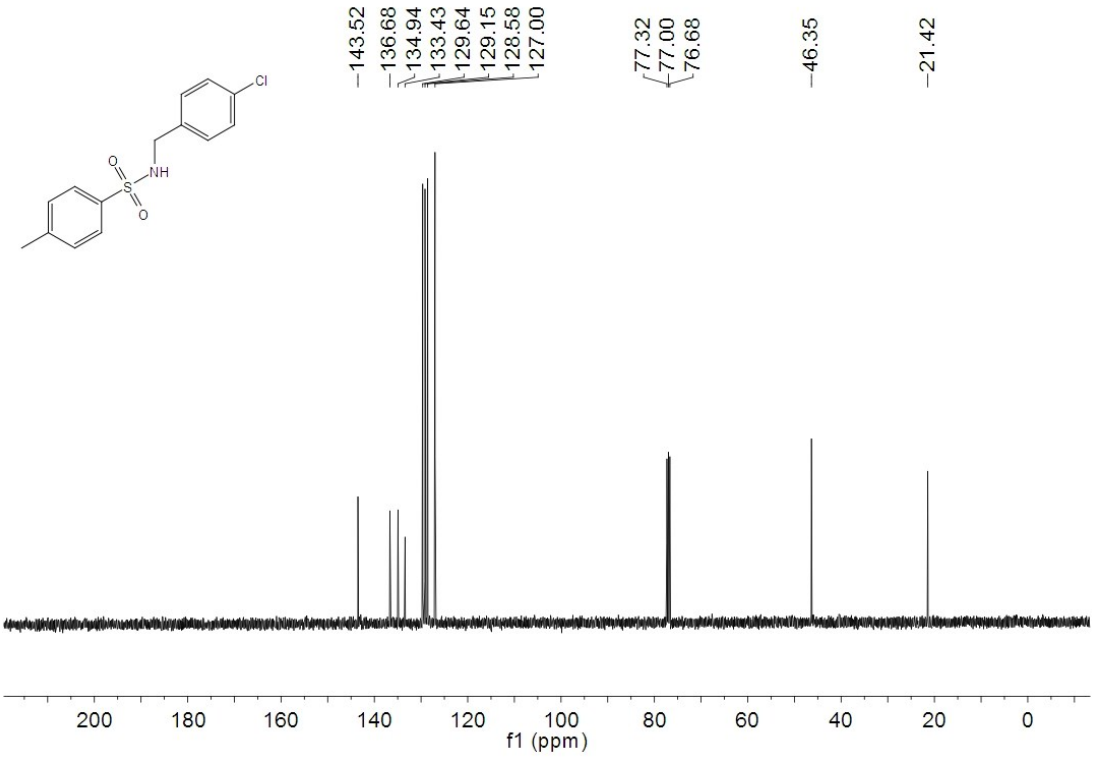


Fig. S-14. ^{13}C-NMR spectrum of **3e$_3$**.

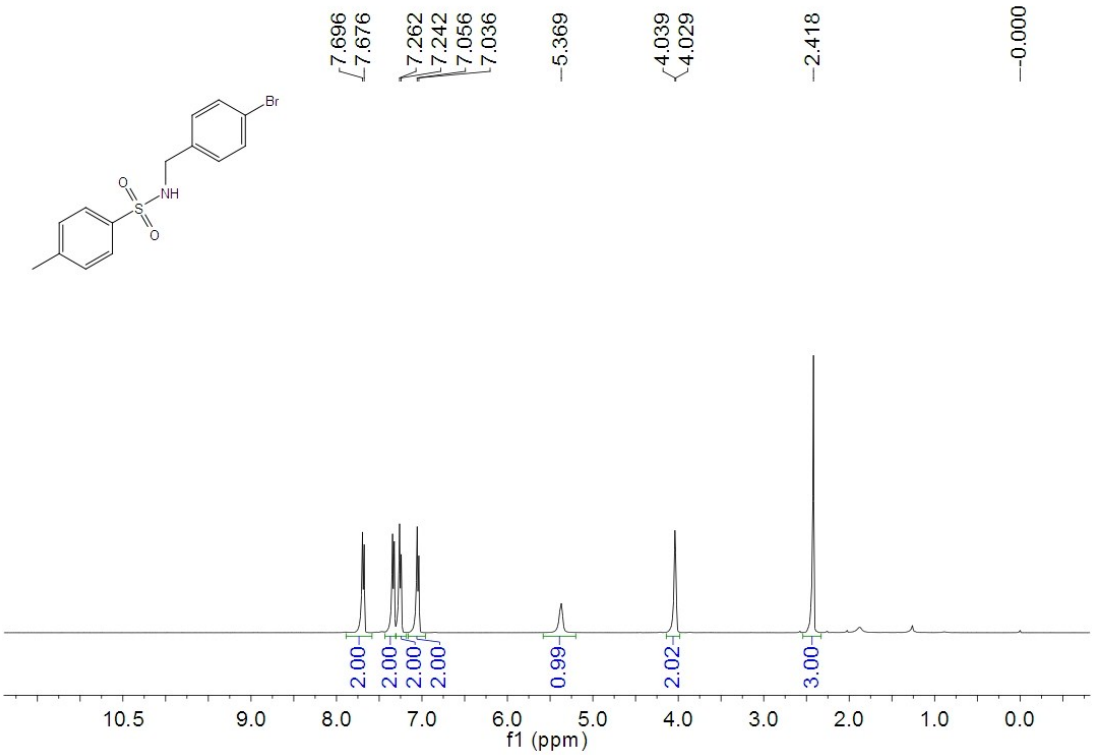


Fig. S-15. ^{1}H-NMR spectrum of **3e$_4$**.

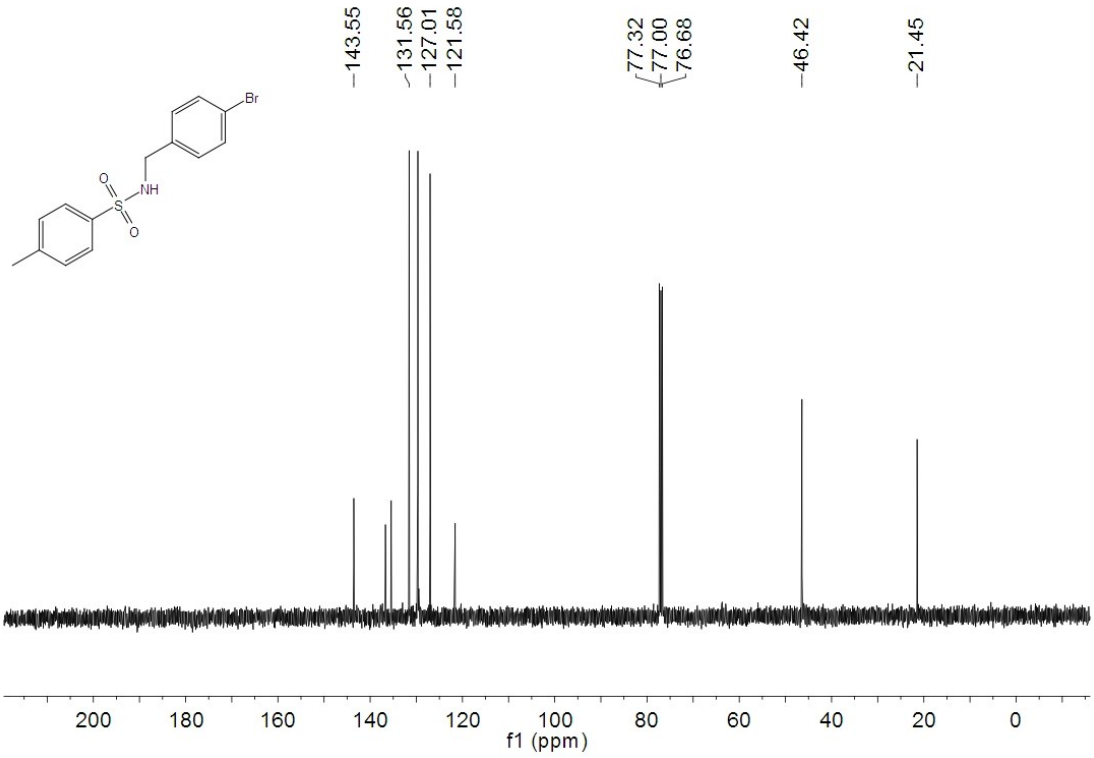


Fig. S-16. ^{13}C-NMR spectrum of **3e$_4$**.

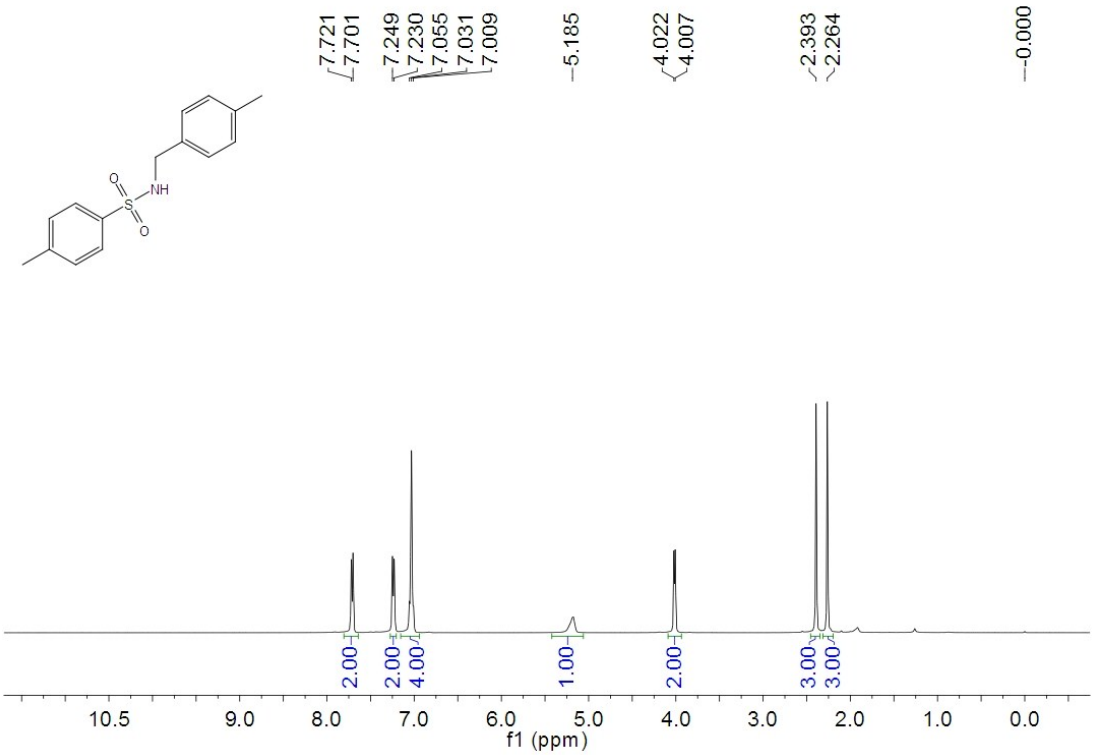


Fig. S-17. ^{1}H-NMR spectrum of **3e$_5$**.

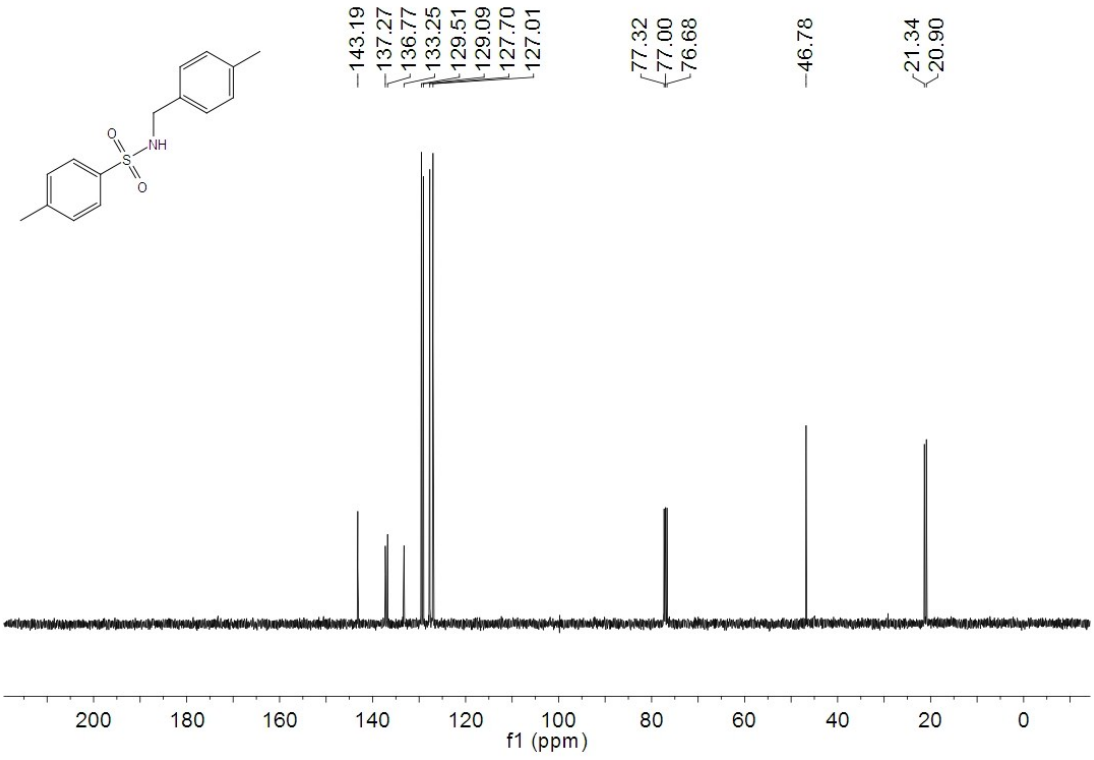


Fig. S-18. ^{13}C-NMR spectrum of **3e$_5$**.

