Supporting Information

Facile Synthesis of Diiodinated Dihydronaphthalenes and Naphthalenes via Iodine Mediated Electrophilic Cyclization

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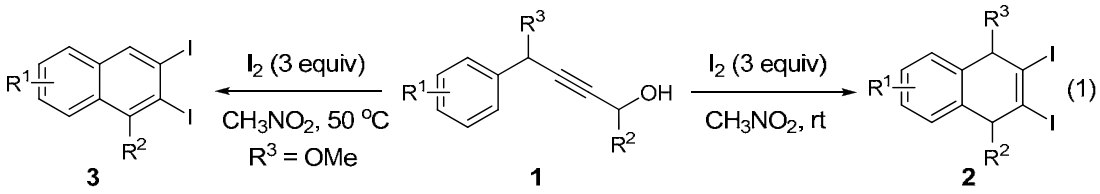
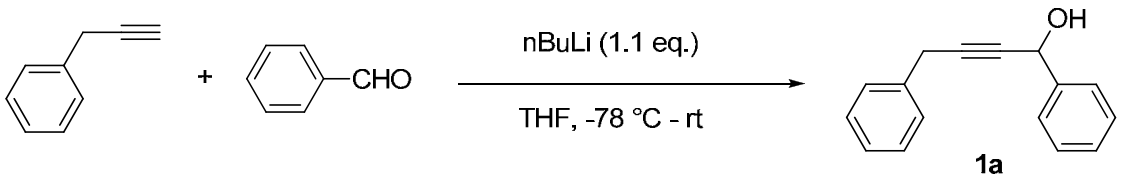


General Information. ^{1}H NMR and ^{13}C NMR spectra were recorded on JEOL JMTC-270/54/SS (JASTEC, 300 MHz, 400 MHz, 500 MHz) spectrometers. ^{1}H NMR spectra are reported as follows: chemical shift in ppm (δ) relative to the chemical shift of $CDCl_3$ at 7.26 ppm, integration, multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and br = broadened), and coupling constants (Hz). ^{13}C NMR spectra reported in ppm (δ) relative to the central line of triplet for $CDCl_3$ at 77 ppm. IR spectra were recorded on JASCO FT/IR-4100 spectrometer; absorptions are reported in cm^{-1}. High-resolution mass spectra were obtained on a BRUKER APEXIII spectrometer. Column chromatography was carried out employing Slica gel 60 N (spherical, neutral, 40~100 μm, KANTO Chemical Co.). Analytical thin-layer chromatography (TLC) was performed on 0.2 mm precoated plate Kieselgel 60 F_{254} (Merk).

Materials. Anhydrous dichloromethane, CH_3CN, MeOH, acetonitrile, THF (WAKO), I_2 (TCI), IBr (Aldrich), DDQ (Aldrich) were purchased and used as received. Various substituted aryl propargyl alcohols **1** were prepared by the modified literature methods. The structures of products were determined by ^{1}H, ^{13}C NMR, NOE, DEPT, COSY, HMBC, HMQC, high-resolution mass (ESI, EI), and reported analytical data.

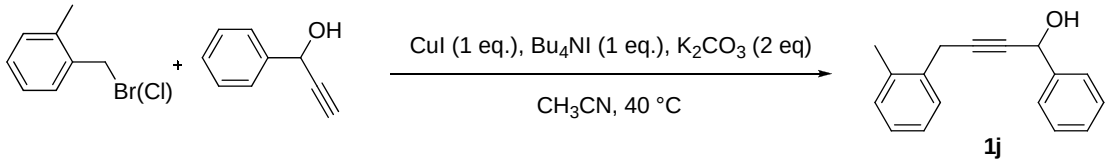
Experimental Section

1. Representative procedures for the preparation of (1a-i, 1q)[1]



To a solution of THF (10 mL) and prop-2-ynylbenzene (232 mg, 2 mmol) was added *n*-BuLi (1.4 ml, 1.65 M in hexanes) at -78ºC, and the reaction mixture was stirred for 1 h at same temperature. Benzaldehyde (212 mg, 2 mmol) was added. The reaction mixture was warmed to room temperature for 1 h and then quenched with aqueous NH_4Cl. The mixture was extracted with diethyl ether, washed with brine, dry over Na_2SO_4, The filtrate was evaporated. After chromatography on silica gel (Merck KGaA, 230-400 mesh ASTM) afforded 1,4-diphenylbut-2-yn-1-ol **1a** as a colorless oil (73%, 325 mg).

2. Representative procedures for the preparation of (1j-p)[2]



1-Phenylprop-2-yn-1-ol (182 µL, 1.5 mmol) was added via microsyringe to a stirring mixture of 1-(chloromethyl)-2-methylbenzene (232 mg, 1.5 mmol), CuI (285.7 mg, 1.5 mmol), K_2CO_3 (414.63 mg, 3 mmol), and *n*-Bu_4NI (554.07 mg, 1.5 mmol) in dry acetonitrile (2 mL). The resulting slurry was stirred at 40 °C for 72 h. The reaction mixture was then diluted with saturated aqueous ammonium chloride (20 mL), and extracted twice with diethyl ether (20 mL). The combined organic layers were dried using anhydrous Na_2SO_4, filtered, and evaporated. Chromaatography on silica gel (Merck KGaA, 230-400 mesh ASTM) afforded 1-phenyl-4-o-tolylbut-2-yn-1-ol **1j** as a colorless oil (78%, 276 mg).

Representative procedure for synthesis of 2,3-diiodo-1-phenyl-1,4-dihydronaphthalene (2a)

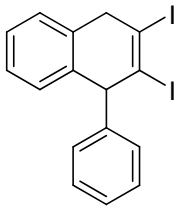
I_2 (0.6 mmol, 152 mg) was added to a solution of 1,4-diphenylbut-2-yn-1-ol **1a** (0.2 mmol, 44.5 mg) in acetonitrile (2 mL), and the resulting mixture was stirred at room temperature for 2-5 h. The reaction mixture was diluted with 10 mL dichlomethane, washed with a saturated sodium thiosulfate. The organic layer was dried with an anhydrous $MgSO_4$. After concentration of the filtrate, the residue was purified by chromatography on silica gel to

afford 2,3-diiodo-1-phenyl-1,4-dihydronaphthalene **2a** (80.6 mg, 88%) as a brown solid.

1. C. W. Downey, B. D. Mahoney, V. R. Lipari, *J. Org. Chem.* **2009**, *74*, 2904-2906.
2. J. Castro, A. Moyano, M. A. Pericàs, A. Riera, *Synthesis* **1997**, 518-520.
3. P. G. Cozzi, J. Rudolph, C. Bolm, P.-O. Norrby, C. Tomasini, *J. Org. Chem.* **2005**, *70*, 5733-5736.

Spectra data

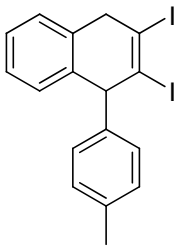
2,3-Diiodo-1-phenyl-1,4-dihydronaphthalene (2a)



2a

White solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.30 (3H, m), 7.27-7.06 (6H, m), 5.19 (1H, dd, *J* = 3.9, 3.3 Hz), 4.33 (1H, dd, *J* = 21.3, 3.3 Hz), 4.24 (1H, dd, *J* = 21.3, 3.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 46.49, 60.57, 107.79, 114.04, 126.62, 126.92, 127.01, 127.15, 128.01, 128.40, 128.73, 132.18, 136.03, 143.67; IR(neat): 696, 745, 1286, 1441, 1540, 1663, 3053, 3441 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{12}I_2Na$ $(M+Na)^+$: 480.8921, found: 480.8920.

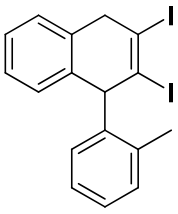
2,3-Diiodo-1-*p*-tolyl-1,4-dihydronaphthalene (2b)



2b

Brown oil; 1H NMR (300 MHz, $CDCl_3$) δ 7.17-7.01 (8H, m), 5.11 (1H, dd, *J* = 3.6, 3.3 Hz), 4.28 (1H, dd, *J* = 21.3, 3.3 Hz), 4.18 (1H, dd, *J* = 21.3, 3.6 Hz), 2.33 (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) 21.11, 46.48, 60.22, 107.57, 114.40, 126.52, 126.88, 126.99, 127.83, 128.33, 129.43, 132.14, 136.22, 136.79, 140.76; IR(neat): 733, 905, 1509, 1602, 1738, 3020 cm-^{1}; HRMS (ESI) calcd for $C_{17}H_{14}I_2Na$ $(M+Na)^+$: 494.9077, found: 494.9076.

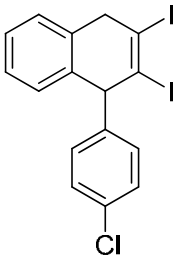
2,3-Diiodo-1-*o*-tolyl-1,4-dihydronaphthalene (2c)



2c

White solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.19-7.06 (7H, m), 6.88-6.85 (2H, m), 5.38 (1H, t, *J* = 4.5 Hz), 4.26 (2H, d, *J* = 4.5 Hz), 2.46 (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 19.67, 46.26, 55.68, 107.63, 114.50, 126.53, 126.58, 126.78, 126.99, 127.09, 128.44, 129.41, 130.90, 131.98, 135.03, 135.85, 143.16; IR(neat): 739, 905, 1376, 1601, 1738, 2922 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{14}I_2Na$ $(M+Na)^+$: 494.9077, found: 494.9076.

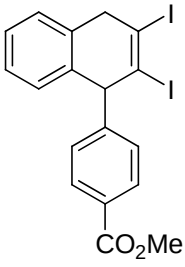
1-(4-Chlorophenyl)-2,3-diiodo-1,4-dihydronaphthalene (2d)



2d

White solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29-7.25 (2H, m), 7.21-7.07 (5H, m), 6.97-6.94 (1H, m), 5.10 (1H, dd, *J* = 3.9, 3.0 Hz), 4.26 (1H, dd, *J* = 21.3, 3.0 Hz), 4.16 (1H, dd, *J* = 21.3, 3.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 46.37, 59.77, 108.21, 113.43, 126.84, ,127.02, 127.11, 128.32, 128.91, 129.35, 132.13, 132.96, 135.47, 142.21 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{11}I_2ClNa$ $(M+Na)^+$: 514.8531, found: 514.8529.

Methyl 4-(2,3-diiodo-1,4-dihydronaphthalen-1-yl)benzoate (2e)

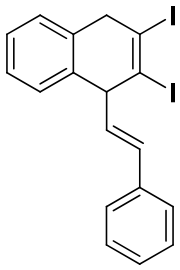


2e

Brown oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.97 (2H, m), 7.28-7.24 (2H, m), 7.20-7.09 (3H, m), 6.97-6.95 (1H, m), 5.19 (1H, dd, *J* = 4.0, 3.2 Hz), 4.27 (1H, dd, *J* = 21.6, 3.2 Hz), 4.22 (1H, dd, *J* = 21.6, 4.0 Hz), 3.89 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 46.43, 52.09, 60.32, 108.50, 112.67, 126.97, 127.10, 127.16, 128.11, 128.35, 129.08, 130.15, 132.19, 135.24, 148.69, 166.71; IR(neat): 729, 905, 1108, 1274, 1433, 1607, 1715, 2947, 3025 cm^{-1}; HRMS (ESI) calcd

for $C_{18}H_{14}I_2O_2Na$ $(M+Na)^+$: 538.8975, found: 538.8977.

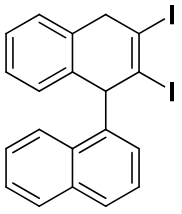
(*E*)-2,3-Diiodo-1-styryl-1,4-dihydronaphthalene (2f)



2f

Slight yellow solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44-7.25 (7H, m), 7.22-7.12 (2H, m), 6.52 (1H, d, *J* = 15.6 Hz), 6.15 (1H, dd, *J* = 8.4, 15.6 Hz), 4.77 (1H, dt, *J* = 8.4, 3.3 Hz), 4.20 (1H, dd, *J* = 20.7, 2.1 Hz), 4.04 (1H, dd, *J* = 20.7, 3.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 46.29, 58.62, 107.86, 112.34, 126.51, 126.93, 126.96x2, 127.62, 128.18, 128.49, 129.60, 131.17, 132.88, 134.60, 136.54; IR(neat): 687, 737, 957, 1446, 1491, 1738, 2923, 3021 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{14}I_2Na$ $(M+Na)^+$: 506.9077, found: 506.9076.

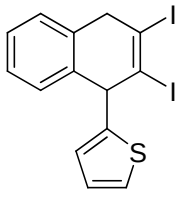
2,3-Diiodo-1,4-dihydro-1,1'-binaphthyl (2g)



2g

White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (1H, d, *J* = 7.6 Hz), 7.89 (1H, d, *J* = 7.6 Hz), 7.78 (1H, d, *J* = 8.4 Hz), 7.57 (1H, t, *J* = 7.6 Hz), 7.50 (1H, t, *J* = 7.6 Hz), 7.42 (1H, t, *J* = 7.6 Hz), 7.30-7,27 (1H, m), 7.15-7.09 (2H, m), 6.98 (1H, t, *J* = 7.6 Hz), 6.90 (1H, d, *J* = 7.6 Hz), 5.94 (1H, bs), 4.39 (1H, dd, *J* = 21.1, 5.2 Hz), 4.33 (1H, dd, *J* = 21.1, 4.4 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 46.34, 54.76, 107.83, 114.56, 123.13, 125.59, 125.72, 126.41, 126.66, 126.97, 126.99, 127.25, 127.91, 128.03, 129.00, 131.15, 131.68, 134.13, 136.32, 141.04; IR(neat): 737, 773, 1216, 1366, 1738, 2921 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{14}I_2Na$ $(M+Na)^+$: 530.9077, found: 530.9076.

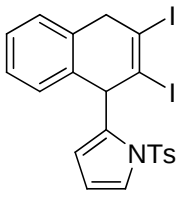
2-(2,3-Diiodo-1,4-dihydronaphthalen-1-yl)thiophene (2h)



2h

Yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28-7.14 (6H, m), 6.97-6.94 (2H, m), 5.49 (1H, dd, *J* = 3.3, 2.4 Hz), 4.25 (1H, dd, *J* = 20.7, 2.4 Hz), 4.14 (1H, dd, *J* = 20.4, 3.3 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 46.30, 55.83, 108.56, 112.35, 125.01, 125.07, 126.59, 126.98, 127.01, 127.09, 127.99, 132.55, 135.97, 146.41 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{10}I_2SNa$ $(M+Na)^+$: 486.8485, found: 486.8484.

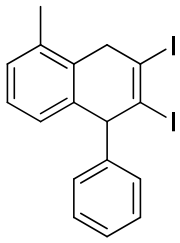
2-(2,3-Diiodo-1,4-dihydronaphthalen-1-yl)-1-tosyl-1*H*-pyrrole (2i)



2i

White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (2H, bs Hz), 7.43 (1H, bs), 7.27-7.23 (3H, m), 7.13-7.08 (2H, m), 6.98 (1H, d, *J* = 8.4 Hz), 6.19 (1H, t, *J* = 3.2 Hz), 6.00 (1H, bs), 5.76 (1H, bs), 4.10 (2H, s), 2.37 (3H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.58, 46.26, 50.98, 109.43, 112.25, 123.59, 126.65, 126.82, 126.97, 127.49(x2), 128.27, 129.85, 136.50, 136.91, 145.01; IR(neat): 671, 745, 1171, 1351, 1738, 2926 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{17}I_2NO_2SNa$ $(M+Na)^+$: 623.8962, found: 623.8958.

2,3-Diiodo-5-methyl-1-phenyl-1,4-dihydronaphthalene (2j)

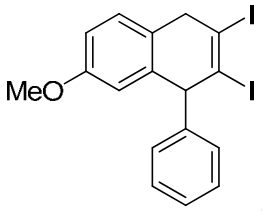


2j

White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.13 (5H, m), 7.02-6.98 (2H, m), 6.82-6.79 (1H, m), 5.09 (1H, dd, *J* = 4.0, 3.2 Hz), 4.12 (1H, dd, *J* = 21.2, 3.2 Hz), 3.95 (1H, dd, *J* = 21.2, 4.0 Hz), 2.23 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.41, 44.86, 60.65, 107.88, 114.34, 126.28, 126.76, 127.02, 127.93, 127.98, 128.62, 130.70, 134.65, 135.76, 144.14; IR(neat): 694, 733, 788, 1215, 1450, 1738, 3019 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{14}I_2Na$ $(M+Na)^+$: 494.9077,

found: 494.9076.

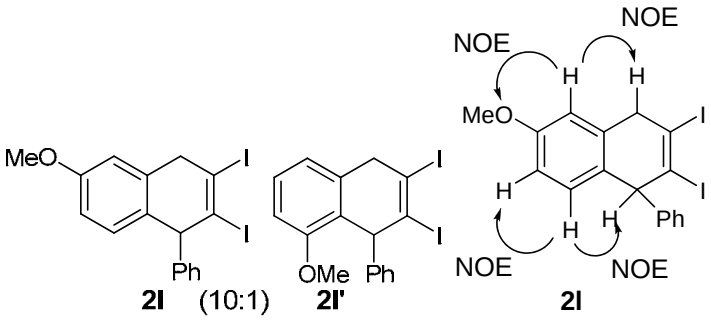
2,3-Diiodo-7-methoxy-1-phenyl-1,4-dihydronaphthalene (2k)

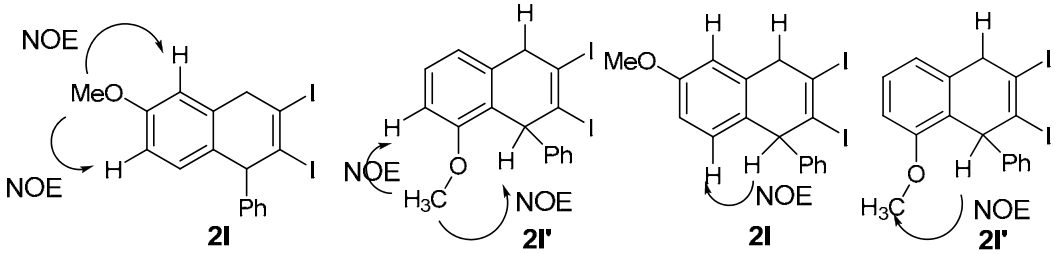


2k

Brown solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31-7.14 (5H, m), 6.98 (1H, d, *J* = 8.4 Hz), 6.71 (1H, dd, *J* = 8.4, 2,7 Hz), 6.48 (1H, d, *J* = 2.7 Hz), 5.05 (1H, dd, *J* = 3.6 Hz), 4.17 (1H, dd, *J* = 20.6, 3.6 Hz), 4.08 (1H, dd, *J* = 20.6, 3.6 Hz), 3.66 (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 45.91, 55.18, 60.82, 108.21, 112.89, 113.14, 113.78, 124.58, 127.17, 127.95, 128.00, 128.74, 137.11, 143.57, 158.30 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{14}I_2ONa$ $(M+Na)^+$: 510.9026, found: 510.9025.

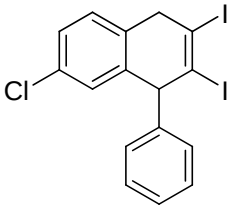
2,3-Diiodo-6-methoxy-1-phenyl-1,4-dihydronaphthalene (2l)





2l (containing 1/10 of **2l'**); Brown oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31-7.13 (5H, m), 6.88 (1H, d, *J* = 8.4 Hz), 6.67 (1H, dd, *J* = 8.4, 2.7 Hz), 6.57 (1H, d, *J* = 2.7 Hz), 5.35 (**2l'**, 1H, dd, J = 3.6, 2.4 Hz), 5.05 (**2l**, 1H, t, *J* = 3.6 Hz), 4.18 (**2l**+**2l'**, 2H, bs), 3.75 (**2l**, 3H, s), 3.69 (**2l'**, 3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 46.69, 55.22, 59.88, 107.57, 110.99, 113.71, 114.54, 127.06, 127.97, 128.45, 128.68, 129.59, 133.28, 144.06, 158.05; IR(neat): 696, 726, 1032, 1227, 1501, 1611, 1738, 2926, 3456 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{14}I_2ONa$ $(M+Na)^+$: 510.9026, found: 510.9025.

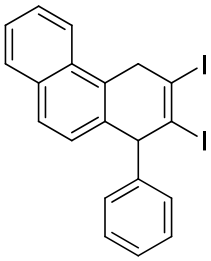
7-Chloro-2,3-diiodo-1-phenyl-1,4-dihydronaphthalene (2m)



2m

White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.24 (3H, m), 7.16-7.12 (3H, m), 7.02 (1H, d, *J* = 8.4 Hz), 6.98 (1H, d, *J* = 2.4 Hz), 5.05 (1H, dd, *J* = 3.6, 3.2 Hz), 4.21 (1H, dd, *J* = 21.2, 3.2 Hz), 4.13 (1H, dd, *J* = 21.2, 3.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 45.85, 60.24, 107.32, 113.45, 127.03, 127.48, 127.99, 128.25, 128.36, 128.92, 130.66, 132.56, 137.69, 143.01; IR(neat): 691, 802, 1216, 1366, 1738, 2924 cm^{-1}; HRMS (EI) calcd for $C_{16}H_{11}I_2$ (m/z): 491.8639, found: 491.8637.

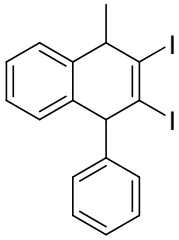
2,3-Diiodo-1-phenyl-1,4-dihydrophenanthrene (2n)



2n

White solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 (1H, d, *J* = 8.4 Hz), 7.82 (1H, d, *J* = 8.4 Hz), 7.66-7.52 (3H, m), 7.35-7.23 (5H, m), 7.10 (1H, d, *J* = 8.4 Hz), 5.24 (1H, dd, *J* = 5.1, 3.6 Hz), 4.75 (1H, dd, *J* = 21.2, 3.6 Hz), 4.51 (1H, dd, *J* = 21.2, 5.1 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 44.14, 60.62, 107.82, 114.49, 122.63, 125.82, 126.51, 126.69, 127.30, 127.59, 128.49, 128.53, 128.71(x2), 130.15, 132.02, 133.08, 143.74; IR(neat): 697, 744, 764, 807, 1216, 1738, 3022 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{14}I_2Na$ $(M+Na)^+$: 530.9077, found: 530.9076.

