Electronic Supplementary Material (ESI) for ChemComm.

A Mild Carbon-Boron Bond Formation from Diaryliodonium Salts

Núria Miralles, R. Martín Romero, Elena Fernández* and Kilian Muñiz*

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1. GENERAL REMARKS

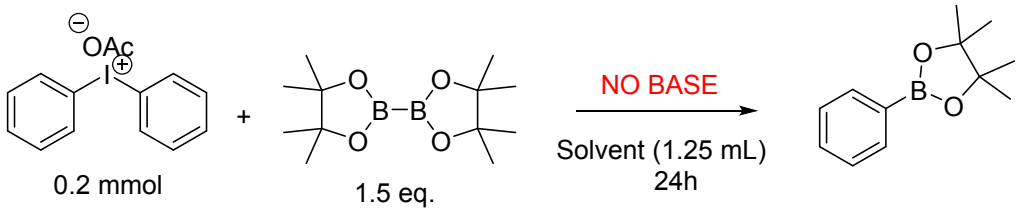
All solvents, reagents and deuterated solvents were purchased from Aldrich, Alfa Aesar, Maybridge and TCI. Methanol was dried by calcium hydride destillation. Dichloromethane, tetrahydrofuran, *n*-hexane and acetonitrile were dried and purified with a solvent purification system Pure SOLV system-4. Column chromatography was performed with silica gel (Merck, type 60, 0.063-0.2 mm). NMR spectra were recorded on a Bruker Avance 300 MHz, 400 MHz or 500 MHz spectrometer, Varian Gemini 300 MHz and Varian Mercury 400 MHz. All chemical shifts in NMR experiments are reported as ppm downfield from TMS. The following calibrations were used: $CDCl_3$ δ = 7.26 and 77.0 ppm, CD_3OD δ = 3.31 and 49.0 ppm, DMSO-d_6 δ = 2.50 and 39.5 ppm. MS (ESI-LCMS) experiments were performed using an Agilent 1100 HPLC with a Bruker micro-TOF instrument (ESI). Unless otherwise stated, a Supelco C8 (5 cm x 4.6 mm, 5 µm particles) column was used with a linear elution gradient from 100% H_2O (0.5% HCO_2H) to 100% CH_3CN in 13 min at a flow rate of 0.5 mL/min. MS (ESI) and HRMS experiments were performed on a Kratos MS 50 within the service centers at ICIQ. GC analyses were performed on a HP6890 gas chromatograph and an Agilent Technologies 5973 Mass selective detector (Waldbronn, Germany) equipped with an achiral capillary column HP-5 (30m, 0.25mm i. d., 0.25µm thickness) using He as the carrier gas. IR spectra were measured on a Bruker Alpha instrument in the solid state.

The following comercially available compounds were used as received: Diphenyliodonium chloride, diphenyliodonium triflate, diphenyliodonium hexafluorophosphate, sodium periodate, iodine, anhydrous sodium acetate, peracetic acid, *m*-CPBA, potassium bromide, p-toluensulfonic acid monohydrate, boron trifluoride diethyl etherate, trifluoromethanesulfonic acid, hydrobromic acid, ammonium chloride, silver acetate, 4-iodobenzotrifluoride, 2-iodo-1,3-dimethylbenzene, 4-iodo-1,1'-biphenyl, 3-methyl-iodobenzene, 4-iodoanisole, 2-iodonaphtalene, anisole, 3- iodobenzotrifluoride, diphenylether, chlorobenzene, bromobenzene, fluorobenzene, 4-(trifluoromethyl)phenylboronic acid, methyl benzoate, 3-(trifluoromethyl)phenylboronic acid, nitrobenzene,

mesitylene, 2,6-dimethylphenylboronic acid, m-xilene, 4-biphenylboronic acid, 3-methylphenylboronic acid, toluene, diacetoxyiodobenzene, 1,3,5-triisopropylbenzene, 2-naphthylboronic acid, bis(pinacolato)diboron, bis(neopentyl glycolato)diboron, bis(hexylene glycolato)diboron, bis(catecholato)diboron. Diboron reagent Bpin-Bdan (pin = pinacolate, dan = 1,8-diaminonaphtalene) was synthesized following previously reported procedures.[1]

2. OPTIMIZATION OF REACTION CONDITIONS

SCREENING OF SOLVENTS:



Solvent	T (º C)	Yield (%)[1]
MeOH	rt	59
MeOH	50	68
EtOH	50	52
iPrOH	50	13
THF	50	39
Dry MeOH	50	83

[1]Yield from two independent runs calculated by GC spectroscopy with mesitylene as internal standard.

3. SYNTHESIS OF DIACETOXYIODOARENES

METHOD I (GP1):

Arene (10 mmol, 1 equiv.) was dissolved in AcOH (24 mL) and AcOOH (2.6 equiv.) was added dropwise and the final mixture was stirred at rt for 20h. The solvent was removed *in vacuo* and, the final crude was triturated with hexane and filtrated, or the residue was dissolved in the minimum amount of CH_2Cl_2 and cyclohexane was added and solvents were evaporated under reduced pressure to afford the desired diacetoxyiodoarene.

METHOD II (GP2):

Following the procedure described previously in literature.[2] $NaIO_4$ (1.03 equiv.) and anhydrous sodium acetate (2.2 equiv.) were suspended in a stirred mixture of glacial AcOH (15 mL) with Ac_2O (1.5 mL). Arene was added and the mixture was stirred and boiled under reflux for 2.5h, cooled to rt and poured into water. The mixture was extracted with CH_2Cl_2 (3x), dried over Na_2SO_4 and concentrated *in vacuo*. The resulting crude mixture was triturated with Et_2O and filtered to obtain the desired diacetoxyiodoarene.

4. SYNTHESIS OF DIARYLIODONIUM SALTS

METHOD A (GP3):

Following the procedure described previously in literature,[3] powered I_2 (0.72 g, 2.84 mmol) and $NaIO_4$ (0.92 g, 4.30 mmol) were suspended in stirred conc. H_2SO_4 (10 mL), and the vigorous stirring was continued at 75 ºC for 1 h. Next, Ac_2O (0-10 mL depending the substrate) was slowly added dropwise, with stirring, to the cooled suspension of the formed yellow $(IO)_2SO_4$. Arene (26 mmol) was added portionwise or dropwise, and the resulting mixture was stirred at rt for 20 h. The final mixture was poured into stirred ice-H_2O (300 g). Any precipitates were filtered off and discarded. The cold filtrates were extracted with Et_2O (4x50 mL) and the ethereal extracts were again discarded. KBr (2.0 g, 16.8 mmol) was added to the vigorously stirred remaining aqueous solution.

After 1 h, the precipitate was collected by filtration and washed with cold H_2O until the filtrates were neutral in pH and then were air-dried in the dark to give the diaryliodonium bromides.

METHOD B (GP4):

Following the procedure described previously in literature[3]. Arene (26 mmol) was dissolved or suspended in conc H_2SO_4 or in a glacial AcOH-conc. H_2SO_4 mixture (depending the substrate) and the resulting mixture was warmed up, with stirring, to 55 ºC. While keeping the same temperature, $NaIO_4$ (2.14 g, 10 mmol) was slowly added portion wise over 1.5h. The cooled final mixtures were poured into stirred ice-H_2O (300 g). Any precipitates were filtered off and rejected. The cold filtrates were extracted with Et_2O (4 x 50 mL) and the ethereal extracts were discarded. KBr (2.0 g, 16.8 mmol) was added to the vigorously stirred remaining aqueous solution. After 1h, the precipitate was collected by filtration and washed with cold H_2O until the filtrates were pH-neutral and then air-dried in the dark to give the diaryliodonium bromides.

METHOD C (GP5):

Following the procedure described previously in literature,[4] *m*-CPBA (2.5 equiv.), $TsOH \cdot H_2O$ (3.4 equiv.) and the arene (3.5 equiv.) were dissolved in CH_2Cl_2. Then, I_2 (5.71 mmol, 1.0 equiv.) was added and the final mixture was stirred at rt for 15 h. The solution was diluted with H_2O and extracted with CH_2Cl_2 (3x), dried over $NaSO_4$ and concentrated. The resulting residue was triturated with Et_2O and filtered to give the desired diaryliodonium tosylate.

METHOD D (GP6):

Based on the procedures described previously in literature,[5] arylboronic acid (1.1 equiv.) was dissolved in CH_2Cl_2 (35 mL) under an inert atmosphere. At 0 ºC, $BF_3{\cdot}OEt_2$ (1.5 equiv.) was added and the mixture was stirred at this temperature during 10 minutes. Then, diacetoxyiodoarene (7 mmol, 1.0 equiv.) was added and the final mixture was stirred at rt for 1.5 h. After re-cooling again to 0 ºC, TfOH (1.1 equiv.) was added and the mixture was warmed to rt and stirred for 20 minutes. The solvent was removed under reduced pressure and Et_2O was added to the residual mixture. The resulting mixture was cooled to -20 ºC for 10 h and the precipitate (diaryliodonium trifluoromethanesulfonate) was collected by filtration and then washed with cold Et_2O. The obtained solid was dissolved in CH_3OH (12 mL) and a solution of HBr (48% aq., 0.9 mL) in CH_3OH (12 mL) was added, stirring the resulting mixture at rt for 45 minutes. Finally, the precipitate was collected by filtration to give the desired diaryliodonium bromide.

METHOD E (GP7):

Following the procedure described previously in literature,[6] Ac_2O (20 mL) was added to a well stirred mixture of diacetoxyiodoarene (6 mmol, 1 equiv.) in arene (3.7 equiv.) and then, at 0 ºC, H_2SO_4 (2 mL) was added dropwise, stirring the final mixture at 0 ºC during 1h, and at rt for another 15 h. The final mixture was poured onto ice and the cold solution was extracted with Et_2O discarding the ethereal extracts. KBr (10 equiv.) was added to the vigorously stirred remaining aqueous solution. After 1 h, the precipitate was collected by filtration and washed with cold H_2O and air-dried in the dark to give the diaryliodonium bromides.

METHOD F (GP8):

Following a modified procedure of the described previously in literature,[7] the respective arene (1.1 equiv.) was added to a solution of iodoarene (9 mmol, 1 equiv.) and *m*-CPBA (1.1 equiv.) in CH_2Cl_2 (40 mL). Then, at 0 ºC, TfOH (1.7 equiv.) was added dropwise, and the final mixture was stirred at 0 ºC during 2 h, and at rt during another 15h. The solvent was removed under reduced pressure

and Et_2O was added. The final mixture was cooled to -20 ºC and kept for 10 h. The precipitates (diarliodonium trifluoromethanesulfonate) were collected by filtration and washed with cold Et_2O. The obtained solid was dissolved in CH_3OH (15 mL), a solution of HBr (48% aq., 1.1 mL) in CH_3OH (15mL) was added, and the resulting mixture was stirred at rt for 45 minutes. Finally, the formed precipitate was collected by filtration to give the desired diaryliodonium bromide.

5. ION EXCHANGE

EXCHANGE BETWEEN TOSILATE AND CHLORIDE AS COUNTER-ION (GP9)

Following the procedure described previously in literature,[8] water was added dropwise to a solution of diaryliodonium tosilate (4 mmol, 1 equiv.) in hot acetonitrile (5 mL, 60 ºC) until the mixture became clear. NH_4Cl (5 equiv.) in H_2O (4 mL) was added dropwise to the resulting solution. A solid precipitated on cooling the reaction mixture to room temperature. This precipitate was filtered off, washed with Et_2O and dried in air to give the corresponding diaryliodonium chloride.

EXCHANGE BETWEEN HALIDE AND ACETOXY AS COUNTER-ION (GP10)

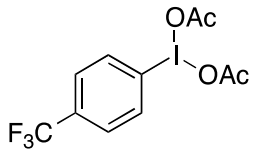
Following a modified procedure of the described previously in literature,[9] a preheated solution of AgOAc (1 equiv.) in AcOH (10 mL) and CH_3CN (3.3 mL) in an aluminium foil-covered-flask was added to a 60 ºC heated solution of diaryliodonium halide (4 mmol, 1 equiv.) in AcOH (10 mL) in an aluminium foil-covered-flask and the final mixture was stirred at 60 ºC for 45 minutes. The mixture was cooled and filtered though a Celite plug, washed with CH_3CN and concentrated under reduced pressure. The residue was triturated with Et_2O and dried under vacuum at 50 ºC overnight (if necessary) to afford the desired diaryliodonium acetate.

6. EXPERIMENTAL PROCEDURE FOR THE METAL-FREE BORYLATION OF DIARYL DIARYLIODONIUM SALTS (GP 11)

To an oven-dried Schlenk-type tube with a magnetic stir bar, B_2pin_2 (76.2 mg, 0.3 mmol) was added under argon. Then, the diaryliodonium salt (0.2 mmol) and the MeOH as solvent (1.25 mL) were added. The vial was sealed with a cteflon septum cap and heated to 50 ºC in an oil bath for 24 hours. The reaction mixture was then cooled to room temperature. An aliquot of 20 µL was diluted with 40 µL of a prepared solution of mesitylene as interal standard and analyzed by GC-MS to determine yield and selectivity, respectively. The reaction mixture and all the volatiles were removed under reduced pressure and the crude product was purified by column chromatography.

7. CHARACTERIZATION OF I(III) COMPOUNDS

1-Diacetoxyiodo-4-(trifluoromethyl)benzene



Synthesized according to GP1 as a white solid in 83% of yield.

^{1}H-NMR (400 MHz, $CDCl_3$): δ = 2.02 (s, 6H), 7.74 (d, *J* = 8.2 Hz, 2H), 8.22 (d, *J* = 8.2 Hz, 2H).

^{13}C-NMR (100 MHz, CD_3OD): δ = 20.5, 123.3 (q, *J* = 273.3 Hz), 124.6, 127.9 (q, *J* = 3.7 Hz), 133.8 (q, *J* = 33.4 Hz), 135.5, 176.7.

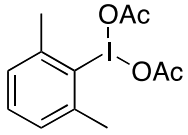
^{19}F-NMR (376 MHz, CD_3OD): δ = -63.3.

IR ν(cm^{-1}): 3095, 3044, 2989, 1641, 1597, 1425, 1400, 1369, 1323, 1294, 1270, 1192, 1166, 1115, 1069, 1054, 1005, 954, 926, 825.

HRMS (MALDI+): calc. for $[C_9H_7F_3IO_2]^+$: 330.9437; found: 330.9438.

m.p.(ºC): 103-104.

2-Diacetoxyiodo-1,3-dimethylbenzene



Synthesized according to GP1 as a pale yellow solid in 99% of yield. Data in agreement with those reported previously.[8]

^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.97 (s, 6H), 2.75 (s, 6H), 7.28-7.31 (m, 2H), 7.36-7.40 (m, 1H).

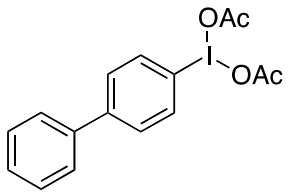
^{13}C-NMR (125 MHz, $CDCl_3$): δ = 20.4, 27.1, 128.2, 132.6, 133.1, 141.6, 176.6.

IR ν(cm^{-1}): 3066, 2930, 1463, 1585, 1456, 1368, 1268, 1166, 1032, 1008, 926.

HRMS (MALDI+): calc. for $[C_{10}H_{12}IO_2]^+$: 290.9876; found: 290.9820.

m.p.(ºC): 142-147.

4-Diacetoxyiodo-1,1'-biphenyl



Synthesized according to GP1 as a pale yellow solid in 68% of yield. Data in agreement with those reported previously.[10]

^{1}H-NMR (400 MHz, $CDCl_3$): δ = 2.03 (s, 6H), 7.37-7.52 (m, 3H), 7.54-7.61 (m, 2H), 7.68 (d, *J* = 8.5 Hz, 2H), 8.15 (d, *J* = 8.6 Hz, 2H).

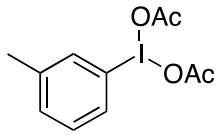
^{13}C-NMR (100 MHz, $CDCl_3$): δ = 20.6, 120.2, 127.5, 128.7, 129.2, 129.8, 135.6, 139.3, 145.1, 176.6.

IR ν(cm^{-1}): 3066, 2930, 1642, 1585, 1456, 1367, 1267, 1245, 1166, 1032, 1008, 998, 926.

HRMS (MALDI+): calc. for $[C_{14}H_{12}IO_2]^+$: 338.9876; found: 338.9848.

m.p.(ºC): 135-137.

3-Methyl-diacetoxyiodobenzene



Synthesized according to GP1 as a pale yellow solid in 93% of yield. Data in agreement with those reported previously.[11]

^{1}H-RMN (400 MHz, $CDCl_3$): δ = 2.01 (s, 6H), 2.43 (s, 3H), 7.36-7.42 (m, 2H), 7.86-7.94 (m, 2H).

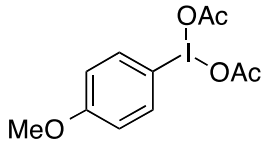
^{13}C-RMN (100 MHz, $CDCl_3$): δ = 20.5, 21.6, 121.6, 130.8, 132.2, 132.8, 135.5, 141.6, 176.5.

IR ν(cm^{-1}): 3044, 2924, 1640, 1595, 1473, 1427, 1364, 1291, 1267, 1097, 1042, 1008, 986, 925, 892.

HRMS (MALDI+): calc. for $[C_9H_{10}IO_2]^+$: 276.9720; found: 276.9729.

m.p.(ºC): 147-151.

1-Diacetoxyiodo-4-methoxybenzene



Synthesized according to GP2 as a white solid in 73% of yield. Data in agreement with those reported previously.[2]

^{1}H-NMR (400 MHz, $CDCl_3$): δ = 1.99 (s, 6H), 3.86 (s, 3H), 6.96 (d, *J* = 9.1 Hz, 2H), 8.01 (d, *J* = 9.1 Hz, 2H).

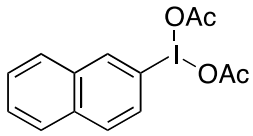
^{13}C-NMR (100 MHz, $CDCl_3$): δ = 20.5, 55.7, 111.8, 116.8, 137.3, 162.3, 176.5.

IR ν(cm^{-1}): 3088, 2978, 2934, 2840, 1642, 1572, 1492, 1456, 1441, 1364, 1289, 1250, 1181, 1021, 1007, 923, 827.

HRMS (MALDI+): calc. for $[C_9H_{10}IO_3]^+$: 292.9669; found: 292.9657.

m.p.(ºC): 85-87.

2-Diacetoxyiodonaphtalene



Synthesized according to GP2 as a white solid in 60% of yield. Data in agreement with those reported previously.[12]

^{1}H-NMR (400 MHz, $CDCl_3$): δ = 2.00 (s, 6H), 7.60-7.67 (m, 2H), 7.90-7.97 (m, 3H), 8.10 (dd, *J* = 1.8, 8.8 Hz, 1H), 8.66 (s, 1H).

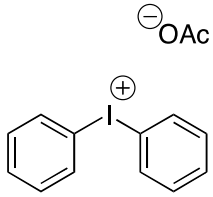
^{13}C-NMR (100 MHz, $CDCl_3$): δ = 20.5, 118.8, 127.7, 128.2, 128.7, 128.9, 130.5, 130.9, 134.1, 134.2, 136.2, 176.6.

IR ν(cm^{-1}): 3055, 1639, 1580, 1500, 1433, 1363, 1264, 1157, 1129, 1041, 1008, 935, 922, 893, 808.

HRMS (MALDI+): calc. for $[C_{12}H_{10}IO_2]^+$: 312.9720; found: 312.9757.

m.p.(ºC): 128-131.

diPhenyliodonium acetate (1d)



Isolated according to GP10 as a white solid in 90% of yield.

^{1}H-NMR (400 MHz, CD_3Cl): δ = 1.87 (s, 3H), 7.37 (t, *J* = 7.7 Hz, 4H), 7.47-7.52 (m, 2H), 7.89 (d, *J* = 7.5 Hz, 4H).

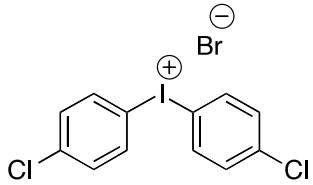
^{13}C-NMR (100 MHz, CD_3Cl): δ = 24.5, 120.3, 131.1, 131.4, 134.5, 179.0.

IR ν(cm^{-1}): 3050, 2995, 2914, 1566, 1545, 1471, 1448, 1439, 1384, 1328, 1272, 1179, 1096, 1066, 1010, 989, 929, 911, 833.

HRMS (ESI+): calc. for $[C_{12}H_{10}I]^+$: 280.9822; found: 280.9823.

m.p.(ºC): 160-161.

bis(4-Chlorophenyl)iodonium bromide



Synthesized according to GP3 (3 mL Ac_2O) as a brown solid in 52% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.58 (d, *J* = 8.6 Hz, 4H), 8.21 (d, *J* = 8.6 Hz, 4H).

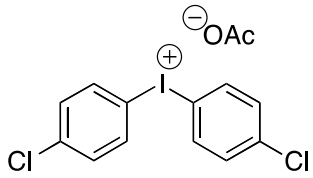
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 117.2, 131.5, 136.8, 136.9.

IR ν(cm^{-1}): 3073, 3041, 3022, 1563, 1472, 1442, 1387, 1265.

HRMS (ESI+): calc. for $[C_{12}H_8Cl_2I]$+: 348.9042; found: 348.9041.

m.p.(ºC): 193-194.

bis(4-Chlorophenyl)iodonium acetate (4)



Isolated according to GP10 as a brown solid in 87% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.88 (s, 3H), 7.56 (d, *J* = 8.5 Hz, 4H), 8.15 (d, *J* = 8.5 Hz, 4H).

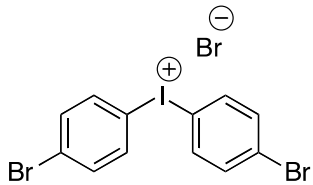
^{13}C-NMR (100 MHz, CD_3OD): δ = 20.9, 114.4, 133.1, 138.1, 140.1, 175.4.

IR ν(cm^{-1}): 3075, 3057, 1544, 1472, 1447, 1385, 1332, 1270.

HRMS (ESI+): calc. for $[C_{12}H_8Cl_2I]^+$: 348.9042; found: 348.9057.

m.p.(ºC): 153-154.

bis(4-Bromophenyl)iodonium bromide



Synthesized according to GP4 (20 mL H_2SO_4, 10 mL AcOH) as a pale yellow solid in 94% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.68 (d, *J* = 8.4 Hz, 4H), 8.12 (d, *J* = 8.4 Hz, 4H).

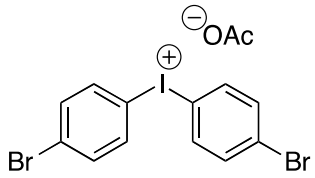
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 118.8, 125.5, 134.3, 136.8.

IR ν(cm^{-1}): 3067, 1619, 1551, 1464, 1433, 1411, 1375, 1262, 1169, 1100, 1087, 1060, 986.

HRMS (ESI+): calc. for $[C_{12}H_8Br_2I]^+$: 436.8032; found: 436.8040.

m.p.(ºC): 188-190.

bis(4-Bromophenyl)iodonium acetate (5)



Isolated according to GP10 as a pale brown solid in 86% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.91 (s, 3H), 7.69 (d, *J* = 8.6 Hz, 4H), 8.06 (d, *J* = 8.6 Hz, 4H).

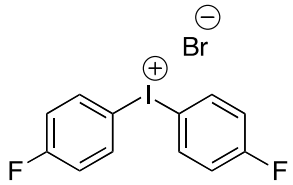
^{13}C-NMR (100 MHz, CD_3OD): δ = 23.1, 115.1, 128.5, 136.2, 138.1.

IR ν(cm^{-1}): 3072, 3055, 1544, 1472, 1443, 1424, 1382, 1334, 1265, 1230, 1181, 1111, 1091, 1067, 1051, 992, 915, 881.

HRMS (ESI+): calc. for $[C_{12}H_8Br_2I]^+$: 436.8032; found: 436.8028.

m.p.(ºC): 163-164.

bis(4-Fluorophenyl)iodonium bromide



Synthesized according to GP4 (20 mL H_2SO_4, 10 mL AcOH) as a pale yellow solid in 86% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.37 (t, *J* = 8.9 Hz, 4H), 8.24-8.29 (m, 4H).

^{13}C-NMR (100 MHz, DMSO-d_6): δ = 113.6, 118.9 (d, *J* = 22.5 Hz), 137.7 (d, *J* = 9.2 Hz), 163.7 (d, *J* = 250.8 Hz).

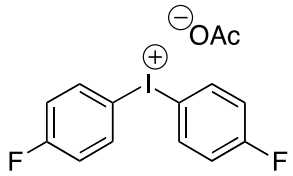
^{19}F-NMR (376 MHz, DMSO-d_6): δ = -107.0.

IR ν(cm^{-1}): 3093, 3054, 3025, 2959, 1644, 1579, 1484, 1398, 1281, 1237, 1221, 1163, 1093, 1049, 1009, 1000, 887.

HRMS (ESI+): calc. for $[C_{12}H_8F_2I]^+$: 316.9633; found: 316.9630.

m.p.(ºC): 166-167.

bis(4-Fluorophenyl)iodonium acetate (6)



Isolated according to GP10 as a pale brown solid in 56% of yield.

^{1}H-NMR (500 MHz, CD_3OD): δ = 1.88 (s, 3H), 7.29 (t, *J* = 8.7 Hz, 4H), 8.23-8.25 (m, 4H).

^{13}C-NMR (125 MHz, CD_3OD): δ = 24.1, 110.8, 120.4 (d, *J* = 23.2 Hz), 139.2 (d, *J* = 9.1 Hz), 166.3 (d, *J* = 253.6 Hz), 180.1.

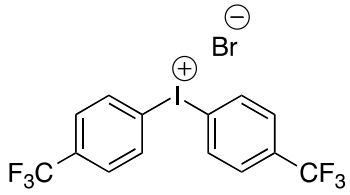
^{19}F-NMR (470 MHz, CD_3OD): δ = -107.2.

IR ν(cm^{-1}): 3096, 3034, 2964, 1592, 1575, 1554, 1480, 1390, 1330, 1309, 1292, 1222, 1174, 1158, 1070, 1002, 960.

HRMS (ESI+): calc. for $[C_{12}H_8F_2I]^+$: 316.9633; found: 316.9635.

m.p.(ºC): 166-167.

bis(4-(Trifluoromethyl)phenyl)iodonium bromide



Synthesized according to GP6 as a white solid in 59% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.86 (d, *J* = 8.3 Hz, 4H), 8.45 (d, *J* = 8.2 Hz, 4H).

^{13}C-NMR (100 MHz, DMSO-d_6): δ = 123.5 (q, *J* = 273.2 Hz), 124.8, 128.1 (q, *J* = 3.6 Hz), 131.4 (q, *J* = 32.4 Hz), 135.9.

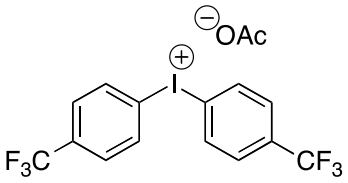
^{19}F-NMR (376 MHz, DMSO-d_6): δ = -61.3.

IR ν(cm^{-1}): 3062, 3032, 2969, 1595, 1400, 1326, 1158, 1126, 1105, 1066, 1047, 1004, 998, 969, 961, 849, 836, 770, 719.

HRMS (ESI+): calc. for $[C_{14}H_8F_6I]^+$: 416.9569; found: 416.9574.

m.p.(ºC): 217-218.

bis(4-(Trifluoromethyl)phenyl)iodonium acetate (7)



Isolated according to GP10 as a white solid in 85% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.87 (s, 3H), 7.85 (d, *J* = 8.3 Hz, 4H), 8.40 (d, *J* = 8.3 Hz, 4H).

^{13}C-NMR (100 MHz, CD_3OD): δ = 24.1, 120.9, 124.7 (q, *J* = 272.5 Hz), 129.8 (q, *J* = 3.7 Hz), 135.2 (q, *J* = 33.3 Hz), 137.4.

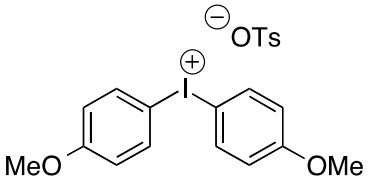
^{19}F-NMR (376 MHz, CD_3OD): δ = -64.6.

IR ν(cm^{-1}): 3097, 3037, 1594, 1535, 1396, 1320, 1158, 1118, 1106, 1067, 1047, 1000, 956, 920, 831, 822.

HRMS (ESI+): calc. for $[C_{14}H_8F_6I]^+$: 416.9569; found: 416.9573.

m.p.(ºC): 157-158.

bis(4-Methoxyphenyl)iodonium 4-methylbenzenesulfonate



Synthesized according to GP5 as a pale yellow solid in 99% of yield.

^{1}H-NMR (400 MHz, $CDCl_3$): δ = 2.32 (s, 3H), 3.80 (s, 6H), 6.85 (d, *J* = 9.1 Hz, 4H), 7.06 (d, *J* = 7.9 Hz, 2H), 7.61 (d, *J* = 8.1 Hz, 2H), 7.84 (d, *J* = 9.2 Hz, 4H).

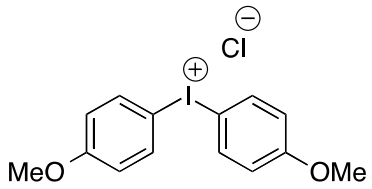
^{13}C-NMR (100 MHz, $CDCl_3$): δ = 21.4, 55.7, 104.4, 117.6, 126.2, 128.6, 136.9, 139.4, 143.0, 162.4.

IR ν(cm^{-1}): 3090, 2941, 2841, 1571, 1486, 1460, 1439, 1404, 1297, 1252, 1211, 1165, 1117, 1030, 1020, 1008, 991.

HRMS (ESI+): calc. for $[C_{14}H_{14}IO_2]^+$: 341.0033; found: 341.0032.

m.p.(ºC): 150-151.

bis(4-Methoxyphenyl)iodonium chloride



Isolated according to GP9 as a white solid in 79% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 3.84 (s, 6H), 7.05 (d, *J* = 8.7 Hz, 4H), 8.03 (d, *J* = 8.7 Hz, 4H).

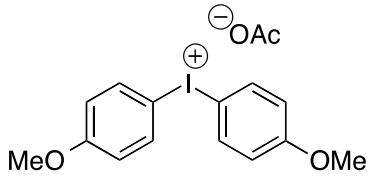
^{13}C-NMR (100 MHz, CD_3OD): δ = 56.3, 105.3, 118.7, 138.1, 164.4.

IR ν(cm^{-1}): 3030, 2967, 2941, 2839, 1570, 1484, 1456, 1401.

HRMS (ESI+): calc. for $[C_{14}H_{14}IO_2]^+$: 341.0033; found: 341.0029.

m.p.(ºC): 215-216.

bis(4-Methoxyphenyl)iodonium acetate (8)



Isolated according to GP10 as a grey semisolid in 60% of yield.

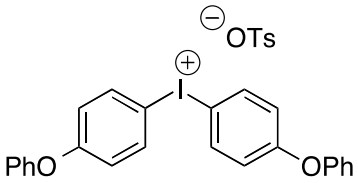
^{1}H-NMR (400 MHz, CD_3OD): δ = 1.90 (s, 3H), 3.84 (s, 6H), 7.04 (d, *J* = 9.0 Hz, 4H), 8.03 (d, *J* = 9.1 Hz, 4H).

^{13}C-NMR (100 MHz, CD_3OD): δ = 22.9, 56.3, 105.3, 118.7, 138.1, 164.4.

IR ν(cm^{-1}): 2968, 2941, 2840, 1571, 1542, 1486, 1460, 1440, 1389, 1295, 1247, 1173, 1117, 1018, 991.

HRMS (ESI+): calc. for $[C_{14}H_{14}IO_2]^+$: 341.0033; found: 341.0031.

bis(4-Phenoxyphenyl)iodonium 4-methylbenzenesulfonate



Synthesized according to GP5 as a pale yellow solid in 97% of yield.

^{1}H-NMR (400 MHz, $CDCl_3$) δ = 2.30 (s, 3H), 6.83-6.87 (m, 4H), 6.98-7.05 (m, 6H), 7.18-7.22 (m, 2H), 7.35-7.40 (m, 4H), 7.48-7.51 (m, 2H), 7.85-7.90 (m, 4H).

