Regiospecific One-Pot Synthesis of Diaryliodonium Tetrafluoroborates from Arylboronic Acids and Aryl Iodides

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SUPPORTING INFORMATION

1. General Experimental Conditions: S2

2. Analytical Data of Diaryliodonium Salts **3** and **4**: S2-S9

3. ^{1}H and ^{13}C NMR spectra of Salts **3** and **4**: S10-S59

1. General Experimental Conditions

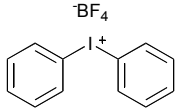
NMR spectra were recorded using DMSO-d_6 as solvent. Chemical shifts are given in ppm relative to the residual peak of DMSO (^{1}H NMR δ 2.50, ^{13}C NMR 39.52) with multiplicity (s=singlet, d=doublet, t=triplet, m=multiplet), integration and coupling constants (Hz). Chemical shifts for ^{19}F NMR are given relative to fluorobenzene (^{19}F NMR δ -112.6 in DMSO-d_6). The reactions were run without any precaution to avoid moisture or air, *i.e.* without inert gas or dry solvent. In reactions carried out above the boiling point of CH_2Cl_2, sealed tubes were used.

In our first report on *m*CPBA-oxidation of aryl iodides, the *m*CPBA was used without drying.[1] However, new commercially available cans of *m*CPBA were found to contain large and variable amounts of H_2O. Thus, *m*CPBA was dried under vacuum at room temperature for 1 h to obtain reproducible results. The percentage of active oxidizing agent in *m*CPBA was determined by iodometric titration after drying.[2] $BF_3{\cdot}Et_2O$ was stored under an argon atmosphere. All other chemicals were used as received without further purification.

The *in-situ* generation of tetrafluoroborate anion was determined by ^{19}F-NMR where the fluoride atoms resonate with two peaks given in brackets ([ ]). This is due to the natural isotope distribution of ^{10}B and ^{11}B and resonance frequencies for BF_4^- are between -147 and -149 ppm relative to fluorobenzene.[3,4]

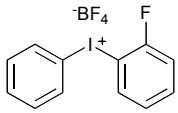
2. Analytical Data of Diaryliodonium Salts 3a-x and 4:

Diphenyliodonium tetrafluoroborate (3a):[5,6]



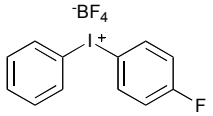
Synthesized from iodobenzene (**1a**) and phenylboronic acid (**2a**) in 82% yield as a white solid; mp: 132-134 °C (ref[6] 135-137 °C); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.25 (4 H, d, *J* = 8.3 Hz), 7.67 (2 H, t, *J* = 7.5 Hz), 7.53 (4 H, app. t, *J* = 7.7 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 135.2, 132.1, 131.8, 116.5; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.72, -147.78] (4 F); HRMS (ESI): calcd for $C_{12}H_{10}I$ ([M-BF_4^-]$^+$): 280.9822, found 280.9832.

2-Fluorophenyl(phenyl)iodonium tetrafluoroborate (3b):



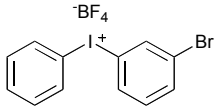
Synthesized from iodobenzene (**1a**) and 2-fluorophenylboronic acid (**2b**) in 88% yield as a white solid; mp: 141-143 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.42 (1 H, ddd, *J* = 7.6, 6.0, 1.6 Hz), 8.23 (2 H, d, *J* = 7.2 Hz), 7.77-7.64 (2 H, m), 7.62-7.52 (3 H, m), 7.38 (1 H, td, *J* = 8.0, 1.2 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 159.2 (d, *J* = 248 Hz), 137.1, 135.6 (d, *J* = 8 Hz), 135.1, 132.3, 132.0, 127.7 (d, *J* = 3 Hz), 117.0, 116.8, 103.8 (d, *J* = 23.3 Hz); ^{19}F NMR (376 MHz, DMSO-d_6): δ -97.45 (1 F), [-147.77, -147.82] (4 F); HRMS (ESI) calcd for $C_{12}H_9FI$ ([M-BF_4^-]$^+$): 298.9727, found 298.9718;

4-Fluorophenyl(phenyl)iodonium tetrafluoroborate (3c):



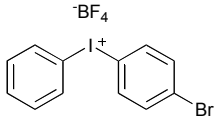
Synthesized from iodobenzene (**1a**) and 4-fluorophenylboronic acid (**2c**) in 58% yield as a white solid; mp: 138-140 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.32 (2 H, dd, J = 8.8, 5.2 Hz), 8.25 (2 H, d, J = 7.5 Hz), 7.67 (1 H, t, J = 7.5 Hz), 7.54 (2 H, app t, J = 7.8 Hz), 7.42 (2 H, t, J = 8.8 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.0 (d, J = 251 Hz), 138.1 (d, J = 9.0 Hz), 135.1, 132.1, 131.8, 119.2 (d, J = 23 Hz), 116.9, 110.7 (d, J = 3.1 Hz); ^{19}F NMR (376 MHz, DMSO-d_6): δ -106.3 (1 F, m), [-147.75, -147.80] (4 F); HRMS (ESI) calcd for $C_{12}H_9FI$ ([M-BF_4^-]$^+$): 298.9727, found 298.9717.

3-Bromophenyl(phenyl)iodonium tetrafluoroborate (3d):



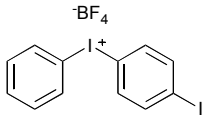
Synthesized from iodobenzene (**1a**) and 3-bromophenylboronic acid (**2d**) in 75% yield as a white solid; mp: 121-123 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.58 (1 H, app. t, J = 1.8 Hz), 8.31-8.23 (3 H, m), 7.87 (1 H, ddd, J = 8.0, 1.9, 0.8 Hz), 7.69 (1 H, t, J = 7.5 Hz), 7.56 (2 H, app. t, J = 7.6 Hz), 7.49 (1 H, app. t, J = 7.9 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 136.9, 135.3, 135.0, 134.0, 133.5, 132.3, 131.9, 123.2, 117.1, 116.8; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.76, -147.81] (4 F); HRMS (ESI) calcd for $C_{12}H_9BrI$ ([M-BF_4^-]$^+$): 358.8927, found 358.8920.

4-Bromophenyl(phenyl)iodonium tetrafluoroborate (3e):



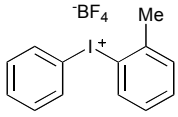
Synthesized from iodobenzene (**1a**) and 4-bromophenylboronic acid (**2e**) by the general procedure in 78% yield, as a white solid; mp: 124-126 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.25 (2 H, d, J = 8.0 Hz), 8.17 (2 H, d, J = 8.8 Hz), 7.76 (2 H, d, J = 8.8 Hz), 7.68 (1 H, t, J = 7.5 Hz), 7.54 (2 H, app t, J = 7.7 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 137.1, 135.2, 134.7, 132.2, 131.8, 126.2, 116.8 115.1; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.76, -147.81] (4 F); HRMS (ESI) calcd for $C_{12}H_9BrI$ ([M-BF_4^-]$^+$): 358.8927, found 358.8917.

4-Iodopheny(phenyl)liodonium tetrafluoroborate (3f):



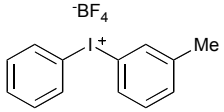
Synthesized from iodobenzene (**1a**) and 4-iodophenylboronic acid (**2f**) in 73% yield as a white solid; mp: 147-149 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.24 (2 H, dd, J = 8.4, 1.2 Hz), 7.99 (2 H, d, J = 8.4 Hz), 7.90 (2 H, d, J = 8.4 Hz), 7.68 (1 H, t, J = 7.4 Hz), 7.54 (2 H, app t, J = 7.8 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 140.4, 136.8, 135.2, 132.2, 131.8, 116.7, 115.9, 100.3; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.76, -147.81] (4 F); HRMS (ESI) calcd for $C_{12}H_9I_2$ ([M-BF_4^-]$^+$): 406.8788, found 406.8791.

2-Methylphenyl(phenyl)iodonium tetrafluoroborate (3g):[7]



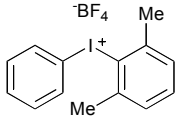
Synthesized from iodobenzene (**1a**) and 2-methylphenylboronic acid (**2g**) in 80% yield as a white solid. The previous reference does not contain analytical data. mp: 154-156 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.40 (1 H, d, J = 8.0 Hz), 8.21 (2 H, dd, J = 7.2, 0.8 Hz), 7.66 (1 H, t, J = 7.4 Hz), 7.61-7.49 (4 H, m), 7.34-7.29 (1 H, m), 2.62 (3 H, s); ^{13}C NMR (100 MHz, DMSO-d_6): δ 140.6, 137.1, 135.1, 132.9, 132.0, 131.8, 131.5, 129.3, 121.4, 115.8, 25.0; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.77, -147.83] (4 F); HRMS (ESI) calcd for $C_{13}H_{12}I$ ([M-BF_4^-]$^+$): 294.9978, found 294.9971.

3-Methylphenyl(phenyl)iodonium tetrafluoroborate (3h):[8]



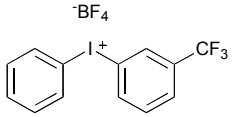
Synthesized from iodobenzene (**1a**) and 3-methylphenylboronic acid (**2h**) in 84% yield as a white solid; mp: 122-124 °C (ref[8] 123.5-125°C); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.23 (2 H, dd, J = 8.4, 1.0 Hz), 8.11 (1 H, s), 8.05 (1 H, d, J = 8.0 Hz), 7.67 (1 H, t, J = 7.4 Hz), 7.56-7.46 (3 H, m), 7.42 (1 H, app. t, J = 7.8 Hz), 2.34 (3 H, s); ^{13}C NMR (100 MHz, DMSO-d_6): δ 141.8, 135.3, 135.1, 132.7, 132.2, 132.0, 131.8, 131.5, 116.4, 116.3, 20.8; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.76, -147.81] (4 F); HRMS (ESI) calcd for $C_{13}H_{12}I$ ([M-BF_4^-]$^+$): 294.9978, found 294.9985.

2,6-Dimethylphenyl(phenyl)iodonium tetrafluoroborate (3i):



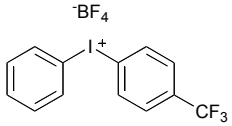
Synthesized from iodobenzene (**1a**) and 2,6-dimethylphenylboronic acid (**2i**) in 81% yield as a white solid; mp: 175-177 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.01 (2 H, dd, J = 8.4, 1.2 Hz), 7.65 (1 H, t, J = 7.2 Hz), 7.53-7.46 (3 H, m), 7.42-7.38 (2 H, m), 2.65 (6 H, s); ^{13}C NMR (100 MHz, DMSO-d_6): δ 141.8, 134.7, 132.9, 131.9, 131.9, 129.1, 126.2, 114.4, 26.5; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.78, -147.83] (4 F); HRMS (ESI) calcd for $C_{14}H_{14}I$ ([M-BF_4^-]$^+$): 309.0135, found 309.0134.

3-Trifluoromethylphenyl(phenyl)iodonium tetrafluoroborate (3j):



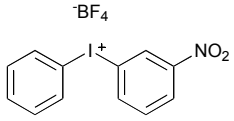
Synthesized from iodobenzene (**1a**) and 3-trifluoromethylphenylboronic acid (**2j**) in 73% yield as a white solid; mp: 132-134 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.76 (1 H, s), 8.55 (1 H, d, J = 8.4 Hz), 8.31 (2 H, dd, J = 7.6, 1.0 Hz), 8.05 (1 H, d, J = 8.0 Hz), 7.76 (1 H, app. t, J = 8.0 Hz), 7.69 (1 H, app. t, J = 7.4, 1.2 Hz), 7.56 (2 H, app t, J = 8.0 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 139.2, 135.3, 132.7, 132.3, 131.9, 131.8 (q, J = 3.7 Hz), 131.3 (q, J = 32.4 Hz), 128.8 (q, J = 3.5 Hz), 122.9 (q, J = 272 Hz), 116.9, 116.8; ^{19}F NMR (376 MHz, DMSO-d_6): δ -60.79 (3 F, s), [-147.75, -147.81] (4 F); HRMS (ESI) calcd for $C_{13}H_9F_3I$ ([M-BF_4^-]$^+$): 348.96.96, found 348.9685.

4-Trifluoromethylphenyl(phenyl)iodonium tetrafluoroborate (3k):[7]



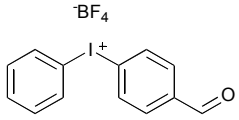
Synthesized from iodobenzene (**1a**) and 4-trifluoromethylphenylboronic acid (**2k**) in 69% yield as a white solid. The previous reference does not contain analytical data. mp: 106-108 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.45 (2 H, d, J = 8.4 Hz), 8.30 (2 H, dd, J = 8.0, 1.0 Hz), 7.92 (2 H, d, J = 8.4, Hz), 7.70 (1 H, t, J = 7.6, 1.4 Hz), 7.56 (2 H, app. t, J = 8.0 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 135.8, 135.4, 132.4, 132.0, 131.9 (q, J = 32.0 Hz), 128.4 (q, J = 3.7 Hz), 123.5 (q, J = 268.1 Hz), 116.7; ^{19}F NMR (376 MHz, DMSO-d_6): δ -61.25 (3 F, s), [-147.75, -147.80] (4 F); HRMS (ESI) calcd for $C_{13}H_9F_3I$ ([M-BF_4^-]$^+$): 348.9696, found 348.9698.

3-Nitrophenyl(phenyl)iodonium tetrafluoroborate (3l):



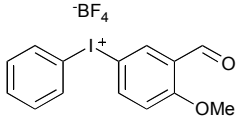
Synthesized from iodobenzene (**1a**) and 3-nitrophenylboronic acid (**2l**) in 56% yield as a white solid; mp: 108-110 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.18 (1 H, app. t, J = 2.0 Hz), 8.65 (1 H, ddd, J = 8.0, 1.6, 0.8 Hz), 8.45 (1 H, ddd, J = 8.4, 2.4, 0.8 Hz), 8.33 (2 H, dd, J = 8.4, 0.8 Hz), 7.82 (1 H, app t, J = 8.0 Hz), 7.69 (1 H, t, J = 7.4 Hz), 7.56 (2 H, app t, J = 7.8 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 148.4, 141.1, 135.4, 132.8, 132.4, 131.9, 129.8, 126.8, 117.0, 116.3; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.75, -147.80] (4 F); HRMS (ESI) calcd for $C_{12}H_9INO_2$ ([M-BF_4^-]$^+$): 325.9672, found 325.9677.

4-Formylphenyl(phenyl)iodonium tetrafluoroborate (3m):



Synthesized from iodobenzene (**1a**) and 4-formylphenylboronic acid (**2m**) in 65% yield as an off-white solid (*Note*: The product was isolated through precipitation with diethyl ether, rather than silica plug, and then purified by decantation as described in the general procedure); mp: 135-138 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.02 (1 H, s), 8.45 (2 H, d, J = 8.0 Hz), 8.29 (2 H, d, J = 8.0 Hz), 8.00 (2 H, d, J = 8.0 Hz), 7.69 (1 H, t, J = 7.4 Hz), 7.55 (2 H, app t, J = 7.8 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 192.5, 137.9, 135.8, 135.4, 132.3, 131.9, 131.9, 122.2, 116.7; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.77, -147.82] (4 F); HRMS (ESI) calcd for $C_{13}H_{10}IO$ ([M-BF_4^-]$^+$): 308.9771, found 308.9774.

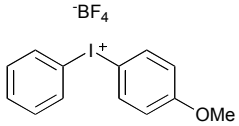
3-Formyl-4-methoxyphenyl(phenyl)iodonium tetrafluoroborate (3n):



Synthesized from iodobenzene (**1a**) and 3-formyl-4-methoxyphenylboronic acid (**2n**) in 85% yield as an off-white solid (*Note*: The product was isolated through precipitation with diethyl ether, rather than silica plug, and then purified by decantation as described in the general procedure); mp: 185-187 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.27 (1 H, s), 8.51-8.44 (2 H, m), 8.26 (2 H, d, J = 7.6 Hz), 7.66

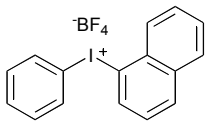
(1 H, t, *J* = 7.4 Hz), 7.53 (2 H, app. t, *J* = 7.8 Hz), 7.39 (1 H, d, *J* = 8.8 Hz), 3.96 (3 H, s); ^{13}C NMR (100 MHz, DMSO-d_6): δ 187.8, 163.4, 142.5, 135.1, 135.0, 132.1, 131.7, 126.2, 117.0, 116.6, 106.2, 56.7; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.78, -147.84] (4 F); HRMS (ESI) calcd for $C_{14}H_{12}IO_2$ ([M-BF_4^-]$^+$): 338.9876, found 338.9880.

4-Methoxyphenyl(phenyl)iodonium tetrafluoroborate (3o):[9,10]



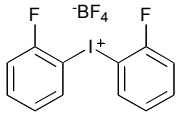
Synthesized from iodobenzene (**1a**) and 4-methoxyphenylboronic acid (**2o**) in 84% yield as a white solid (*Note*: **2o** was added at -78 ˚C and stirred at that temperature for 15 minutes, then brought up to room temperature and worked up as described in the general procedure.); mp: 95-97 ˚C (ref[10] 95-96 ˚C); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.21-8.15 (4 H, m), 7.65 (1 H, t, *J* = 7.4 Hz), 7.52 (2 H, app t, *J* = 7.6 Hz), 7.07 (2 H, d, *J* = 7.2 Hz), 3.79 (3 H, s); ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.0, 137.2, 134.8, 131.9, 131.7, 117.5, 117.0, 105.4, 55.7; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.78, -147.83] (4 F); HRMS (ESI) calcd for $C_{13}H_{12}IO$ ([M-BF_4^-]$^+$): 310.9927, found 310.9929.

1-Naphtyl(phenyl)iodonium tetrafluoroborate (3p):[11]



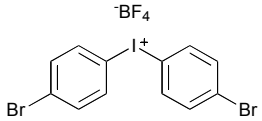
Synthesized from iodobenzene (**1a**) and 1-naphtylboronic acid (**2p**) in 81% yield as a white solid (*Note*: **2p** was added at -78 ˚C and stirred at that temperature for 15 minutes, then brought up to room temperature and worked up as described in the general procedure.); mp: 177-179 ˚C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.81 (1 H, dd, *J* = 7.6, 1.0 Hz), 8.34 (1 H, *J* = 8.0 Hz), 8.31-8.26 (3 H, m), 8.07 (1 H, d, *J* = 8.0 Hz), 7.81 (1 H, app. t, *J* = 7.2, 1.2 Hz), 7.72 (1 H, app. t, *J* = 8.0, 0.8 Hz), 7.64 (1 H, app t, *J* = 7.8 Hz), 7.59 (1 H, t, *J* = 7.2 Hz), 7.47 (2 H, app t, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 137.4, 134.9, 134.1, 133.5, 131.9, 131.7, 130.9, 129.8, 129.5, 128.8, 128.0, 127.6, 119.3, 116.2; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.77, -147.82] (4 F); HRMS (ESI) calcd for $C_{16}H_{12}I$ ([M-BF_4^-]$^+$): 330.9978, found 330.9989.

Bis(2-fluorophenyl)iodonium tetrafluoroborate (3q):



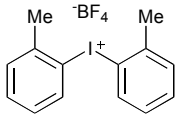
Synthesized from 2-fluoroiodobenzene (**1b**) and 2-fluorophenylboronic acid (**2b**) in 85% yield as a white solid; mp: 174-175 ˚C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.41 (2 H, ddd, *J* = 8.0, 6.4, 1.2 Hz), 7.74 (2 H, m), 7.59 (2 H, td, *J* = 8.4, 1.6 Hz), 7.38 (2 H, td, *J* = 7.8, 1.0 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 159.1 (d, *J* = 248.5 Hz), 137.1, 135.8 (d, *J* = 7.2 Hz), 127.8, 117.0 (d, *J* = 22 Hz), 104.1 (d, *J* = 23.5 Hz); ^{19}F NMR (376 MHz, DMSO-d_6): δ -97.44 (2 F, m) [-147.77, -147.83] (4 F); HRMS (ESI) calcd for $C_{12}H_8F_2I$ ([M-BF_4^-]$^+$): 316.9633, found 316.9624.

Bis(4-bromophenyl)iodonium tetrafluoroborate (3r):



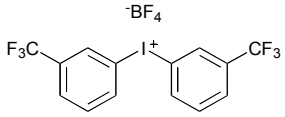
Synthesized from 4-bromoiodobenzene (**1c**) and 4-bromophenylboronic acid (**2e**) in 66% yield as a white solid; mp: 178-180 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.17 (4 H, d, J = 8.8 Hz), 7.77 (4 H, d, J = 8.8 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 137.1, 134.7, 126.4, 115.3; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.76, -147.82] (4 F); HRMS (ESI) calcd for $C_{12}H_8Br_2I$ ([M-BF_4^-]$^+$): 436.8032, found 436.8017.

Bis(2-methylphenyl)iodonium tetrafluoroborate (3s):



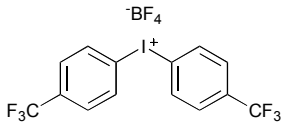
Synthesized from 2-iodotoluene (**1d**) and 2-methylphenylboronic acid (**2g**) in 74% yield as a white solid; mp: 160-162 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.31 (2 H, d, J = 7.6 Hz), 7.60-7.53 (4 H, m), 7.33-7.27 (2 H, m), 2.61 (6 H, s); ^{13}C NMR (100 MHz, DMSO-d_6): δ 140.6, 137.2, 132.8, 131.6, 129.3, 120.5, 25.0; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.78, -147.83] (4 F); HRMS (ESI) calcd for $C_{14}H_{14}I$ ([M-BF_4^-]$^+$): 309.0135, found 309.0132.

Bis(3-trifluoromethylphenyl)iodonium tetrafluoroborate (3t):



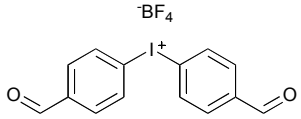
Synthesized from 3-trifluoromethyliodobenzene (**1e**) and 3-trifluoromethylphenylboronic acid (**2j**) in 51% yield as a white solid (*Note*: Pre-oxidation time was increased to 60 minutes and 30 minutes was needed for the second step); mp: 163-165 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.82 (2 H, s), 8.61 (2 H, d, J = 8.4 Hz), 8.08 (2 H, d, J = 8.4 Hz), 7.79 (2 H, app. t, J = 8.0 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 139.3, 132.8, 132.0, (q, J = 3.7 Hz), 131.4 (q, J = 32.5 Hz), 129.1 (q, J = 3.5 Hz), 122.9 (q, J = 270.3 Hz), 117.2; ^{19}F NMR (376 MHz, DMSO-d_6): δ -60.82 (6 F, s) [-147.76, -147.82] (4 F); HRMS (ESI) calcd for $C_{14}H_8F_6I$ ([M-BF_4^-]$^+$): 416.9569, found 416.9585.

Bis(4-trifluoromethylphenyl)iodonium tetrafluoroborate (3u):



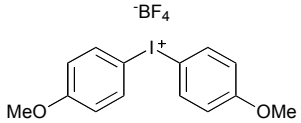
Synthesized from 4-trifluoromethyliodobenzene (**1f**) and 4-trifluoromethylphenylboronic acid (**2k**) in 56% yield as a white solid (*Note*: Pre-oxidation time was increased to 60 minutes and 30 minutes was needed for the second step); mp: 189-191 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.51 (4 H, d, 8.0 Hz), 7.95 (4 H, d, 8.0 Hz); ^{13}C NMR (125 MHz, DMSO-d_6): δ 136.3, 132.1 (q, J = 32.3 Hz), 128.6 (q, J = 3.5 Hz), 123.4 (q, J = 271.5 Hz), 121.0; ^{19}F NMR (376 MHz, DMSO-d_6): δ -61.25 (6 F, s), [-147.78, -147.83] (4 F); HRMS (ESI) calcd for $C_{14}H_8F_6I$ ([M-BF_4^-]$^+$): 416.9569, found 416.9574.

Bis(4-formylphenyl)iodonium tetrafluoroborate (3v):



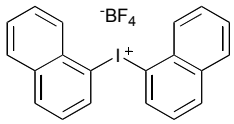
Synthesized from 4-formyliodobenzene (**1g**) and 4-formylphenylboronic acid (**2m**) in 31% yield as an off-white solid (*Note*: The reaction was carried out in MeCN (1 mL) due to solubility problems.); mp: 109-111 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.02 (2 H, s), 8.49 (4 H, d, J = 8.4 Hz), 8.01 (4 H, d, J = 8.4 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 192.5, 138.0, 136.0, 132.0, 122.4; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.78, -147.83] (4 F); HRMS (ESI) calcd for $C_{14}H_{10}IO_2$ ([M-BF_4^-]$^+$): 336.9720, found 336.9707.

Bis(4-methoxyphenyl)iodonium tetrafluoroborate (3w):



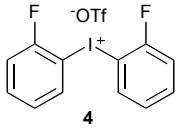
Synthesized from 4-methoxyiodobenzene (**1h**) and 4-methoxyphenylboronic acid (**2o**) by the modified procedure in 46% yield, as a grey solid; mp: 170 °C (decomposed.); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.12 (4 H, d, J = 7.2 Hz), 7.06 (4 H, d, J = 7.2 Hz), 3.79 (6 H, s); ^{13}C NMR (100 MHz, DMSO-d_6): δ 161.8, 136.8, 117.3, 105.9, 55.7; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.78, -147.84] (4 F); HRMS (ESI) calcd for $C_{14}H_{14}IO_2$ ([M-BF_4^-]$^+$): 341.0033, found 341.0029.

Bis(1-Naphtyl)iodonium tetrafluoroborate (3x):



Synthesized from 1-iodonaphthalene (**1i**) and 1-naphtylboronic acid (**2p**) by the modified procedure in 37% yield as a grey solid; mp: 155 °C (decomposed); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.88 (2 H, dd, 7.6, 1.0 Hz), 8.49 (2 H, d, 8.8 Hz), 8.21 (2 H, d, 8.0 Hz), 8.01 (2 H, d, 8.0 Hz), 7.81 (2 H, app. t, 6.8, 1.4 Hz), 7.68 (2 H, app. t, 8.0, 0.8 Hz), 7.57 (2 H, app. t, 7.8 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 137.3, 134.2, 133.4, 130.9, 129.7, 129.4, 128.8, 128.0, 127.5, 119.2; ^{19}F NMR (376 MHz, DMSO-d_6): δ [-147.78, -147.84] (4 F); HRMS (ESI) calcd for $C_{20}H_{14}I$ ([M-BF_4^-]$^+$): 381.0135, found 381.0136.

Bis(2-fluorophenyl)iodonium triflate (4):



4

Synthesized from 2-fluoroiodobenzene (**1b**) and 2-fluorophenylboronic acid (**2b**) in 75% yield as a white solid; mp: 181-183 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.41 (2 H, ddd, J = 8.0, 6.4, 1.6 Hz), 7.74 (2 H, m), 7.59 (2 H, td, J = 8.4, 1.2 Hz), 7.38 (2 H, td, J = 8.0, 1.3 Hz); ^{13}C NMR (100 MHz, DMSO-d_6): δ 159.1 (d, J = 248.4 Hz), 137.1, 135.8 (d, J = 7.7 Hz), 127.8, 120.8 (q, 320.5 Hz, $F_3SO_3^-$), 116.8 (d, J = 22.1 Hz), 104.1 (d, J = 24.0 Hz); ^{19}F NMR (376 MHz, DMSO-d_6): δ -77.3 (3 F, s, $CF_3SO_3^-$), -97.43 (2 F, m); HRMS (ESI) calcd for $C_{12}H_8F_2I$ ([M-BF_4^-]$^+$): 316.9633, found 316.9624.

References

(1) Bielawski, M.; Olofsson, B. *Chem. Commun.* **2007**, 2521-2523.

(2) Vogel, A. I.; Furniss, B. S.; Hannaford, A. J.; Rogers, V.; Smith, P. W. G.; Tatchell, A. R. *Vogel's Textbook of Practical Organic Chemistry*, 1978.

(3) Plakhotnik, V. N.; Ernst, L.; Sakhaii, P.; Tovmash, N. F.; Schmutzler, R. *J. Fluorine Chem.* **1999**, *98*, 133-135.

(4) Padelidakis, V.; Tyrra, W.; Naumann, D. *J. Fluorine Chem.* **1999**, *99*, 9-15.

(5) Ochiai, M.; Toyonari, M.; Nagaoka, T.; Chen, D.-W.; Kida, M. *Tetrahedron Lett.* **1997**, *38*, 6709-6712.

(6) Kazmierczak, P.; Skulski, L. *Synthesis* **1995**, 1027-1032.

(7) Ochiai, M.; Suefuji, T.; Miyamoto, K.; Tada, N.; Goto, S.; Shiro, M.; Sakamoto, S.; Yamaguchi, K. *J. Am. Chem. Soc.* **2003**, *125*, 769-773.

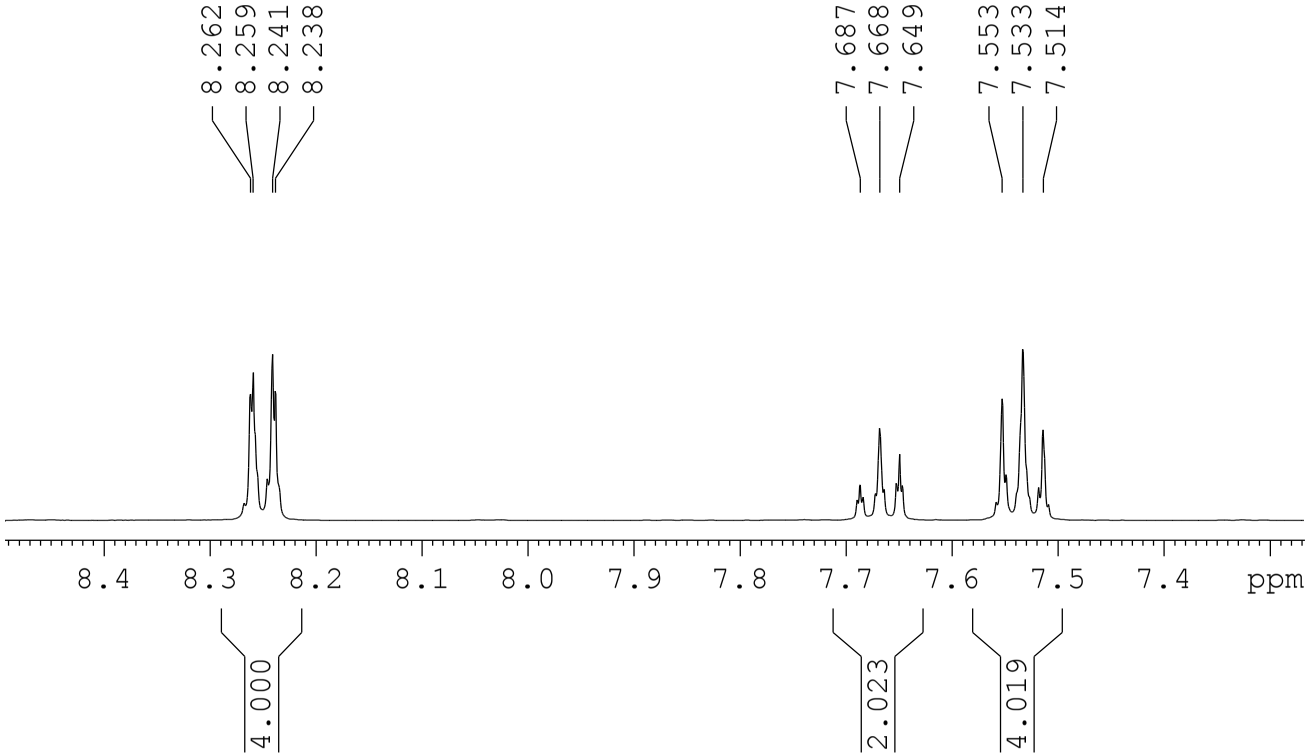
(8) Lubinkowski, J. J.; Gimenez Arrieche, C.; McEwen, W. E. *J. Org. Chem.* **1980**, *45*, 2076-2079.

