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Oxyhalogenation of thiols and disulfides into sulfonyl chlorides/ bromides in water using oxone-KX(X= Cl or Br)

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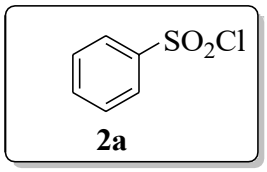
General Information: Oxone®($2KHSO_5 \cdot KHSO_4 \cdot K_2SO_4$), *N*-chlorosuccinimide, KBr, were purchased from Sigma-Aldrich India Ltd.,. aq. HCl, aq. HBr, NaCl, KCl, KI and other solvents used in this study were procured from SD Fine Chem. Ltd., India. Melting points of the compounds were recorded on Veego programmable melting point apparatus in open capillaries and are uncorrected. IR spectra were recorded on a PerkinElmer FT-IR 240-C spectrophotometer using KBr & neat optics. ^{1}H NMR spectra were recorded on Bruker AV 300 MHz in $CDCl_3$ using TMS as the internal standard. All the reactions were monitored by thin layer chromatography (TLC) on precoated silica gel 60 F254 (mesh); spots were visualized

with UV light or by charring with anisaldehyde solution. Merck silica gel (60-120 mesh) was used for column chromatography.

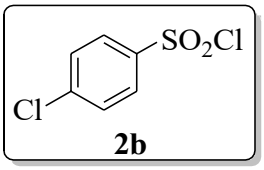
General procedure for the preparation of sulfonyl chlorides with oxone-KX: A mixture of thiol (3.4 mmol), oxone (8.6 mmol) and KCl (3.4 mmol), water (10 mL) was taken into a round bottomed flask and stirred at room temperatur. This reaction is slightly exothermic and temperature of the mixture rose to 45°C. After completion of the reaction (TLC), the reaction mixture was extracted with ethyl acetate (4x5 mL). The combined organic layers was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product obtained was purified by normal column chromatography (silica gel 60-120 mesh, *n*-hexane) to obtain corresponding sulfonyl chloride. A similar procedure was used for preparation of sulfonyl bromides with oxone-KBr.

General procedure for the preparation of sulfonyl chlorides from disulfides: A mixture of disulfide (1.7 mmol), oxone (3.5 mmol) and KCl (3.5 mmol), water (10 mL) was taken into a round bottomed flask and stirred at room temperature. After completion of the reaction (TLC), the reaction mixture was extracted with ethyl acetate (4x5 mL) and the combined organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product obtained was purified by normal column chromatography (silica gel 60-120 mesh, *n*-hexane) to obtain corresponding sulfonyl chloride.

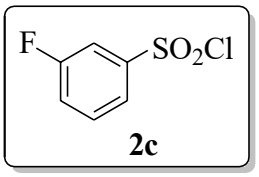
Characterization data:



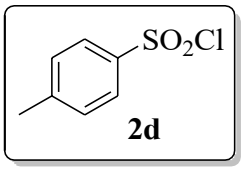
Benzenesulfonyl chloride (2a). Colorless oil, (0.31 g, 98%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.05-8.04 (m, 2H), 7.76 (t, J = 7.3 Hz, 1H), 7.62 (t, J = 7.7 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ =136.4, 133.6, 131.3, 129.3, 128.7, 127.4; IR (neat): υ 3065, 2926, 2855, 1444, 1326, 1144, 1077, 750, 593 cm^{-1}. EI-MS 176, 159, 112, 95, 75, 57; EI-HRMS: Exact mass observed for $C_6H_5ClO_2S$: 175.9695 (calculated: 175.9698).



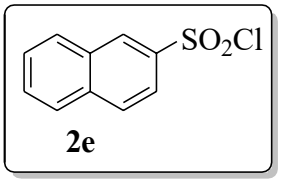
4-chlorobenzene-1-sulfonyl chloride (2b). White solid, m.p. 51-52°C (lit[1] 52-54) (0.28 g, 97%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.98 (d, J = 8.6 Hz, 2H) 7.60 (d, J = 8.6 Hz, 2H), ^{13}C NMR (75 MHz, $CDCl_3$): δ = 142.4, 140.4, 137.6; IR (neat): υ 3109, 3052, 1573, 1474, 1184, 1088, 825, 755, 559 cm^{-1}. EI-MS 212, 201, 177, 175, 111, 75, 69; EI-HRMS: Exact mass observed for $C_6H_4Cl_2O_2S$: 209.9319(calculated: 209.9309).



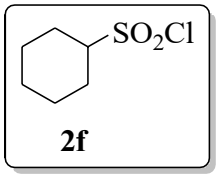
3-Fluorobenzene-1-sulfonyl chloride (2c). Pale yellow low-melting solid, m.p. 6-8 $^{\circ}$C (Lit[2] 7 °C), (0.28 g, 95%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.98 (d, J = 8.1 Hz, 2H), 7.30 (d, J =8.1Hz, 2H), 6.69 (s, 1H), 2.44 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 185.5, 145.6, 129.7, 129.5, 128.0, 39.9, 21.7; IR (neat): υ 3108, 3056, 2927, 2757, 1593, 1474, 1368, 1228, 1163, 884, 595 cm^{-1}. EI-HRMS: Exact mass observed for $C_6H_4ClFO_2S$: 193.9602(calculated: 193.9604).



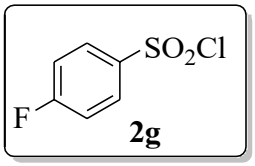
4-Methylbenzene-1-sulfonyl chloride (2d): White solid m.p. 72-73 ^{o}C (lit[3] 71-72) (0.29 g, 96%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.89 (d, J = 8.4 Hz, 2H), 7.43 (d, J = 8.4 Hz, 2H), 2.59 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ =146.8, 130.2, 126.9, 21.7; IR (KBr): υ 3009, 1690, 1598, 1481, 1231, 993, 796, cm^{-1}. EI-MS 191, 175, 128, 111, 75, 55; EI-HRMS: Exact mass observed for $C_7H_7ClO_2S$: 189.9854(calculated: 189.9855).



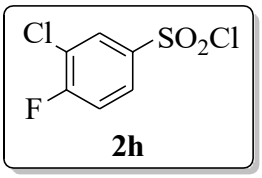
Naphthalene-1-sulfonyl chloride (2e). White solid, m.p. 74-75 ^{o}C (lit[1] 73-74); (0.25 g, 92%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.61 (s, 1H), 8.08-7.96 (m, 4H), 7.78-7.67 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ= 130.3, 130.2, 129.8, 128.9, 128.3, 128.1, 121.2; IR (neat): υ 3108, 3073, 2928, 1589, 1492, 1380, 1182, 1081, 840 cm^{-1}. EI-MS: 226, 210, 208, 146, 127, 115, 77, 57; EI-HRMS: Exact mass observed for $C_{10}H_7ClO_2S$: 225.9854 (calculated: 225.9855).



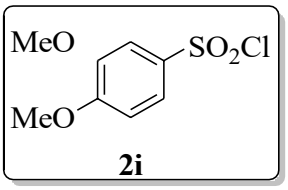
Cyclohexanesulfonyl chloride (2f). Colorless oil (0.27 g, 89%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.56-3.46 (m, 1H), 2.42-2.37 (m, 2H), 2.01-1.95 (m, 2H), 1.75-1.62 (m, 3H), 1.48-1.18 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 74.7, 27.0, 24.8, 24.5; IR (neat): υ 2938, 2859, 1451, 1369, 1219, 1160, 751, 589 cm^{-1}; EI-MS: m/z. 182, 118, 99, 83, 67, 55; EI-HRMS: Exact mass observed for $C_6H_{11}ClO_2S$: 182.0164 (caluculated:182.0168).



4-Fluorobenzene-1-sulfonyl chloride (2g). Colorless oil (0.28 g, 95). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.10-8.07 (m, 2H), 7.33-7.28 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 168.1, 130.1, 130.0, 117.2, 116.9; IR (neat): υ 3108, 3073, 2928, 1589, 1492, 1380, 1182, 840, 569 cm^{-1}. EI-HRMS: Exact mass observed for $C_6H_4ClFO_2S$: 193.9601(calculated: 193.9604).

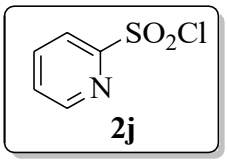


3-Chloro-4-fluorobenzene-1-sulfonyl chloride (2h). Colorless oil (0.25 g, 92). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.14 (dd, J = 2.4 Hz, 1H), 8.01-7.92 (m, 1H), 7.43-7.39 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ =163.7, 130.1, 127.8, 127.6, 118.2, 117.8; IR (neat): υ 2927, 2757, 1593, 1474, 1228, 1163, 1101, 884 cm^{-1}. EI-MS: 227, 224, 195, 193, 129. 129, 109, 94, 79; EI-HRMS: Exact mass observed for $C_6H_3Cl_2FO_2S$: 227.9211(calculated: 227.9214).



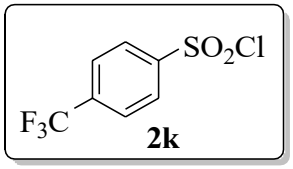
3,4-Dimethoxybenzene-1-sulfonyl chloride (2i). White solid, 63-65, (lit[4] 64-65), (0.24 g, 90%).^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.68 (dd, J = 2.2 Hz, 1H), 7.44 (d, J = 2.2 Hz, 1H), 7.68 (d, J = 8.5 Hz, 1H), 3.99 (s, 3H), 3.97 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ =154.6, 149.2, 135.8, 121.6110.4, 108.956.4, 56.3; IR (neat): υ 2929, 2862, 1461, 1406,

1229, 1183, 1119, 1034, 819 cm^{-1} . EI-MS: 236, 201, 153, 137, 94, 79; EI-HRMS: Exact mass observed for $C_8H_{10}ClO_4S$: 235.9916(calculated: 235.9910).



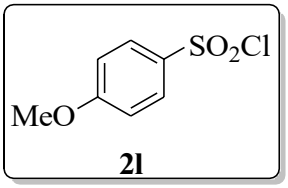
2j

Pyridine-2-sulfonyl chloride (2j). Colorless oil (0.28 g, 88%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.64-8.55 (m, 1H), 7.98-7.85 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-D_6): δ = 155.2, 146.0, 140, 126.1, 123.3; IR (neat): υ 3029, 1453, 1028, 638 cm^{-1}.EI-MS 177, 175, 159, 111, 69, 57; EI-HRMS: Exact mass observed for $C_5H_4ClO_2S$:176.96526(calculated: 176.96513).



2k

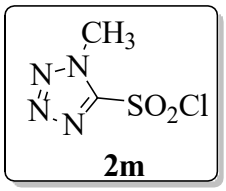
4-(Trifluoromethyl) benzene-1-sulfonyl chloride (2k): Colorless oil (024 g, 90%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.20 (d, J = 8.3 Hz, 2H), 7.92 (d, J = 8.3 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 147.0, 136.9136.4, 136.1, 127.6, 127.0, 126.9; IR (neat): υ 3026, 1454, 1242, 1145, 1014, 940, 780 cm^{-1}; EI-HRMS: Exact mass observed for $C_7H_4ClF_3O_2S$: 243.9569(calculated: 243.9572).



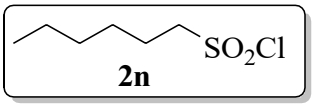
2l

4-Methoxybenzene-1-sulfonyl chloride (2l). Colorless oil (0.27 g, 93%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.97 (d, J = 9.0 Hz, 2H), 7.04 (d, J = 9.0 Hz, 2H), 3.92 (s, 3H) ^{13}C NMR (75 MHz, $CDCl_3$): δ = 159.8, 132.6, 128.3, 114.5, 55.3; IR (neat): υ 3052, 3007, 2926, 2854, 1574, 1463, 1367, 1281, 1171, 1088, 944 cm^{-1}. EI-MS 206, 190, 175, 142,

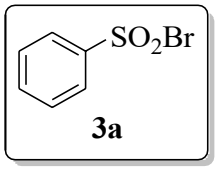
75, 55; EI-HRMS: Exact mass observed for $C_7H_7ClO_3S$:205.9803 (calculated: 205.9804).



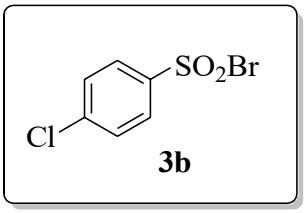
1-Methyl-1*H*-tetrazole-5-sulfonyl chloride (2m). Colorless oil (0.27 g, 88%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 40.18 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 143.08; IR (neat): υ 2928, 2858, 1633, 1343, 1056, 771cm^{-1}. Mass: ESI-MS:181(M+H), 203 (M+Na).



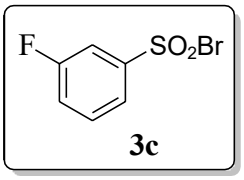
Hexane-1-sulfonyl chloride (2n). Colorless oil (0.21 g, 68). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.38 (t, J = 7.3, 7.4 Hz, 2H), 1.69-1.62 (m, 2H), 1.41-1.30 (m, 6H), 0.89 (t, J = 6.6, 6.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 62.5, 36.1, 31.0, 23.3, 22.3, 13.8; IR (neat): υ 2929, 2857, 1463, 1378, 1256, 988, 725 cm^{-1}. EI-MS 184, 141, 125, 109, 77, 69, 57; EI-HRMS: Exact mass observed for $C_6H_{13}ClO_2S$: 184.0322 (calculated: 184.0324).



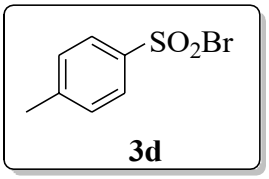
Benzenesulfonyl bromide (3a). Colorless oil (0.38 g, 96%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.02-7.99 (m, 2H), 7.76 (t, J = 7.3 Hz, 1H), 7.63 (t, J = 7.7 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ =136.4, 133.6, 131.3, 129.3, 128.7, 127.4; IR (neat): υ 3022, 2919, 1489, 1329, 1143, 806, 653 cm^{-1}. EI-MS 220, 171, 158, 137, 97, 69; EI-HRMS: Exact mass observed for $C_6H_5ClO_2S$: 219.91920 (calculated: 219.91936).



4-Chlorobenzene-1-sulfonyl bromide (3b). White solid, m.p. 74-75°C (lit[5] 73-74); (0.35 g, 95%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.98 (d, J = 8.6 Hz, 2H) 7.60 (d, J = 8.6 Hz, 2H), ^{13}C NMR (75 MHz, $CDCl_3$): δ = 142.4, 140.4, 130.0; IR (neat): υ 3109, 3052, 1573, 1474, 1184, 1088, 825, 755, 559 cm^{-1}. EI-MS 256, 254, 177, 175, 113, 111, 75, 76; EI-HRMS: Exact mass observed for $C_6H_4Cl_2O_2S$: 253.87998(calculated: 253.88039).

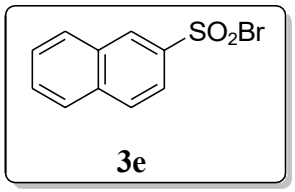


3-Fluorobenzene-1-sulfonyl bromide (3c). Colorless oil (0.34 g, 92%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.84-7.80 (m, 2H), 7.72-7.60 (m, 2H), 7.49-7.43 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 163.5, 131.4, 122.7, 122.4, 114.1, 113.7; IR (neat): υ 3073, 2938, 1564, 1485, 1378, 1192, 846 cm^{-1}. EI-HRMS: Exact mass observed for $C_6H_4BrFO_2S$: 237.9096(calculated: 237.9099).