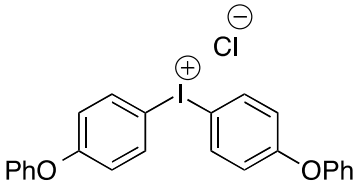
^{13}C-NMR (100 MHz, $CDCl_3$) δ = 21.5, 107.1, 120.4, 125.2, 126.1, 128.6, 130.3, 137.2, 139.5, 142.8, 155.0, 161.0.

IR ν(cm^{-1}): 3059, 1569, 1478, 1222, 1152, 1006, 750.

HRMS (ESI+): calc. for $[C_{24}H_{18}IO_2]^+$: 465.0346; found: 465.0331.

m.p.(ºC): 161-163.

bis(4-Phenoxyphenyl)iodonium chloride



Isolated according to GP9 as a white solid in 81% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.02 (d, *J* = 9.0 Hz, 4H), 7.07-7.11 (m, 4H), 7.21-7.28 (m, 2H), 7.41-7.48 (m, 4H), 8.12 (d, *J* = 9.0 Hz, 4H).

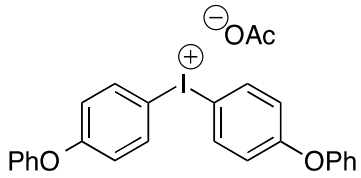
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 120.0, 120.1, 124.9, 130.4, 137.0, 154.7, 159.7.

IR ν(cm^{-1}): 3051, 3012, 1592, 1569, 1475, 1397, 1284, 1230, 1195, 1168, 1111, 1068, 1021, 997, 953, 939, 912 862.

HRMS (ESI+): calc. for $[C_{24}H_{18}IO_2]^+$: 465.0346; found: 465.0345.

m.p.(ºC): 213-214.

bis(4-Phenoxyphenyl)iodonium acetate (9)



Isolated according to GP10 as a brown solid in 87% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 7.01-7.10 (m, 8H), 7.22-7.28 (m, 2H), 7.40-7.47 (m, 4H), 8.09 (d, *J* = 9.1 Hz, 4H).

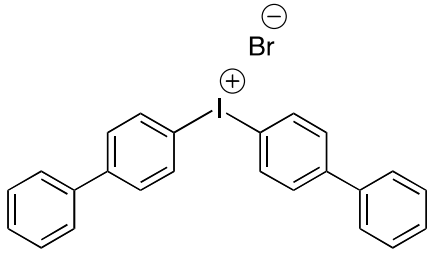
^{13}C-NMR (100 MHz, CD_3OD): δ = 23.9, 107.6, 121.5, 121.6, 126.4, 131.4, 138.5, 156.3, 160.0.

IR ν(cm^{-1}): 3057, 3038, 1712, 1590, 1570, 1543, 1477, 1390, 1333, 1299, 1282, 1234, 1192, 1164, 1114, 1097, 1072, 1049, 1022, 999, 963, 912, 891, 863, 852, 828, 793, 776, 750.

HRMS (ESI+): calc. for $[C_{24}H_{18}IO_2]^+$: 465.0346; found: 465.0348.

m.p.(ºC): 160-162.

di([1,1'-Biphenyl]-4-yl)iodonium bromide



Synthesized according to GP6 as a brown solid in 85% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.38-7.44 (m, 2H), 7.45-7.50 (m, 4H), 7.66-7.71 (m, 4H), 7.77 (d, *J* = 8.6 Hz, 4H), 8.31 (d, *J* = 8.5 Hz, 4H).

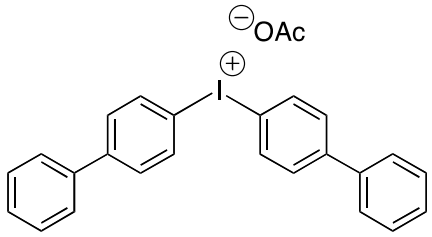
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 117.8, 127.0, 128.5, 129.1, 129.6, 135.6, 138.2, 143.1.

IR ν(cm^{-1}): 3047, 3030, 1599, 1580, 1557, 1475, 1446, 1389, 1309, 1281, 1191, 1157, 1119, 1103, 1076, 1028, 1009, 994, 915.

HRMS (ESI+): calc. for $[C_{24}H_{18}I]^+$: 433.0448; found: 433.0443.

m.p.(ºC): 186-187.

di([1,1'-Biphenyl]-4-yl)iodonium acetate (10)



Isolated according to GP10 as a pale brown solid in 95% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.90 (s, 3H), 7.39-7.43 (m, 2H), 7.45-7.49 (m, 4H), 7.62-7.65 (m, 4H), 7.78 (d, *J* = 8.6 Hz, 4H), 8.25 (d, *J* = 8.6 Hz, 4H).

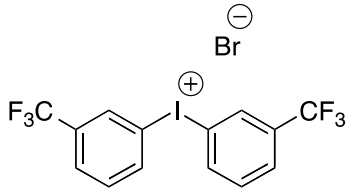
^{13}C-NMR (100 MHz, CD_3OD): δ = 23.6, 114.6, 128.3, 129.9, 130.2, 131.5, 136.9, 140.0, 146.9.

IR ν(cm^{-1}): 3054, 3025, 1708, 1539, 1475, 1445, 1389, 1334, 1282, 1245, 1190, 1153, 1105, 1074, 1027, 996, 913.

HRMS (ESI+): calc. for $[C_{24}H_{18}I]^+$: 433.0448; found: 433.0449.

m.p.(ºC): 142-145.

bis(3-(Trifluoromethyl)phenyl)iodonium bromide



Synthesized according to GP3 (0 mL Ac_2O) as a white solid in 47% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.71 (t, *J* = 7.9 Hz, 2H), 7.98 (d, *J* = 7.7 Hz, 2H), 8.54 (d, *J* = 7.8 Hz, 2H), 8.75 (s, 2H).

^{13}C-NMR (100 MHz, DMSO-d_6): δ = 120.9, 123.0 (q, *J* = 273.2 Hz), 128.2 (q, *J* = 3.5 Hz), 131.0 (q, *J* = 32.3 Hz), 131.6 (q, *J* = 3.8 Hz), 132.3, 139.0.

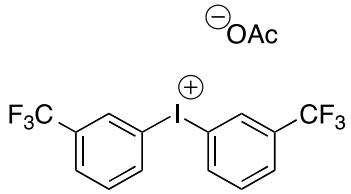
^{19}F-RMN (376 MHz, DMSO-d_6): δ = -60.9.

IR ν(cm^{-1}): 3053, 3035, 3019, 1599, 1422, 1320, 1305, 1277, 1191, 1169, 1122, 1094, 1082, 1053, 992, 915.

HRMS (ESI+): calc. for $[C_{14}H_8F_6I]^+$: 416.9569; found: 416.9570.

m.p.(ºC): 196-205.

bis(3-(Trifluoromethyl)phenyl)iodonium acetate (11)



Isolated according to GP10 as a grey liquid in 99% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.92 (s, 3H), 7.73 (t, *J* = 7.9 Hz, 2H), 8.00 (d, *J* = 7.8 Hz), 8.48 (d, *J* = 7.9 Hz, 2H), 8.66 (s, 2H).

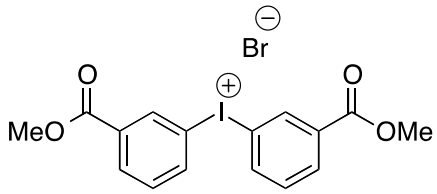
^{13}C-NMR (100 MHz, CD_3OD): δ = 22.4, 117.8, 124.2 (q, *J* = 272.5 Hz), 130.3, 133.3 (q, *J* = 3.9 Hz), 133.8, 134.5 (q, *J* = 33.6 Hz), 140.2, 177.7.

^{19}F-RMN (376 MHz, DMSO-d_6): δ = -64.1.

IR ν(cm^{-1}): 3066, 1711, 1536, 1421, 1394, 1318, 1306, 1276, 1169, 1123, 1095, 1079, 1055, 1006, 993, 934, 886, 793.

HRMS (ESI+): calc. for $[C_{14}H_8F_6I]^+$: 416.9569; found: 416.9575.

bis(3-(Methoxycarbonyl)phenyl)iodonium bromide



Synthesized according to GP3 (0 mL Ac_2O) as a pale brown solid in 70% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 3.38 (s, 6H), 7.64 (t, *J* = 7.4 Hz, 2H), 8.14 (d, *J* = 7.4 Hz, 2H), 8.52 (d, *J* = 7.4 Hz, 2H), 8.83 (s, 2H).

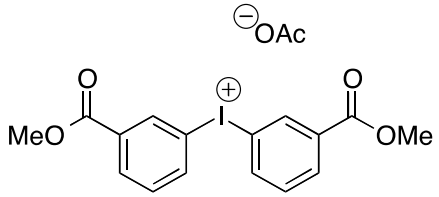
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 52.7, 119.0, 131.9, 132.1, 132.2, 135.5, 139.5, 164.6.

IR ν(cm^{-1}): 3029, 2952, 1722, 1708, 1590, 1559, 1433, 1417.

HRMS (ESI+): calc. for $[C_{16}H_{14}IO_4]^+$: 396.9931; found: 396.9930.

m.p.(ºC): 140-145.

bis(3-(Methoxycarbonyl)phenyl)iodonium acetate (12)



Isolated according to GP10 as a brown solid in 60% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.94 (s, 3H), 3.93 (s, 6H), 7.64 (t, *J* = 7.9 Hz, 2H), 8.25 (d, *J* = 7.6 Hz, 2H), 8.43 (d, *J* = 7.4 Hz, 2H), 8.81 (s, 2H).

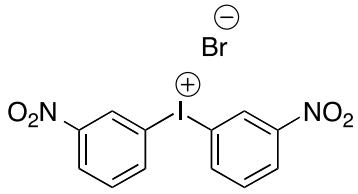
^{13}C-NMR (100 MHz, CD_3OD): δ = 22.1, 53.3, 117.0, 133.1, 134.0, 134.7, 137.1, 140.7, 166.0, 177.1.

IR ν(cm^{-1}): 3056, 2952, 1713, 1589, 1540, 1435, 1390, 1363, 1261, 1194, 1115, 1082, 1057, 994, 961, 878.

HRMS (ESI+): calc. for $[C_{16}H_{14}IO_4]^+$: 396.9931; found: 396.9926.

m.p.(ºC): 113-114.

bis(3-Nitrophenyl)iodonium bromide



Synthesized according to GP3 (0 mL Ac_2O) as a yellow solid in 66% of yield.

^{1}H-NMR (500 MHz, DMSO-d_6): δ = 7.77 (t, *J* = 8.2 Hz, 2H), 8.40 (d, *J* = 7.8 Hz, 2H), 8.67 (d, *J* = 8.0 Hz, 2H), 9.21 (s, 2H).

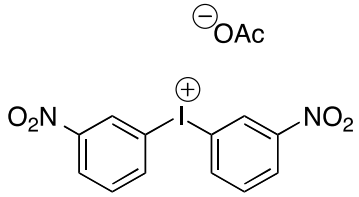
^{13}C-NMR (125 MHz, DMSO-d_6): δ = 120.8, 126.3, 129.8, 132.4, 141.1, 148.2.

IR ν(cm^{-1}): 3083, 3034, 3006, 2862, 1595, 1524, 1460, 1422, 1344, 1303, 1259.

HRMS (ESI+): calc. for $[C_{12}H_8IN_2O_4]^+$: 370.9523; found: 370.9529.

m.p.(ºC): 199-200.

bis(3-Nitrophenyl)iodonium acetate (13)



Isolated according to GP10 as a yellow solid in 75% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.86 (s, 3H), 7.78 (t, *J* = 8.1 Hz, 2H), 8.50 (d, *J* = 8.6 Hz, 2H), 8.60 (d, *J* = 8.0 Hz, 2H), 9.20 (s, 2H).

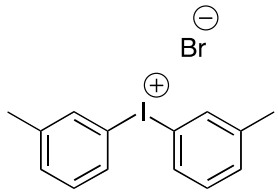
^{13}C-NMR (100 MHz, CD_3OD): δ = 24.0, 117.7, 128.2, 131.4, 133.9, 142.1, 150.5, 180.1.

IR ν(cm^{-1}): 3088, 3036, 2866, 1596, 1525, 1466, 1418, 1396, 1347, 1300.

HRMS (ESI+): calc. for $[C_{12}H_8IN_2O_4]^+$: 370.9523; found: 370.9530.

m.p.(ºC): 124-125.

bis(3-Methylphenyl)iodonium bromide



Synthesized according to GP6 as a white solid in 88% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 2.32 (s, 3H), 7.37 (t, *J* = 7.7 Hz, 1H), 7.43 (d, *J* = 7.4 Hz, 1H), 7.98 (d, *J* = 7.7 Hz, 1H), 8.05 (s, 1H).

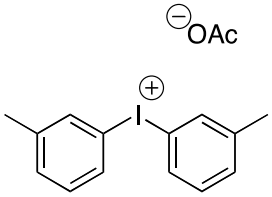
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 20.8, 118.2, 131.2, 132.0, 132.2, 135.1, 141.4.

IR ν(cm^{-1}): 3042, 3021, 2998, 2916, 1593, 1561, 1474, 1442, 1378, 1274, 1210, 1093, 1056, 987, 917.

HRMS (ESI+): calc. for $[C_{14}H_{14}I]^+$: 309.0135; found: 309.0133.

m.p.(ºC): 221-224.

bis(3-Methylphenyl)iodonium acetate (14)



Isolated according to GP10 as a grey solid in 99% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.87 (s, 3H), 2.40 (s, 6H), 7.40 (t, *J* = 7.8 Hz, 2H), 7.51 (d, *J* = 7.7 Hz, 2H), 7.94 (d, *J* = 7.9 Hz, 2H), 8.01 (s, 2H).

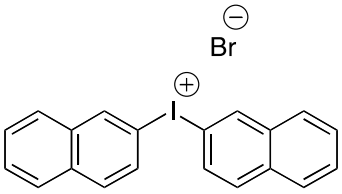
^{13}C-NMR (100 MHz, CD_3OD): δ = 21.2, 24.2, 115.9, 132.8, 133.4, 134.3, 136.5, 144.4.

IR ν(cm^{-1}): 3033, 3007, 2918, 1596, 1547, 1472, 1416, 1380, 1327, 1276, 1213, 1171, 1068, 1040, 1001, 984, 908.

HRMS (ESI+): calc. for $[C_{14}H_{14}I]^+$: 309.0135; found: 309.0126.

m.p.(ºC): 140-143.

di(Naphalen-2-yl)iodonium bromide



Synthesized according to GP6 as a brown solid in 18% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.64-7.68 (m, 2H), 7.96-8.03 (m, 3H), 8.23 (dd, *J* = 1.8, 8.8 Hz, 1H), 8.94-8.96 (m, 1H).

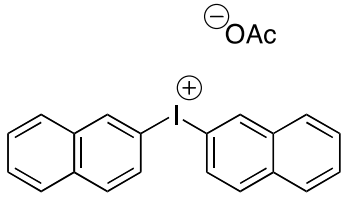
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 115.8, 127.7, 128.0, 128.1, 128.6, 130.2, 131.1, 133.2, 133.8, 135.7.

IR ν(cm^{-1}): 3040, 3022, 1762, 1662, 1577, 1498, 1347, 1267, 1239, 1196, 1160, 1144, 1131, 1019, 962, 949, 931, 856.

HRMS (ESI+): calc. for $[C_{20}H_{14}I]^+$: 381.0135; found: 381.0141.

m.p.(ºC): 161-165.

di(Naphalen-2-yl)iodonium acetate (15)



Isolated according to GP10 as a brown solid in 43% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.92 (s, 3H), 7.65-7.70 (m, 2H), 7.94-8.03 (m, 3H), 8.14 (dd, *J* = 1.7, 8.9 Hz, 1H), 8.87 (s, 1H).

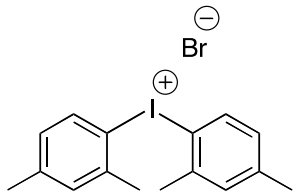
^{13}C-NMR (100 MHz, CD_3OD): δ = 23.0, 112.7, 129.2, 129.3, 129.4, 130.4, 130.8, 133.1, 135.6, 135.9, 137.7.

IR ν(cm^{-1}): 3051, 2926, 1704, 1666, 1513, 1381, 1343, 1265, 1158, 1130, 1033, 962, 930, 882, 855.

HRMS (ESI+): calc. for $[C_{20}H_{14}I]^+$: 381.0135; found: 381.0128.

m.p.(ºC): 130-140.

Bis(2,4-Dimethylphenyl)iodonium bromide



Synthesized according to GP4 (5 mL H_2SO_4, 35 mL AcOH) as a yellow solid in 52% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 2.37 (s, 6H), 2.60 (s, 6H), 7.09-7.14 (m, 2H), 7.36-7.39 (m, 2H), 8.03 (d, *J* = 8.3 Hz, 2H).

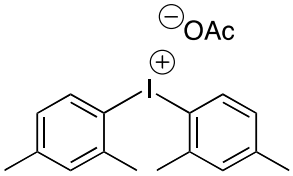
^{13}C-NMR (100 MHz, CD_3OD): δ = 21.2, 25.3, 116.6, 131.3, 133.7, 138.2, 142.2, 145.6.

IR ν(cm^{-1}): 2913, 1590, 1440, 1377, 1274, 1234, 1166, 1142, 1035, 1001, 990.

HRMS (ESI+): calc. for $[C_{16}H_{18}I]^+$: 337.0448; found: 337.0448.

m.p.(ºC): 162-163.

Bis(2,4-Dimethylphenyl)iodonium acetate (16)



Isolated according to GP10 as a pale yellow solid in 50% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.91 (s, 3H), 2.37 (s, 6H), 2.60 (s, 6H), 7.12 (dd, *J* = 1.7, 8.3 Hz, 2H), 7.38 (s, 2H), 8.03 (d, *J* = 8.3 Hz, 2H).

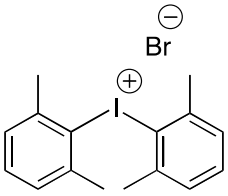
^{13}C-NMR (100 MHz, CD_3OD): δ = 21.2, 23.2, 25.3, 116.4, 131.3, 133.7, 138.2, 142.2, 145.7.

IR ν(cm^{-1}): 3001, 2955, 2918, 2860, 1552, 1471, 1444, 1385, 1328, 1233, 1170, 1146, 1035, 1003, 915.

HRMS (ESI+): calc. for $[C_{16}H_{18}I]^+$: 337.0448; found: 337.0451.

m.p.(ºC): 138-139.

Bis(2,6-Dimethylphenyl)iodonium bromide



Synthesized according to GP6 as a pale yellow solid in 36% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 7.30-7.34 (m, 4H), 7.41-7.46 (m, 2H).

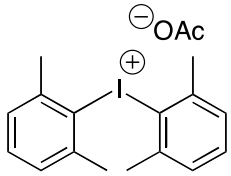
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 25.7, 124.8, 129.4, 132.1, 141.9.

IR ν(cm^{-1}): 2973, 1580,1459, 1375, 1304, 1252, 1022, 784.

HRMS (ESI+): calc. for $[C_{16}H_{18}I]^+$: 337.0448; found: 337.0454.

m.p.(ºC): 144-145.

Bis(2,6-Dimethylphenyl)iodonium acetate (17)



Isolated according to GP10 as a white solid in 96% of yield.

^{1}H-RMN (400 MHz, CD_3OD): δ = 1.88 (s, 3H), 2.59 (s, 12H), 7.35-7.40 (m, 4H), 7.45-7.51 (m, 2H).

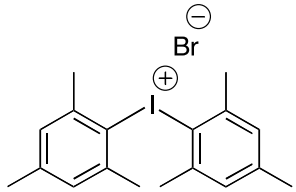
^{13}C-RMN (100 MHz, CD_3OD): δ = 24.2, 26.3, 122.8, 131.1, 134.2, 144.0.

IR ν(cm^{-1}): 3050, 2963, 2917, 1574, 1552, 1455, 1381, 1325, 1248, 1170, 1112, 1030, 986.

HRMS (ESI+): calc. for $[C_{16}H_{18}I]^+$: 337.0448; found: 337.0447.

m.p.(ºC): 126-127.

bis(Mesityl)iodonium bromide



Synthesized according to GP4 (5 mL H_2SO_4, 35 mL AcOH) as a yellow solid in 13% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 2.27 (s, 6H), 2.46 (s, 12H), 7.15 (s, 4H).

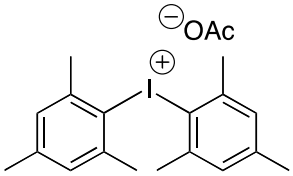
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 20.4, 25.4, 120.3, 130.2, 141.8, 142.4.

IR ν(cm^{-1}): 3017, 2953, 2920, 1588, 1570, 1443, 1375, 1298, 1247, 1177, 1033, 983.

HRMS (ESI+): calc. for $[C_{18}H_{22}I]^+$: 365.0761; found: 365.0762.

m.p.(ºC): 134-135.

bis(Mesityl)iodonium acetate (18)



Isolated according to GP10 as a pale yellow solid in 73% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.93 (s, 3H), 2.35 (s, 6H), 2.53 (s, 12), 7.19 (s, 4H).

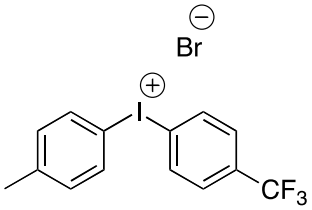
^{13}C-NMR (100 MHz, CD_3OD): δ = 20.8, 22.8, 26.1, 119.1, 131.8, 143.6, 145.3.

IR ν(cm^{-1}): 3017, 2967, 2920, 1666, 1563, 1447, 1375, 1351, 1297, 1178, 1104, 1035, 986, 944, 851.

HRMS (ESI+): calc. for $[C_{18}H_{22}I]^+$: 365.0761; found: 365.0757.

m.p.(ºC): 117-119.

***p*-Tolyl(4-(trifluoromethyl)phenyl)iodonium bromide**



Synthesized according to GP7 (*p*-diacetoxyiodo-trifluorotoluene as diacetoxyiodoarene and toluene as arene) as a white solid in 54% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 2.41 (s, 3H), 7.37 (d, *J* = 8.2 Hz, 2H), 7.81 (d, *J* = 8.3 Hz, 2H), 8.08 (d, *J* = 8.4 Hz, 2H), 8.33 (d, *J* = 8.1 Hz, 2H).

^{13}C-NMR (100 MHz, CD_3OD): δ = 21.4, 113.5, 121.0, 124.8 (q, *J* = 272.8 Hz), 129.5 (q, *J* = 3.8 Hz), 134.0, 134.9 (q, *J* = 33.6 Hz), 136.6, 136,9, 145.3.

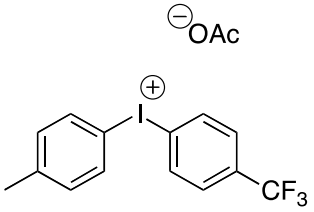
^{19}F-NMR (376 MHz, CD_3OD): δ = -64.8.

IR ν(cm^{-1}): 3028, 1595, 1487, 1398, 1322, 1168, 1124.

HRMS (ESI+): calc. for $[C_{14}H_{11}F_3I]^+$: 362.9852; found: 362.9852.

m.p.(ºC): 218-219.

_p_-Tolyl(4-(trifluoromethyl)phenyl)iodonium acetate (19)



Isolated according to GP10 as a white solid in 55% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.87 (s, 3H), 2.42 (s, 3H), 7.37 (d, *J* = 8.2 Hz, 2H), 7.81 (d, *J* = 8.4 Hz, 2H), 8.08 (d, *J* = 8.4 Hz, 2H), 8.32 (d, *J* = 8.3 Hz, 2H).

^{13}C-NMR (100 MHz, CD_3OD): δ = 21.4, 24.2, 113.0, 120.5, 123.4 (q, *J* = 272.0 Hz), 129.5 (q, *J* = 3.7 Hz), 134.0, 134.9 (q, *J* = 33.1 Hz), 136.7, 136.9, 145.4, 180.2.

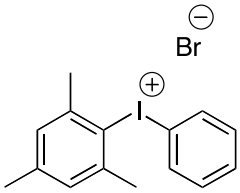
^{19}F-NMR (376 MHz, CD_3OD): δ = -64.50.

IR ν(cm^{-1}): 3030, 2920, 1596, 1552, 1486, 1393, 1321, 1208, 1162, 1114, 1102, 1069, 1051, 1002.

HRMS (ESI+): calc. for $[C_{14}H_{11}F_3I]^+$: 362.9852; found: 362.9844.

m.p.(ºC): 148-149.

Mesityl(phenyl)iodonium bromide



Synthesized according to GP7 (diacetoxyiodobenzene as diacetoxyiodoarene and mesitylene as arene) as a white solid in 77% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 2.36 (s, 3H), 2.66 (s, 6H), 7.23 (s, 2H), 7.47-7.52 (m, 2H), 7.60-7.65 (m, 1H), 7.86-7.91 (m, 2H).

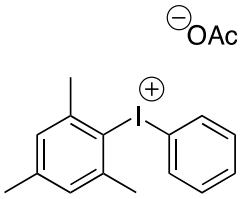
^{13}C-NMR (100 MHz, CD_3OD): δ = 21.0, 27.0, 115.2, 123.3, 131.3, 133.1, 133.2, 135.1, 143.3, 145.7.

IR ν(cm^{-1}): 3049, 2977, 2919, 1588, 1562, 1469, 1454, 1440, 1377, 1324, 1298, 1269, 1244, 1175.

HRMS (ESI+): calc. for $[C_{15}H_{16}I]^+$: 323.0291; found: 323.0289.

m.p.(ºC): 172-173.

Mesityl(phenyl)iodonium acetate (20)



Isolated according to GP10 as a white solid in 60% of yield.

^{1}H-NMR (300 MHz, CD_3OD): δ = 1.88 (s, 3H), 2.36 (s, 3H), 2.66 (s, 6H), 7.24 (s, 2H), 7.51 (t, *J* = 7.7 Hz, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.90 (d, *J* = 7.6 Hz, 2H).

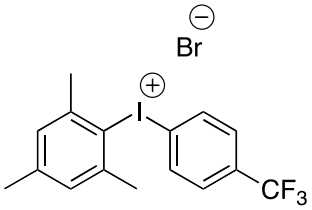
^{13}C-NMR (75 MHz, CD_3OD): δ = 21.0, 24.2, 27.0, 114.3, 122.4, 131.3, 133.3, 135.2, 143.5, 145.8.

IR ν(cm^{-1}): 3052, 2970, 2915, 1571, 1539, 1477, 1462, 1444, 1418, 1385, 1328, 1296.

HRMS (ESI+): calc. for $[C_{15}H_{16}I]^+$: 323.0291; found: 323.0291.

m.p.(ºC): 130-131.

Mesityl(4-(trifluoromethyl)phenyl)iodonium bromide



Synthesized according to GP8 (*p*-iodo-trifluorotoluene as iodoarene and mesitylene as arene) as a grey solid in 27% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 2.29 (s, 3H), 2.59 (s, 6H), 7.19 (s, 2H), 7.80 (d, *J* = 8.4 Hz, 2H), 8.06 (d, *J* = 8.2 Hz, 2H).

^{13}C-NMR (100 MHz, DMSO-d_6): δ = 20.5, 26.3, 123.1 (q, *J* = 1.4 Hz), 123.5 (q, *J* = 272.8 Hz), 125.5, 128.0 (q, *J* = 3.8 Hz), 129.7, 131.0 (q, *J* = 32.5 Hz), 134.5, 141.0, 142.6.

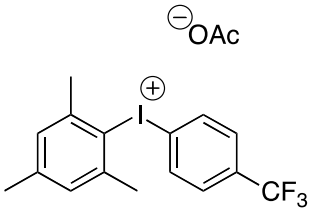
^{19}F-NMR (376 MHz, DMSO-d_6): δ = -61.3.

IR ν(cm^{-1}): 3027, 2980, 2955, 2917, 1592, 1485, 1451, 1392, 1319, 1245, 1163, 1127, 1101, 1064, 1045, 1031, 1002, 990, 954.

HRMS (ESI+): calc. for $[C_{16}H_{15}F_3I]^+$: 391.0165; found: 391.0159.

m.p.(ºC): 167-168.

Mesityl(4-(trifluoromethyl)phenyl)iodonium acetate (21)



Isolated according to GP10 as a white solid in 71% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.87 (s, 3H), 2.38 (s, 3H), 2.66 (s, 6H), 7.27 (s, 2H), 7.81 (d, *J* = 8.4 Hz, 2H), 8.07 (d, *J* =8.4 Hz, 2H).

^{13}C-NMR (100 MHz, CD_3OD): δ = 21.1, 24.1, 27.0, 118.4, 122.6, 123.0 (q, *J* = 272.4 Hz), 129.8 (q, *J* = 3.7 Hz), 131.5, 134.8 (q, *J* = 33.3 Hz), 135.8, 143.6, 146.2.

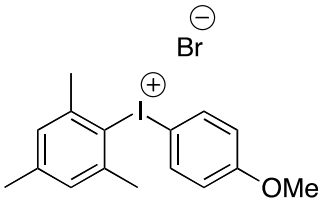
^{19}F-NMR (376 MHz, CD_3OD): δ□= −64.5.

IR ν(cm^{-1}): 2977, 2924, 1594, 1544, 1490, 1461, 1422, 1389, 1320, 1299, 1239, 1165, 1127, 1105, 1068, 1049, 1029, 1003, 915.

HRMS (ESI+): calc. for $[C_{16}H_{15}F_3I]^+$: 391.0165; found: 391.0160.

m.p.(ºC): 147-148.

Mesityl(4-methoxyphenyl)iodonium bromide



Synthesized according to GP7 (*p*-diacetoxyiodo-anisole as diacetoxyiodoarene and mesitylene as arene) as a white solid in 48% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 2.27 (s, 3H), 2.59 (s, 6H), 3.76 (s, 3H), 6.99 (d, *J* = 9.1 Hz, 2H), 7.15 (s, 2H), 7.84 (d, *J* = 9.1 Hz, 2H).

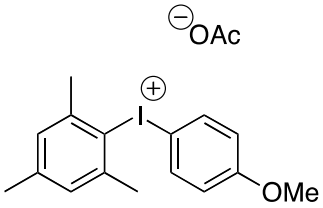
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 20.5, 26.2, 55.6, 106.1, 117.2, 125.0, 129.5, 136.0, 140.9, 142.3, 161.3.

IR ν(cm^{-1}): 2978, 2943, 2916, 2834, 1580, 1569, 1484, 1457, 1444, 1398, 1380, 1297, 1249, 1168, 1113, 1100, 1051, 1024, 989.

HRMS (ESI+): calc. for $[C_{16}H_{18}IO]^+$: 353.0397; found: 353.0398.

m.p.(ºC): 163-164.

Mesityl(4-methoxyphenyl)iodonium acetate (22)



Isolated according to GP10 as a pale brown solid in 83% of yield.

^{1}H-RMN (300 MHz, CD_3OD): δ = 1.88 (s, 3H), 2.35 (s, 3H), 2.67 (s, 6H), 3.83 (s, 3H), 7.04 (d, *J* = 9.0 Hz, 2H), 7.21 (s, 2H), 7.85 (d, *J* = 9.2 Hz, 2H).

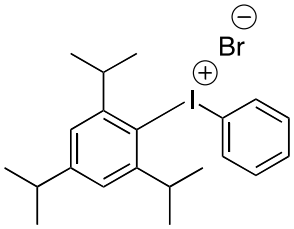
^{13}C-RMN (75 MHz, CD_3OD): δ = 21.0, 24.2, 27.0, 56.3, 102.8, 118.9, 122.9, 131.2, 137.4, 143.2, 145.5, 164.2.

IR ν(cm^{-1}): 3003, 2970, 2917, 2840, 1572, 1546, 1487, 1461, 1447, 1376, 1324, 1295, 1250, 1172, 1117, 1104, 1034, 1018, 994, 947, 909

HRMS (ESI+): calc. for $[C_{16}H_{18}IO]^+$: 353.0397; found: 353.0390.

m.p.(ºC): 166-167.

Phenyl(2,4,6-triisopropylphenyl)iodonium bromide



Synthesized according to GP8 (iodobenzene as iodoarene and mesitylene as 1,3,5-triisopropylbenzene as arene) as a white solid in 47% of yield.