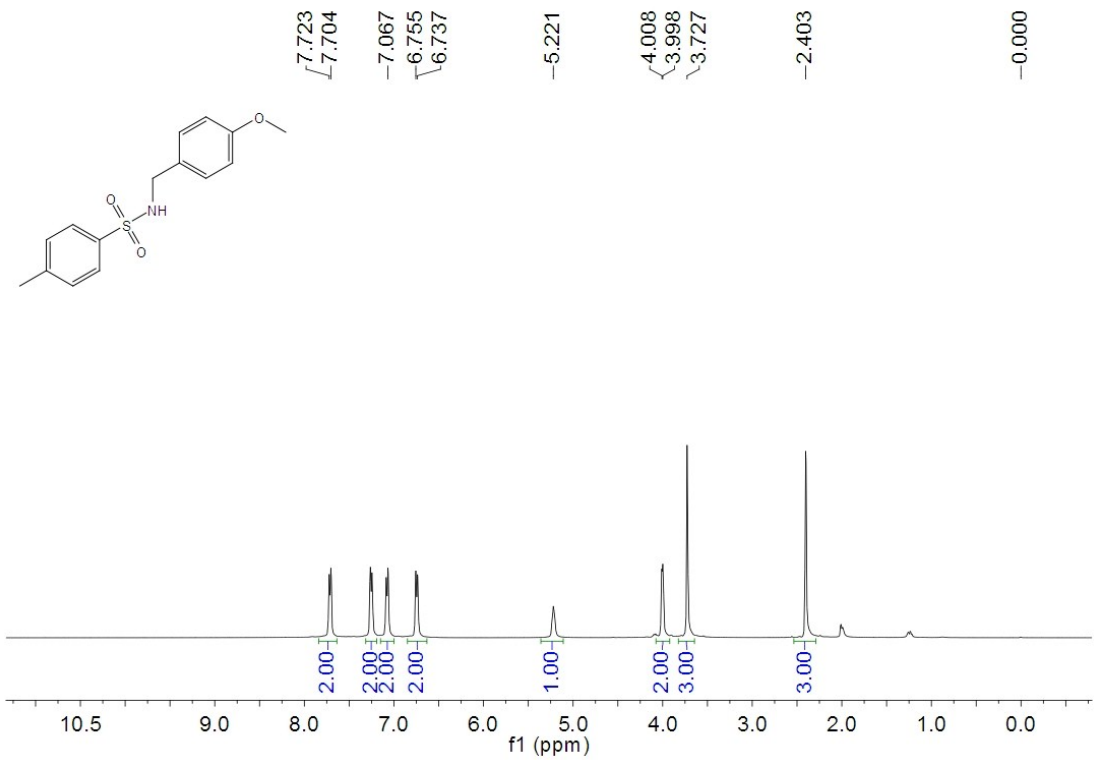


Fig. S-19. ^{1}H-NMR spectrum of **3e$_6$**.

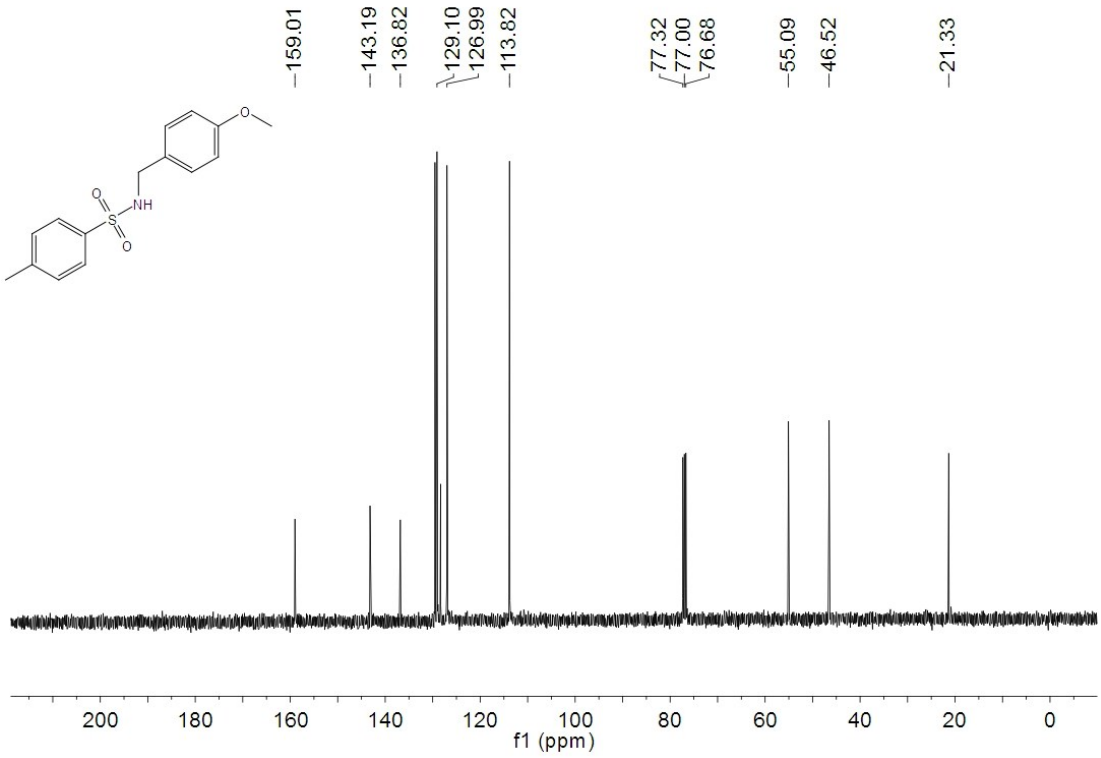


Fig. S-20. ^{13}C-NMR spectrum of **3e$_6$**.

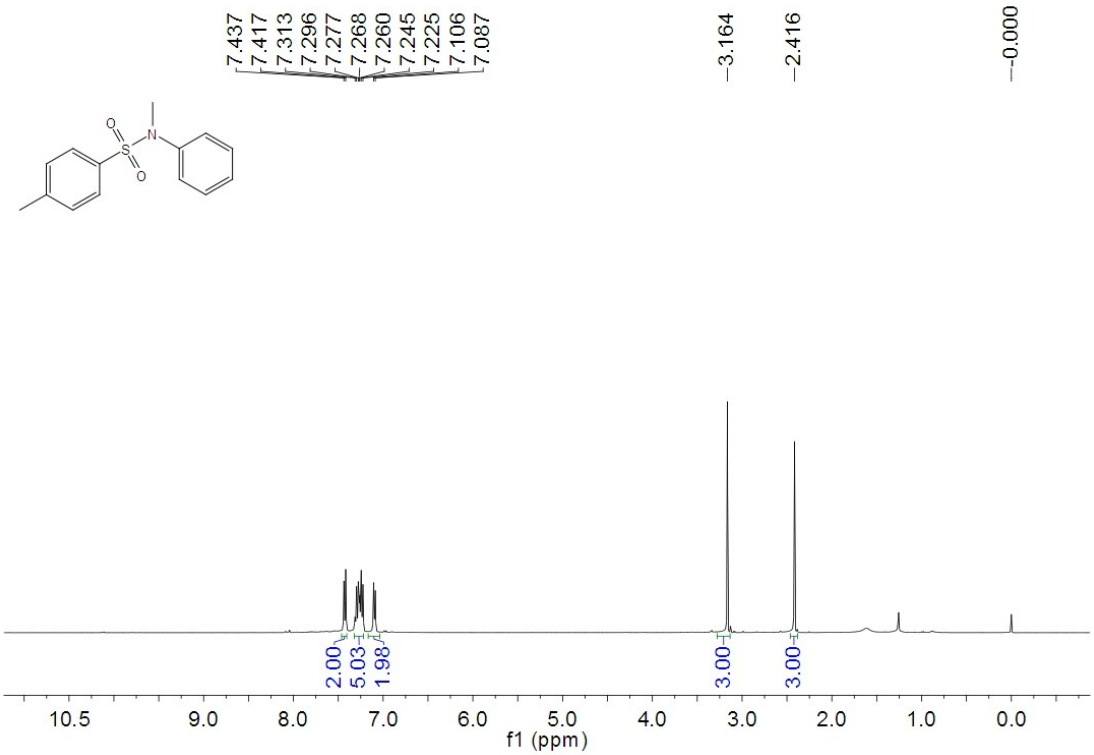


Fig. S-21. ^{1}H-NMR spectrum of **3f$_1$**.

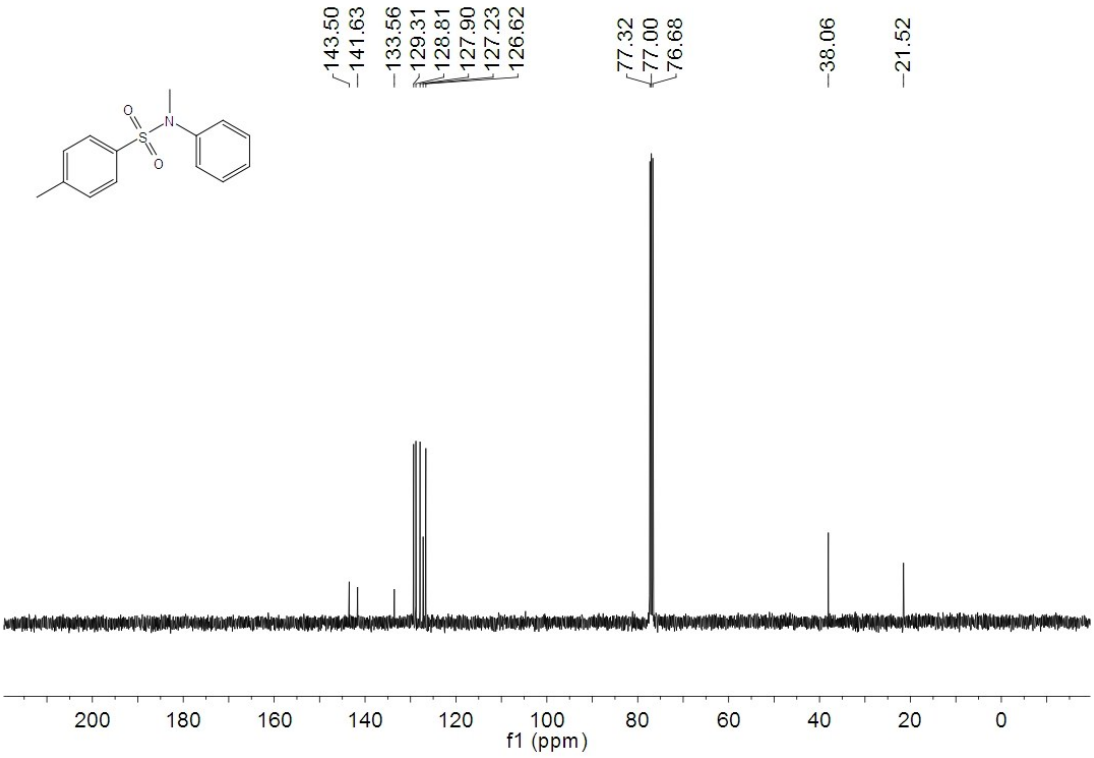


Fig. S-22. ^{13}C-NMR spectrum of **3f$_1$**.

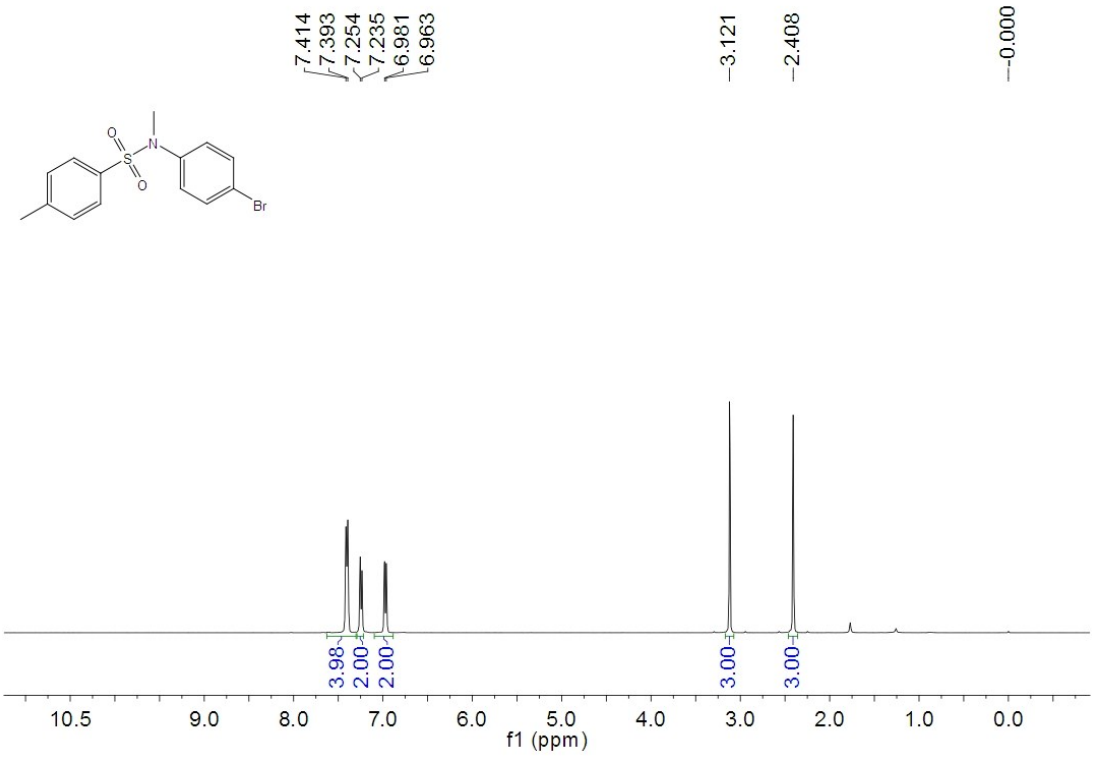


Fig. S-23. ^{1}H-NMR spectrum of **3f$_2$**.

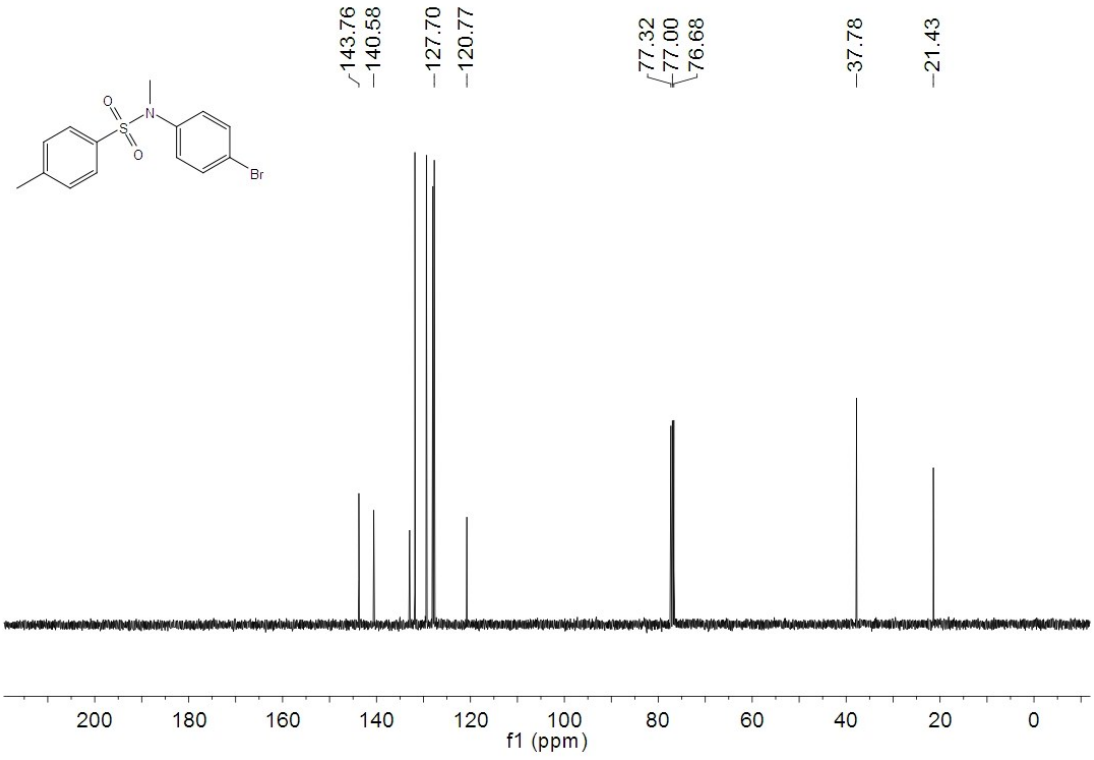


Fig. S-24. ^{13}C-NMR spectrum of **3f$_2$**.