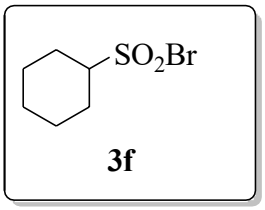


4-Methylbenzene-1-sulfonyl chloride (3d). White solid m.p. 74-75 ^{o}C (lit[6] 73-74); (0.33 g, 89%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.87 (d, J = 8.0 Hz, 2H), 7.39 (d, J = 8.0 Hz, 2H), 2.46 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ =142.20, 136.81, 130.06, 126.40, 21.80; IR (KBr): υ 3010, 2923, 1584, 1457, 1027, 696 cm^{-1}.EI-MS 234, 202, 186, 171, 139,

123, 107, 92, 77; EI-HRMS: Exact mass observed for $C_7H_7BrO_2S$: 233.9348(calculated: 233.9350).



Naphthalene-2-sulfonyl bromide (3e). pale yellow solid m.p. 74-75 ^{o}C (lit[6] 73-74); (0.30 g, 90%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.62 (s, 1H), 8.10-7.99 (m, 4H), 7.80-7.70 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ= 135.6, 132.1, 130.3, 130.1, 129.9, 128.3, 128.1, 121.0; IR (neat): υ 3063, 2946, 1589, 1455, 1237, 1025, 840 cm^{-1}. EI-MS: 270, 185, 149, 139, 123, 69, 57; EI-HRMS: Exact mass observed for $C_{10}H_7ClO_2S$: 269.93500 (calculated: 269.93501).



Cyclohexanesulfonyl bromide (3f). Colorless oil (0.37 g, 95%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.54-3.44 (m, 1H), 2.40-2.36 (m, 2H), 1.97-1.94 (m, 2H), 1.73-1.60 (m, 3H), 1.45-1.12 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 74.7, 27.0, 24.8, 24.5; IR (neat): υ 2938, 2859, 1451, 1369, 1219, 1160, 751, 589 cm^{-1}; EI-MS: m/z. 227, 225, 191, 163, 148, 111, 97, 83, 69, 55; EI-HRMS: Exact mass observed for $C_6H_{11}BrO_2S$: 225.9661 (caluculated:225.9663).

References:

1. K. Bahrami, M. M. Khodaei, M. Soheilizad, K. Donya, *Tetrahedron Lett.*, 2012, **53**, 354-

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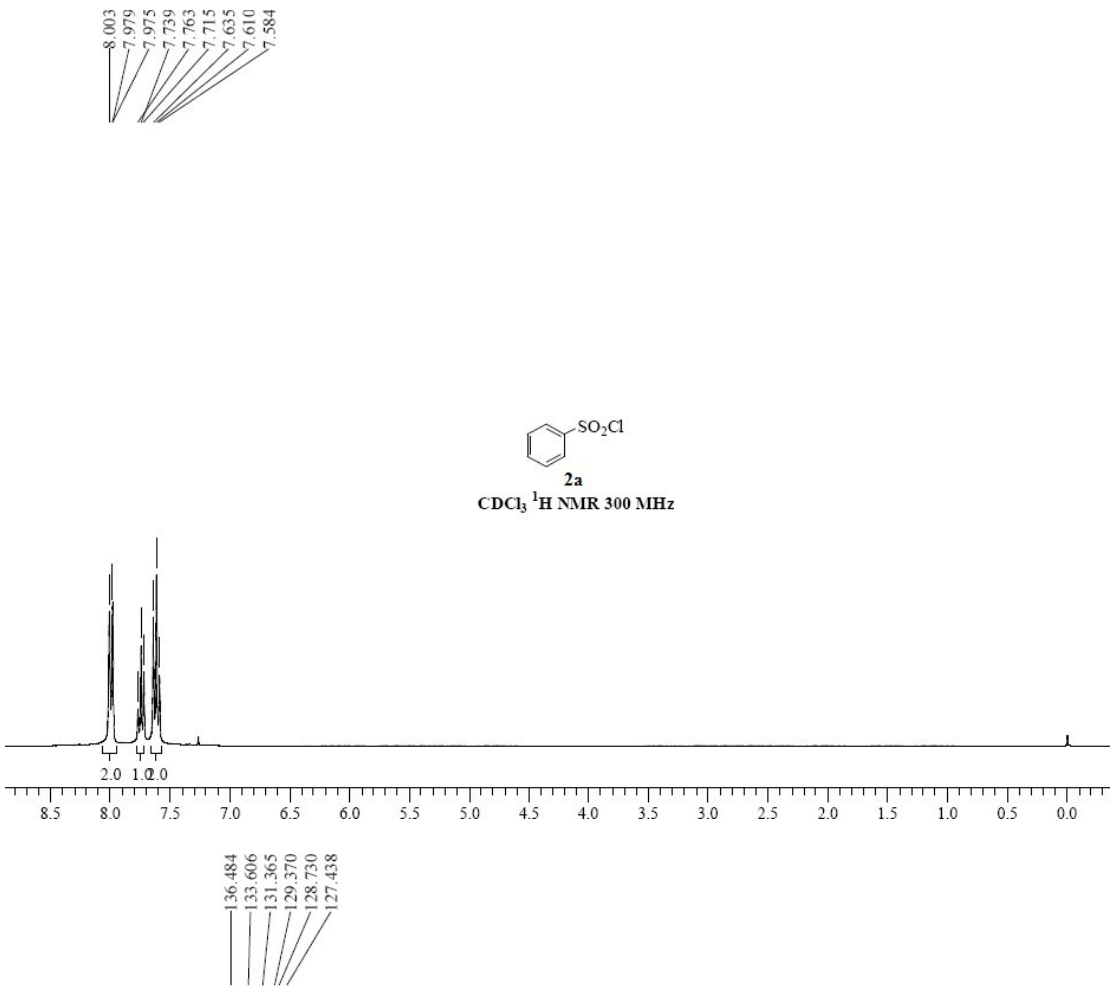
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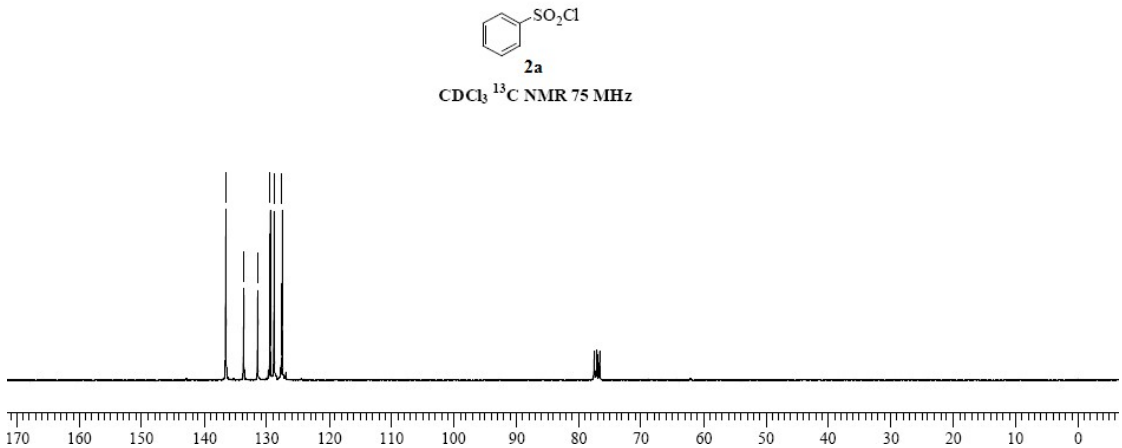
7.979
7.975
7.739
7.763
7.715
7.635
7.610
7.584
SO2Cl
2a
CDCl3 1H NMR 300 MHz
2.0 1.0 2.0
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
136.484
133.606
131.365
129.370
128.730
127.438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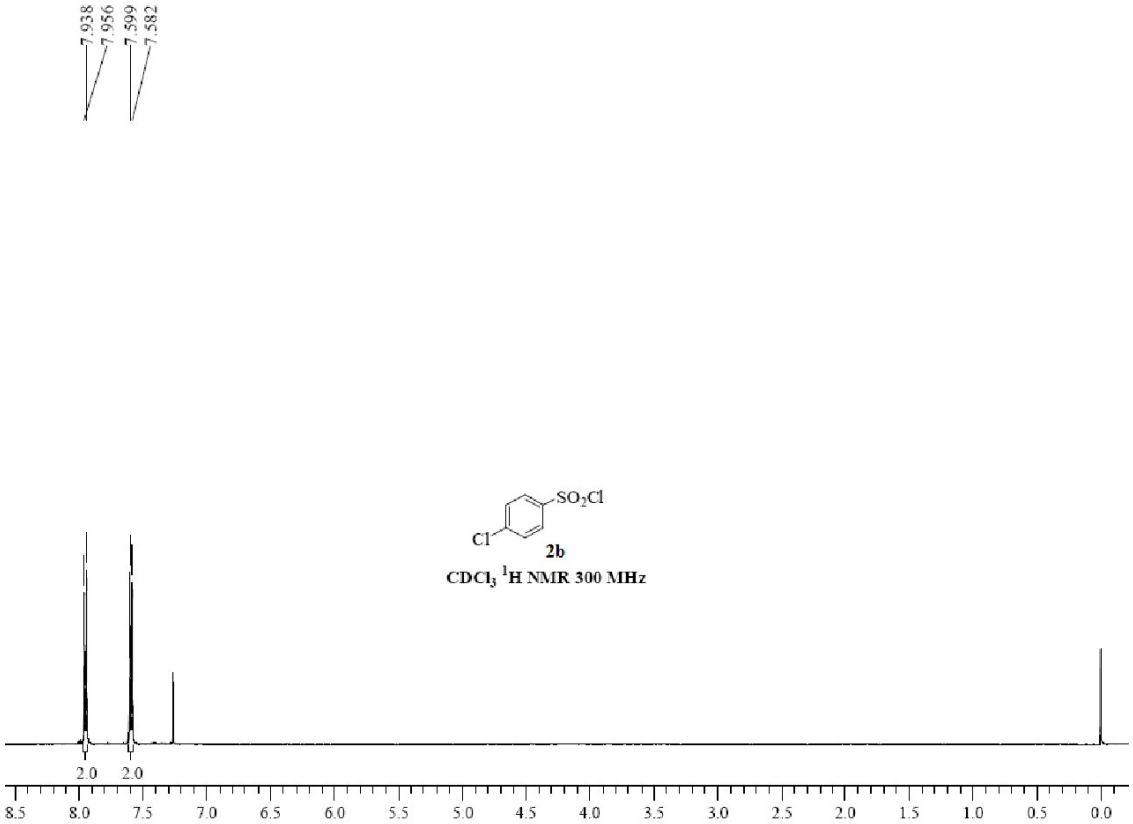
2a
CDCl3 13C NMR 75 MHz
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


7.938
7.956
7.599
7.582
SO2Cl
Cl
2b
CDCl3 1H NMR 300 MHz
2.0
2.0
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
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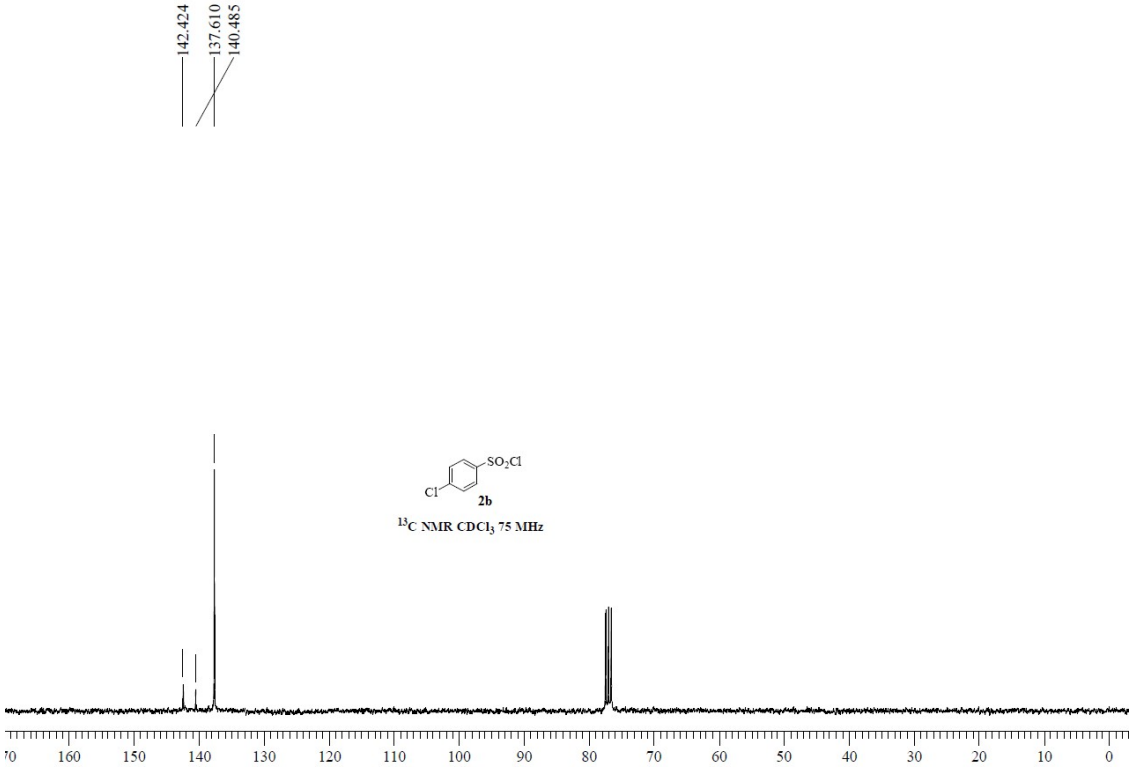
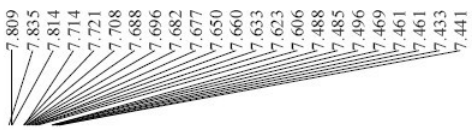
0.0


