Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information

Copper-mediated sulfonylation of aryl iodides and bromides with arylsulfonyl hydrazides in PEG-400

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General information

Hydrogen nuclear magnetic resonance spectra (^{1}H NMR) was obtained at 300 MHz. Spectra was recorded in $CDCl_3$ solutions. Carbon-13 nuclear magnetic resonance spectra (^{13}C NMR) was obtained at 75 MHz. Spectra was recorded in $CDCl_3$ solutions. Chemical shifts are reported in ppm relative to the TMS (^{1}H NMR) and to the solvent (^{13}C NMR). Melting points were obtained of a XT4A melting point apparatus and were uncorrected. Gas chromatography mass spectra (GC/MS) were recorded on a Saturn 2000GC/MS instrument. Thin layer chromatography (TLC) was performed using Merck Silica Gel GF254, 0.25 mm thickness.

Experimental procedure

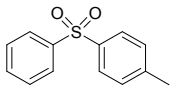
Preparation of arylsulfonyl hydrazides

Arylsulfonyl hydrazides were prepared according to a literature procedure[1]. Hydrazine monohydrate (80%) (275 mg, 4.4 mmol) was added water (260 mg) and was cooled to 0 °C. To this solution was added dropwise a solution of arylsulfonyl chloride (2.0 mmol) in THF (10 ml) at 0 °C. The mixture was further stirred at 0 °C for 30 min., followed by addition of diethyl ether (10 ml). The mixture was extracted with saturated brine (3 × 10 ml). The organic layer was dried over sodium sulfate, filtered through Celite. The combined organic extracts were concentrated and the resulting residue was purified by column chromatography on silica gel to provide the desired product.

General procedure for the coupling of aryl halides and arylsulfonyl hydrazide

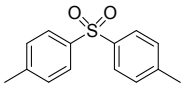
Aryl halide (1.2 mmol), arylsulfonyl hydrazide (1.0 mmol), cupric acetate (1.0 mmol), and PEG-400 (2.0 ml) were taken in a 25 ml sealed tube. The reaction mixture was stirred at 100 ^{o}C for 12 hours in air. Water (10 ml) was added and then the mixture was extracted with EtOAc (4×10 ml). The extracts were combined and washed by brine (3×10 ml), dried over $MgSO_4$, filtered, and evaporated, and purified by chromatography on silica gel to obtain the desired products with ethyl acetate/hexane (v/v=1:3～1:10). The products were characterized by their spectral and analytical data and compared with those of the known compounds.

Analytical data



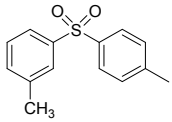
phenyl *p*-tolyl sulfone (Table 2, 3aa)[2]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.92 (d, J=6.6 Hz, 2H), 7.82 (d, J=8.1 Hz, 2H), 7.52-7.44 (m, 3H), 7.27 (d, J=8.1 Hz, 2H), 2.36 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.2, 142.0, 138.6, 133.0, 129.9, 129.2, 127.7, 127.4, 21.5. GC-MS (EI, m/z): 232 [M+].



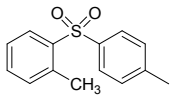
di-*p*-tolyl sulfone (Table 2, 3ab) [2]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.81 (d, J=8.4 Hz, 4H), 7.27 (d, J=8.1 Hz, 4H), 2.38 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.9, 139.0, 129.9, 127.5, 21.6. GC-MS (EI, m/z): 246[M+].



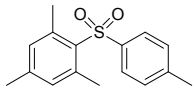
***m*-methylphenyl *p*-tolyl sulfone (Table 2, 3ac)** [2]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.82 (d, J=8.4 Hz, 2H), 7.73-7.71 (m, 2H), 7.36-7.27 (m, 4H), 2.38 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.0, 141.8, 139.5, 138.8, 133.8, 129.8, 129.1, 127.8, 127.6, 124.6, 21.5, 21.3. GC-MS (EI, m/z): 246[M+].



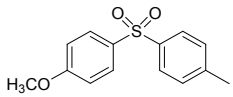
***o*-methylphenyl *p*-tolyl sulfone (Table 2, 3ad)** [3]

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.19 (d, J=7.8 Hz, 1H), 7.74 (d, J=7.8 Hz, 2H), 7.46-7.20 (m, 5H), 2.44 (s, 3H), 2.39 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.3, 143.5, 139.2, 133.6, 133.0, 129.9, 129.6, 127.8, 127.5, 127.3, 21.6, 21.5. GC-MS (EI, m/z): 246[M+].



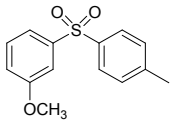
2,4,6-trimethylphenyl *p*-tolyl sulfone (Table 2, 3ae) [4]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.67 (d, J=8.4 Hz, 2H), 7.25 (d, J=7.8 Hz, 2H), 6.93 (s, 2H), 2.59 (s, 6H), 2.39 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.3, 143.2, 140.7, 140.0, 134.2, 132.2, 129.5, 126.3, 22.8, 21.5, 20.9. GC-MS (EI, m/z): 274[M+].



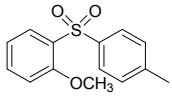
***p*-methoxyphenyl *p*-tolyl sulfone (Table 2, 3af)** [5]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.87-7.78 (m, 4H), 7.26 (d, *J*=8.1 Hz, 2H), 6.94 (d, *J*=8.8 Hz, 2H), 3.82 (s, 3H), 2.37 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 163.2, 143.8, 139.3, 133.4, 129.9, 129.6, 127.3, 114.5, 55.7, 21.5. GC-MS (EI, m/z): 262[M+].



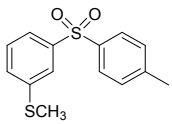
***m*-methoxyphenyl *p*-tolyl sulfone (Table 2, 3ag)** [2]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.82 (d, *J*=8.4 Hz, 2H), 7.50-7.28 (m, 5H), 7.05 (d, *J*=8.1 Hz, 1H), 3.83 (s, 3H), 2.39 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 160.0, 144.2, 143.0, 138.5, 130.4, 129.9, 127.6, 119.7, 119.3, 112.1, 55.7, 21.6. GC-MS (EI, m/z): 262[M+].



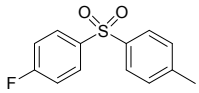
***o*-methoxyphenyl *p*-tolyl sulfone (Table 2, 3ah)** [3]

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.11 (d, *J*=7.8 Hz, 1H), 7.84 (d, *J*=8.4 Hz, 2H), 7.50-7.49 (m, 1H), 7.26 (d, *J*=8.4 Hz, 2H), 7.06 (m, 1H), 6.88 (d, *J*=8.4 Hz, 1H), 3.73 (s, 3H), 2.37 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 157.1, 143.7, 138.8, 135.3, 129.5, 129.1, 128.3, 120.4, 112.7, 55.8, 21.5. GC-MS (EI, m/z): 262[M+].



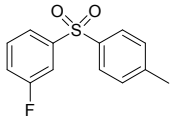
***m*-methylthiophenyl *p*-tolyl sulfone (Table 2, 3ai)**

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.82 (d, *J*=8.4 Hz, 2H), 7.76 (s, 1H), 7.64-7.63 (m, 1H), 7.37-7.27 (m, 4H), 2.48 (s, 3H), 2.38 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.3, 142.6, 141.1, 138.4, 130.4, 130.0, 129.5, 127.7, 124.3, 123.7, 21.5, 15.4. GC-MS (EI, m/z): 278[M+].



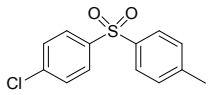
***p*-fluorophenyl *p*-tolyl sulfone (Table 2, 3aj)** [2]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.97-7.92 (m, 2H), 7.82 (d, *J*=8.1 Hz, 2H), 7.29 (d, *J*=8.1 Hz, 2H), 7.19-7.13 (m, 2H), 2.38 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.3 (167.0, 163.6, d, *J* = 255.0 Hz), 144.4, 138.5, 138.1, 130.3, 130.0, 127.6, 116.5 (116.6, 116.3, d, *J* = 22.5 Hz), 21.5. GC-MS (EI, m/z): 250[M+].



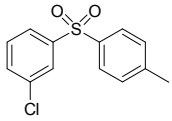
***m*-fluorophenyl *p*-tolyl sulfone (Table 2, 3ak)** [2]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.83 (d, *J*=8.1 Hz, 2H), 7.73 (d, *J*=7.8 Hz, 1H), 7.65-7.60 (m, 1H), 7.49-7.46 (m, 1H), 7.33-7.24 (m, 3H), 2.40 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ162.4 (164.1, 160.7, d, *J* = 255.0 Hz), 144.7, 144.0, 137.9, 131.2, 130.1, 127.8, 123.3, 120.3 (120.4, 120.2, d, *J* = 15.0 Hz), 114.8 (114.9, 114.6, d, *J* = 22.5 Hz), 21.6. GC-MS (EI, m/z): 250[M+].



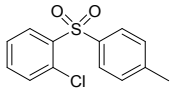
***p*-chlorophenyl *p*-tolyl sulfone (Table 2, 3al)** [2]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.89-7.80 (m, 4H), 7.43 (d, *J*=7.8 Hz, 2H), 7.28 (d, *J*=7.8 Hz, 2H), 2.38 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.5, 140.5, 139.6, 138.2, 130.1, 129.5, 129.0, 127.7, 21.6. GC-MS (EI, m/z): 267[M+].



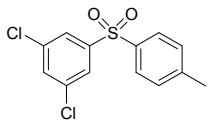
***m*-chlorophenyl *p*-tolyl sulfone (Table 2, 3am)** [6]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.91 (s, 1H), 7.90-7.80 (m, 3H), 7.51-7.40 (m, 2H), 7.32 (d, *J*=8.4 Hz, 2H), 2.40 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.7, 143.8, 138.0, 135.4, 133.1, 130.5, 130.1, 127.8, 127.5, 125.6, 21.5. GC-MS (EI, m/z): 267 [M+].



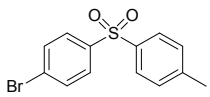
***o*-chlorophenyl *p*-tolyl sulfone (Table 2, 3an)**

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.33 (d, *J*=8.3 Hz, 1H), 7.83 (d, *J*=8.1 Hz, 2H), 7.50-7.38 (m, 3H), 7.29 (d, *J*=8.1 Hz, 2H), 2.39 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.6, 138.6, 137.1, 134.6, 132.7, 132.0, 130.8, 129.5, 128.6, 127.3, 21.6. GC-MS (EI, m/z): 267 [M+].



3,5-dichlorophenyl *p*-tolyl sulfone (Table 2, 3ao) [6]

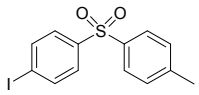
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.84-7.79 (m, 4H), 7.50 (s, 1H), 7.36-7.34 (m, 2H), 2.43 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 145.1, 137.4, 136.2, 132.9, 130.2, 128.0, 125.9, 21.6. GC-MS (EI, m/z): 302[M+]



***p*-bromophenyl *p*-tolyl sulfone (Table 2, 3ap)** [2]

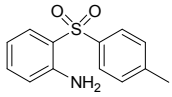
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.83-7.78 (m, 4H), 7.61 (d, *J*=8.4 Hz, 2H), 7.30 (d, *J*=7.8 Hz, 2H), 2.39 (s, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.6, 141.0, 138.1, 132.5, 130.1, 129.0, 128.2, 127.7,

21.6. GC-MS (EI, m/z): 311[M+].



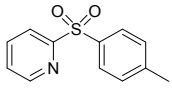
***p*-iodophenyl *p*-tolyl sulfone (Table 2, 3aq)**

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95-7.92 (m, 2H), 7.83 (d, *J*=8.1 Hz, 2H), 7.54-7.45 (m, 2H), 7.29 (d, *J*=8.1 Hz, 2H), 2.38 (s, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.2, 142.0, 138.6, 133.0, 129.9, 129.2, 127.7, 127.5, 21.5. GC-MS (EI, m/z): 358[M+].



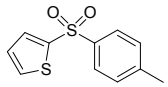
***o*-aminophenyl *p*-tolyl sulfone (Table 2, 3ar)** [7]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.83-7.78 (m, 3H), 7.29-7.24 (m, 3H), 6.79-6.76 (m, 1H), 6.64-6.62 (m, 1H), 5.12 (s, 2H), 2.37 (s, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 146.2, 143.8, 139.1, 134.7, 129.8, 129.6, 126.9, 122.4, 117.7, 117.6, 21.4. GC-MS (EI, m/z): 247[M+].



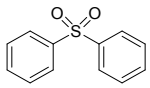
2-pyridyl-*p*-tolyl sulfone (Table 2, 3as) [5]

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.68 (s, 1H), 8.04-7.96 (m, 2H), 7.84-7.82 (m, 2H), 7.55-7.52 (m, 1H), 7.28-7.25 (m, 2H), 2.37 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 157.6, 150.7, 145.8, 139.5, 134.8, 130.4, 128.6, 128.2, 123.3, 21.9. GC-MS (EI, m/z): 233[M+].



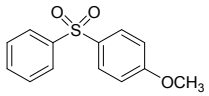
2-thiophyl-*p*-tolyl sulfone (Table 2, 3at) [3]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.86 (d, *J*=8.4 Hz, 2H), 7.67-7.60 (m, 2H), 7.30 (d, *J*=8.1 Hz, 2H), 7.07-7.04 (m, 1H), 2.39 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.4, 143.5, 139.1, 133.6, 133.0, 130.0, 127.8, 127.3, 21.6. GC-MS (EI, m/z): 238[M+].



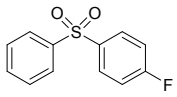
diphenyl Sulfone (Table 2, 3ba) [8]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.96-7.94 (m, 4H), 7.59-7.48 (m, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 141.5, 133.2, 129.3, 127.7. GC-MS (EI, m/z): 218 [M+].



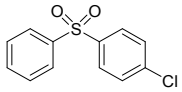
***p*-methoxyphenyl phenyl sulfone (Table 2, 3bb)** [8]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.93-7.86 (m, 4H), 7.52-7.48 (m, 3H), 6.98-6.95 (m, 2 H), 3.83 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 163.4, 142.3, 133.1, 132.9, 129.9, 129.2, 127.3, 114.5, 55.7. GC-MS (EI, m/z): 248 [M+].



***p*-fluorophenyl phenyl sulfone (Table 2, 3bc)** [8]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.99-7.92 (m, 4H), 7.58-7.49 (m, 3H), 7.21-7.15 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ165.4 (167.1, 163.7, d, J = 255 Hz), 141.4, 137.7, 133.4, 130.5 (130.5, 130.4, d, J =7.5 Hz), 129.4, 127.6, 116.7 (116.8, 116.5, d, J = 22.5 Hz). GC-MS (EI, m/z): 236 [M+].



***p*-chlorophenyl phenyl sulfone (Table 2, 3bd)** [8]

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95-7.87 (m, 4H), 7.58-7.45 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 141.1, 140.1, 139.9, 133.5, 129.6, 129.5, 129.1, 127.6. GC-MS (EI, m/z): 253 [M+].