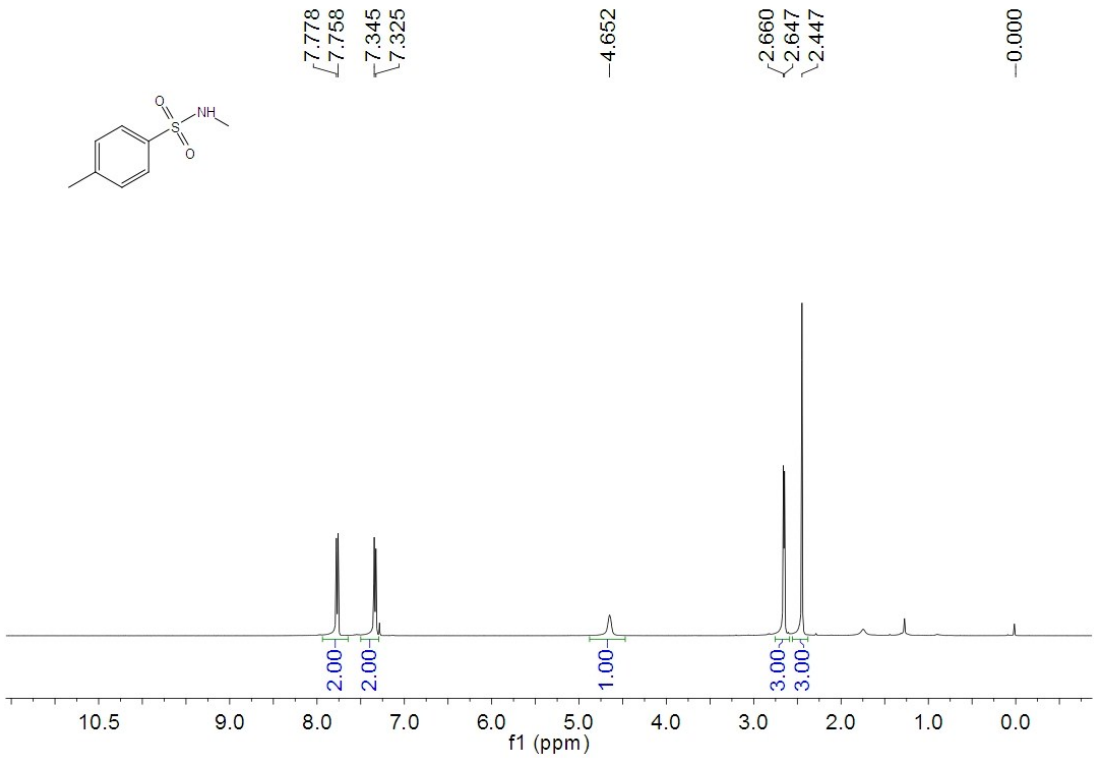


Fig. S-25. ^{1}H-NMR spectrum of **3g$_1$**.

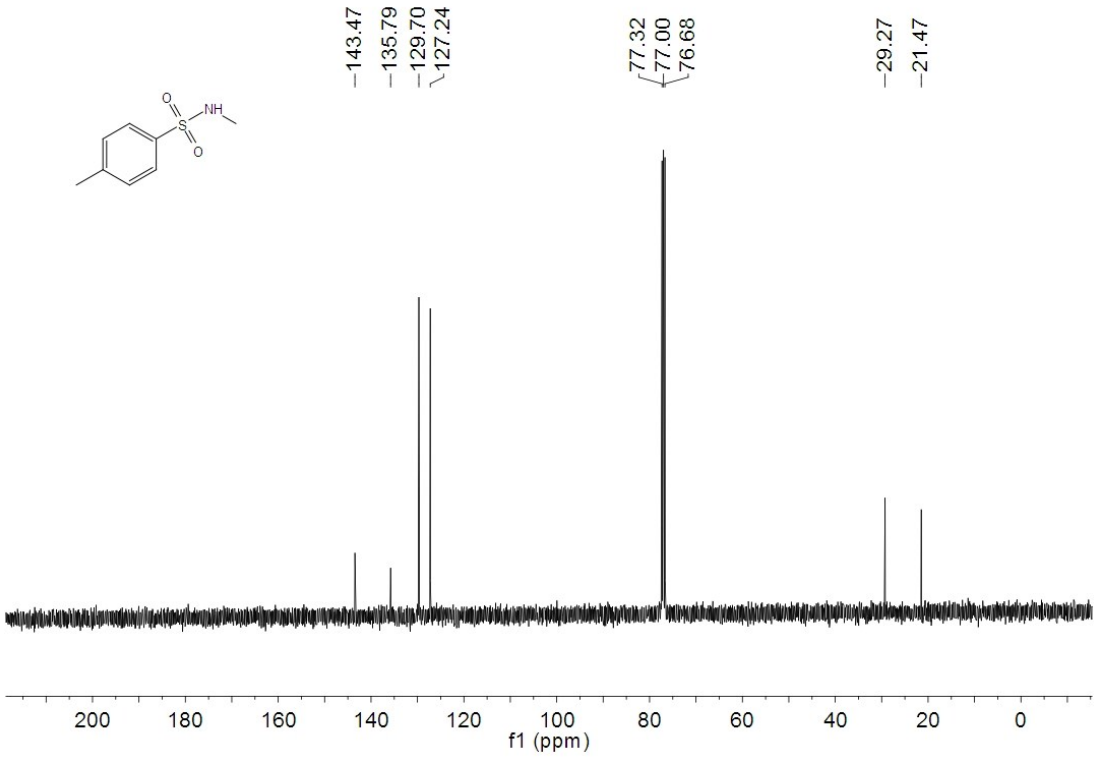


Fig. S-26. ^{13}C-NMR spectrum of **3g$_1$**.

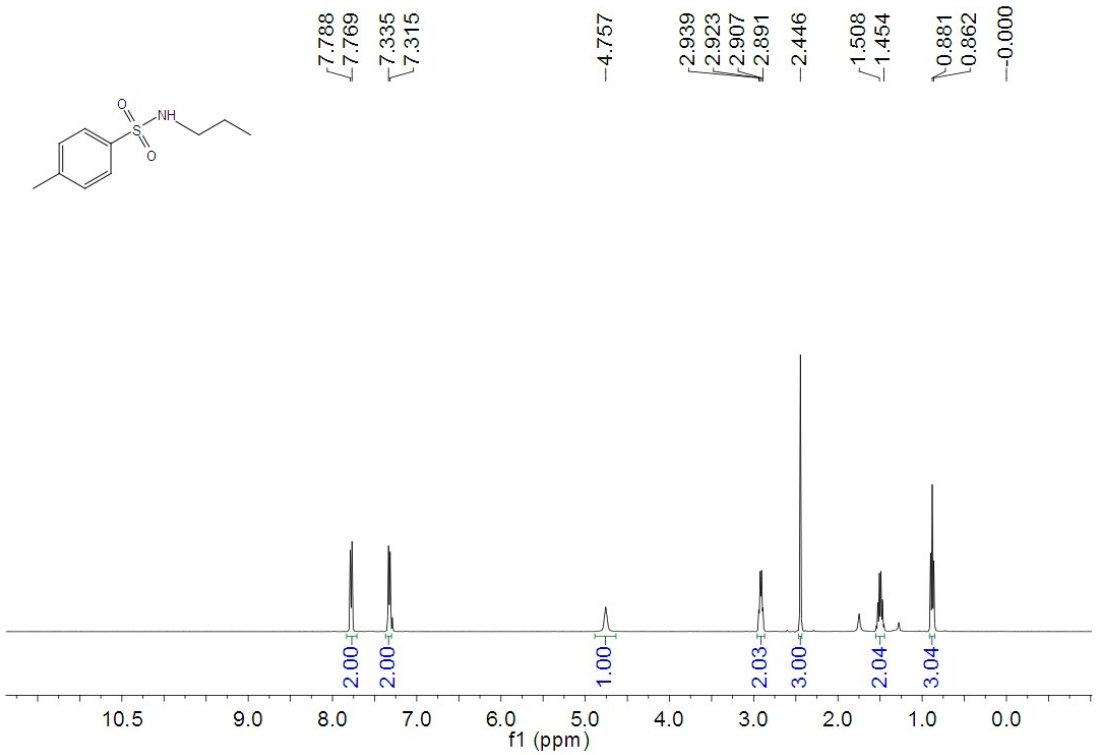


Fig. S-27. ^{1}H-NMR spectrum of **3g$_2$**.

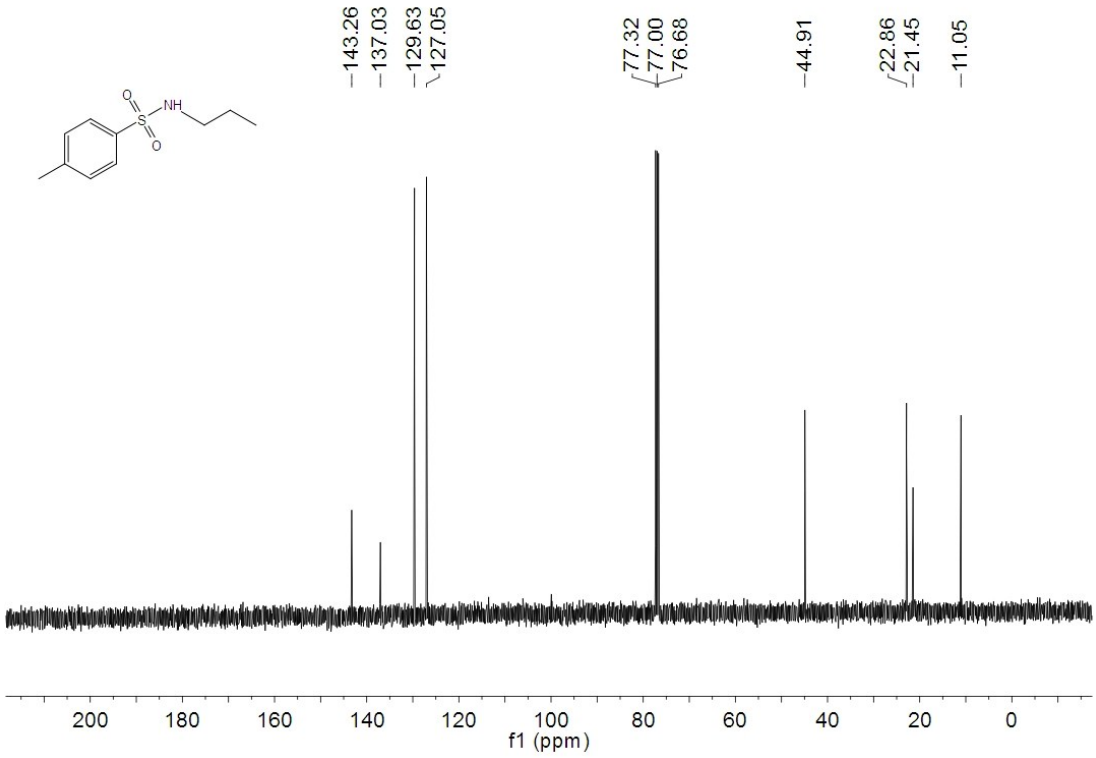


Fig. S-28. ^{13}C-NMR spectrum of **3g$_2$**.

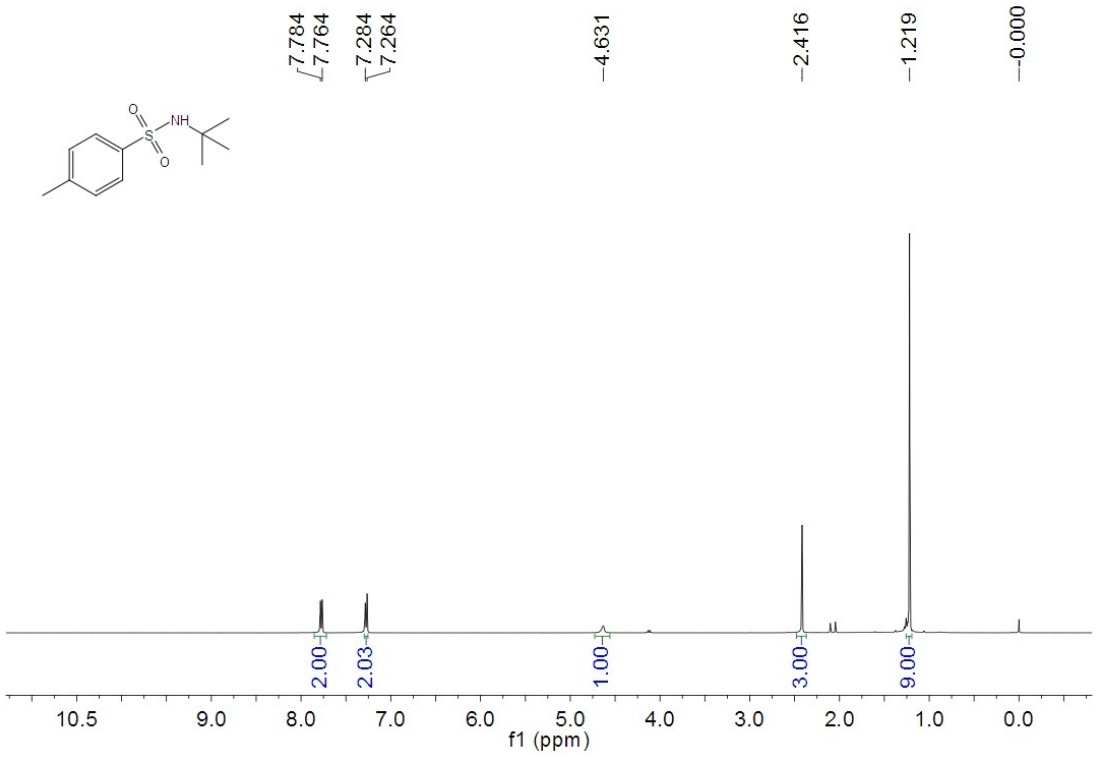


Fig. S-29. ^{1}H-NMR spectrum of **3g$_3$**.