^{1}H-NMR (400 MHz, DMSO-d_6): δ = 1.15-1.30 (m, 18H), 2.96 (hp, *J* = 6.9 Hz, 1H), 3.39 (hp, *J* = 6.7 Hz, 2H), 7.27 (s, 2H), 7.44-7.50 (m, 2H), 7.54-7.59 (m, 1H), 7.84 (d, *J* = 7.7 Hz, 2H).

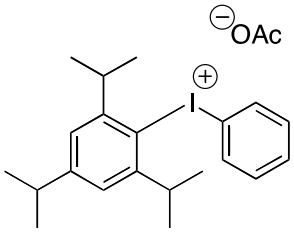
^{13}C-NMR (100 MHz, DMSO-d_6): δ = 23.5, 23.9, 33.3, 38.6, 118.0, 124.3, 125.5, 131.1, 131.6, 133.4, 150.7, 153.5.

IR ν(cm^{-1}): 3049, 2964, 2931, 2867, 1585, 1564, 1462, 1439, 1410, 1385, 1362, 1324, 1308, 1296, 1258, 1191, 1170, 1154, 1098, 1064, 1010, 990, 957, 937, 922, 910.

HRMS (ESI+): calc. for $[C_{21}H_{28}I]^+$: 407.1230; found: 407.1226.

m.p.(ºC): 158-159.

Phenyl(2,4,6-triisopropylphenyl)iodonium acetate (23)



Isolated according to GP10 as grey oil in 99% of yield.

^{1}H-NMR (400 MHz, CD_3OD): δ = 1.25-1.32 (m, 18H), 1.96 (s, 3H), 3.02 (hp, *J* = 6.9 Hz, 1H), 3.42 (hp, *J* = 6.7 Hz, 2H), 7.34 (s, 2H), 7.50-7.54 (m, 2H), 7.61-7.66 (m, 1H), 7.83-7.86 (m, 2H)

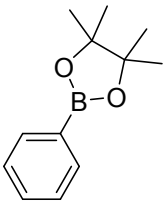
^{13}C-NMR (100 MHz, CD_3OD): δ = 21.8, 24.0, 24.5, 35.4, 40.6, 115.1, 123.4, 126.3, 133.3, 134.7, 153.3, 156.8, 176.7.

IR ν(cm^{-1}): 2965, 2931, 2873, 1709, 1542, 1464, 1441, 1386, 1364, 1242, 1156, 1101, 1068, 1055, 1010, 986, 937, 877, 735.

HRMS (ESI+): calc. for $[C_{21}H_{28}I]^+$: 407.1230; found: 407.1246.

8. CHARACTERIZATION OF THE BORYLATED PRODUCTS

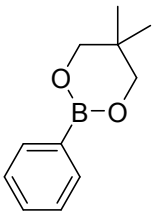
4,4,5,5-Tetramethyl-2-phenyl-1,3,2-dioxaborolane (3a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (2%). It was obtained as yellowish oil (77%). Data in agreement with the literature values.[13]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.34 (s, 12H), 7.34-7.40 (m, 2H), 7.43-7.49 (m, 1H), 7.81 (dd, *J* = 1.4, 8.0 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 25.0, 83.9, 127.9, 131.4, 134.9.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 31.1.
MS (70 eV) m/z: 204.1 [M^+].

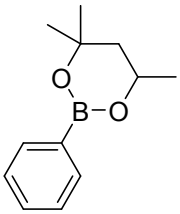
5,5-Dimethyl-2-phenyl-1,3,2-dioxaborinane (3b)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (2%). It was obtained as white solid (37%). Data in agreement with the literature values.[14]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.03 (s, 6H), 3.78 (s, 4H), 7.36 (ddd, *J* = 1.0, 4.3, 8.2 Hz, 2H), 7.41-7.46 (m, 1H), 7.80 (dd, *J* = 1.4, 8.0 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 29.8, 32.0, 72.4, 127.7, 130.8, 133.9.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 29.3.
MS (70 eV) m/z: 190.1 [M^+].

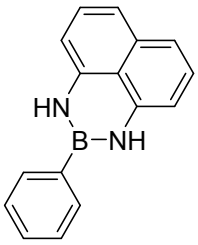
4,4,6-Trimethyl-2-phenyl-1,3,2-dioxaborinane (3c)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (2%). It was obtained as yellowish oil (34%). Data in agreement with the literature values.[15]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.35 (d, *J* = 6.2 Hz, 3H), 1.37 (s, 3H), 1.38 (s, 3H), 1.56-1.63 (m, 1H), 1.87 (dd, *J* = 13.9, 2.9 Hz, 1H), 4.35 (bs, 1H), 7.32-7.36 (m, 2H), 7.38-7.82 (m, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 23.5, 28.5, 31.6, 46.3, 65.3, 71.3, 127.8, 130.7, 134.1.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 27.0.
MS (70 eV) m/z: 204.13 [M^+].

2-Phenyl-2,3-dihydro-1*H*-naphtol[1,8-*de*][1,3,2]diazaborinine (3e)

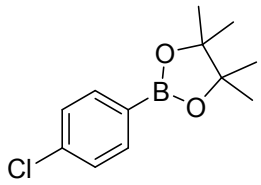


The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (2%). It was obtained as pink oil (52%). Data in agreement with the literature values.[16]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.04 (bs, 2H), 6.43 (d, *J* = 7.3 Hz, 2H), 7.08 (d, *J* = 8.3 Hz, 2H), 7.17 (dd, *J* = 1.2, 7.2, Hz, 2H), 7.44-7.50 (m, 3H), 7.66 (dd, *J* = 1.7, 7.7 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 106.2, 117.9, 119.9, 127.7, 130.4, 131.5, 136.4, 141.7.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 29.5.

MS (70 eV) m/z: 244.1 [M^+].

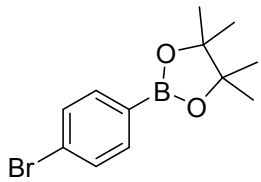
4,4,5,5-Tetramethyl-2-(4-chlorophenyl)-1,3,2-dioxaborolane (4a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was obtained as colorless oil (41%). Data in agreement with the literature values.[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.34 (s, 12H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 8.0 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 24.8, 83.9, 127.9, 136.1, 137.5.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 30.8
MS (70 eV) m/z: 238.1 [M^+].

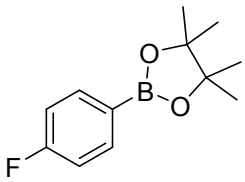
4,4,5,5-Tetramethyl-2-(4-bromophenyl)-1,3,2-dioxaborolane (5a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (2%). It was obtained as yellow oil (36.2%). Data in agreement with the literature values.[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.34 (s, 12H), 7.51 (d, *J* = 8.0 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 2H).
^{11}B NMR (128 MHz, $CDCl_3$) δ = 31.3.
MS (70 eV) m/z: 282.0 [M^+].

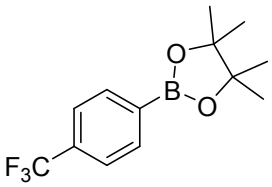
4,4,5,5-Tetramethyl-2-(4-fluorophenyl)-1,3,2-dioxaborolane (6a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (15:1). It was obtained as colorless oil (45%). Data in agreement with the literature values.[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.34 (s, 12H), 7.07-702 (m, 2H), 7.81-7.77 (m, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 24.8, 83.9, 114.8 (d, *J* = 20.2 Hz), 136.9 (d, *J* = 8.3 Hz), 165.1 (d, J = 150.8 Hz).
MS (70 eV) m/z: 222.1 [M^+].

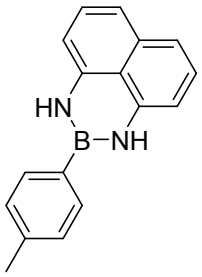
4,4,5,5-Tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane (7a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (15:1). It was obtained as white solid (56%). Data in agreement with the literature values.[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.36 (s, 12H), 7.61 (d, *J* = 8.0 Hz, 2H), 7.91 (d, *J* = 8.0 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 24.9, 84.2, 124.1 (q, *J* = 272.7 Hz), 124.3 (q, *J* = 3.8 Hz), 132.8 (q, *J* = 32.0 Hz), 135.0.
MS (70 eV) m/z: 272.1 [M^+].

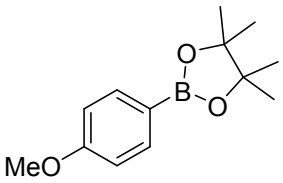
2-(*p*-tolyl)-2,3-dihydro-1*H*-naphtol[1,8-*de*][1,3,2]diazaborinine (19e):



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (2%). It was afforded as white solid. Data in agreement with the literature values.[27]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.30 (s, 3H), 6.01 (br, 2H), 6.31 (d, *J* = 7.8 Hz, 2H), 6.95 (d, *J* = 8.0 Hz, 2H), 7.04 (t, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.44 (d, *J* = 7.8 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.7, 106.2, 117.9, 119.9, 127.7, 130.2, 131.5, 136.4, 140.5, 141.3.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 29.7.

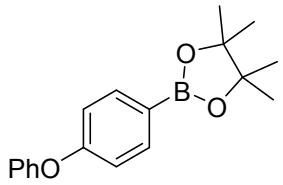
4,4,5,5-Tetramethyl-2-(4-methoxyphenyl)-1,3,2-dioxaborolane (8a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was obtained as colorless oil (76%). Data in agreement with the literature values.[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.34 (s, 12H), 3.83 (s, 3H), 6.90 (d, *J* = 8.8 Hz, 2H), 7.74 (d, *J* = 8.8 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 24.8, 55.1, 83.5, 113.3, 136.5, 162.1.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 31.3.
MS (70 eV) m/z: 234.2 [M^+].

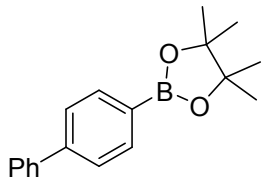
4,4,5,5-Tetramethyl-2-(4-phenoxyphenyl)-1,3,2-dioxaborolane (9a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was afforded as yellow oil (71%).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.34 (s, 12H), 6.95-7.00 (m, 2H), 7.00-7.06 (m, 2H), 7.13 (dd, *J* = 2.1, 8.5 Hz, 1H), 7.31-7.39 (m, 2H), 7.75-7.81 (m, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 24.8, 83.7, 117.6, 119.4, 121.4, 123.6, 129.8, 136.6, 156.5, 160.1.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 31.3.
HRMS (EI): calc. for $[C_{18}H_{22}BO_3]^+$ ($[M+H]^+$): 297.1584, Found: 297.1646.

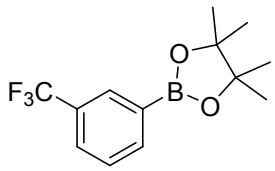
4,4,5,5-tetramethyl-2-(4-biphenylyl)-1,3,2-dioxaborolane (10a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was afforded as white solid (72%). Data in agreement with the literature values:[18]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.34 (s, 12H), 7.34-7.41 (m, 1H), 7.42-7.50 (m, 2H), 7.59-7.70 (m, 4H), 7.91 (d, *J* = 7.9 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 24.9, 83.8, 126.5, 127.3, 127.6, 128.8, 135.3, 141.0, 143.9.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 31.7.

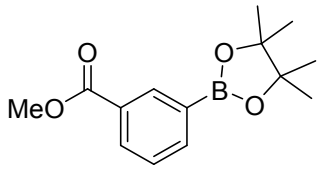
4,4,5,5-Tetramethyl-2-(2-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane (11a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was afforded as colorless oil (65%). Data in agreement with the literature values:[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.36 (s, 12H), 7.45-7.52 (m, 1H), 7.67-7.73 (m, 1H), 7.97 (d, *J* = 7.4 Hz, 1H), 8.06 (s, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 25.0, 84.4, 124.4 (q, *J* = 272.7 Hz), 127.9 (q, *J* = 3.8 Hz), 128.2, 130.2 (q, *J* = 31.5 Hz), 131.5 (q, *J* = 3.7 Hz), 138.1.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 30.8**.**
^{19}F NMR (376 MHz, $CDCl_3$) δ = -62.2.
MS (70 eV) m/z: 272.1 [M^+].

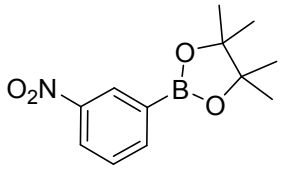
Methyl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (12a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (10:1). It was afforded as yellowish solid (41%). Data in agreement with the literature values:[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.35 (s, 12H), 3.92 (s, 3H), 7.45 (t, *J* = 7.6 Hz, 1H), 7.98 (dt, *J* = 1.3, 7.4 Hz, 1H), 8.10-8.15 (m, 1H), 8.47 (s, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 24.8, 52.0, 84.0, 127.8, 129.5, 132.8, 135.8, 139.1, 167.2.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 31.3.
MS (70 eV) m/z: 262.1 [M^+].

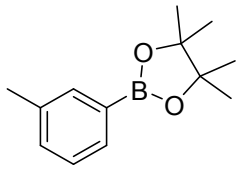
4,4,5,5-tetramethyl-2-(3-nitrophenyl)-1,3,2-dioxaborolane (13a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (50:1). It was afforded as yellow solid (31%). Data in agreement with the literature values:[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.36 (s, 12H), 7.54 (t, *J* = 7.8 Hz, 1H), 8.09 (d, *J* = 7.3Hz, 1H), 8.26-8.31 (m,1H), 8.64 (s, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 24.9, 84.6, 125.9, 128.8, 129.4, 140.7.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 30.4.
MS (70 eV) m/z: 249.1 [M^+].

4,4,5,5-Tetramethyl-2-(3-methyl)-1,3,2-dioxaborolane (14a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was afforded as colorless oil (40%). Data in agreement with the literature values:[13]

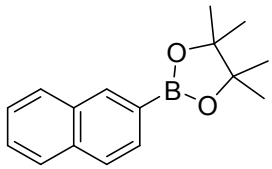
^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.34 (s, 12H), 2.35 (s, 3H), 7.26-7.29 (m, 2H), 7.60-7.63 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.2, 24.8, 83.7, 127.7, 131.7, 132.0, 135.3, 137.1.

^{11}B NMR (128 MHz, $CDCl_3$) δ = 31.0.

MS (70 eV) m/z: 218.3 [M^+].

4,4,5,5-Tetramethyl-2-(naphthalen-2-yl)-1,3,2-dioxaborolane (15a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was afforded as yellow solid (51%). Data in agreement with the literature values:[19]

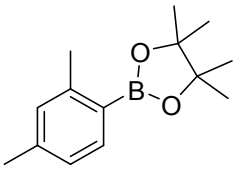
^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.40 (s, 12H), 7.43-7.58 (m, 3H), 7.80-7.92 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 24.9, 83.9, 125.8, 126.9, 127.7, 128.7, 130.3, 132.8, 134.9, 136.2.

^{11}B NMR (128.3 MHz, $CDCl_3$) δ = 31.3

MS (70 eV) m/z: 254.3 [M^+].

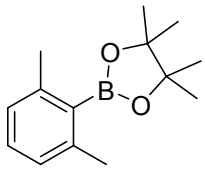
4,4,5,5-Tetramethyl-2-(2,4-dimethylphenyl)-1,3,2-dioxaborolane (16a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was afforded as colorless oil (57%). Data in agreement with the literature values:[13]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.34 (s, 12H), 2.33 (s, 3H), 2.52 (s, 3H), 7.00 (d, *J* = 8.0 Hz, 1H), 7.01 (s, 1H), 7.68 (d, *J* = 8.0 Hz, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.5, 22.1, 24.8, 83.2, 125.5, 130.7, 136.1, 140.8, 144.9.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 32.0
MS (70 eV) m/z: 232.2 [M^+].

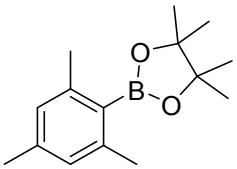
4,4,5,5-Tetramethyl-2-(2,6-dimethylphenyl)-1,3,2-dioxaborolane (17a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was afforded as colorless oil (51%). Data in agreement with the literature values:[13]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.39 (s, 12H), 2.39 (s, 6H), 6.92-6.97 (m, 2H), 7.12 (t, *J* = 7.5 Hz, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 22.2, 24.9, 83.7, 126.4, 129.2, 141.7.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 32.2.
MS (70 eV) m/z: 232.2 [M^+].

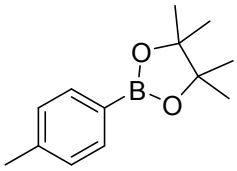
2-Mesityl-4,4,5,5-tetramethyl-2-phenyl-1,3,2-dioxaborolane (18a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was afforded as yellowish oil (34%). Data in agreement with the literature values:[13]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.37 (s, 12H), 2.24 (s, 3H), 2.37 (s, 6H), 6.78 (s, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.2, 22.2, 24.9, 83.4, 127.4, 138.9, 142.1.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 32.3.
MS (70 eV) m/z: 246.2 [M^+].

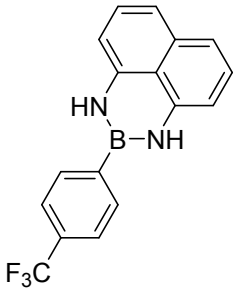
4,4,5,5-Tetramethyl-2-*p*-tolyl-1,3,2-dioxaborolane (19a)



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was afforded as white solid (22%). Data in agreement with the literature values:[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.34 (s, 12H), 2.36 (s, 3H), 7.19 (d, *J* = 7.9 Hz), 7.71 (d, *J* = 7.9 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.7, 24.9, 83.6, 128.5, 134.8, 141.4.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 31.0.

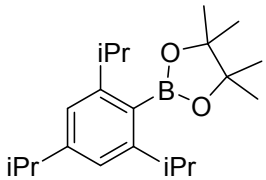
2-(4-(trifluoromethyl)phenyl)-2,3-dihydro-1*H*-naphtol[1,8-*de*][1,3,2]diaza-borinine (7e):



The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (2%). It was afforded as white solid. Data in agreement with the literature values:[27]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.05 (bs, 2H), 6.43 (dd, *J* = 0.9, 7.3 Hz, 2H), 7.08 (d, *J* = 7.8 Hz, 2H), 7.17 (dd, *J* = 1.2, 7.2, Hz, 2H), 7.66 (d, *J* = 7.7 Hz, 2H), 7.74 (d, *J* = 7.7 Hz, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 106.3, 118.3, 120.1, 124.2 (q, *J* = 272 Hz), 125.1 (q, *J* = 3.8 Hz), 127.7, 131.9, 132.2 (q, *J* = 31 Hz), 136.4, 140.6.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 29.9.

4,4,5,5-Tetramethyl-2-(2,4,6-triisopropylphenyl)-1,3,2-dioxaborolane (24a):

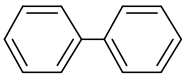


The product was purified by flash column chromatography using as eluent a mixture of petroleum ether/EtOAc (20:1). It was afforded as white solid. Data in agreement with the literature values.[28]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.22-1.31 (m, 18H), 1.37 (s, 12H), 2.92 (hp, *J* = 6.9 Hz, 1H), 3.01 (hp, *J* = 6.9 Hz, 2H), 7.05 (s, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.2, 22.2, 24.9, 83.4, 127.4, 138.9, 142.1.
^{11}B NMR (128 MHz, $CDCl_3$) δ = 32.1; **MS (70 eV) m/z:** 33.30 [M^+].

9. CHARACTERIZATION OF THE CROSS COUPLING PRODUCTS.

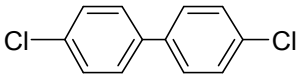
Biphenyl (25)



The product was purified by flash column chromatography using hexane as eluent. It was obtained as a white solid (87%). Data in agreement with the literature values.[20]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.47-7.51 (m, 6H), 7.64-7.66 (m, 4H).
MS (70 eV) m/z: 154.1 [M^+].

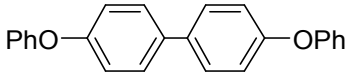
4,4'-Dichlorobiphenyl (26)



The product was purified by flash column chromatography using hexane as eluent. It was obtained as a white solid (72%). Data in agreement with the literature values.[20]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39 (d, *J* = 8.5 Hz, 4H), 7.46 (d, *J* = 8.6 Hz, 4H).
MS (70 eV) m/z: 222.1 [M^+].

4,4'-Diphenoxybiphenyl (27)



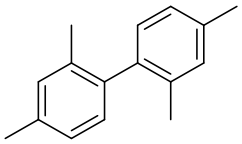
The product was purified by flash column chromatography using hexane as eluent. It was obtained as a white solid (42%). Data in agreement with the literature values.[21]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.07-7.10 (m, 8H), 7.11-7.17 (m, 2H), 7.33-7.41 (m, 4H), 7.51-7.57 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 118.8, 118.9, 123.2, 128.0, 129.6, 135.4, 156.4, 156.9.

MS (70 eV) m/z: 338.3 [M^+].

2,2',4,4'-Tetramethylbiphenyl (28)



The product was purified by flash column chromatography using hexane as eluent. It was obtained as colorless oil (65%). Data in agreement with the literature values.[22]

^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.06 (s, 6H), 2.39 (s, 6H), 7.00-7.07 (m, 4H), 7.11 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ = 19.8, 21.1, 126.2, 129.4, 130.5, 135.8, 136.5, 138.6.

MS (70 eV) m/z: 210.2 [M^+].

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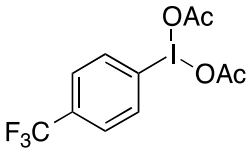
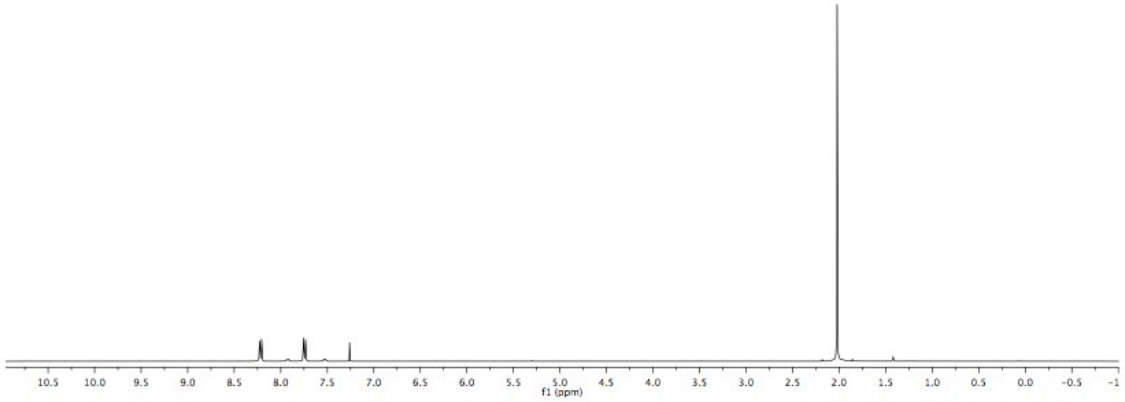
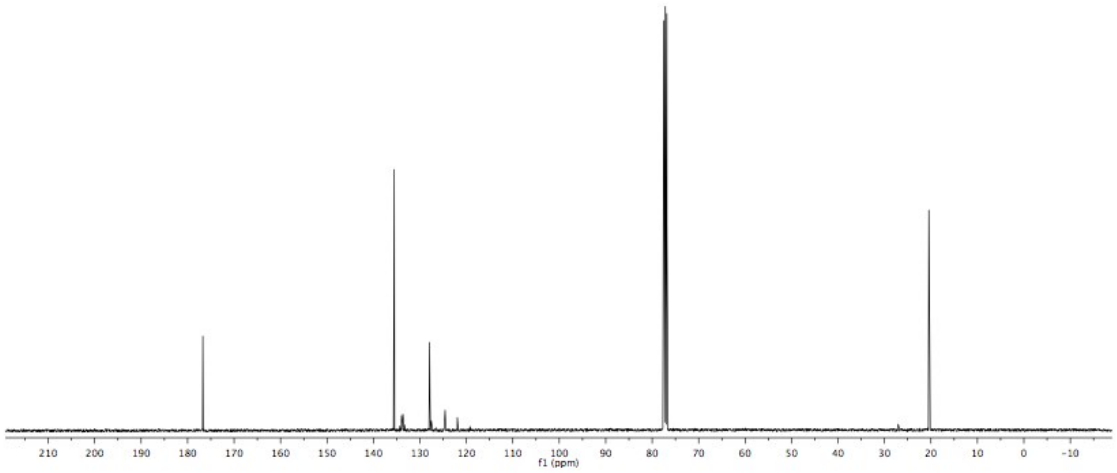
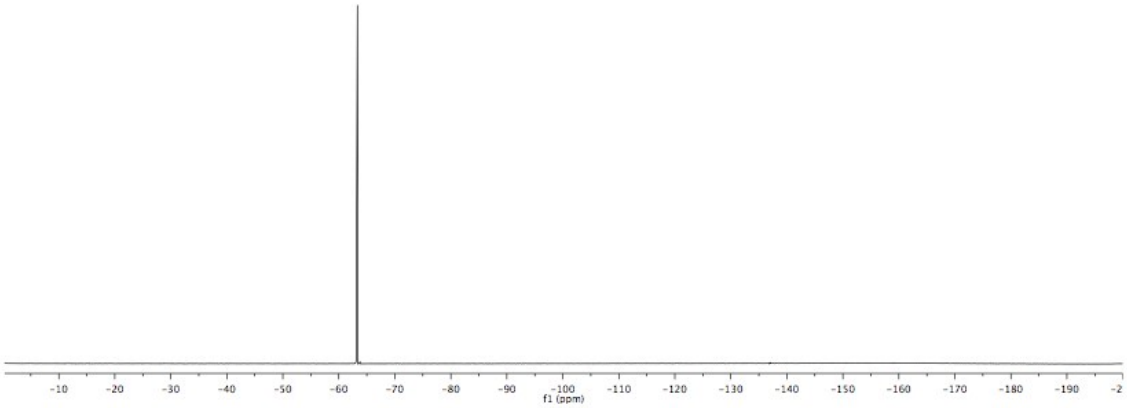
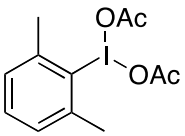
11. SPECTRAL CHARACTERIZATION OF I(III) COMPOUNDS