References

1. X. Z. Yu, X. W. Li and B. S. Wan. *Org. Biomol. Chem.*, 2012, **10**, 7479-7482.
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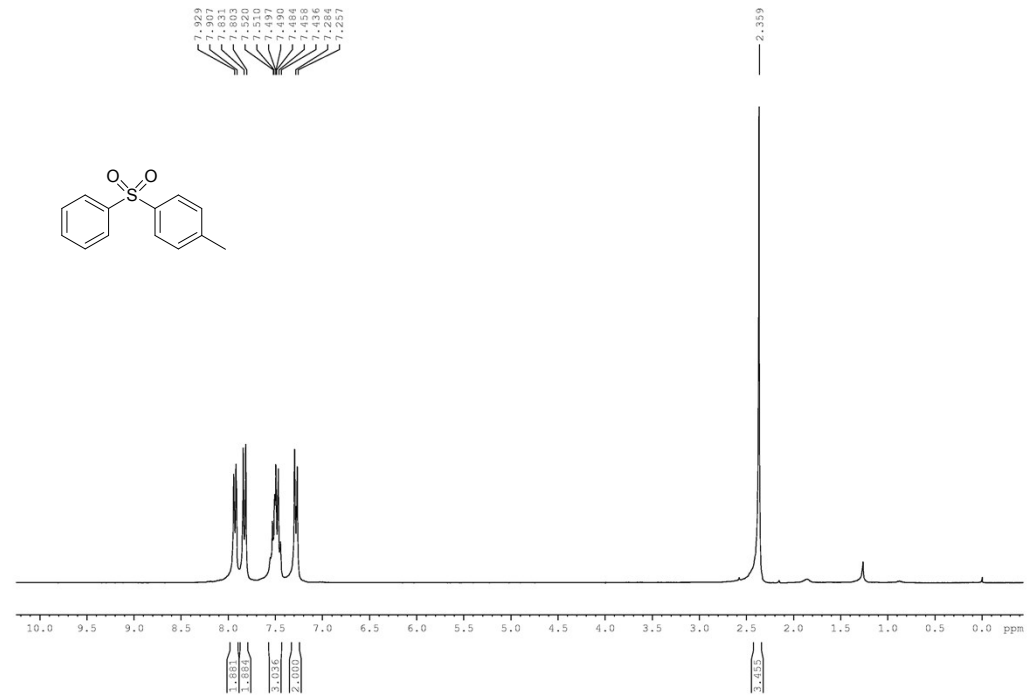
3aa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7.907
7.883
7.863
7.520
7.510
7.497
7.490
7.484
7.468
7.436
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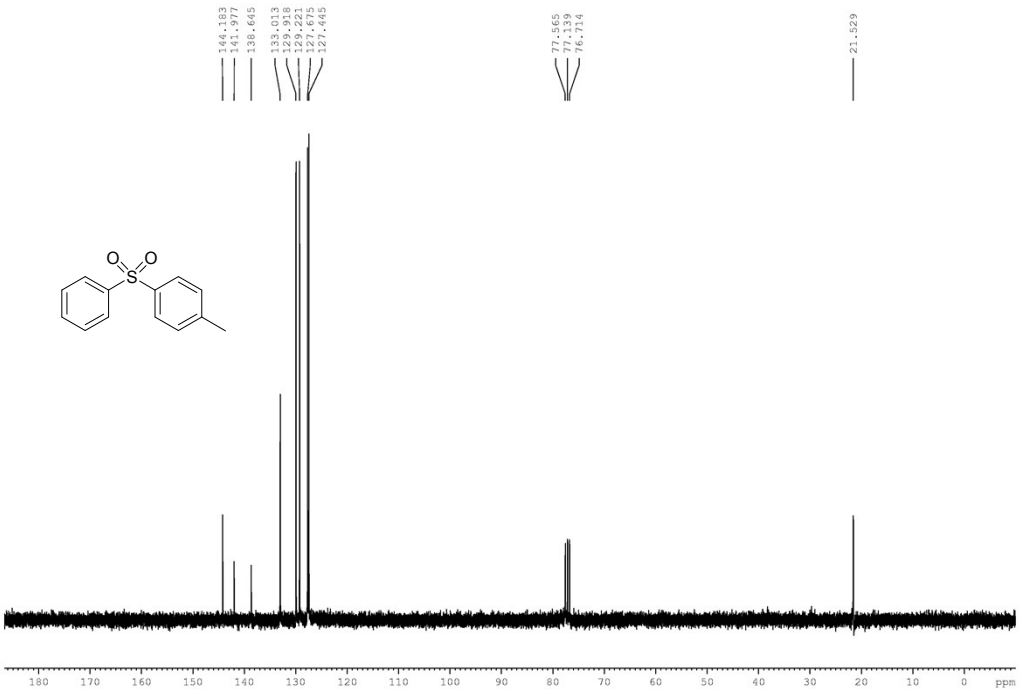
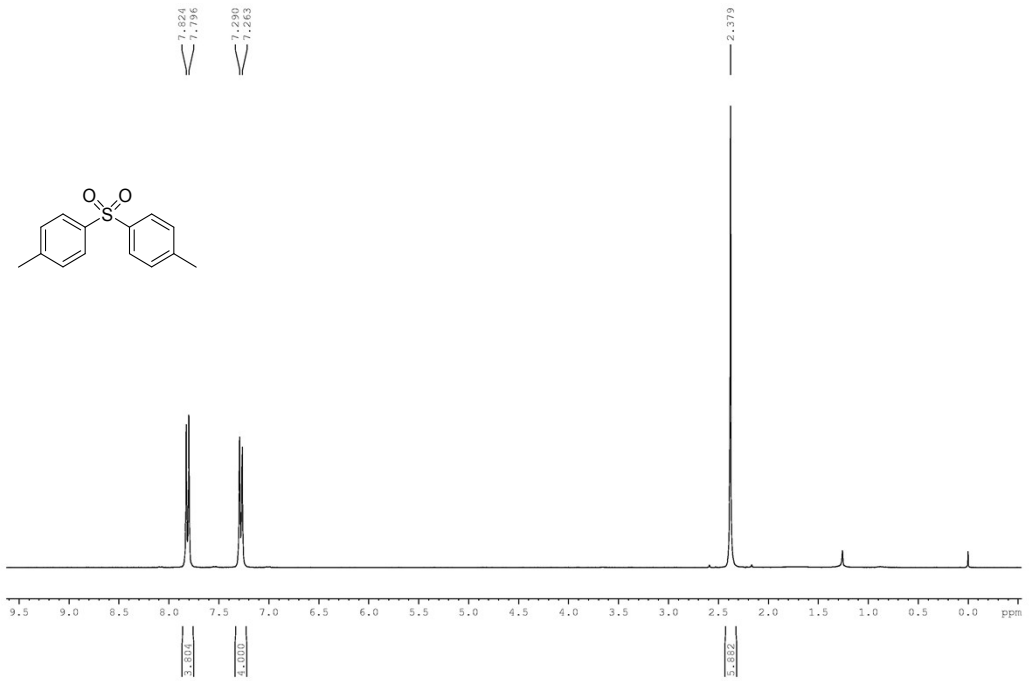
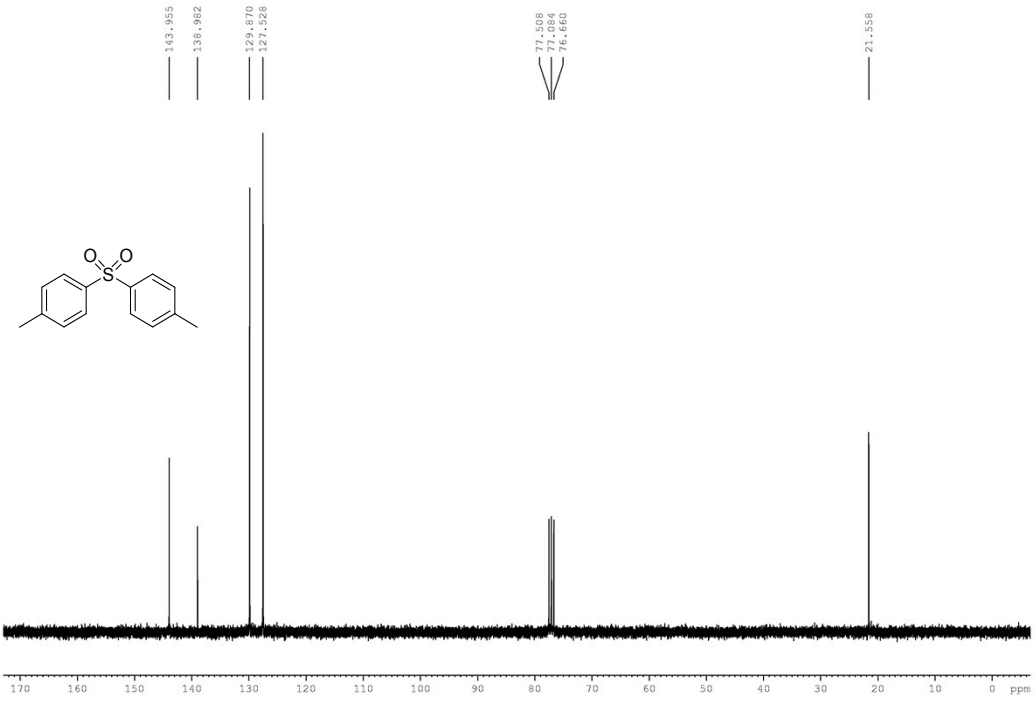
7.257
2.359
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.881
1.884
3.036
2.000
3.455

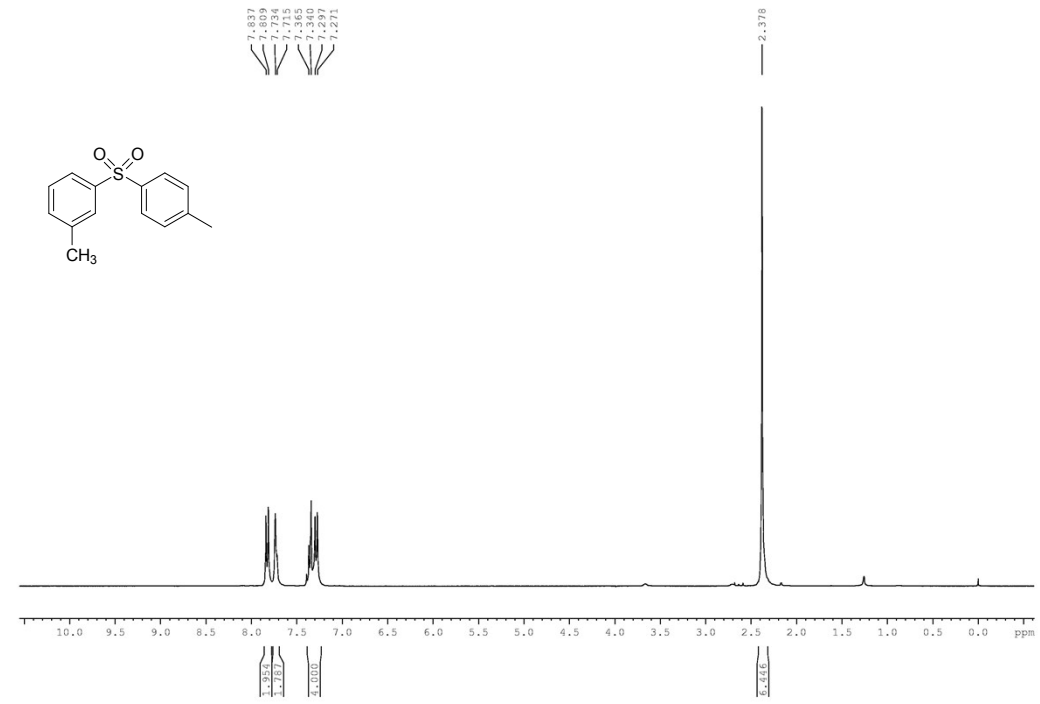

144.193
141.977
138.645
133.013
129.918
129.221
127.675
127.445
77.565
77.139
76.714
21.529
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

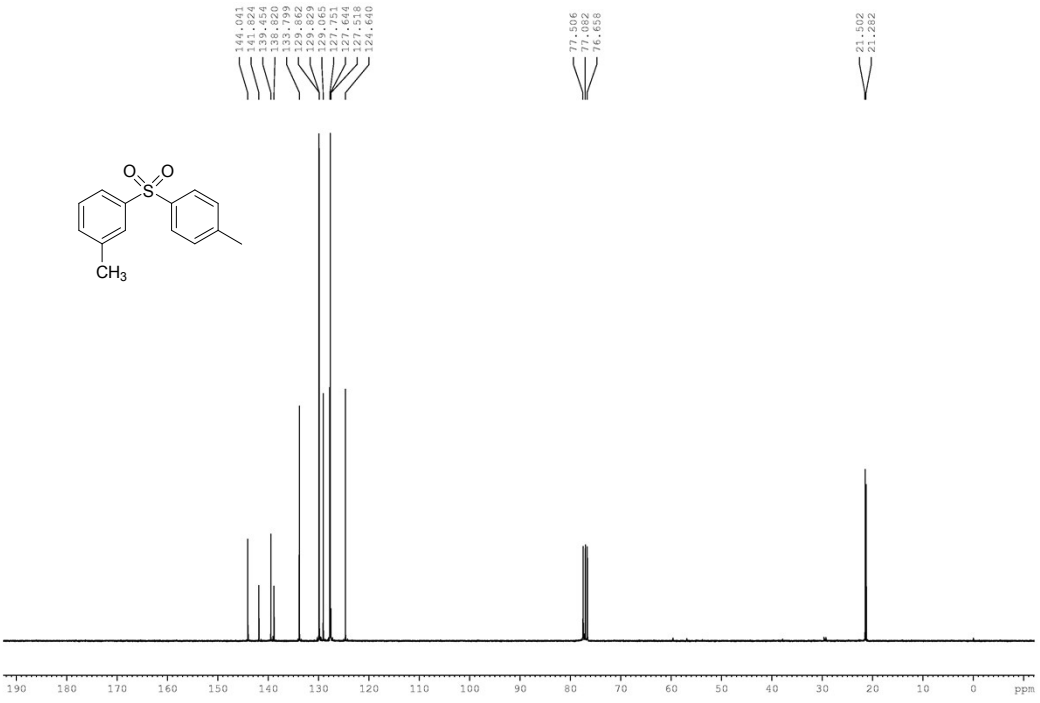
3ab


7.804
7.796
7.290
7.263
2.379
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 ppm
3.804
4.000
5.882

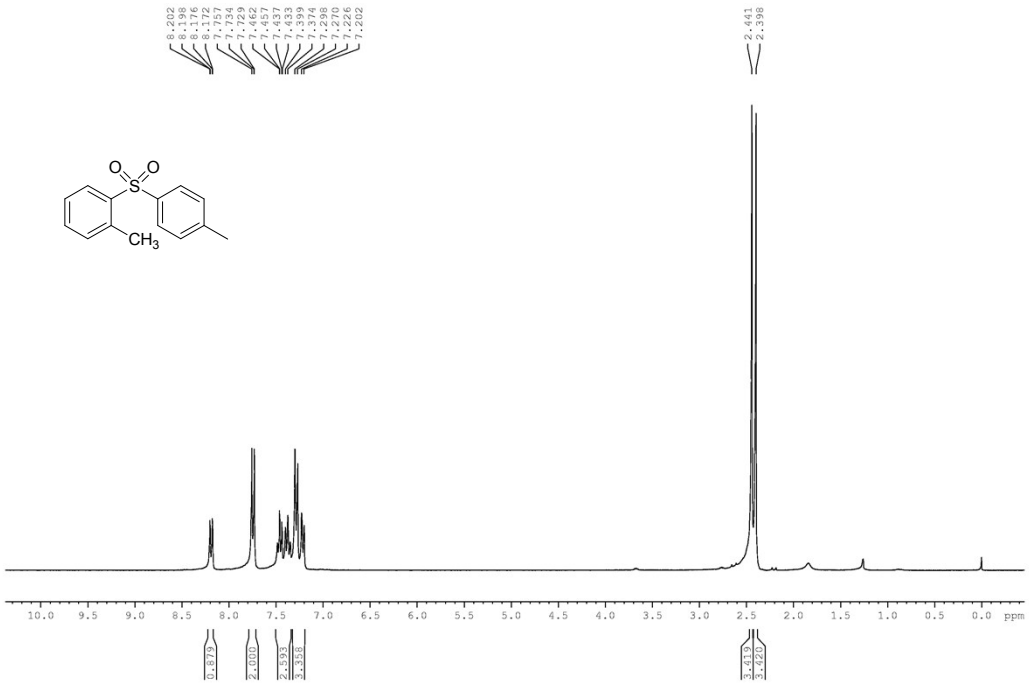

143.955
138.962
129.870
127.528
77.508
77.084
76.660
21.558
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

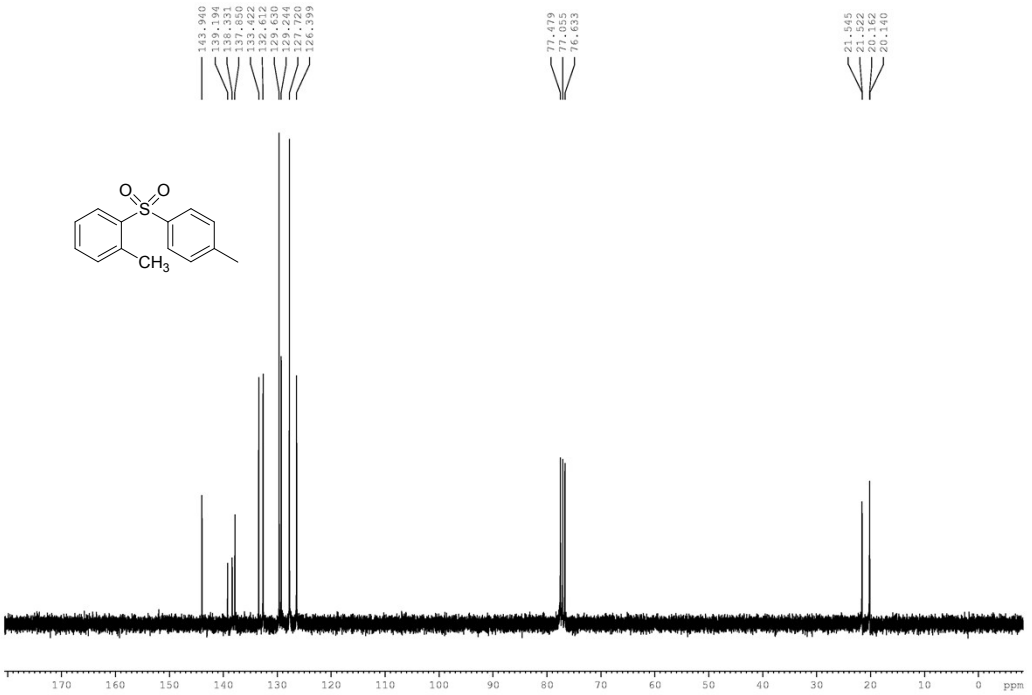
3ac


7.837
7.809
7.734
7.725
7.365
7.340
7.297
7.271
2.378
O
O
S
CH3
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 ppm
1.954
1.787
4.000
6.466