(9) Ochiai, M.; Toyonari, M.; Sueda, T.; Kitagawa, Y. *Tetrahedron Lett.* **1996**, *37*, 8421-8424.

(10) Kasumov, T. M.; Brel, V. K.; Koz'min, A. S.; Zefirov, N. C. *Synthesis* **1995**, 775-776.

(11) Kaafarani, B. R.; Gu, H.; Pinkerton, A. A.; Neckers, D. C. *J. Chem. Soc., Dalton Trans.* **2002**, 2318-2321.

3. ^{1}H and ^{13}C NMR spectra of Diaryliodonium Salts 3 and 4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7.553
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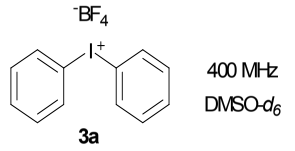
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
ppm
4.000
2.023
4.019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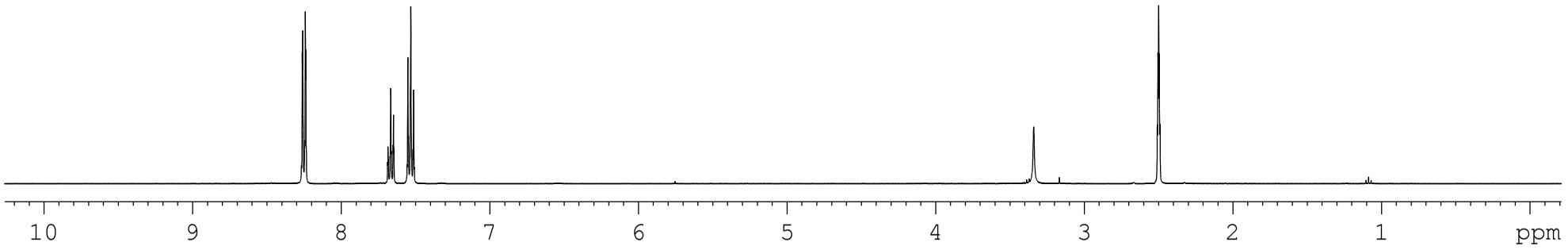
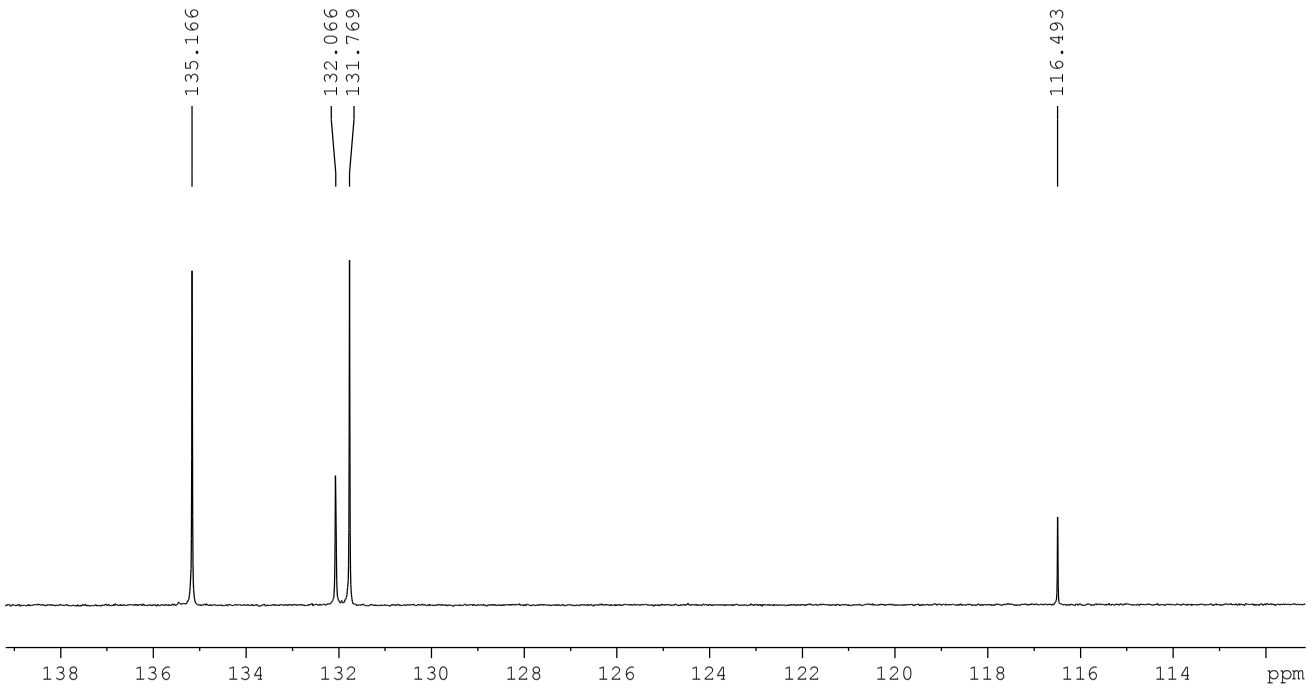
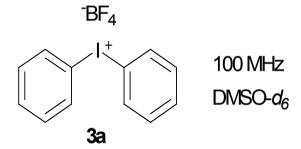
$^{+}$
3a
400 MHz
DMSO-d_6


10
9
8
7
6
5
4
3
2
1
ppm





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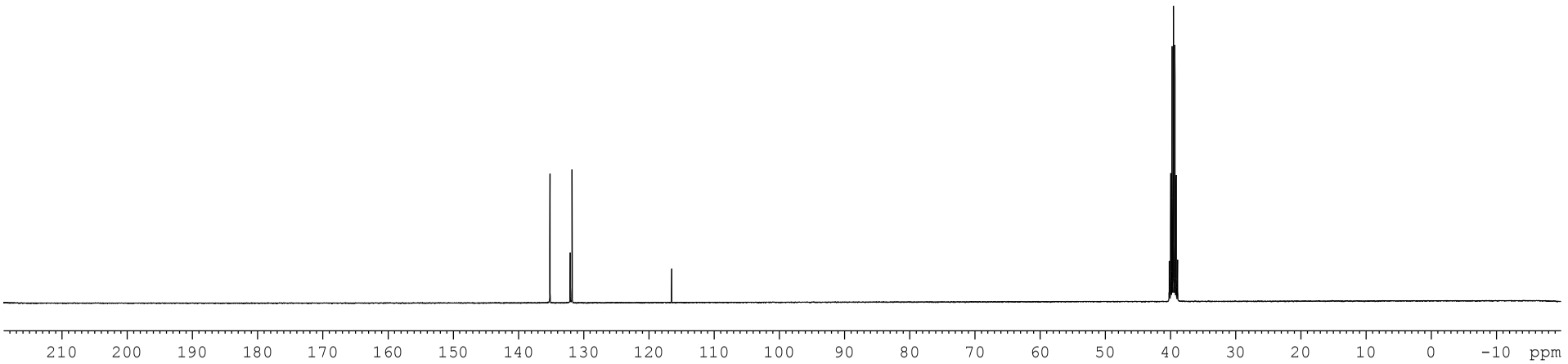
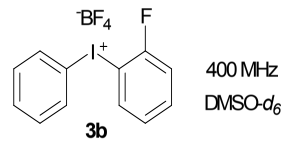
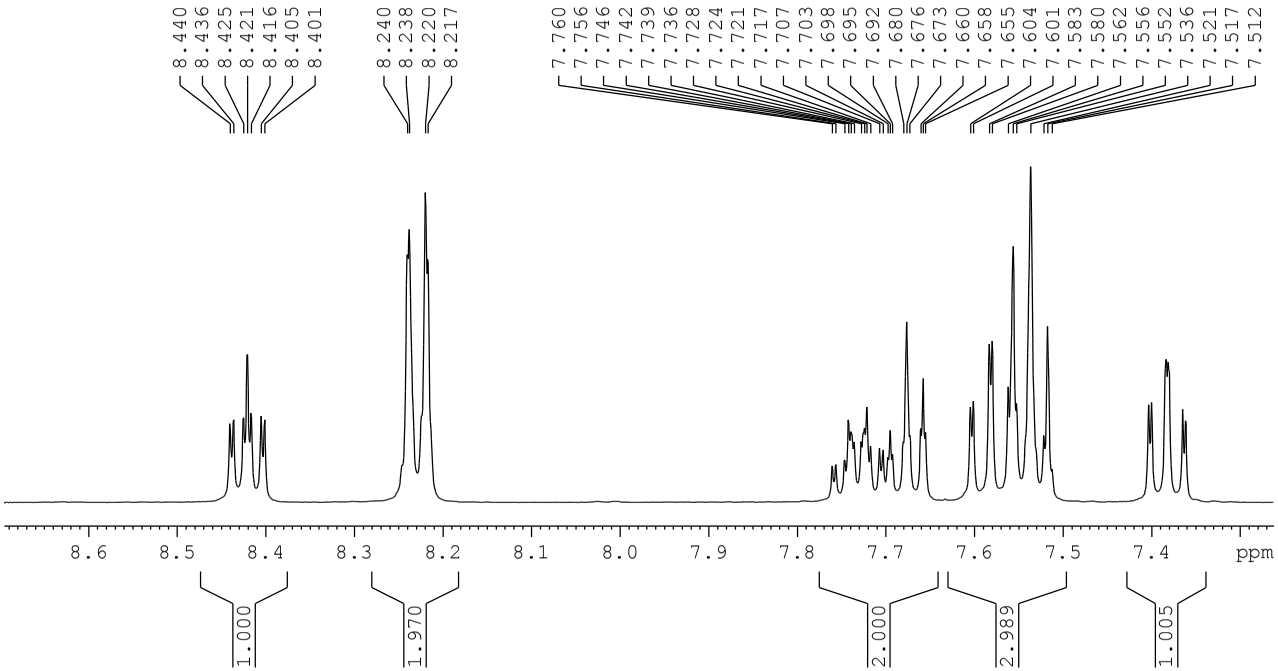
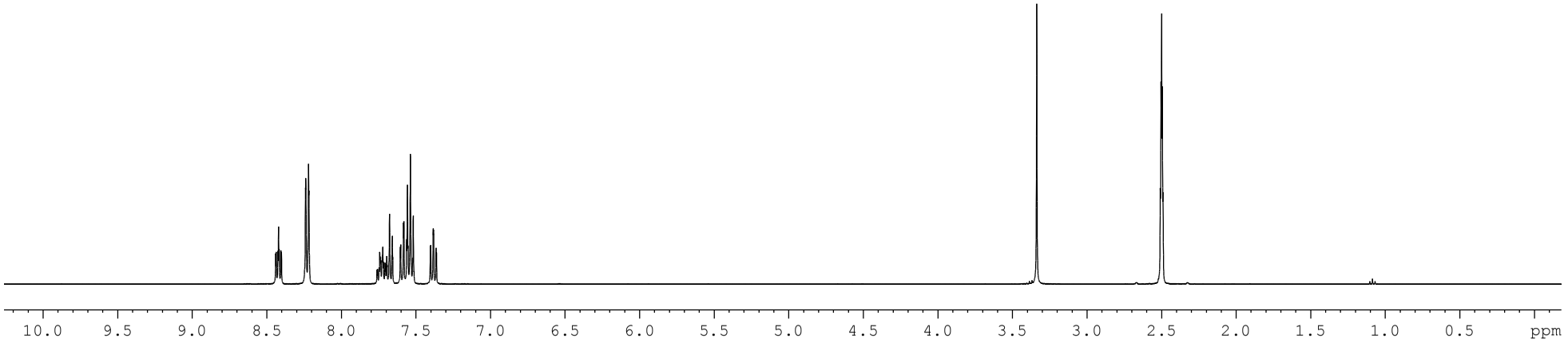
DMSO-d_6

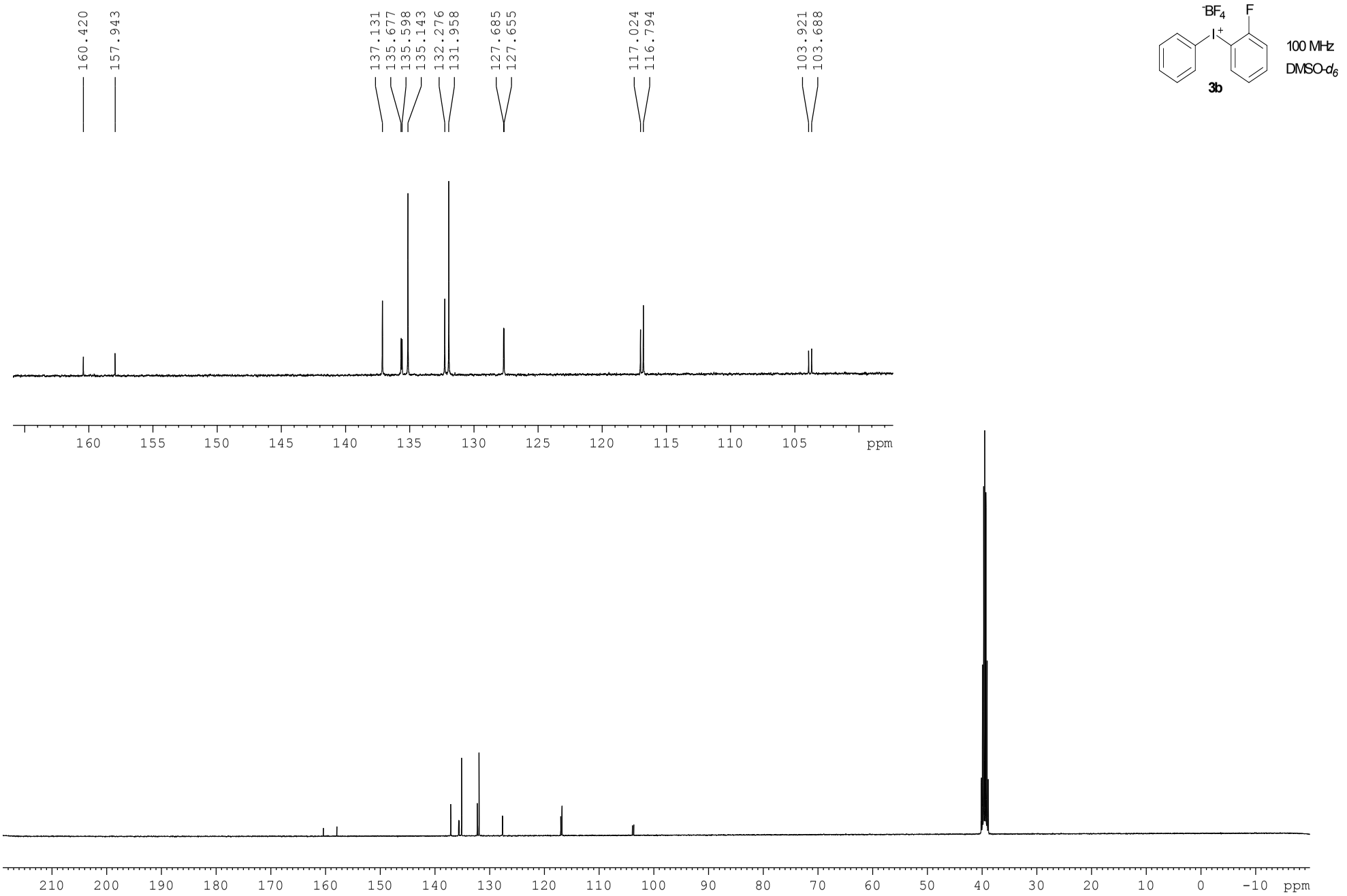




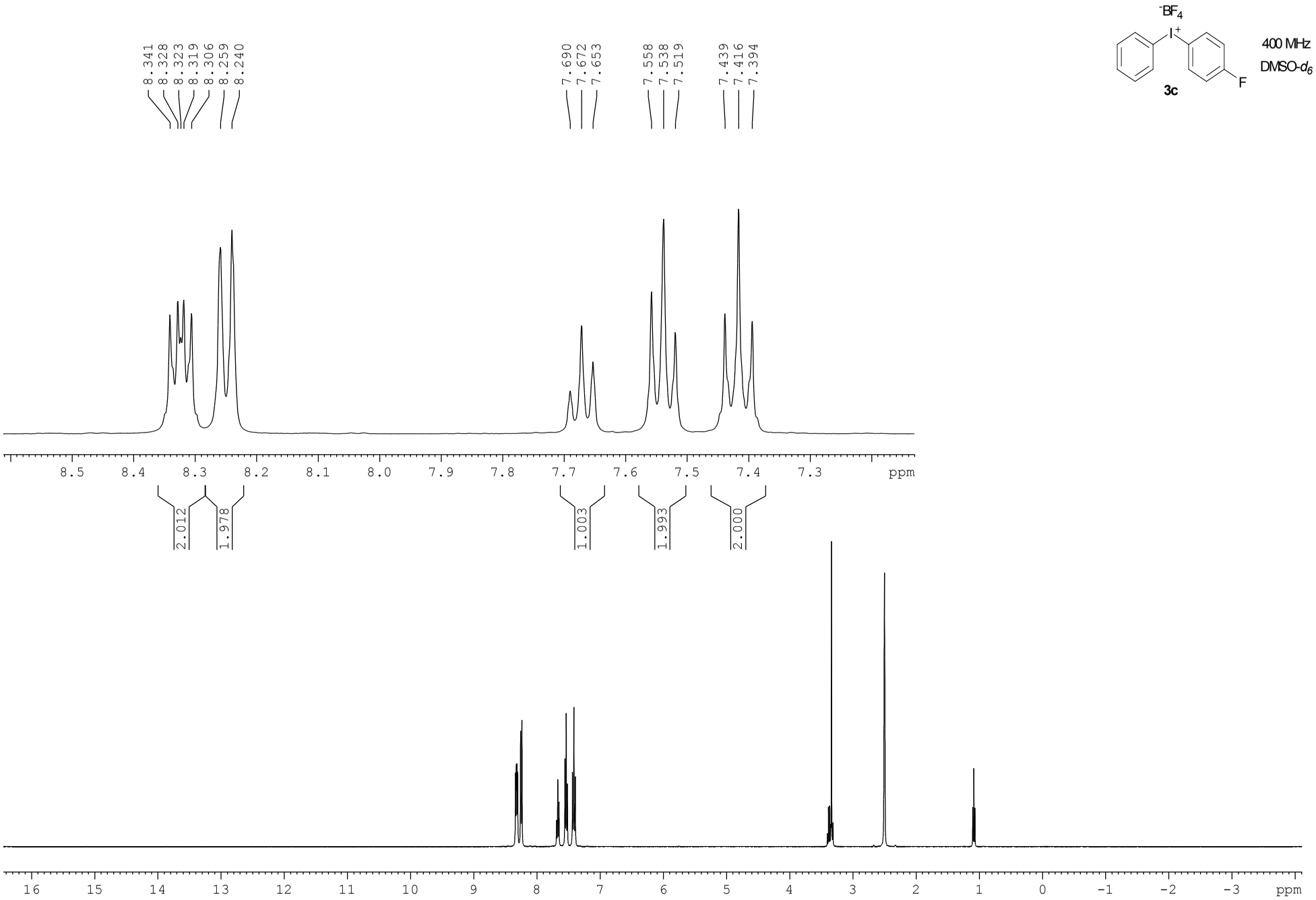
400 MHz
DMSO-d_6







100 MHz
DMSO-d_6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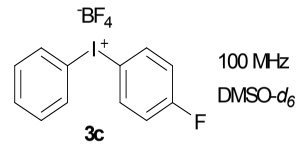
ppm
ppm
$^{-}BF_4$
$^{+}$
F
3c
400 MHz
DMSO-d_6



100 MHz
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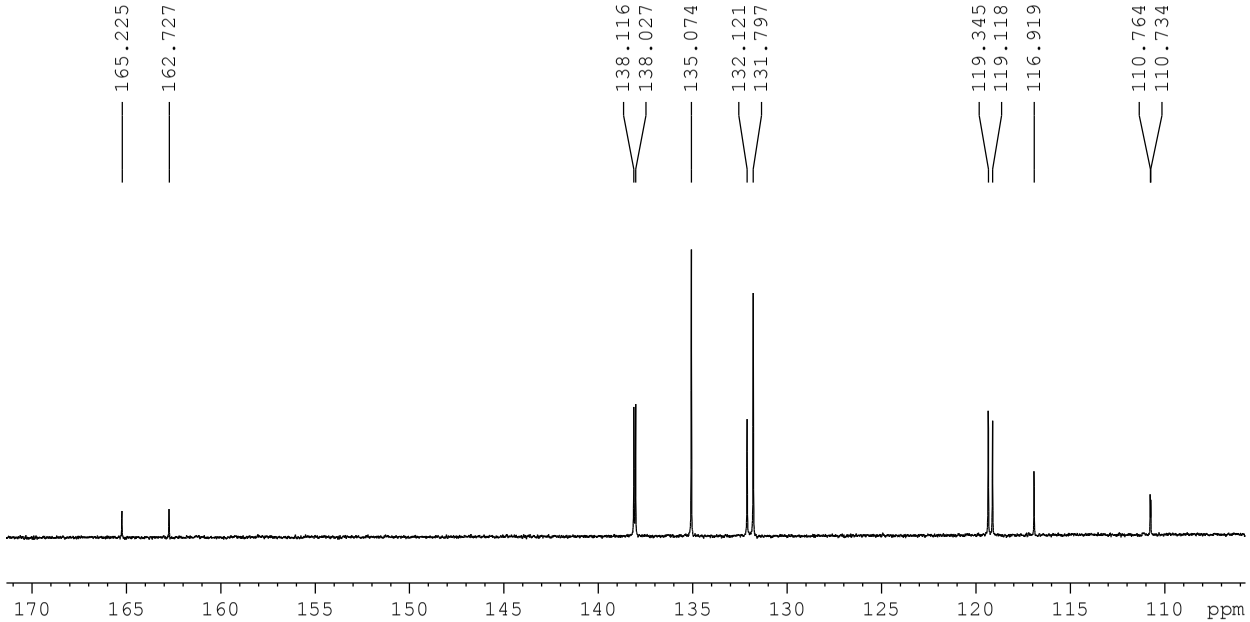

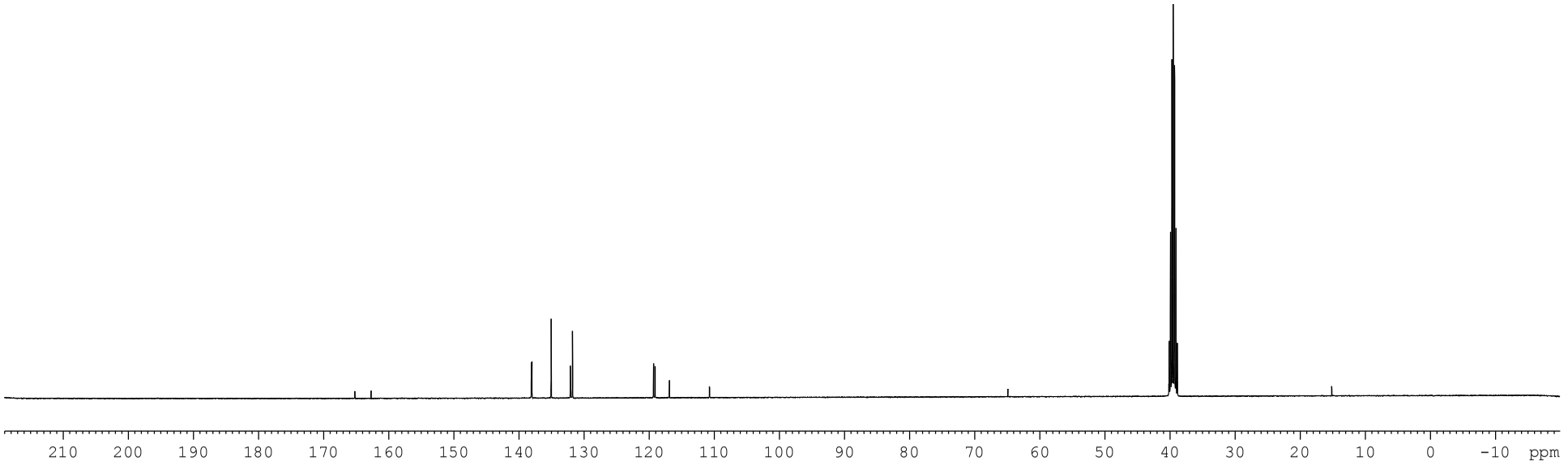

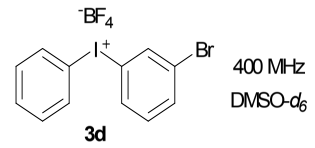

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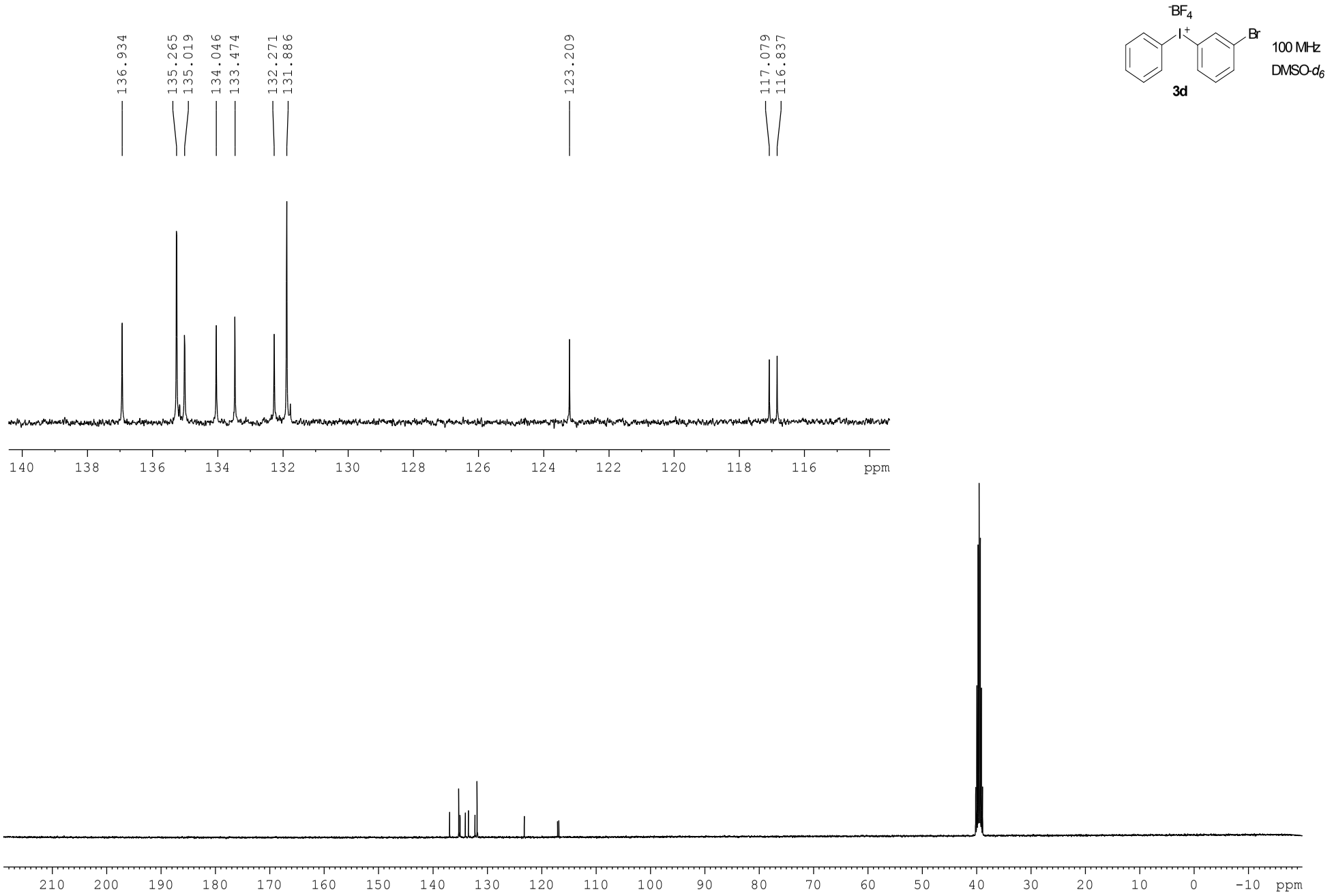

