

Supporting Information  
For

**Visible light catalyzed methylsulfoxidation of (het)aryl diazonium salts with DMSO**

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## 1. General Information

All reactions were monitored by TLC, visualization was effected with UV and/or by developing in iodine. Melting points were recorded on a Precision melting point apparatus and are uncorrected. IR spectra were recorded on a Perkin Elmer's RX I FTIR spectrophotometer. NMR spectra were recorded on a Bruker Avance spectrometer at 400 MHz ( $^1\text{H}$ ) and 100 MHz ( $^{13}\text{C}$ ). Chemical shifts are reported in  $\delta$  (ppm) relative to TMS as the internal standard. To describe spin multiplicity, standard abbreviations such as s, d, t, q, m, dd referring to singlet, doublet, triplet, quartet, multiplet and doublet of doublet respectively, are used. The ESI-HRMS spectra were recorded on Agilent 6520- Q-TofLC/MS system.

The diazonium salts were synthesized from corresponding anilines following **Method A**<sup>1a</sup> or **Method B**<sup>1b</sup> as described below. DMSO was freshly distilled over  $\text{CaH}_2$  before the reaction. All other chemicals and catalysts were purchased from commercial sources and used as received.

## 2. General Procedures

### General procedure for the synthesis of (het)aryl diazonium tetrafluoroborates

#### Method A (for 1a, 1g-i, 1k, 1m-n, 1r-u, 1w-1za)

To a stirred solution of aniline (10.0 mmol) in absolute ethanol (3.0 mL) and aqueous  $\text{HBF}_4$  (50%, 2.5 mL), *tert*-butyl nitrite (2.7 mL) was added dropwise at 0 °C. The reaction was stirred at room temperature for 1 h followed by addition of diethyl ether (20 mL). The resulting precipitate of corresponding aryldiazonium tetrafluoroborate was filtered off, washed with diethyl ether ( $3 \times 10$  mL), dried in vacuo and used without further purification.

#### Method B (for 1b-f, 1j, 1l, 1o-q, 1v, 1zb)

To a stirred solution of aniline (10.0 mmol) in water (2.0 mL) and aqueous  $\text{HBF}_4$  (50%, 2.5 mL) was added an aqueous solution of  $\text{NaNO}_2$  (1.5 gm in 2 mL  $\text{H}_2\text{O}$ ) dropwise at 0 °C. The reaction was stirred at 0 °C for 45 minutes. The resulting precipitate was filtered off and washed successively with ice water ( $3 \times 10$  mL) and diethyl ether ( $3 \times 10$  mL). The solid was recrystallized with acetone and cold diethyl ether, dried in vacuo and used without further purification.

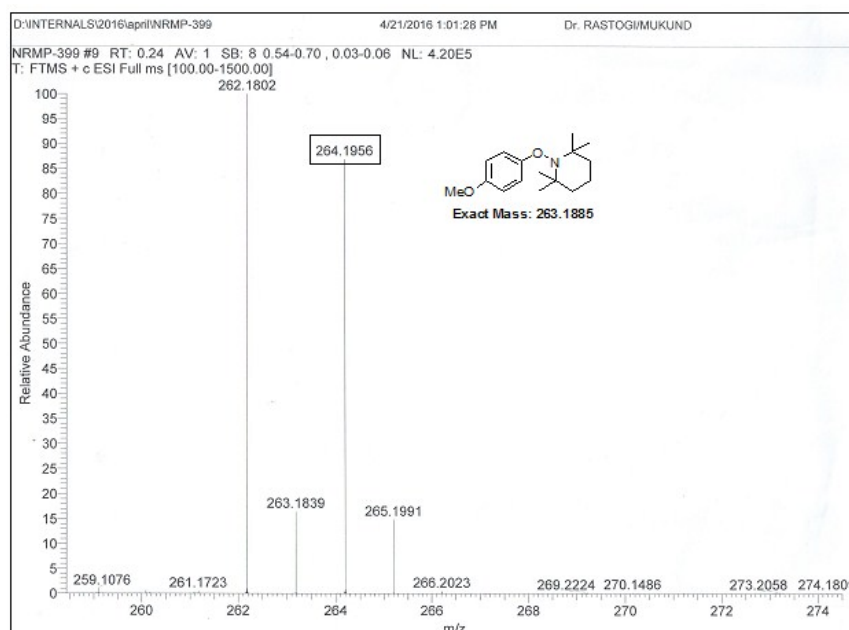
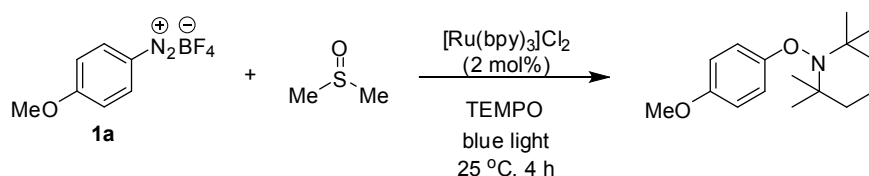
### General procedure for methylsulfoxidation with DMSO

In a 5 mL snap vial equipped with magnetic stirring bar, the (het)aryldiazonium tetrafluoroborate **1** (0.5 mmol) and photocatalyst  $[\text{Ru}(\text{bpy})_3]\text{Cl}_2$  (0.01 mmol, 2 mol%)

were dissolved in anhydrous DMSO (1.0 mL). The resulting reaction mixture was degassed by three “pump-freeze-thaw” cycles via a syringe needle. The vial was irradiated using 450 nm blue LEDs with a cooling device maintaining a temperature around 25 °C. After 2-4h of irradiation (TLC monitoring) the reaction mixture was diluted with water (10 mL) and extracted with ethyl acetate (3 x 20 mL). The combined organic layer was dried (Na<sub>2</sub>SO<sub>4</sub>) and concentrated under reduced pressure. Purification of the crude product was achieved by column chromatography on silica gel using hexane/ethyl acetate as eluent to afford the pure product **3**.

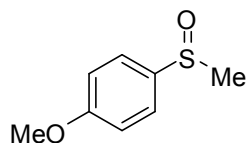
### Details of TEMPO trapping experiment

In a 5 mL snap vial equipped with magnetic stirring bar, the 4-methoxybenzenediazonium tetrafluoroborate **1a** (0.5 mmol), [Ru(bpy)<sub>3</sub>]Cl<sub>2</sub> (0.01 mmol, 2 mol%) and TEMPO (0.75 mmol) were dissolved in anhydrous DMSO (1.0 mL). The resulting reaction mixture was degassed by three “pump-freeze-thaw” cycles via a syringe needle. The vial was irradiated using 450 nm blue LEDs with a cooling device maintaining a temperature around 25 °C. After 4h of irradiation the reaction mixture was diluted with water (10 mL) and extracted with ethyl acetate (3 x 20 mL). The combined organic layer was dried (Na<sub>2</sub>SO<sub>4</sub>) and concentrated under reduced pressure. The crude product was analyzed by High Resolution Mass Spectrometry.



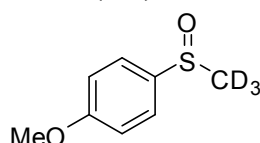
### 3. Product Characterization

#### 1-Methoxy-4-(methylsulfinyl)benzene (3a)<sup>2</sup>



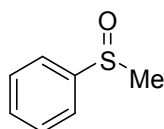
Colorless solid; isolated yield 68% (58 mg).  $R_f$  0.50 (60% EtOAc/hexane); Mp 42-44 °C (lit.<sup>2</sup> 42-43.6 °C); **IR** (Film,  $\text{cm}^{-1}$ ): 1068, 1218, 1259, 1645; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.50 – 7.54 (m, 2H), 6.94 – 6.97 (m, 2H), 3.78 (s, 3H), 2.63 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  162.0, 136.5, 125.5, 114.9, 55.5, 43.9; **HRMS** for  $\text{C}_8\text{H}_{10}\text{O}_2\text{S}$ : calcd. ( $\text{MH}^+$ ): 171.0474, found: 171.0473

#### 1-Methoxy-4-(methylsulfinyl)benzene-*d*<sub>3</sub> (3a')



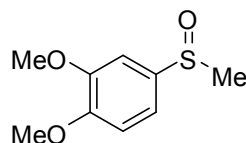
Colorless oil; isolated yield 68% (59 mg).  $R_f$  0.50 (60% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1068, 1219, 1385, 1638; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52 (d,  $J = 8.8$  Hz, 2H), 6.96 (d,  $J = 8.8$  Hz, 2H), 3.78 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  162.0, 136.5, 125.5, 114.9, 55.5; **HRMS** for  $\text{C}_8\text{H}_7\text{D}_3\text{O}_2\text{S}$ : calcd. ( $\text{MH}^+$ ): 174.0663, found: 174.0657

#### Methylsulfinylbenzene (3b)<sup>3</sup>



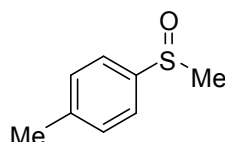
Yellow oil; isolated yield 40% (28 mg).  $R_f$  0.50 (50% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1047, 1644, 3019; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.56 – 7.59 (m, 2H), 7.42 – 7.48 (m, 3H), 2.65 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  145.6, 131.0, 129.3, 123.4, 43.9; **HRMS** for  $\text{C}_7\text{H}_8\text{OS}$ : calcd. ( $\text{MH}^+$ ): 141.0369, found: 141.0367

#### 1,2-Dimethoxy-4-(methylsulfinyl)benzene (3c)



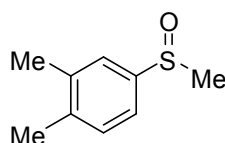
Brown oil; isolated yield 67% (67 mg).  $R_f$  0.50 (70% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1069, 1219, 1638; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.20 (s, 1H), 6.89 (dd,  $J = 2.0, 8.3$  Hz, 1H), 6.89 (d,  $J = 8.3$  Hz, 1H), 3.89 (s, 3H), 3.86 (s, 3H), 2.64 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  151.5, 150.2, 137.0, 116.9, 111.2, 105.9, 56.2, 56.2, 44.2; **HRMS** for  $\text{C}_9\text{H}_{12}\text{O}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 201.0580, found: 201.0578

### 1-Methyl-4-(methylsulfinyl)benzene (3d)<sup>2</sup>



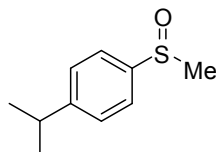
Brown gummy solid; isolated yield 35% (27 mg).  $R_f$  0.50 (60% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1039, 1646, 3019; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47 (d,  $J = 8.1$  Hz, 2H), 7.26 (d,  $J = 7.9$  Hz, 2H), 2.63 (s, 3H), 2.35 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  142.5, 141.5, 130.0, 123.6, 44.0, 21.4; **HRMS** for  $\text{C}_8\text{H}_{10}\text{OS}$ : calcd. ( $\text{MH}^+$ ): 155.0525, found: 155.0522

### 1,2-Dimethyl-4-(methylsulfinyl)benzene (3e)<sup>4</sup>



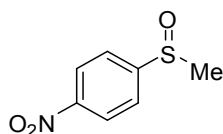
Brown gummy solid; isolated yield 32% (27 mg).  $R_f$  0.50 (60% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1026, 1156, 1637, 3019; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36 (s, 1H), 7.28 (d,  $J = 7.9$  Hz, 1H), 7.21 (s, 1H), 2.63 (s, 3H), 2.25 (s, 3H), 2.26 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  142.7, 140.2, 138.2, 130.5, 124.4, 121.1, 44.0, 19.8, 19.7; **HRMS** for  $\text{C}_9\text{H}_{12}\text{OS}$ : calcd. ( $\text{MH}^+$ ): 169.0682, found: 169.0684

### 1-Isopropyl-4-(methylsulfinyl)benzene (3f)<sup>3</sup>



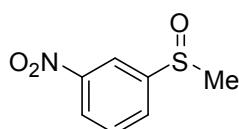
Brown oil; isolated yield 62% (57 mg).  $R_f$  0.50 (50% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1054, 1155, 1644, 3019; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.50 (d,  $J = 8.2$  Hz, 2H), 7.31 (d,  $J = 8.2$  Hz, 2H), 2.85 – 2.95 (m, 1H), 2.65 (s, 3H), 1.20 (d,  $J = 6.9$  Hz, 6H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  152.4, 142.8, 127.5, 123.7, 43.9, 34.1, 23.8; **HRMS** for  $\text{C}_{10}\text{H}_{14}\text{OS}$ : calcd. ( $\text{MH}^+$ ): 183.0838, found: 183.0836

### 1-(Methylsulfinyl)-4-nitrobenzene (3g)<sup>2</sup>



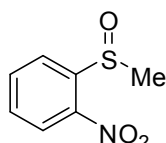
Yellow solid; isolated yield 80% (74 mg).  $R_f$  0.50 (60% EtOAc/hexane); Mp 148-150 °C (150.6-151.9 °C); **IR** (Film,  $\text{cm}^{-1}$ ): 1055, 1347, 1529, 1644, 3019; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.31 – 8.34 (m, 2H), 7.75 – 7.79 (m, 2H), 2.72 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  153.3, 129.1, 124.7, 124.5, 43.9; **HRMS** for  $\text{C}_7\text{H}_7\text{NO}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 186.0219, found: 186.0218

### 1-(Methylsulfinyl)-3-nitrobenzene (3h)<sup>2</sup>



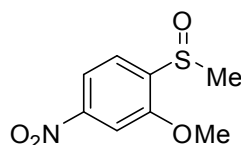
Yellow solid; isolated yield 69% (64 mg).  $R_f$  0.50 (60% EtOAc/hexane); Mp 115-116 °C (lit.<sup>2</sup> 114.3-115.7 °C); **IR** (Film,  $\text{cm}^{-1}$ ): 1068, 1352, 1534, 1644, 3019; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.43 (s, 1H), 8.30 (d,  $J = 8.0$  Hz, 1H), 7.94 (d,  $J = 7.6$  Hz, 1H), 7.70 (t,  $J = 8.0$  Hz, 1H), 2.74 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  148.8, 148.7, 130.6, 129.3, 125.7, 119.0, 44.0; **HRMS** for  $\text{C}_7\text{H}_7\text{NO}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 186.0219, found: 186.0215

### 1-(Methylsulfinyl)-2-nitrobenzene (3i)<sup>3</sup>



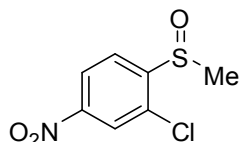
Yellow solid; isolated yield 78% (73 mg).  $R_f$  0.50 (60% EtOAc/hexane); Mp 56-58 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 1068, 1345, 1529, 3019; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.31 (dd,  $J = 1.3, 7.9$  Hz, 1H), 8.25 (dd,  $J = 1.2, 8.2$  Hz, 1H), 7.89 – 7.94 (m, 1H), 7.63 – 7.68 (m, 1H), 2.86 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  145.3, 144.6, 135.7, 131.5, 126.1, 125.1, 43.8; **HRMS** for  $\text{C}_7\text{H}_7\text{NO}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 186.0219, found: 186.0223

### 2-Methoxy-1-(methylsulfinyl)-4-nitrobenzene (3j)



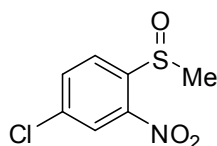
Brown solid; isolated yield 73% (79 mg).  $R_f$  0.50 (70% EtOAc/hexane); Mp 128-130 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 1037, 1347, 1531, 1719, 3021; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.93 – 8.01 (m, 2H), 7.70 (d,  $J = 1.8$  Hz, 1H), 3.95 (s, 3H), 2.76 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  155.1, 150.8, 141.1, 125.9, 116.7, 105.8, 56.6, 40.9; **HRMS** for  $\text{C}_8\text{H}_9\text{NO}_4\text{S}$ : calcd. ( $\text{MH}^+$ ): 216.0325, found: 216.0320

### 2-Chloro-1-(methylsulfinyl)-4-nitrobenzene (3k)



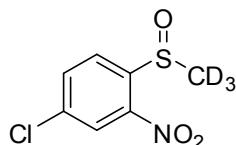
Brown solid; isolated yield 75% (82 mg).  $R_f$  0.5f0 (60% EtOAc/hexane); Mp 87-88 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 669, 1064, 1344, 1536, 1644, 3019; **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.31 (dd,  $J = 1.6, 8.6$  Hz, 1H), 8.21 (d,  $J = 1.9$  Hz, 1H), 8.11 (d,  $J = 8.6$  Hz, 1H), 2.82 (s, 3H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  151.3, 150.0, 130.8, 126.8, 125.0, 123.0, 41.4; **HRMS** for  $\text{C}_7\text{H}_6\text{ClNO}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 219.9830, found: 219.9829

#### 4-Chloro-1-(methylsulfinyl)-2-nitrobenzene (3l)



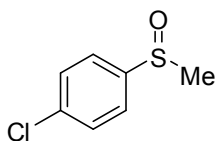
Brown solid; isolated yield 67% (74 mg).  $R_f$  0.50 (60% EtOAc/hexane); Mp 124-126 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 669, 1064, 1344, 1538, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.25 (d,  $J = 8.4$  Hz, 1H), 8.23 (d,  $J = 2.0$  Hz, 1H), 7.87 (dd,  $J = 2.1, 8.4$  Hz, 1H), 2.86 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  145.0, 143.8, 137.9, 135.7, 127.6, 125.1, 43.8; **HRMS** for  $\text{C}_7\text{H}_6\text{ClNO}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 219.9830, found: 219.9831

#### 4-Chloro-1-(methylsulfinyl)-2-nitrobenzene- $d_3$ (3l')



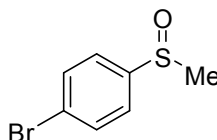
Brown solid; isolated yield 69% (77 mg).  $R_f$  0.50 (60% EtOAc/hexane); Mp 118-120 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 522, 1061, 1344, 1538, 1638, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.22 - 8.25 (m, 2H), 7.86 (dd,  $J = 2.0, 8.4$  Hz, 1H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  145.0, 143.7, 137.9, 135.7, 127.6, 125.1; **HRMS** for  $\text{C}_7\text{H}_3\text{D}_3\text{ClNO}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 223.0018, found: 223.0016

#### 1-Chloro-4-(methylsulfinyl)benzene (3m)<sup>2</sup>



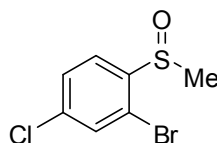
Colorless solid; isolated yield 60% (52 mg).  $R_f$  0.50 (50% EtOAc/hexane); Mp 45-47 °C (lit.<sup>2</sup> 45-46.3 °C); **IR** (Film,  $\text{cm}^{-1}$ ): 771, 822, 1052, 1644;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.51 - 7.54 (m, 2H), 7.43 - 7.46 (m, 2H), 2.65 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  144.2, 137.3, 129.7, 125.0, 44.0; **HRMS** for  $\text{C}_7\text{H}_7\text{ClOS}$ : calcd. ( $\text{MH}^+$ ): 174.9979, found: 174.9978

#### 1-Bromo-4-(methylsulfinyl)benzene (3n)<sup>5</sup>



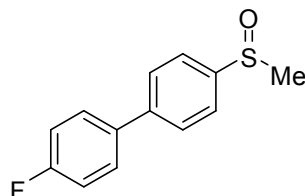
Yellow solid; isolated yield 50% (55 mg).  $R_f$  0.50 (50% EtOAc/hexane); Mp 85-87 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 669, 1068, 1645, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 (d,  $J = 8.5$  Hz, 2H), 7.46 (d,  $J = 8.5$  Hz, 2H), 2.65 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  144.9, 132.6, 125.5, 125.2, 44.0; **HRMS** for  $\text{C}_7\text{H}_7\text{BrOS}$ : calcd. ( $\text{MH}^+$ ): 218.9474, found: 218.9479

#### 2-Bromo-4-chloro-1-(methylsulfinyl)benzene (3o)



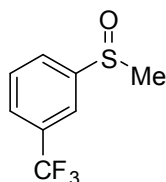
Yellow solid; isolated yield 62% (78 mg).  $R_f$  0.50 (40% EtOAc/hexane); Mp 98-99 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 669, 771, 1023, 1059, 1646, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.81 (d,  $J = 8.3$  Hz, 1H), 7.49 – 7.52 (m, 2H), 2.74 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  144.1, 137.8, 132.6, 129.2, 126.9, 118.7, 41.9; **HRMS** for  $\text{C}_7\text{H}_6\text{BrClOS}$ : calcd. ( $\text{MH}^+$ ): 252.9084, found: 252.9085

#### 4-Fluoro-4'-(methylsulfinyl)biphenyl (3p)



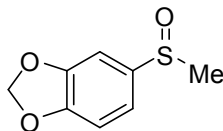
Yellow solid; isolated yield 63% (74 mg).  $R_f$  0.50 (60% EtOAc/hexane); Mp 113-114 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 1046, 1159, 1603, 3020;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.61 – 7.66 (m, 4H), 7.47 – 7.51 (m, 2H), 7.09 (t,  $J = 8.6$  Hz, 2H), 2.69 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  162.9 (d,  $J_{\text{C-F}} = 246.5$  Hz), 144.6, 143.2, 135.9 (d,  $J_{\text{C-F}} = 3.1$  Hz), 128.9 (d,  $J_{\text{C-F}} = 8.3$  Hz), 127.9, 124.1, 115.9 (d,  $J_{\text{C-F}} = 21.6$  Hz), 44.0; **HRMS** for  $\text{C}_{13}\text{H}_{11}\text{FOS}$ : calcd. ( $\text{MH}^+$ ): 235.0587, found: 235.0590

#### 1-(Methylsulfinyl)-3-(trifluoromethyl)benzene (3q)<sup>2</sup>



Brown oil; isolated yield 64% (67 mg).  $R_f$  0.50 (50% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 769, 1064, 1326, 3395;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 (br s, 1H), 7.80 (d,  $J = 7.3$  Hz, 1H), 7.71 (d,  $J = 7.7$  Hz, 1H), 7.60 – 7.64 (m, 1H), 2.74 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  147.3, 131.9, 130.0, 127.9, 127.8, 126.9, 120.7, 44.0; **HRMS** for  $\text{C}_8\text{H}_7\text{F}_3\text{OS}$ : calcd. ( $\text{MH}^+$ ): 209.0242, found: 209.0245

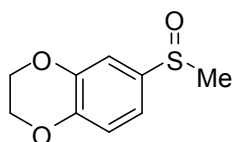
#### 5-(Methylsulfinyl)benzo[d][1,3]dioxole (3r)



Brown oil; isolated yield 65% (60 mg).  $R_f$  0.50 (70% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1040, 1241, 1637, 3018;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.09 (d,  $J = 1.3$  Hz, 1H), 7.05 (d,  $J = 8.0$  Hz, 1H), 6.84 (d,  $J = 8.0$  Hz, 1H), 5.98 (s, 2H), 2.61 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  150.3, 148.8, 138.9, 118.4, 108.8, 103.8, 101.9, 44.2; **HRMS** for  $\text{C}_8\text{H}_8\text{O}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 185.0267, found: 185.0268

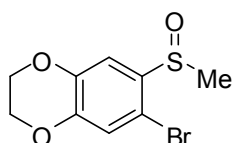


### 6-(Methylsulfinyl)-2,3-dihydrobenzo[b][1,4]dioxine (3s)



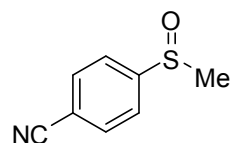
Brown oil; isolated yield 65% (65 mg).  $R_f$  0.50 (70% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1067, 1253, 1285, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.11 (d,  $J = 2.1$  Hz, 1H), 7.03 (dd,  $J = 2.1, 8.4$  Hz, 1H), 6.91 (d,  $J = 8.4$  Hz, 1H), 4.21 (s, 4H), 2.61 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  146.1, 144.4, 137.8, 118.3, 116.9, 113.0, 64.4, 64.3, 44.0; **HRMS** for  $\text{C}_9\text{H}_{10}\text{O}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 199.0423, found: 199.0421

### 6-Bromo-7-(methylsulfinyl)-2,3-dihydrobenzo[b][1,4]dioxine (3t)



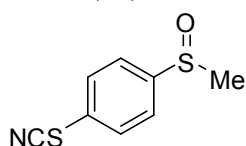
Brown gummy solid; isolated yield 67% (93 mg).  $R_f$  0.50 (70% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1066, 1089, 1258, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.38 (s, 1H), 7.00 (s, 1H), 4.22 (s, 4H), 2.69 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  146.5, 144.6, 137.2, 121.5, 114.7, 108.5, 64.5, 64.2, 42.3; **HRMS** for  $\text{C}_9\text{H}_9\text{BrO}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 276.9529, found: 276.9528

### 4-(methylsulfinyl)benzonitrile (3u)<sup>2</sup>



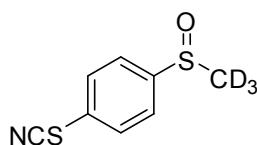
Colorless solid; isolated yield 65% (54 mg).  $R_f$  0.50 (50% EtOAc/hexane); **Mp** 87-89 °C (lit.<sup>2</sup> 86.3-87.9 °C); **IR** (Film,  $\text{cm}^{-1}$ ): 1054, 1644, 2233, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.77 (d,  $J = 8.5$  Hz, 2H), 7.70 (d,  $J = 8.5$  Hz, 2H), 2.70 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  151.5, 133.0, 124.3, 117.7, 114.9, 43.8; **HRMS** for  $\text{C}_8\text{H}_7\text{NOS}$ : calcd. ( $\text{MH}^+$ ): 166.0321, found: 166.0325

### 1-(Methylsulfinyl)-4-thiocyanatobenzene (3v)



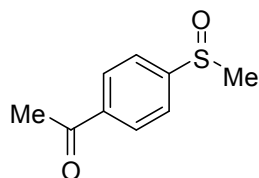
Brown gummy solid; isolated yield 74% (73 mg).  $R_f$  0.50 (60% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 669, 1052, 2162, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60-7.67 (m, 4H), 2.68 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  147.7, 130.0, 128.2, 125.3, 109.2, 44.0; **HRMS** for  $\text{C}_8\text{H}_7\text{NOS}_2$ : calcd. ( $\text{MH}^+$ ): 198.0042, found: 198.0042