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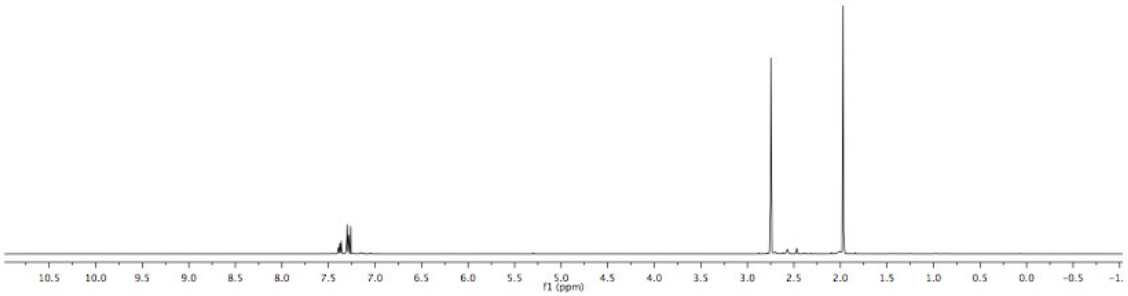
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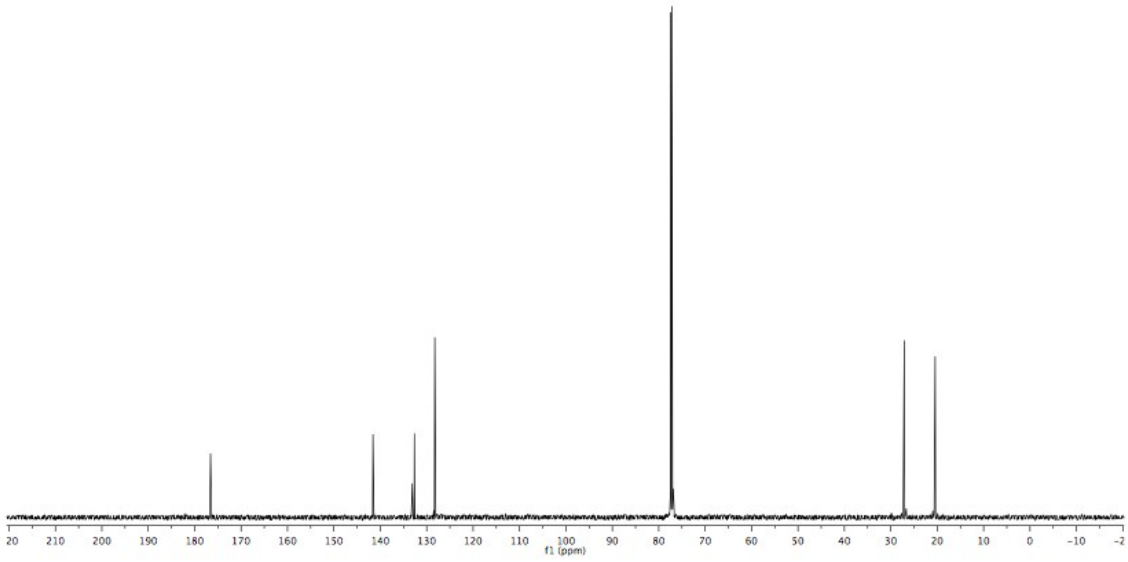
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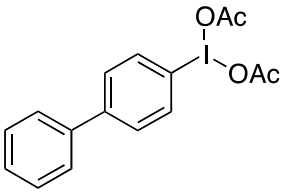
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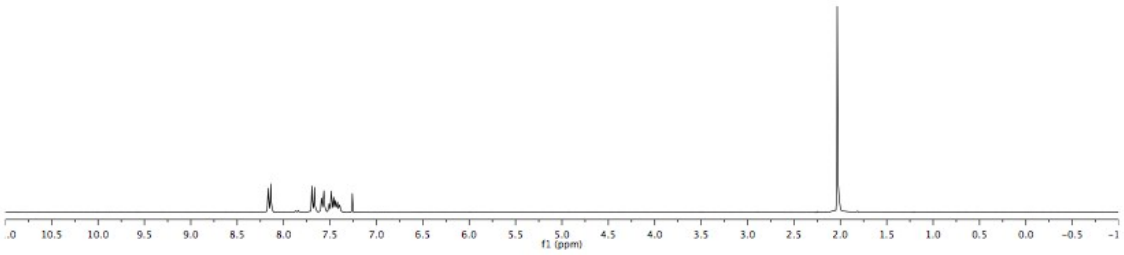
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5.0
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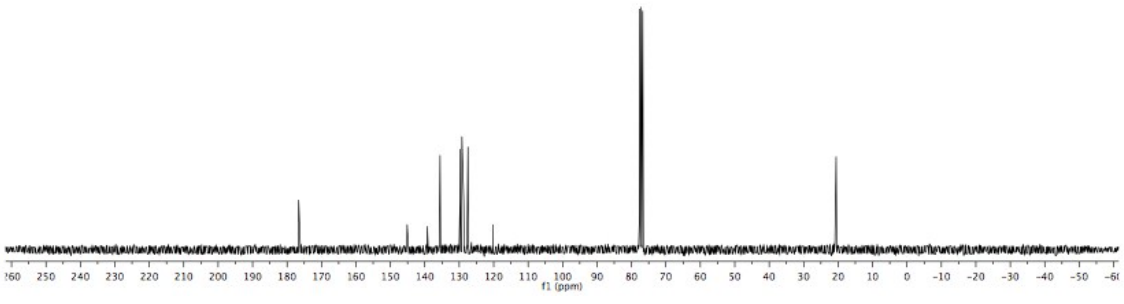
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5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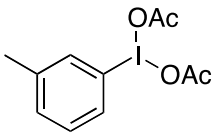
−50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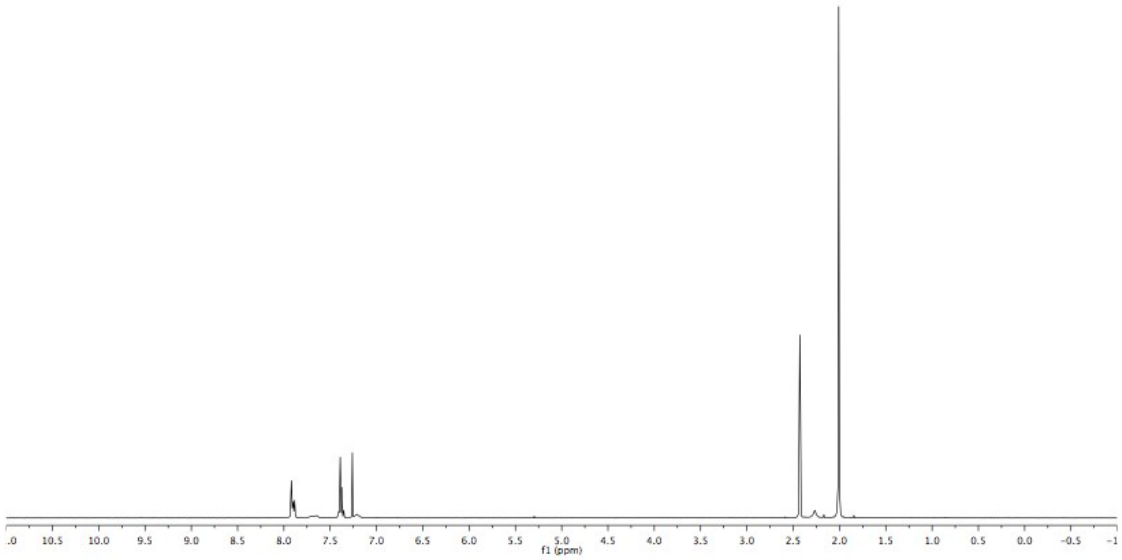
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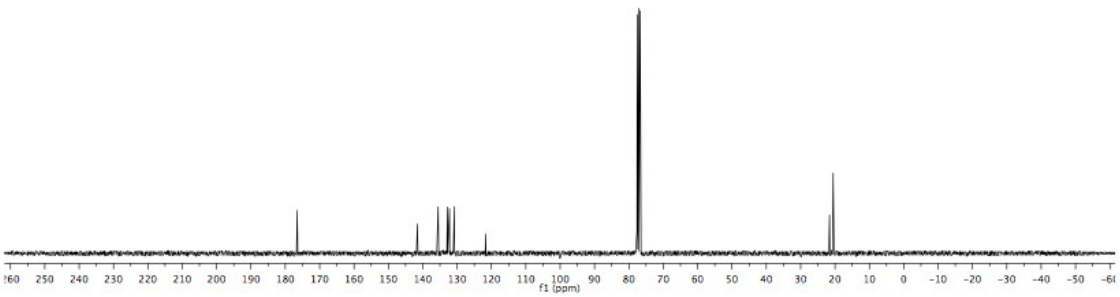
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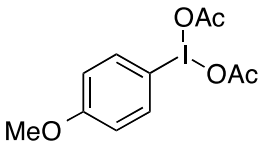
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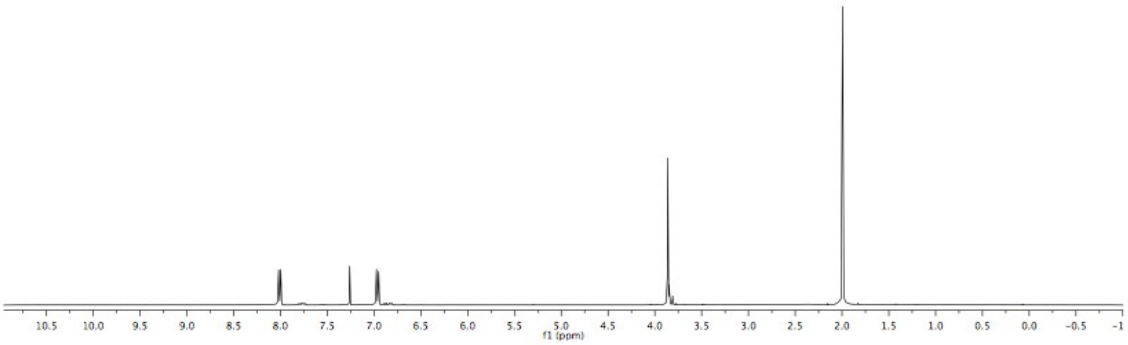
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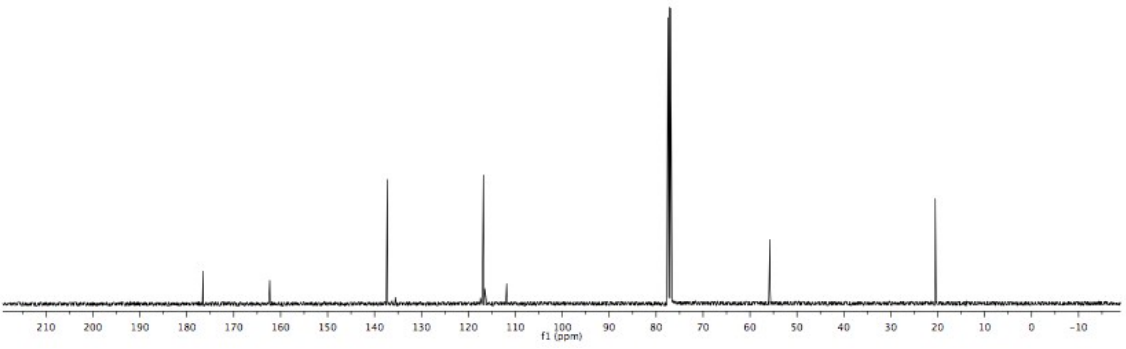
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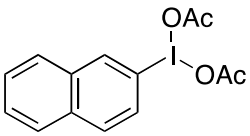
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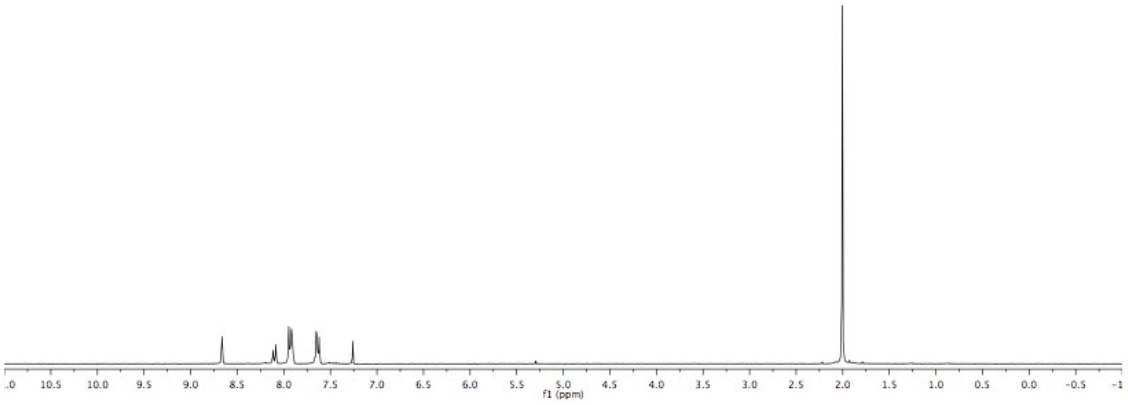
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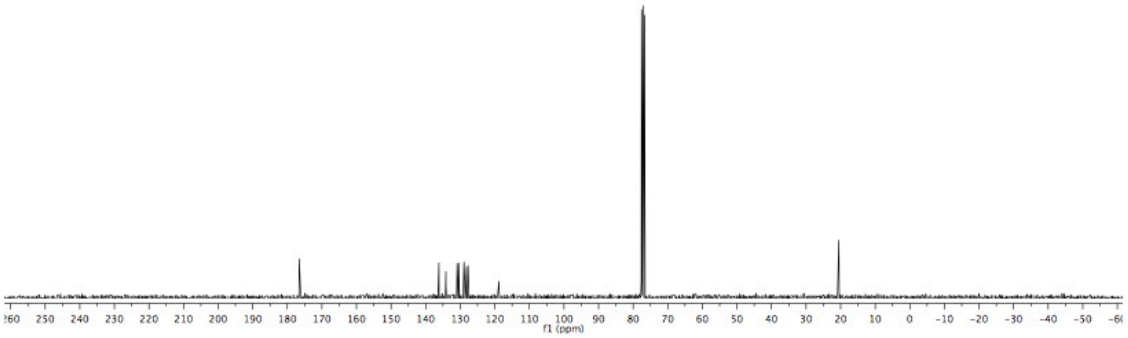
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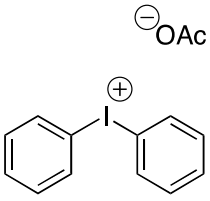
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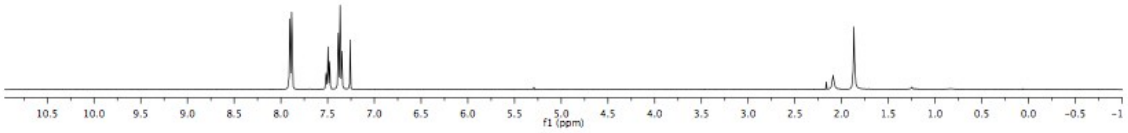
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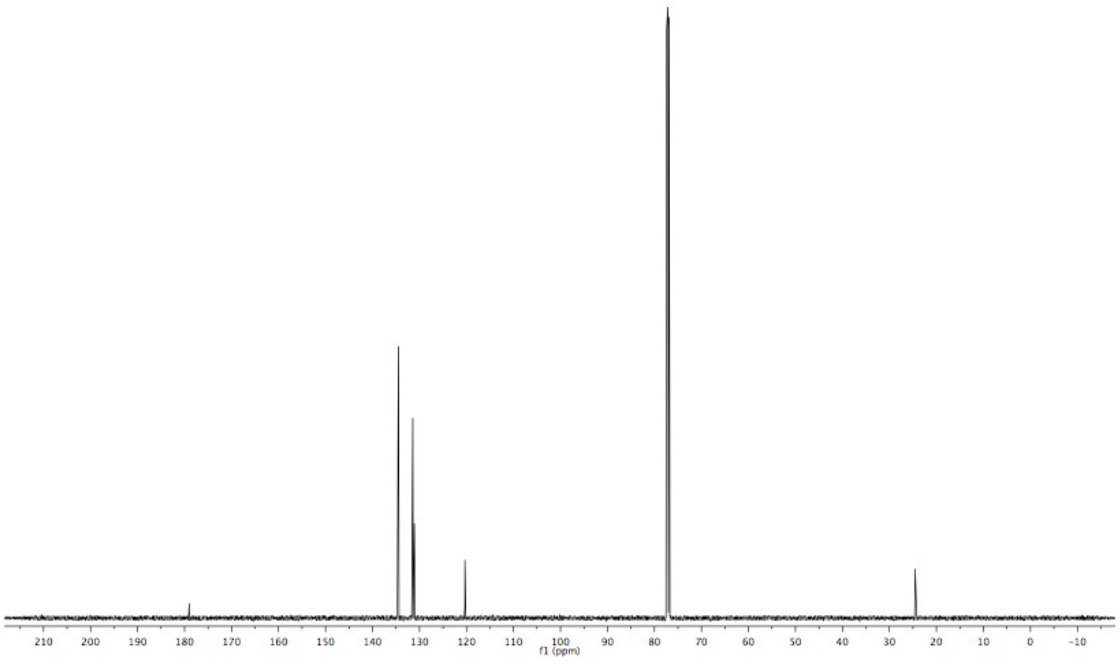
f1 (ppm)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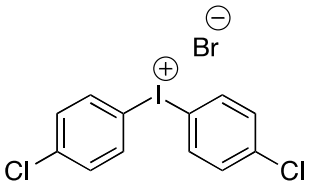
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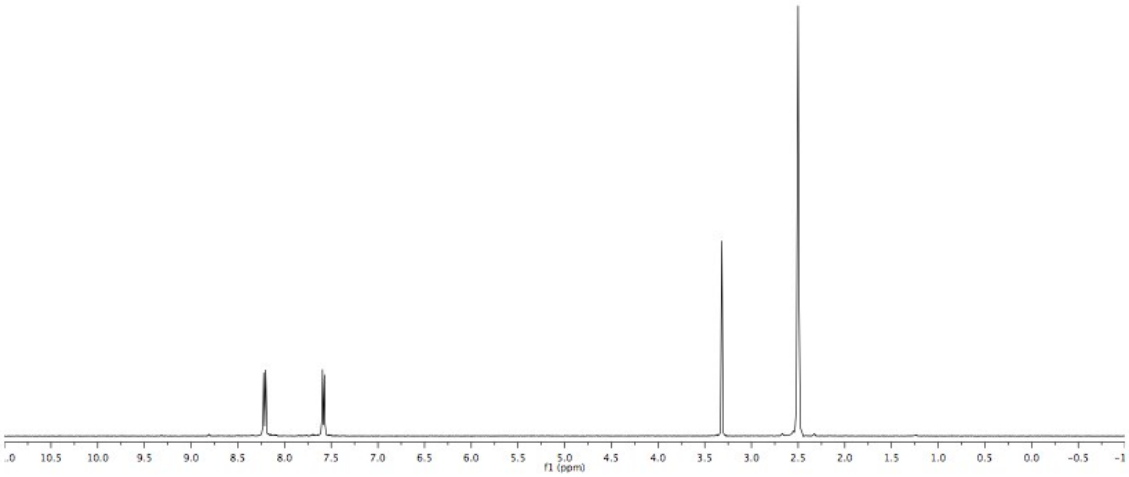
Cl
Cl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5.0
f1 (ppm)
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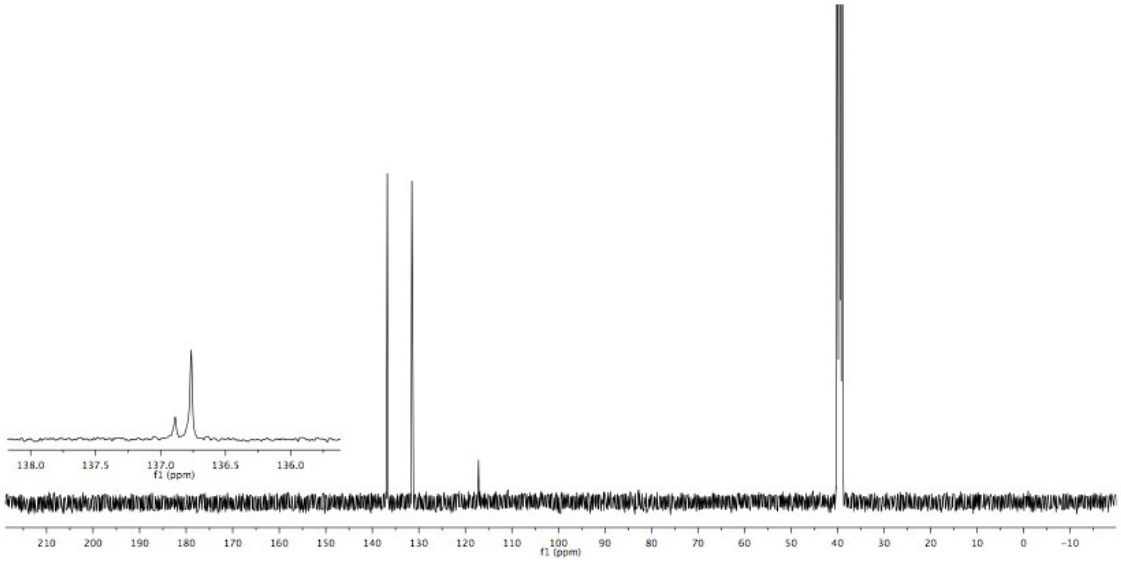
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5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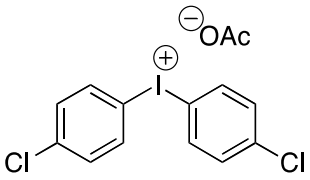
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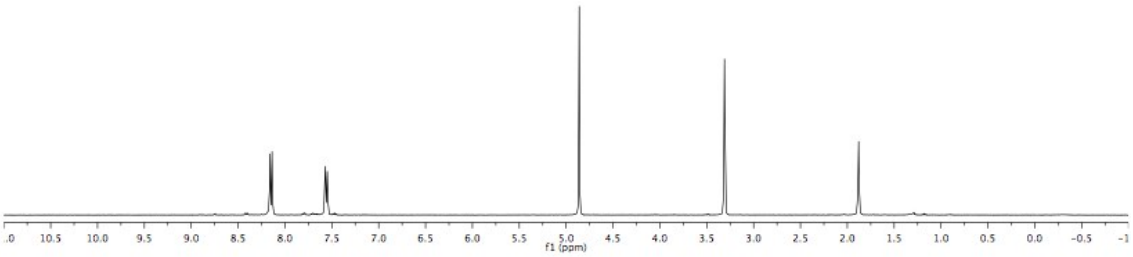
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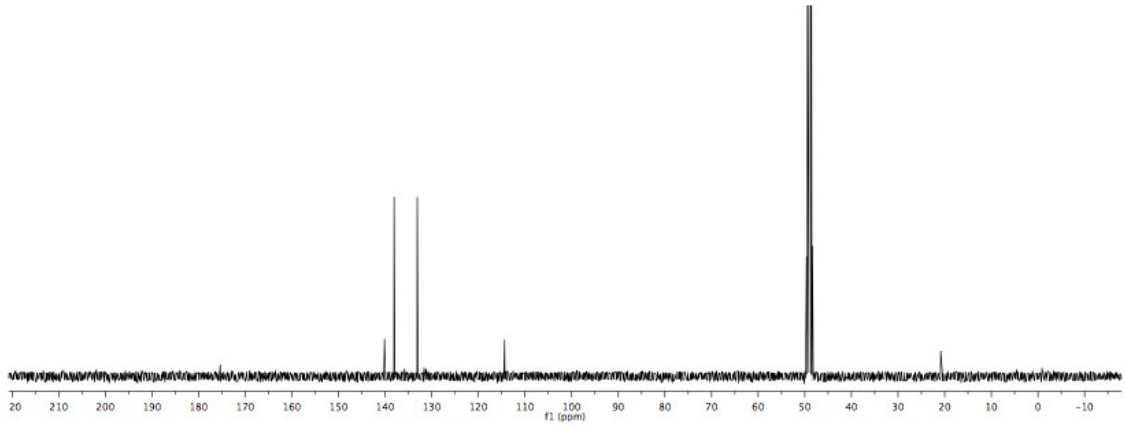
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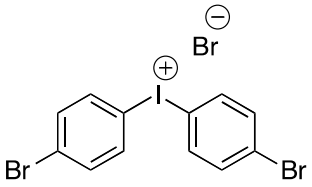
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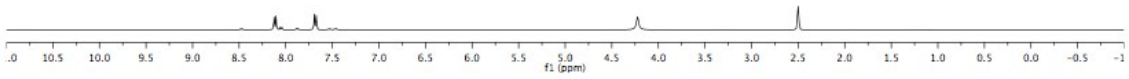
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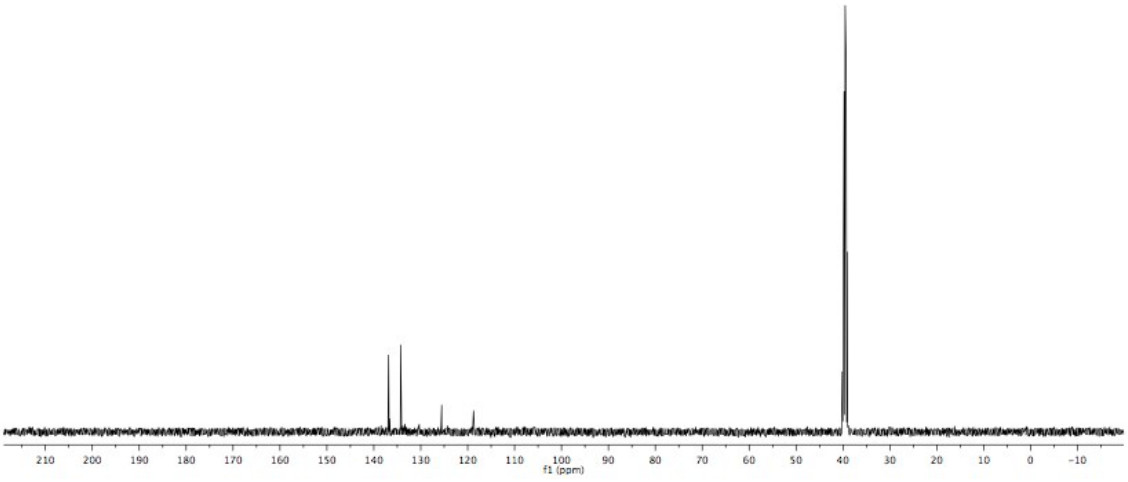
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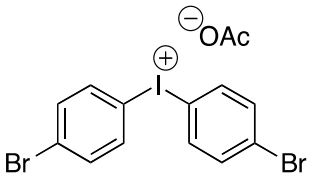
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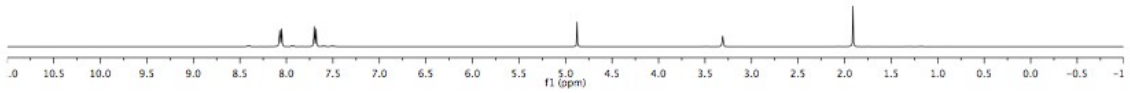
I
Br
Br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0.5
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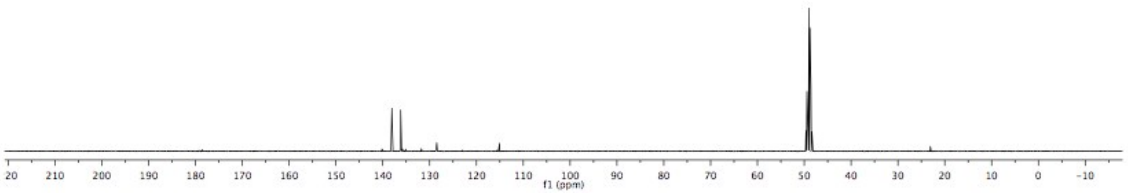
−0.5
f1 (ppm)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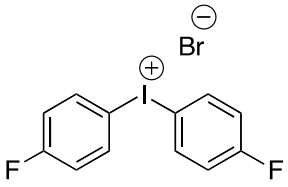
f1 (ppm)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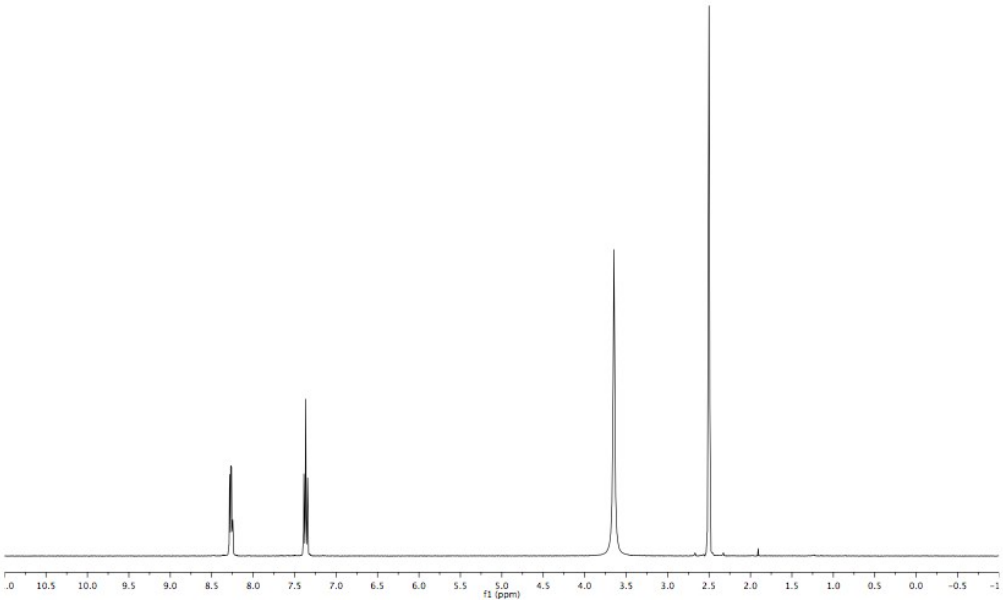
I
F
F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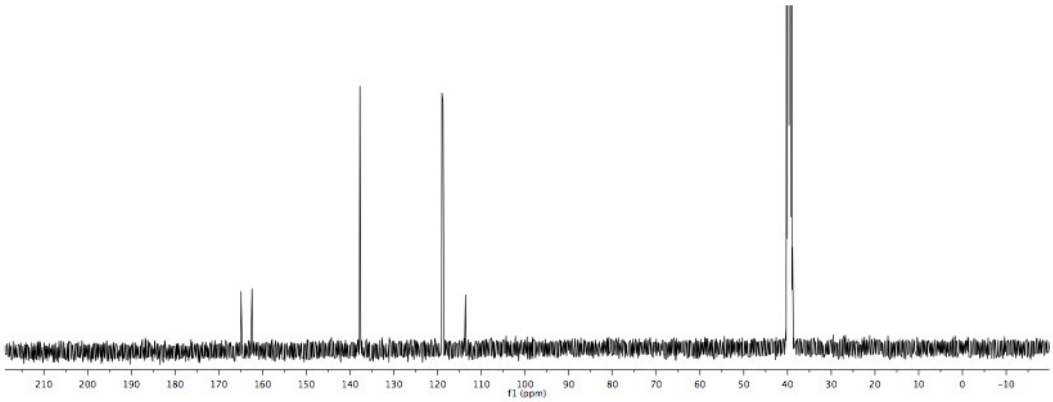
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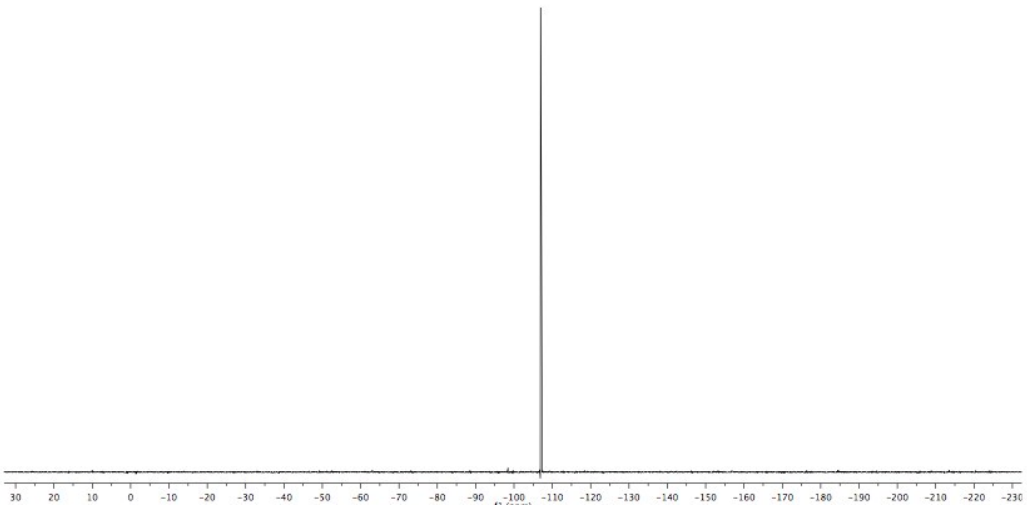
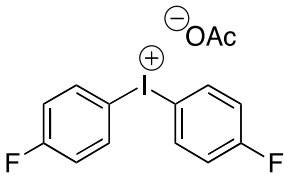
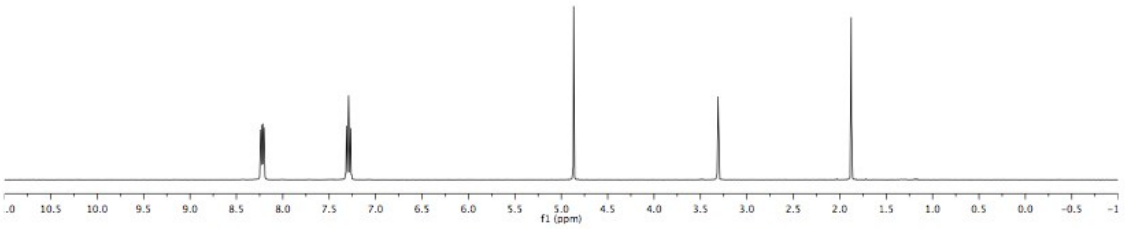



OAc
I
F
F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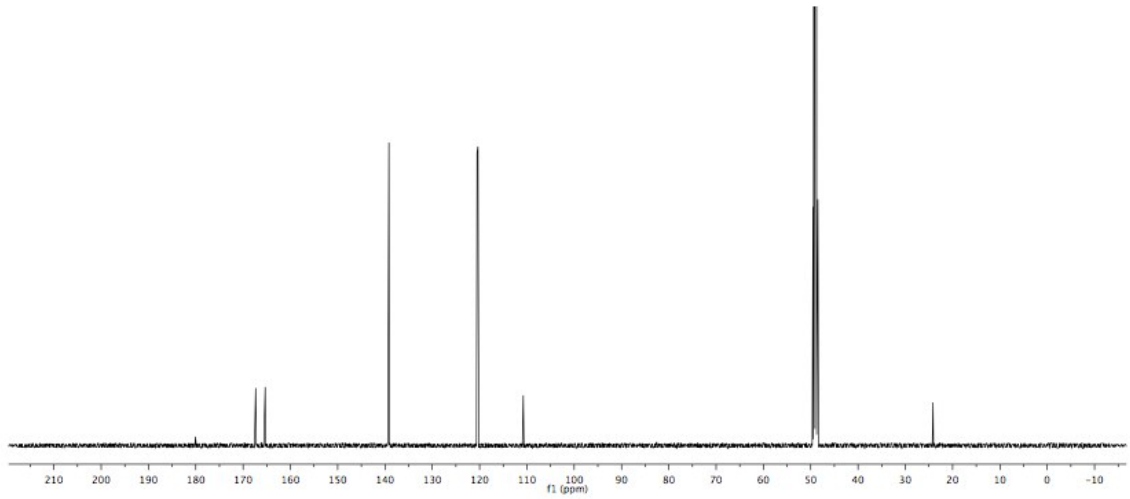
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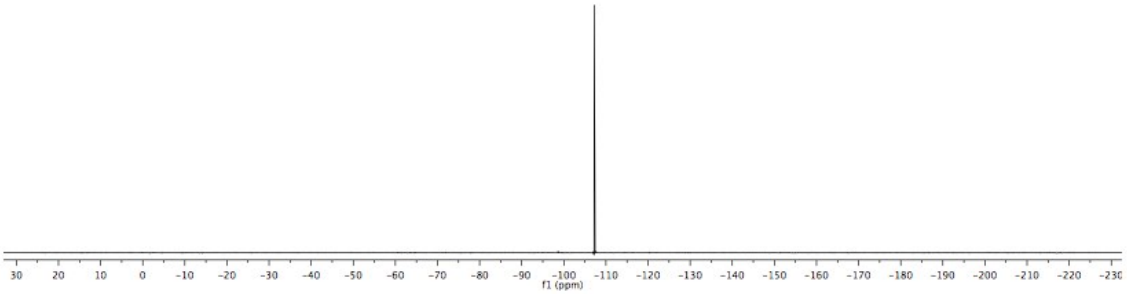
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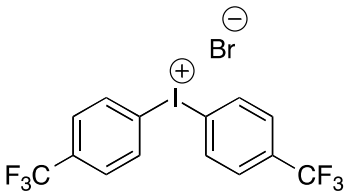
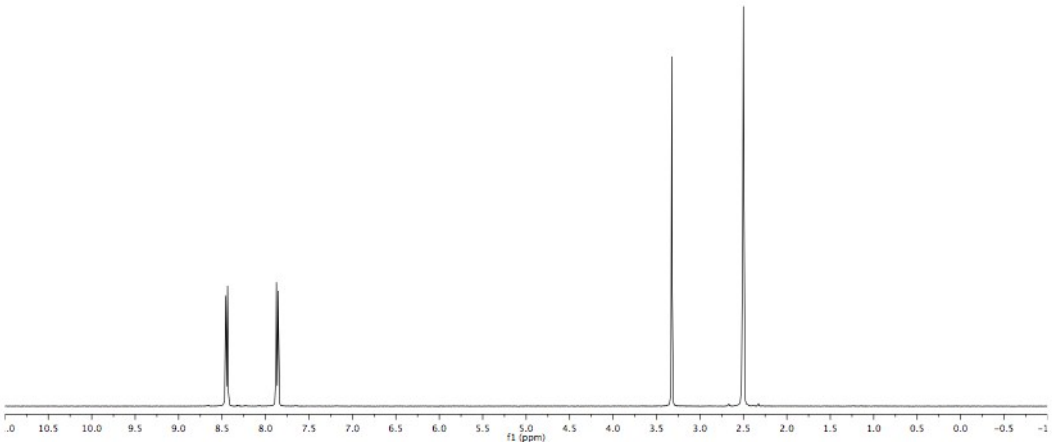