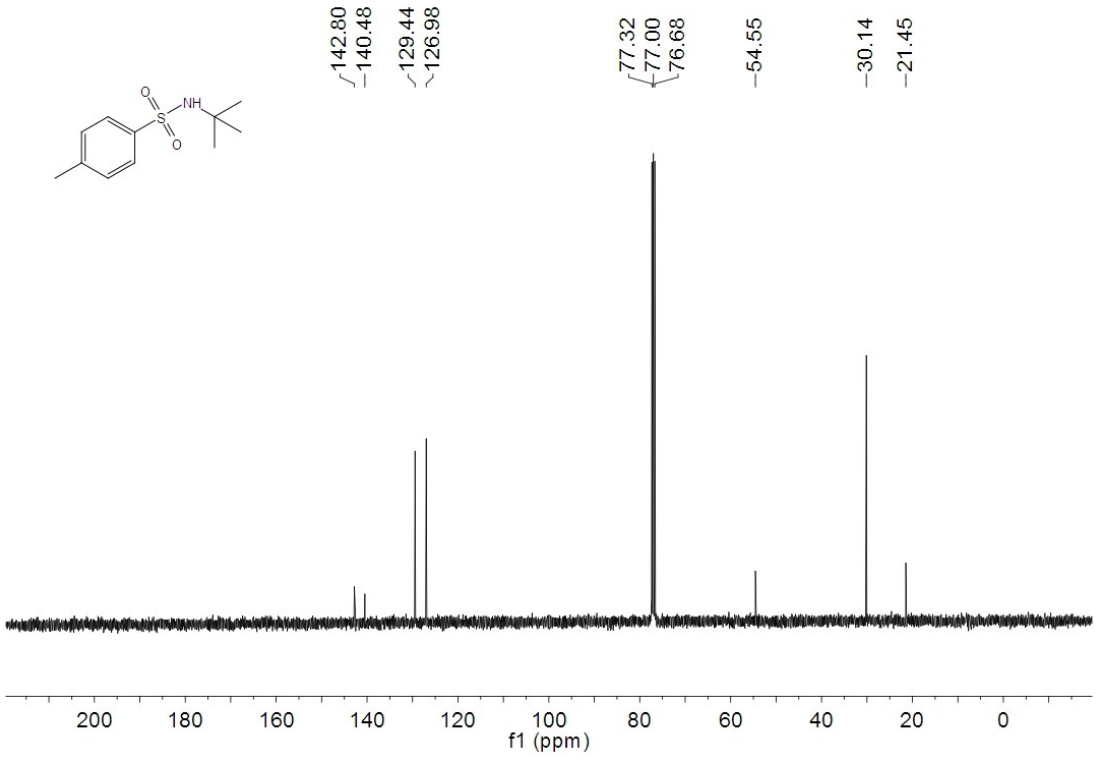


Fig. S-30. ^{13}C-NMR spectrum of **3g$_3$**.

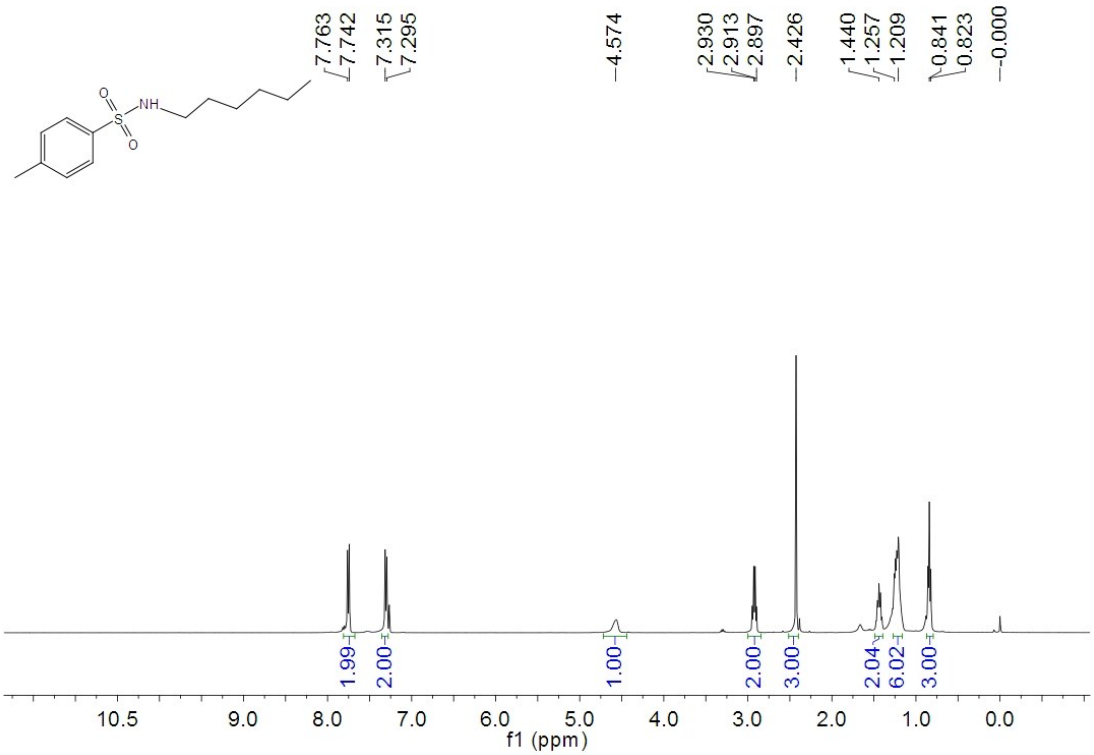


Fig. S-31. ^{1}H-NMR spectrum of **3g$_4$**.

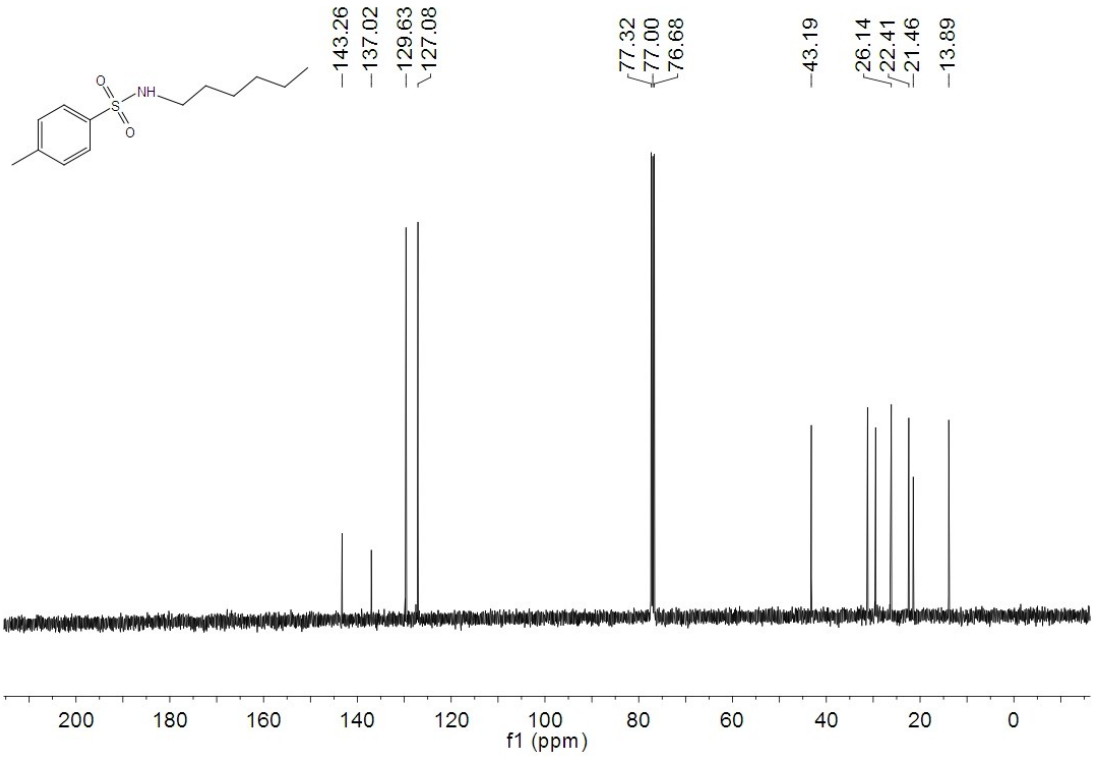


Fig. S-32. ^{13}C-NMR spectrum of **3g$_4$**.

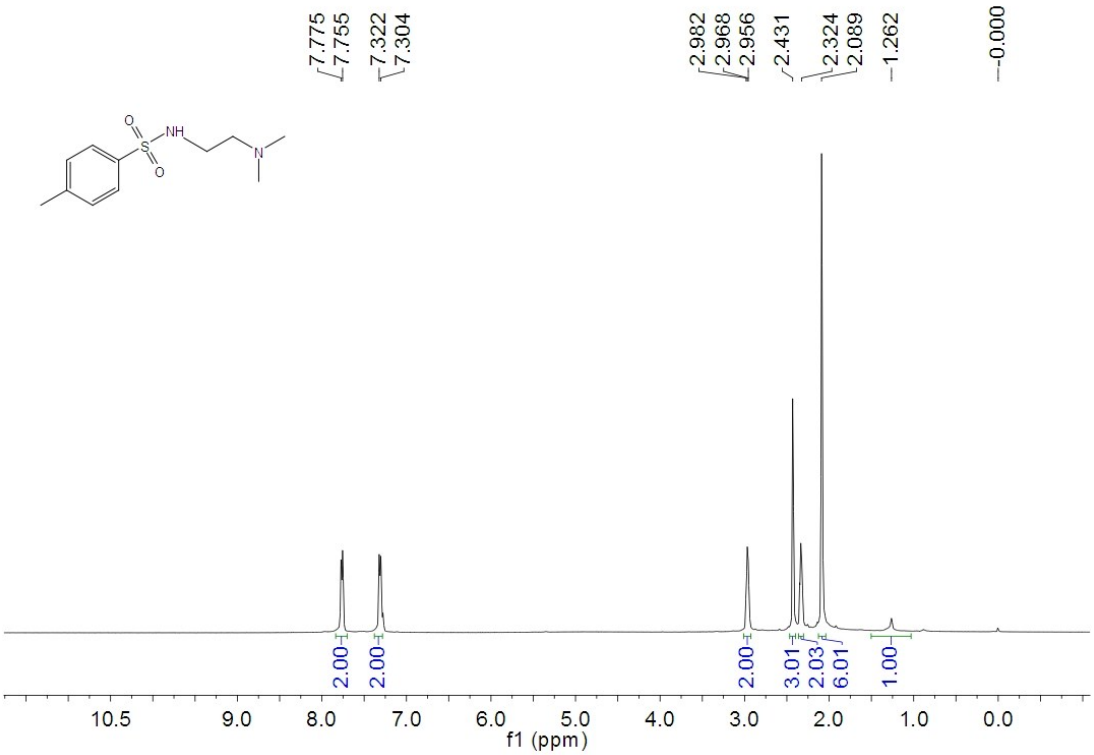


Fig. S-33. ^{1}H-NMR spectrum of **3h**.

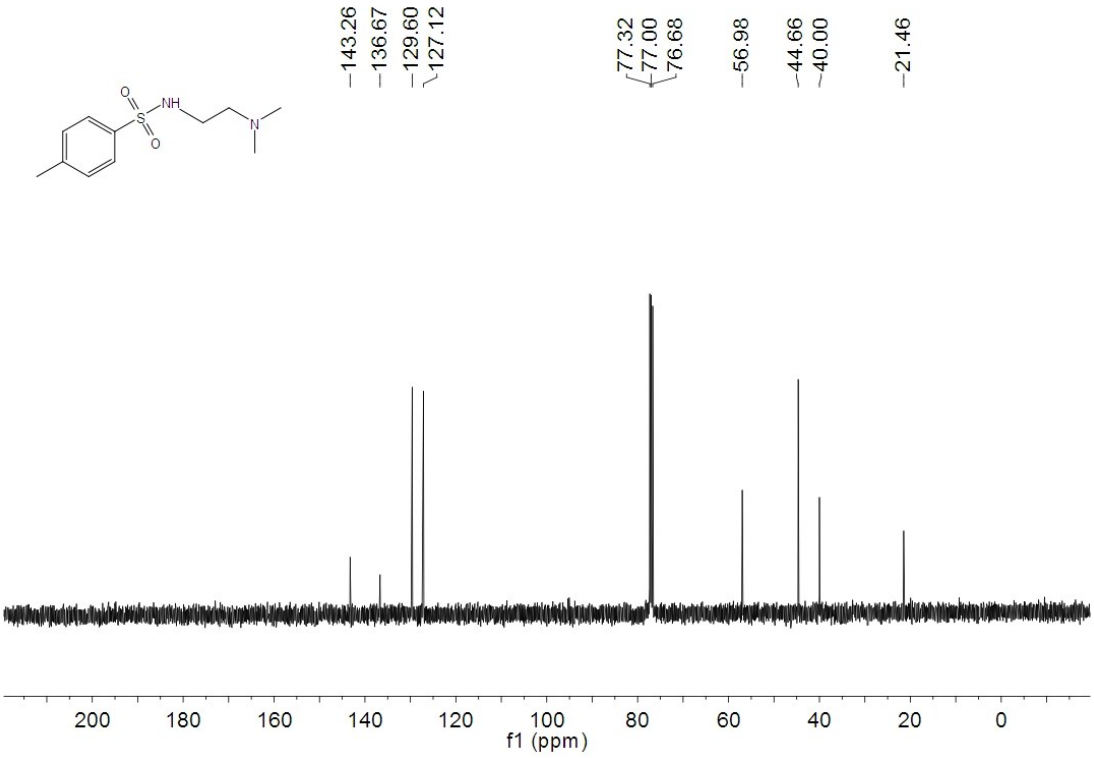


Fig. S-34. ^{13}C-NMR spectrum of **3h**.