### 1-(Methylsulfinyl)-4-thiocyanatobenzene-*d*<sub>3</sub> (3v')



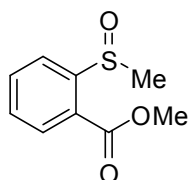
Brown gummy solid; isolated yield 75% (75 mg).  $R_f$  0.50 (60% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 669, 1054, 1637, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60-7.66 (m, 4H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  147.6, 130.0, 128.2, 125.3, 109.2; **HRMS** for  $\text{C}_8\text{H}_4\text{D}_3\text{NOS}_2$ : calcd. ( $\text{MH}^+$ ): 201.0230, found: 201.0231

### 1-(4-(methylsulfinyl)phenyl)ethanone (3w)<sup>2</sup>



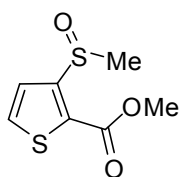
Brown solid; isolated yield 71% (65 mg).  $R_f$  0.50 (50% EtOAc/hexane); Mp 108-109 °C (lit.<sup>2</sup> 107.1-108.0 °C); **IR** (Film,  $\text{cm}^{-1}$ ): 1068, 1687, 3020;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.04 (d,  $J$  = 8.1 Hz, 2H), 7.68 (d,  $J$  = 8.0 Hz, 2H), 2.69 (s, 3H), 2.58 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  197.0, 150.9, 139.1, 129.2, 123.8, 43.8, 26.8; **HRMS** for  $\text{C}_9\text{H}_{10}\text{O}_2\text{S}$ : calcd. ( $\text{MH}^+$ ): 183.0474, found: 183.0477

### Methyl 2-(methylsulfinyl)benzoate (3x)<sup>6</sup>



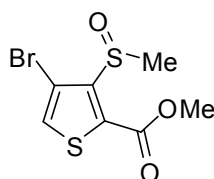
Brown oil; isolated yield 49% (49 mg).  $R_f$  0.50 (50% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1065, 1644, 1714, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.25 (d,  $J$  = 7.9 Hz, 1H), 8.02 (d,  $J$  = 7.8 Hz, 1H), 7.76 (t,  $J$  = 7.7 Hz, 1H), 7.50 (t,  $J$  = 7.6 Hz, 1H), 3.88 (s, 3H), 2.78 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.8, 150.4, 134.1, 130.7, 130.2, 126.5, 124.2, 52.6, 44.1; **HRMS** for  $\text{C}_9\text{H}_{10}\text{O}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 199.0423, found: 199.0428

### Methyl 3-(methylsulfinyl)thiophene-2-carboxylate (3y)



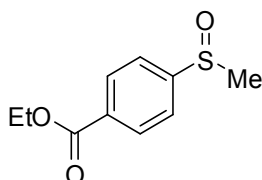
Colorless solid; isolated yield 73% (75 mg).  $R_f$  0.50 (50% EtOAc/hexane); Mp 105-106 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 1056, 1156, 1275, 1644, 1705, 3020;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 – 7.61 (m, 2H), 3.84 (s, 3H), 2.83 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  161.3, 154.2, 132.3, 127.1, 126.1, 52.7, 42.7; **HRMS** for  $\text{C}_7\text{H}_8\text{O}_3\text{S}_2$ : calcd. ( $\text{MH}^+$ ): 204.9988, found: 204.9989

### Methyl 4-bromo-3-(methylsulfinyl)thiophene-2-carboxylate (3z)



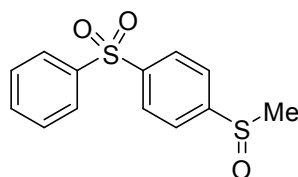
Brown solid; isolated yield 60% (85 mg).  $R_f$  0.50 (50% EtOAc/hexane); Mp 109-110 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 627, 1067, 1637, 1718;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49 (s, 1H), 3.85 (s, 3H), 3.01 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.0, 145.1, 132.6, 130.6, 110.2, 53.1, 40.0; **HRMS** for  $\text{C}_7\text{H}_7\text{BrO}_3\text{S}_2$ : calcd. ( $\text{MH}^+$ ): 282.9093, found: 282.9095

#### Ethyl 4-(methylsulfinyl)benzoate (3za)



Brown oil; isolated yield 75% (80 mg).  $R_f$  0.50 (50% EtOAc/hexane); **IR** (Film,  $\text{cm}^{-1}$ ): 1051, 1277, 1639, 1717, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.13 (d,  $J = 8.2$  Hz, 2H), 7.65 (d,  $J = 8.2$  Hz, 2H), 4.34 (q, 2H), 2.68 (s, 3H), 1.35 (t,  $J = 7.1$  Hz, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.5, 150.7, 133.0, 130.5, 123.5, 61.5, 43.9, 14.3; **HRMS** for  $\text{C}_{10}\text{H}_{12}\text{O}_3\text{S}$ : calcd. ( $\text{MH}^+$ ): 213.0580, found: 213.0577

#### 1-(Methylsulfinyl)-4-(phenylsulfonyl)benzene (3zb)



Brown solid; isolated yield 80% (112 mg).  $R_f$  0.50 (100% EtOAc); Mp 85-86 °C; **IR** (Film,  $\text{cm}^{-1}$ ): 607, 1068, 1637, 3019;  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.01 – 8.04 (m, 2H), 7.88 – 7.91 (m, 2H), 7.69 – 7.72 (m, 2H), 7.52 – 7.56 (m, 1H), 7.45 – 7.49 (m, 2H), 2.67 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  151.7, 144.3, 140.8, 133.7, 129.5, 128.6, 127.9, 124.5, 43.8; **HRMS** for  $\text{C}_{13}\text{H}_{12}\text{O}_3\text{S}_2$ : calcd. ( $\text{MH}^+$ ): 281.0301, found: 141.0367

## 4. References

1. (a) J. Wu, Y. Gu, X. Leng and Q. Shen, *Angew. Chem., Int. Ed.*, 2015, **54**, 7648; (b) K. Zhang, X.-H. Xu and F.-L. Qing, *J. Org. Chem.*, 2015, **80**, 7658
2. P. Hanson, R. A. A. J. Hendrickx and J. R. L. Smith, *Org. Biomol. Chem.*, 2008, **6**, 745
3. J. Sun, C. Zhu, Z. Dai, M. Yang, Y. Pan, and H. Hu, *J. Org. Chem.*, 2004, **69**, 8500
4. M. Akazome, Y. Ueno, H. Ooiso and K. Ogura, *J. Org. Chem.*, 2000, **65**, 68
5. L. Zhao, H. Zhang and Y. Wang, *J. Org. Chem.*, 2016, **81**, 129
6. W. Dai, G. Li, L. Wang, B. Chen, S. Shang, Y. Lv and S. Gao, *RSC Adv.*, 2014, **4**, 46545

## 5. Spectra

NRMP-393

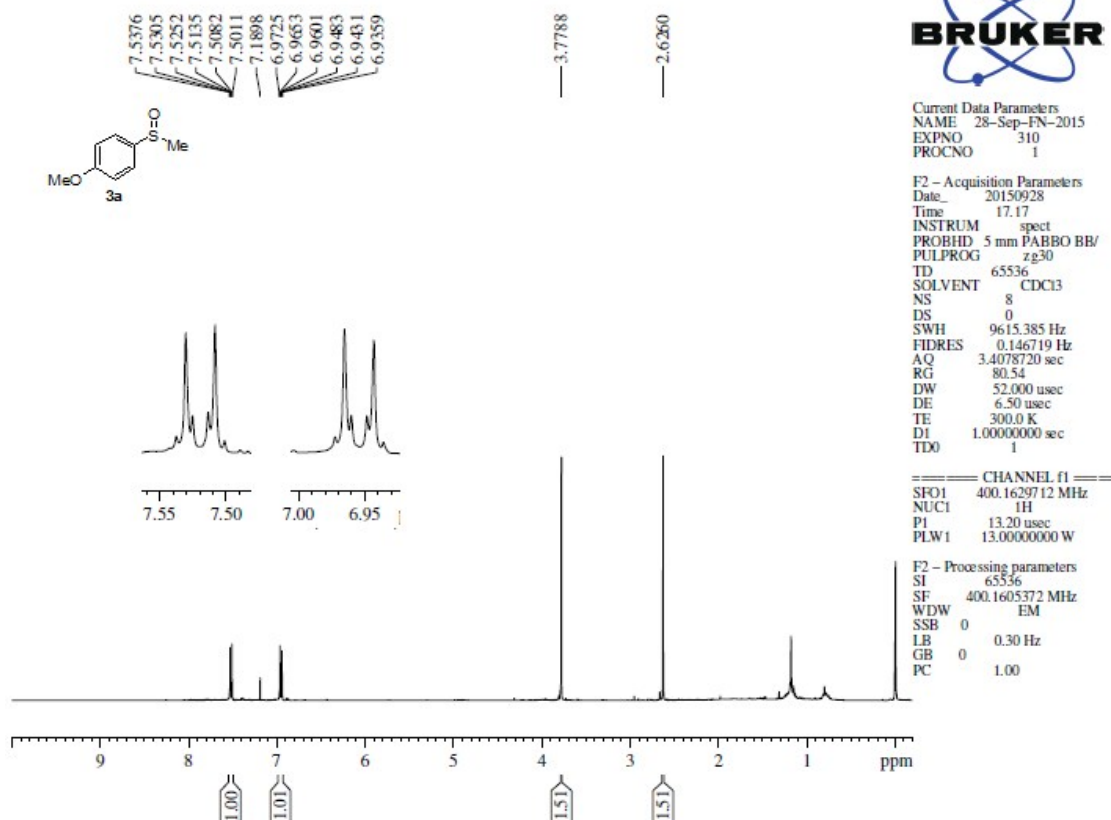


Figure 1:  $^1\text{H}$  NMR spectrum of 3a

NRMP-393

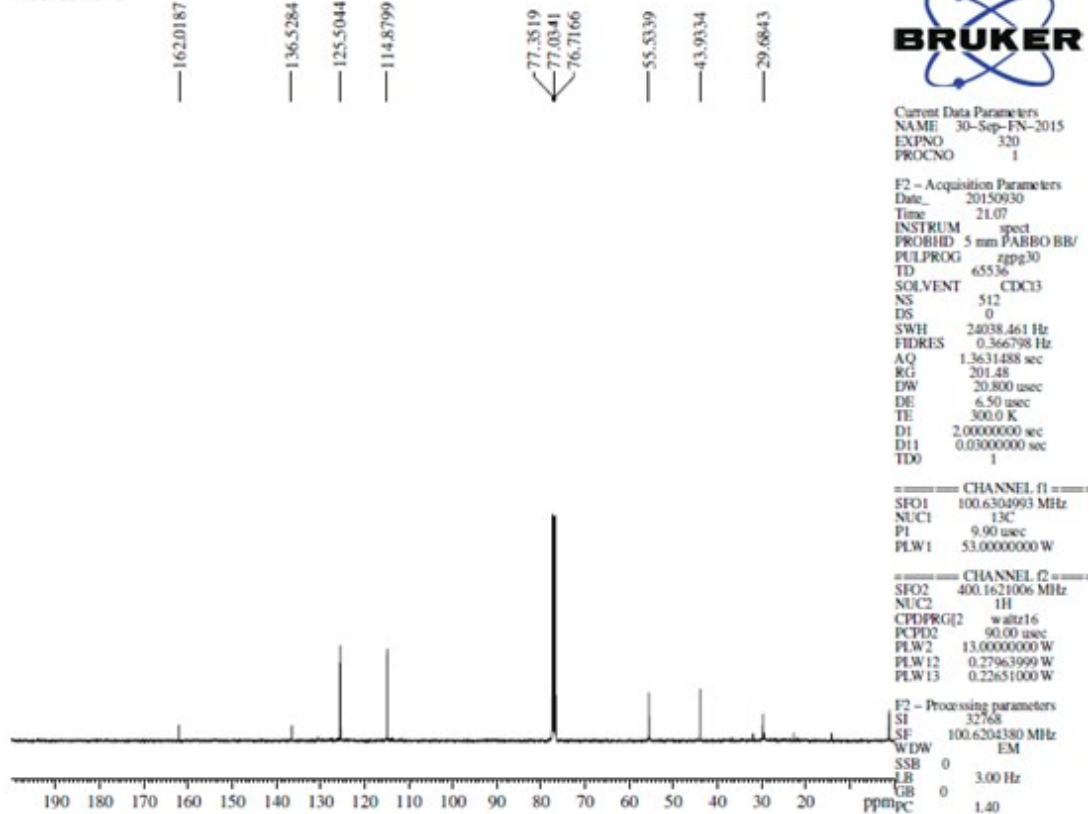


Figure 2:  $^{13}\text{C}$  NMR spectrum of 3a

NRMP-508

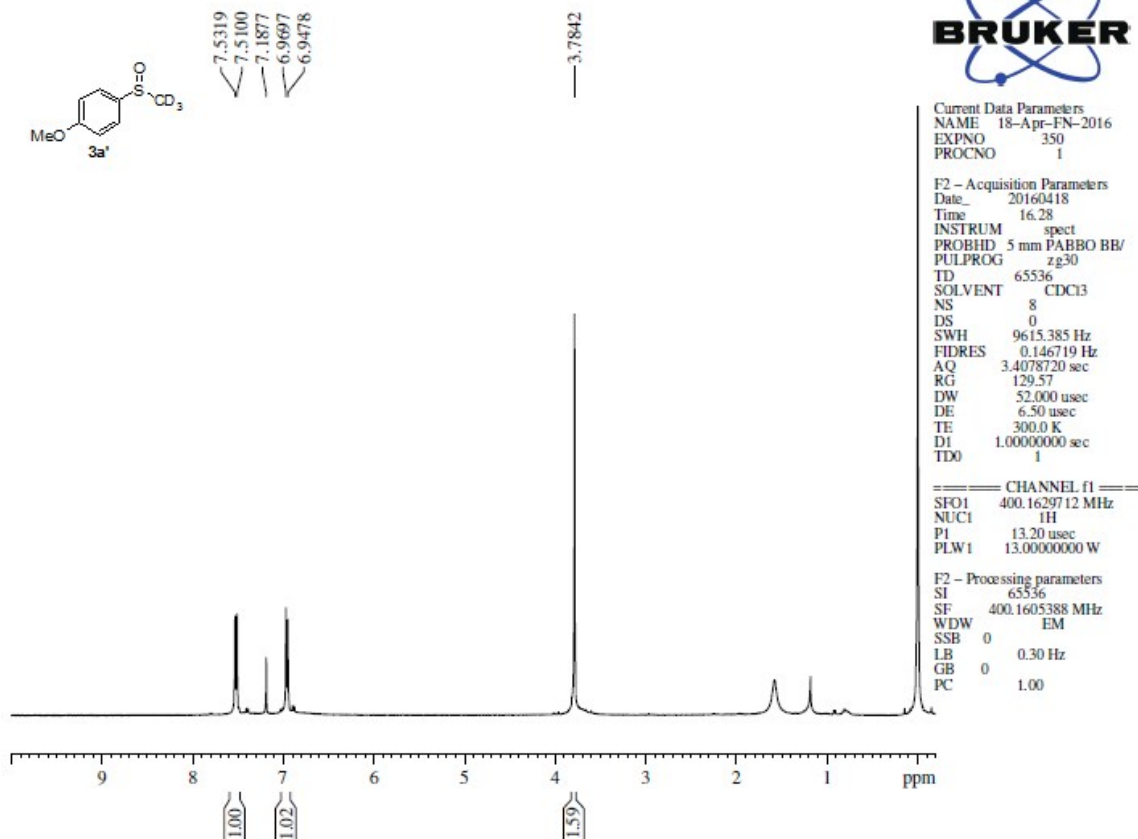


Figure 3: <sup>1</sup>H NMR spectrum of 3a'

NRMP-508

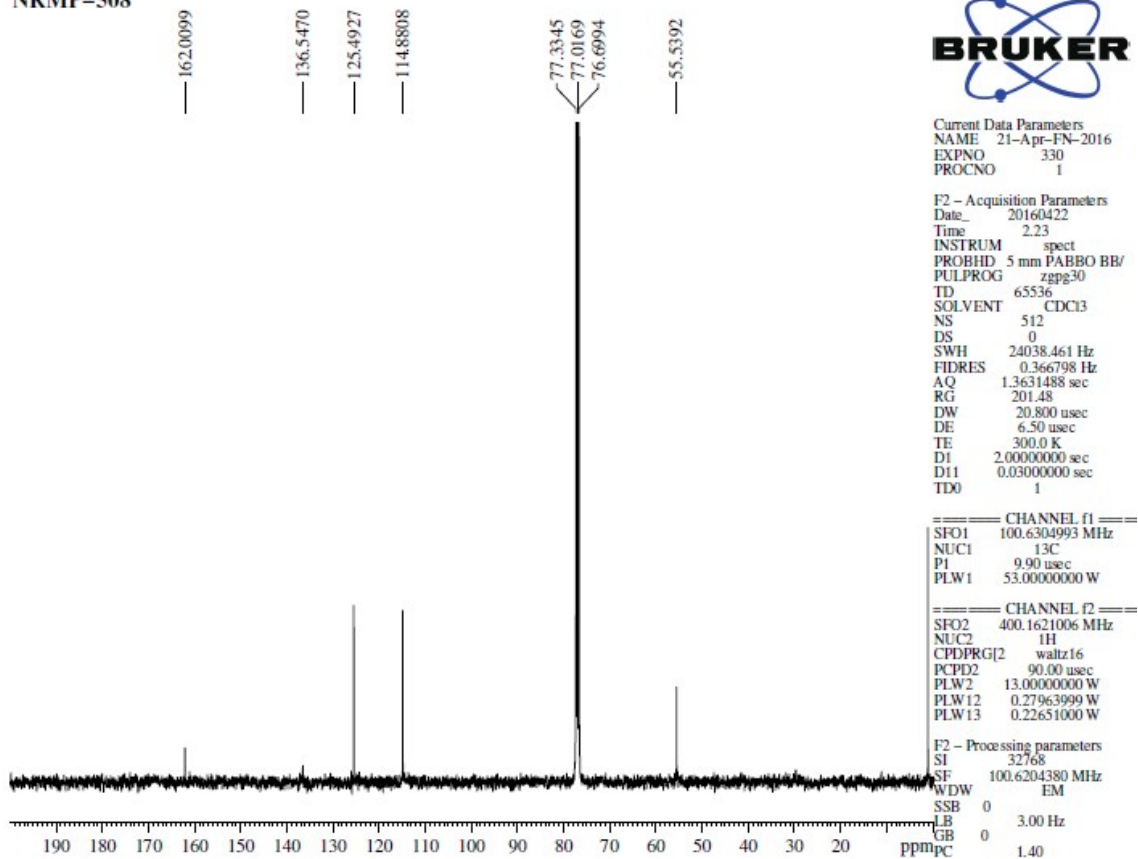
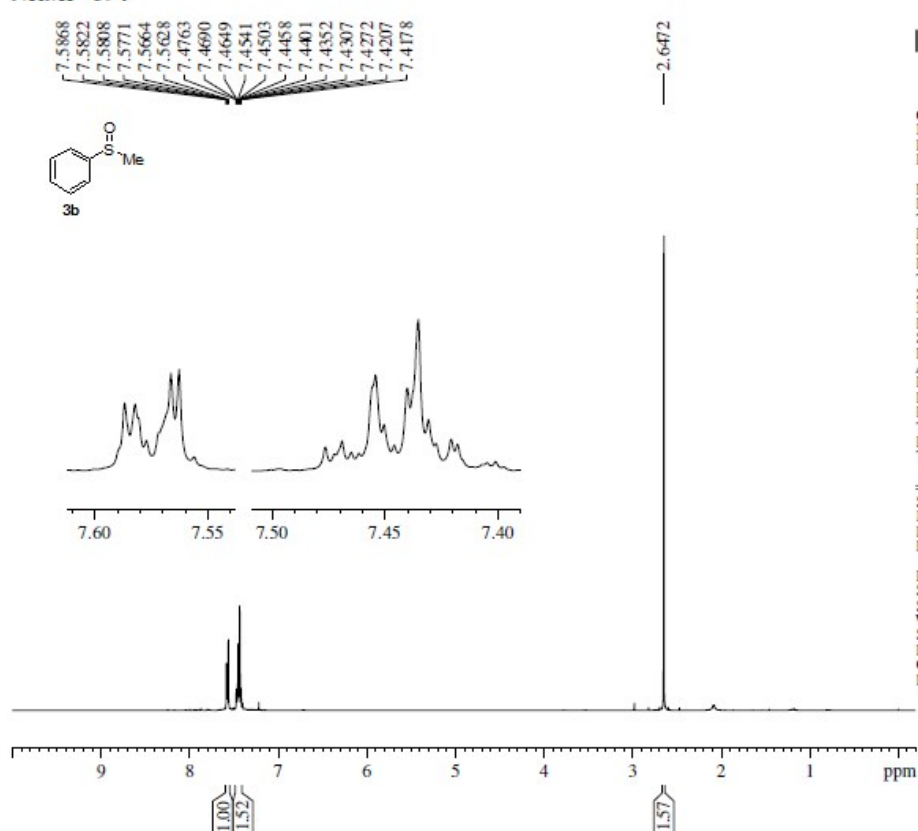


Figure 4: <sup>13</sup>C NMR spectrum of 3a'

NRMP-394



Current Data Parameters  
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 PROCNO 1

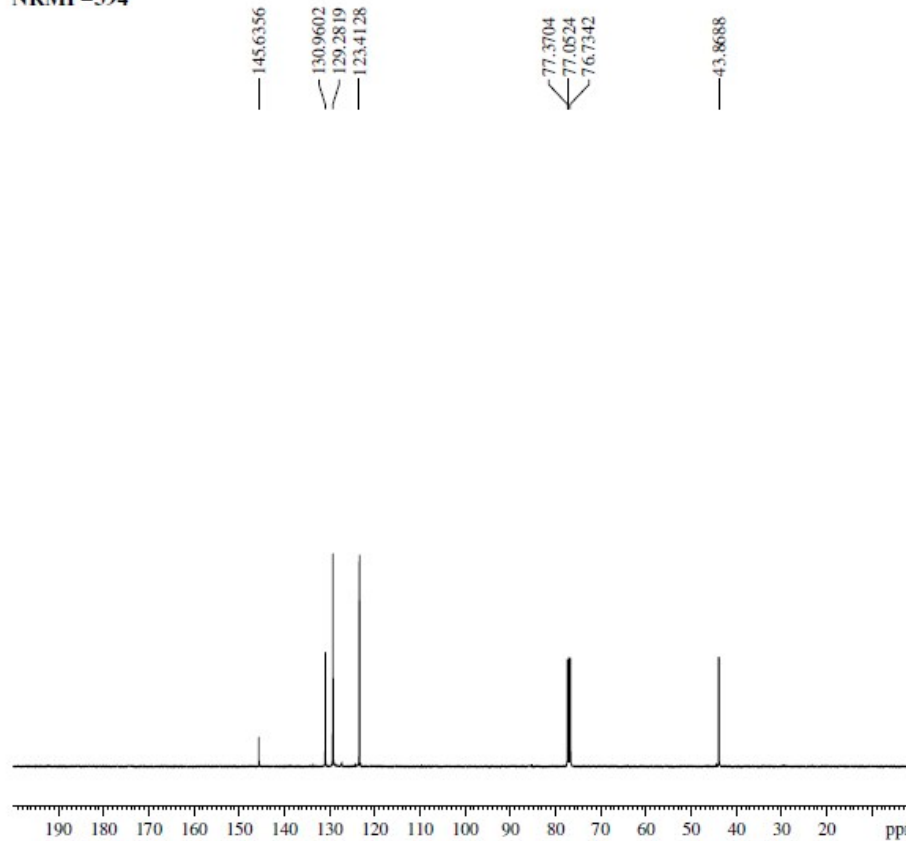
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 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 58.68  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 12.85 usec  
 PLW1 13.1000038 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605262 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 5: <sup>1</sup>H NMR spectrum of 3b

NRMP-394



Current Data Parameters  
 NAME 27-Oct-FN-2015  
 EXPNO 350  
 PROCNO 1

F2 - Acquisition Parameters  
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 PULPROG zgpg30  
 TD 65536  
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 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

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 NUC1 13C  
 P1 9.00 usec  
 PLW1 61.09999847 W

==== CHANNEL f2 ====  
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 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.1000038 W  
 PLW12 0.26705000 W  
 PLW13 0.21630999 W

F2 - Processing parameters  
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 SF 100.6204444 MHz  
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 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 6: <sup>13</sup>C NMR spectrum of 3b