2,3-Diiodo-1-methyl-4-phenyl-1,4-dihydronaphthalene (2o)

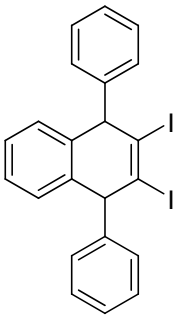


2o (d.r. = 2.3:1.0)

d.r. = 2.3:1.0; Slight yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34-6.74 (9H, m), 5.21 (minor isomer: 1H, d, *J* = 2.0 Hz), 4.92 (major isomer: 1H, d, *J* = 3.5 Hz), 4.14-4.09 (mixture of two

isomers: 1H, m), 1.71 (minor isomer: 3H, d, *J* = 7.0 Hz), 1.64 (major isomer: 1H, d, *J* = 7.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.21, 27.26, 48.84, 49.81, 58.35, 60.92, 114.27, 116.58, 118.15, 118.34, 126.60, 126.70, 126.77, 127.07, 127.13, 127.18(x2), 127.25, 127.87, 128.28, 128.48, 128.67, 129.08, 129.97, 134.76, 135.34, 136.30, 136.69, 142.70, 146.70 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{14}I_2Na$ $(M+Na)^+$: 494.9077, found: 494.9077.

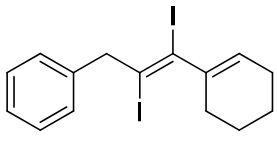
2,3-Diiodo-1,4-diphenyl-1,4-dihydronaphthalene (2p)



2p (d.r. = 6.0:1.0)

d.r. = 6.0:1.0; White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-6.92 (14H, m), 5.35 (2H, s), 5.27 (minor diastereomer: 2H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 59.43, 59.83, 116.47, 116.71, 126.73, 126.79, 127.17, 127.26, 128.47, 128.60, 128.62, 128.72, 129.09, 129.23, 134.56, 134.66, 143.55, 145.67; IR(neat): 638, 695, 748, 1216, 1366, 1596, 1738, 3019 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{16}I_2Na$ $(M+Na)^+$: 556.9234, found: 556.9234.

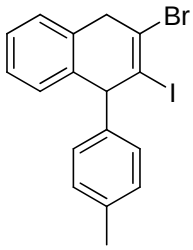
(*E*)-(3-Cyclohexenyl-2,3-diiodoallyl)benzene (4q)



4q

White solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38-7.27 (5H, m), 5.81-5.78 (1H, m), 4,14 (2H, s), 2.19-2.17 (2H, m), 2.12-2.06 (2H, m), 1.80-1.73 (2H, m), 1.66-1.59 (2H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.63, 22.23, 25.08, 26.46, 55.13, 100.1, 101.87, 126.92, 128.51, 128.71, 129.14, 137.15, 144.95; IR(neat): 696, 740, 917, 1451, 1492, 1738, 2925 cm^{-1}; HRMS (EI) calcd for $C_{15}H_{16}I_2$ (m/z): 449.9341, found: 449.9340.

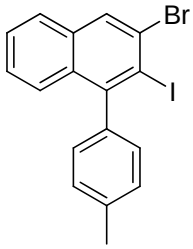
3-Bromo-2-iodo-1-p-tolyl-1,4-dihydronaphthalene (2b')



2b'

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19-7.07 (7H, m), 7.03-7.01 (1H, m), 5.04 (1H, dd, *J* = 4.0, 3.2 Hz), 4.16 (1H, dd, *J* = 21.2, 4.0 Hz), 4.08 (1H, dd, *J* = 21.2, 3.2 Hz), 2.32 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.19, 41.39, 60.25, 105.78, 126.54, 126.98, 127.22, 127.68, 127.90, 128.31, 129.37, 131.34, 136.04, 136.78, 140.76; IR(neat): 733, 906, 1111, 1509, 1619, 1738, 3021 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{14}BrIONa$ $(M+Na)^+$: 446.9216, found: 446.9214.

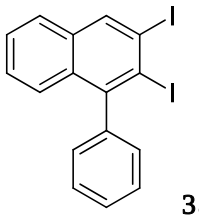
3-Bromo-2-iodo-1-p-tolylnaphthalene (3b')



3b'

White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (1H, s), 7.75 (1H, d, *J* = 8.0 Hz), 7.51-4.47 (1H, m), 7.36-7.33 (4H, m), 7.12-7.09 (2H, d, *J* = 8.0 Hz), 2.50 (3H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.51, 105.77, 126.64, 126.81, 126.85, 127.00, 127.65, 129.14, 129.34, 130.49, 131.45, 133.67, 137.68, 141.64, 147.85 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{12}BrIONa$ $(M+Na)^+$: 444.9059, found: 444.9059.

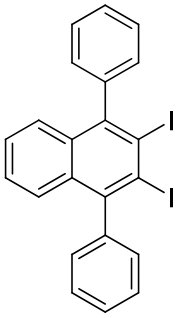
2,3-Diiodo-1-phenylnaphthalene (3a)



3a

White solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.59 (1H, s), 7.78 (1H, d, *J* = 8.1 Hz), 7.62-7.51 (4H, m), 7.42-7.32 (2H, m), 7.27-7.24 (2H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 105.38, 110.97, 126.78, 126.86, 127.22, 127.65, 128.00, 128.50, 129.46, 131.68, 134.15, 138.28, 145.59, 146.97 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{10}I_2Na$ $(M+Na)^+$: 478.8764, found: 478.8763.

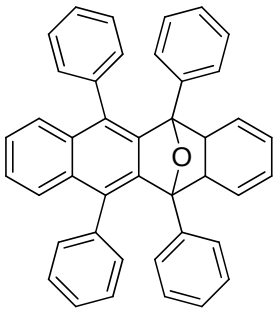
2,3-Diiodo-1,4-diphenylnaphthalene (3p)



3p

White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.49 (6H, m), 7.37-7.26 (8H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 112.63, 126.81, 127.77, 127.90, 128.48, 129.48, 132.26, 146.29, 146.57; IR(neat): 697, 719, 838, 1599, 1738, 3019 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{14}I_2ONa$ $(M+Na)^+$: 554.9077, found: 554.9078.

(6p)

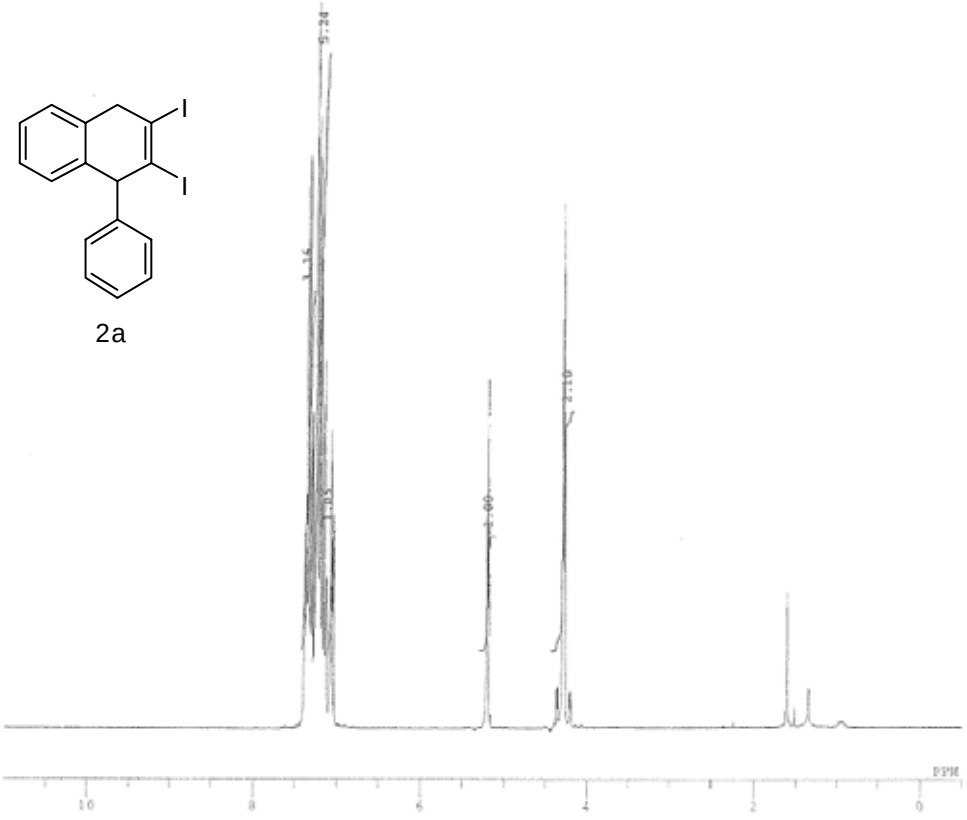


6p

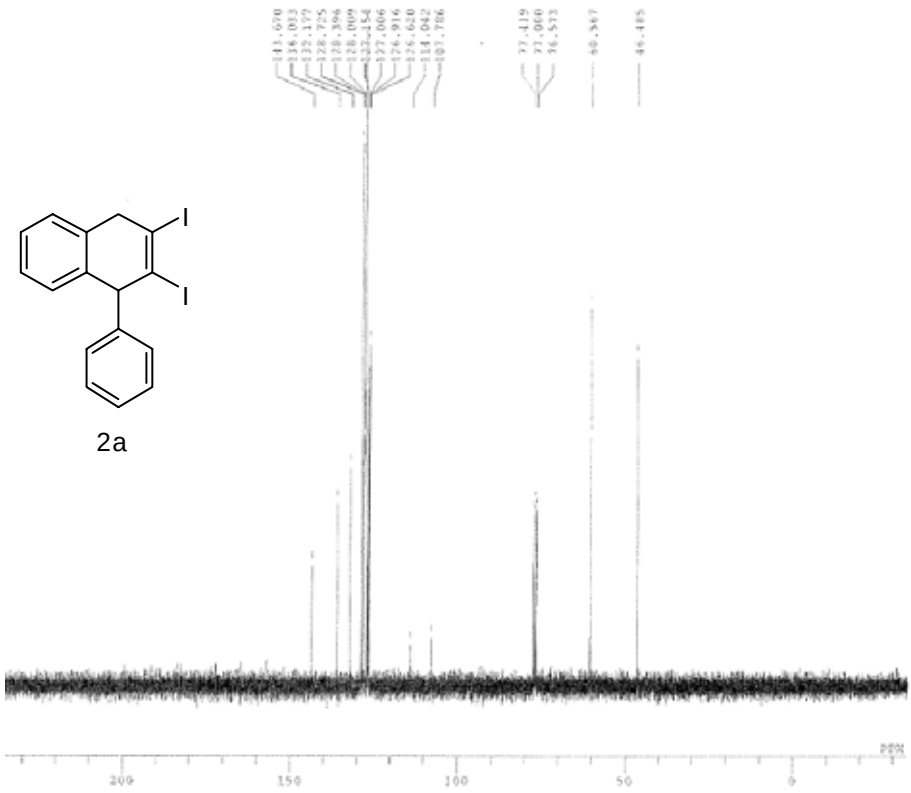
White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79-7.77 (2H, m), 7.61-7.56 (6H, m), 7.32-6.97 (20H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 90.88, 122.54, 125.88, 126.10, 126.30, 126.92, 127.45, 127.54, 127.80, 128.59, 129.80, 131.04, 131.96, 132.04, 134.56, 136.89, 144.62, 149.91; IR(neat): 697, 719, 838, 1599, 1738, 3019 cm^{-1}; HRMS (ESI) calcd for $C_{42}H_{28}ONa$ $(M+Na)^+$: 571.2032, found: 571.2030.

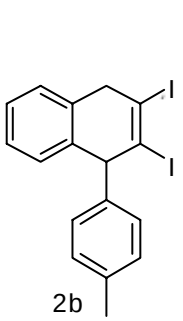
NMR spectra

2a

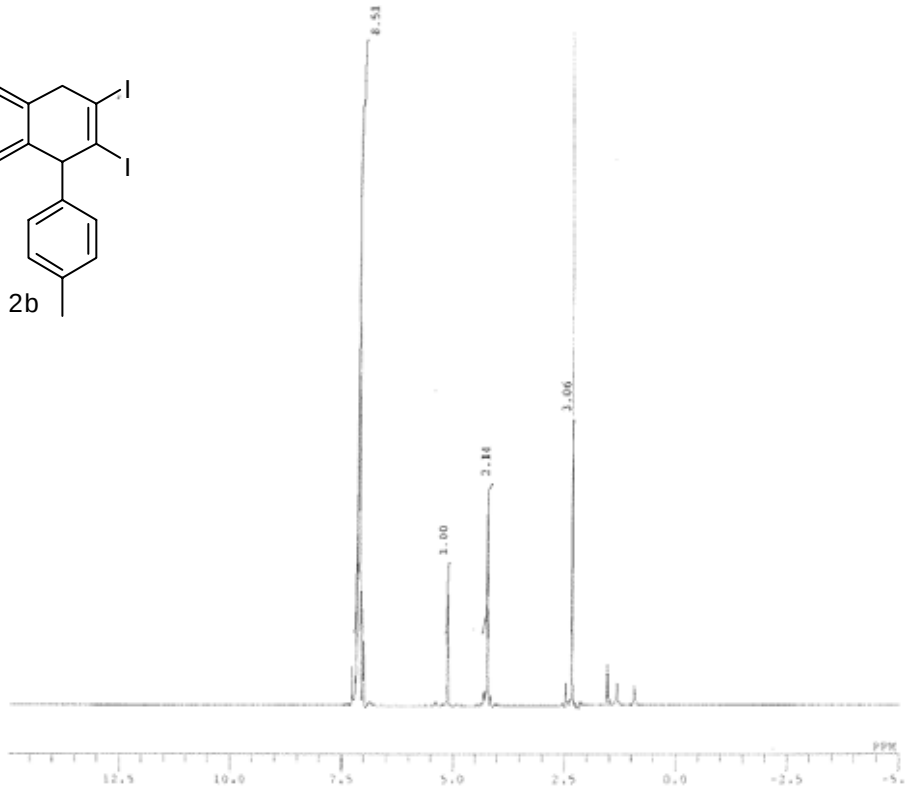


2a




I
I

2b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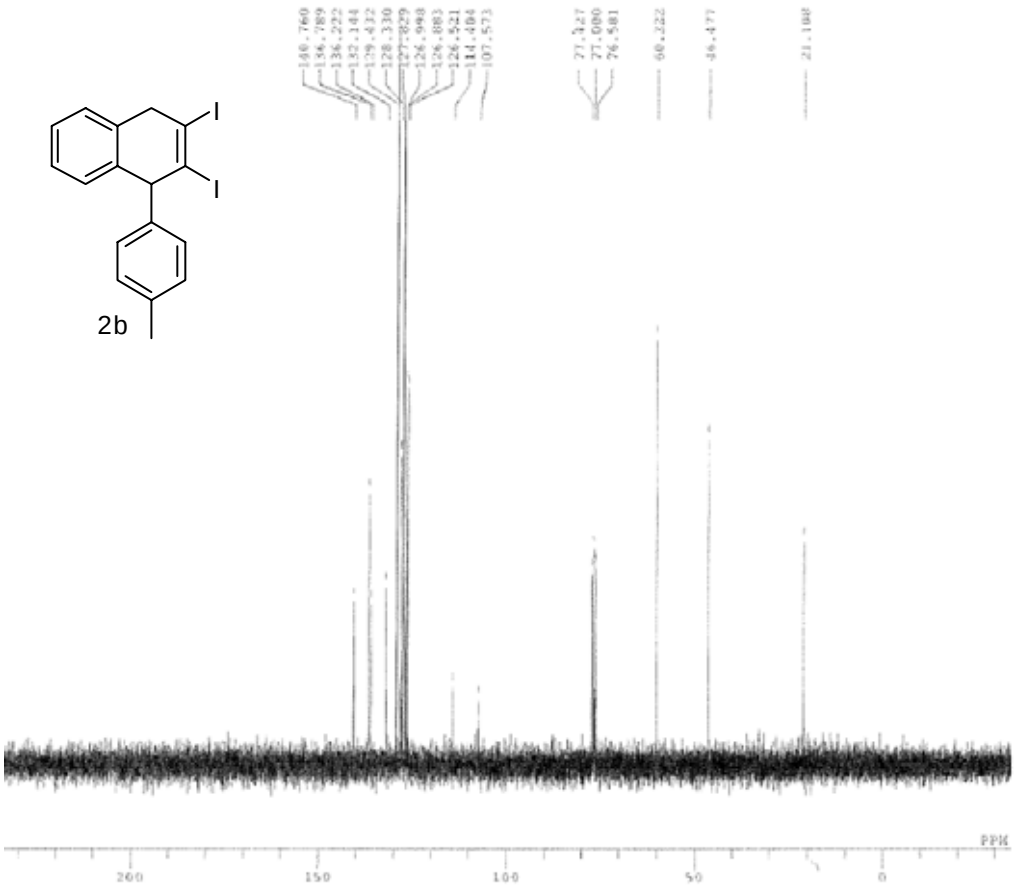
1.00
2.18
3.06
PPM
12.5
10.0
7.5
5.0
2.5
0.0
-2.5

I
I

2b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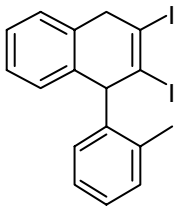
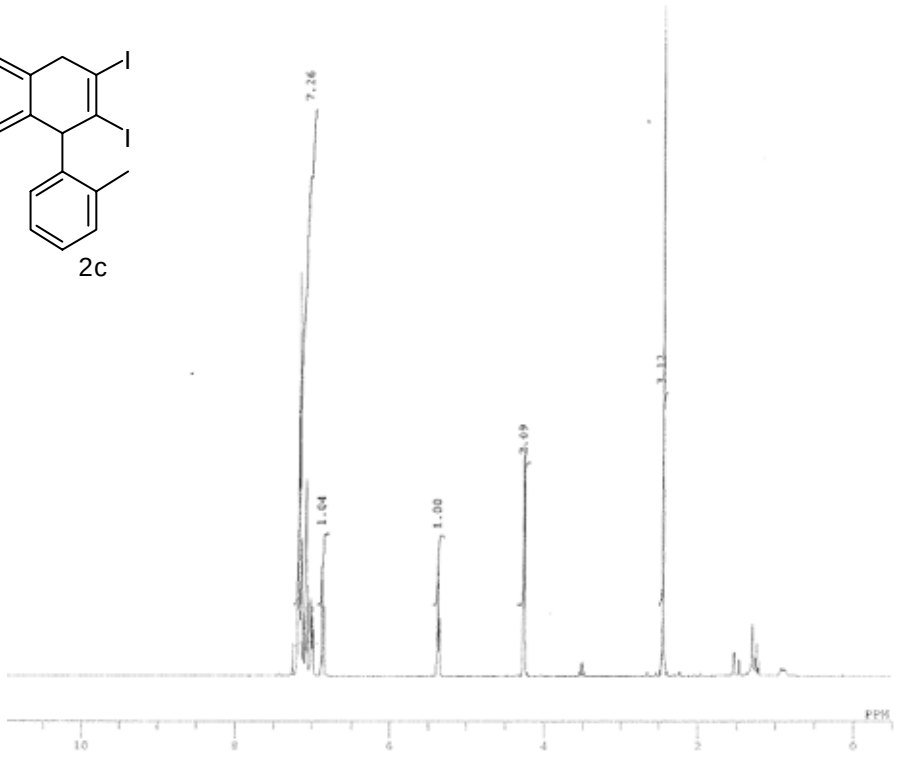
136.789
136.222
132.144
129.432
128.330
127.829
126.998
126.883
126.521
114.404
107.573
77.327
77.000
76.581
60.222
36.477
21.180
PPM
200
150
100
50
0