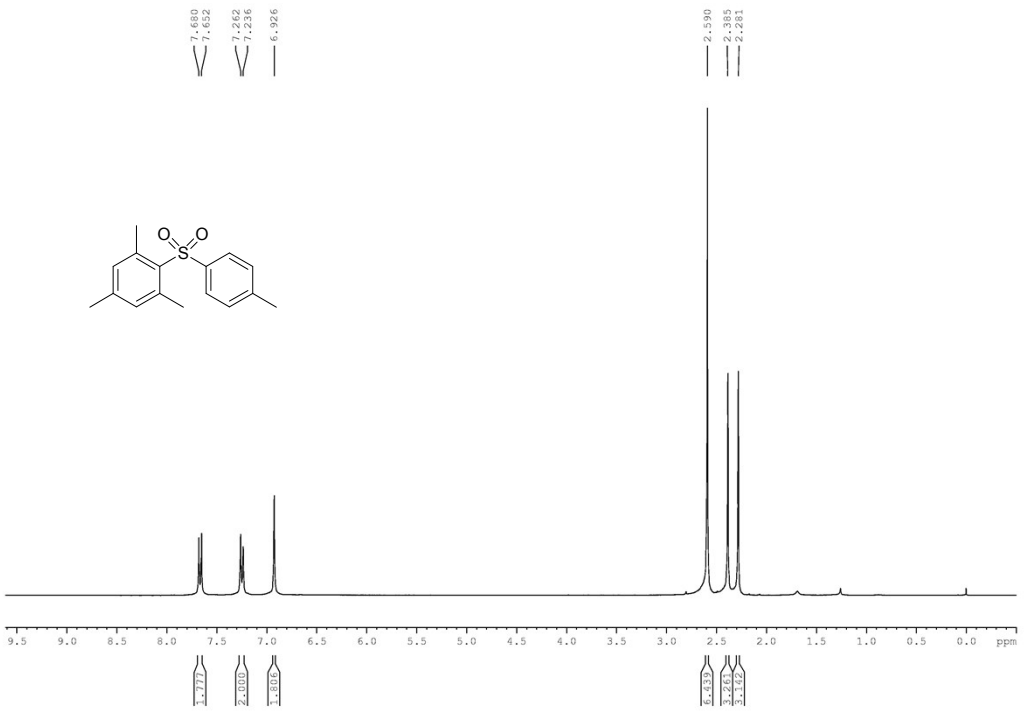

144.041
141.824
139.454
138.720
138.139
129.862
129.829
129.065
127.751
127.644
127.118
124.640
77.506
77.082
76.658
21.502
21.282
O
O
S
CH3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

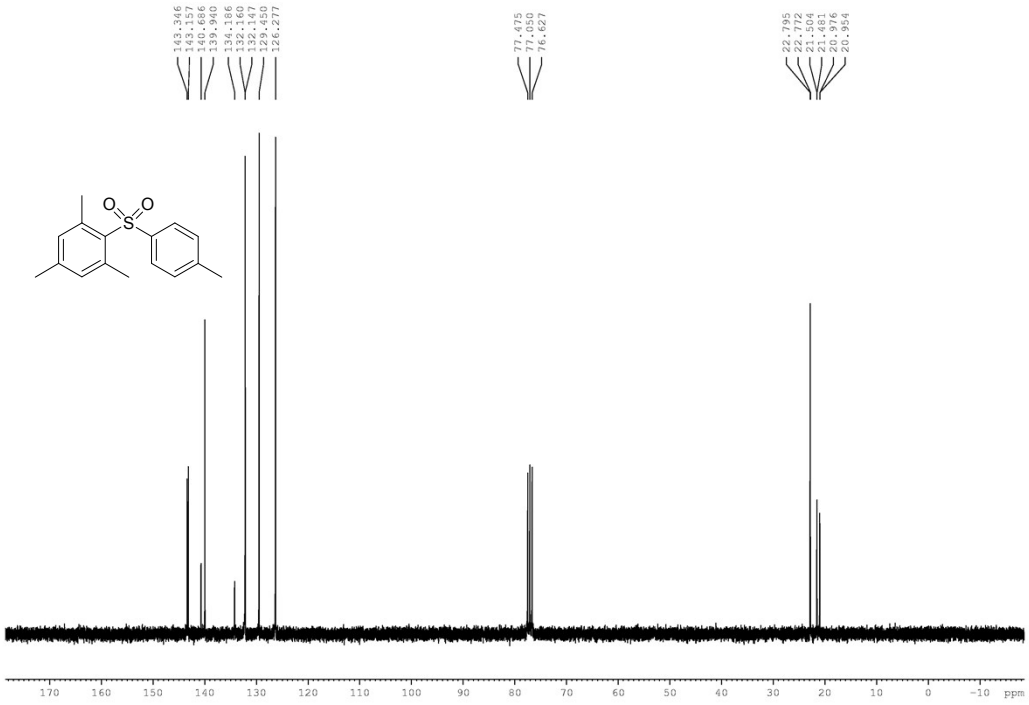
3ad


8.202
8.198
8.176
8.172
7.757
7.734
7.729
7.462
7.457
7.437
7.393
7.389
7.374
7.298
7.270
7.226
7.202
2.441
2.398
O
O
S
CH3
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 ppm
0.879
2.000
2.593
3.358
3.419
3.420

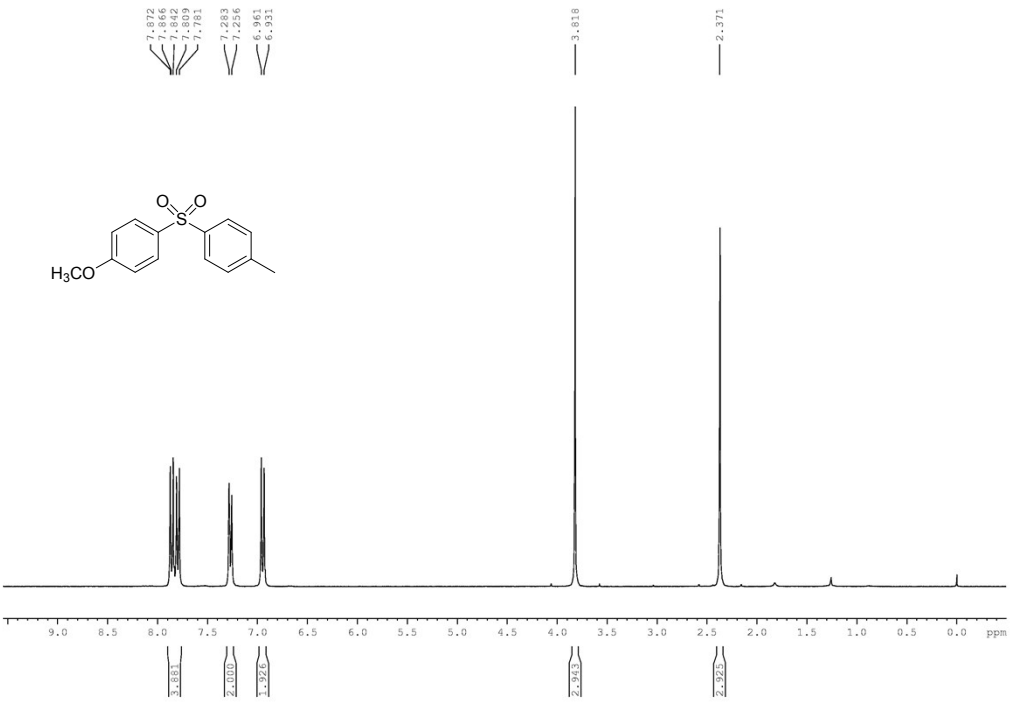

143.940
139.194
138.331
137.850
133.422
132.612
129.630
129.244
127.720
126.399
77.479
77.055
76.633
21.545
21.522
20.162
20.140
O
O
S
CH3
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

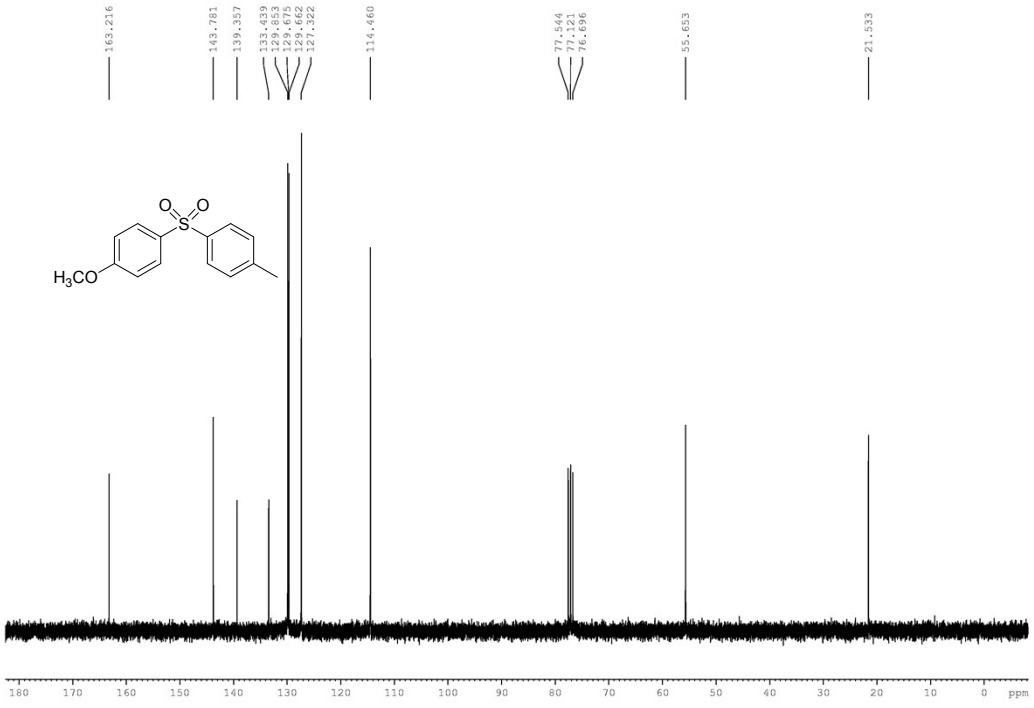
3ae


7.680
7.652
7.262
7.236
6.926
2.590
2.385
2.281
O
O
S
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 ppm
1.777
2.000
1.886
6.439
3.261
3.162

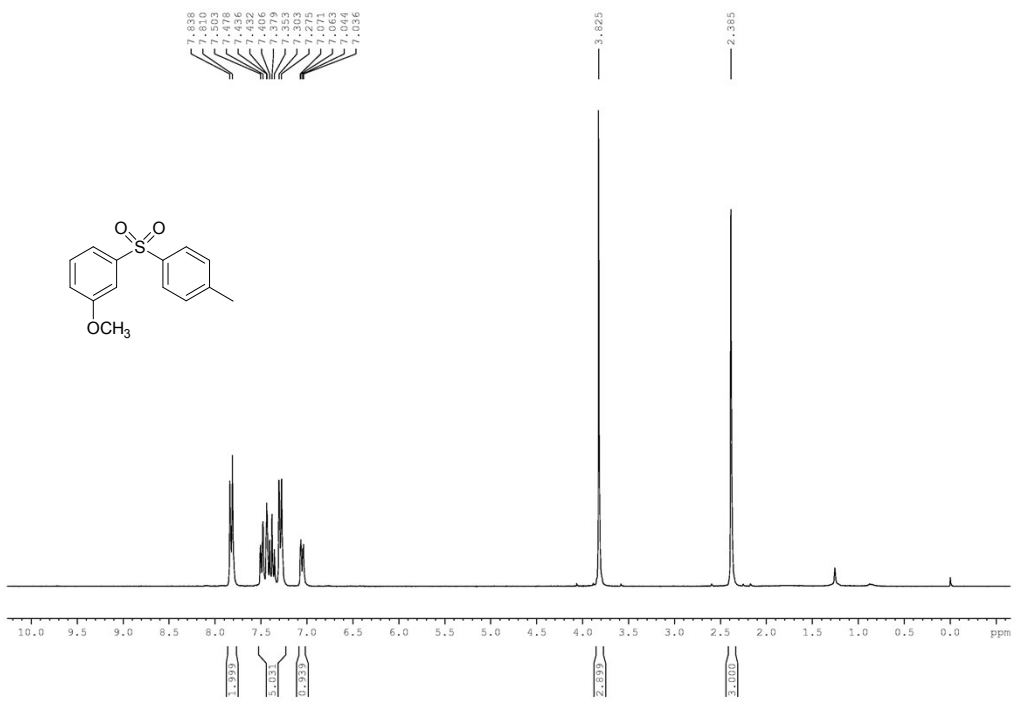

143.346
142.157
140.686
139.940
134.186
132.160
132.147
129.450
126.277
77.475
77.050
76.627
22.795
22.772
21.504
21.481
20.976
20.954
O
O
S
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

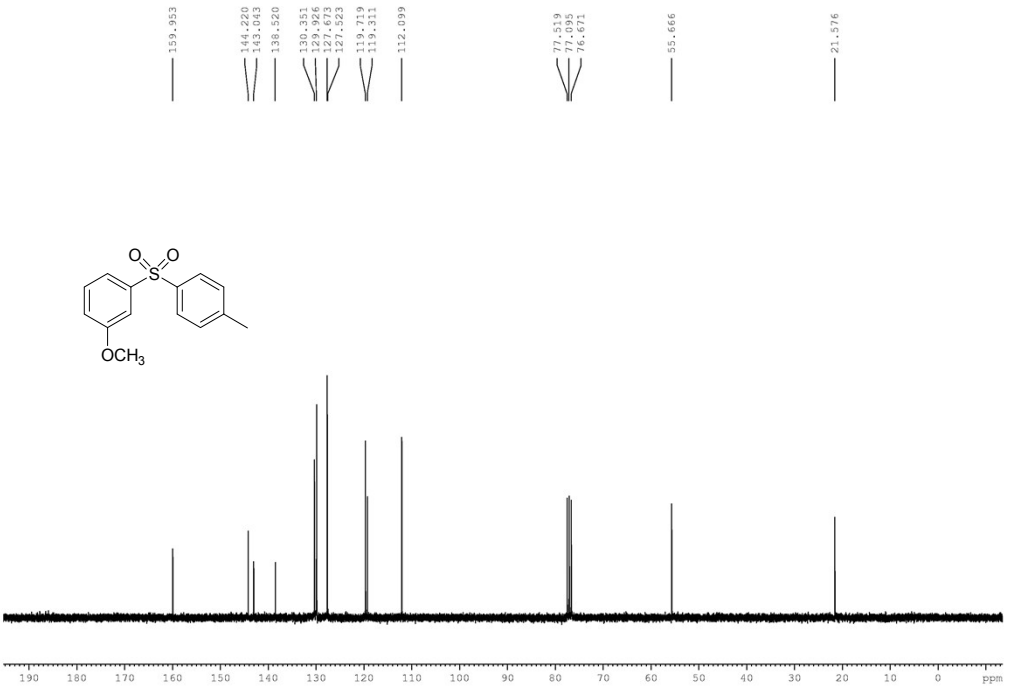
3af


7.872
7.866
7.862
7.809
7.781
7.283
7.256
6.961
6.931
3.818
2.371
O
O
S
H3CO
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 ppm
3.881
2.000
1.926
2.943
2.925

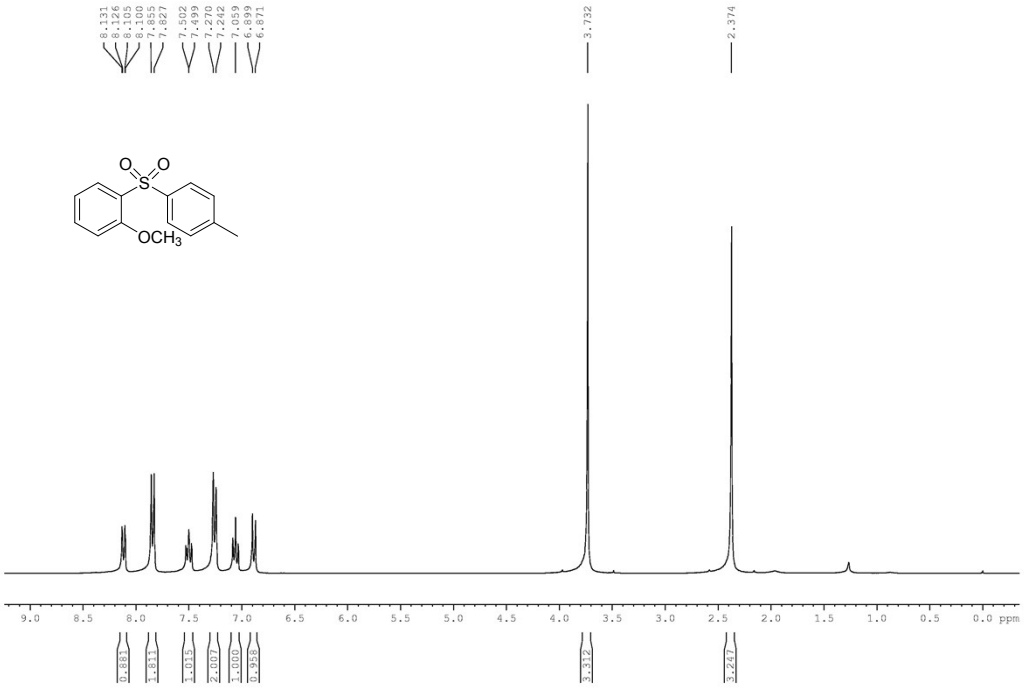

163.216
143.781
139.357
133.439
129.853
129.675
129.662
127.322
114.460
77.544
77.121
76.696
55.653
21.533
O
O
S
H3CO
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