Br
I
F3C
CF3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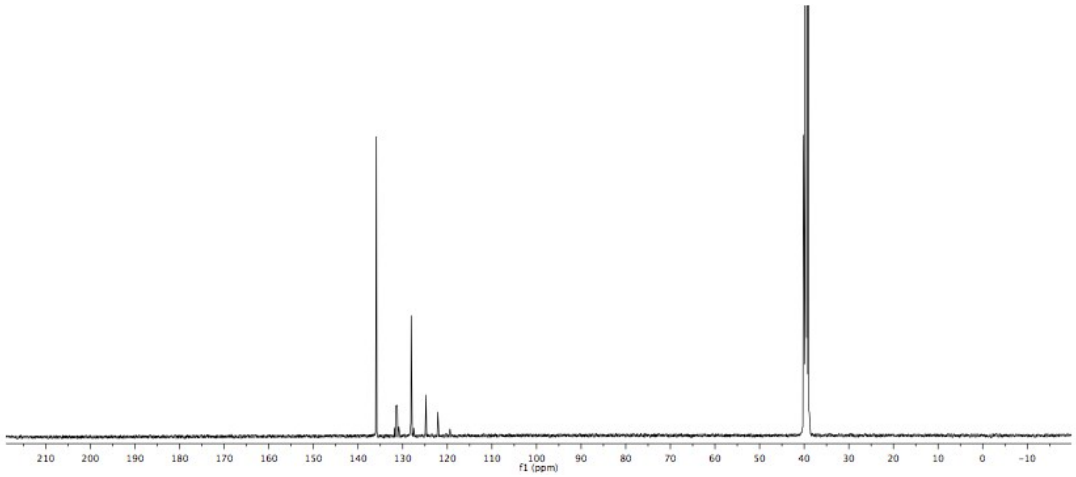
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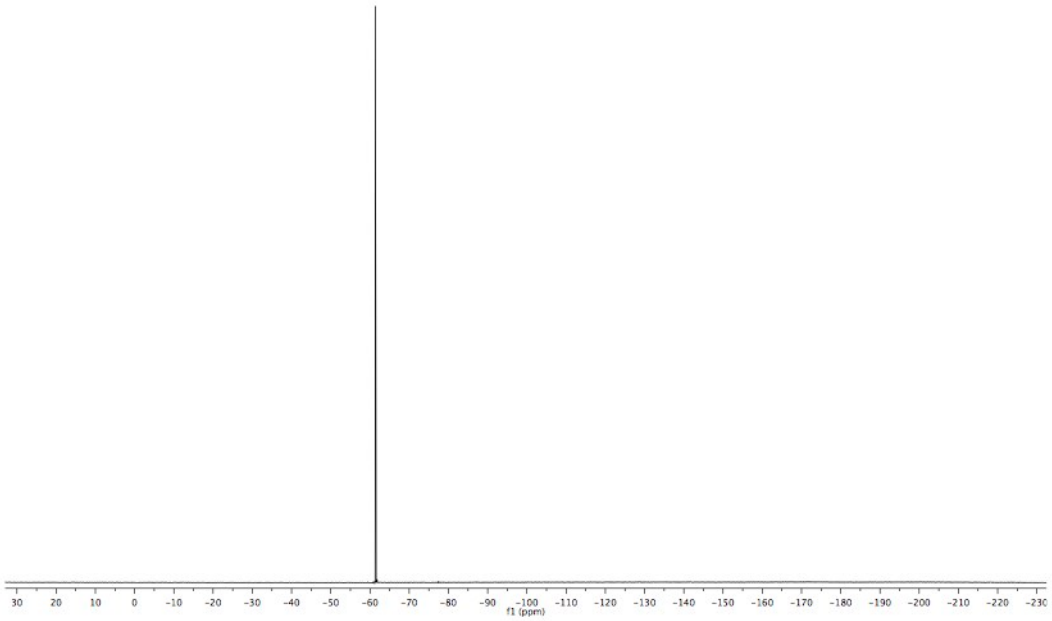
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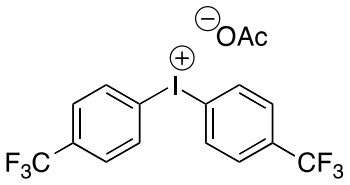
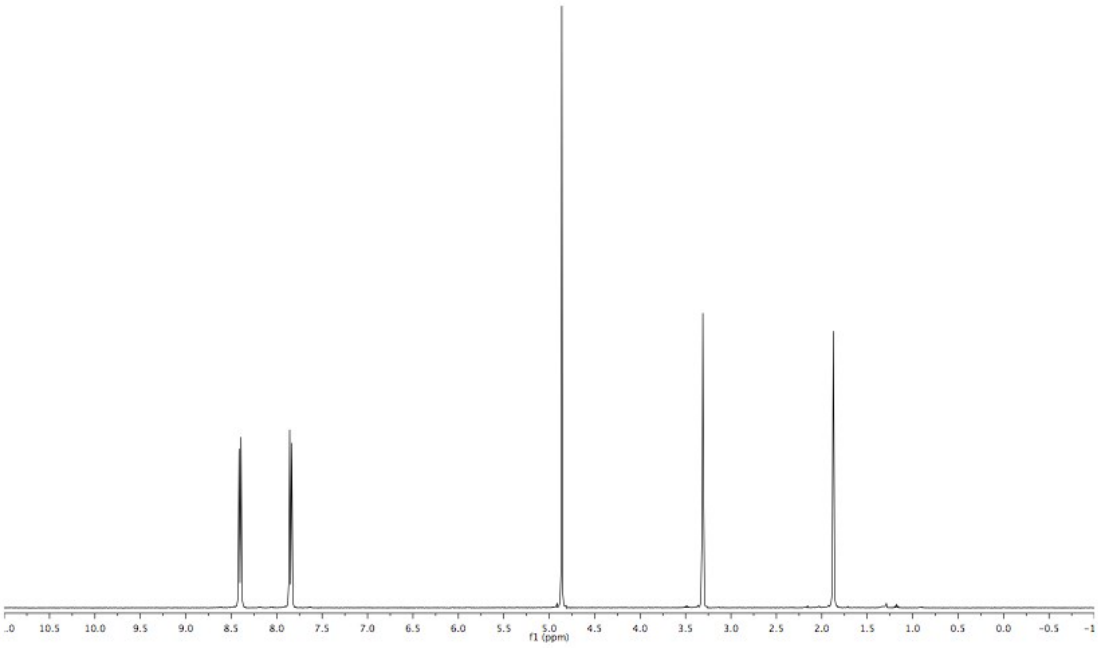

OAc
I
F3C
CF3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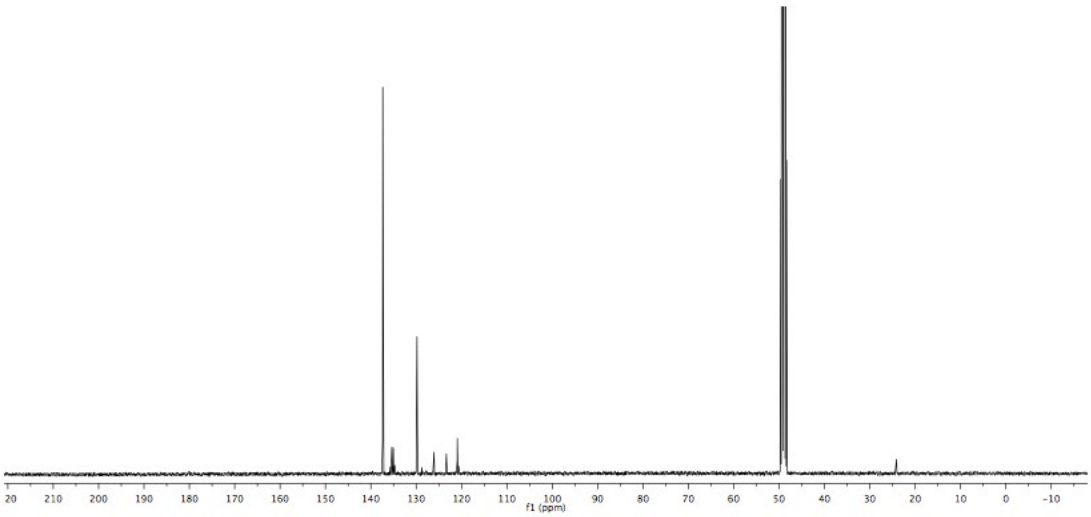
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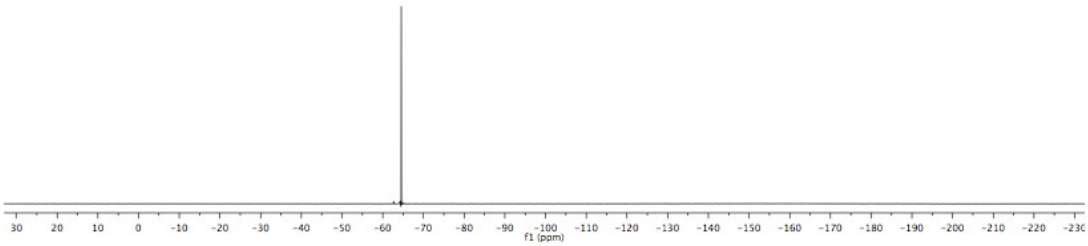
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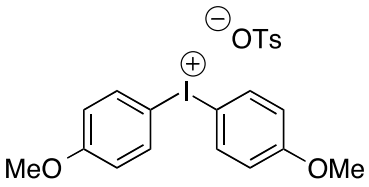
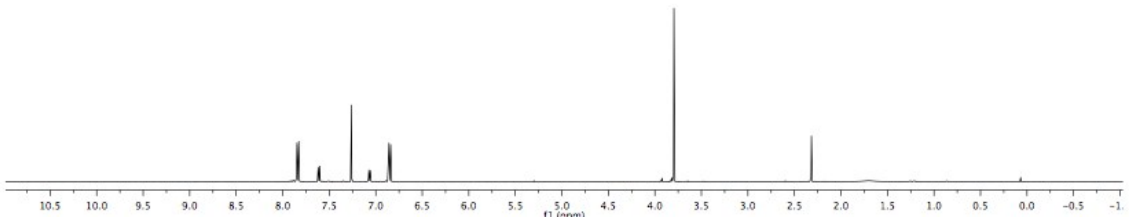

OTs
MeO
OMe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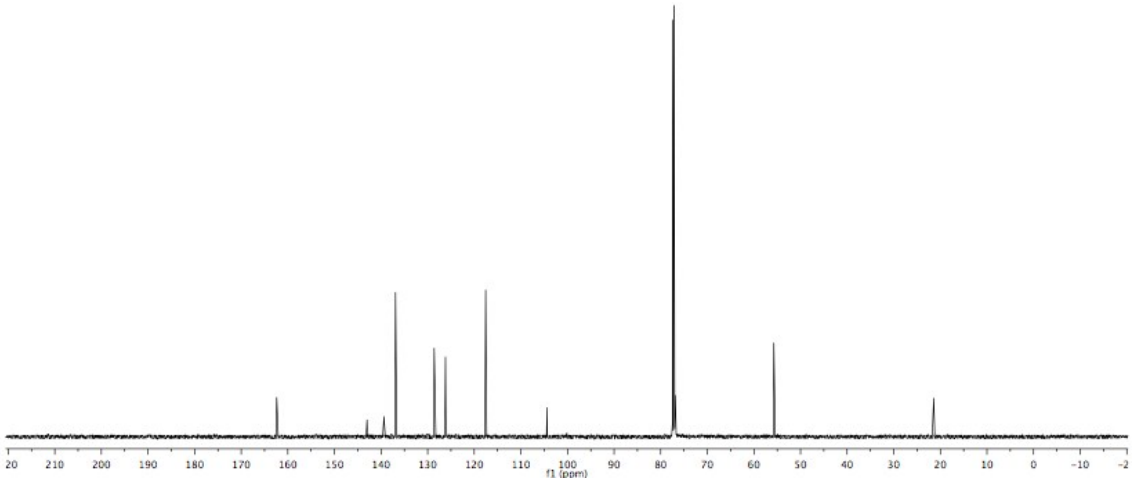
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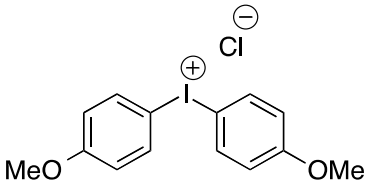
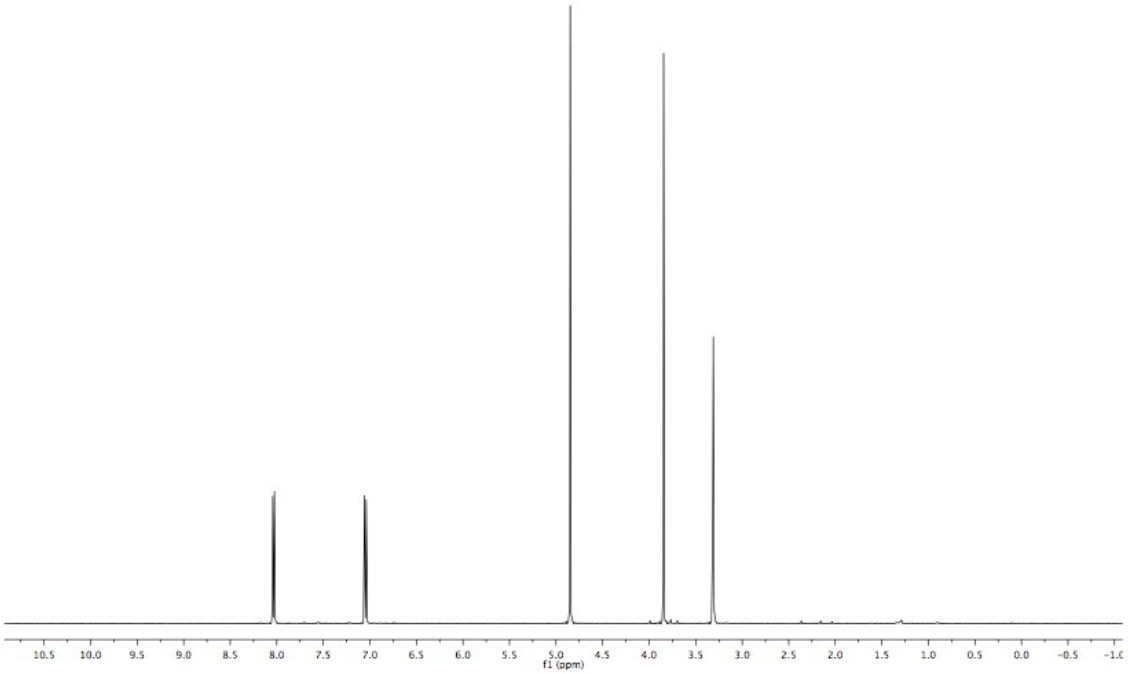

Cl
I
MeO
OMe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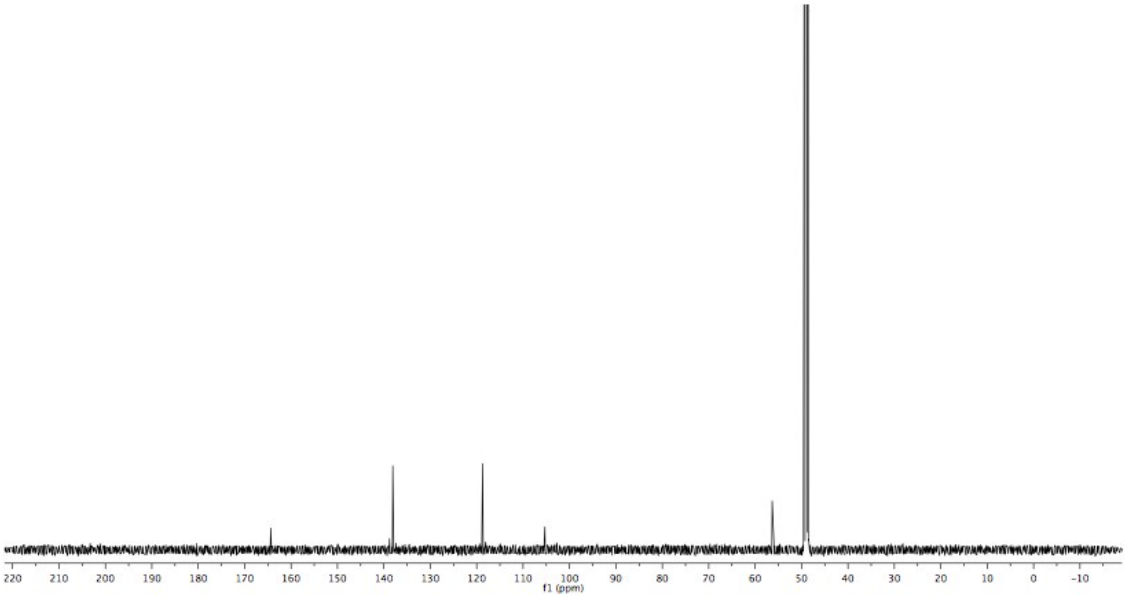
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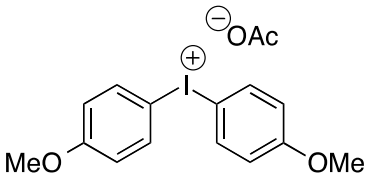
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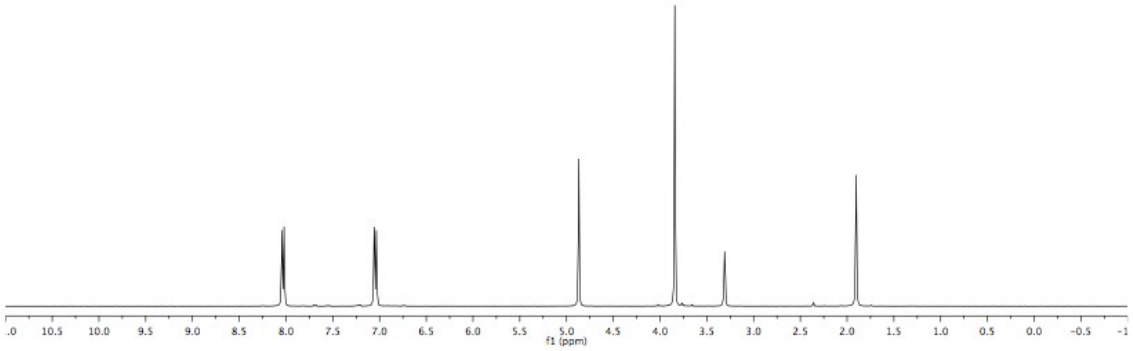
I
MeO
OMe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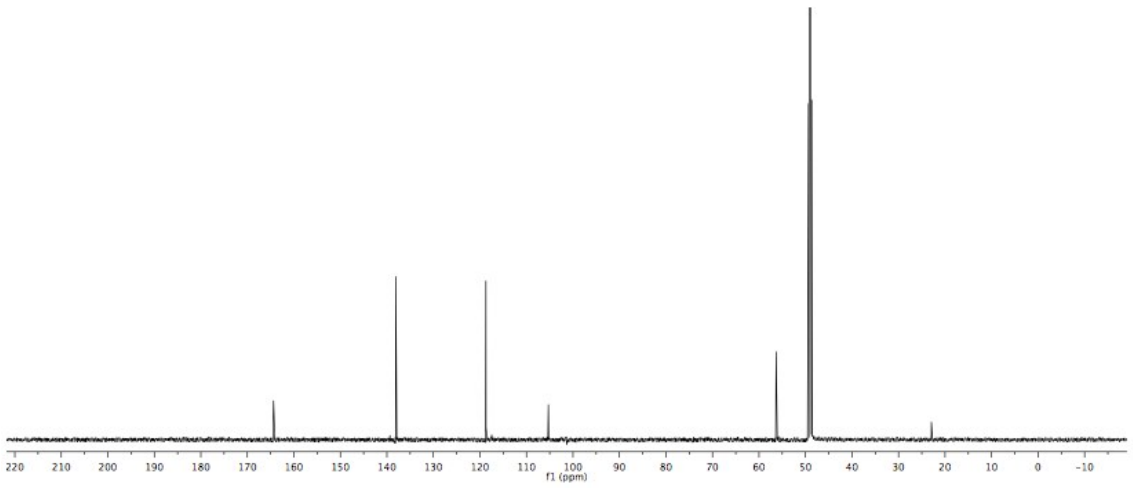
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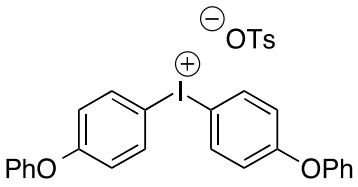
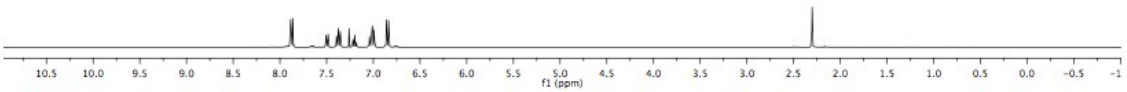

OTs
PhO
OPh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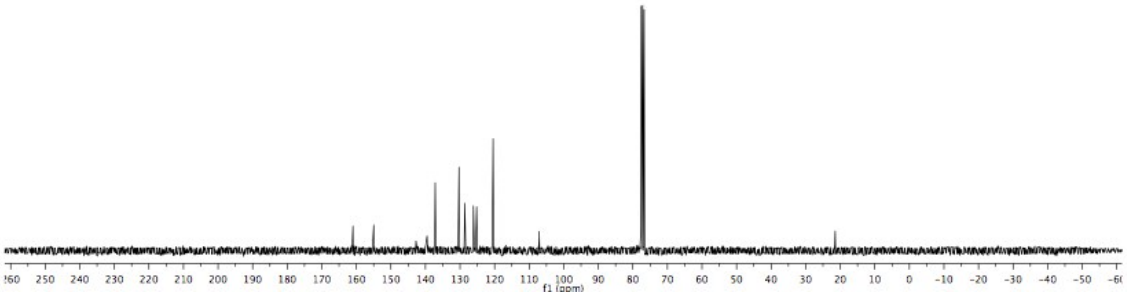
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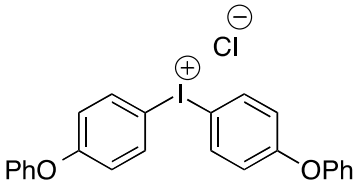
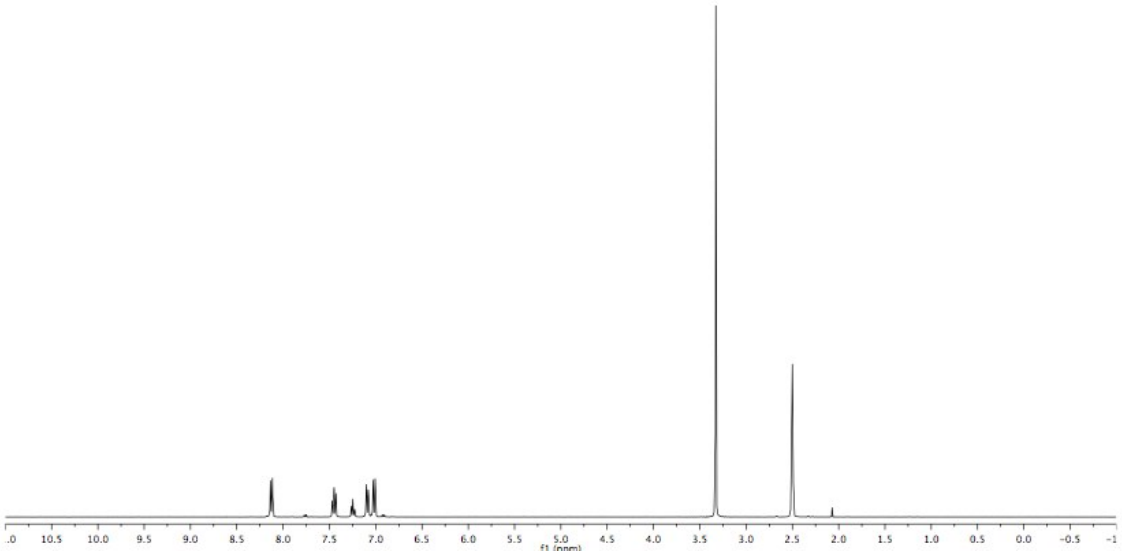

Cl
I
PhO
OPh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8.0
7.5
7.0
6.5
6.0
5.5
5.0
f1 (ppm)
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4.0
3.5
3.0
2.5
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1.5
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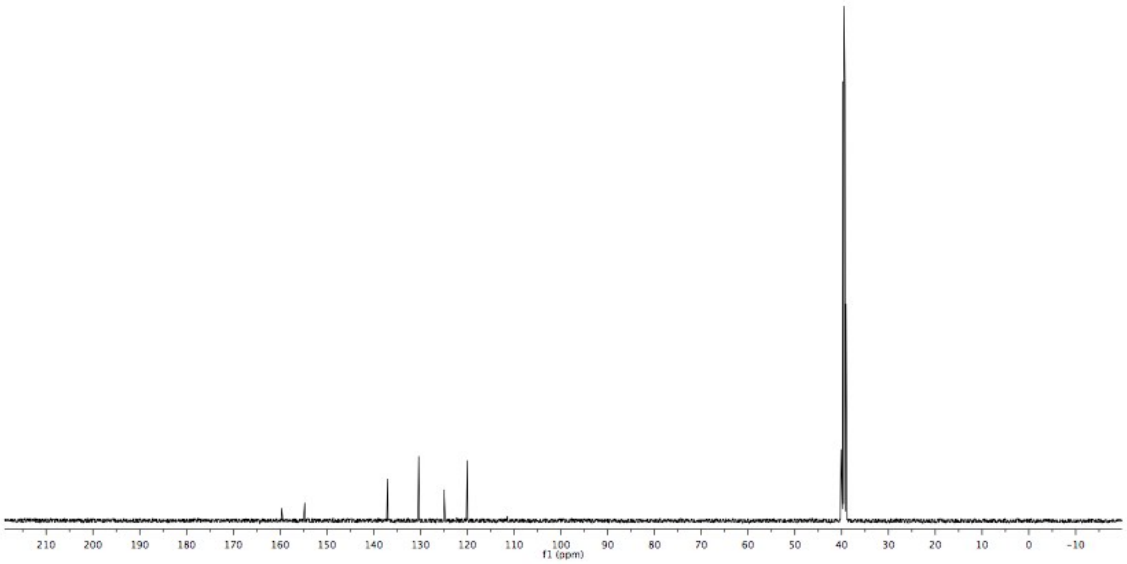
0.5
0.0
-0.5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f1 (ppm)
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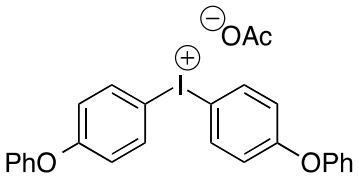
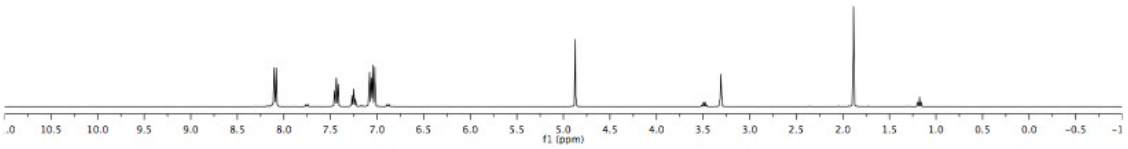
0
-10


OAc
PhO
OPh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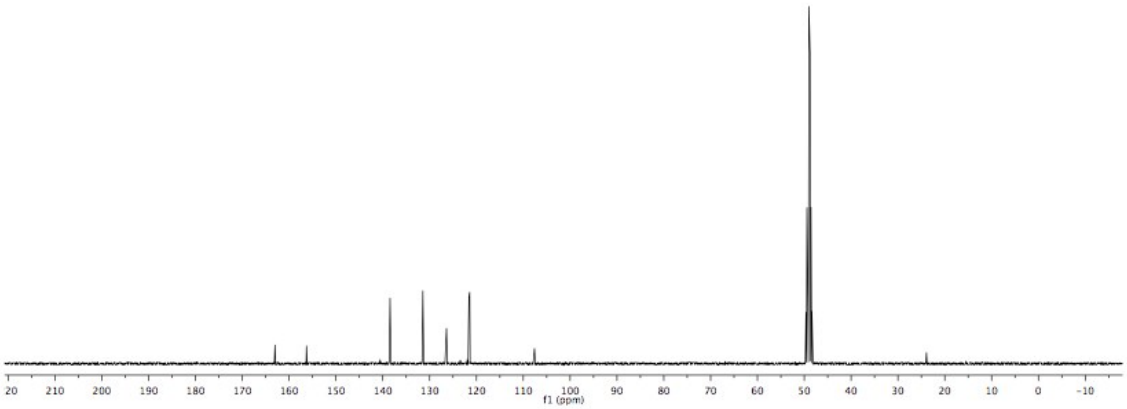
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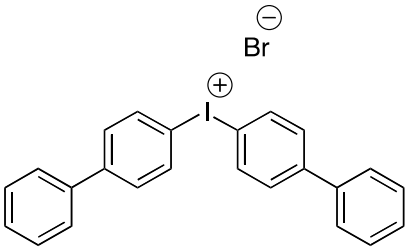
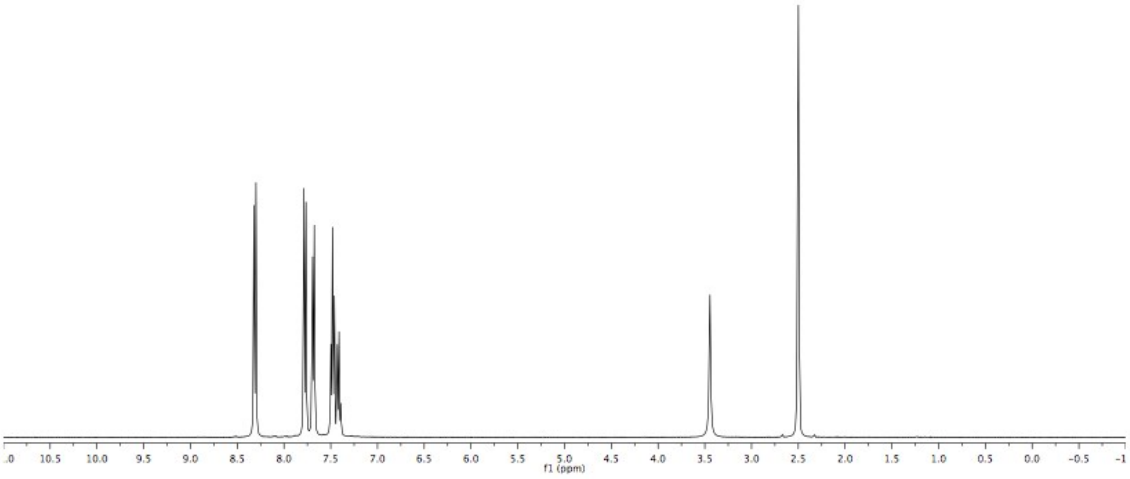