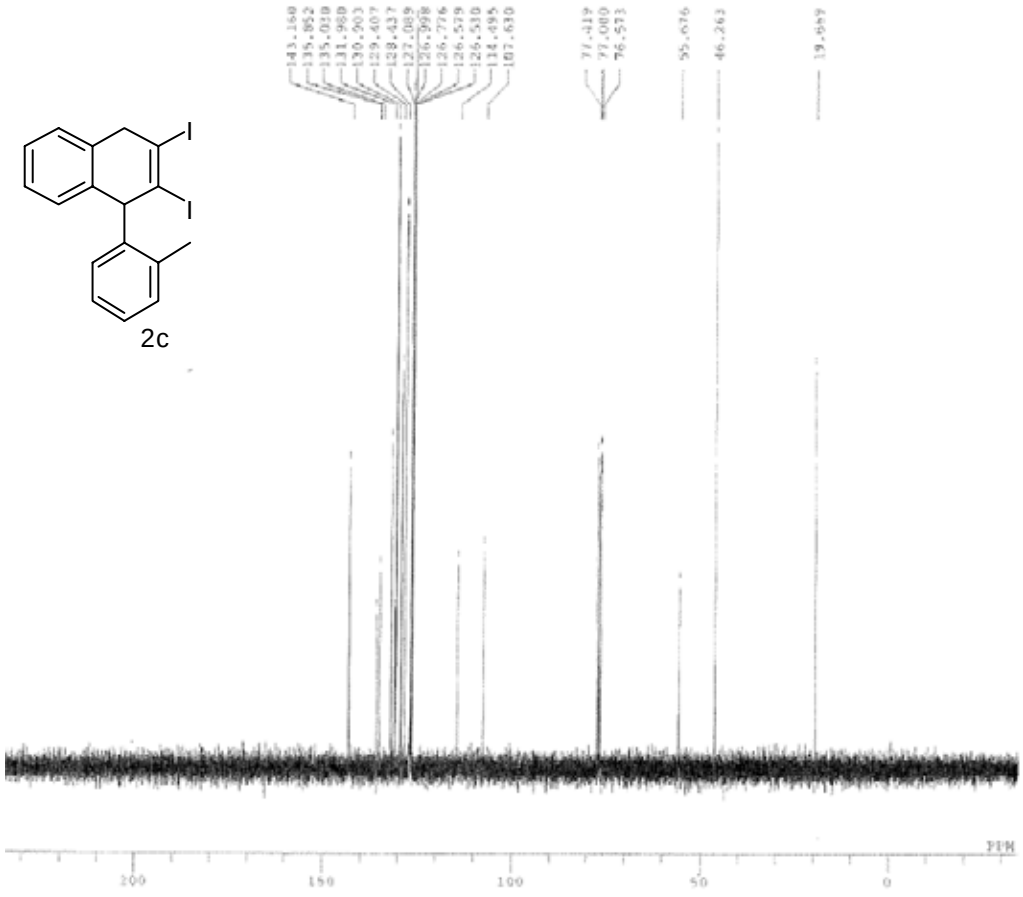

I
I

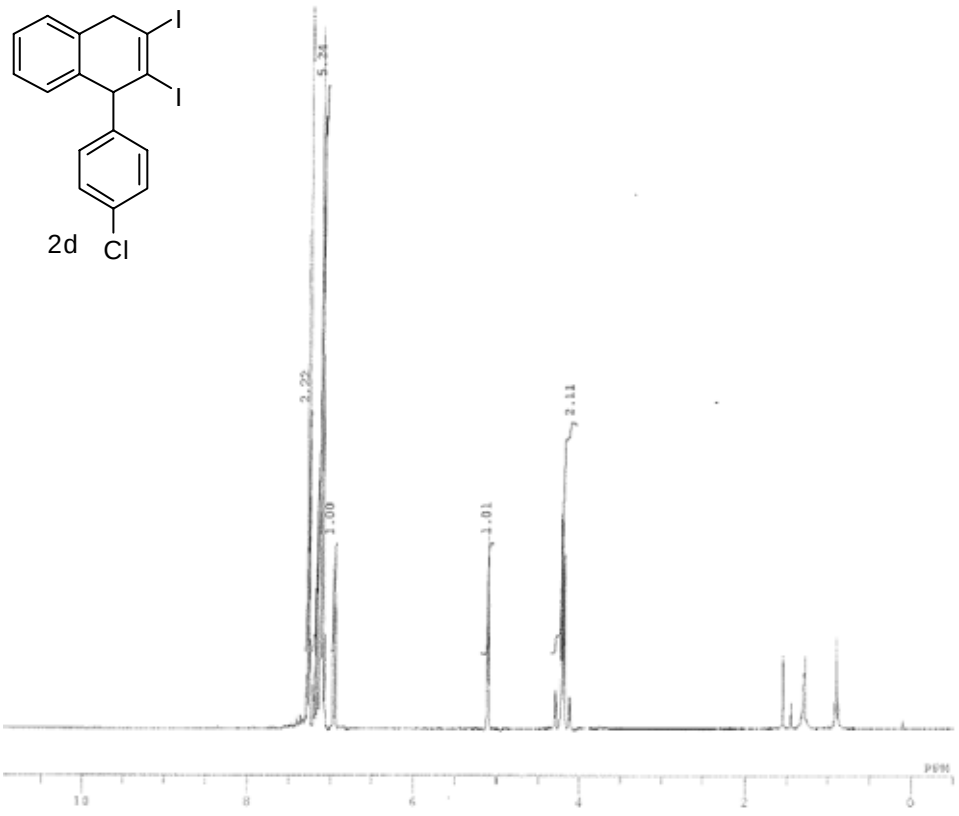
2c

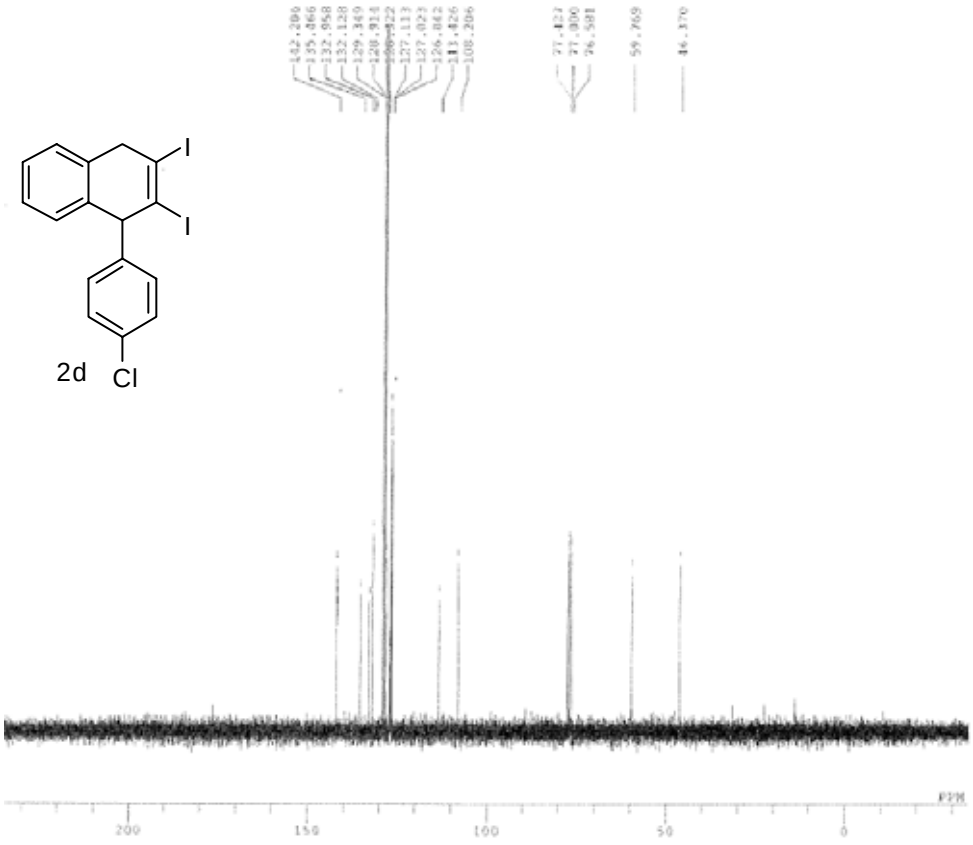

7.26
1.64
1.00
2.69
3.12
PPM
10
8
6
4
2
0

I
I

2c


143.160
135.852
135.038
131.968
130.903
129.607
128.437
127.089
126.998
126.776
126.529
126.530
114.495
107.630
77.417
77.000
76.583
55.676
46.263
19.669
PPM
200
150
100
50
0


I
I
2d
Cl
5.24
2.22
2.11
1.01
1.00
PPM
10
8
6
4
2
0


I
I
2d
Cl
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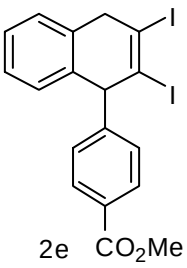
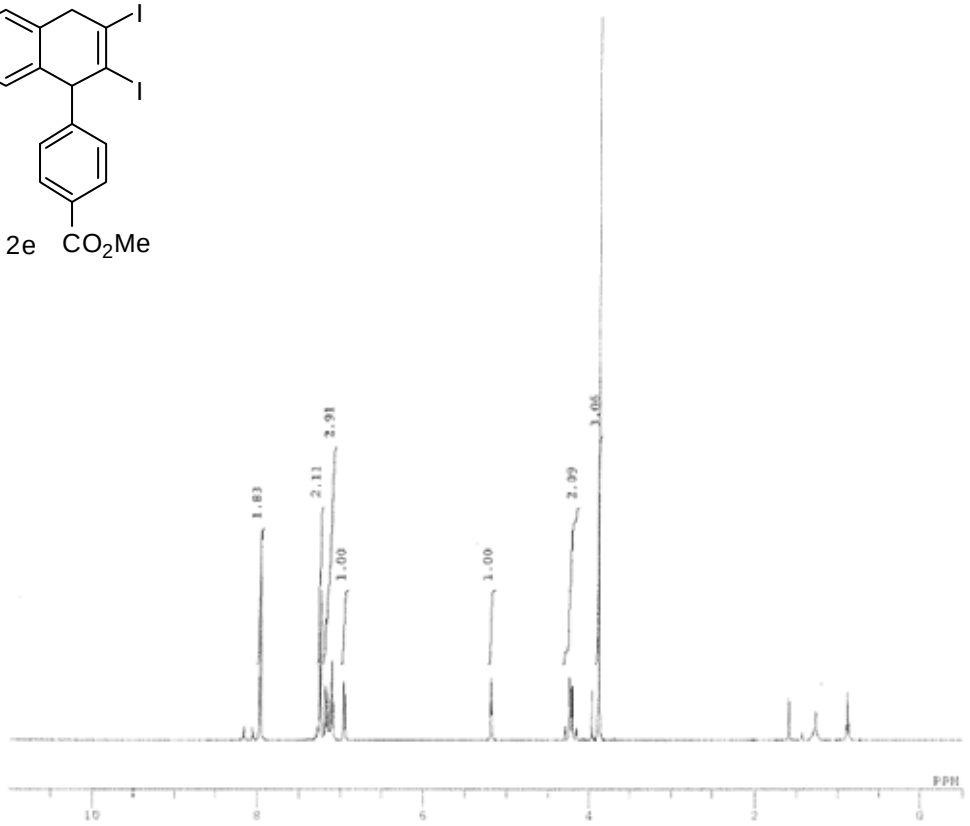
132.958
132.128
129.349
128.911
128.522
127.113
127.023
126.842
115.426
108.206
77.422
77.000
76.581
59.769
46.370
PPM
200
150
100
50
0


I
I
2e
CO2Me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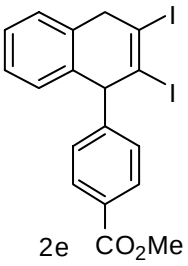
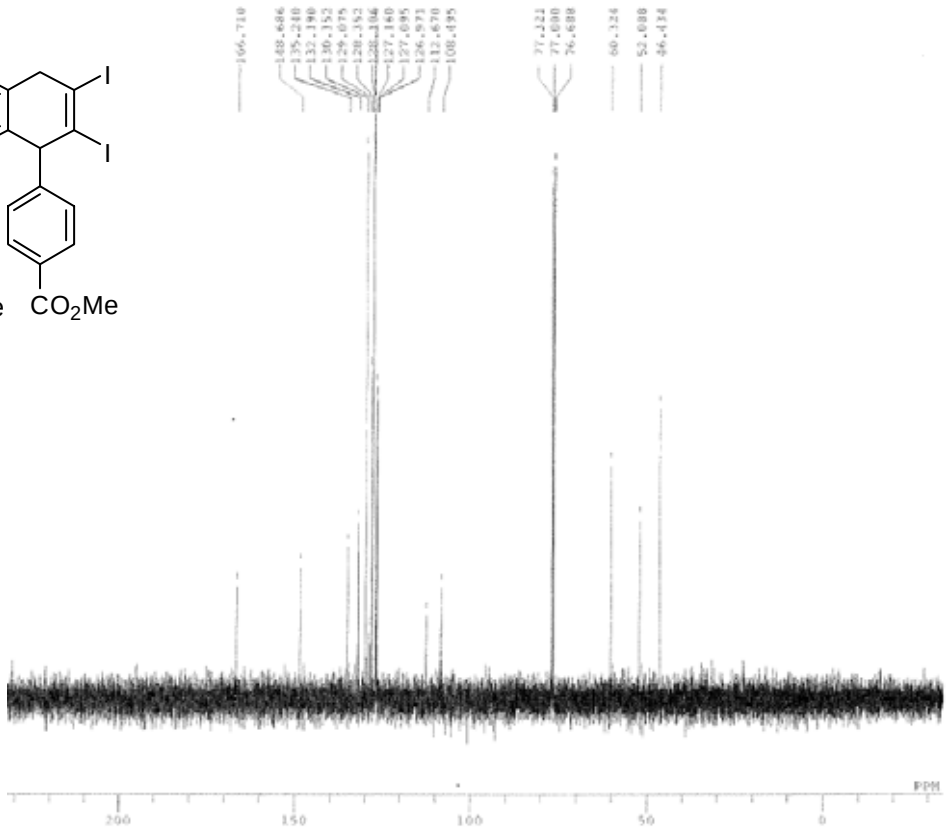
2.91
1.00
1.00
2.99
3.05
PPM
10
8
6
4
2
0


I
I
2e
CO2Me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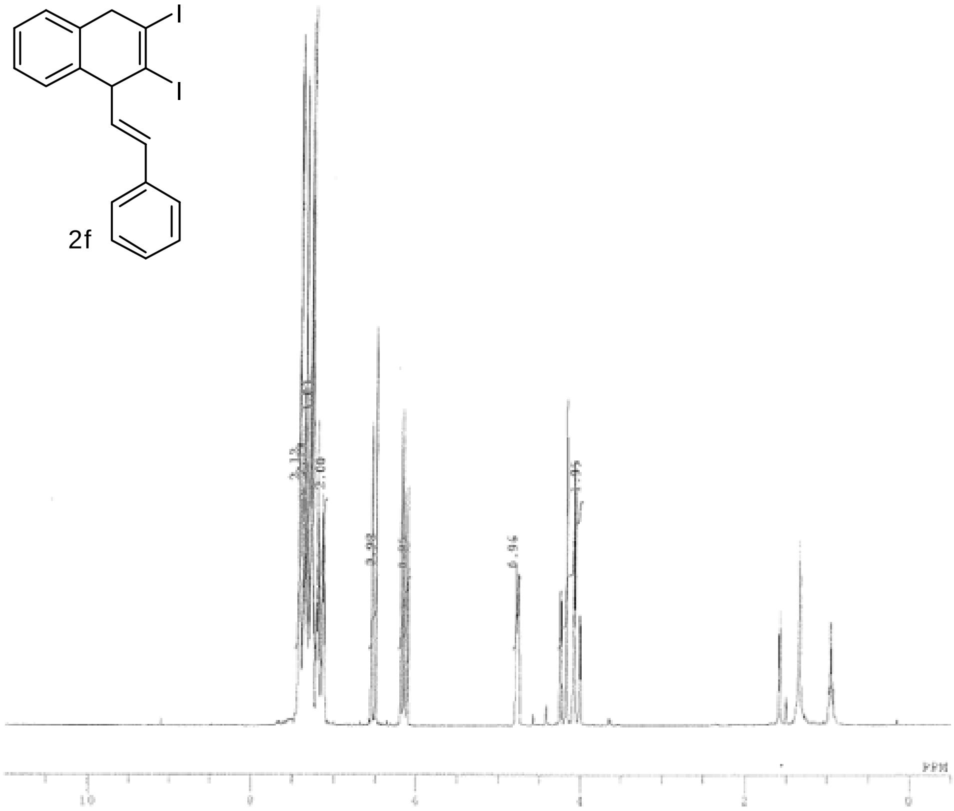
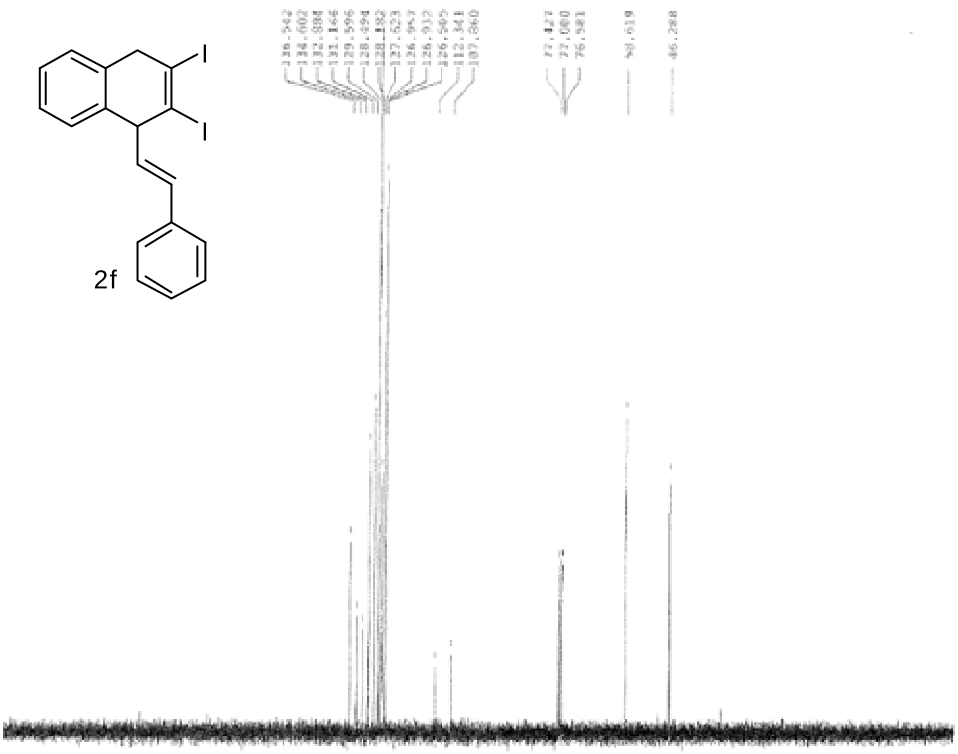
135.240
132.190
130.152
129.675
128.152
127.140
127.695
126.973
112.670
108.495
77.322
77.000
76.688
60.324
52.088
46.434
PPM
200
150
100
50
0


2f
PPM
10
8
6
4
2
0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132.884
131.166
129.596
128.694
128.182
127.623
126.957
126.932
126.505
112.341
107.860
77.427
77.000
76.581
58.619
46.288
PPM
200
150
100
50
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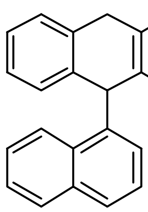
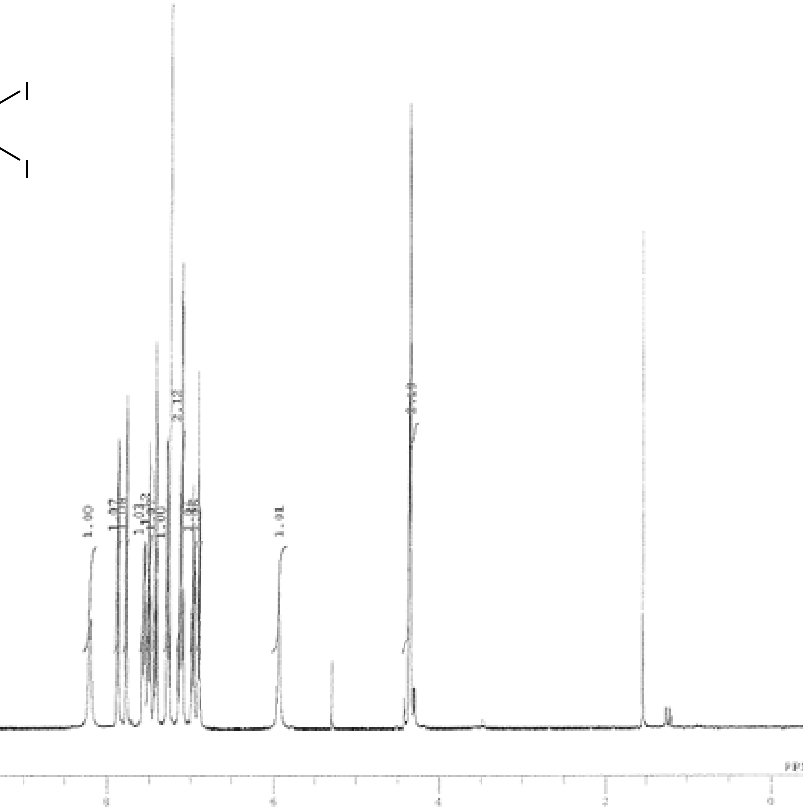
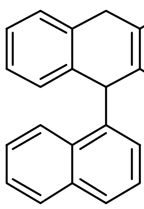
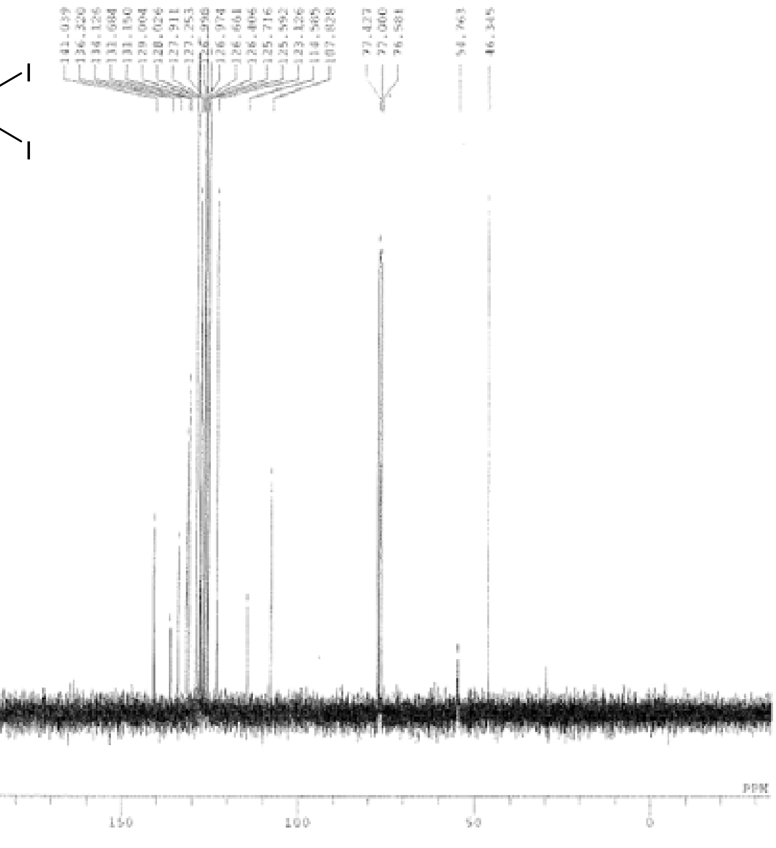