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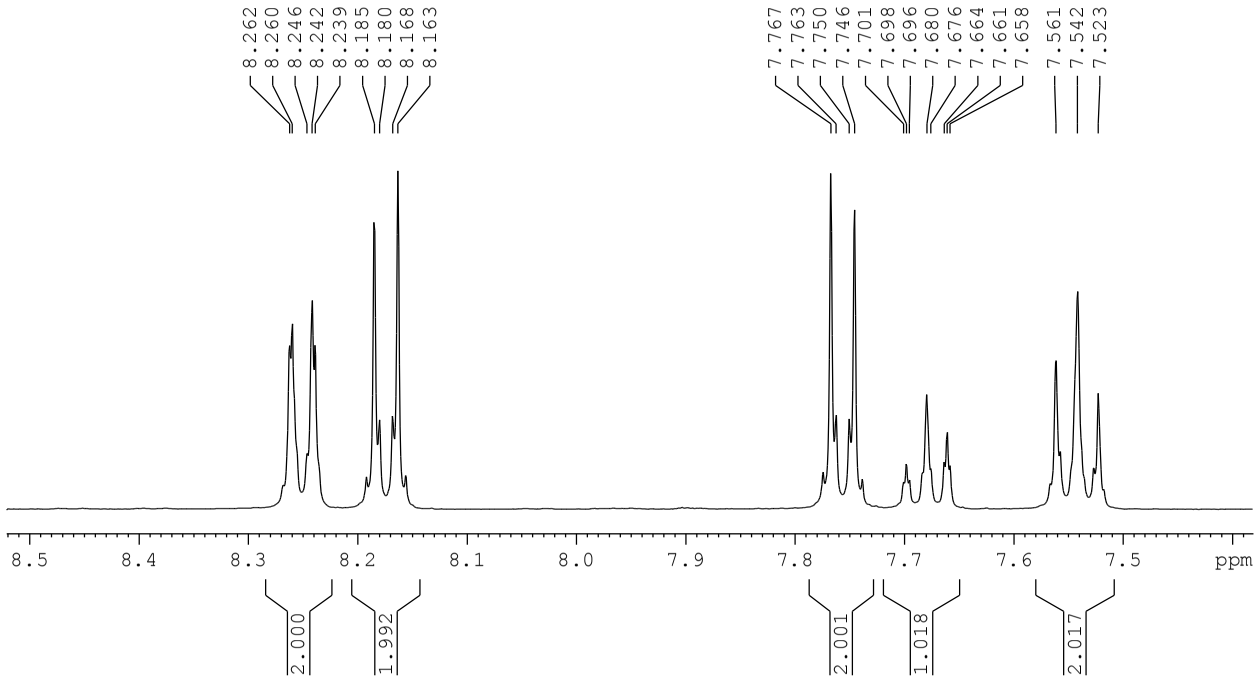
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7.676
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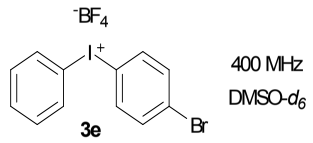
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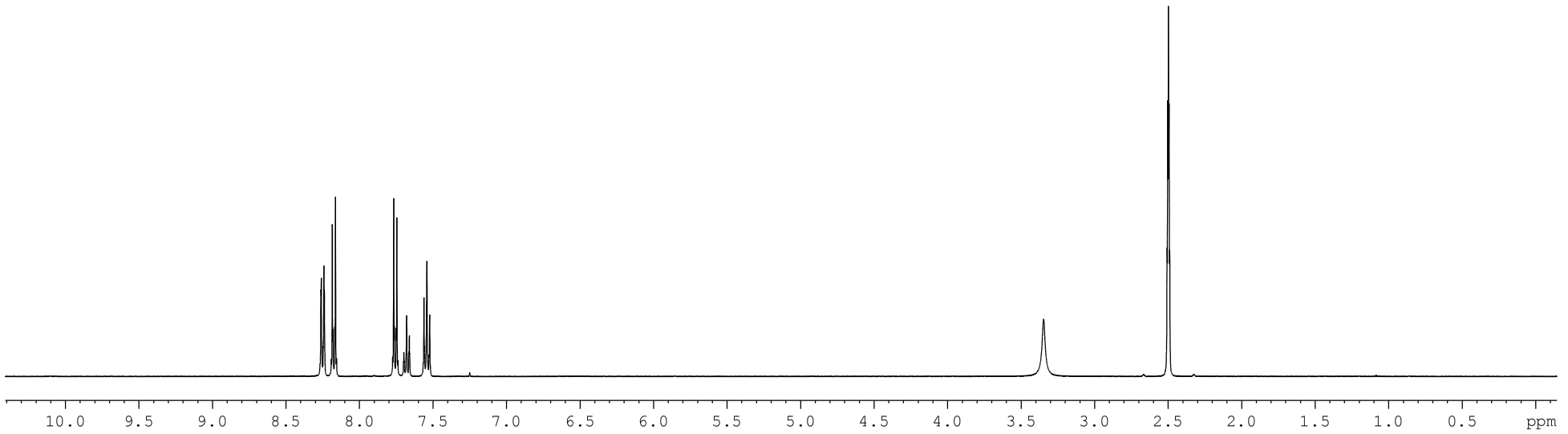
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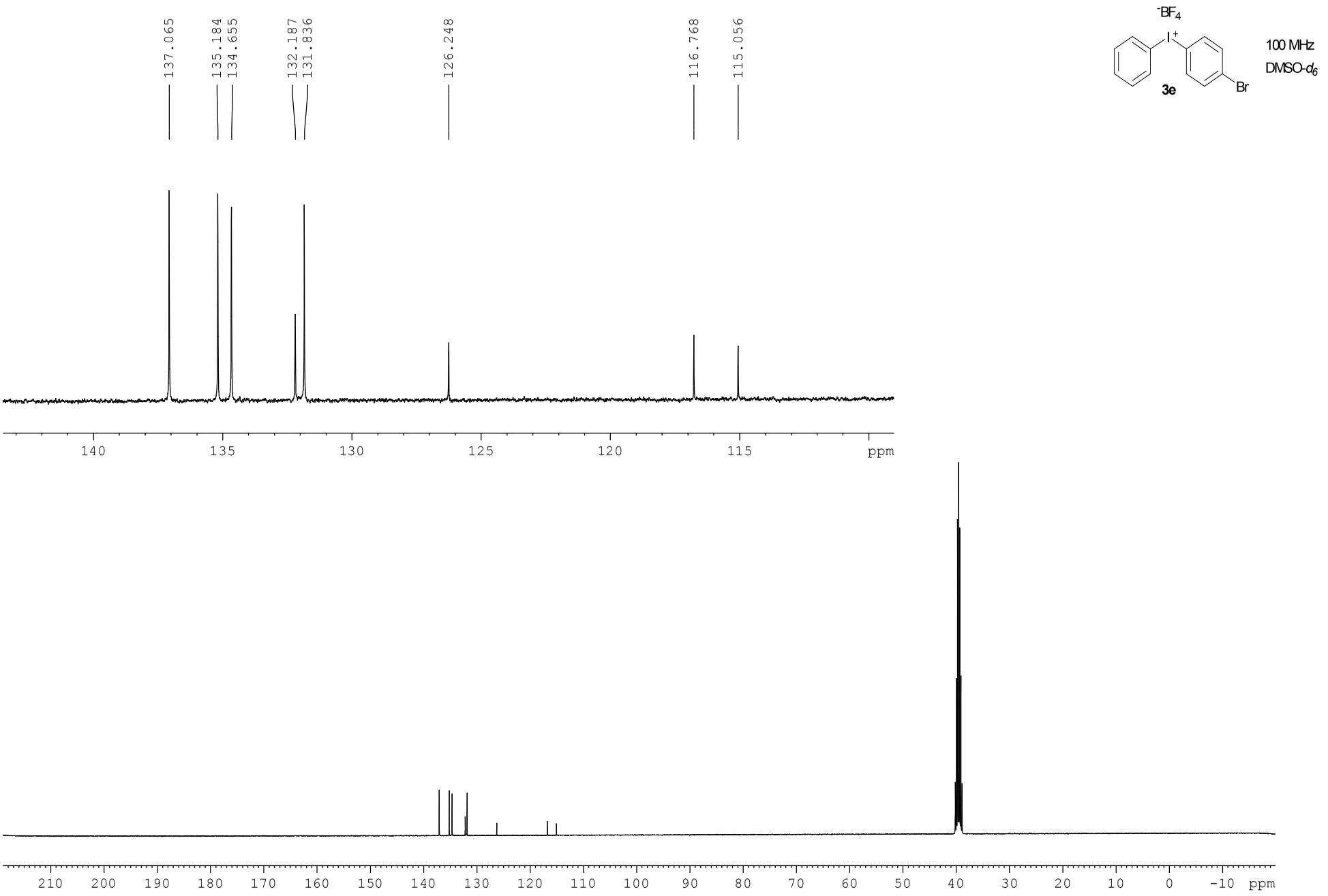
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130
125
120
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200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
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10
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-10
ppm

$^{-}BF_4$
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Br
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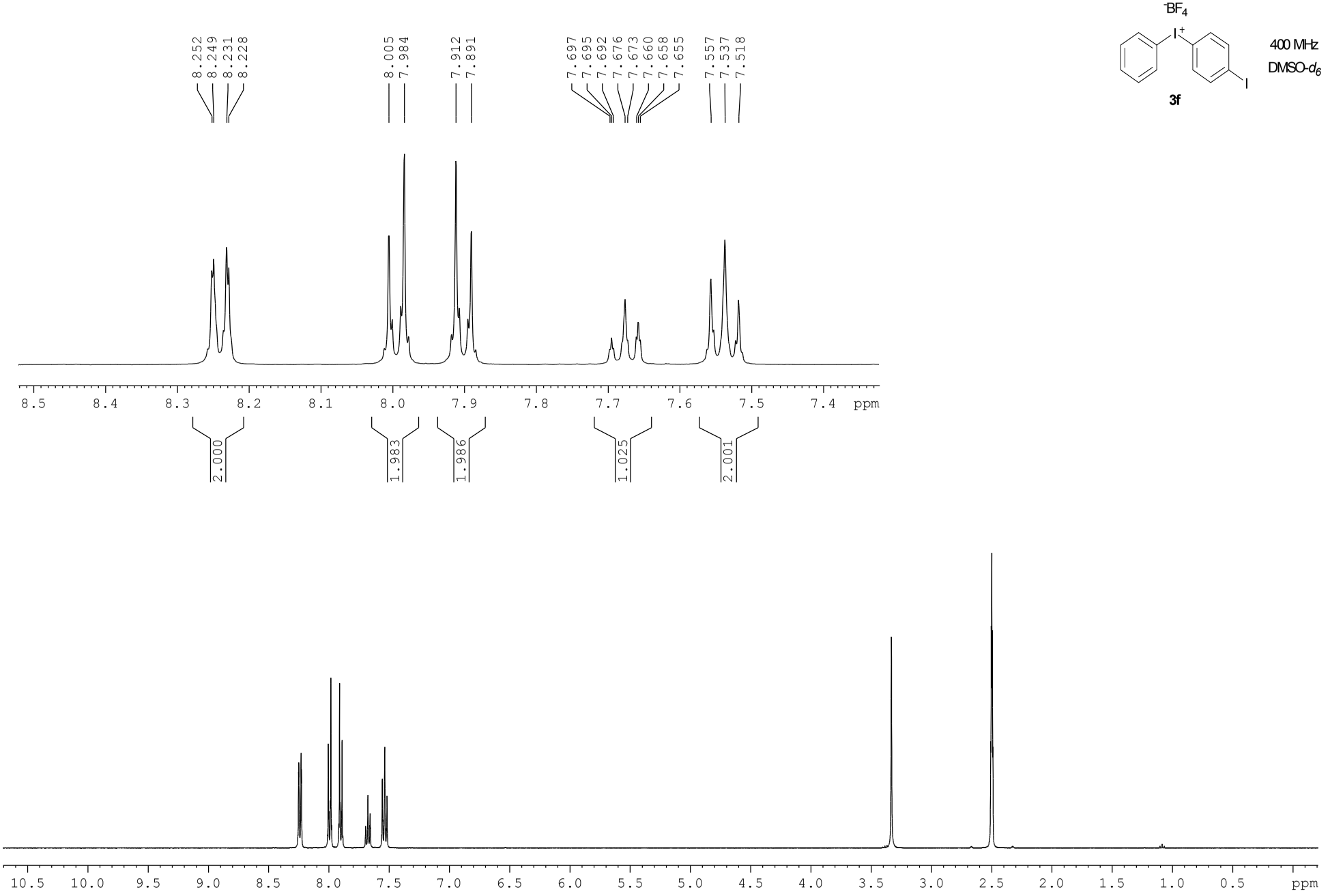
100 MHz
DMSO-d_6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7.5
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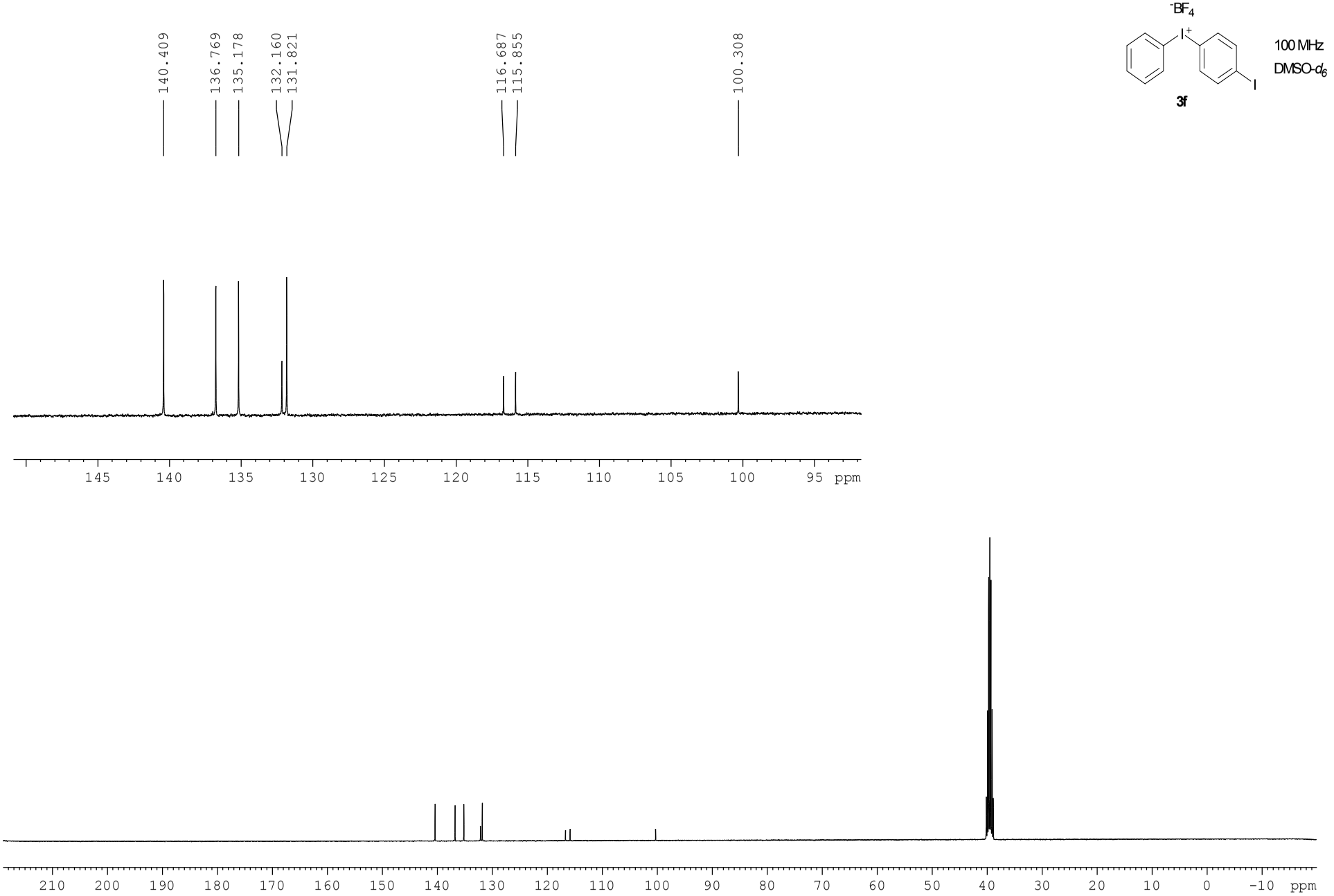
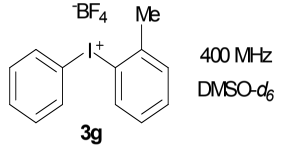
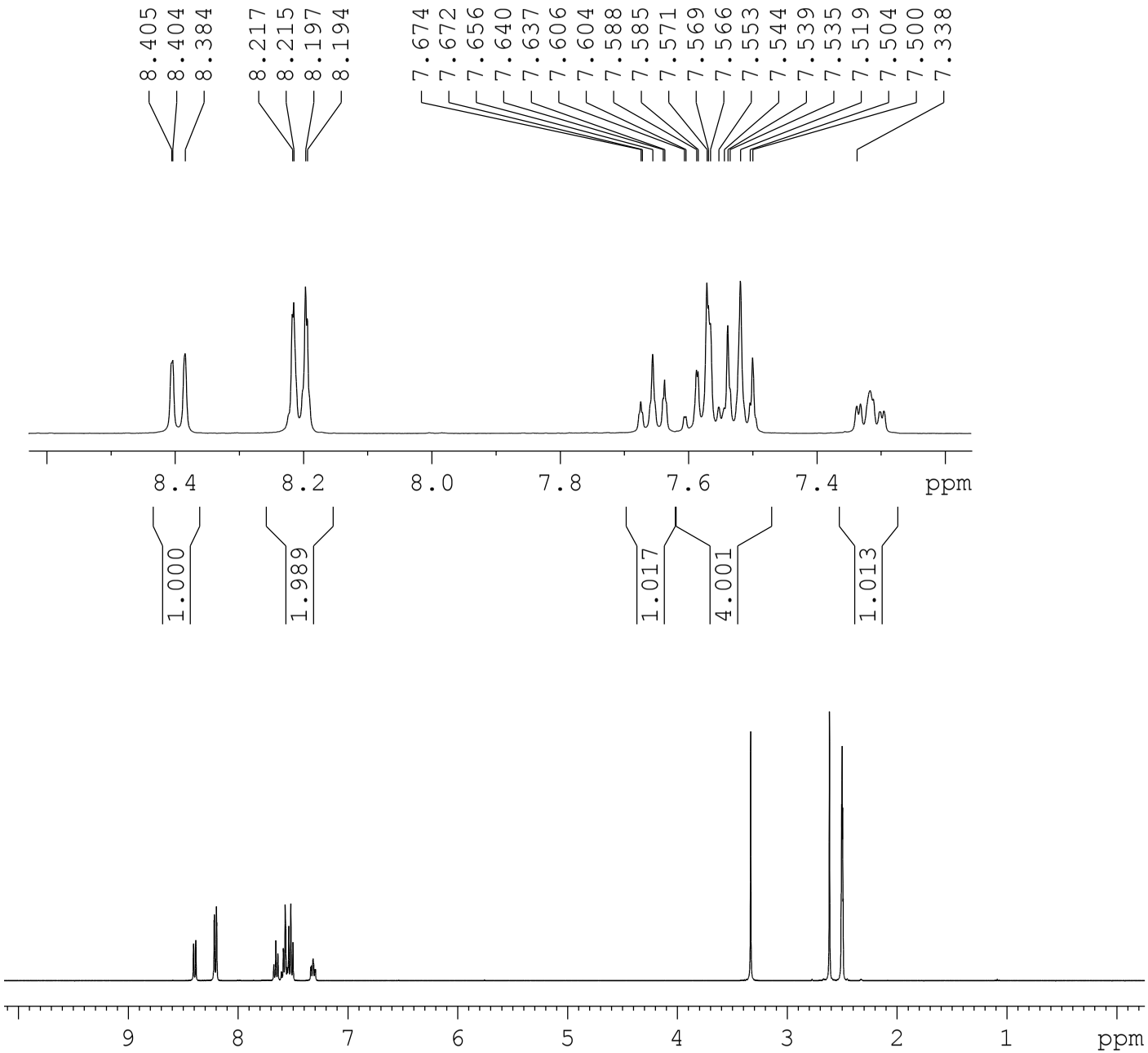
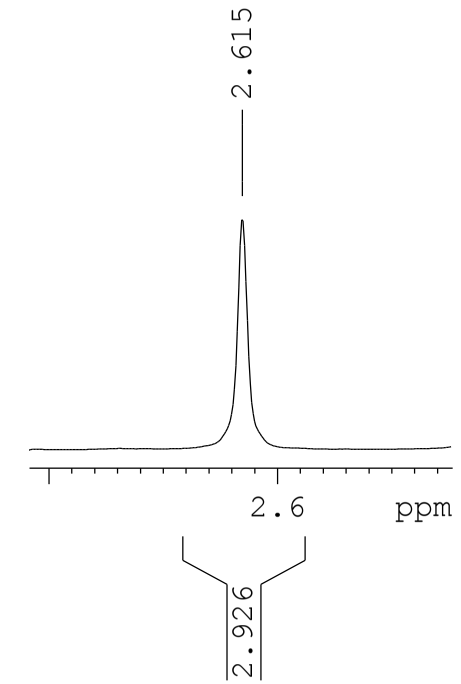
2.000
1.983
1.986
1.025
2.001
$^{-}BF_4$
$^{+}$
I
I
3f
400 MHz
DMSO-d_6
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

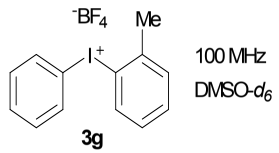

140.409
136.769
135.178
132.160
131.821
116.687
115.855
100.308
145 140 135 130 125 120 115 110 105 100 95 ppm
$^{-}BF_4$
$^{+}$
I
100 MHz
DMSO-d_6
3f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm



400 MHz
DMSO-d_6

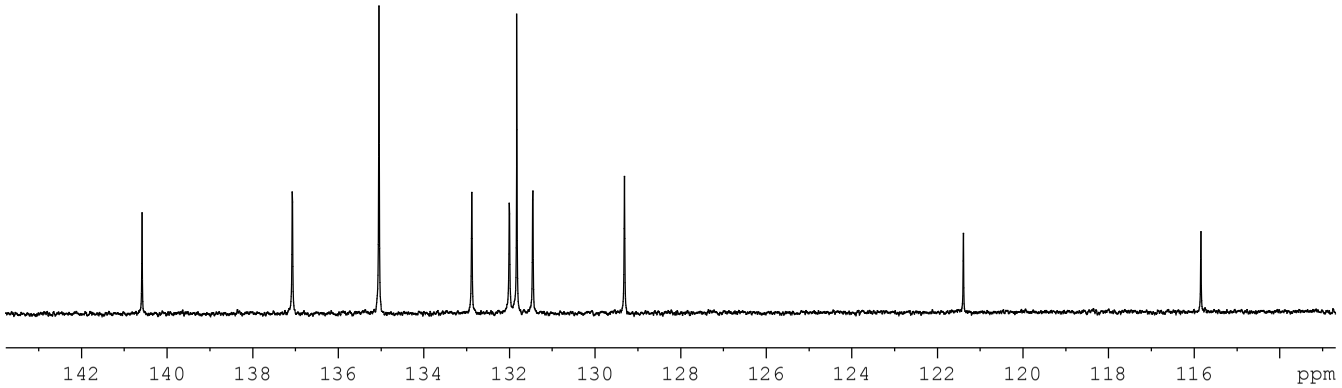




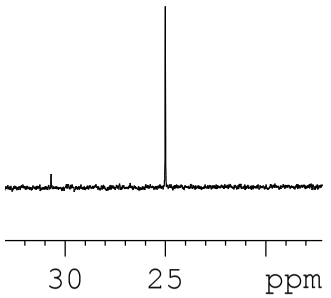


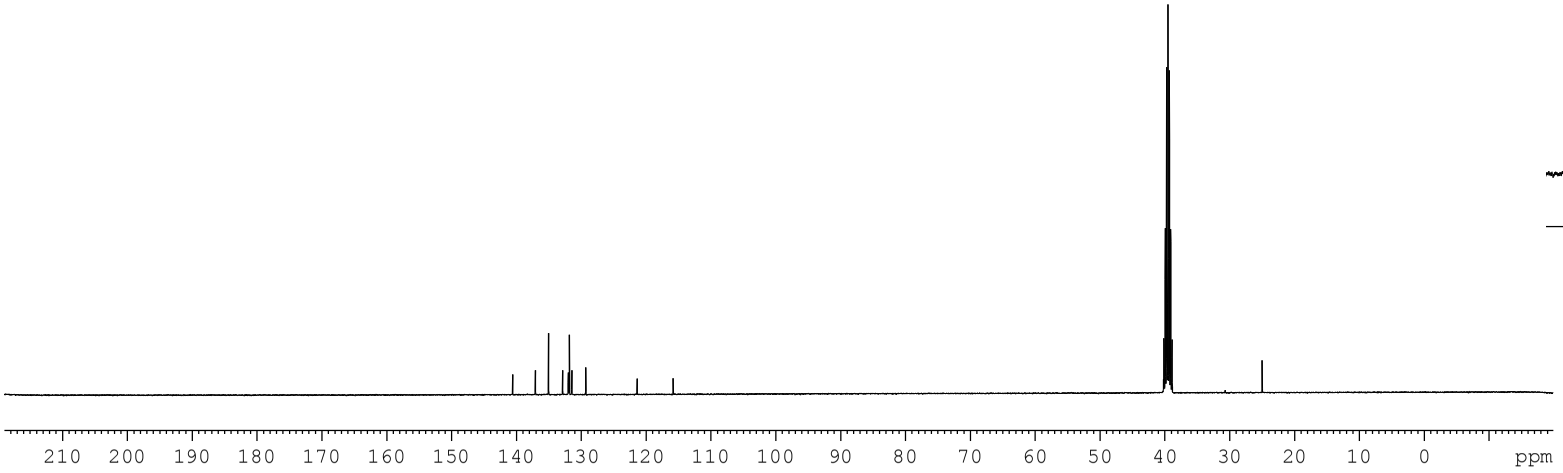
100 MHz
DMSO-d_6

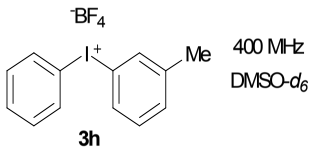
140.587
137.077
135.052
132.880
132.002
131.829
131.453
129.314
121.390
115.841

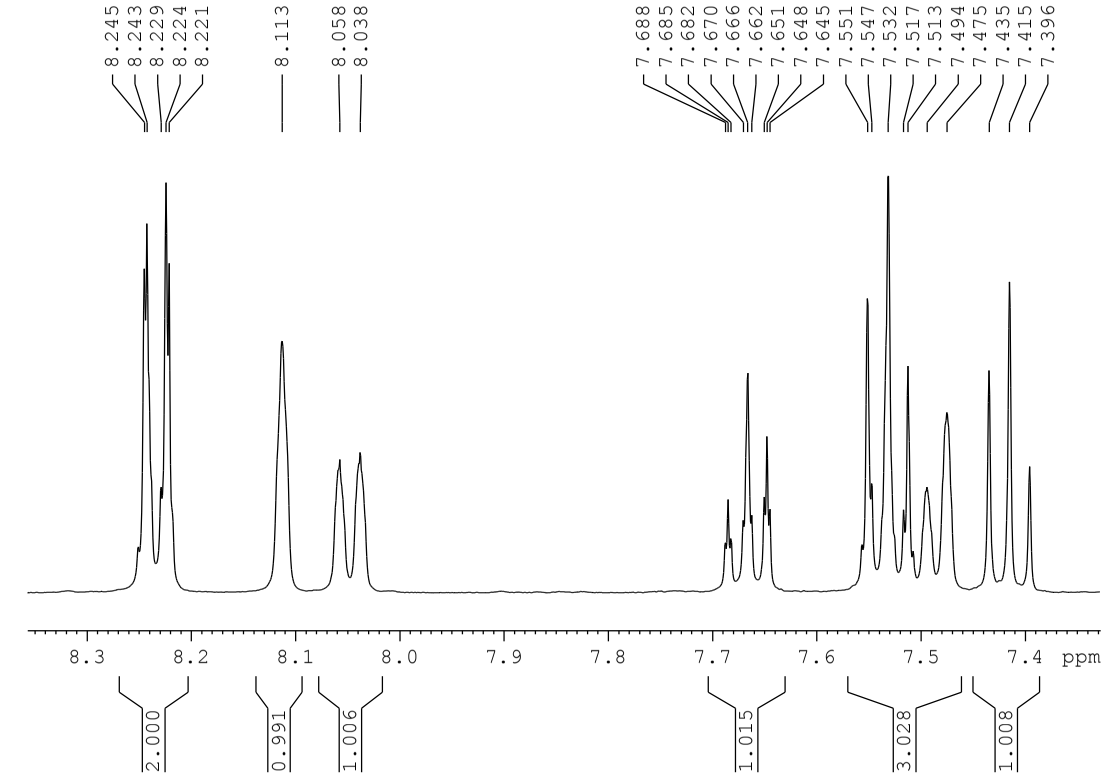


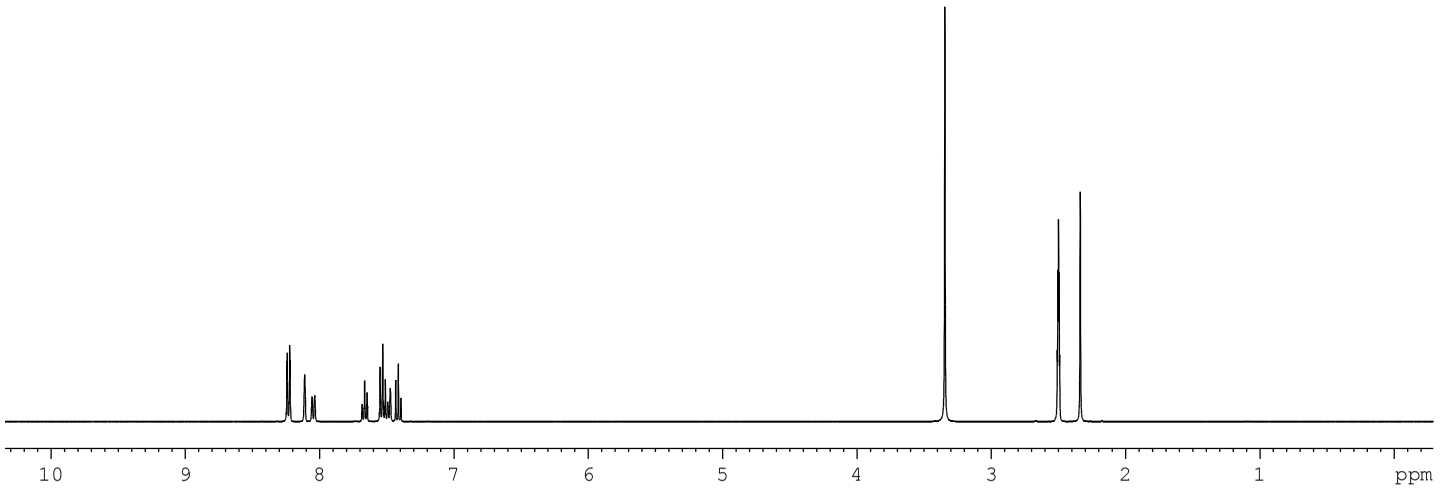
25.004

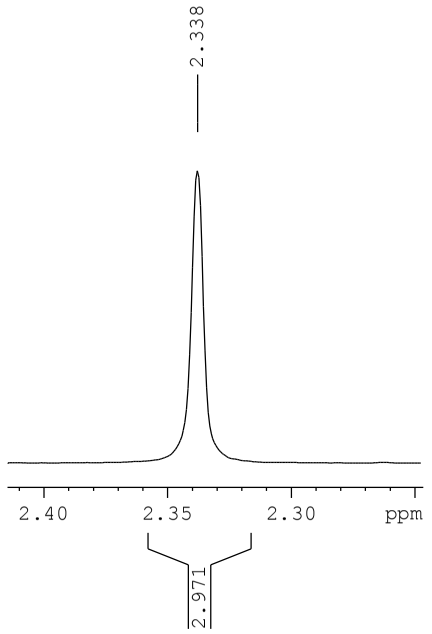


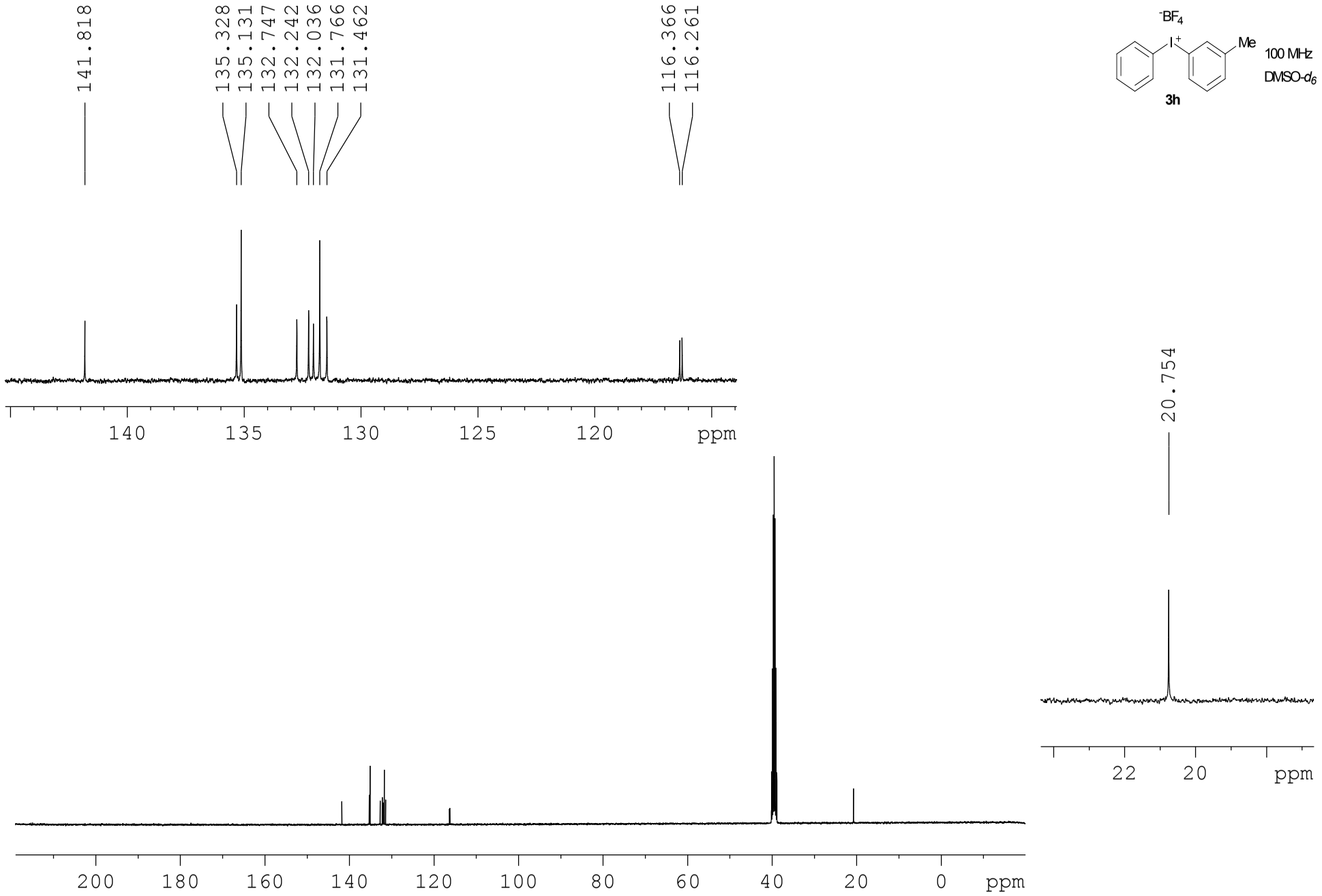



$^{-}BF_4$
Me
400 MHz
DMSO-d_6
3h

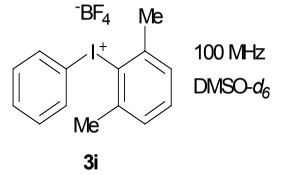

8.245
8.243
8.229
8.224
8.221
8.113
8.058
8.038
7.688
7.685
7.682
7.670
7.666
7.662
7.651
7.648
7.645
7.551
7.547
7.532
7.517
7.513
7.494
7.475
7.435
7.415
7.396
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
ppm
2.000
0.991
1.006
1.015
3.028
1.008