Br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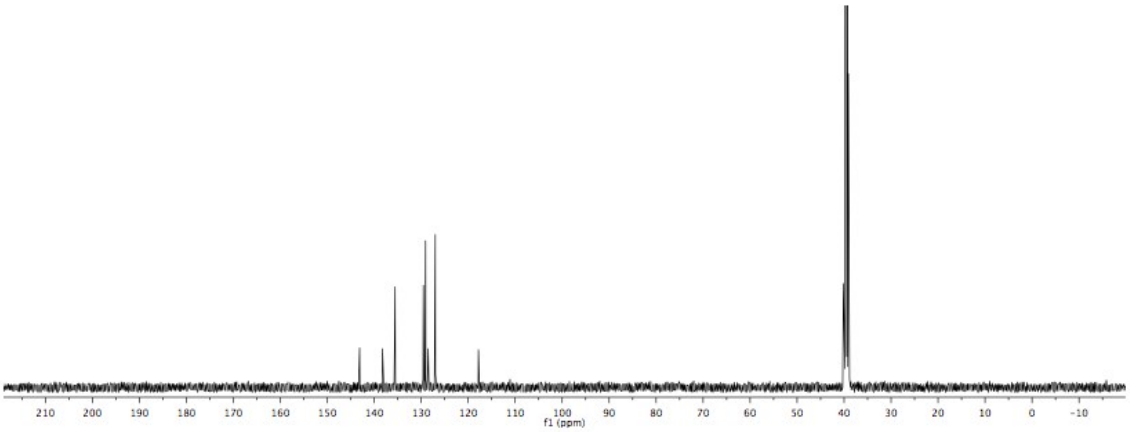
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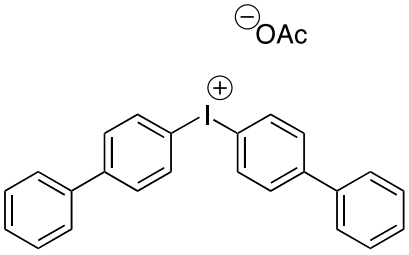
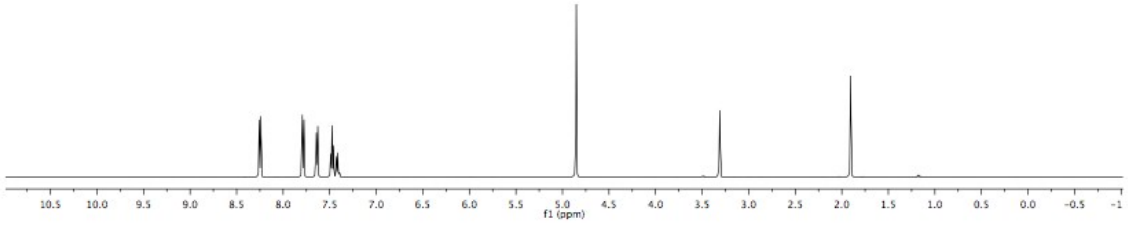

OAc


10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
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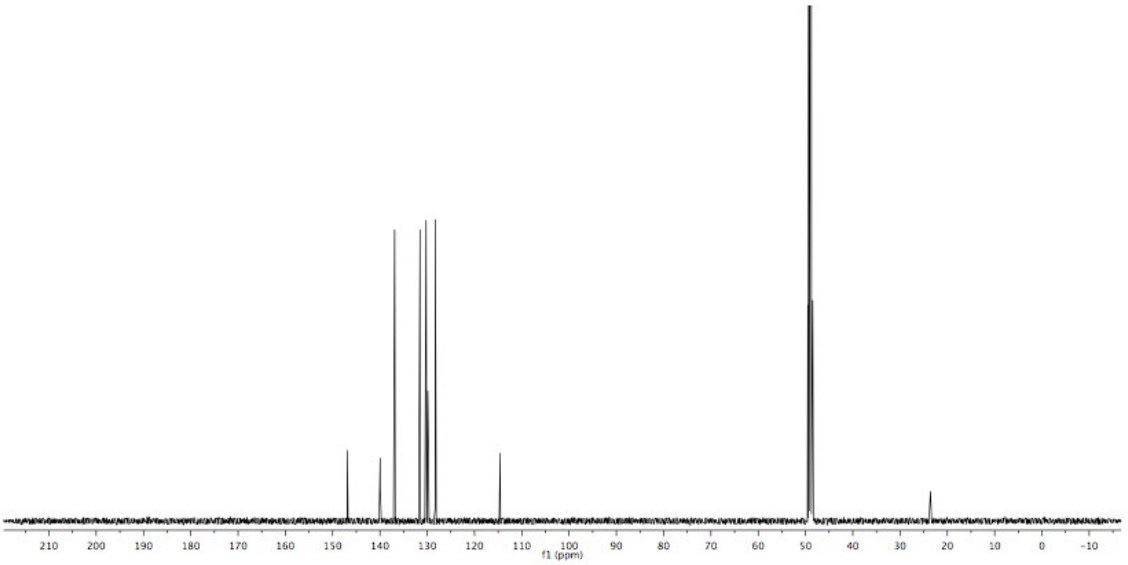
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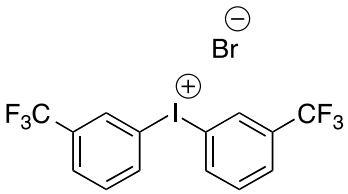
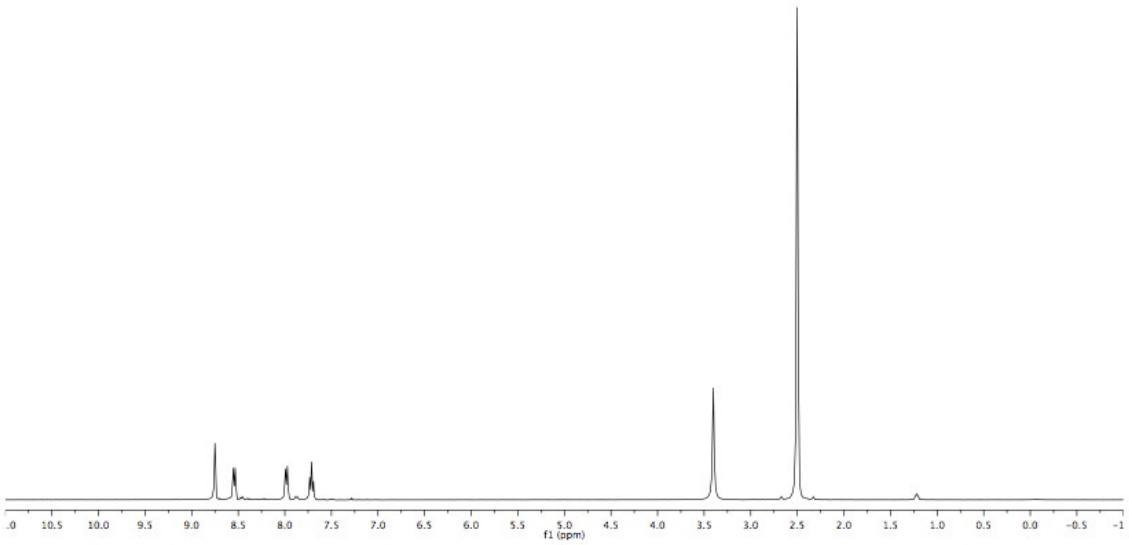
f1 (ppm)