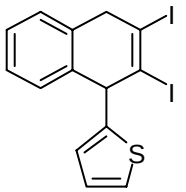
2g



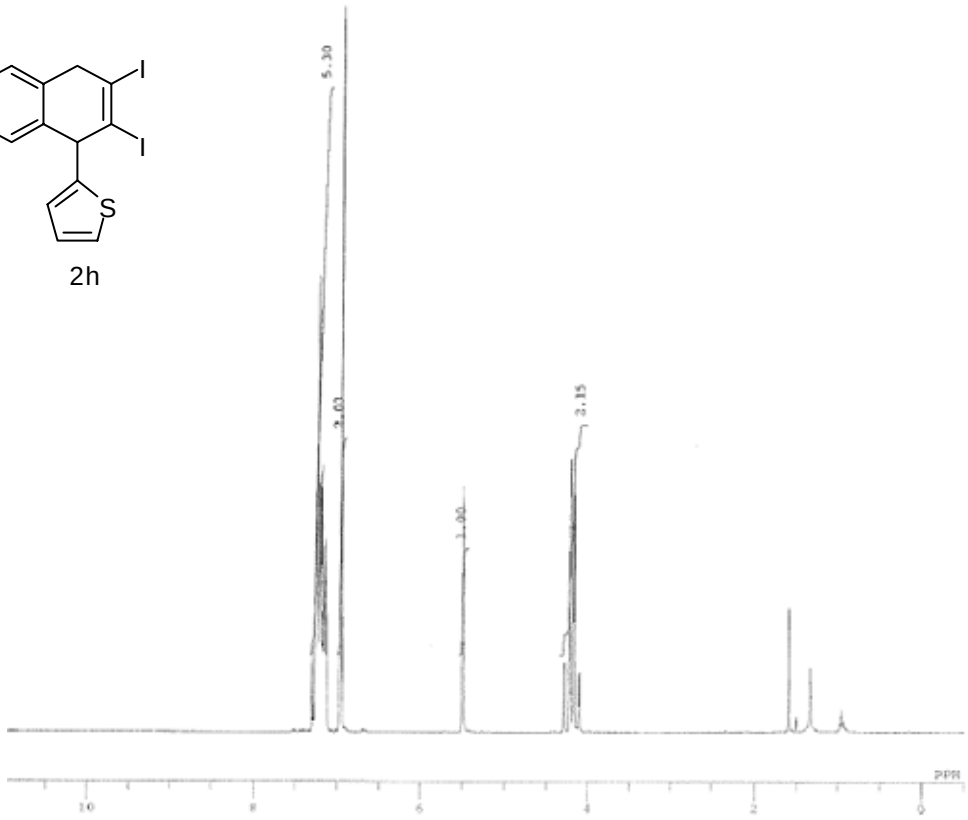


2g



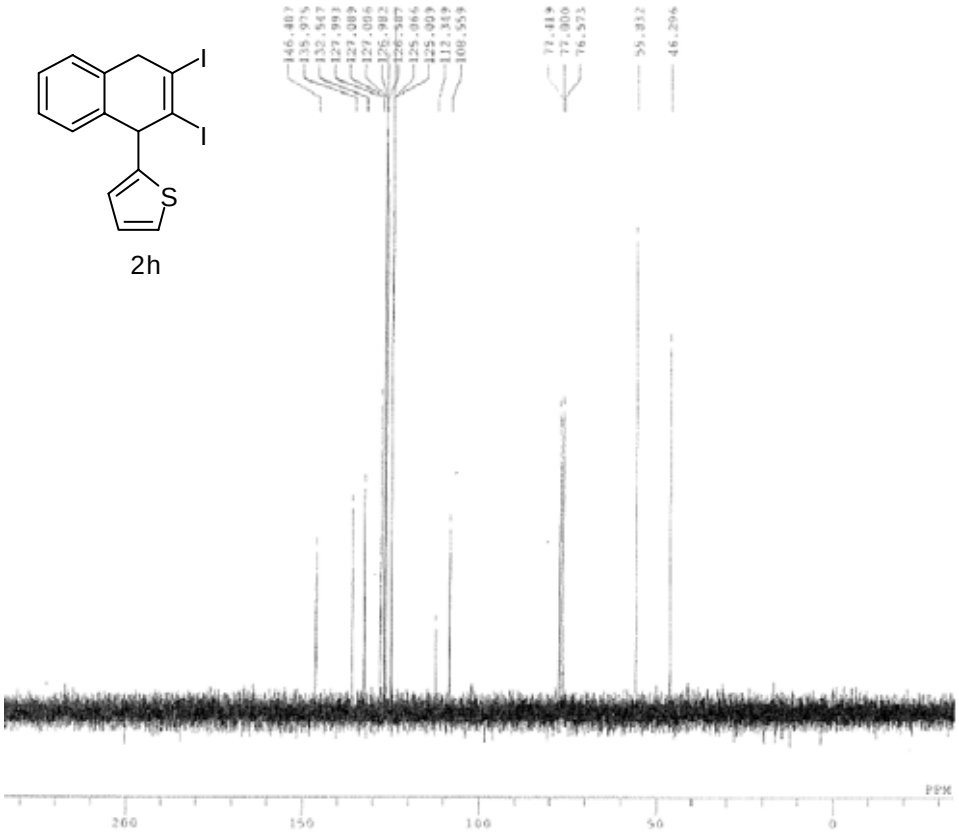

I
I
S

2h


5.30
2.03
1.00
3.15
PPM
10
8
6
4
2
0

I
I
S

2h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127.993
127.089
127.056
126.982
126.587
125.066
125.009
112.349
108.559
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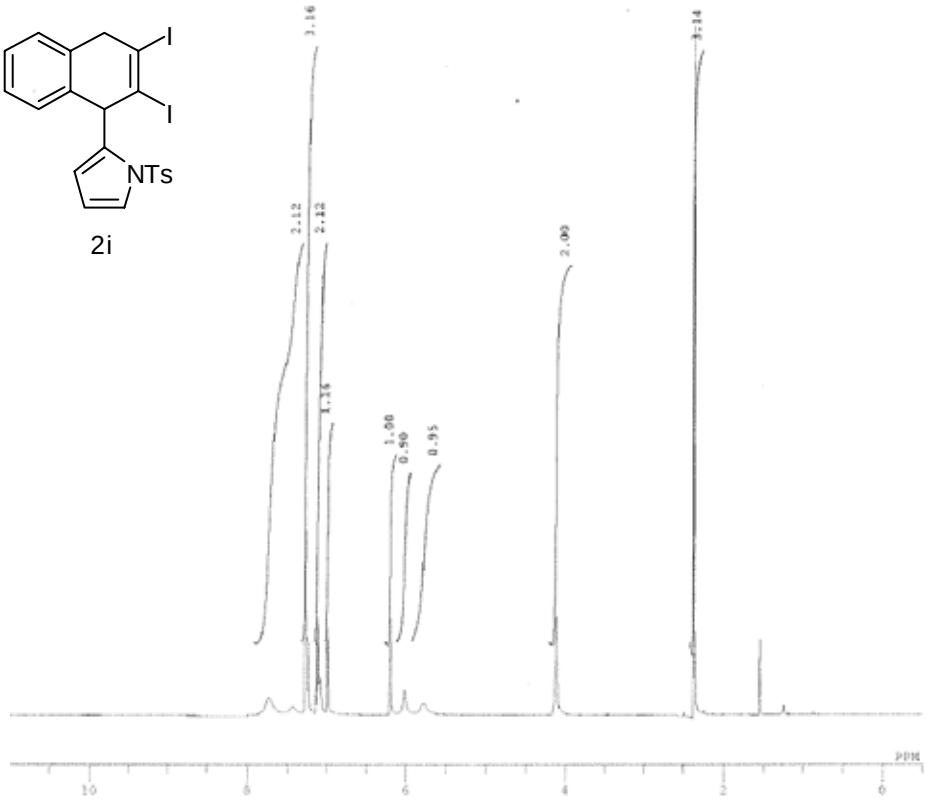
77.000
76.573
55.832
46.296
PPM
200
150
100
50
0

I
I
NTs

2i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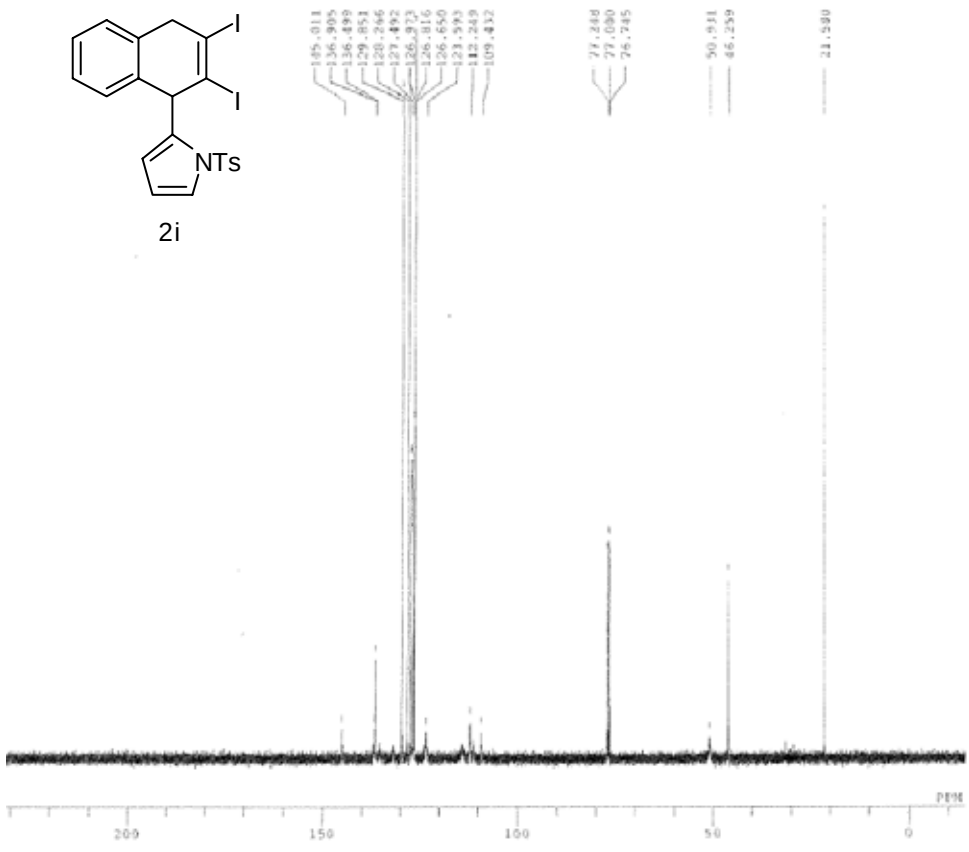
3.14
2.12
2.12
2.00
1.35
1.00
0.50
0.95
PPM
10
8
6
4
2
0

I
I
NTs

2i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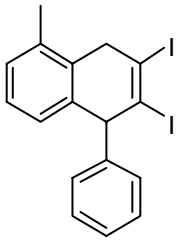
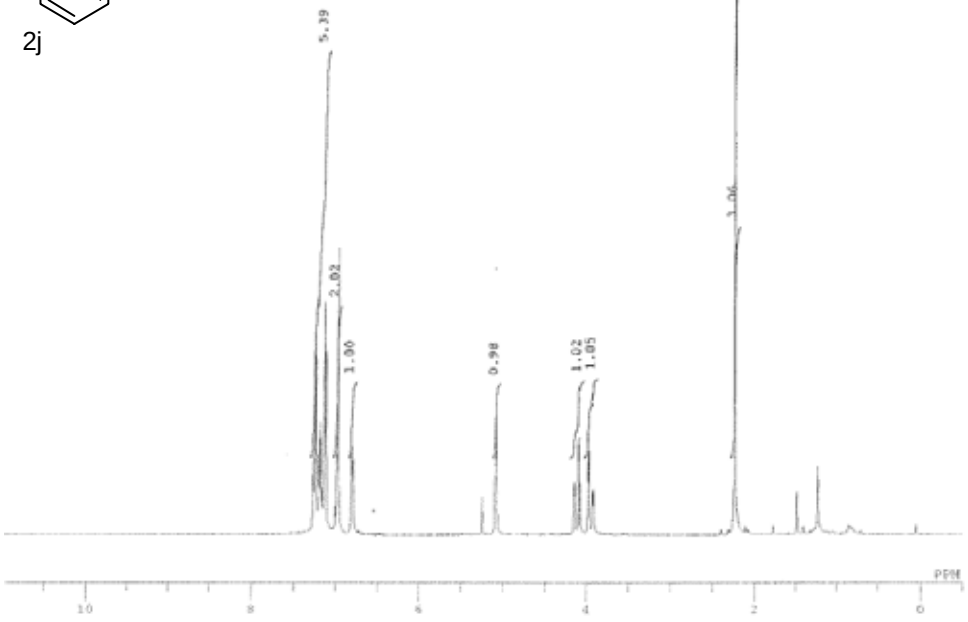
136.499
129.851
128.266
127.492
126.972
126.816
126.650
123.593
122.249
109.432
77.248
77.080
76.745
50.931
46.259
21.590
PPM
200
150
100
50
0

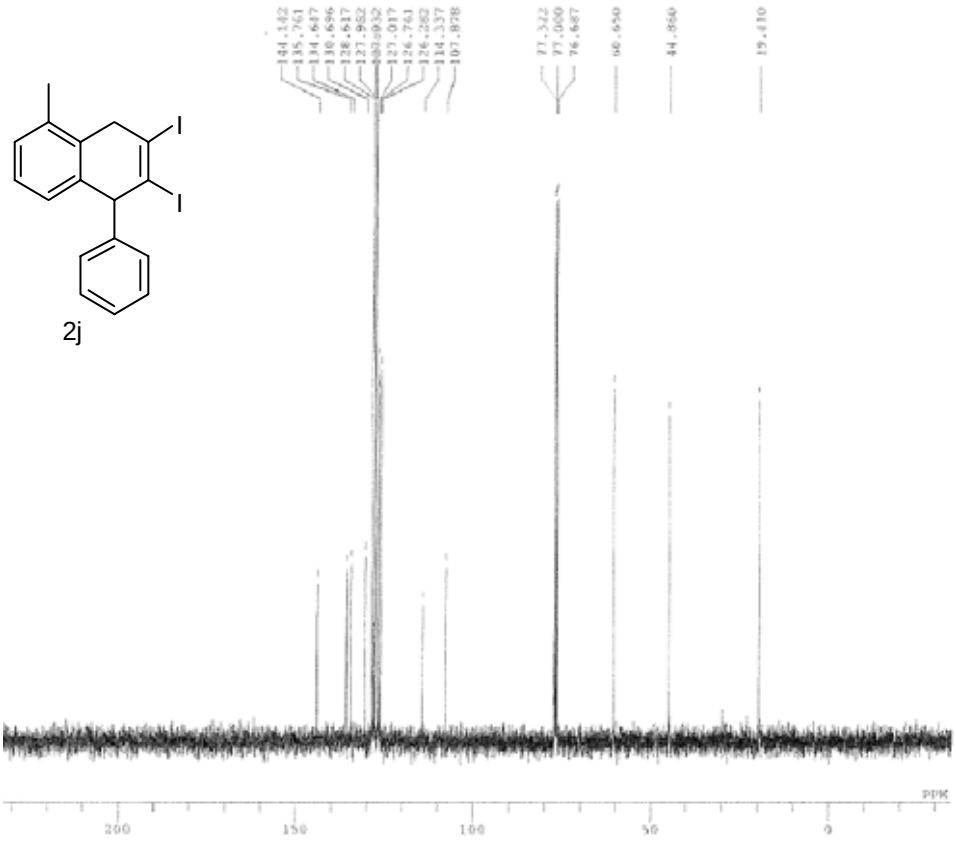

I
I

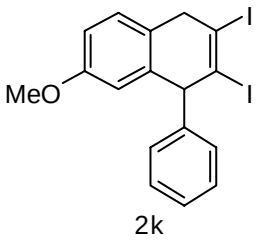
2j


5.39
2.02
1.00
0.98
1.02
1.05
3.06
PPM
10
8
6
4
2
0

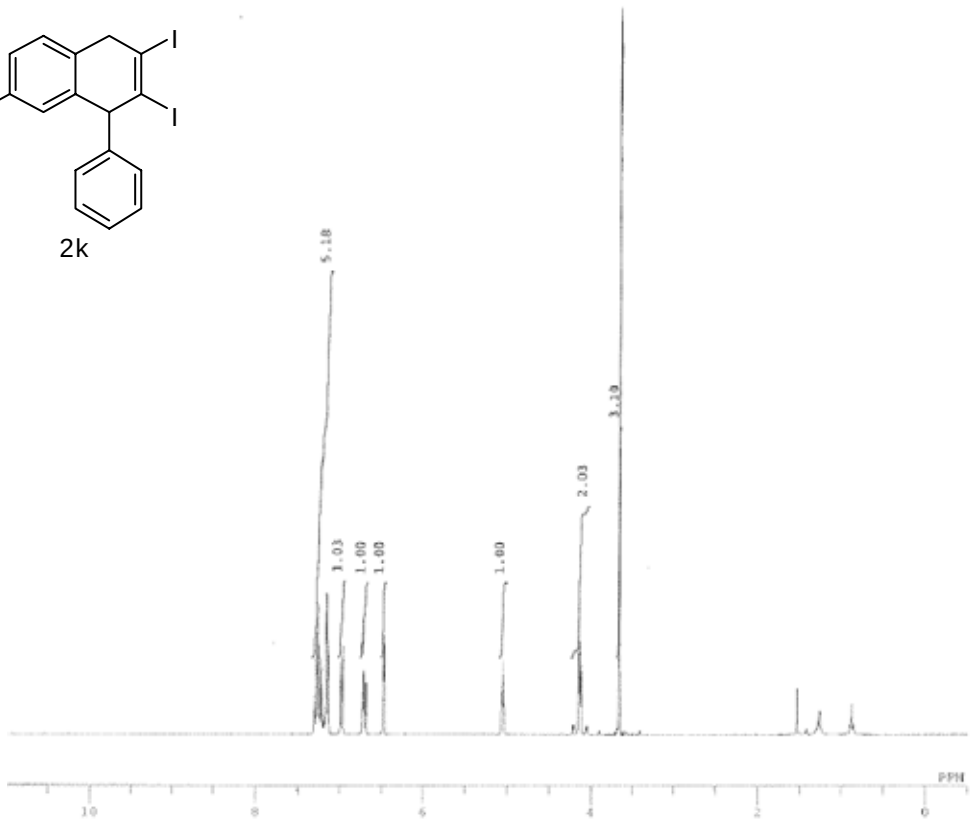
I
I

2j


144.142
135.761
134.647
130.696
128.617
127.952
127.932
127.017
126.761
126.282
114.337
107.878
77.342
77.000
76.687
66.650
44.960
15.410
PPM
200
150
100
50
0


I
MeO
I

2k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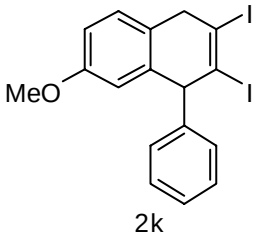
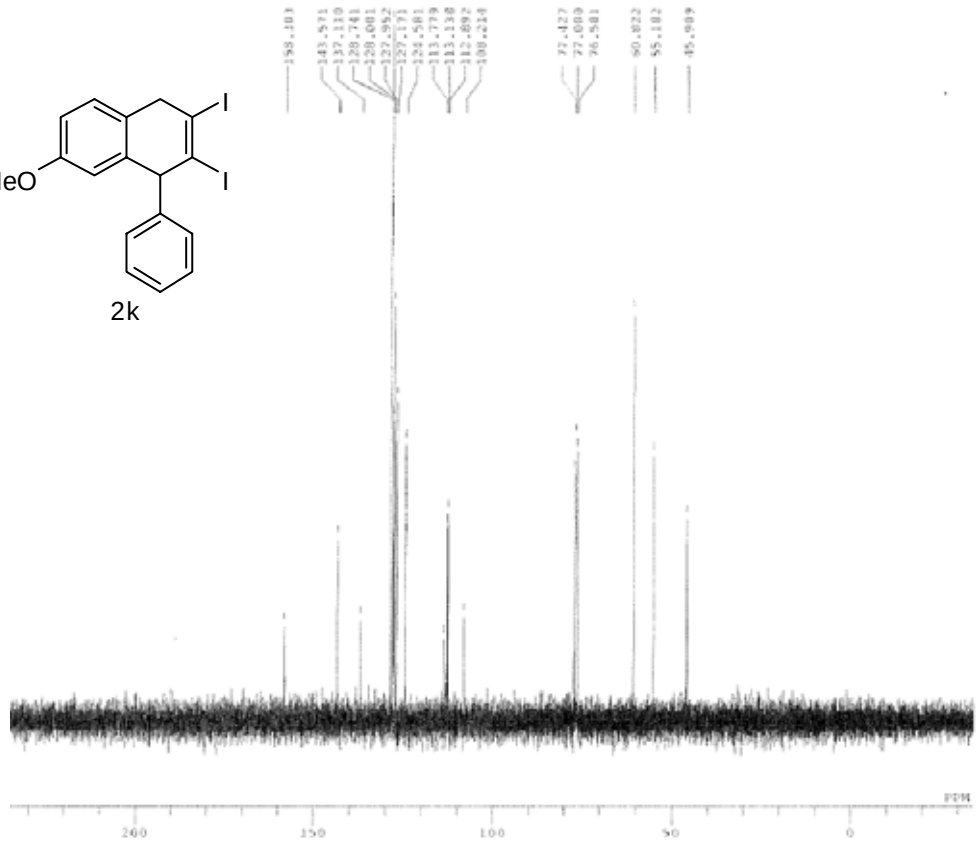
3.03
1.00
1.00
1.00
2.03
3.29
PPM
10
8
6
4
2
0