NRMP-501

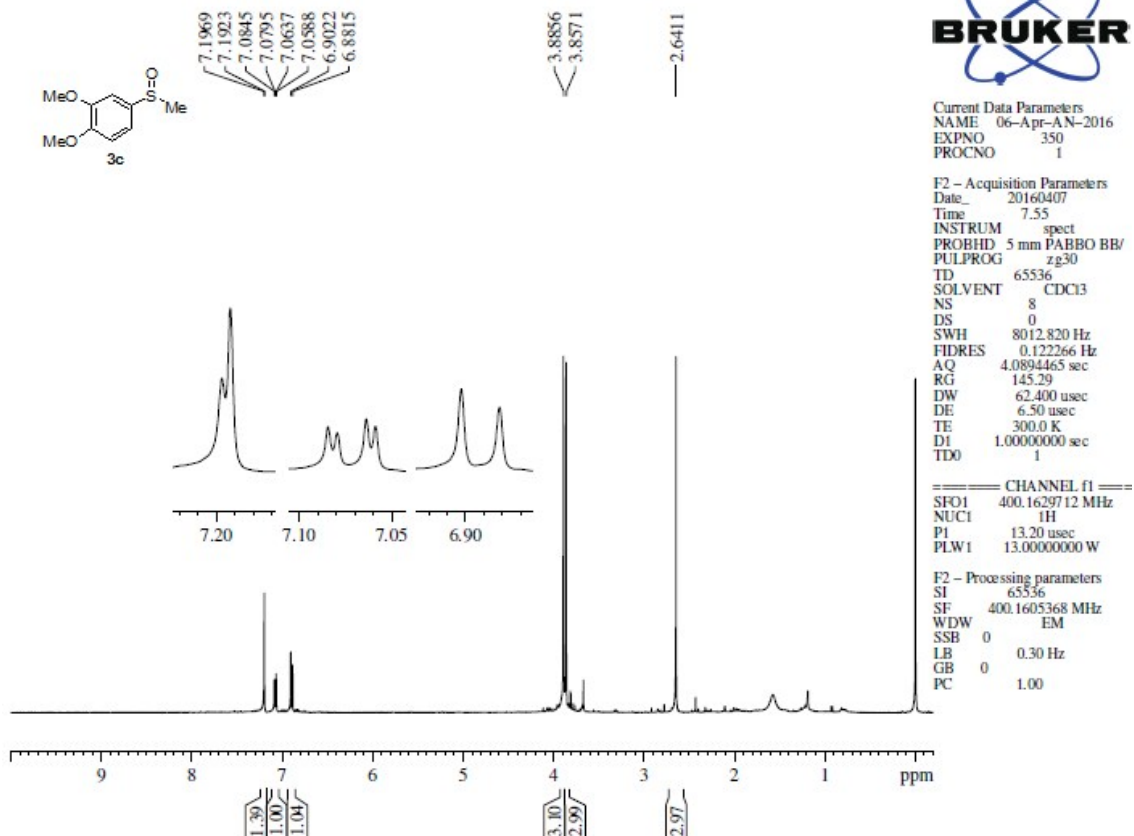


Figure 7: <sup>1</sup>H NMR spectrum of 3c

NRMP-501

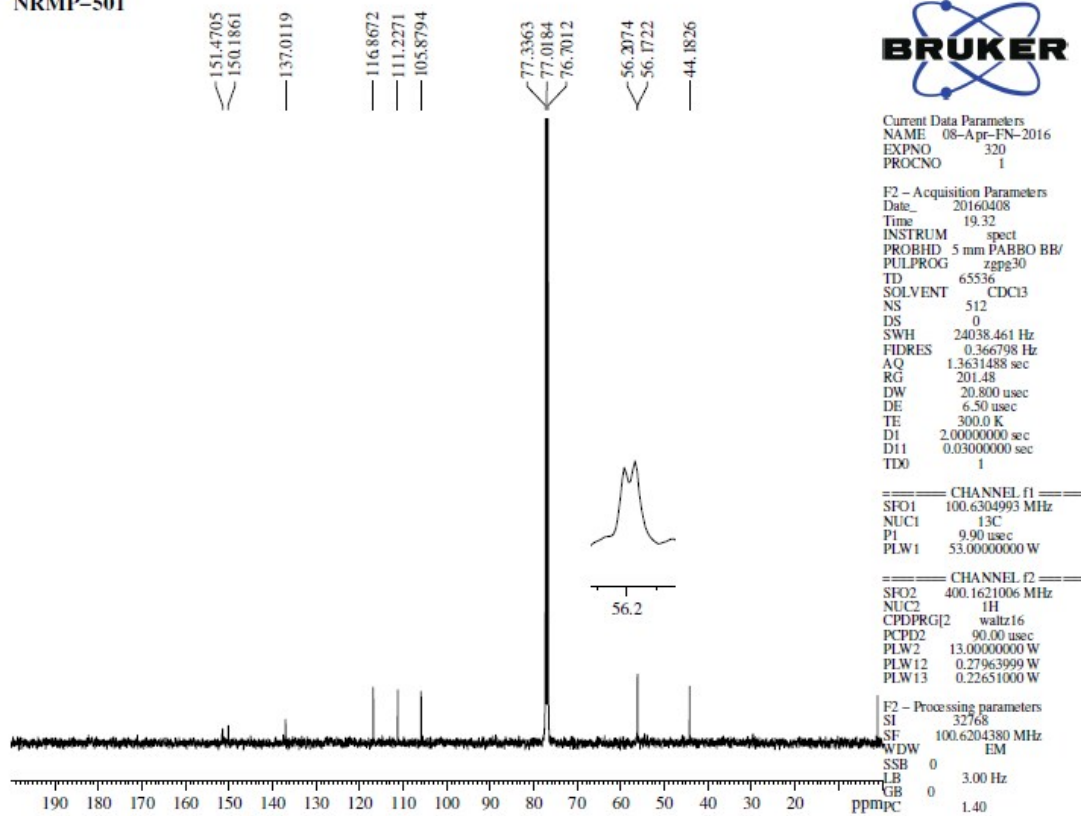


Figure 8: <sup>13</sup>C NMR spectrum of 3c

NRMP-496

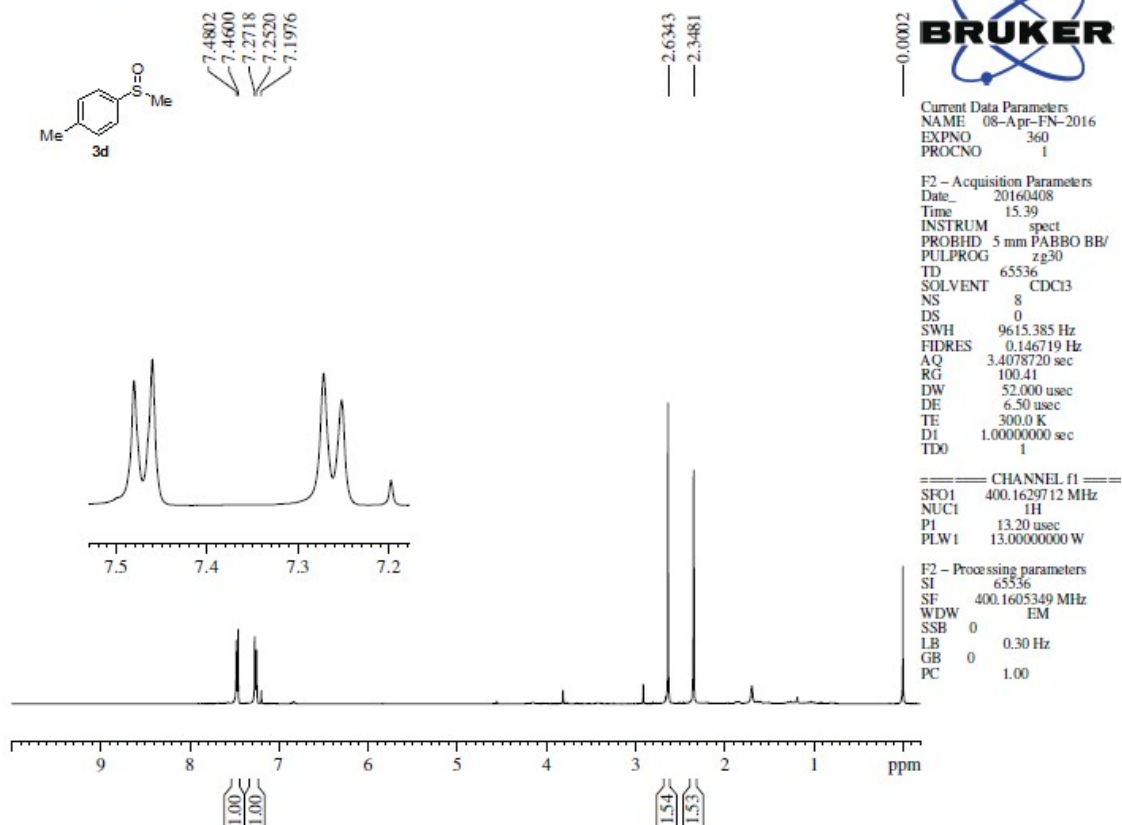


Figure 9: <sup>1</sup>H NMR spectrum of 3d

NRMP-496

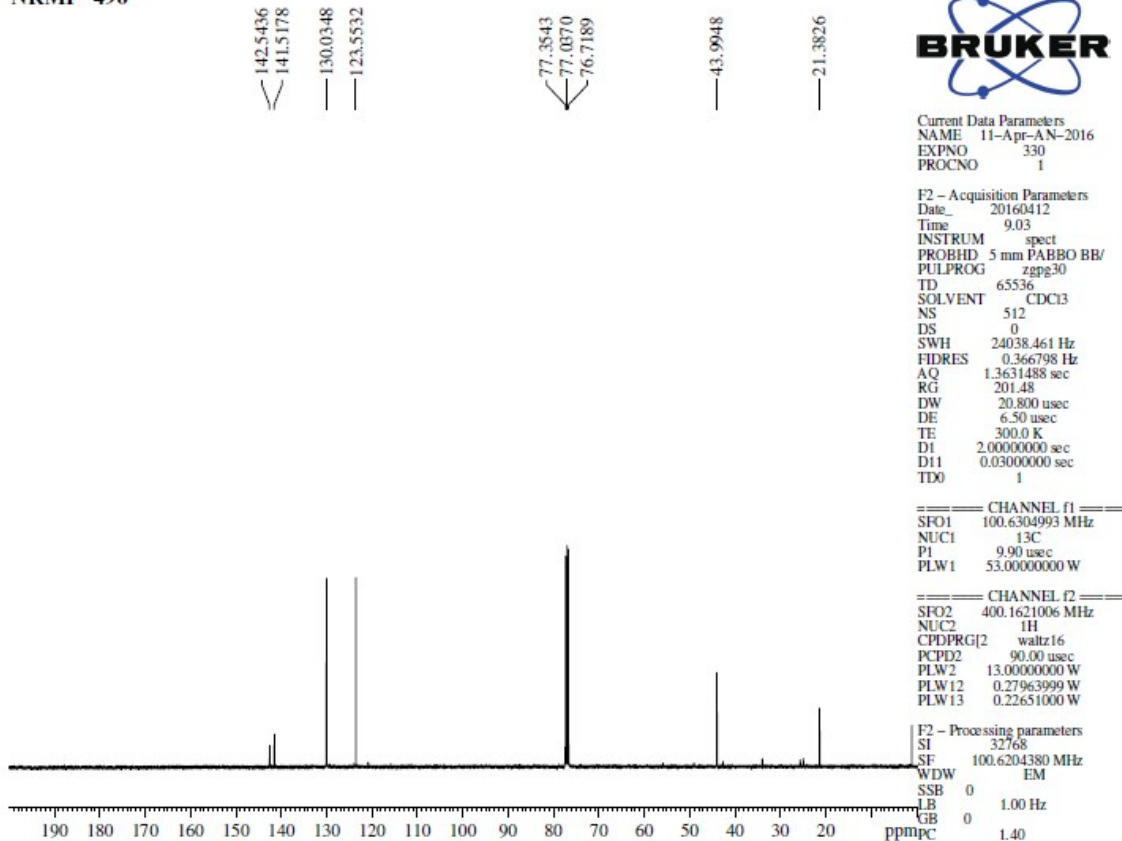


Figure 10: <sup>13</sup>C NMR spectrum of 3d



NRMP-499

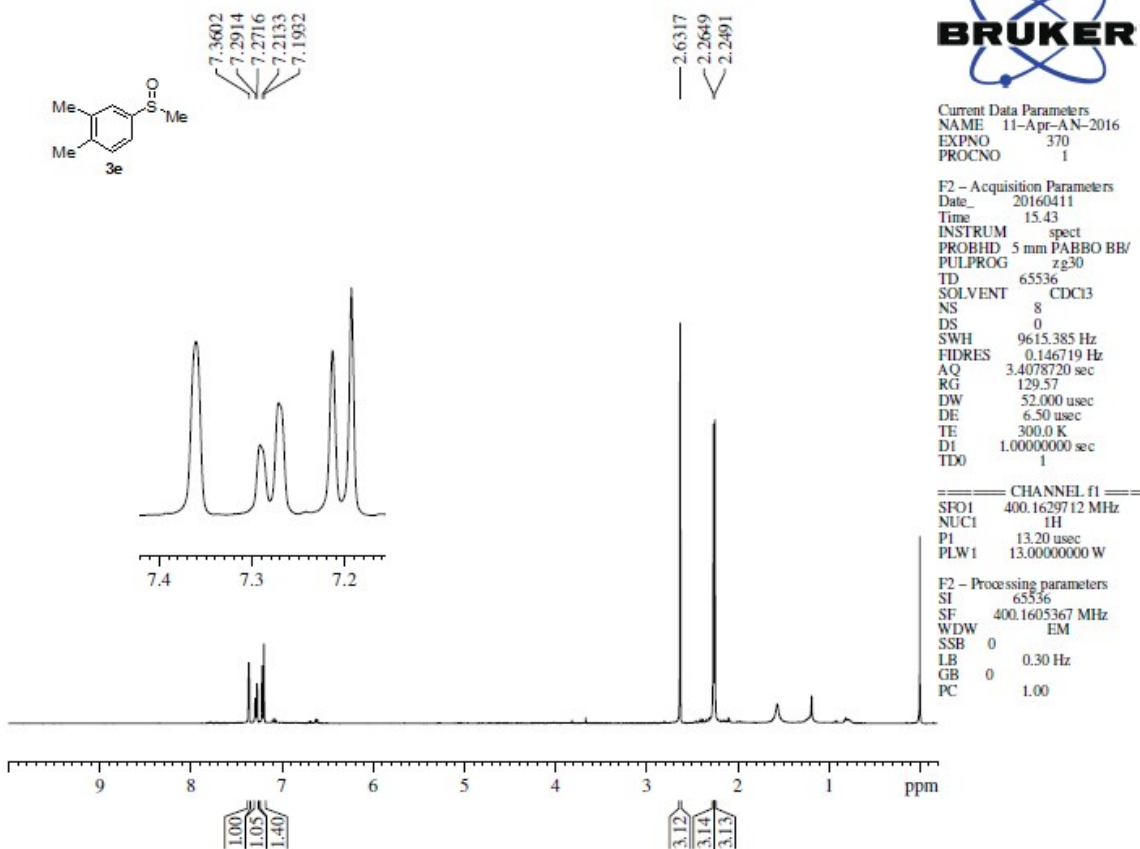


Figure 11: <sup>1</sup>H NMR spectrum of 3e

NRMR-499

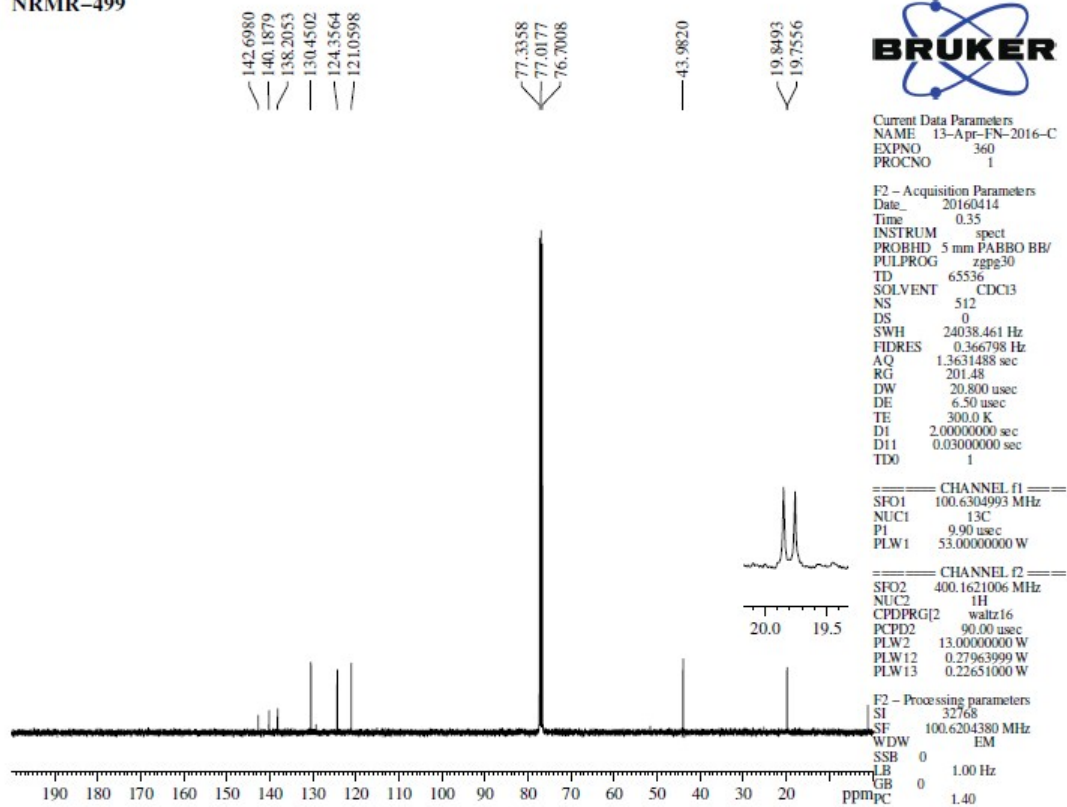


Figure 12: <sup>13</sup>C NMR spectrum of 3e

NRMP-412

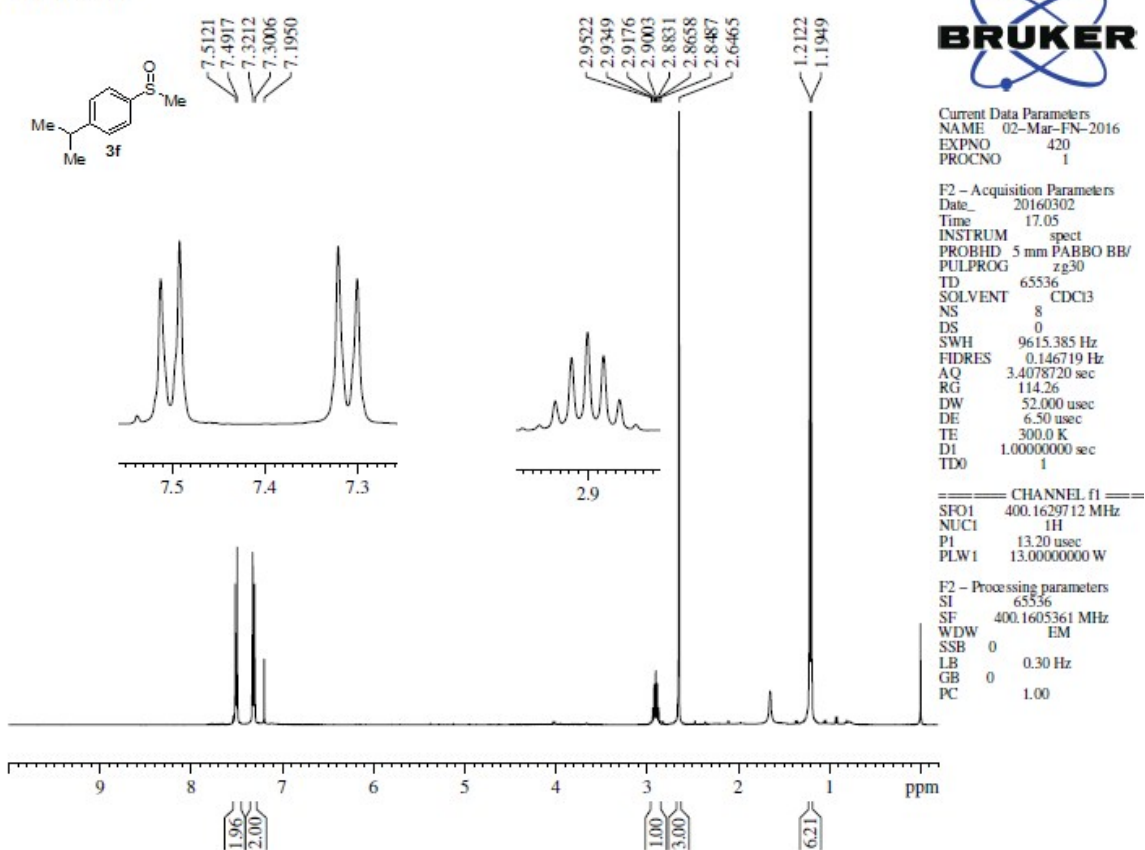


Figure 13: <sup>1</sup>H NMR spectrum of 3f

NRMP-412

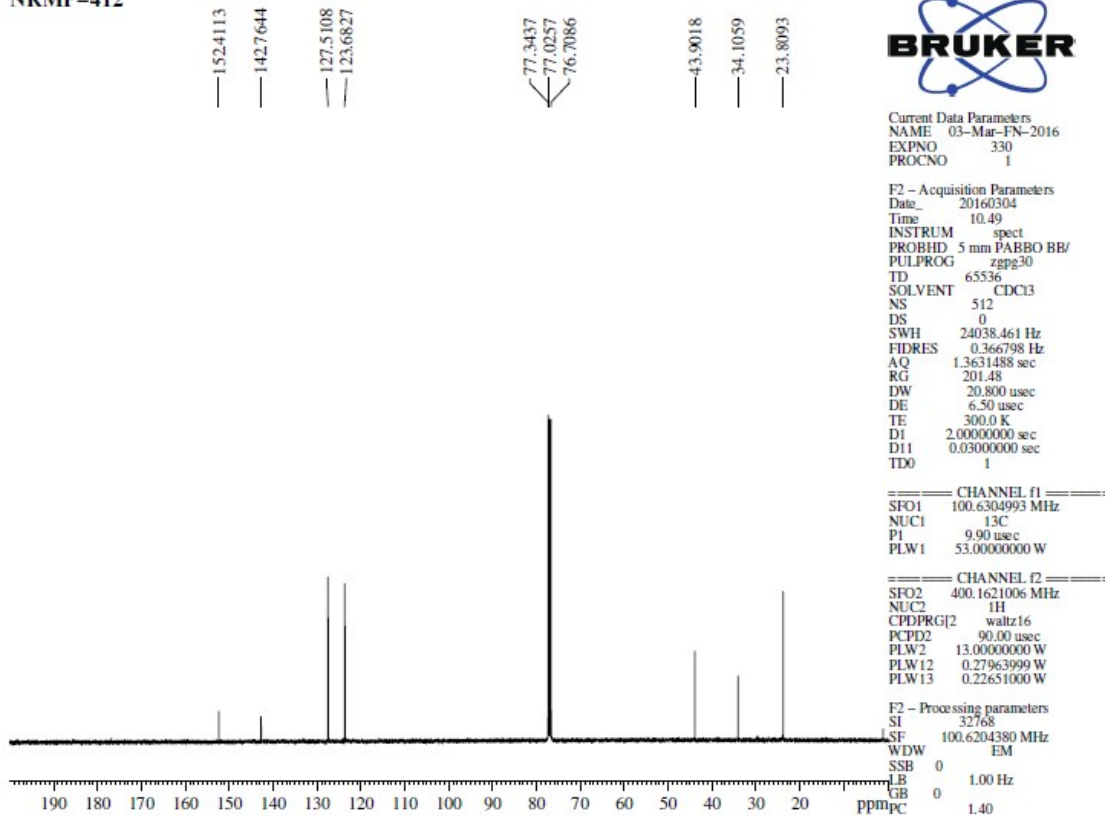
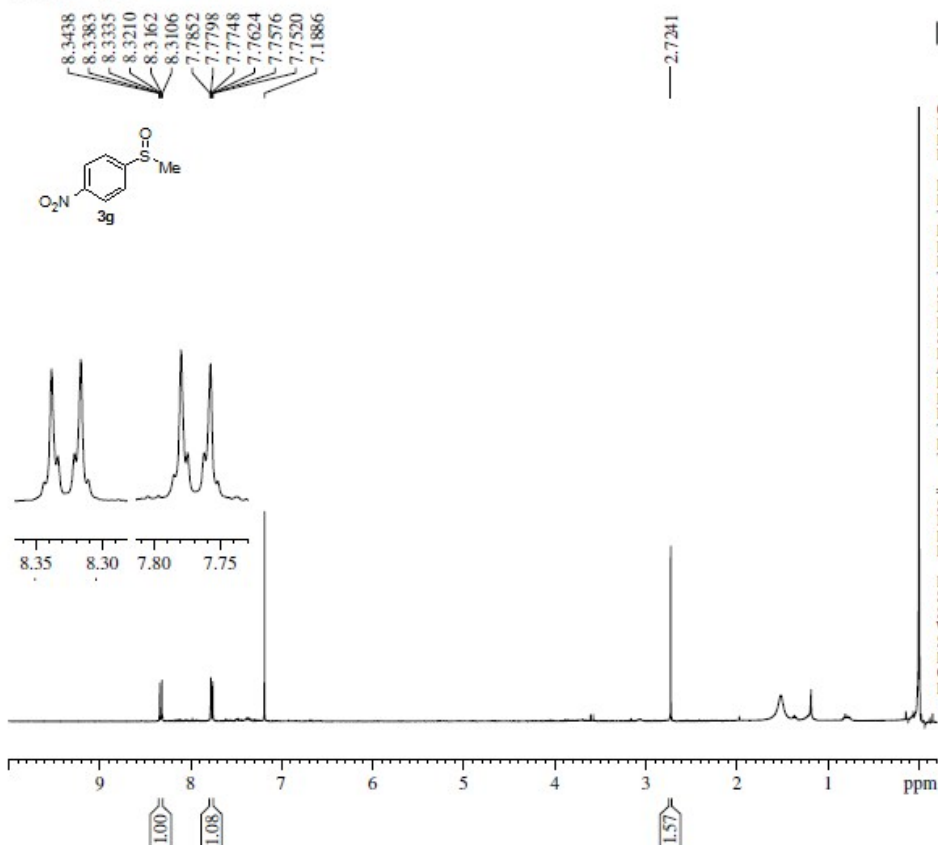