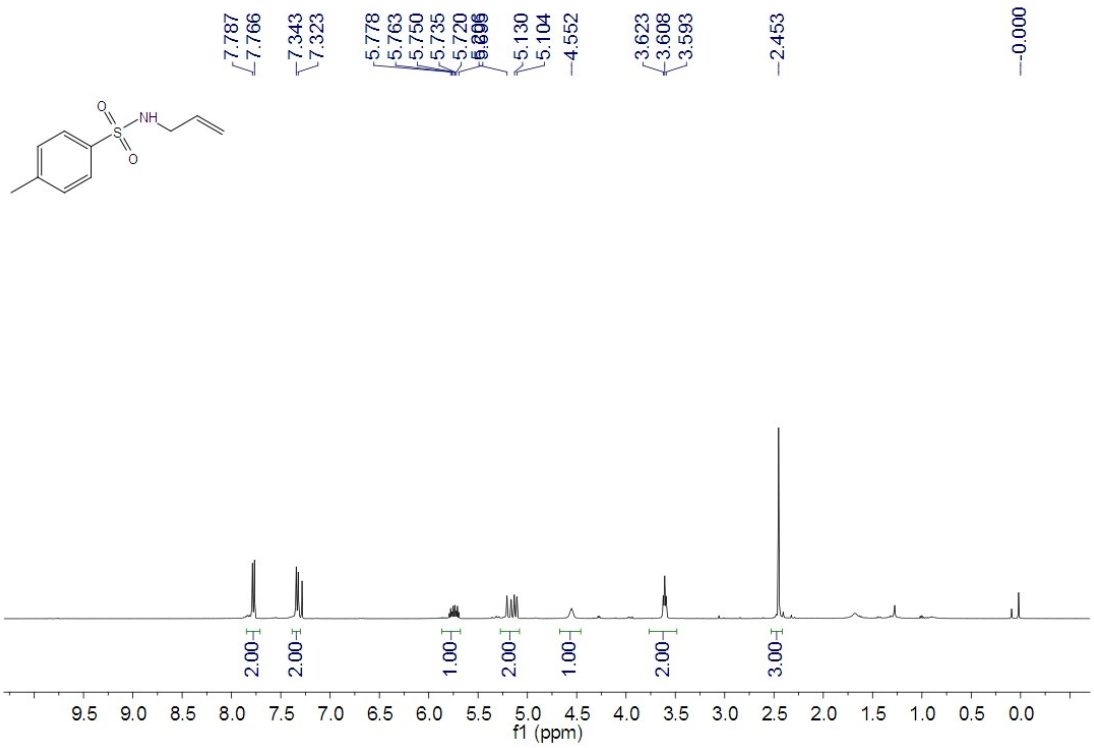


Fig. S-35. ^{1}H-NMR spectrum of **3i**.

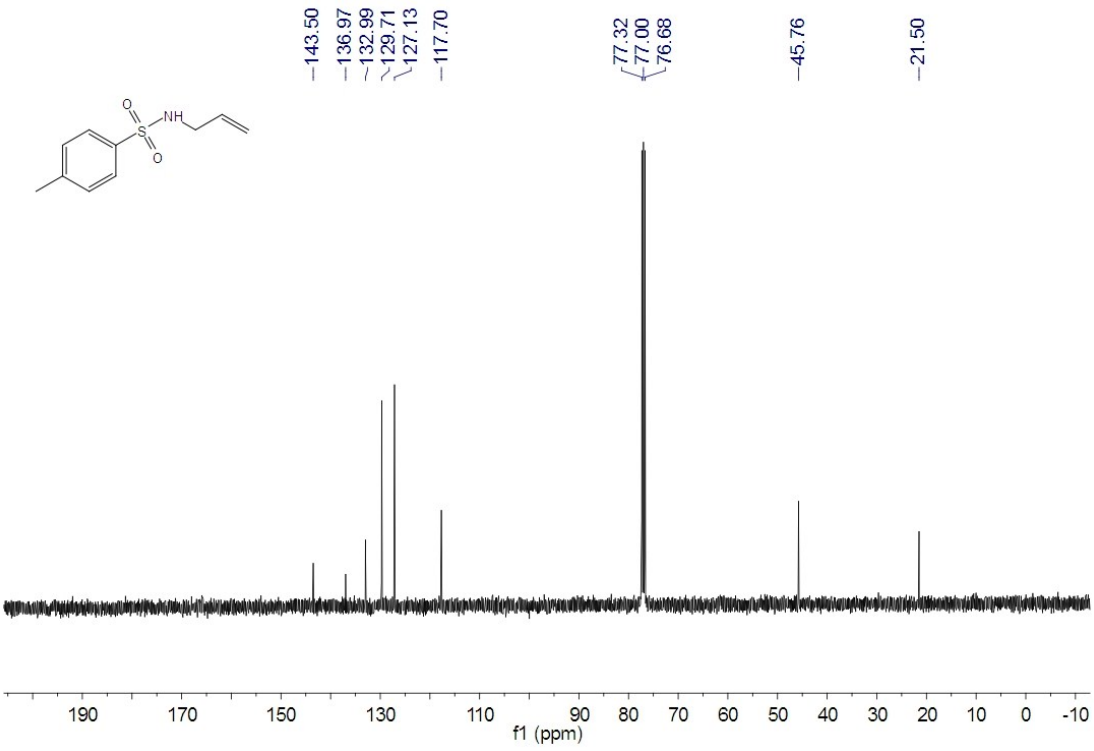


Fig. S-36. ^{13}C-NMR spectrum of **3i**.

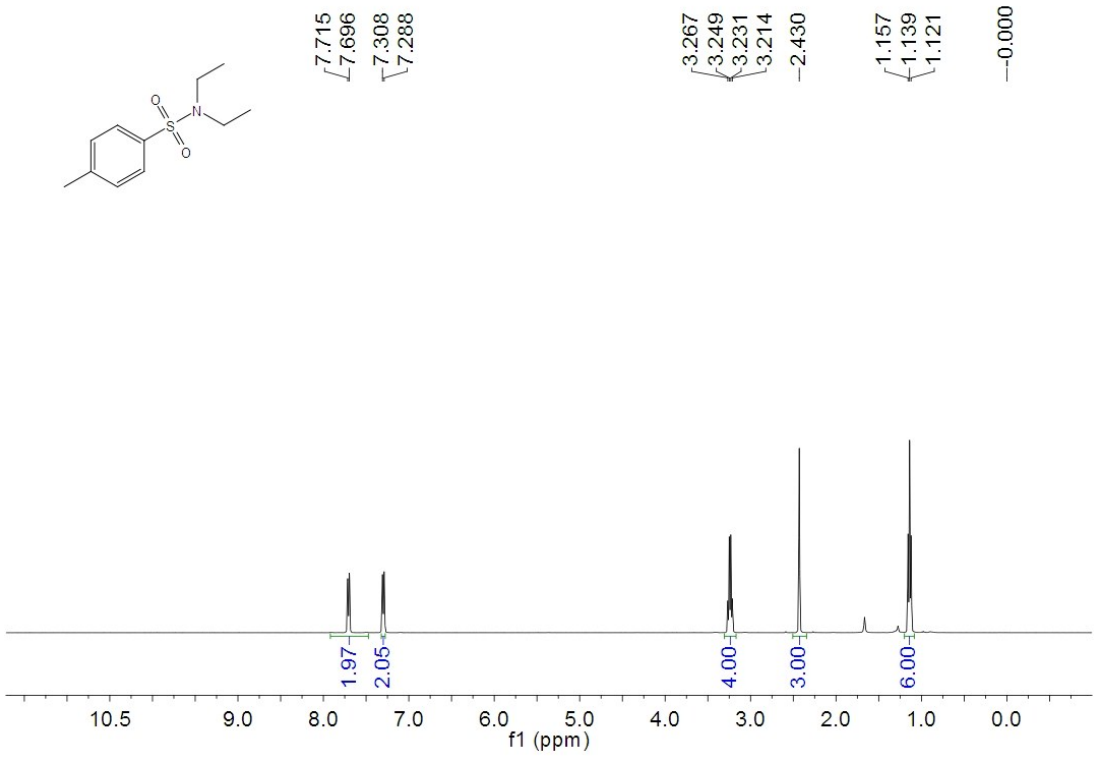


Fig. S-37. ^{1}H-NMR spectrum of **3j**.

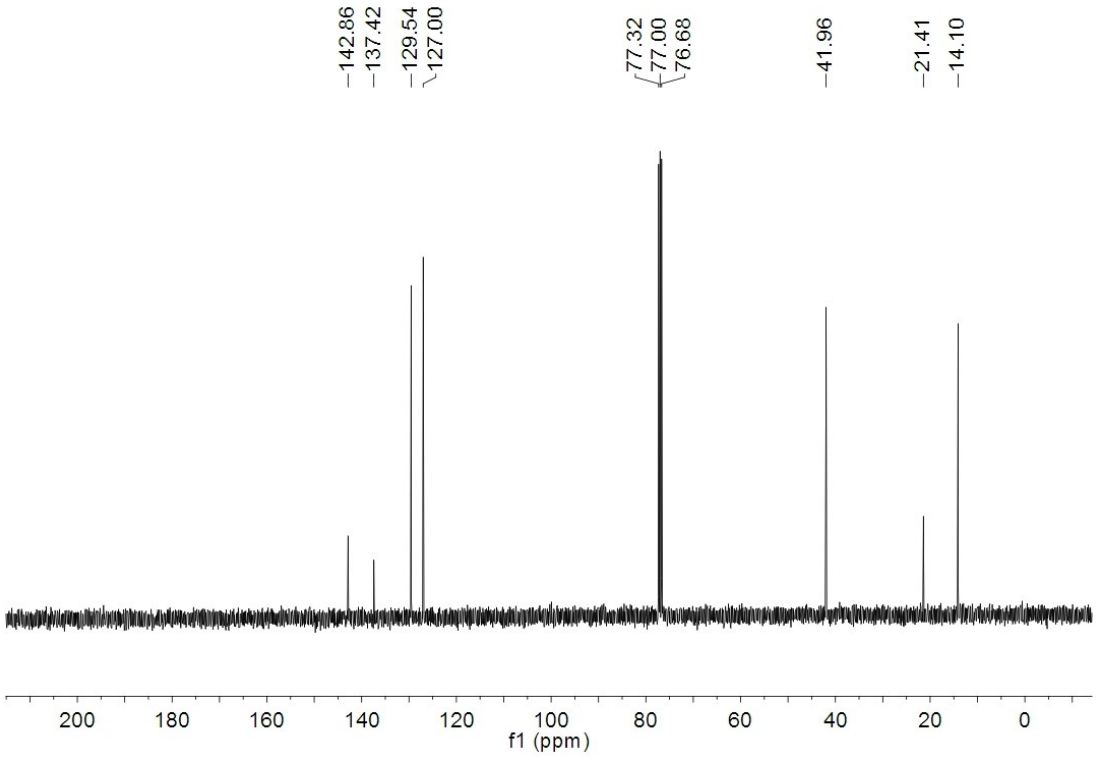


Fig. S-38. ^{13}C-NMR spectrum of **3j**.

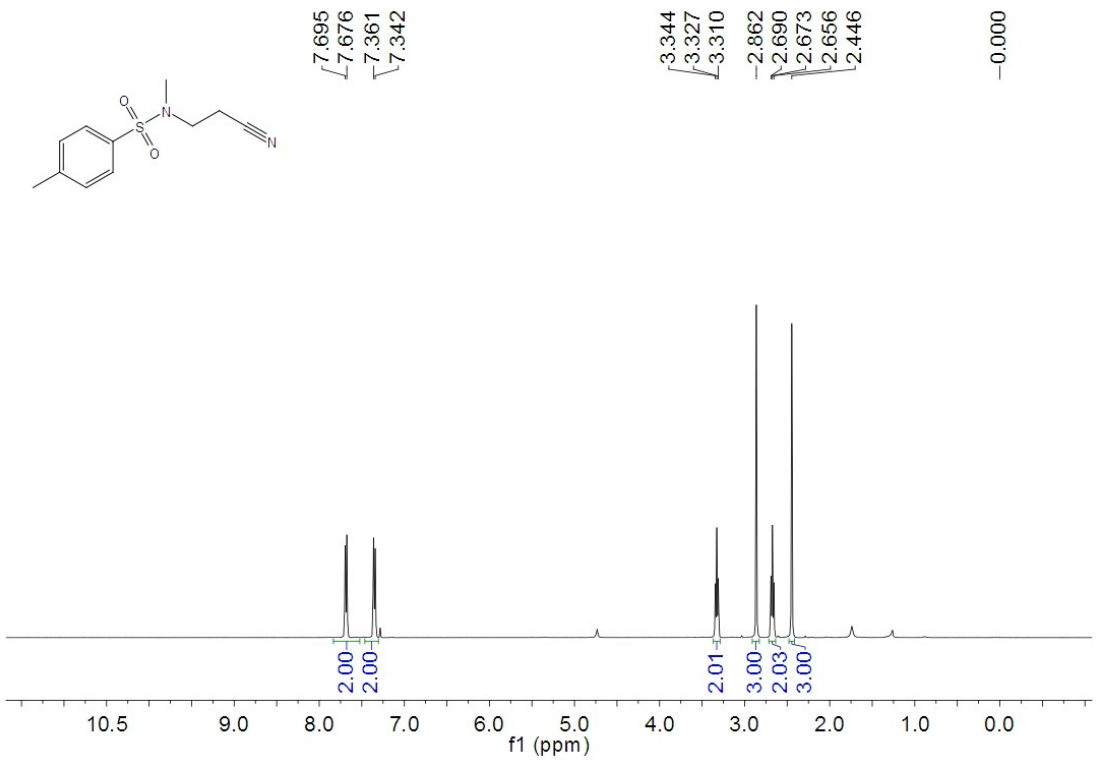


Fig. S-39. ^{1}H-NMR spectrum of **3k**.

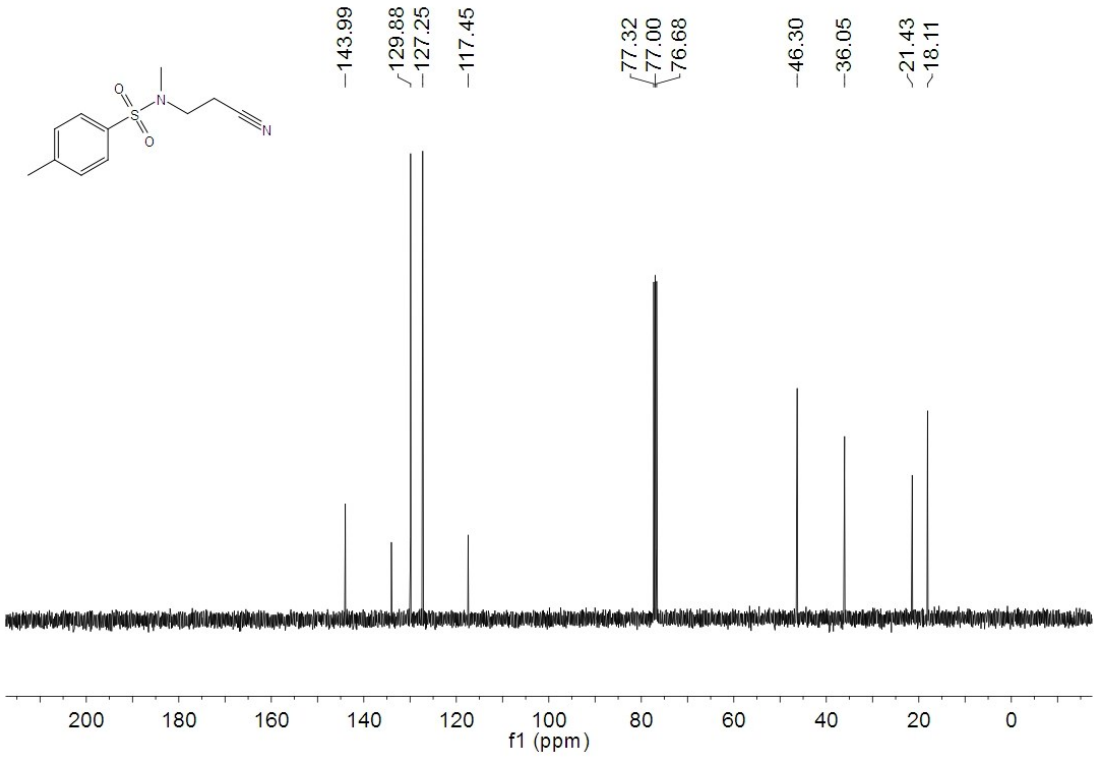


Fig. S-40. ^{13}C-NMR spectrum of **3k**.