Br
F3C
I
CF3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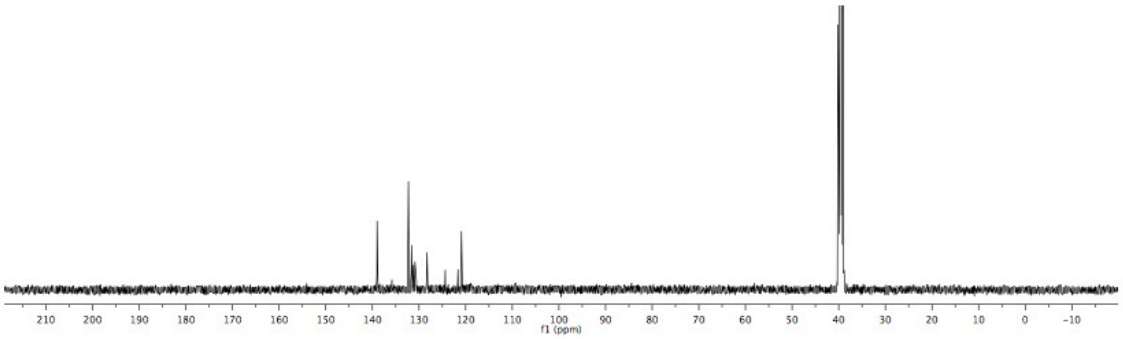
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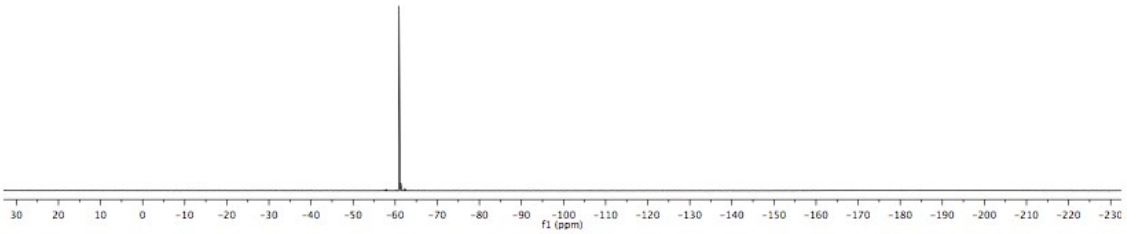
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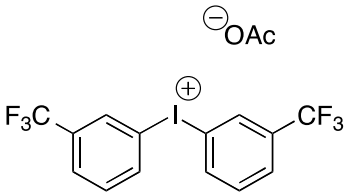
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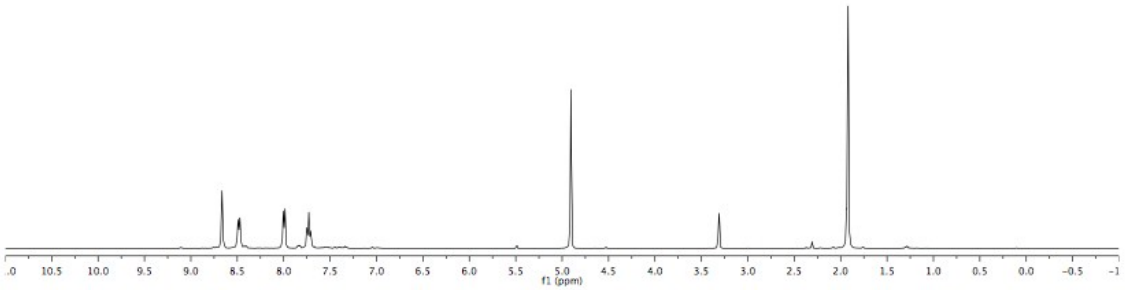
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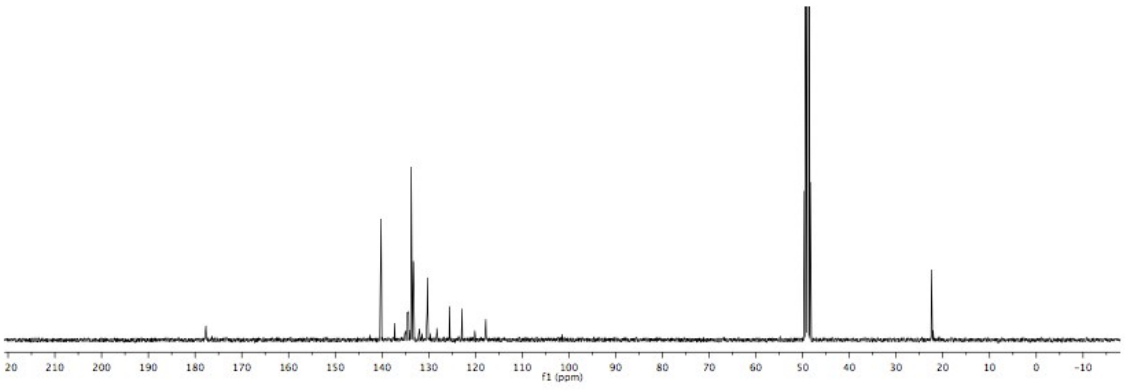
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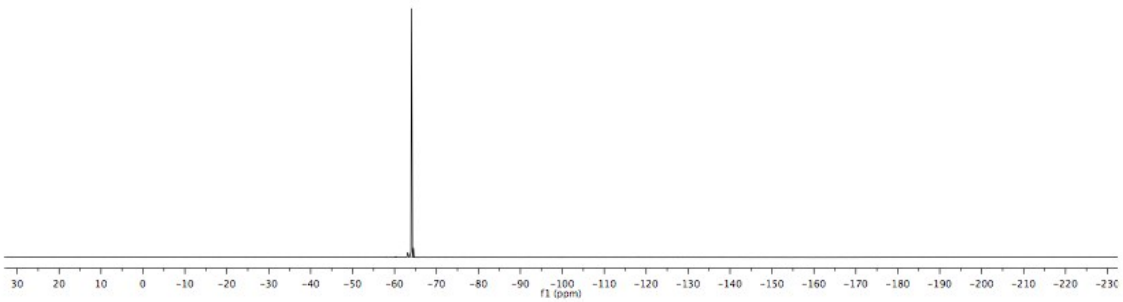
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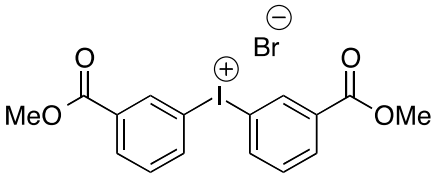
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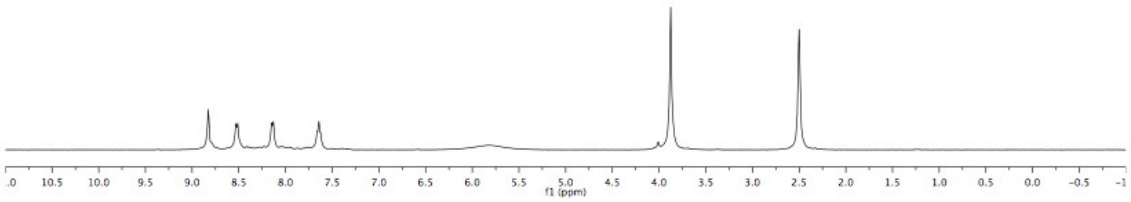
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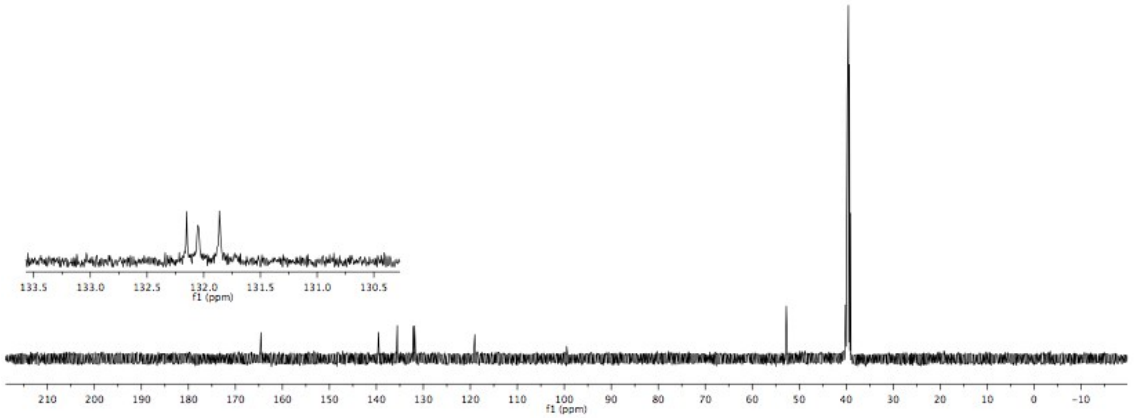
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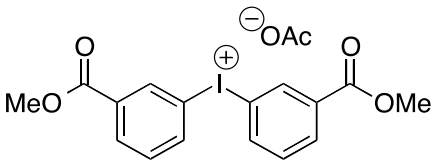
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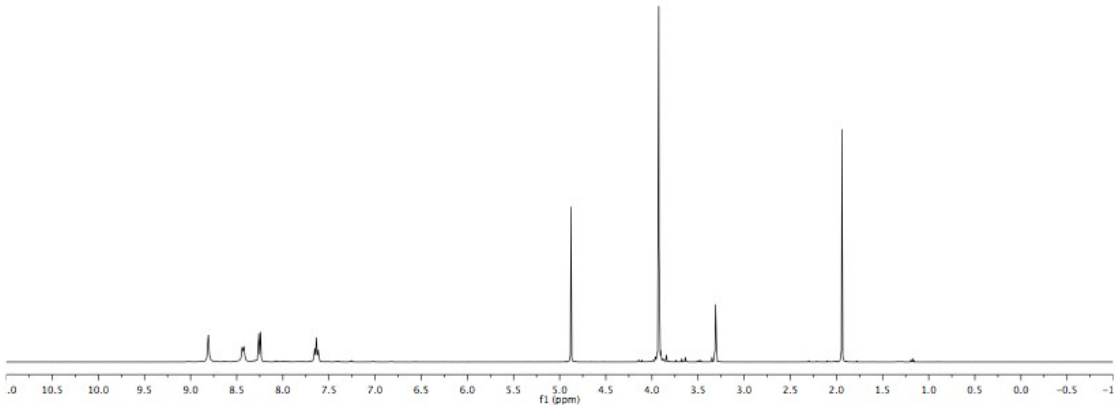
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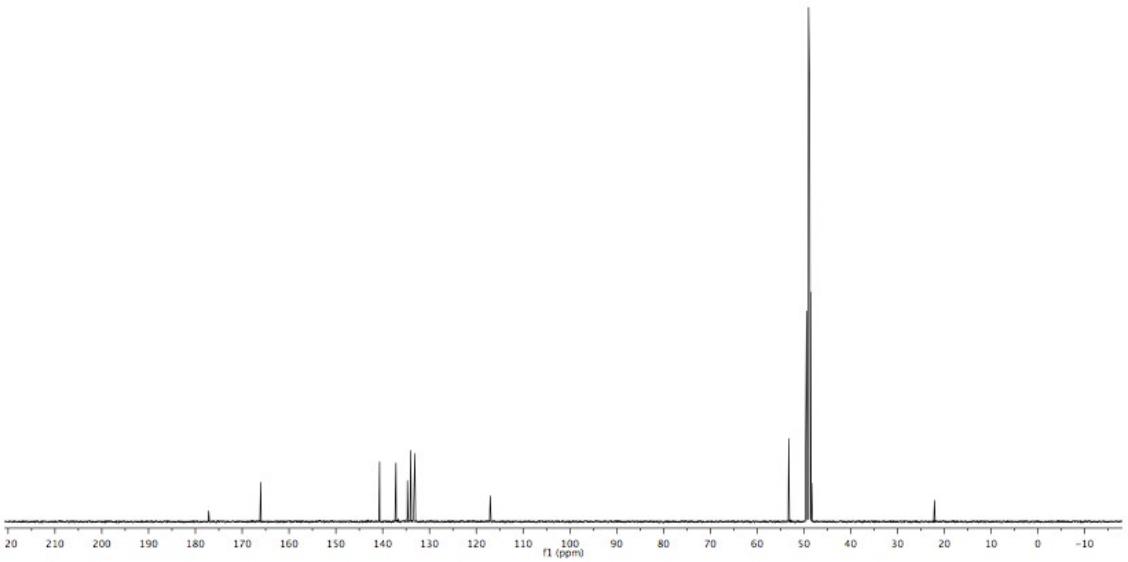
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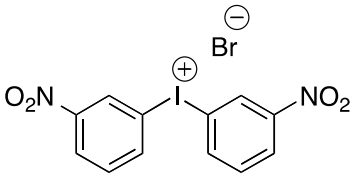
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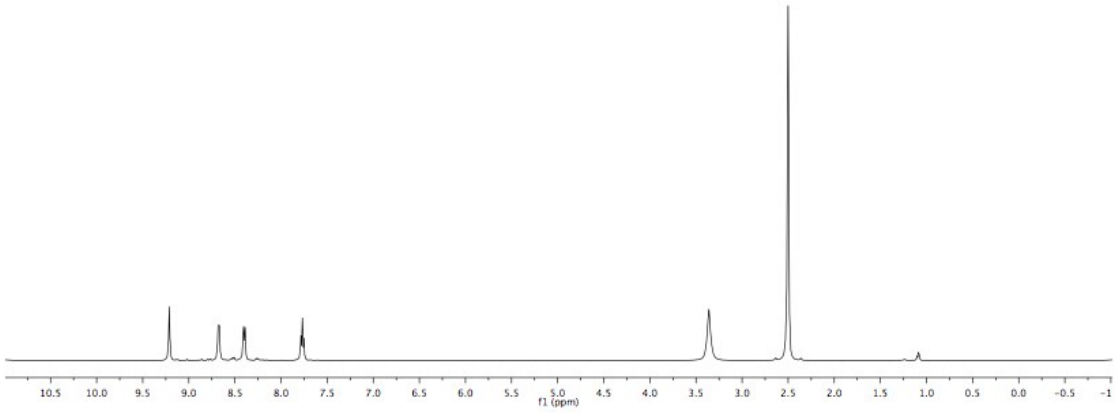
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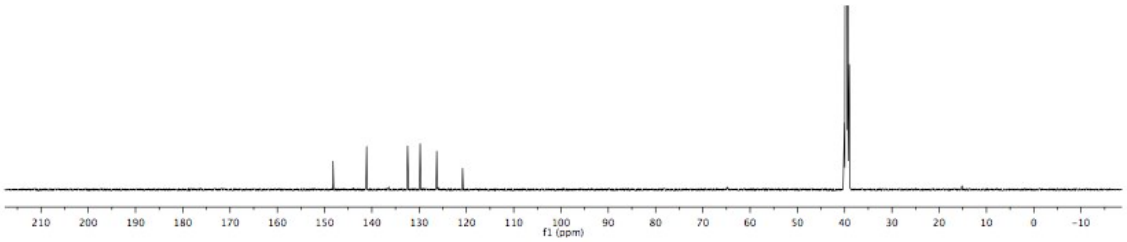
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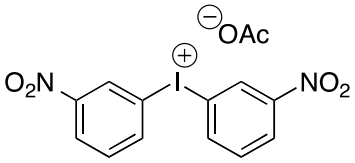
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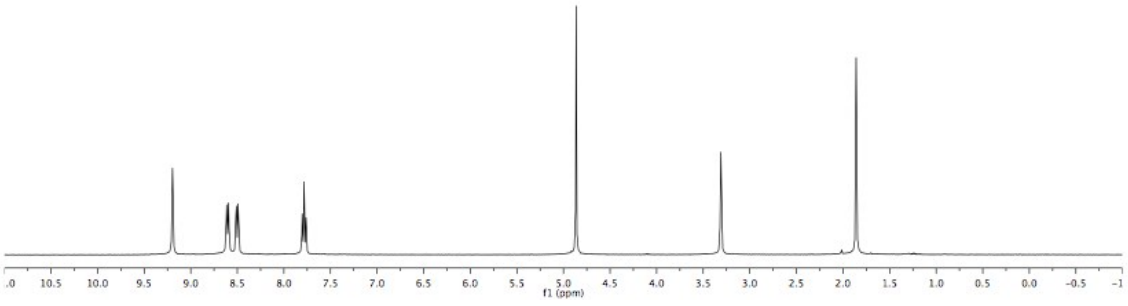
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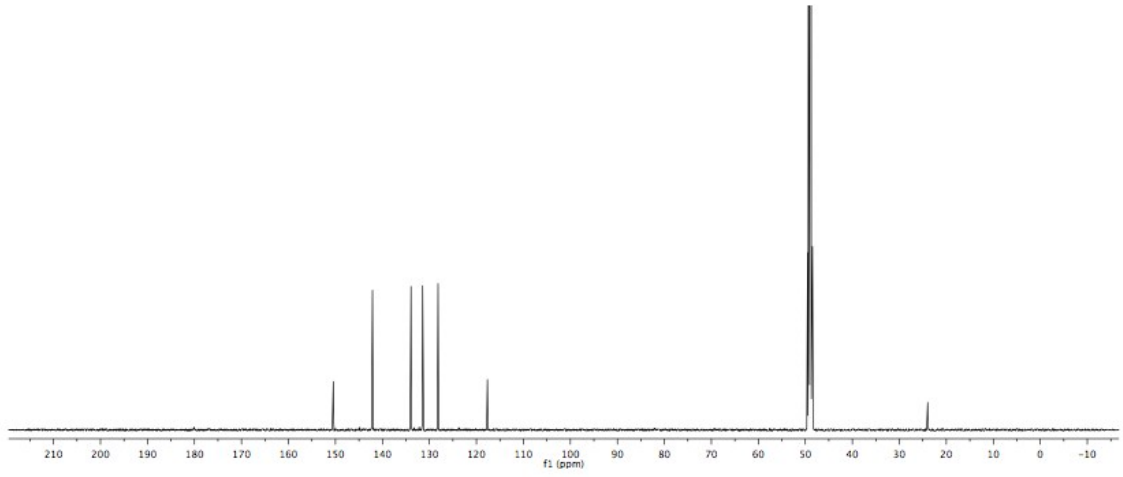
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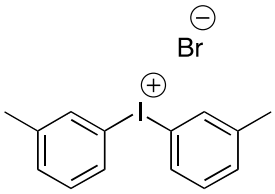
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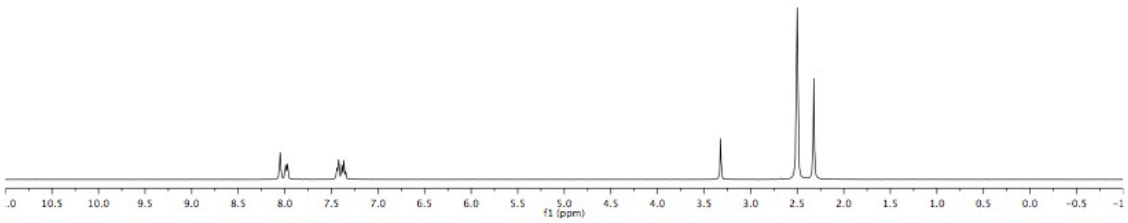
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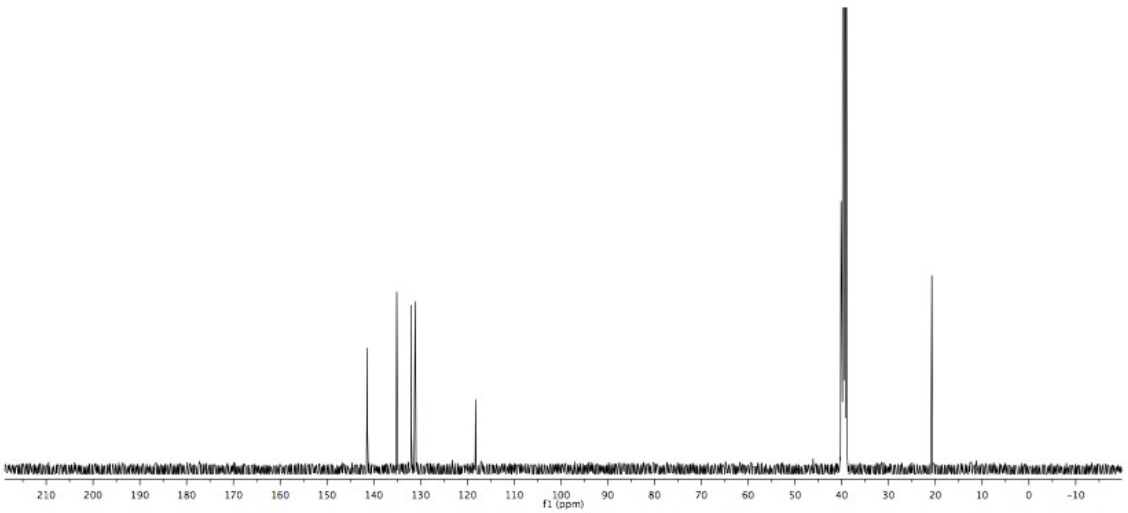
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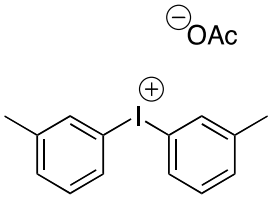
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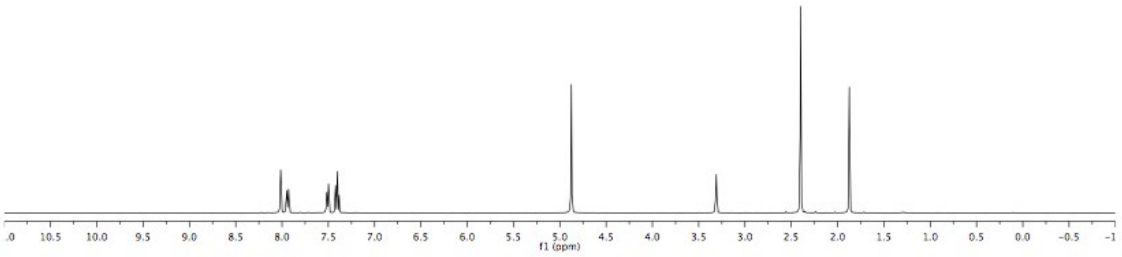
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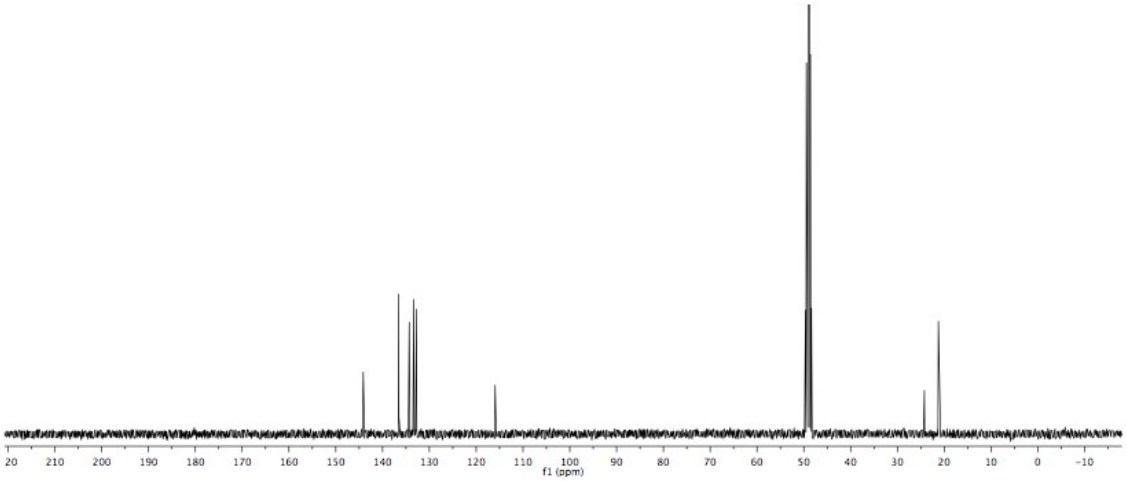
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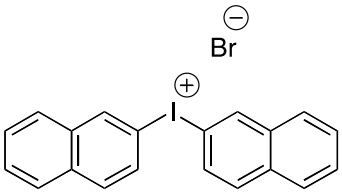
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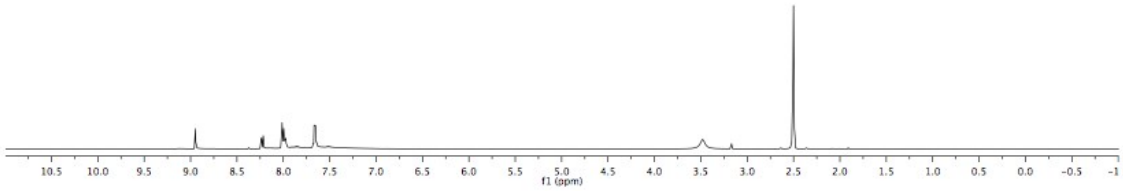
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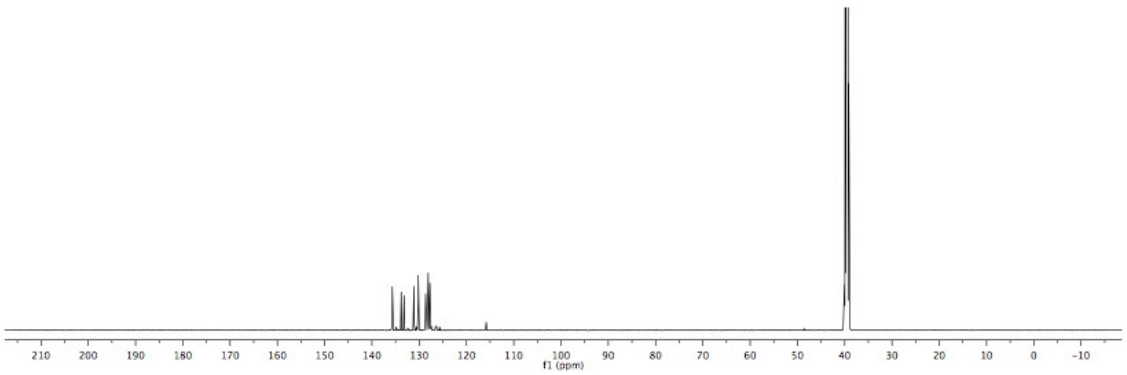
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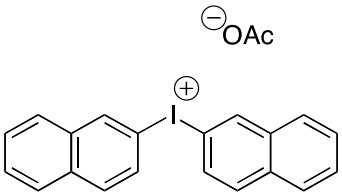
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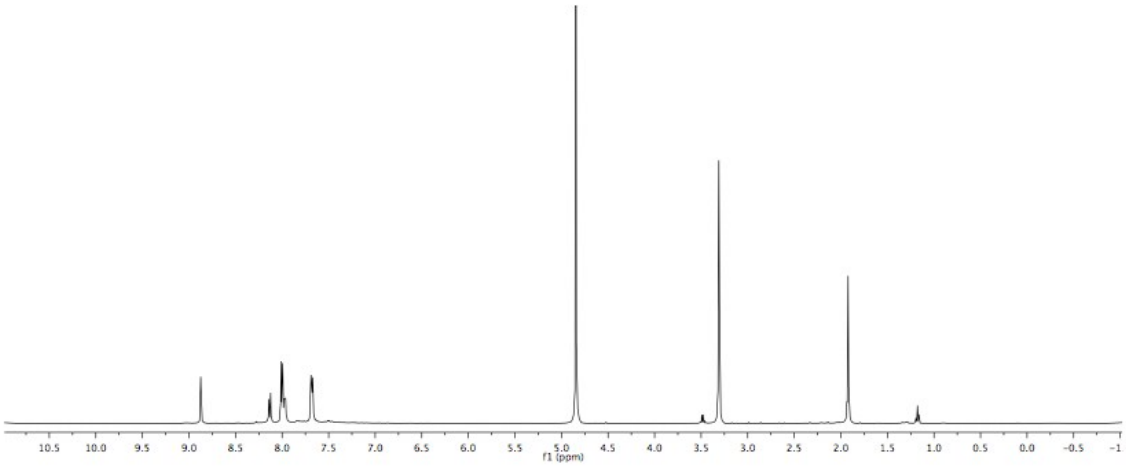
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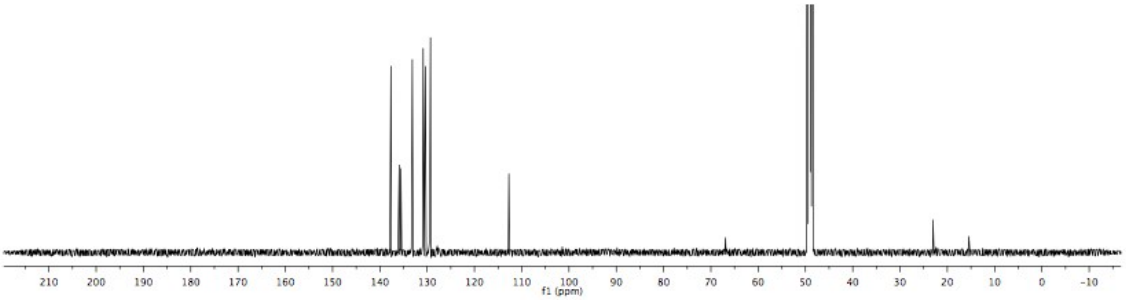
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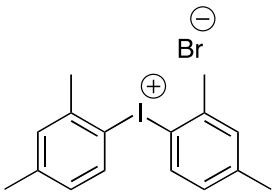
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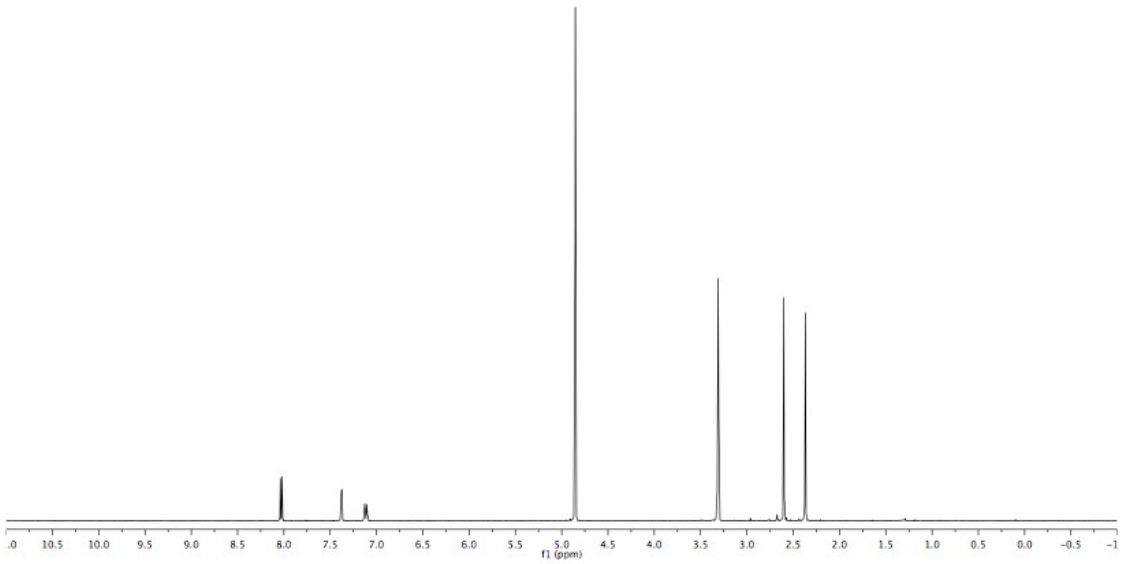
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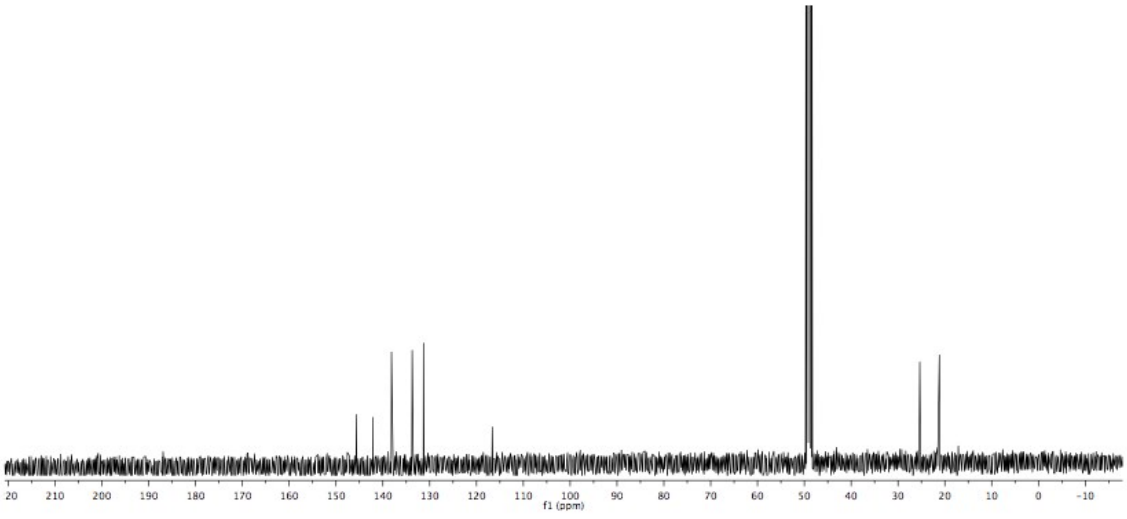
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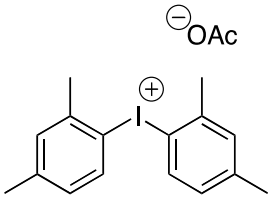
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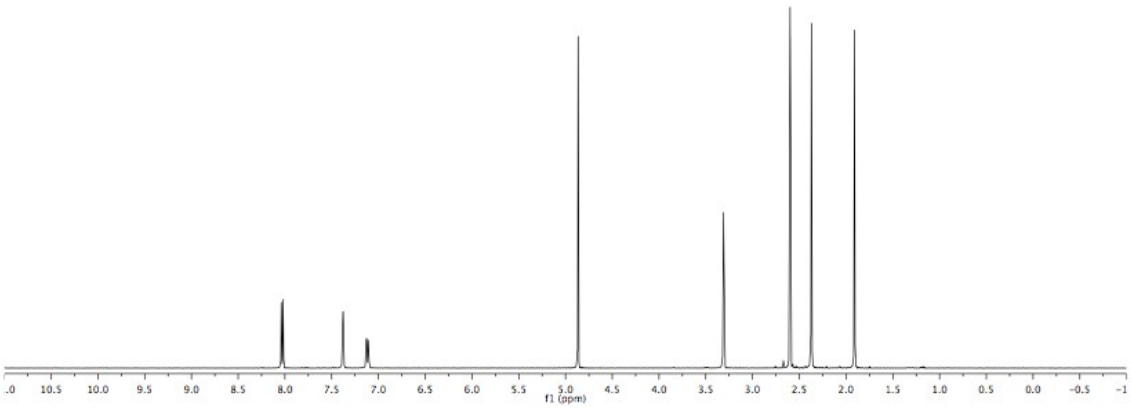
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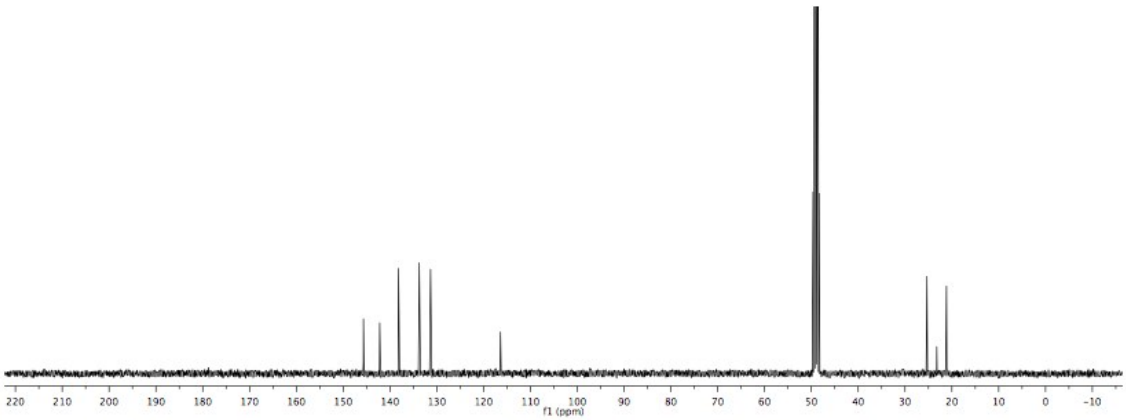
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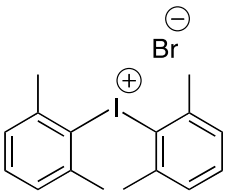
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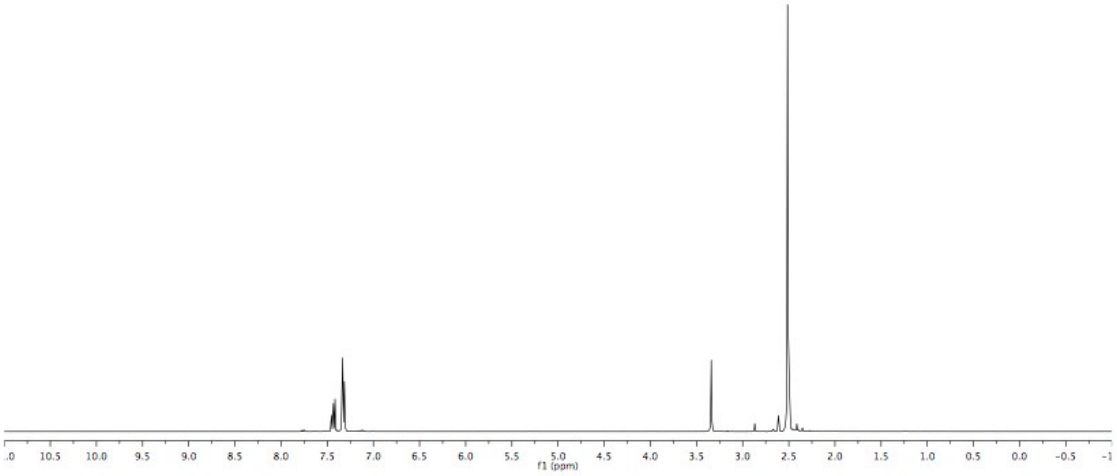
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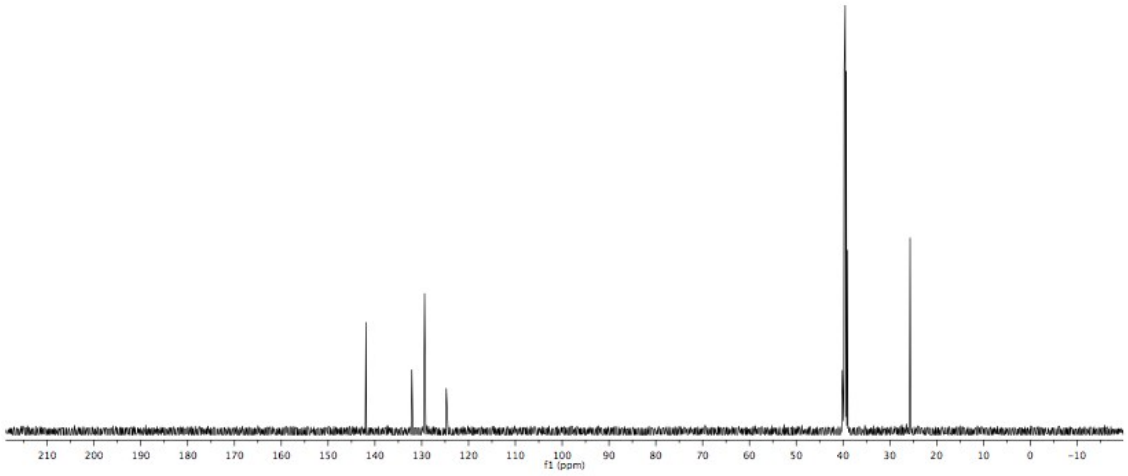
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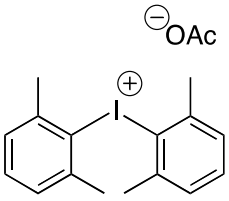
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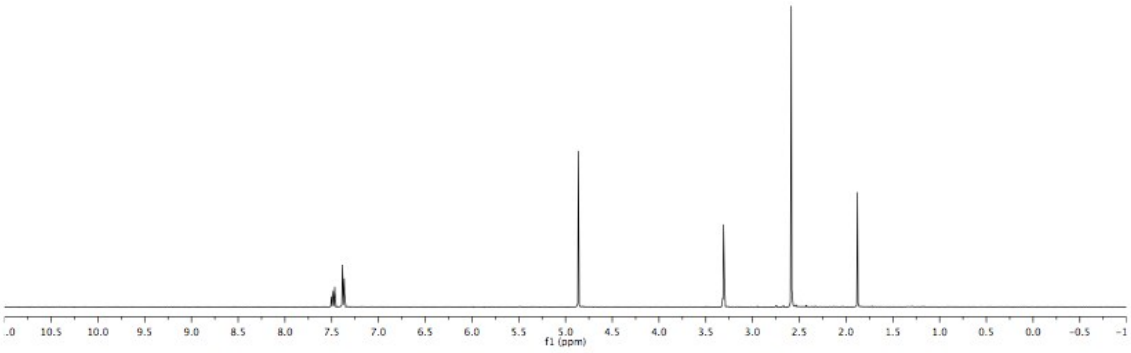