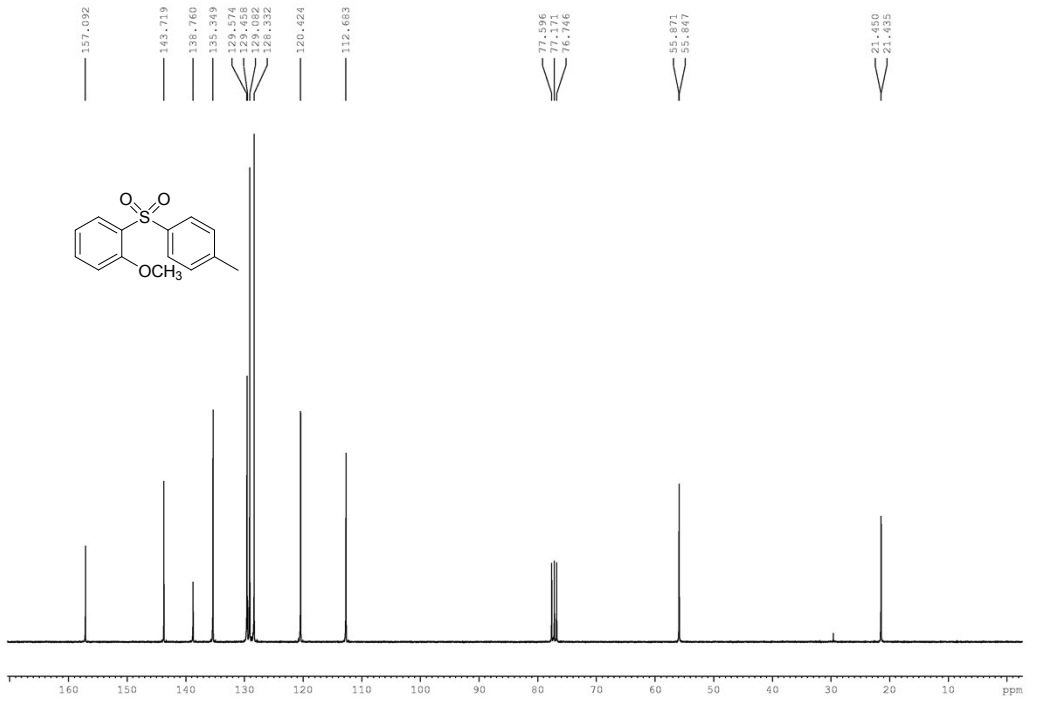
3ag


7.838
7.810
7.503
7.478
7.436
7.432
7.406
7.379
7.353
7.303
7.275
7.071
7.063
7.044
7.036
3.825
2.385
O
O
S
OCH3
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 ppm
1.939
5.031
0.939
2.899
3.000

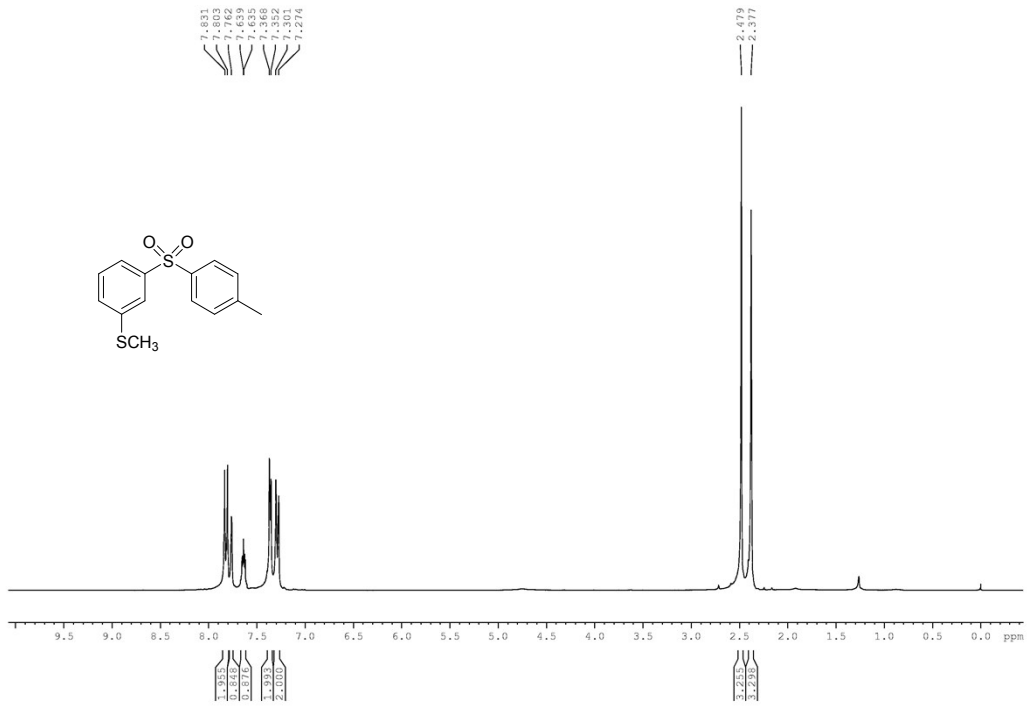

159.953
144.220
143.043
138.520
130.351
129.926
127.673
127.523
119.719
119.311
112.099
77.519
77.095
76.671
55.666
21.576
O
O
S
OCH3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

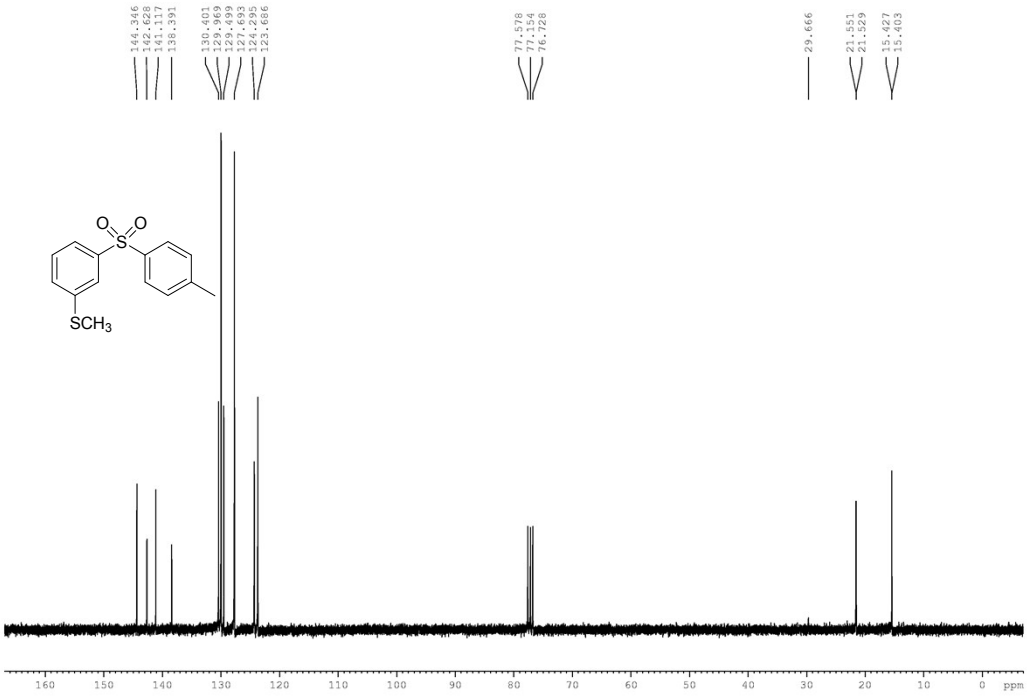
3ah


8.131
8.126
8.105
8.100
7.855
7.827
7.502
7.499
7.270
7.242
7.059
6.899
6.871
3.732
2.374
O
S
O
OCH3
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 ppm
0.881
1.831
1.015
2.007
3.000
0.958
3.312
3.247

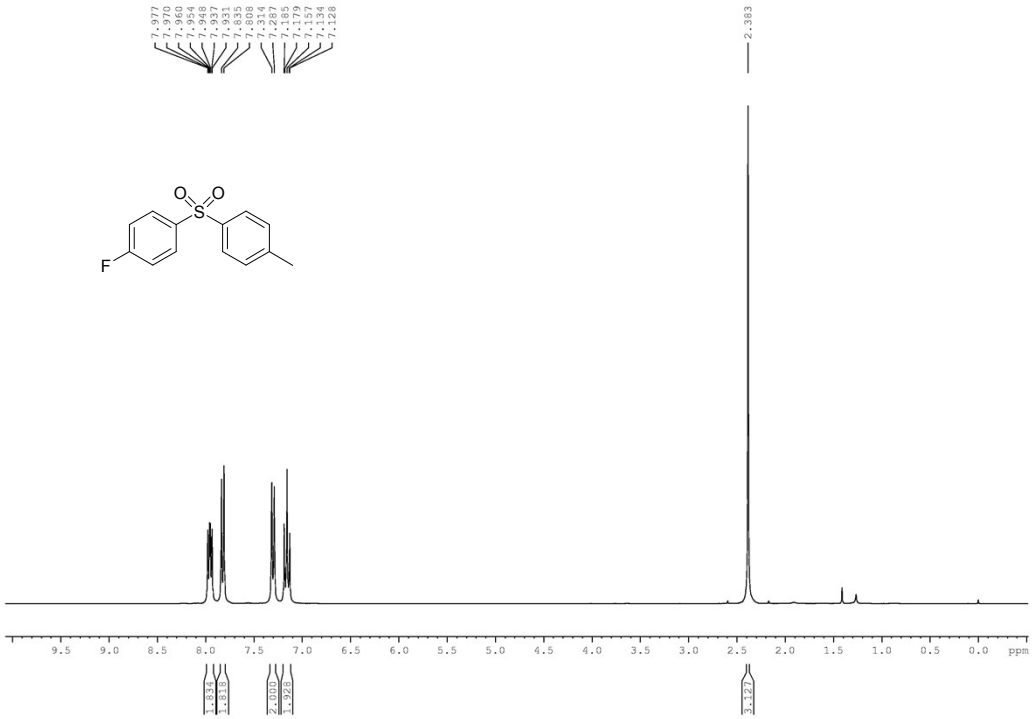

157.092
143.719
138.760
135.349
129.574
129.458
129.082
128.332
120.424
112.683
77.596
77.171
76.746
55.871
55.847
21.450
21.435
O
S
O
OCH3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

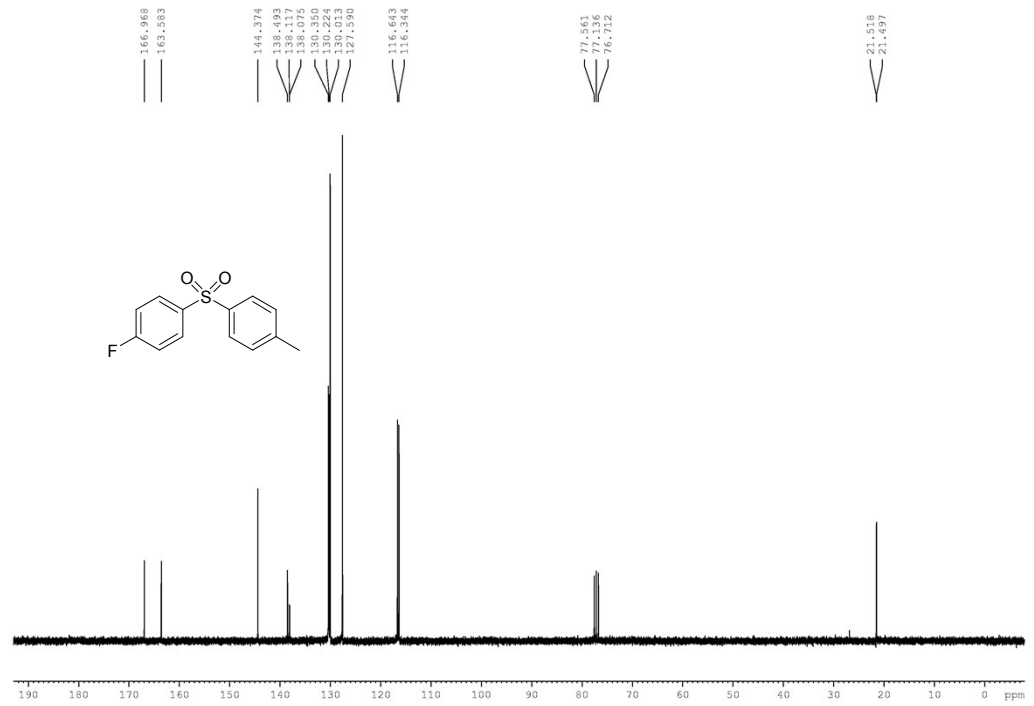
3ai


7.831
7.803
7.762
7.639
7.635
7.368
7.352
7.301
7.274
2.479
2.377
O
O
S
SCH3
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 ppm
1.965
0.948
0.876
1.993
2.000
3.265
3.289

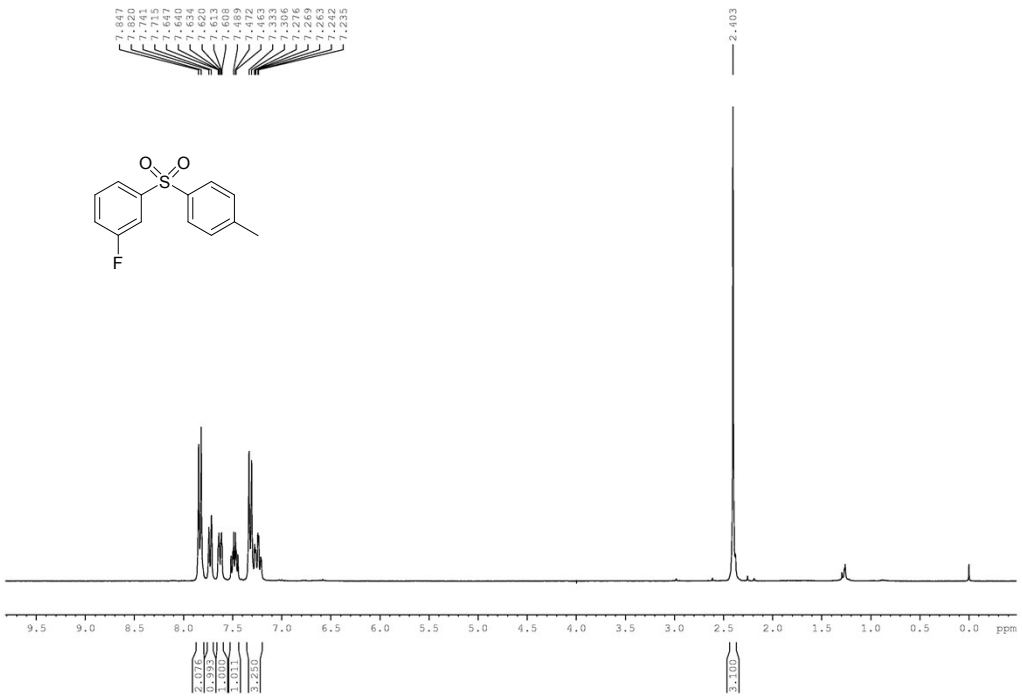

144.346
142.628
141.117
138.391
130.401
129.969
129.499
127.693
124.295
123.686
77.578
77.154
76.728
29.666
21.551
21.529
15.427
15.403
O
O
S
SCH3
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