Figure 14: <sup>13</sup>C NMR spectrum of 3f

NRMP-405



Current Data Parameters  
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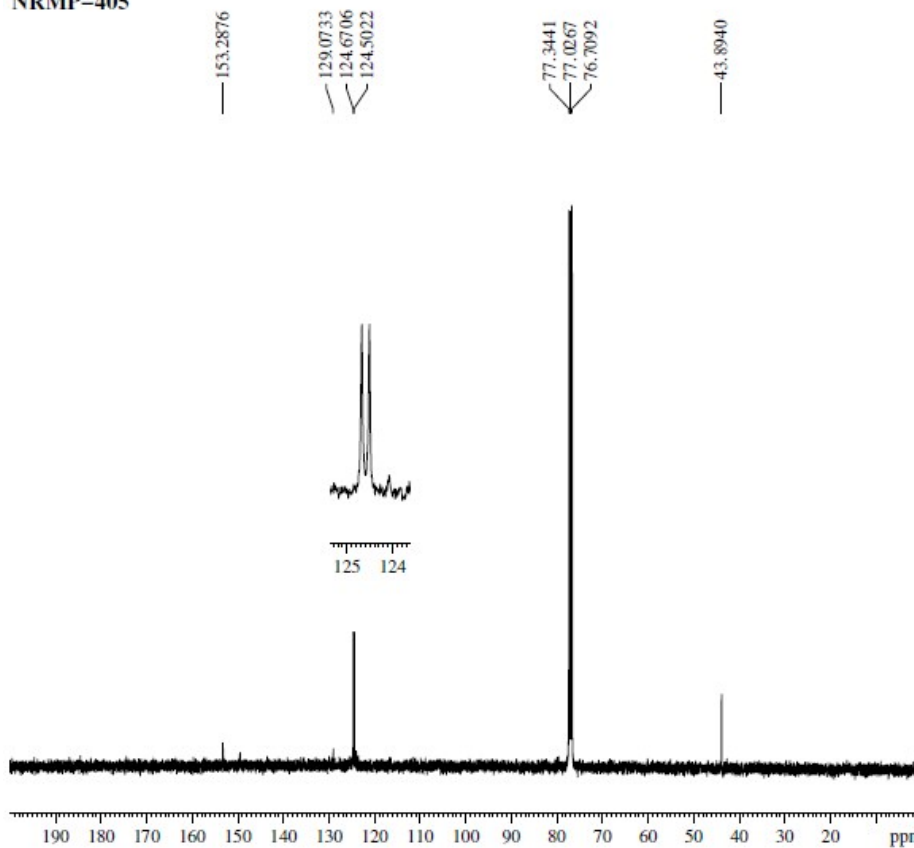
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 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 159.22  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 ====  
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 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605379 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 15: <sup>1</sup>H NMR spectrum of 3g

NRMP-405



Current Data Parameters  
 NAME 21-Mar-FN-2016  
 EXPNO 360  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160323  
 Time 1.54  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

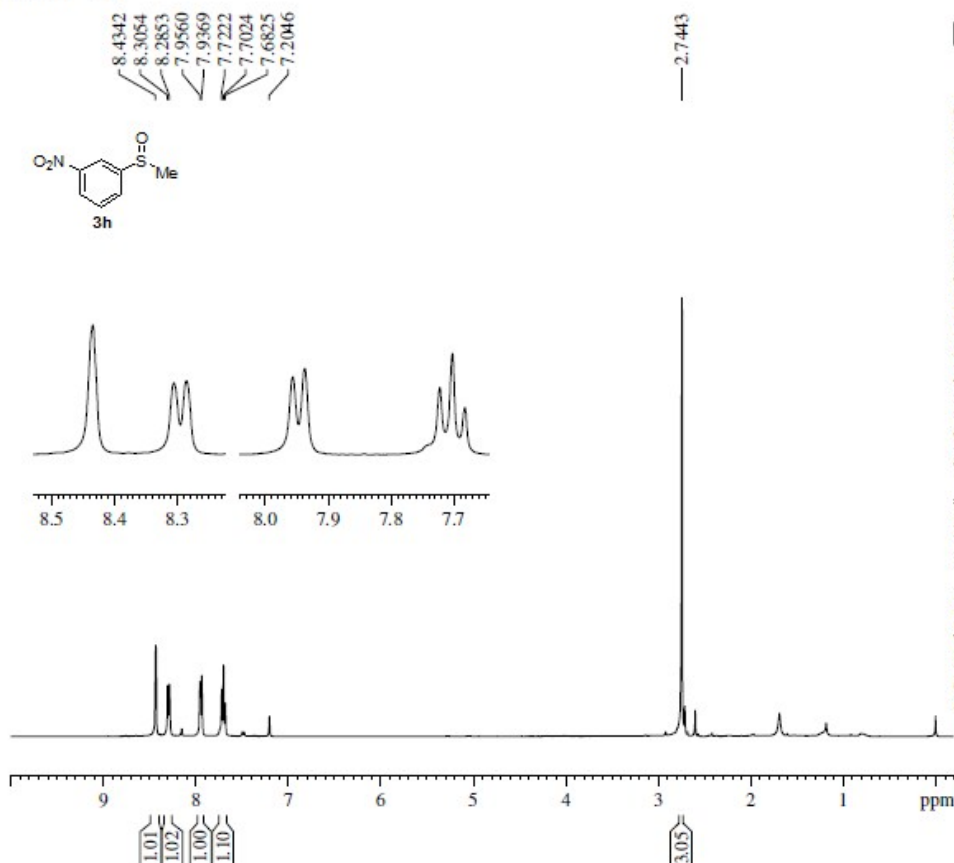
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure 16: <sup>13</sup>C NMR spectrum of 3g

NRMP-411



Current Data Parameters  
 NAME 07-Mar-AN-2016  
 EXPNO 420  
 PROCNO 1

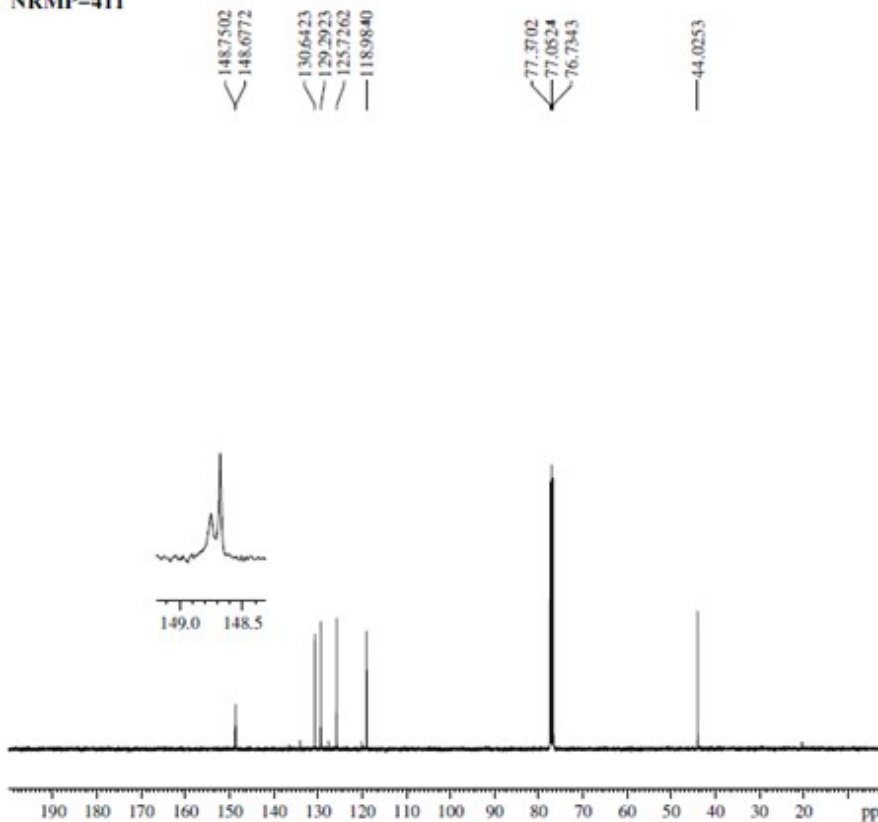
F2 - Acquisition Parameters  
 Date\_ 20160307  
 Time 17.23  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 100.41  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605320 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 17: <sup>1</sup>H NMR spectrum of 3h

NRMP-411



Current Data Parameters  
 NAME 08-Mar-AN-2016  
 EXPNO 320  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160309  
 Time 10.37  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

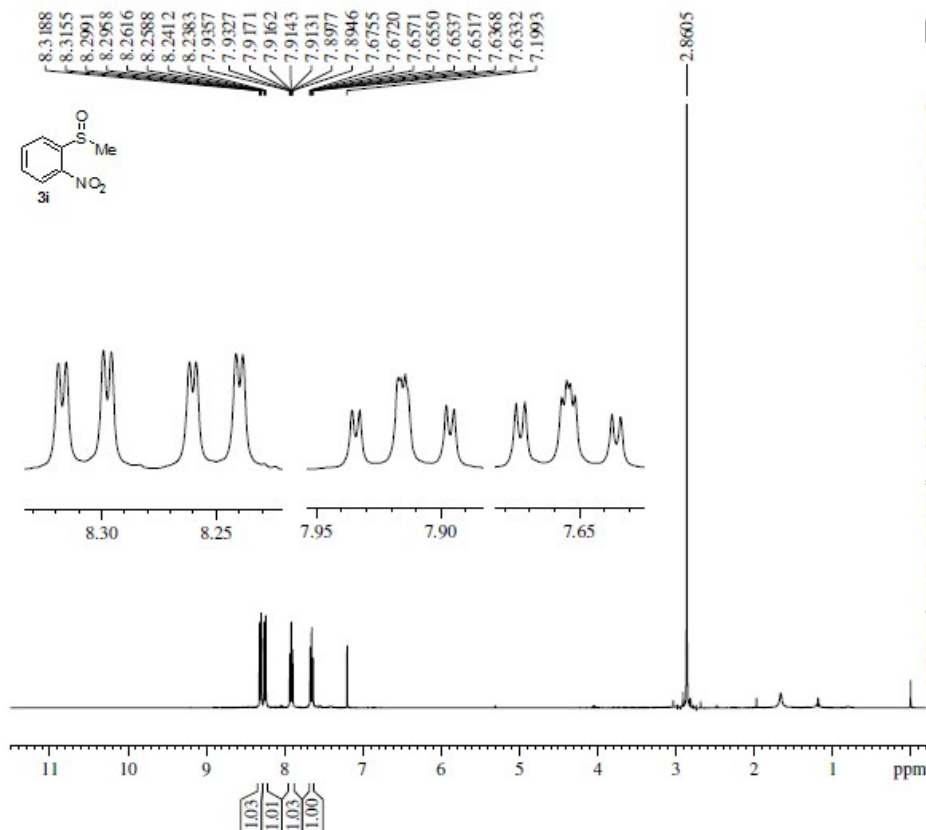
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure 18: <sup>13</sup>C NMR spectrum of 3h

NRMP-416



Current Data Parameters  
 NAME 27-Oct-FN-2015  
 EXPNO 350  
 PROCNO 1

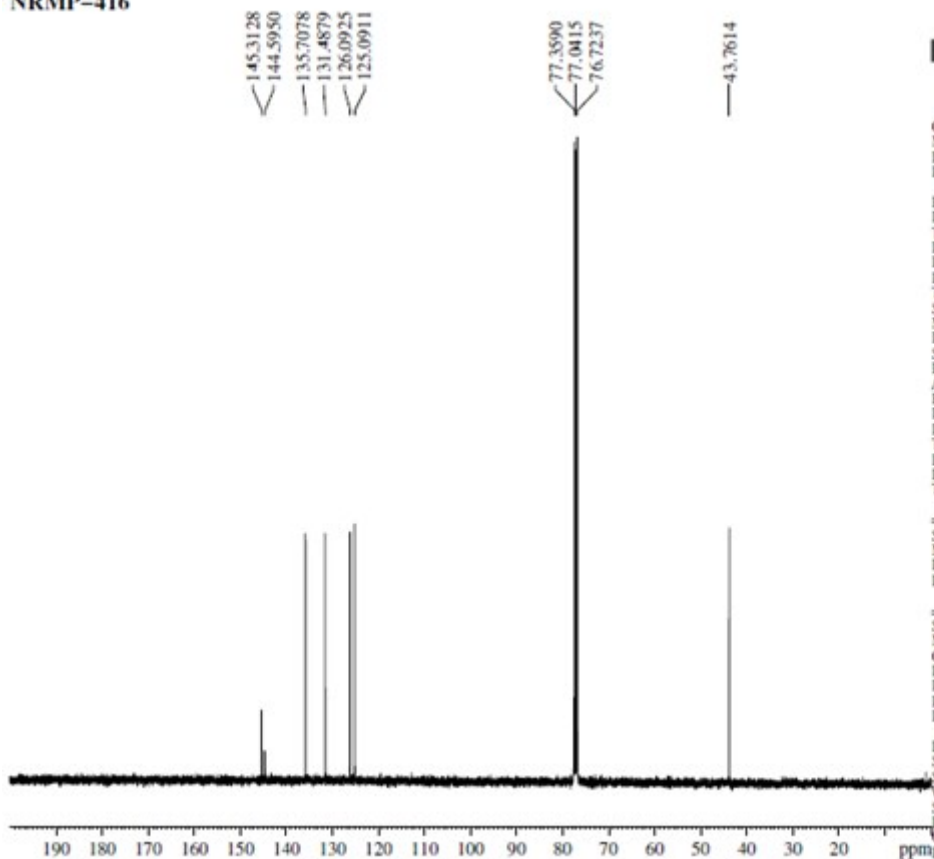
F2 - Acquisition Parameters  
 Date\_ 20151027  
 Time 16.39  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 9  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 114.26  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 12.85 usec  
 PLW1 13.10000038 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605336 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 19: <sup>1</sup>H NMR spectrum of 3i

NRMP-416



Current Data Parameters  
 NAME 05-Nov-AN-2015  
 EXPNO 310  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20151106  
 Time 12.13  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

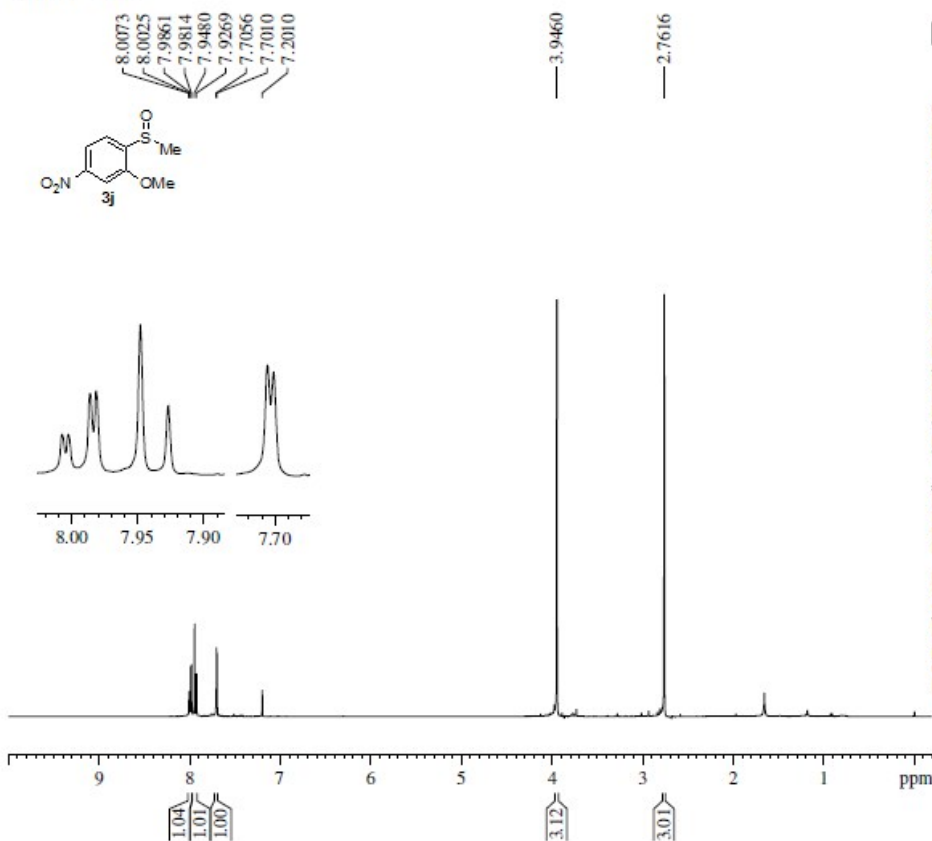
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.00 usec  
 PLW1 61.09999847 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.10000038 W  
 PLW12 0.26705000 W  
 PLW13 0.21630999 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure 20: <sup>13</sup>C NMR spectrum of 3i

NRMP-420



Current Data Parameters  
 NAME 03-Mar-FN-2016  
 EXPNO 410  
 PROCNO 1

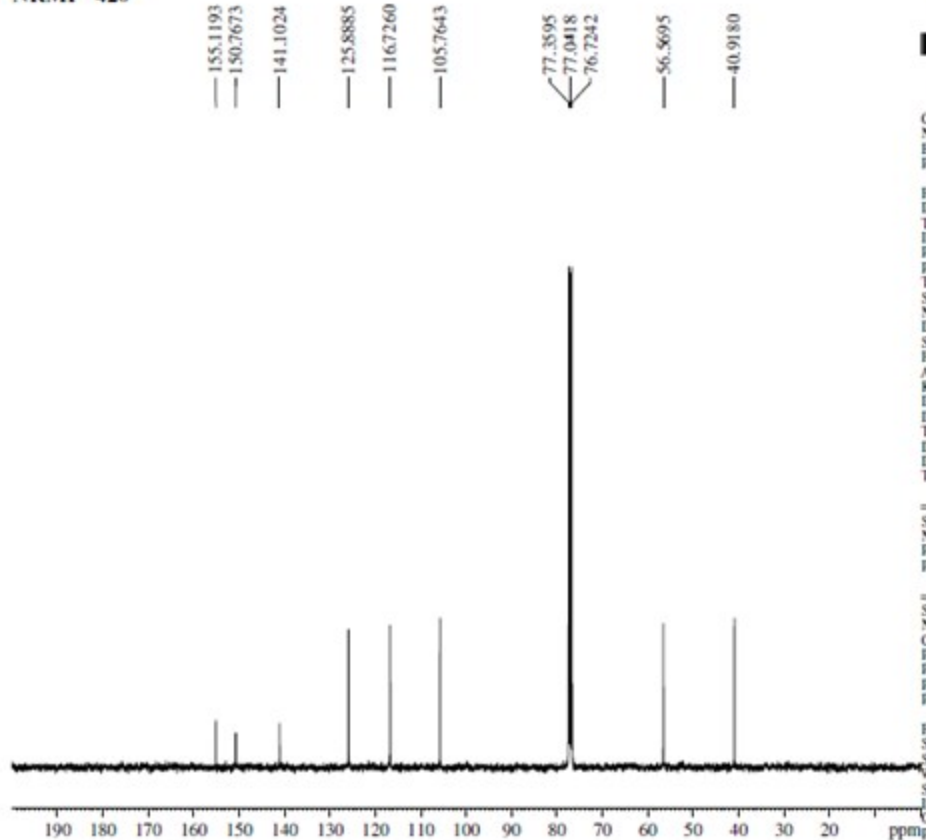
F2 - Acquisition Parameters  
 Date\_ 20160303  
 Time 16.47  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 100.41  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605336 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 21: <sup>1</sup>H NMR spectrum of 3j

NRMP-420



Current Data Parameters  
 NAME 07-Mar-AN-2016  
 EXPNO 350  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160307  
 Time 21.23  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

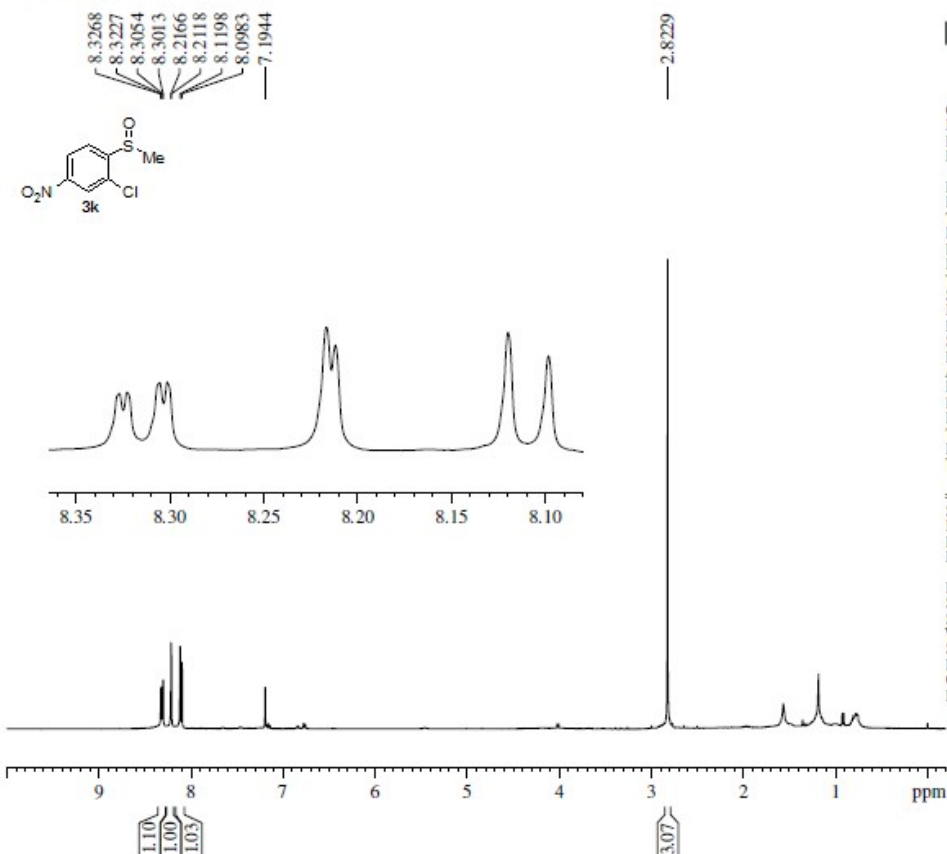
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 22: <sup>13</sup>C NMR spectrum of 3j

NRMP-455



Current Data Parameters  
 NAME 07-Jan-FN-2016  
 EXPNO 360  
 PROCNO 1

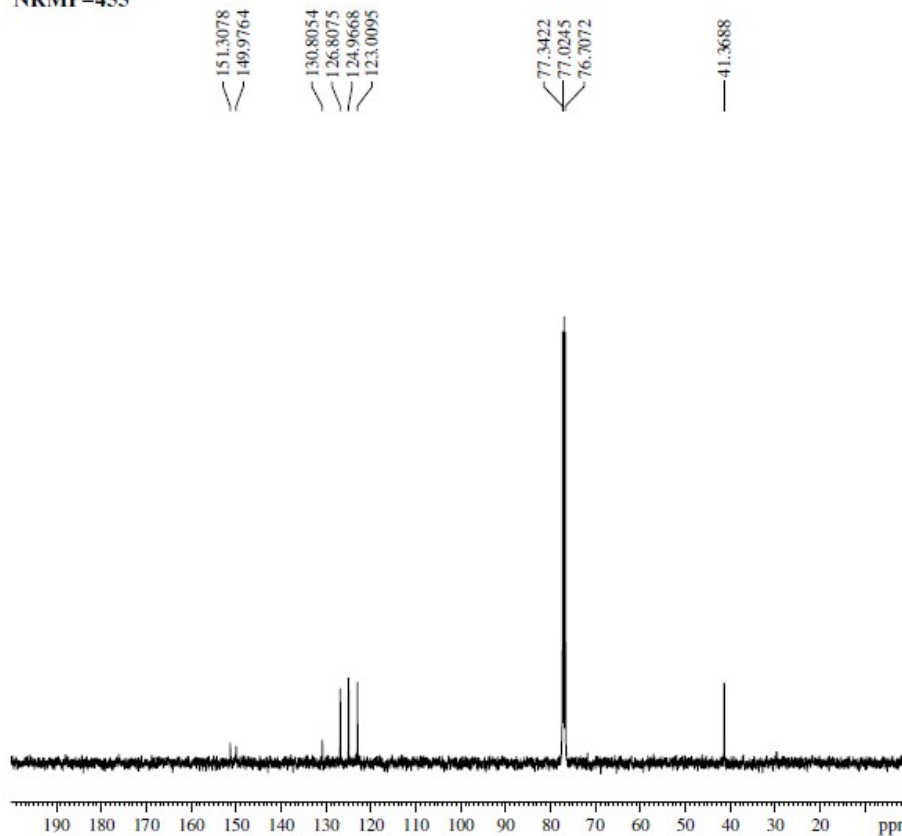
F2 - Acquisition Parameters  
 Date\_ 20160107  
 Time 15.35  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDC13  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 129.57  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 DI 1.00000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605362 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 23: <sup>1</sup>H NMR spectrum of 3k

NRMP-455



Current Data Parameters  
 NAME 11-Jan-AN-2016  
 EXPNO 330  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160112  
 Time 14.13  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 131  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.1 K  
 DI 2.00000000 sec  
 DI1 0.03000000 sec  
 TDO 1

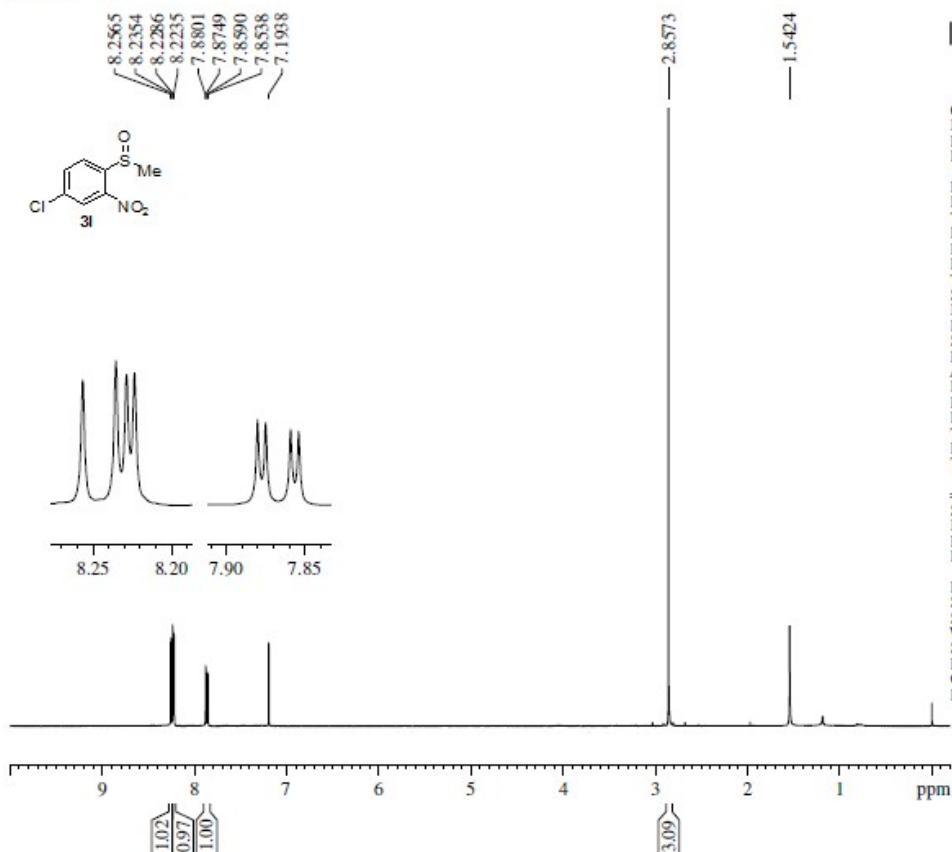
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 24: <sup>13</sup>C NMR spectrum of 3k