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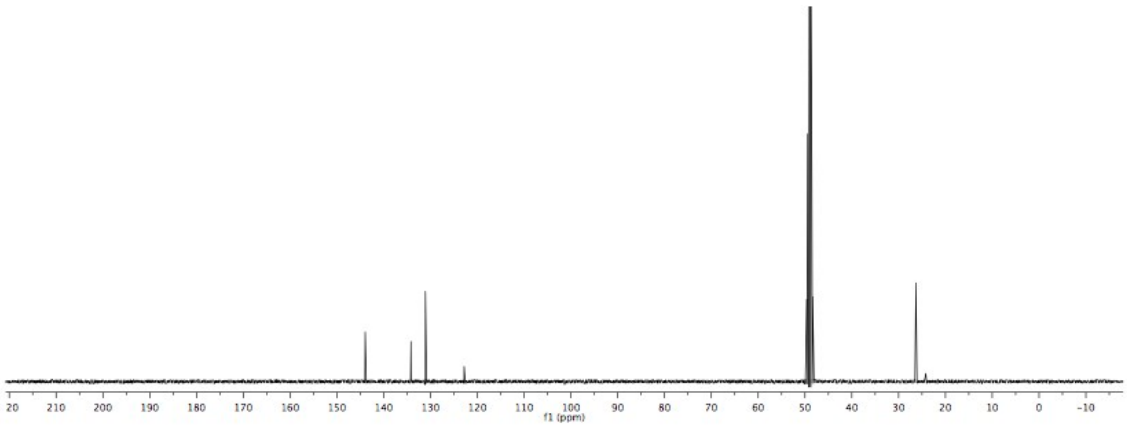
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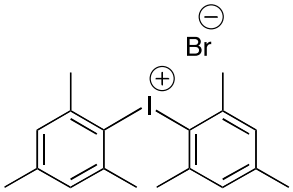
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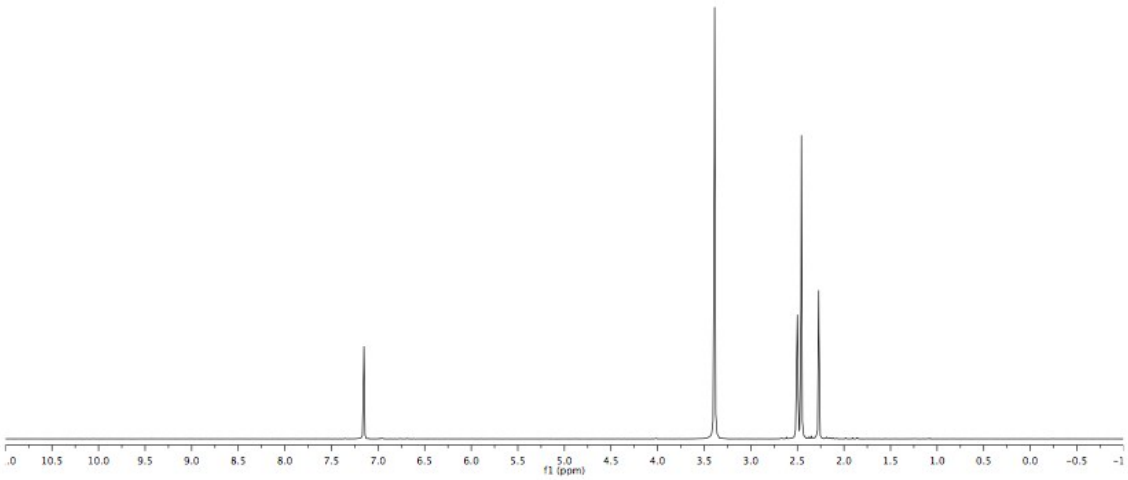
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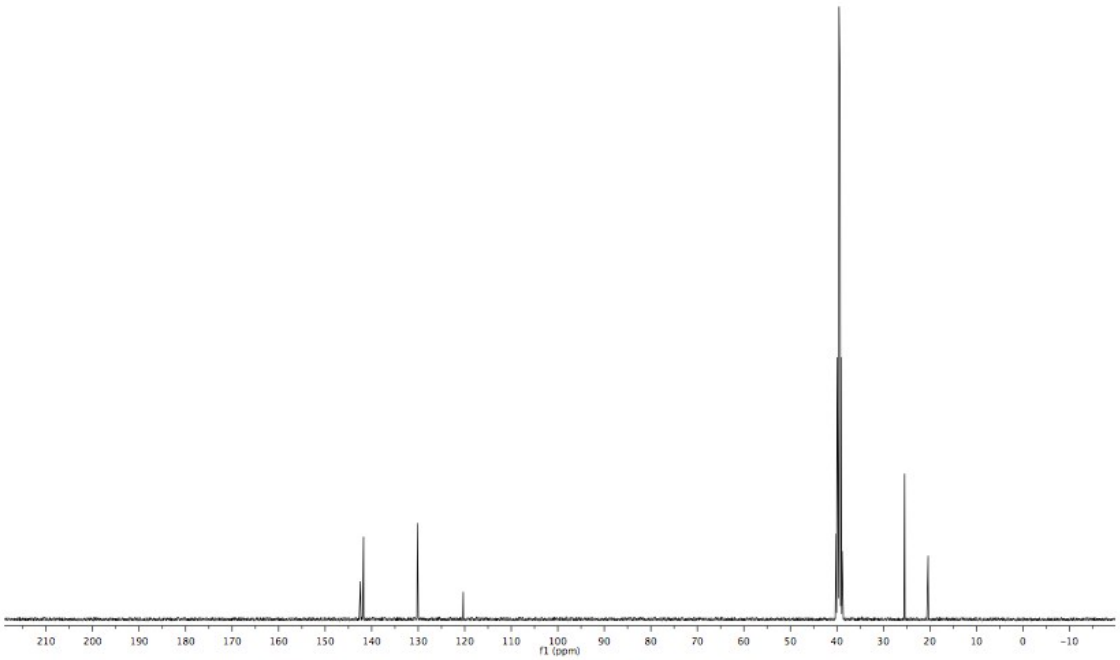
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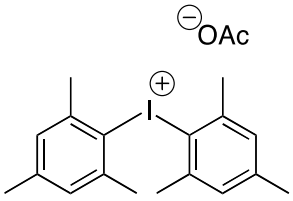
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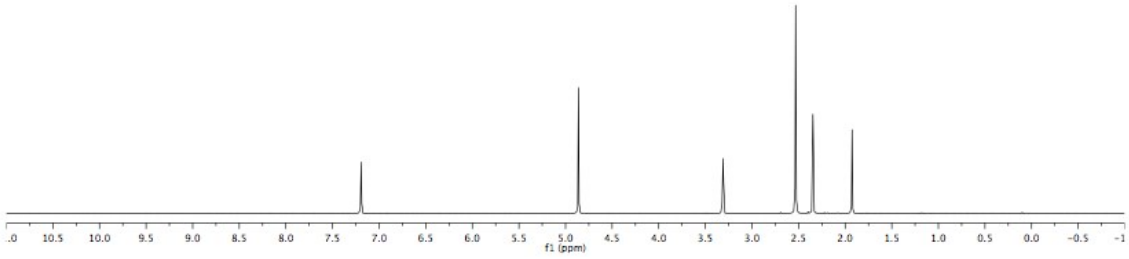
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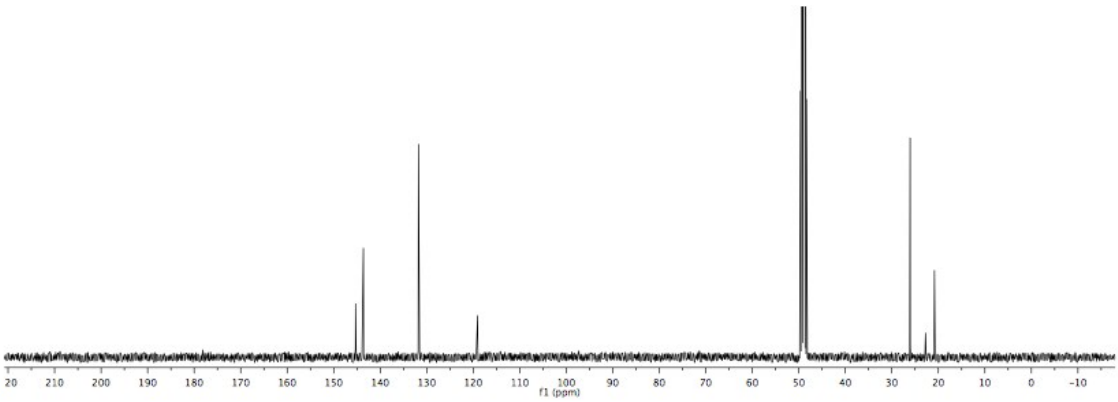
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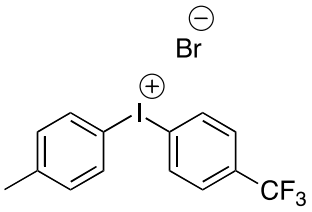
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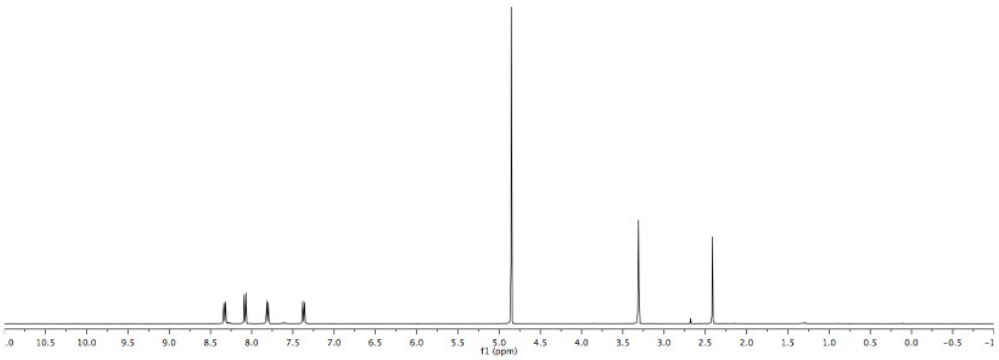
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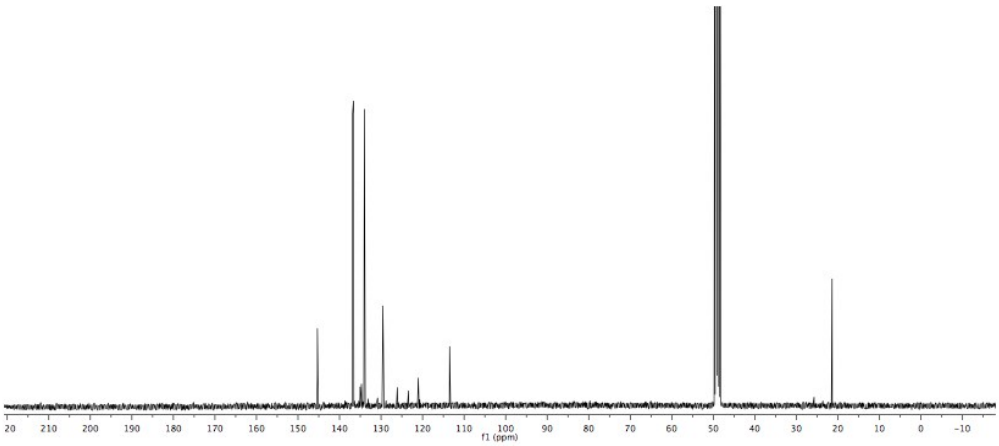
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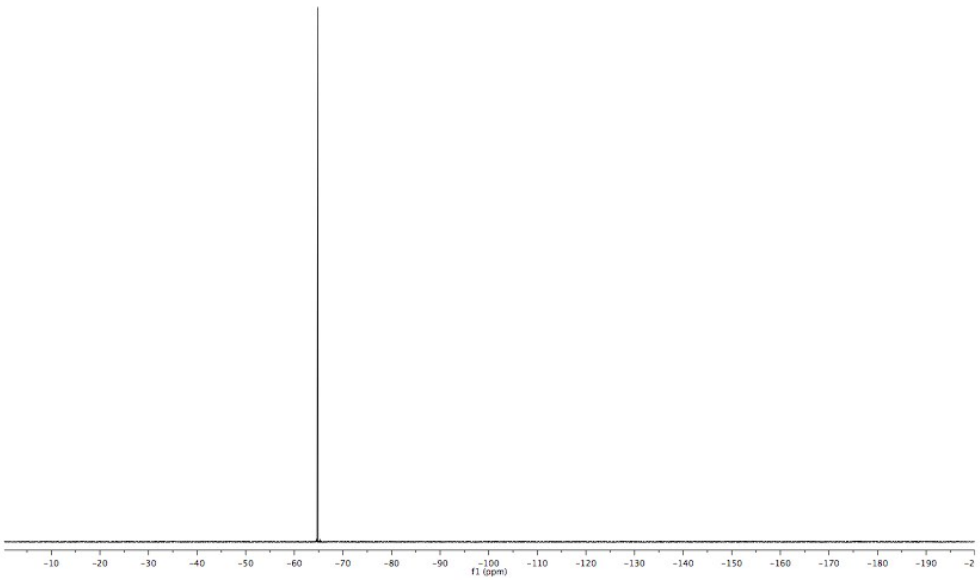
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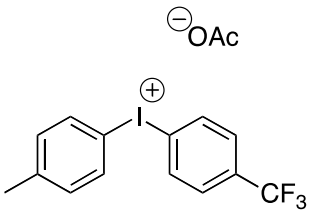
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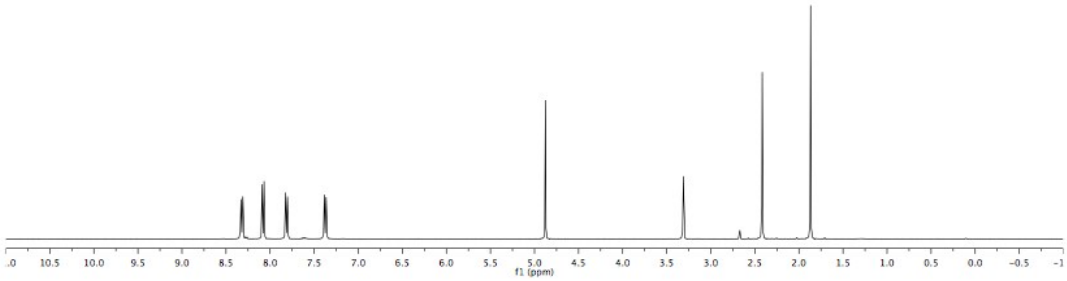
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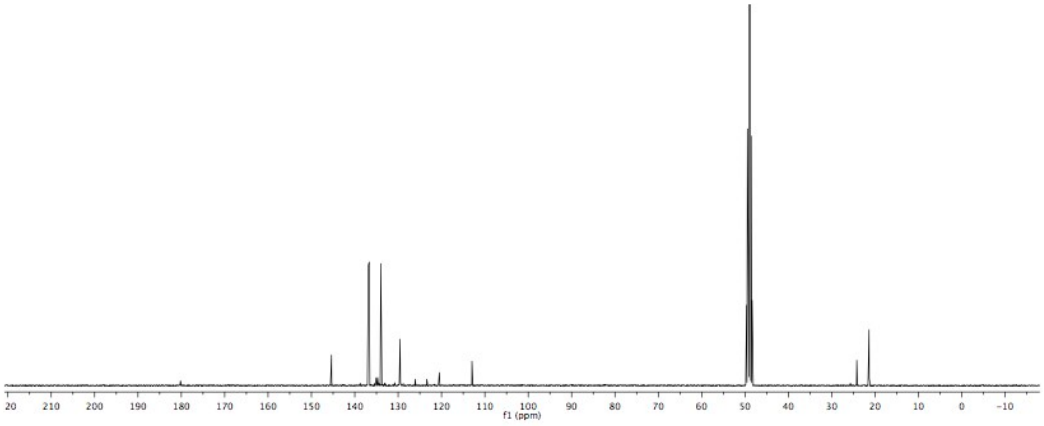
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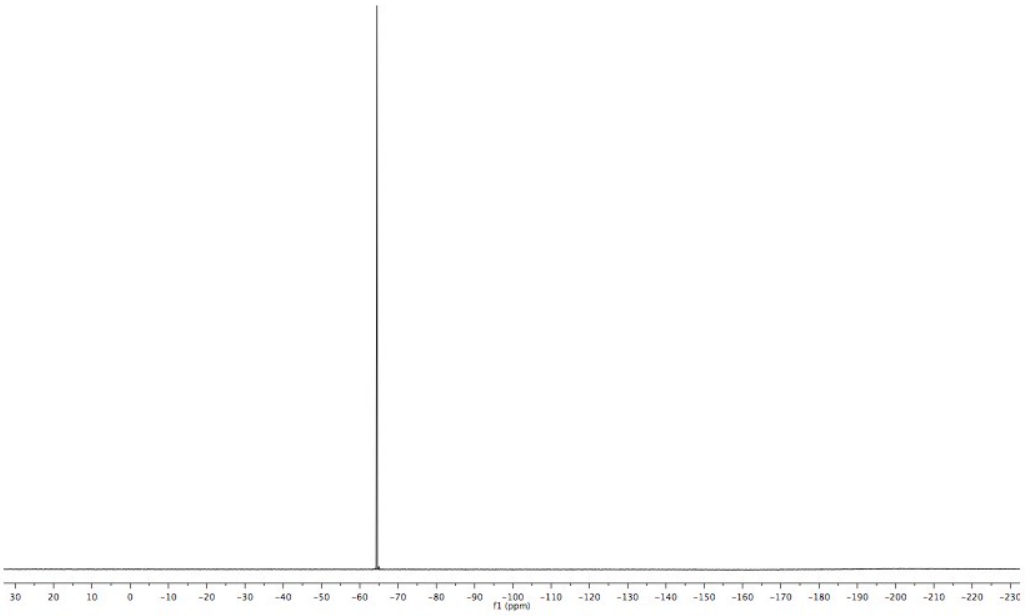
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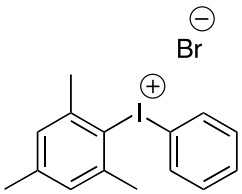
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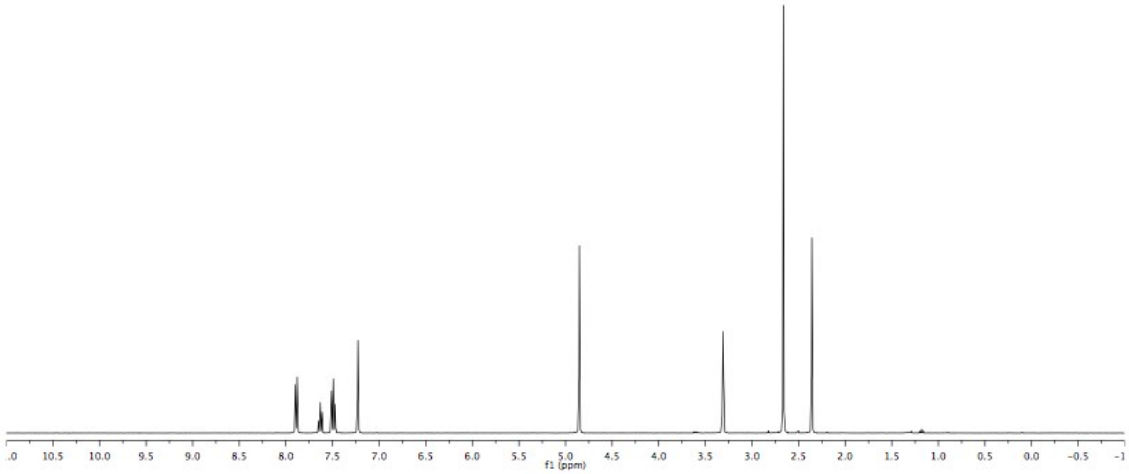
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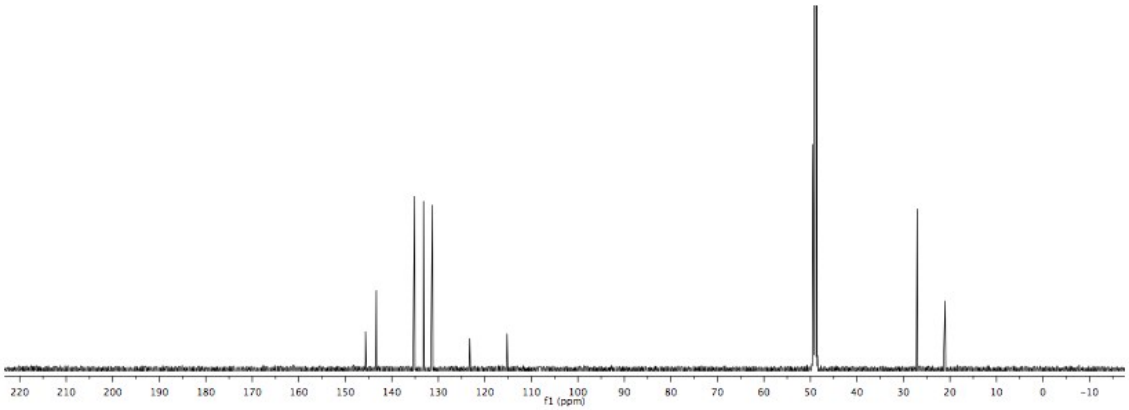
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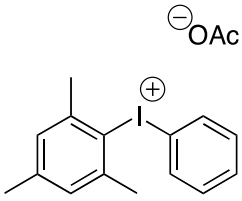
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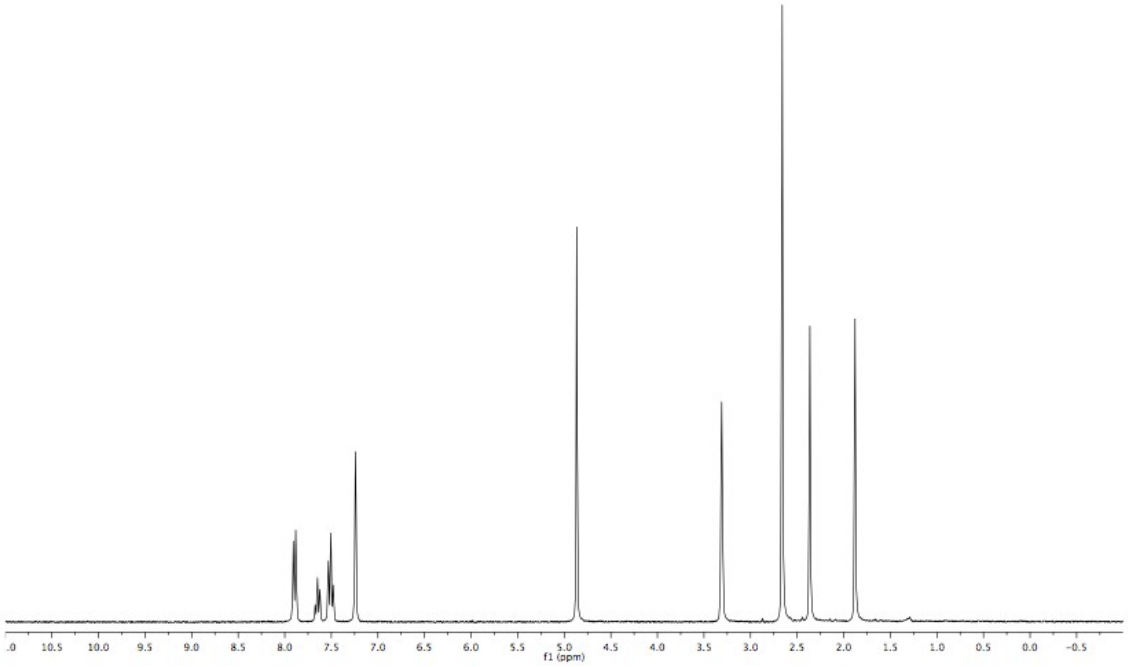
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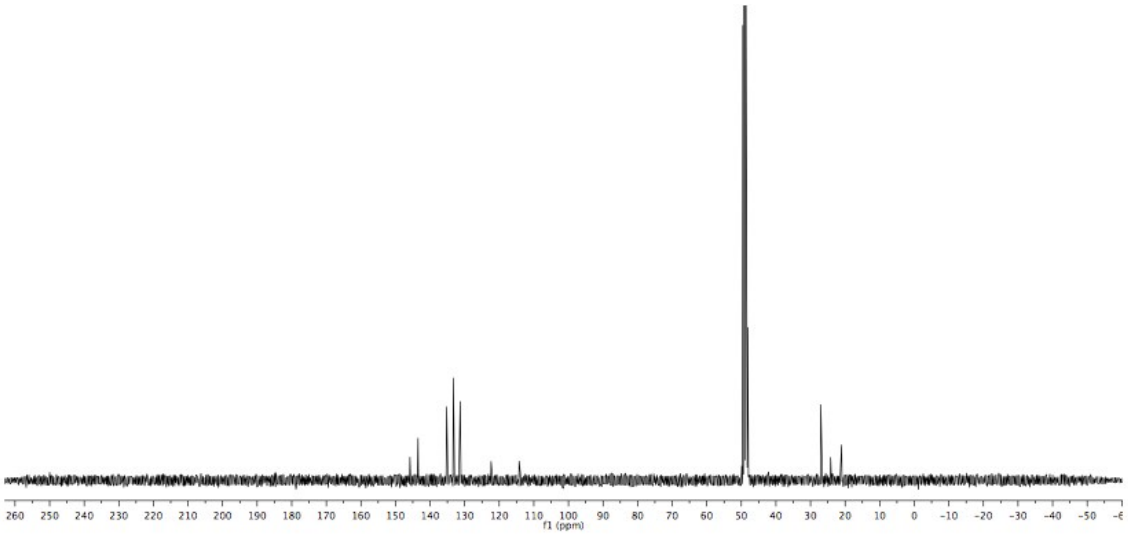
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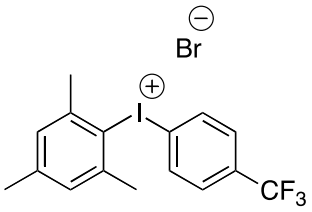
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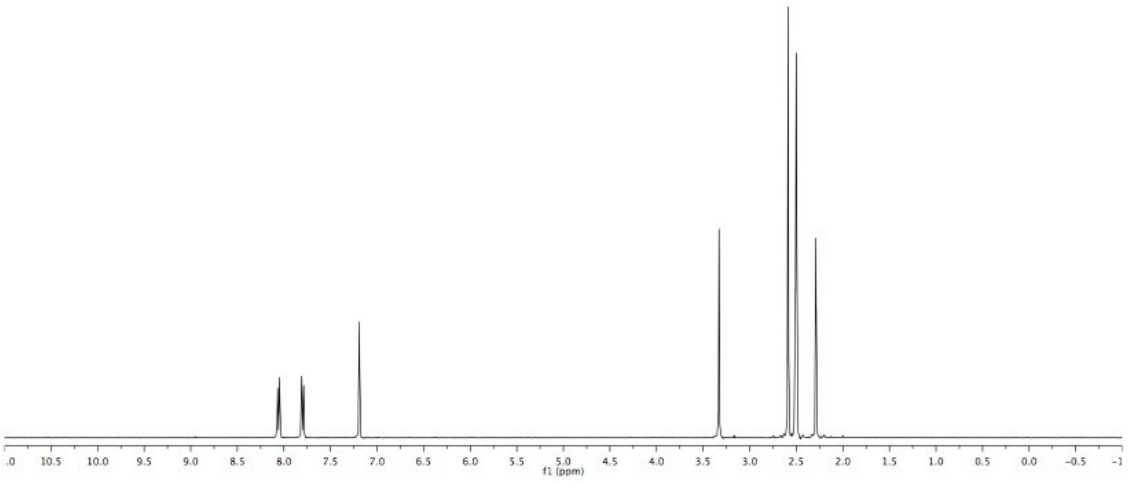
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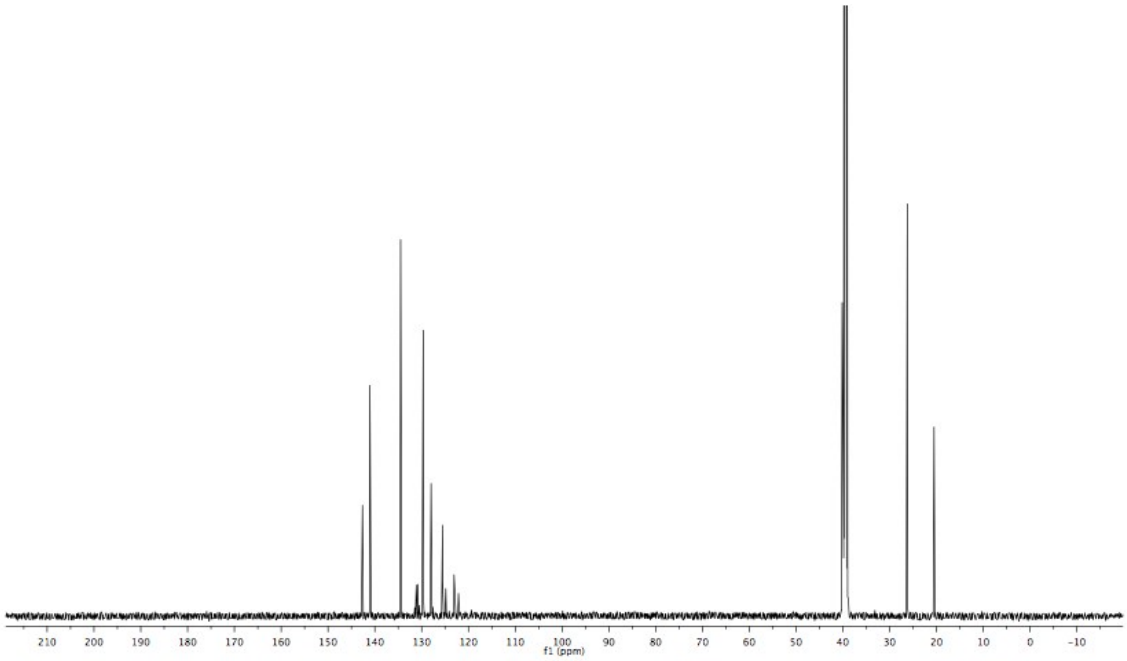
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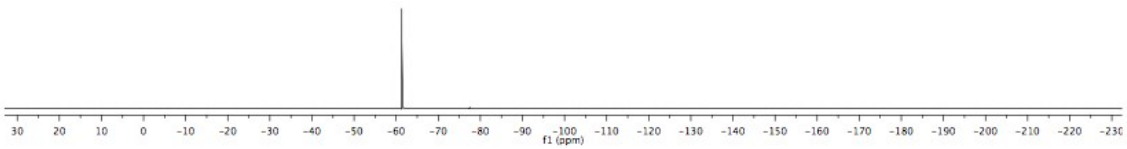
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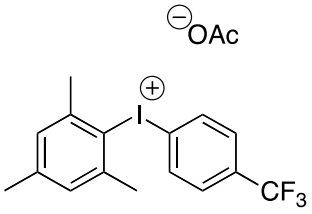
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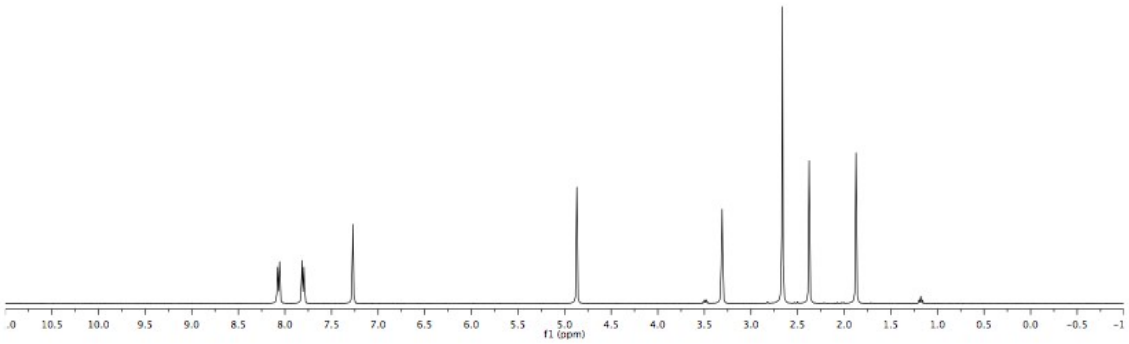
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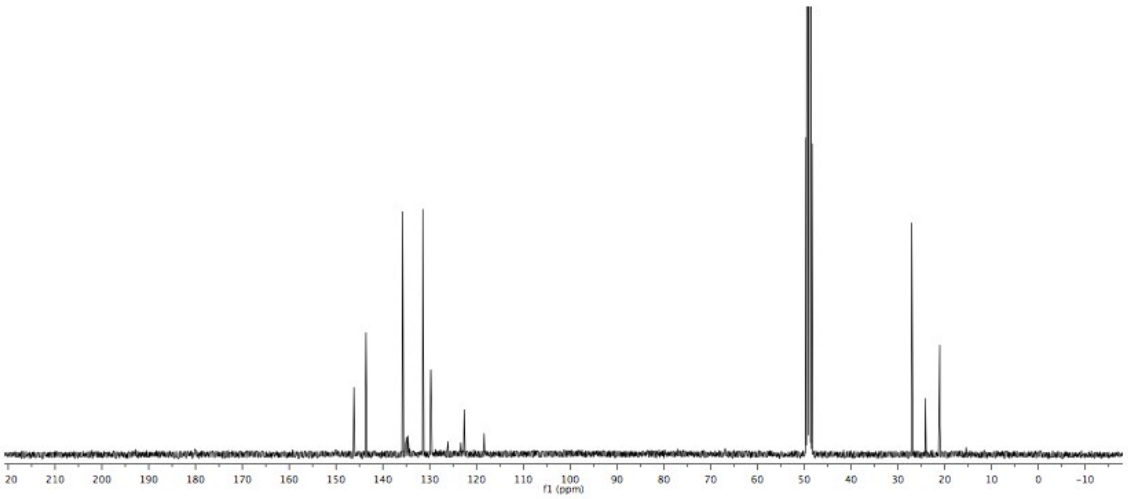
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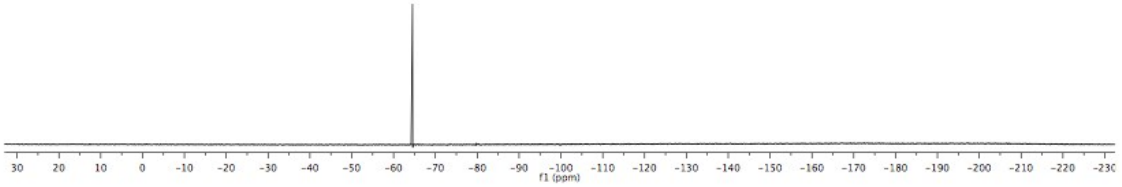
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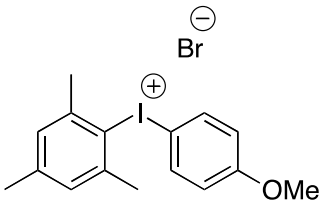
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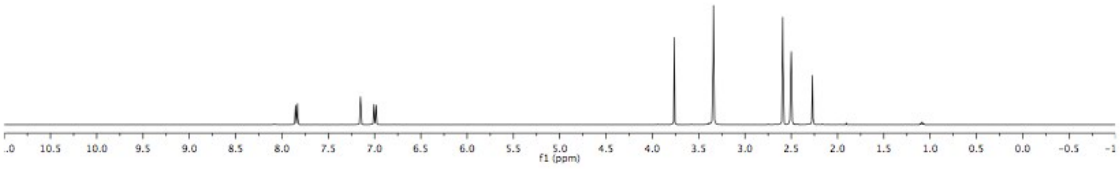
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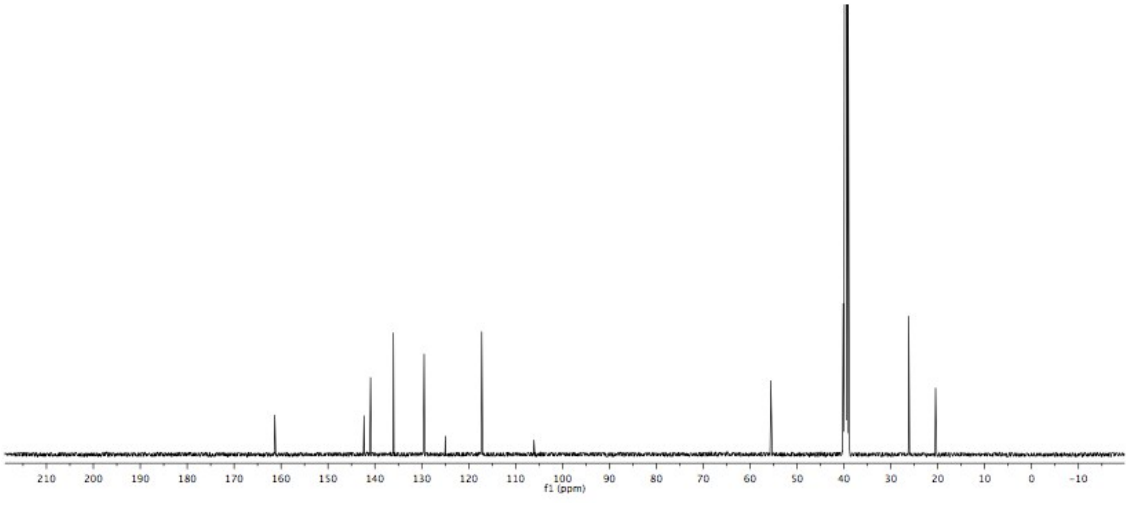
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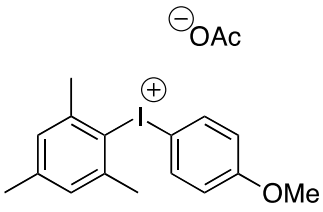
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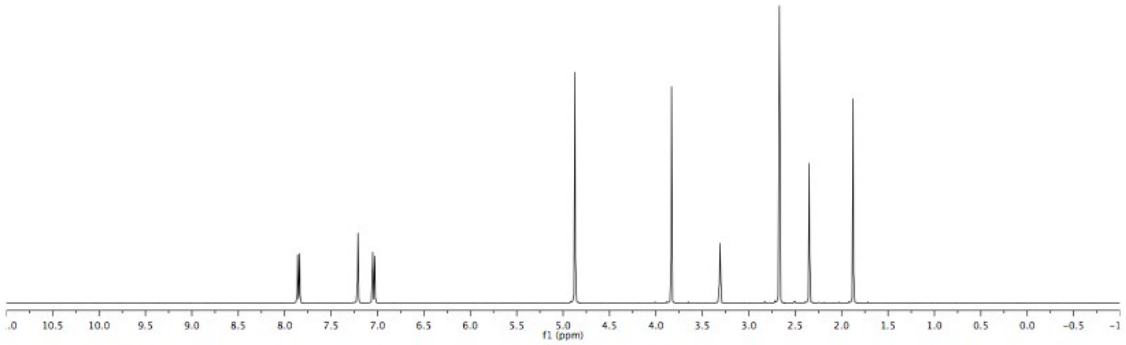
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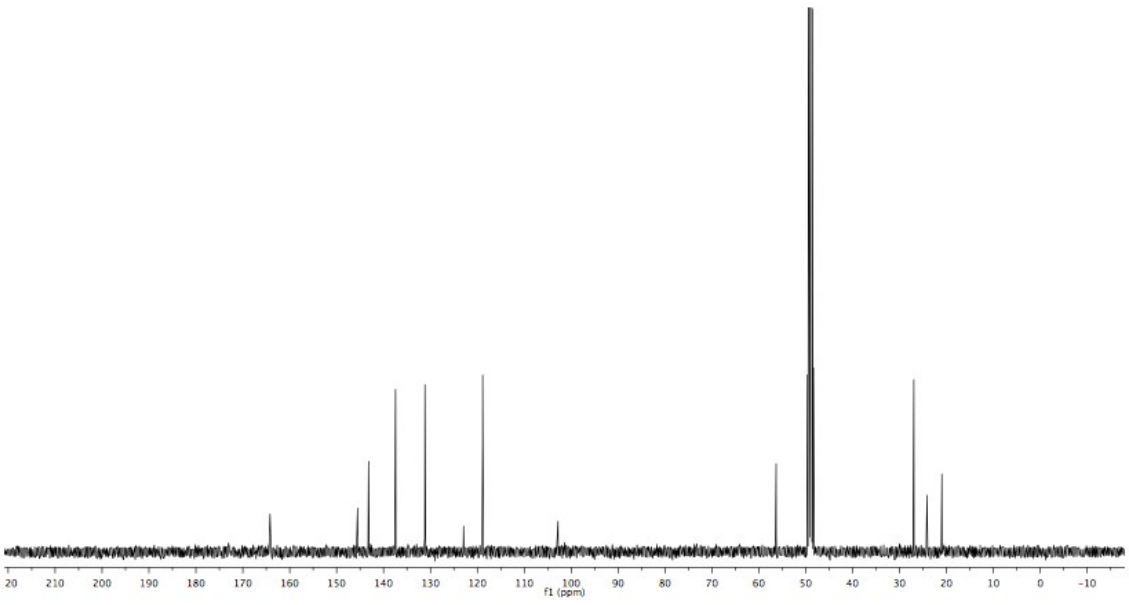
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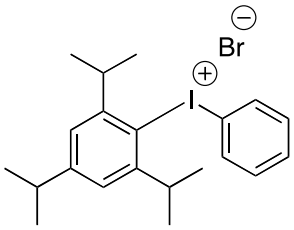
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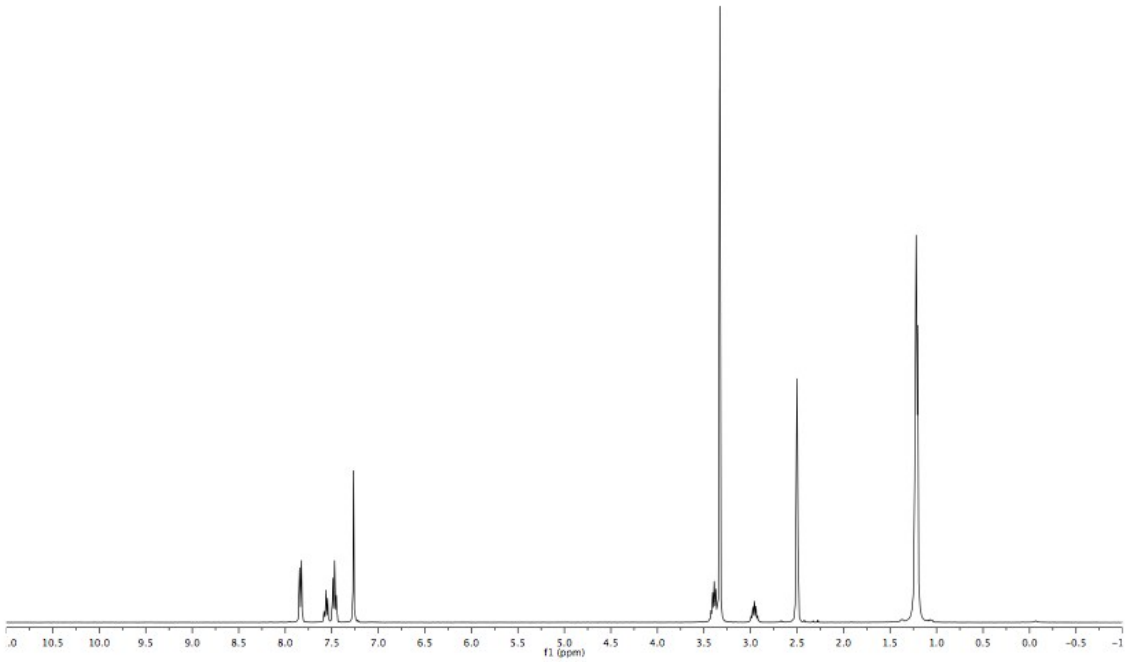
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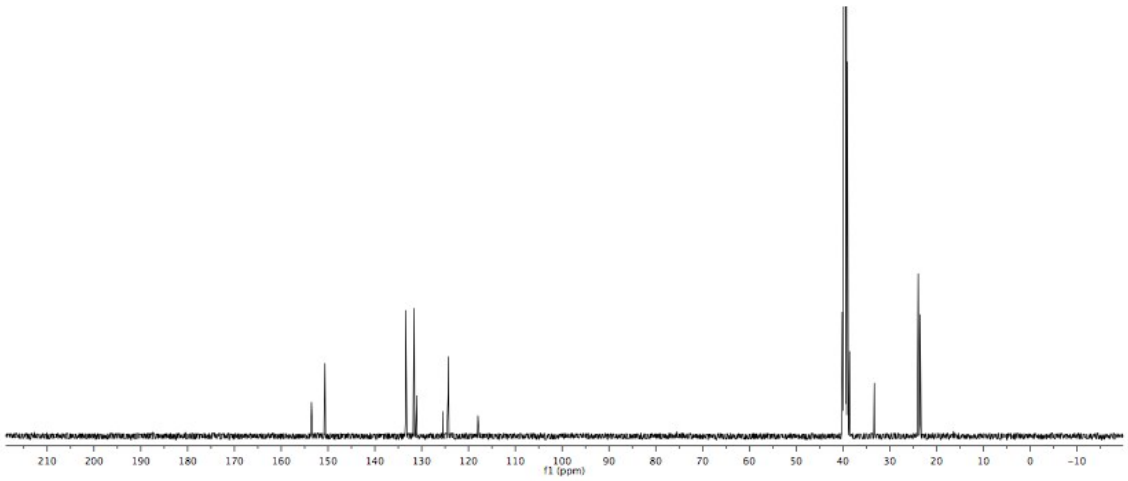
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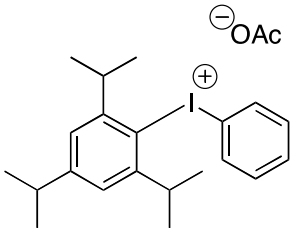
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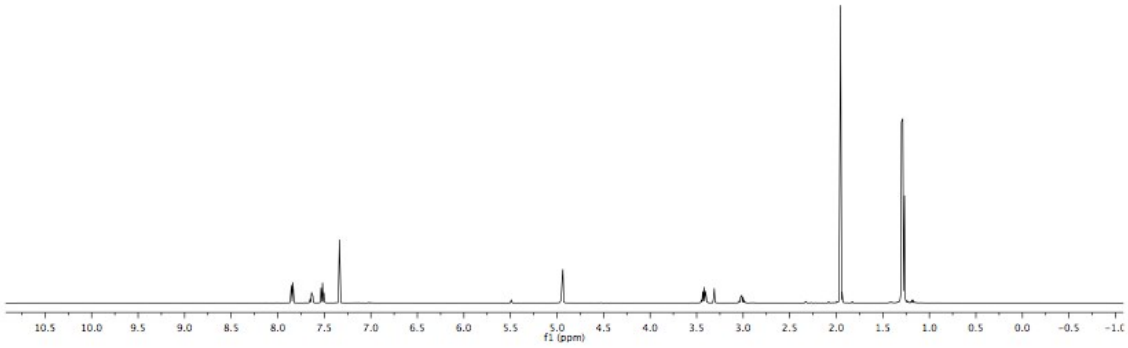
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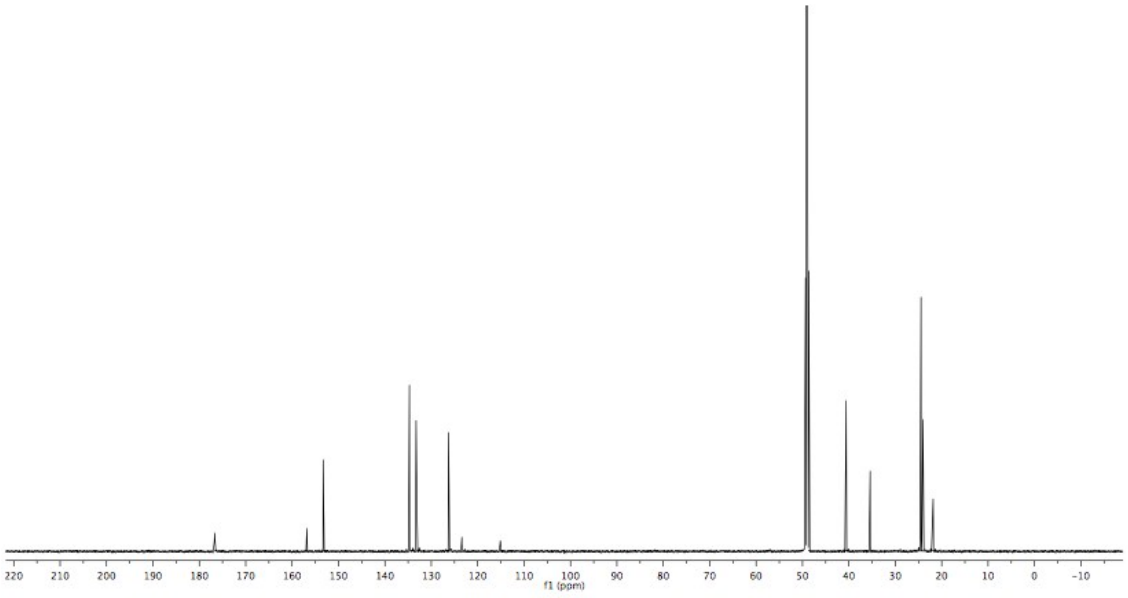
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