142.424
137.610
140.485
SO2Cl
Cl
2b
13C NMR CDCl3 75 MHz
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0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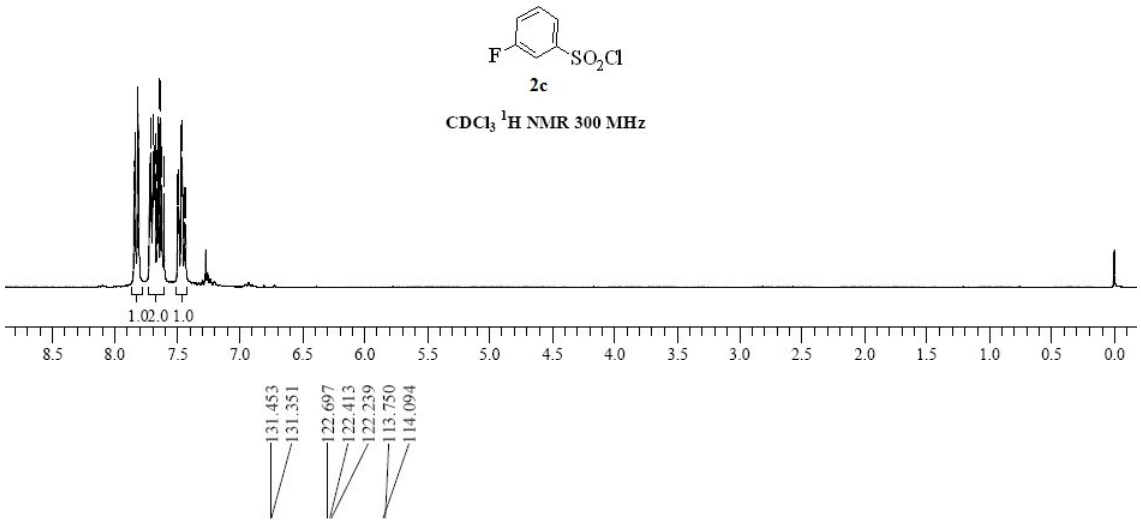
7.835
7.814
7.714
7.721
7.708
7.688
7.696
7.682
7.677
7.650
7.660
7.633
7.623
7.606
7.488
7.485
7.496
7.469
7.461
7.461
7.433
7.441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2c
CDCl3 1H NMR 300 MHz
1.02.0 1.0
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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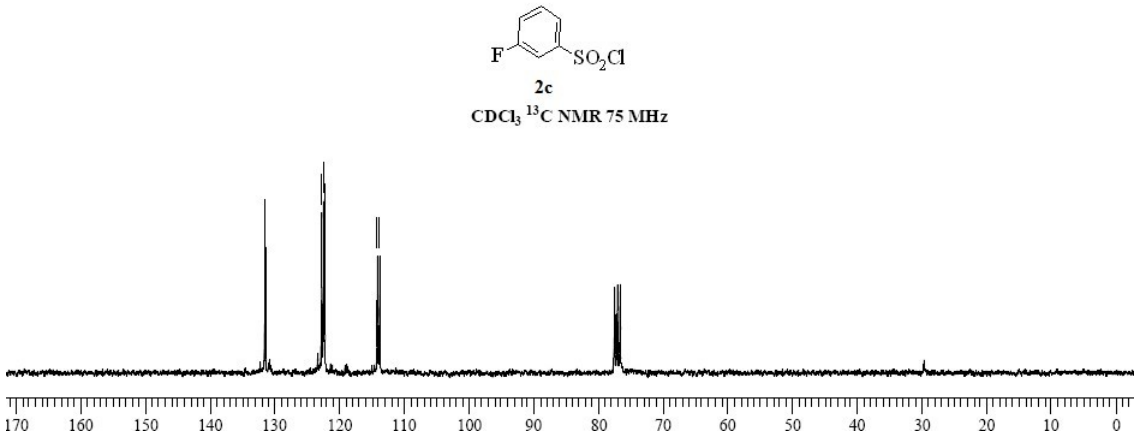
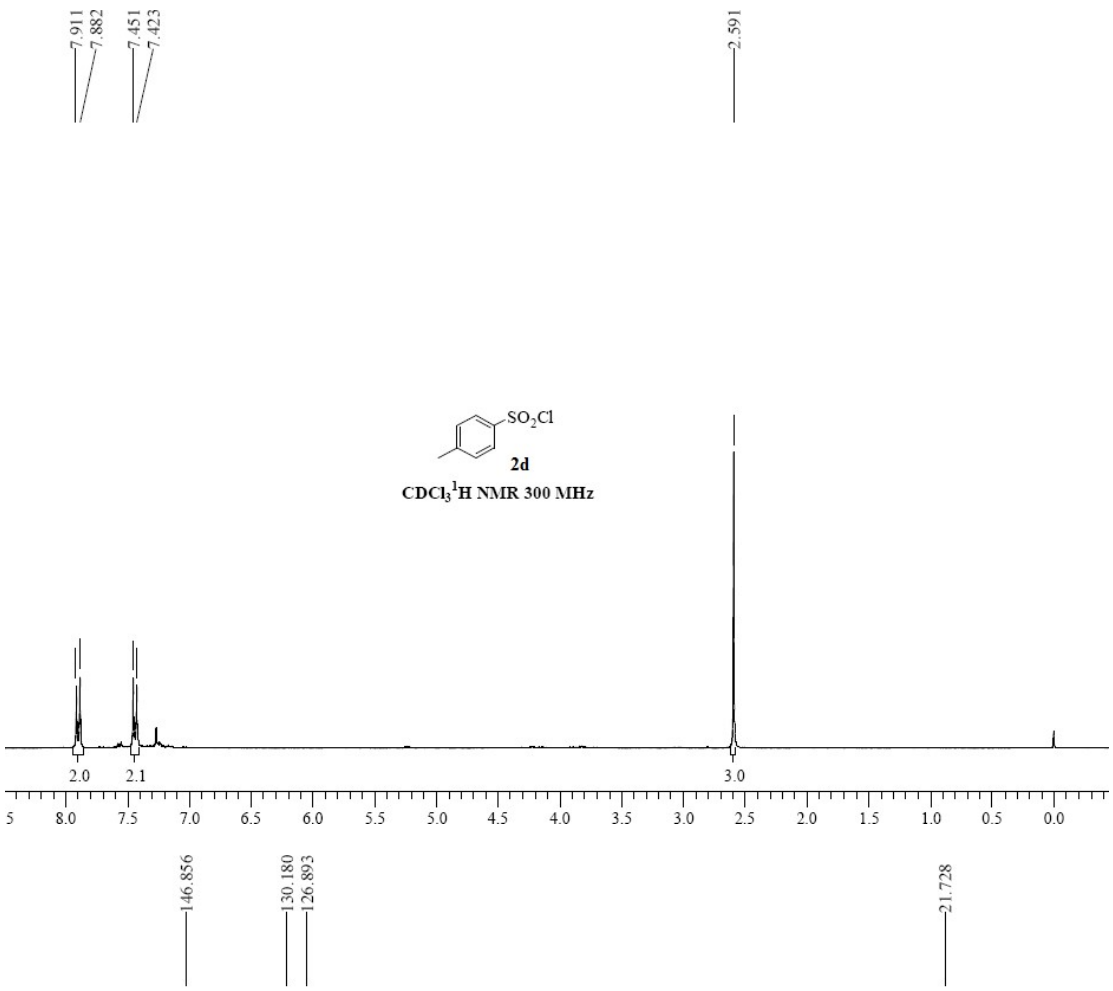
131.351
122.697
122.413
122.239
113.750
114.094


F
SO2Cl
2c
CDCl3 13C NMR 75 MHz
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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7.882
7.451
7.423
2.591
SO2Cl
2d
CDCl3 1H NMR 300 MHz
2.0
2.1
3.0
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
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1.0
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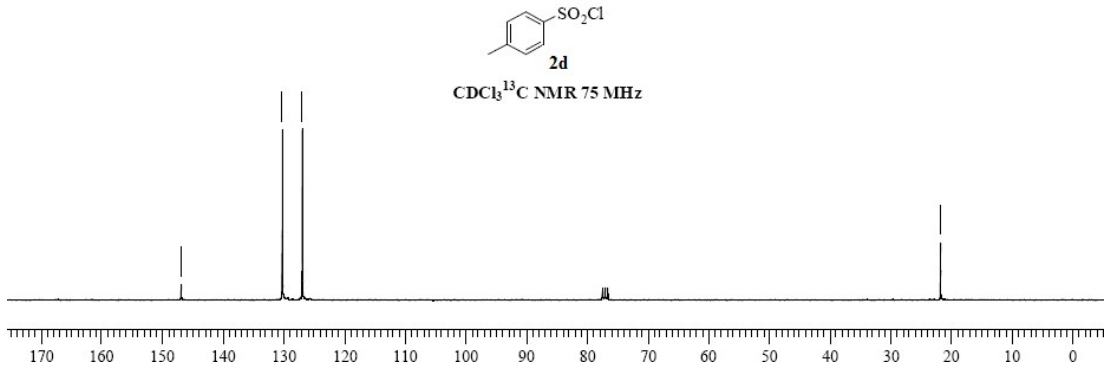
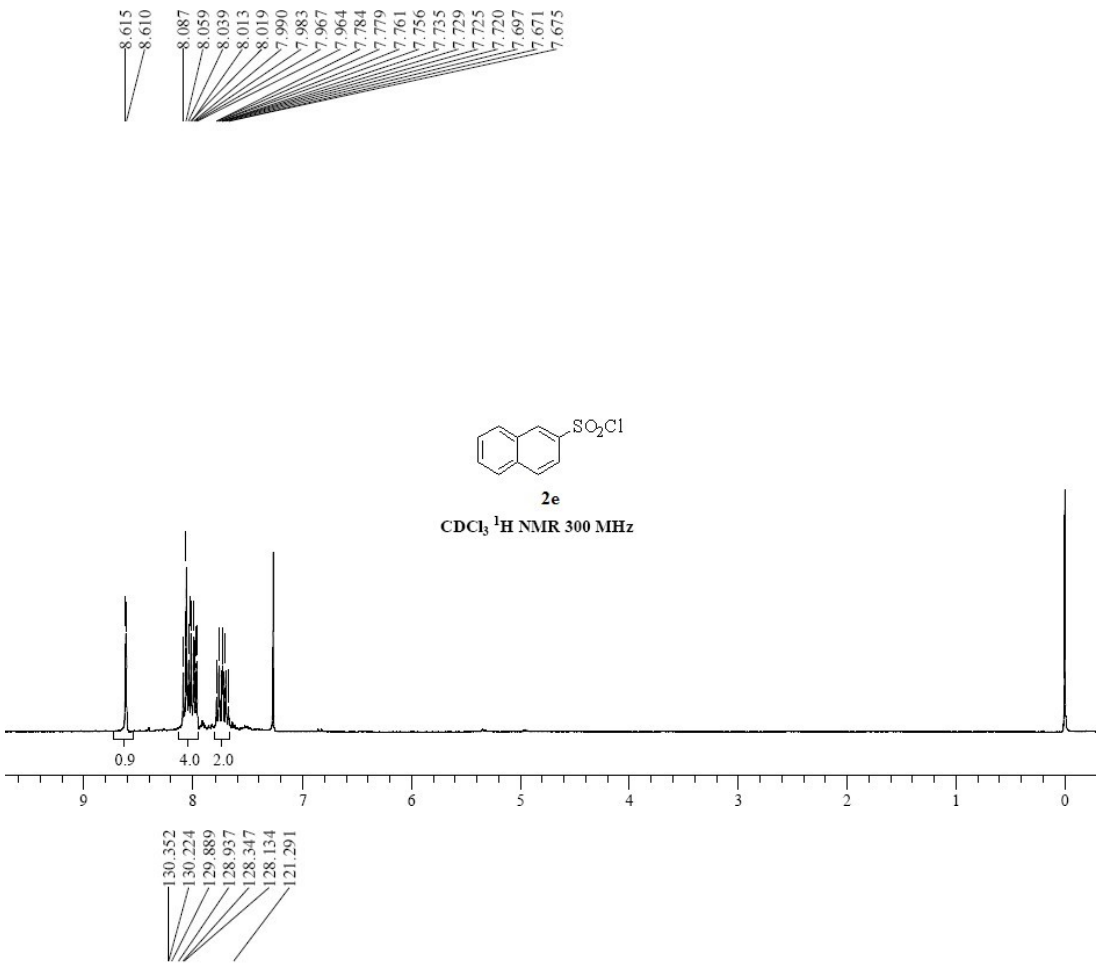
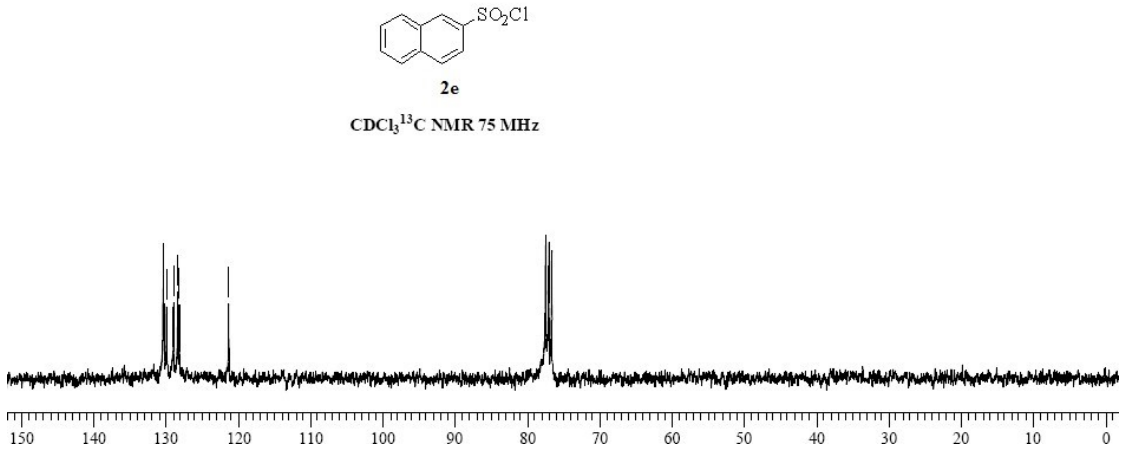
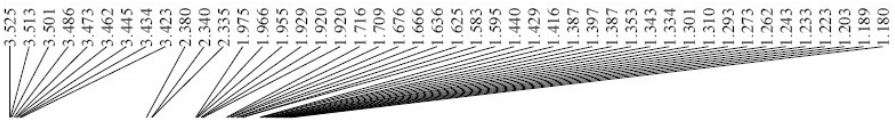
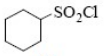
0.0
146.856
130.180
126.893
21.728


SO2Cl
2d
CDCl3 13C NMR 75 MHz
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


8.615
8.610
8.087
8.059
8.039
8.013
8.019
7.990
7.983
7.967
7.964
7.784
7.779
7.761
7.756
7.735
7.729
7.725
7.720
7.697
7.671
7.675
SO2Cl
2e
CDCl3 1H NMR 300 MHz
0.9
4.0
2.0
9
8
7
6
5
4
3
2
1
0
130.352
130.224
129.889
128.937
128.347
128.134
121.291

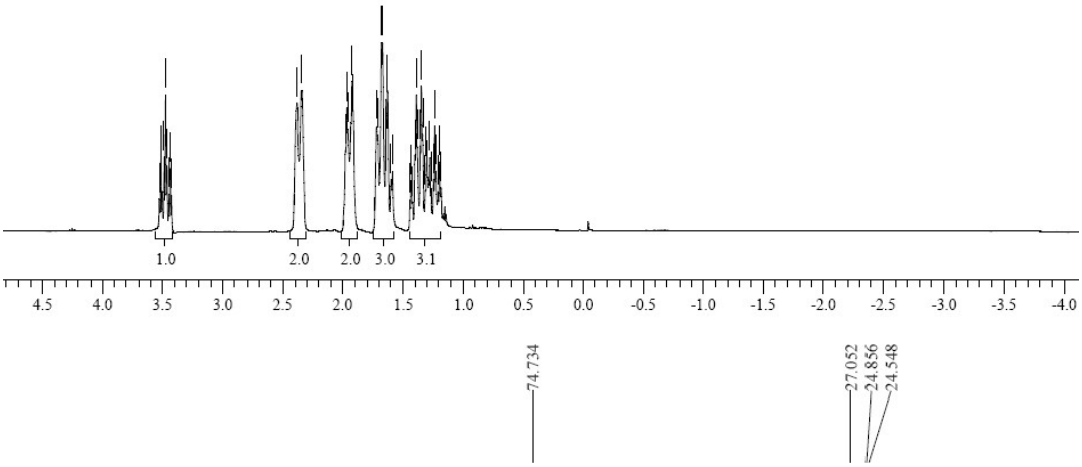

SO2Cl
2e
CDCl3 13C NMR 75 MHz
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