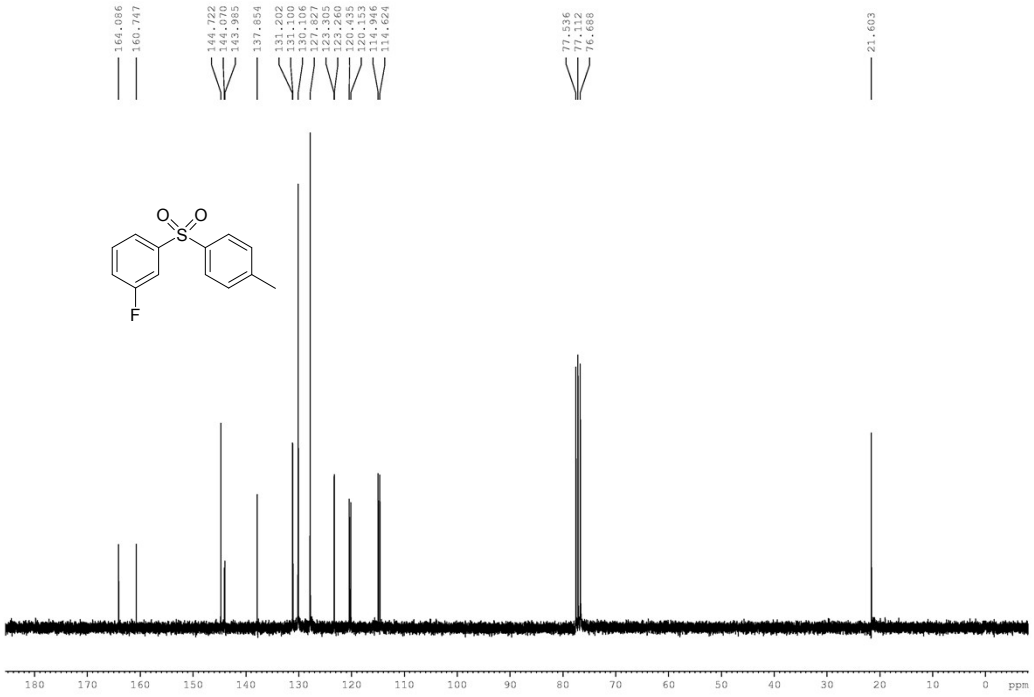
3aj


7.977
7.970
7.960
7.954
7.948
7.937
7.931
7.835
7.808
7.314
7.287
7.185
7.179
7.157
7.134
7.128
2.383
O
O
S
F
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 ppm
1.834
1.818
2.000
1.928
3.127

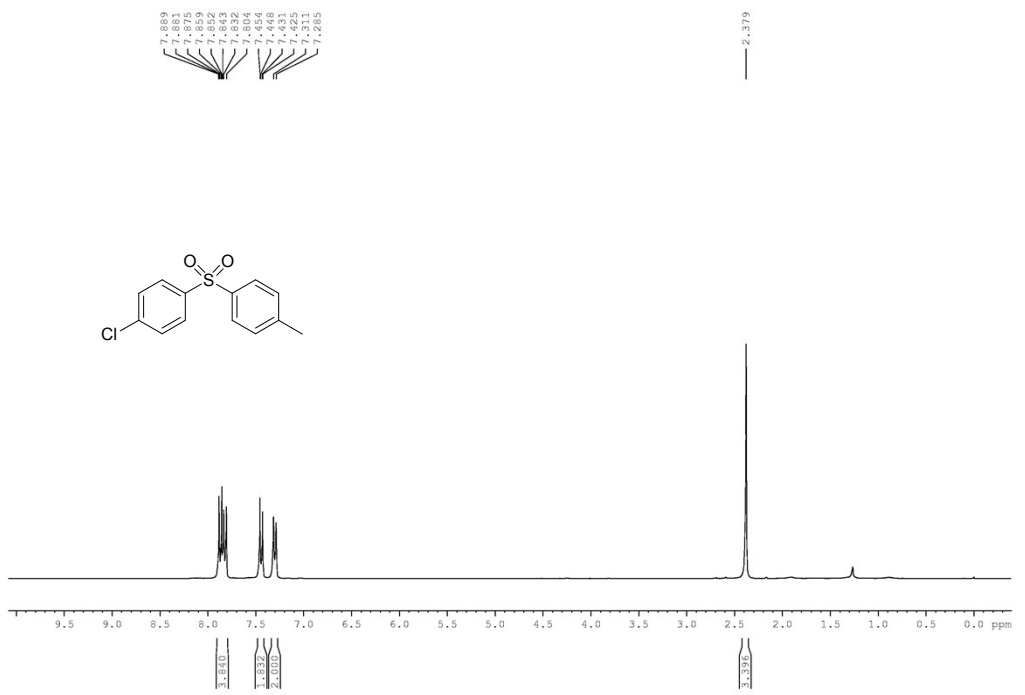

166.968
163.583
144.374
138.493
138.117
138.075
130.350
130.224
130.033
127.990
116.643
116.344
77.561
77.136
76.712
21.518
21.497
O
O
S
F
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

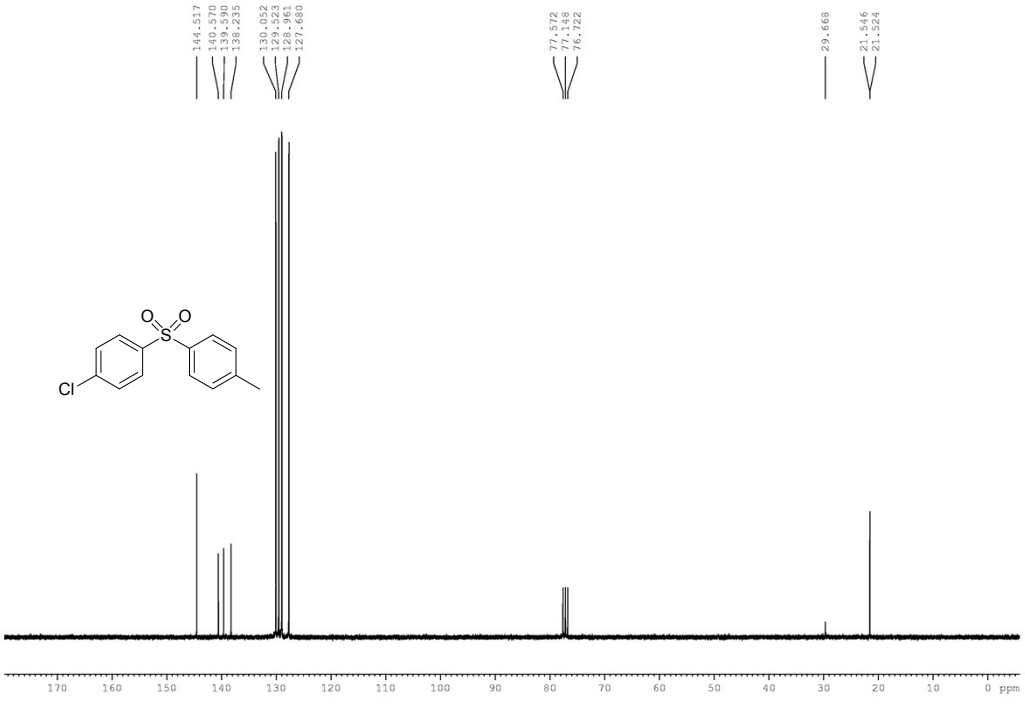
3ak


7.847
7.820
7.741
7.715
7.647
7.640
7.624
7.620
7.613
7.608
7.489
7.472
7.463
7.383
7.306
7.276
7.269
7.263
7.242
7.235
2.403
O
O
S
F
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 ppm
2.076
0.993
1.000
1.011
3.250
3.100

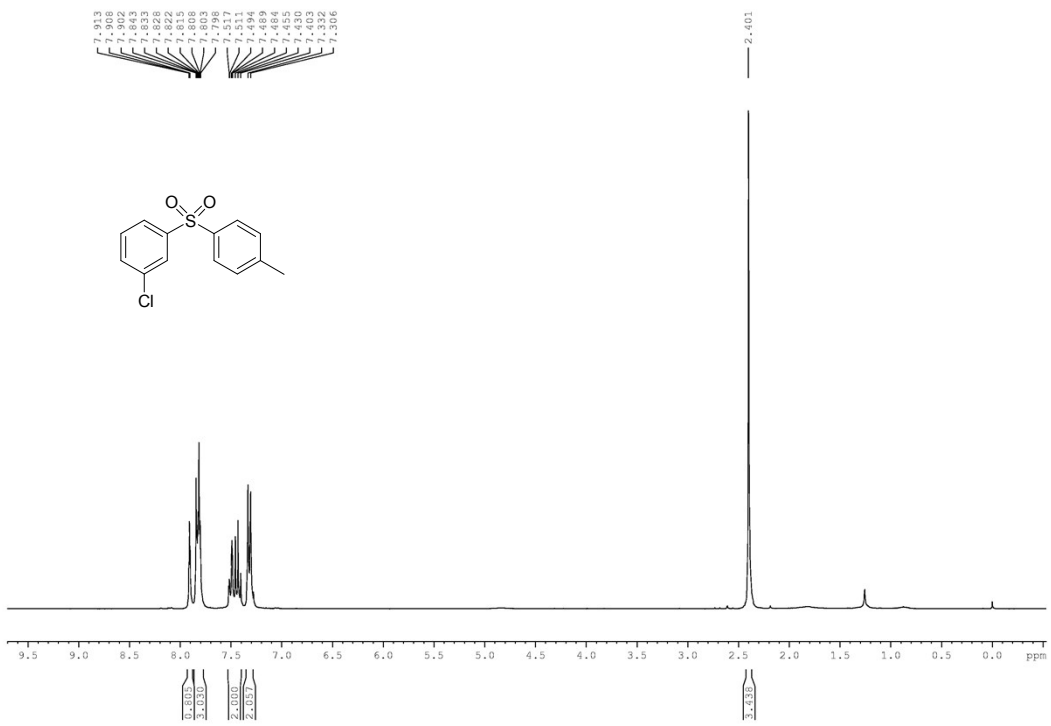

164.086
160.747
144.722
144.070
143.985
137.854
131.202
131.100
130.106
127.827
123.305
123.260
120.435
120.153
114.946
114.624
77.536
77.112
76.688
21.603
O
O
S
F
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

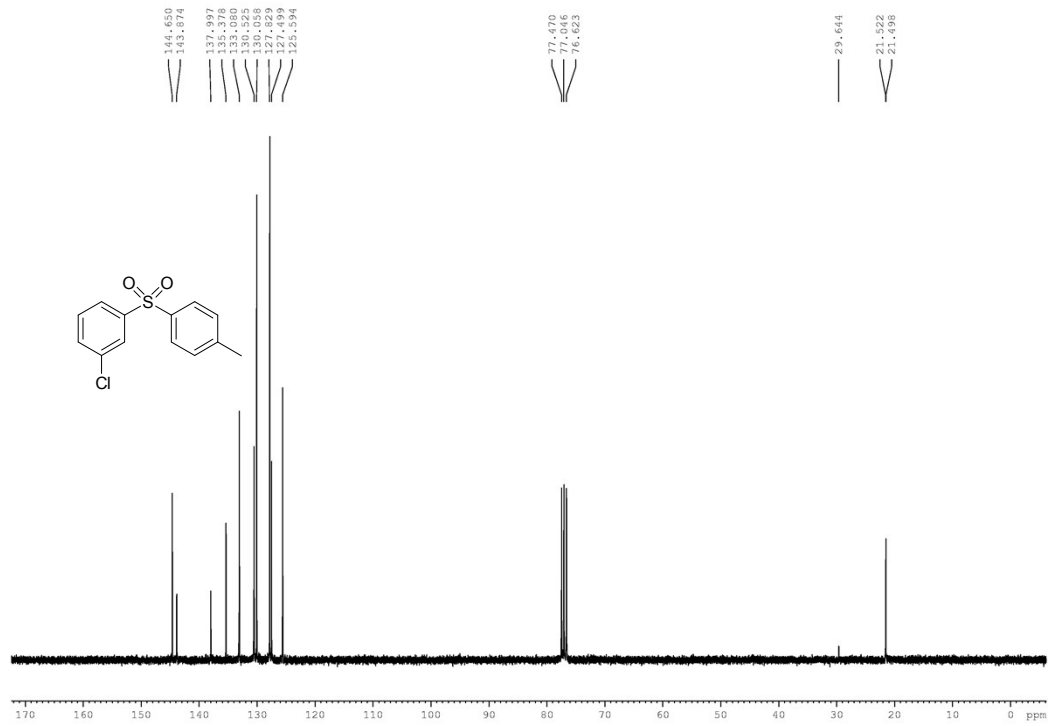
3al


7.869
7.881
7.875
7.859
7.852
7.843
7.832
7.804
7.454
7.448
7.431
7.425
7.311
7.285
2.379
O
O
S
Cl
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 ppm
3.840
1.832
2.000
3.396

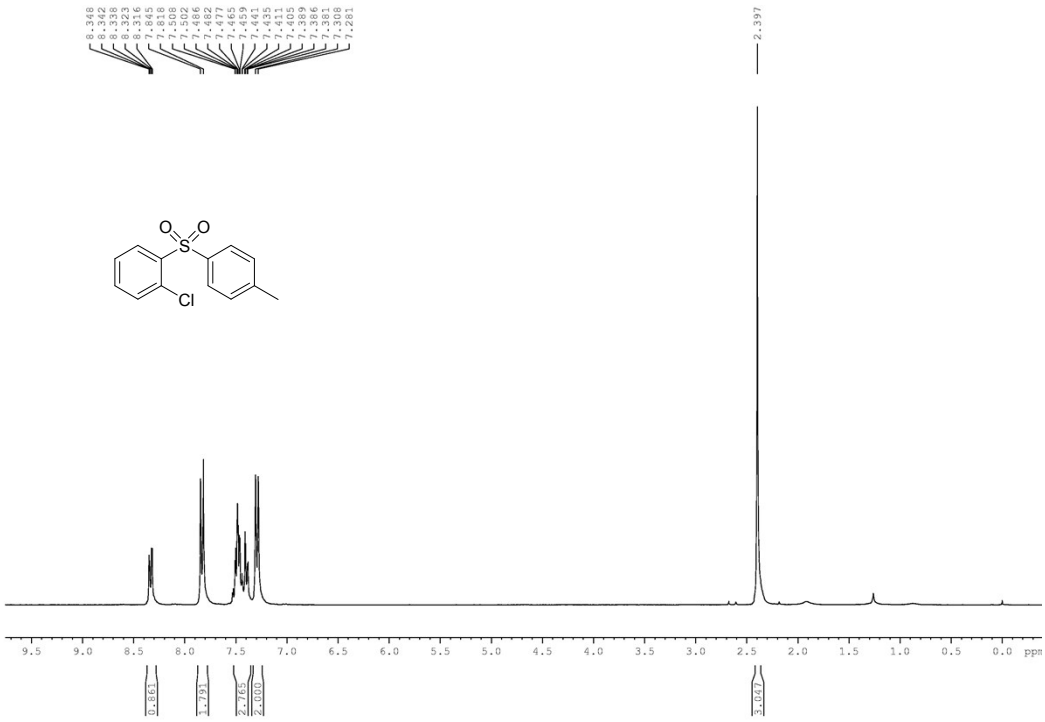

144.517
140.570
139.590
138.235
130.052
129.523
128.961
127.680
77.572
77.148
76.722
29.668
21.546
21.524
O
O
S
Cl
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

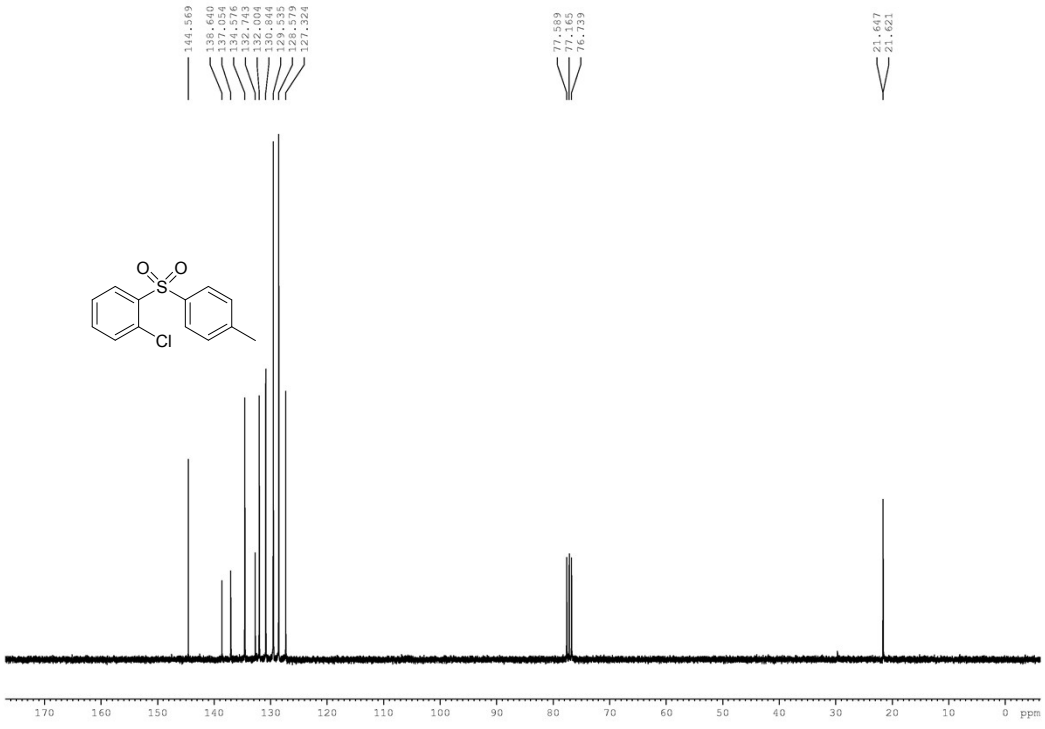
3am


7.913
7.908
7.902
7.863
7.833
7.828
7.822
7.815
7.808
7.803
7.798
7.517
7.511
7.494
7.489
7.484
7.455
7.430
7.403
7.332
7.306
2.401
O
O
S
Cl
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 ppm
0.805
3.036
2.000
2.057
3.438