NRMP-409



Current Data Parameters  
 NAME 12-Oct-AN-2015  
 EXPNO 370  
 PROCNO 1

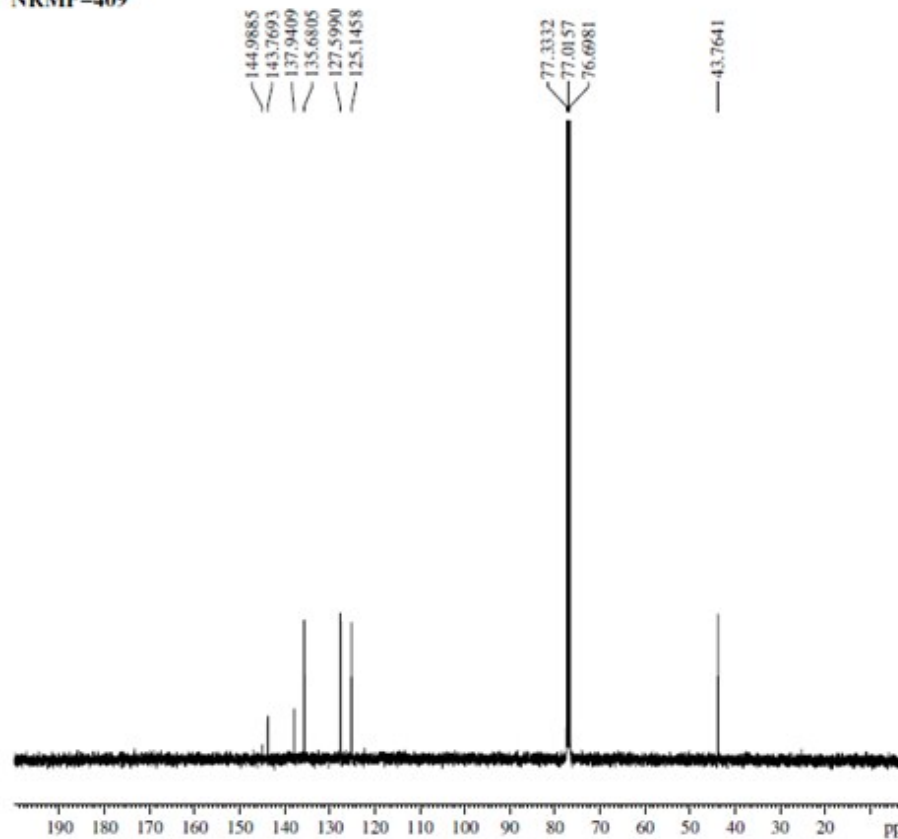
F2 - Acquisition Parameters  
 Date\_ 20151013  
 Time 2.35  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDC13  
 NS 8  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.0894465 sec  
 RG 179.93  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.0000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 12.85 usec  
 PLW1 13.1000038 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605358 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 25: <sup>1</sup>H NMR spectrum of 3l

NRMP-409



Current Data Parameters  
 NAME 14-Oct-AN-2015  
 EXPNO 340  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20151015  
 Time 14.46  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.0300000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.00 usec  
 PLW1 61.09999847 W

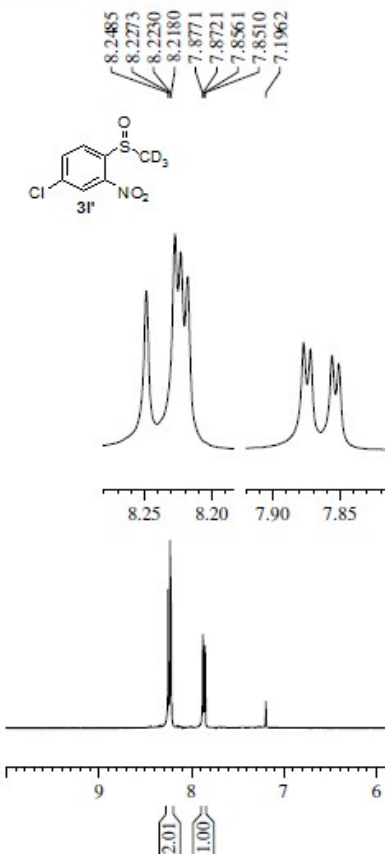
==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.1000038 W  
 PLW12 0.26705000 W  
 PLW13 0.21630999 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure 26: <sup>13</sup>C NMR spectrum of 3l



NRMP-509



Current Data Parameters  
 NAME 18-Apr-FN-2016  
 EXPNO 360  
 PROCNO 1

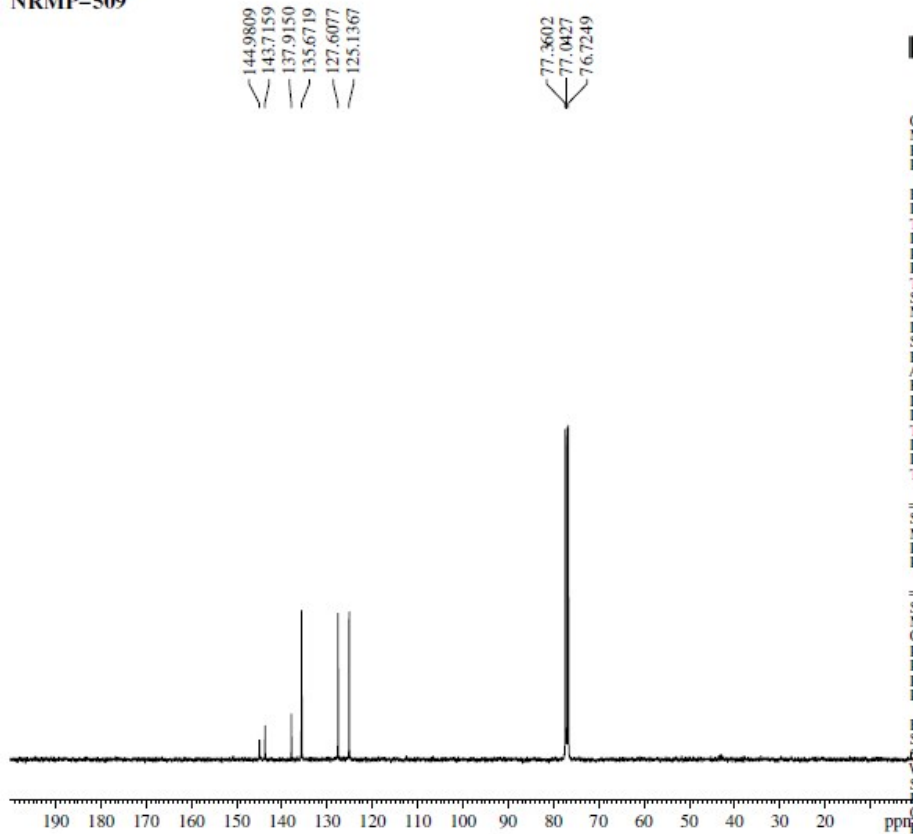
F2 - Acquisition Parameters  
 Date\_ 20160418  
 Time 16.32  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDC13  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 145.29  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.0000000 sec  
 TDX 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605354 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 27: <sup>1</sup>H NMR spectrum of 3I'

NRMP-509



Current Data Parameters  
 NAME 21-Apr-FN-2016  
 EXPNO 340  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160422  
 Time 2.57  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.03000000 sec  
 TDX 1

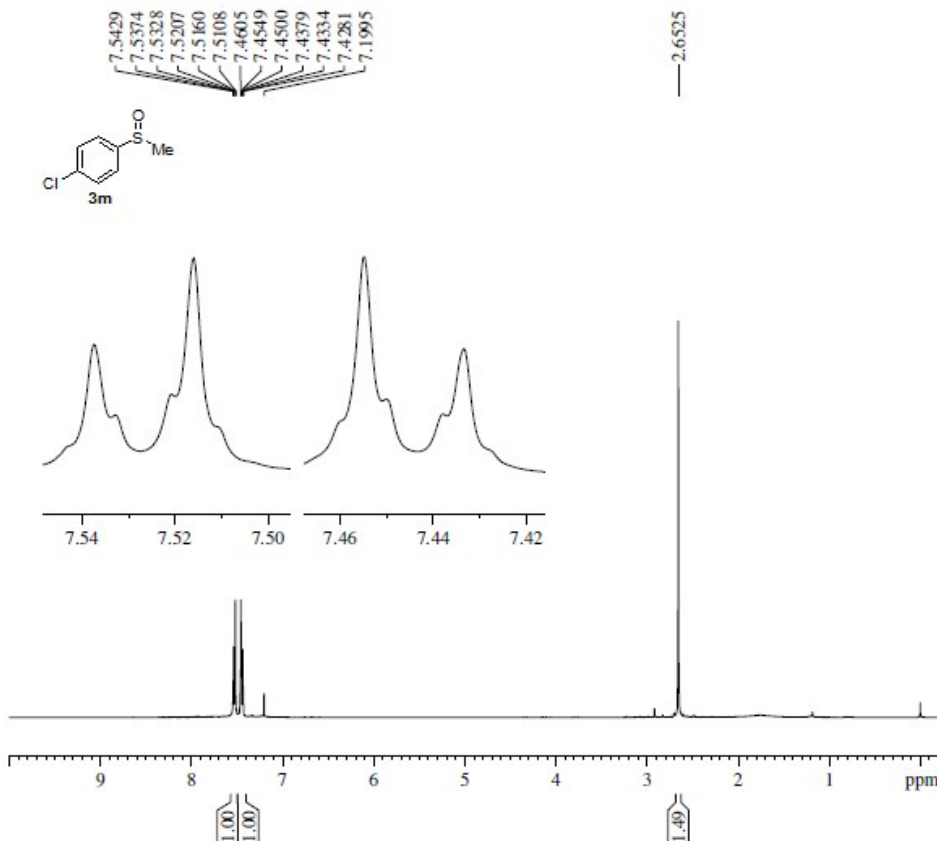
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CDP[RG]2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 28: <sup>13</sup>C NMR spectrum of 3I'

NRMP-415



Current Data Parameters  
 NAME 26-Oct-FN-2015  
 EXPNO 370  
 PROCNO 1

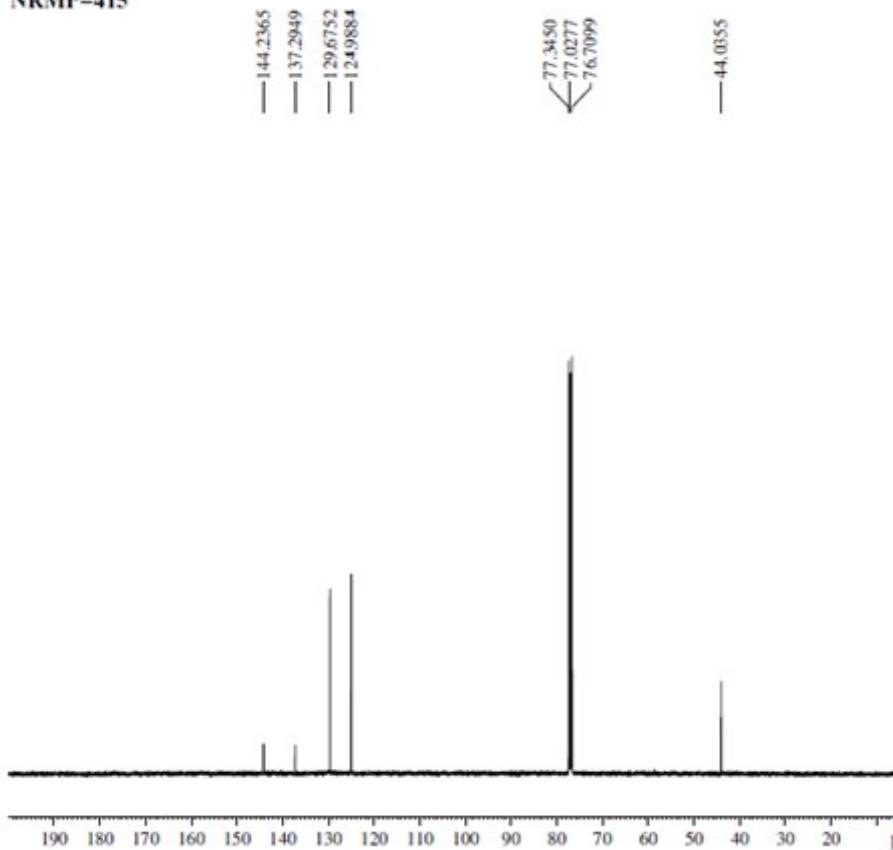
F2 - Acquisition Parameters  
 Date\_ 20151026  
 Time 17.02  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 A Q 3.4078720 sec  
 RG 114.26  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 12.85 usec  
 PLW1 13.10000038 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605335 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 29: <sup>1</sup>H NMR spectrum of 3m

NRMP-415



Current Data Parameters  
 NAME 04-Mar-FN-2016  
 EXPNO 340  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160306  
 Time 0.08  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 A Q 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

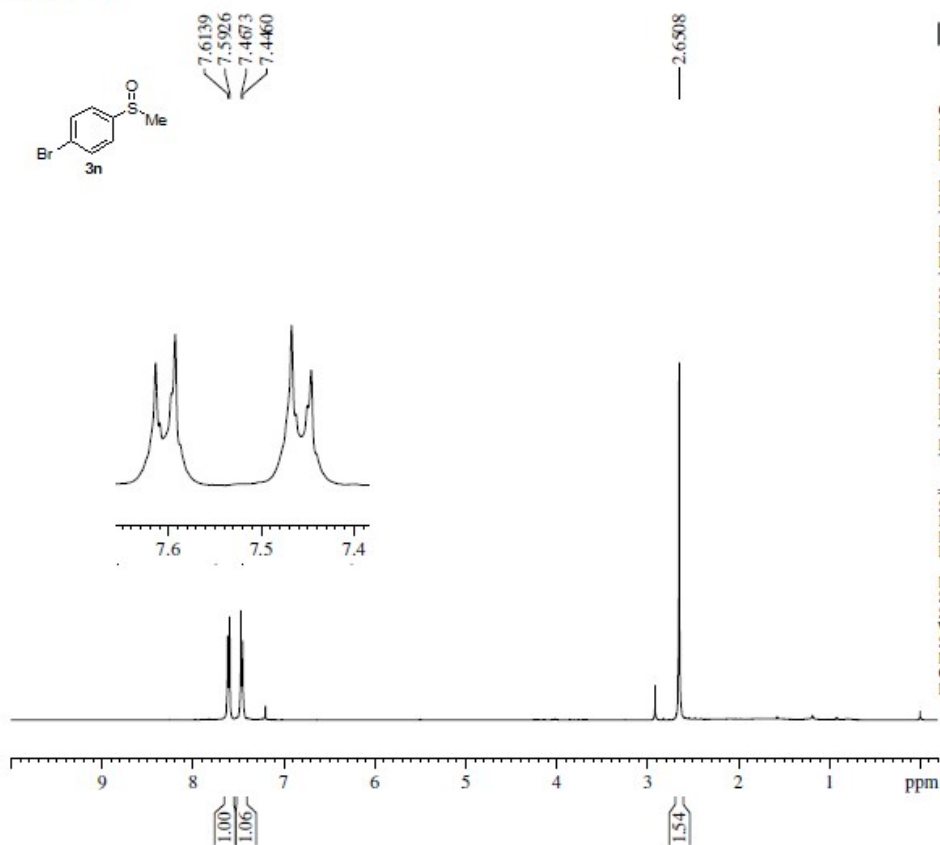
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure 30: <sup>13</sup>C NMR spectrum of 3m

NRMP-446



Current Data Parameters  
 NAME 17-Mar-AN-2016  
 EXPNO 340  
 PROCNO 1

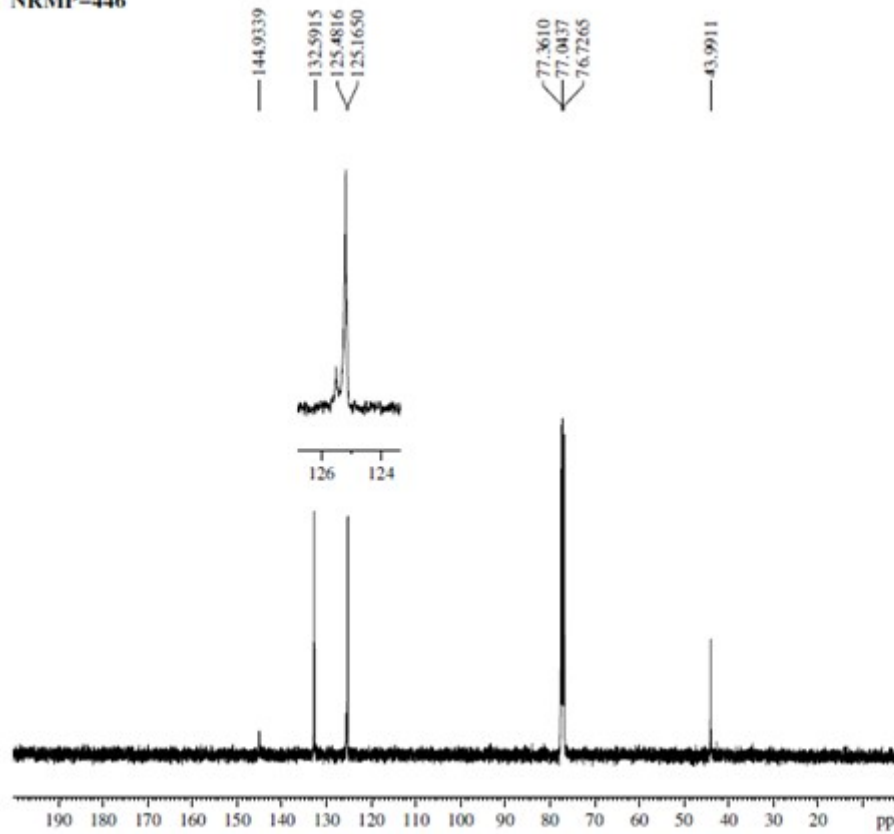
F2 - Acquisition Parameters  
 Date\_ 20160318  
 Time 0.08  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 114.26  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.0000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.0000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605338 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 31: <sup>1</sup>H NMR spectrum of 3n

NRMP-446



Current Data Parameters  
 NAME 18-Mar-AN-2016  
 EXPNO 350  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160319  
 Time 20.12  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.0300000 sec  
 TD0 1

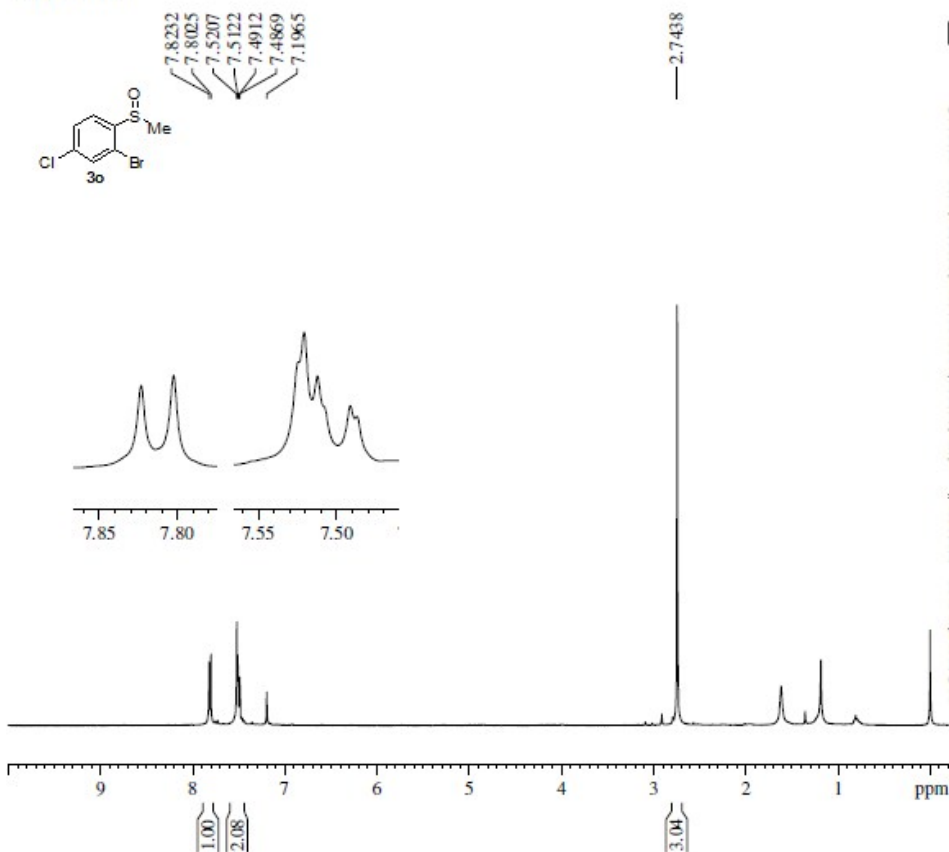
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.0000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.0000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure 32: <sup>13</sup>C NMR spectrum of 3n

NRMP-404



Current Data Parameters  
 NAME 09-Oct-FN-2015  
 EXPNO 410  
 PROCNO 1

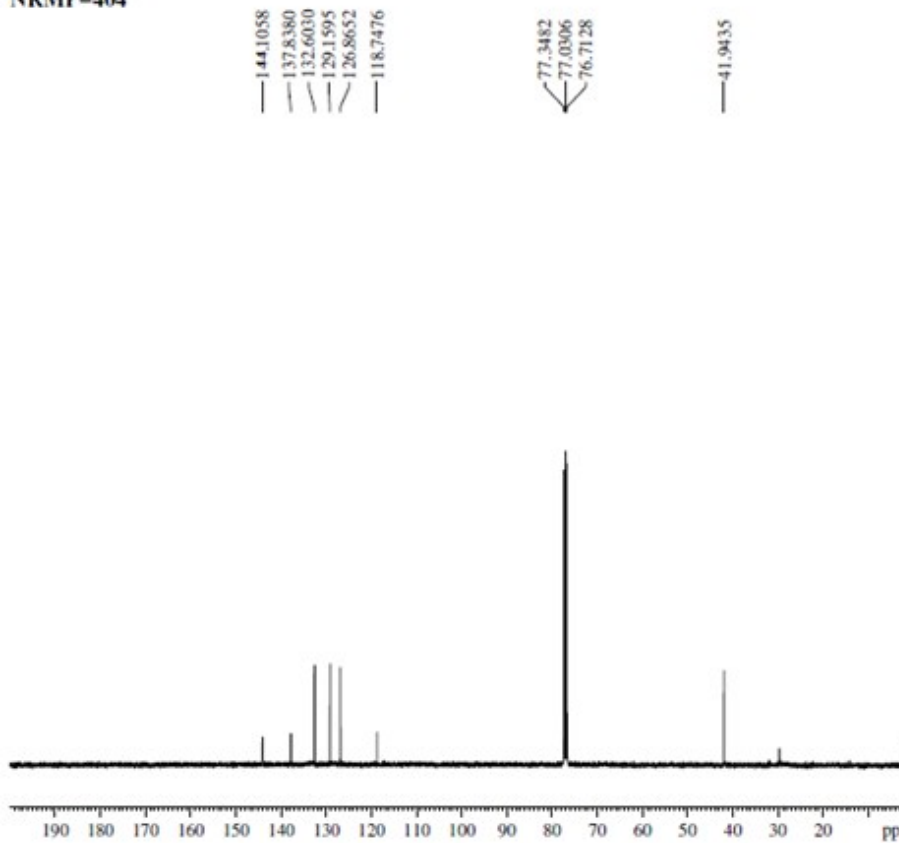
F2 - Acquisition Parameters  
 Date\_ 20151009  
 Time 17.42  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 129.57  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.0000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605346 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 33: <sup>1</sup>H NMR spectrum of 3o

NRMP-404



Current Data Parameters  
 NAME 14-Oct-AN-2015  
 EXPNO 330  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20151015  
 Time 14.12  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

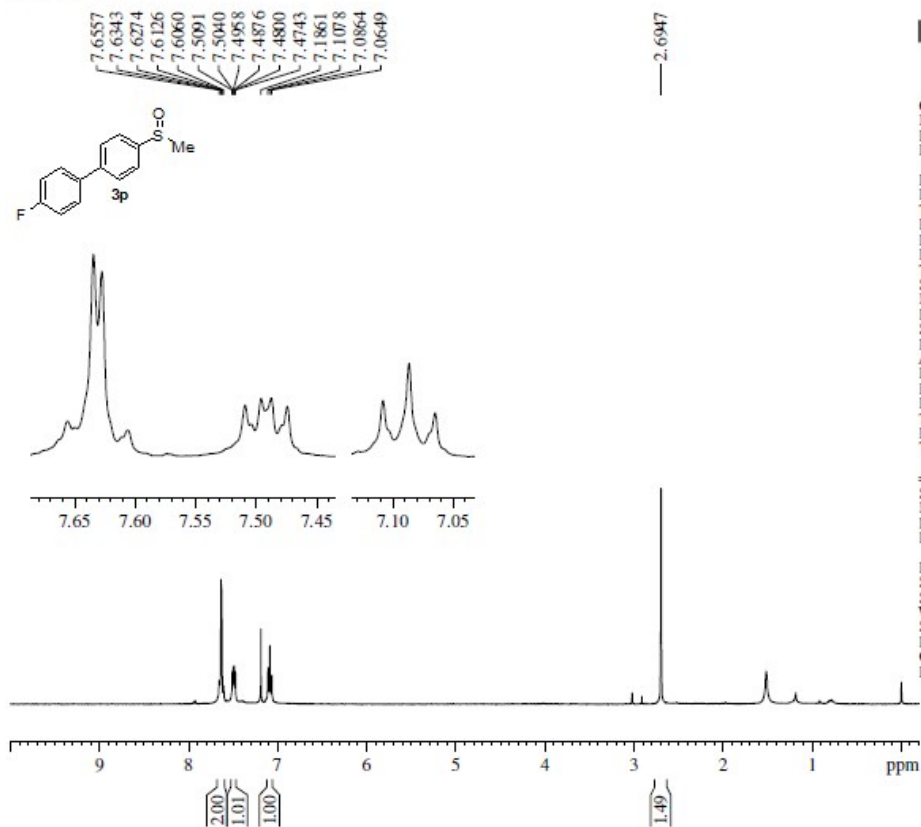
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.00 usec  
 PLW1 61.09999847 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.10000038 W  
 PLW12 0.26705000 W  
 PLW13 0.21630999 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure 34: <sup>13</sup>C NMR spectrum of 3o