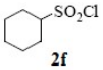




2f

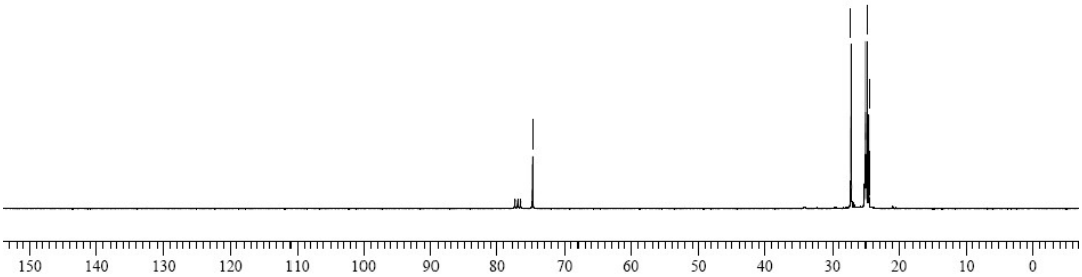
$CDCl_3$ ^{1}H NMR 300 MHz

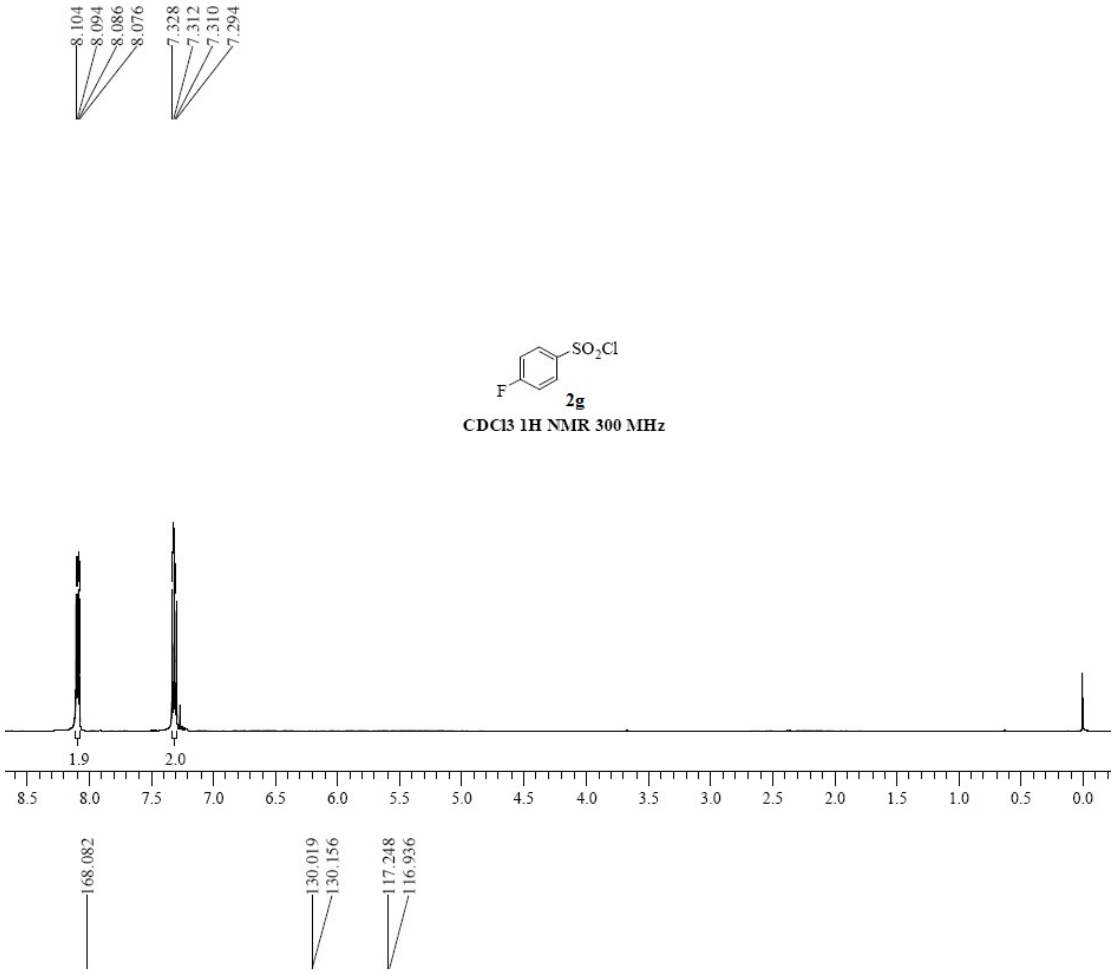


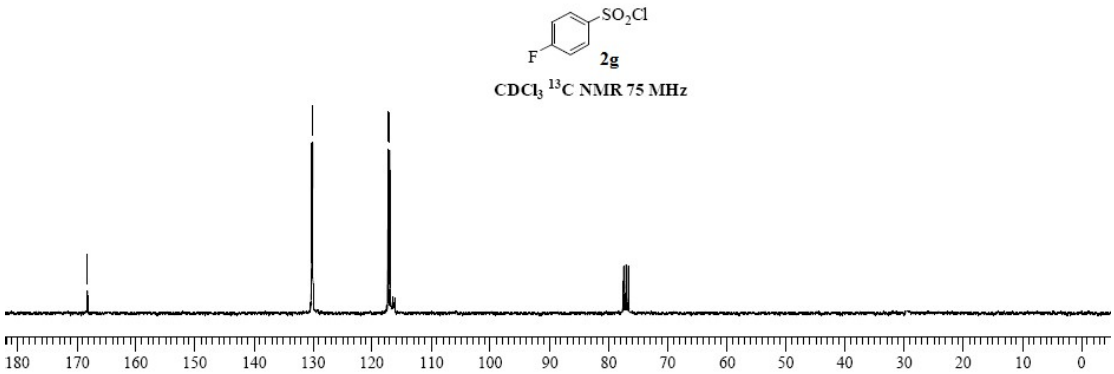


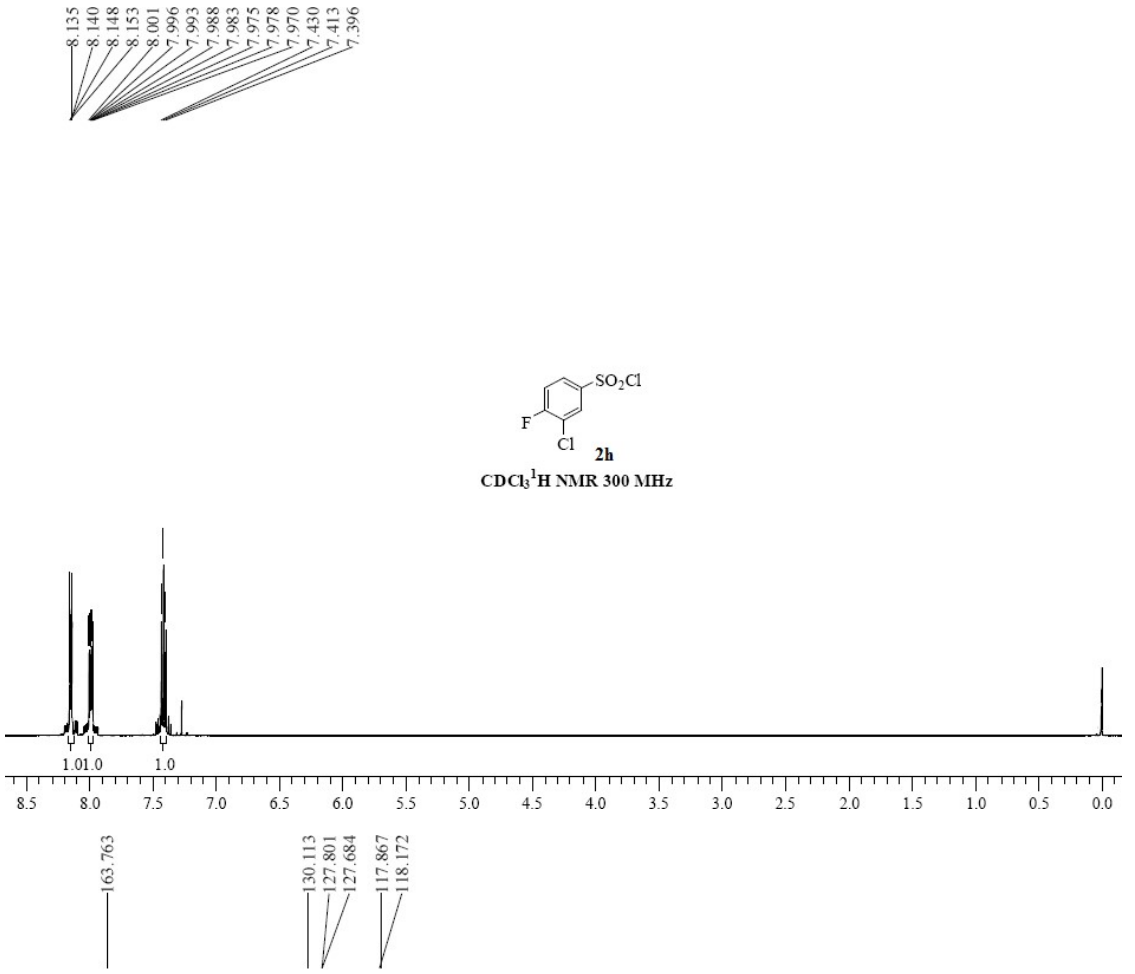
2f

$CDCl_3$ ^{13}C NMR 75 MHz




8.104
8.094
8.086
8.076
7.328
7.312
7.310
7.294
SO2Cl
F
2g
CDCl3 1H NMR 300 MHz
1.9
2.0
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
168.082
130.019
130.156
117.248
116.936


SO2Cl
F
2g
CDCl3 13C NMR 75 MHz
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