10
9
8
7
6
5
4
3
2
1
ppm


2.338
2.40
2.35
2.30
ppm
2.971

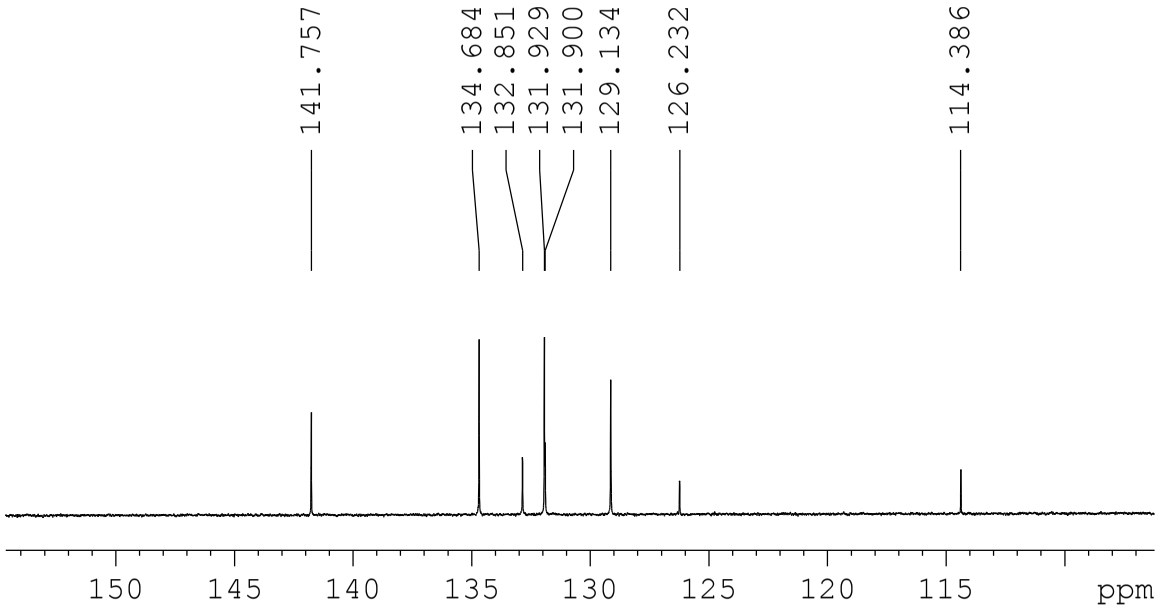

141.818
135.328
135.131
132.747
132.242
132.036
131.766
131.462
116.366
116.261
20.754
$^{-}BF_4$
Me
100 MHz
DMSO-d_6
3h
140
135
130
125
120
ppm
22
20
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm

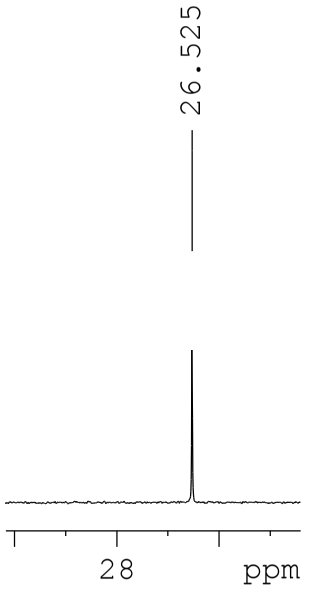
10
9
8
7
6
5
4
3
2
1
ppm
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
ppm
2.000
1.010
3.035
2.000
8.024
8.021
8.003
8.000
7.667
7.664
7.662
7.650
7.646
7.642
7.630
7.627
7.625
7.528
7.525
7.509
7.493
7.490
7.473
7.410
7.391
2.70
2.65
ppm
5.944
2.652
Me
Me
3i
$^{-}BF_4$
400 MHz
DMSO-d_6

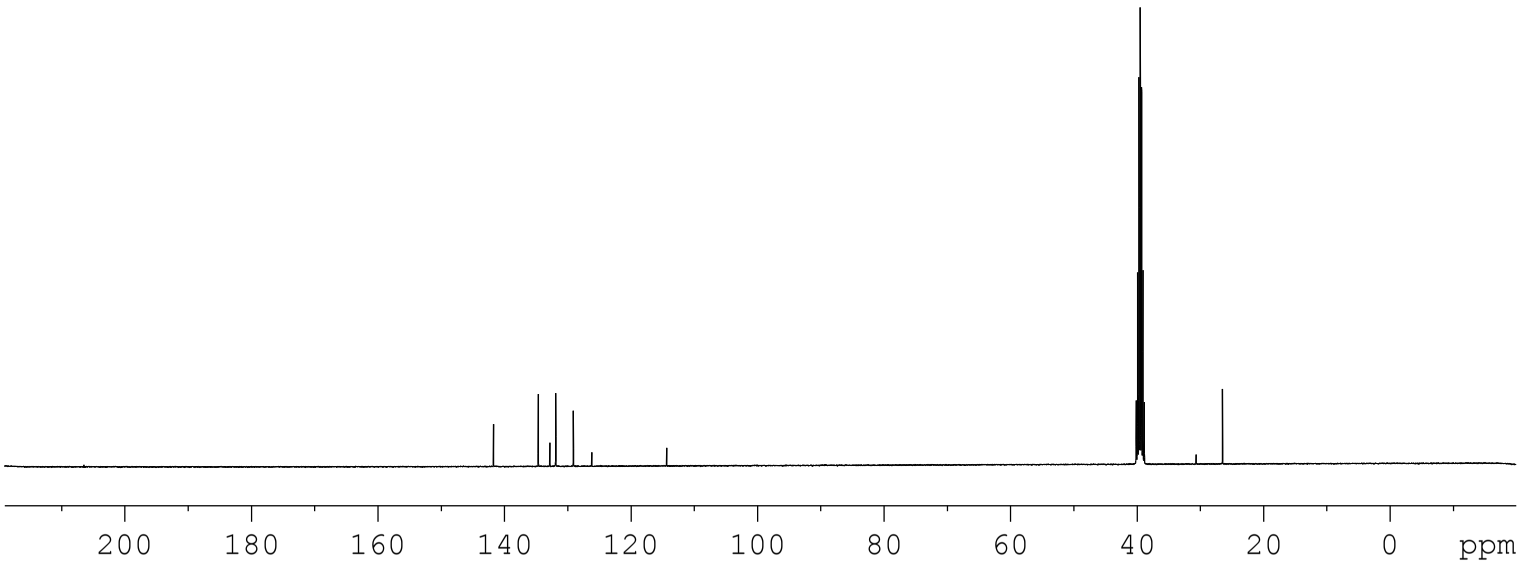


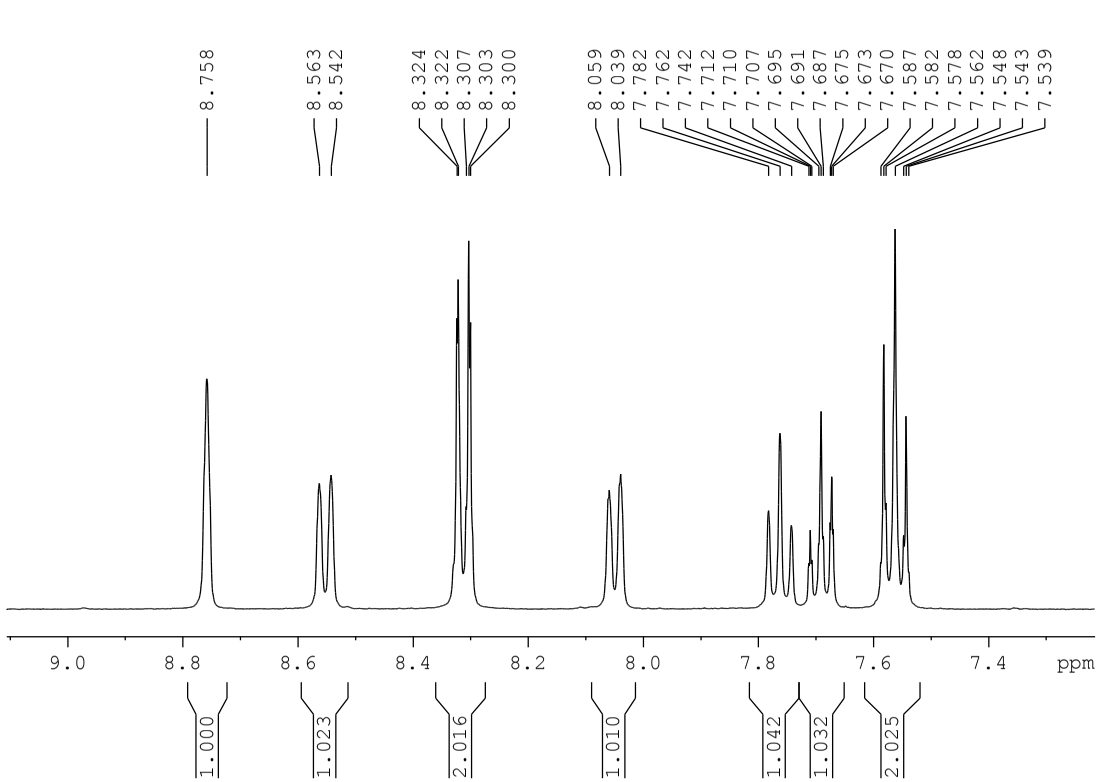
100 MHz
DMSO-d_6

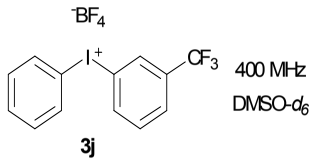
3i

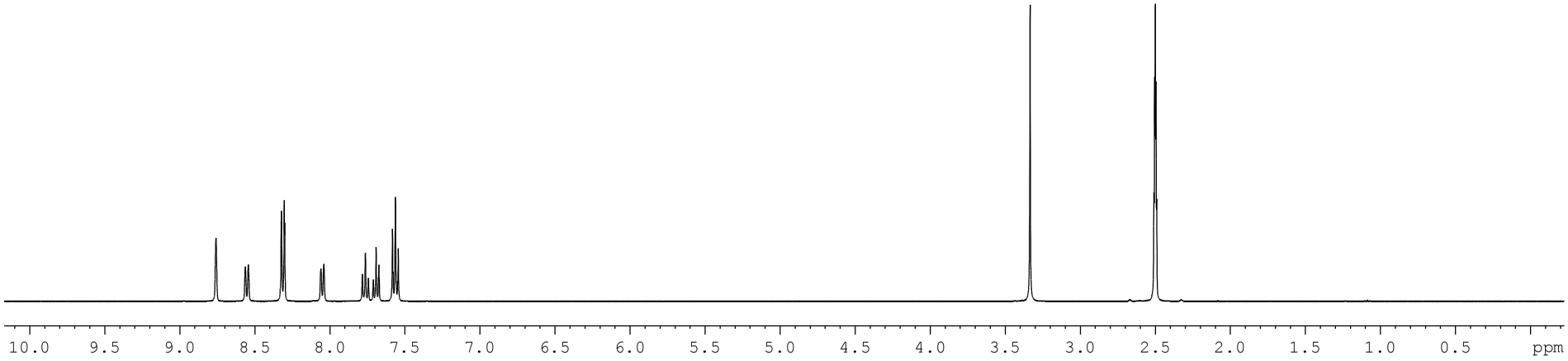


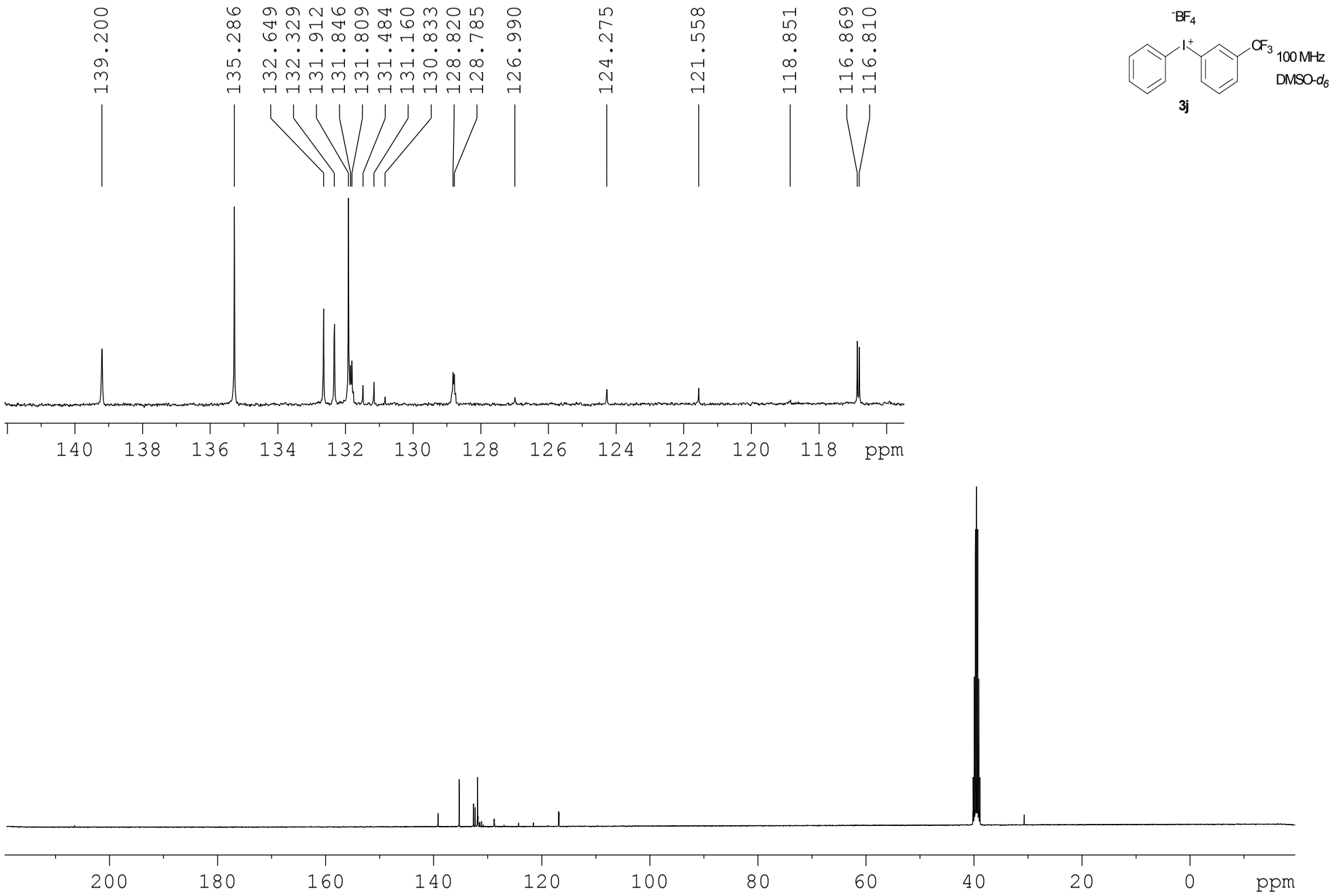


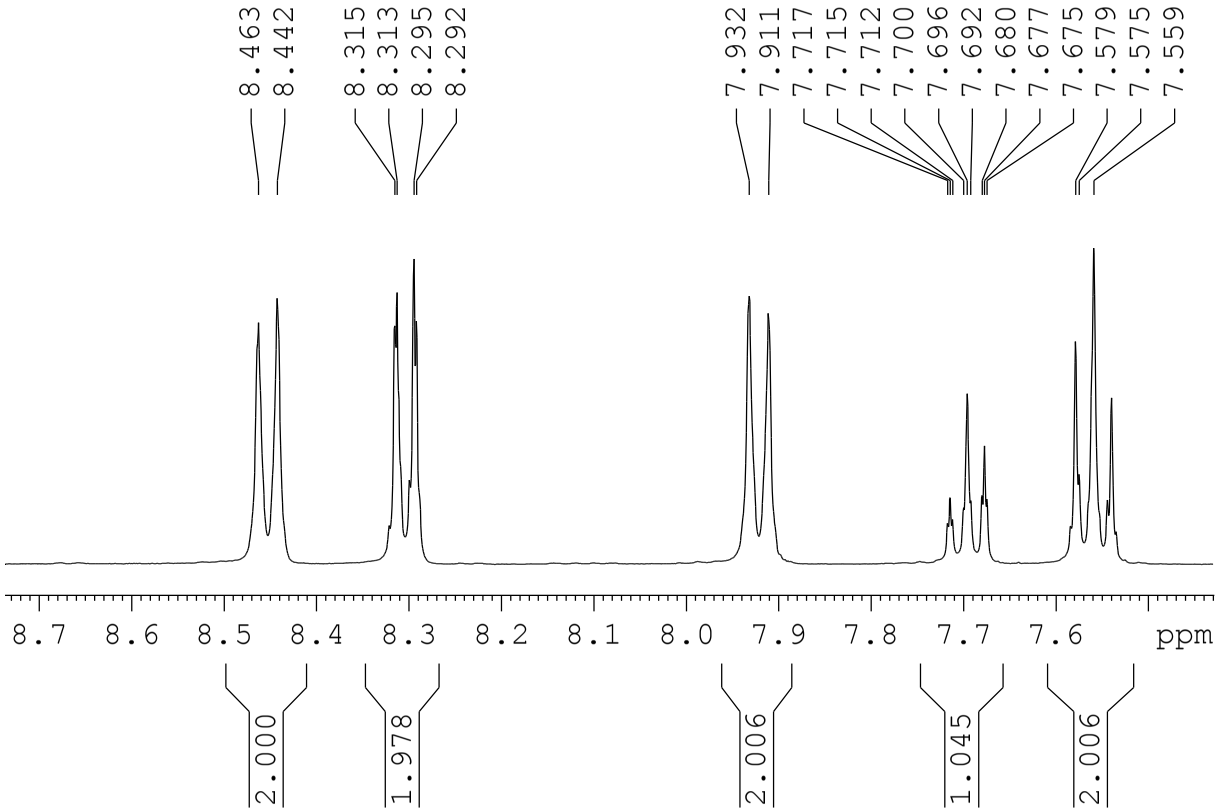


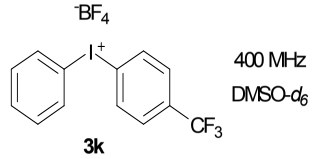

8.758
8.563
8.542
8.324
8.322
8.307
8.303
8.300
8.059
8.039
7.782
7.762
7.742
7.712
7.710
7.707
7.695
7.691
7.687
7.675
7.673
7.670
7.587
7.582
7.578
7.562
7.548
7.543
7.539
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
ppm
1.000
1.023
2.016
1.010
1.042
1.032
2.025

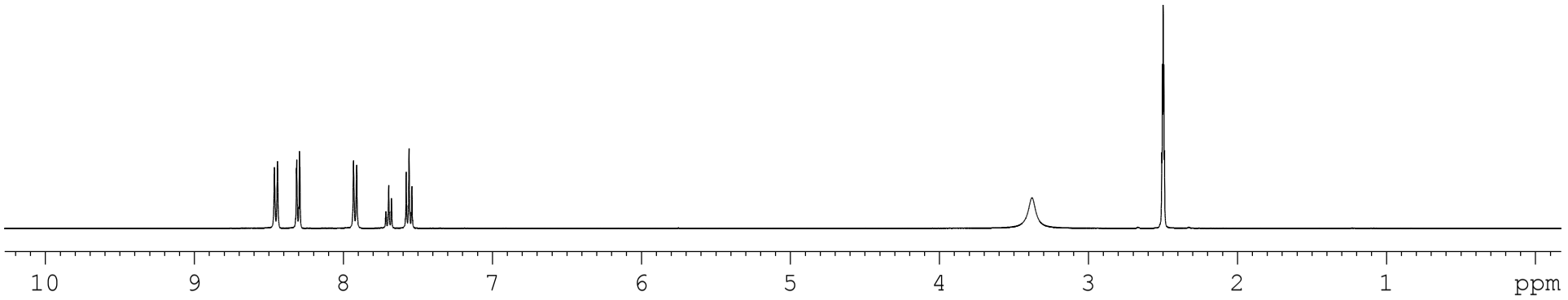

$^{-}BF_4$
I^{+}
CF_3
400 MHz
DMSO-d_6
3j

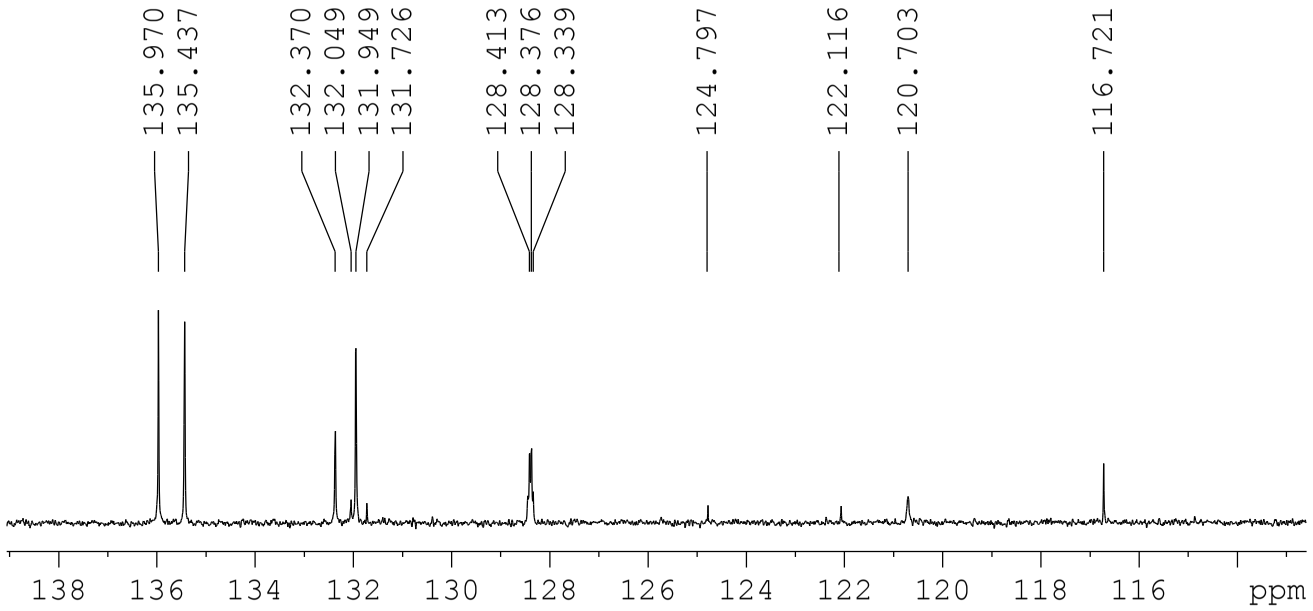

10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

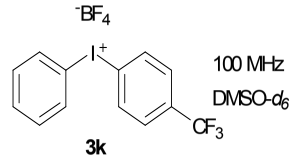

139.200
135.286
132.649
132.329
131.912
131.846
131.809
131.484
131.160
130.833
128.820
128.785
126.990
124.275
121.558
118.851
116.869
116.810
$^{-}BF_4$
I^{+}
CF_3
100 MHz
DMSO-d_6
3j
140 138 136 134 132 130 128 126 124 122 120 118 ppm
200 180 160 140 120 100 80 60 40 20 0 ppm

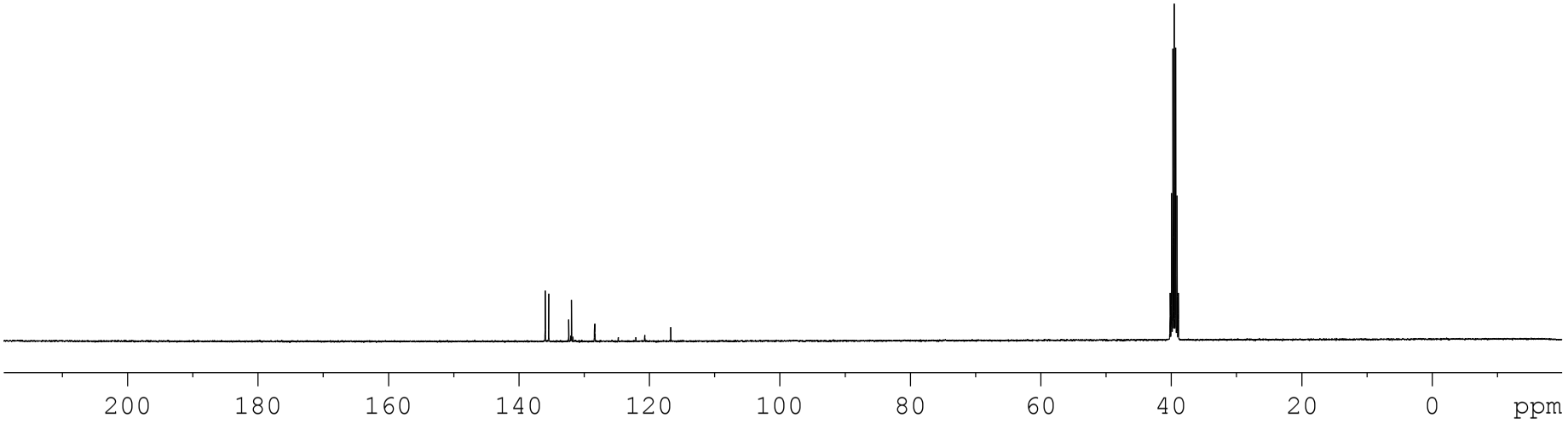

8.463
8.442
8.315
8.313
8.295
8.292
7.932
7.911
7.717
7.715
7.712
7.700
7.696
7.692
7.680
7.677
7.675
7.579
7.575
7.559
8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 ppm
2.000
1.978
2.006
1.045
2.006