NRMP-470



Current Data Parameters  
 NAME 25-Jan-AN-2016  
 EXPNO 310  
 PROCNO 1

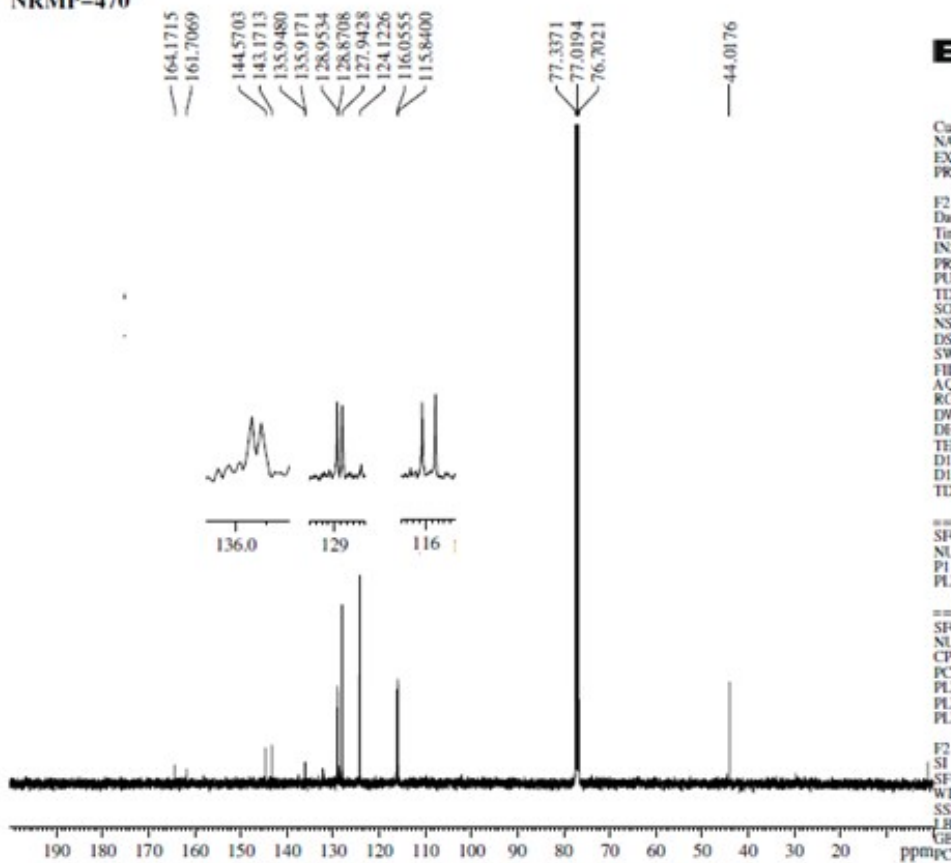
F2 - Acquisition Parameters  
 Date\_ 20160125  
 Time 19.50  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 159.22  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605392 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 35: <sup>1</sup>H NMR spectrum of 3p

NRMP-470



Current Data Parameters  
 NAME 04-Feb-FN-2016  
 EXPNO 340  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160205  
 Time 11.04  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

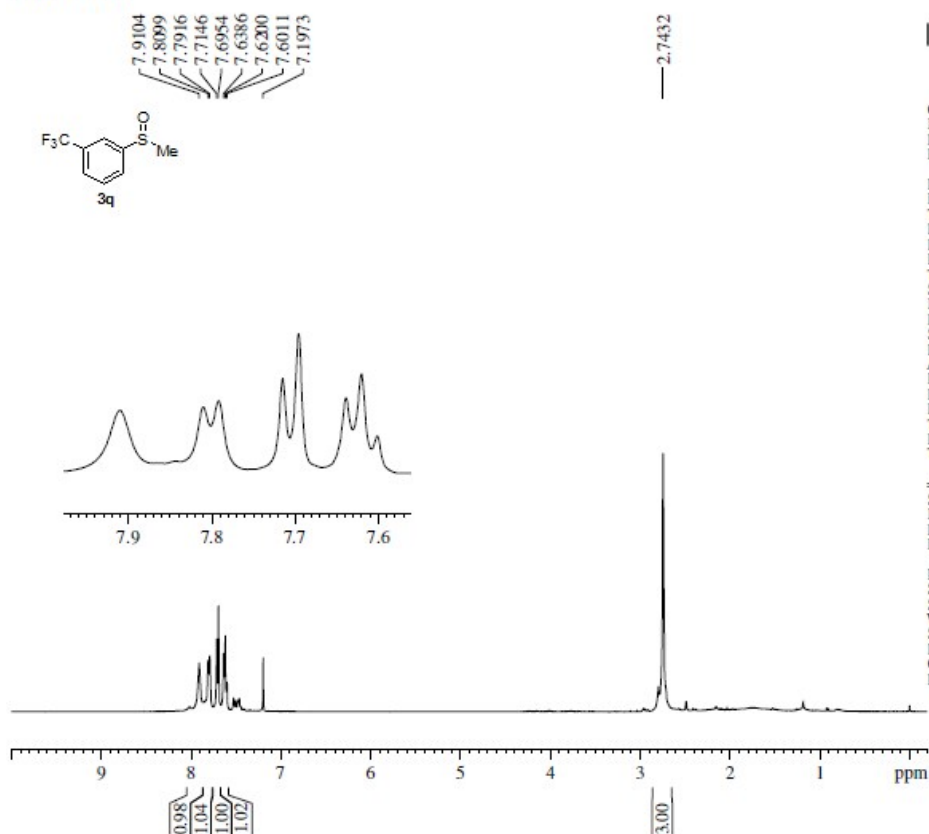
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure 36: <sup>13</sup>C NMR spectrum of 3p

NRMP-459



Current Data Parameters  
 NAME 10-Mar-FN-2016  
 EXPNO 370  
 PROCNO 1

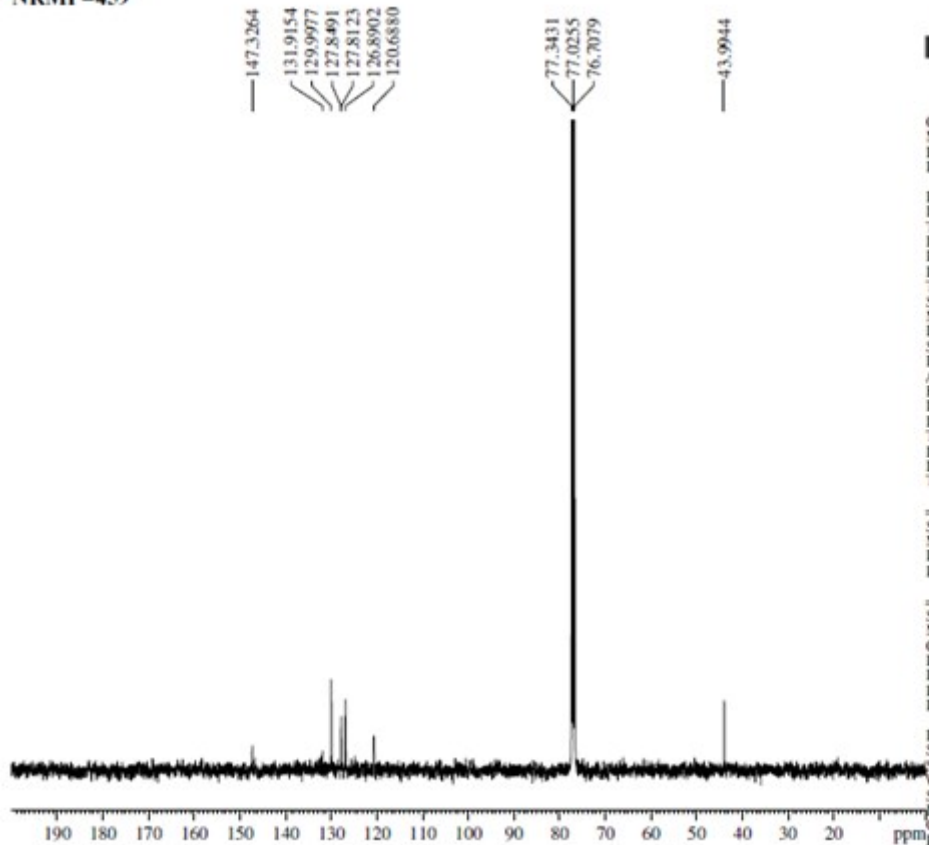
F2 - Acquisition Parameters  
 Date\_ 20160310  
 Time 15.55  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 114.26  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605350 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 37: <sup>1</sup>H NMR spectrum of 3q

NRMP-459



Current Data Parameters  
 NAME 14-Mar-AN-2016  
 EXPNO 320  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160315  
 Time 14.17  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

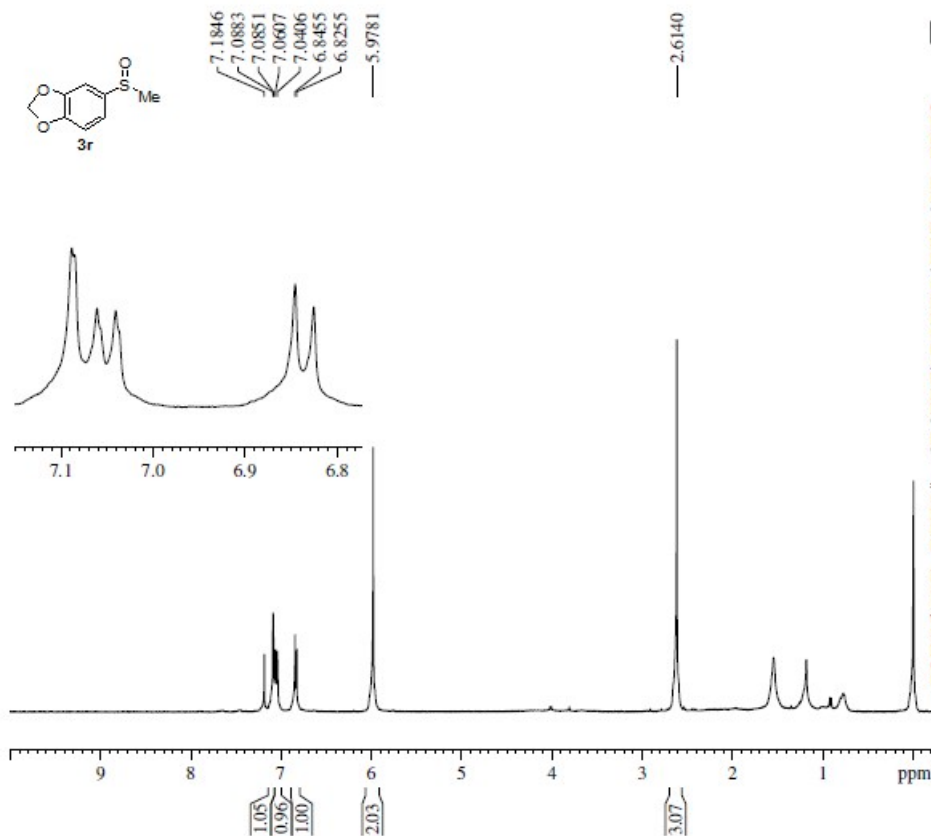
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 38: <sup>13</sup>C NMR spectrum of 3q

NRMP-494



Current Data Parameters  
 NAME 21-Mar-FN-2016  
 EXPNO 420  
 PROCNO 1

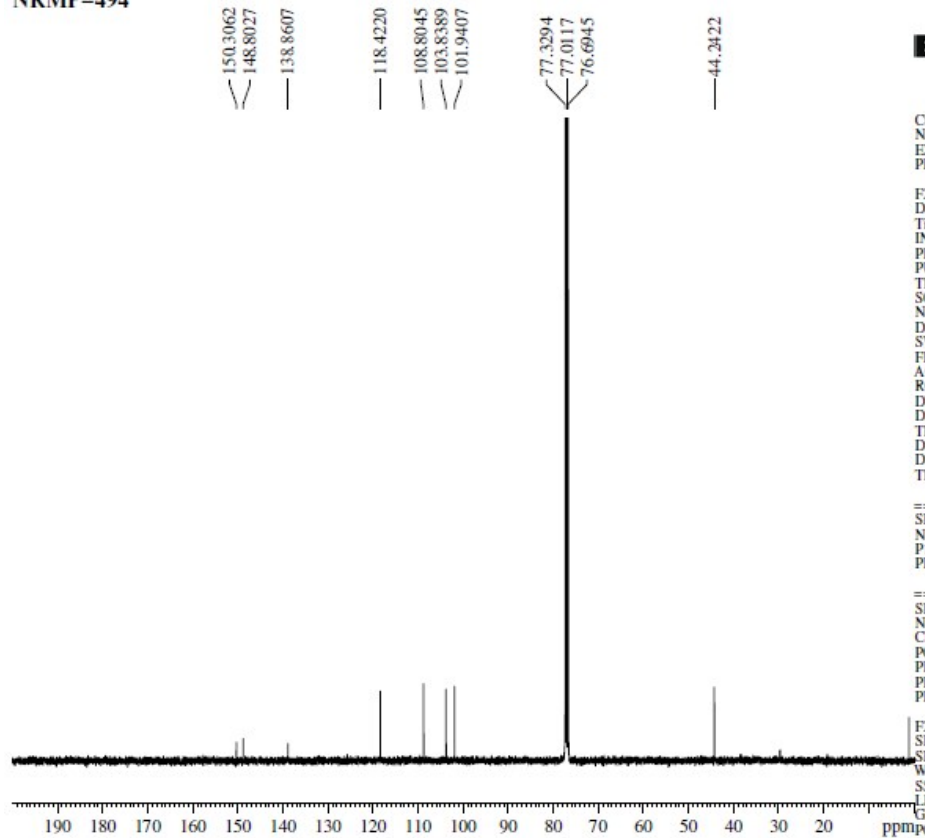
F2 - Acquisition Parameters  
 Date\_ 20160321  
 Time 16.59  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 201.48  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605397 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 39: <sup>1</sup>H NMR spectrum of 3r

NRMP-494



Current Data Parameters  
 NAME 24-Mar-FN-2016  
 EXPNO 310  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160324  
 Time 20.57  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 1024  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

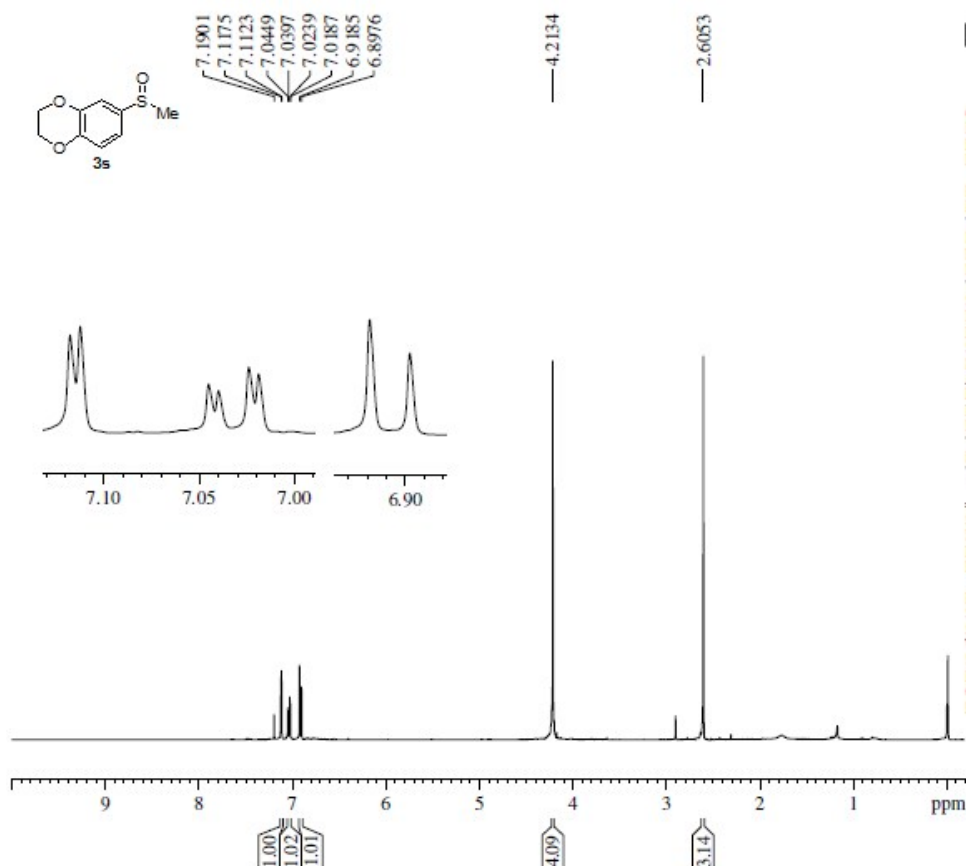
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure 40: <sup>13</sup>C NMR spectrum of 3r

NRMP-461



Current Data Parameters  
 NAME 29-Feb-FN-2016  
 EXPNO 360  
 PROCNO 1

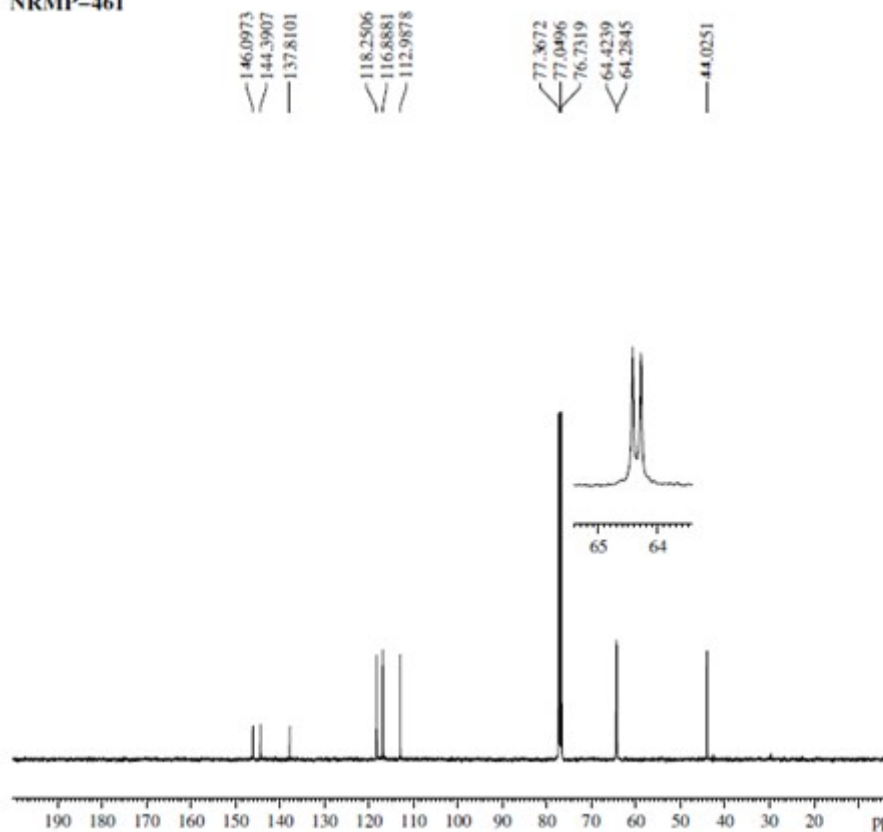
F2 - Acquisition Parameters  
 Date\_ 20160229  
 Time 16.13  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 89.7  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605380 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 41: <sup>1</sup>H NMR spectrum of 3s

NRMP-461



Current Data Parameters  
 NAME 02-Mar-FN-2016  
 EXPNO 330  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160302  
 Time 20.29  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

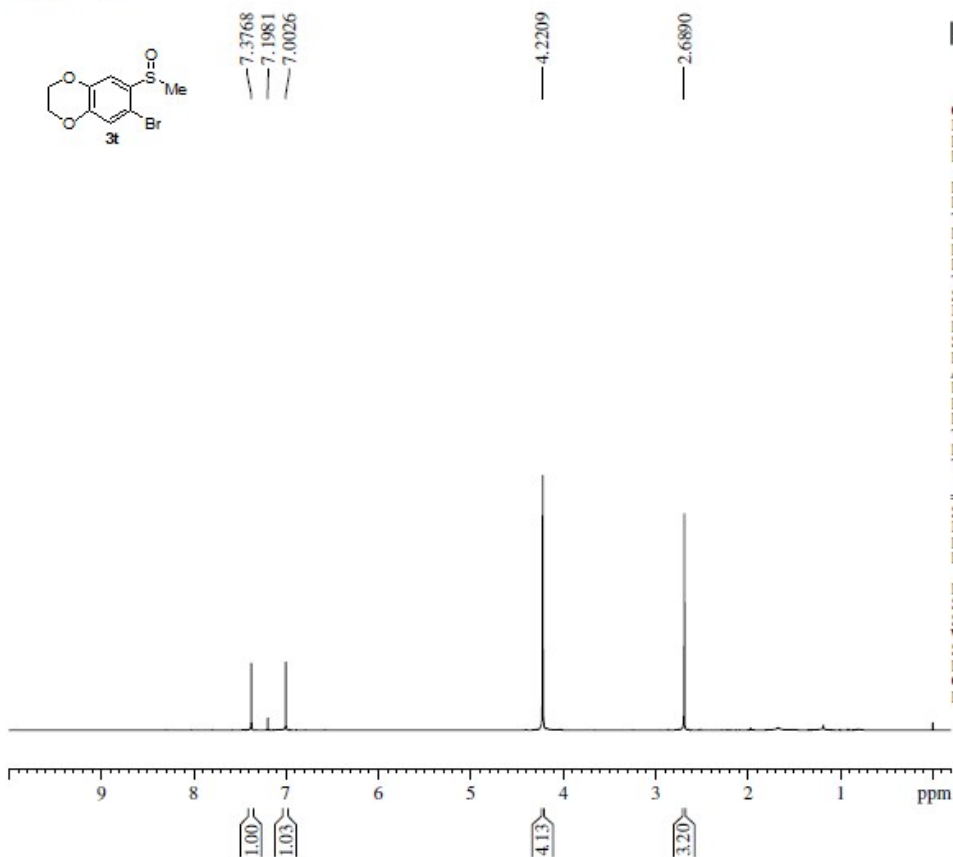
==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 FCFD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 42: <sup>13</sup>C NMR spectrum of 3s



NRMP-480



Current Data Parameters  
 NAME 05-Feb-FN-2016  
 EXPNO 380  
 PROCNO 1

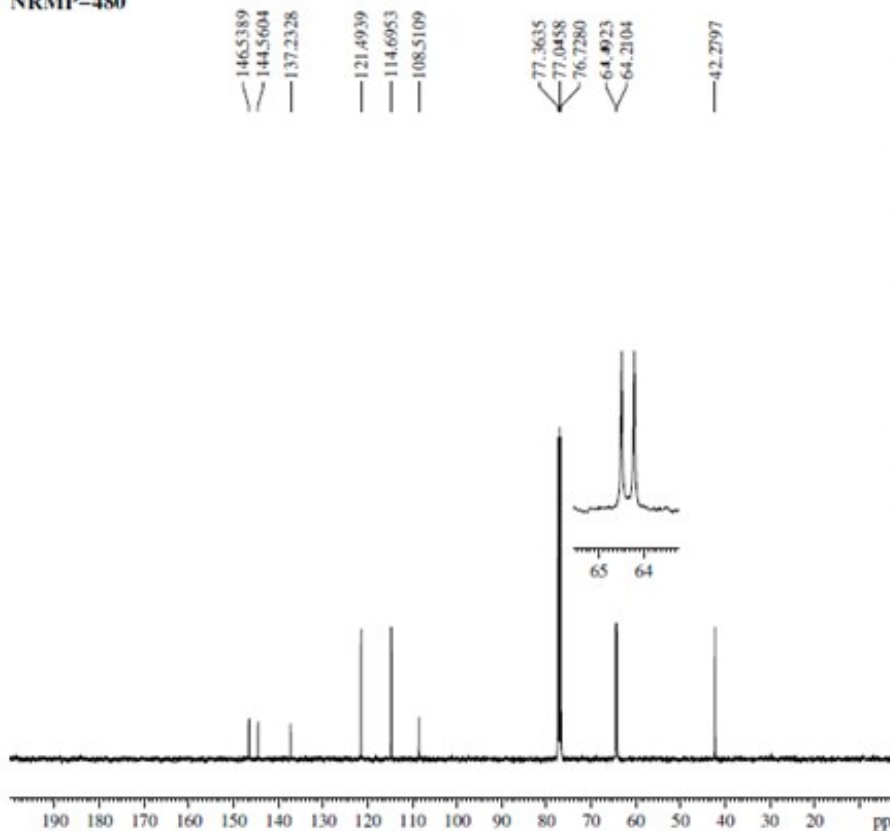
F2 - Acquisition Parameters  
 Date\_ 20160205  
 Time 16.51  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 100.41  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605347 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 43: <sup>1</sup>H NMR spectrum of 3t