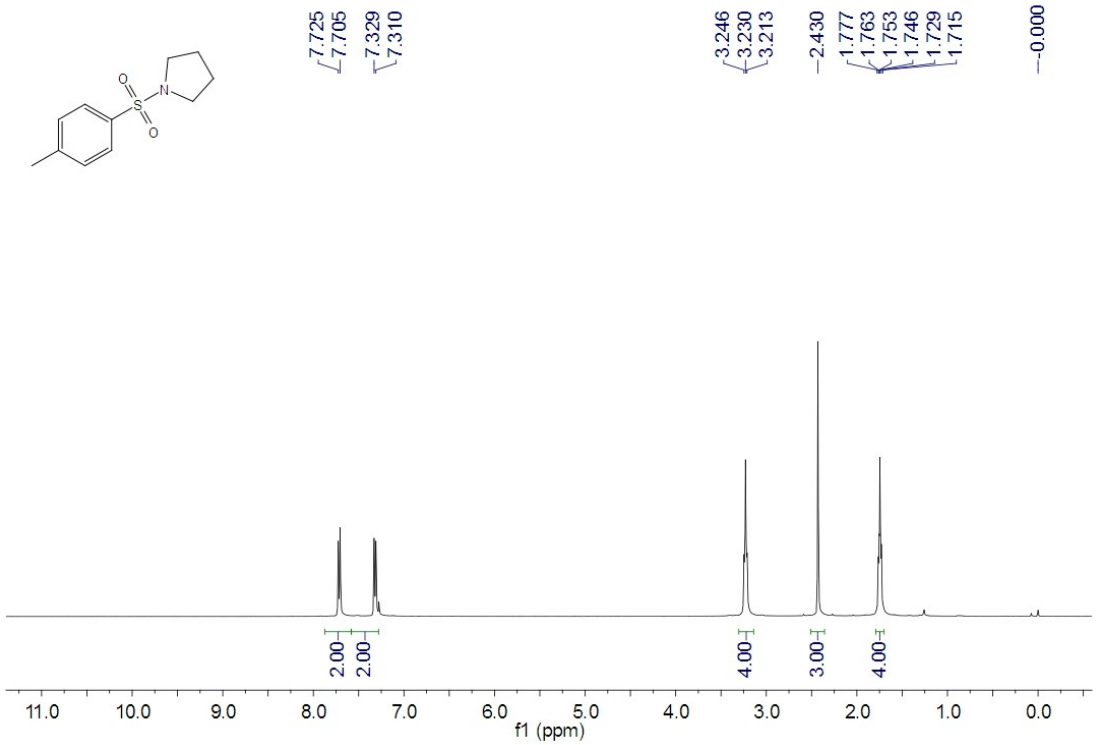


Fig. S-41. ^{1}H-NMR spectrum of **3l**.

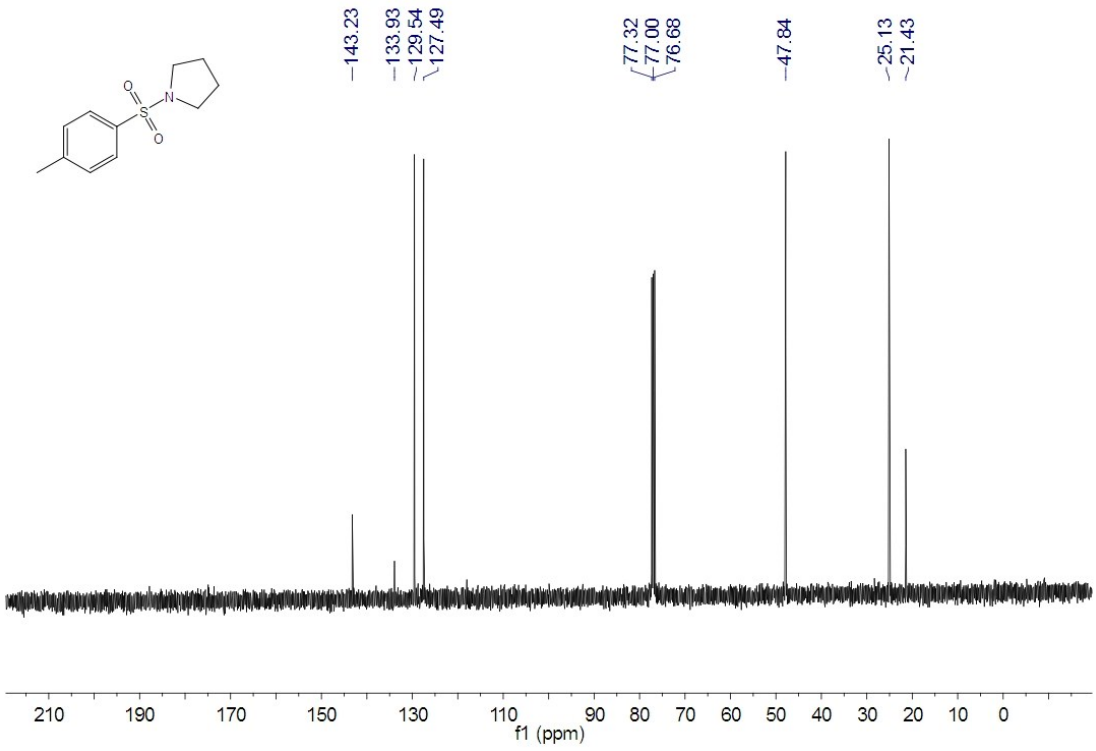


Fig. S-42. ^{13}C-NMR spectrum of **3l**.

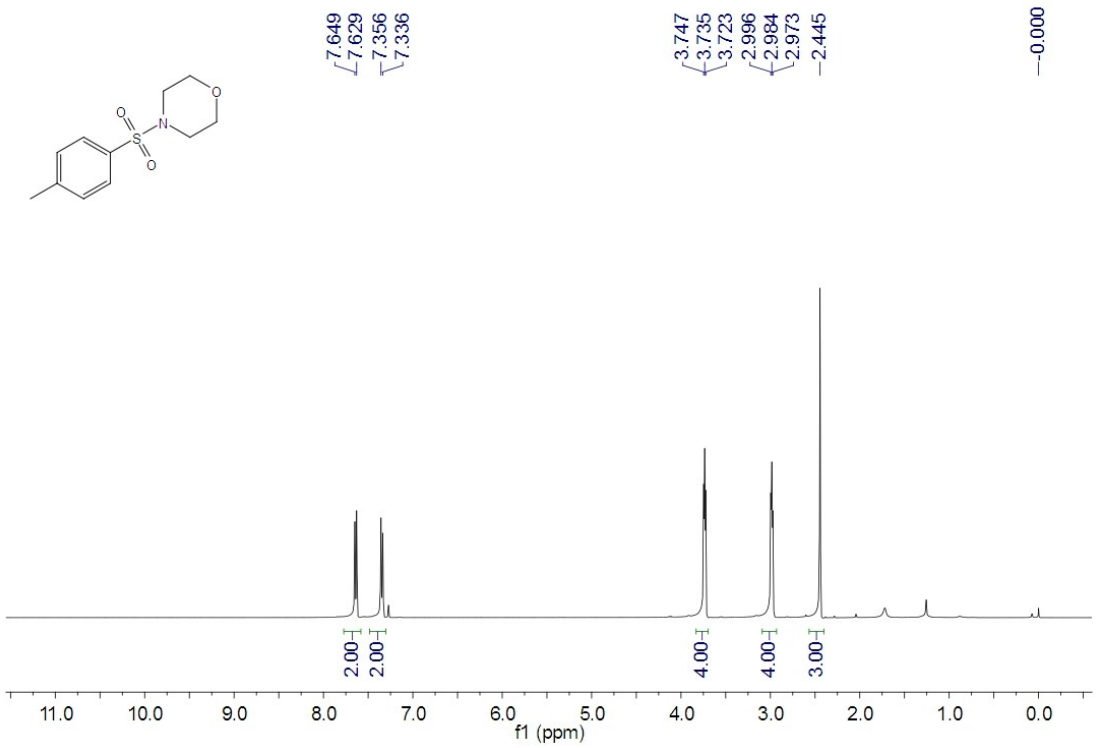


Fig. S-43. ^{1}H-NMR spectrum of **3m**.

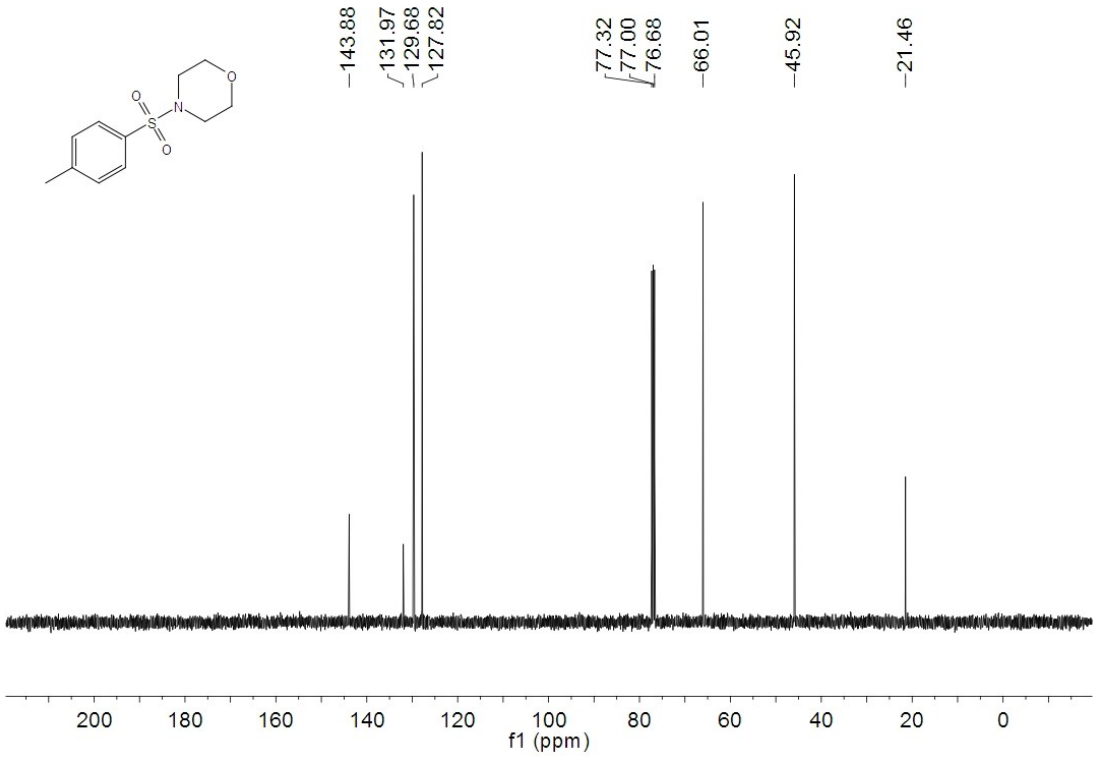


Fig. S-44. ^{13}C-NMR spectrum of **3m**.

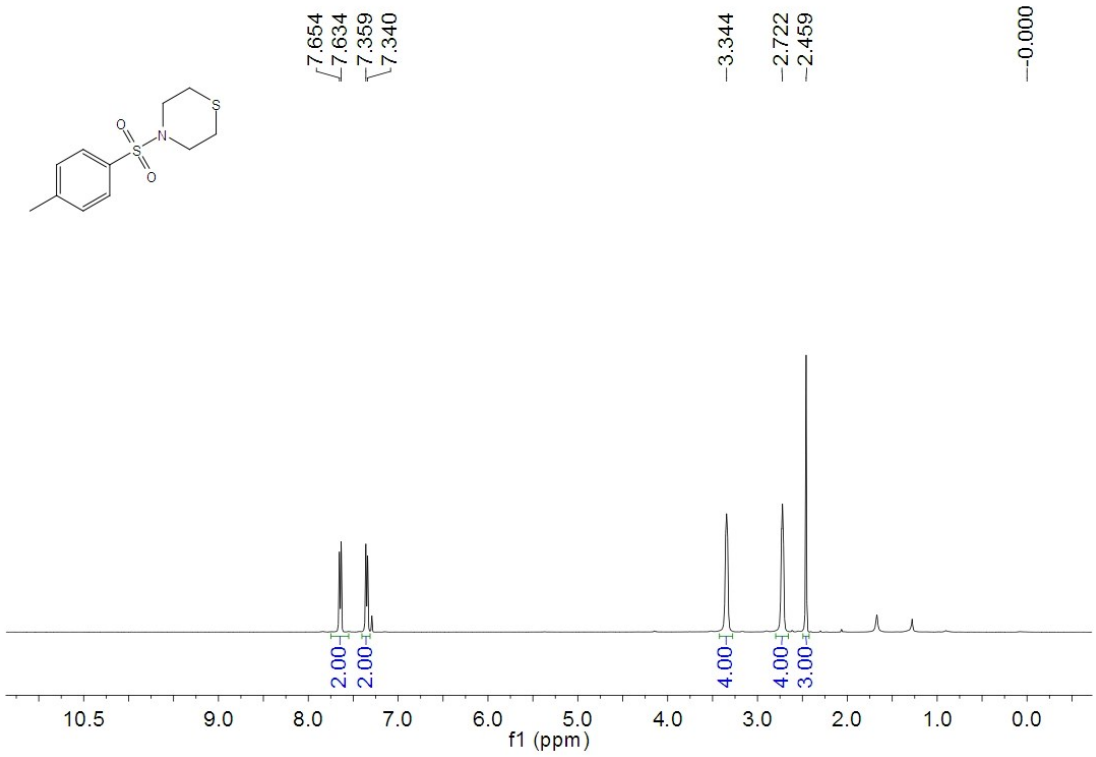


Fig. S-45. ^{1}H-NMR spectrum of **3n**.