8.135
8.140
8.148
8.153
8.001
7.996
7.993
7.988
7.983
7.975
7.978
7.970
7.430
7.413
7.396
SO2Cl
F
Cl
2h
CDCl3 1H NMR 300 MHz
1.01.0
1.0
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
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0.0
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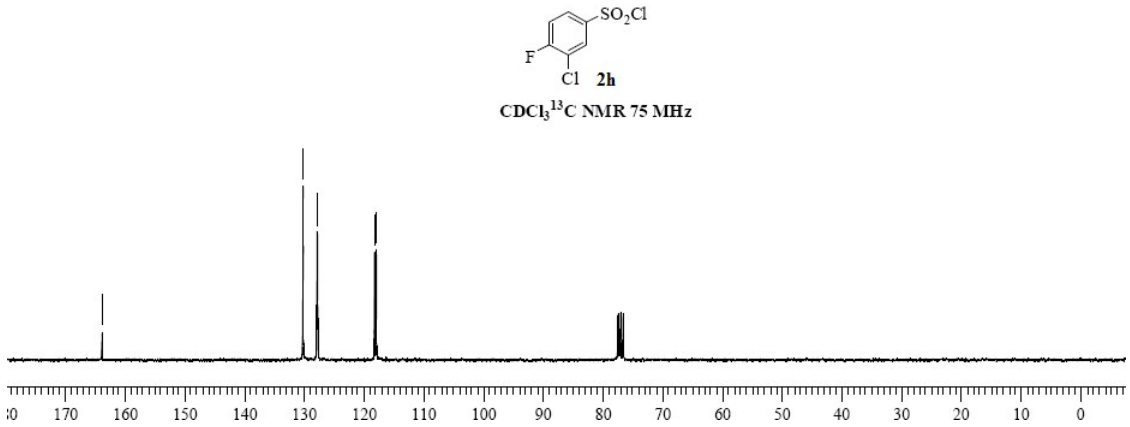
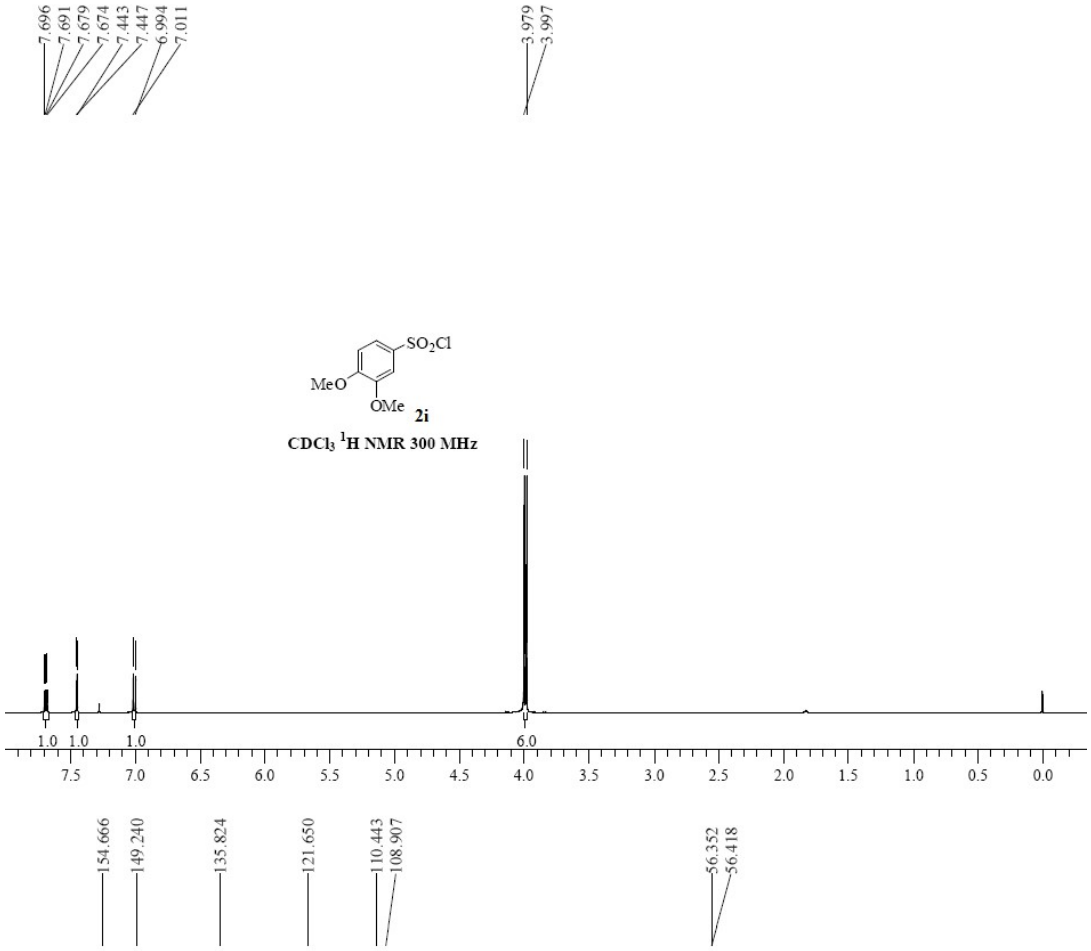
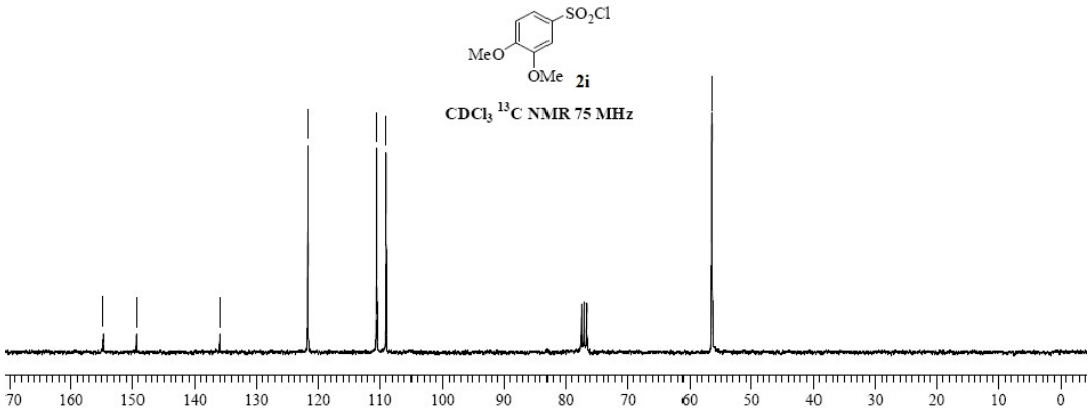
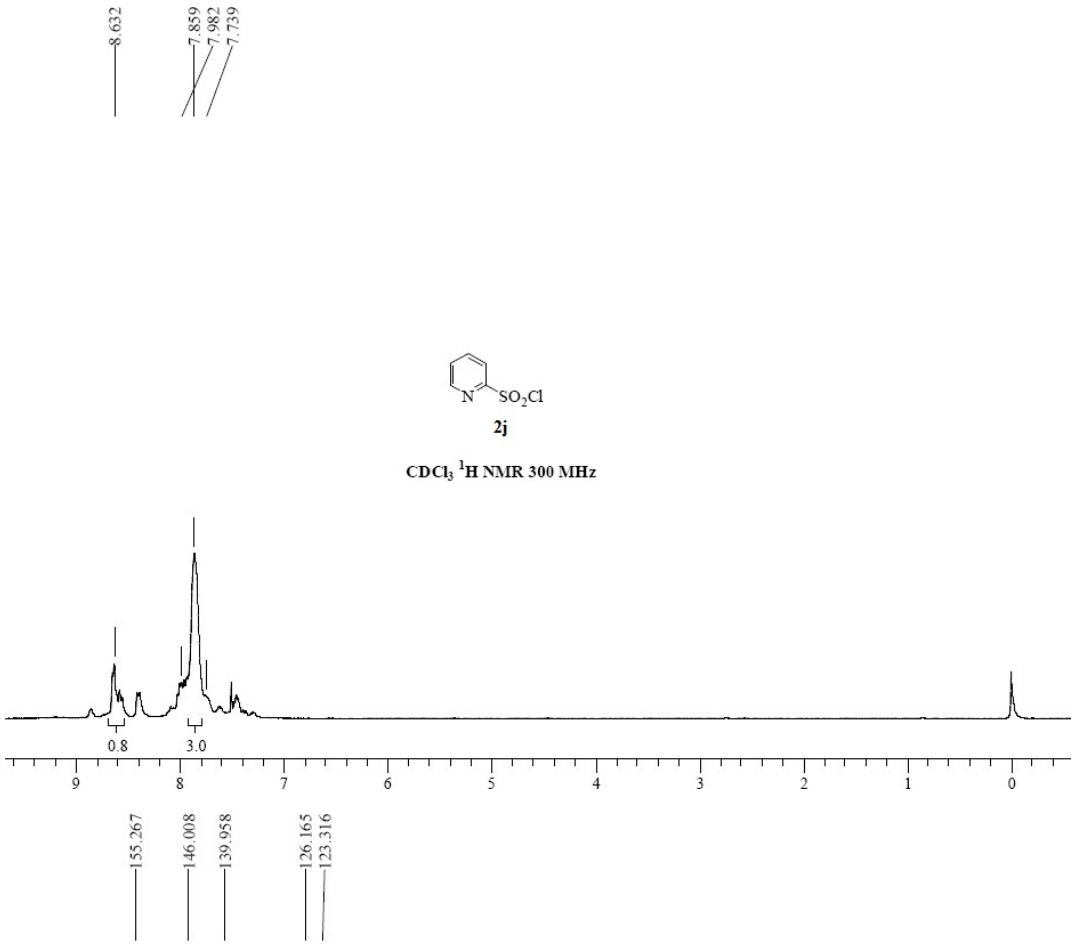
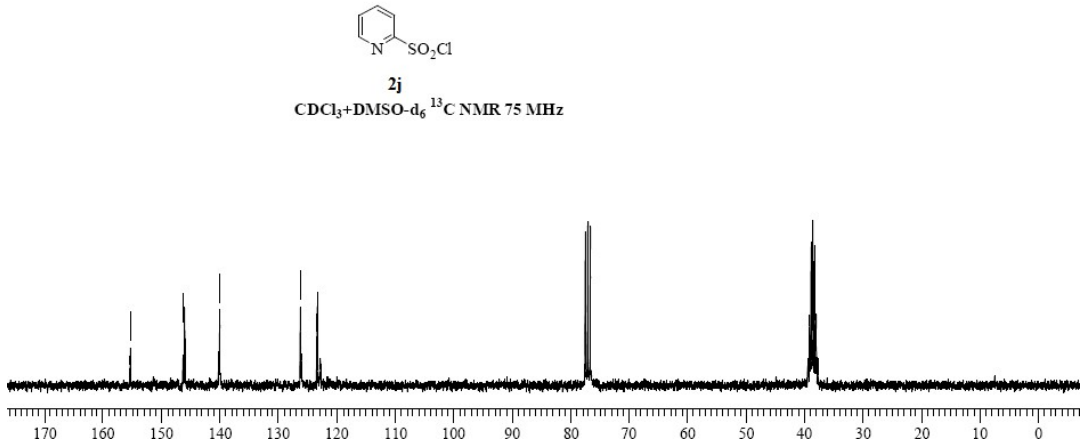
130.113
127.801
127.684
117.867
118.172

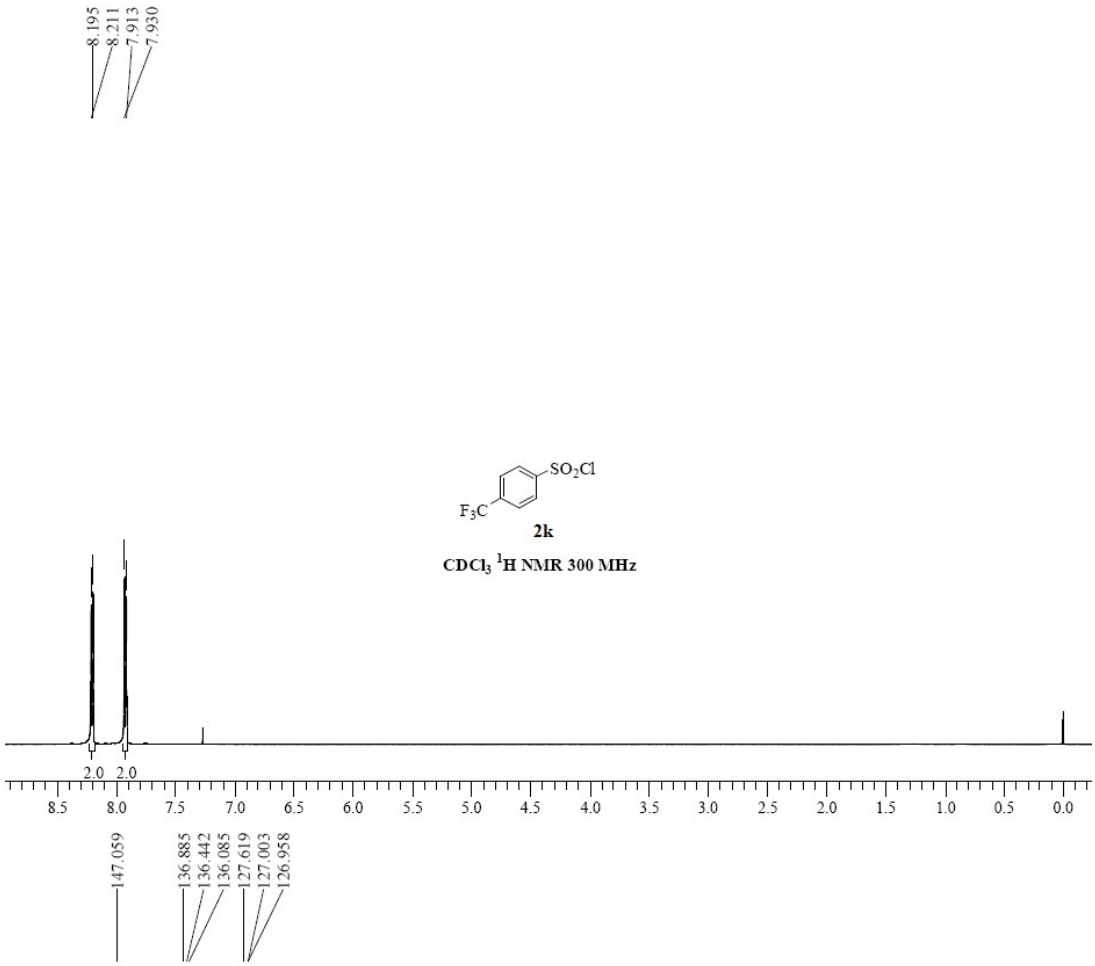

SO2Cl
F
Cl
2h
CDCl3 13C NMR 75 MHz
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

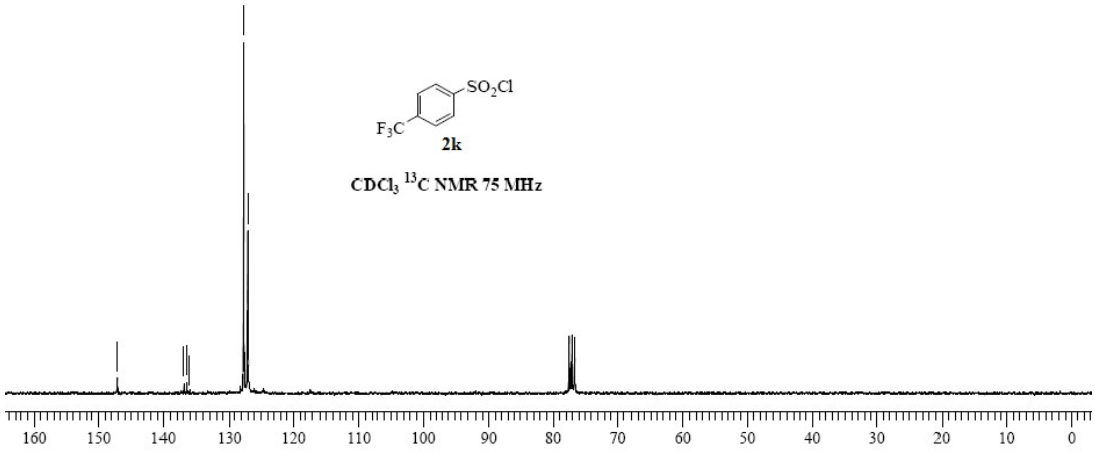

7.696
7.691
7.679
7.674
7.443
7.447
6.994
7.011
3.979
3.997
SO2Cl
MeO
OMe
2i
CDCl3 1H NMR 300 MHz
1.0 1.0 1.0
6.0
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
154.666
149.240
135.824
121.650
110.443
108.907
56.352
56.418


SO2Cl
MeO
OMe
2i
CDCl3 13C NMR 75 MHz
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


8.632
7.859
7.982
7.739
N
SO2Cl
2j
CDCl3 1H NMR 300 MHz
0.8
3.0
9
8
7
6
5
4
3
2
1
0
155.267
146.008
139.958
126.165
123.316


N
SO2Cl
2j
CDCl3+DMSO-d6 13C NMR 75 MHz
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


8.195
8.211
7.913
7.930
SO2Cl
F3C
2k
CDCl3 1H NMR 300 MHz
2.0 2.0
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
147.059
136.885
136.442
136.085
127.619
127.003
126.958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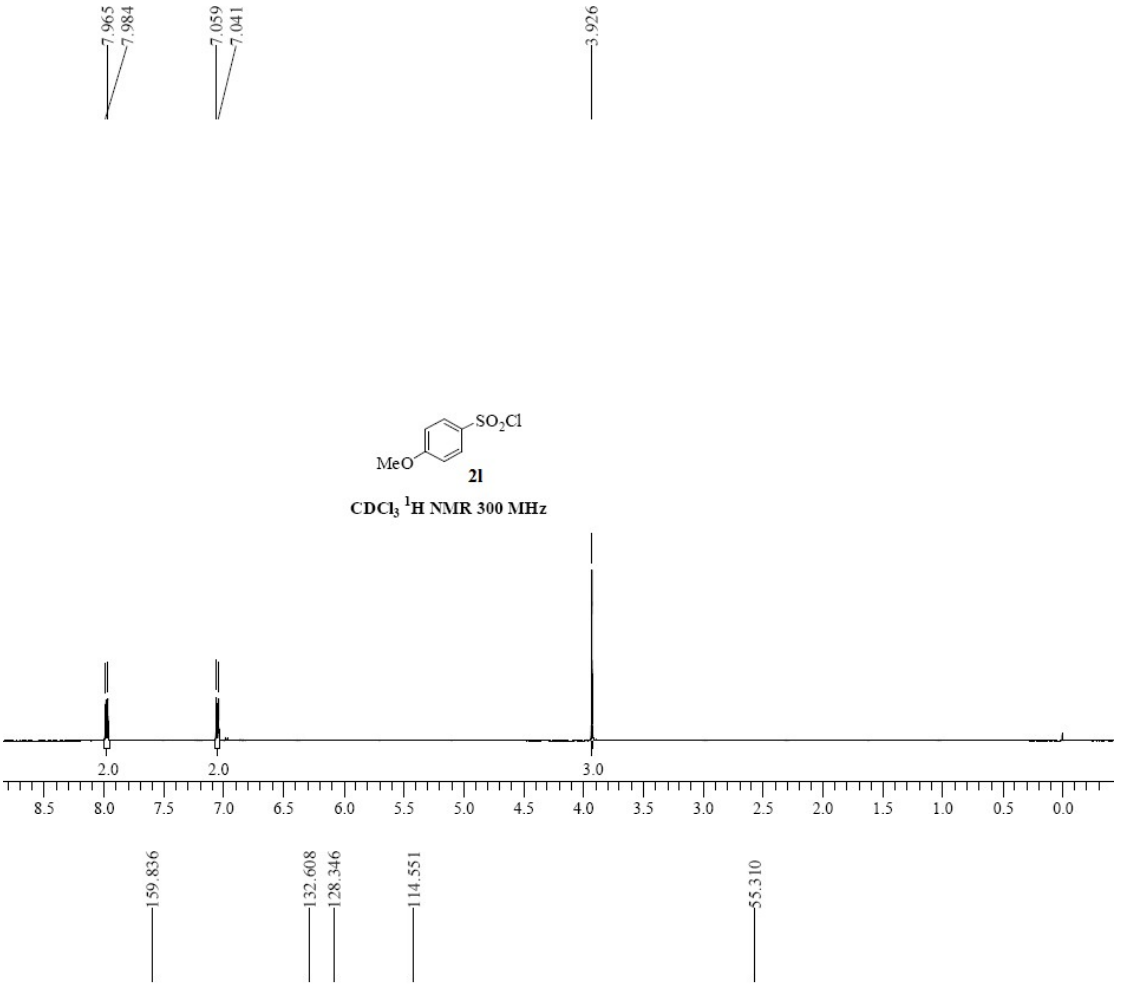
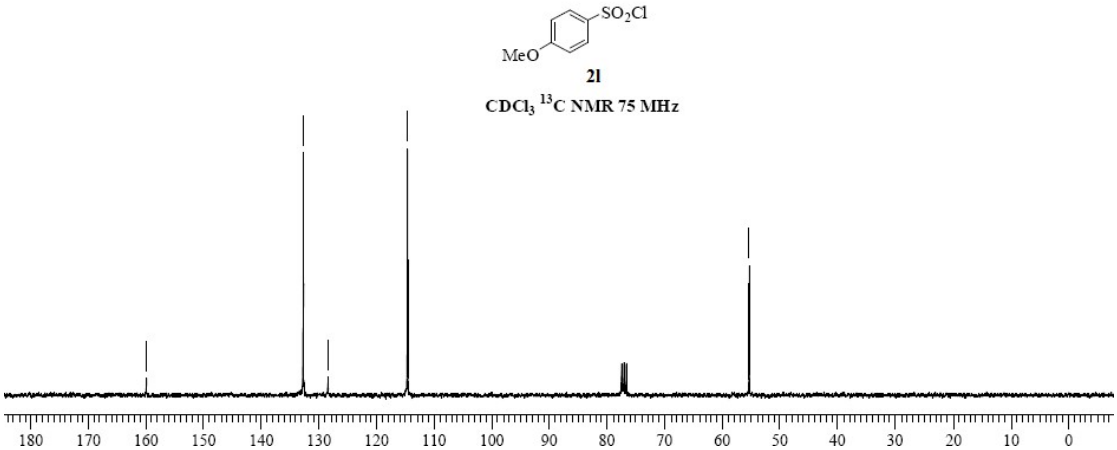
F3C
2k
CDCl3 13C NMR 75 MHz
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