I
MeO
I

2k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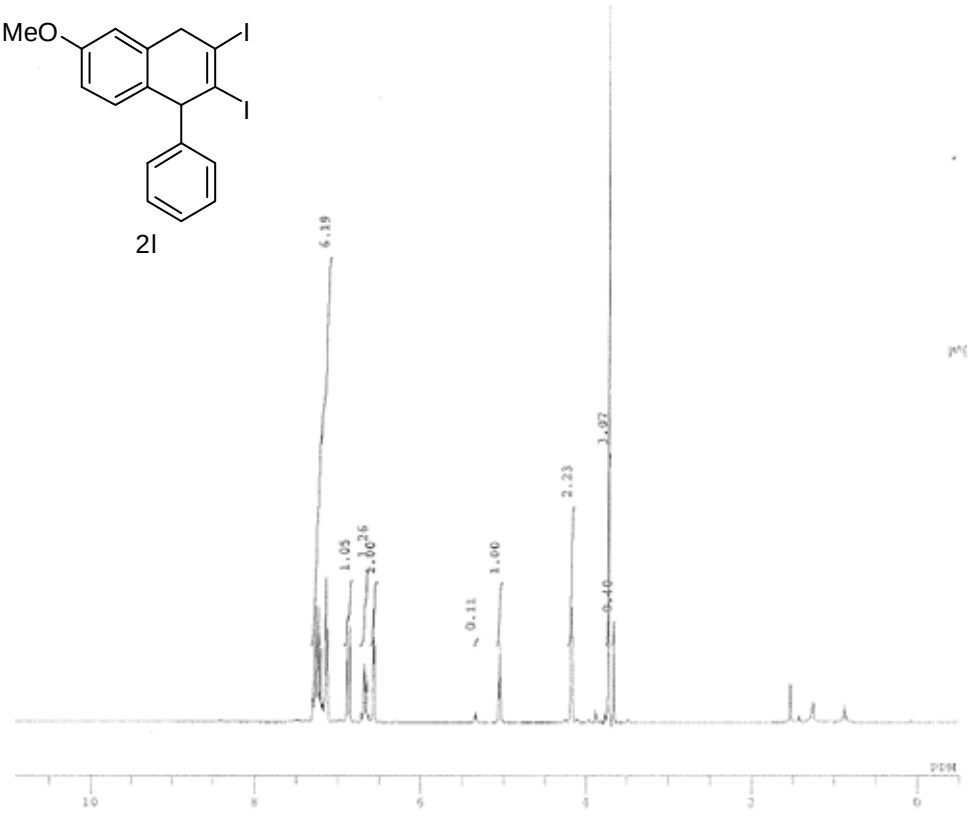
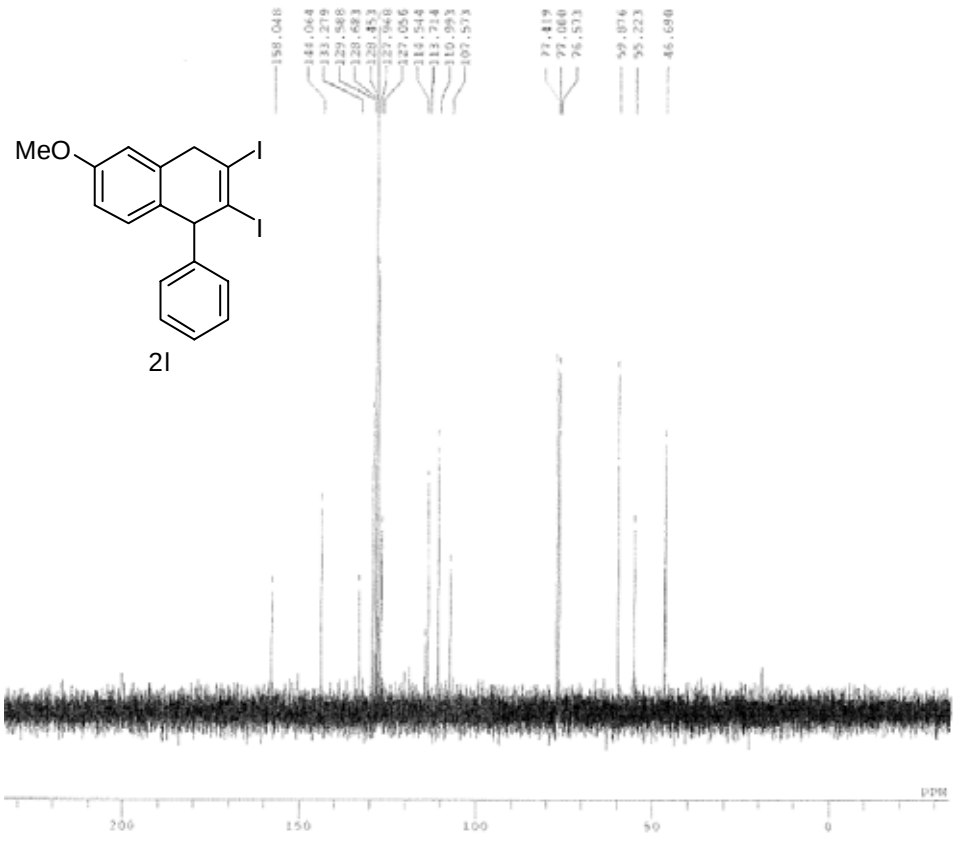
145.571
137.110
129.741
129.001
127.952
127.171
126.581
113.779
113.138
112.892
100.214
77.427
77.000
76.581
60.822
55.182
45.909
PPM
200
150
100
50
0

MeO
I
I

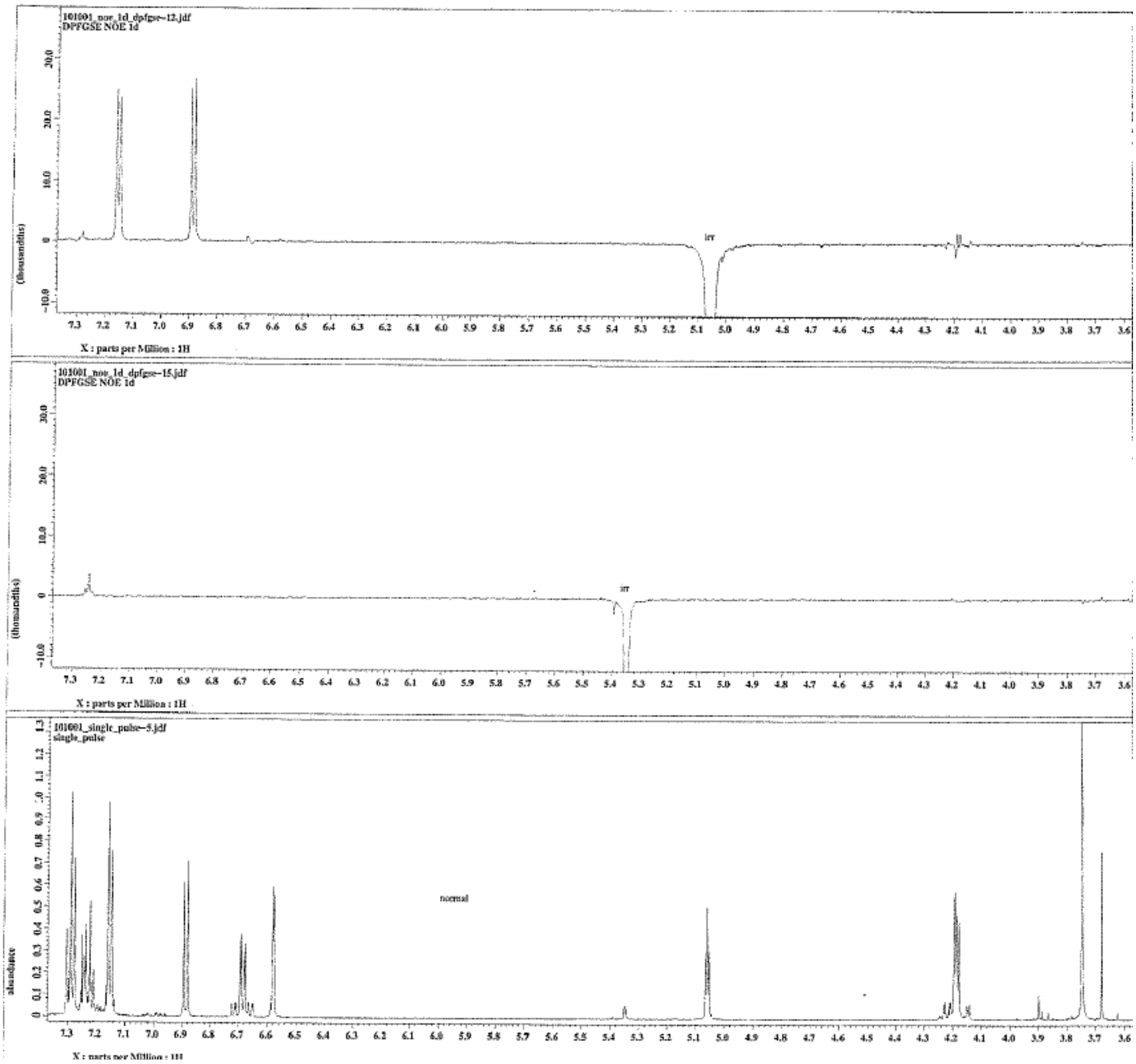
2l

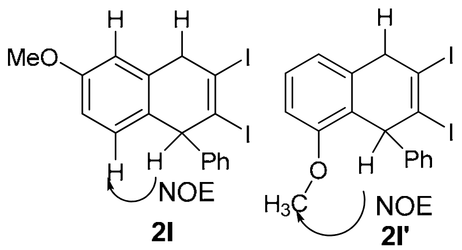

6.19
1.05
2.06
0.11
1.00
2.23
3.07
0.40
PPM
10
8
6
4
2
0

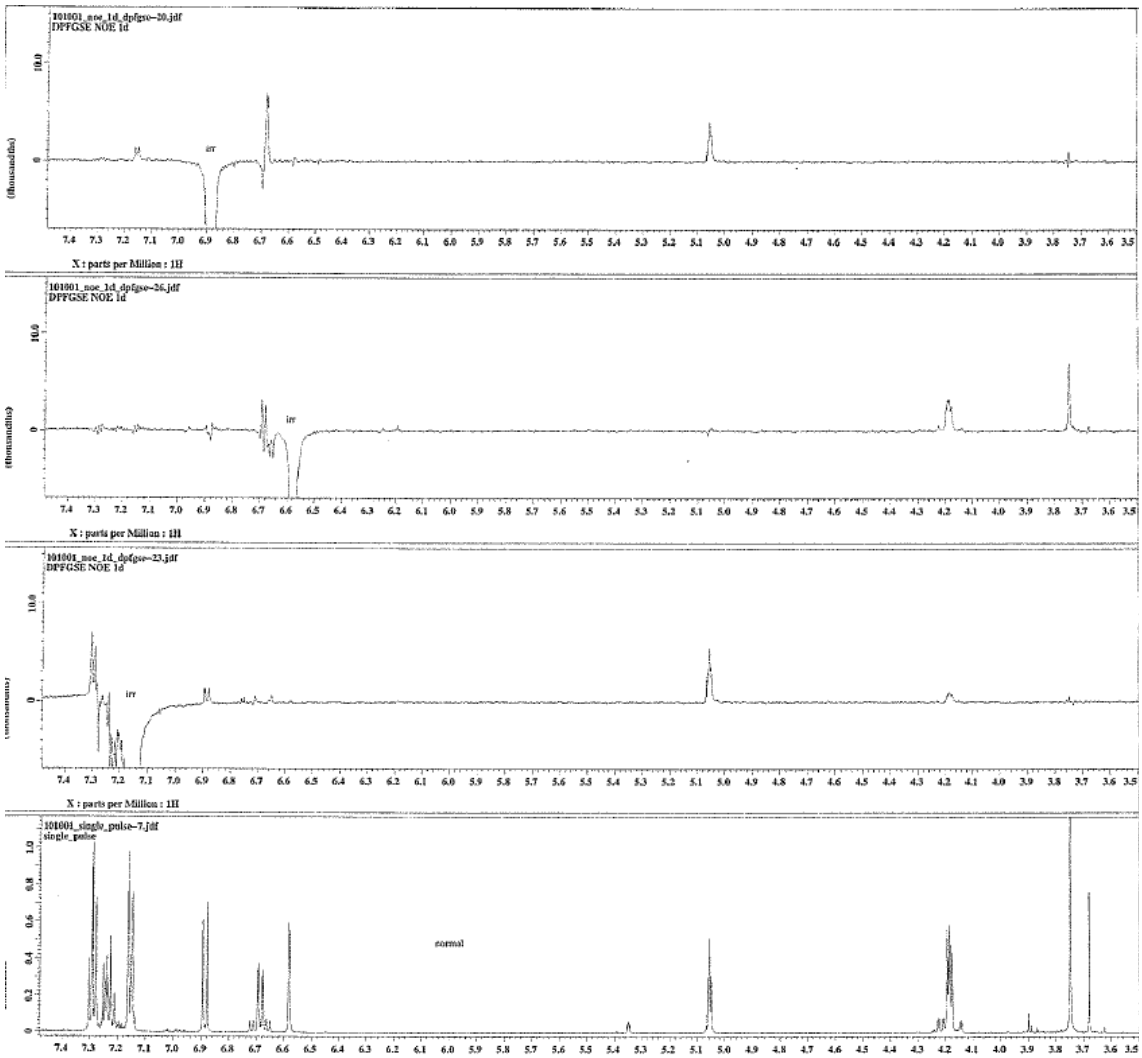

158.048
144.064
133.279
129.588
128.683
128.453
127.946
127.056
118.544
113.714
110.993
107.573
77.419
77.060
76.573
59.876
55.223
46.690
PPM
200
150
100
50
0

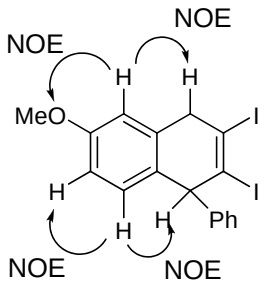
MeO
I
I

2l

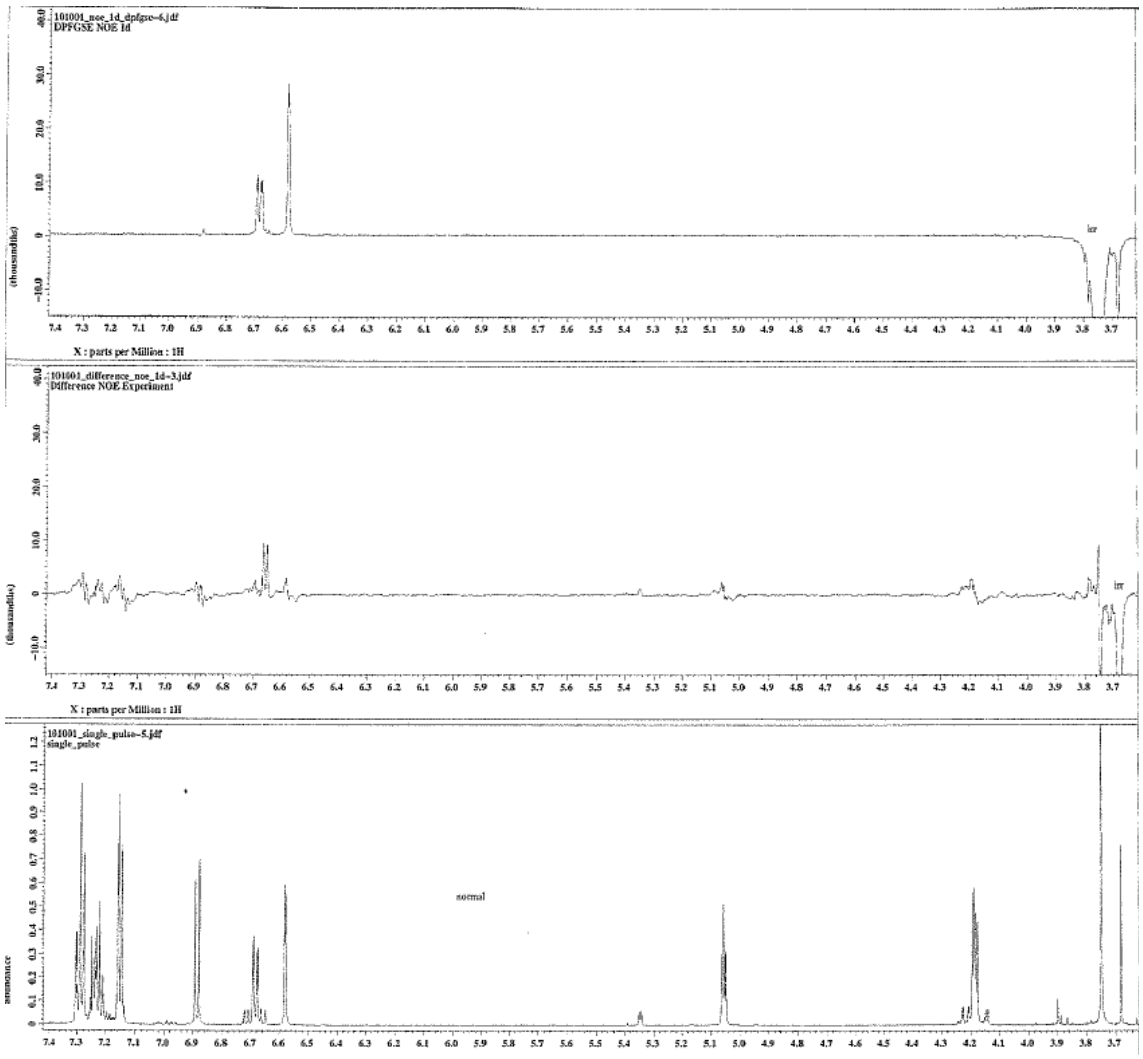

101001_noe_1d_dpfgse-12.jdf
DPFGSE NOE 1d
irr
X : parts per Million : 1H
101001_noe_1d_dpfgse-15.jdf
DPFGSE NOE 1d
irr
X : parts per Million : 1H
101001_single_pulse-5.jdf
single_pulse
normal
abundance

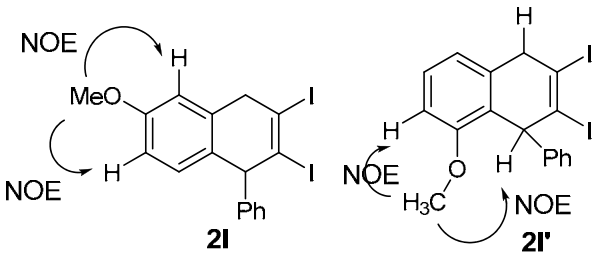

MeO
I
I
Ph
NOE
2l
O
H_3C
NOE
2l'

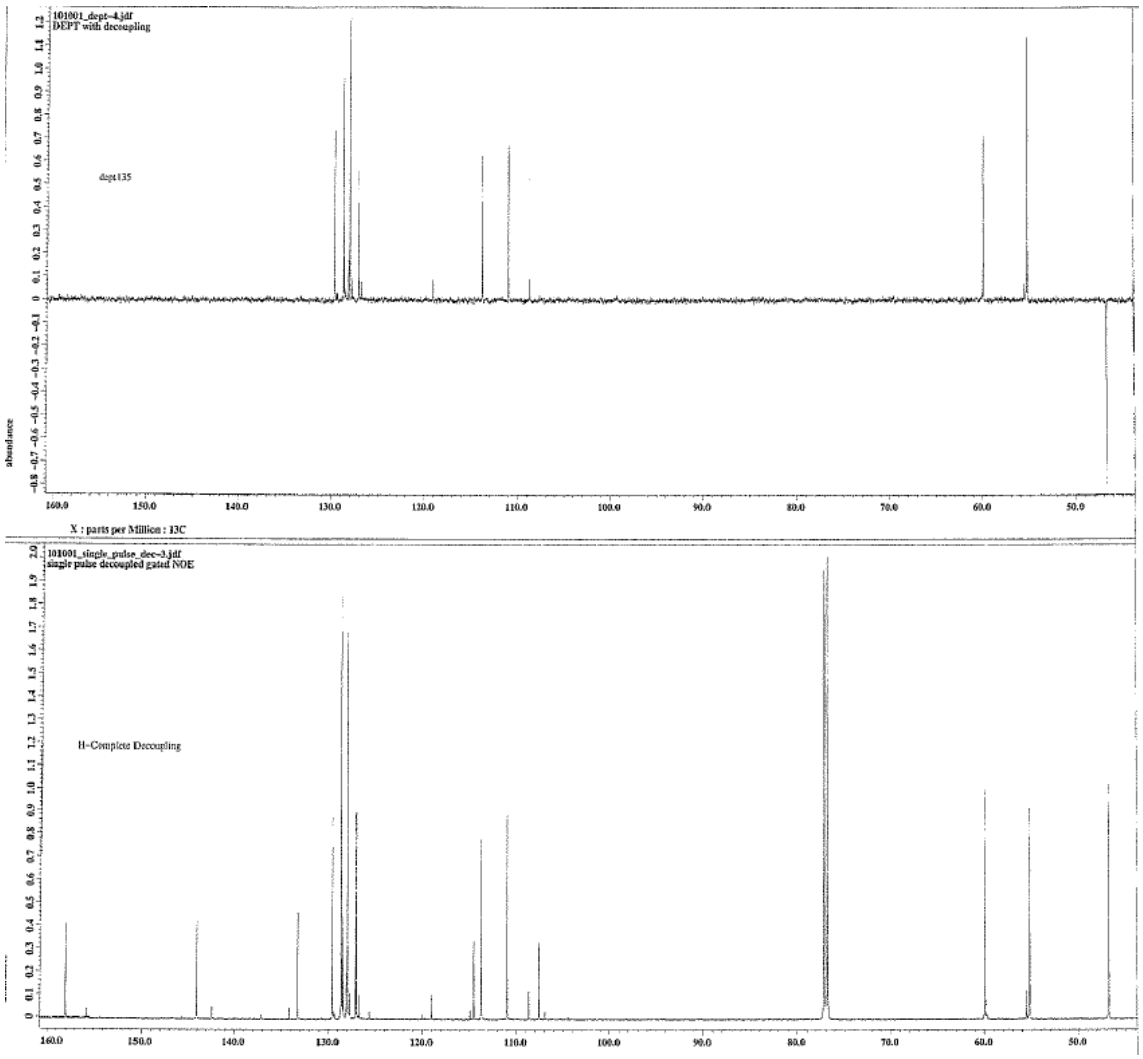

101001_noe_1d_dpfgse-20.jdf
DPFGSE NOE 1d
irr
X : parts per Million : 1H
101001_noe_1d_dpfgse-26.jdf
DPFGSE NOE 1d
irr
X : parts per Million : 1H
101001_noe_1d_dpfgse-23.jdf
DPFGSE NOE 1d
irr
X : parts per Million : 1H
101001_single_pulse-7.jdf
single_pulse
normal

