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

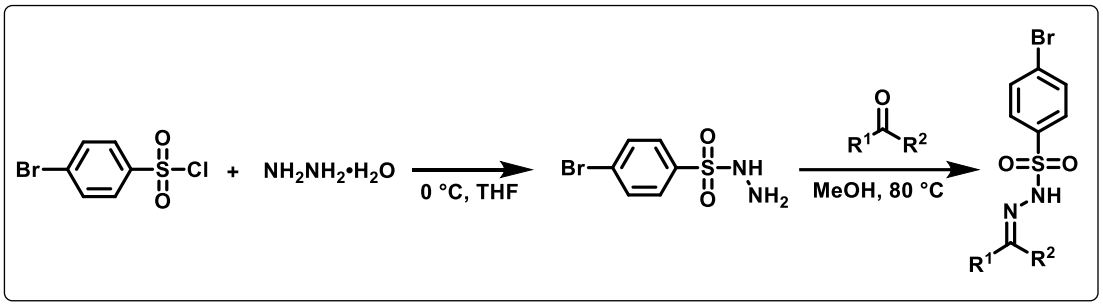
Copper-catalyzed sulfonylation of *N*-tosylhydrazones followed by a one-pot C-N bond formation

Kena Zhang, Olivier Provot, Christine Tran, Mouad Alami, and Abdallah Hamze*

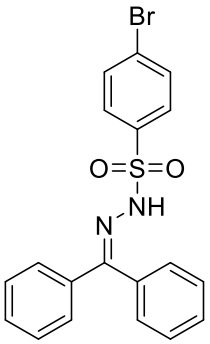
Université Paris-Saclay, BioCIS, Faculté de Pharmacie, CNRS, 92290 Châtenay-Malabry, France

Content

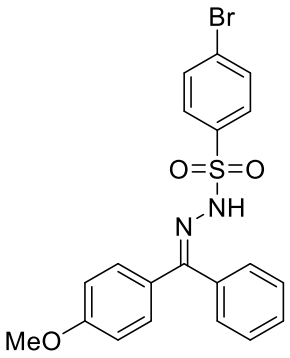
1. General procedure for the preparation of compounds 1:



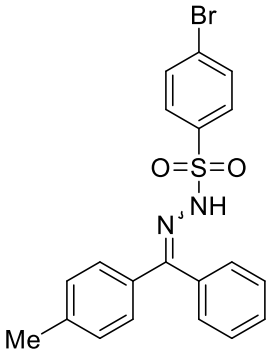
Hydrazine hydrate (12.5 mmol) was added dropwise to a solution of sulfonyl chloride (5.0 mmol) in dry THF (25.0 mL) at 0 °C under N_2. After vigorous stirring for 30 min at 0 °C, ethyl acetate (60.0 mL) was added, and the mixture was washed repeatedly with ice-cold 10% aqueous sodium chloride solution (3 × 20.0 mL). The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. Sulfonyl hydrazide was obtained and used for the next step without further purification. To a rapidly stirred suspension of sulfonyl hydrazides (1 eq) in dry methanol (2 mL/mmol) at 80 °C, the ketone (1 eq) was added dropwise. Within 2 - 12 hours, the *N*-sulfonyl hydrazones began to precipitate. The mixture was cooled to 0 °C, and the product was collected on a Büchner funnel, washed with petroleum ether, then was dried in vacuo to afford the pure product.



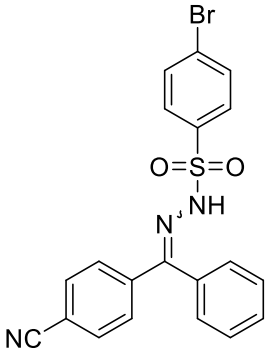
4-bromo-*N'*-(diphenylmethylene)benzenesulfonohydrazide (1a)[1] White solid (1.85g, 89%); **mp** 215 – 218 °C; **IR (film, cm^{-1})**: 3194, 1574, 1445, 1389, 1375, 1349, 1317, 1172, 1092, 1067, 1053, 1026, 1009; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.87 – 7.83 (m, 2H), 7.71 – 7.69 (m, 2H), 7.58 (bs, 1H), 7.55 – 7.49 (m, 3H), 7.47 – 7.43 (m, 2H), 7.39 – 7.26 (m, 3H), 7.16 – 7.13 (m, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 155.0, 137.7, 136.4, 132.5, 131.1, 130.4, 130.2, 130.0, 129.6, 128.5, 128.44, 128.37, 127.7; **HRMS** (ESI) *m/z*: $(M + Na)^+$ $C_{19}H_{15}BrN_2NaO_2S$ calcd. 438.9915, found 438.9910.



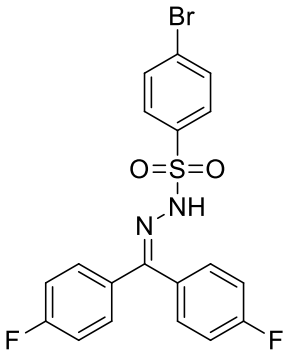
4-bromo-*N′*-((4-methoxyphenyl)(phenyl)methylene)benzenesulfonohydrazide (1b) White solid (1.62g, 73%); **mp** 131 – 133 °C; **IR (film, cm^{-1})**: 3185, 1606, 1573, 1509, 1457, 1445, 1374, 1349, 1317, 1300, 1251, 1171, 1054, 1029, 1010; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.86 – 7.82 (m, 2H), 7.69 – 7.66 (m, 2.5H), 7.52 – 7.26 (m, 3.3H), 7.13 – 7.01 (m, 3.3H), 6.88-6.80 (m, 0.7H), 3.87 – 3.80 (m, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 161.2, 160.8, 154.9, 154.8, 137.6, 136.7, 132.3, 130.1, 130.0, 129.9, 129.8, 129.4, 129.1, 128.9, 128.3, 128.2, 128.2, 127.7, 122.1, 115.2, 113.7, 55.4, 55.3; **HRMS** (ESI) *m*/*z*: $(M + Na)^+$ $C_{20}H_{17}BrN_2NaO_3S$ calcd. 469.0020, found 469.0019.



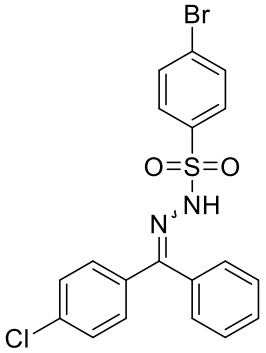
4-bromo-*N′*-(phenyl(p-tolyl)methylene)benzenesulfonohydrazide (1c) White solid (1.89g, 88%); **mp** 184 – 188 °C; **IR (film, cm^{-1})**: 3191, 1573, 1371, 1345, 1315, 1297, 1171, 1091, 1066, 1051, 1009; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.87 – 7.82 (m, 2H), 7.70 – 7.67 (m, 2H), 7.61 (s, 1H), 7.53 – 7.43 (m, 2H), 7.38 – 7.26 (m, 5H), 7.13 – 7.01 (m, 2H), 2.44 – 2.34 (m, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 155.1, 140.4, 137.6, 136.4, 132.3, 130.5, 130.0, 129.4, 128.2, 128.2, 127.8, 127.6, 21.4; **HRMS** (ESI) *m*/*z*: $(M + Na)^+$ $C_{20}H_{17}BrN_2NaO_2S$ calcd. 453.0071, found 453.0069.



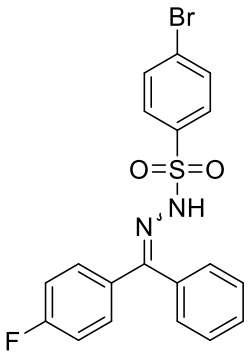
4-bromo-*N'*-((4-cyanophenyl)(phenyl)methylene)benzenesulfonohydrazide (1d) Light yellow solid (1.92g, 87%); **mp** 188 – 192 °C; **IR (film, cm^{-1})**: 3125, 2234, 1573, 1405, 1351, 1318, 1279, 1162, 1113, 1092, 1068, 1054, 1025, 1011; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.85 – 7.80 (m, 3H), 7.72 – 7.68 (m, 2H), 7.60 – 7.52 (m, 7H), 7.15 – 7.12 (m, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 152.5, 140.4, 137.4, 132.6, 132.2, 130.9, 130.3, 130.0, 129.5, 128.9, 128.2, 128.0, 118.5, 113.4; **HRMS** (ESI) *m/z*: $(M + H)^+$ $C_{20}H_{15}BrN_3O_2S$ calcd. 442.0048, found 442.0042.



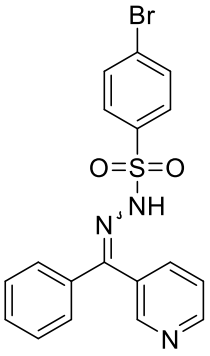
***N'*-(bis(4-fluorophenyl)methylene)-4-bromobenzenesulfonohydrazide (1e) White solid** (1.85g, 82%); **mp** 187 – 191 °C; **IR (film, cm^{-1})**: 3203, 1600, 1573, 1505, 1381, 1346, 1310, 1225, 1173, 1155, 1090, 1066, 1050, 1009; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.85 – 7.81 (m, 2H), 7.71 – 7.68 (m, 2H), 7.59 (bs, 1H), 7.42 – 7.37 (m, 2H), 7.26 – 7.13 (m, 4H), 7.03 – 6.96 (m, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 164.1 (d, J = 251.4 Hz), 163.7 (d, J = 251.8 Hz), 152.9, 137.5, 132.5, 130.6 (d, J = 8.4 Hz), 129.7, 129.6, 128.7, 126.8 (d, J = 3.4 Hz), 117.3 (d, J = 21.7 Hz), 115.6 (d, J = 21.9 Hz) ; **^{19}F NMR** (188 MHz, $CDCl_3$) δ (ppm): -108.68, -110.04; **HRMS** (ESI) *m/z*: $(M + Na)^+$ $C_{19}H_{13}BrF_2N_2NaO_2S$ calcd. 474.9726, found 474.9726.



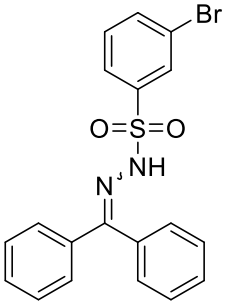
4-bromo-*N'*-((4-chlorophenyl)(phenyl)methylene)benzenesulfonohydrazide (1f) White solid (1.87g, 83%); **mp** 154 – 158 °C; **IR (film, cm^{-1})**: 3183, 1650, 1575, 1486, 1444, 1389, 1347, 1316, 1279, 1171, 1089, 1068, 1051, 1010; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.80 – 7.74 (m, 2H), 7.64 - 7.60 (m, 2H), 7.53 (s, 1H), 7.48 – 7.43 (m, 3H), 7.36 – 7.26 (m, 2H), 7.24 – 7.18 (m, 2H), 7.07 – 7.03 (m, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 153.7, 137.6, 136.4, 136.1, 134.9, 132.5, 130.7, 130.6, 130.4, 130.3, 130.1, 130.0, 129.6, 129.5, 128.9, 128.7, 128.5, 128.3, 127.7; **HRMS** (ESI) *m/z*: $(M + Na)^+$ $C_{19}H_{14}BrClN_2NaO_2S$ calcd. 472.9525, found 472.9516.



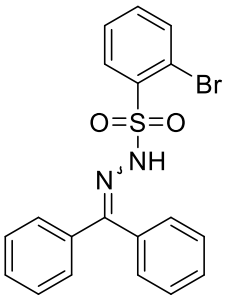
4-bromo-*N'*-((4-fluorophenyl)(phenyl)methylene)benzenesulfonohydrazide (1g) White solid (1.86g, 86%); **mp** 203 – 208 °C; **IR (film, cm^{-1})**: 3210, 1573, 1505, 1380, 1347, 1318, 1230, 1170, 1066, 1051, 1008; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.87 – 7.82 (m, 2H), 7.73 – 7.66 (m, 2H), 7.58 (s, 1H), 7.56 – 7.50 (m, 1H), 7.46 – 7.10 (m, 5H), 7.04 – 6.93 (m, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 164.1 (d, J = 251.2 Hz), 163.6 (d, J = 251.6 Hz), 153.94, 137.6 (d, J = 5.1 Hz), 136.31, 132.51, 130.76, 130.65, 130.51, 130.34, 130.07, 129.73, 129.61, 129.56, 128.50, 128.29, 127.70, 117.2 (d, J = 25.0 Hz), 115.51 (d, J = 21.8 Hz); **^{19}F NMR** (188 MHz, $CDCl_3$) δ -109.05, -110.32 ; **HRMS** (ESI) *m/z*: $(M + H)^+$ $C_{19}H_{15}BrFN_2O_2S$ calcd. 435.0001, found 434.9999.



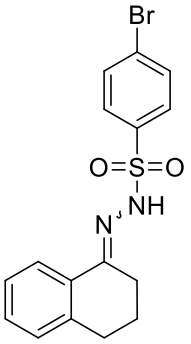
4-bromo-*N'*-(phenyl(pyridin-3-yl)methylene)benzenesulfonohydrazide (1h) White solid (708mg, 34%); **mp** 167 – 172 °C; **IR (film, cm^{-1})**: 1591, 1574, 1469, 1440, 1414, 1388, 1346, 1331, 1282, 1157, 1092, 1050, 1027, 1010; **^{1}H NMR** (300 MHz, DMSO-d_6) δ (ppm): 10.85 (bs, 1H), 8.71 (d, J = 3.9 Hz, 1H), 8.43 (s, 1H), 7.90 – 7.83 (m, 4H), 7.69 (dt, J_1 = 1.7 Hz, J_2 = 7.8, 1H), 7.56 (dd, J_1 = 4.9 Hz, J_2 = 7.7 Hz, 1H), 7.43 – 7.27 (m, 2H); **^{13}C NMR** (75 MHz, DMSO) δ (ppm): 152.2, 150.2, 149.0, 138.2, 136.7, 136.6, 132.2, 129.9, 129.6, 128.7, 128.5, 127.1, 127.0, 123.9; **HRMS** (ESI) *m/z*: $(M + H)^+$ $C_{18}H_{15}BrN_3O_2S$ calcd. 418.0048, found 418.0043



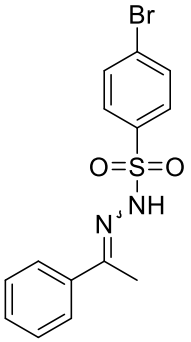
3-bromo-*N'*-(diphenylmethylene)benzenesulfonohydrazide (1i) White solid (1.76g, 85%); **mp** 134 – 137 °C; **IR (film, cm^{-1})**: 3191, 1444, 1394, 1346, 1321, 1162, 1116, 1062, 1032; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 8.15 (t, *J* = 1.8 Hz, 1H), 7.92 (dt, J_1 = 1.1 Hz, J_2 =7.9 Hz, 1H), 7.74 (ddd, J_1 = 1.0 Hz, J_2 =1.9 Hz, J_3 = 8.0 Hz, 1H), 7.60 (s, 1H), 7.56 – 7.52 (m, 3H), 7.47 – 7.43 (m, 3H), 7.36 – 7.28 (m, 3H), 7.19 – 7.13 (m, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 155.2, 140.4, 136.4, 131.0, 130.7, 130.4, 130.2, 130.0, 128.4, 128.4, 127.8, 126.6, 123.1; **HRMS** (ESI) *m/z*: $(M + H)^+ C_{19}H_{16}BrN_2O_2S$ calcd. 417.0095, found 417.0098.



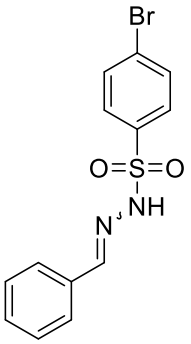
2-bromo-*N'*-(diphenylmethylene)benzenesulfonohydrazide (1j) Yellow solid (1.80g, 87%); **mp** 94 – 99 °C; **IR (film, cm^{-1})**: 1576, 1447, 1379, 1345, 1320, 1306, 1169, 1127, 1104, 1058, 1026; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 8.39 (dd, J_1 = 1.8 Hz, J_2 =7.9 Hz, 1H), 8.19 (bs, 1H), 7.72 (dd, J_1 = 1.3 Hz, J_2 =7.9 Hz, 1H), 7.63 – 7.54 (m, 4H), 7.47 (td, J_1 = 1.8 Hz, J_2 =7.7 Hz, 1H), 7.35 – 7.23 (m, 7H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 155.2, 137.8, 136.3, 135.0, 134.4, 133.2, 131.1, 130.3, 130.1, 129.7, 128.7, 128.3, 128.1, 127.7, 119.7; **HRMS** (ESI) *m/z*: $(M + Na)^+ C_{19}H_{15}BrN_2NaO_2S$ calcd. 438.9915, found 438.9913.



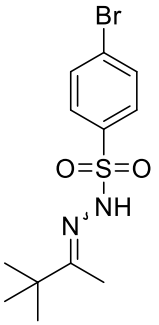
4-bromo-*N'*-(3,4-dihydronaphthalen-1(2H)-ylidene)benzenesulfonohydrazide (1k) White solid (1.59g, 84%); **mp** 178 – 181 °C; **IR (film, cm^{-1})**: 3200, 1570, 1468, 1389, 1338, 1315, 1279, 1167, 1078, 1065, 1050, 1006; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.96 (dd, J_1 = 1.3 Hz, J_2 = 7.7 Hz, 1H), 7.91 (dt, J_1 = 2.3 Hz, J_2 = 9.1 Hz, 2H), 7.69 – 7.65 (m, 2H),7.27 – 7.18 (m, 2H), 7.10 (d, *J* = 7.1 Hz, 1H), 2.75 – 2.71 (m, 2H), 2.48 (t, *J* = 6.6 Hz, 2H), 1.90 (p, *J* = 6.6 Hz, 2H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 140.0, 137.6, 132.4, 131.4, 130.0, 129.8, 128.6, 126.7, 125.1, 123.2, 29.4, 25.6, 21.5; **HRMS** (ESI) *m/z*: $(M + Na)^+ C_{16}H_{15}BrN_2NaO_2S$ calcd. 402.9915, found 402.9914.



4-bromo-*N′*-(1-phenylethylidene)benzenesulfonohydrazide (1l)[2] White solid (1.46g, 83%); **mp** 171 – 175 °C; **IR (film, cm^{-1})**: 3213, 1576, 1409, 1389, 1342, 1303, 1168, 1086, 1069, 1049, 1011; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.98 – 7.78 (m, 3H), 7.68 – 7.54 (m, 4H), 7.49 – 7.32 (m, 3H), 2.21 – 2.18 (m, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 153.5, 137.5, 137.2, 132.4, 130.0, 129.8, 128.6, 126.4, 13.6; **HRMS** (ESI) *m*/*z*: $(M + Na)^+$ $C_{14}H_{13}BrN_2NaO_2S$ calcd. 376.9758, found 376.9755.

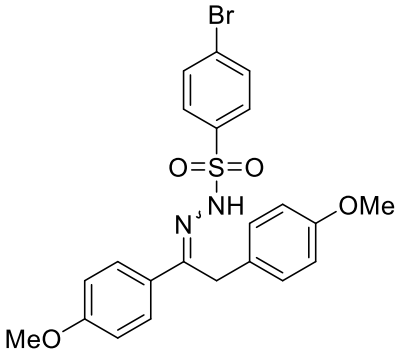


***N′*-benzylidene-4-bromobenzenesulfonohydrazide (1m)[3] White solid** (1.30g, 77%); **mp** 159 – 161 °C; **IR (film, cm^{-1})**: 3177, 1573, 1420, 1389, 1359, 1327, 1309, 1225, 1173, 1091, 1067, 1042, 1010; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 8.13 (bs, 1H), 7.88 – 7.84 (m, 2H), 7.78 (s, 1H), 7.68 – 7.63 (m, 2H), 7.58 – 7.55 (m, 2H), 7.339 – 7.32 (m, 3H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 148.7, 137.4, 133.0, 132.5, 130.9, 129.6, 128.9, 128.7, 127.6; **HRMS** (ESI) *m*/*z*: $(M + Na)^+$ $C_{13}H_{11}BrN_2NaO_2S$ calcd. 362.9602, found 362.9600.

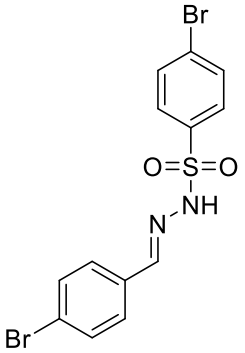


4-bromo-*N′*-(3,3-dimethylbutan-2-ylidene)benzenesulfonohydrazide (1n) White solid (1.27g, 76%); **mp** 179 – 182 °C; **IR (film, cm^{-1})**: 3218, 1574, 1468, 1389, 1341, 1234, 1172, 1125, 1090, 1069, 1040, 1003; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.85 – 7.81 (m, 2H), 7.66 – 7.62 (m, 2H), 1.74 (s, 3H), 1.03 (s, 9H); **^{13}C NMR** (75

MHz, $CDCl_3$) δ (ppm): 164.8, 137.4, 132.1, 129.9, 128.3, 39.0, 27.6, 12.0; **HRMS** (ESI) *m*/*z*: $(M + Na)^+$ $C_{12}H_{17}BrN_2NaO_2S$ calcd. 357.0071, found 357.0068.

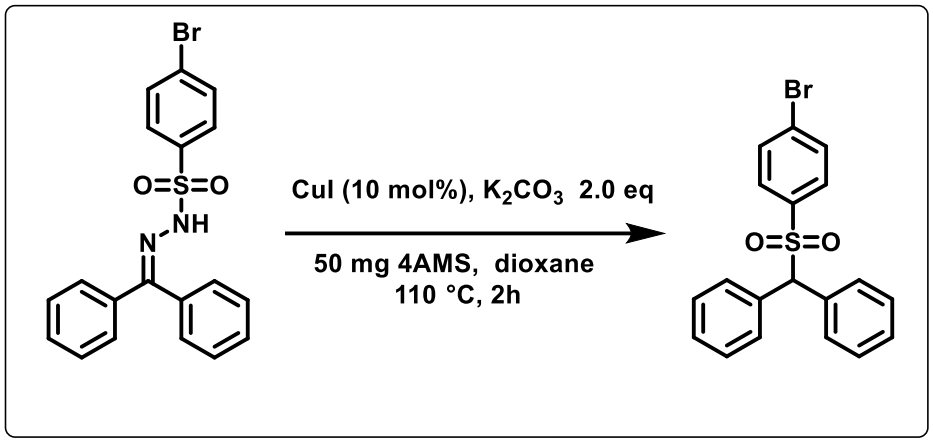


***N'*-(1,2-bis(4-methoxyphenyl)ethylidene)-4-bromobenzenesulfonohydrazide (1o) Yellow solid** (1.74g, 71%); **mp** 142 – 145 °C; **IR (film, cm^{-1})**: 3201, 1610, 1576, 1510, 1470, 1397, 1363, 1318, 1298, 1260, 1244, 1178, 1167, 1067, 1030, 1002; **^{1}H NMR** (300 MHz, $CDCl_3$) δ (ppm): 7.76 – 7.56 (m, 6H), 6.90 – 6.80 (m, 4H), 6.72 (d, *J* = 8.7 Hz, 2H), 3.91 (s, 1.4H), 3.81 (s, 2.2H), 3.78 (s, 2.6H), 3.77 (s, 1.3H), 3.67 (s, 0.5H) ; **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 161.3, 159.0, 154.7, 137.4, 132.4, 132.2, 131.1, 130.5, 130.2, 129.8, 129.6, 128.9, 128.6, 128.3, 128.2, 125.3, 115.0, 114.1, 114.0, 55.5, 55.4, 55.3, 44.5, 44.0, 32.6; **HRMS** (ESI) *m*/*z*: $(M + Na)^+$ $C_{22}H_{21}BrN_2NaO_4S$ calcd. 513.0283, found 513.0279.



(*E*)-4-bromo-*N'*-(4-bromobenzylidene)benzenesulfonohydrazide (1p) White solid (mg, 87%); **mp** 213 - 214 °C; **R_f** = 0.3 (cyclohexane/ethyl acetate 8/2); **IR** (film, cm^{-1}): 3176, 1572, 1466, 1362, 1334, 1278, 1169, 1088, 1063, 1005; **^{1}H NMR** (300 MHz, DMSO-d_6) δ (ppm): 11.72 (s, 1H), 7.91 (s, 1H), 7.83 – 7.78 (m, 4H), 7.59 (d, *J* = 8.5 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 2H); **^{13}C NMR** (75 MHz, DMSO-d_6) δ (ppm): 146.5, 138.1, 132.8, 132.4, 131.8, 129.2, 128.7, 127.1, 123.5; **HRMS** (ESI) *m*/*z*: $(M + H)^+$ $C_{13}H_{11}Br_2N_2O_2S$ calcd. 418.8888, found 418.8882.

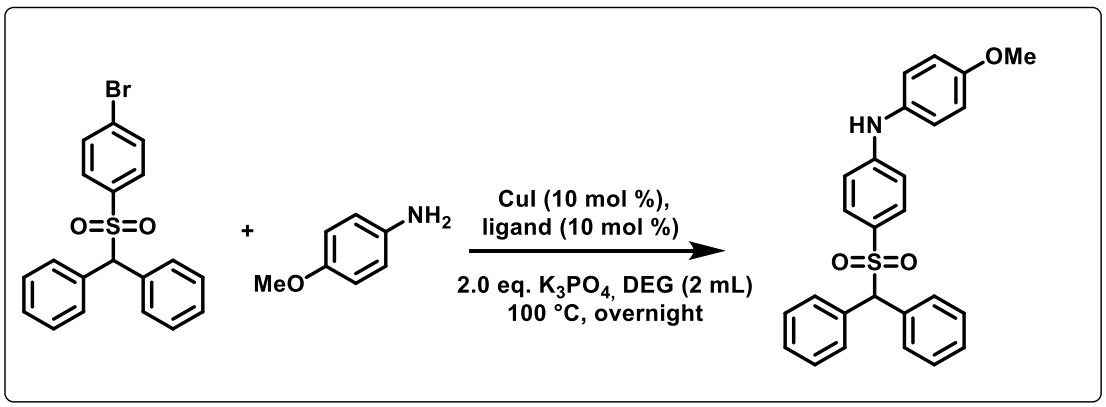
2. General procedure for the first step:



A 10 mL sealed tube with a magnetic stir bar charged with sulfonylhydrazones (0.3 mmol, 1 equiv), CuI (5.7 mg, 0.1 equiv), K_2CO_3 (83 mg, 2 equiv), 50 mg 4A MS, and the mixture was put under argon atmosphere for five minutes, and then dioxane (2 mL) was added. The reaction vessel was placed in a preheated oil bath at 110 °C and then stirring at this temperature for 2 h. After the total consumption of the starting material (TLC), the reaction mixture was then allowed to cool to ambient temperature. The crude was purified by column chromatography flash on silica gel using cyclohexane and ethyl acetate (8/2) as the eluent to afford the desired product.

(((4-bromophenyl)sulfonyl)methylene)dibenzene (2a) White solid (108 mg, 93%); **mp** 172 – 173 °C; $\mathbf{R_f}$ = 0.3 (cyclohexane/ethyl acetate 7/3); **^{1}H NMR** (400 MHz, $CDCl_3$) δ (ppm): 7.55 - 7.45 (m, 8H), 7.35 - 7.31 (m, 6H), 5.28 (s, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ (ppm): 137.5, 132.8, 132.1, 130.7, 130.1, 129.1, 129.0, 128.9, 76.7; **HRMS** (ESI) *m/z*: $(M + Na)^+$ $C_{19}H_{15}BrNaO_2S$ calcd. 408.9874, found 408.9878.

3. General procedure for the second step:

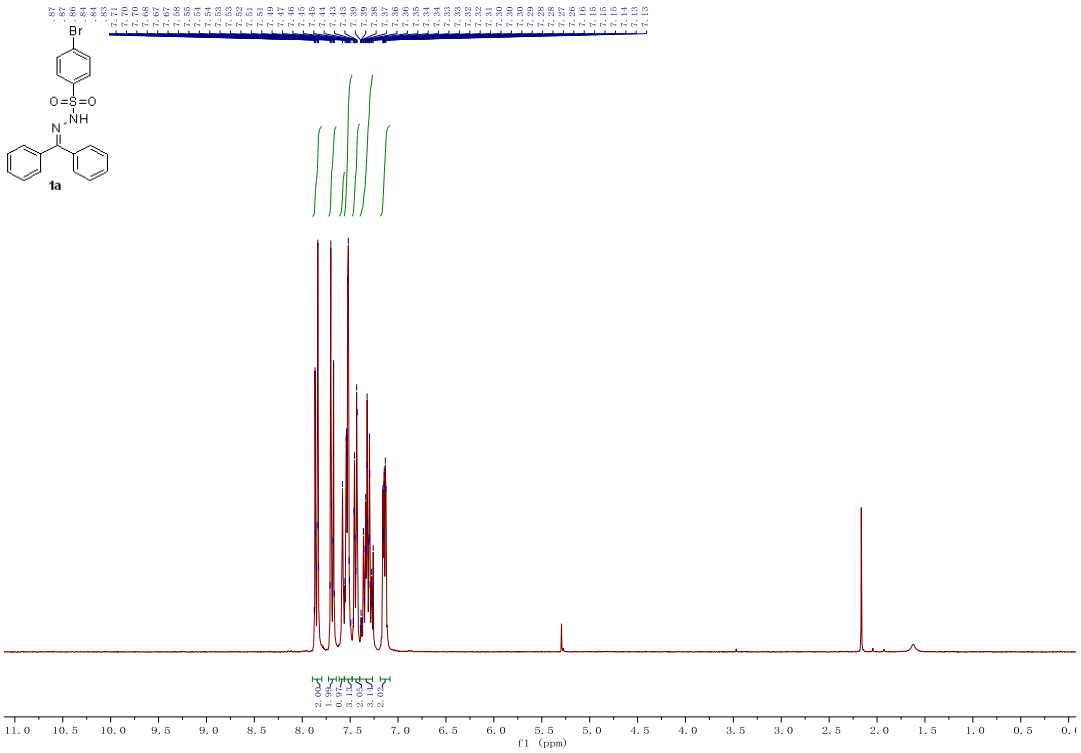


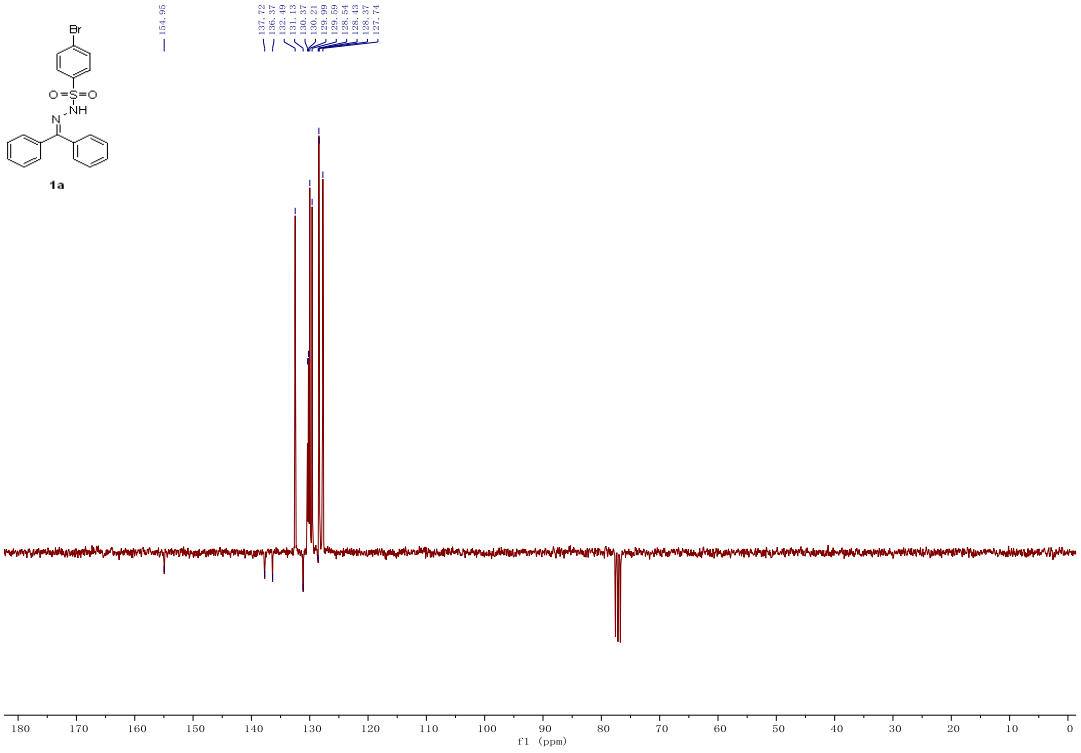
(((4-bromophenyl)sulfonyl)methylene)dibenzene (0.3 mmol, 1.0 eq.), corresponding amine (1.2 eq),

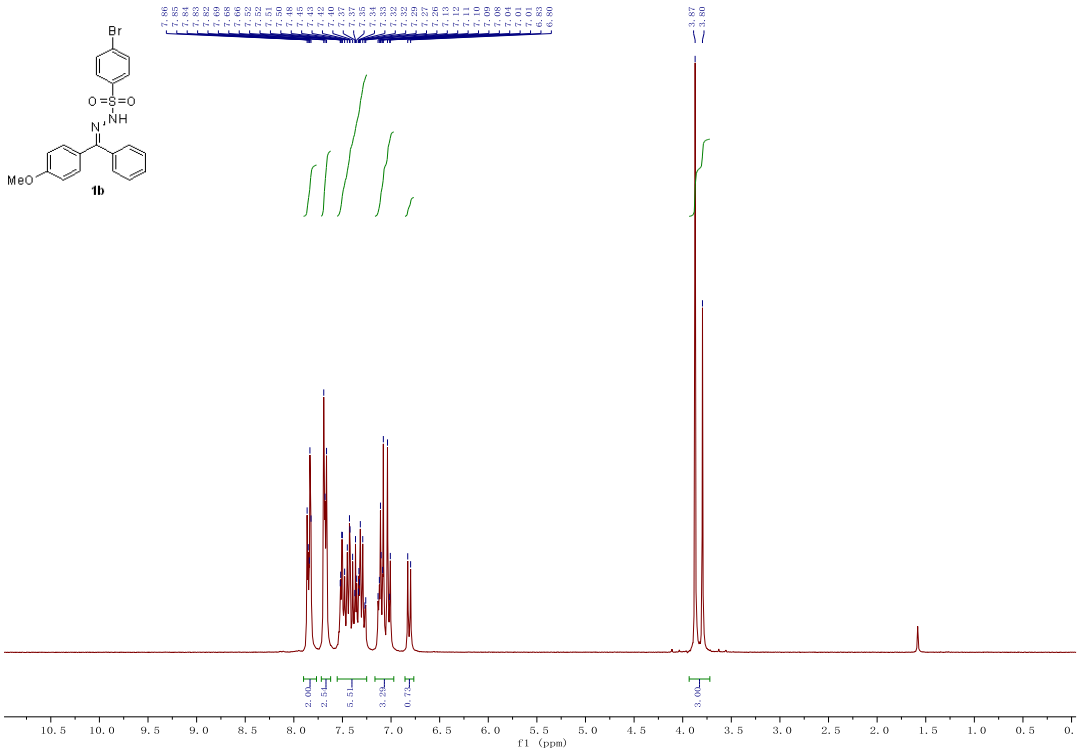
CuI (5.7 mg, 0.1 equiv), ligand (0.1 eq), K_3PO_4 (127 mg, 2.0 eq) were mixed under argon atmosphere for five minutes, and 2 mL of solvent was added to the mixture. The reaction vessel was placed in a preheated oil bath and then stirring at this temperature overnight. After the total consumption of the starting material (TLC), the reaction mixture was then allowed to cool to ambient temperature and then extracted with MeOH/DCM (V/V 1/9). The organic layer was combined and dried over $MgSO_4$. The crude was purified by column chromatography flash on silica gel using cyclohexane and ethyl acetate (7/3) as eluent to afford the desired product.

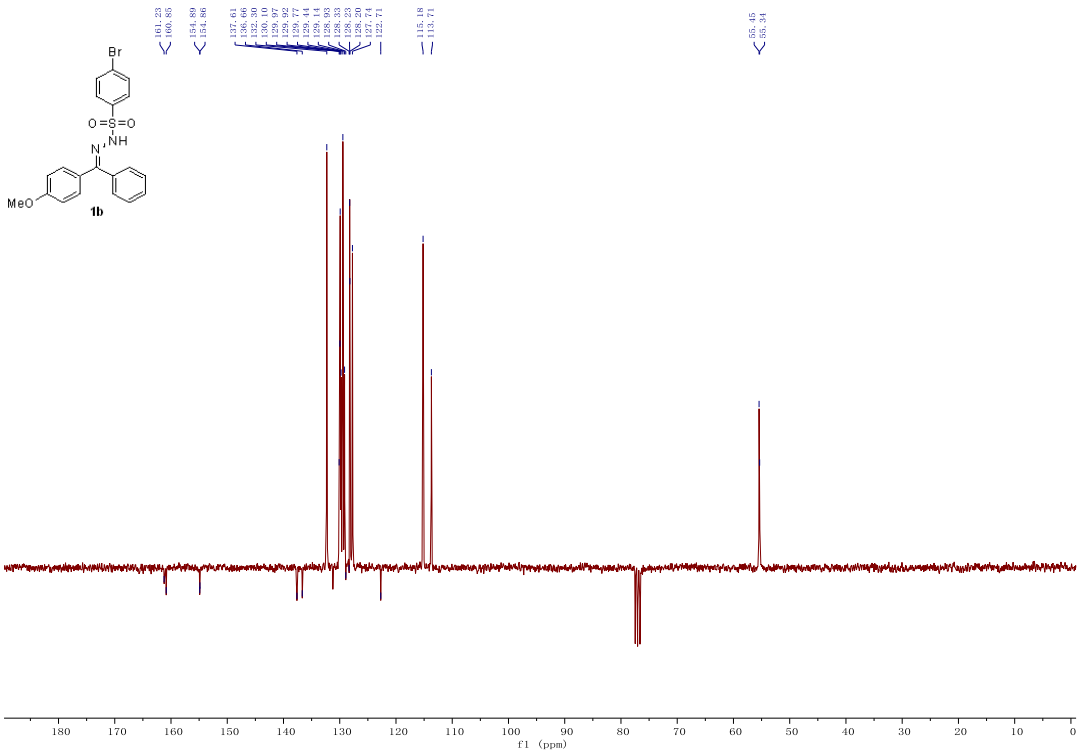
4. ^{1}H, ^{13}C and ^{19}F spectrum:

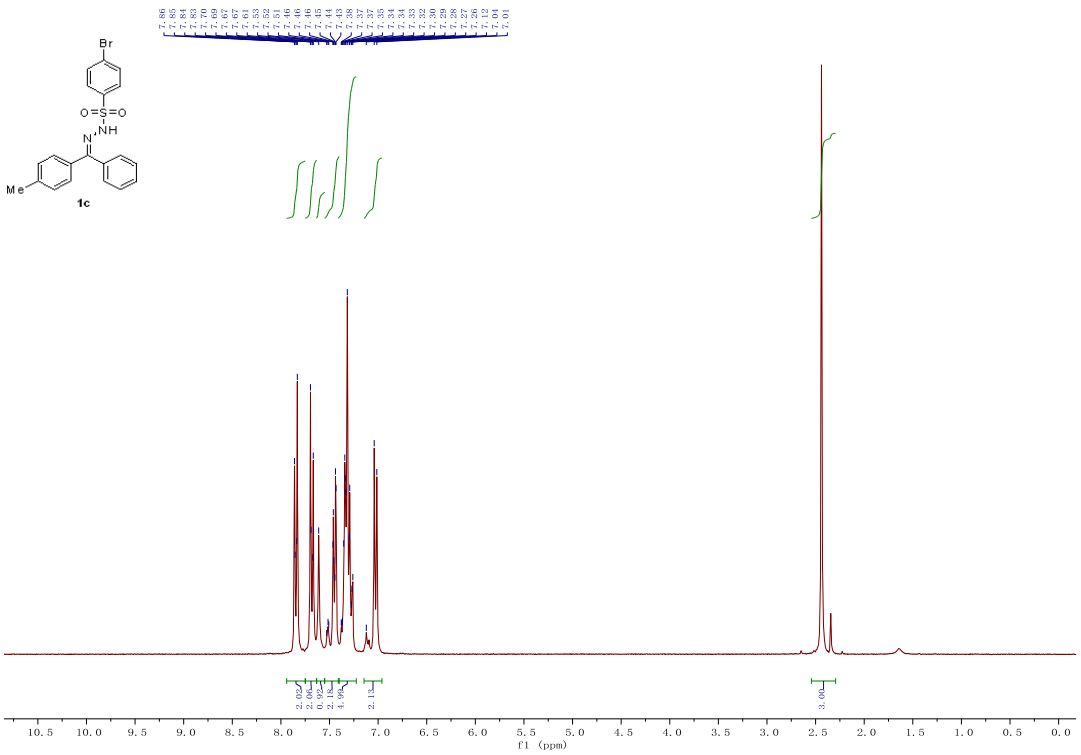
Starting materials

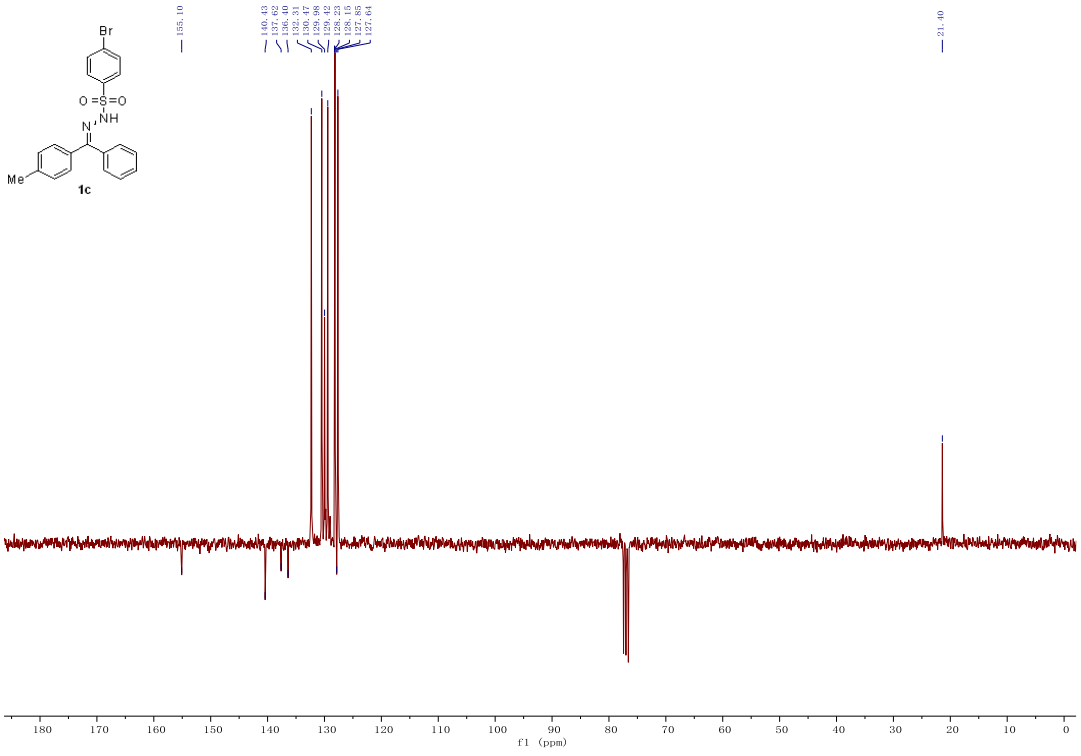


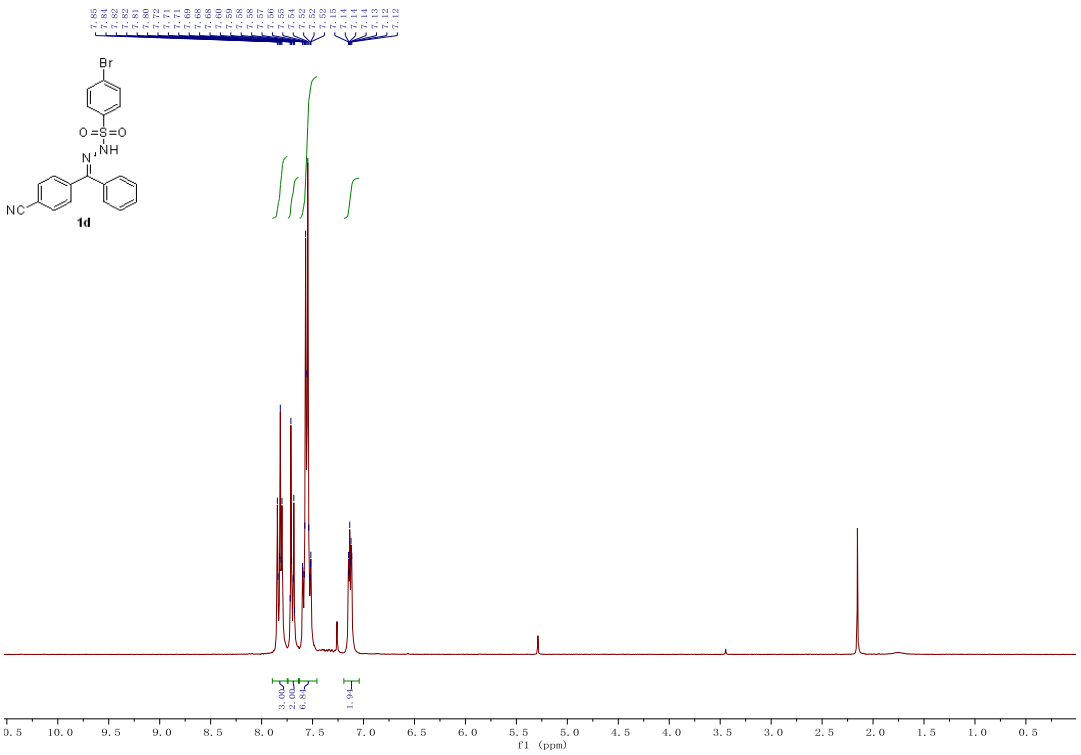


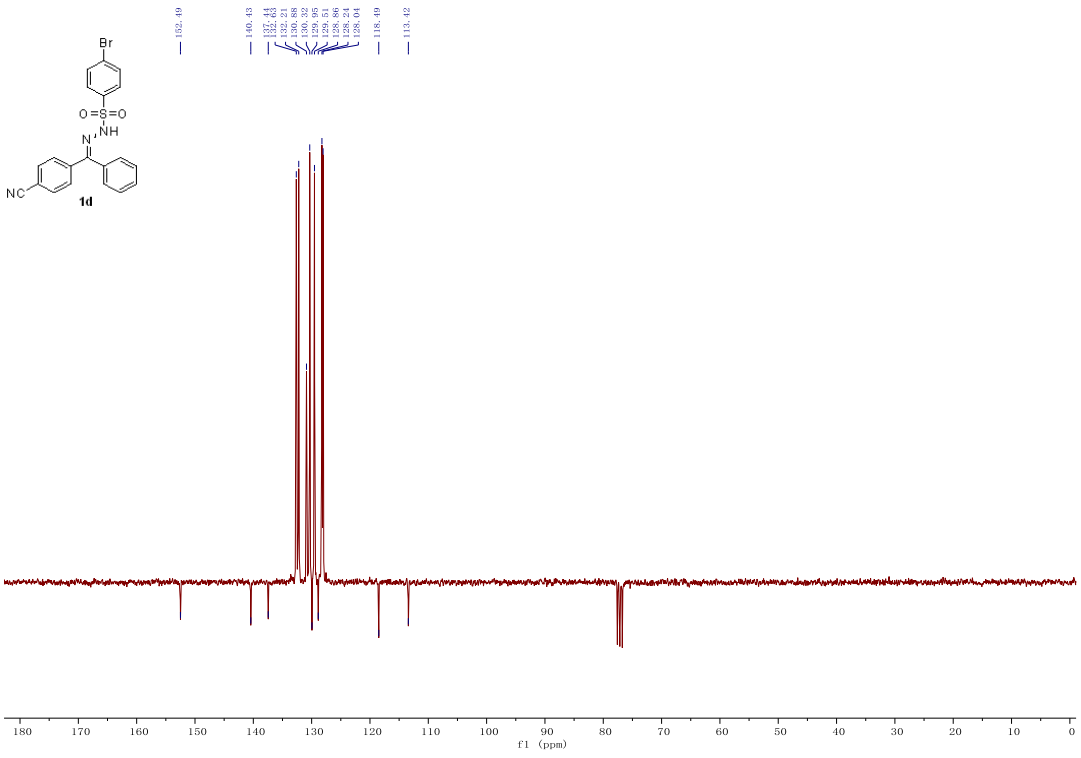

Br
O=S=O
NH
N
MeO
1b
7.86
7.85
7.84
7.83
7.82
7.69
7.68
7.66
7.52
7.52
7.51
7.50
7.48
7.45
7.43
7.42
7.40
7.37
7.37
7.35
7.34
7.33
7.32
7.32
7.29
7.27
7.26
7.13
7.12
7.11
7.10
7.09
7.08
7.04
7.01
7.01
6.83
6.80
3.87
3.80
2.00
2.54
5.51
3.39
0.73
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)

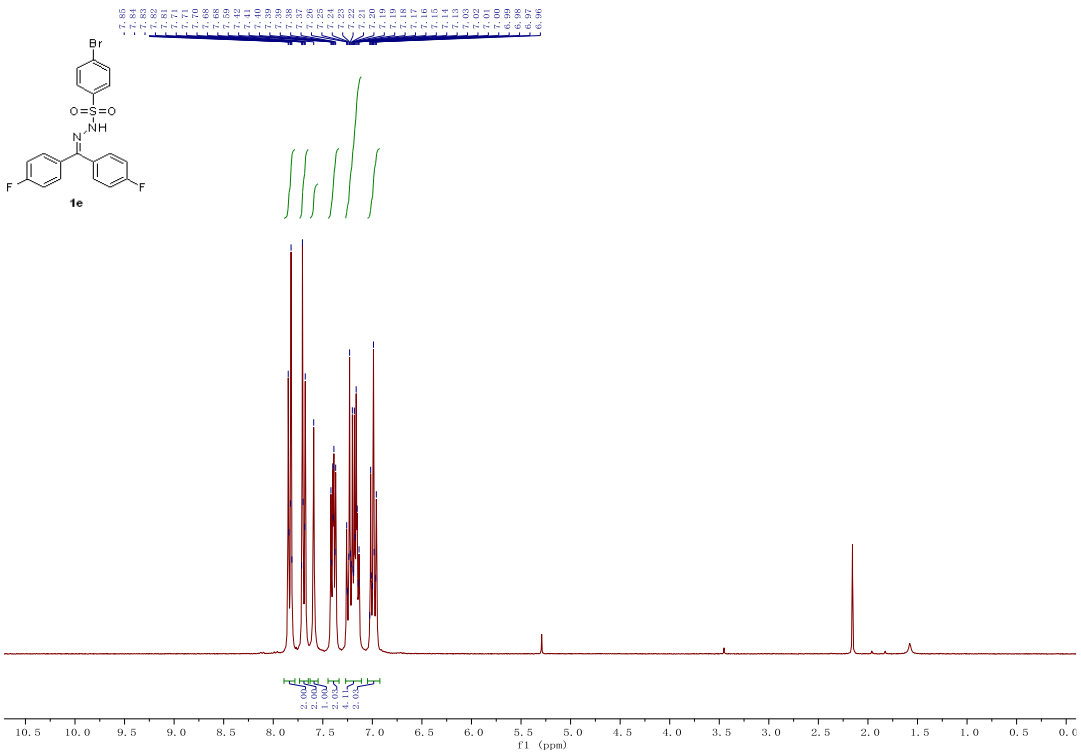

Br
O=S=O
NH
N
MeO
1b
161.23
160.85
154.89
154.86
137.61
136.66
132.30
130.10
129.97
129.92
129.77
129.44
129.14
128.93
128.33
128.23
128.20
127.74
122.71
115.18
115.71
55.45
55.34
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

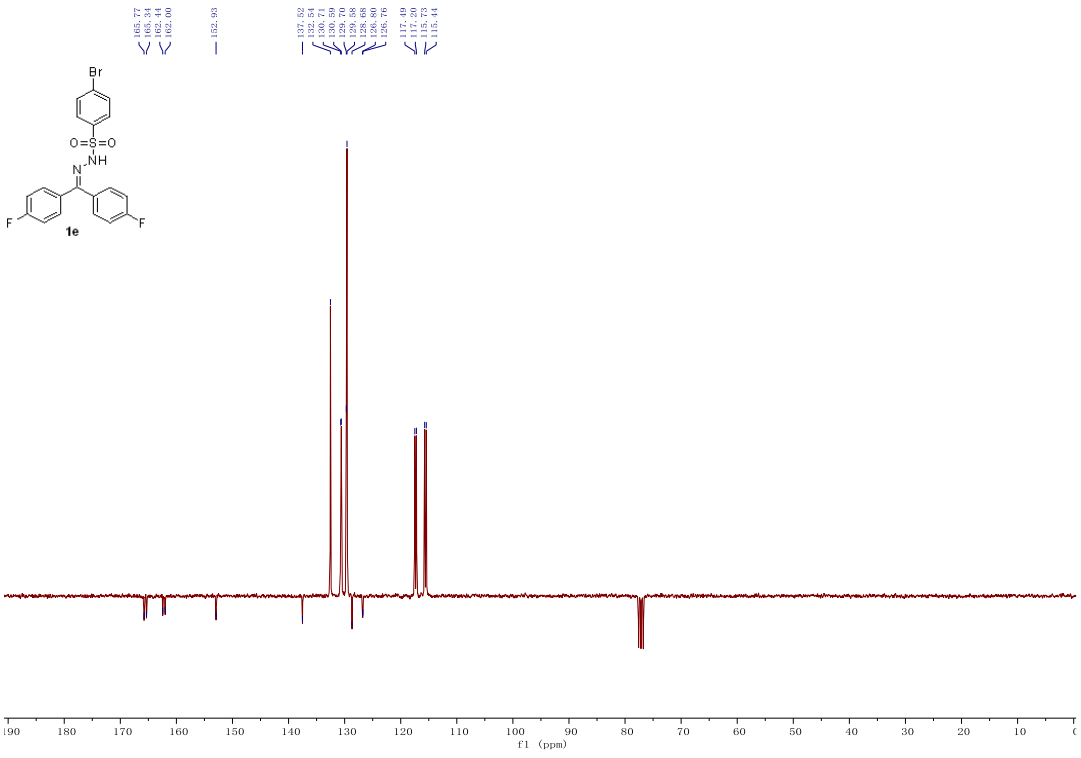

Br
O=S=O
NH
N
Me
1c
7.86
7.85
7.84
7.83
7.70
7.69
7.67
7.67
7.61
7.53
7.52
7.51
7.46
7.46
7.46
7.45
7.44
7.43
7.38
7.37
7.37
7.35
7.34
7.34
7.33
7.32
7.30
7.29
7.28
7.27
7.26
7.12
7.01
2.02
2.06
0.92
1.82
4.99
2.13
3.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

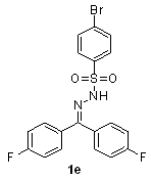

Br
O=S=O
NH
N
Me
1c
155.10
140.43
135.62
136.40
132.31
130.47
129.98
129.42
128.23
128.15
127.85
127.64
21.40
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.85
7.84
7.82
7.82
7.81
7.80
7.72
7.71
7.71
7.69
7.68
7.68
7.60
7.59
7.58
7.56
7.56
7.55
7.54
7.54
7.52
7.52
7.15
7.14
7.14
7.14
7.13
7.12
7.12
Br
O=S=O
NH
N
NC
1d
3.00
2.04
6.84
1.94
0.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


162.49
146.43
137.44
132.68
132.21
130.88
130.32
129.95
129.51
128.86
128.24
128.04
118.49
113.42
Br
O=S=O
NH
N
NC
1d
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

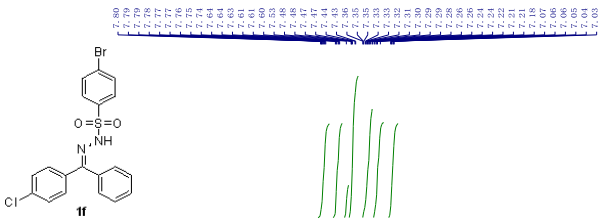

7.85
7.84
7.83
7.82
7.81
7.81
7.71
7.70
7.69
7.68
7.68
7.59
7.42
7.41
7.40
7.39
7.38
7.37
7.26
7.25
7.24
7.23
7.22
7.21
7.20
7.19
7.18
7.17
7.16
7.15
7.14
7.13
7.03
7.02
7.01
7.00
6.99
6.98
6.97
6.96
Br
O=S=O
NH
N
F
F
1e
2.00
2.00
1.00
2.03
4.11
2.03
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


165.77
163.34
162.44
162.00
152.93
137.52
132.54
130.71
130.59
129.70
129.58
128.68
128.60
126.76
117.49
117.20
115.73
115.44
Br
O=S=O
NH
N
F
F
1e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Br
O=S=O
NH
N
F
F

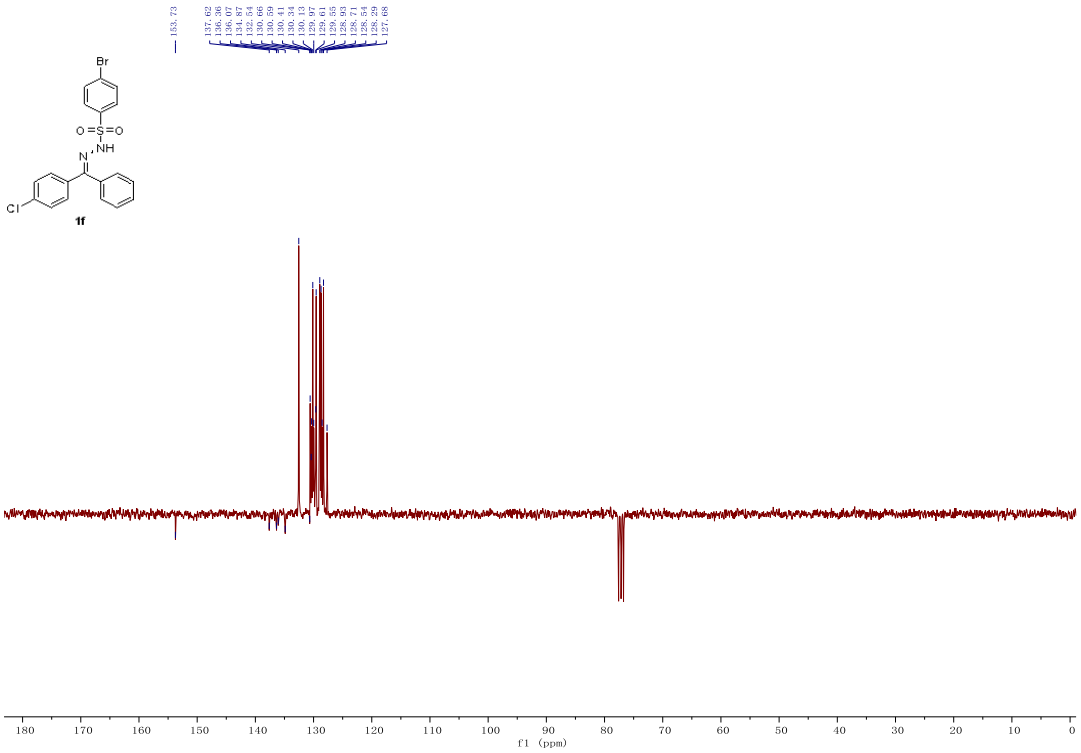
1e

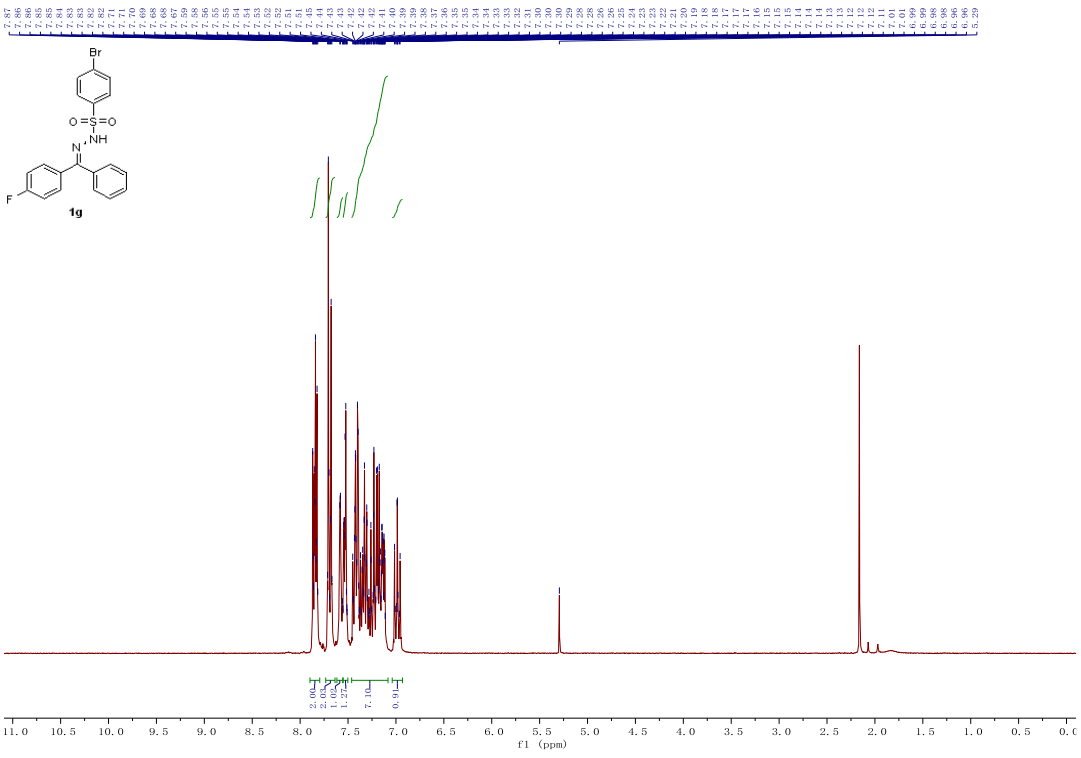
-108.68
-110.04
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

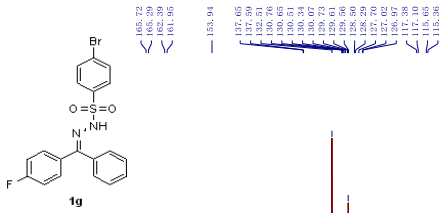

Br
O=S=O
NH
N
Cl

1f

11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)

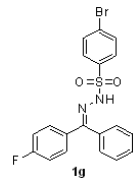

183.73
137.62
136.58
136.07
134.87
132.64
130.68
130.59
130.41
130.34
130.13
129.97
129.61
129.55
128.93
128.71
128.54
128.39
127.68
Br
O=S=O
NH
N
Cl
1f
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.87 7.86 7.86 7.85 7.85 7.84 7.83 7.83 7.82 7.82 7.71 7.71 7.70 7.69 7.68 7.68 7.67 7.59 7.58 7.56 7.55 7.55 7.54 7.54 7.53 7.52 7.52 7.51 7.51 7.45 7.44 7.43 7.43 7.42 7.42 7.42 7.41 7.40 7.39 7.39 7.38 7.37 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.32 7.31 7.31 7.30 7.30 7.29 7.28 7.28 7.26 7.25 7.24 7.23 7.23 7.22 7.21 7.20 7.19 7.18 7.18 7.17 7.17 7.16 7.15 7.15 7.15 7.14 7.14 7.14 7.13 7.13 7.12 7.12 7.12 7.11 7.01 7.01 6.99 6.99 6.98 6.98 6.96 6.96 6.29
Br
O=S=O
NH
N
F
1g
2.10 2.03 1.07 1.12 7.10 0.91
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Br
O=S=O
NH
N
F

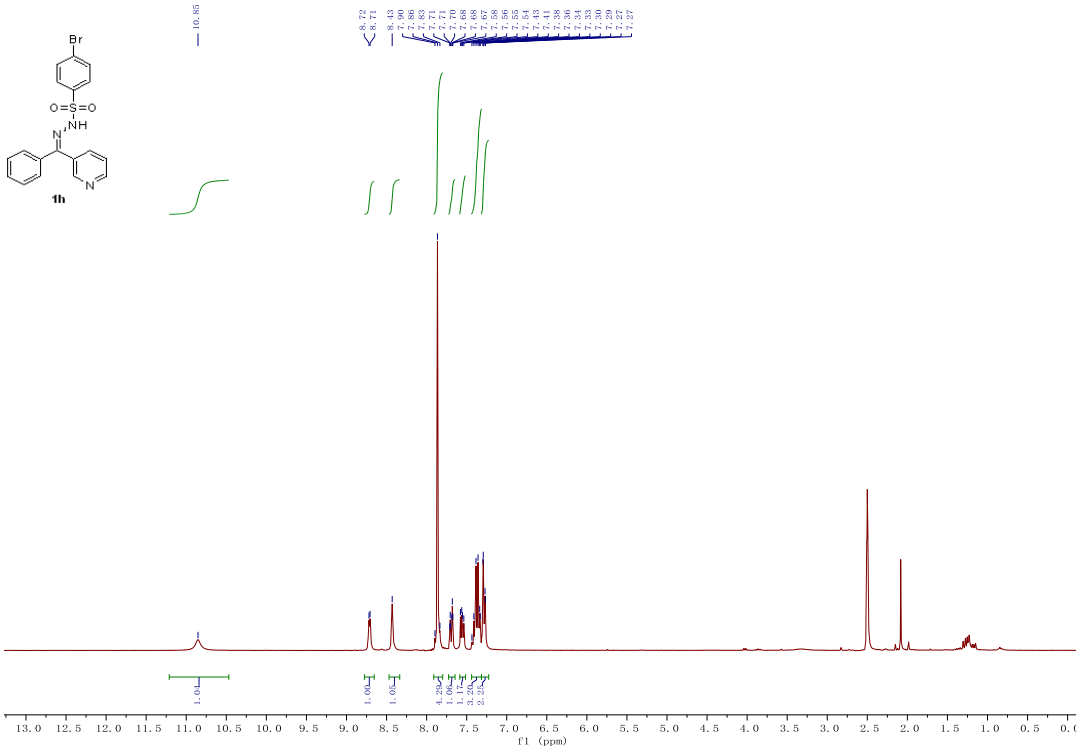
1g

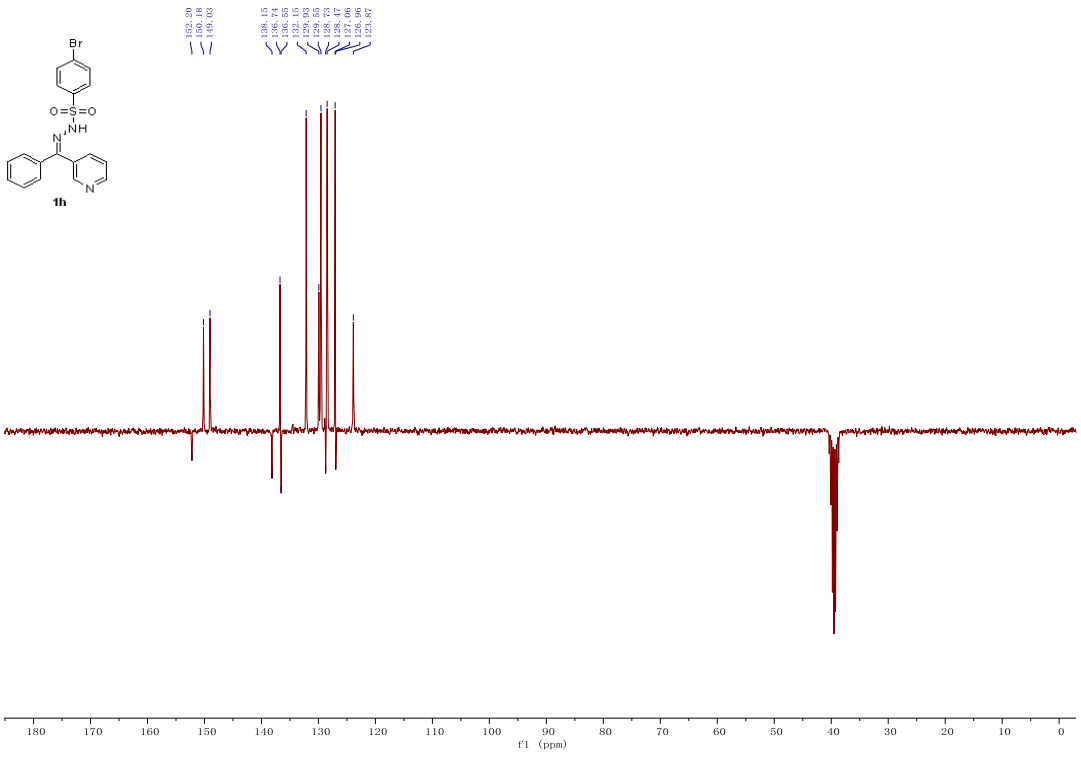
165.72
165.39
162.39
161.95
153.94
137.65
137.59
132.51
130.76
130.65
130.51
130.34
130.07
129.73
129.61
129.56
128.50
128.29
127.70
127.02
126.97
117.38
117.10
115.65
115.36
f1 (ppm)

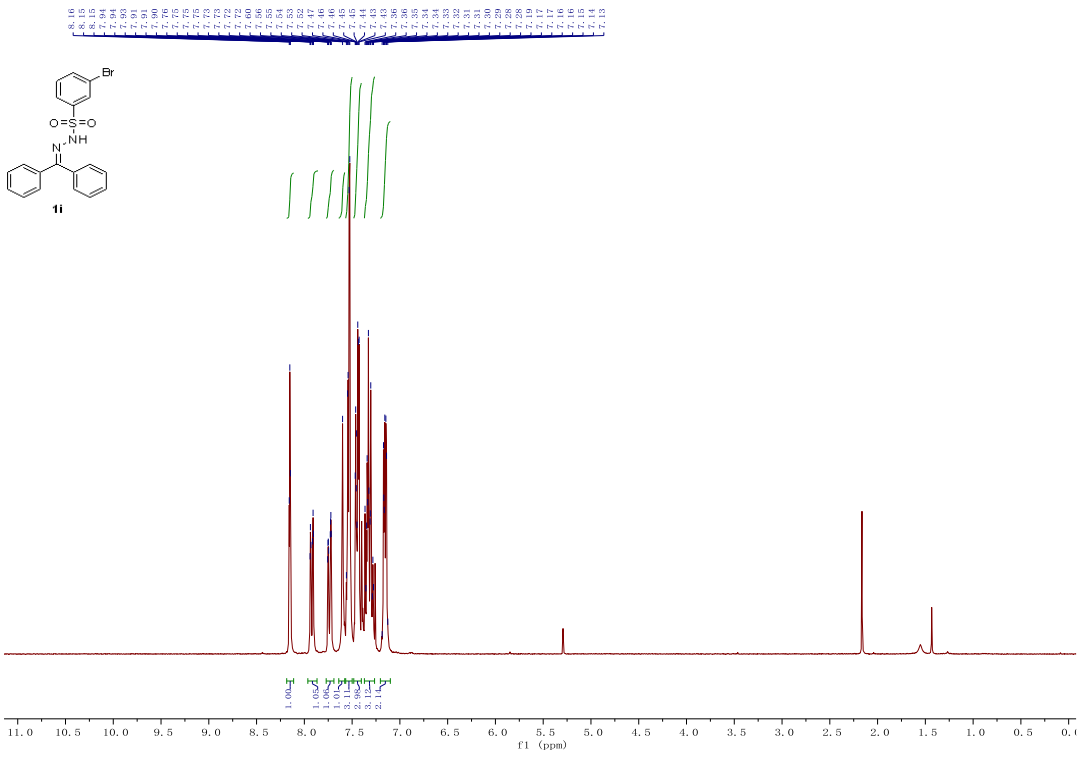

Br
O=S=O
NH
N
F

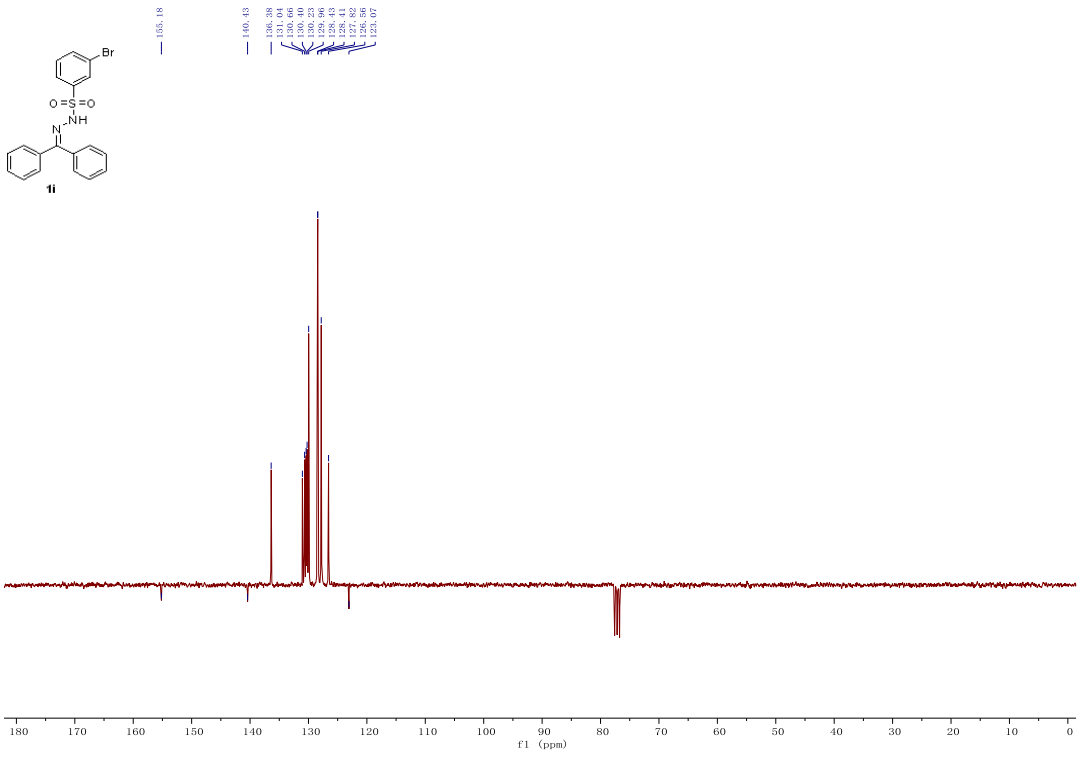
1g

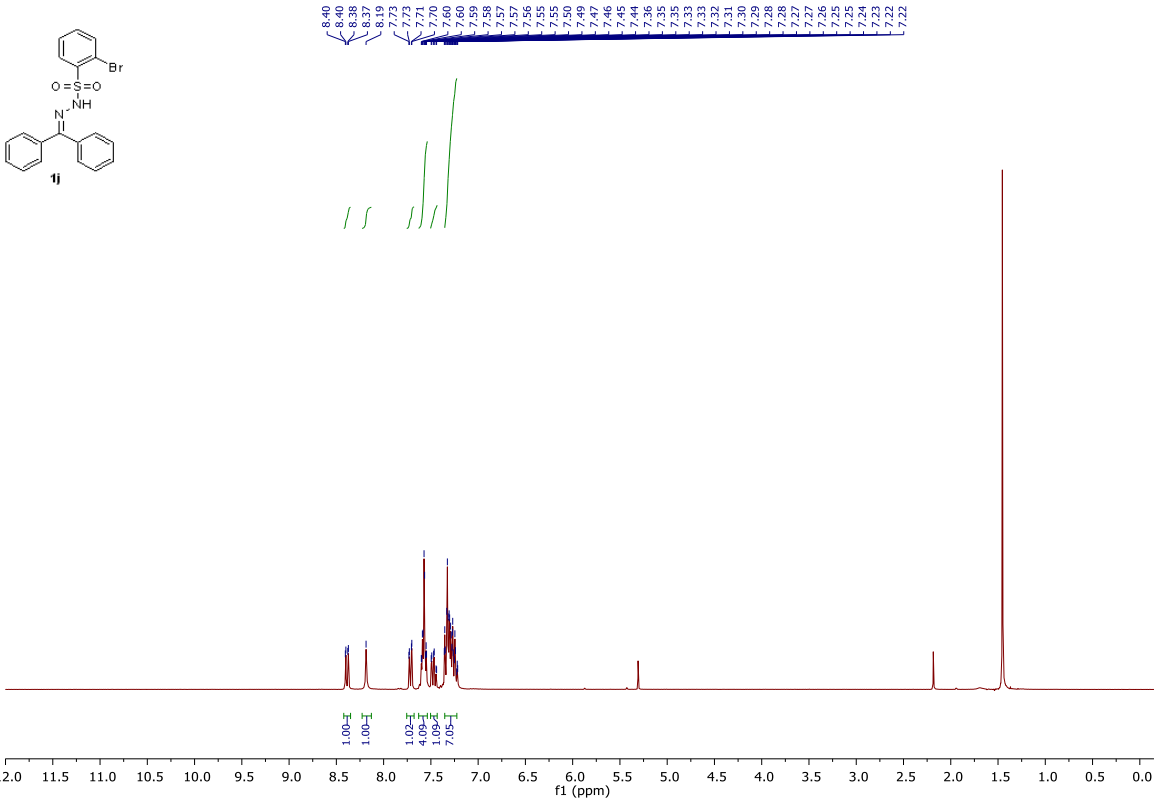
-109.05
-110.32
f1 (ppm)

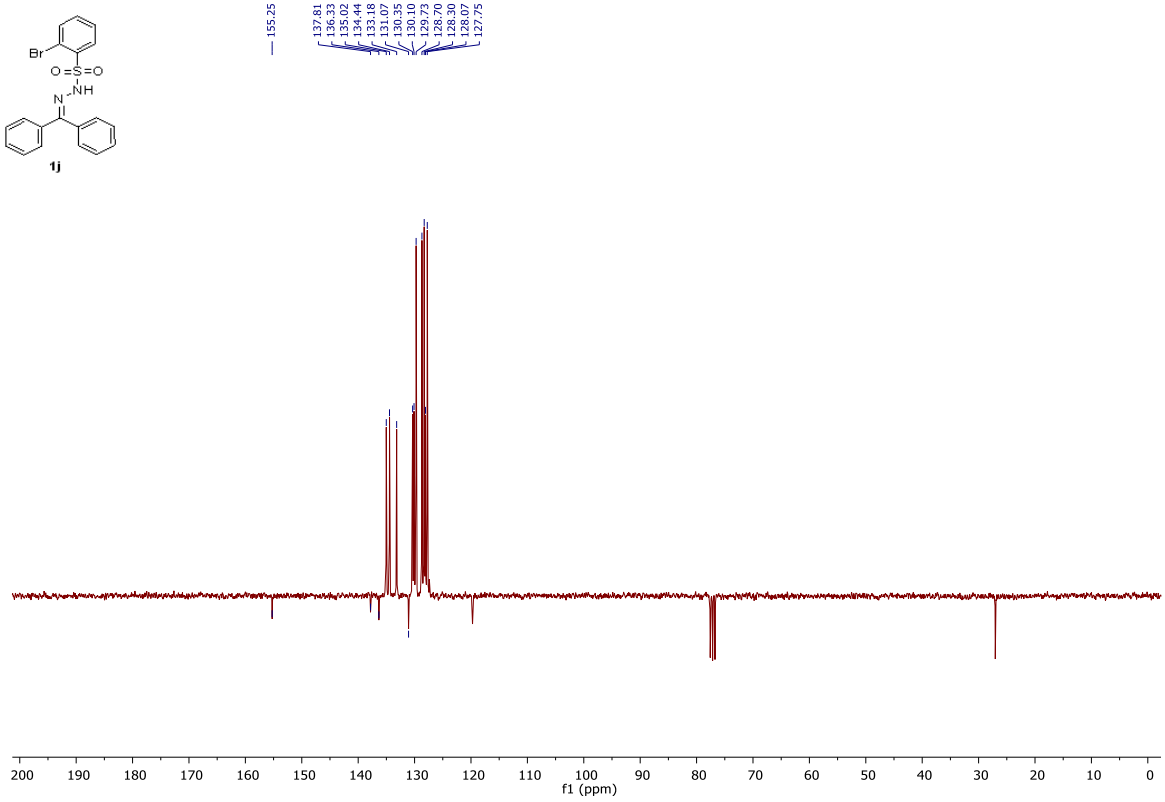

Br
O=S=O
NH
N
N
1h
10.85
8.72
8.71
8.43
8.40
7.86
7.83
7.71
7.71
7.70
7.68
7.66
7.65
7.58
7.56
7.55
7.54
7.43
7.41
7.38
7.36
7.34
7.33
7.30
7.29
7.27
7.27
1.04
1.00
1.05
4.29
1.06
1.17
3.12
2.35
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

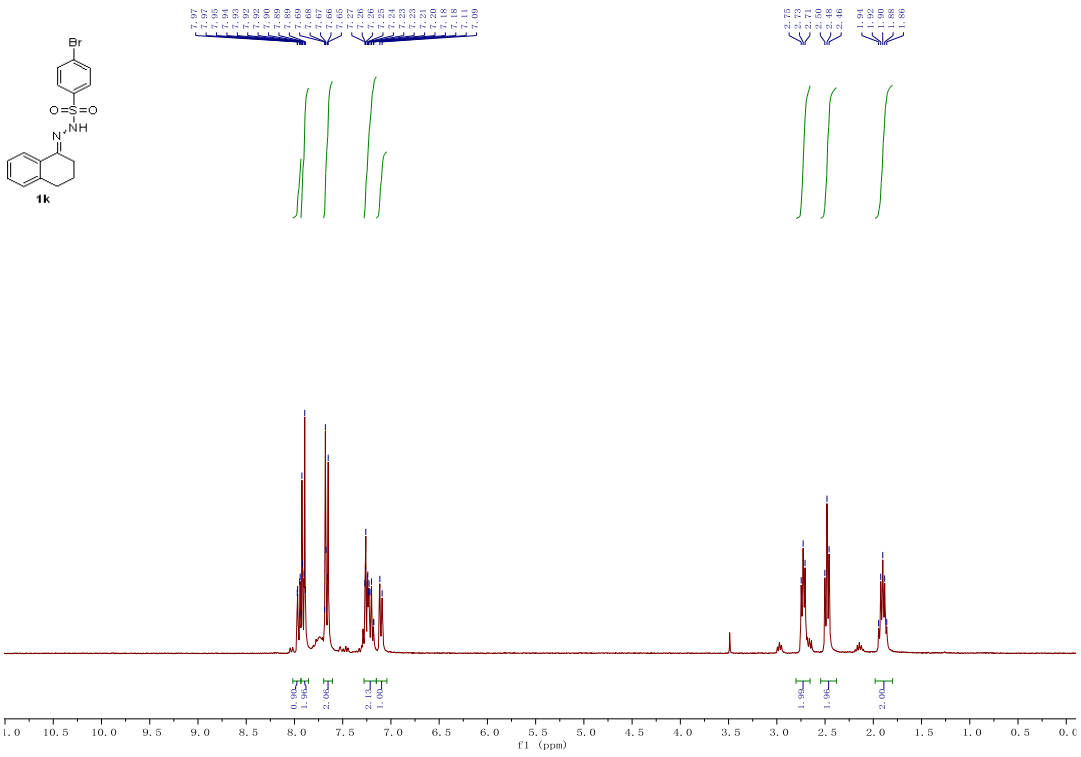

Br
O=S=O
NH
N
N
1h
152.20
150.18
149.03
138.15
136.74
136.55
132.15
129.93
129.55
128.73
128.47
127.06
126.96
123.87
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