7.965
7.984
7.059
7.041
3.926
SO2Cl
MeO
2l
CDCl3 1H NMR 300 MHz
2.0
2.0
3.0
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
159.836
132.608
128.346
114.551
55.310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MeO
2l
CDCl3 13C NMR 75 MHz
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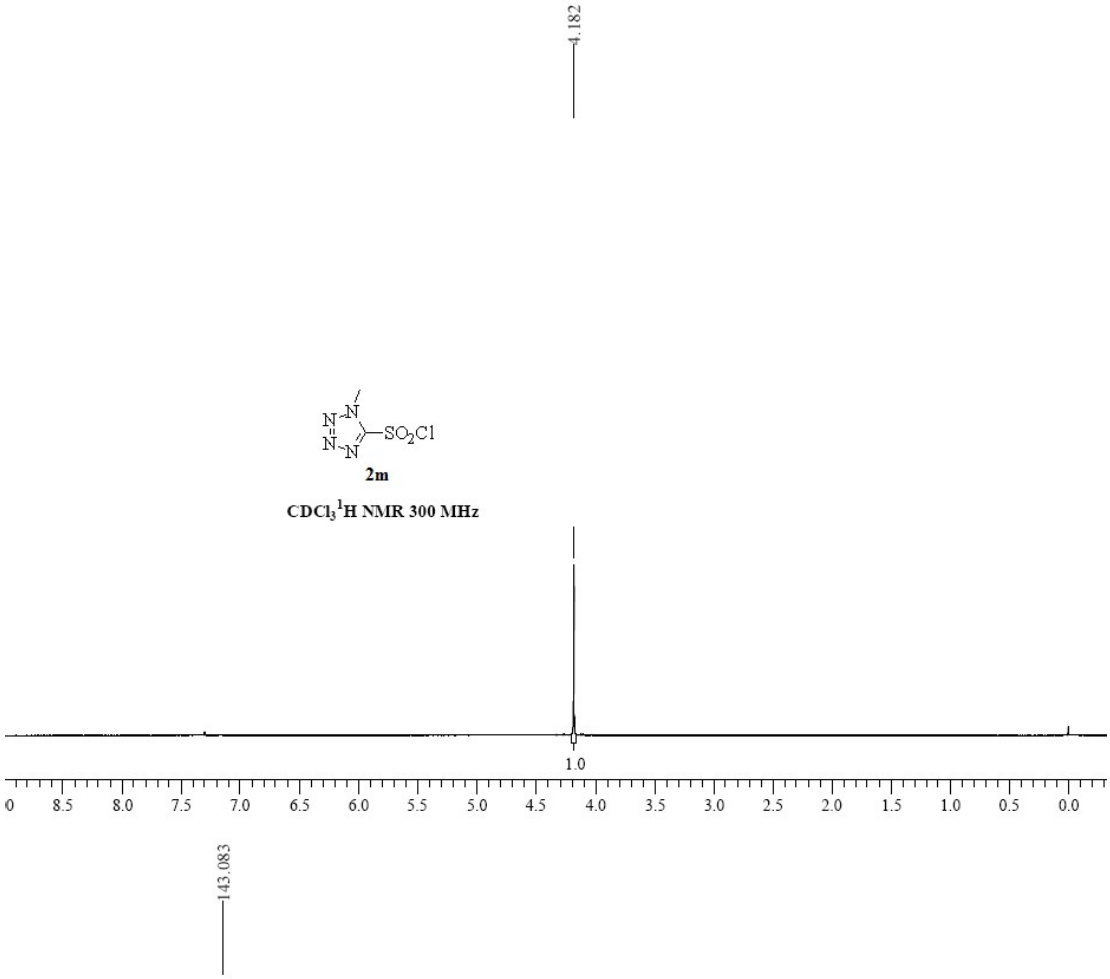
4.182
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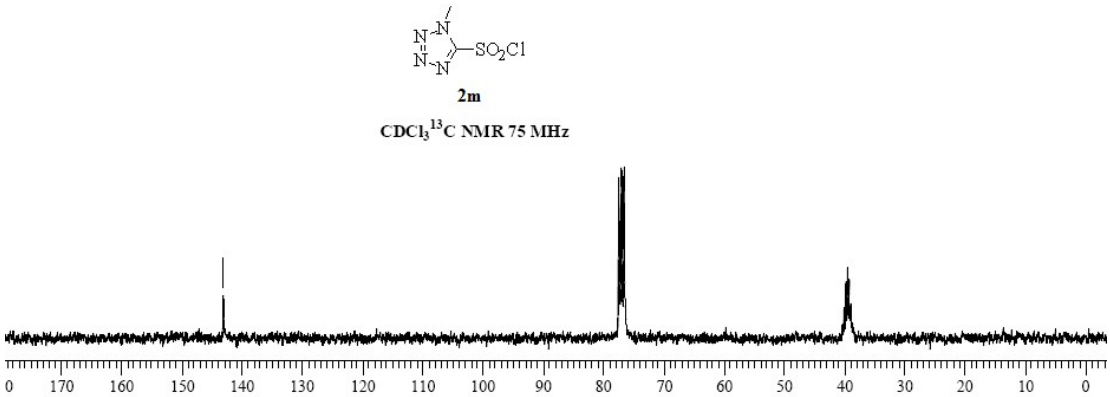
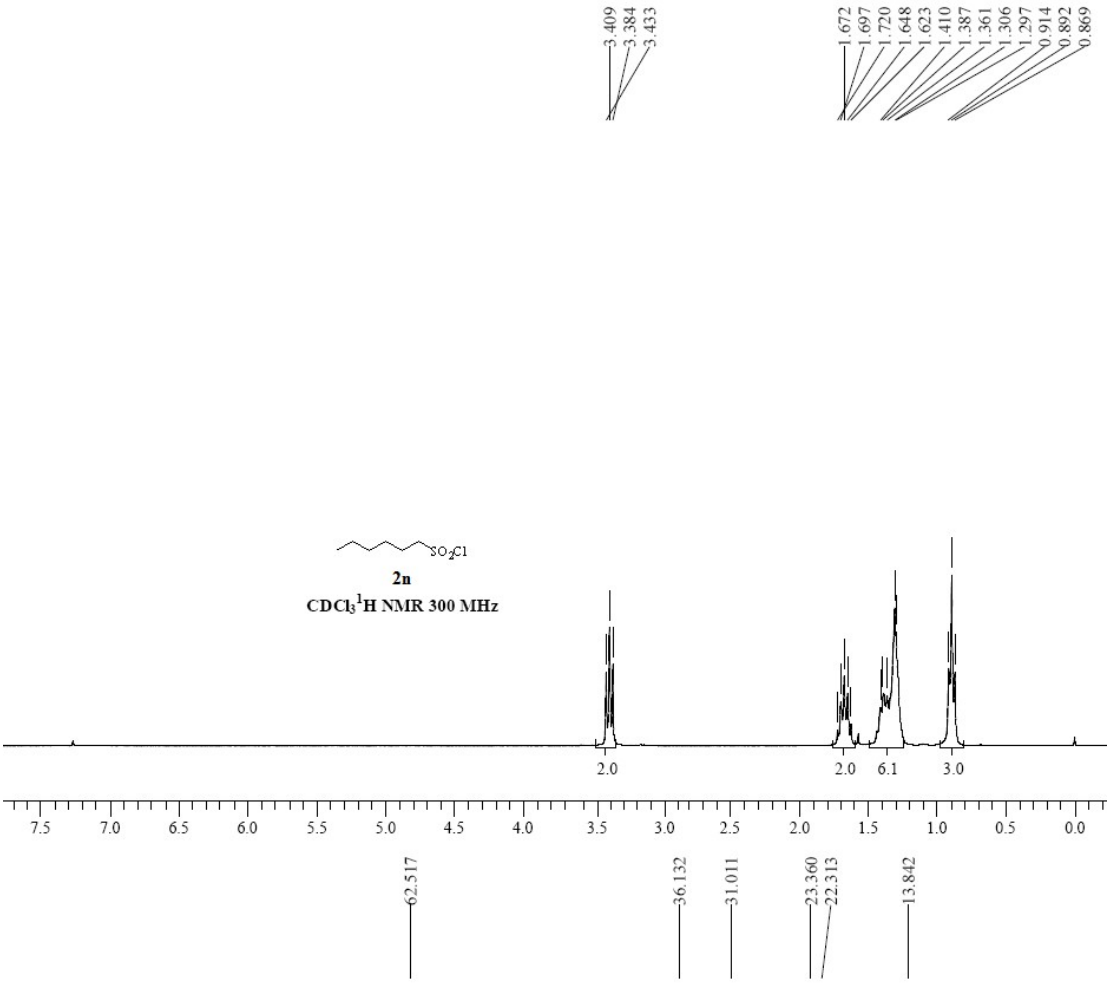
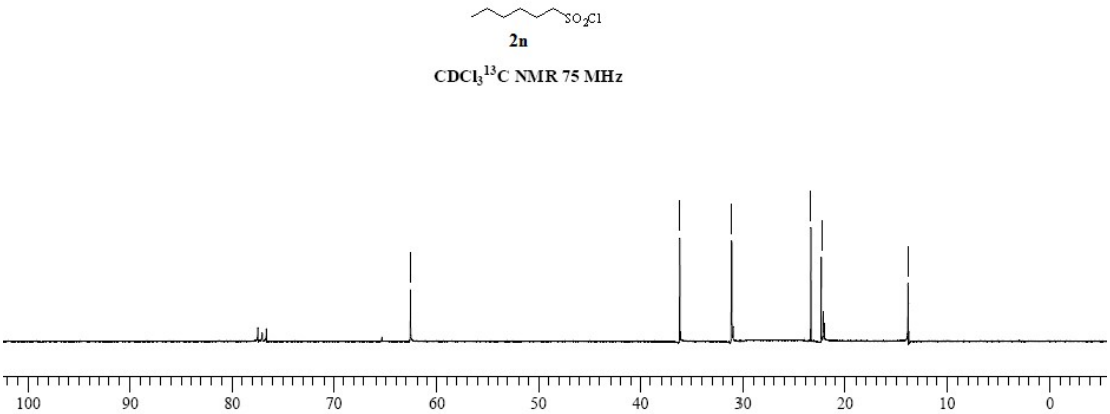
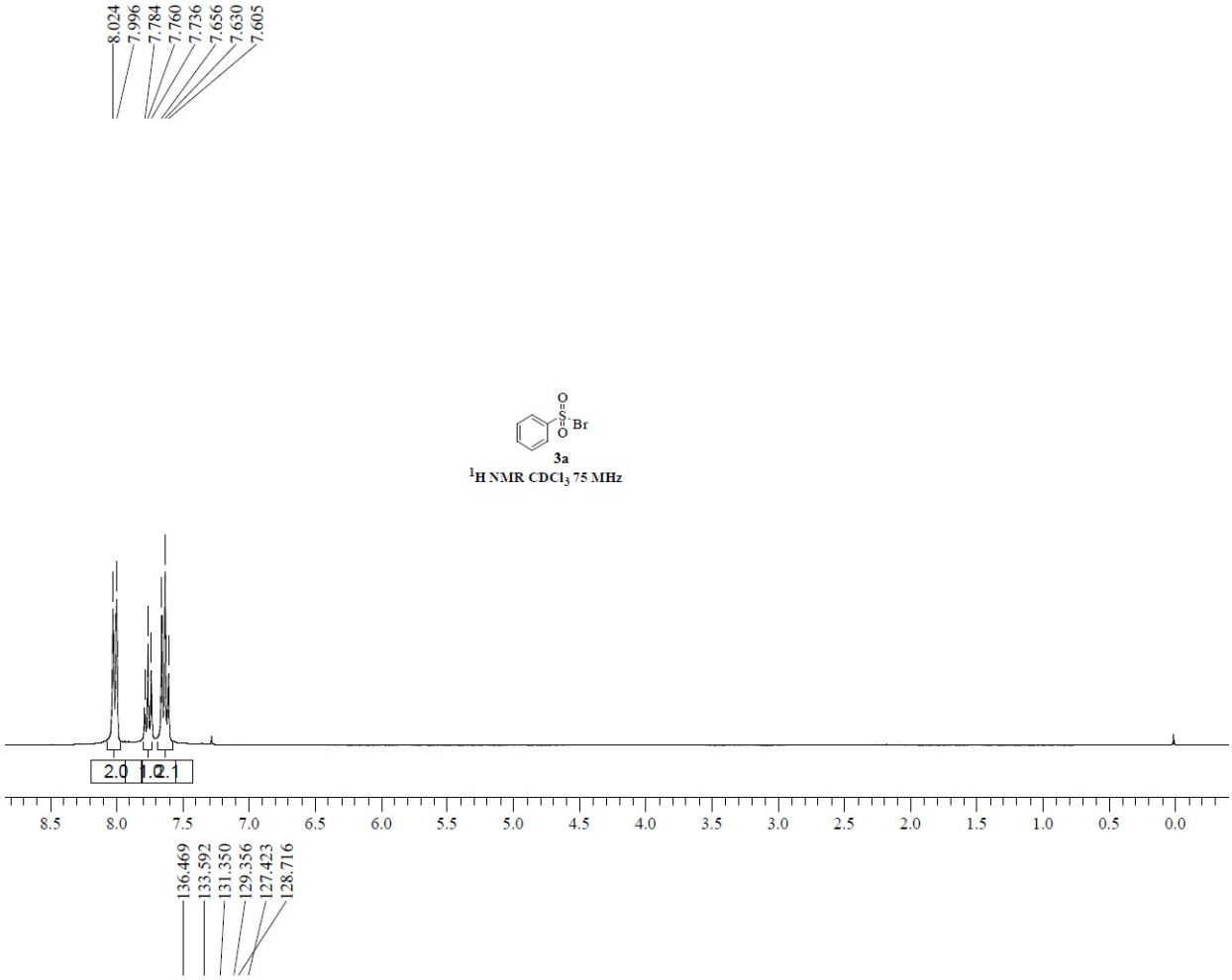
2m
CDCl3 1H NMR 300 MHz
1.0
143.083