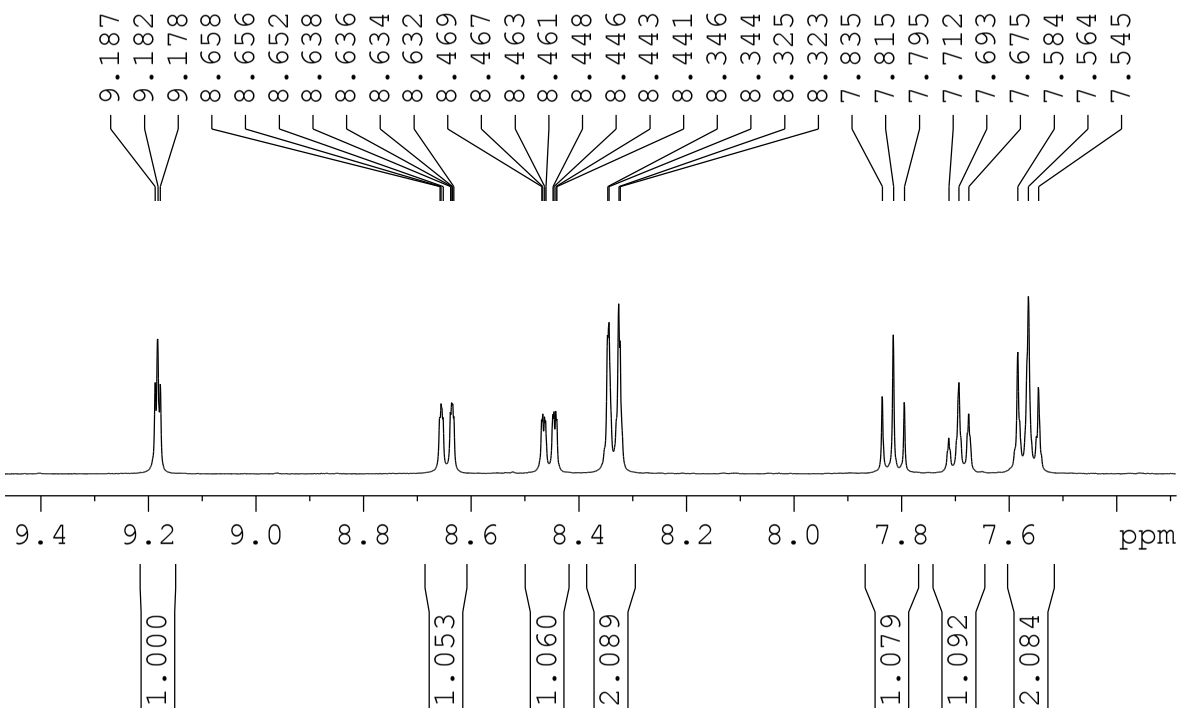

$^{-}BF_4$
$^{+}$
400 MHz
DMSO-d_6
CF_3
3k

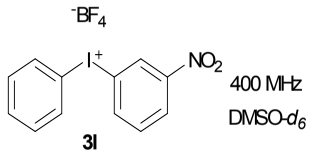

10 9 8 7 6 5 4 3 2 1 ppm

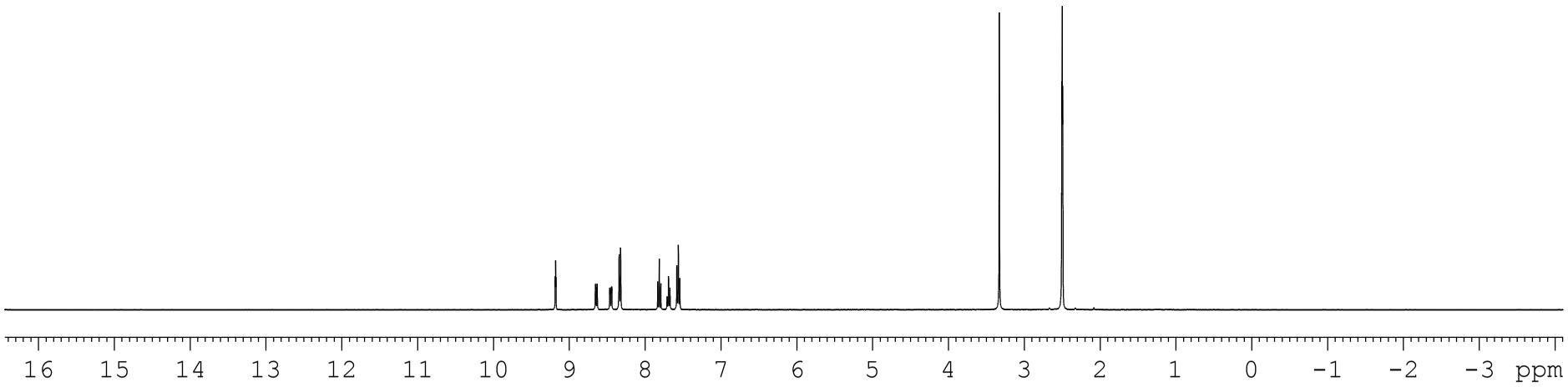

135.970
135.437
132.370
132.049
131.949
131.726
128.413
128.376
128.339
124.797
122.116
120.703
116.721
138
136
134
132
130
128
126
124
122
120
118
116
ppm

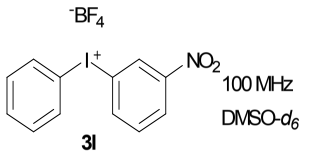

$^{-}BF_4$
$^{+}$
100 MHz
DMSO-d_6
CF_3
3k


200
180
160
140
120
100
80
60
40
20
0
ppm

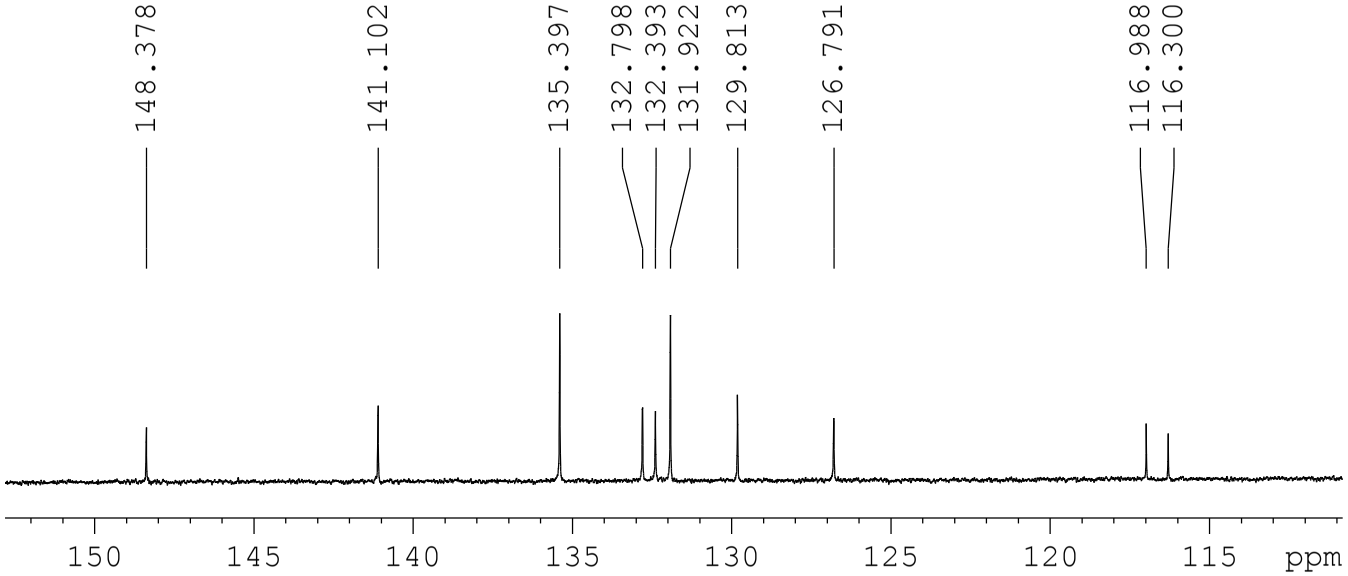

9.187
9.182
9.178
8.658
8.656
8.652
8.638
8.636
8.634
8.632
8.469
8.467
8.463
8.461
8.448
8.446
8.443
8.441
8.346
8.344
8.325
8.323
7.835
7.815
7.795
7.712
7.693
7.675
7.584
7.564
7.545
9.4
9.2
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
ppm
1.000
1.053
1.060
2.089
1.079
1.092
2.084

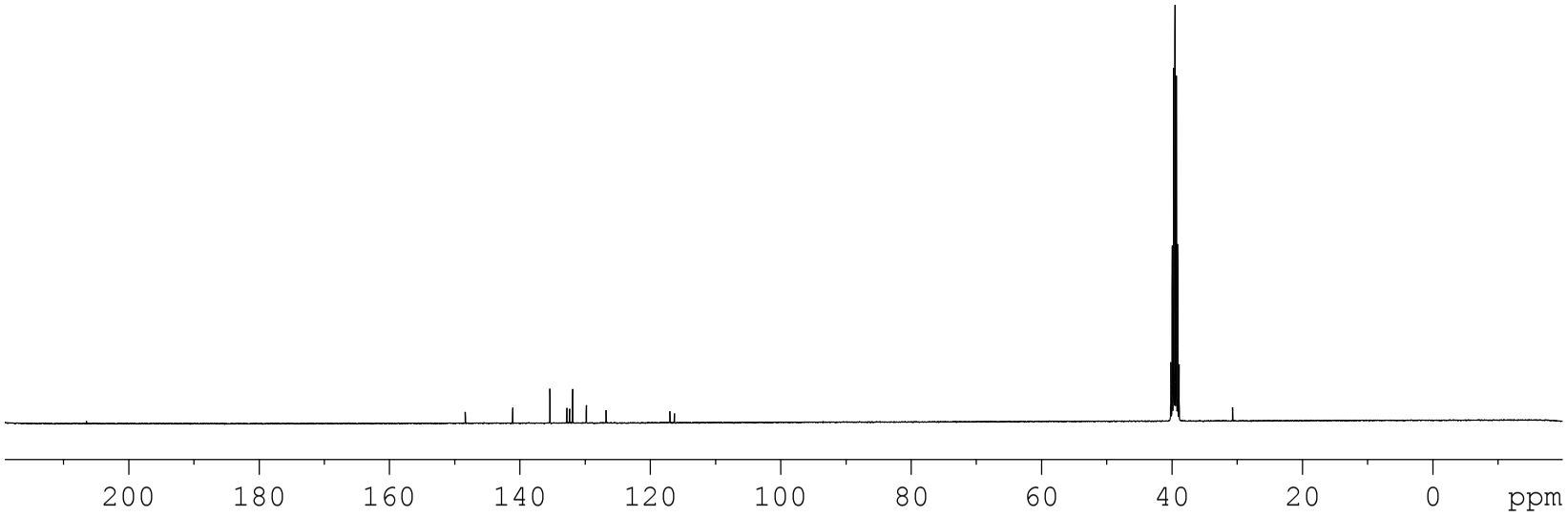

-BF4
I+
NO2
3l
400 MHz
DMSO-d6

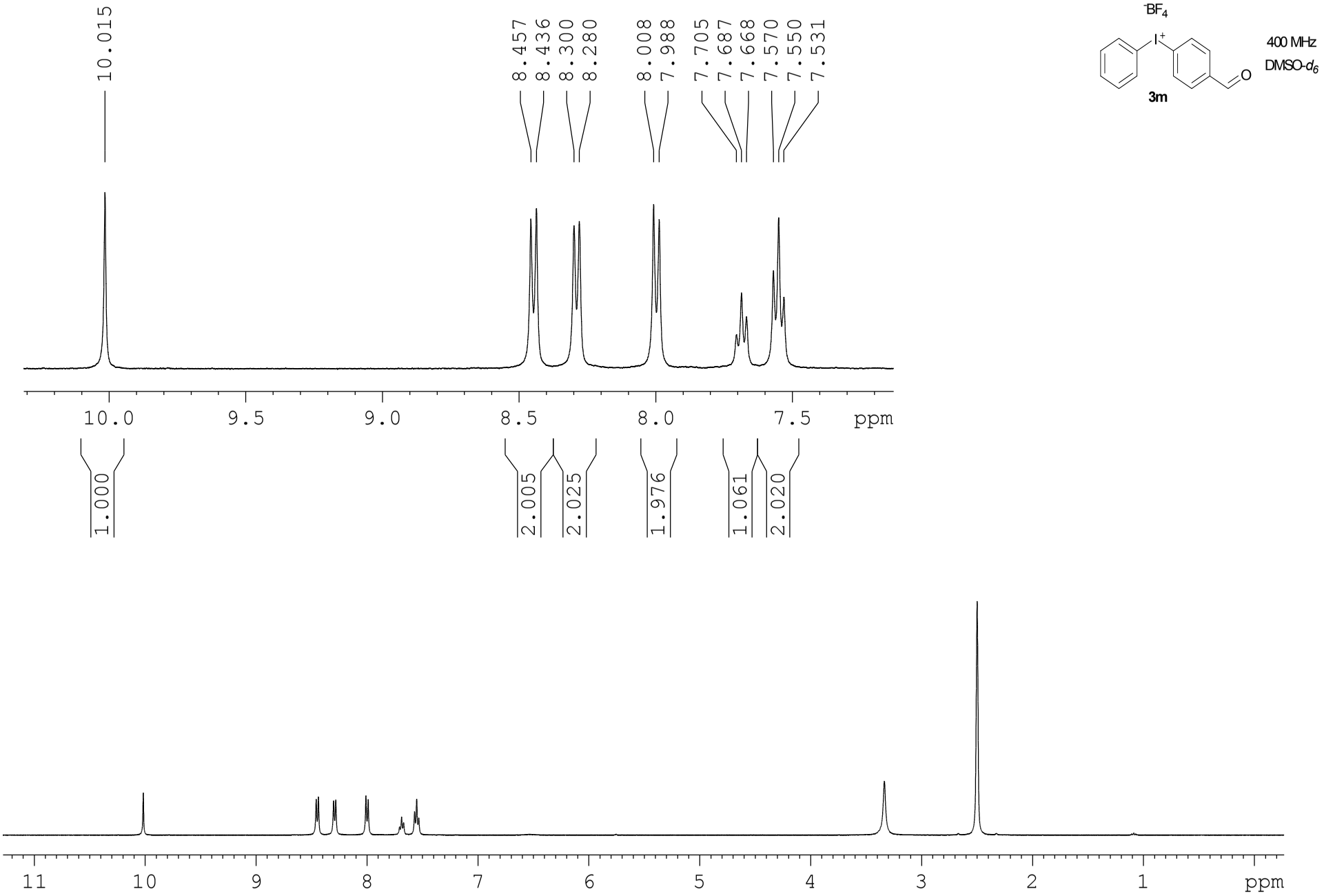

16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
ppm

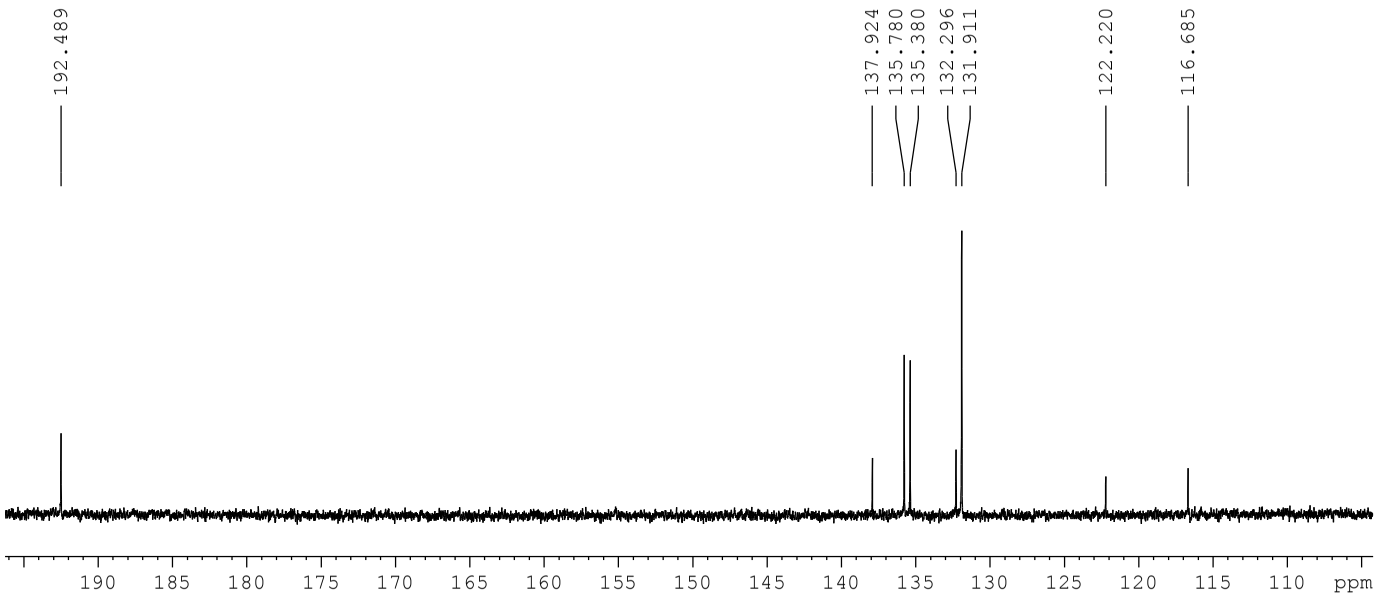


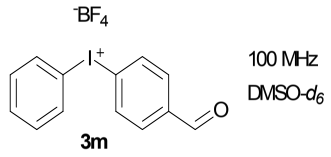
100 MHz
DMSO-d_6

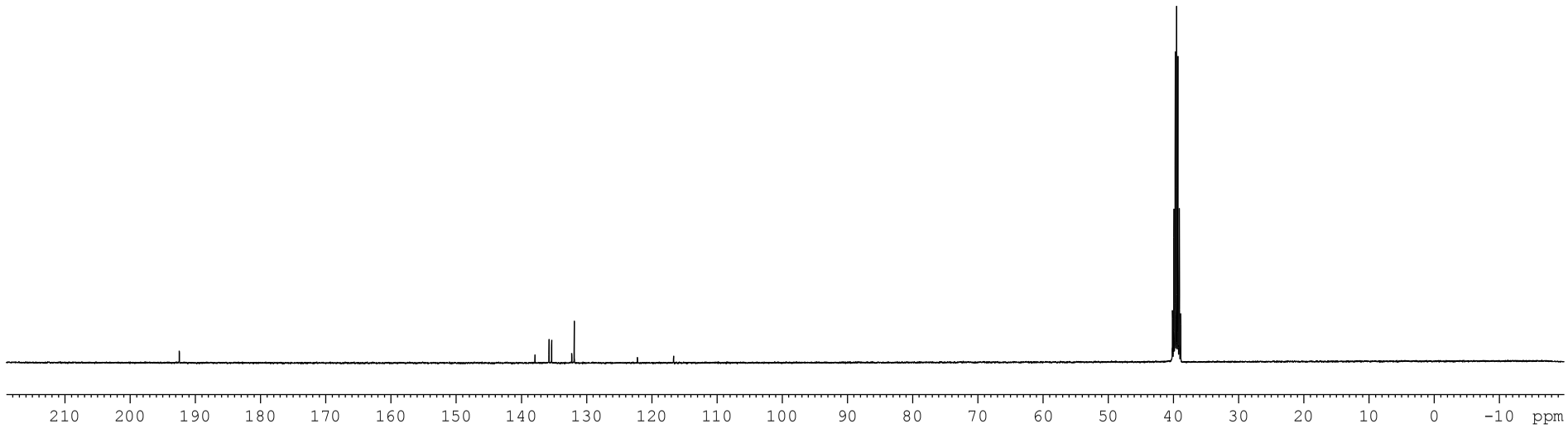


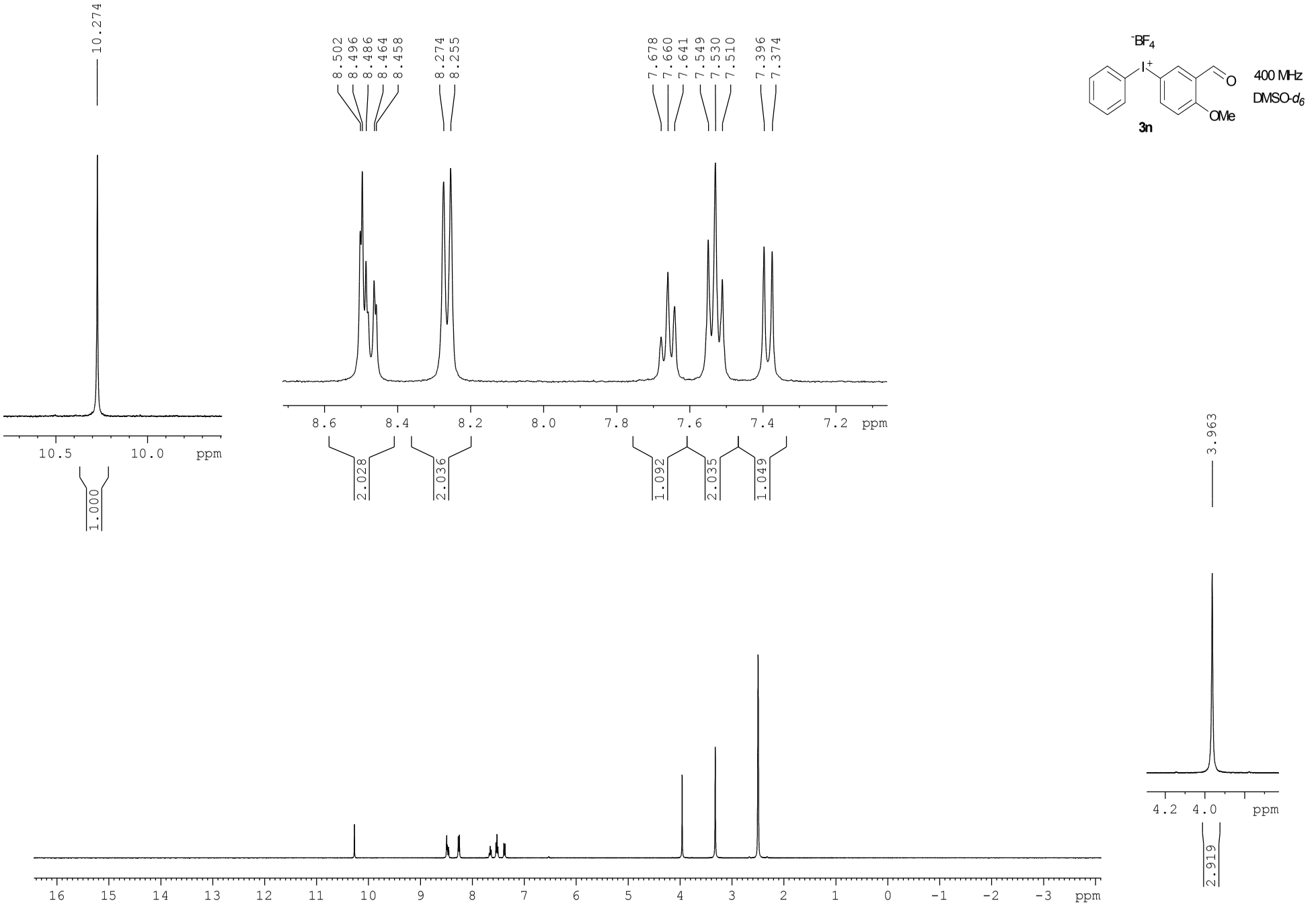


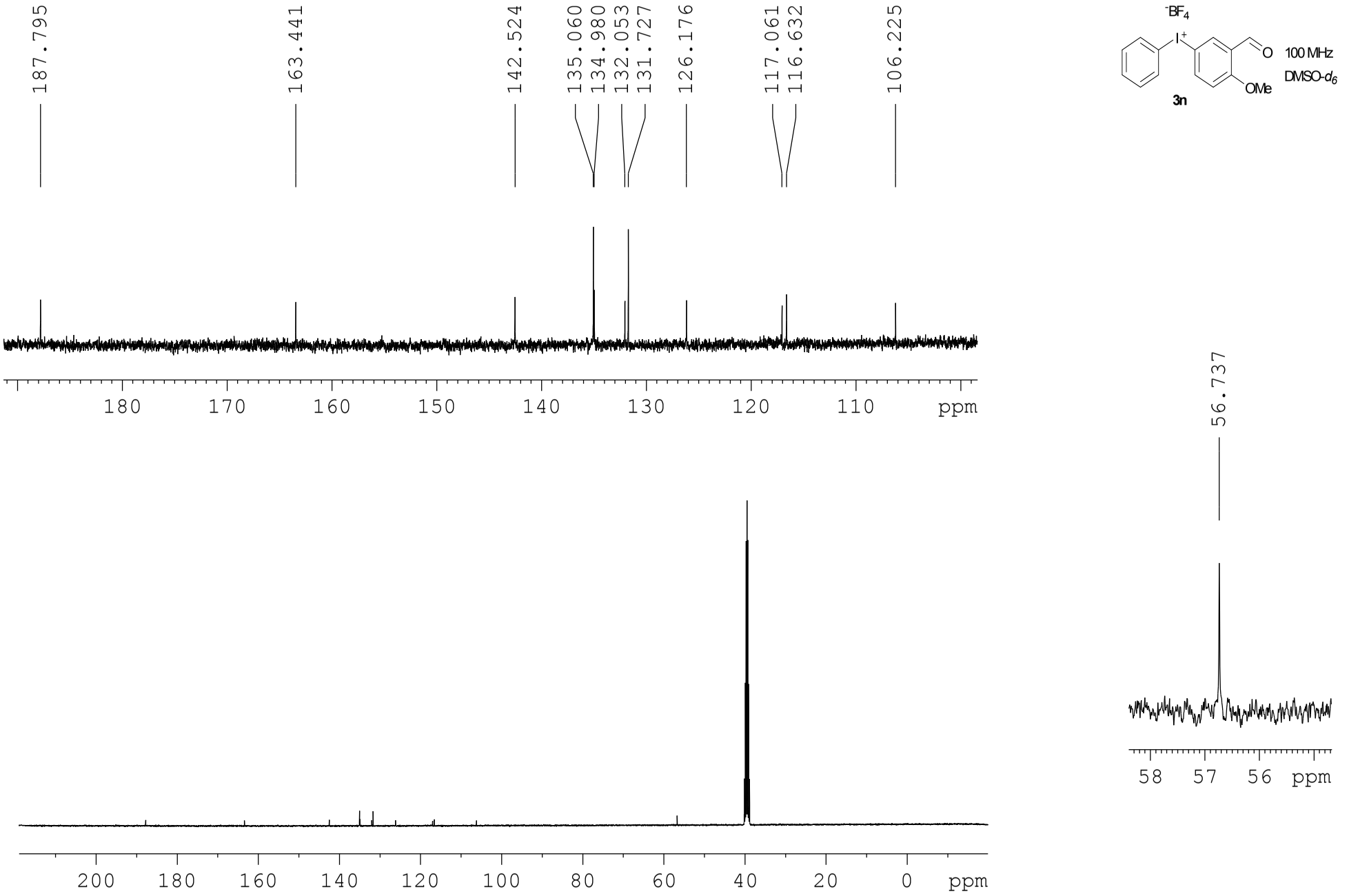

10.015
8.457
8.436
8.300
8.280
8.008
7.988
7.705
7.687
7.668
7.570
7.550
7.531
10.0
9.5
9.0
8.5
8.0
7.5
ppm
1.000
2.005
2.025
1.976
1.061
2.020
11
10
9
8
7
6
5
4
3
2
1
ppm
$^{-}BF_4$
I^{+}
O
3m
400 MHz
DMSO-d_6

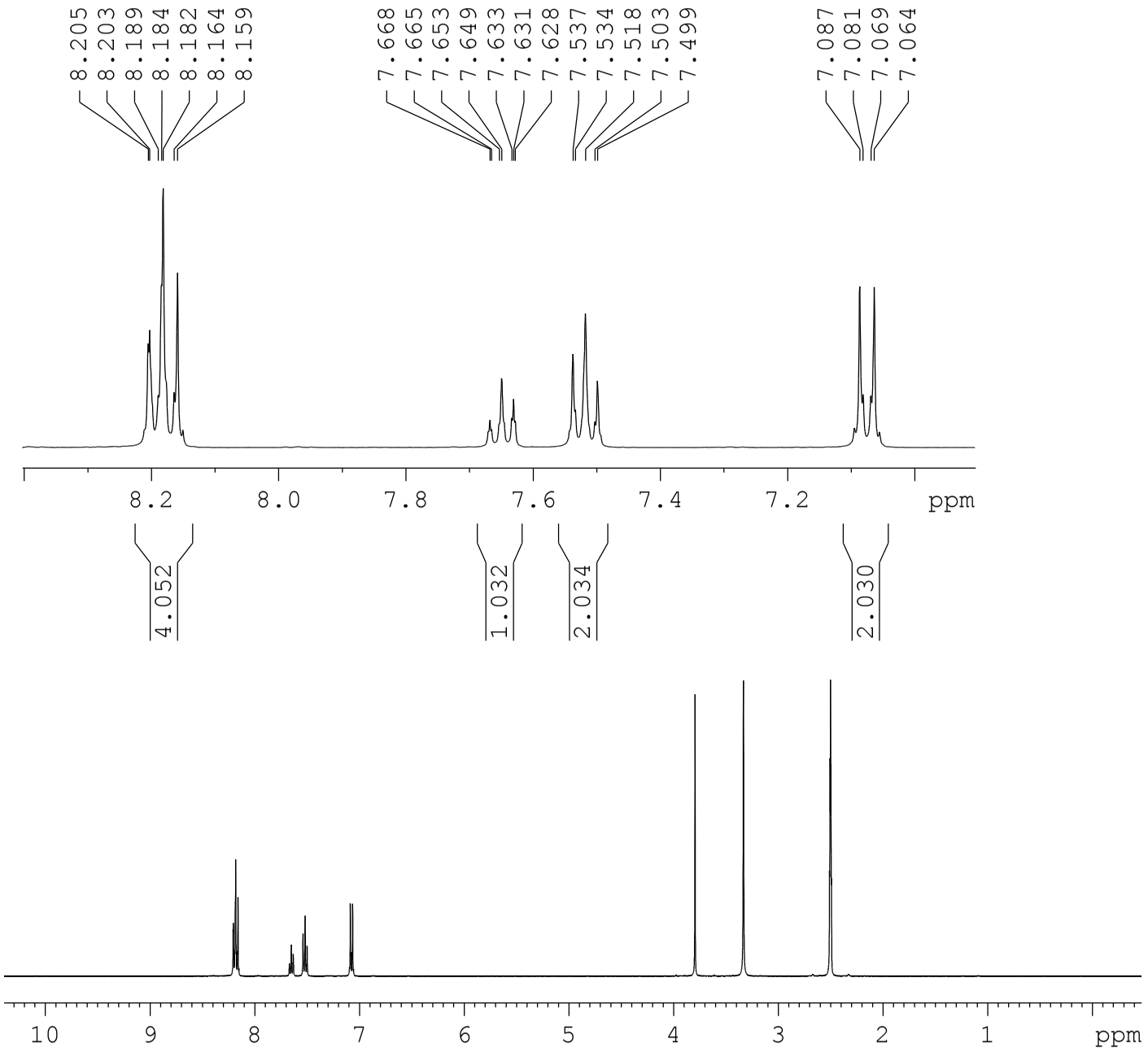

192.489
137.924
135.780
135.380
132.296
131.911
122.220
116.685
190 185 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 ppm