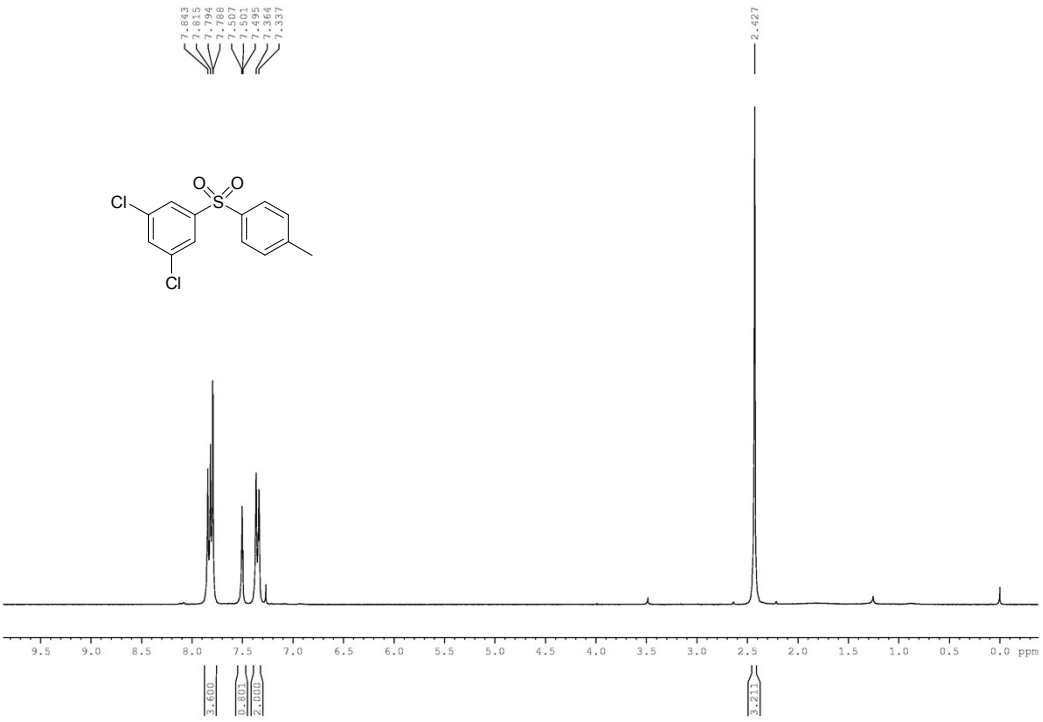

144.650
143.674
137.997
135.378
133.080
132.225
130.058
127.829
127.499
125.594
77.470
77.046
76.623
29.644
21.522
21.498
O
O
S
Cl
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

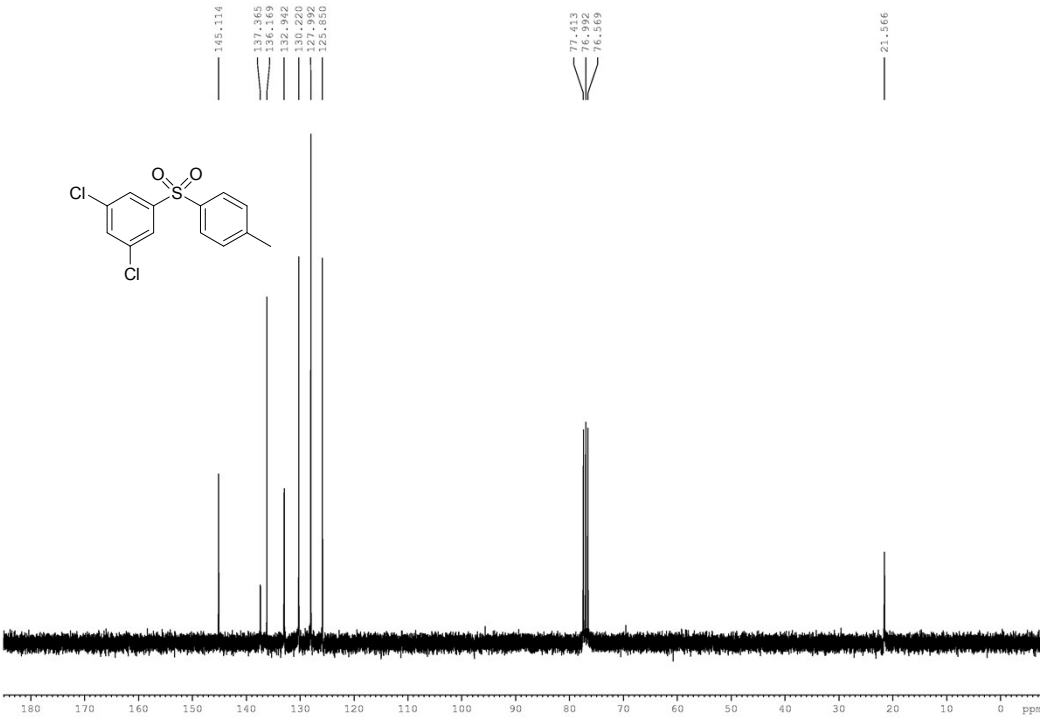
3an


8.348
8.342
8.338
8.323
8.316
7.645
7.618
7.508
7.502
7.486
7.482
7.477
7.465
7.459
7.441
7.435
7.411
7.405
7.389
7.386
7.381
7.308
7.281
2.397
O
O
S
Cl
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 ppm
0.861
1.791
2.765
2.000
3.067

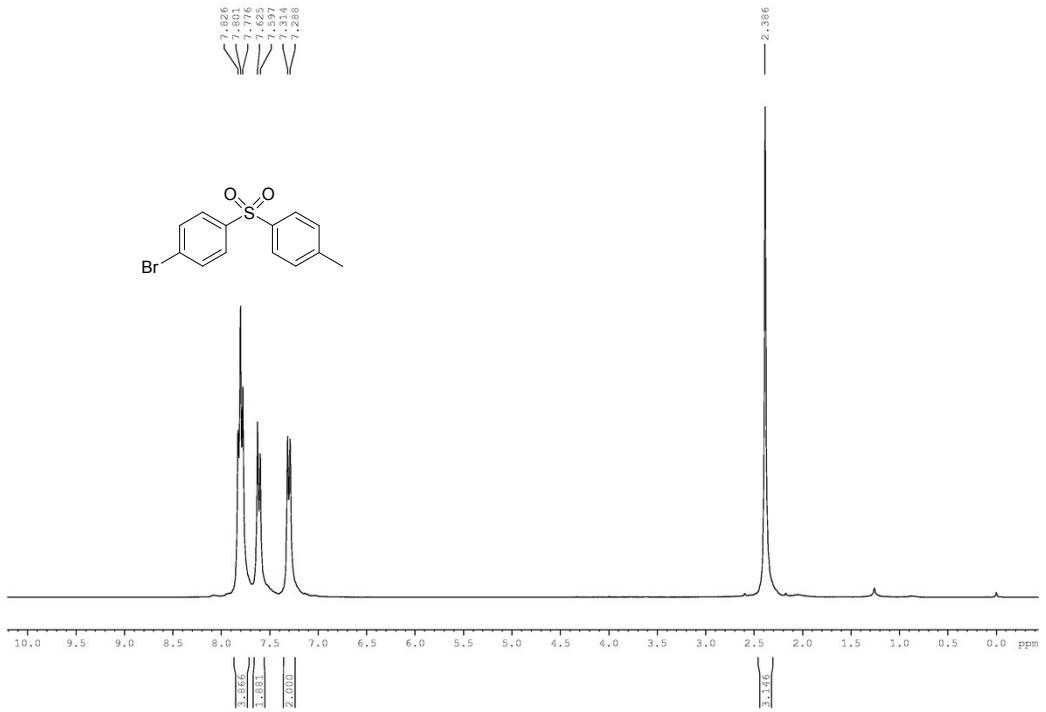

144.569
138.640
137.054
134.576
132.743
132.004
130.844
129.435
128.579
127.324
77.589
77.165
76.739
21.647
21.621
O
O
S
Cl
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

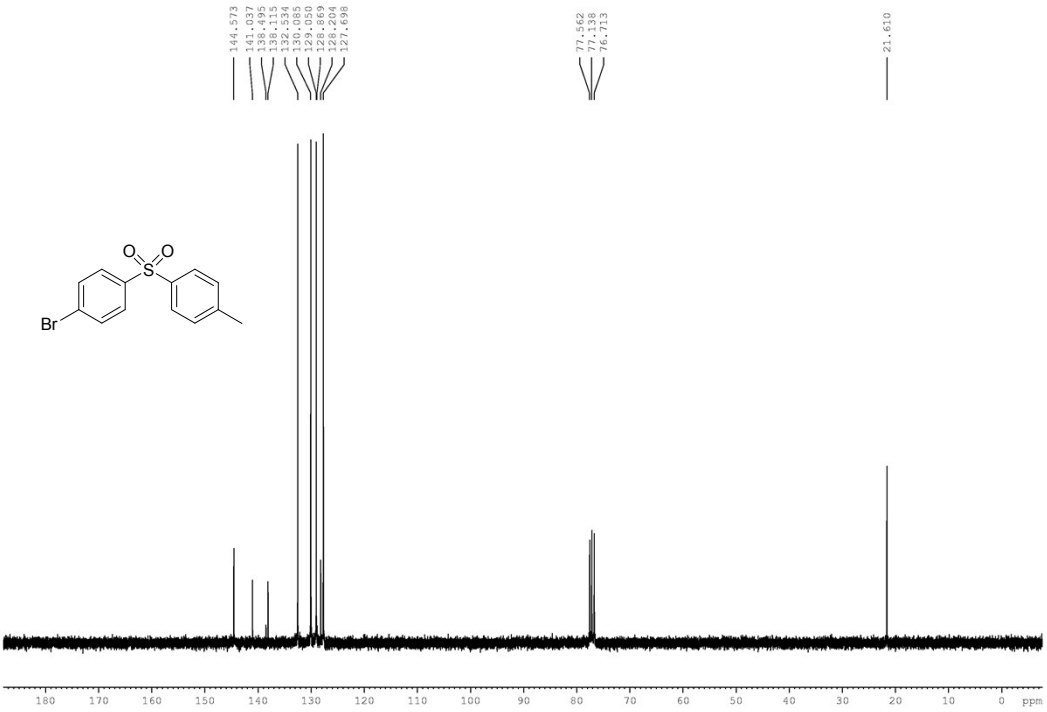
3ao


7.843
7.515
7.494
7.488
7.488
7.507
7.501
7.495
7.364
7.337
2.427
Cl
O
O
S
Cl
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 ppm
3.460
0.881
2.000
3.211

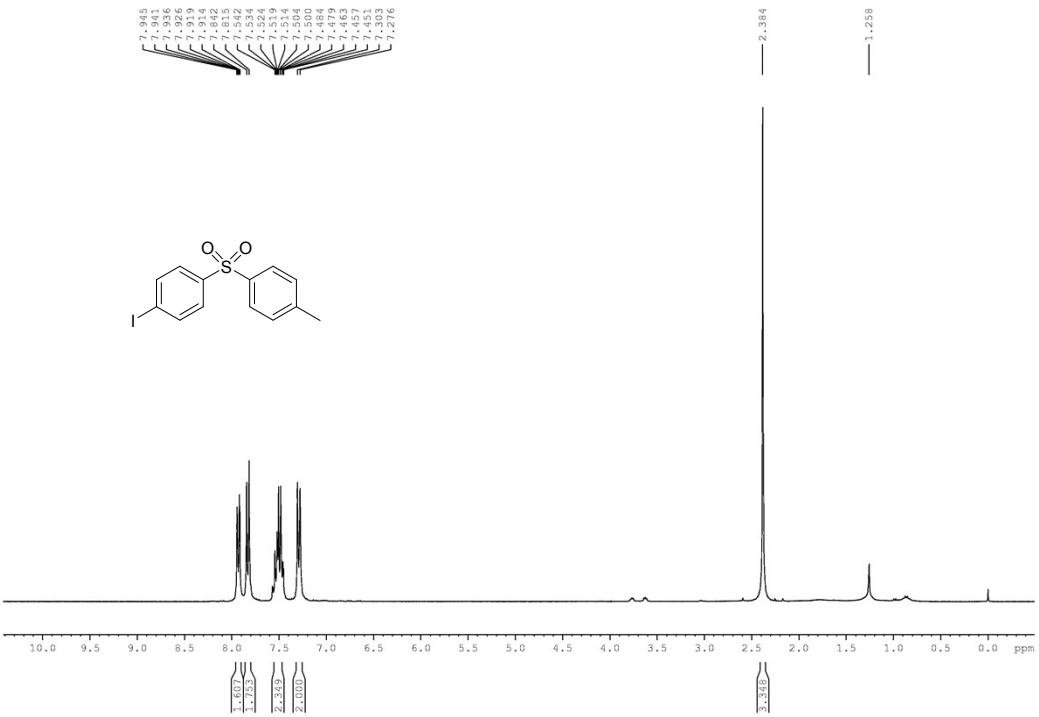

145.114
137.365
136.169
132.942
130.220
127.992
125.850
77.413
76.992
76.569
21.566
Cl
O
O
S
Cl
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

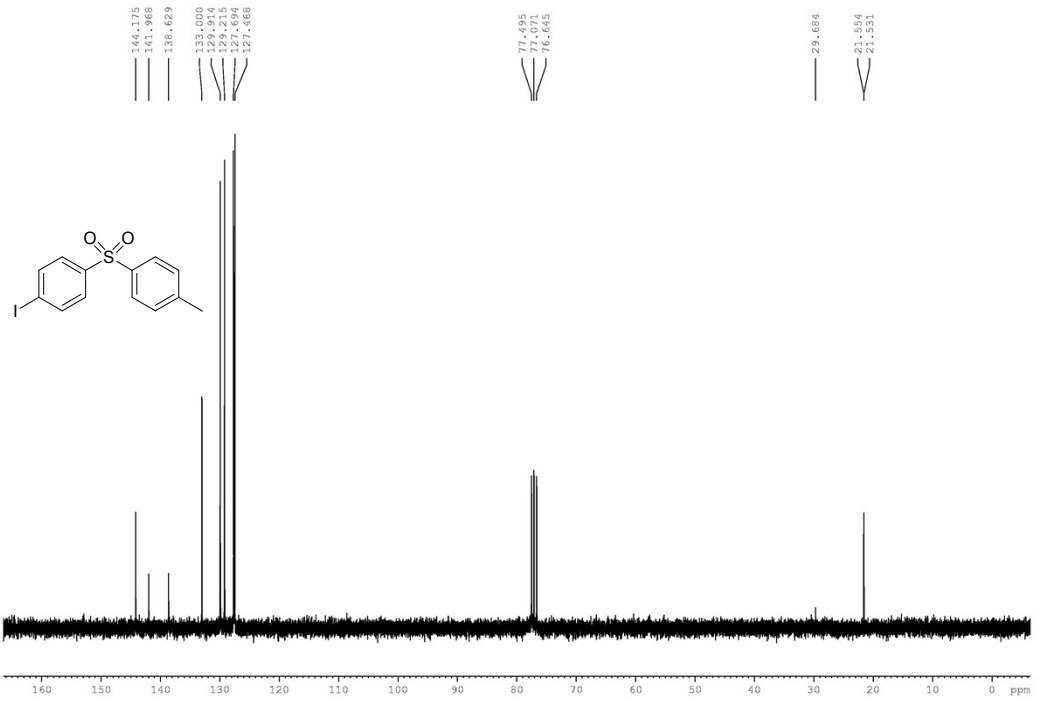
3ap


7.826
7.801
7.776
7.625
7.597
7.344
7.288
2.386
O
O
S
Br
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 ppm
3.366
1.681
2.000
3.146

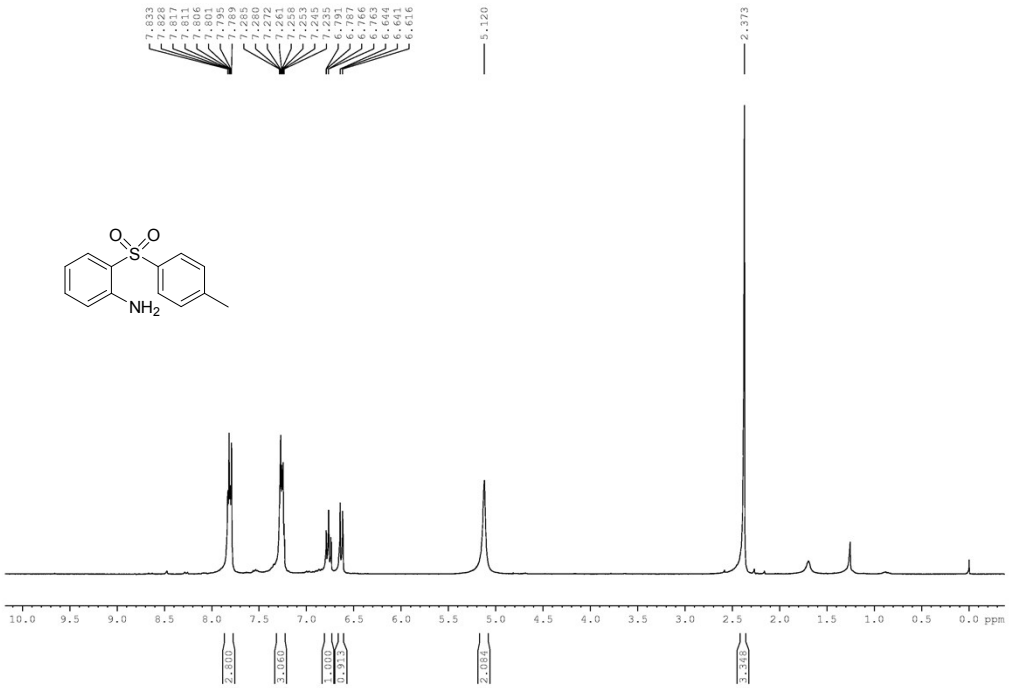

144.573
141.037
138.495
138.115
132.534
130.385
129.050
128.869
128.204
127.698
77.562
77.138
76.713
21.610
O
O
S
Br
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