NRMP-480



Current Data Parameters  
 NAME 08-Feb-FN-2016  
 EXPNO 330  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160209  
 Time 14.09  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

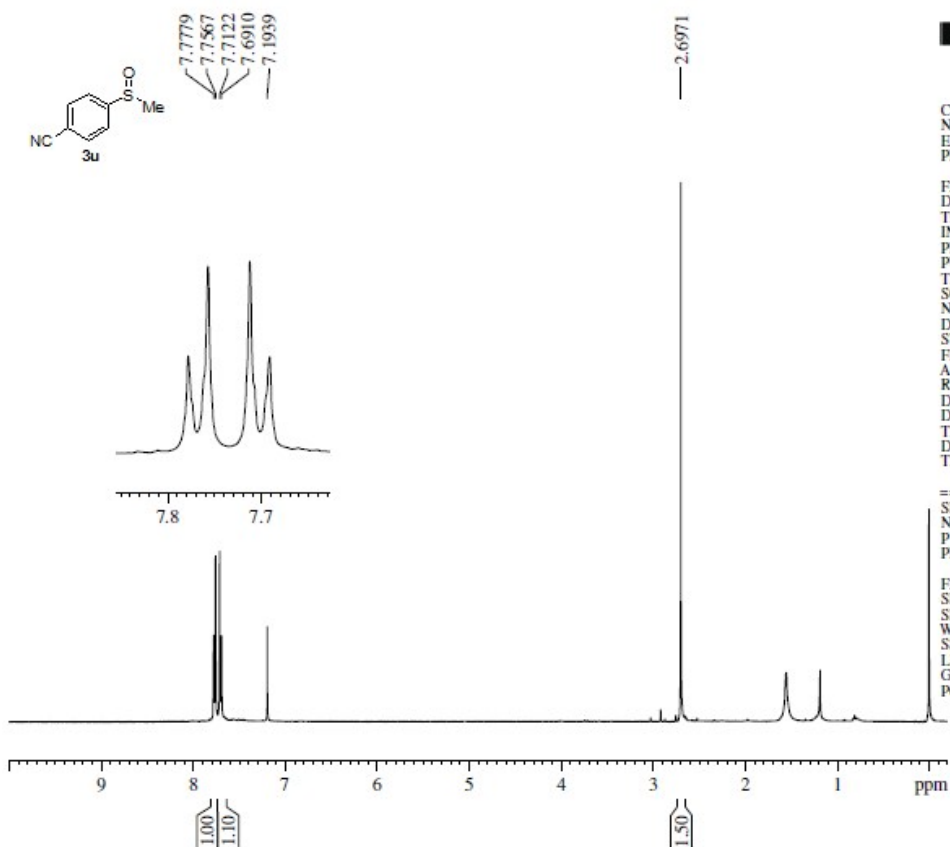
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPGPRG[2] waltz16  
 P1[2] 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 44: <sup>13</sup>C NMR spectrum of 3t

NRMP-418



Current Data Parameters  
 NAME 04-Nov-AN-2015  
 EXPNO 390  
 PROCNO 1

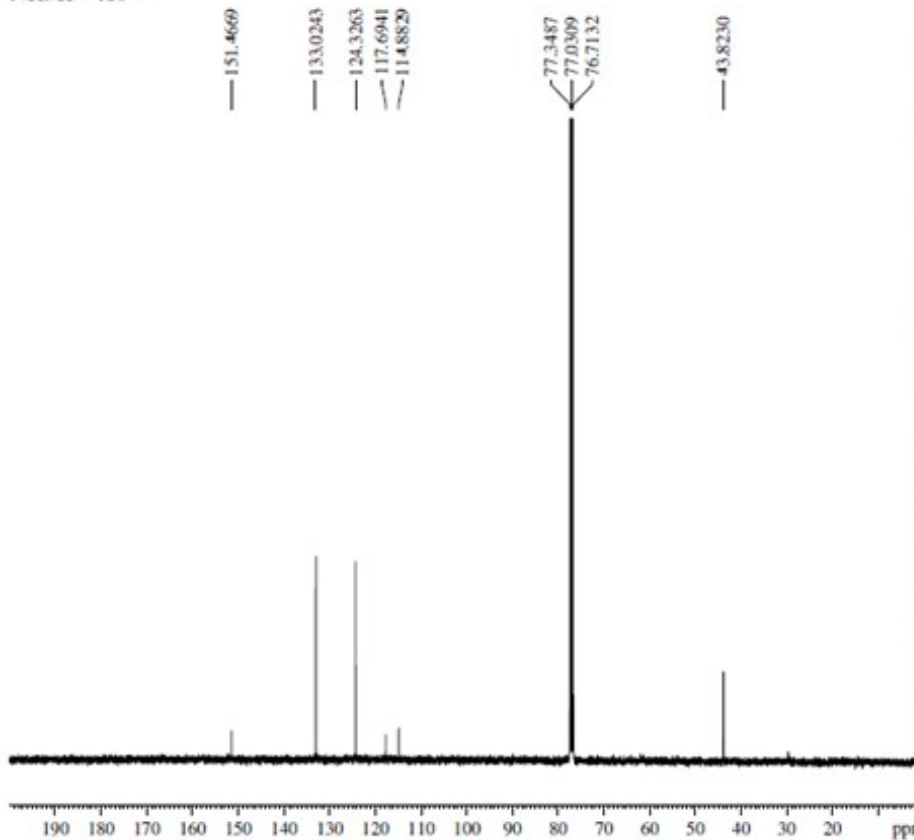
F2 - Acquisition Parameters  
 Date\_ 20151105  
 Time 2.05  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.0894465 sec  
 RG 159.22  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 12.85 usec  
 PLW1 13.10000038 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605355 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 45: <sup>1</sup>H NMR spectrum of 3u

NRMP-418



Current Data Parameters  
 NAME 19-Nov-AN-2015  
 EXPNO 340  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20151120  
 Time 10.05  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 299.3 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

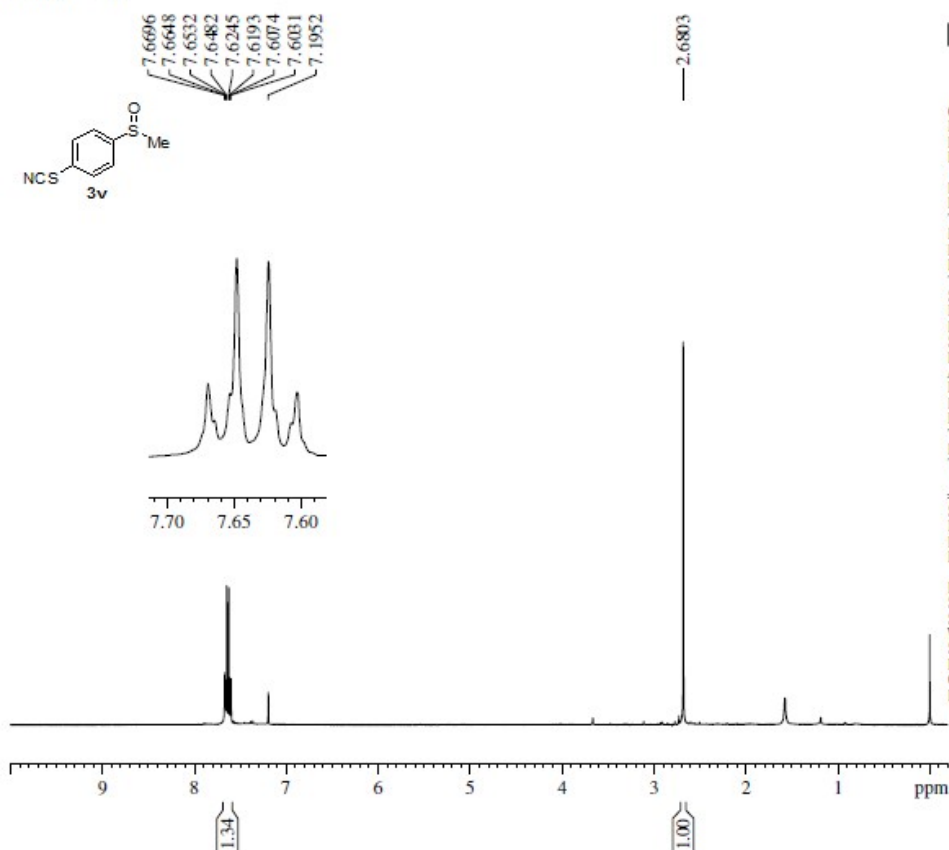
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CDPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Figure 46: <sup>13</sup>C NMR spectrum of 3u

NRMP-507



Current Data Parameters  
 NAME 13-Apr-FN-2016  
 EXPNO 370  
 PROCNO 1

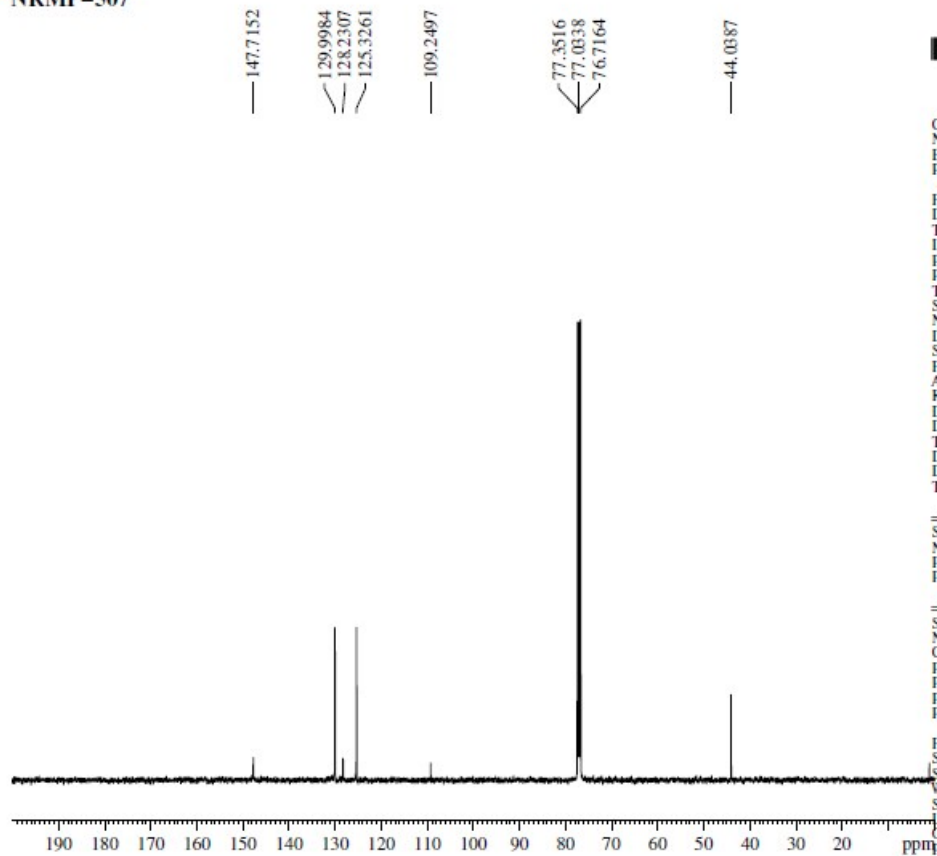
F2 - Acquisition Parameters  
 Date\_ 20160413  
 Time 18.43  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.0894465 sec  
 RG 145.29  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605357 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 47: <sup>1</sup>H NMR spectrum of 3v

NRMP-507



Current Data Parameters  
 NAME 19-Apr-FN-2016  
 EXPNO 320  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160419  
 Time 19.59  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

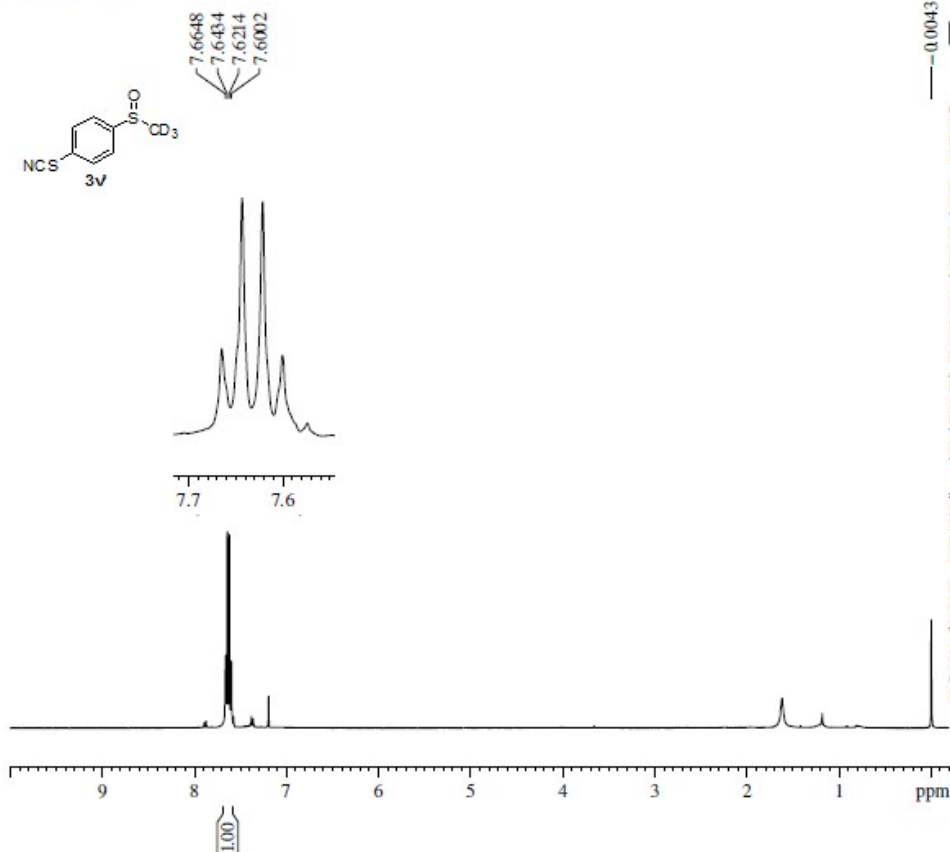
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 48: <sup>13</sup>C NMR spectrum of 3v

NRMP-511



Current Data Parameters  
 NAME 18-Apr-FN-2016  
 EXPNO 380  
 PROCNO 1

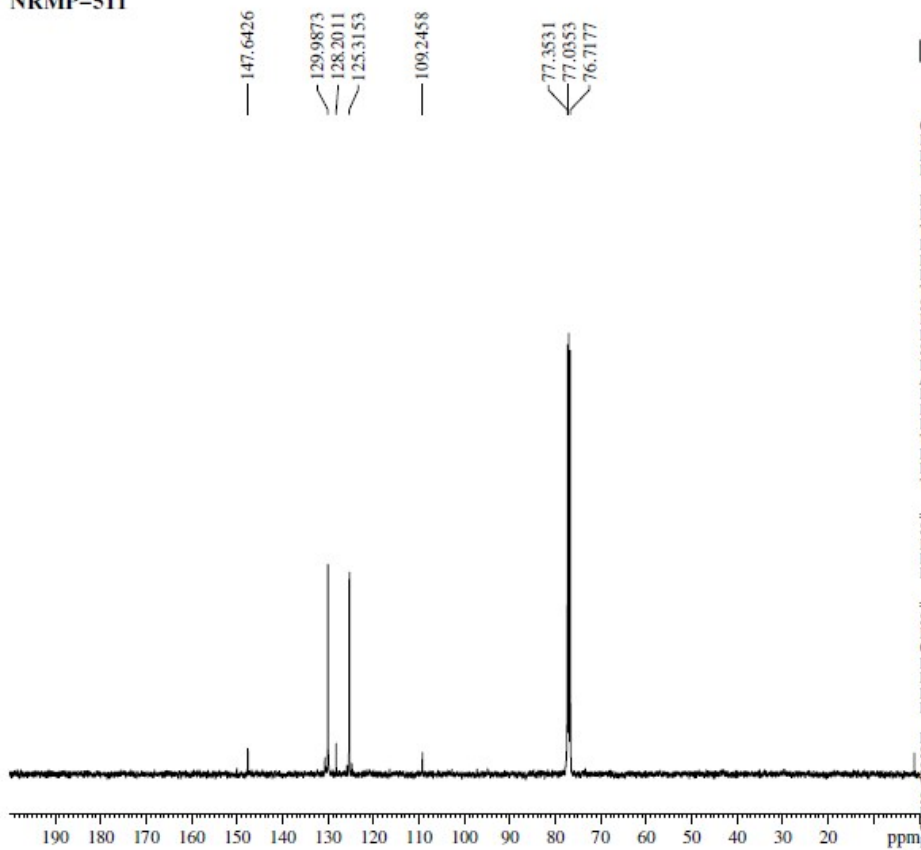
F2 - Acquisition Parameters  
 Date\_ 20160418  
 Time 16.41  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 145.29  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605361 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 49: <sup>1</sup>H NMR spectrum of 3v'

NRMP-511



Current Data Parameters  
 NAME 21-Apr-FN-2016  
 EXPNO 350  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160422  
 Time 3.30  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

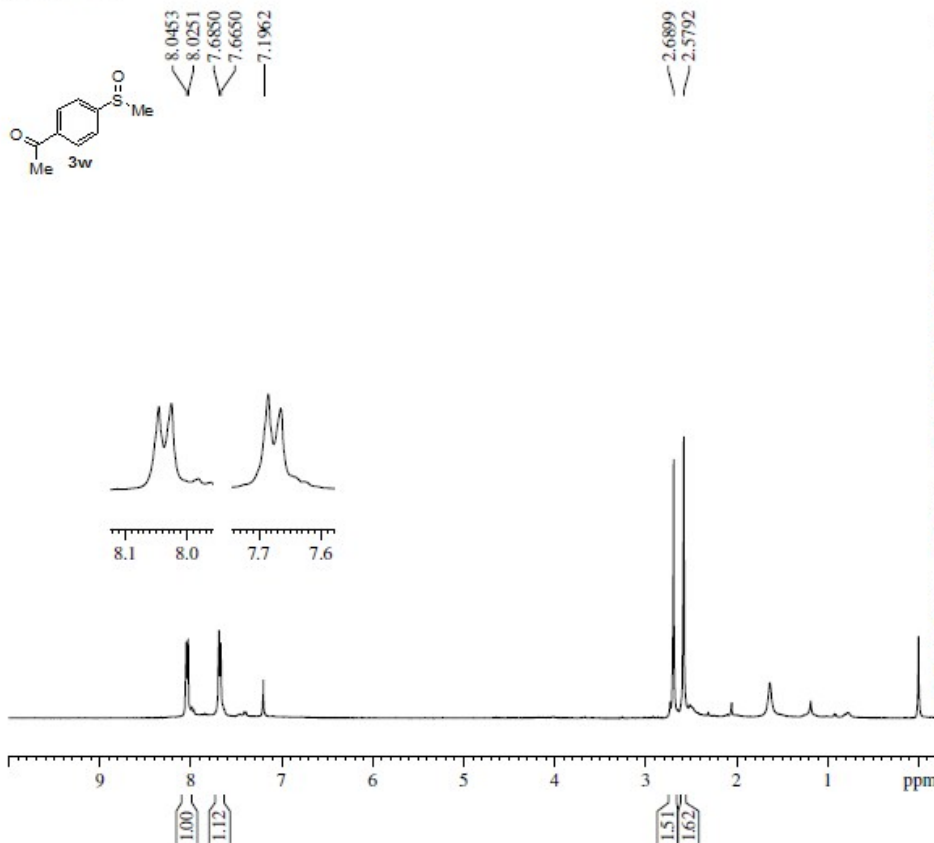
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204391 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 50: <sup>13</sup>C NMR spectrum of 3v'

NRMP-419



Current Data Parameters  
 NAME 14-Mar-FN-2016  
 EXPNO 340  
 PROCNO 1

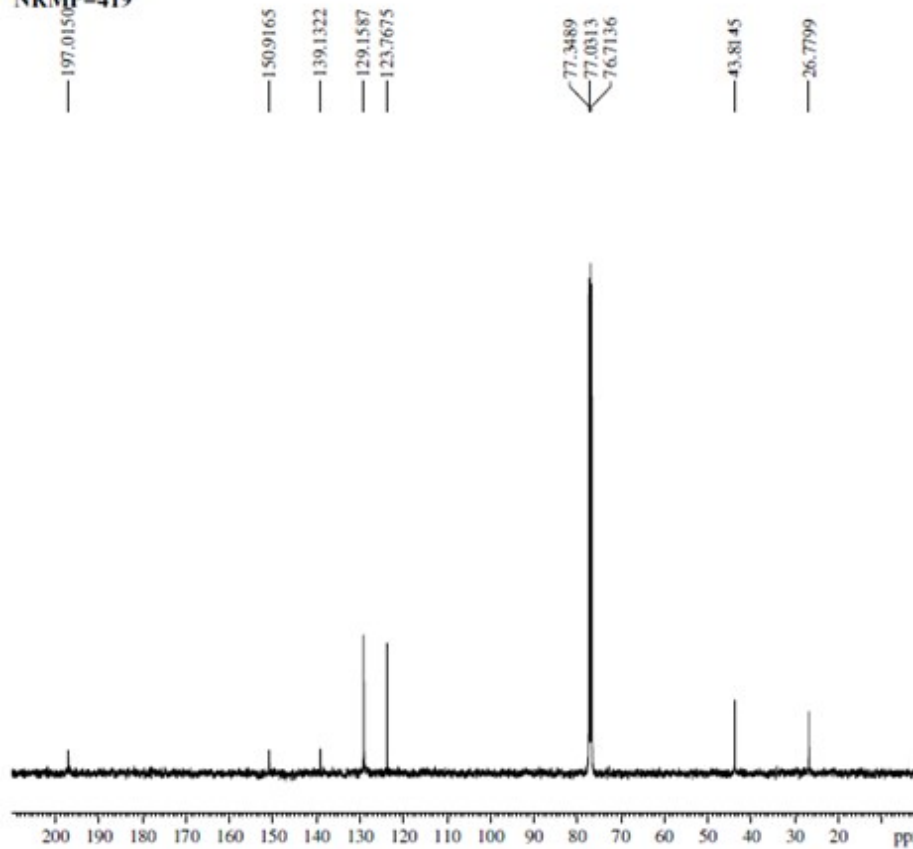
F2 - Acquisition Parameters  
 Date\_ 20160314  
 Time 19.16  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 114.26  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605350 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 51: <sup>1</sup>H NMR spectrum of 3w

NRMP-419



Current Data Parameters  
 NAME 16-Mar-AN-2016  
 EXPNO 360  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160317  
 Time 21.58  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

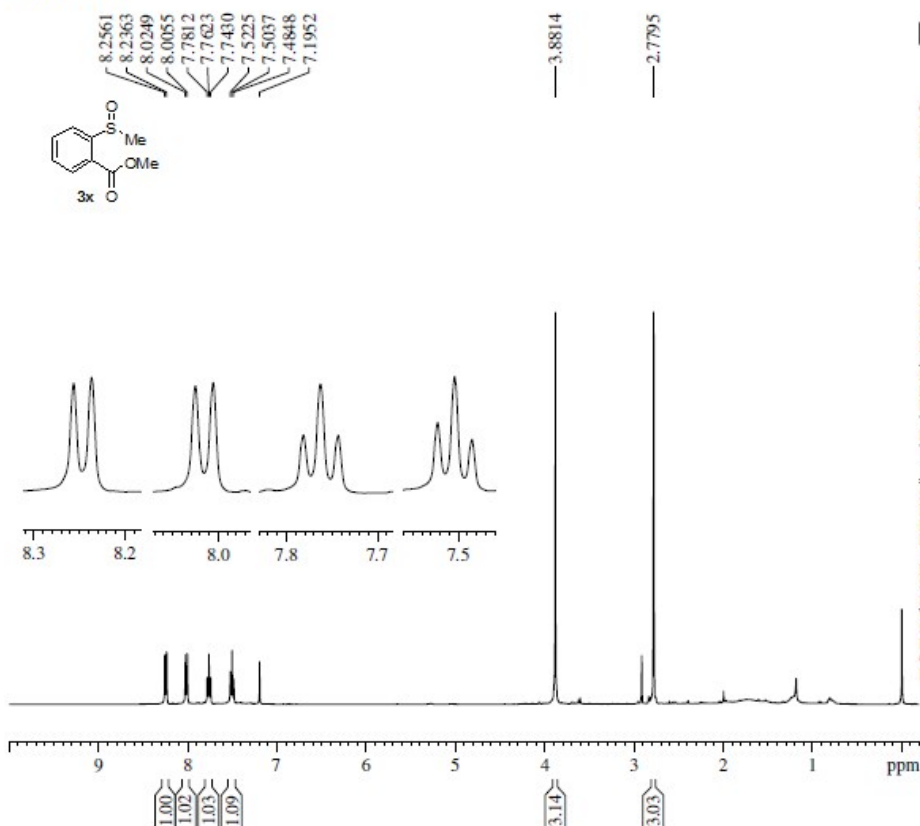
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.228651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 52: <sup>13</sup>C NMR spectrum of 3w

NRMP-417



Current Data Parameters  
 NAME 11-Mar-FN-2016  
 EXPNO 360  
 PROCNO 1

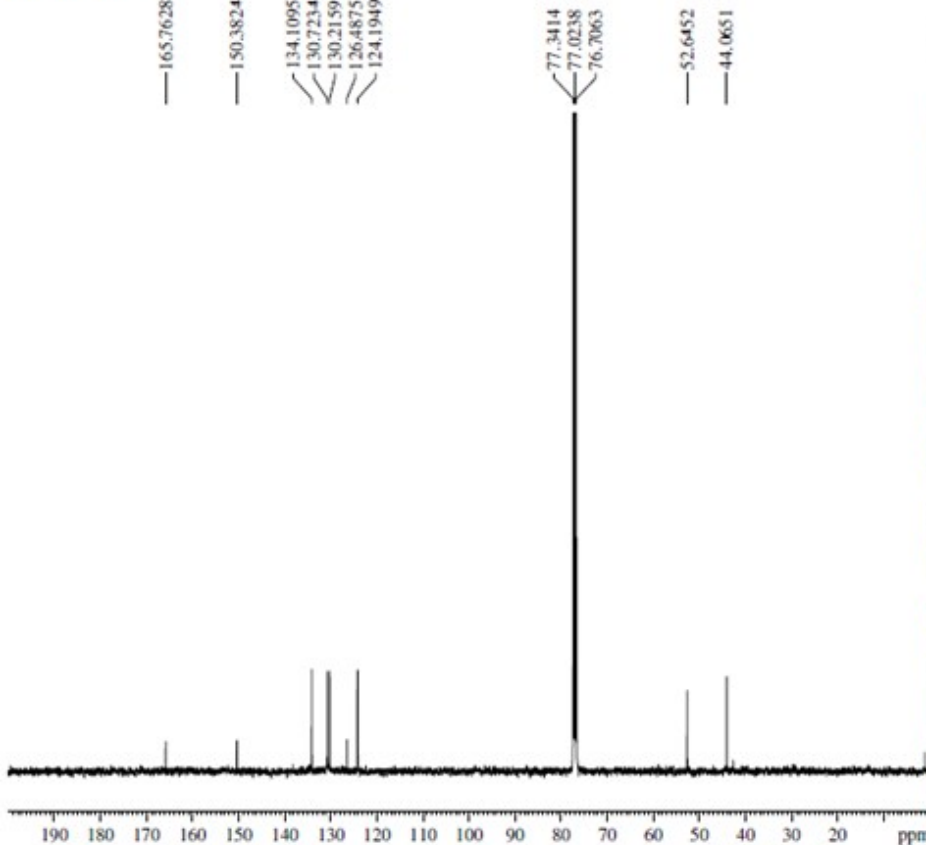
F2 - Acquisition Parameters  
 Date\_ 20160311  
 Time 16.37  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 114.26  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605360 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.00

Figure 53: <sup>1</sup>H NMR spectrum of 3x

NRMP-417



Current Data Parameters  
 NAME 14-Mar-AN-2016  
 EXPNO 310  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160315  
 Time 14.08  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

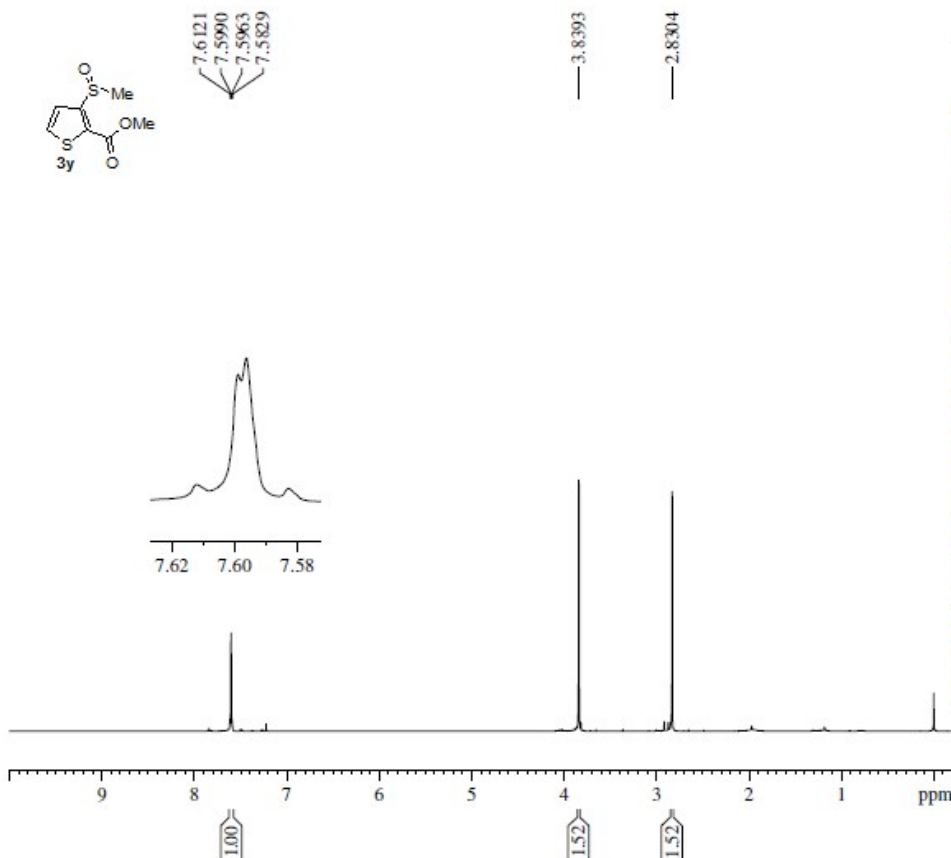
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG2 waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 54: <sup>13</sup>C NMR spectrum of 3x

NRMP-471



Current Data Parameters  
 NAME 29-Feb-AN-2016  
 EXPNO 310  
 PROCNO 1

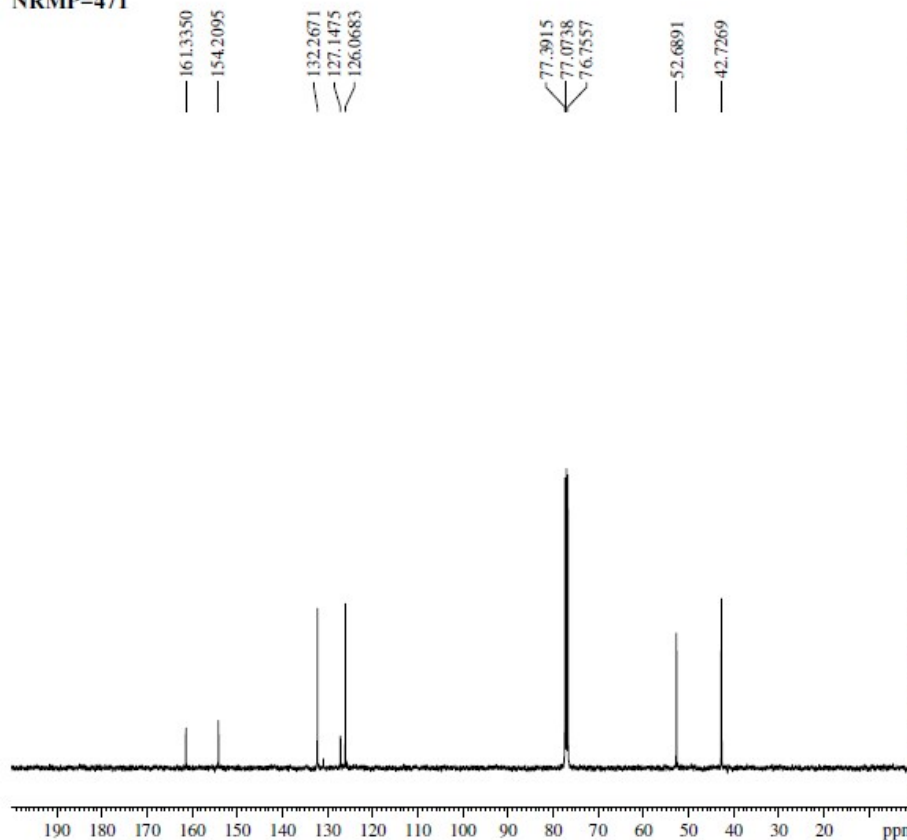
F2 - Acquisition Parameters  
 Date\_ 20160301  
 Time 5.07  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDC13  
 NS 8  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.122266 Hz  
 AQ 4.0894465 sec  
 RG 80.54  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.0000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605265 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 55: <sup>1</sup>H NMR spectrum of 3y

NRMP-471



Current Data Parameters  
 NAME 25-Feb-FN-2016  
 EXPNO 330  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160225  
 Time 18.49  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.03000000 sec  
 TDO 1

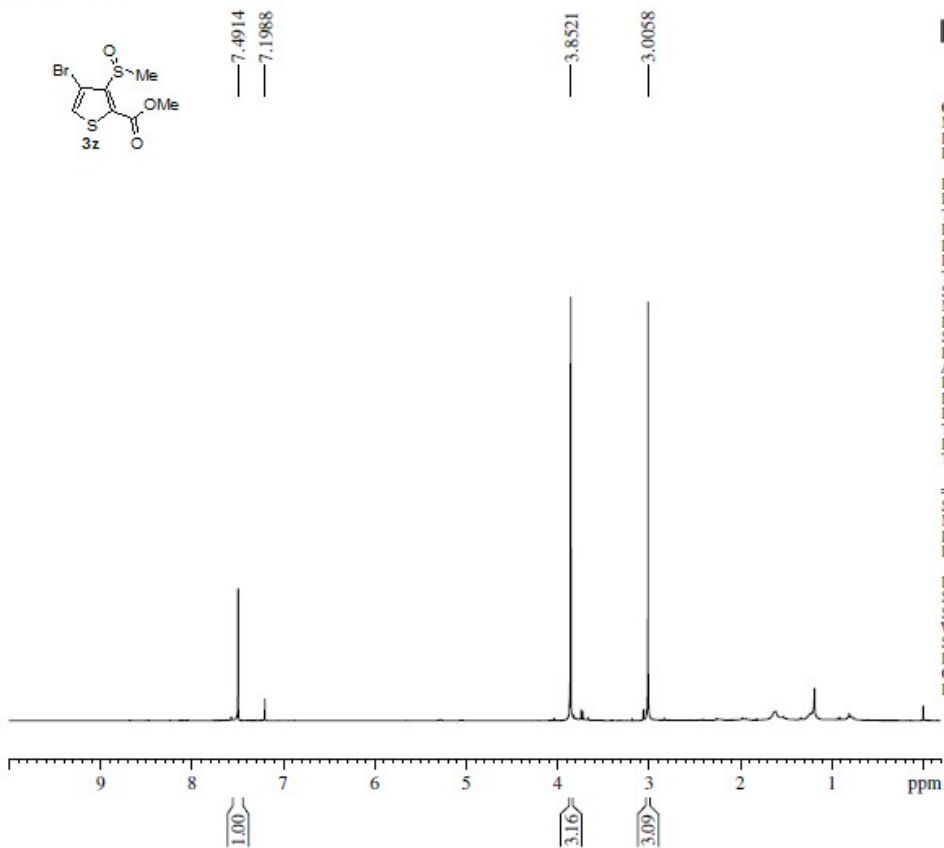
==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.40

Figure 56: <sup>13</sup>C NMR spectrum of 3y

NRMP-500



Current Data Parameters  
 NAME 04-Apr-FN-2016  
 EXPNO 480  
 PROCNO 1

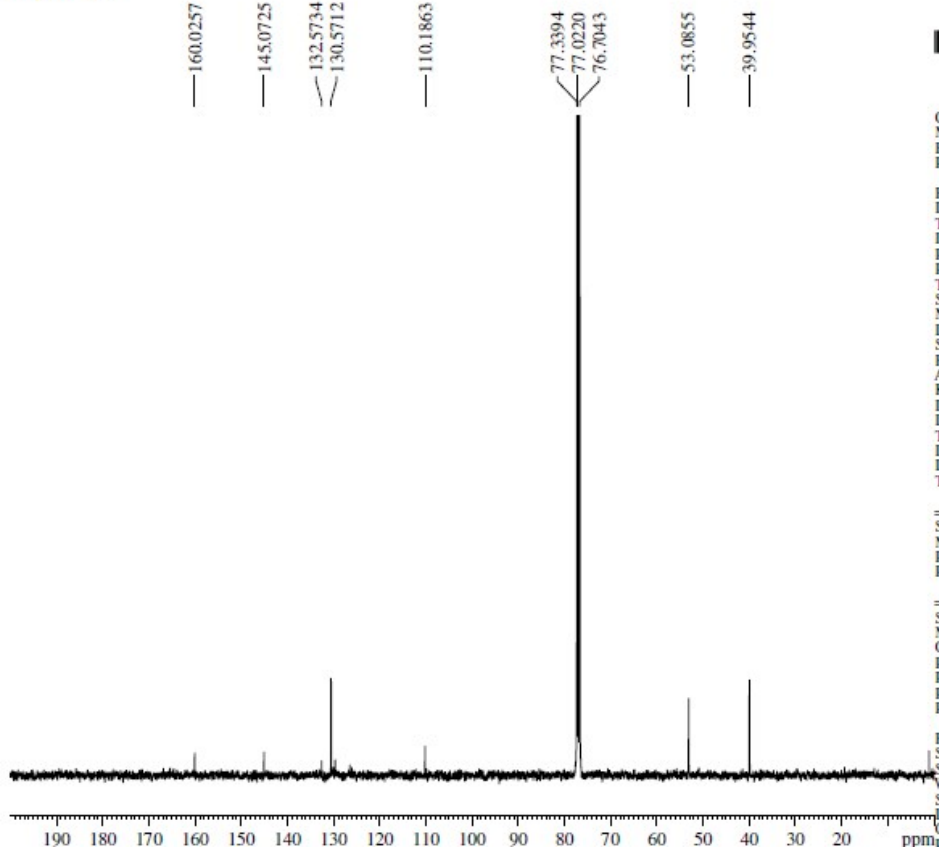
F2 - Acquisition Parameters  
 Date\_ 20160404  
 Time 17.07  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDC13  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 114.26  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605341 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 57: <sup>1</sup>H NMR spectrum of 3z

NRMP-500



Current Data Parameters  
 NAME 19-Apr-FN-2016  
 EXPNO 310  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160419  
 Time 19.26  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TD0 1

==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 3.00 Hz  
 GB 0  
 PC 1.00

Figure 58: <sup>13</sup>C NMR spectrum of 3z



NRMP-481

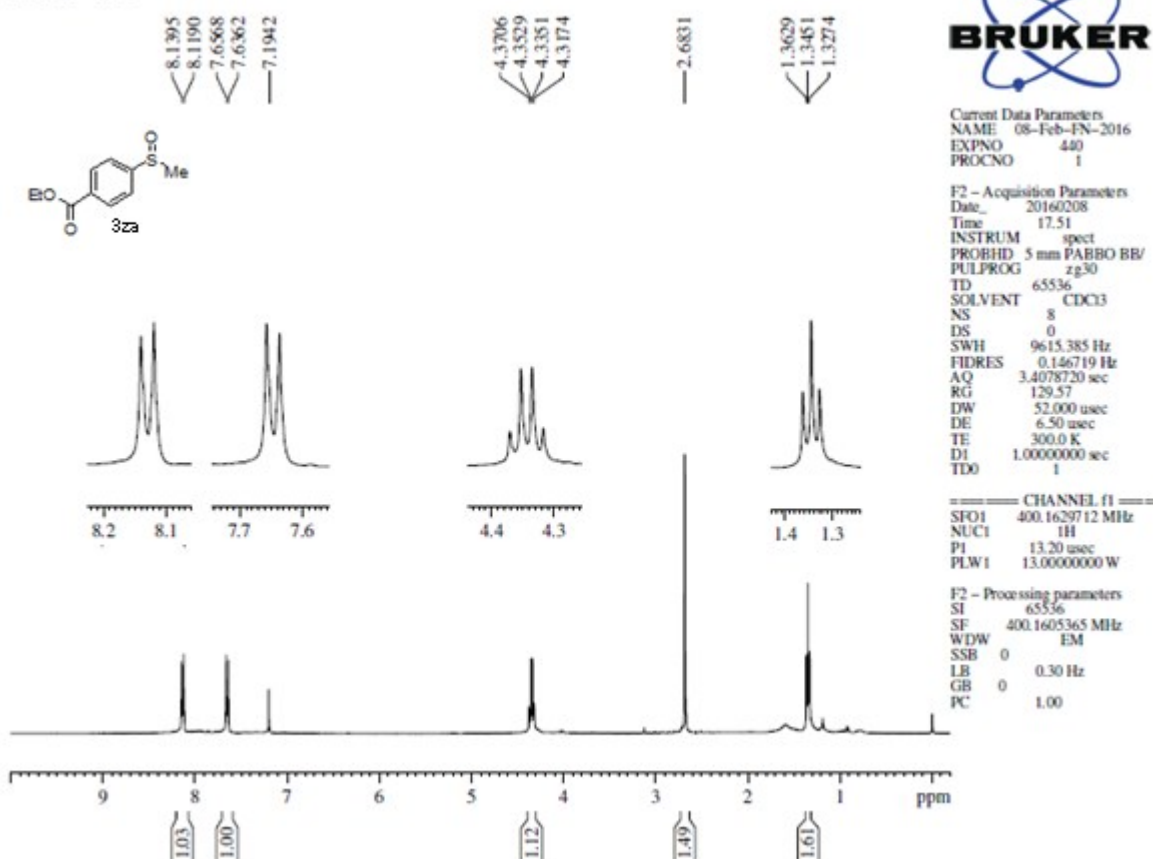


Figure 59: <sup>1</sup>H NMR spectrum of 3za

NRMP-481

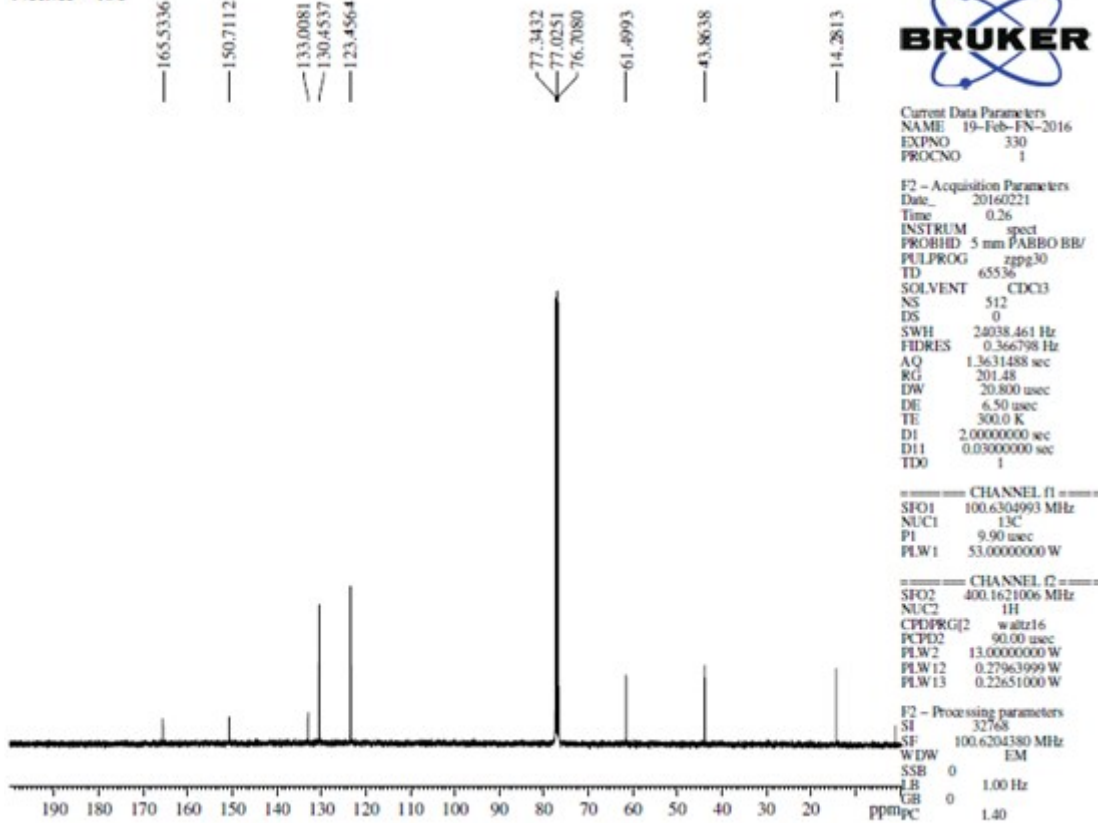
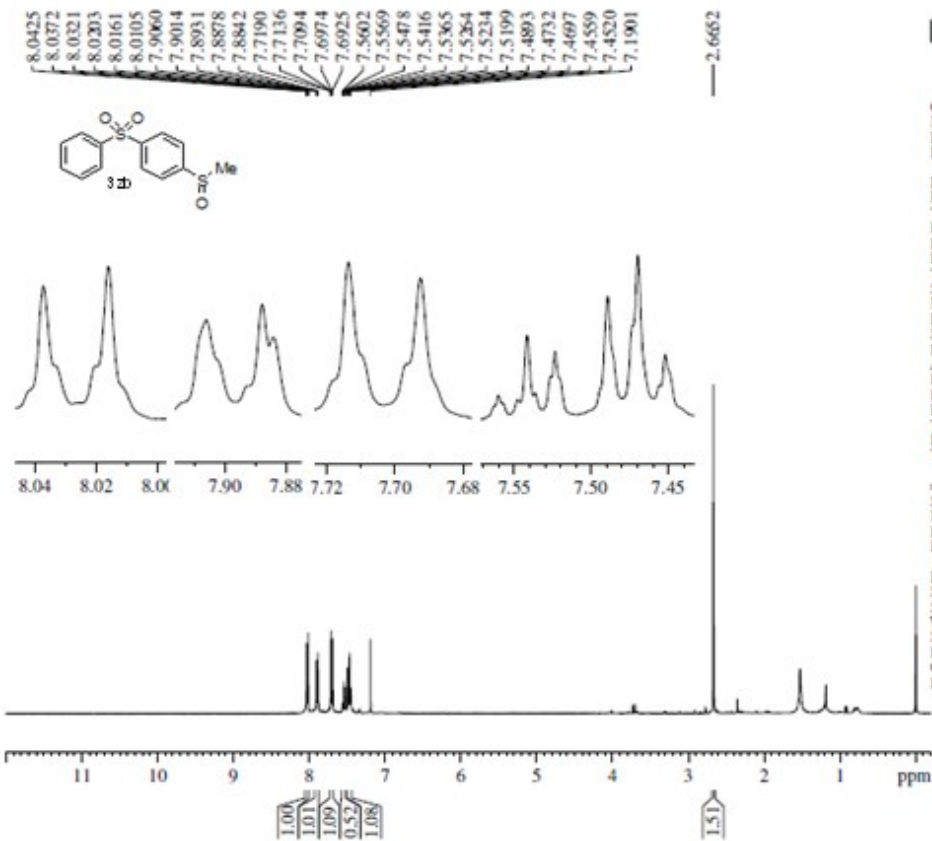


Figure 60: <sup>13</sup>C NMR spectrum of 3za

NRMP-498



Current Data Parameters  
 NAME 31-Mar-FN-2016  
 EXPNO 420  
 PROCNO 1

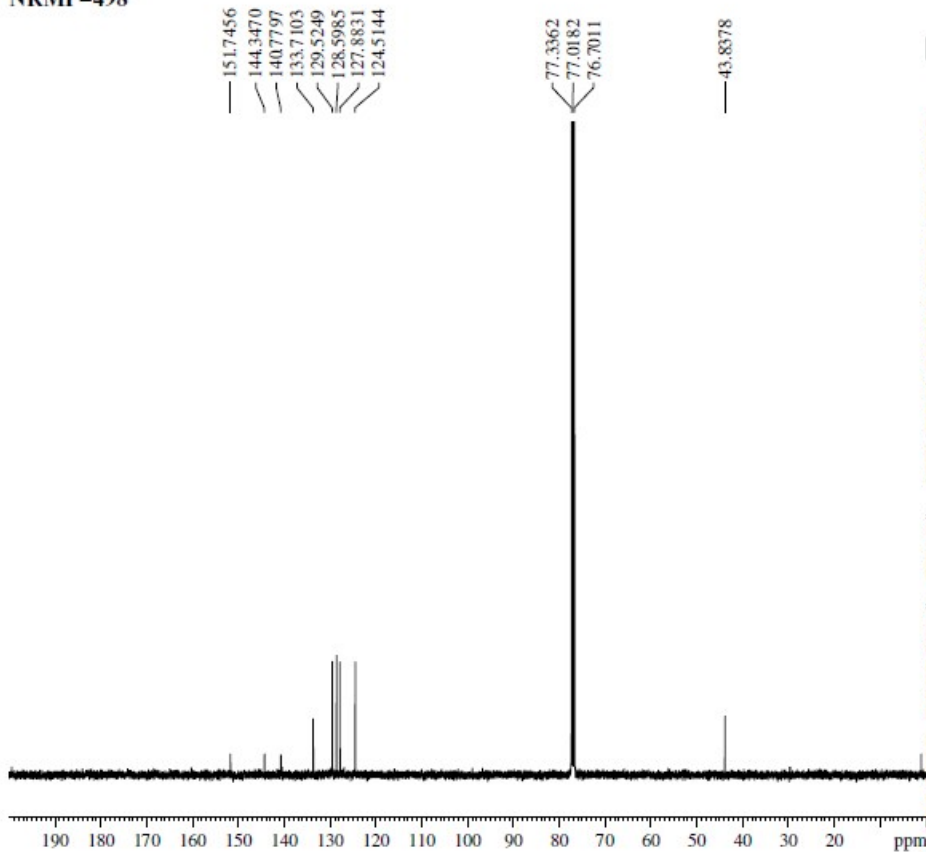
F2 - Acquisition Parameters  
 Date\_ 20160331  
 Time 18.27  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 9615.385 Hz  
 FIDRES 0.146719 Hz  
 AQ 3.4078720 sec  
 RG 145.29  
 DW 52.000 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.00000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 400.1629712 MHz  
 NUC1 1H  
 P1 13.20 usec  
 PLW1 13.00000000 W

F2 - Processing parameters  
 SI 65536  
 SF 400.1605378 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

Figure 61: <sup>1</sup>H NMR spectrum of 3zb

NRMP-498



Current Data Parameters  
 NAME 04-Apr-FN-2016  
 EXPNO 320  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160405  
 Time 2.48  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB/  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 0  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 201.48  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 TDO 1

==== CHANNEL f1 ====  
 SFO1 100.6304993 MHz  
 NUC1 13C  
 P1 9.90 usec  
 PLW1 53.00000000 W

==== CHANNEL f2 ====  
 SFO2 400.1621006 MHz  
 NUC2 1H  
 CPDPRG[2] waltz16  
 PCPD2 90.00 usec  
 PLW2 13.00000000 W  
 PLW12 0.27963999 W  
 PLW13 0.22651000 W

F2 - Processing parameters  
 SI 32768  
 SF 100.6204380 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

<sup>13</sup>C NMR spectrum of 3zb