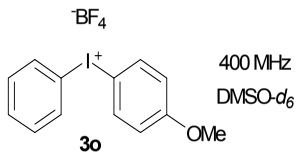

$^{-}BF_4$
$^{+}$
O
3m
100 MHz
DMSO-d_6

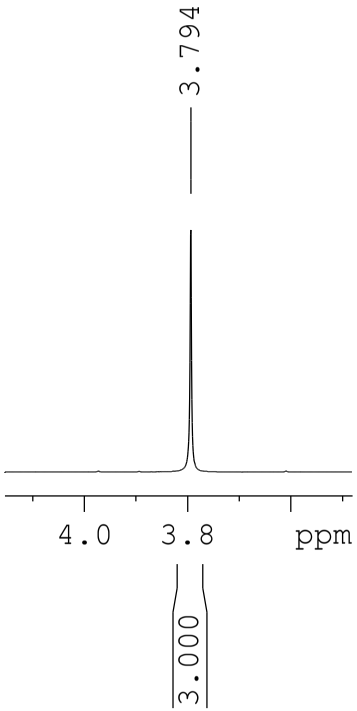

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

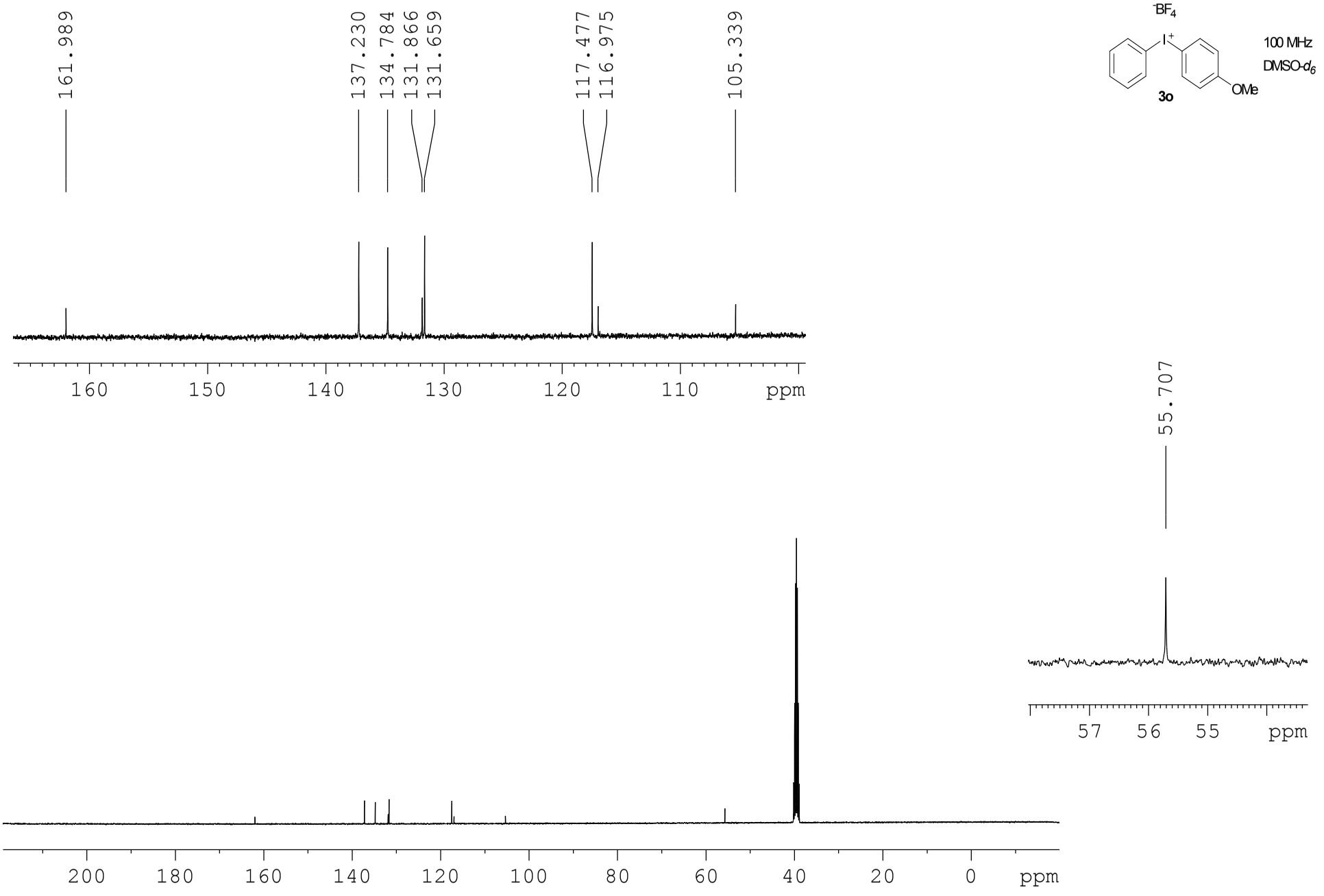

10.274
8.502
8.496
8.486
8.464
8.458
8.274
8.255
7.678
7.660
7.641
7.549
7.530
7.510
7.396
7.374
3.963
$^{-}BF_4$
I^{+}
O
OMe
3n
400 MHz
DMSO-d_6
ppm
1.000
2.028
2.036
1.092
2.035
1.049
2.919

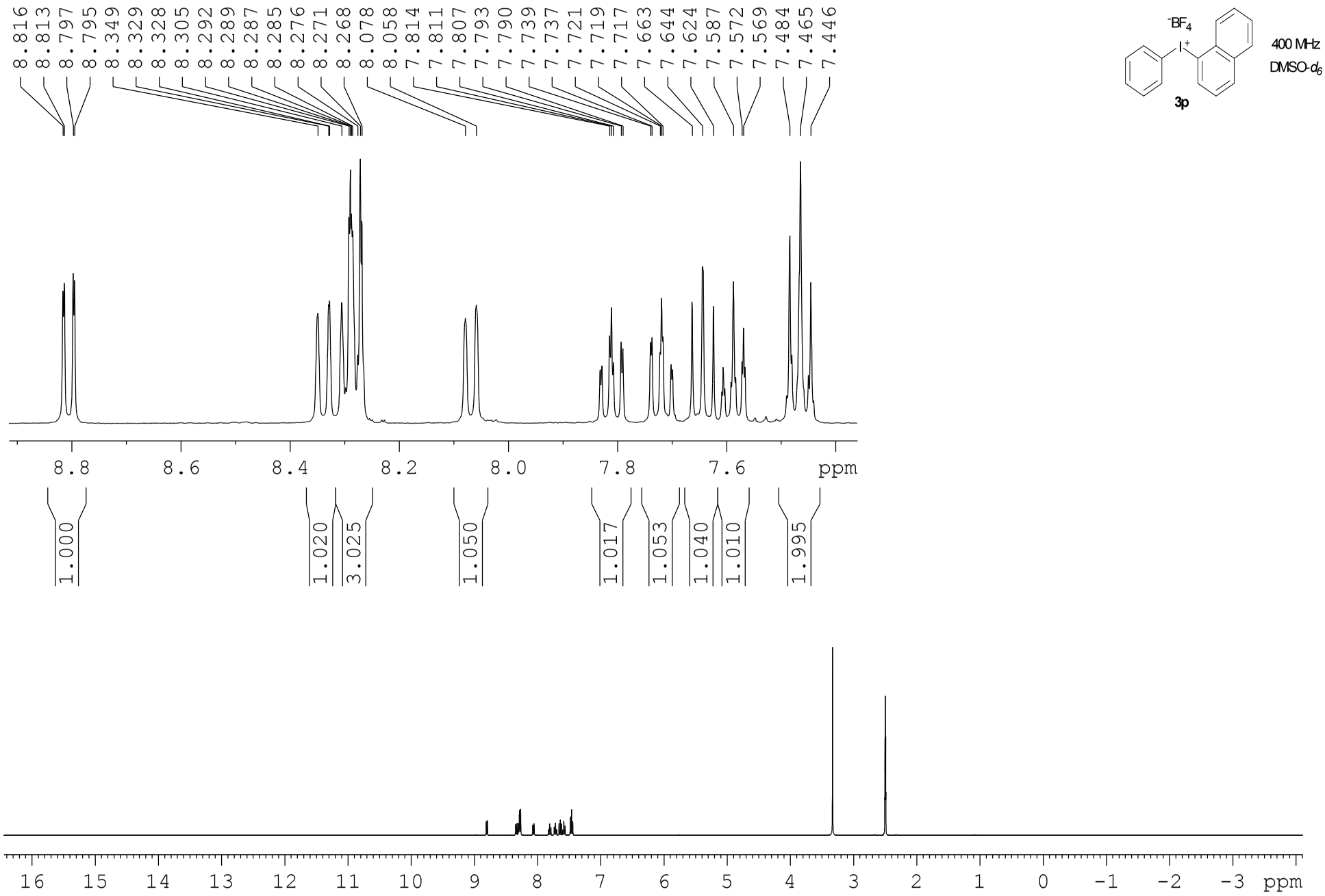

187.795
163.441
142.524
135.060
134.980
132.053
131.727
126.176
117.061
116.632
106.225
180
170
160
150
140
130
120
110
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm
56.737
58
57
56
ppm
$^{-}BF_4$
$^{+}$
O
OMe
3n
100 MHz
DMSO-d_6


8.205
8.203
8.189
8.184
8.182
8.164
8.159
7.668
7.665
7.653
7.649
7.633
7.631
7.628
7.537
7.534
7.518
7.503
7.499
7.087
7.081
7.069
7.064
8.2
8.0
7.8
7.6
7.4
7.2
ppm
4.052
1.032
2.034
2.030
10
9
8
7
6
5
4
3
2
1
ppm

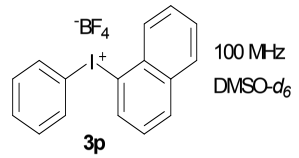

$^{-}BF_4$
I^{+}
400 MHz
DMSO-d_6
OMe
3o


3.794
4.0
3.8
ppm
3.000


161.989
137.230
134.784
131.866
131.659
117.477
116.975
105.339
160
150
140
130
120
110
ppm
$^{-}BF_4$
$^{+}$
OMe
3o
100 MHz
DMSO-d_6
55.707
57
56
55
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm


8.816
8.813
8.797
8.795
8.349
8.329
8.328
8.305
8.292
8.289
8.287
8.285
8.276
8.271
8.268
8.078
8.058
7.814
7.811
7.807
7.793
7.790
7.739
7.737
7.721
7.719
7.717
7.663
7.644
7.624
7.587
7.572
7.569
7.484
7.465
7.446
8.8
8.6
8.4
8.2
8.0
7.8
7.6
ppm
1.000
1.020
3.025
1.050
1.017
1.053
1.040
1.010
1.995
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
ppm

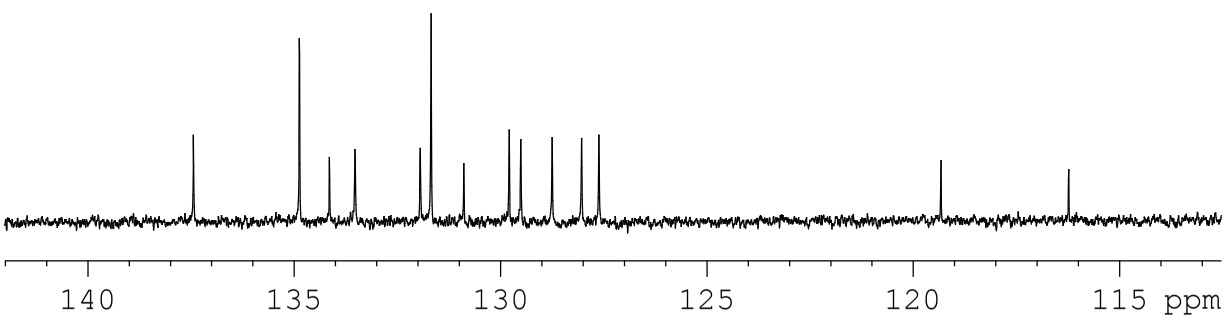
BF4
I+
3p
400 MHz
DMSO-d6

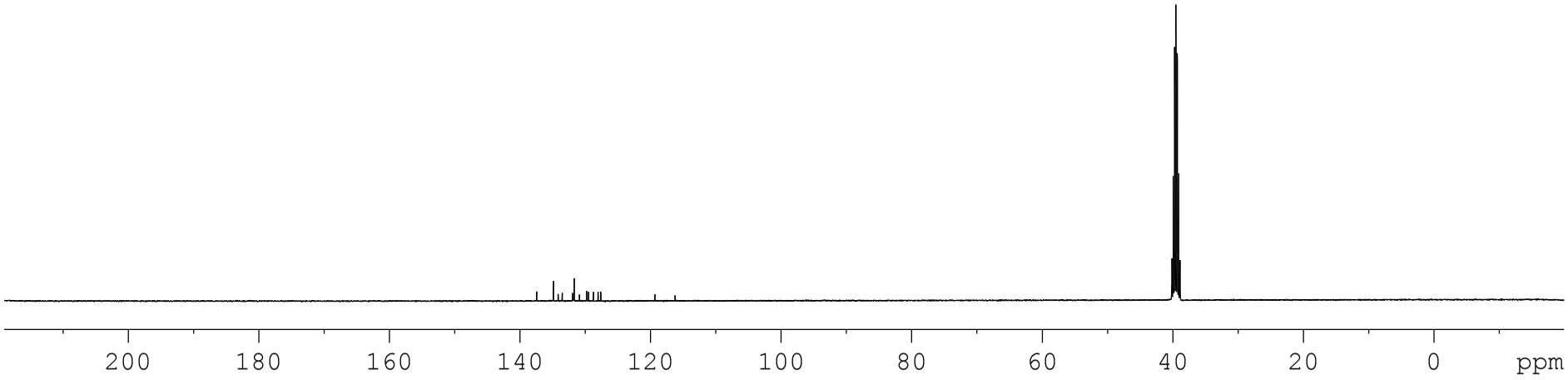


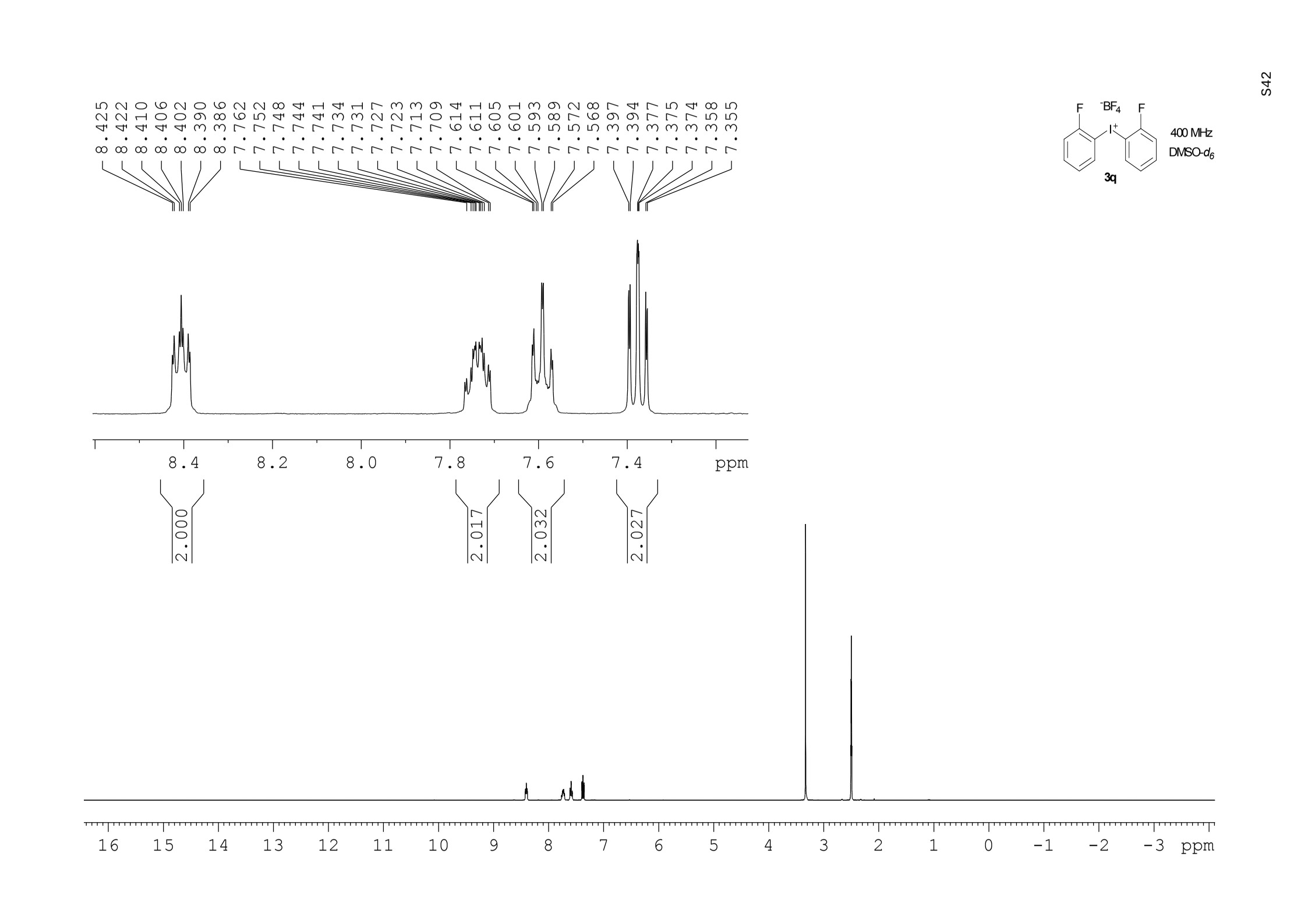
3p

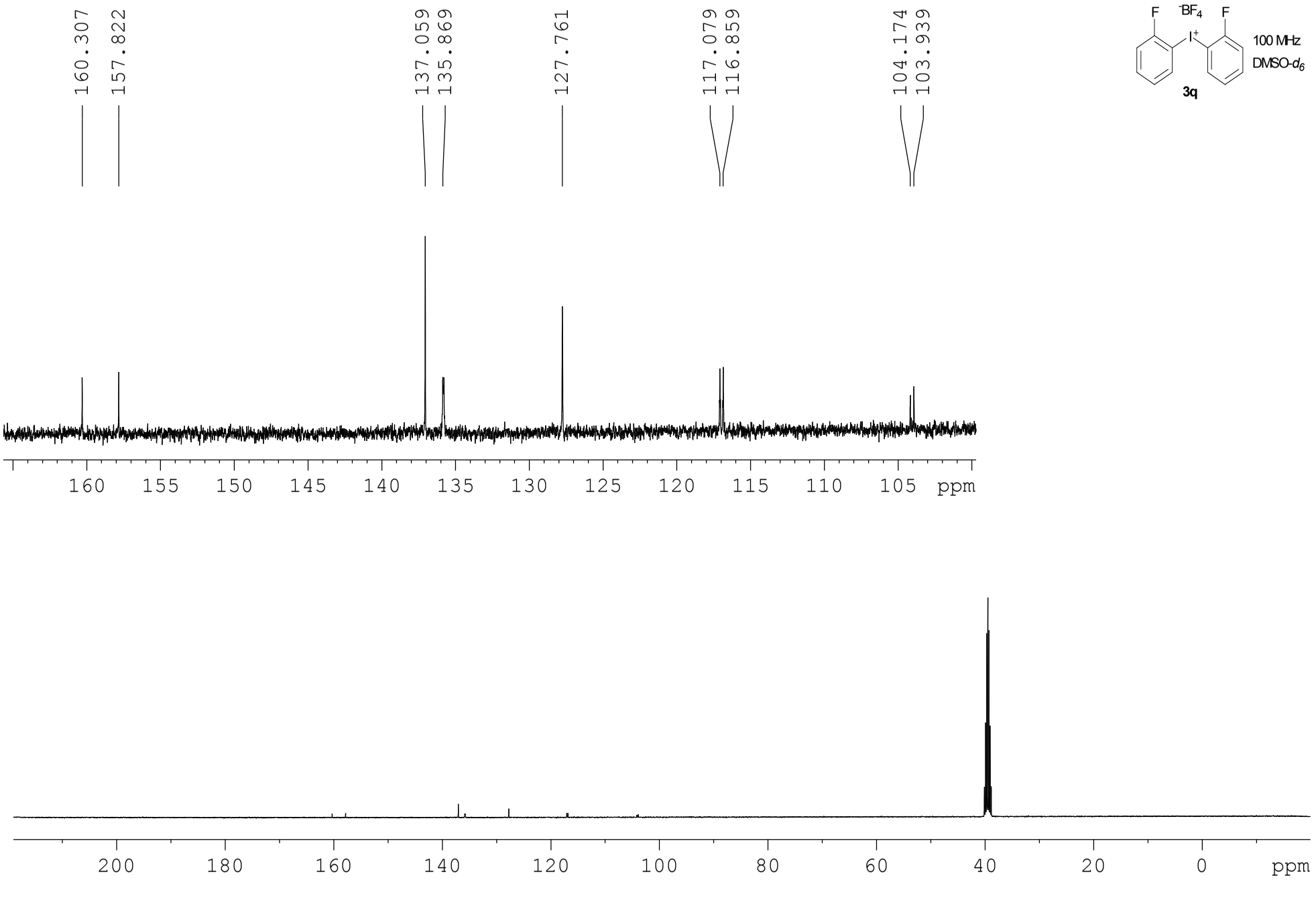
100 MHz
DMSO-d_6

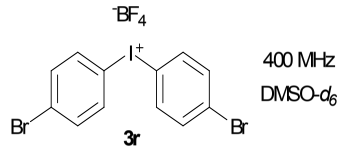
137.443
134.874
134.147
133.526
131.947
131.684
130.895
129.795
129.510
128.752
128.037
127.621
119.333
116.235

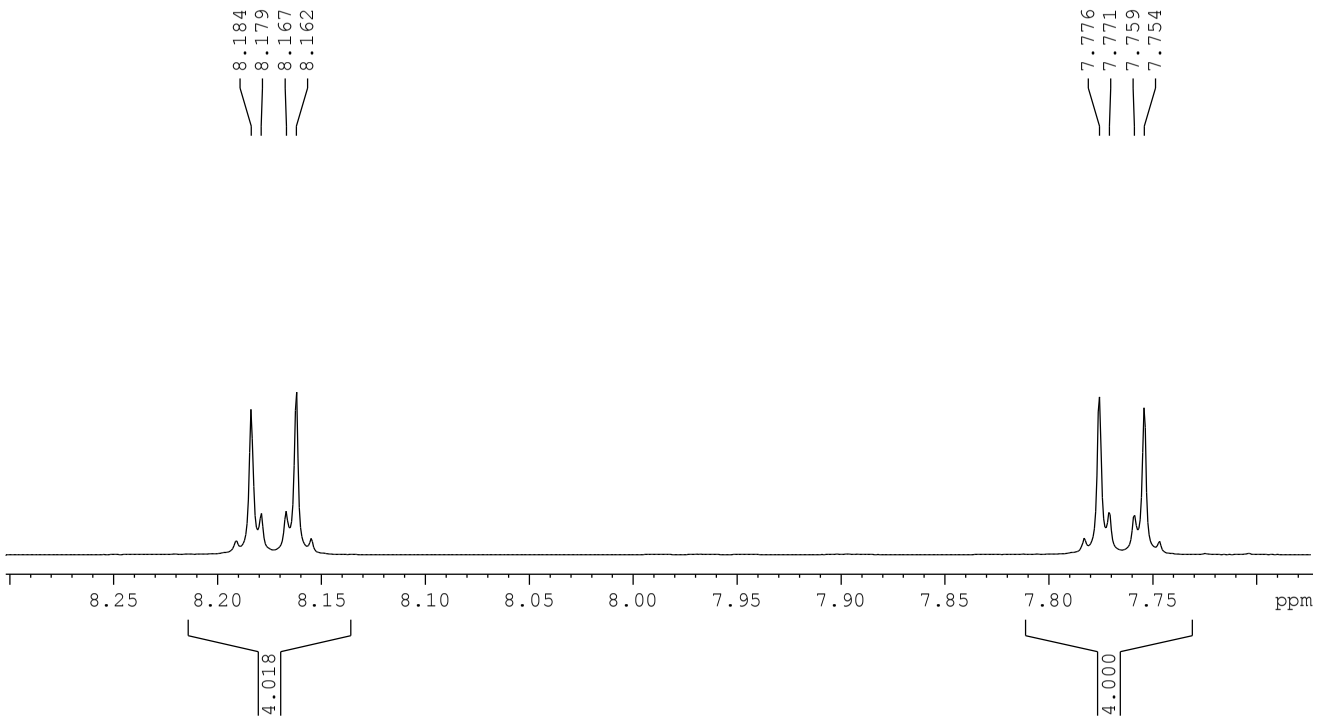


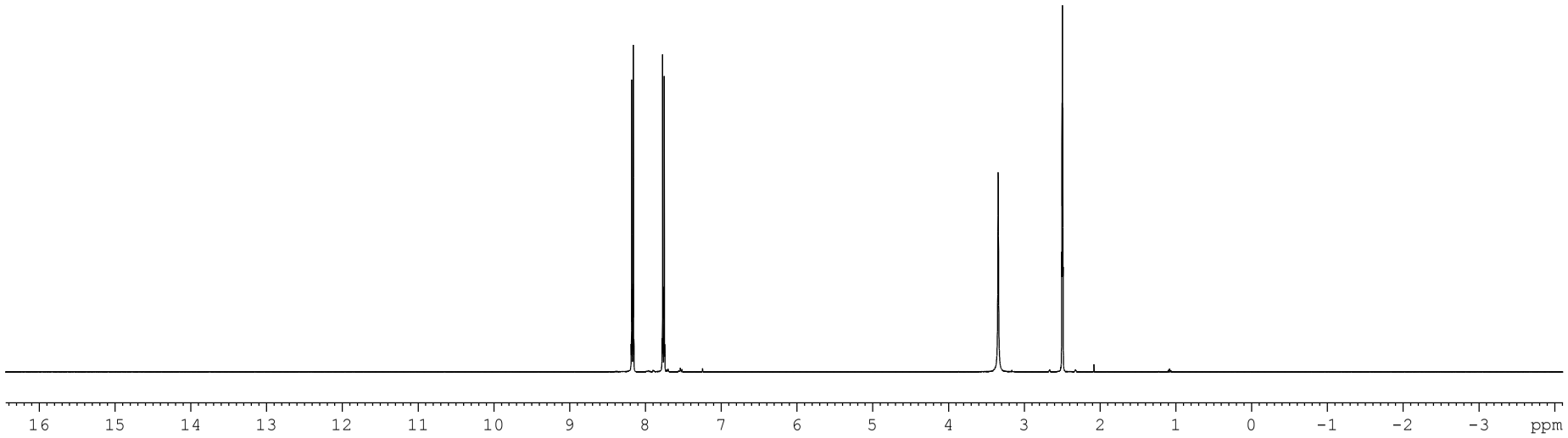


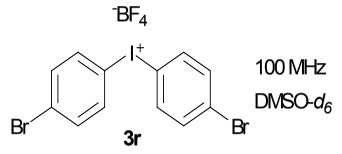

8.425
8.422
8.410
8.406
8.402
8.390
8.386
7.762
7.752
7.748
7.744
7.741
7.734
7.731
7.727
7.723
7.713
7.709
7.614
7.611
7.605
7.601
7.593
7.589
7.572
7.568
7.397
7.394
7.377
7.375
7.374
7.358
7.355
8.4
8.2
8.0
7.8
7.6
7.4
ppm
2.000
2.017
2.032
2.027
F
BF4
F
400 MHz
DMSO-d6
3q
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
ppm

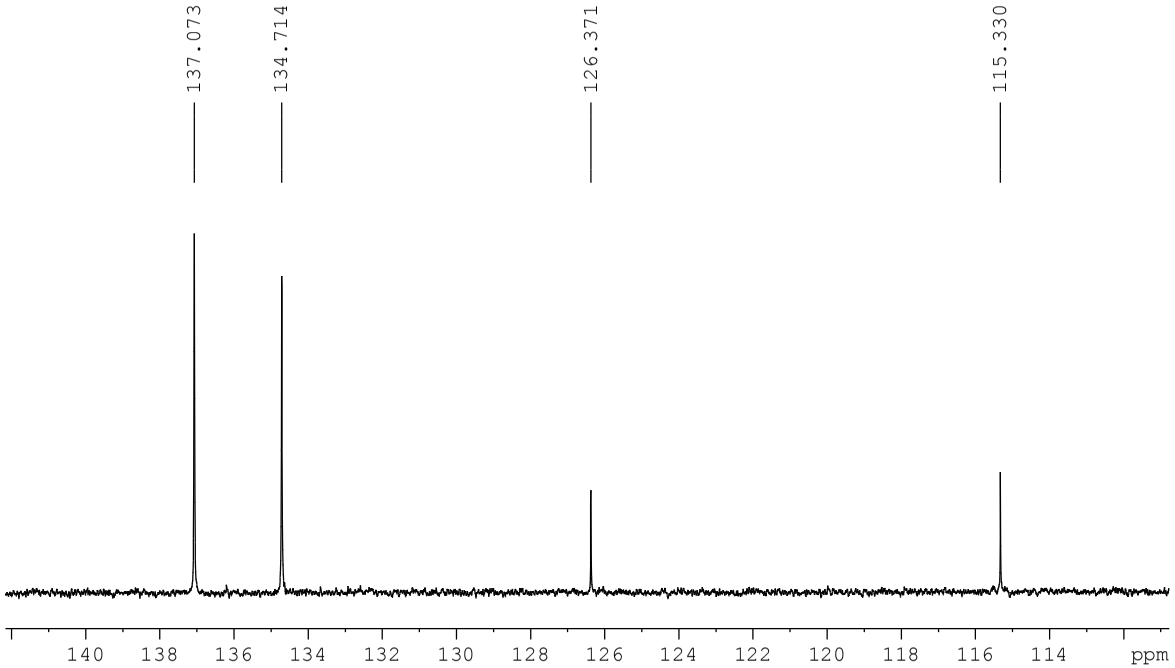

160.307
157.822
137.059
135.869
127.761
117.079
116.859
104.174
103.939
F
$^{-}BF_4$
F
I^{+}
100 MHz
DMSO-d_6
3q
160
155
150
145
140
135
130
125
120
115
110
105
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm

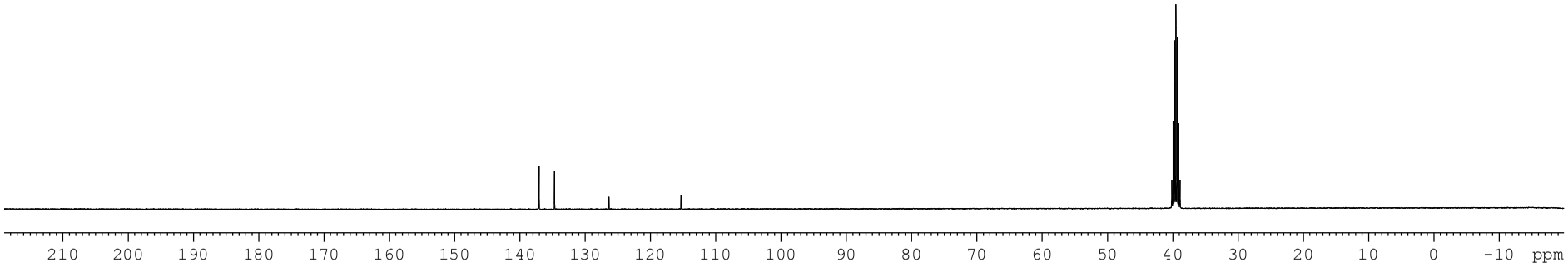

$^-BF_4$
$^+$
Br
Br
3r
400 MHz
DMSO-d_6


8.184
8.179
8.167
8.162
7.776
7.771
7.759
7.754
8.25
8.20
8.15
8.10
8.05
8.00
7.95
7.90
7.85
7.80
7.75
ppm
4.018
4.000