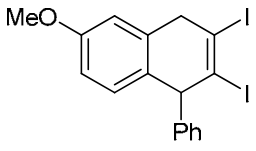

NOE
NOE
H
H
MeO
I
H
I
H
H
Ph
NOE
NOE

2l

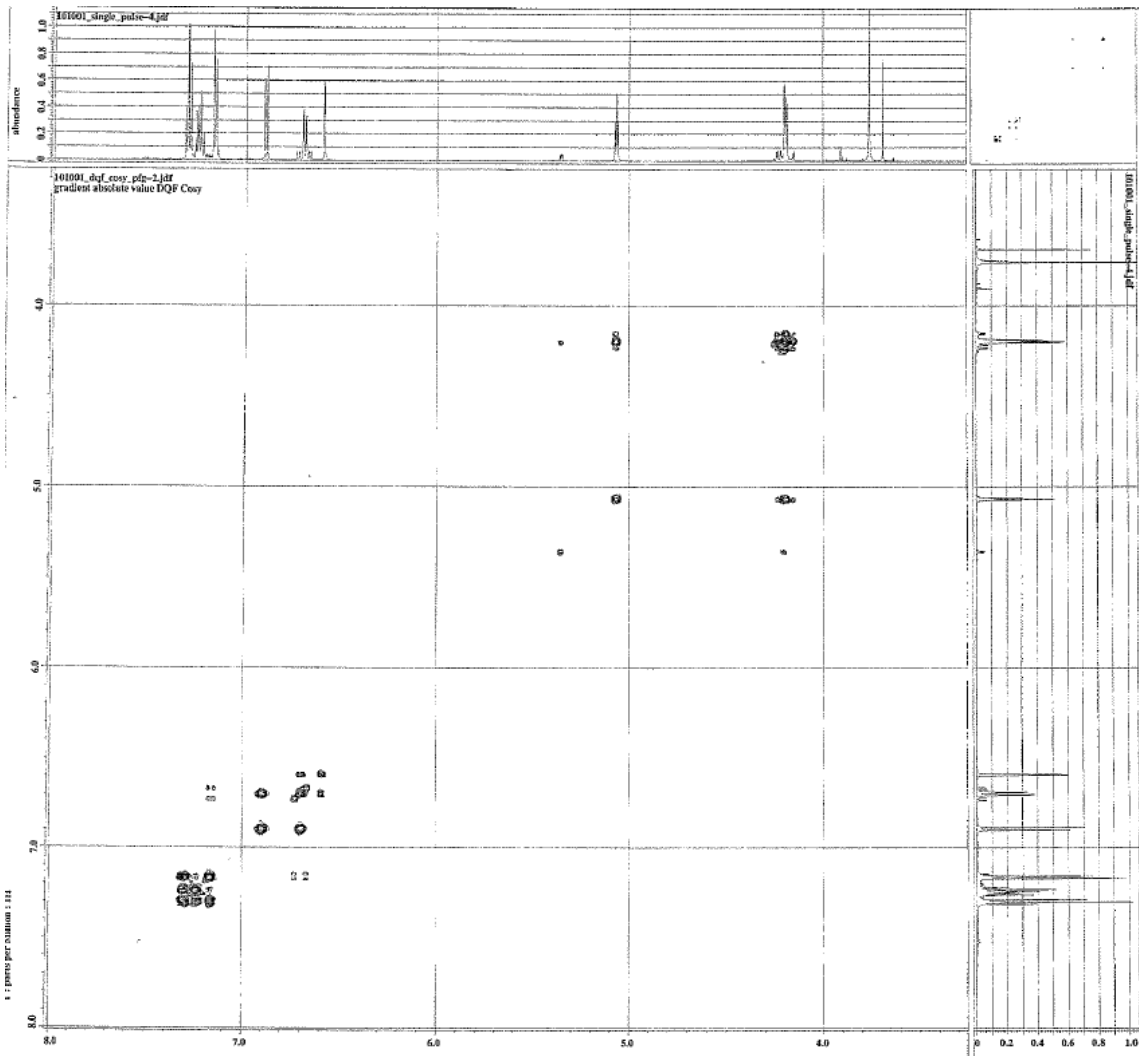

101001_noe_1d_dpfgse-6.jdf
DPFGSE NOE 1d
irr
X : parts per Million : 1H
101001_difference_noe_1d-3.jdf
Difference NOE Experiment
irr
X : parts per Million : 1H
101001_single_pulse-5.jdf
single_pulse
normal

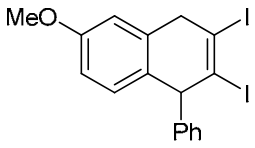

NOE
H
MeO
I
H
I
NOE
Ph
2l
H
I
H
I
NOE
O
H
Ph
H3C
NOE
2l'


101001_dept-4.jdf
DEPT with decoupling
dept135
abundance
X : parts per Million : 13C
101001_single_pulse_dec-3.jdf
single pulse decoupled gated NOE
H-Complete Decoupling

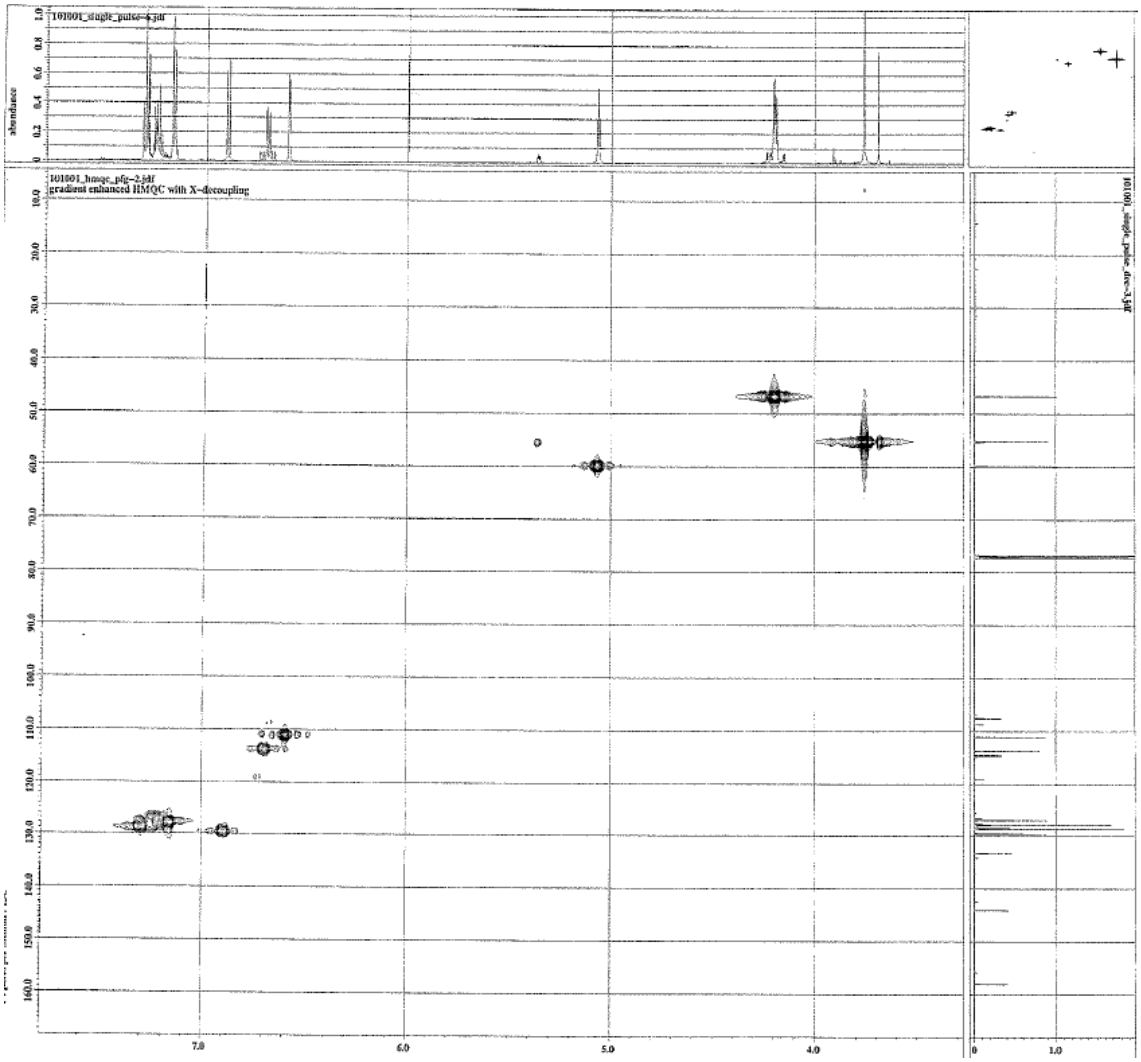

MeO
I
I
Ph

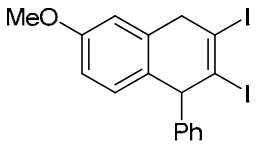
2l **dept**


101001_single_pulse_4.jdf
absorbance
101001_dqf_cosy_pfg_2.jdf
gradient absolute value DQF Cosy
8.0
7.0
6.0
5.0
4.0

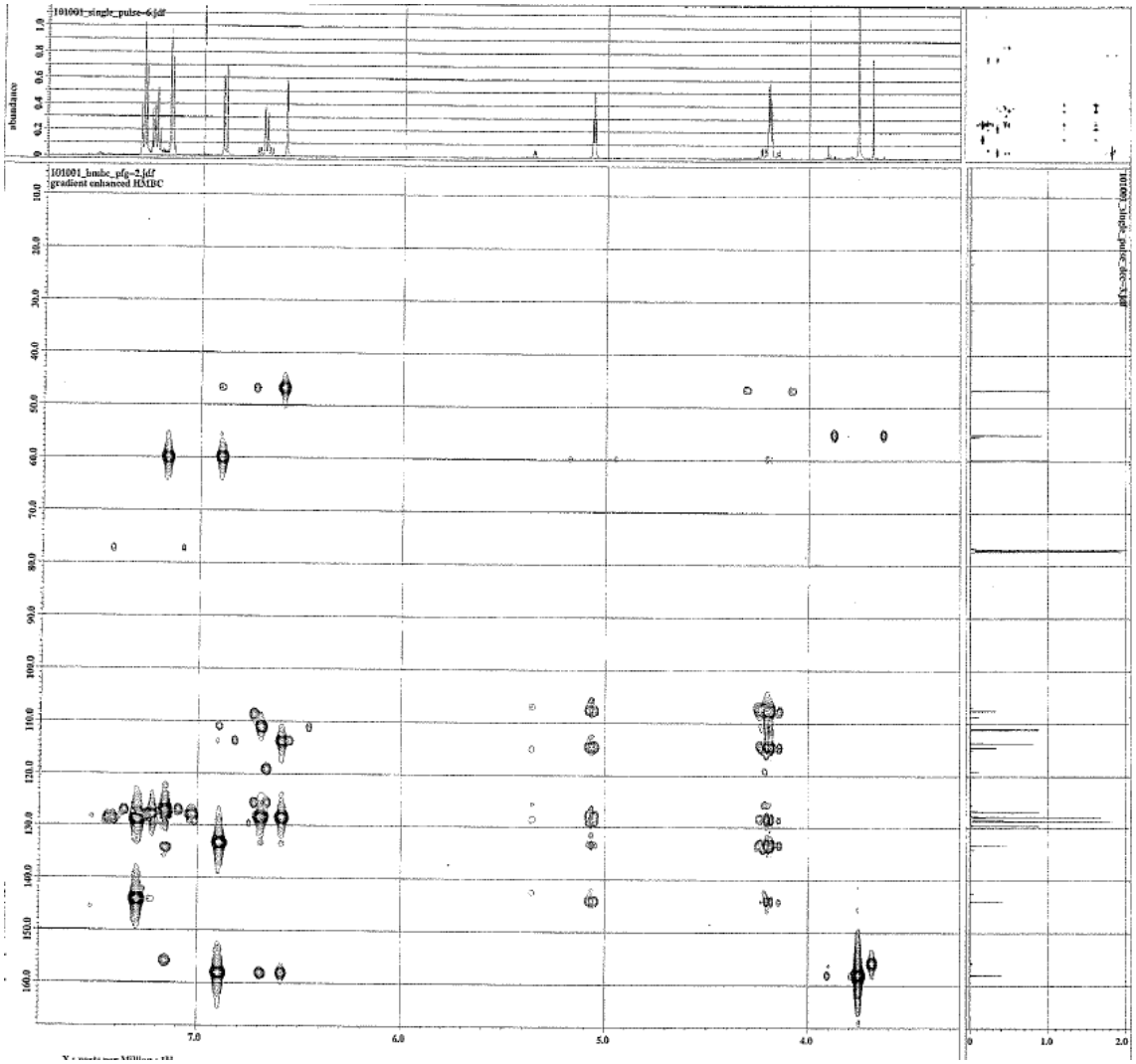

MeO
I
I
Ph

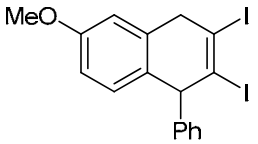
2l **COSY**





2l **hmqc**

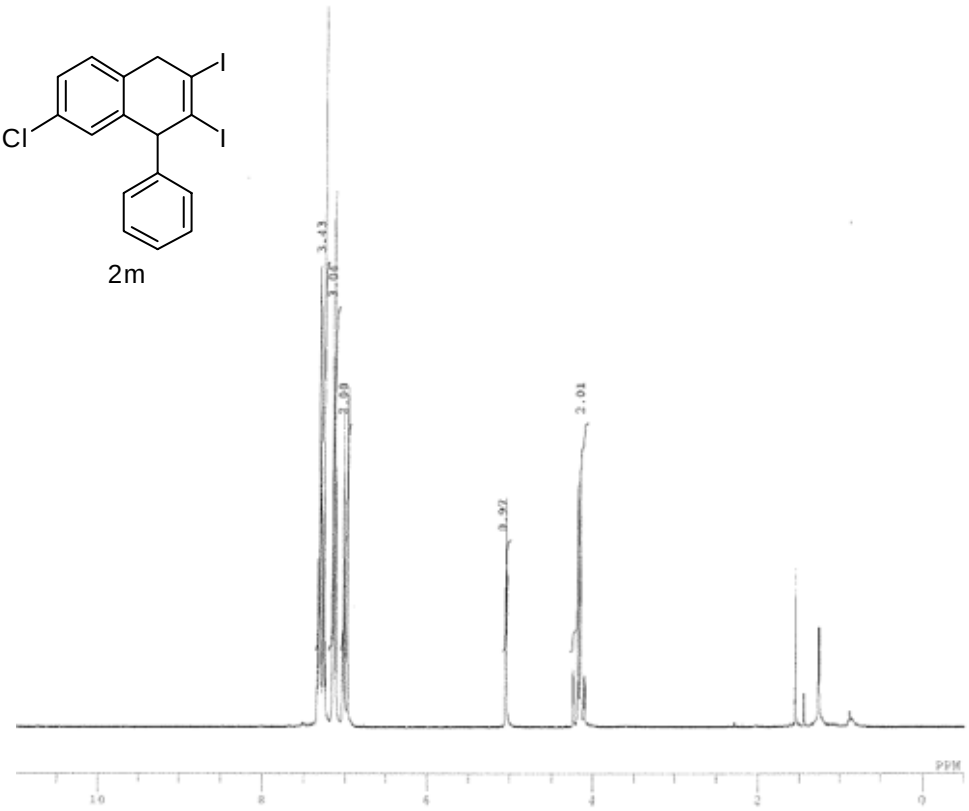




2l **hmbc**

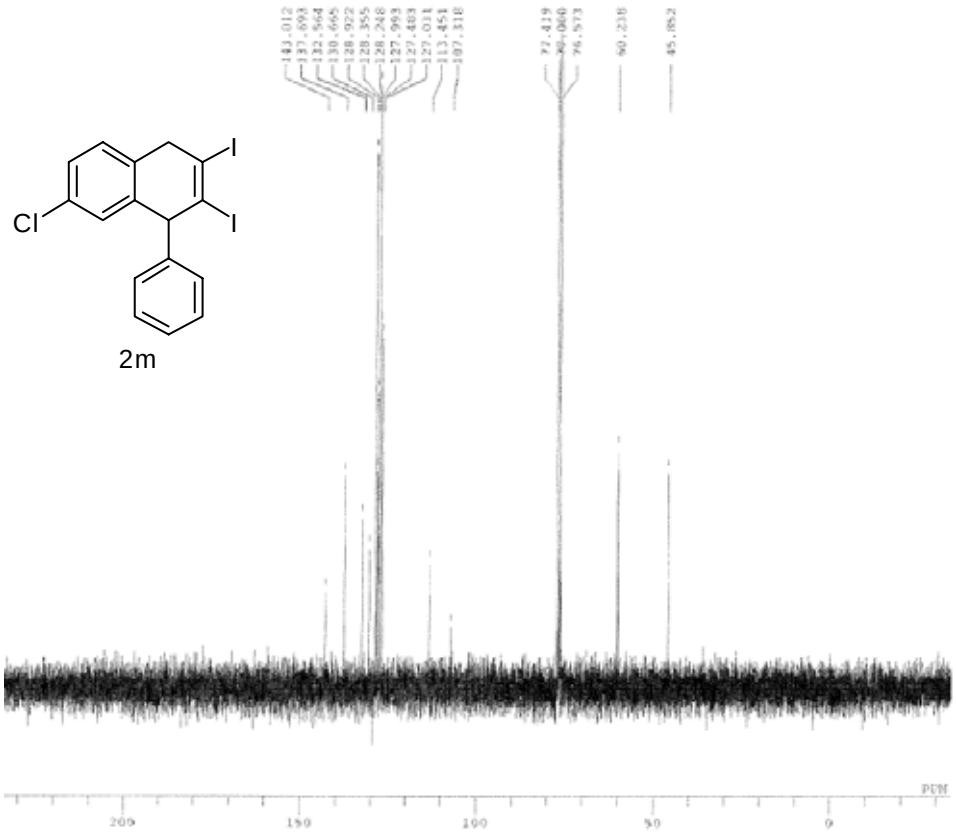
I
Cl
I

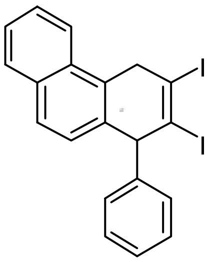
2m


3.43
3.00
3.00
3.92
2.01
PPM
10
8
6
4
2
0

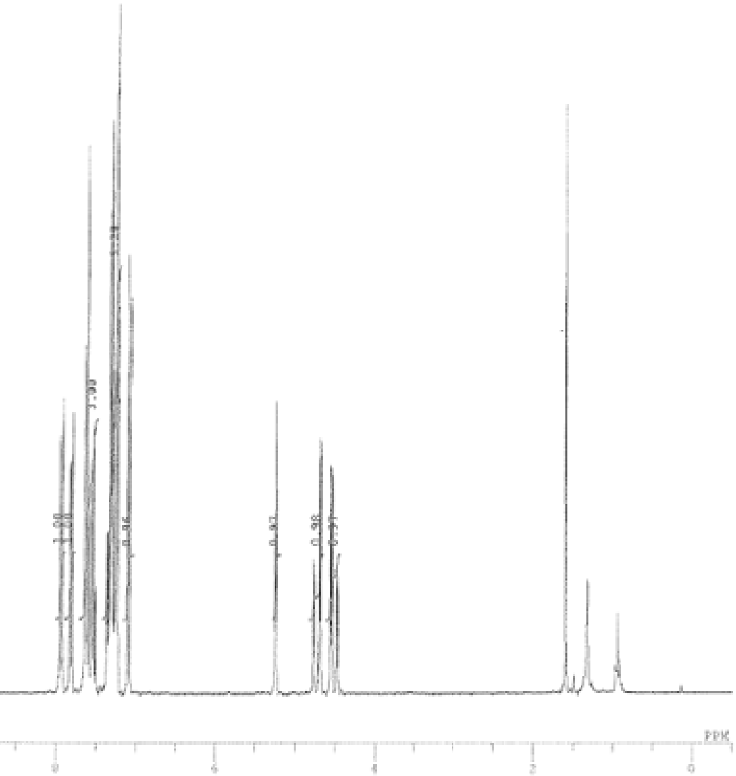
I
Cl
I

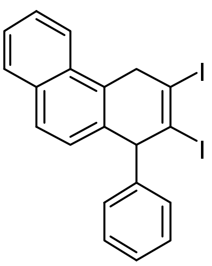
2m


143.012
137.698
132.964
130.665
128.922
128.355
128.248
127.993
127.483
127.031
113.451
107.318
77.419
76.060
76.573
60.238
45.082
PPM
200
150
100
50
0

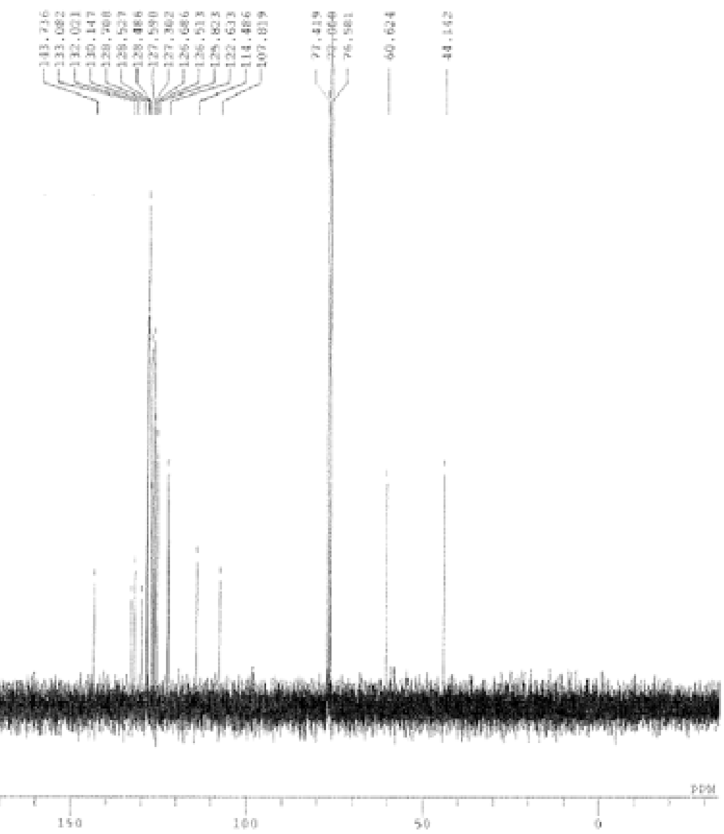


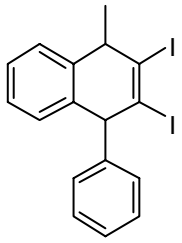
2n



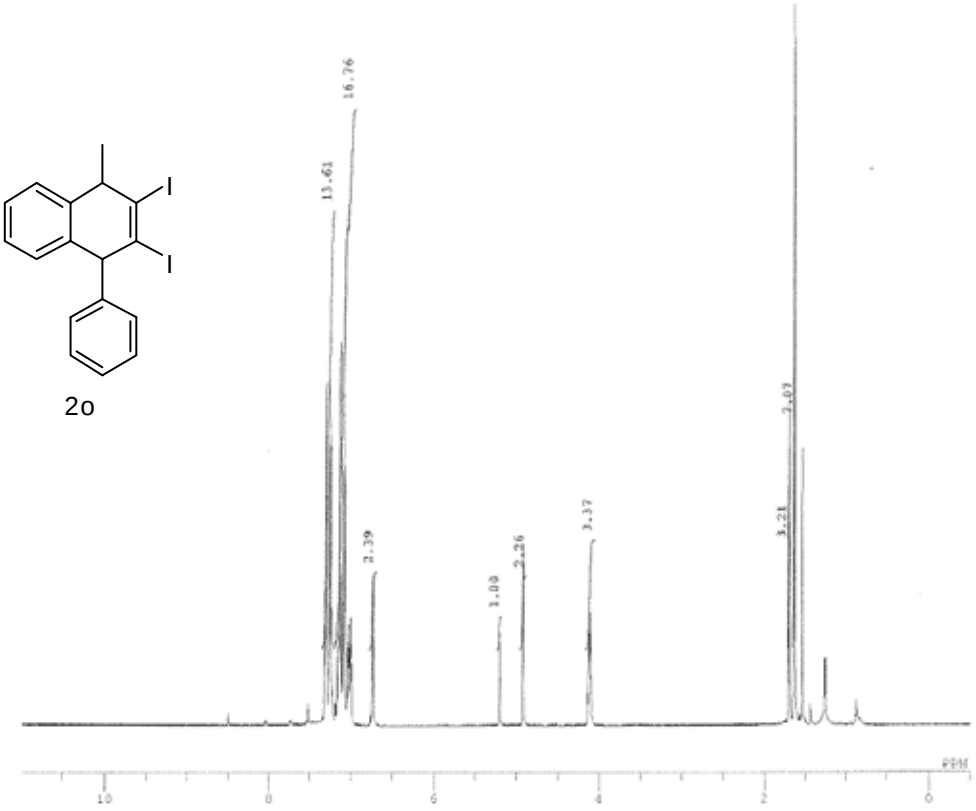


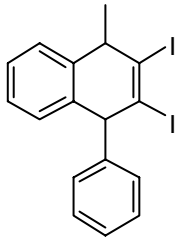
2n



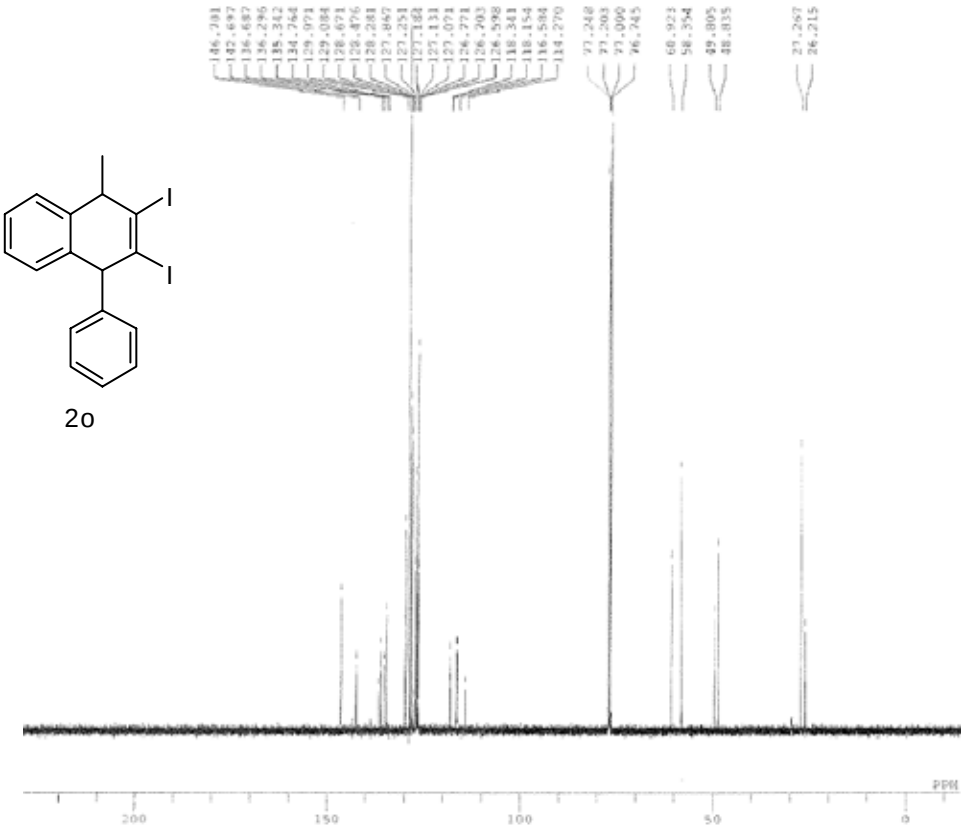


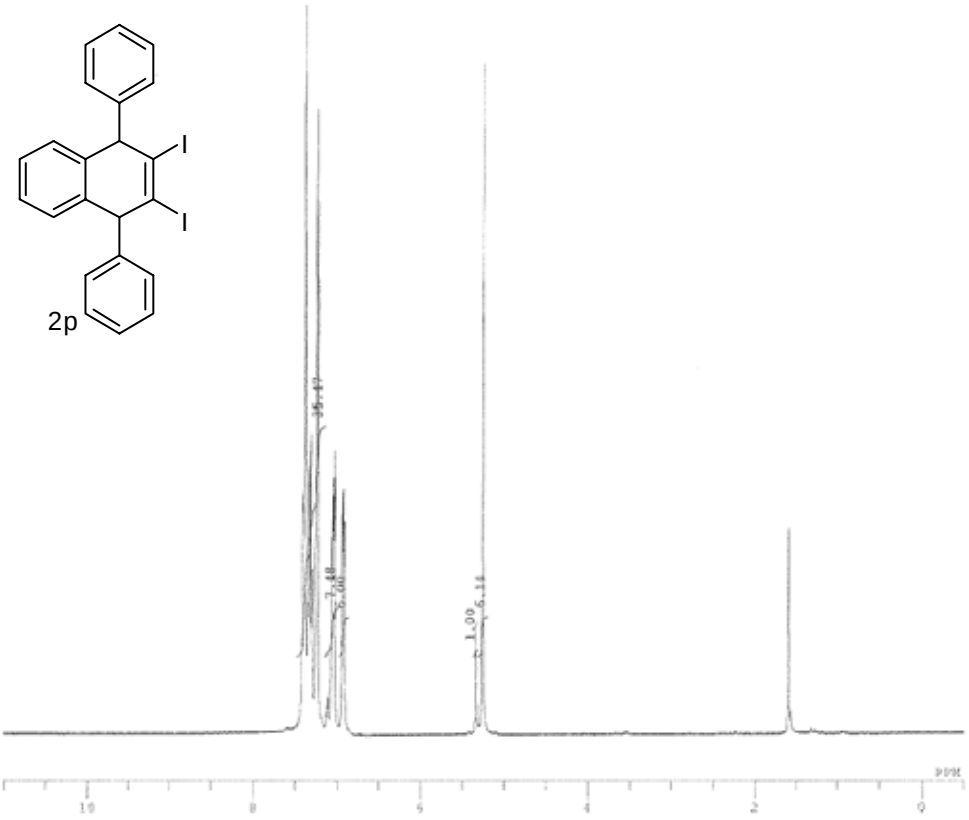
2o

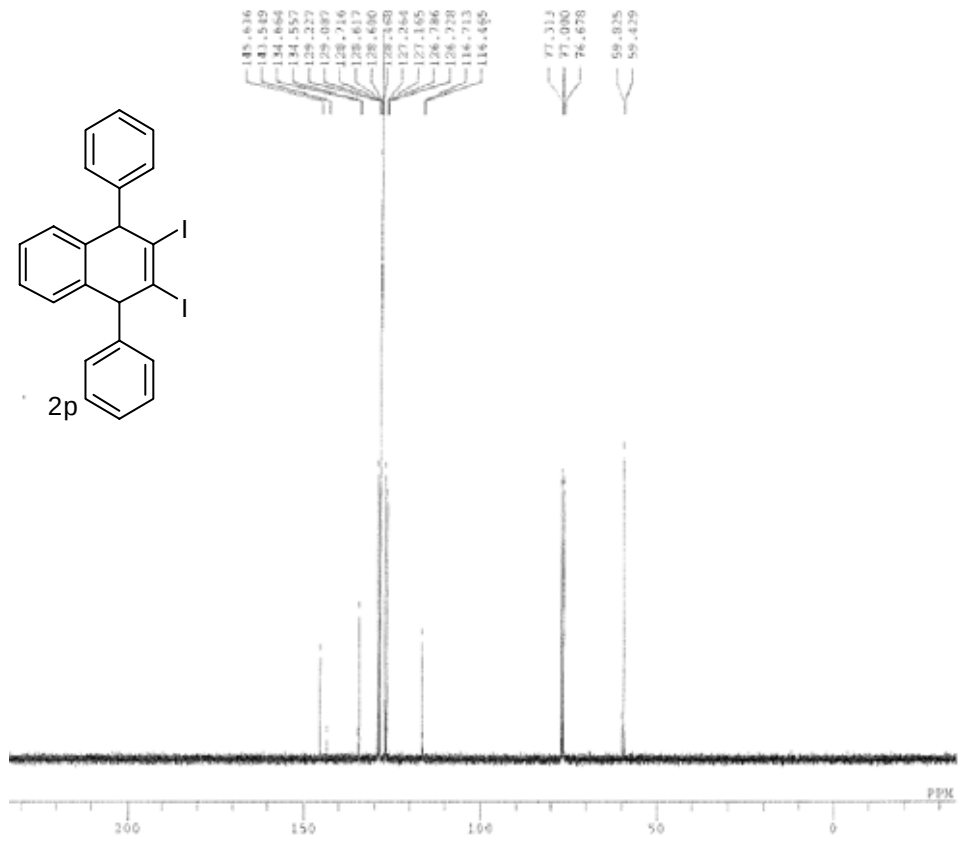

13.61
16.76
2.39
1.00
2.26
3.37
3.21
3.03
PPM
10
8
6
4
2
0

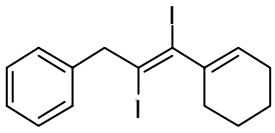


2o

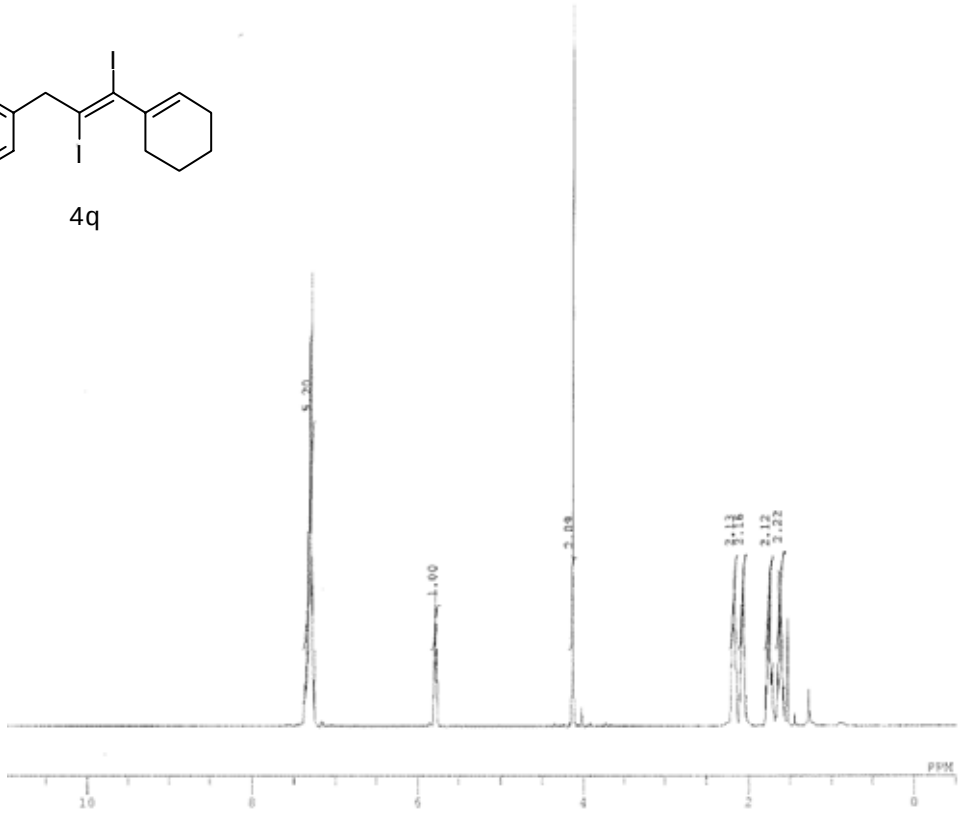

146.381
142.697
136.687
136.296
135.342
134.764
129.971
129.084
128.671
128.476
128.281
127.867
127.251
127.182
127.133
127.071
126.771
126.703
126.598
118.341
118.154
116.584
114.270
77.248
77.000
76.745
68.923
58.554
49.805
48.835
27.267
26.215
PPM
200
150
100
50
0


I
I
2p
1.00
6.14
PPM

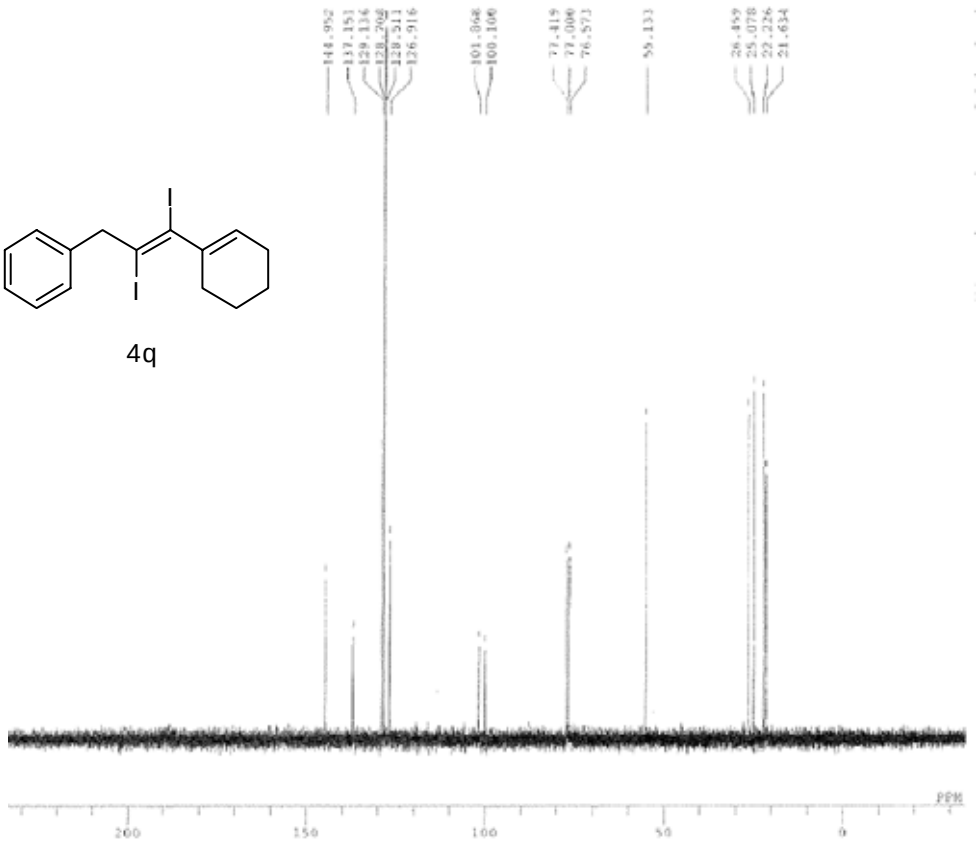

I
I
2p
PPM

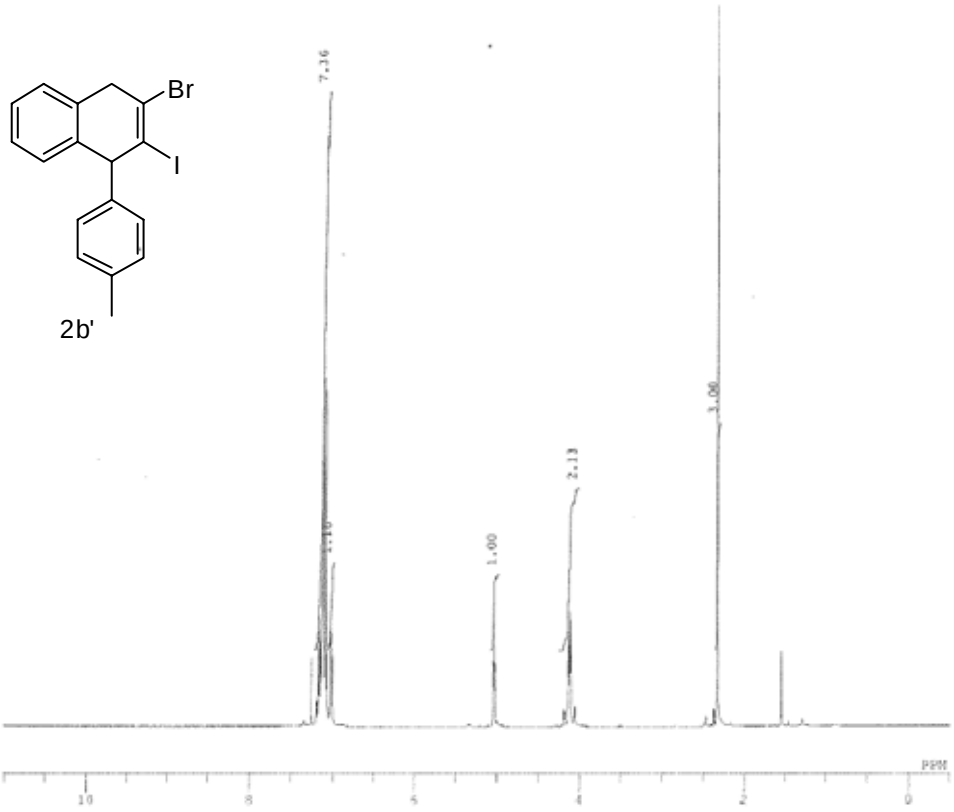


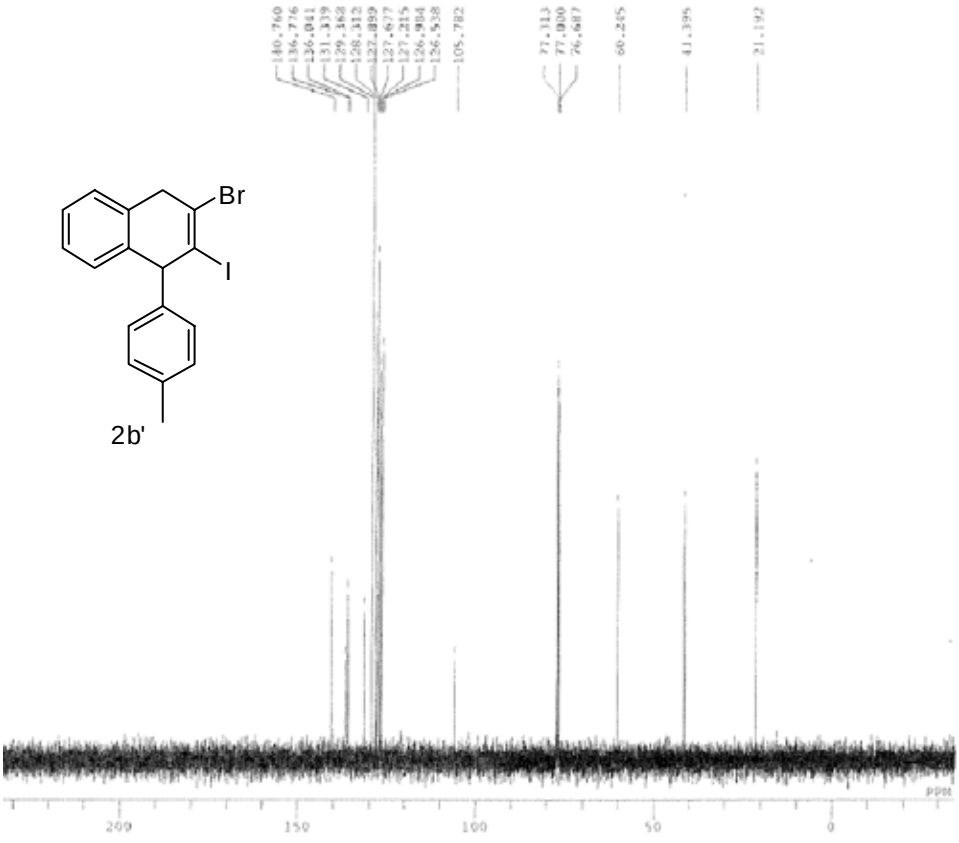
4q

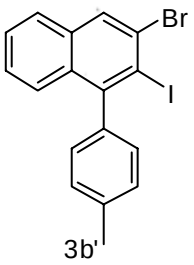

5.20
1.00
2.08
3.13
3.16
2.12
2.22
PPM
10
8
6
4
2
0

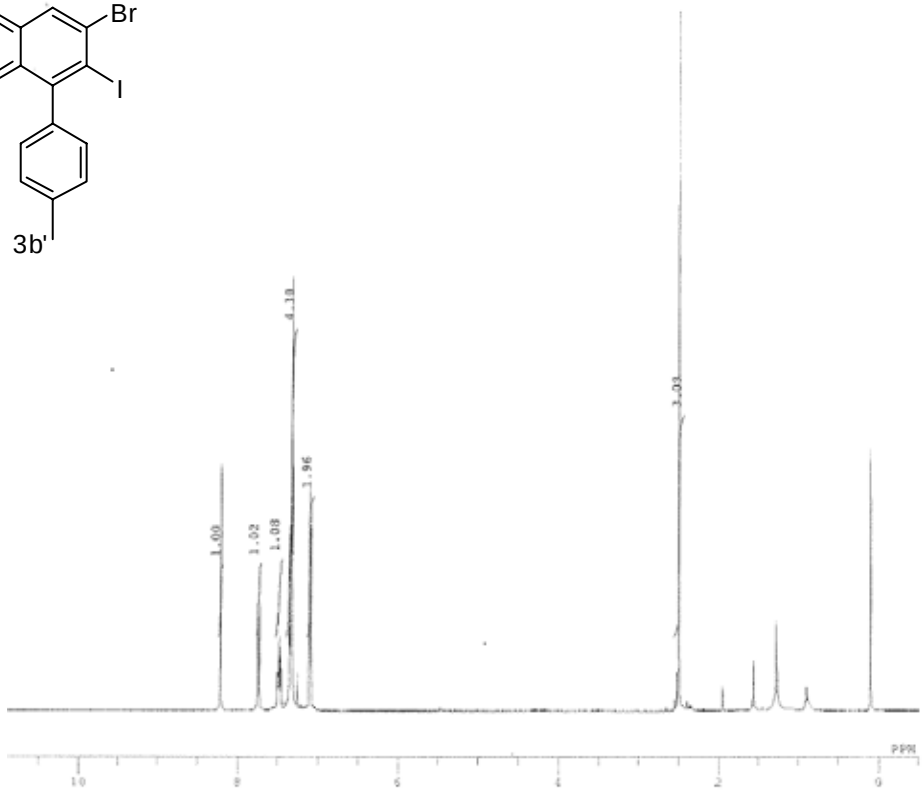
4q


144.952
137.151
129.136
128.298
128.041
126.916
101.048
100.100
77.419
77.000
76.573
59.133
26.849
25.078
22.226
21.634
PPM
200
150
100
50
0