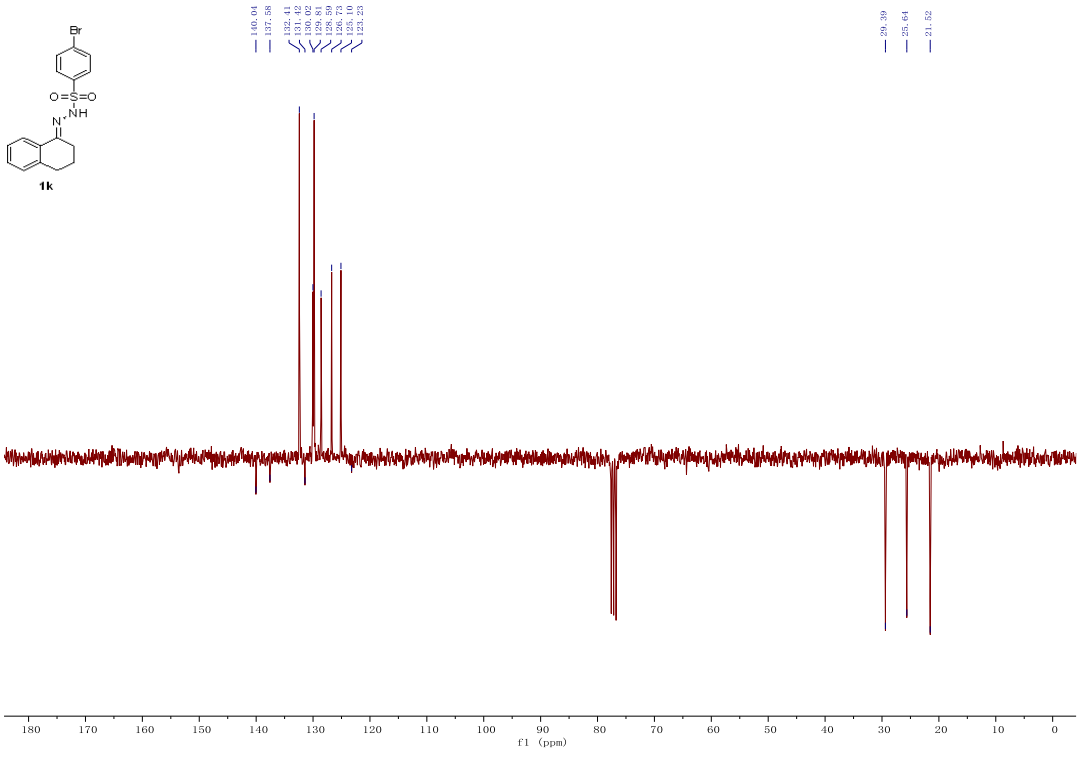

8.16
8.15
8.15
7.94
7.94
7.93
7.93
7.91
7.91
7.90
7.76
7.75
7.75
7.75
7.73
7.73
7.72
7.72
7.66
7.66
7.55
7.54
7.53
7.52
7.47
7.46
7.46
7.45
7.45
7.44
7.43
7.43
7.36
7.36
7.35
7.34
7.34
7.33
7.32
7.31
7.31
7.30
7.29
7.28
7.28
7.19
7.17
7.17
7.16
7.15
7.15
7.14
7.13
Br
O=S=O
NH
N
1i
1.00
1.05
1.06
1.01
1.11
2.98
3.12
2.14
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

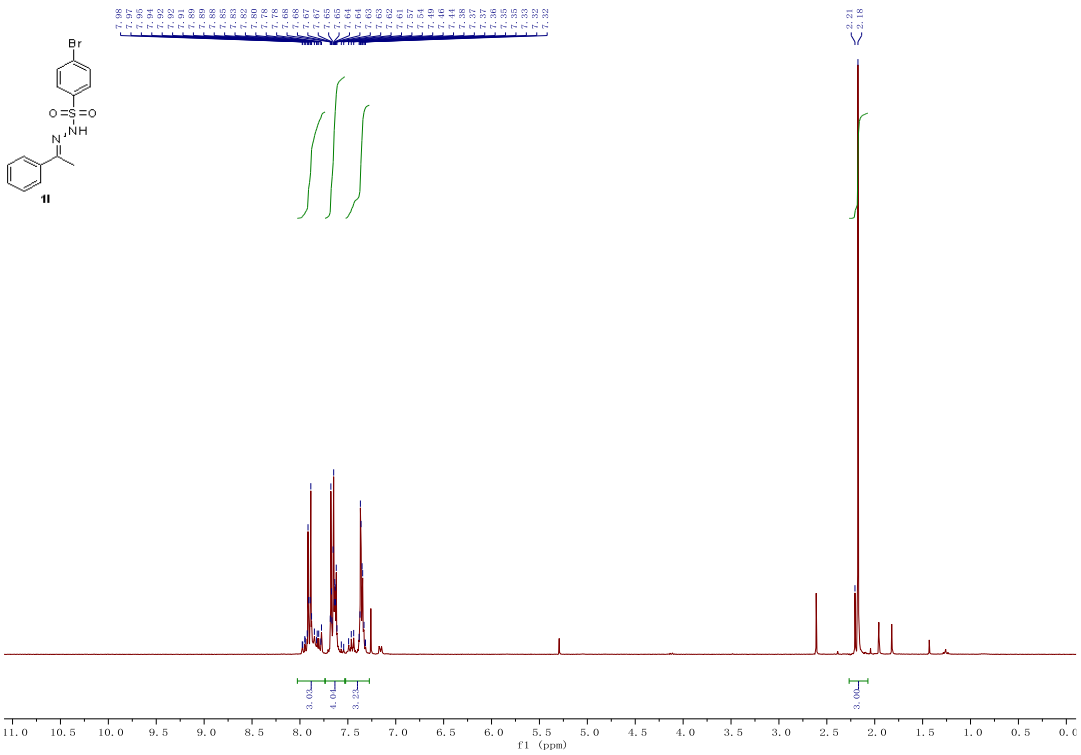

155.18
140.43
136.38
131.04
130.66
130.40
130.23
129.96
129.45
128.41
127.82
126.56
125.07
Br
O=S=O
NH
N
1i
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

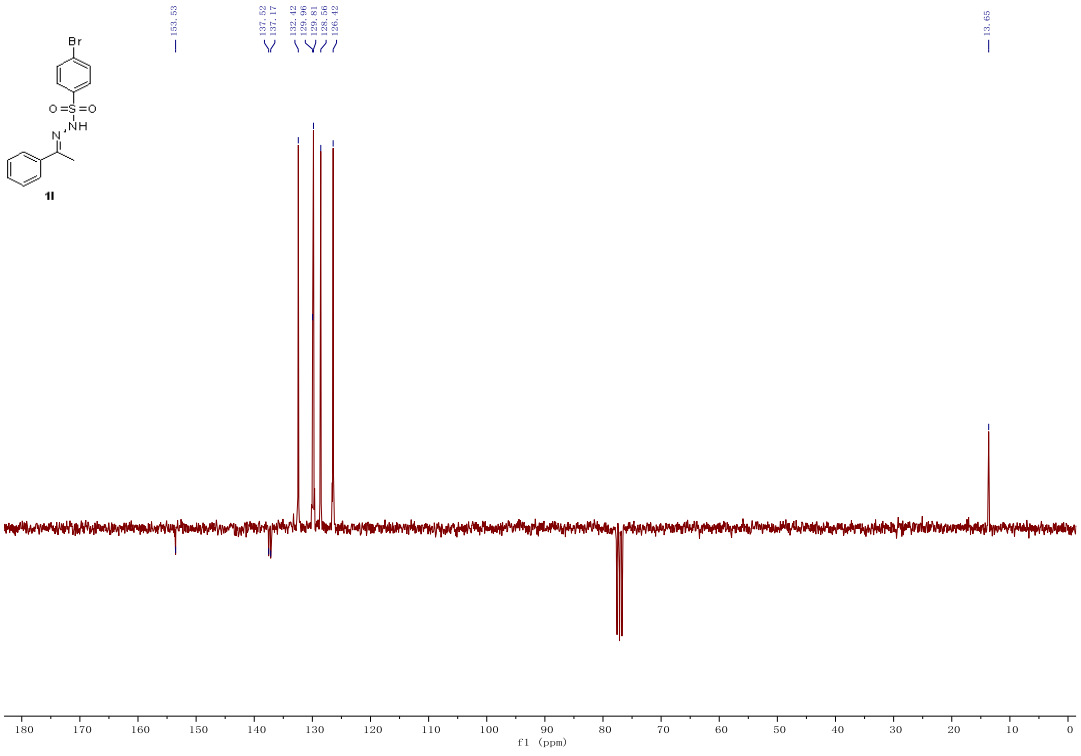

1j
8.40
8.40
8.38
8.37
8.19
7.73
7.71
7.70
7.60
7.60
7.59
7.58
7.57
7.57
7.56
7.55
7.55
7.50
7.49
7.47
7.46
7.45
7.44
7.36
7.35
7.35
7.33
7.33
7.32
7.31
7.30
7.29
7.28
7.28
7.27
7.27
7.26
7.25
7.25
7.24
7.23
7.22
7.22
1.00
1.00
1.02
4.09
1.09
7.05
f1 (ppm)

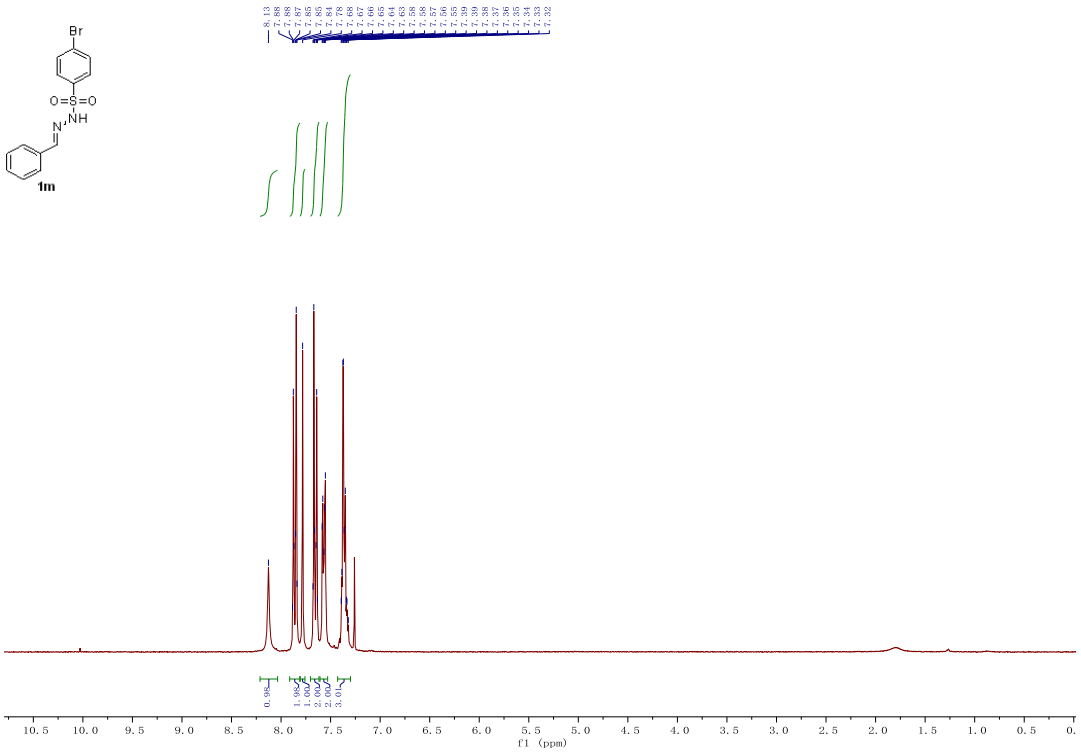

1j
155.25
137.81
136.33
135.02
134.44
133.18
131.07
130.35
130.10
129.73
128.70
128.30
128.07
127.75
f1 (ppm)

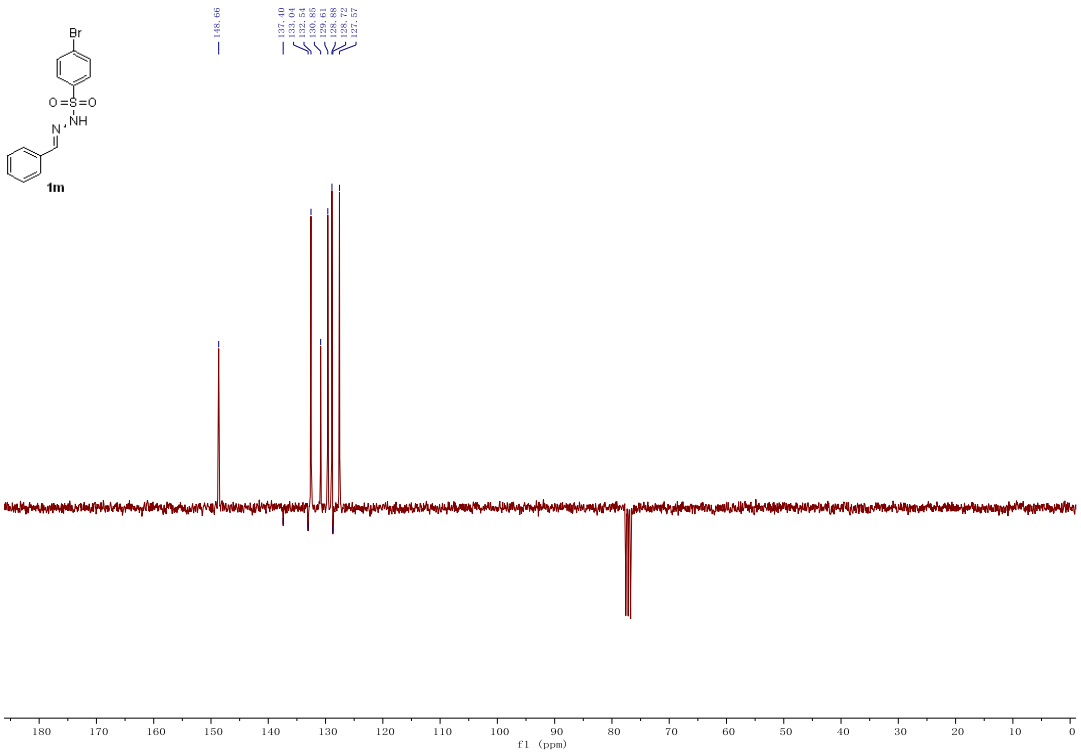

Br
O=S=O
NH
N
1k
7.97
7.97
7.95
7.94
7.93
7.92
7.92
7.90
7.89
7.89
7.69
7.68
7.67
7.66
7.65
7.27
7.26
7.25
7.24
7.23
7.23
7.21
7.20
7.18
7.18
7.11
7.09
2.75
2.73
2.71
2.50
2.48
2.46
1.94
1.92
1.90
1.88
1.86
0.98
1.96
2.06
2.13
1.00
1.99
1.98
2.00
f1 (ppm)

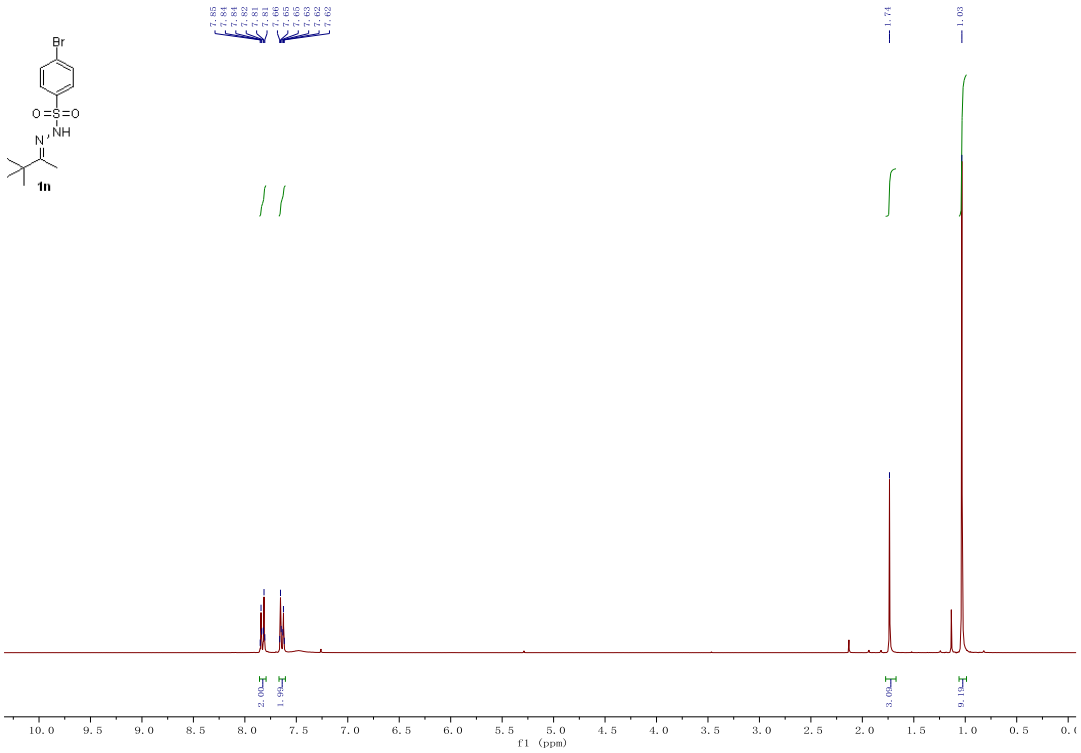

Br
O=S=O
NH
N
1k
140.04
137.58
132.41
131.42
130.02
129.94
128.59
126.73
125.10
125.25
29.39
25.64
21.52
f1 (ppm)

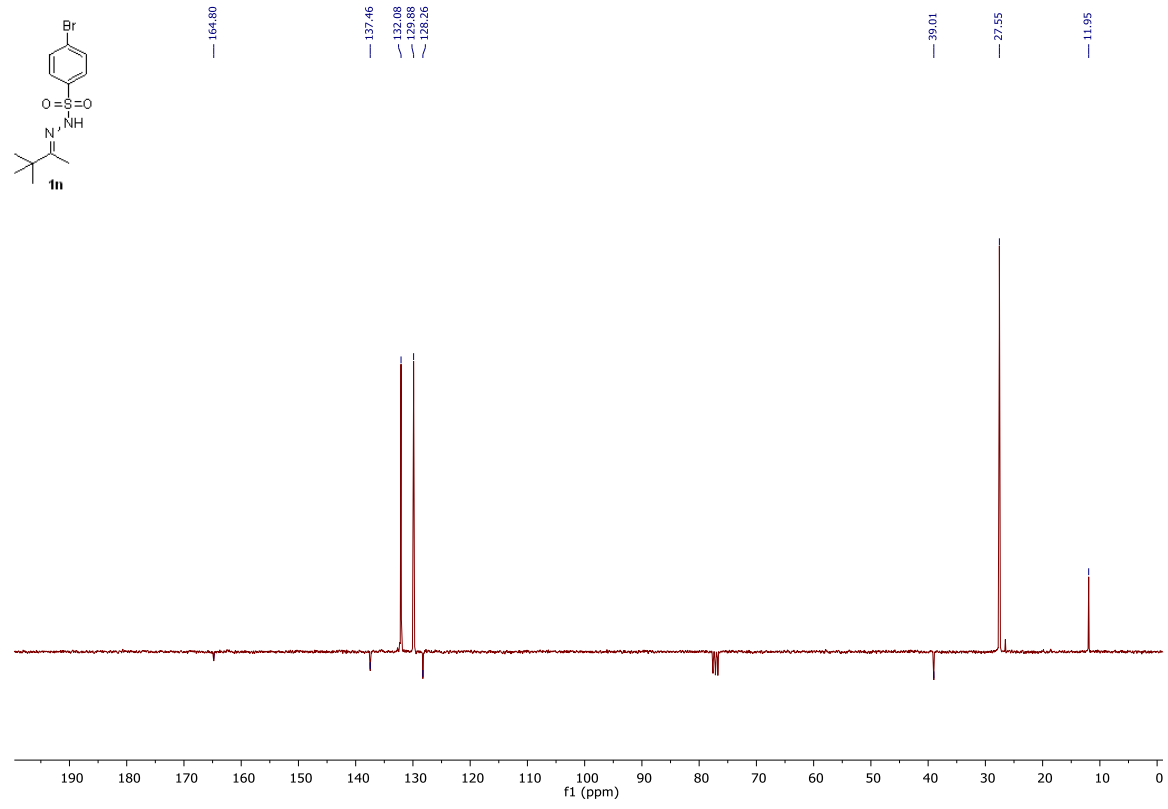

7.98
7.97
7.95
7.94
7.92
7.92
7.91
7.89
7.88
7.85
7.83
7.82
7.80
7.78
7.78
7.68
7.67
7.67
7.66
7.65
7.64
7.64
7.63
7.62
7.61
7.57
7.54
7.49
7.46
7.44
7.38
7.37
7.37
7.36
7.35
7.35
7.33
7.32
7.32
2.21
2.18
Br
O=S=O
NH
N
1l
3.03
4.04
3.23
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

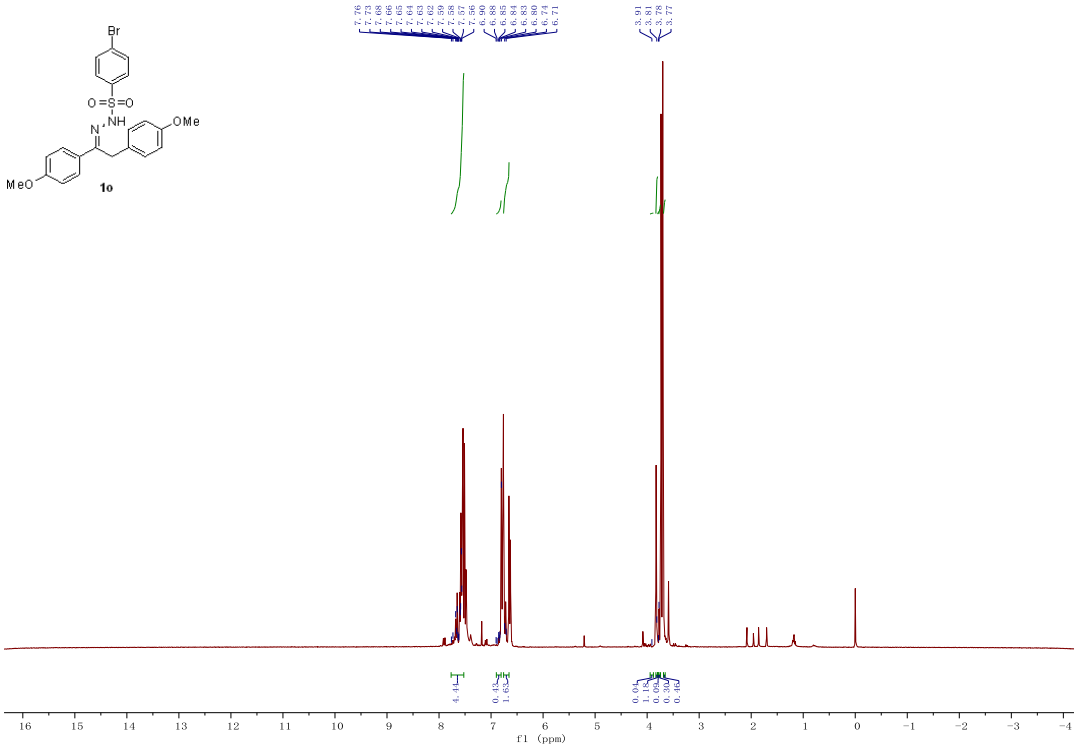

153.53
137.52
137.17
132.42
129.96
129.81
128.56
126.42
13.65
Br
O=S=O
NH
N
1l
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

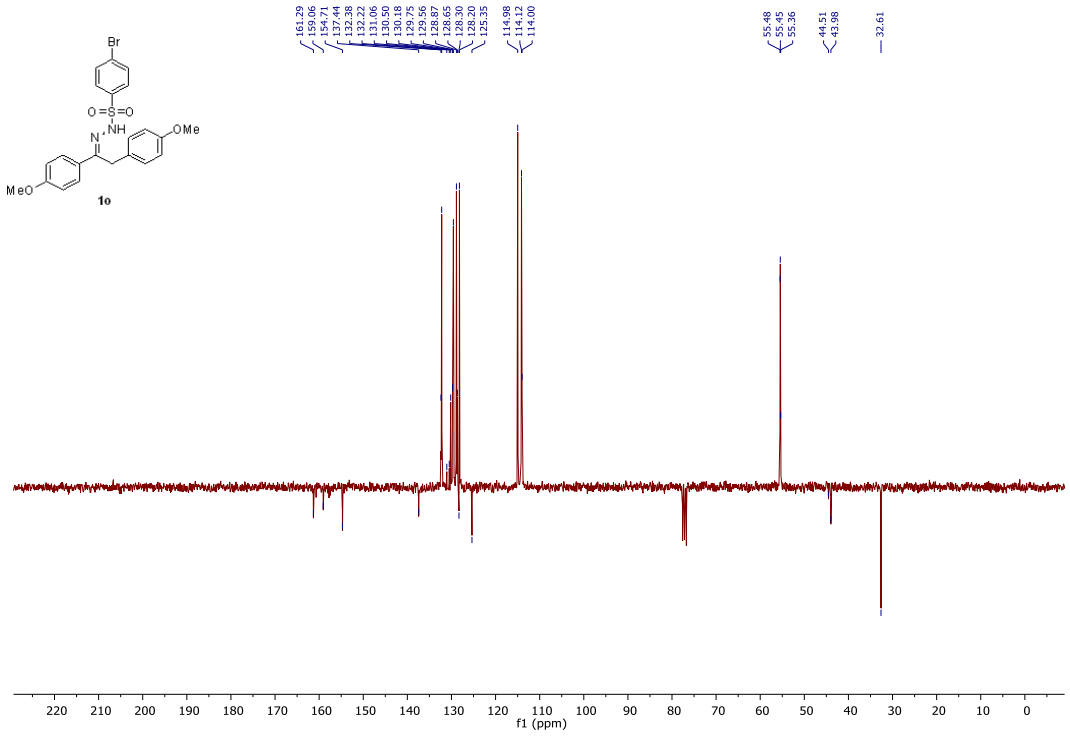

Br
O=S=O
NH
N
1m
8.13
7.88
7.88
7.87
7.85
7.85
7.84
7.78
7.68
7.67
7.66
7.65
7.64
7.63
7.58
7.58
7.57
7.56
7.55
7.39
7.38
7.37
7.36
7.35
7.34
7.33
7.32
0.98
1.98
1.00
2.00
2.00
3.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)


Br
O=S=O
NH
N
1m
148.66
137.40
132.64
132.54
130.85
129.61
128.88
128.72
127.57
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

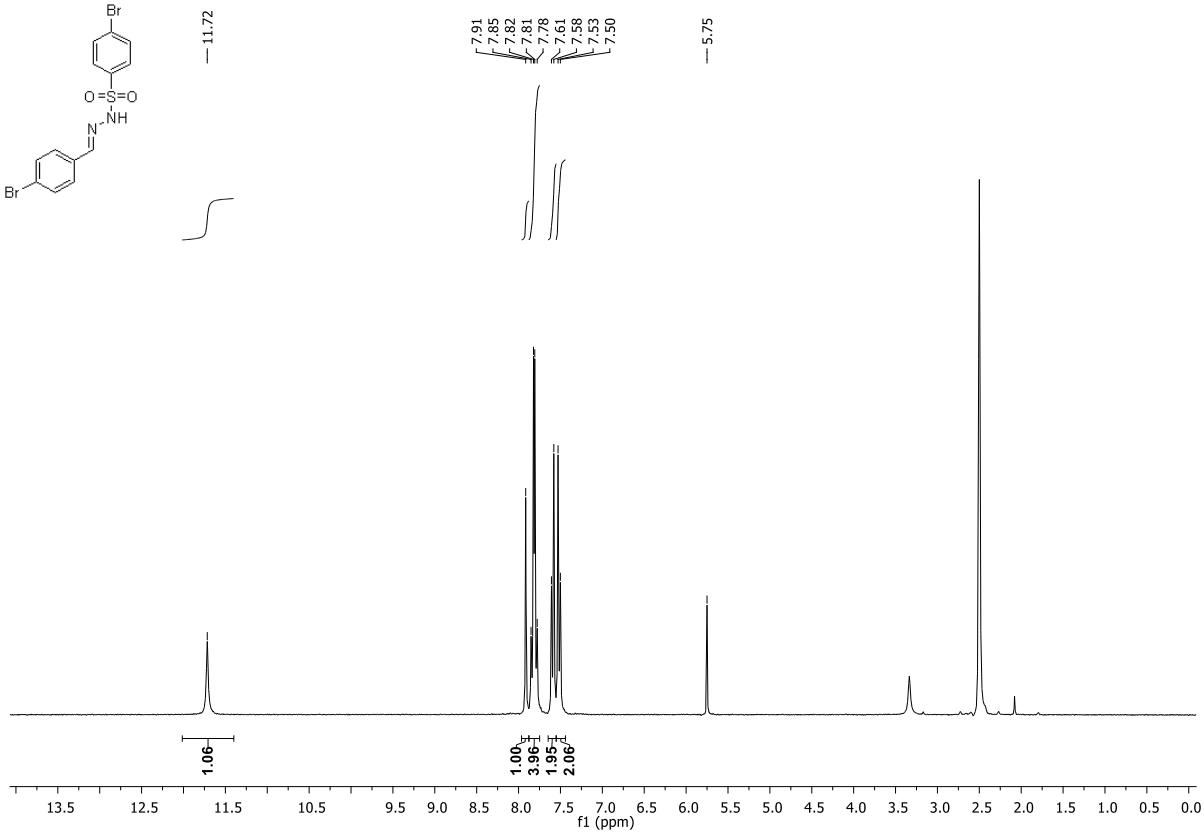

Br
O=S=O
NH
N
1n
7.85
7.84
7.84
7.82
7.81
7.81
7.66
7.65
7.63
7.62
7.62
7.62
1.74
1.03
2.00
1.99
3.09
9.13
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


Br
O=S=O
NH
N
1n
164.80
137.46
132.08
129.88
128.26
39.01
27.55
11.95
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

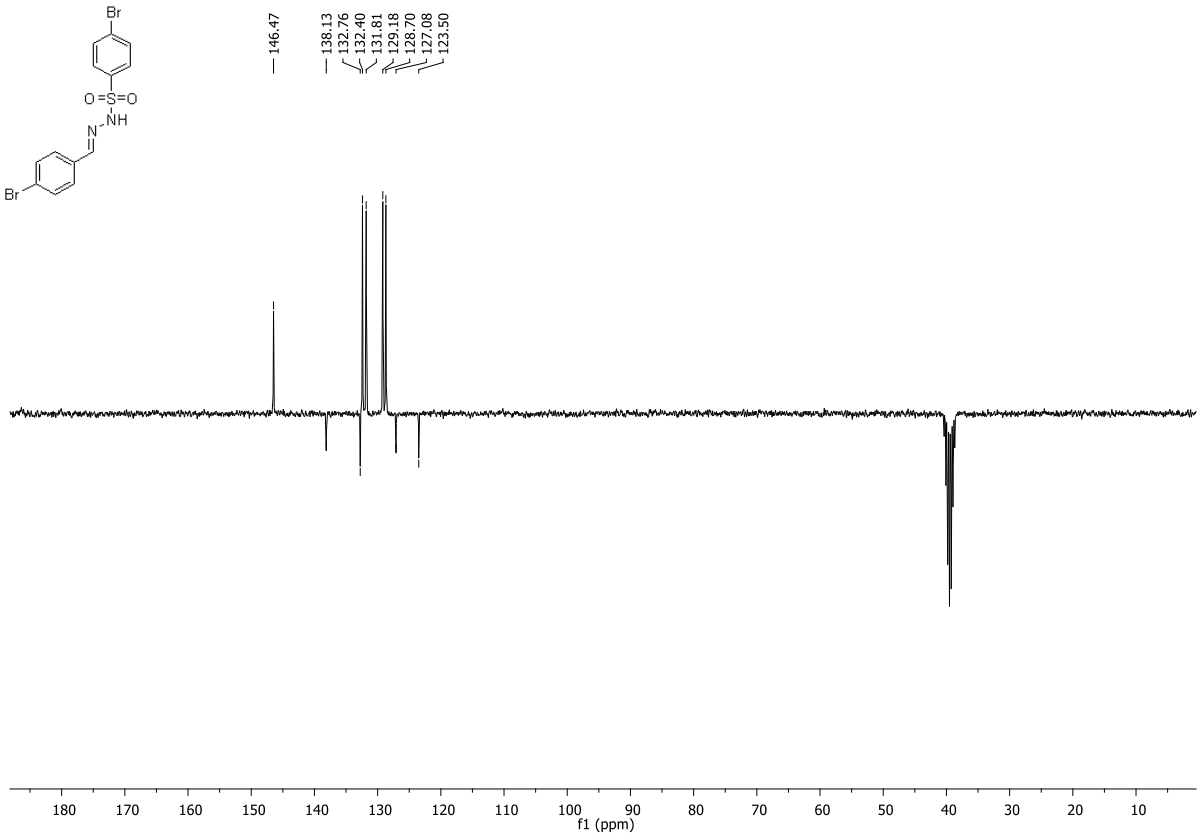

Br
O=S=O
NH
N
OMe
MeO
1o
7.76
7.73
7.68
7.66
7.65
7.64
7.63
7.62
7.59
7.58
7.57
7.56
6.90
6.88
6.85
6.84
6.83
6.80
6.74
6.71
3.91
3.81
3.78
3.77
4.44
0.43
1.65
0.04
1.18
0.69
0.50
0.46
f1 (ppm)

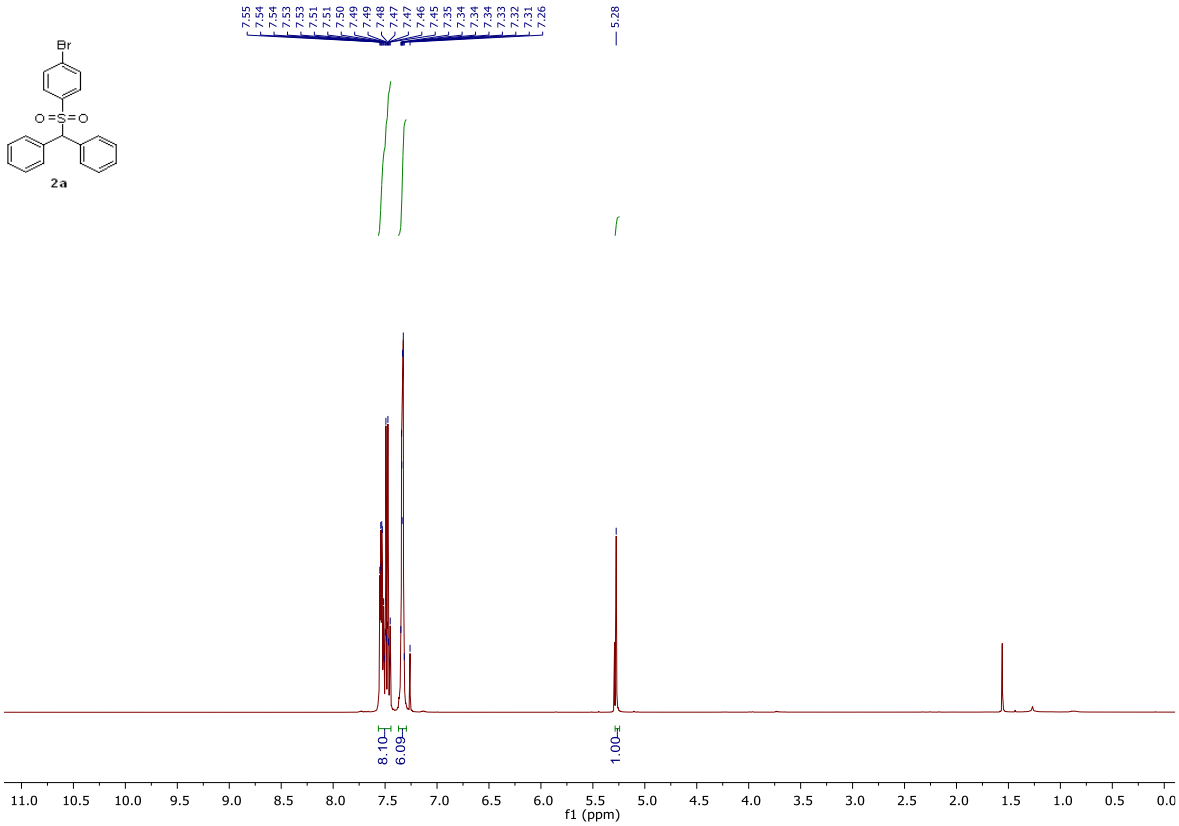

Br
O=S=O
NH
N
OMe
MeO
1o
161.29
159.06
154.71
137.44
132.38
132.22
131.06
130.50
130.18
129.75
129.56
128.87
128.65
128.30
128.20
125.35
114.98
114.12
114.00
55.48
55.45
55.36
44.51
43.98
32.61
f1 (ppm)

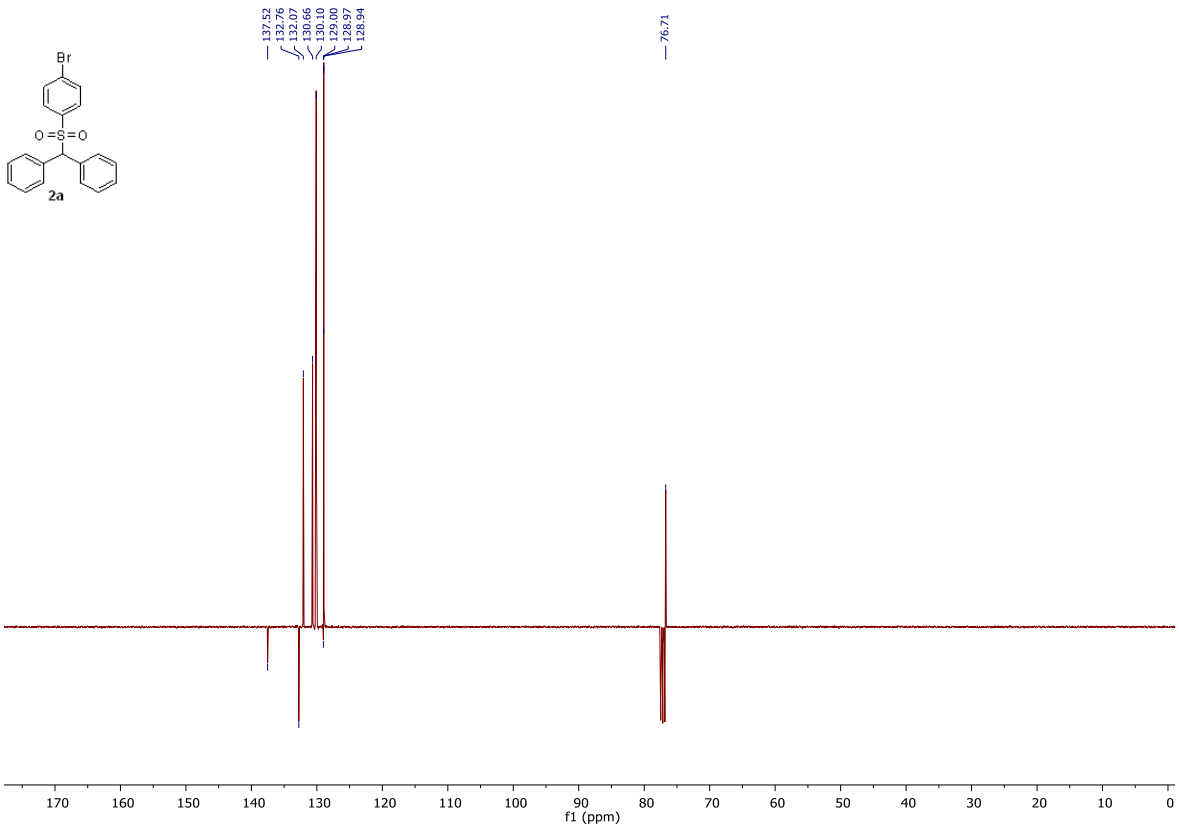
1p


Br
O=S=O
NH
N
Br
11.72
7.91
7.85
7.82
7.81
7.78
7.61
7.58
7.53
7.50
5.75
1.06
1.00
3.96
1.95
2.06
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