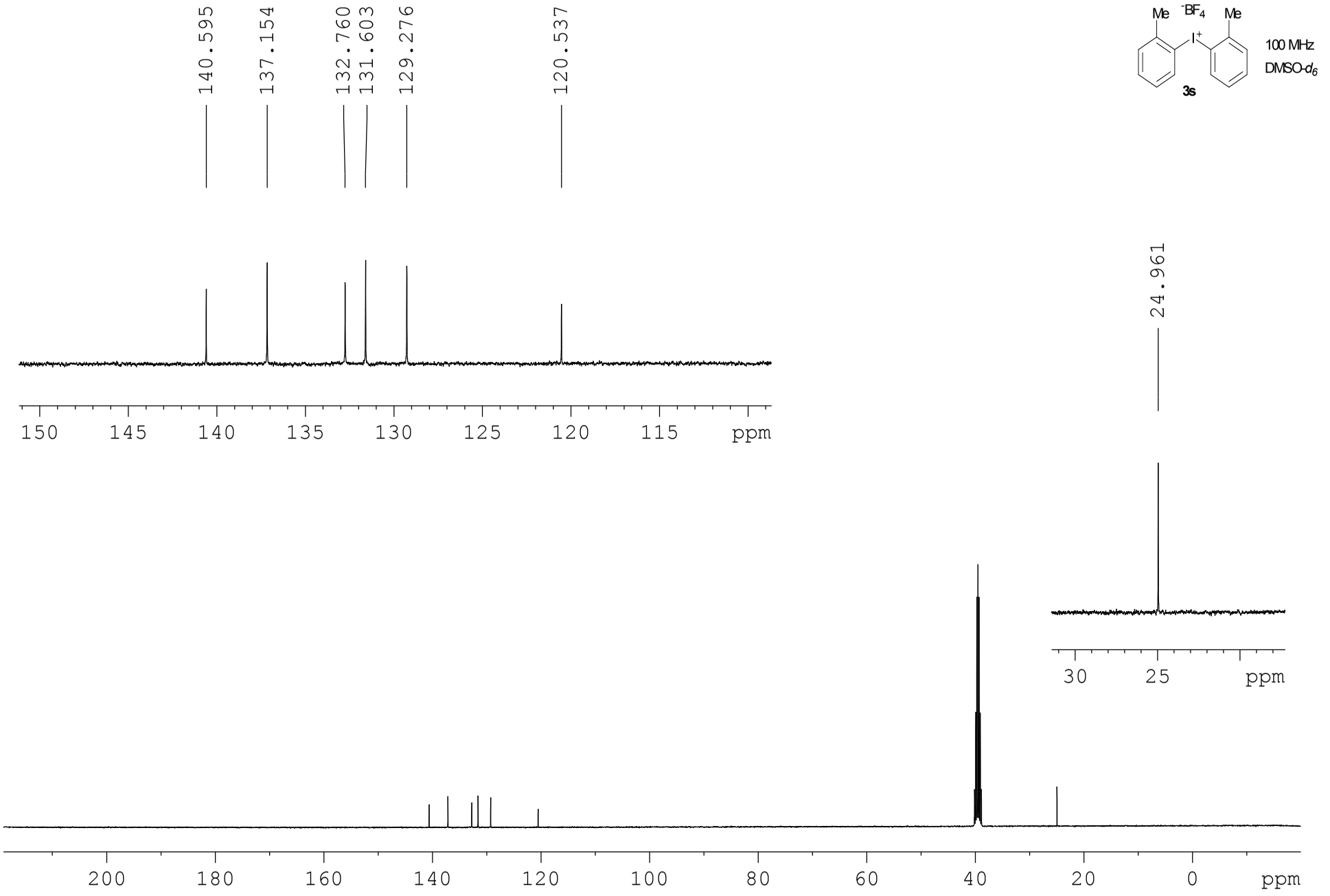

16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
ppm

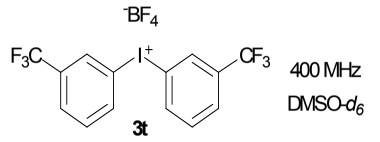

$^{-}BF_4$
I^{+}
Br
Br
3r
100 MHz
DMSO-d_6


137.073
134.714
126.371
115.330
140 138 136 134 132 130 128 126 124 122 120 118 116 114 ppm

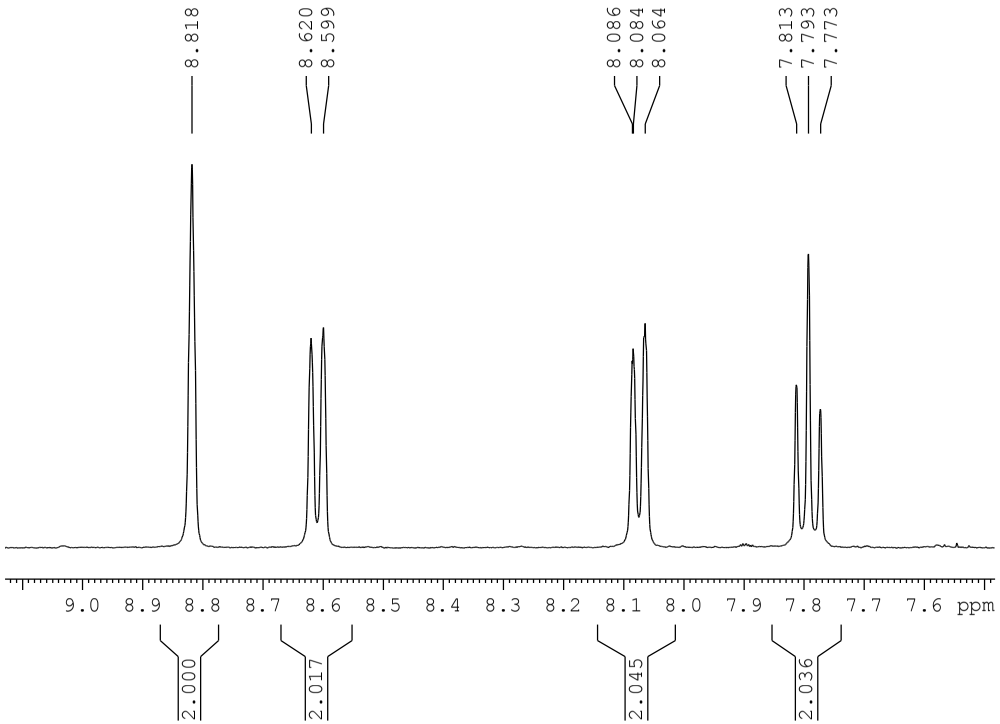

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

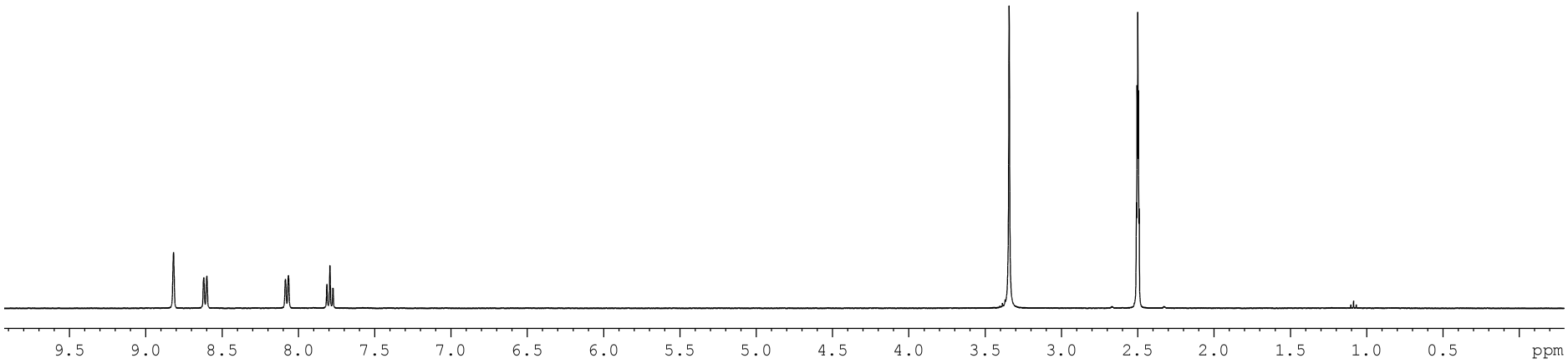
8.325
8.323
8.304
7.599
7.597
7.580
7.578
7.567
7.564
7.562
7.549
7.542
7.321
7.314
7.303
7.300
7.294
7.286
7.284
7.279
2.000
4.013
2.029
5.961
2.608
ppm
Me
BF4
Me
3s
400 MHz
DMSO-d6

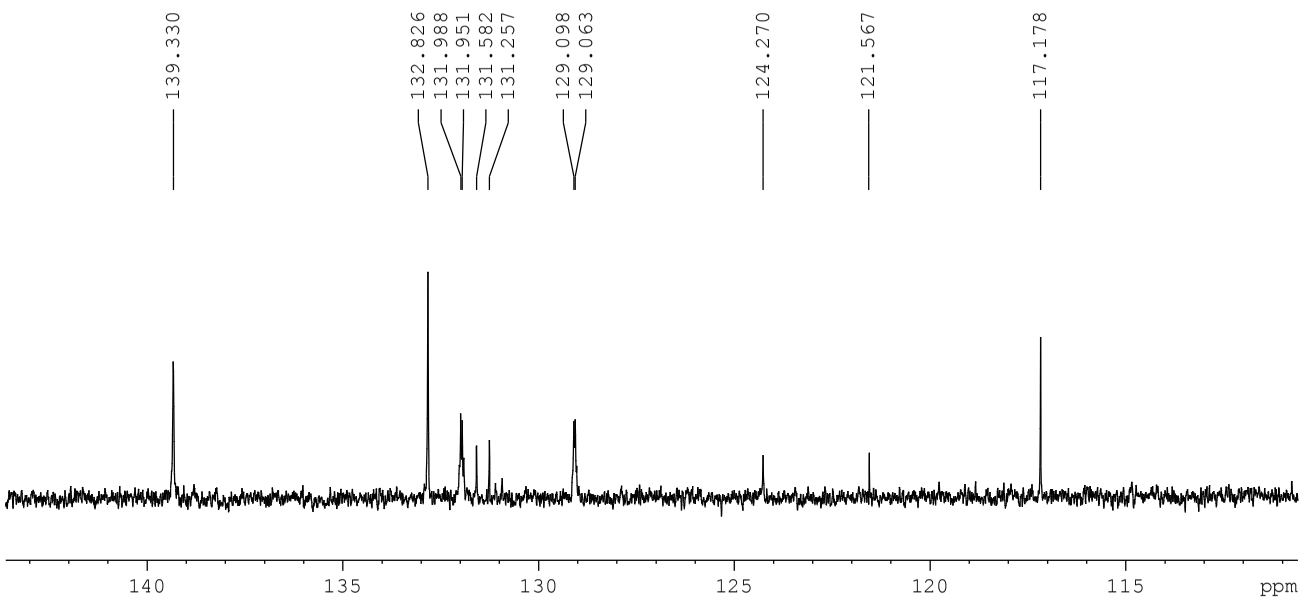

140.595
137.154
132.760
131.603
129.276
120.537
24.961
Me
$^{-}BF_4$
Me
I^{+}
3s
100 MHz
DMSO-d_6
150
145
140
135
130
125
120
115
ppm
30
25
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm

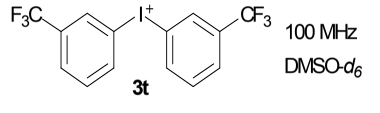


400 MHz
DMSO-d_6

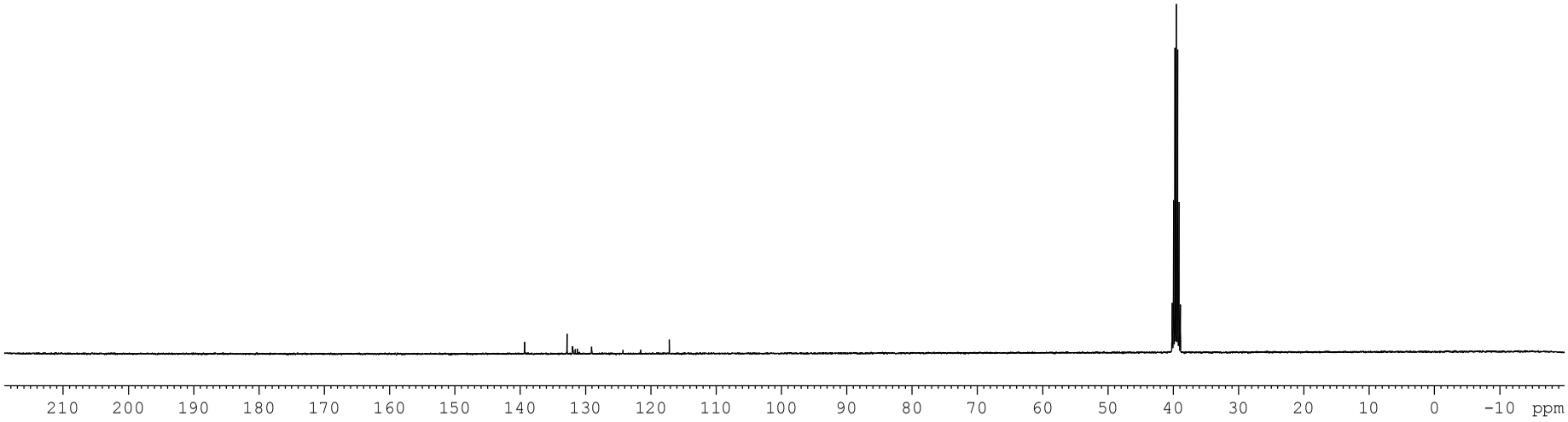


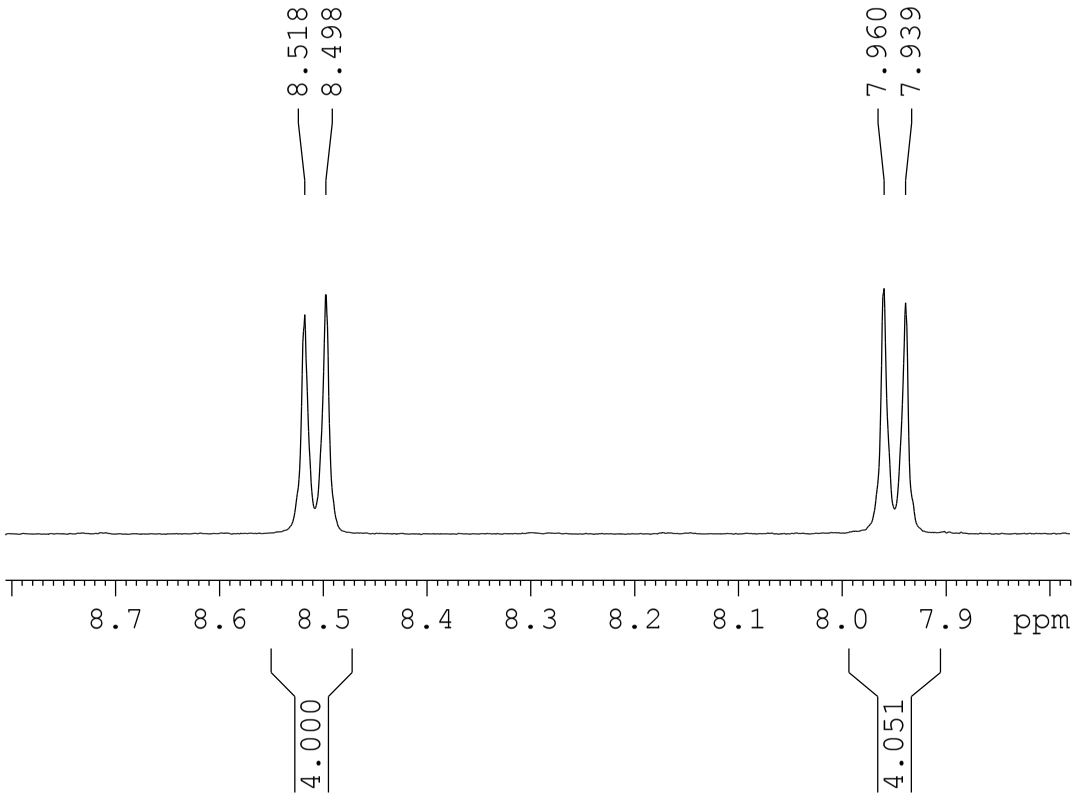


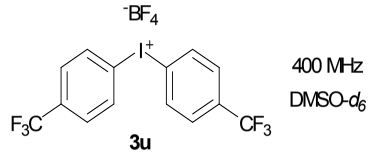


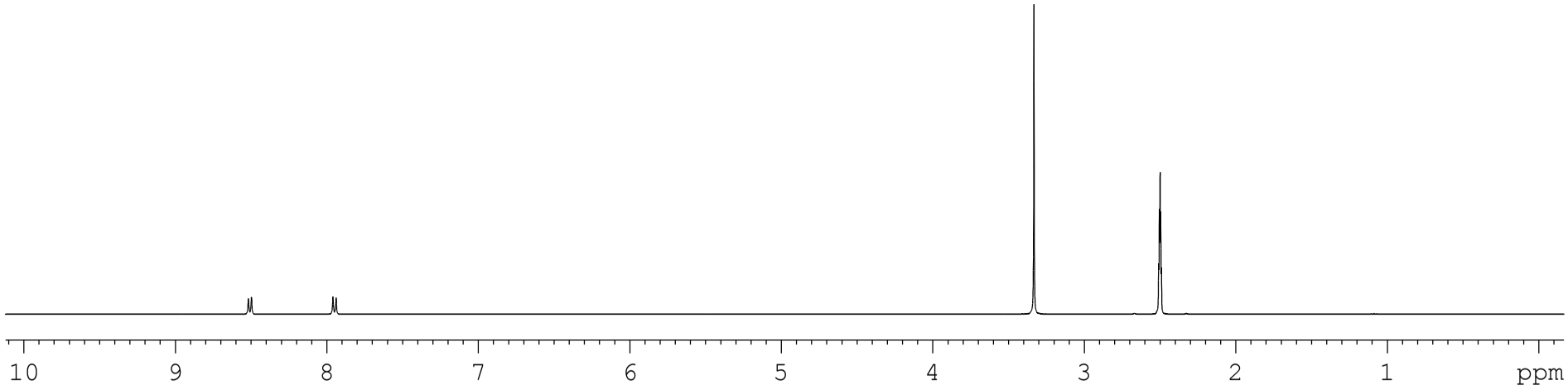


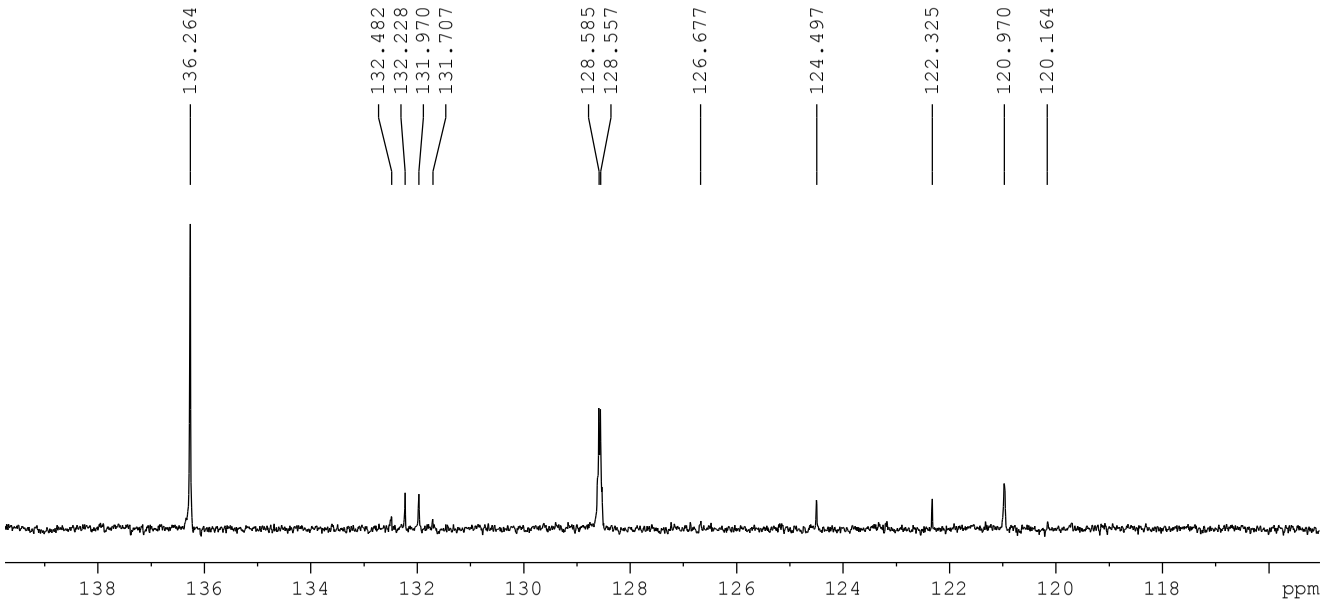
100 MHz
DMSO-d_6

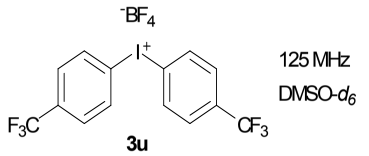


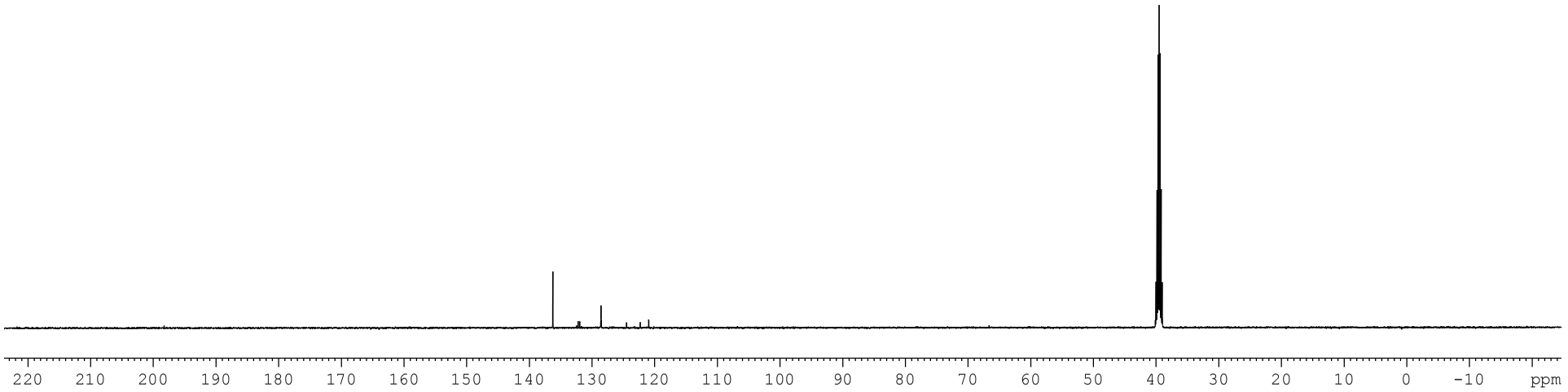

8.518
8.498
7.960
7.939
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
ppm
4.000
4.051

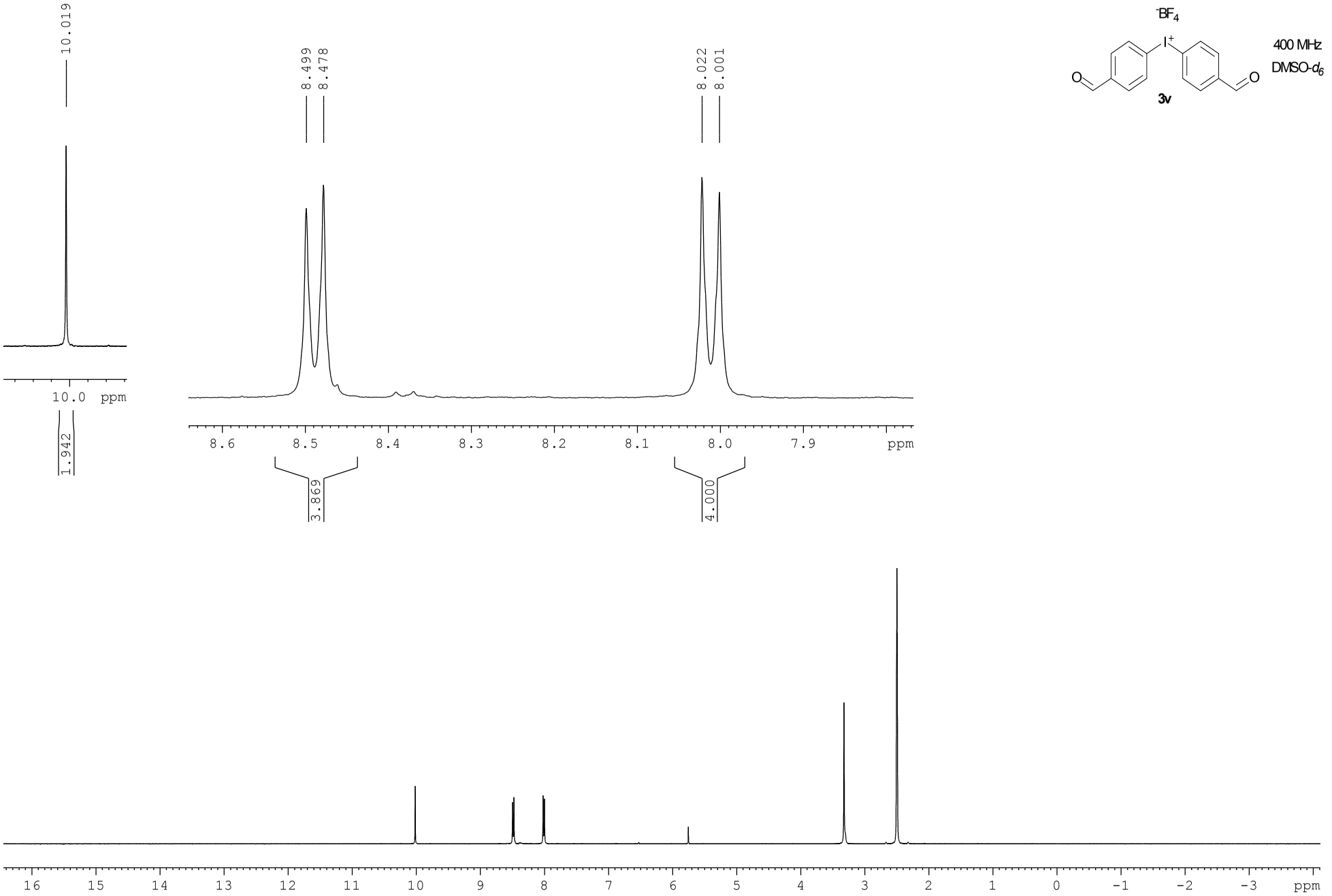

-BF4
I+
F3C
CF3
3u
400 MHz
DMSO-d6


10
9
8
7
6
5
4
3
2
1
ppm

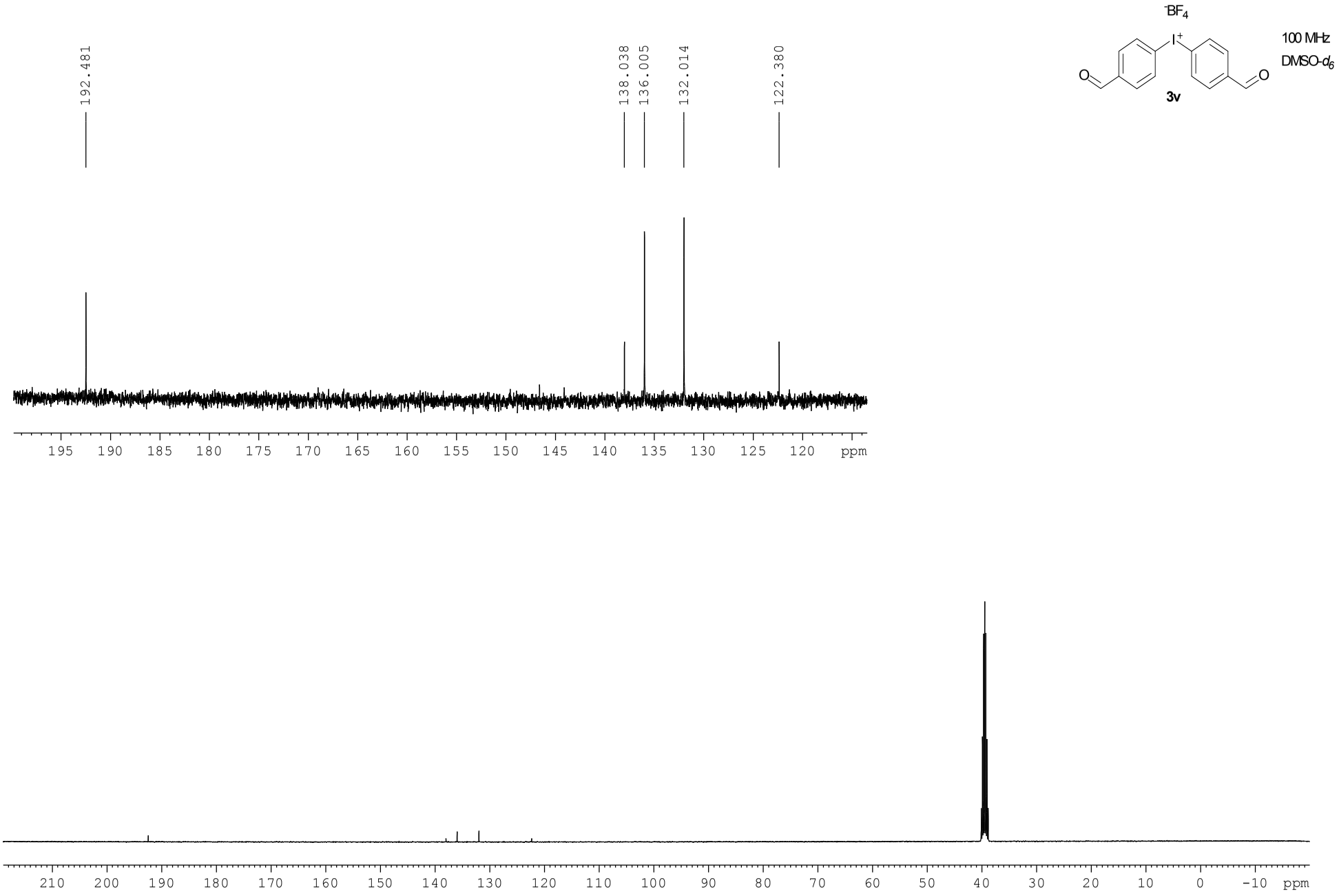

136.264
132.482
132.228
131.970
131.707
128.585
128.557
126.677
124.497
122.325
120.970
120.164
138
136
134
132
130
128
126
124
122
120
118
ppm

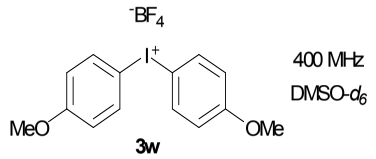

$^{-}BF_4$
I^{+}
F_3C
CF_3
3u
125 MHz
DMSO-d_6

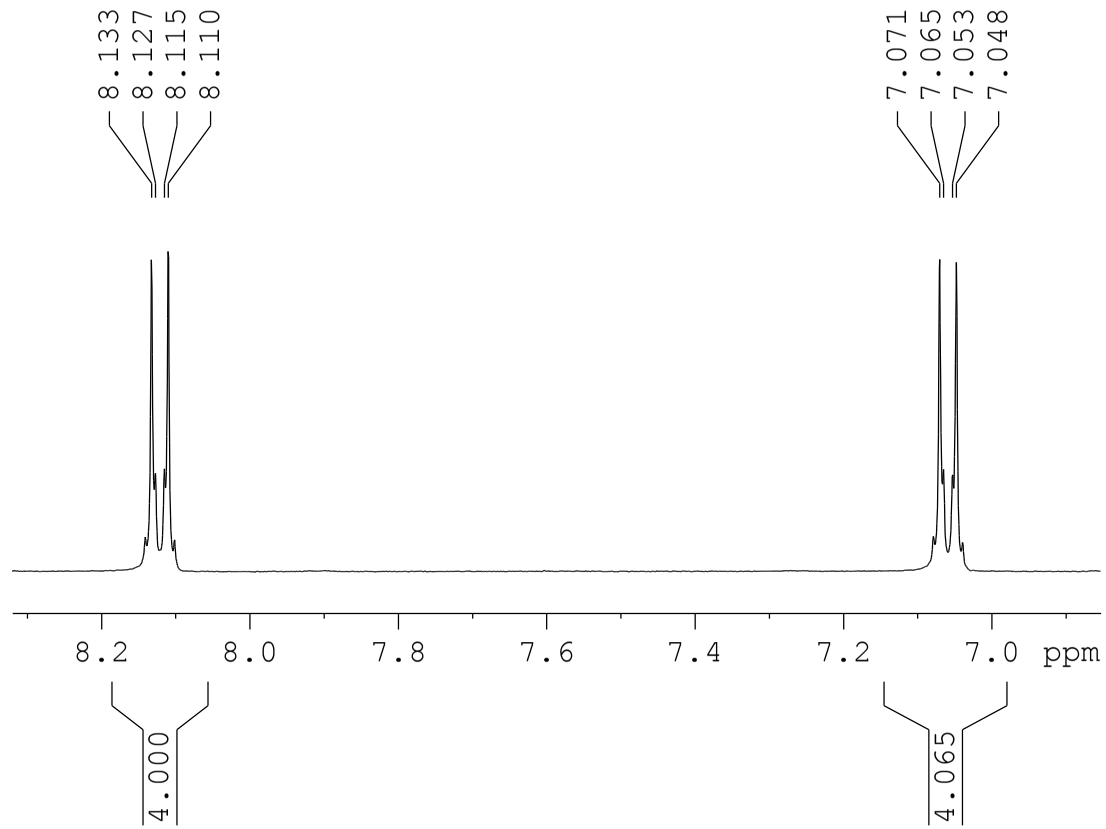

220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm

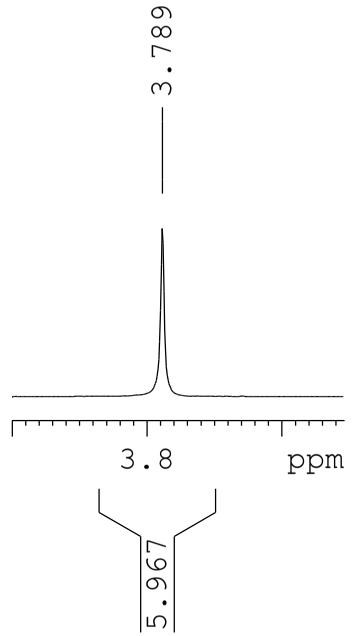


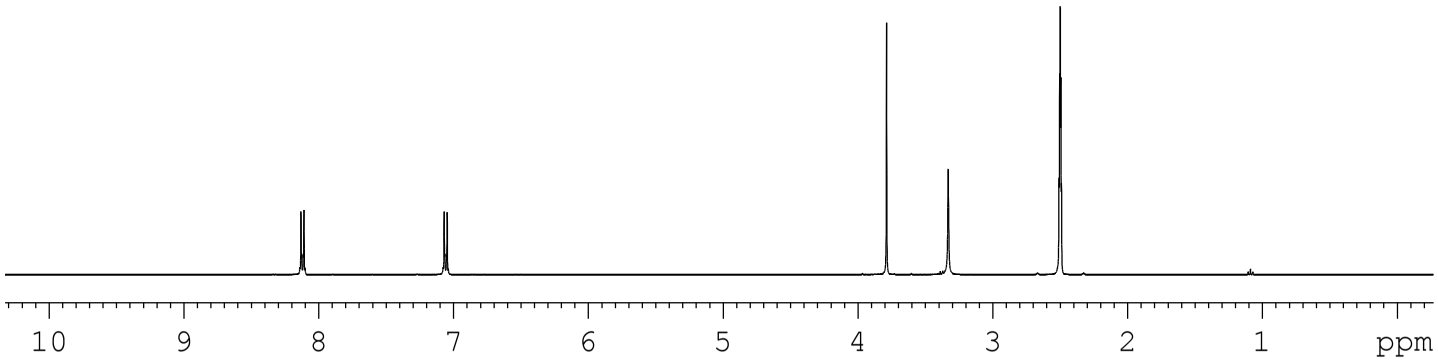
400 MHz
DMSO-d_6

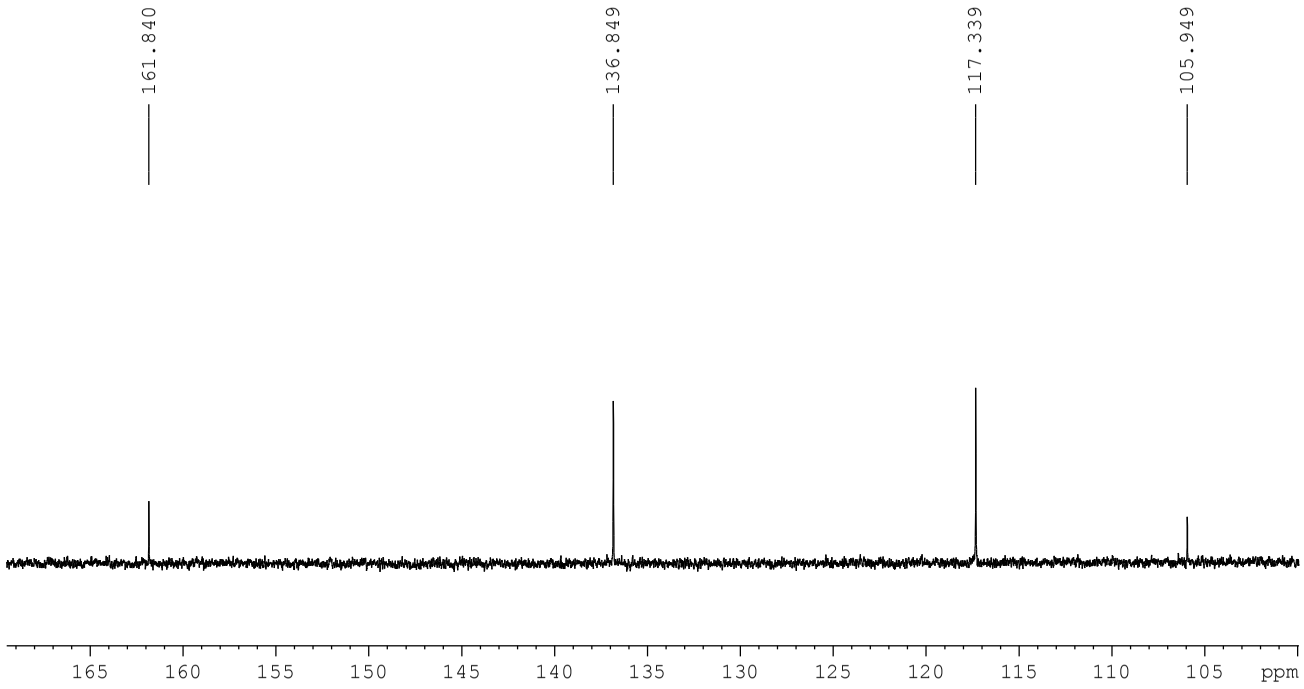

192.481
138.038
136.005
132.014
122.380
195 190 185 180 175 170 165 160 155 150 145 140 135 130 125 120 ppm
$^{-}BF_4$
I^{+}
O
O
3v
100 MHz
DMSO-d_6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

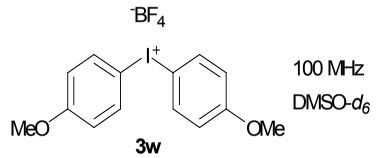

$^{-}BF_4$
I^{+}
MeO
OMe
3w
400 MHz
DMSO-d_6

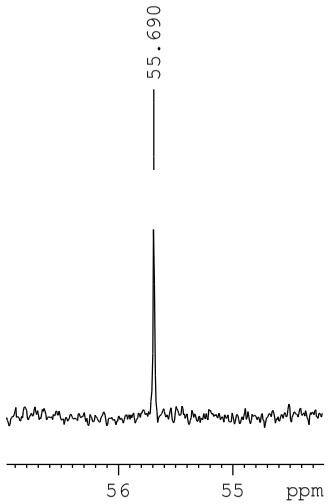

8.133
8.127
8.115
8.110
7.071
7.065
7.053
7.048
8.2
8.0
7.8
7.6
7.4
7.2
7.0
ppm
4.000
4.065

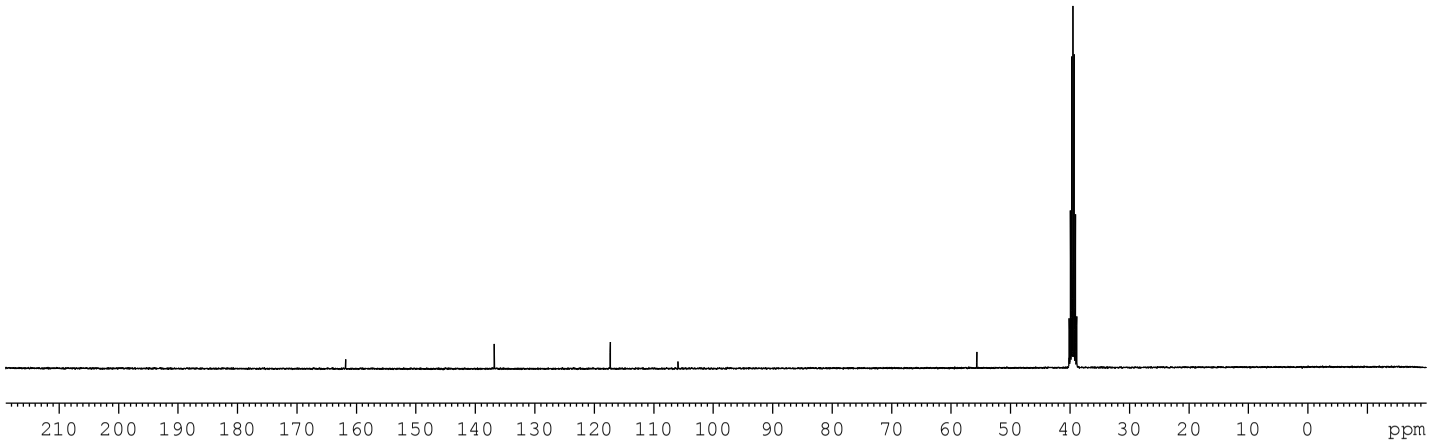

3.789
3.8
ppm
5.967

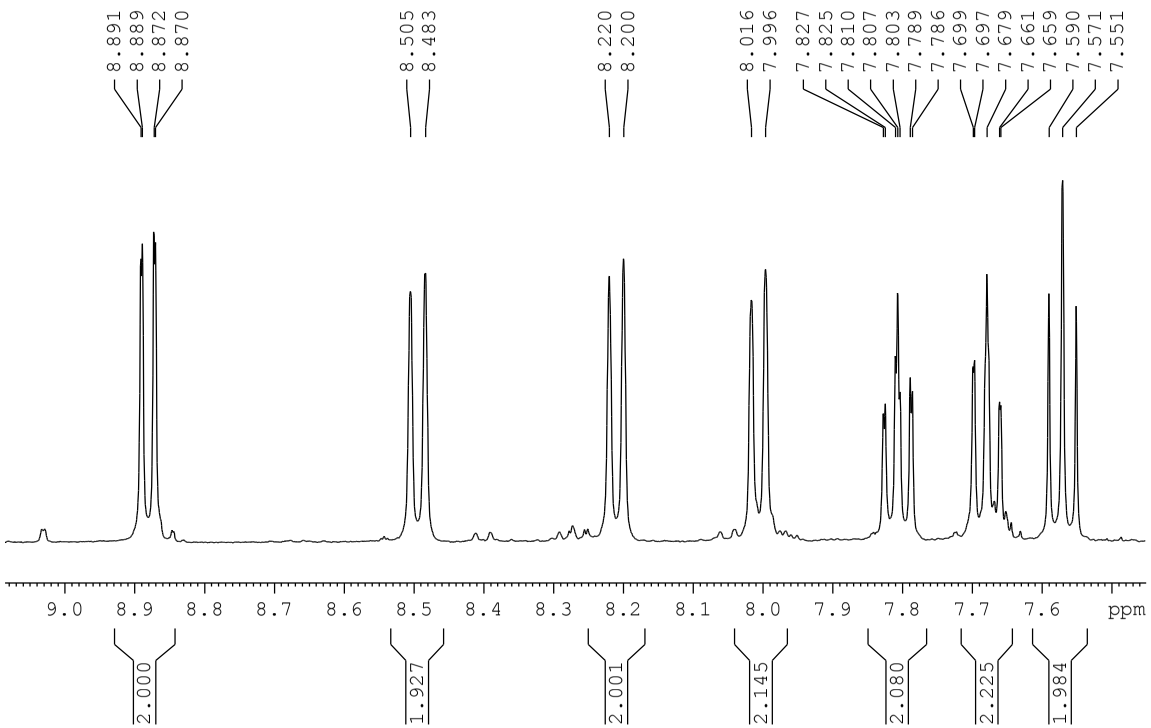

10
9
8
7
6
5
4
3
2
1
ppm

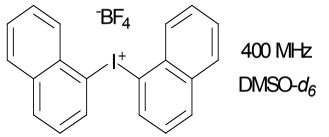

161.840
136.849
117.339
105.949
165
160
155
150
145
140
135
130
125
120
115
110
105
ppm

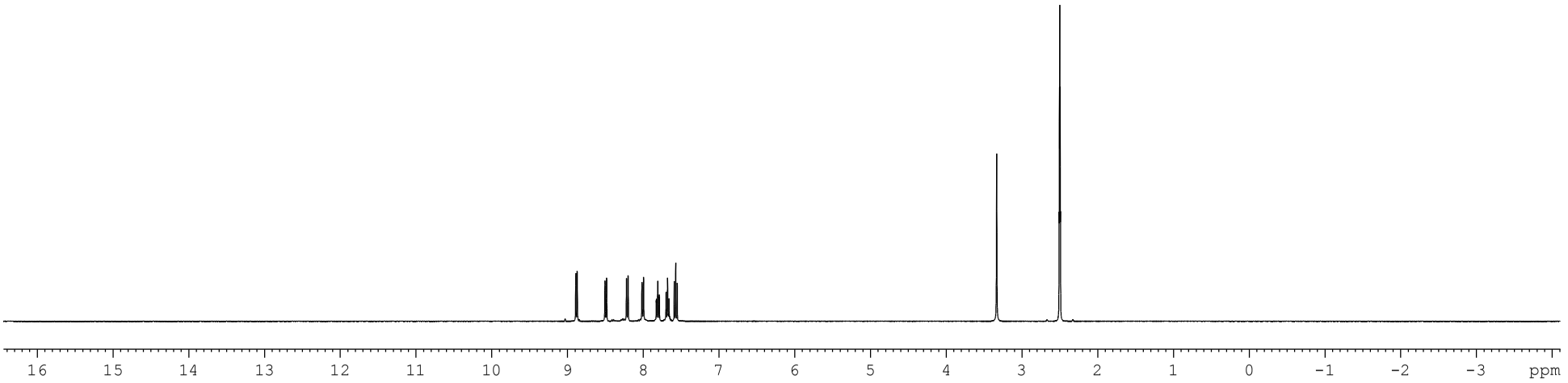

BF4
I+
MeO
OMe
3w
100 MHz
DMSO-d6

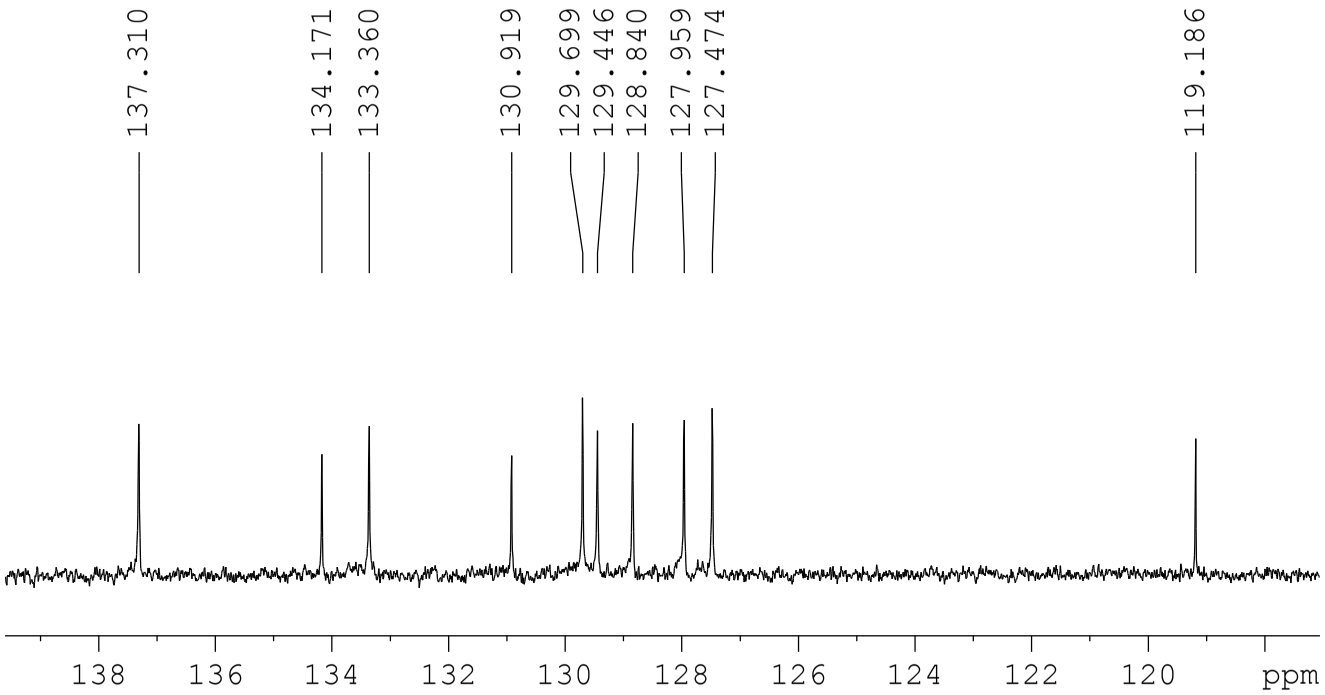

55.690
56
55
ppm

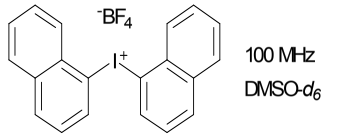

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.891
8.889
8.872
8.870
8.505
8.483
8.220
8.200
8.016
7.996
7.827
7.825
7.810
7.807
7.803
7.789
7.786
7.699
7.697
7.679
7.661
7.659
7.590
7.571
7.551
9.0
8.9
8.8
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
ppm
2.000
1.927
2.001
2.145
2.080
2.225
1.984

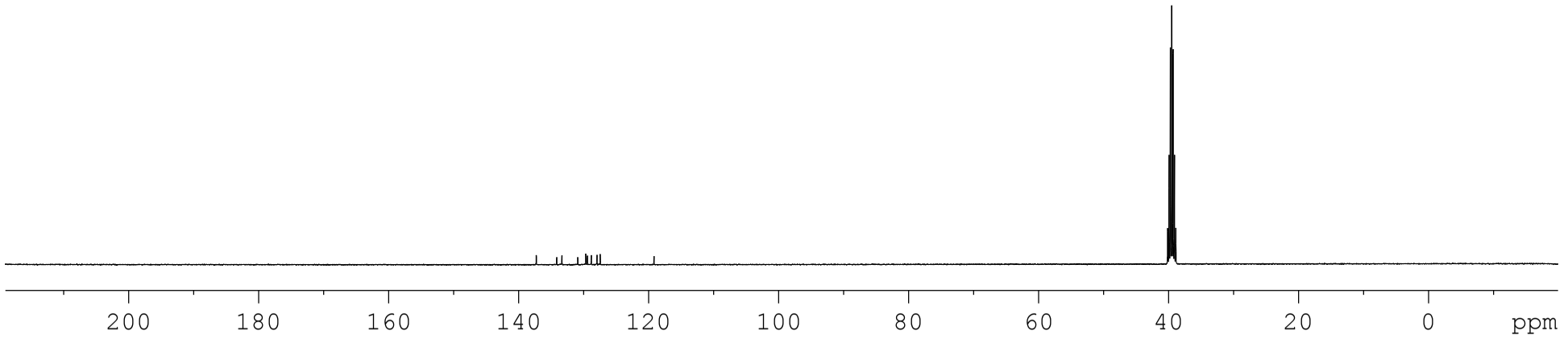

$^{-}BF_4$
I^{+}
400 MHz
DMSO-d_6

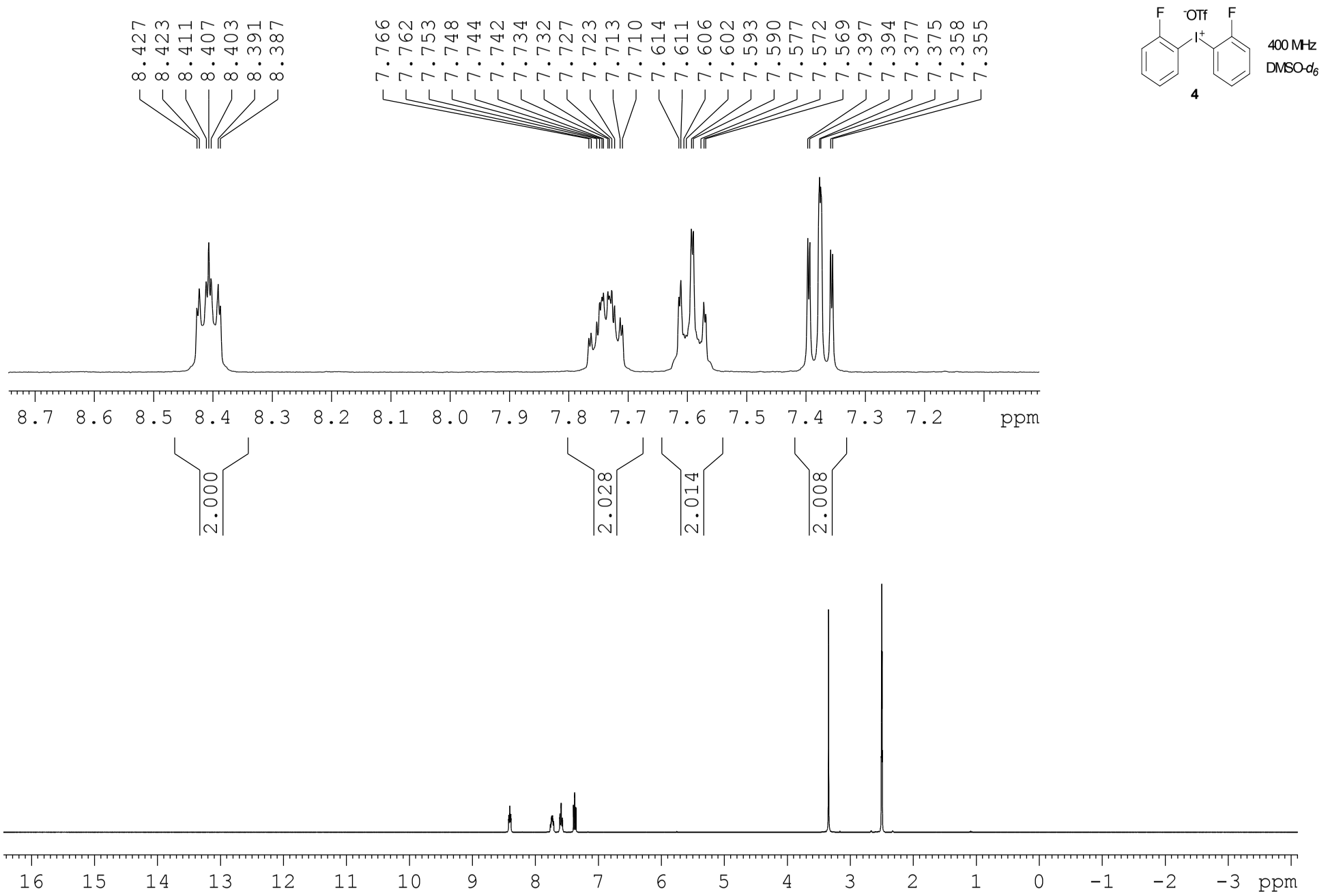

16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
ppm

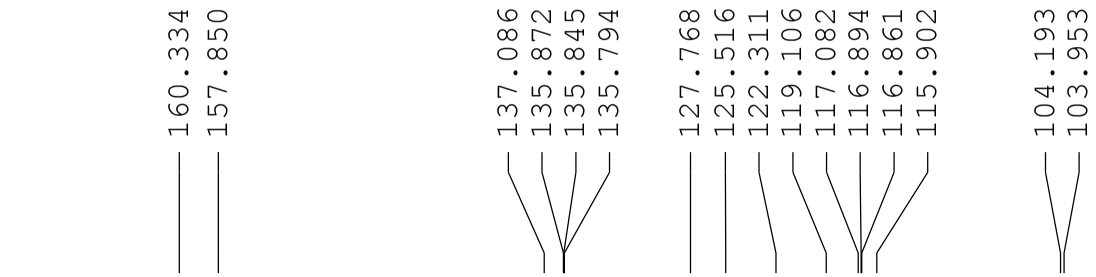


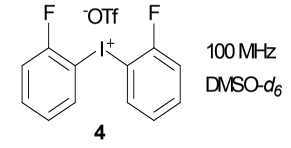


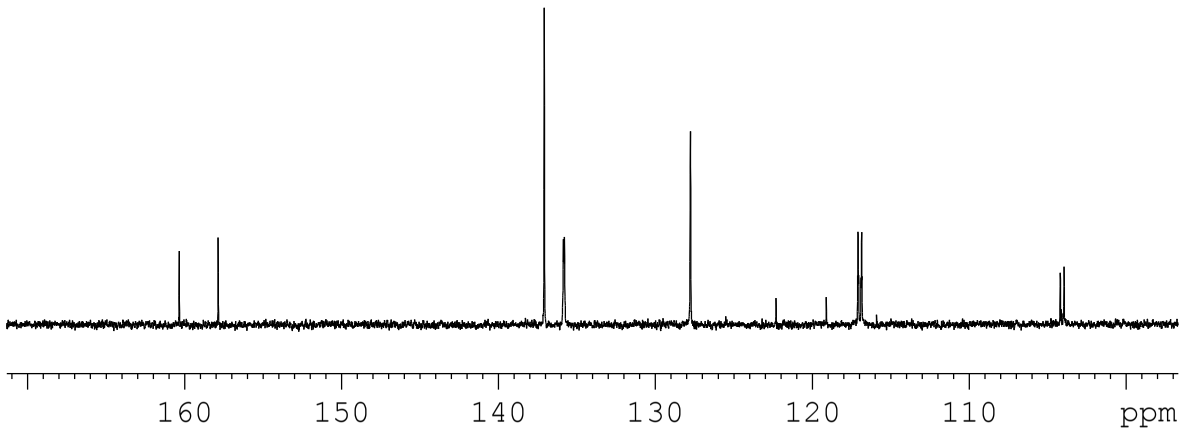
100 MHz
DMSO-d_6

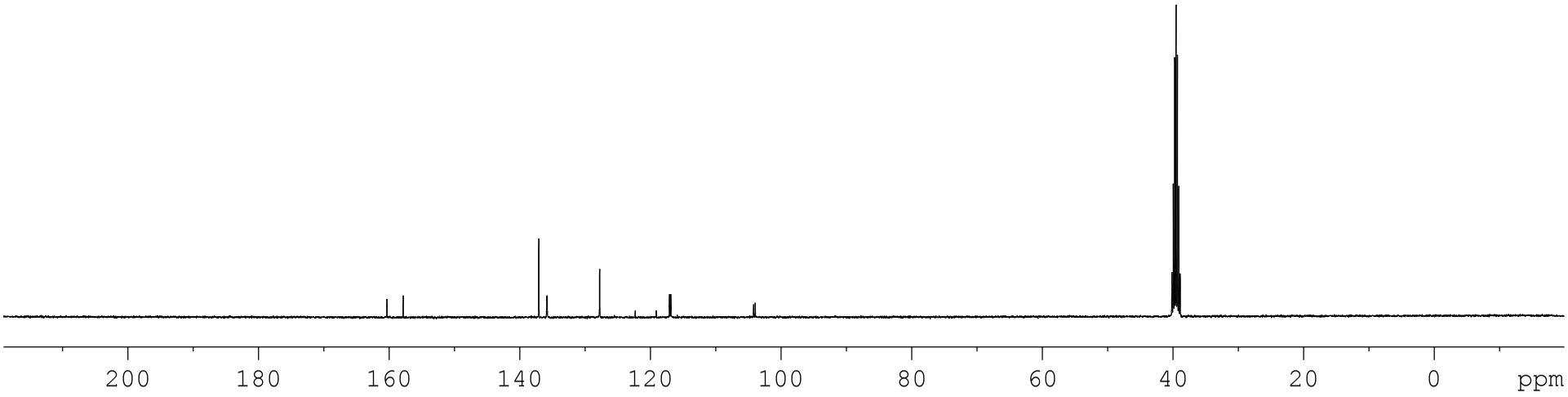



8.427
8.423
8.411
8.407
8.403
8.391
8.387
7.766
7.762
7.753
7.748
7.744
7.742
7.734
7.732
7.727
7.723
7.713
7.710
7.614
7.611
7.606
7.602
7.593
7.590
7.577
7.572
7.569
7.397
7.394
7.377
7.375
7.358
7.355
F
OTf
F
I+
4
400 MHz
DMSO-d6
8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm
2.000
2.028
2.014
2.008
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 ppm


160.334
157.850
137.086
135.872
135.845
135.794
127.768
125.516
122.311
119.106
117.082
116.894
116.861
115.902
104.193
103.953


F
-OTf
F
I+
100 MHz
DMSO-d6
4


160
150
140
130
120
110
ppm


200
180
160
140
120
100
80
60
40
20
0
ppm