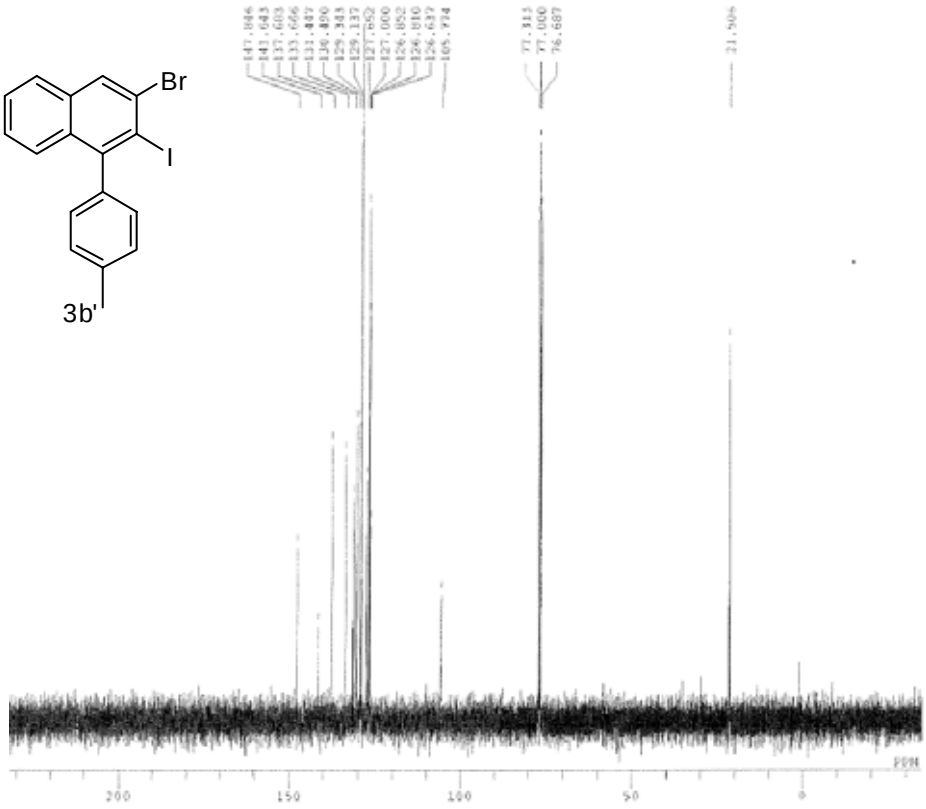

Br
I
2b'
7.16
2.10
1.00
2.13
3.00
PPM
10
8
6
4
2
0

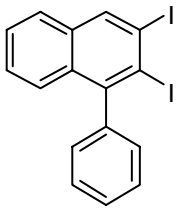

140.760
136.776
136.041
131.339
129.162
128.312
127.899
127.677
127.215
126.984
126.538
105.782
77.313
77.000
76.687
60.245
41.395
21.192
Br
I
2b'
PPM
200
150
100
50
0


Br
I
3b'

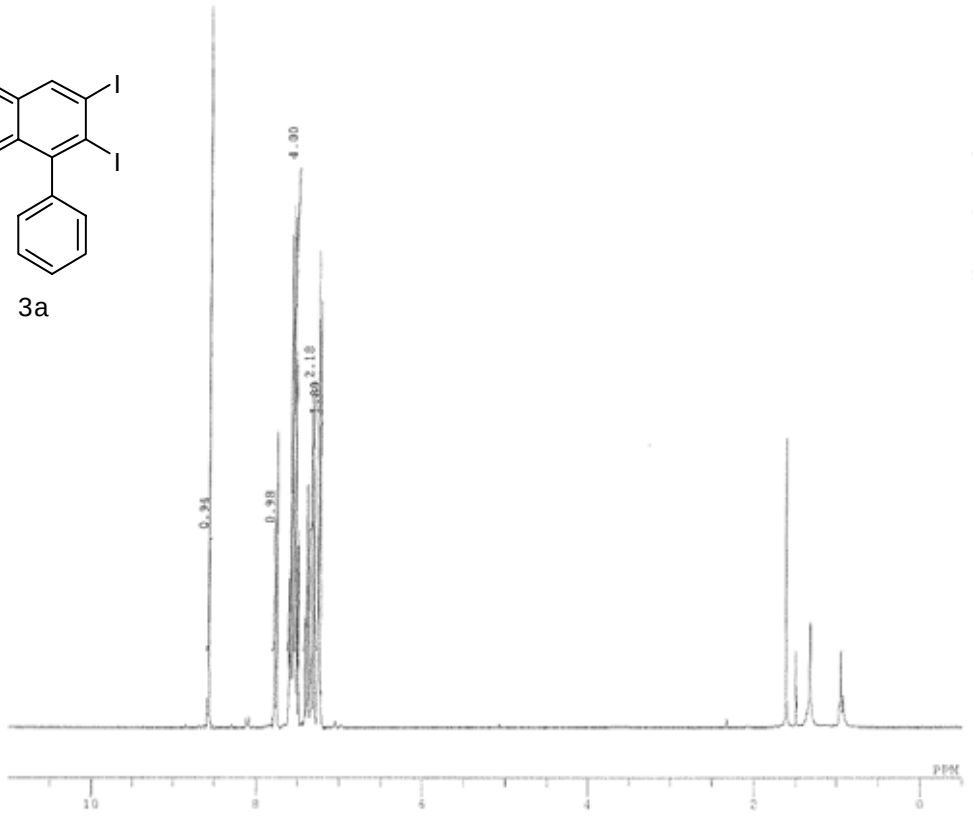

1.00
1.02
1.08
4.18
1.96
3.03
PPM
10
8
6
4
2
0

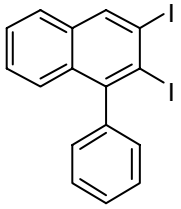
Br
I
3b'


147.886
141.643
137.603
133.666
131.487
130.890
129.343
129.137
127.852
127.000
126.852
126.010
126.637
105.774
77.313
77.000
76.687
31.505
PPM
200
150
100
50
0

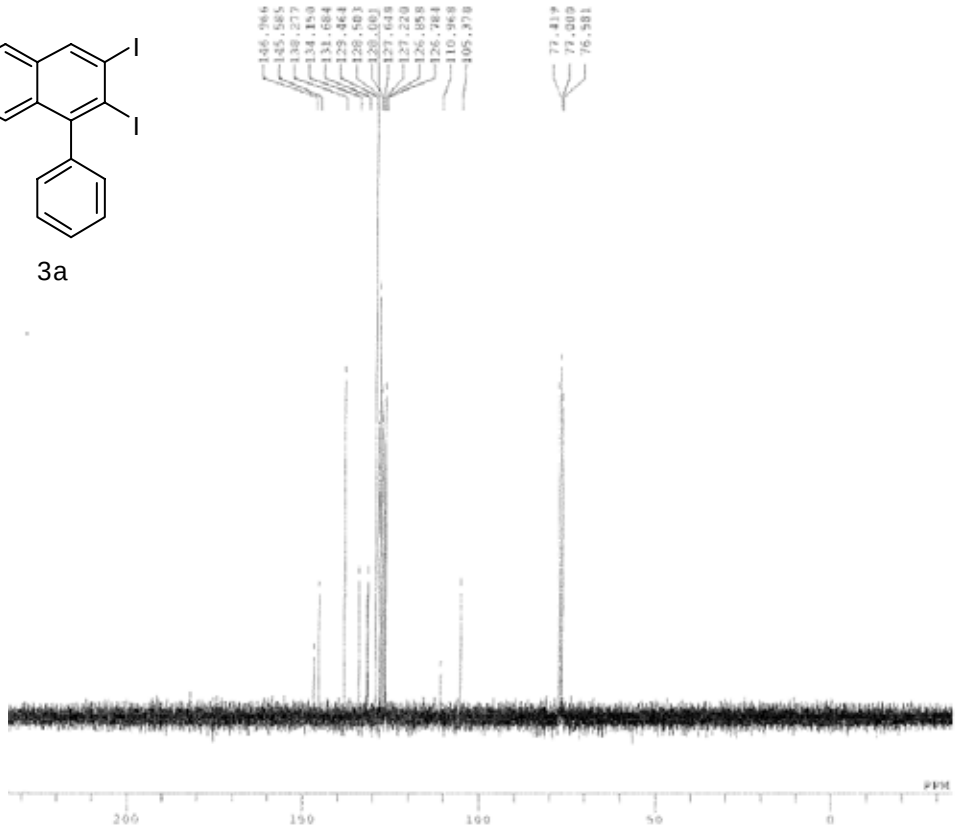


3a


0.95
0.98
4.00
2.18
PPM
10
8
6
4
2
0

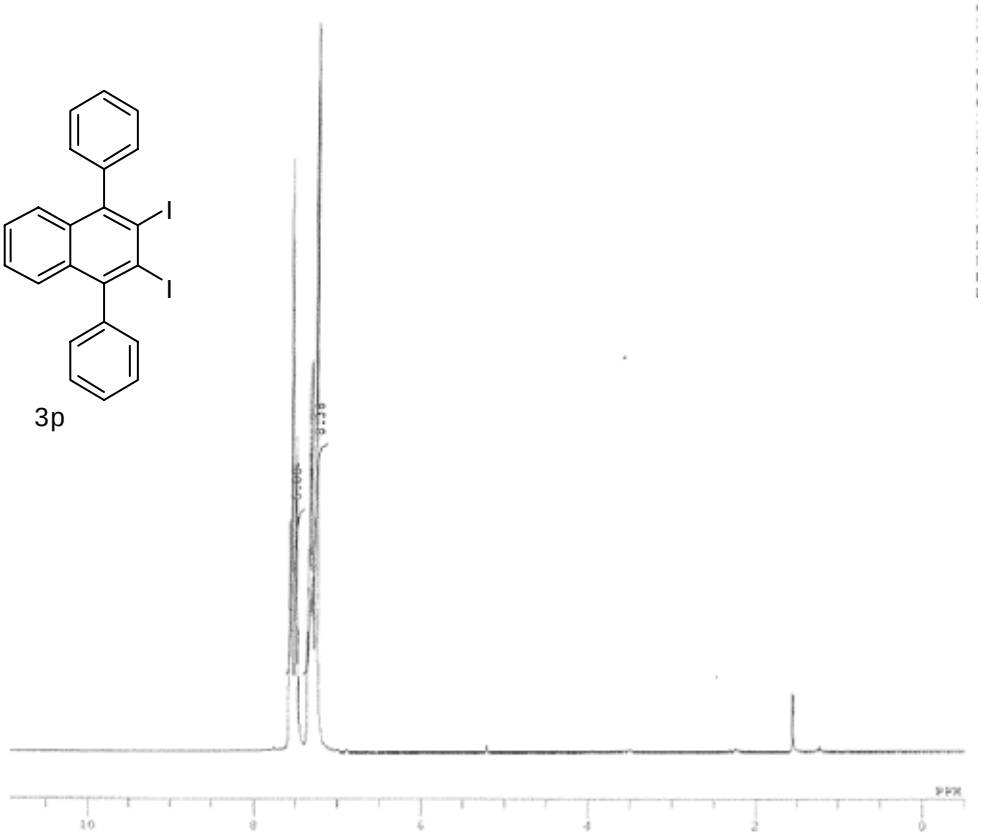


3a


146.766
145.565
138.277
134.150
131.684
129.464
128.583
128.001
127.648
127.220
126.858
126.784
110.968
105.378
77.417
77.009
76.581
PPM
200
150
100
50
0

I
I

3p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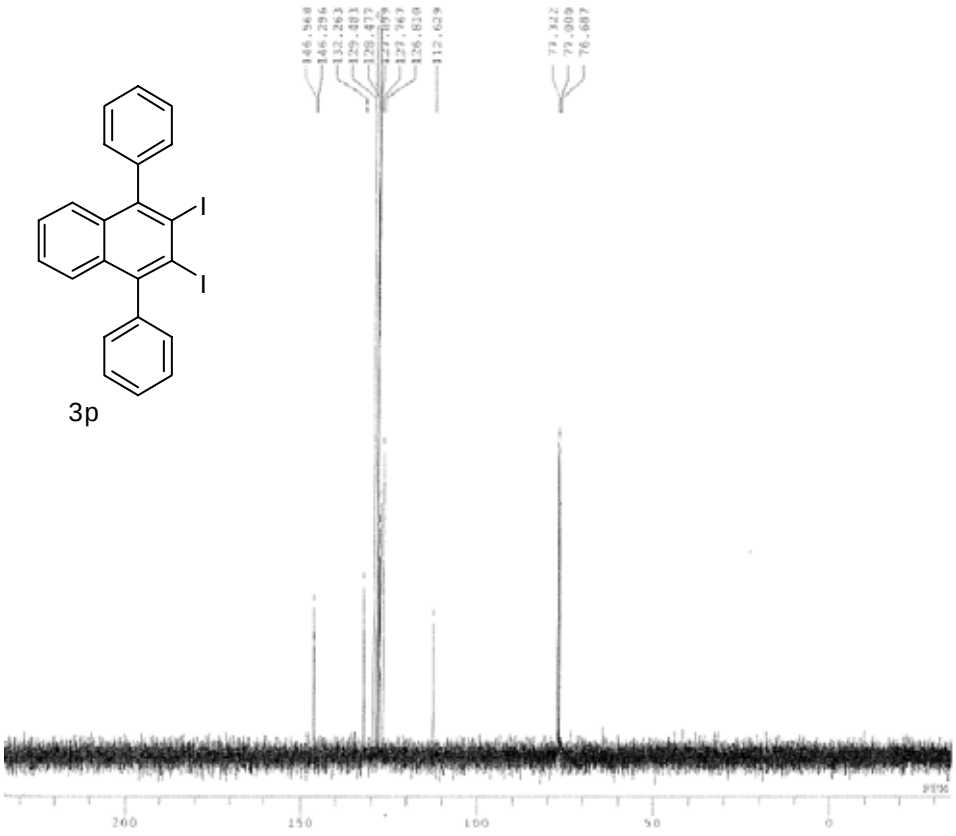
I
I

3p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129.477
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126.810
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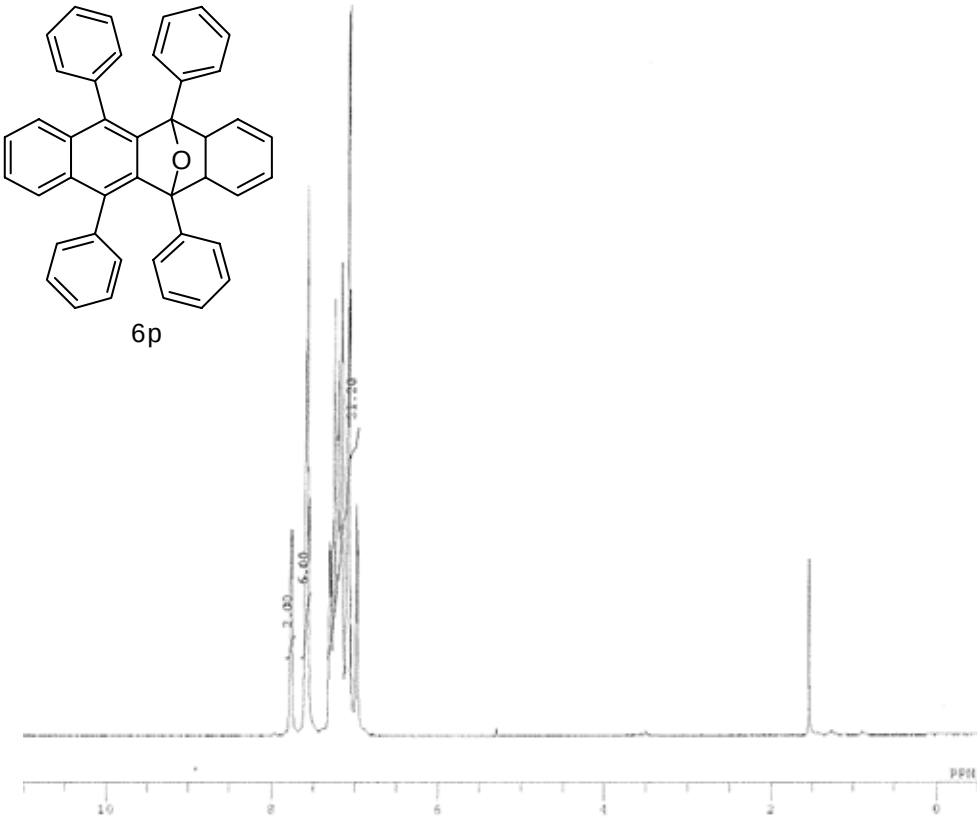
73.322
73.009
76.687
PPM
200
150
100
50
0

6p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21.00
PPM
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8
6
4
2
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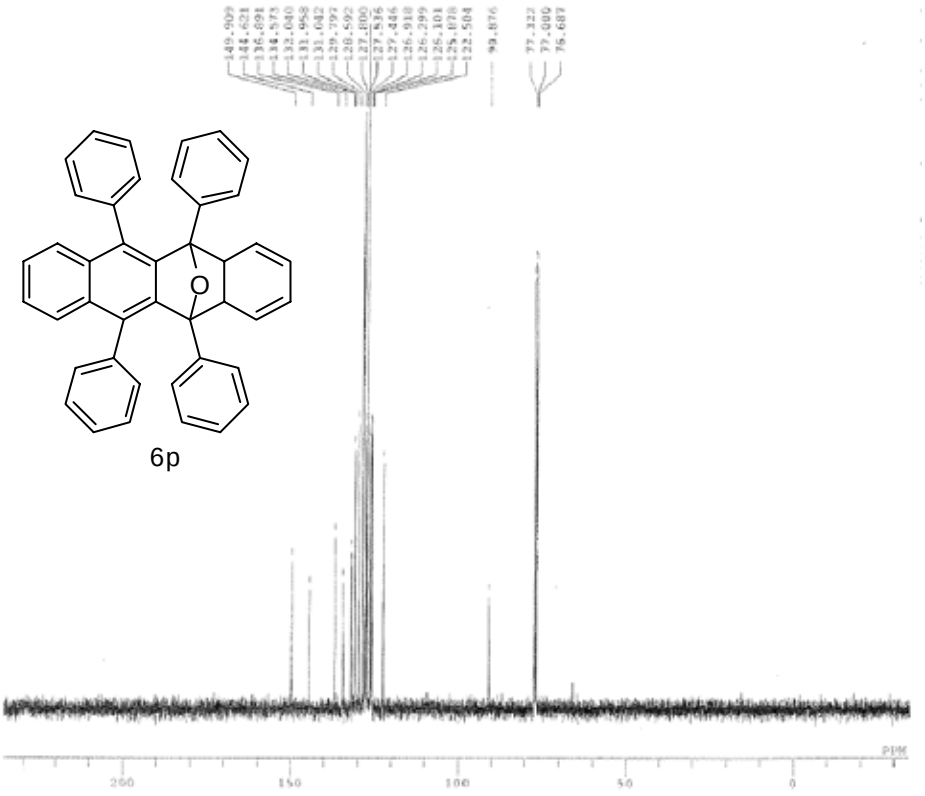
6p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131.956
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127.800
127.536
127.446
126.918
126.299
126.101
125.878
122.504
99.876
77.322
77.000
76.687
PPM
200
150
100
50
0