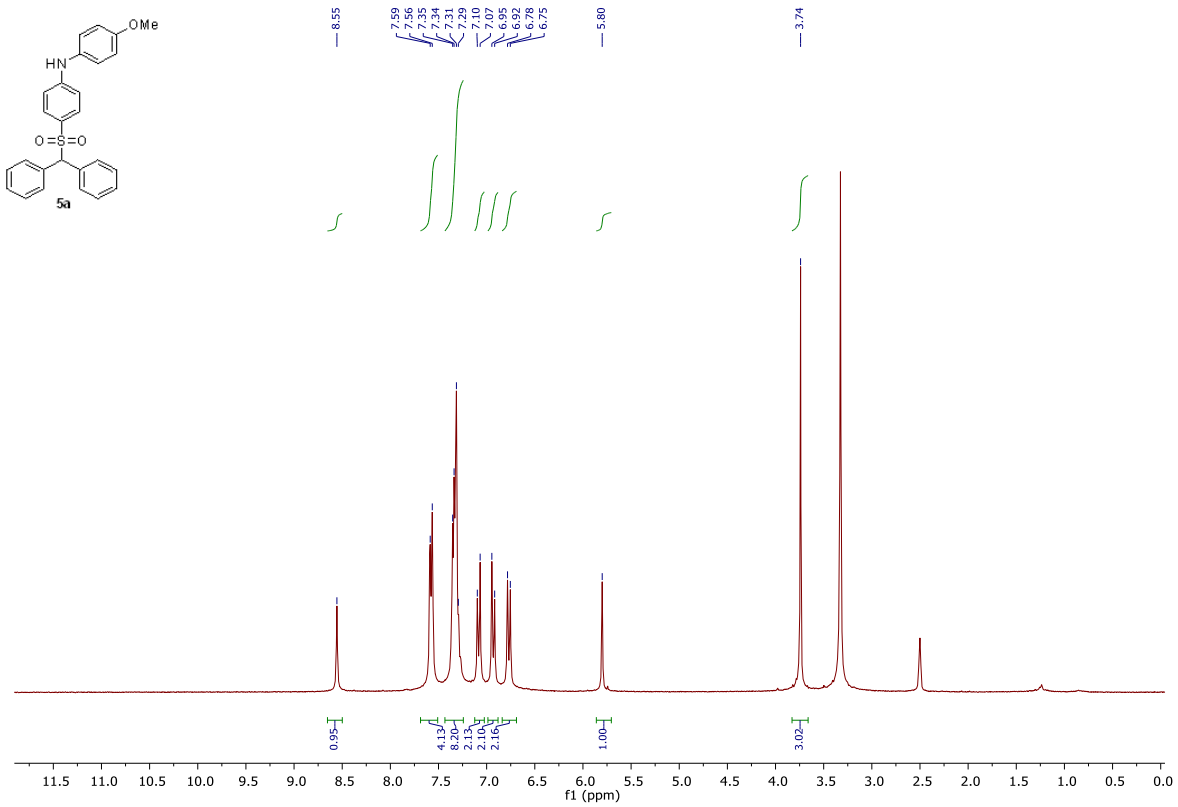
1p

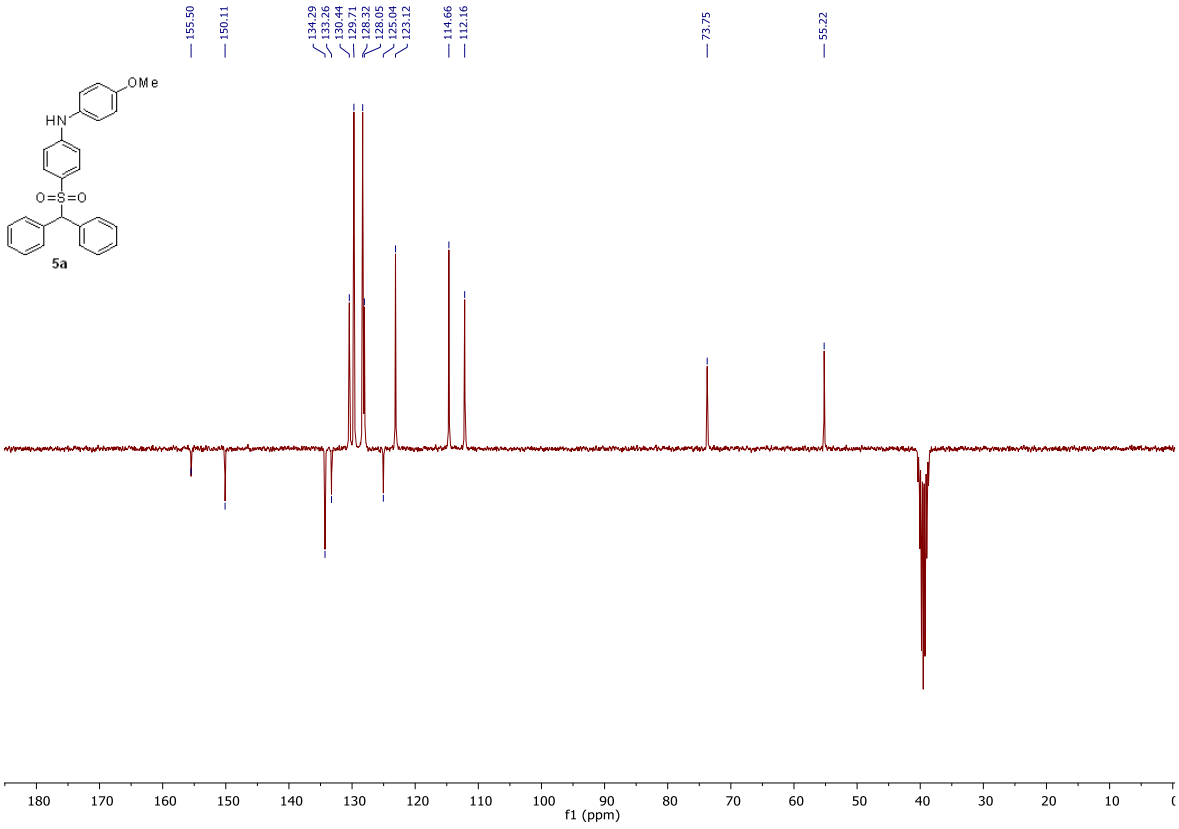

Br
O=S=O
NH
N
Br
146.47
138.13
132.76
132.40
131.81
129.18
128.70
127.08
123.50
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

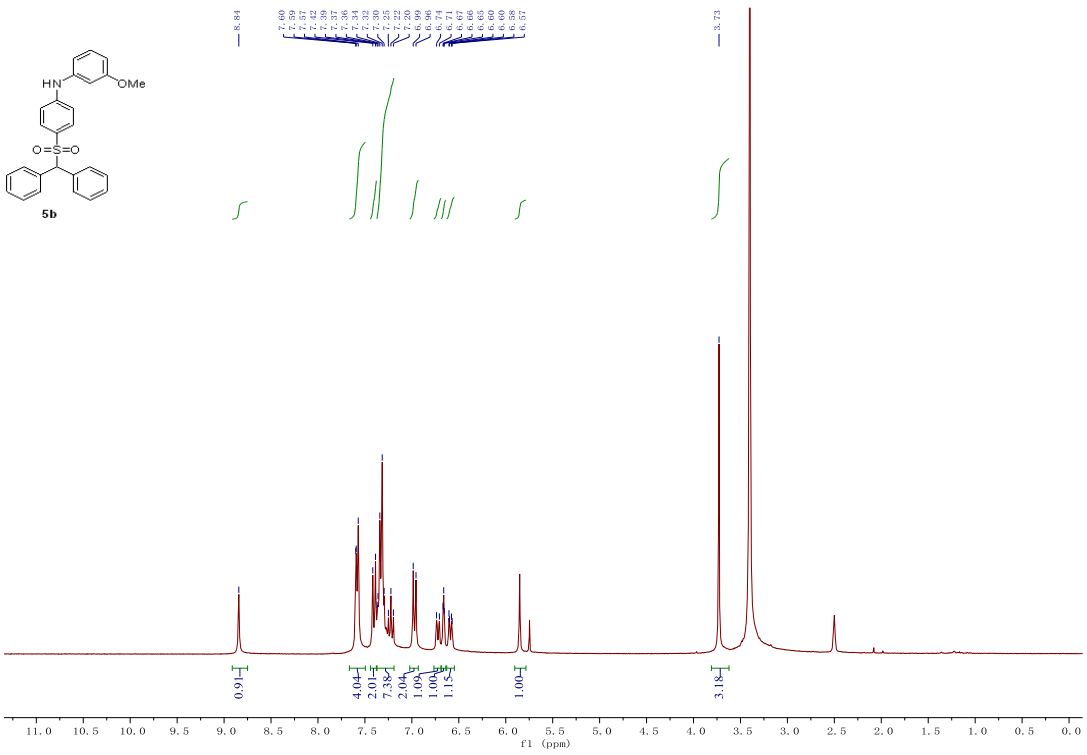

Br
O=S=O
2a
7.55
7.54
7.54
7.53
7.53
7.51
7.51
7.50
7.49
7.49
7.48
7.47
7.47
7.46
7.45
7.35
7.34
7.34
7.34
7.33
7.32
7.31
7.26
5.28
8.10
6.09
1.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

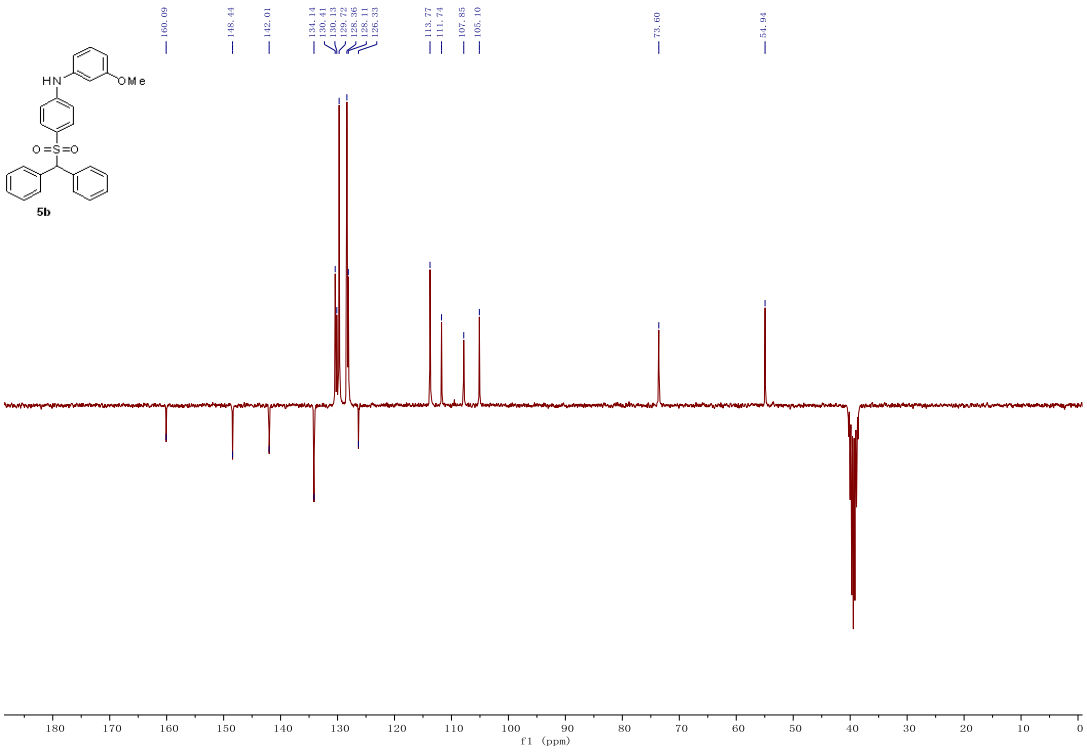

Br
O=S=O
2a
137.52
132.76
132.07
130.66
130.10
129.00
128.97
128.94
76.71
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

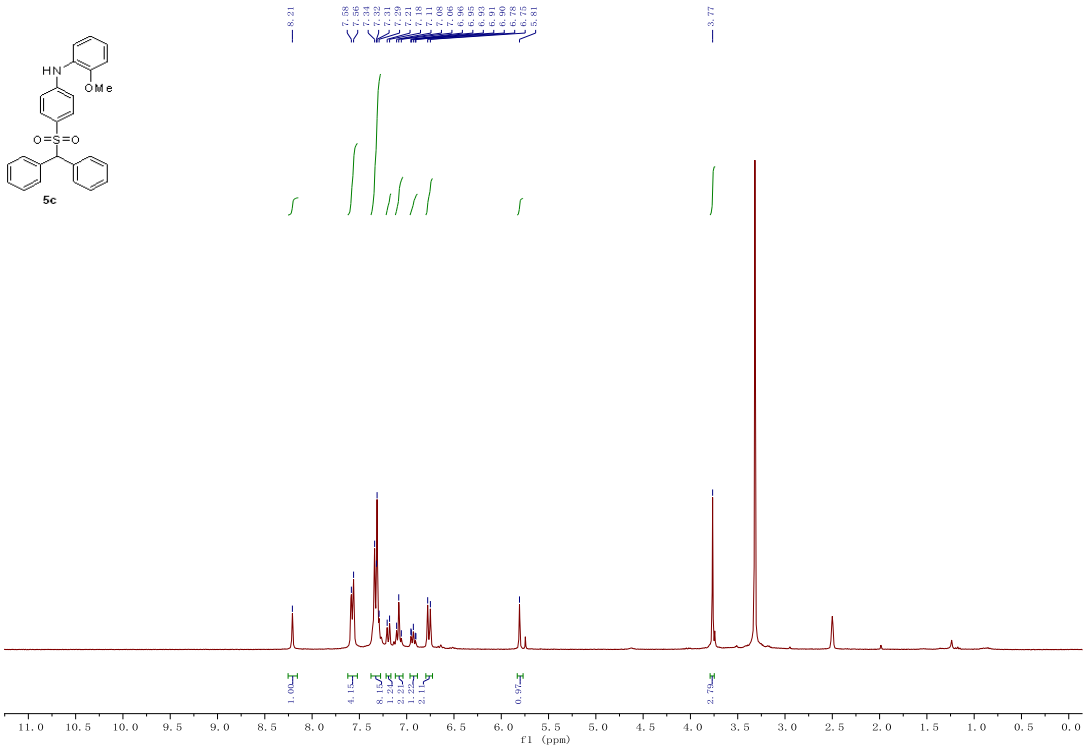
Final product

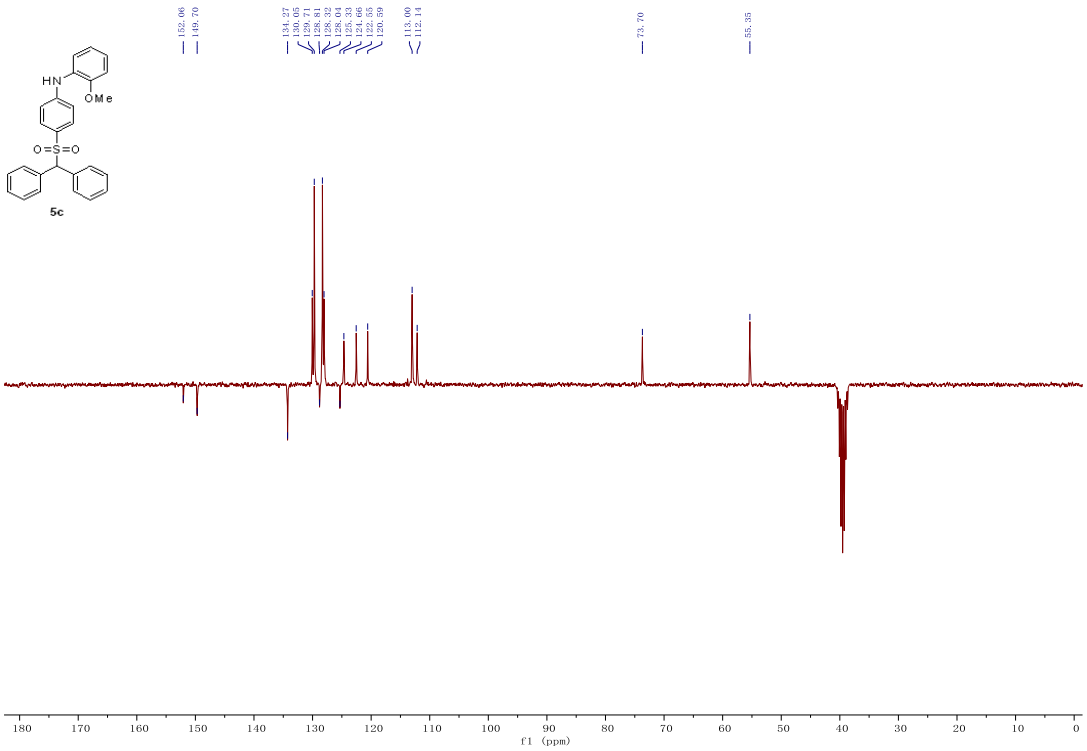


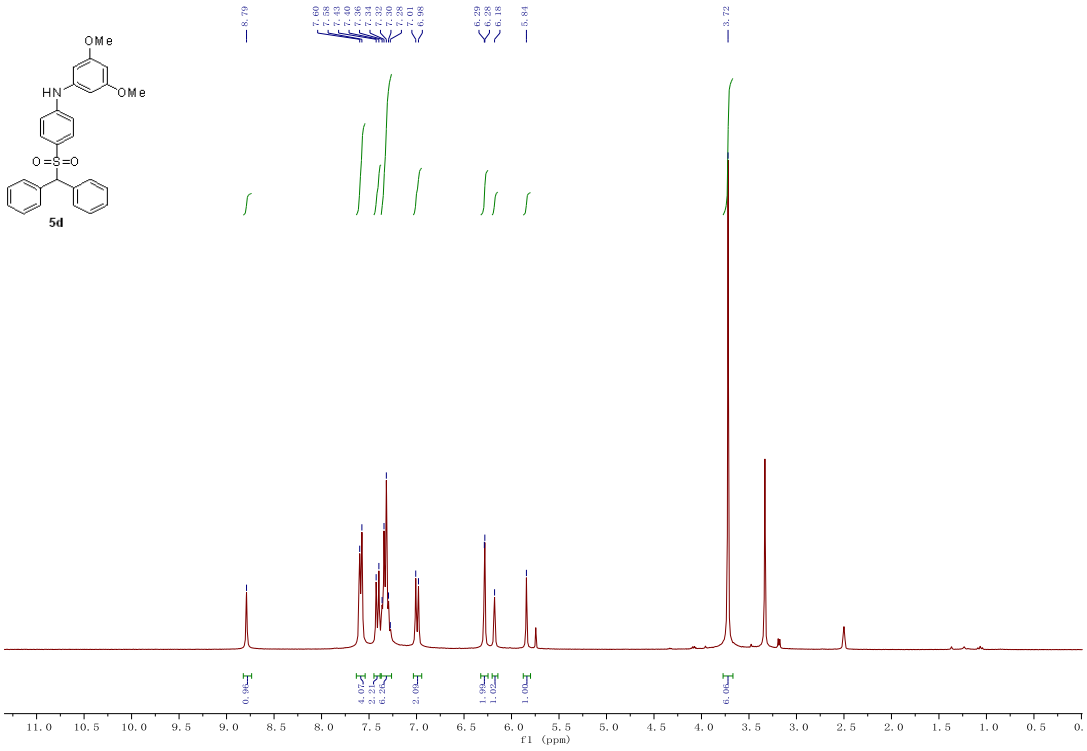


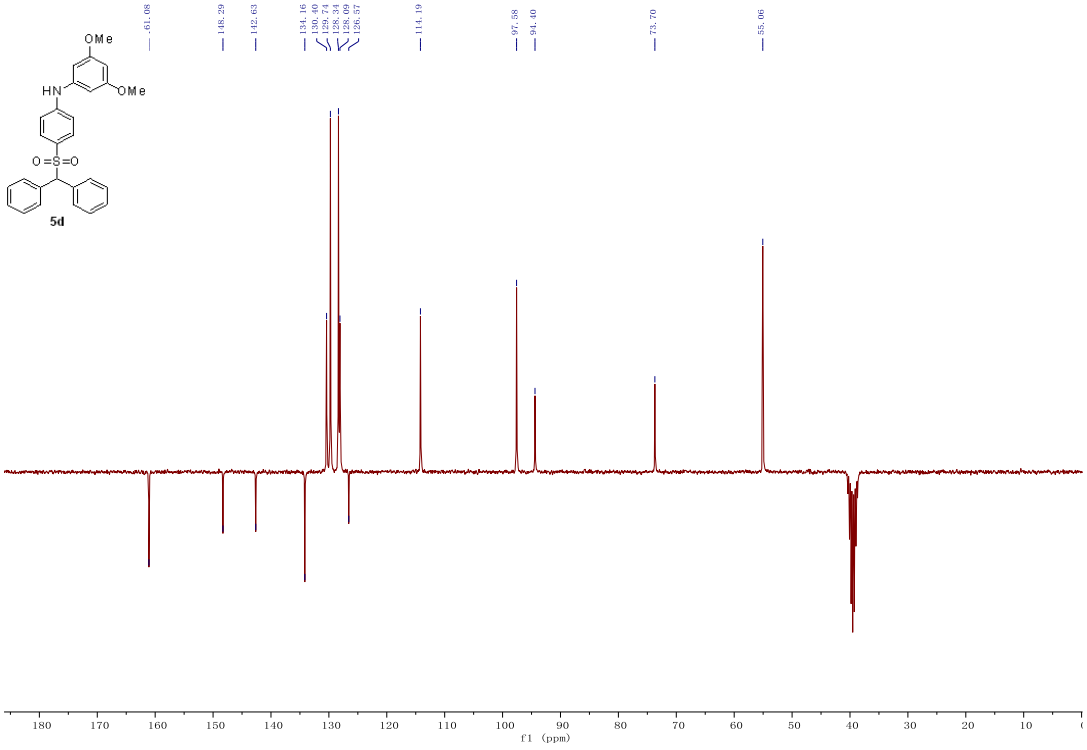

8.84
7.60
7.59
7.57
7.42
7.39
7.37
7.36
7.34
7.32
7.30
7.25
7.22
7.20
6.99
6.96
6.74
6.71
6.67
6.66
6.65
6.60
6.60
6.58
6.58
6.57
3.73
HN
OMe
O=S=O
5b
0.91
4.04
2.01
7.38
2.04
1.09
1.00
1.13
1.00
3.18
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


160.09
148.44
142.01
134.14
130.41
130.13
129.72
128.36
128.11
126.33
113.77
111.74
107.85
105.10
73.60
54.94
HN
OMe
O=S=O
5b
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

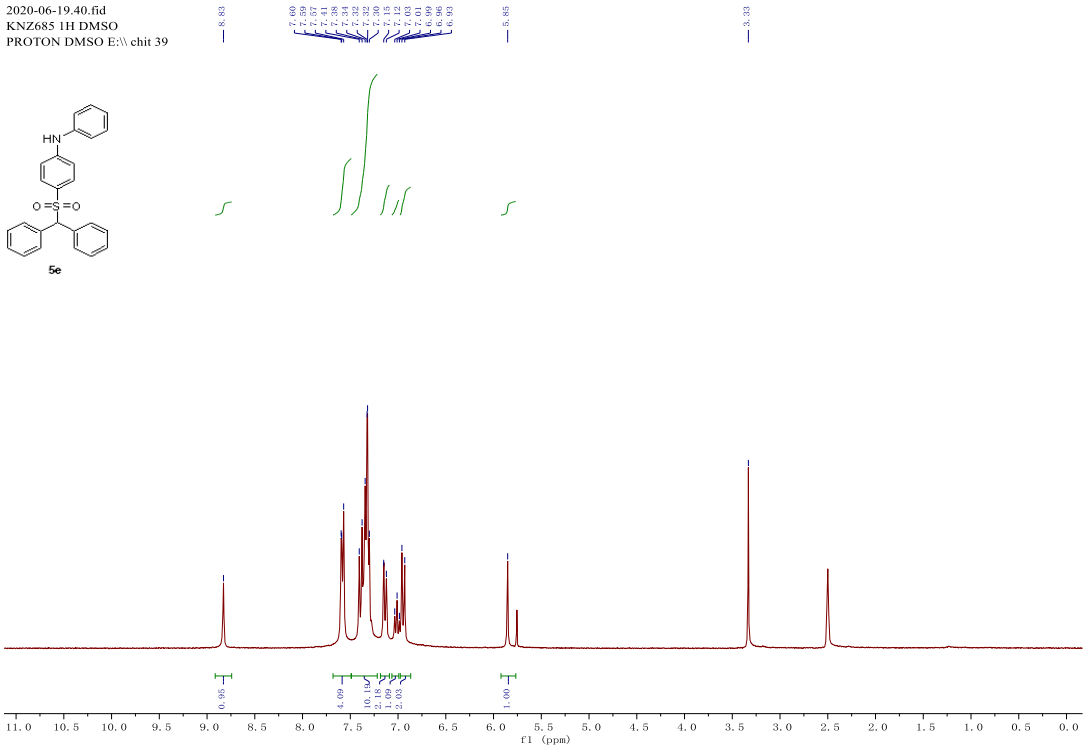

8.21
7.58
7.56
7.34
7.32
7.31
7.29
7.21
7.18
7.11
7.08
7.06
6.96
6.95
6.93
6.91
6.90
6.38
6.75
5.81
3.77
HN
OMe
O=S=O
5c
1.00
4.15
8.18
1.24
2.24
1.22
2.11
0.97
2.79
f1 (ppm)

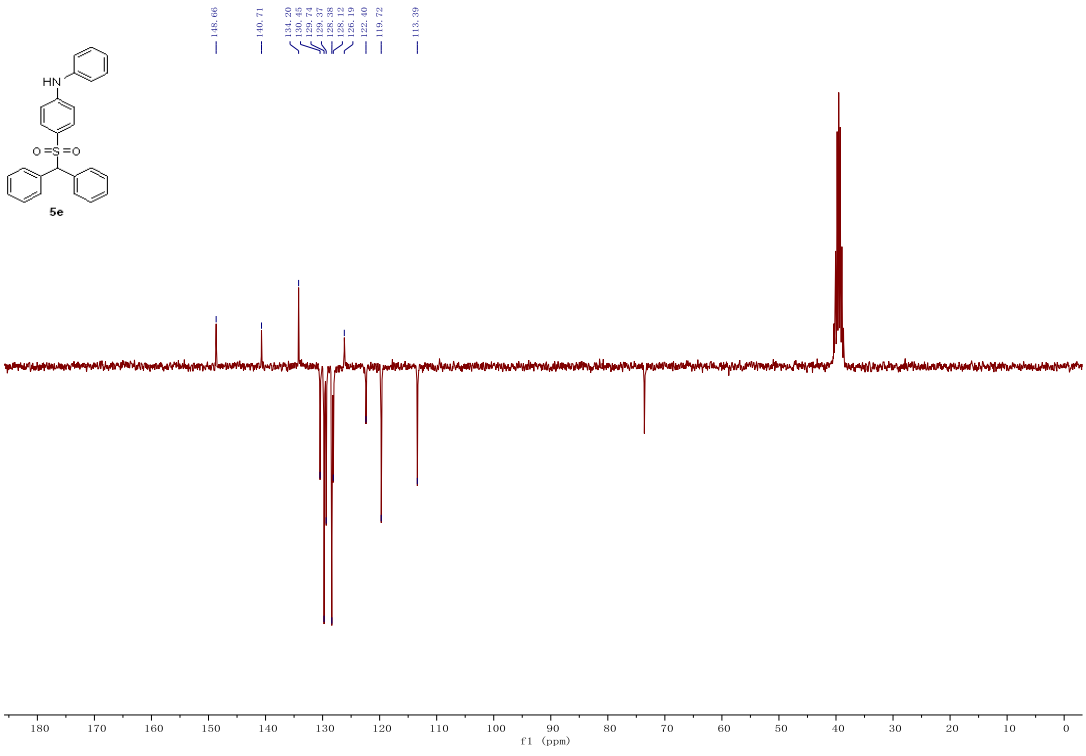

152.06
149.70
134.27
130.05
129.71
128.81
128.32
128.04
125.33
124.66
122.55
120.59
113.09
112.14
73.70
55.35
HN
OMe
O=S=O
5c
f1 (ppm)

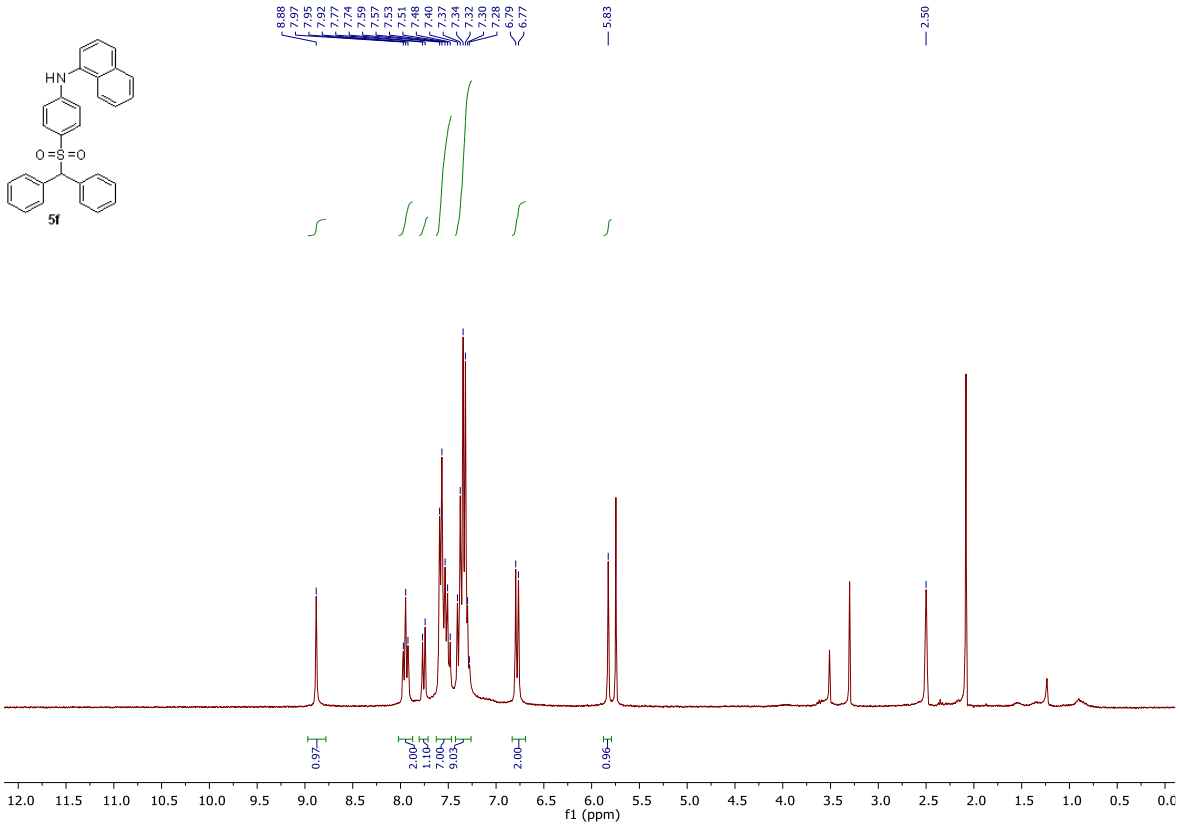

OMe
HN
OMe
O=S=O
5d
8.79
7.60
7.58
7.43
7.40
7.36
7.34
7.32
7.30
7.28
7.01
6.98
6.29
6.28
6.18
5.84
3.72
0.90
4.07
2.21
6.26
2.09
1.99
1.02
1.00
6.06
f1 (ppm)

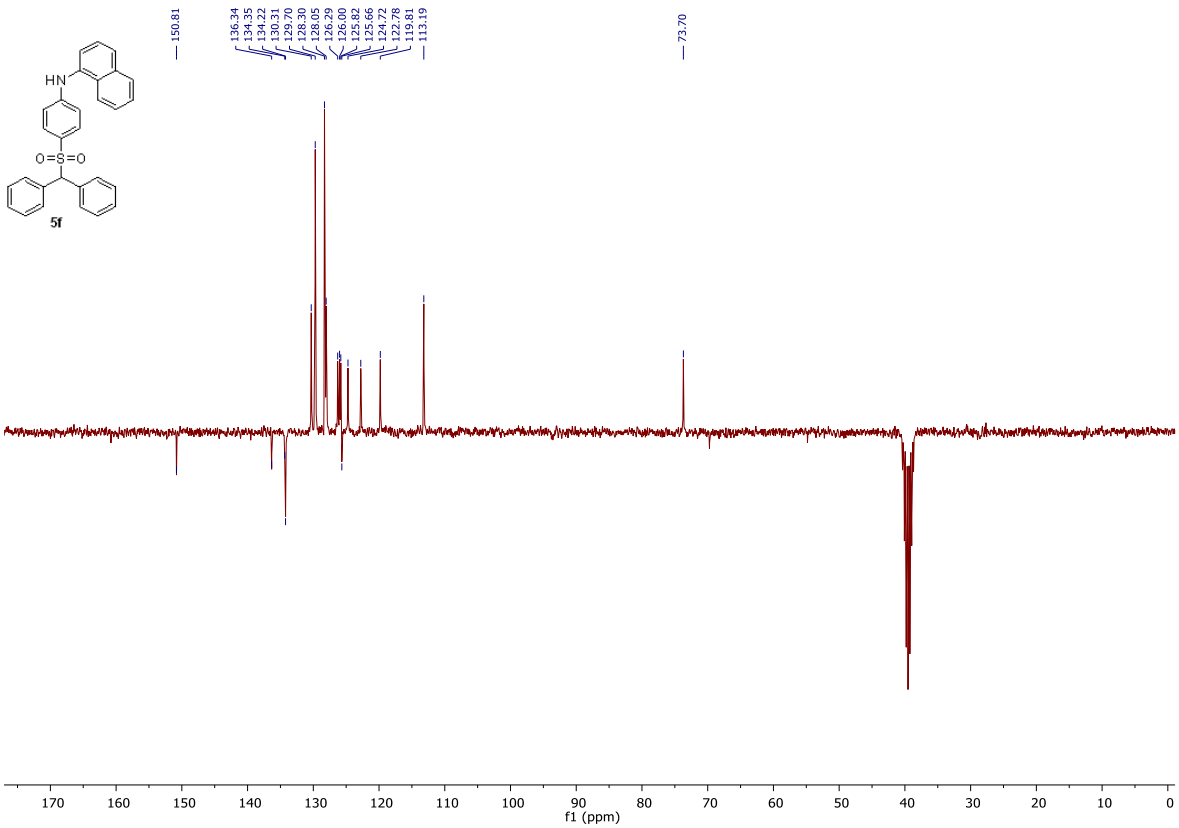

OMe
HN
OMe
O=S=O
5d
161.08
148.29
142.63
134.16
130.40
129.74
128.34
128.09
126.57
114.19
97.58
94.40
73.70
55.06
f1 (ppm)

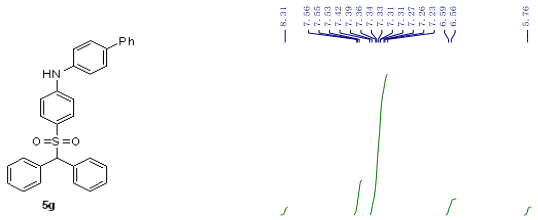
2020-06-19.40.fid
KNZ685 1H DMSO
PROTON DMSO E:\\ chit 39

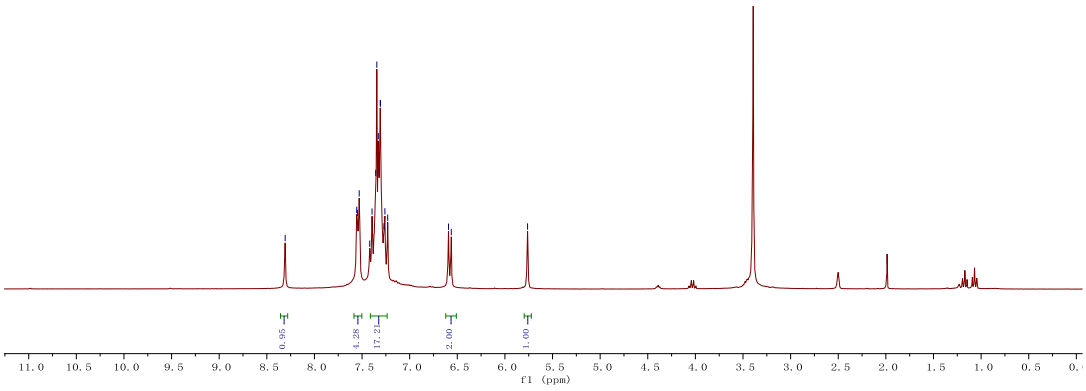


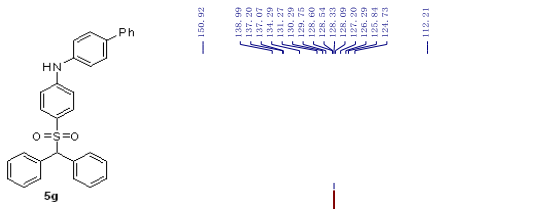


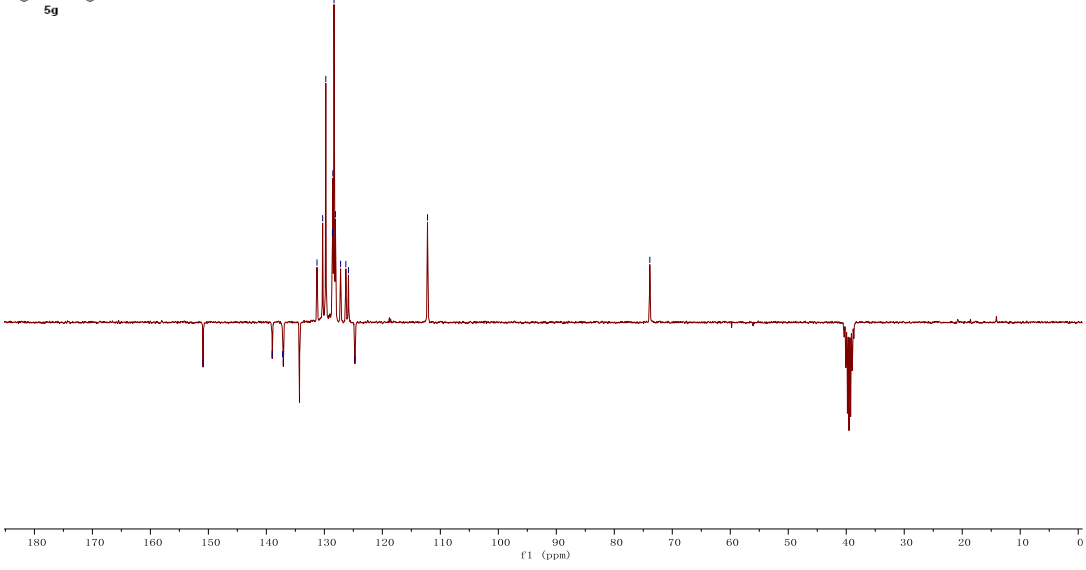

8.88
7.97
7.95
7.92
7.77
7.74
7.59
7.57
7.53
7.51
7.48
7.40
7.37
7.34
7.32
7.30
7.28
6.79
6.77
5.83
2.50
HN
O=S=O
5f
0.97
2.00
1.10
7.00
9.03
2.00
0.96
f1 (ppm)

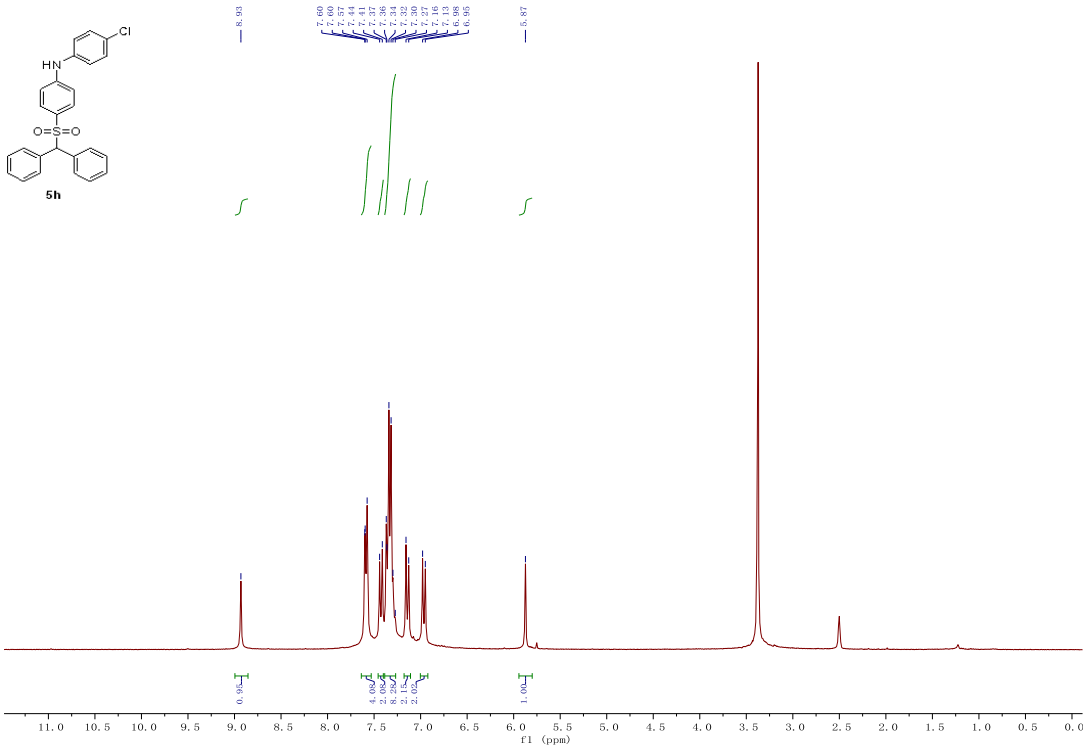

150.81
136.34
134.35
134.22
130.31
129.70
128.30
128.05
126.29
126.00
125.82
125.66
124.72
122.78
119.81
113.19
73.70
HN
O=S=O
5f
f1 (ppm)

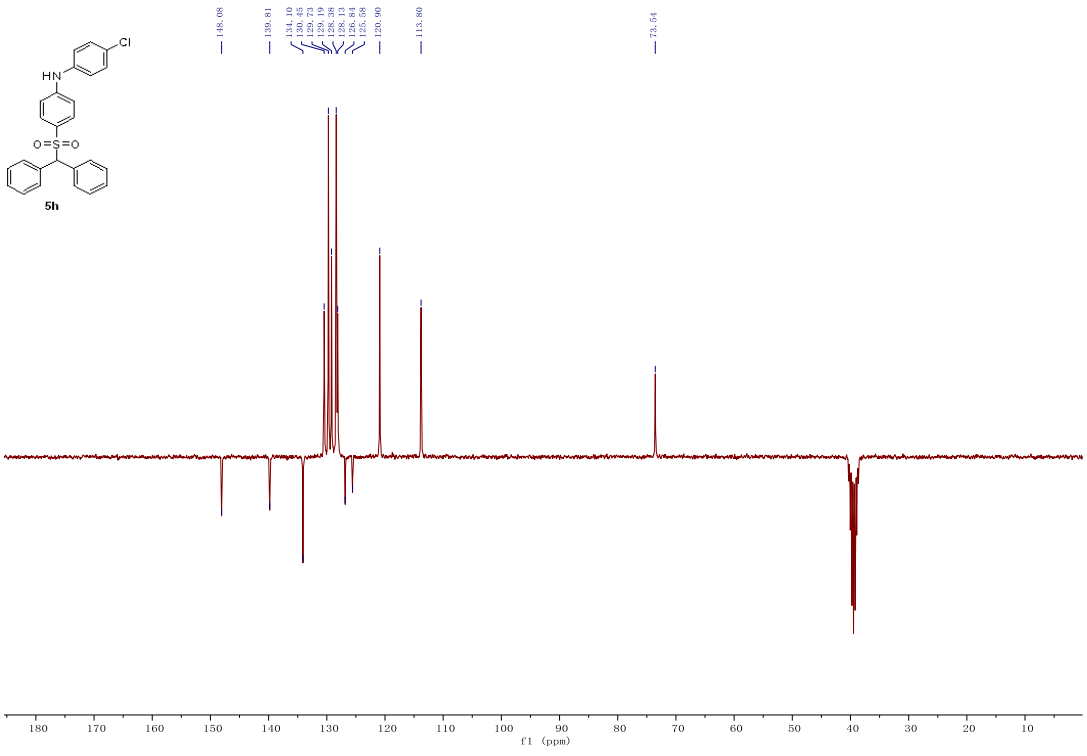

8.31
7.56
7.55
7.53
7.42
7.39
7.36
7.34
7.33
7.31
7.31
7.27
7.26
7.23
6.59
6.56
5.76
Ph
HN
O=S=O
5g