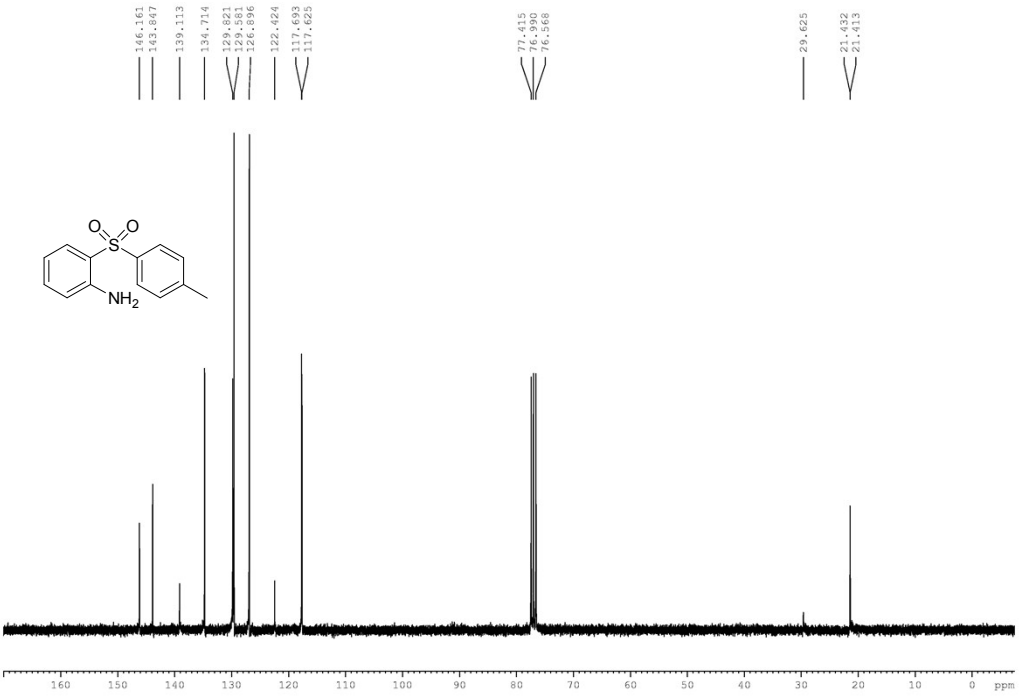
3aq


7.945
7.941
7.936
7.926
7.919
7.914
7.842
7.835
7.672
7.534
7.524
7.519
7.514
7.504
7.500
7.484
7.479
7.463
7.457
7.451
7.303
7.276
2.384
1.258
O
S
O
I
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 ppm
1.607
1.753
2.349
2.000
3.348

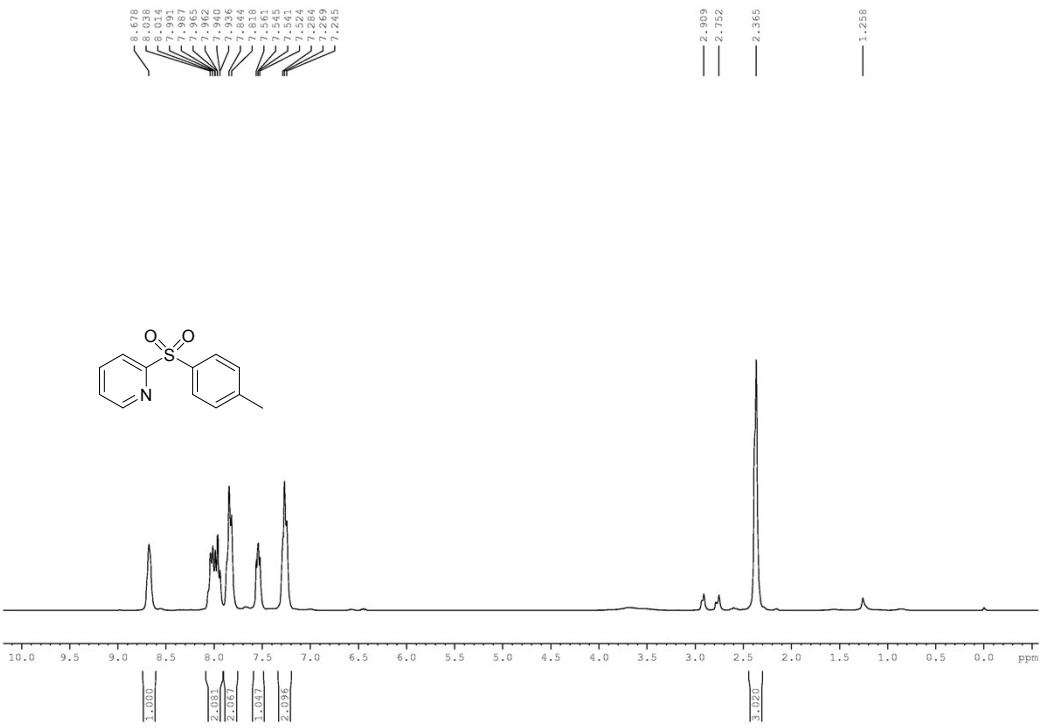

144.175
141.968
138.629
133.000
129.914
129.215
127.694
127.468
77.495
77.071
76.645
29.684
21.554
21.531
O
S
O
I
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

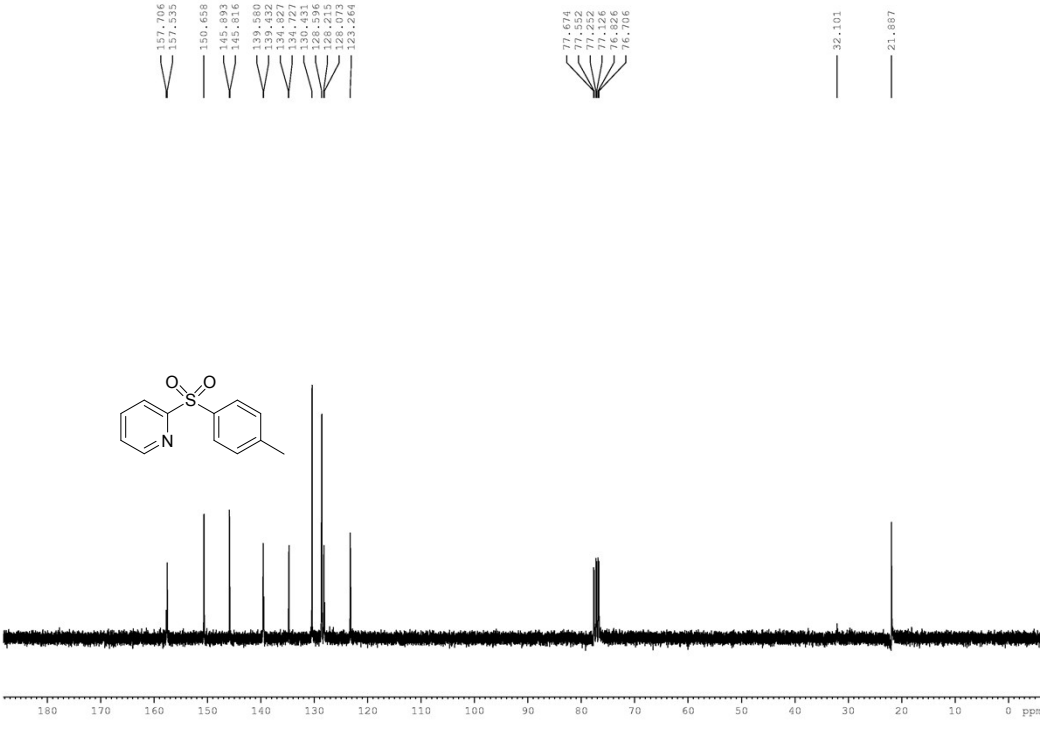
3ar


7.833
7.828
7.817
7.811
7.806
7.801
7.795
7.789
7.285
7.280
7.272
7.261
7.258
7.253
7.245
7.235
6.791
6.787
6.766
6.763
6.644
6.641
6.616
5.120
2.373
O
O
S
NH2
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 ppm
2.800
3.060
1.000
0.913
2.084
3.348

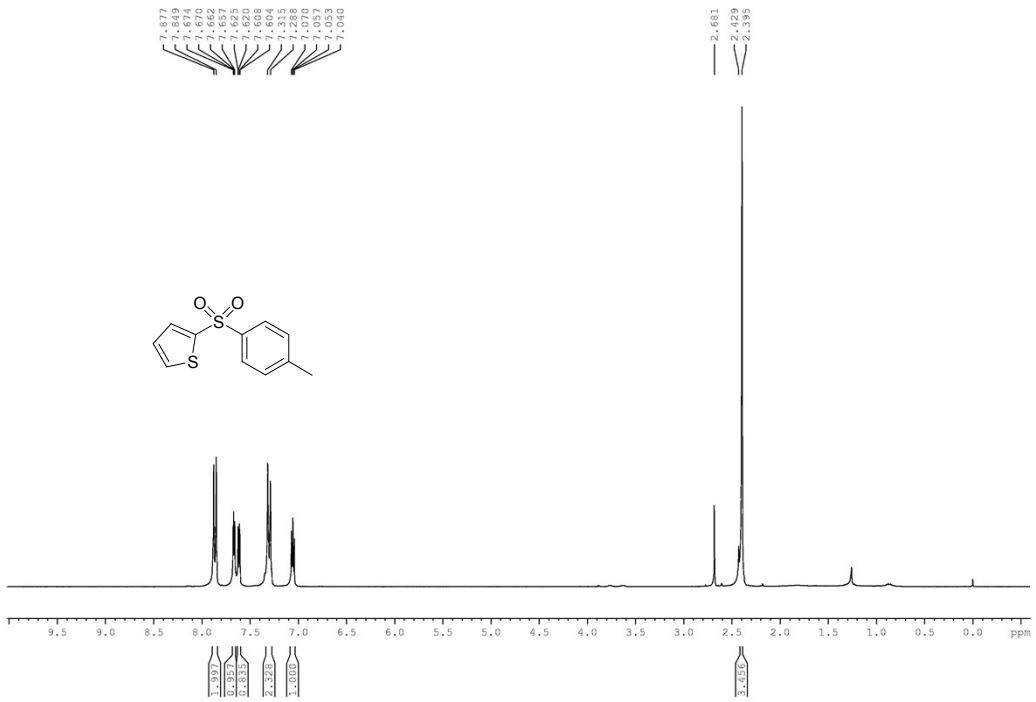

146.161
143.847
139.113
134.714
129.821
129.581
126.896
122.424
117.693
117.625
77.415
76.990
76.568
29.625
21.432
21.413
O
O
S
NH2
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

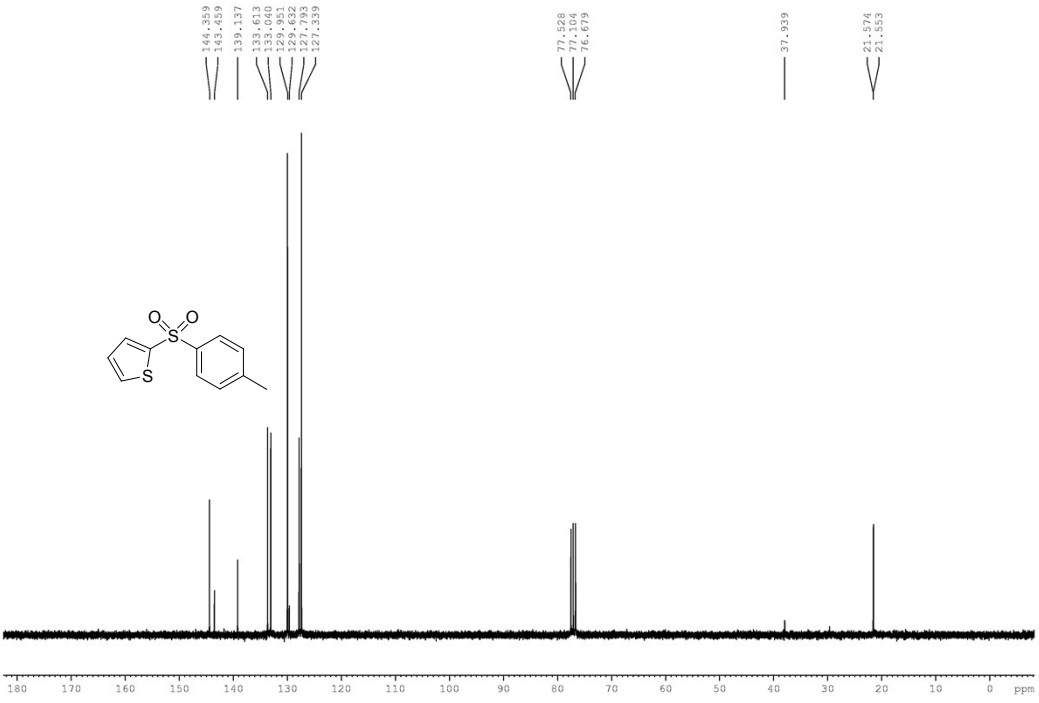
3as


8.678
8.038
8.014
7.991
7.987
7.965
7.962
7.940
7.936
7.844
7.818
7.561
7.545
7.541
7.524
7.284
7.269
7.245
2.909
2.752
2.365
1.258
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 ppm
1.000
2.081
2.067
1.047
2.096
3.020

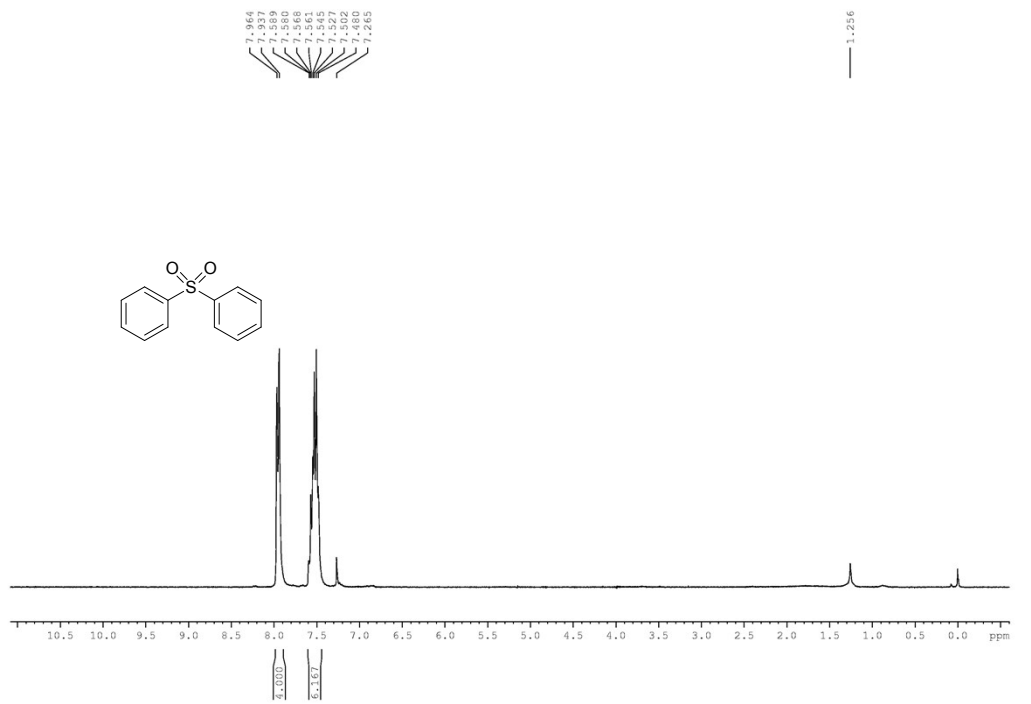

157.706
157.535
150.658
145.893
145.816
139.580
139.432
134.227
134.027
130.431
128.596
128.215
128.073
123.264
77.674
77.552
77.252
77.126
76.826
76.706
32.101
21.887
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