SO2Cl
2m
CDCl3 13C NMR 75 MHz


3.409
3.384
3.433
1.672
1.697
1.720
1.648
1.623
1.410
1.387
1.361
1.306
1.297
0.914
0.892
0.869
SO2Cl
2n
CDCl3 1H NMR 300 MHz
2.0
2.0
6.1
3.0
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
62.517
36.132
31.011
23.360
22.313
13.842


SO2Cl
2n
CDCl3 13C NMR 75 MHz
100
90
80
70
60
50
40
30
20
10
0


8.024
7.996
7.784
7.760
7.736
7.656
7.630
7.605
3a
1H NMR CDCl3 75 MHz
2.0
1.0
2.1
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
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133.592
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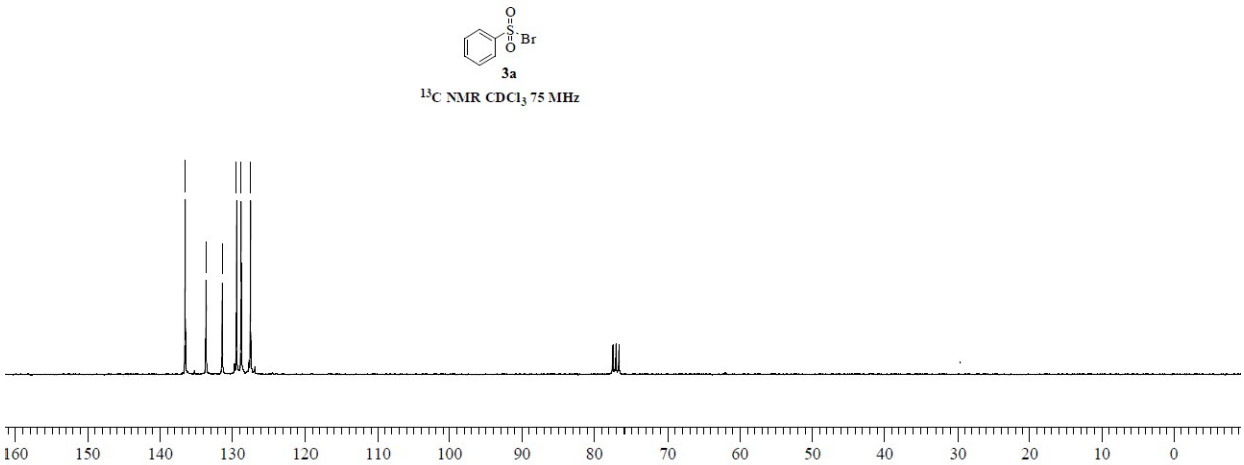
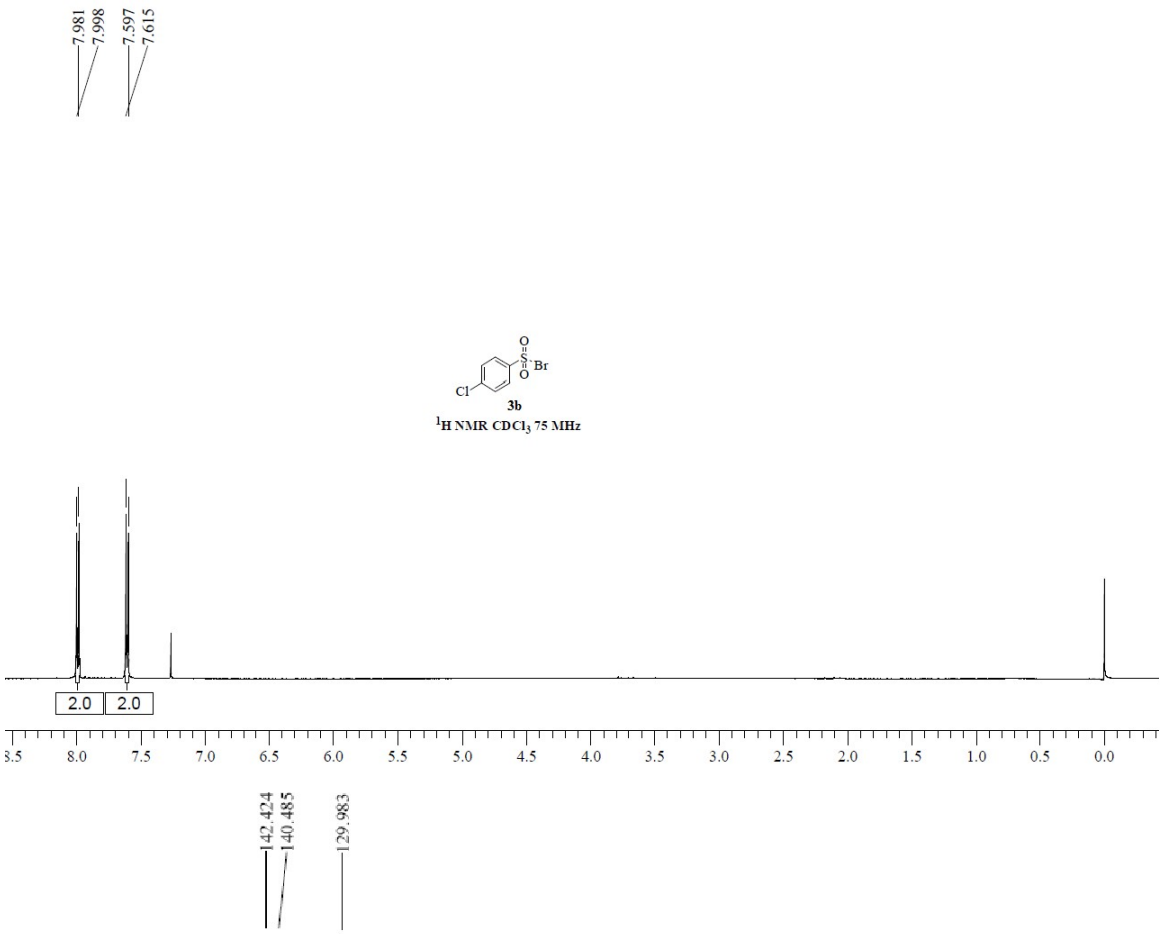
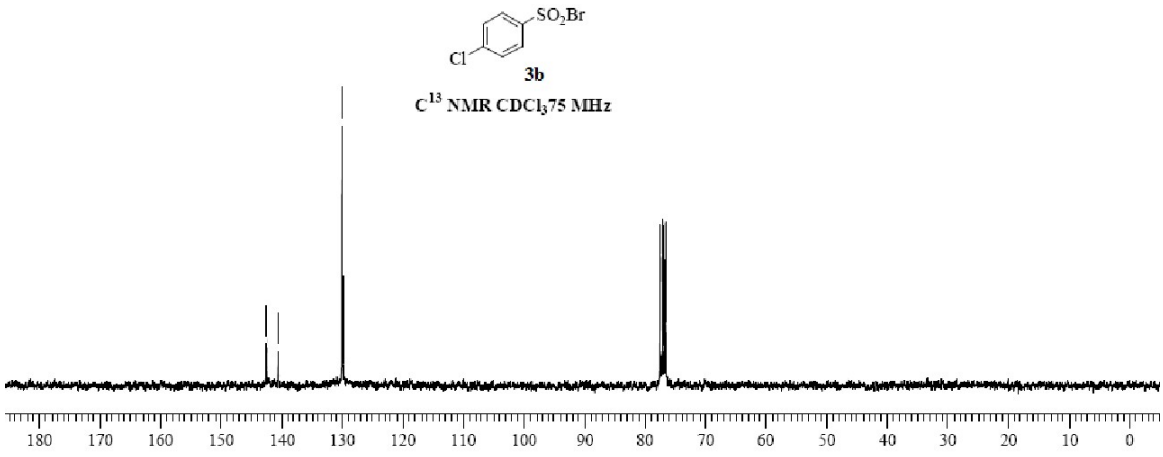
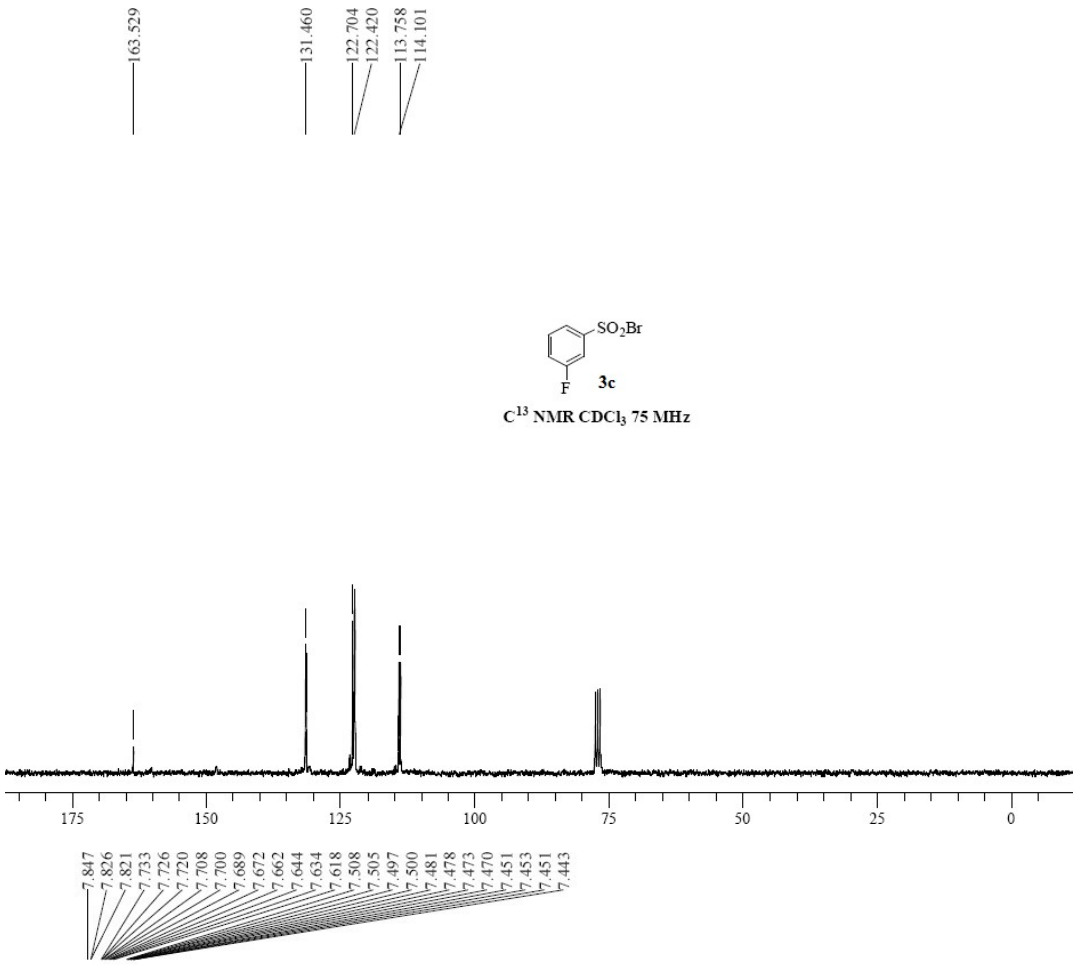
127.423
128.716


3a
13C NMR CDCl3 75 MHz
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


7.981
7.998
7.597
7.615
Cl
Br
3b
^{1}H NMR CDCl$_3$ 75 MHz
2.0
2.0
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
142.424
140.485
129.983

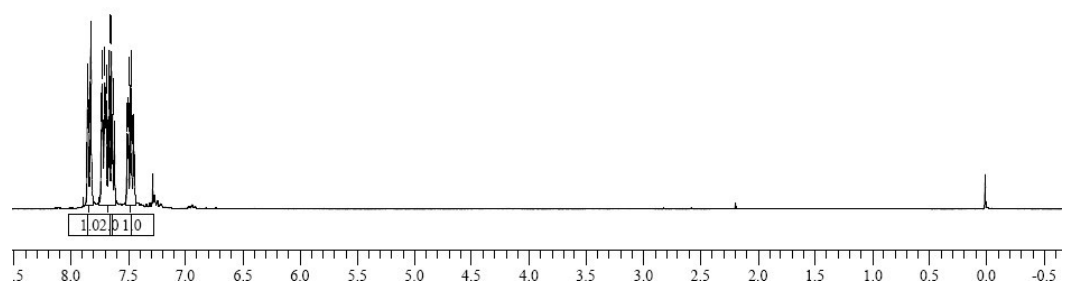

SO$_2$Br
Cl
3b
C^{13} NMR CDCl$_3$75 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0



SO_2Br

F **3c**

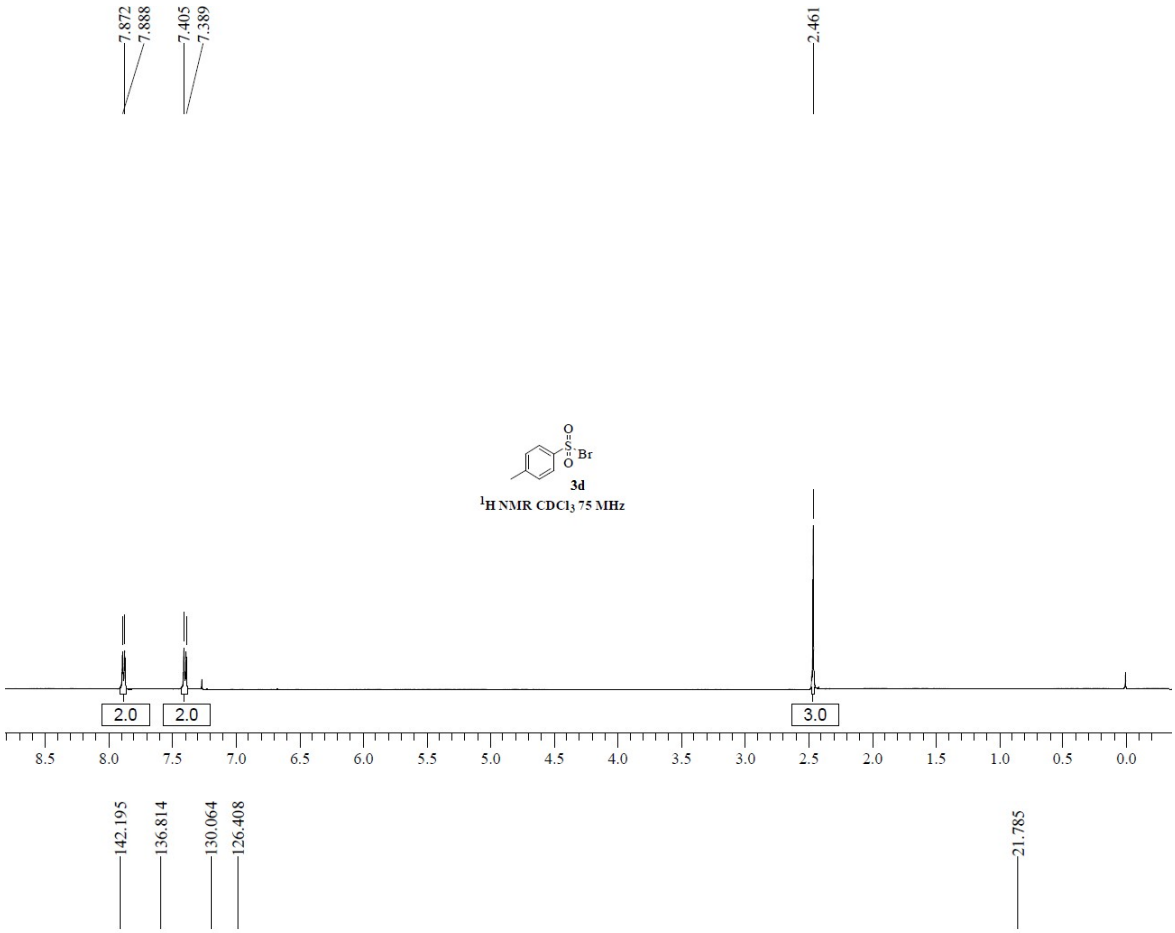
C^{13} NMR $CDCl_3$ 75 MHz

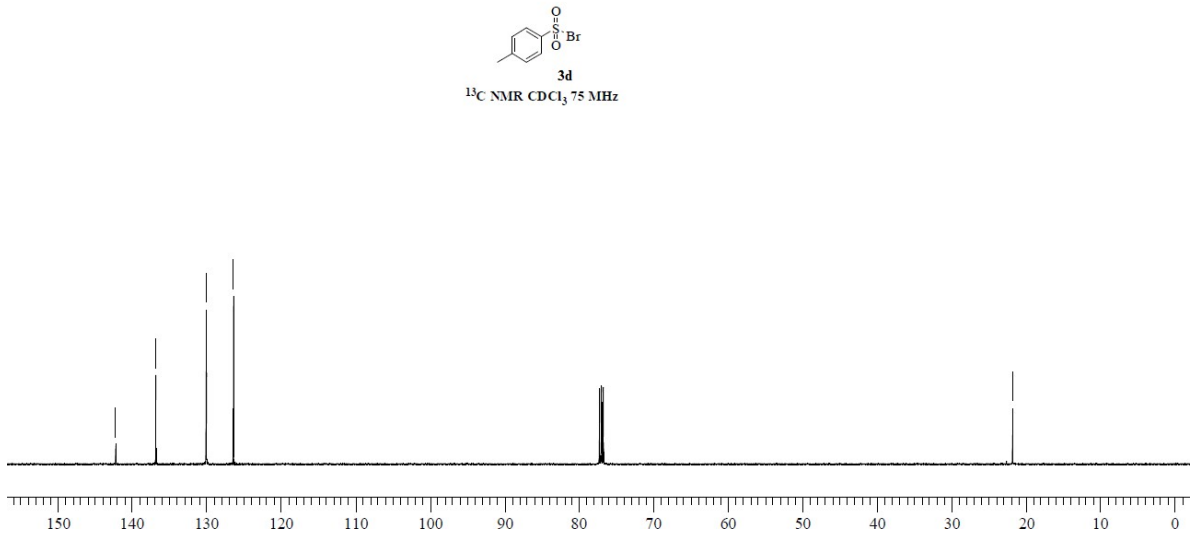


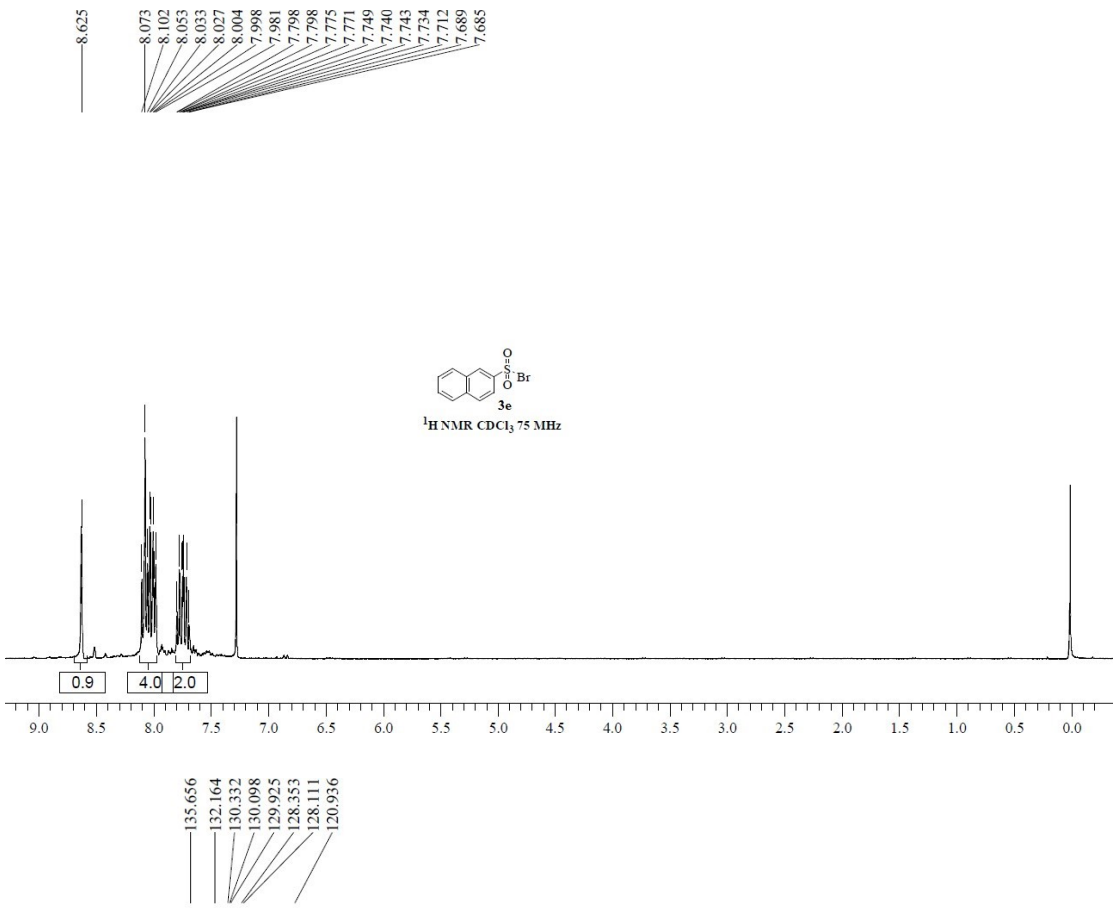
F SO_2Br

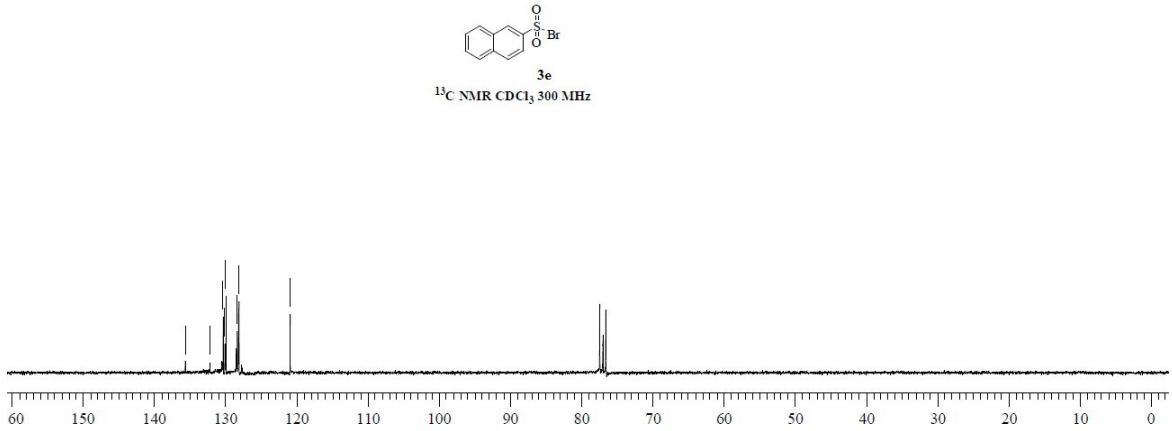
3c

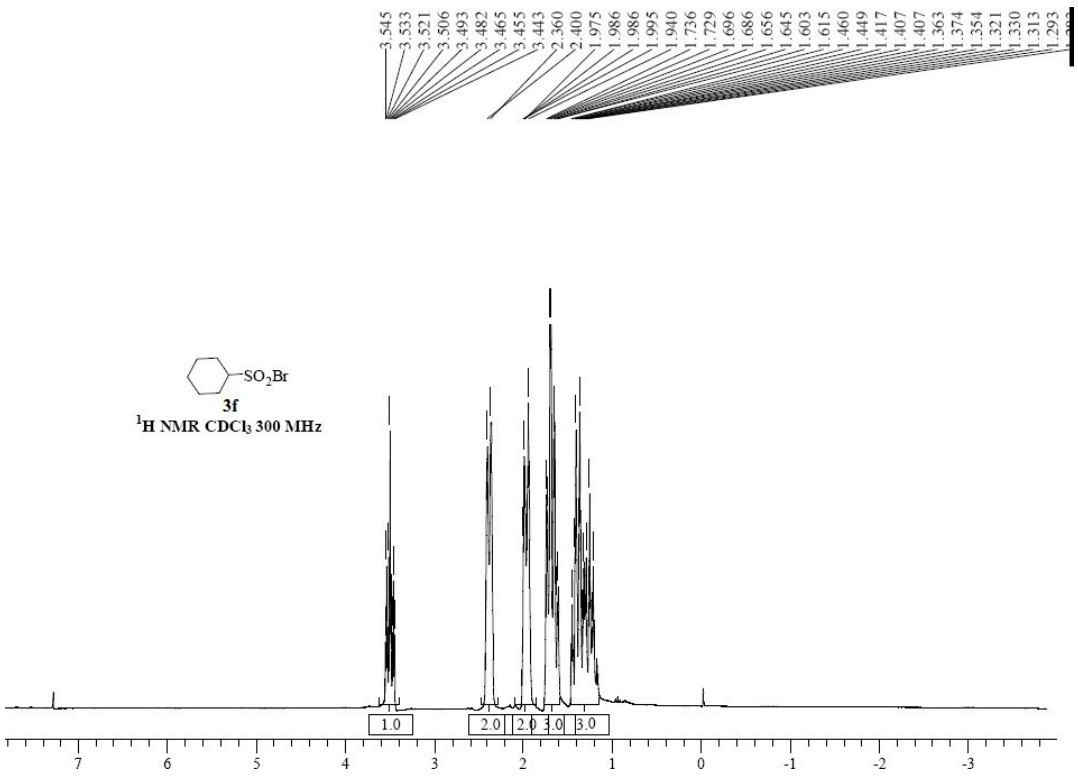
^1H NMR $CDCl_3$ 300 MHz

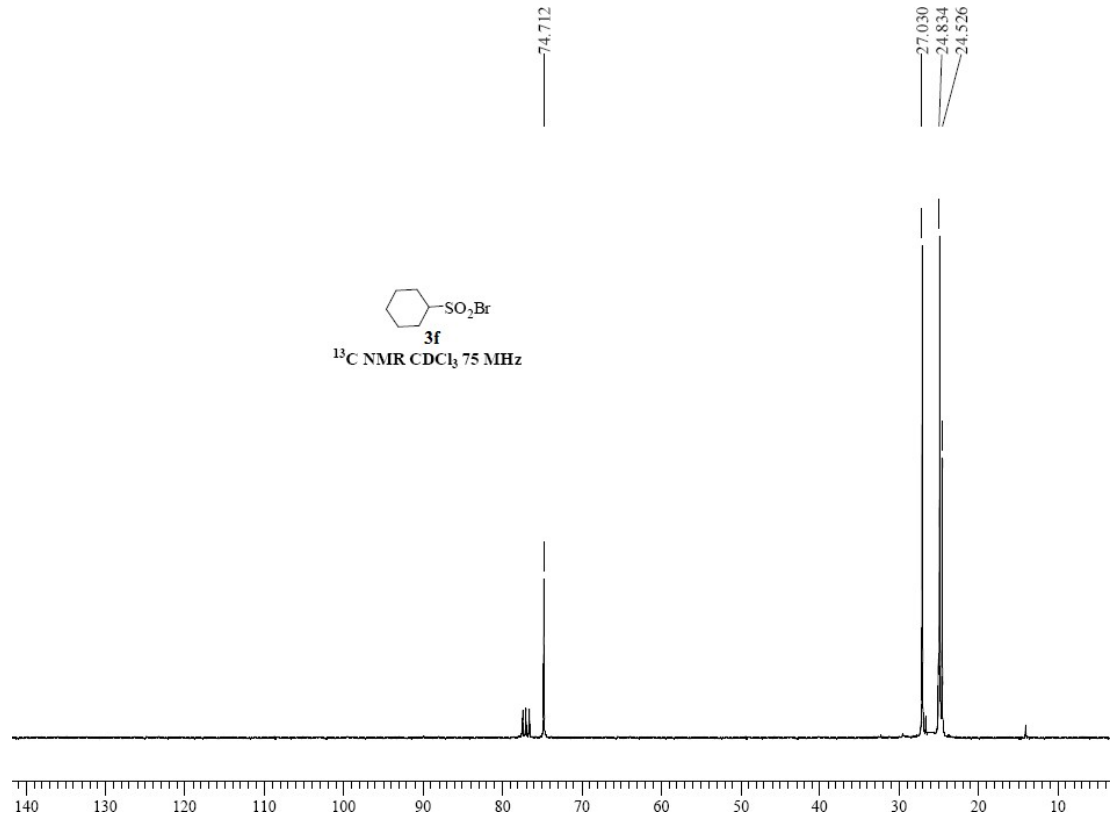

7.872
7.888
7.405
7.389
2.461
3d
^{1}H NMR $CDCl_3$ 75 MHz
2.0
2.0
3.0
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
142.195
136.814
130.064
126.408
21.785


3d
^{13}C NMR $CDCl_3$ 75 MHz
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


8.625
8.073
8.102
8.053
8.033
8.027
8.004
7.998
7.981
7.798
7.798
7.775
7.771
7.749
7.740
7.743
7.734
7.712
7.689
7.685
3e
^{1}H NMR $CDCl_3$ 75 MHz
0.9
4.0
2.0
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
135.656
132.164
130.332
130.098
129.925
128.353
128.111
120.936


3e
^{13}C NMR $CDCl_3$ 300 MHz
60 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


3.545
3.533
3.521
3.506
3.493
3.482
3.465
3.455
3.443
2.360
2.400
1.975
1.986
1.986
1.995
1.940
1.736
1.729
1.696
1.686
1.656
1.645
1.603
1.615
1.460
1.449
1.417
1.407
1.407
1.363
1.374
1.354
1.321
1.330
1.313
1.293
SO2Br
3f
1H NMR CDCl3 300 MHz
1.0
2.0
2.0
3.0
3.0
7
6
5
4
3
2
1
0
-1
-2
-3


74.712
27.030
24.834
24.526
SO2Br
3f
13C NMR CDCl3 75 MHz
140
130
120
110
100
90
80
70
60
50
40
30
20
10