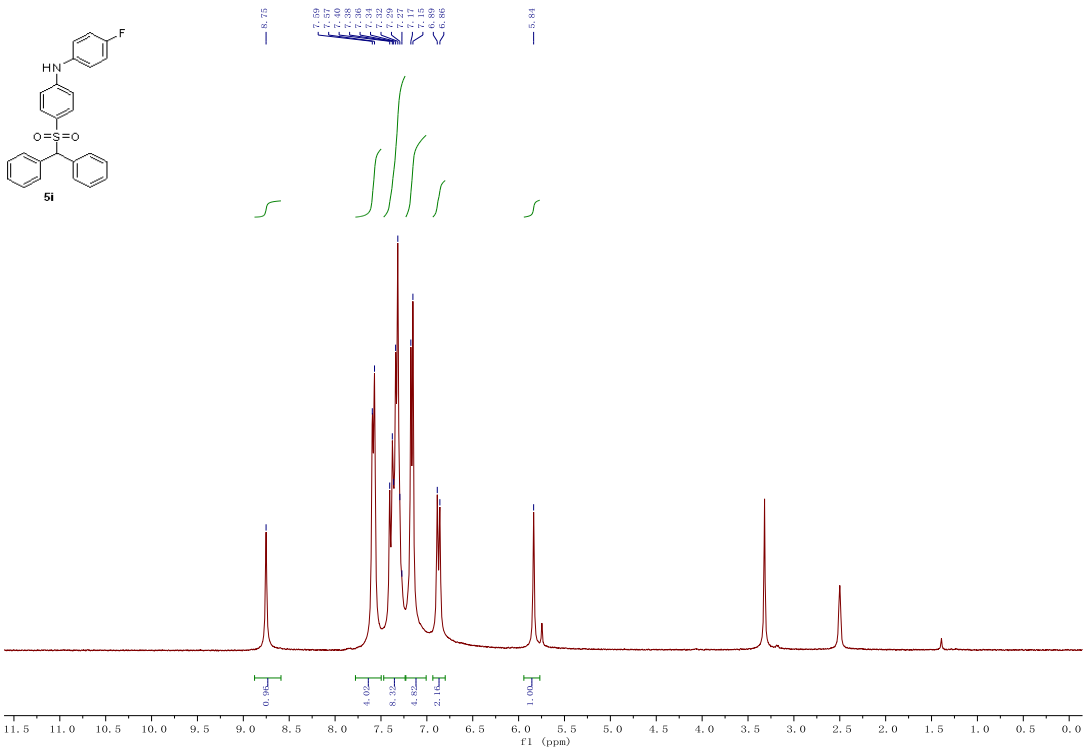

0.95
4.28
17.24
2.00
1.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

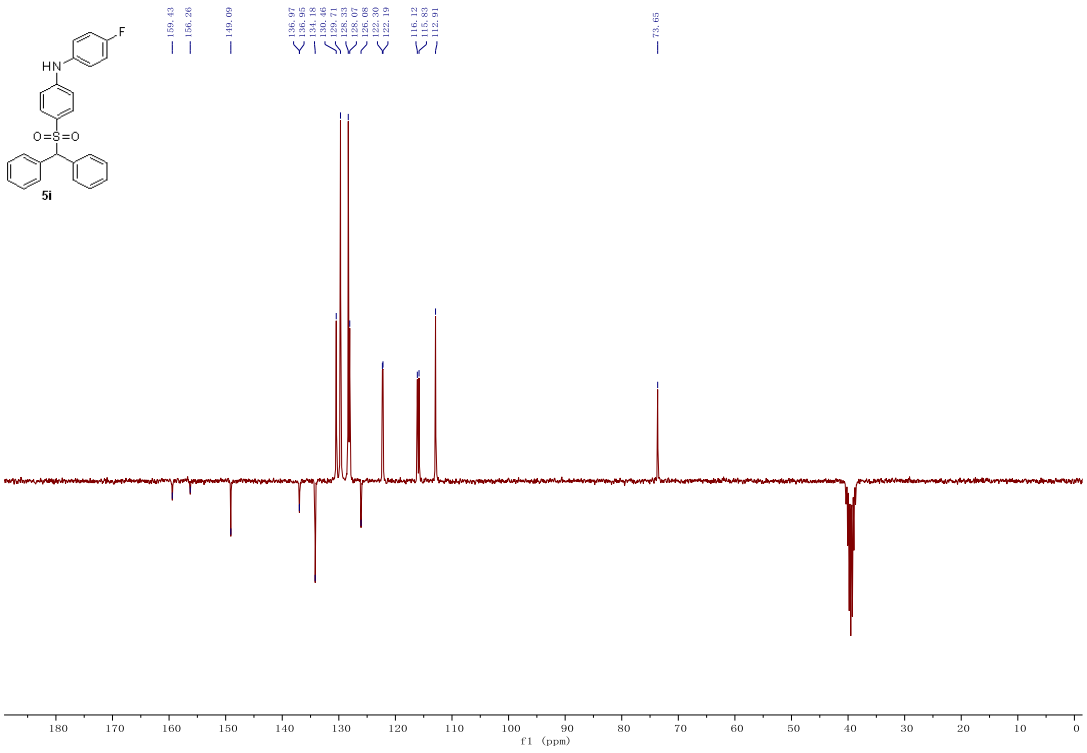

150.92
138.99
137.20
137.07
134.29
131.27
130.29
129.75
128.60
128.54
128.33
128.09
127.20
126.29
125.84
124.73
112.21
73.87
Ph
HN
O=S=O
5g

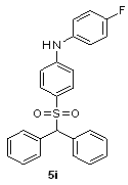

180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Cl
HN
O=S=O
5h
8.93
7.60
7.59
7.57
7.44
7.41
7.37
7.36
7.34
7.32
7.29
7.27
7.16
7.13
6.98
6.95
5.87
0.95
4.08
2.08
8.28
2.15
2.05
1.00
f1 (ppm)


Cl
HN
O=S=O
5h
148.08
139.81
134.10
130.45
129.73
129.39
128.98
128.13
126.84
125.58
120.90
115.80
73.54
f1 (ppm)

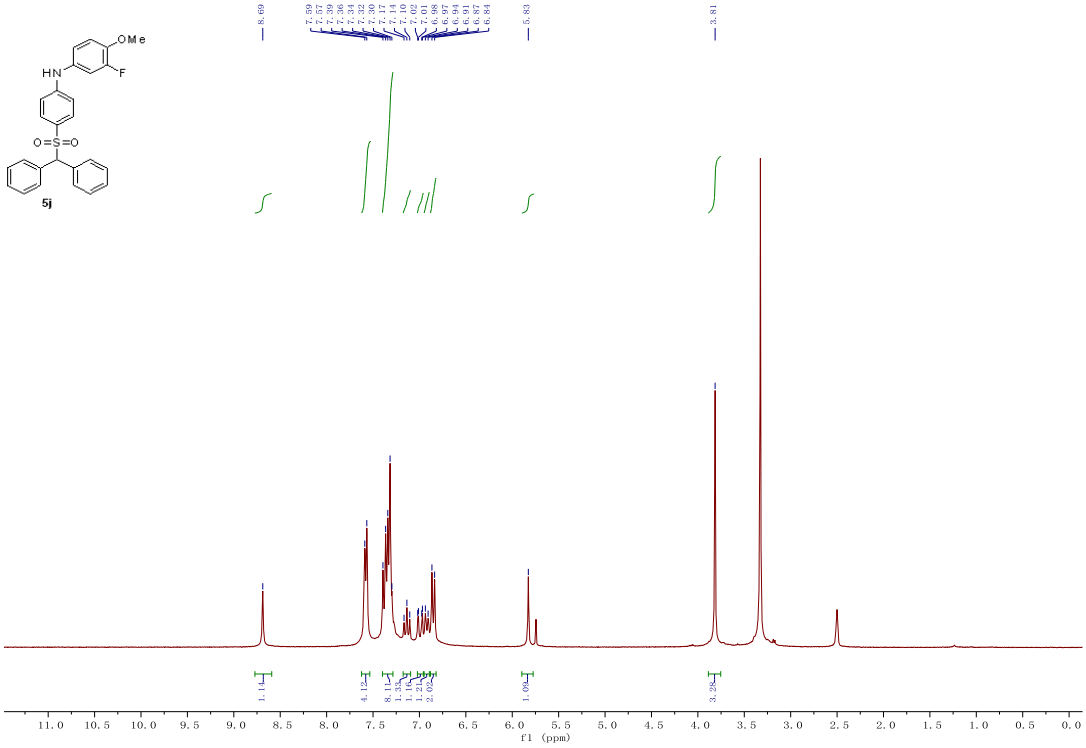

8.75
7.59
7.57
7.40
7.38
7.36
7.34
7.32
7.29
7.27
7.17
7.15
6.89
6.88
5.84
HN
F
O=S=O
5i
0.96
4.02
8.32
4.82
2.16
1.00
f1 (ppm)

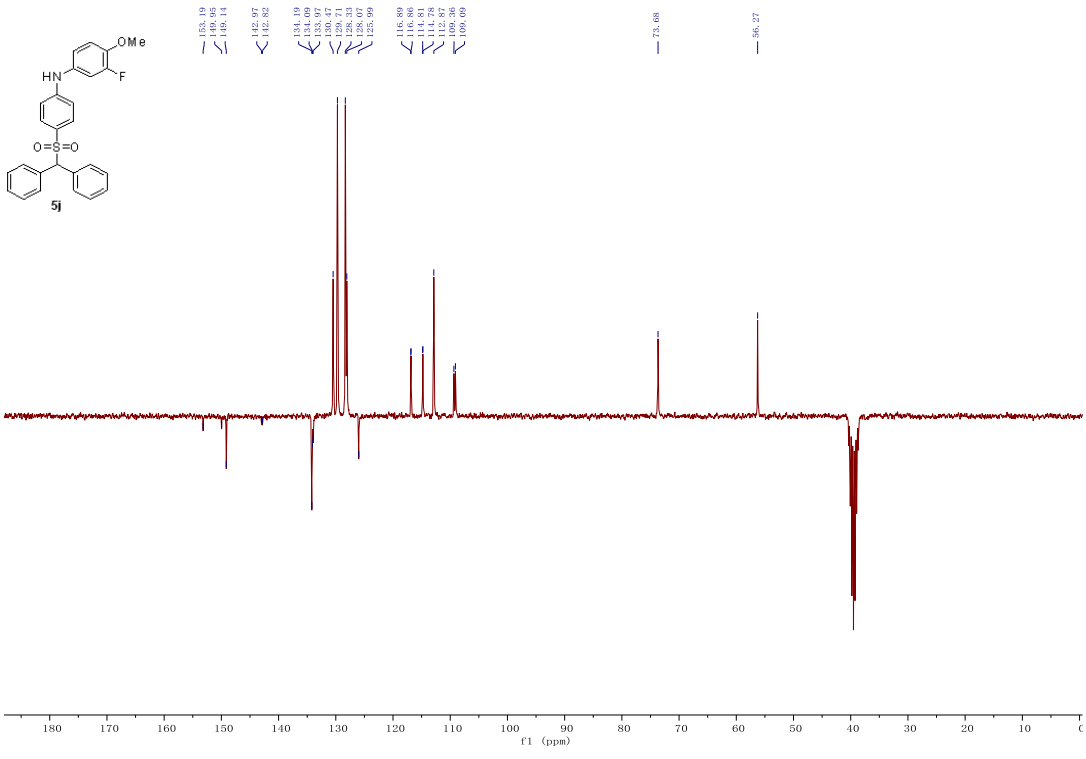

169.43
156.26
149.09
136.97
136.95
134.18
130.46
129.71
128.33
128.07
126.08
120.99
122.19
116.12
115.83
112.91
73.65
HN
F
O=S=O
5i
f1 (ppm)

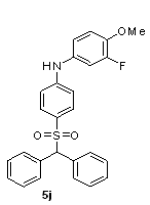

F
HN
O=S=O

5i

-120.10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-2
f1 (ppm)

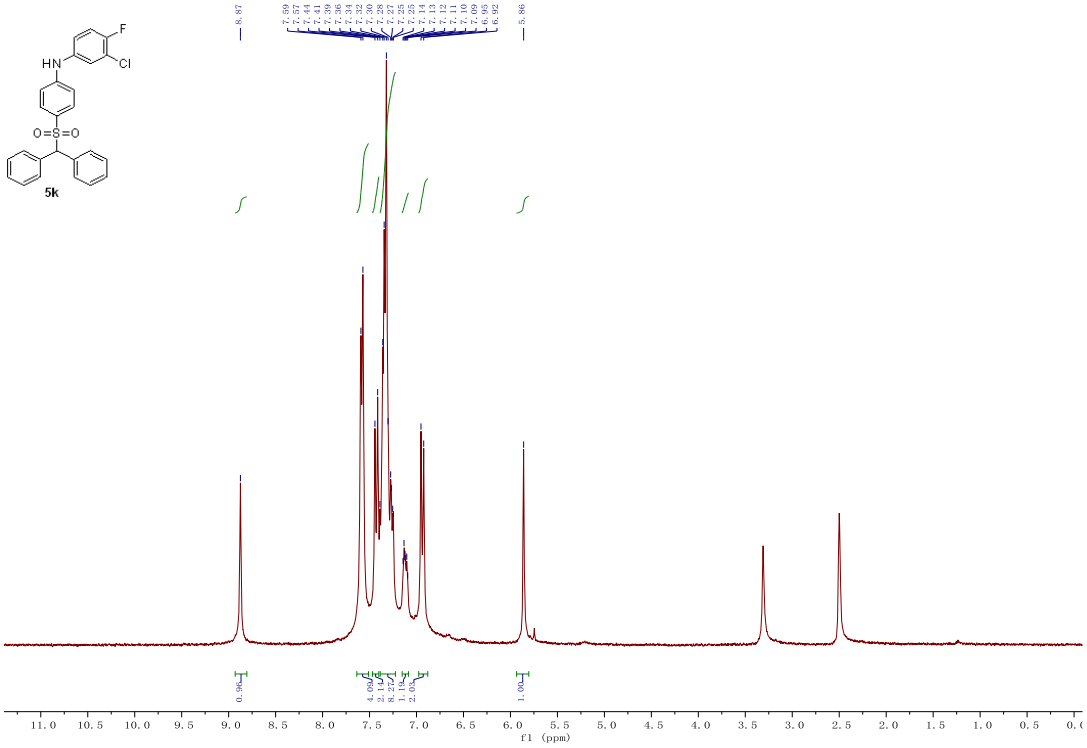

OMe
HN
F
O=S=O
5j
8.69
7.59
7.57
7.39
7.36
7.34
7.32
7.30
7.17
7.14
7.10
7.02
7.01
6.98
6.97
6.94
6.91
6.87
6.84
5.83
3.81
1.14
4.12
8.11
1.35
1.15
1.27
2.02
1.09
3.29
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

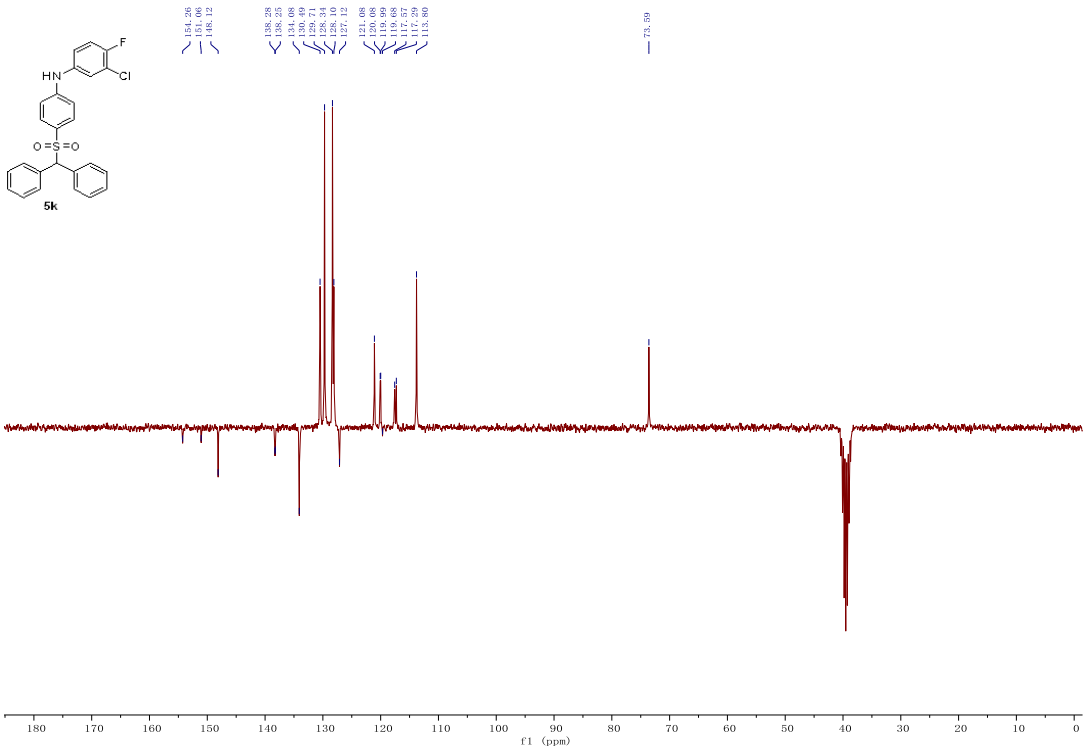

OMe
HN
F
O=S=O
5j
153.19
149.95
149.14
142.97
142.82
134.19
134.09
133.97
130.47
129.71
128.33
126.67
125.99
116.89
116.86
114.81
114.78
112.87
109.36
109.09
73.68
56.27
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

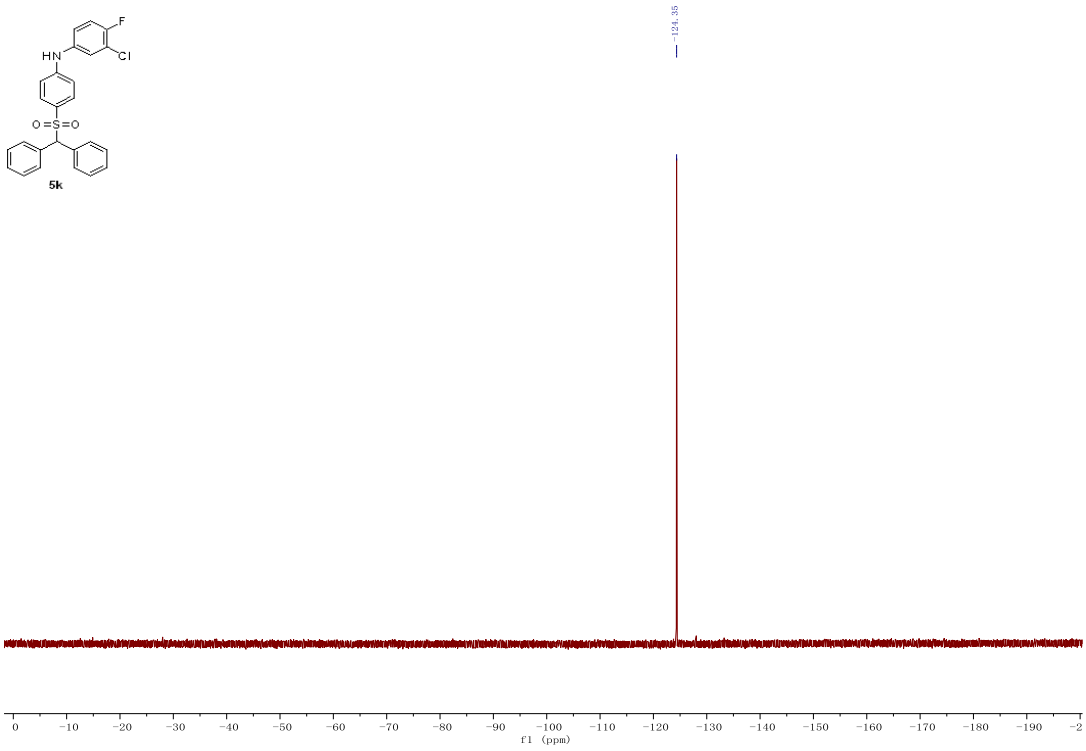

OMe
HN
F
O=S=O

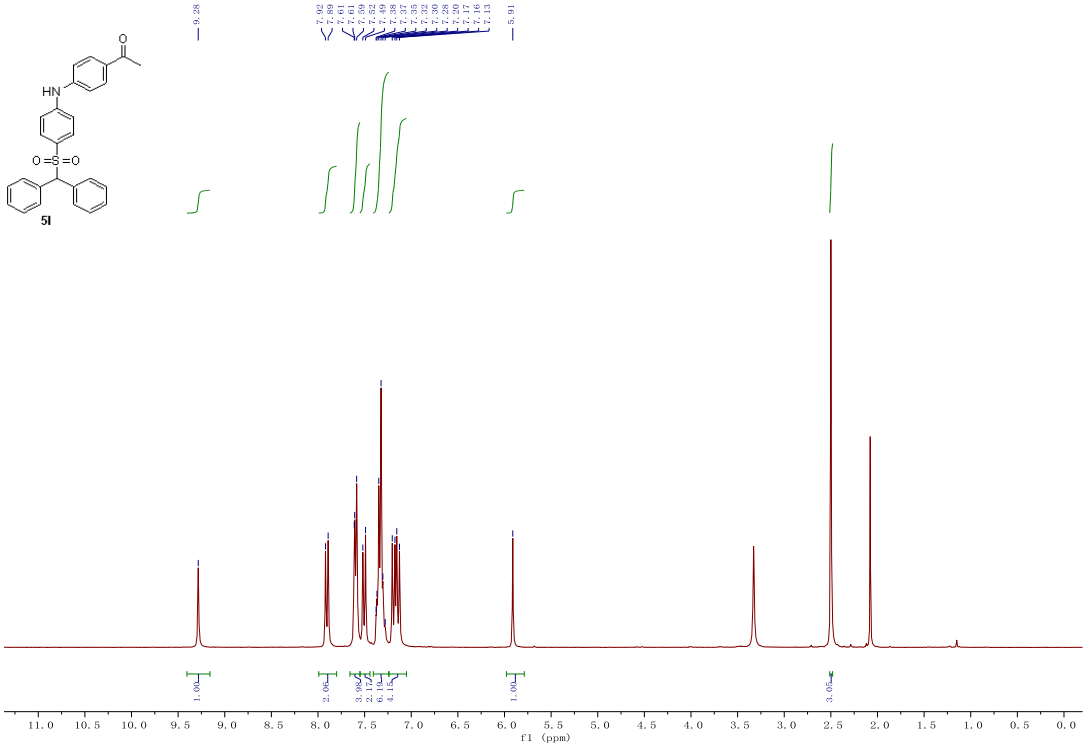
5j

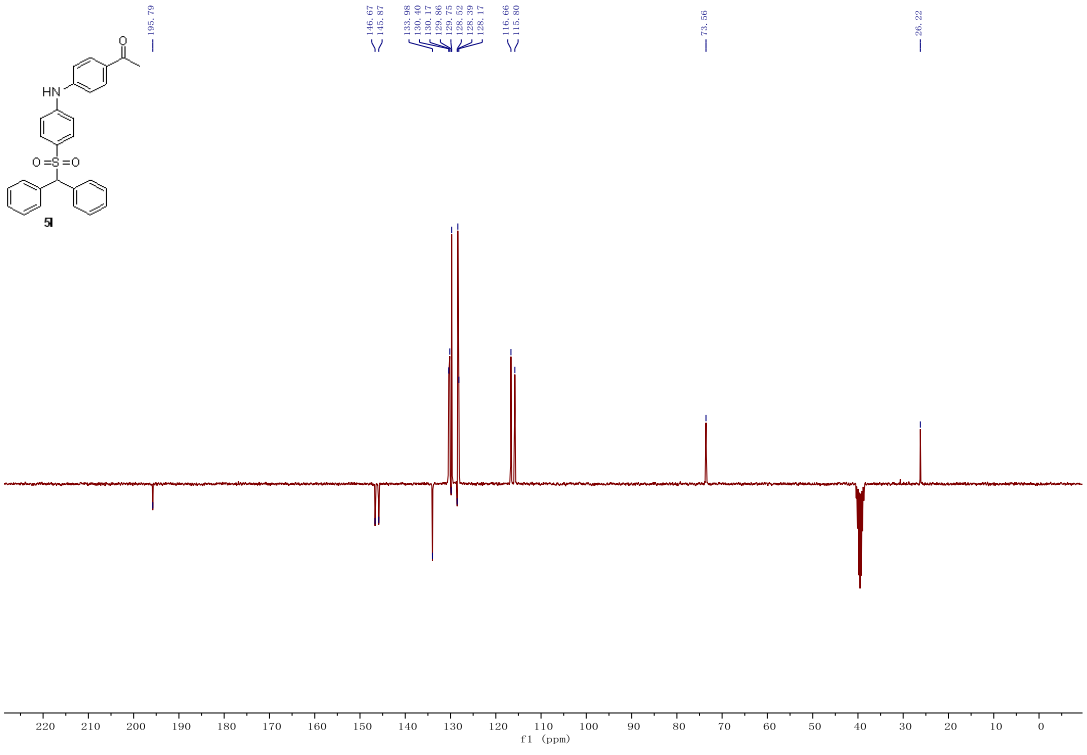
-133.21
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

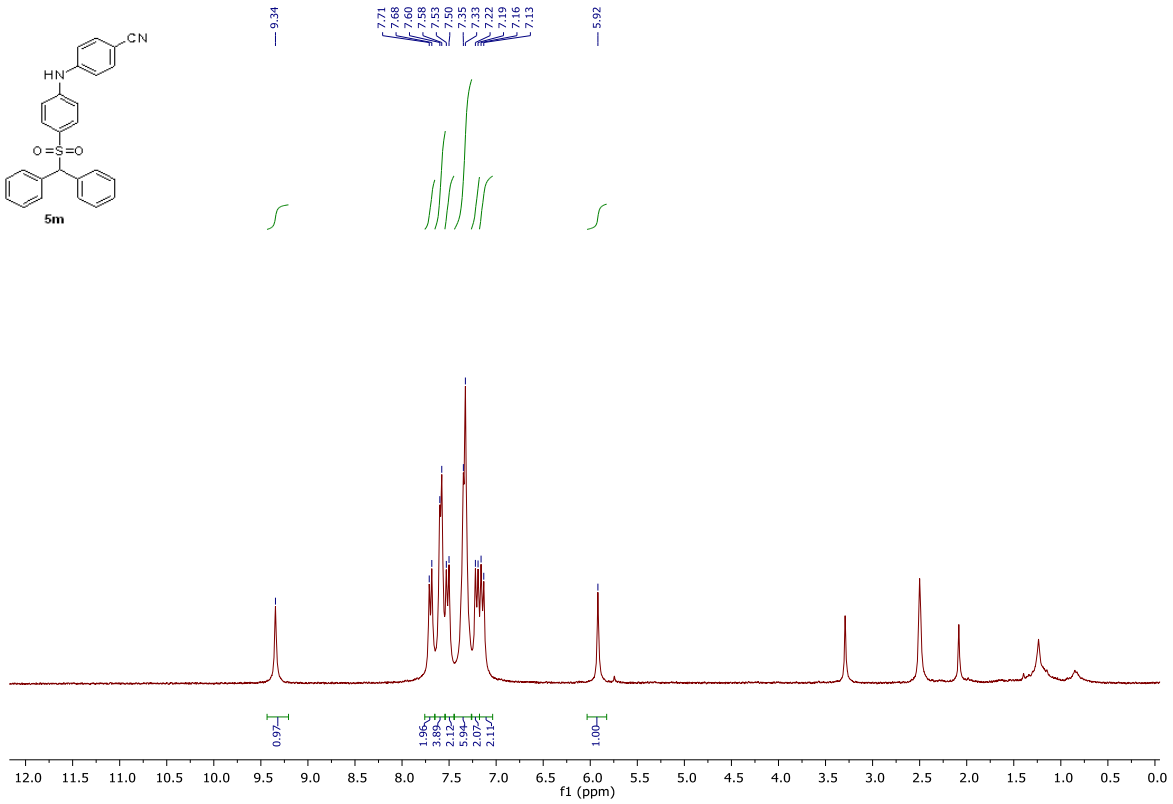

F
Cl
HN
O=S=O
5k
8.87
7.59
7.57
7.44
7.43
7.39
7.36
7.34
7.32
7.30
7.28
7.27
7.25
7.14
7.13
7.12
7.11
7.10
7.09
6.95
6.92
5.86
0.96
4.69
2.11
8.27
1.94
2.03
1.00
f1 (ppm)

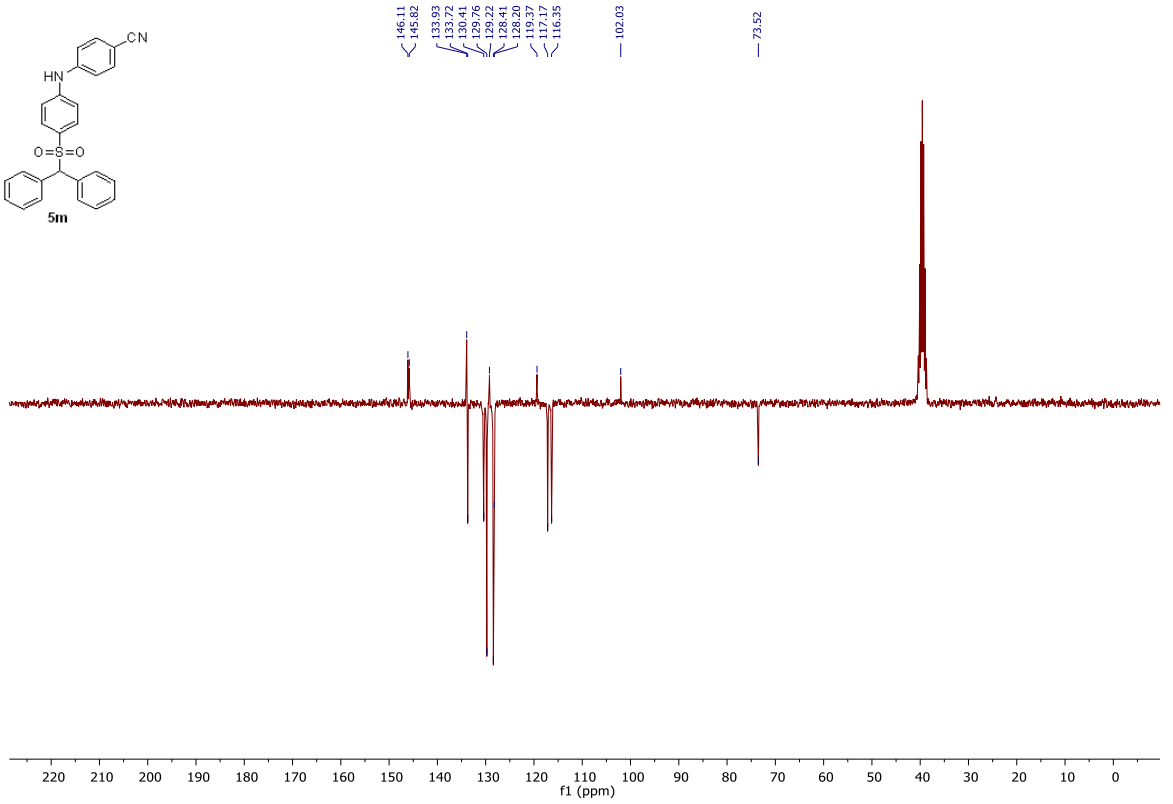

F
Cl
HN
O=S=O
5k
154.26
151.06
148.12
138.28
138.25
134.66
130.49
129.71
128.34
128.10
127.12
121.08
120.86
119.99
119.68
117.57
117.39
113.80
73.59
f1 (ppm)

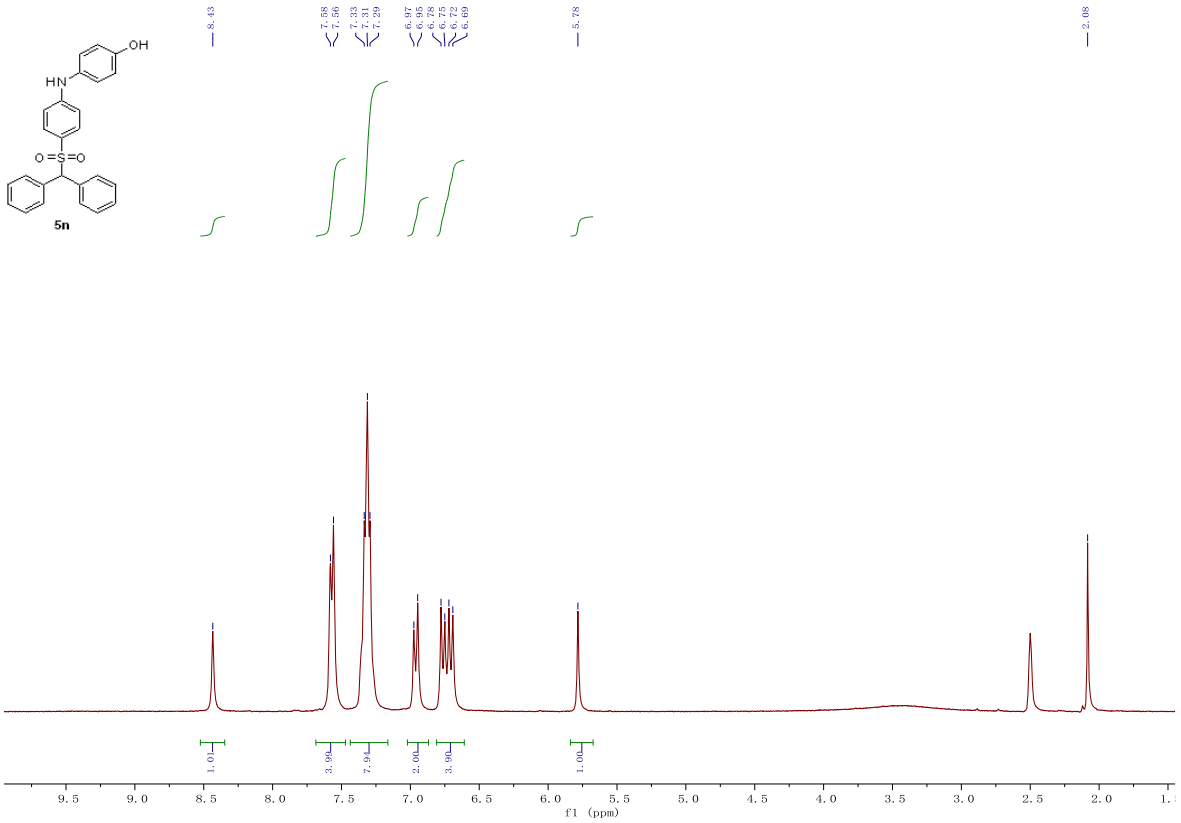

F
Cl
HN
O=S=O
5k
-124.35
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-2
f1 (ppm)

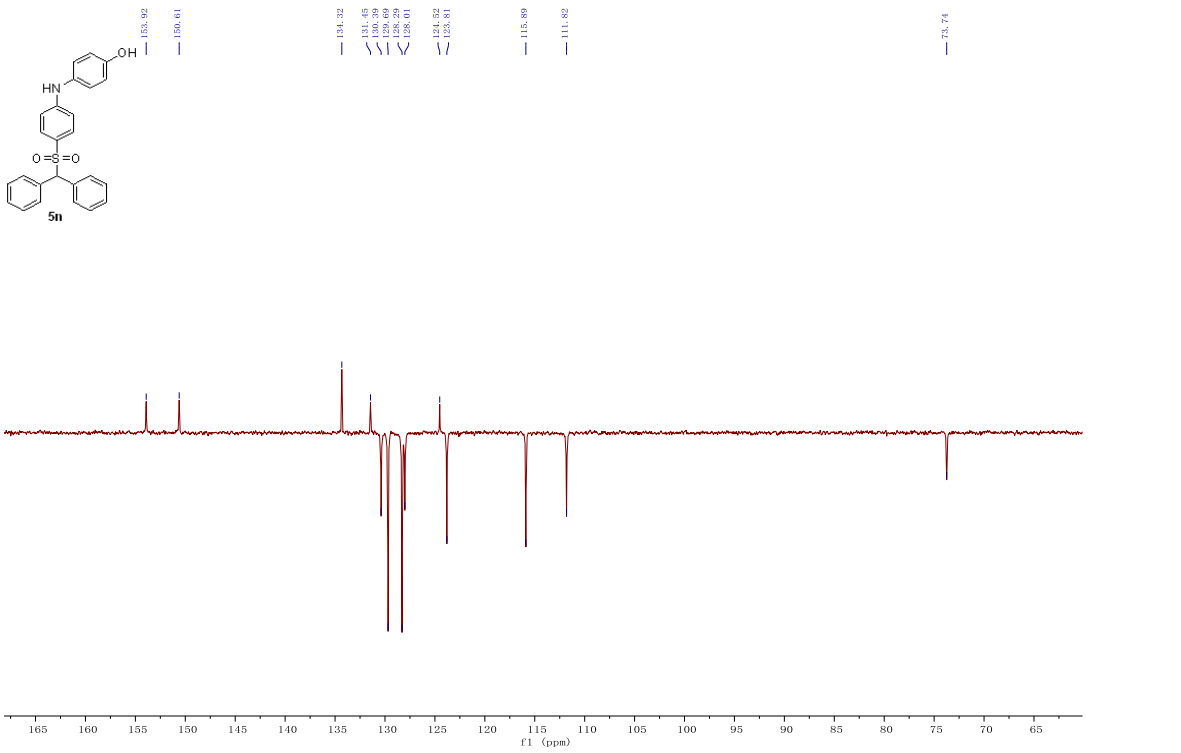

9.28
7.92
7.89
7.61
7.60
7.59
7.52
7.49
7.38
7.37
7.35
7.32
7.30
7.28
7.20
7.17
7.16
7.13
5.91
HN
O=S=O
5l
1.00
2.06
3.98
2.12
6.13
4.15
1.00
3.05
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

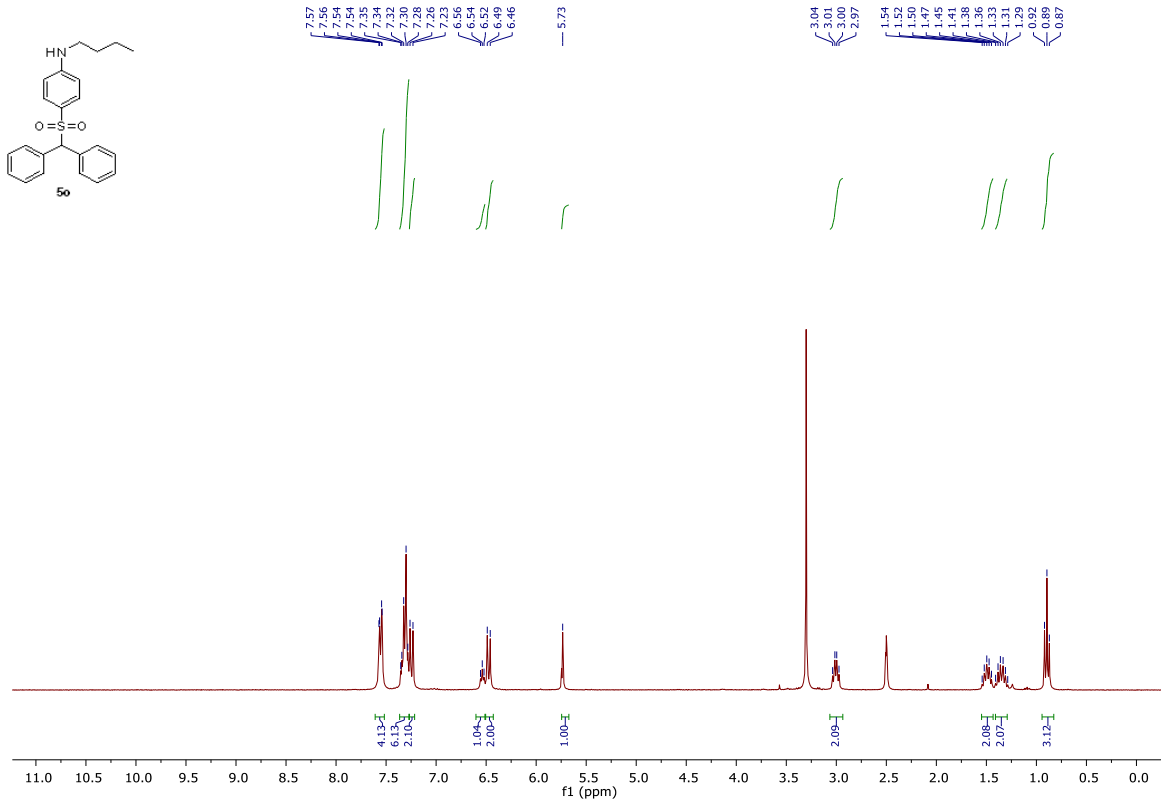

195.79
146.67
145.87
133.98
130.49
130.17
129.86
129.75
128.52
128.39
128.17
116.66
115.80
73.56
26.22
HN
O=S=O
5l
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

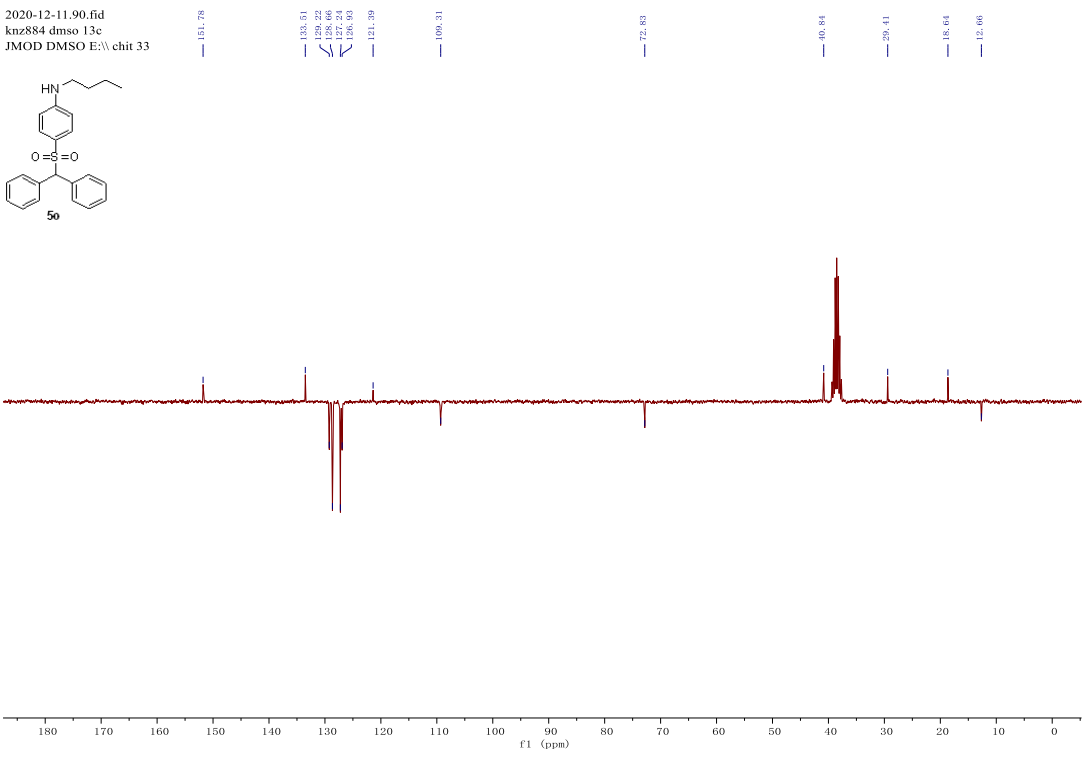

CN
HN
O=S=O
5m
9.34
7.71
7.68
7.60
7.58
7.53
7.50
7.35
7.33
7.22
7.19
7.16
7.13
5.92
0.97
1.96
3.89
2.12
5.94
2.07
2.11
1.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

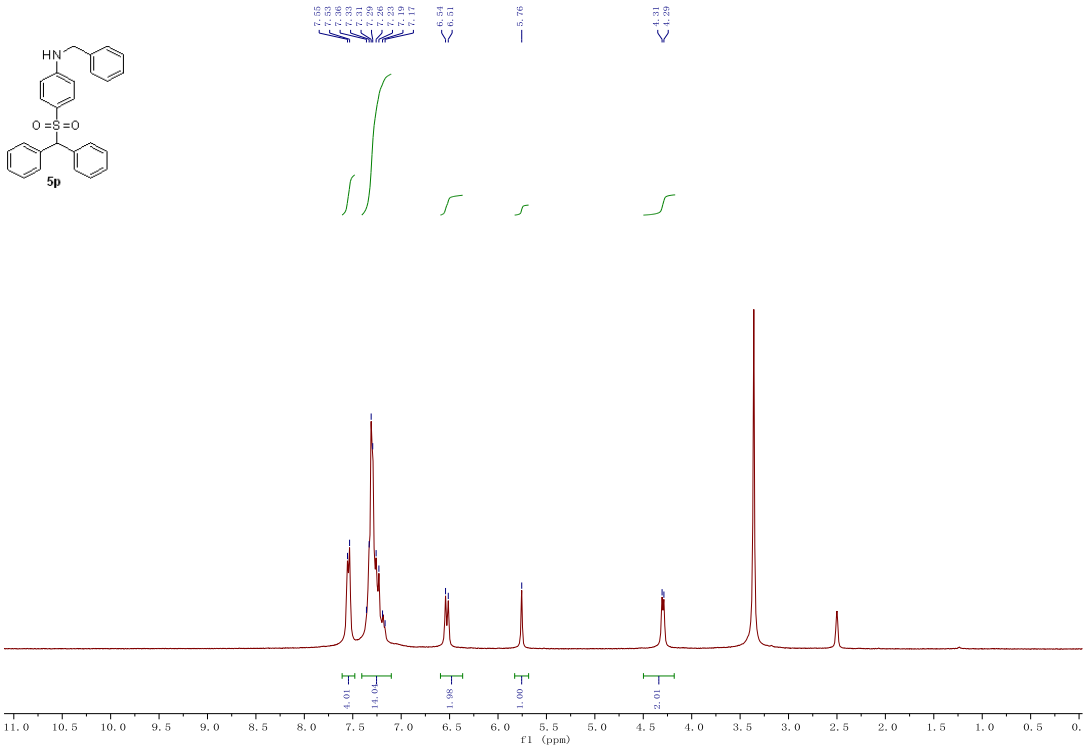

CN
HN
O=S=O
5m
146.11
145.82
133.93
133.72
130.41
129.76
129.22
128.41
128.20
119.37
117.17
116.35
102.03
73.52
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

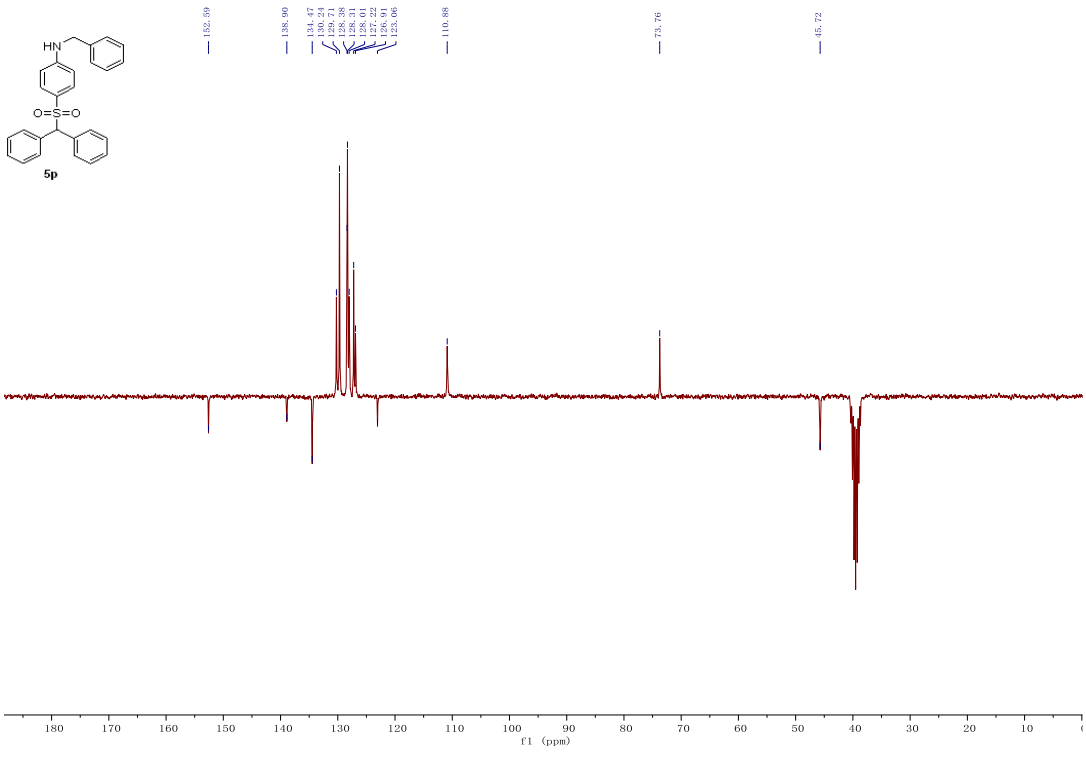

OH
HN
O=S=O
5n
8.43
7.58
7.56
7.33
7.31
7.29
6.97
6.95
6.78
6.75
6.72
6.69
5.78
2.08
1.0
3.96
7.94
2.00
3.96
1.00
f1 (ppm)

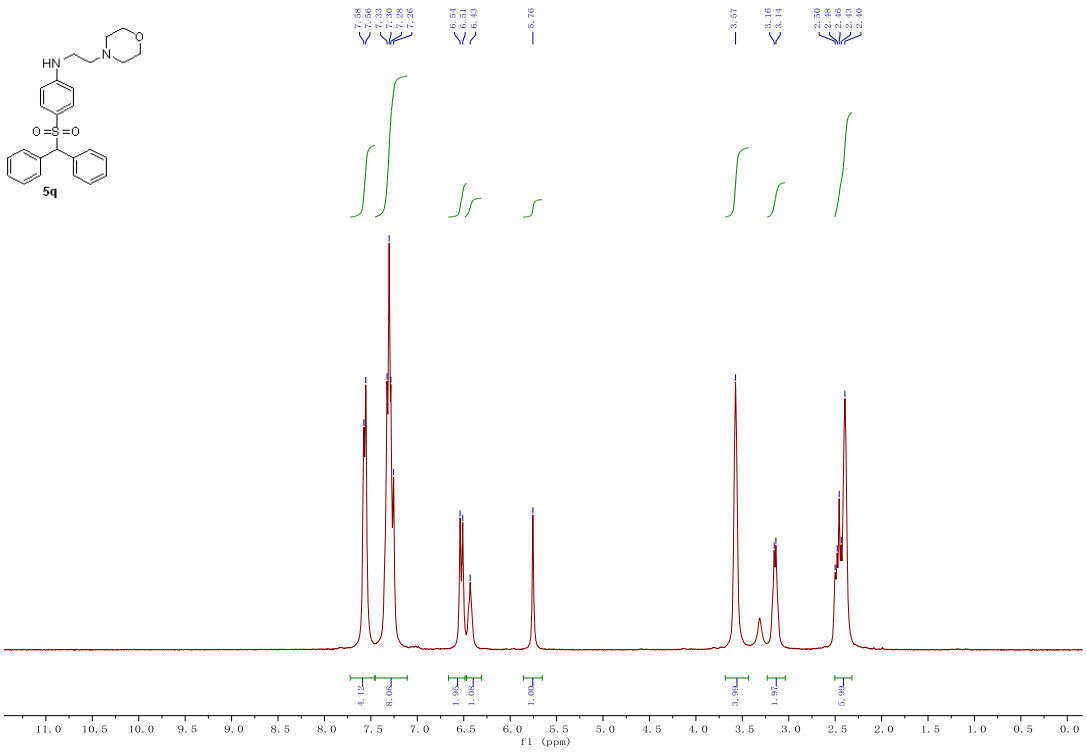

OH
HN
O=S=O
5n
153.92
150.61
134.32
131.45
130.09
129.69
128.29
128.01
124.52
123.81
115.89
111.82
73.74
f1 (ppm)

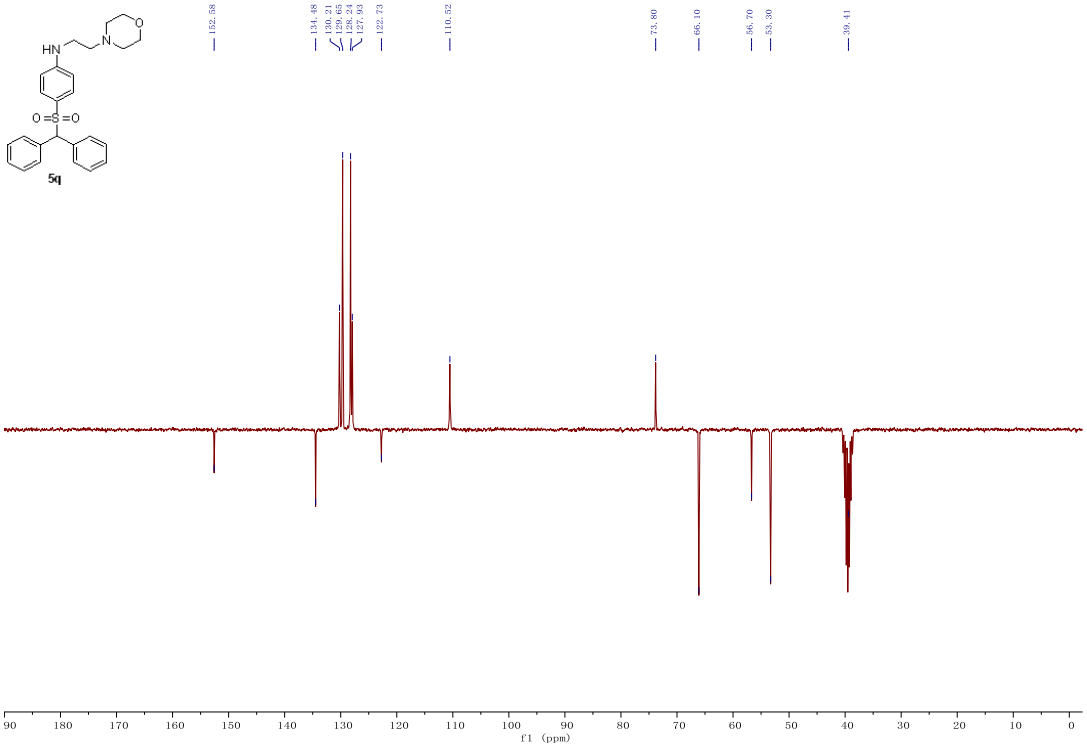

HN
O=S=O
5o
7.57
7.56
7.54
7.54
7.35
7.34
7.32
7.30
7.28
7.26
7.23
6.56
6.54
6.53
6.49
6.46
5.73
3.04
3.01
3.00
2.97
1.54
1.52
1.50
1.47
1.45
1.41
1.38
1.36
1.33
1.31
1.29
0.92
0.89
0.87
4.13
6.13
2.10
1.04
2.06
1.00
2.09
2.08
2.07
3.12
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

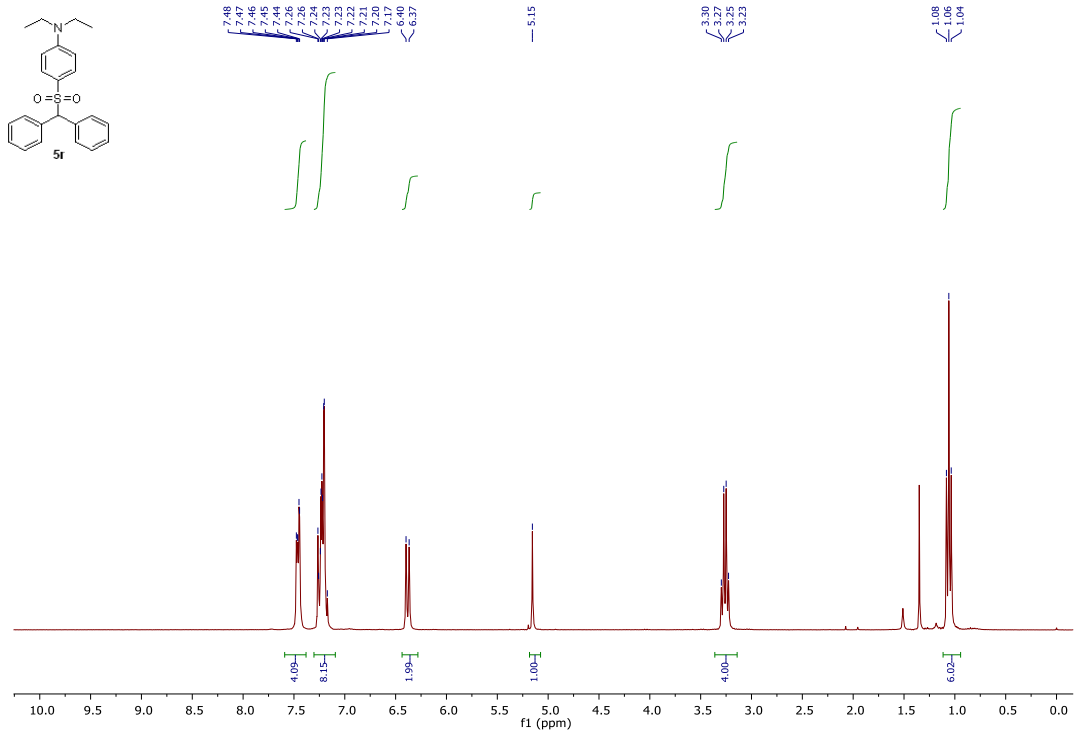

2020-12-11.90.fid
knz884 dmso 13c
JMOD DMSO E:\\ chit 33
151.78
133.61
129.22
128.66
127.24
126.93
121.39
109.31
72.83
40.84
29.41
18.64
12.66
HN
O=S=O
5o
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

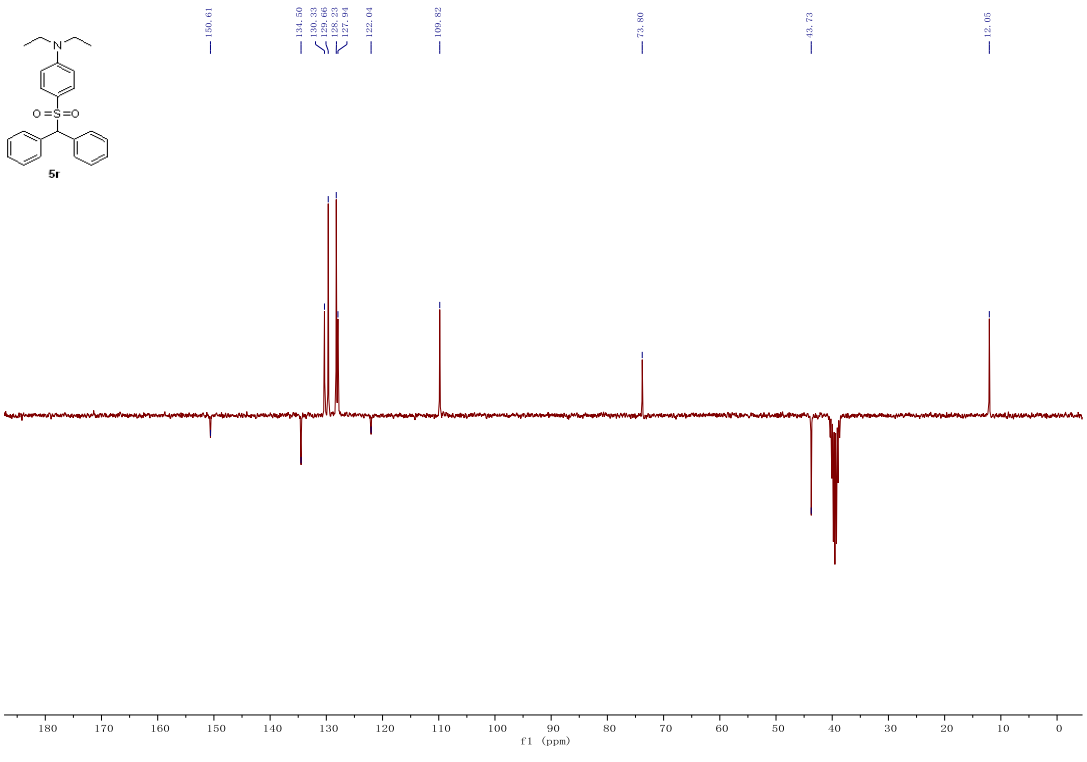

HN
O=S=O
5p
7.55
7.53
7.36
7.33
7.31
7.29
7.26
7.23
7.19
7.17
6.54
6.51
5.76
4.31
4.29
4.01
14.04
1.98
1.00
2.01
f1 (ppm)

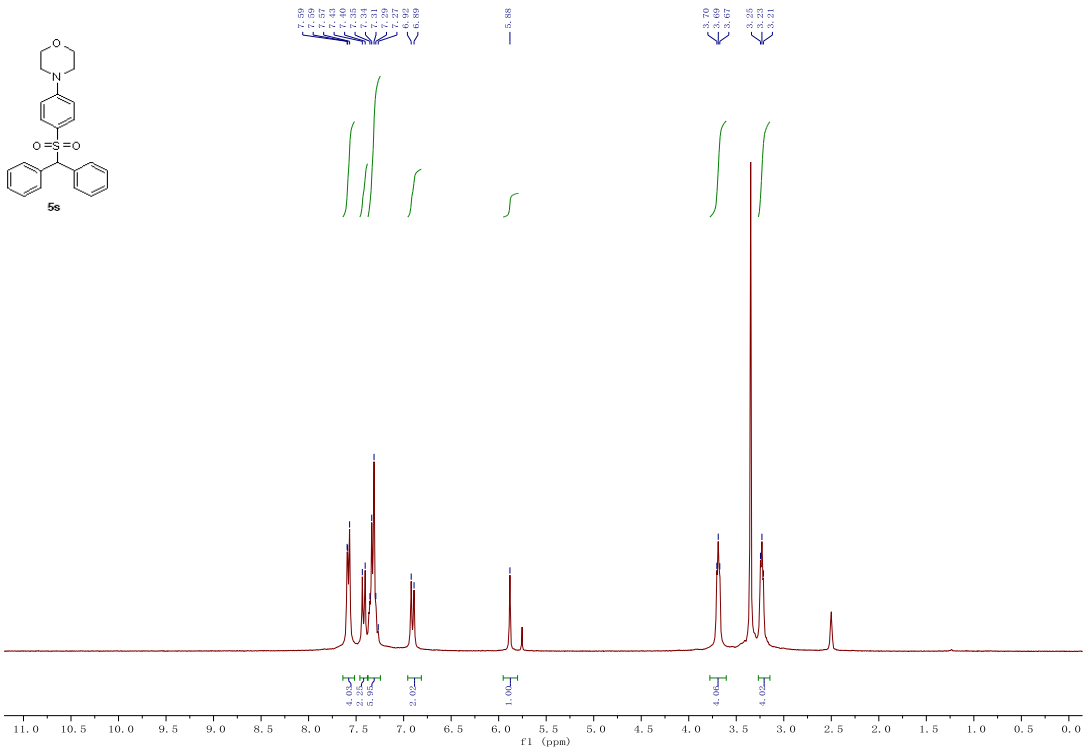

HN
O=S=O
5p
152.69
138.90
134.47
130.24
129.71
128.38
128.31
128.01
127.22
126.91
123.06
110.88
73.76
45.72
f1 (ppm)

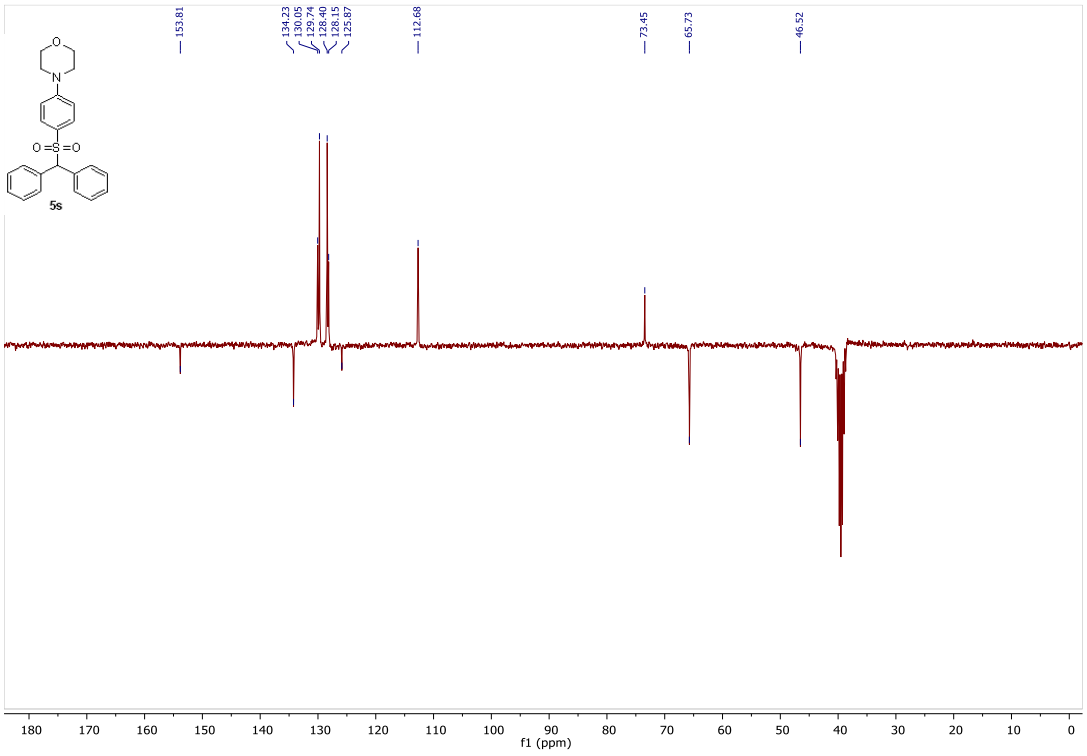

7.58
7.56
7.33
7.30
7.28
7.26
6.54
6.51
6.43
5.76
3.57
3.16
3.14
2.50
2.48
2.45
2.43
2.40
HN
O=S=O
5q
4.12
8.08
1.95
1.08
1.00
3.99
1.97
5.99
f1 (ppm)

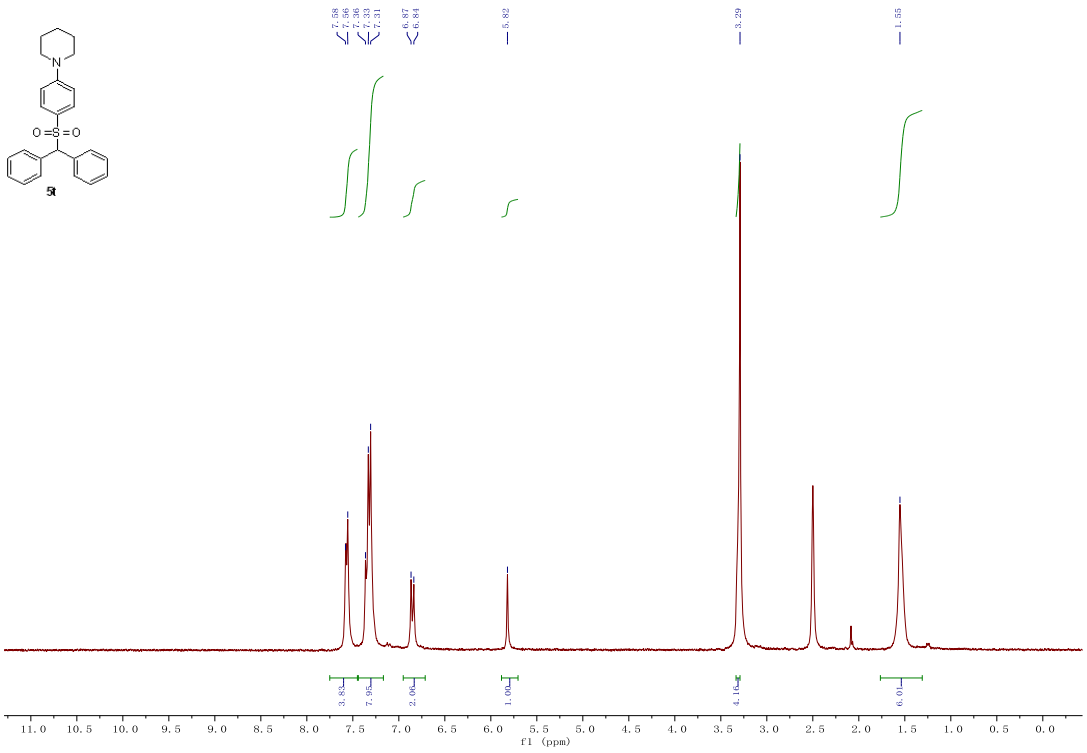

152.58
134.48
130.21
129.65
128.24
127.93
122.73
110.52
73.80
66.10
56.70
53.30
39.41
HN
O=S=O
5q
f1 (ppm)

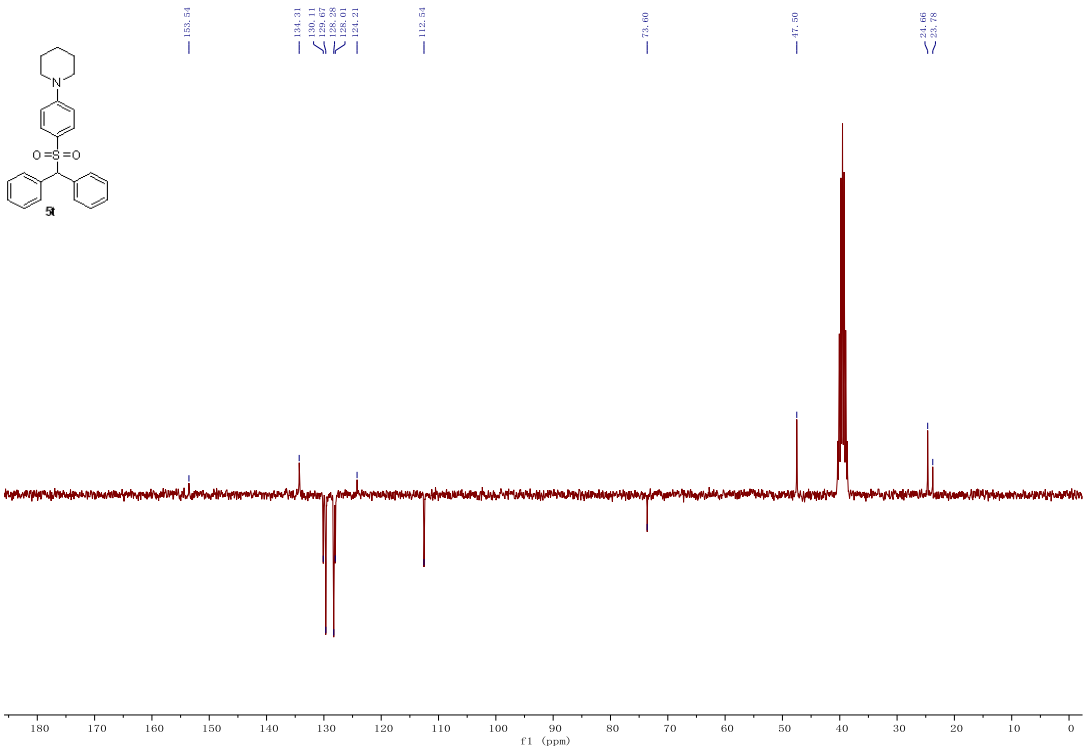

7.48
7.47
7.46
7.45
7.44
7.26
7.24
7.23
7.23
7.22
7.21
7.20
7.17
6.40
6.37
5.15
3.30
3.27
3.25
3.23
1.08
1.06
1.04
5f
4.09
8.15
1.99
1.00
4.00
6.02
f1 (ppm)

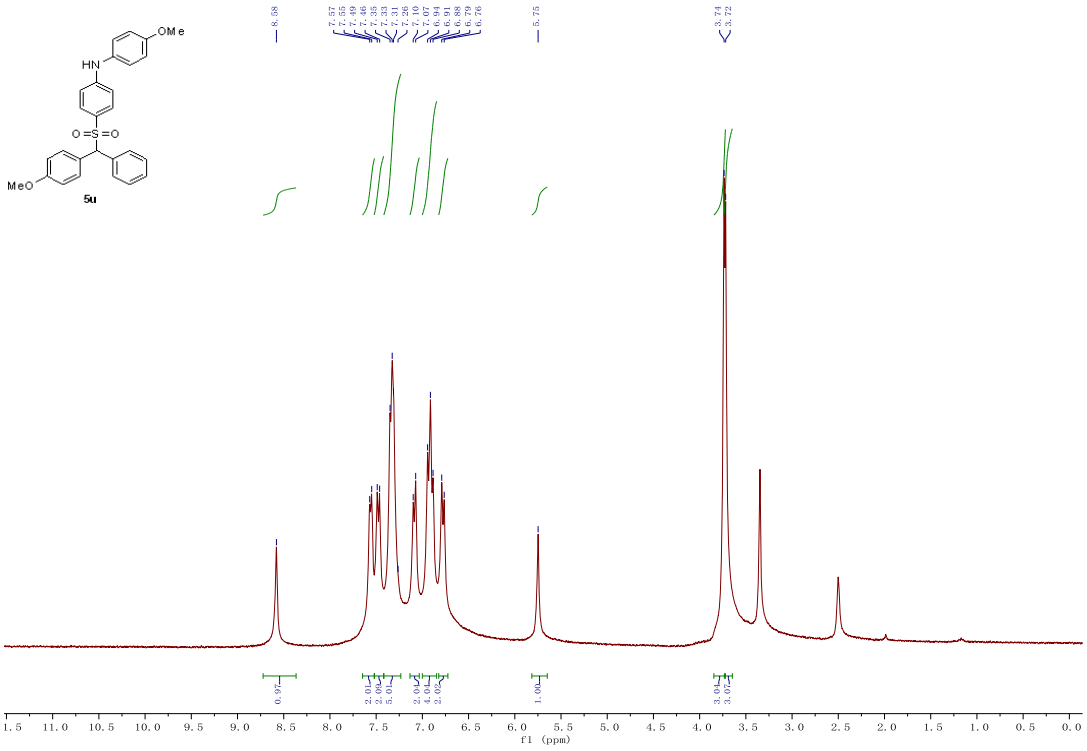

150.61
134.50
130.33
129.66
128.23
127.94
122.04
109.82
73.80
43.73
12.05
5f
f1 (ppm)

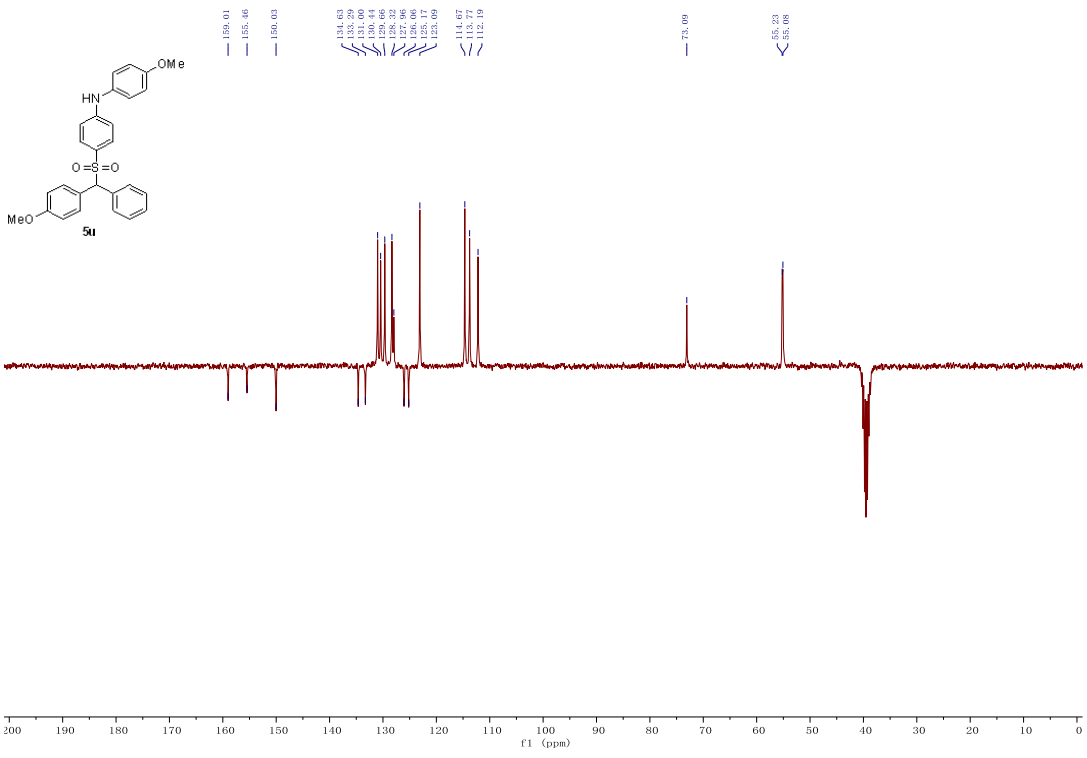

7.59
7.59
7.57
7.43
7.40
7.35
7.34
7.31
7.29
7.27
6.92
6.89
5.88
3.70
3.69
3.67
3.25
3.23
3.21
4.05
2.05
5.95
2.02
1.00
4.06
4.02
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
5s

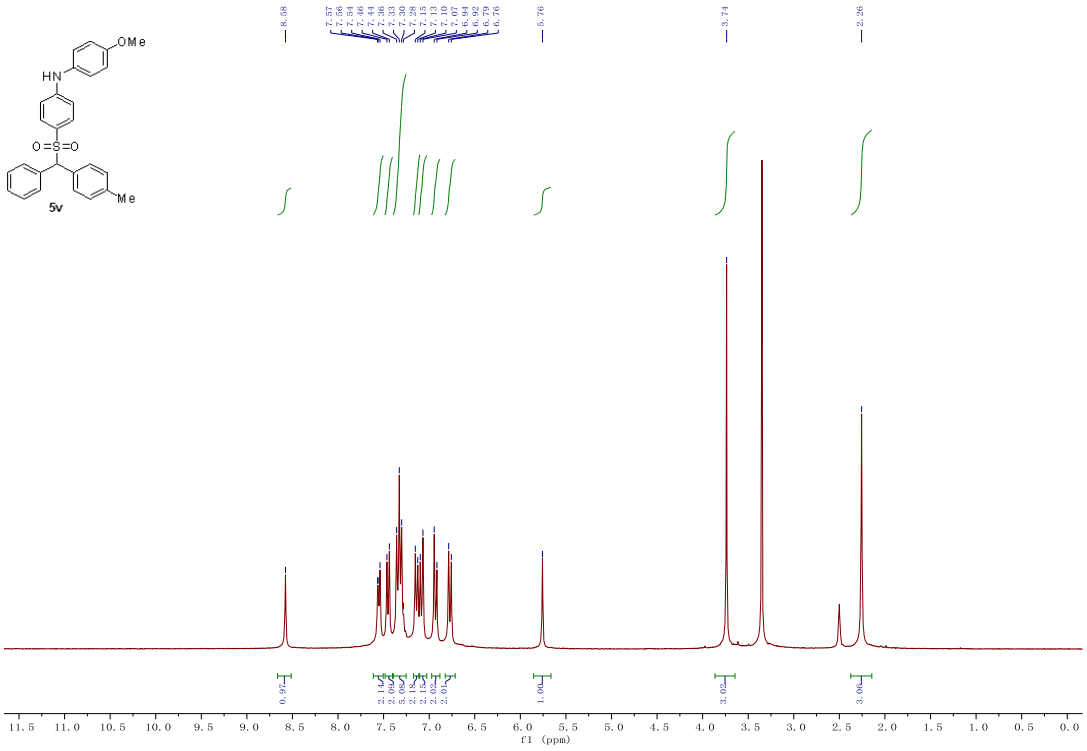

153.81
134.23
130.05
129.74
128.40
126.15
125.87
112.68
73.45
65.73
46.52
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
5s

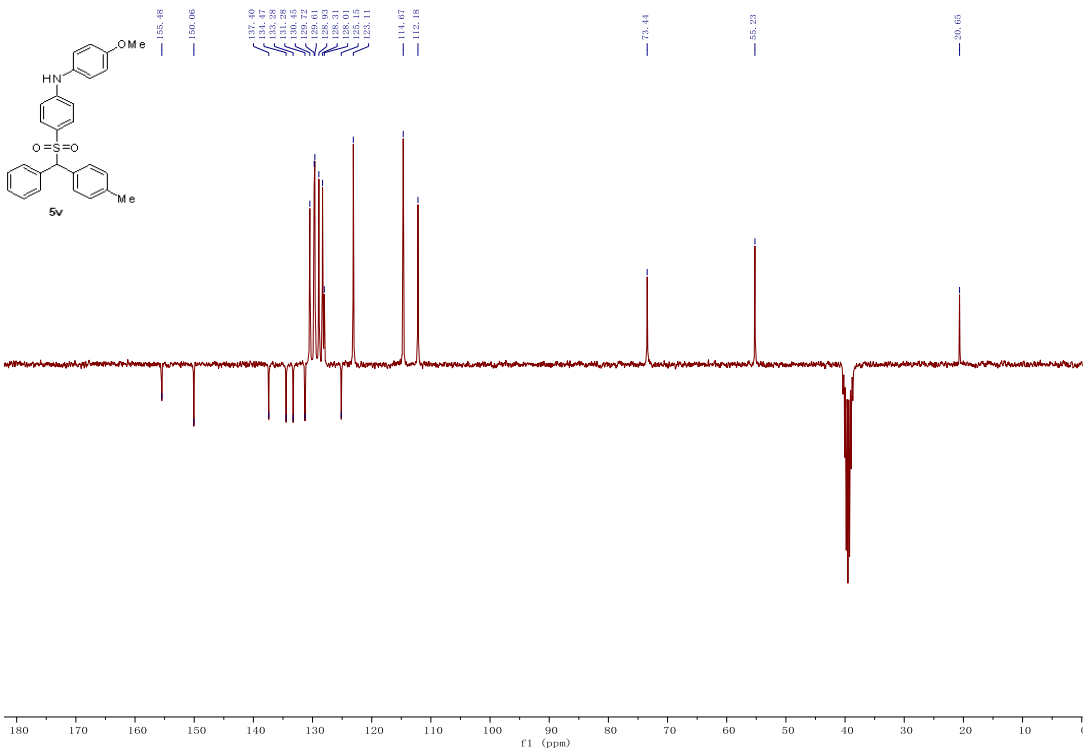

7.58
7.56
7.36
7.33
7.31
6.87
6.84
5.82
3.29
1.55
5t
3.83
7.95
2.06
1.00
4.16
6.01
f1 (ppm)

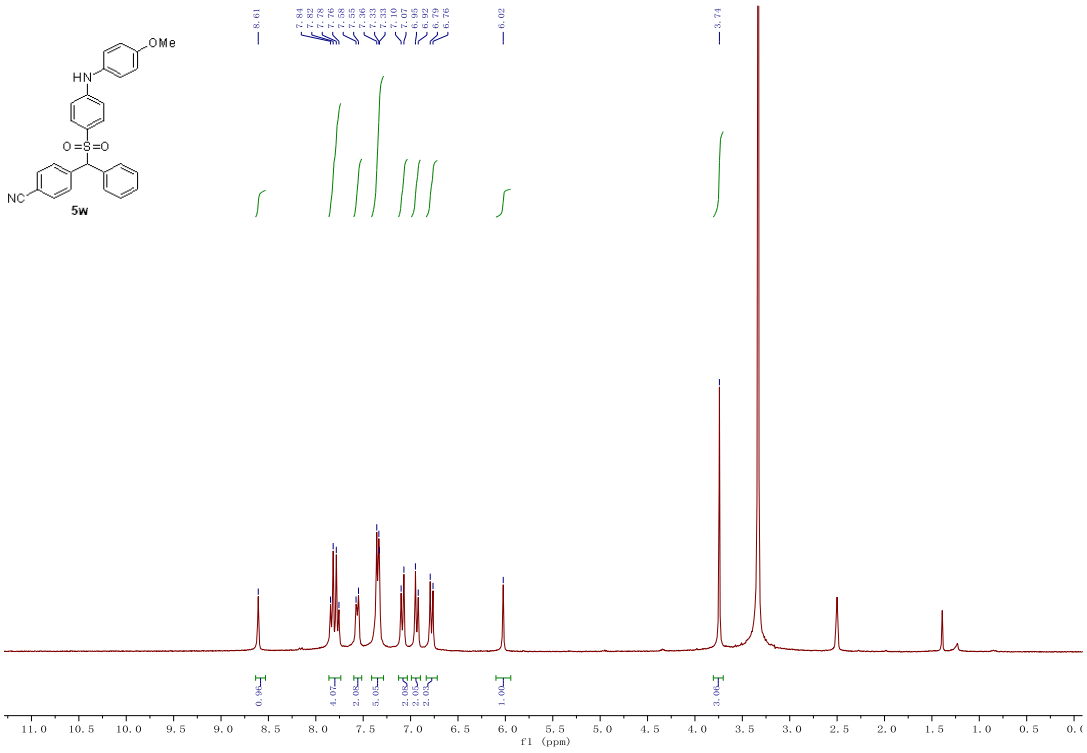

153.54
134.31
130.11
129.67
128.98
128.01
124.21
112.54
73.60
47.50
24.66
23.78
5t
f1 (ppm)

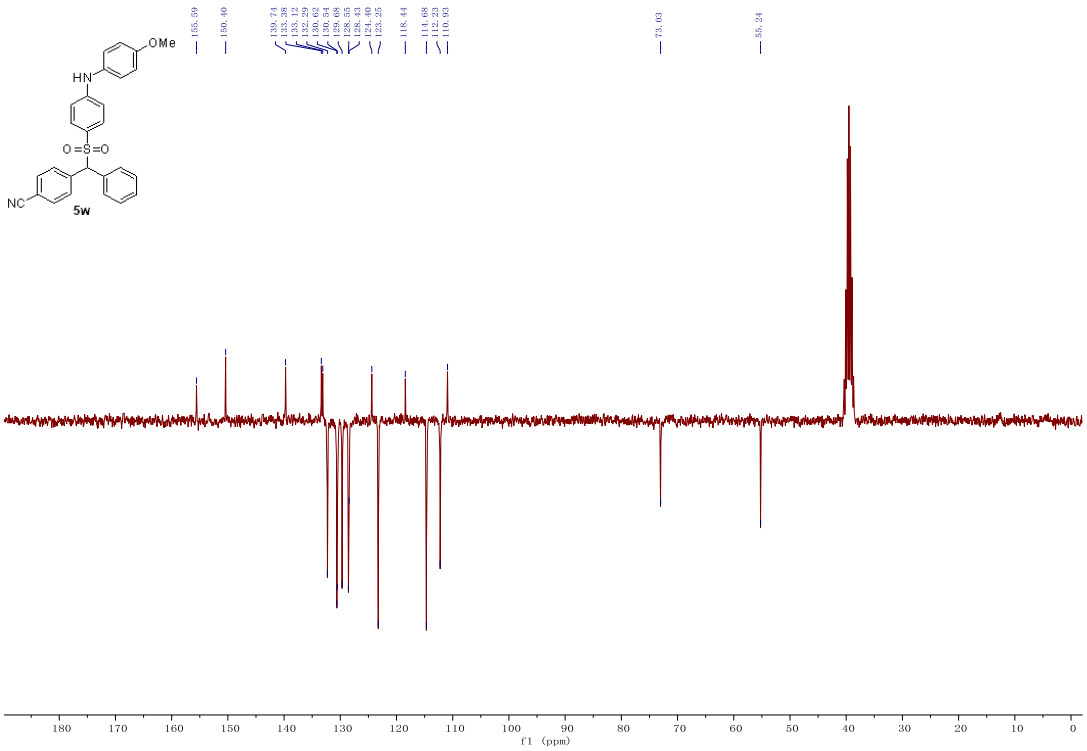

OMe
HN
O=S=O
MeO
5u
8.68
7.57
7.55
7.49
7.46
7.35
7.33
7.31
7.26
7.10
7.07
6.94
6.91
6.88
6.79
6.76
5.75
3.74
3.72
0.97
2.01
2.09
5.01
2.04
4.04
2.02
1.00
3.04
3.07
f1 (ppm)

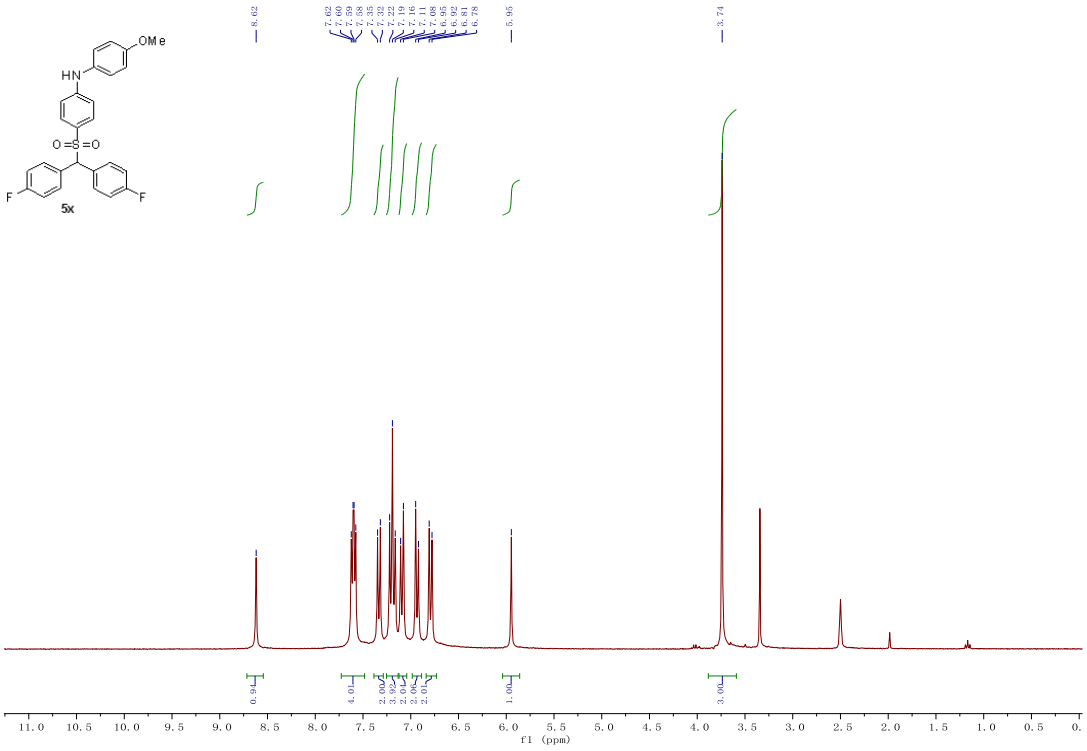

OMe
HN
O=S=O
MeO
5u
159.01
155.46
150.03
134.63
133.29
131.00
130.44
129.66
128.32
127.66
126.06
125.17
125.09
114.67
113.77
112.19
73.09
55.23
55.08
f1 (ppm)

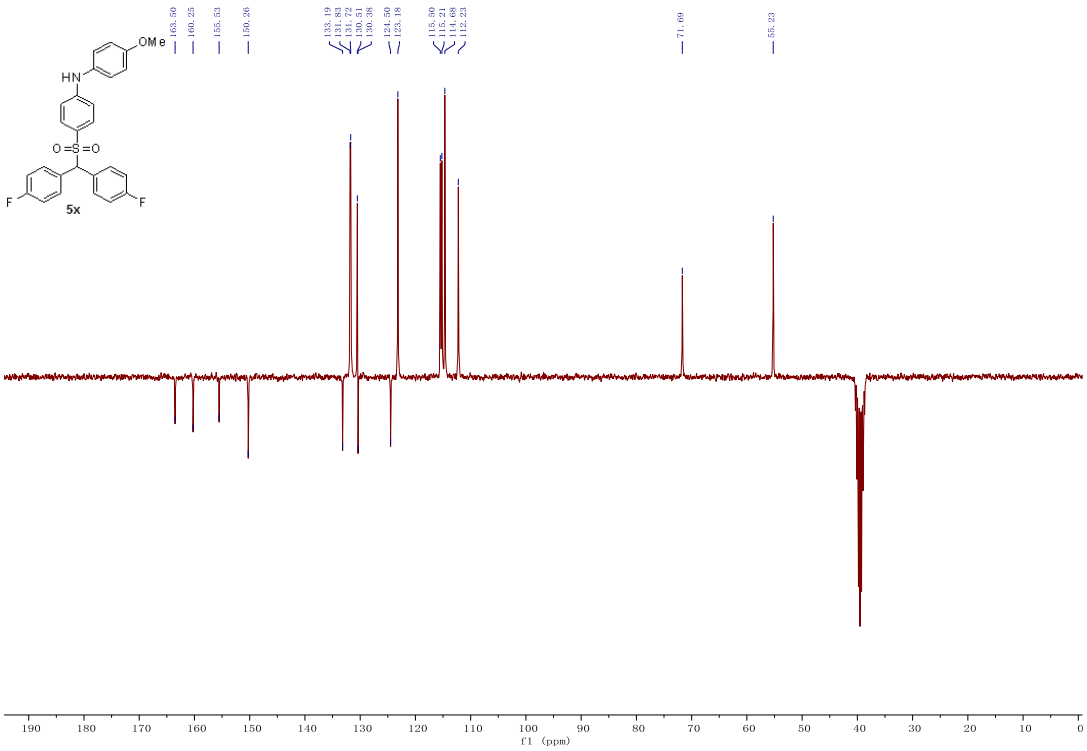

OMe
HN
O=S=O
Me
5v
8.88
7.57
7.56
7.54
7.46
7.44
7.36
7.33
7.30
7.28
7.15
7.13
7.10
7.07
6.94
6.92
6.89
6.76
5.76
3.74
2.96
0.97
2.14
2.09
1.00
2.18
2.15
2.02
2.04
1.04
3.02
3.06
f1 (ppm)

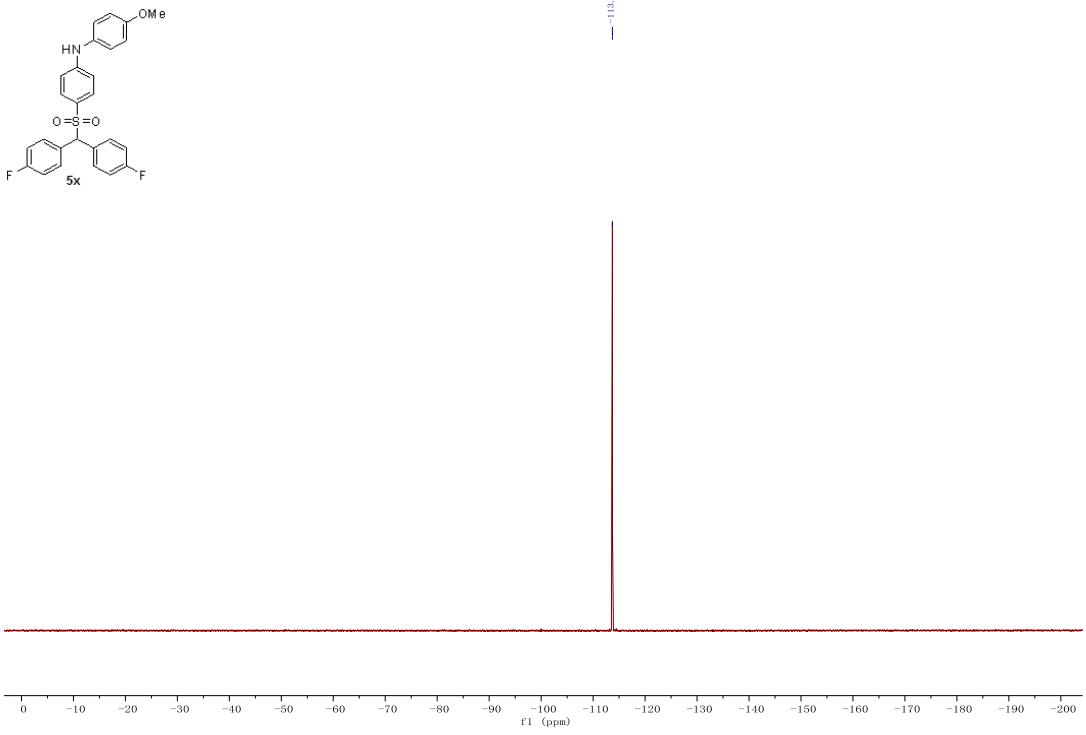

OMe
HN
O=S=O
Me
5v
155.48
150.06
137.40
134.67
133.28
131.28
130.45
130.22
129.61
128.93
128.31
126.01
125.15
123.11
114.67
112.18
73.44
55.23
20.65
f1 (ppm)

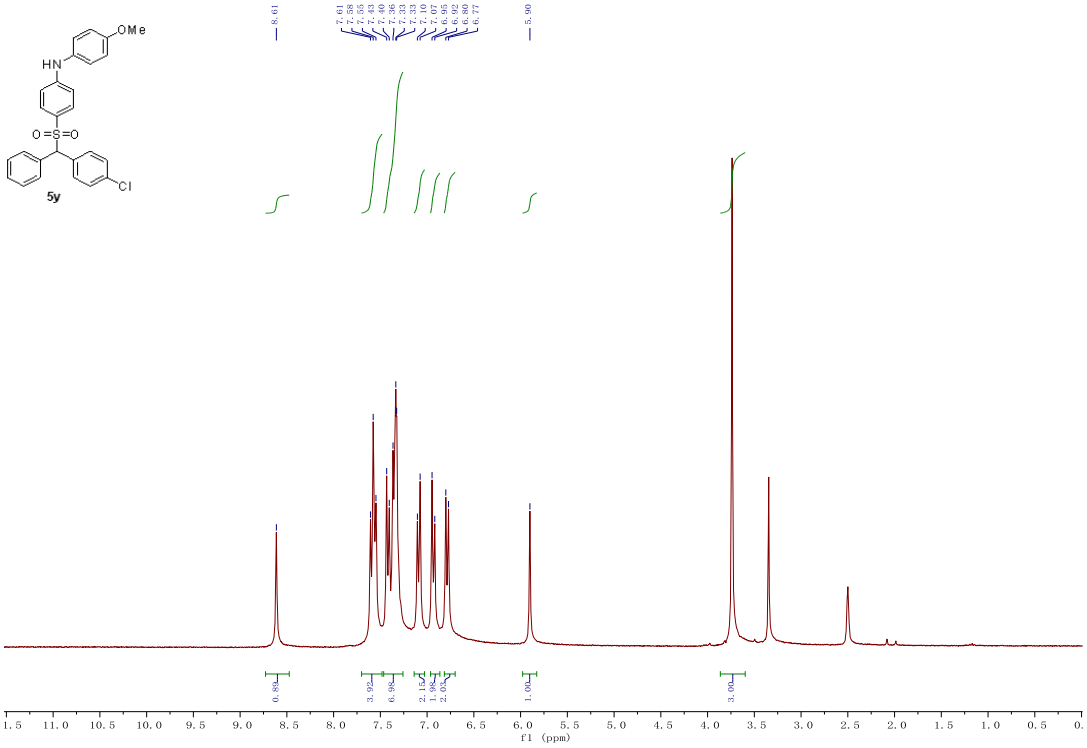

OMe
HN
O=S=O
NC
5w
8.61
7.84
7.82
7.78
7.76
7.58
7.55
7.38
7.33
7.10
6.97
6.95
6.92
6.79
6.76
6.02
3.74
0.96
4.07
2.08
5.05
2.05
2.04
2.03
1.00
3.06
f1 (ppm)

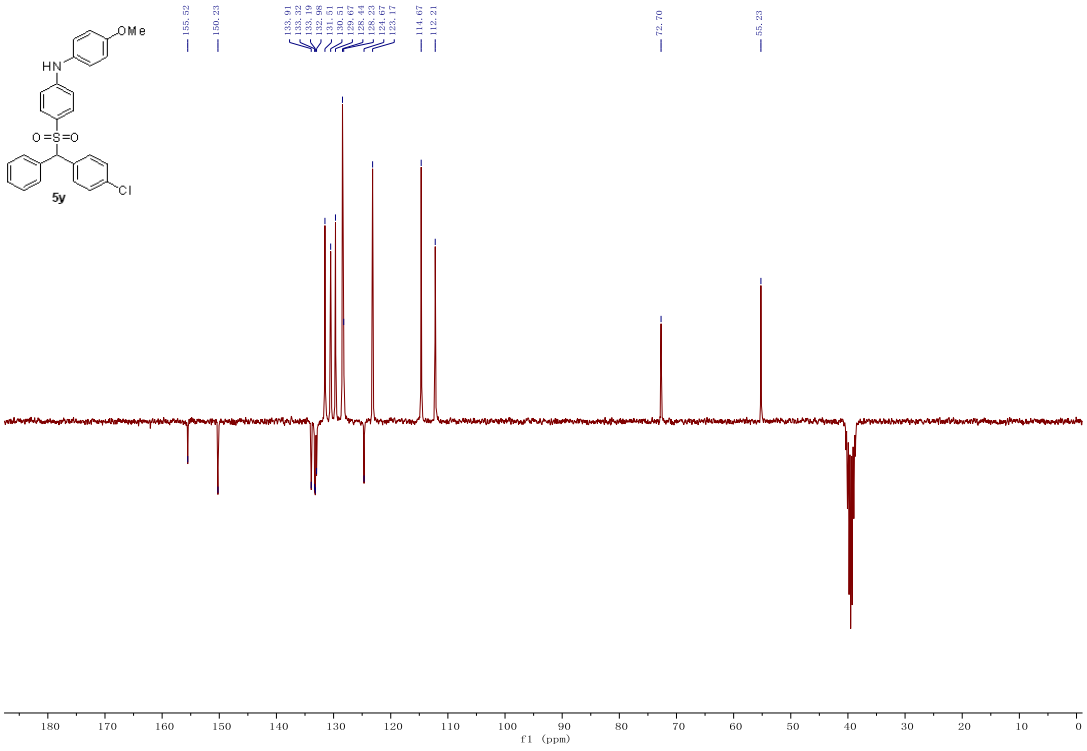

OMe
HN
O=S=O
NC
5w
155.59
150.40
139.74
133.38
133.12
132.39
130.62
130.54
129.68
128.55
128.43
124.40
123.25
118.44
114.68
112.23
110.93
73.03
55.24
f1 (ppm)

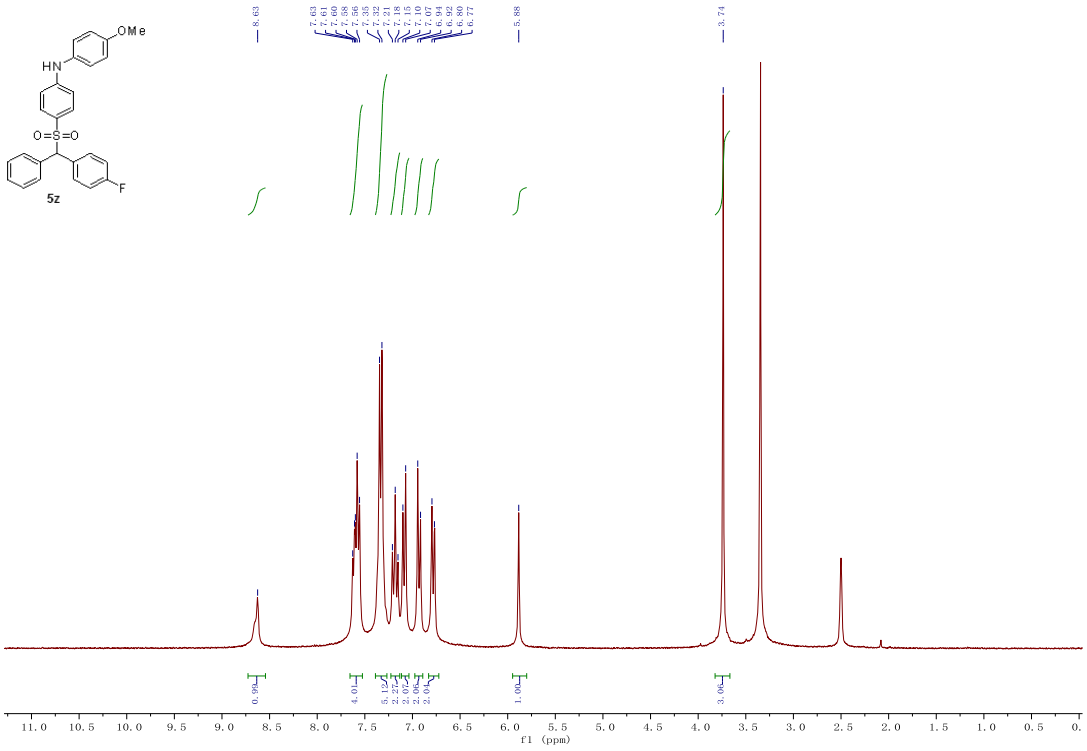

OMe
HN
O=S=O
F
F
5x
8.62
7.62
7.60
7.59
7.38
7.35
7.32
7.22
7.19
7.16
7.11
7.08
6.95
6.92
6.81
6.78
5.95
3.74
0.94
4.01
2.00
3.92
2.04
2.06
2.01
1.00
3.00
f1 (ppm)

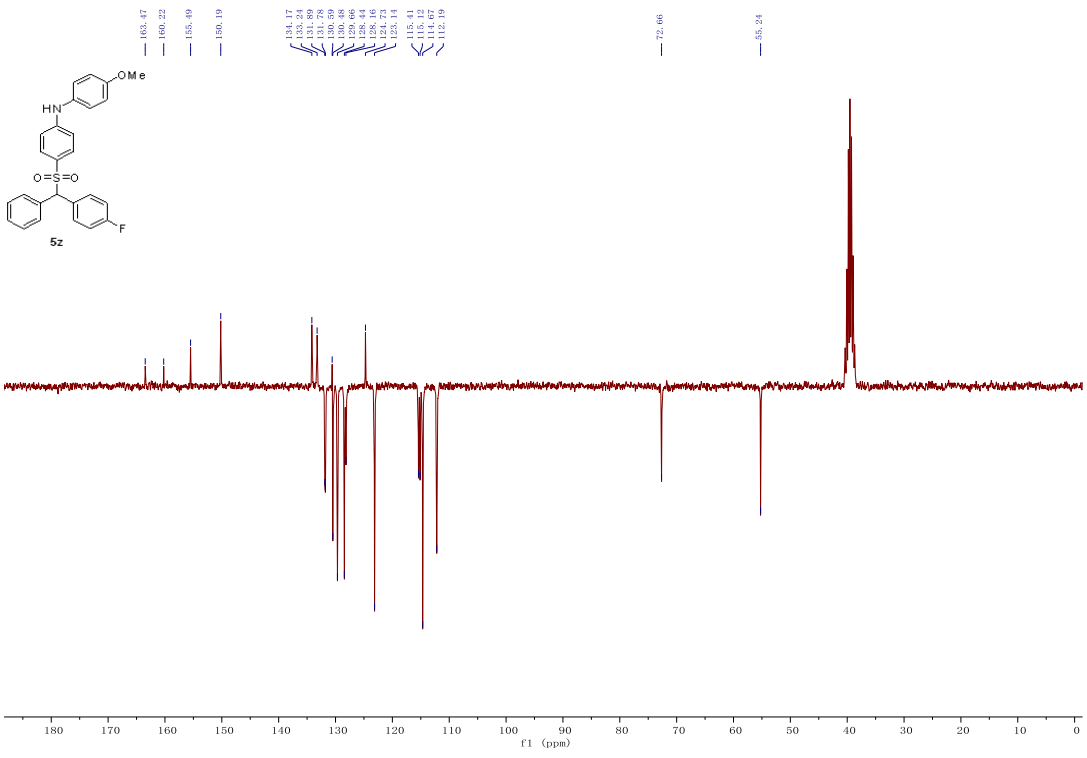

OMe
HN
O=S=O
F
F
5x
163.50
160.25
155.53
150.26
133.19
131.83
131.72
130.51
130.38
124.50
123.18
115.50
115.21
114.68
112.23
71.69
55.23
f1 (ppm)

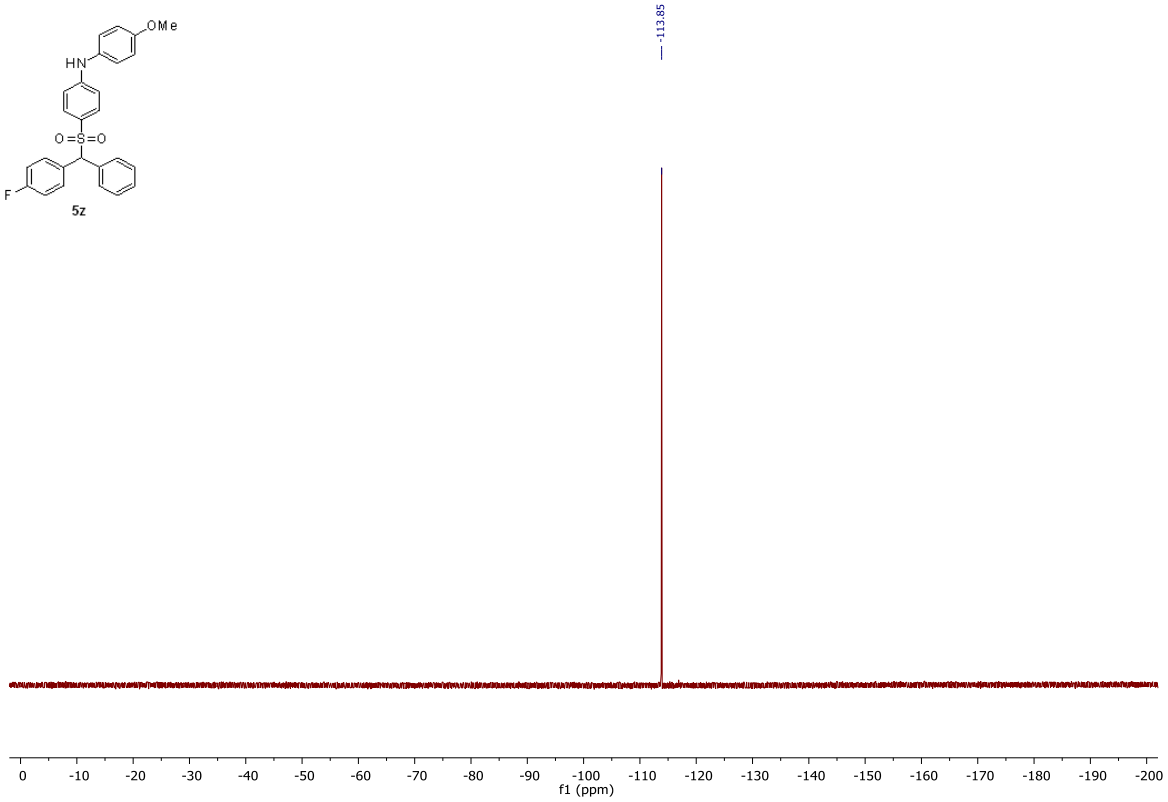

OMe
HN
O=S=O
F
F
5x
-113.71
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

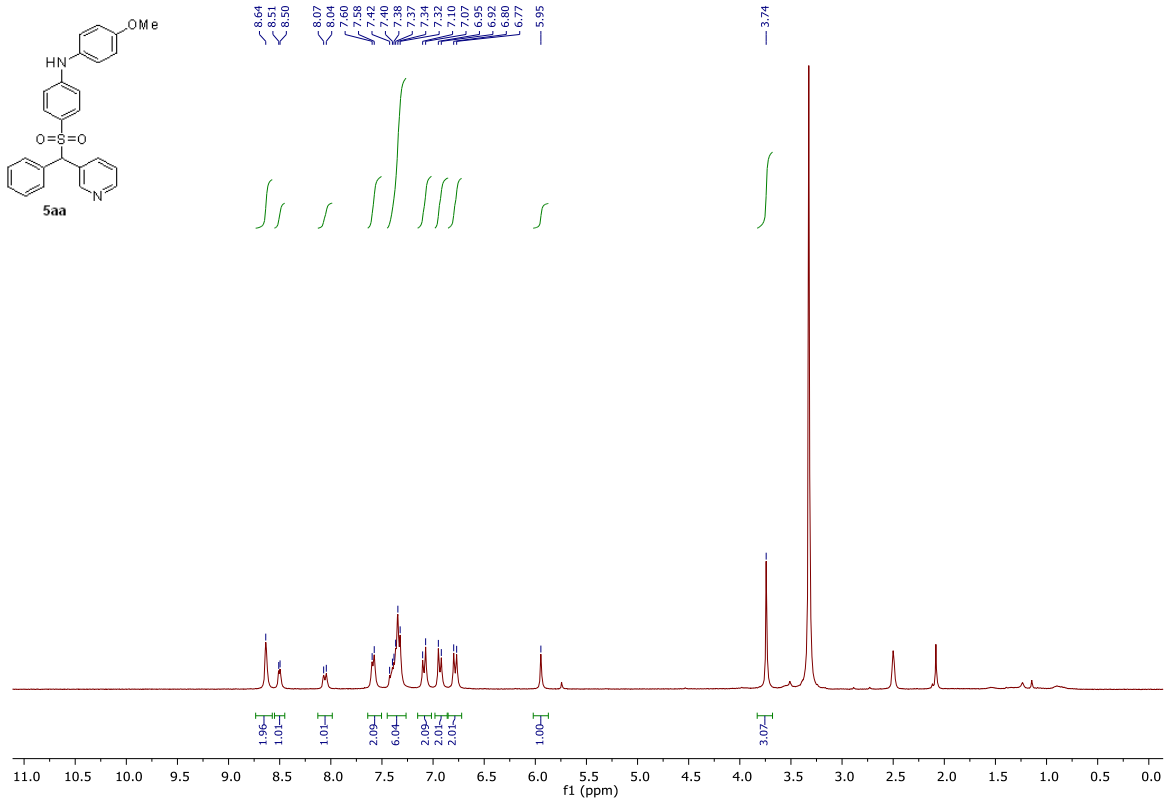

OMe
HN
O=S=O
Cl
5y
8.61
7.61
7.58
7.55
7.43
7.40
7.38
7.33
7.10
7.07
6.95
6.92
6.80
6.77
5.90
0.89
3.92
6.98
2.15
1.98
2.05
1.00
3.00
f1 (ppm)

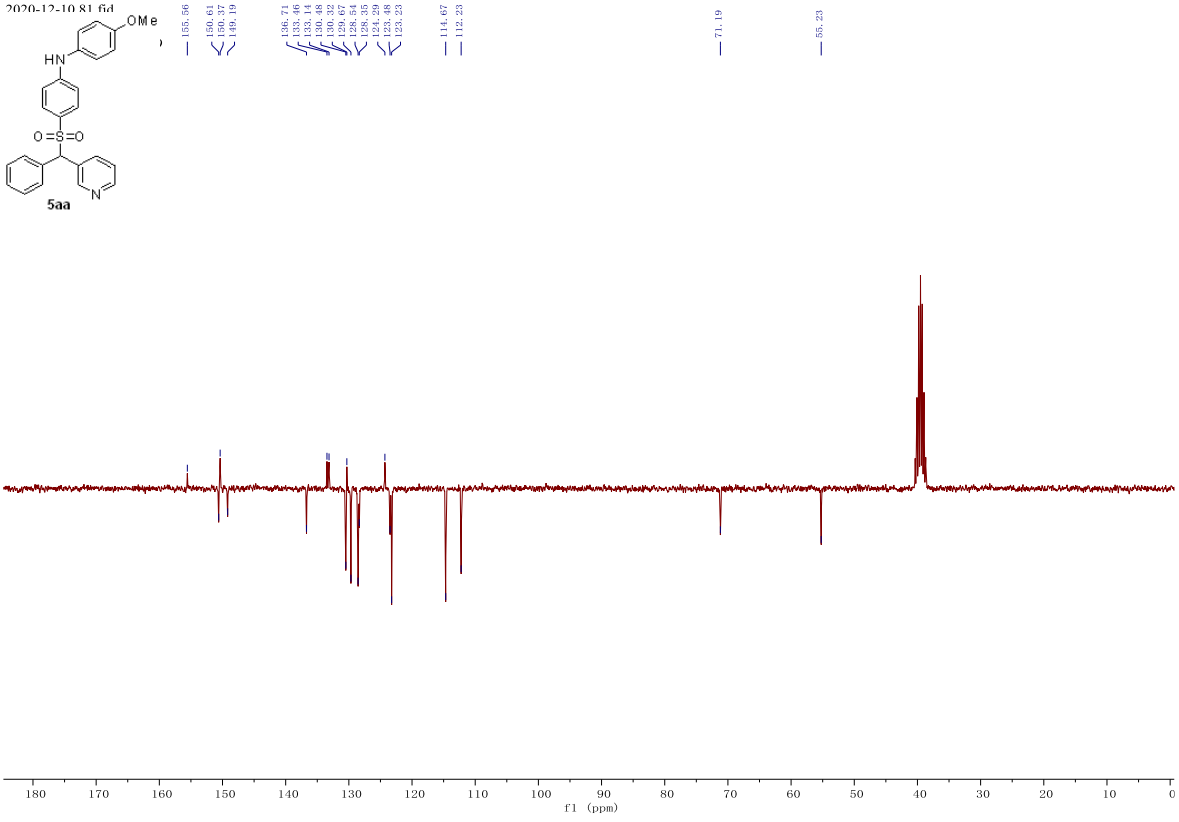

OMe
HN
O=S=O
Cl
5y
155.52
150.23
133.91
133.32
133.19
132.98
131.51
130.51
129.67
129.44
128.23
124.67
123.17
114.67
112.21
72.70
55.23
f1 (ppm)

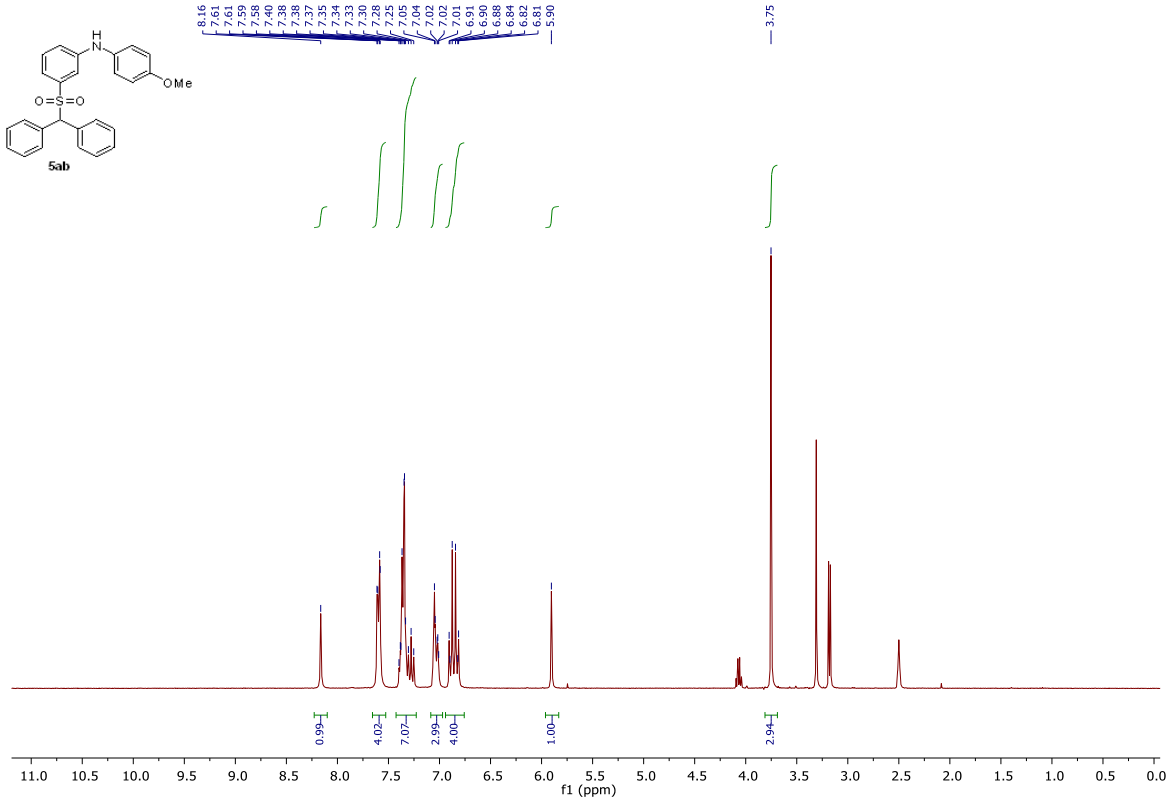

OMe
HN
O=S=O
F
5z
8.63
7.63
7.61
7.60
7.58
7.56
7.35
7.32
7.31
7.18
7.15
7.10
7.07
6.94
6.92
6.89
6.77
5.88
3.74
0.99
4.01
5.12
2.27
2.07
2.06
2.04
1.00
3.06
f1 (ppm)

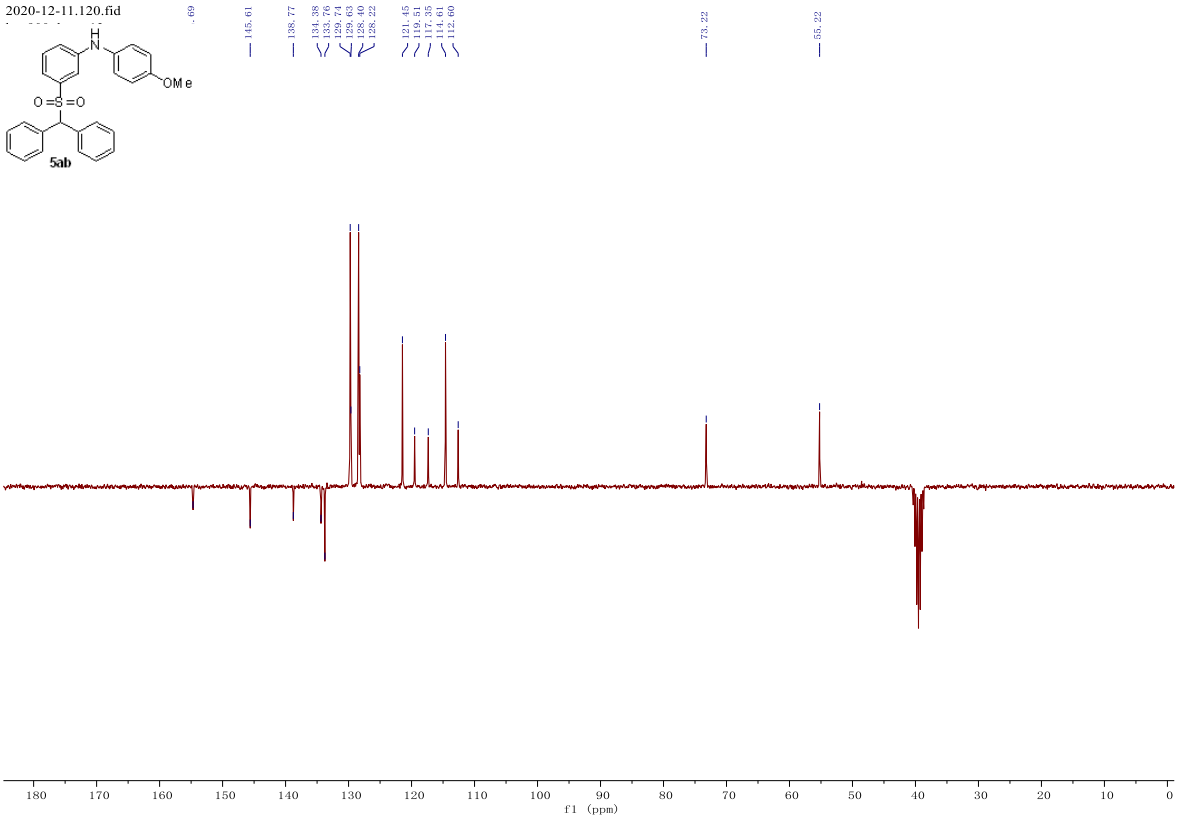

OMe
HN
O=S=O
F
5z
165.47
160.22
155.49
150.19
134.17
133.24
131.89
131.78
130.59
130.48
129.66
128.44
126.16
124.73
123.14
115.41
115.12
114.67
112.19
72.66
55.24
f1 (ppm)

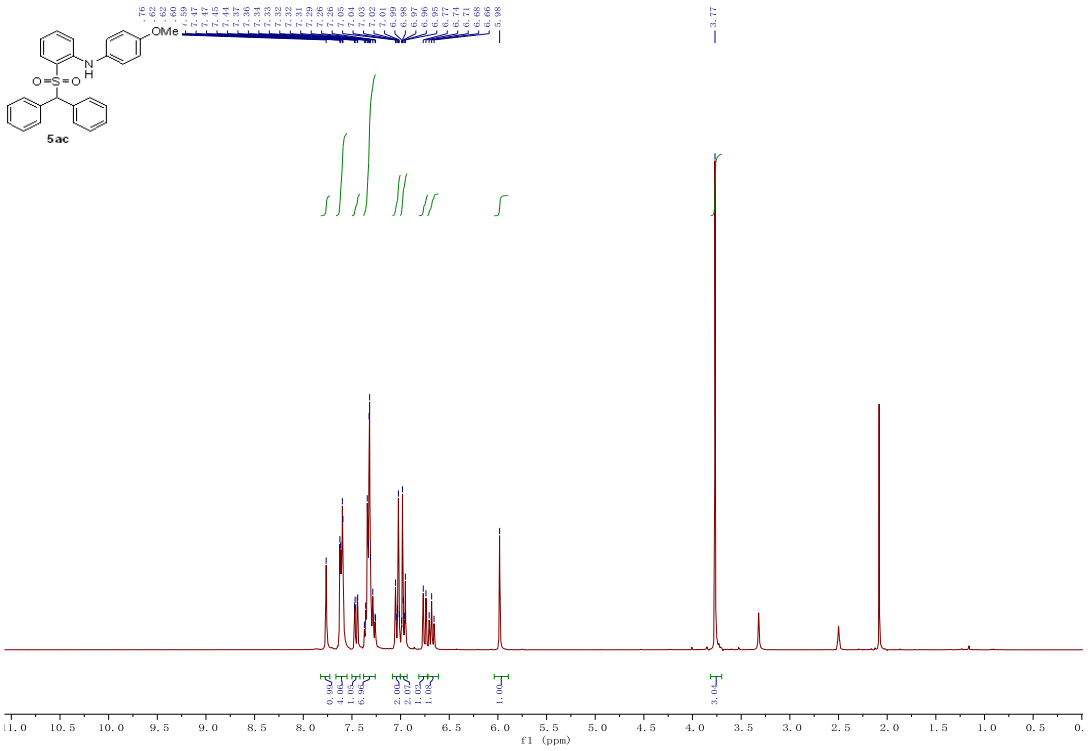

OMe
HN
O=S=O
F
5z
-113.85
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

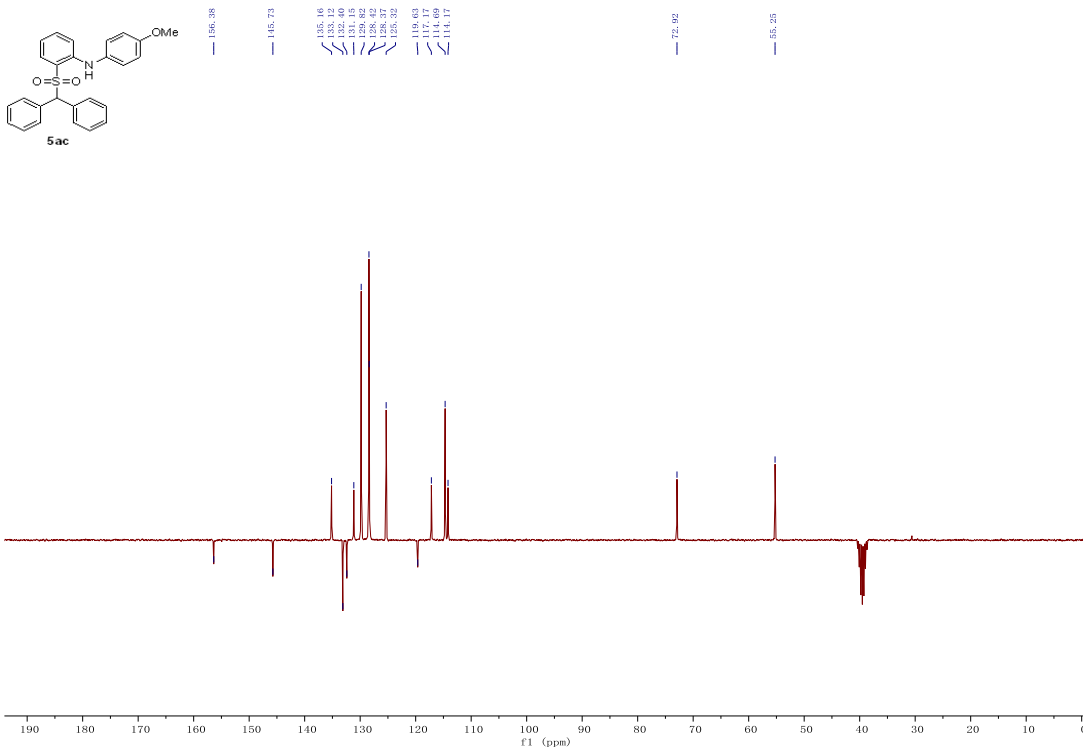

OMe
HN
O=S=O
N
5aa
8.64
8.51
8.50
8.07
8.04
7.60
7.58
7.42
7.40
7.38
7.37
7.34
7.32
7.10
7.07
6.95
6.92
6.80
6.77
5.95
3.74
1.96
1.01
1.01
2.09
6.04
2.09
2.01
2.01
1.00
3.07
f1 (ppm)

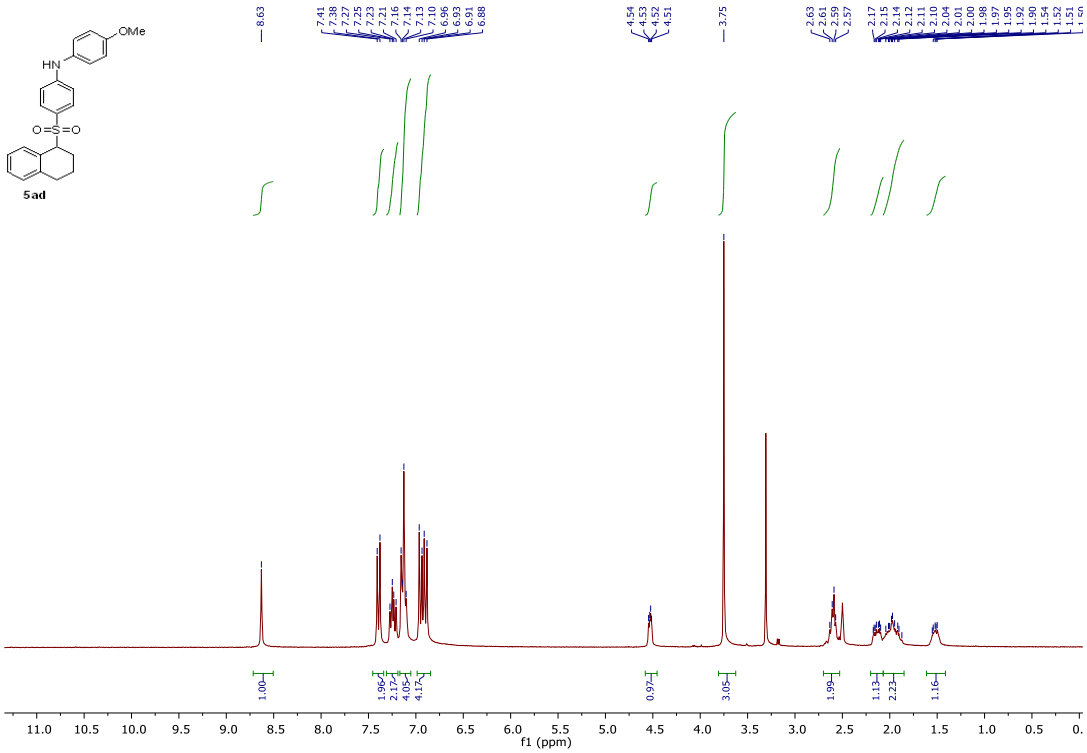

OMe
HN
O=S=O
N
5aa
155.56
150.61
150.37
149.19
136.71
133.46
133.14
130.48
130.32
129.67
128.54
126.35
124.29
123.48
123.23
114.67
112.23
71.19
55.23
f1 (ppm)

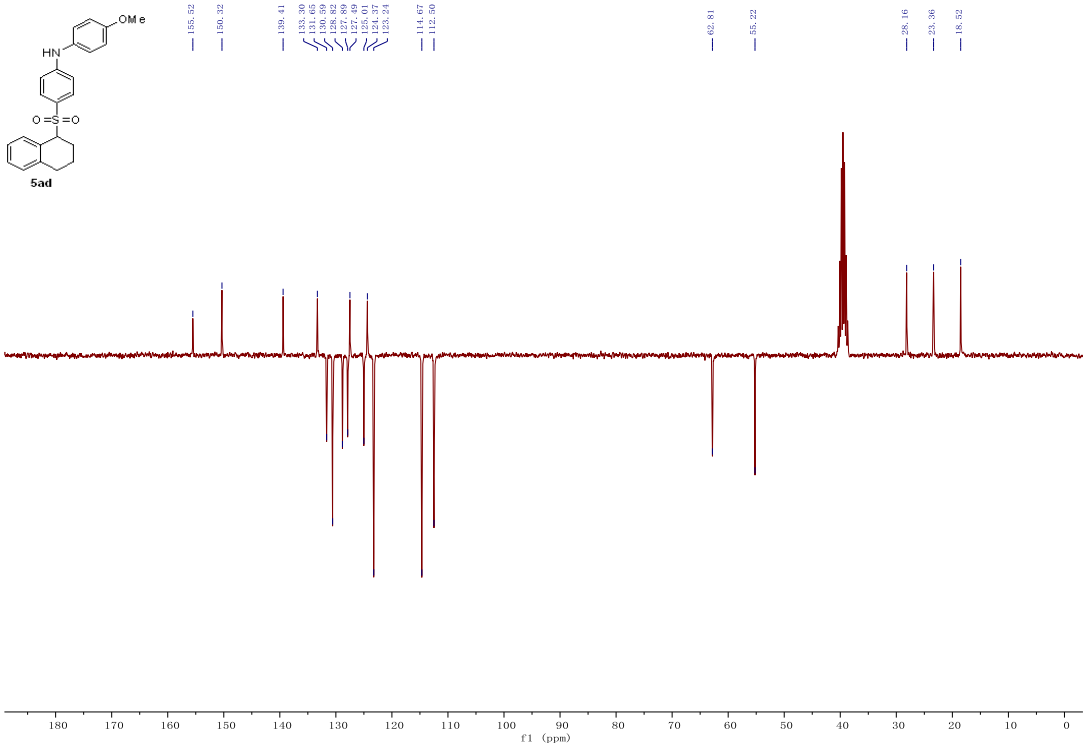

8.16
7.61
7.61
7.59
7.58
7.40
7.38
7.38
7.37
7.35
7.34
7.33
7.30
7.28
7.25
7.05
7.04
7.02
7.02
7.01
6.91
6.90
6.88
6.84
6.82
6.81
5.90
3.75
H
N
OMe
O=S=O
5ab
0.99
4.02
7.07
2.99
4.00
1.00
2.94
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

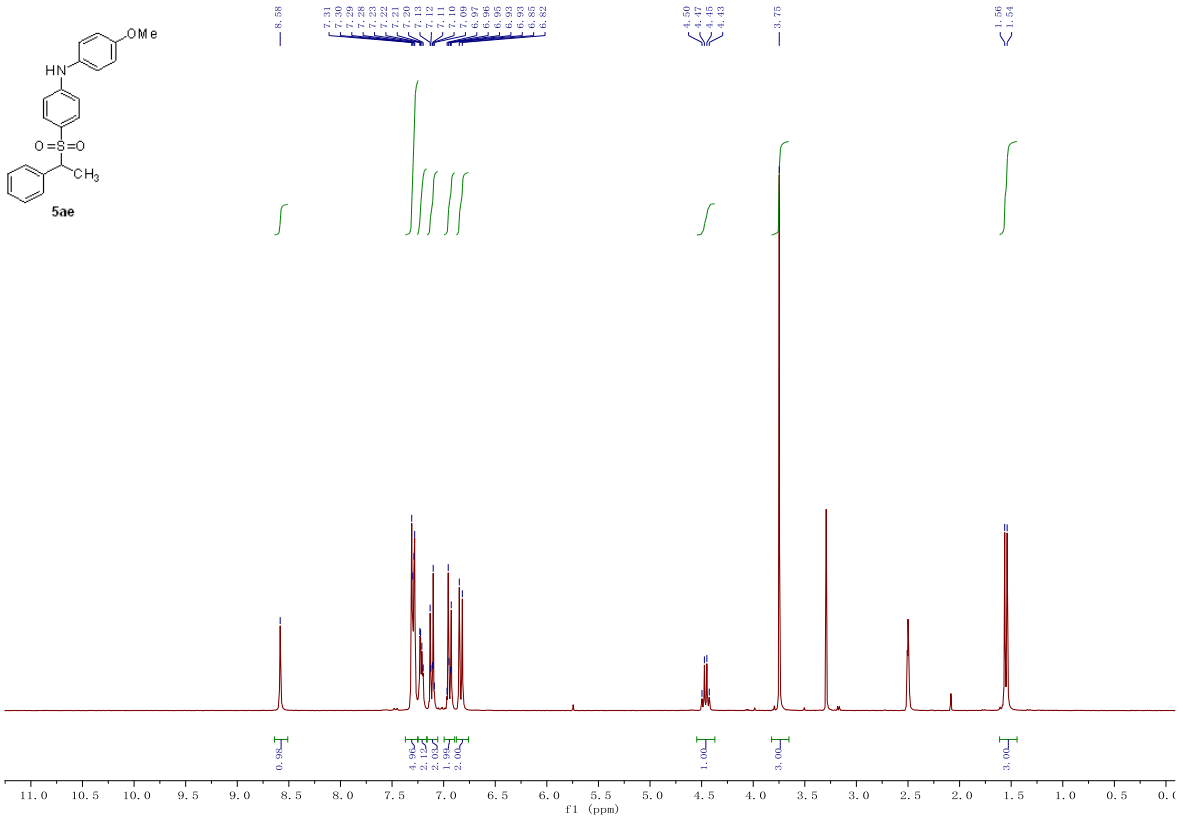

2020-12-11.120.fid
145.61
138.77
134.38
133.76
129.74
129.65
128.40
128.22
121.45
119.51
117.35
114.61
112.60
73.22
55.22
H
N
OMe
O=S=O
5ab
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

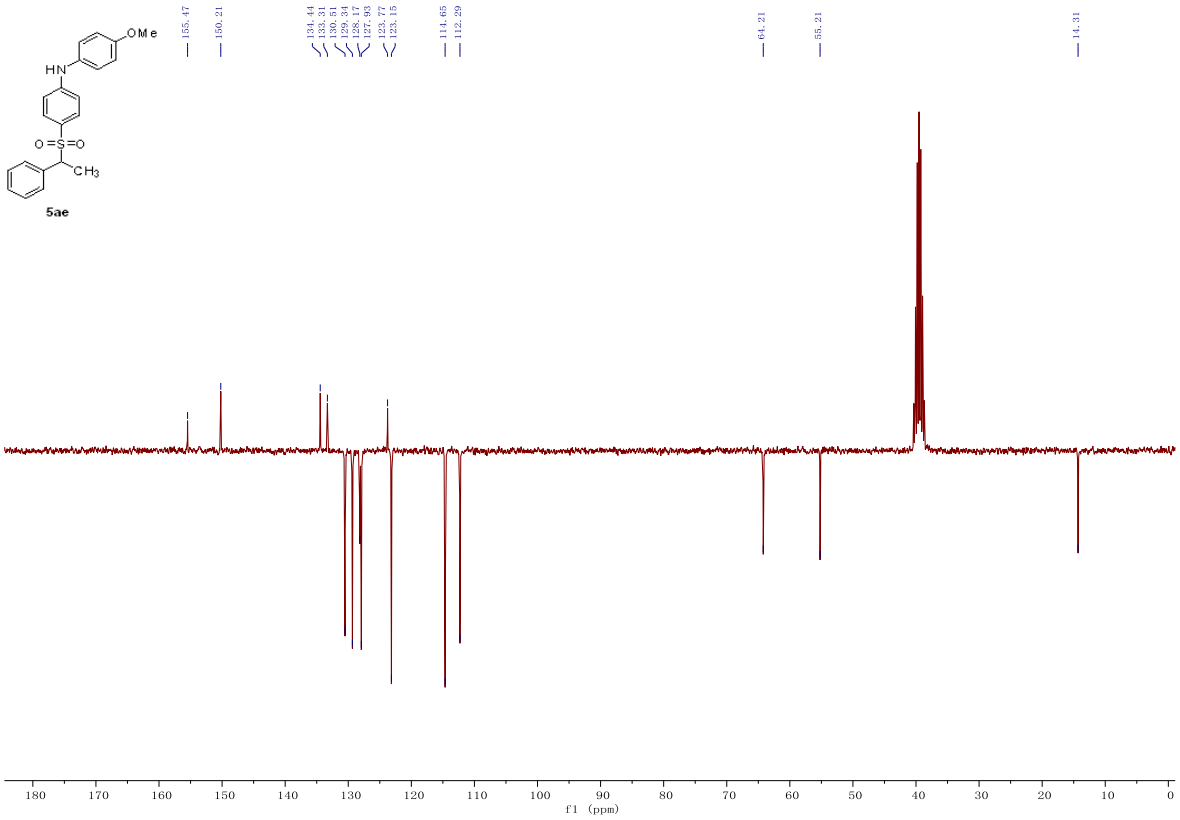

OMe
N
H
O=S=O
5ac
7.76
7.62
7.62
7.60
7.59
7.49
7.47
7.47
7.45
7.44
7.37
7.36
7.34
7.33
7.32
7.32
7.31
7.29
7.28
7.26
7.05
7.04
7.03
7.02
7.01
6.99
6.98
6.97
6.96
6.95
6.77
6.74
6.71
6.68
6.66
5.98
3.77
0.90
4.00
1.05
6.98
2.00
2.02
1.02
1.08
1.00
3.04
f1 (ppm)

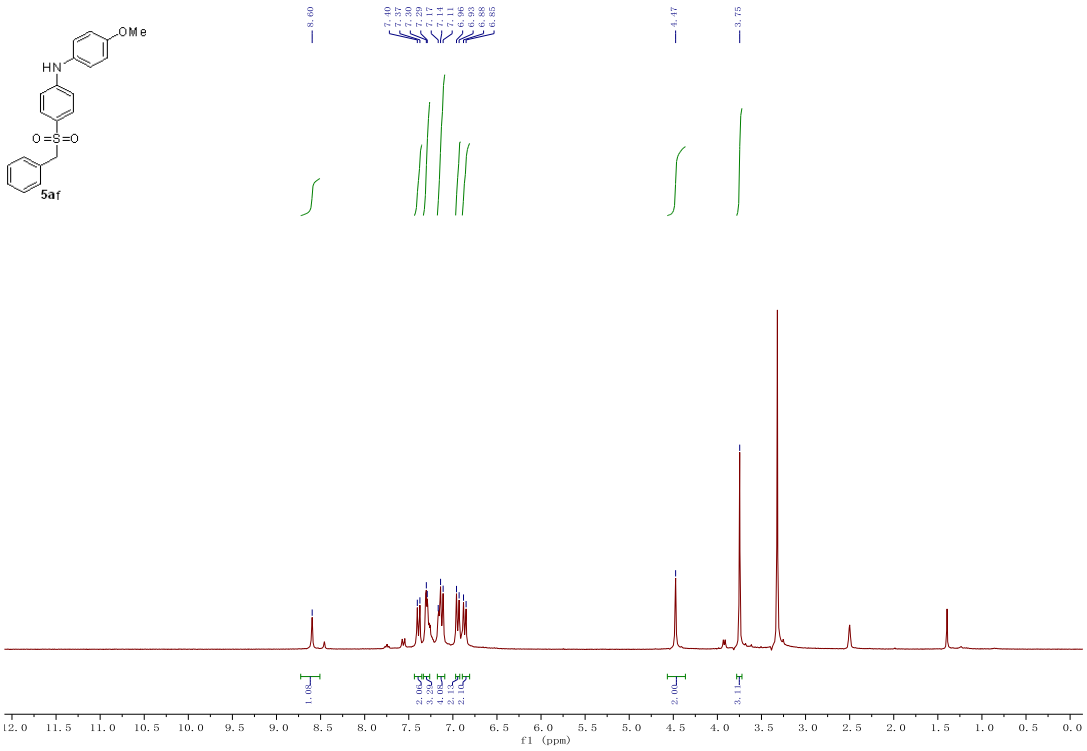

OMe
N
H
O=S=O
5ac
156.38
145.73
135.16
133.12
132.40
131.15
129.82
128.42
128.37
125.32
119.63
117.17
114.69
114.17
72.92
55.25
f1 (ppm)

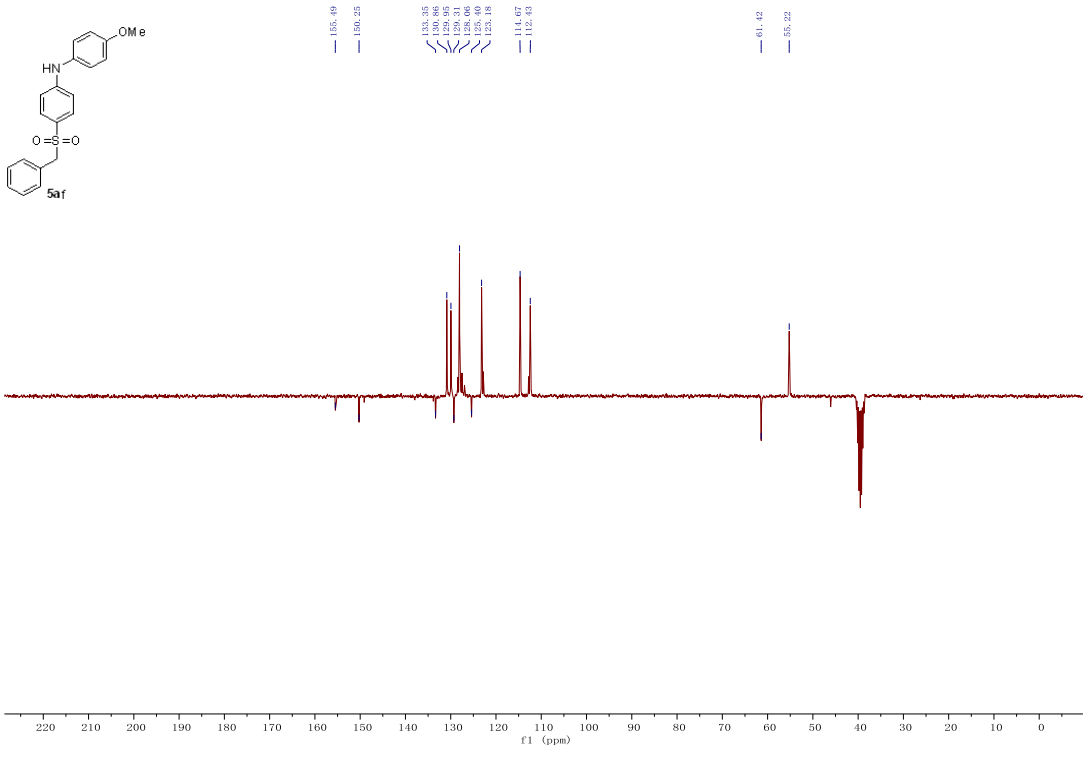

OMe
HN
O=S=O
5ad
8.63
7.41
7.38
7.27
7.25
7.23
7.21
7.16
7.14
7.13
7.10
6.96
6.93
6.91
6.88
4.54
4.53
4.52
4.51
3.75
2.63
2.61
2.59
2.57
2.17
2.15
2.14
2.12
2.11
2.10
2.04
2.01
2.00
1.98
1.97
1.95
1.92
1.90
1.54
1.52
1.51
1.00
1.96
2.13
4.05
4.17
0.97
3.05
1.99
1.13
2.23
1.16
f1 (ppm)

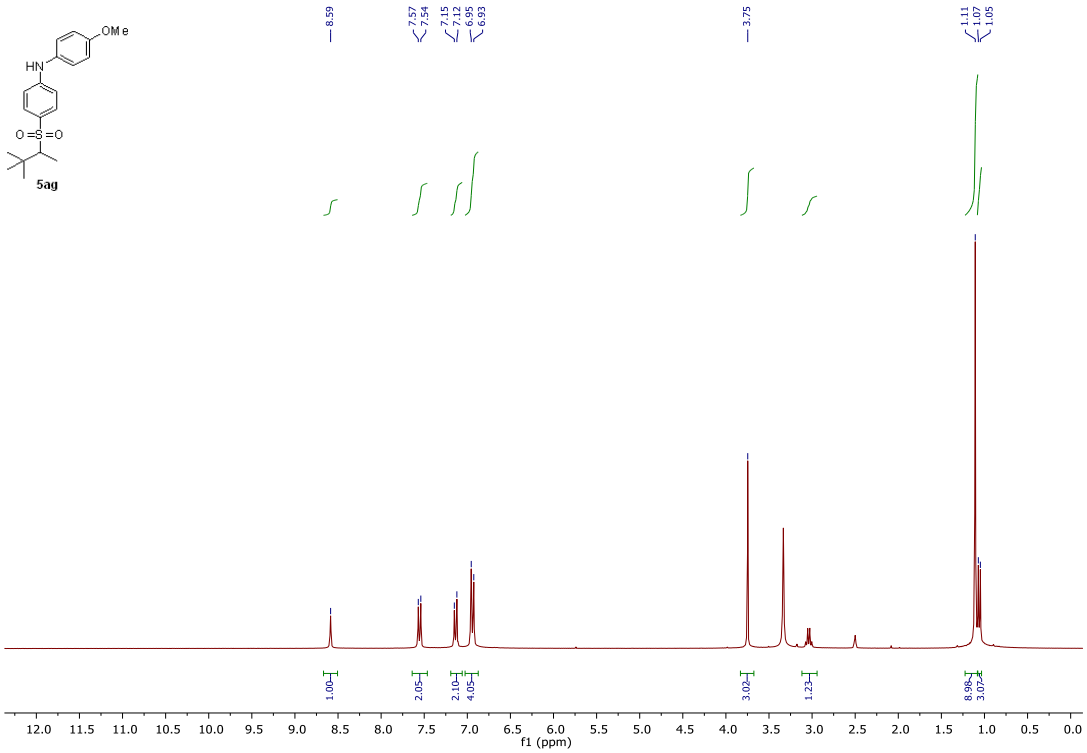

OMe
HN
O=S=O
5ad
155.52
150.32
139.41
133.30
131.65
130.59
128.82
127.89
127.49
126.01
124.37
123.24
114.67
112.50
62.81
55.22
28.16
23.36
18.52
f1 (ppm)

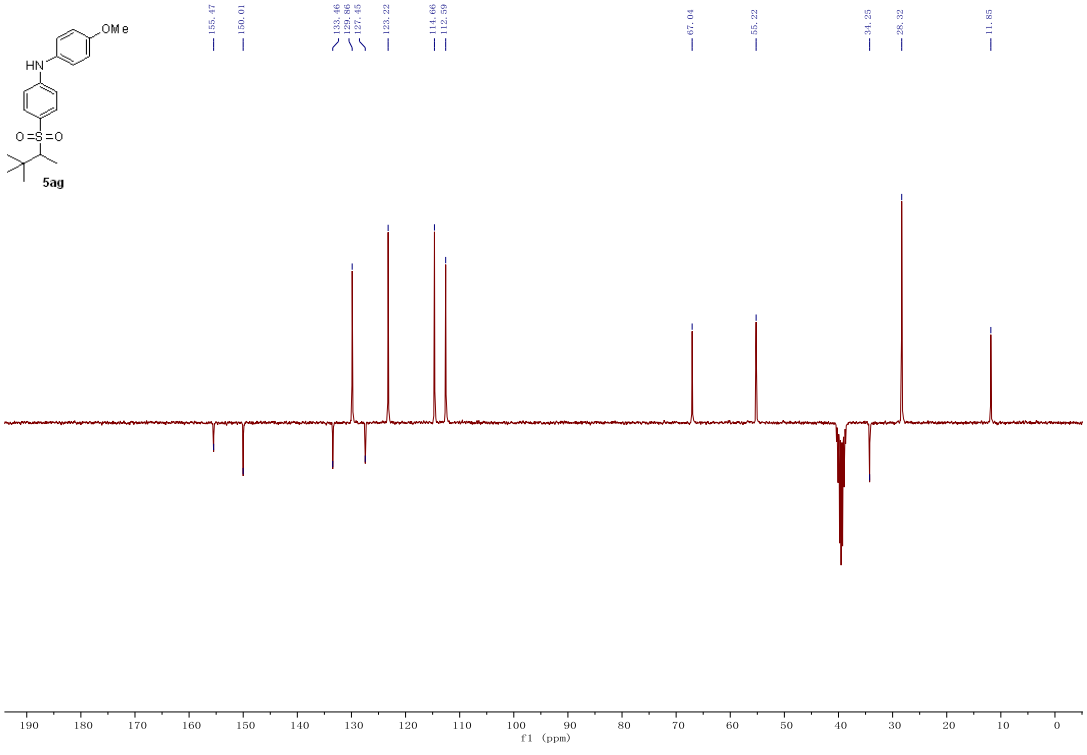

OMe
HN
O=S=O
CH3
5ae
8.58
7.31
7.30
7.29
7.28
7.23
7.22
7.21
7.20
7.13
7.12
7.11
7.10
7.09
6.97
6.96
6.95
6.93
6.93
6.85
6.82
4.50
4.47
4.45
4.43
3.75
1.56
1.54
0.98
4.90
2.15
2.04
1.98
2.00
1.00
3.00
3.00
f1 (ppm)

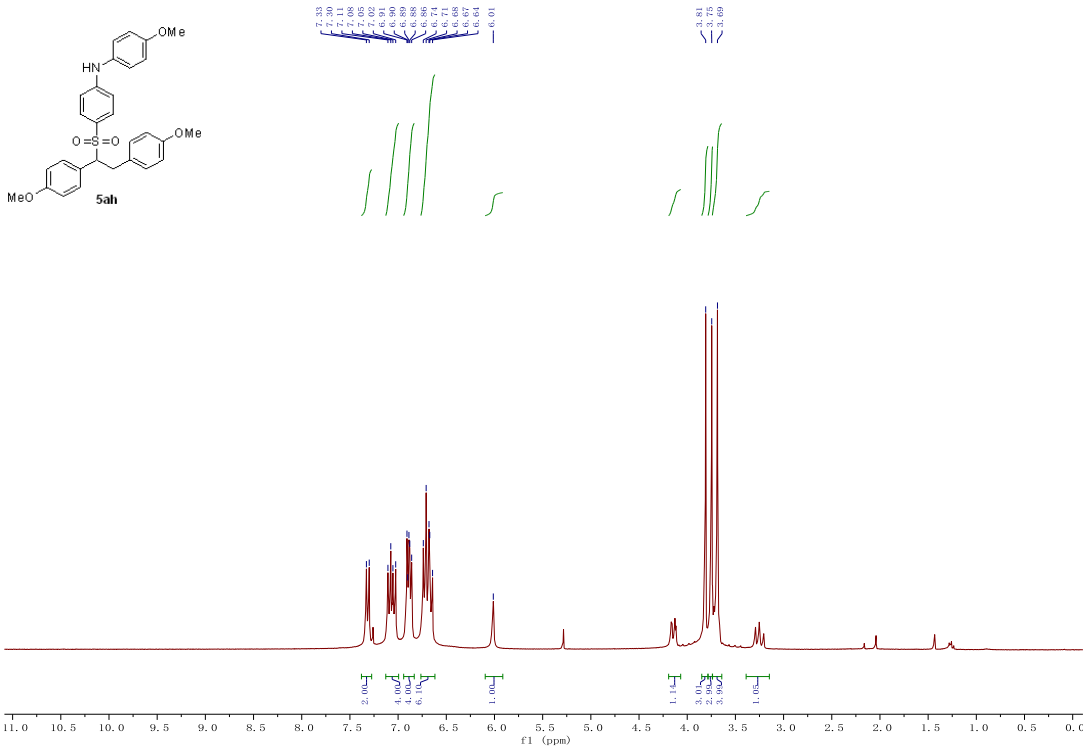

OMe
HN
O=S=O
CH3
5ae
155.47
150.21
134.44
133.31
130.51
129.34
128.17
127.93
123.77
123.15
114.65
112.29
64.21
55.21
14.31
f1 (ppm)

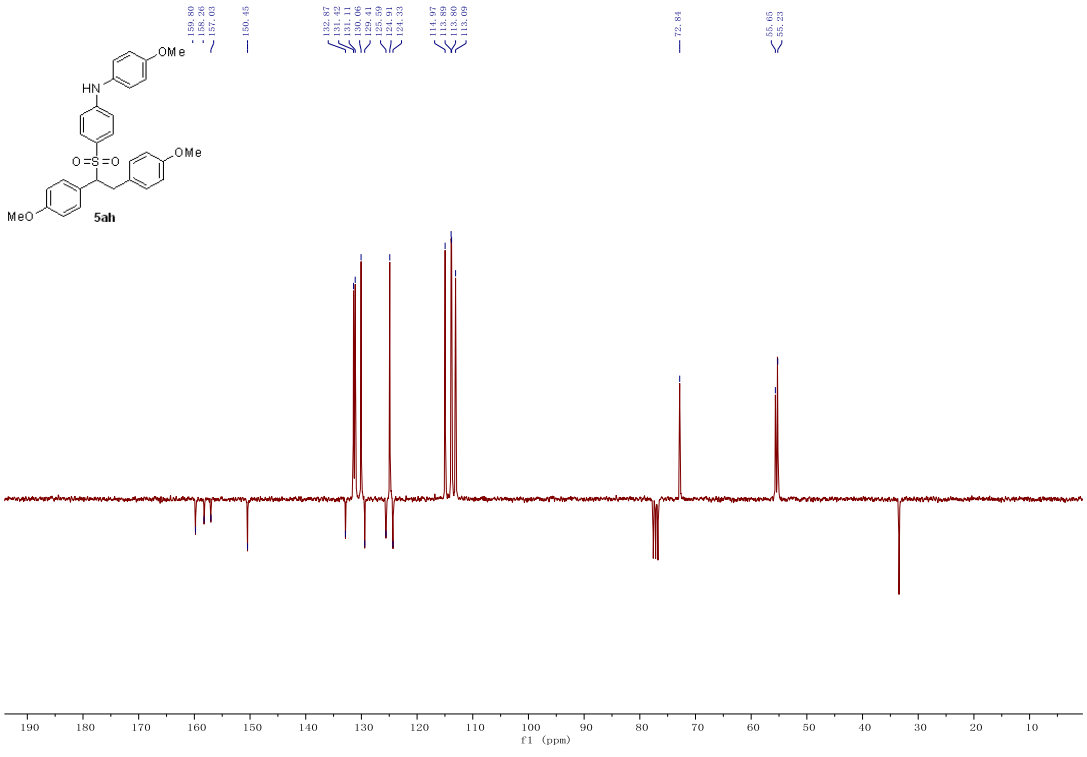

OMe
HN
O=S=O
5af
8.60
7.40
7.37
7.30
7.29
7.17
7.14
7.11
6.96
6.93
6.88
6.85
4.47
3.75
1.08
2.00
3.20
4.08
2.12
2.10
2.00
3.11
f1 (ppm)


OMe
HN
O=S=O
5af
155.49
150.25
133.35
130.86
129.95
129.31
128.06
125.69
123.18
114.67
112.43
61.42
55.22
f1 (ppm)

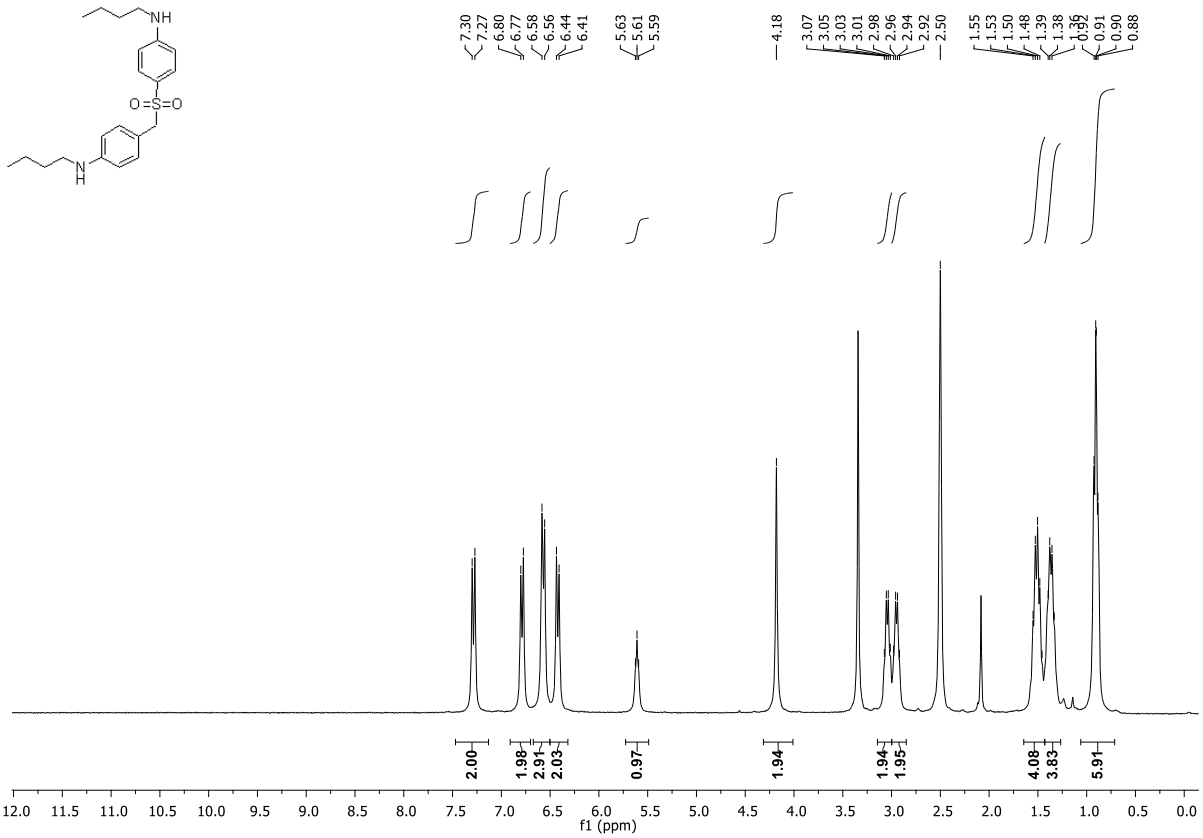

OMe
HN
O=S=O
5ag
8.59
7.57
7.54
7.15
7.12
6.95
6.93
3.75
1.11
1.07
1.05
1.00
2.05
2.10
4.05
3.02
1.23
8.98
3.07
f1 (ppm)


OMe
HN
O=S=O
5ag
155.47
150.01
133.46
129.86
127.45
123.22
114.66
112.59
67.04
55.22
34.25
28.32
11.85
f1 (ppm)

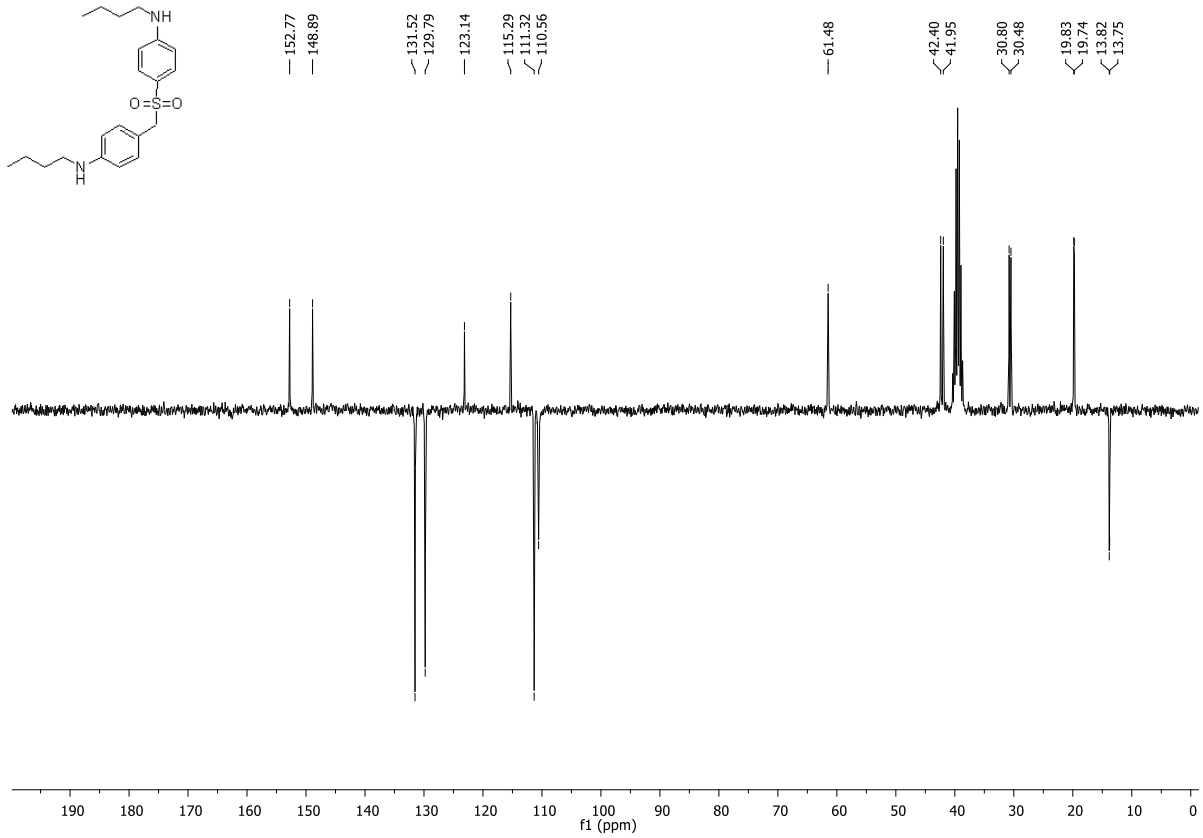

OMe
HN
O=S=O
OMe
MeO
5ah
7.33
7.30
7.11
7.08
7.05
7.02
6.91
6.90
6.89
6.88
6.86
6.74
6.71
6.68
6.67
6.64
6.01
3.81
3.75
3.69
2.00
4.00
4.03
6.10
1.00
1.14
3.01
2.99
3.00
1.05
f1 (ppm)


OMe
HN
O=S=O
OMe
MeO
5ah
159.80
158.28
157.03
150.45
132.67
131.82
131.11
130.06
129.41
125.59
124.91
124.33
114.97
113.89
113.80
113.09
72.84
55.65
55.23
f1 (ppm)

5ai


7.30
7.27
6.80
6.77
6.58
6.56
6.44
6.41
5.63
5.61
5.59
4.18
3.07
3.05
3.03
3.01
2.98
2.96
2.94
2.92
2.50
1.55
1.53
1.50
1.48
1.39
1.38
1.36
0.92
0.91
0.90
0.88
2.00
1.98
2.91
2.03
0.97
1.94
1.94
1.95
4.08
3.83
5.91
f1 (ppm)

5ai


152.77
148.89
131.52
129.79
123.14
115.29
111.32
110.56
61.48
42.40
41.95
30.80
30.48
19.83
19.74
13.82
13.75
f1 (ppm)

1. X.-W. Feng, J. Wang, J. Zhang, J. Yang, N. Wang and X.-Q. Yu, *Org. Lett.*, 2010, **12**, 4408-4411.
2. S. Hu, S. Du, Z. Yang, L. Ni and Z. Chen, *Adv. Synth. Catal.*, 2019, **361**, 3124-3136.
3. D. Choudhary, V. Khatri and A. K. Basak, *Org Lett*, 2018, **20**, 1703-1706.