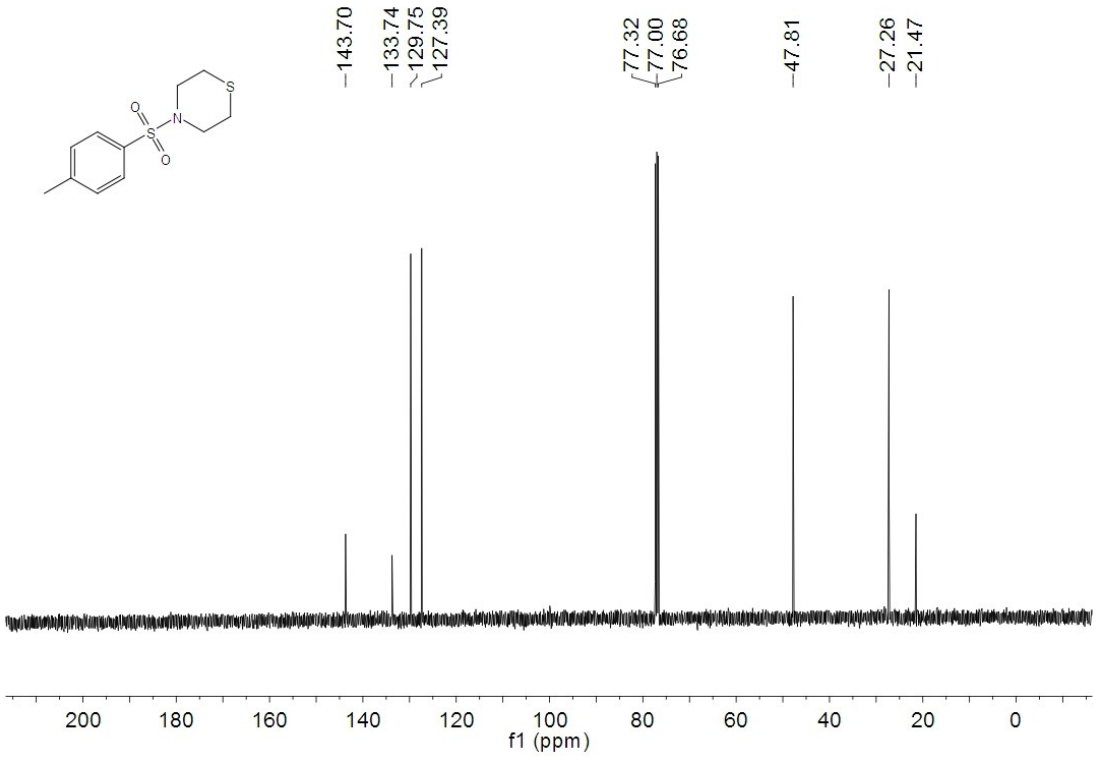


Fig. S-46. ^{13}C-NMR spectrum of **3n**.

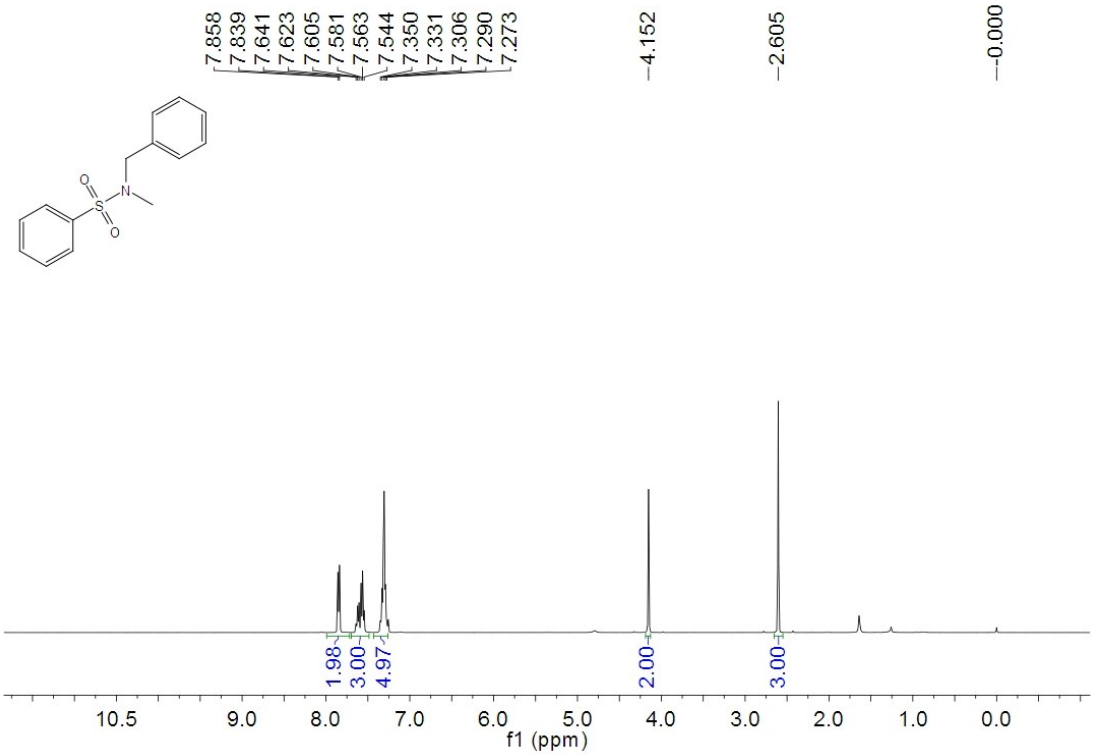


Fig. S-47. ^{1}H-NMR spectrum of **3o$_1$**.

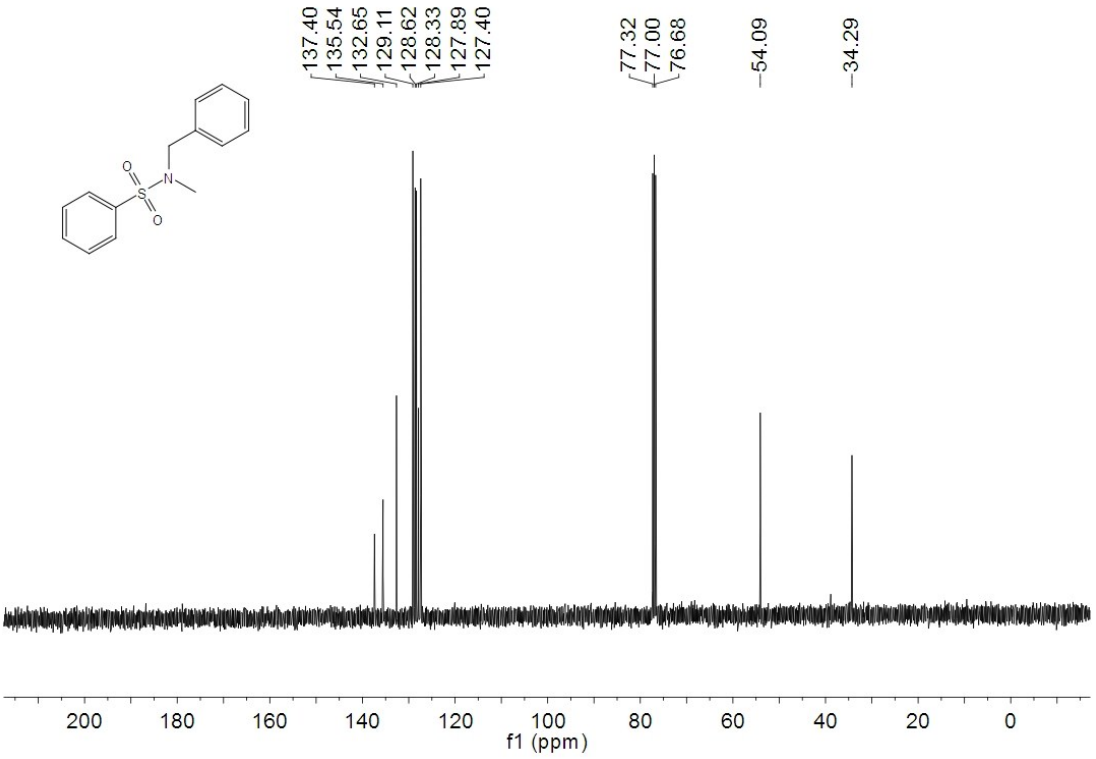


Fig. S-48. ^{13}C-NMR spectrum of **3o$_1$**.

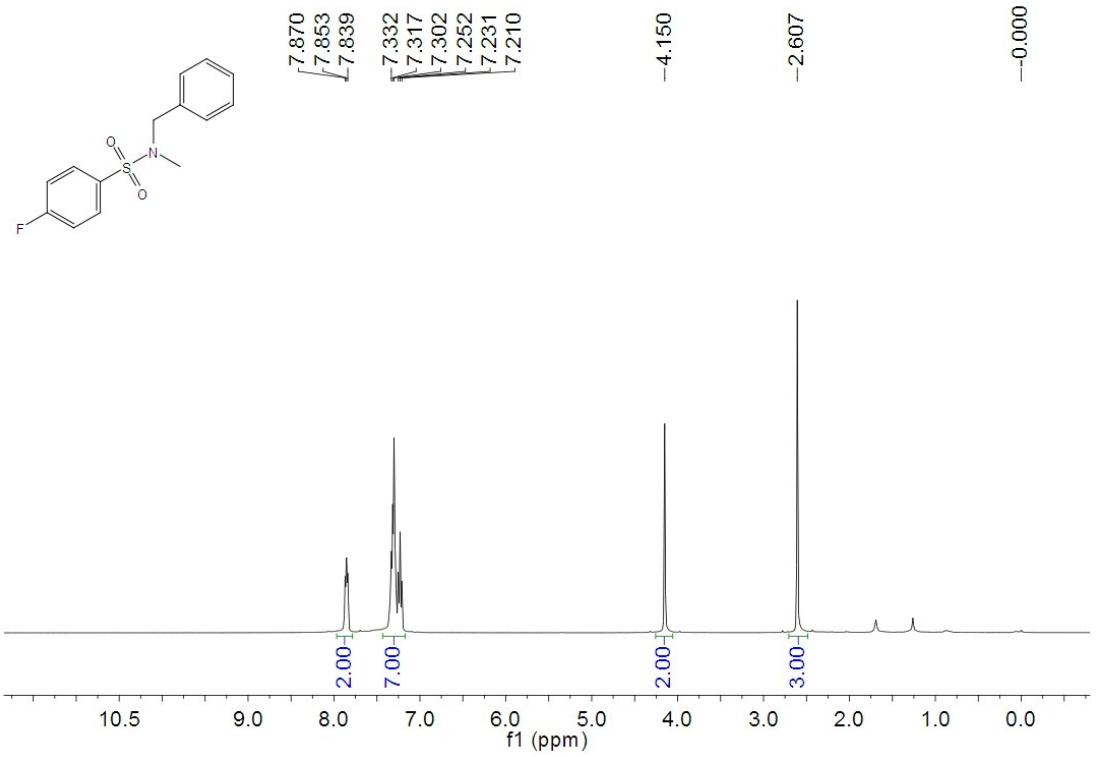


Fig. S-49. ^{1}H-NMR spectrum of **3o$_2$**.

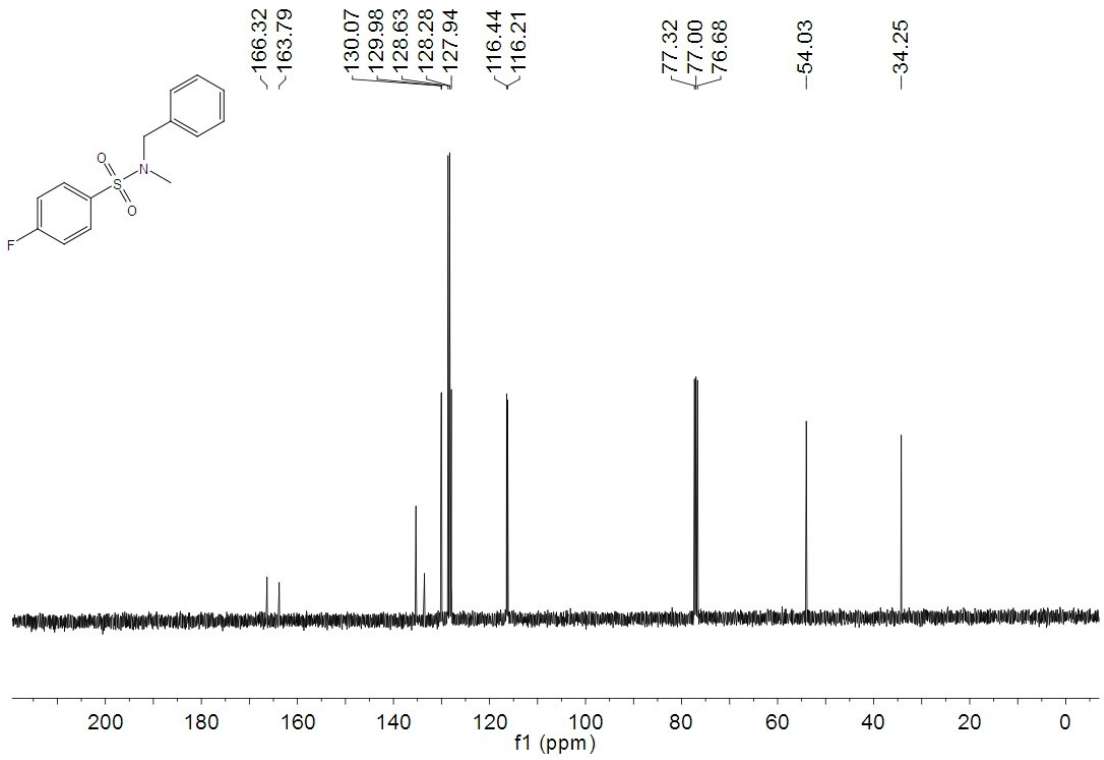


Fig. S-50. ^{13}C-NMR spectrum of **3o$_2$**.

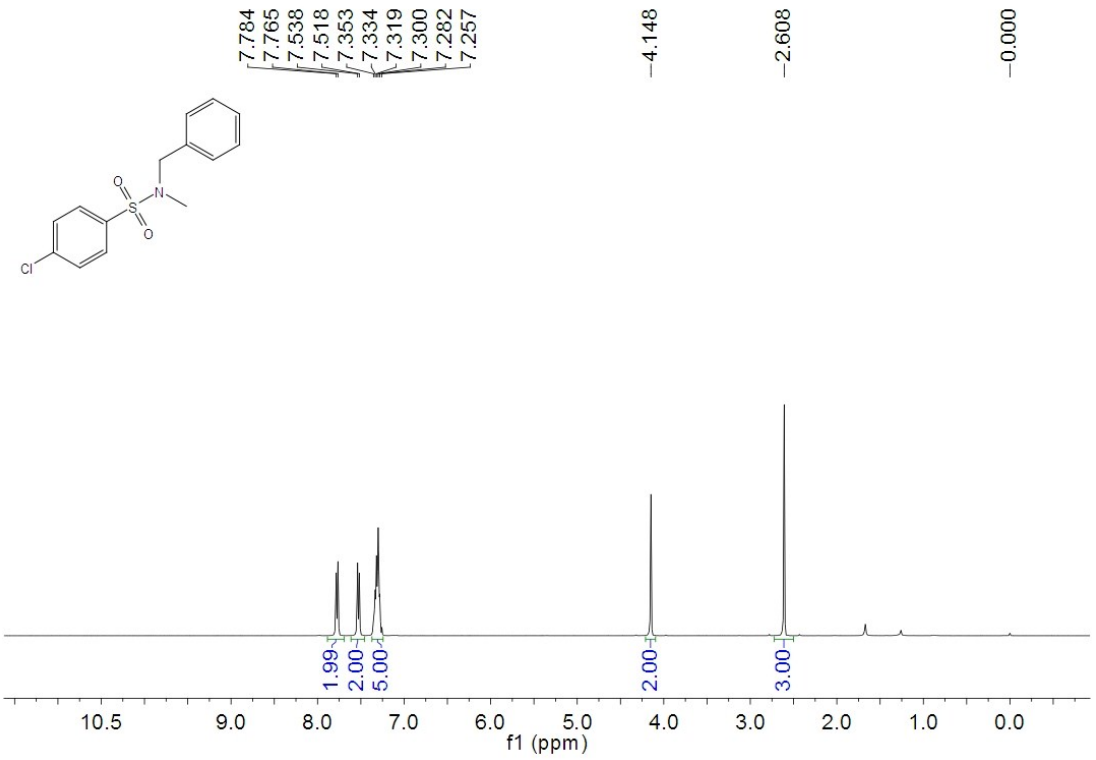


Fig. S-51. ^{1}H-NMR spectrum of **3o$_3$**.

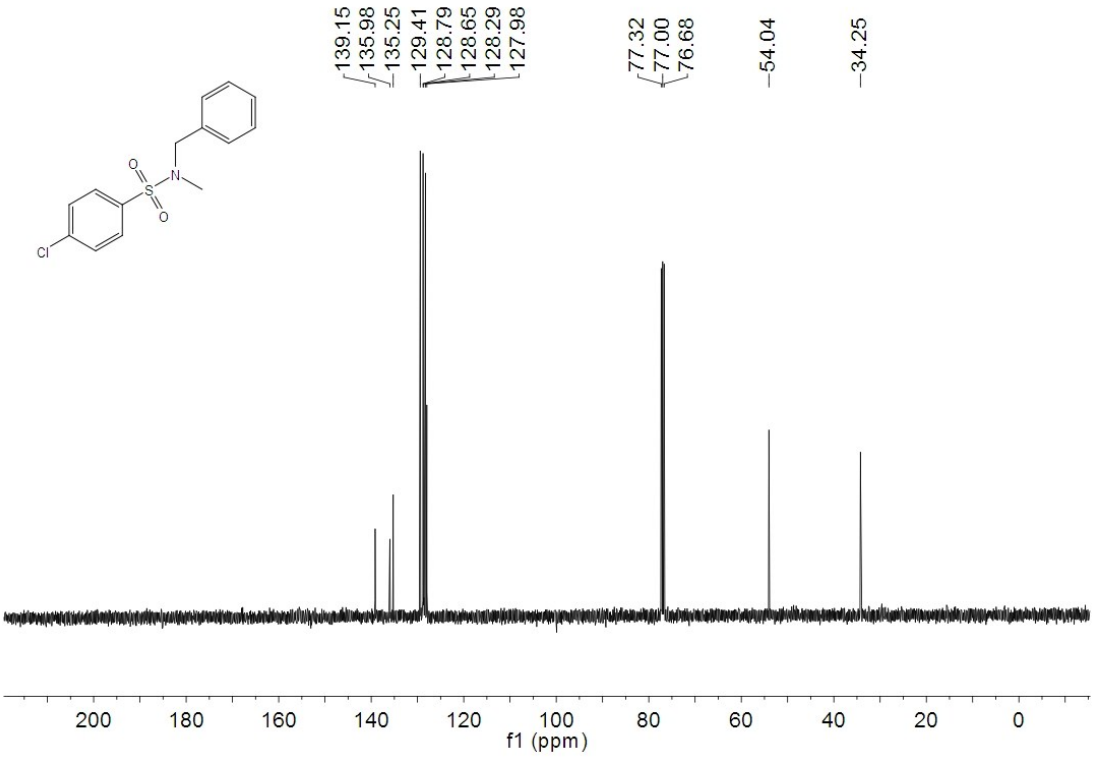


Fig. S-52. ^{13}C-NMR spectrum of **3o$_3$**.

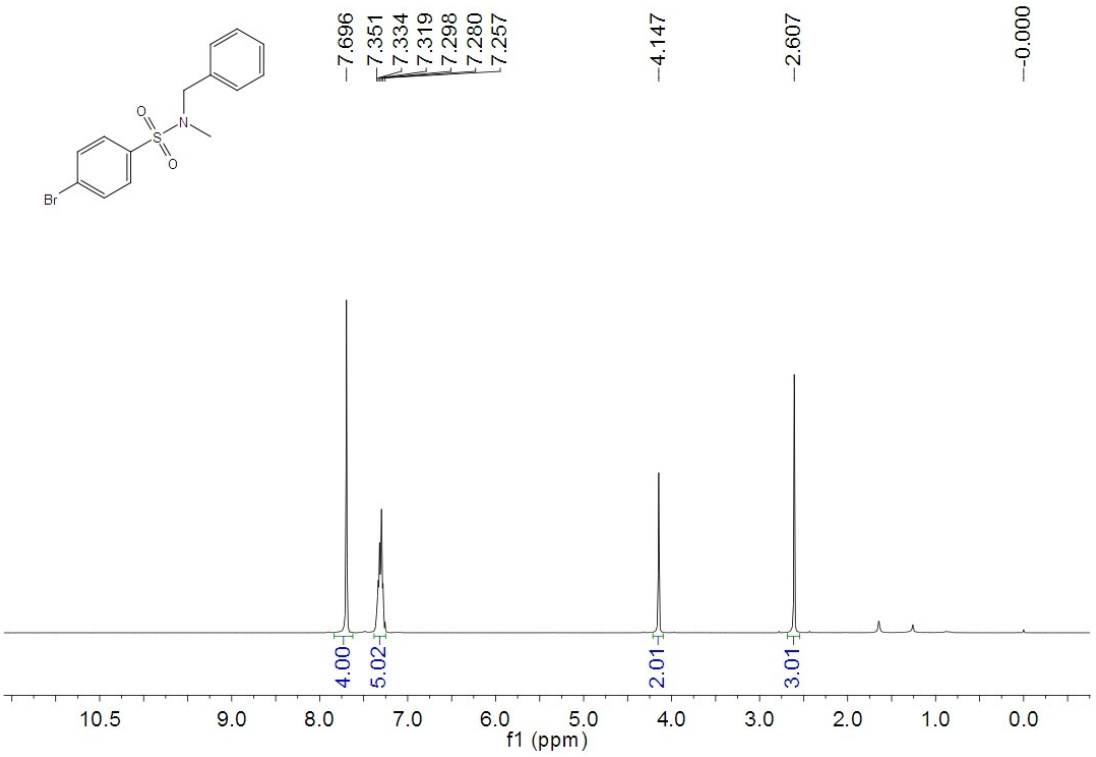


Fig. S-53. ^{1}H-NMR spectrum of **3o$_4$**.

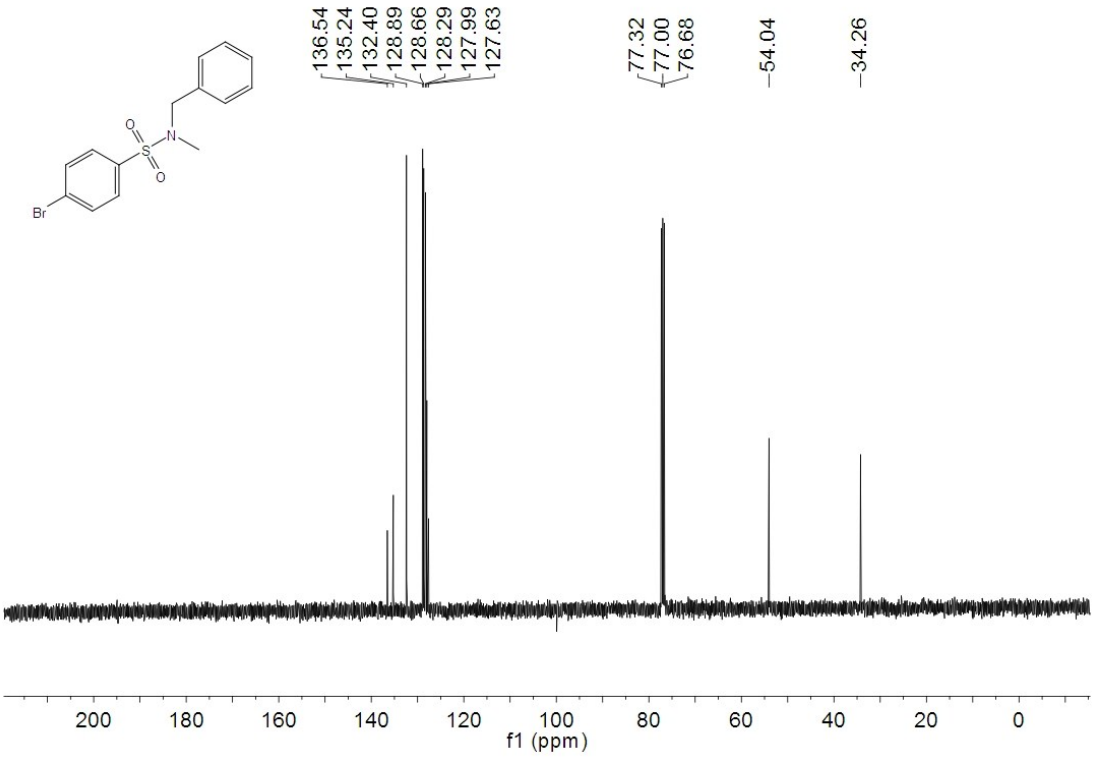


Fig. S-54. ^{13}C-NMR spectrum of **3o$_4$**.

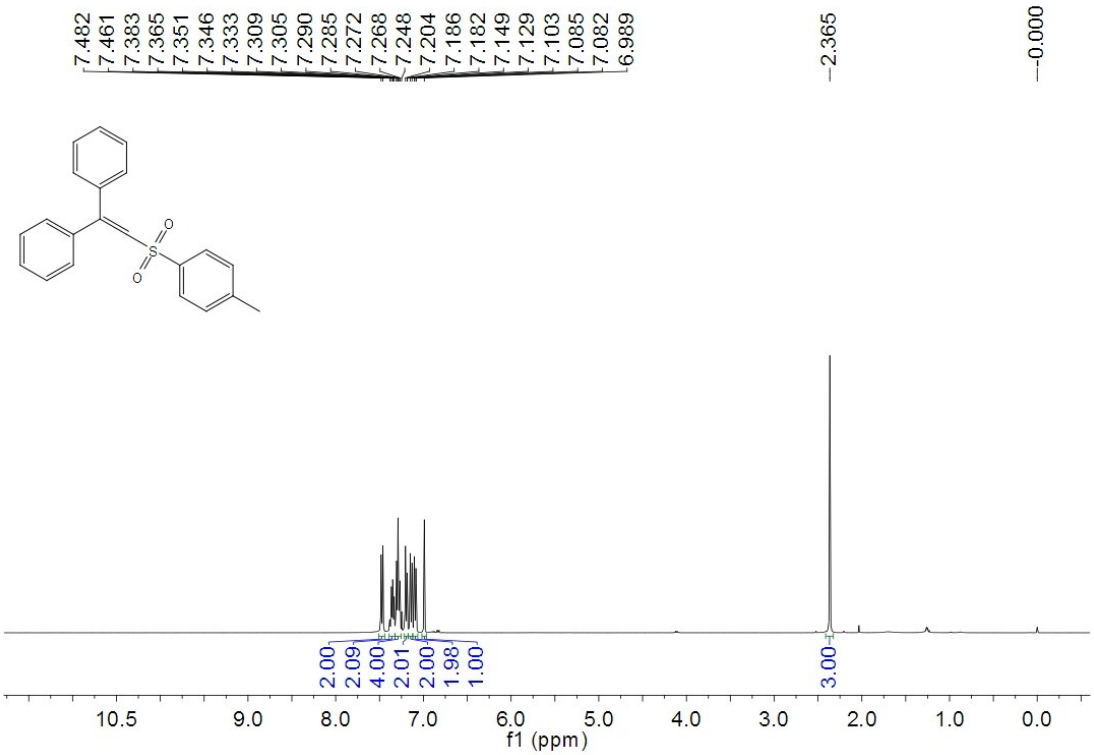


Fig. S-55. ^{1}H-NMR spectrum of **4a**.

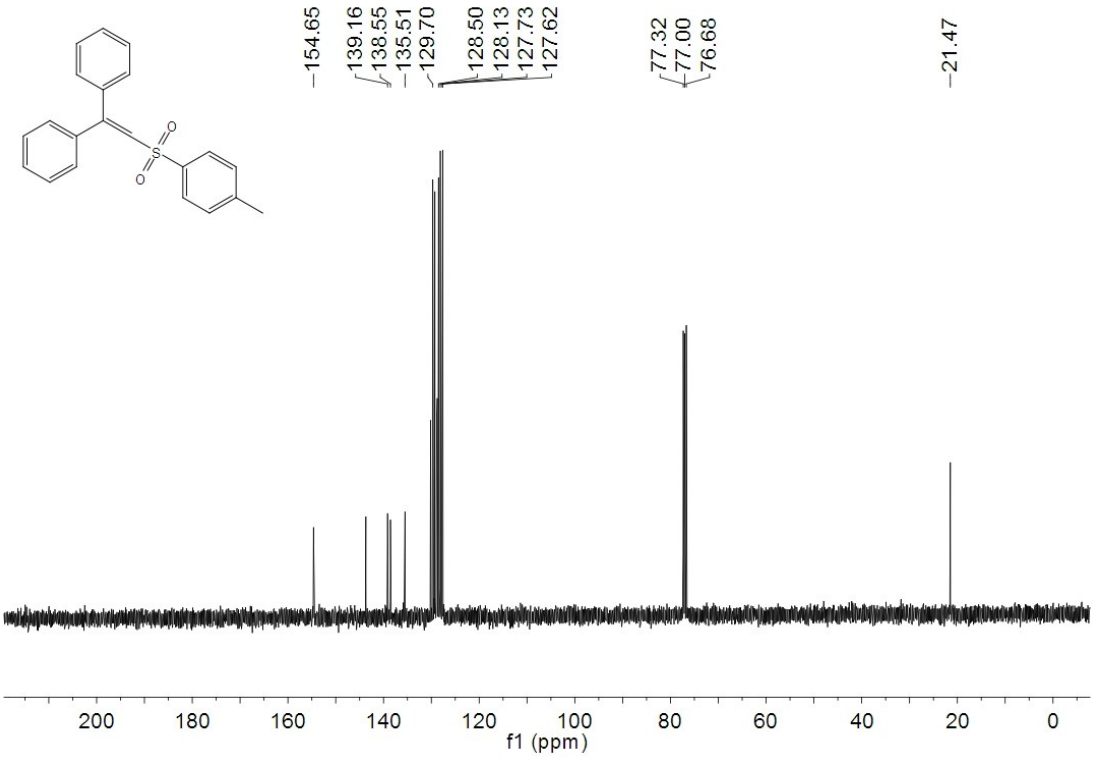


Fig. S-56. ^{13}C-NMR spectrum of **4a**.