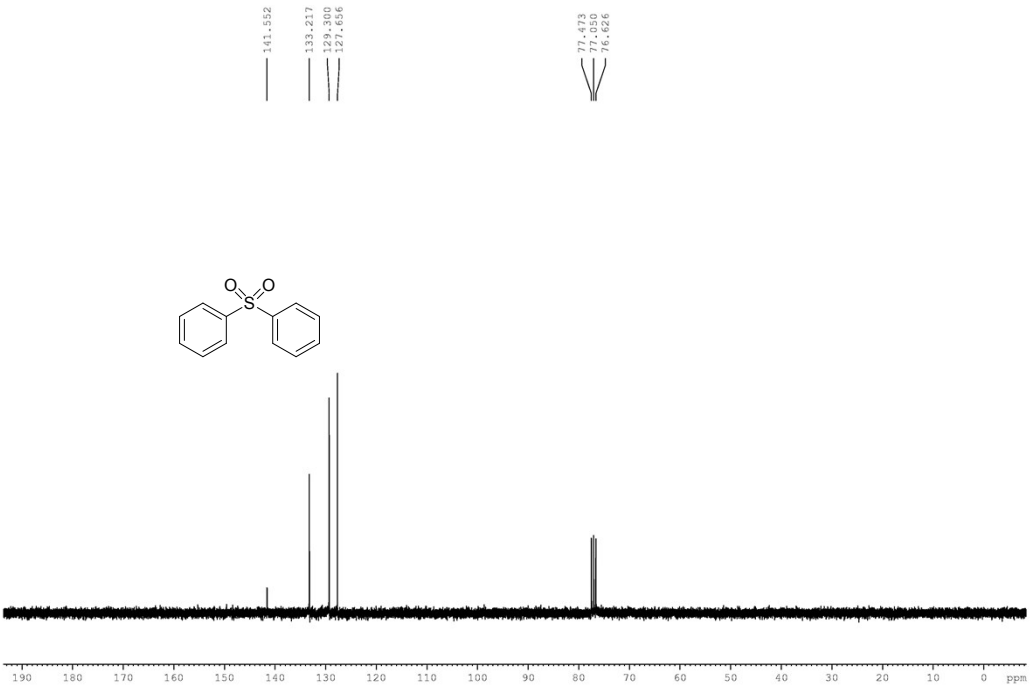
3at


7.877
7.849
7.674
7.670
7.662
7.657
7.625
7.620
7.608
7.604
7.315
7.288
7.070
7.057
7.053
7.040
2.681
2.429
2.395
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 ppm
1.997
0.957
0.835
2.378
1.000
3.456

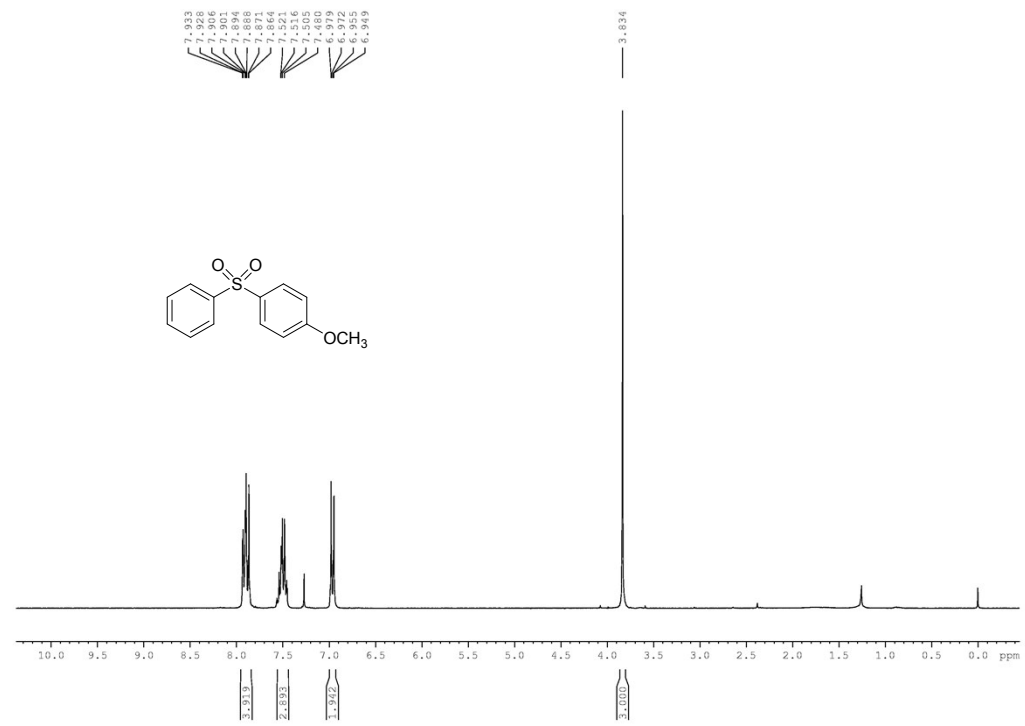

144.359
143.459
139.137
133.613
133.040
129.951
129.632
127.793
127.339
77.526
77.104
76.679
37.939
21.574
21.553
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

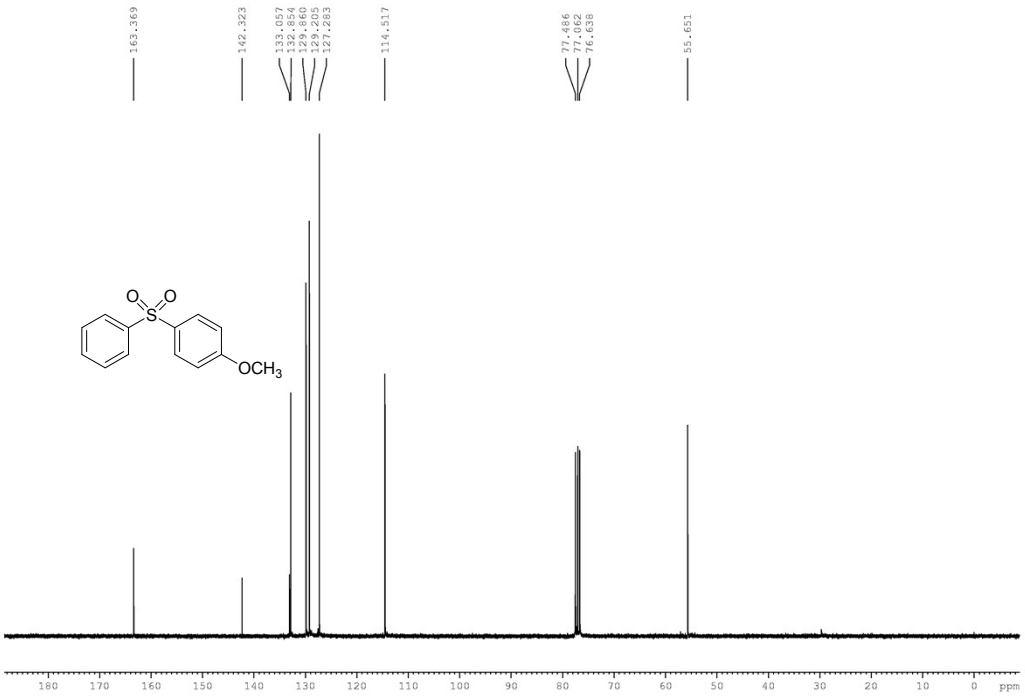
3ba


7.964
7.937
7.589
7.580
7.568
7.561
7.545
7.527
7.502
7.480
7.265
1.256
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 ppm
4.000
6.167

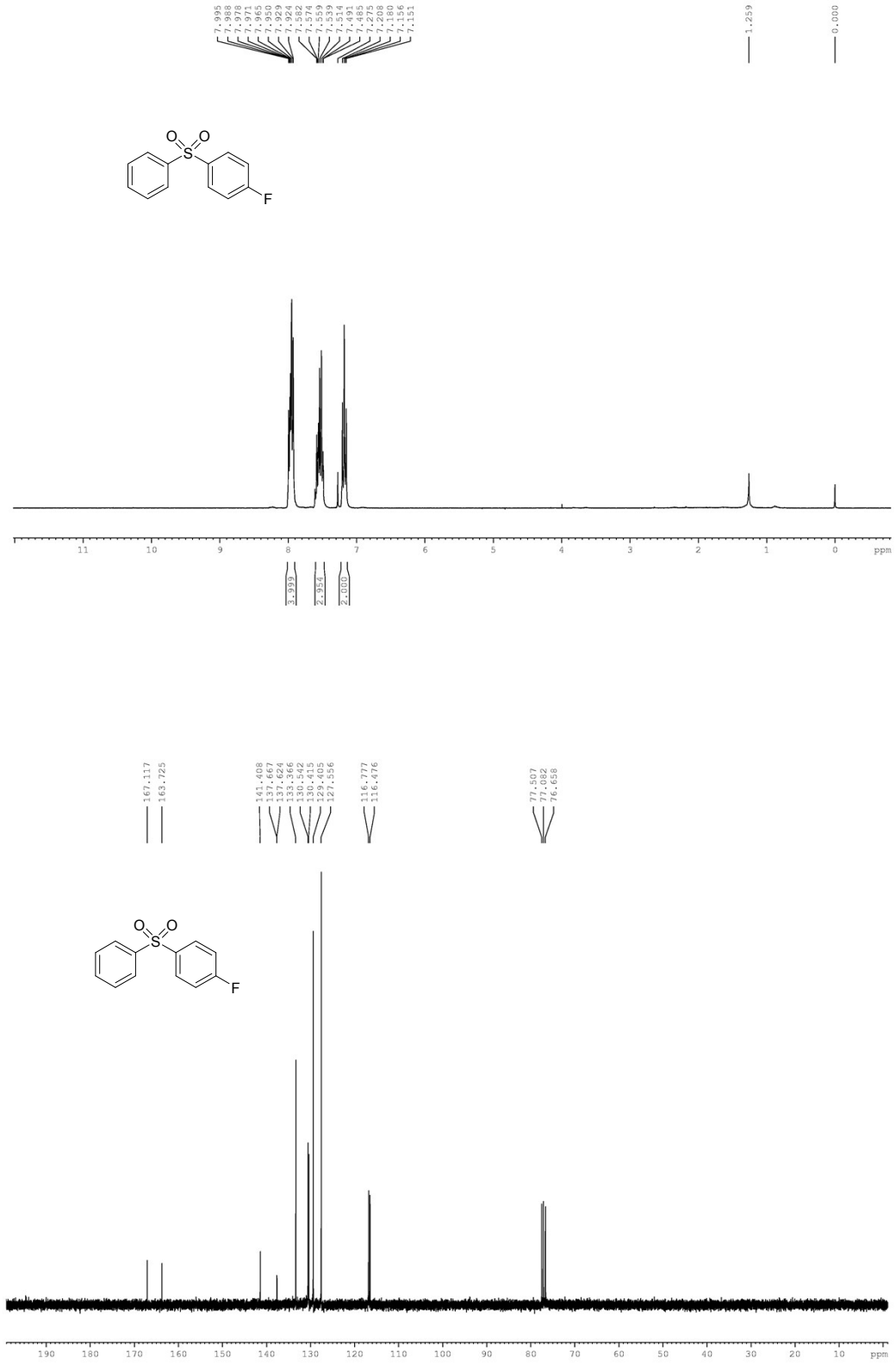

141.552
133.217
129.300
127.656
77.473
77.050
76.626
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3bb

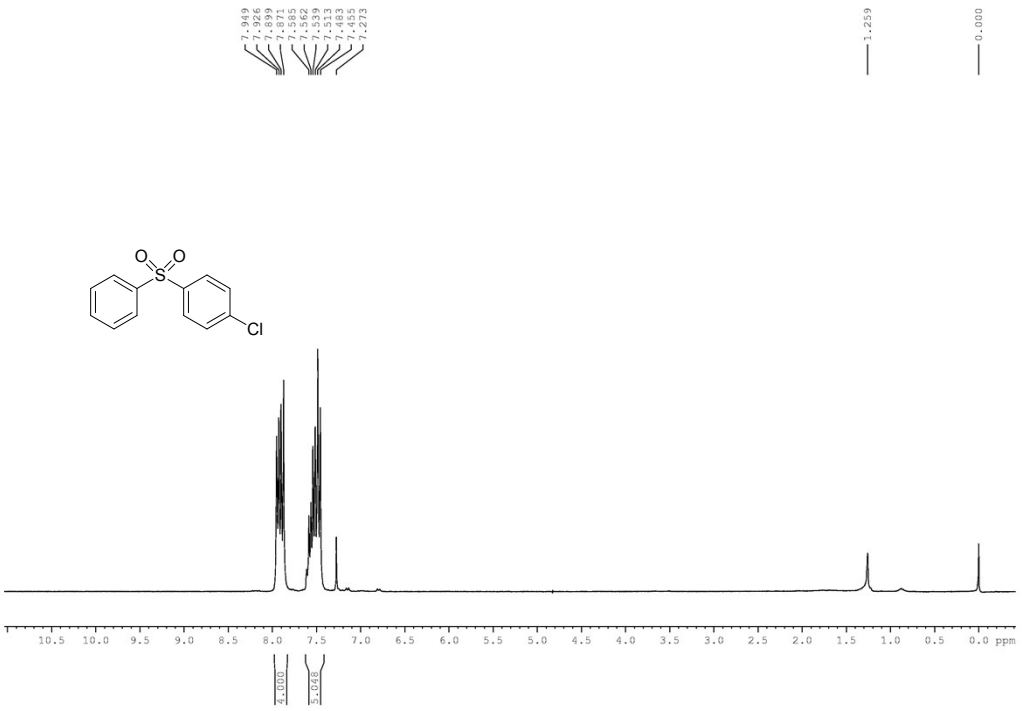

7.933
7.928
7.906
7.901
7.884
7.880
7.871
7.864
7.521
7.516
7.505
7.500
6.979
6.972
6.955
6.949
3.834
O
O
S
OCH3
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
3.919
2.893
1.947
3.000

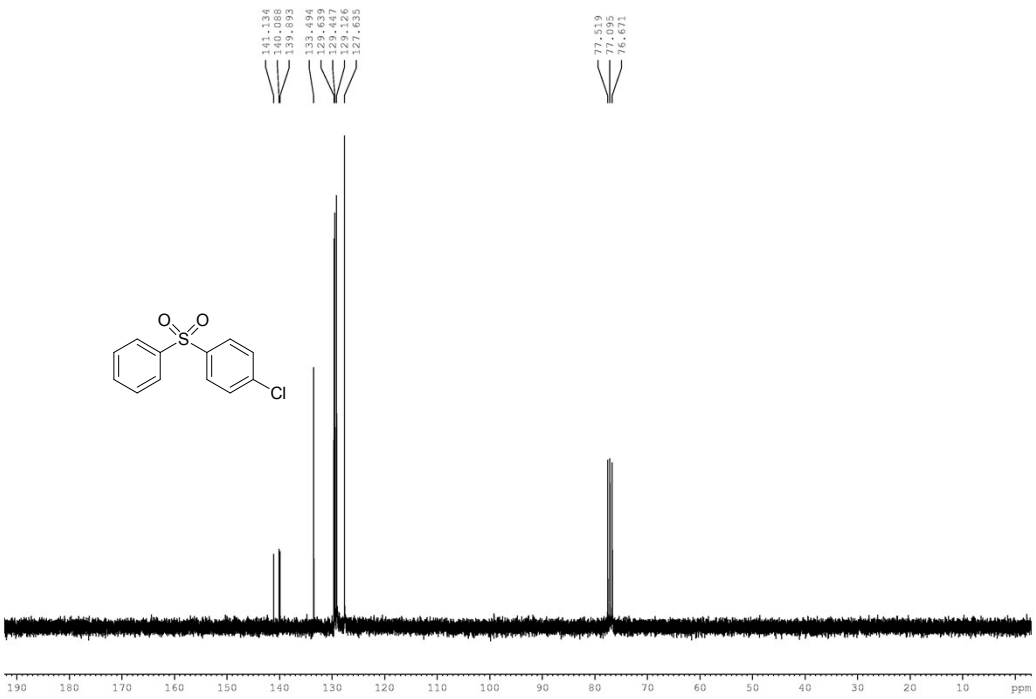

163.369
142.323
133.057
132.554
129.860
129.205
127.283
114.517
77.486
77.062
76.638
55.651
O
O
S
OCH3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3bc


7.995
7.988
7.978
7.971
7.961
7.950
7.929
7.924
7.582
7.574
7.559
7.539
7.514
7.491
7.485
7.275
7.008
7.180
7.156
7.151
1.259
0.000
O
S
O
F
11
10
9
8
7
6
5
4
3
2
1
0
ppm
3.999
2.954
2.000
167.117
163.725
141.408
137.667
137.624
133.366
130.542
130.415
129.405
127.556
116.777
116.476
77.507
77.082
76.658
O
S
O
F
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

3bd


7.949
7.926
7.899
7.871
7.585
7.562
7.539
7.513
7.483
7.455
7.273
1.259
0.000
Cl
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 ppm
4.000
5.046


141.134
140.088
139.993
133.494
129.639
129.447
129.126
127.635
77.519
77.095
76.671
Cl
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm