

Supporting Information

An Aqueous Medium-Controlled Stereospecific Oxidative Iodination of Alkynes: An Efficient Access to (E)-Diiodoalkene Derivatives

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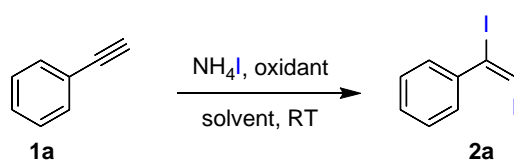
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1. General Information

All chemicals used were reagent grade and used as received without further purification. ^1H NMR spectra were recorded at 300 or 500 MHz and Proton-decoupled ^{13}C NMR spectra were obtained at 75 or 125 MHz in CDCl_3 . The chemical shifts (δ) are reported in ppm units relative to TMS as an internal standard for ^1H NMR and CDCl_3 for ^{13}C NMR spectra. Coupling constants (J) are reported in hertz (Hz) and multiplicities are indicated as follows: s (singlet), br s (broad singlet), d (doublet), dd (doublet of doublet), m (multiplet). TLC inspections were performed on Silica gel 60 F₂₅₄ plates. Column chromatography was performed on silica gel (100-200 mesh) using *n*-hexane-EtOAc as eluent.

2. Reaction Optimization

Table S1. Optimization of reaction conditions.^{a,b}

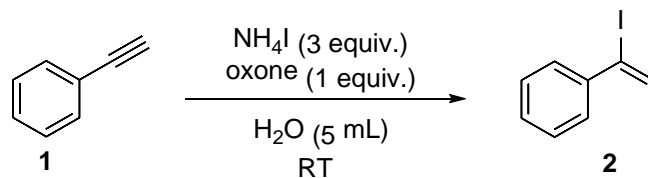


Entry	Solvent	Time (min)	NH_4I (equiv.)	Oxidant (equiv.)	Yield (%)
1	H_2O	20	2	oxone (1)	88
2	H_2O	20	3	oxone (1)	97
3	H_2O	10	3	oxone (1)	61
4	H_2O	20	3	oxone (1.5)	97
5	H_2O	20	3	oxone (0.75)	69
6	H_2O	20	4	oxone (1)	97
7	H_2O	20	3	$\text{K}_2\text{S}_2\text{O}_8$ (1)	30 (93) ^c
8	H_2O	20	3	<i>m</i> CPBA (1)	13 (32) ^d
9	H_2O	20	3	aq. H_2O_2 (1)	00 (04) ^d
10	H_2O	20	3	aq. TBHP (1)	00 (40) ^d

^a Reaction conditions: phenyl acetylene (**1a**) (1 mmol), NH_4I and oxidant in solvent (5 mL), rt. ^b Isolated yields after column chromatography purification. ^c The values shown in parenthesis refers to the yield of the reaction performed for 1 h. ^d The values shown in parenthesis refers to the yield of the reaction performed for 24 h.

3. General Procedure

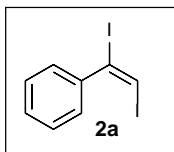
General procedure for the stereo selective vicinal diiodination of alkynes using NH₄I and oxone in aqueous media



In an oven dried double necked round bottom flask equipped with a magnetic stirring bar, alkyne **1** (1 mmol, 1 equiv.), NH₄I (3 mmol, 3 equiv.) and water (5 mL) were taken at room temperature and stirred for some time. Then, oxone (1 mmol, 1 equiv.) was added slowly and the resulting solution was allowed to stir at room temperature for the time indicated in the tables. After completion of the reaction, as indicated by the TLC, the reaction was quenched with aqueous Na₂S₂O₃ and stirred vigorously for few minutes. The reaction mixture was extracted with DCM (15 x 3 mL) and the organic phase was washed with water (2 x 5 mL) and dried over anhydrous Na₂SO₄. After evaporation of the solvent under reduced pressure, the crude reaction mixture was purified by column chromatography (silica gel, hexanes or ethyl acetate/hexanes mixture) to give the corresponding *trans*-diiodoalkene **2**.

4. Spectroscopic data of all compounds:

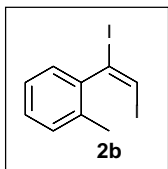
(*E*)-(1,2-diiodovinyl)benzene (**2a**)¹



The compound **2a** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.37-7.30 (m, 5H, Ar-*H*), 7.25 (s, 1H, vinylic-*H*); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 143.03, 128.92, 128.45, 128.38, 96.13, 80.75.

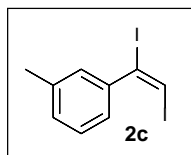
(*E*)-1-(1,2-diiodovinyl)-2-methylbenzene (**2b**)



The compound **2b** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

Yellow solid; mp = 48-50 °C; IR (KBr): ν_{max} 3060, 2933, 1712, 1598, 1450, 1371, 1233, 1147, 1100, 1030, 845, 779, 749, 717, 655, and 593 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.25 (s, 1H, vinylic-*H*), 7.24-7.23 (m, 1H, Ar-*H*), 7.22-7.17 (m, 2H, Ar-*H*), 7.10-7.08 (m, 1H, Ar-*H*), 2.23 (s, 3H, Ar-CH₃); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 142.83, 134.43, 130.52, 129.00, 127.54, 126.22, 96.18, 82.83, 19.44; HR-MS (EI) *m/z* calcd for C₉H₈I₂, 369.86800; Found 369.86645; Anal. Calcd for C₉H₈I₂: C, 29.22; H, 2.18. Found: C, 29.59; H, 2.51.

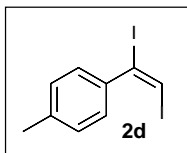
(*E*)-1-(1,2-diiodovinyl)-3-methylbenzene (**2c**)²



The compound **2c** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

^1H NMR (500 MHz, CDCl_3): δ (ppm) = 7.26-7.23 (m, 1H, Ar-*H*), 7.22 (s, 1H, vinylic-*H*), 7.16-7.12 (m, 3H, Ar-*H*), 2.36 (s, 3H, Ar- CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 142.91, 138.12, 129.70, 128.92, 128.23, 125.45, 96.40, 80.45, 21.34.

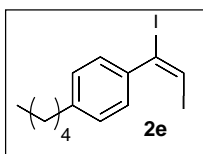
(*E*)-1-(1,2-diiodovinyl)-4-methylbenzene (2d)²



The compound **2d** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.26 (d, J = 8.19 Hz, 2H, Ar-*H*), 7.21 (s, 1H, vinylic-*H*), 7.15 (d, J = 7.94 Hz, 2H, Ar-*H*), 2.35 (s, 3H, Ar- CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 140.10, 139.01, 129.03, 128.42, 96.57, 80.19, 21.41.

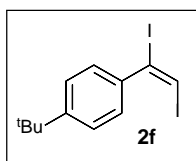
(*E*)-1-(1,2-diiodovinyl)-4-pentylbenzene (2e)¹



The compound **2e** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

^1H NMR (500 MHz, CDCl_3): δ (ppm) = 7.28 (d, J = 8.24 Hz, 2H, Ar-*H*), 7.21 (s, 1H, vinylic-*H*), 7.15 (d, J = 8.24 Hz, 2H, Ar-*H*), 2.60 (t, J = 7.93 Hz, 1 x 2H, benzylic-*H*), 1.66-1.59 (m, 1 x 2H, aliphatic-*H*), 1.36-1.31 (m, 2 x 2H, aliphatic-*H*), 0.90 (t, J = 7.17 Hz, 1 x 3H, aliphatic-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 144.02, 140.12, 128.47, 128.30, 96.73, 80.03, 35.78, 31.53, 30.78, 22.50, 14.02.

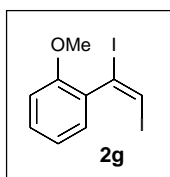
(*E*)-1-(*tert*-butyl)-4-(1,2-diiodovinyl)benzene (2f)



The compound **2f** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): ν_{\max} 3065, 2961, 2903, 2867, 1607, 1499, 1463, 1398, 1363, 1267, 1198, 1156, 1106, 1019, 856, 838, 820, 777, 705 and 600 cm^{-1} ; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.37-7.34 (m, 2H, Ar-*H*), 7.33-7.29 (m, 2H, Ar-*H*), 7.22 (s, 1H, vinylic-*H*), 1.33 (s, 9H, Ar- $\text{C}(\text{CH}_3)_3$); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ (ppm) = 152.04, 139.73, 128.34, 125.21, 96.77, 79.86, 34.76, 31.19; HR-MS (EI) m/z calcd for $\text{C}_{12}\text{H}_{14}\text{I}_2$, 411.92000; Found 411.91849; Anal. Calcd for $\text{C}_{12}\text{H}_{14}\text{I}_2$: C, 34.98; H, 3.42. Found: C, 34.60; H, 3.37.

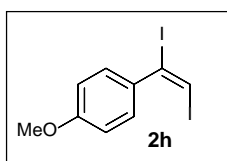
(*E*)-1-(1,2-diiodovinyl)-2-methoxybenzene (2g)



The compound **2g** was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.1:9.9) as an eluent.

IR (Neat): ν_{\max} 3064, 3004, 2935, 2833, 1582, 1482, 1459, 1434, 1288, 1251, 1155, 1108, 1023, 849, 785, 751, 660 and 601 cm^{-1} ; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.34 (t, $J = 8.80\text{ Hz}$, 1H, Ar-*H*), 7.25 (s, 1H, vinylic-*H*), 7.14 (d, $J = 7.58\text{ Hz}$, 1H, Ar-*H*), 6.97 (t, $J = 7.45\text{ Hz}$, 1H, Ar-*H*), 6.90 (d, $J = 8.31\text{ Hz}$, 1H, Ar-*H*), 3.90 (s, 3H, OCH_3); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ (ppm) = 155.17, 131.94, 130.47, 129.34, 120.54, 111.54, 92.53, 83.13, 55.66; HR-MS (EI) m/z calcd for $\text{C}_9\text{H}_8\text{I}_2\text{O}$, 385.86600; Found 385.86645; Anal. Calcd for $\text{C}_9\text{H}_8\text{I}_2\text{O}$: C, 28.01; H, 2.09. Found: C, 28.26; H, 2.10.

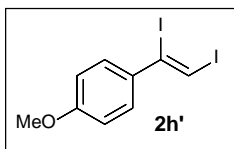
(*E*)-1-(1,2-diiodovinyl)-4-methoxybenzene (2h)¹



The compound **2h** was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.1:9.9) as an eluent.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ (ppm) = 7.33 (d, $J = 8.80\text{ Hz}$, 2H, Ar-*H*), 7.19 (s, 1H, vinylic-*H*), 6.87 (d, $J = 8.92\text{ Hz}$, 2H, Ar-*H*), 3.83 (s, 3H, OCH_3); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ (ppm) = 159.75, 135.22, 130.19, 113.63, 96.53, 79.82, 55.31.

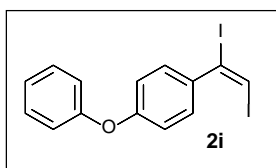
(*Z*)-1-(1,2-diiodovinyl)-4-methoxybenzene (2h')



The compound **2h'** was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.1:9.9) as an eluent.

IR (Neat): ν_{\max} 3027, 2955, 2834, 1601, 1501, 1457, 1294, 1252, 1176, 1030, 865, 831, 771, 679, and 542 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.43 (s, 1H, vinylic-*H*), 7.39 (d, J = 8.68 Hz, 2H, Ar-*H*), 6.81 (d, J = 8.68 Hz, 2H, Ar-*H*), 3.81 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 160.08, 136.40, 129.72, 119.63, 113.63, 90.59, 55.40; HR-MS (EI) m/z calcd for $\text{C}_9\text{H}_8\text{I}_2\text{O}$, 385.86611; Found 385.86645; Anal. Calcd for $\text{C}_9\text{H}_8\text{I}_2\text{O}$: C, 28.01; H, 2.09. Found: C, 28.23; H, 2.15.

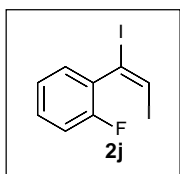
(*E*)-1-(1,2-diiodovinyl)-4-phenoxybenzene (**2i**)



The compound **2i** was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.1:9.9) as an eluent.

Pale yellow solid; mp = 56-58 $^{\circ}\text{C}$; IR (KBr): ν_{\max} 3060, 1610, 1582, 1486, 1280, 1244, 1197, 1148, 1100, 1063, 1018, 869, 823, 778, 744, 687, and 593 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.39-7.32 (m, 4H, Ar-*H*), 7.23 (s, 1H, vinylic-*H*), 7.15 (t, J = 7.45 Hz, 1H, Ar-*H*), 7.06 (d, J = 7.82 Hz, 2H, Ar-*H*), 6.94 (d, J = 8.68 Hz, 2H, Ar-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 157.92, 156.03, 137.29, 130.31, 129.87, 124.02, 119.72, 117.70, 95.87, 80.46; HR-MS (EI) m/z calcd for $\text{C}_{14}\text{H}_{10}\text{I}_2\text{O}$, 447.88100; Found 447.88210; Anal. Calcd for $\text{C}_{14}\text{H}_{10}\text{I}_2\text{O}$: C, 37.53; H, 2.25. Found: C, 38.29; H, 2.25.

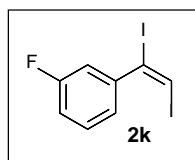
(*E*)-1-(1,2-diiodovinyl)-2-fluorobenzene (**2j**)



The compound **2j** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): ν_{\max} 3064, 2922, 1616, 1577, 1481, 1448, 1269, 1242, 1221, 1151, 1098, 1029, 862, 796, 755, 656, and 605 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ (ppm) = 7.40 (s, 1H, vinylic-*H*), 7.39-7.34 (m, 1H, Ar-*H*), 7.22 (ddd, $J = 1.83, 7.47, 14.64$ Hz, 1H, Ar-*H*), 7.17 (ddd, $J = 1.06, 7.32, 14.95$ Hz, 1H, Ar-*H*), 7.09-7.05 (m, 1H, Ar-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 130.94 (d, $J = 8.06$ Hz), 129.97, 124.25 (d, $J = 3.66$ Hz), 116.28 (d, $J = 20.54$ Hz), 87.88, 84.91; HR-MS (EI) m/z calcd for $\text{C}_8\text{H}_5\text{FI}_2$, 373.84350; Found 373.84646; Anal. Calcd for $\text{C}_8\text{H}_5\text{FI}_2$: C, 25.70; H, 1.35. Found: C, 25.65; H, 1.31.

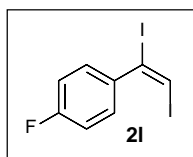
(*E*)-1-(1,2-diiodovinyl)-3-fluorobenzene (**2k**)



The compound **2k** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): ν_{\max} 3064, 2924, 1609, 1582, 1477, 1429, 1263, 1205, 1157, 1120, 1073, 941, 874, 789, 771, 694, and 611 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ (ppm) = 7.37-7.32 (m, 1H, Ar-*H*), 7.30 (s, 1H, vinylic-*H*), 7.15-7.12 (m, 1H, Ar-*H*), 7.07-7.01 (m, 2H, Ar-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 162.16 (d, $J = 247.94$ Hz), 145.00 (d, $J = 8.06$ Hz), 130.07 (d, $J = 8.06$ Hz), 124.23 (d, $J = 2.20$ Hz), 116.00 (d, $J = 21.27$ Hz), 115.61 (d, $J = 22.74$ Hz), 93.94, 81.86; HR-MS (EI) m/z calcd for $\text{C}_8\text{H}_5\text{FI}_2$, 373.84450; Found 373.84646; Anal. Calcd for $\text{C}_8\text{H}_5\text{FI}_2$: C, 25.70; H, 1.35. Found: C, 24.66; H, 1.45.

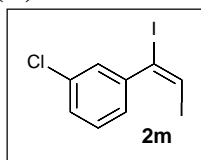
(*E*)-1-(1,2-diiodovinyl)-4-fluorobenzene (**2l**)¹



The compound **2l** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.38-7.32 (m, 2H, Ar-*H*), 7.27 (s, 1H, vinylic-*H*), 7.08-7.02 (m, 2H, Ar-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 162.48 (d, J = 250.14 Hz), 139.11 (d, J = 2.93 Hz), 130.55 (d, J = 8.80 Hz), 115.50 (d, J = 22.00 Hz), 94.78, 81.40.

(*E*)-1-chloro-3-(1,2-diiodovinyl)benzene (2m)

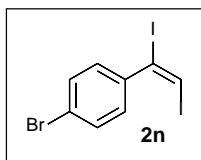


The compound **2m** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): ν_{max} 3064, 2925, 1564, 1465, 1408, 1237, 1158, 1080, 877, 787, 726, 685, and 605 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.34-7.32 (m, 1H, Ar-*H*), 7.31-7.29 (m, 2H, Ar-*H*), 7.30 (s, 1H, vinylic-*H*) 7.26-7.21 (m, 1H, Ar-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 144.66, 134.09, 129.38 (d, J = 69.69 Hz), 127.57 (d, J = 184.86 Hz), 93.76, 82.09.

HR-MS (EI) m/z calcd for $\text{C}_8\text{H}_5\text{ClI}_2$, 389.82000; Found 389.81691; Anal. Calcd for $\text{C}_8\text{H}_5\text{ClI}_2$: C, 24.61; H, 1.29. Found: C, 24.83; H, 1.33.

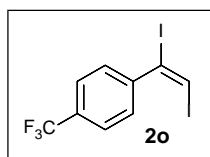
(*E*)-1-bromo-4-(1,2-diiodovinyl)benzene (2n)¹



The compound **2n** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.52-7.49 (m, 2H, Ar-*H*), 7.29 (s, 1H, vinylic-*H*), 7.25-7.21 (m, 2H, Ar-*H*); ^{13}C NMR (125 MHz, CDCl_3): δ (ppm) = 141.96, 131.67, 130.12, 123.08, 94.44, 81.57.

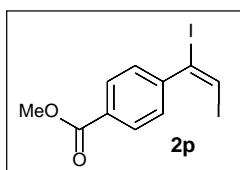
(*E*)-1-(1,2-diiodovinyl)-4-(trifluoromethyl)benzene (2o)²



The compound **2o** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.63 (d, J = 8.19 Hz, 2H, Ar- H), 7.46 (d, J = 8.07 Hz, 2H, Ar- H), 7.36 (s, 1H, vinylic- H); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 146.54, 130.74 (d, J = 33.01 Hz), 128.90, 125.53 (d, J = 3.66 Hz), 123.71 (d, J = 272.88 Hz), 93.64, 82.38.

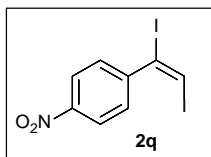
methyl (*E*)-4--(1,2-diiodovinyl)benzoate(2p)⁶



The compound **2p** was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.2:9.8) as an eluent.

^1H NMR (400 MHz, CDCl_3): δ (ppm) = 8.05-8.02 (m, 2H, Ar- H), 7.43-7.39 (m, 2H, Ar- H), 7.33 (s, 1H, vinylic- H), 3.92 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 166.21, 147.30, 130.26, 129.70, 128.49, 94.32, 81.99, 52.24

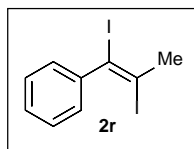
(*E*)-1-(1,2-diiodovinyl)-4-nitrobenzene (2q)



The compound **2q** was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0:10 to 0.1:9.9) as an eluent.

IR (Neat): ν_{max} 3068, 2850, 1599, 1518, 1344, 1315, 1152, 1107, 857, 822, 787, 705, and 598 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 8.24 (d, J = 8.68 Hz, 2H, Ar- H), 7.51 (d, J = 8.80 Hz, 2H, Ar- H), 7.42 (s, 1H, vinylic- H); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 149.31, 147.51, 129.57, 123.84, 92.26, 83.41; HR-MS (EI) m/z calcd for $\text{C}_8\text{H}_5\text{I}_2\text{NO}_2$, 400.84011; Found 400.84096; Anal. Calcd for $\text{C}_8\text{H}_5\text{I}_2\text{NO}_2$: C, 23.97; H, 1.26; N, 3.49. Found: C, 24.04; H, 1.23; N, 4.78.

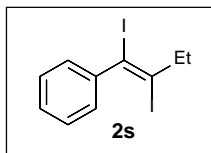
(E)-(1,2-diiodoprop-1-en-1-yl)benzene (2r)⁵



The compound **2r** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

¹H NMR (500 MHz, CDCl₃): δ (ppm) = 7.34 (t, J = 7.01 Hz, 2H, Ar-*H*), 7.28 (t, J = 7.17 Hz, 1H, Ar-*H*), 7.22 (d, J = 7.01 Hz, 2H, Ar-*H*), 2.79 (s, 1 x 3H, allylic-*H*); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 148.04, 128.40, 128.35, 128.20, 96.30, 95.47, 40.17.

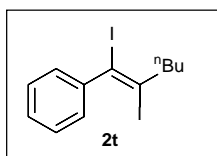
(E)-(1,2-diiodobut-1-en-1-yl)benzene (2s)²



The compound **2s** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.37-7.32 (m, 2H, Ar-*H*), 7.30-7.25 (m, 1H, Ar-*H*), 7.21-7.18 (m, 2H, Ar-*H*), 2.87 (q, J = 7.33 Hz, 1 x 2H, allylic-*H*), 1.17 (t, J = 7.45 Hz, 1 x 3H, aliphatic-*H*); ¹³C NMR (125 MHz, CDCl₃): δ (ppm) = 148.00, 128.44, 128.35, 128.15, 106.49, 93.61, 44.84, 12.90.

(E)-(1,2-diiodohex-1-en-1-yl)benzene (2t)¹

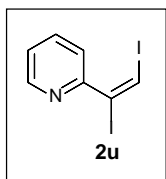


The compound **2t** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.37-7.32 (m, 2H, Ar-*H*), 7.30-7.27 (m, 1H, Ar-*H*), 7.21-7.18 (m, 2H, Ar-*H*), 2.85 (t, J = 7.58 Hz, 1 x 2H, allylic-*H*), 1.70-1.62 (m, 1 x 2H, aliphatic-*H*), 1.51-1.42 (m, 1 x 2H, aliphatic-*H*), 1.00 (t, J = 7.33 Hz, 1 x 3H, aliphatic-*H*);

^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 148.18, 128.44, 128.35, 128.13, 105.28, 94.26, 50.26, 30.52, 21.61, 14.09.

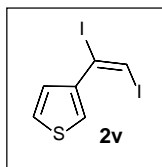
(E)-2-(1,2-diiodovinyl)pyridine (2u)



The compound **2u** was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0.2:9.8 to 0.5:9.5) as an eluent.

IR (Neat): ν_{max} 3046, 2923, 1571, 1454, 1279, 1160, 989, 793, 744, 682 and 598 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 8.68 (d, $J = 4.15$ Hz, 1H, Ar-*H*), 7.56 (ddd, $J = 1.83$, 7.82, 15.52 Hz, 1H, Ar-*H*), 7.47 (d, $J = 7.94$ Hz, 1H, Ar-*H*), 7.43 (s, 1H, vinylic-*H*), 7.27-7.23 (m, 1H, Ar-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 158.94, 149.74, 136.45, 123.53, 123.40, 96.53, 81.73; HR-MS (EI) m/z calcd for $\text{C}_7\text{H}_5\text{I}_2\text{N}$, 356.85400; Found 356.85113; Anal. Calcd for $\text{C}_7\text{H}_5\text{I}_2\text{N}$: C, 23.56; H, 1.41; N, 3.92. Found: C, 23.68; H, 1.71; N, 5.12.

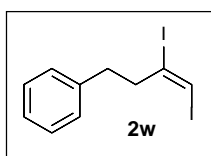
(E)-3-(1,2-diiodovinyl)thiophene (2v)⁶



The compound **2v** was prepared following general procedure and purified by column chromatography using EtOAc/hexanes (0.2:9.8 to 0.5:9.5) as an eluent.

^1H NMR (500 MHz, CDCl_3): δ (ppm) = 7.58-7.56 (m, 1H, Ar-*H*), 7.33-7.30 (m, 1H, Ar-*H*), 7.29-7.25 (m, 1H, Ar-*H*), 7.24 (s, 1H, vinylic-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 142.17, 128.42, 126.46, 125.19, 90.27, 79.67.

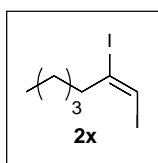
(E)-(3,4-diiodobut-3-en-1-yl)benzene (2w)



The compound **2w** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): ν_{\max} 3067, 3026, 2923, 2854, 1598, 1494, 1449, 1260, 1216, 1161, 1076, 1007, 903, 772, 747, 698, and 557 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 7.32-7.19 (m, 5H, Ar-*H*), 6.84 (s, 1H, vinylic-*H*), 2.82 (s, 2 x 2H, benzylic-*H* + allylic-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 139.52, 128.73, 128.40, 126.34, 102.35, 80.09, 46.81, 34.10; HR-MS (EI) m/z calcd for $\text{C}_{10}\text{H}_{10}\text{I}_2$, 383.88343; Found 383.88719; Anal. Calcd for $\text{C}_{10}\text{H}_{10}\text{I}_2$: C, 31.28; H, 2.63. Found: C, 31.96; H, 2.44.

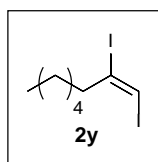
(*E*)-1,2-diiodohept-1-ene (2x)³



The compound **2x** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

^1H NMR (400 MHz, CDCl_3): δ (ppm) = 6.80 (s, 1H, vinylic-*H*), 2.50 (t, $J = 7.21$ Hz, 1 x 2H, allylic-*H*), 1.54 (quint, $J = 7.45$ Hz, 1 x 2H, aliphatic-*H*), 1.40-1.28 (m, 2 x 2H, aliphatic-*H*), 0.91 (t, $J = 6.96$ Hz, 1 x 3H, aliphatic-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 104.44, 78.90, 44.60, 30.29, 27.81, 22.47, 13.97.

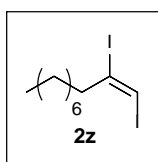
(*E*)-1,2-diiodooct-1-ene (2y)⁴



The compound **2y** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

^1H NMR (500 MHz, CDCl_3): δ (ppm) = 6.80 (s, 1H, vinylic-*H*), 2.50 (t, $J = 7.32$ Hz, 1 x 2H, allylic-*H*), 1.53 (quint, $J = 7.32$ Hz, 1 x 2H, aliphatic-*H*), 1.37-1.30 (m, 3 x 2H, aliphatic-*H*), 0.90 (t, $J = 6.86$ Hz, 1x 3H, aliphatic-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 104.43, 78.86, 44.65, 31.59, 28.11, 27.82, 22.52, 14.05.

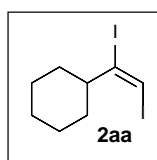
(*E*)-1,2-diiodododec-1-ene (2z)⁵



The compound **2z** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

^1H NMR (400 MHz, CDCl_3): δ (ppm) = 6.80 (s, 1H, vinylic-*H*), 2.50 (t, $J = 7.21$ Hz, 1 x 2H, allylic-*H*), 1.53 (quint, $J = 7.45$ Hz, 1 x 2H, aliphatic-*H*), 1.34-1.26 (m, 5 x 2H, aliphatic-*H*), 0.88 (t, $J = 6.72$ Hz, 1 x 3H, aliphatic-*H*); ^{13}C NMR (125 MHz, CDCl_3): δ (ppm) = 104.44, 78.86, 44.63, 31.82, 29.36, 29.15, 28.16, 28.14, 22.65, 14.12.

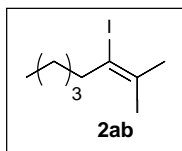
(*E*)-(1,2-diiodovinyl)cyclohexane (**2aa**)



The compound **2aa** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): ν_{max} 3067, 2925, 2851, 2663, 1533, 1446, 1349, 1222, 1133, 1057, 949, 892, 767, 655, and 574 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 6.78 (s, 1H, vinylic-*H*), 2.12-2.05 (m, 1H, allylic-*H*), 1.83-1.55 (m, 5H, cyclohexyl-*H*), 1.49-1.29 (m, 4H, cyclohexyl-*H*), 1.21-1.13 (m, 1H, cyclohexyl-*H*); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm) = 114.34, 76.37, 48.78, 32.28, 25.44, 25.18; HR-MS (EI) m/z calcd for $\text{C}_8\text{H}_{12}\text{I}_2$, 361.90100; Found 361.90284; Anal. Calcd for $\text{C}_8\text{H}_{12}\text{I}_2$: C, 26.54; H, 3.34. Found: C, 26.15; H, 3.15.

(*E*)-2,3-diiodooct-2-ene (**2ab**)

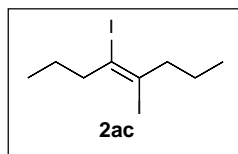


The compound **2ab** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

IR (Neat): ν_{max} 2925, 2858, 1706, 1457, 1375, 1058, 980, 728, and 543 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ (ppm) = 2.68-2.63 (m, 1 x 2H, allylic-*H*), 2.60 (s, 1 x 3H, allylic-*H*), 1.54 (quint, $J = 5.01$ Hz, 1 x 2H, aliphatic-*H*), 1.42-1.25 (m, 2 x 2H, aliphatic-*H*), 0.91 (t, $J = 6.84$

Hz, 1 x 3H, aliphatic-*H*); ^{13}C NMR (125 MHz, CDCl_3): δ (ppm) = 103.06, 92.85, 50.76, 40.34, 30.49, 27.90, 22.51, 13.99; HR-MS (EI) m/z calcd for $\text{C}_8\text{H}_{14}\text{I}_2$, 363.91700; Found 363.91849; Anal. Calcd for $\text{C}_8\text{H}_{14}\text{I}_2$: C, 26.40; H, 3.88. Found: C, 26.92; H, 4.03.

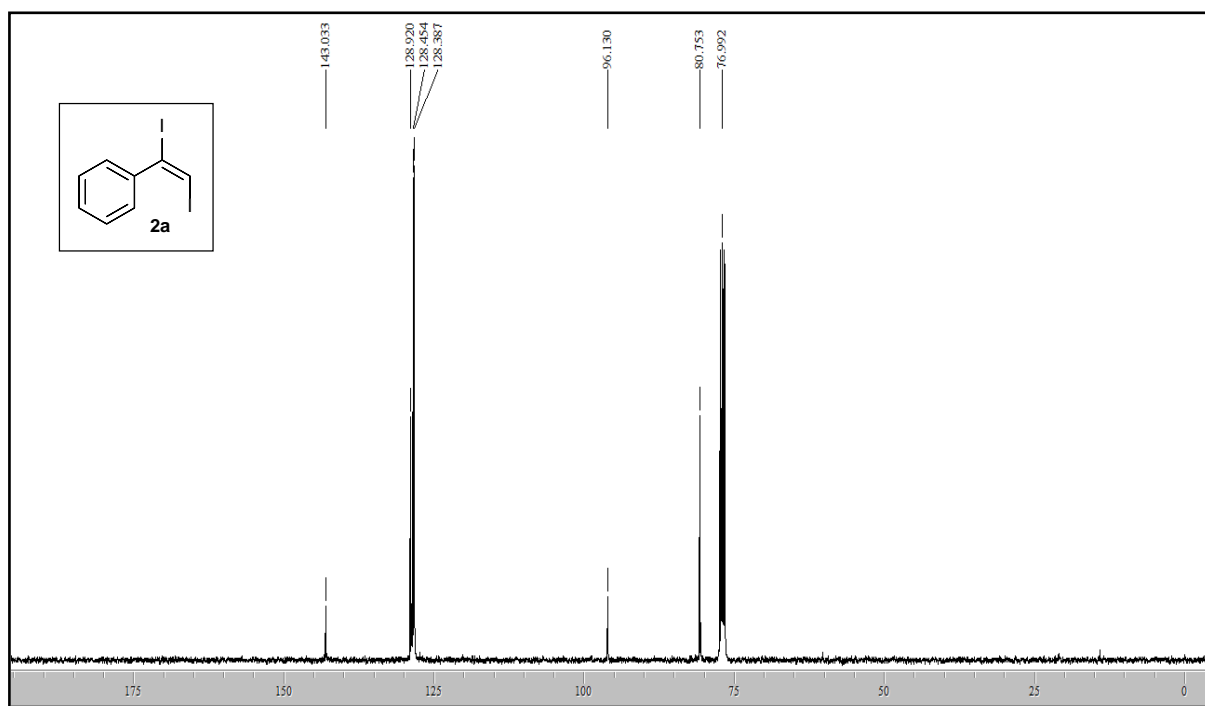
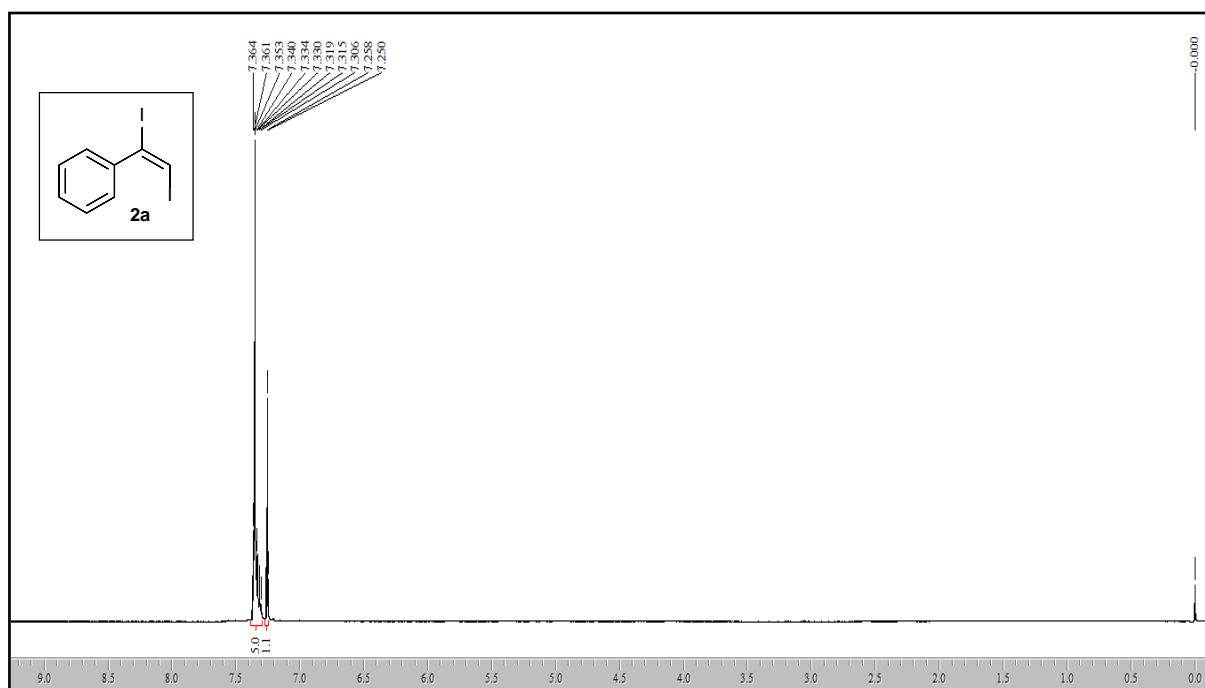
(*E*)-4,5-diiodooct-4-ene (2ac)¹

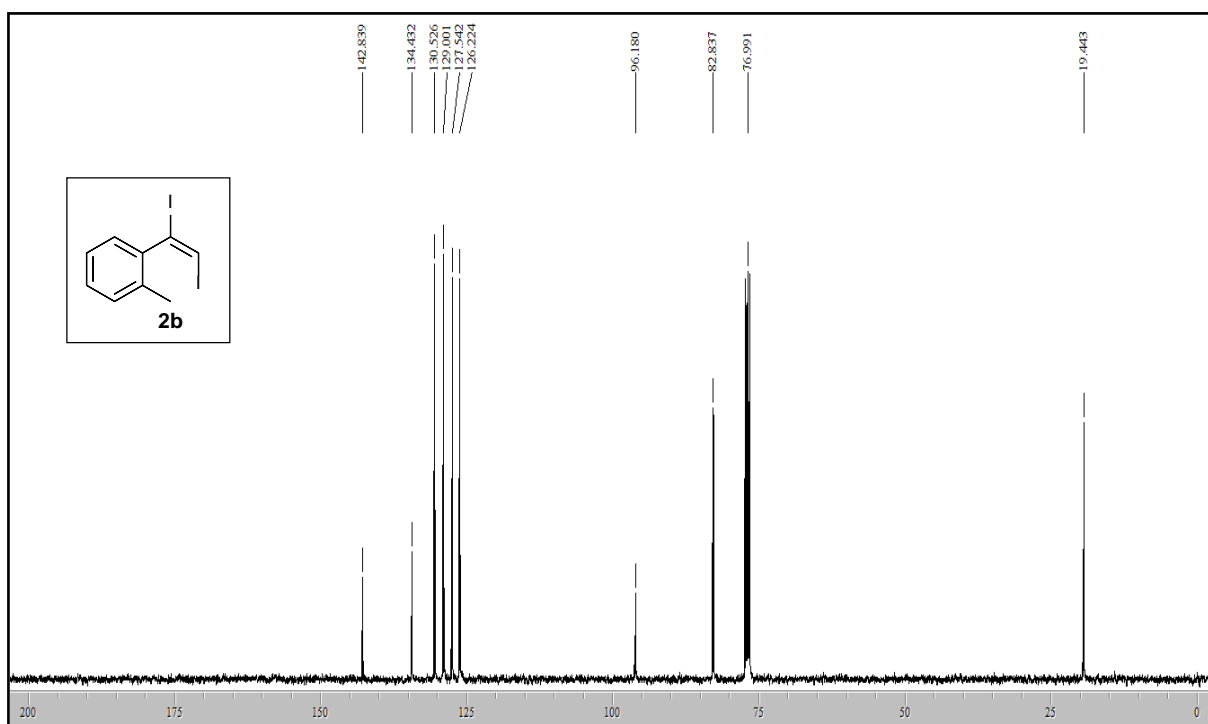
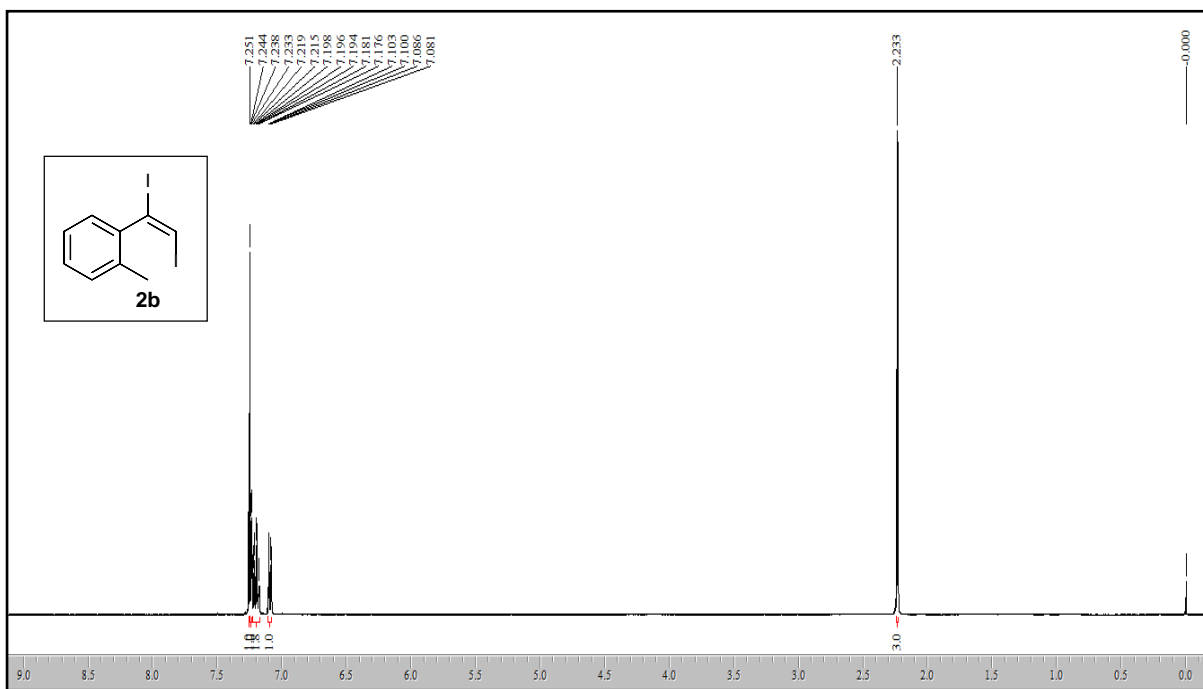


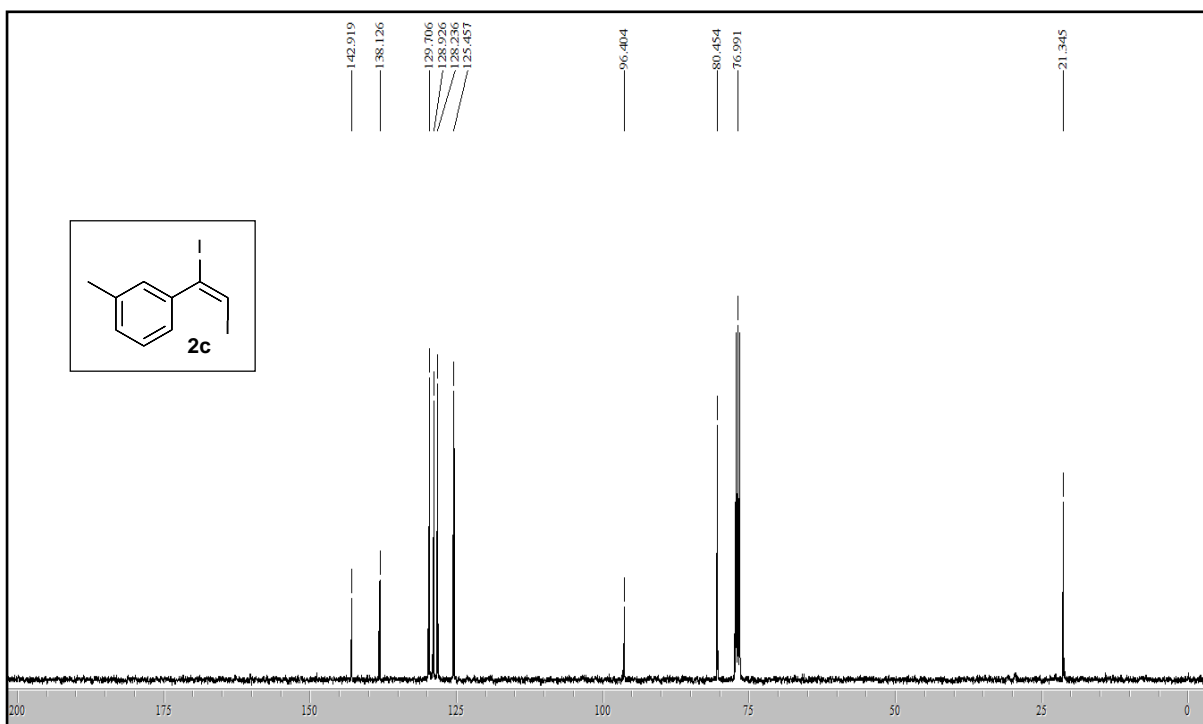
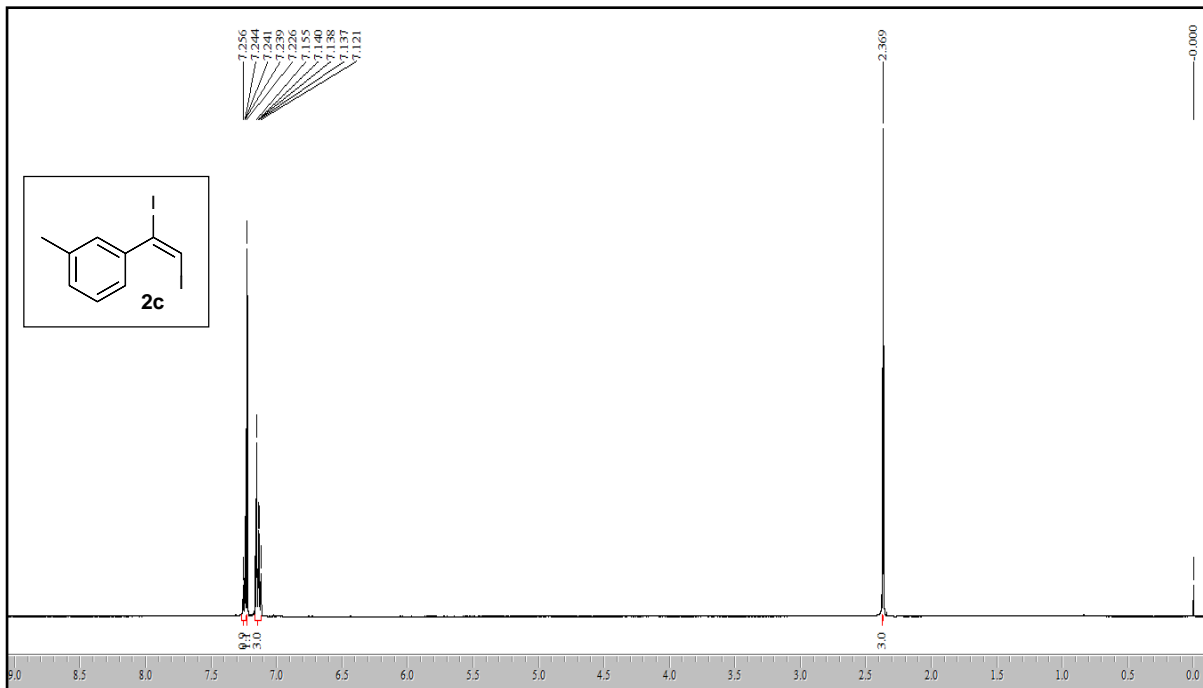
The compound **2ac** was prepared following general procedure and purified by column chromatography using hexanes as an eluent.

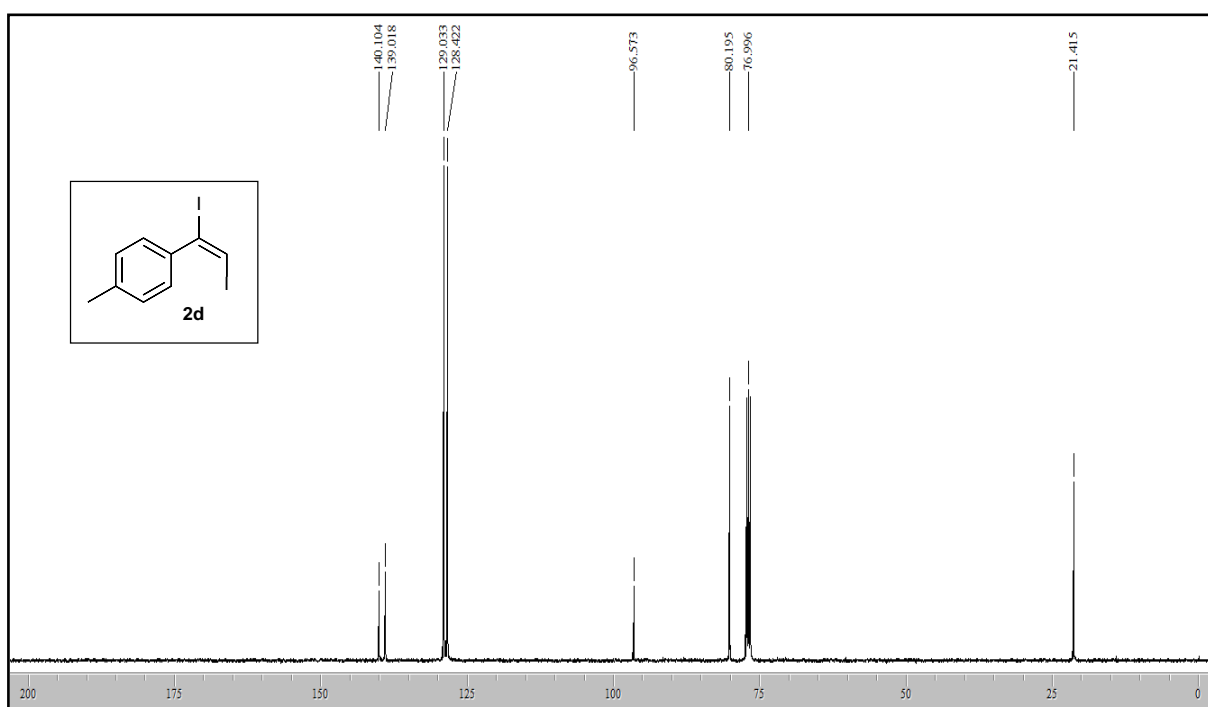
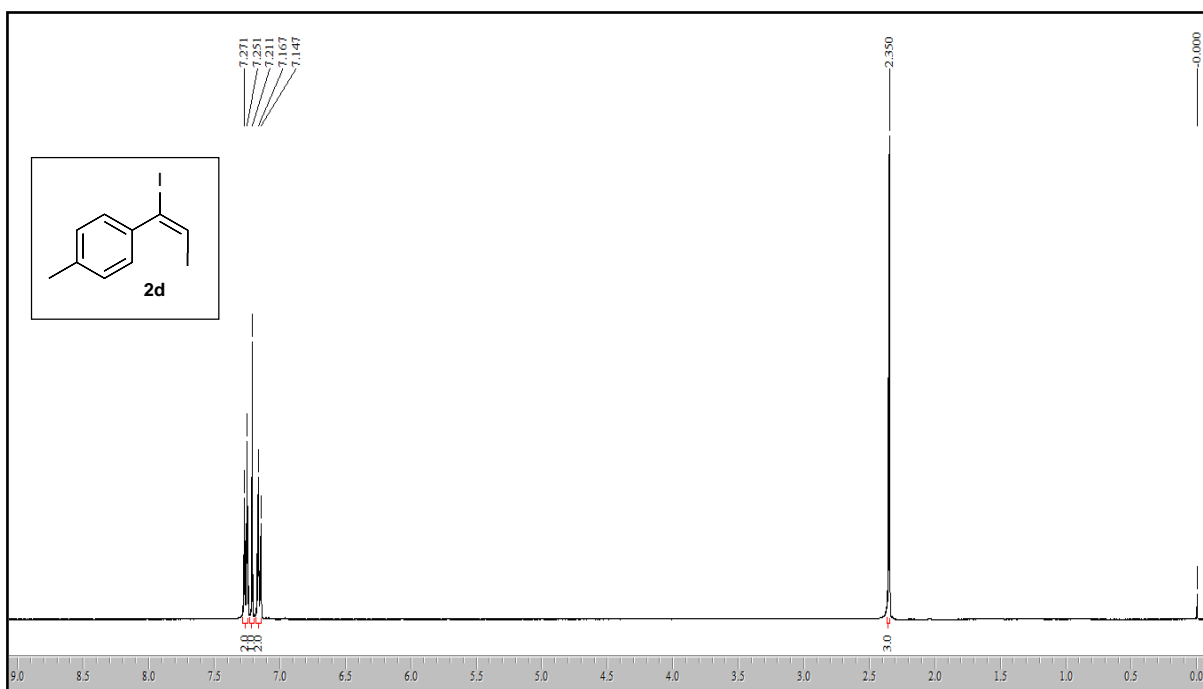
^1H NMR (400 MHz, CDCl_3): δ (ppm) = 2.68 (t, J = 7.33 Hz, 2 x 2H, allylic-*H*), 1.64-1.54 (m, 2 x 2H, aliphatic-*H*), 0.96 (t, J = 7.33 Hz, 2 x 3H, aliphatic-*H*); ^{13}C NMR (125 MHz, CDCl_3): δ (ppm) = 101.96, 52.53, 21.61, 12.80.

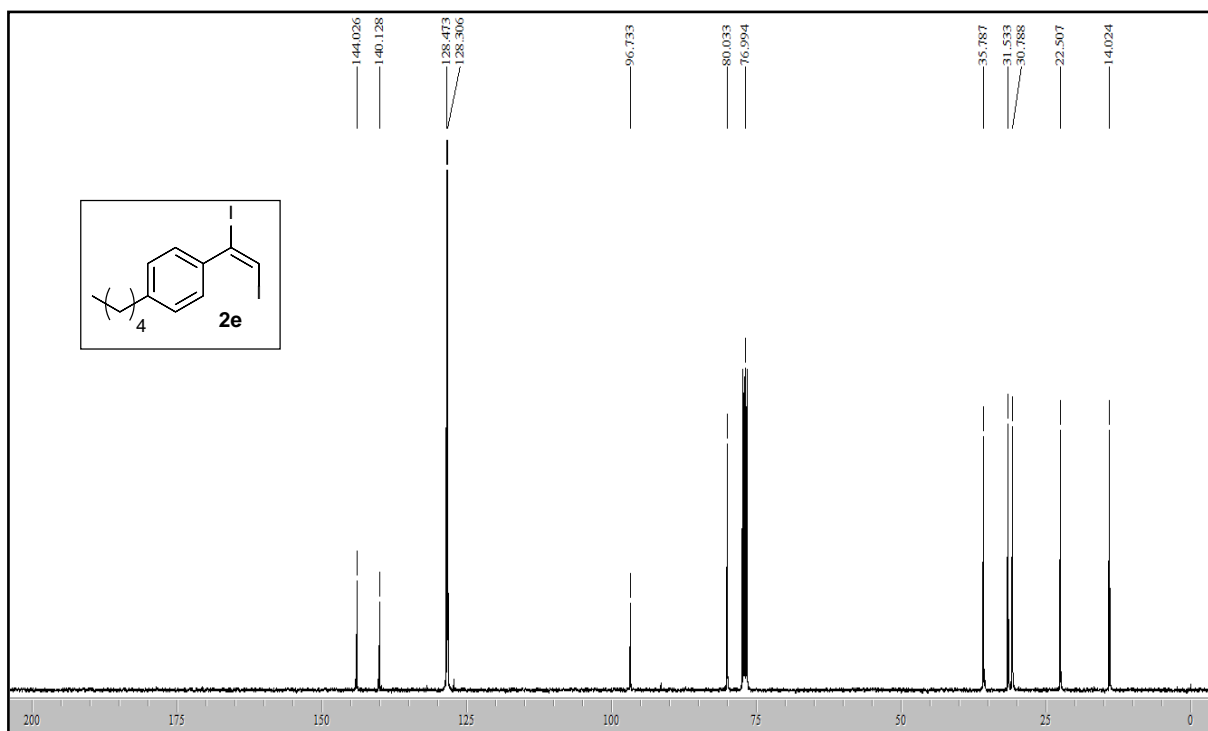
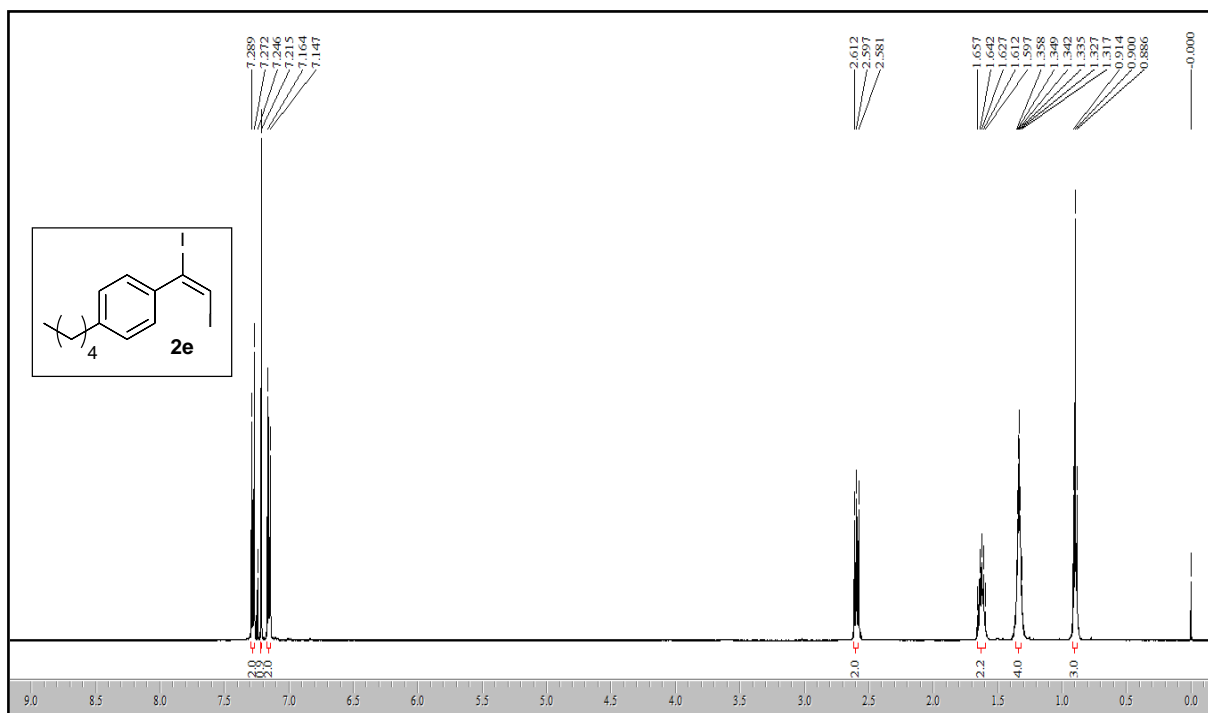
5. Copies of ^1H and ^{13}C NMR spectra of all products:

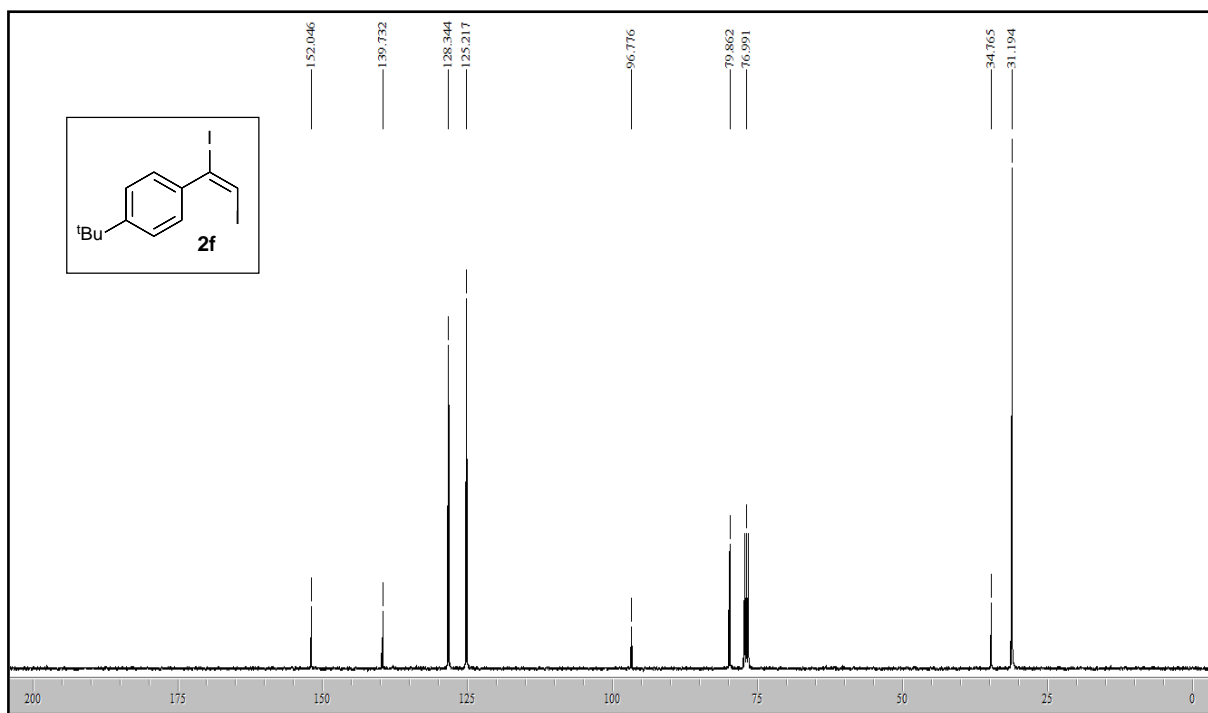
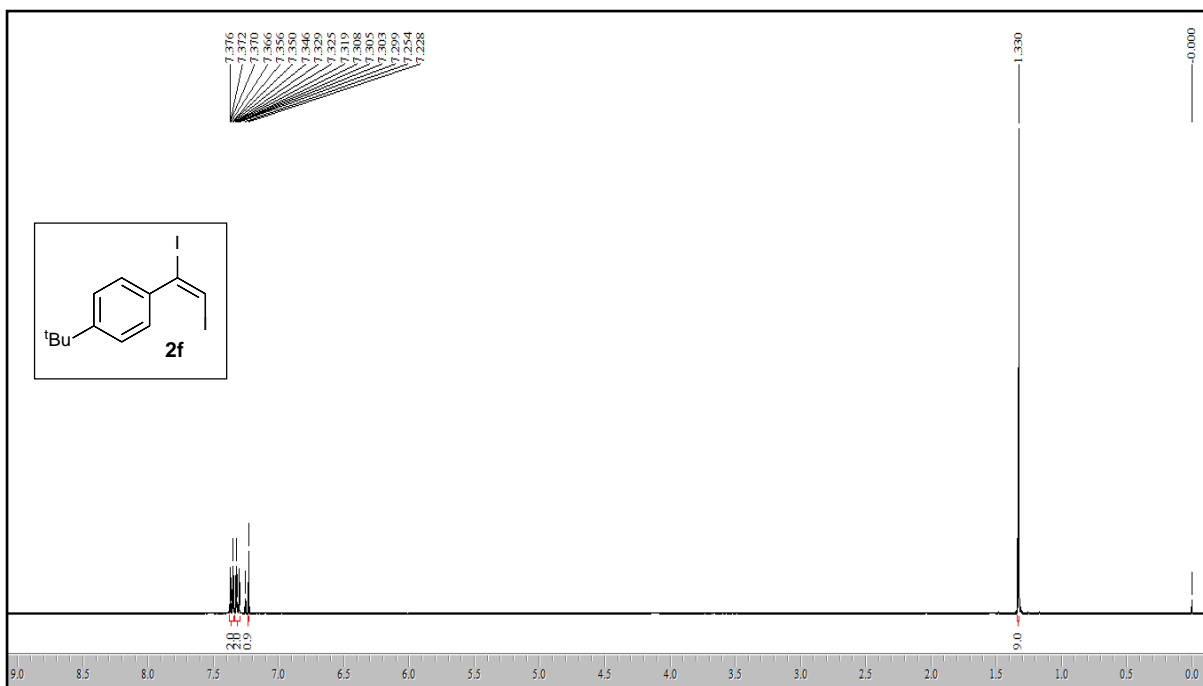


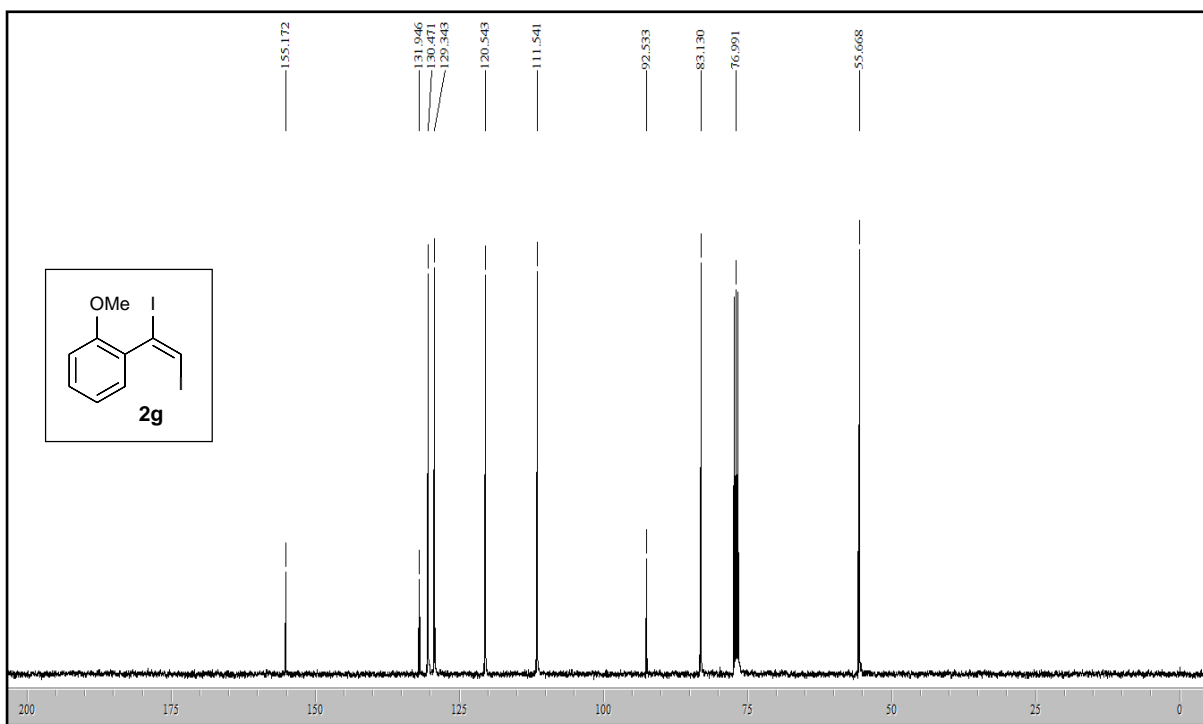
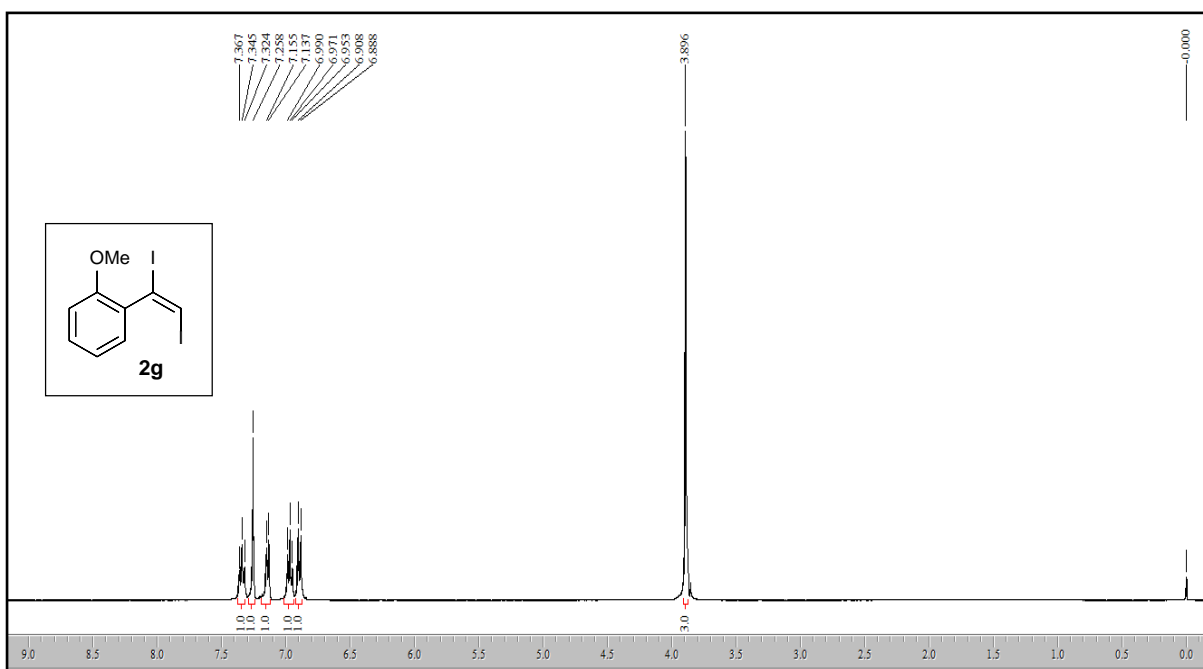


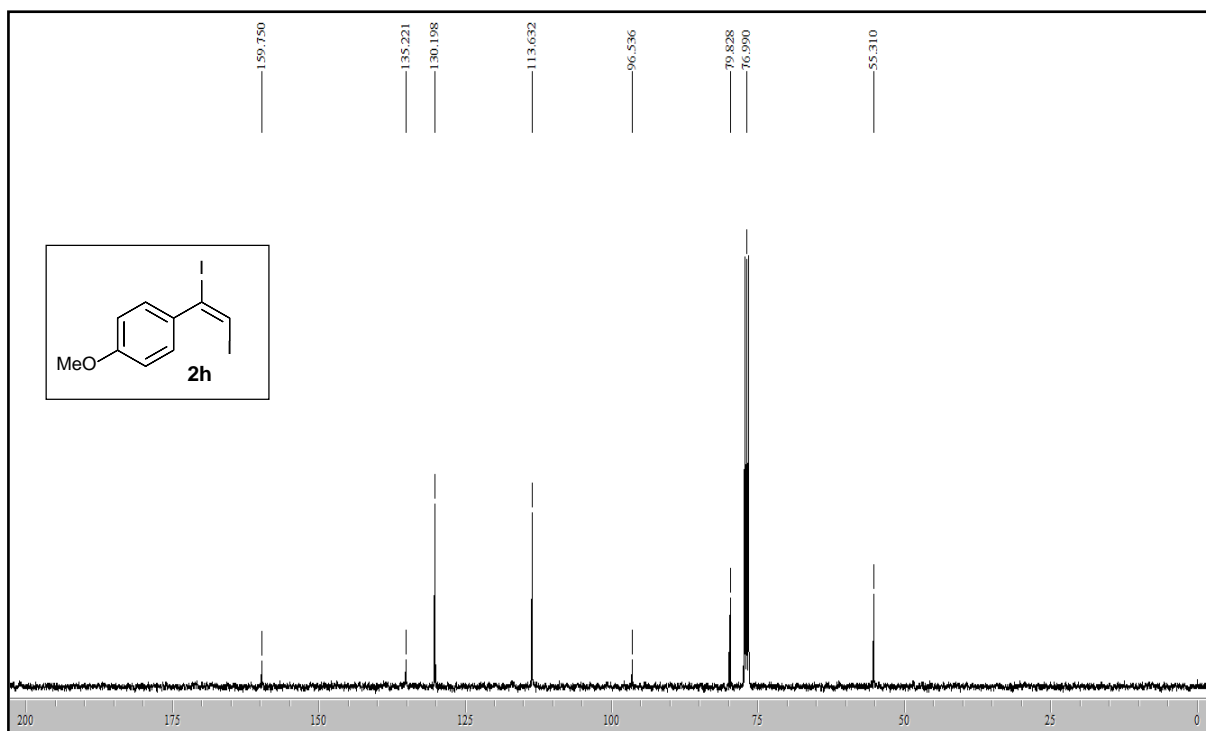
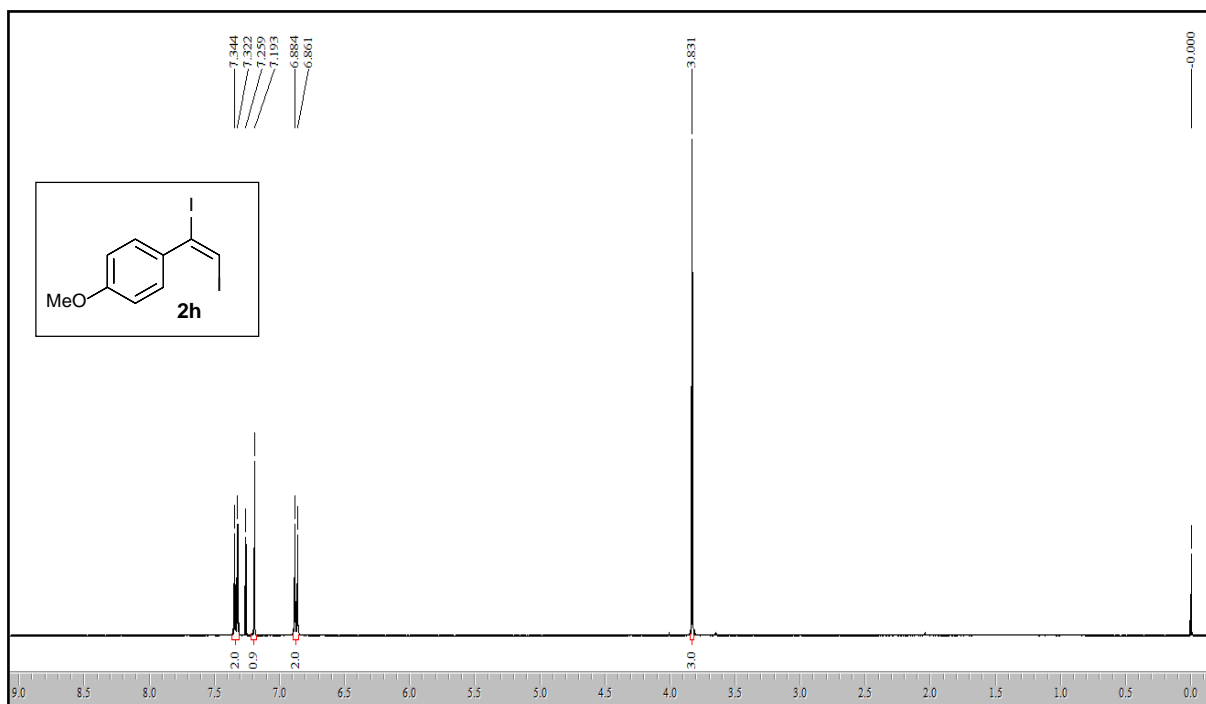


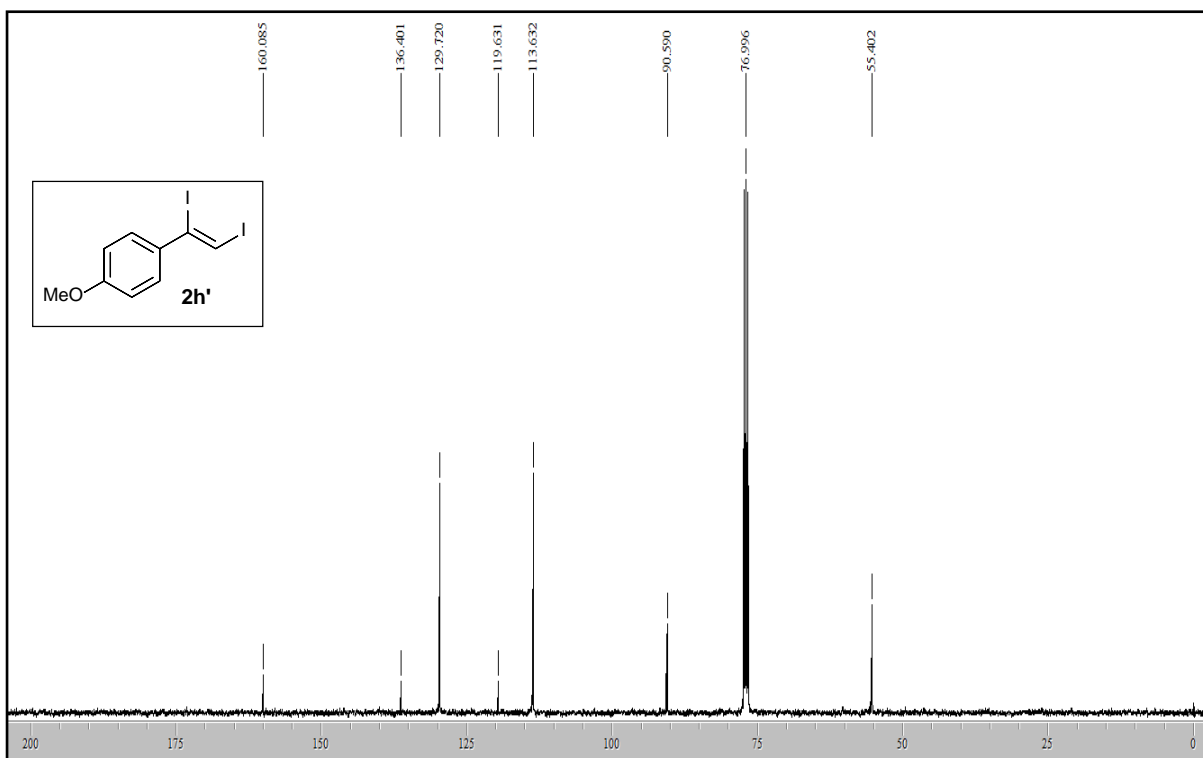
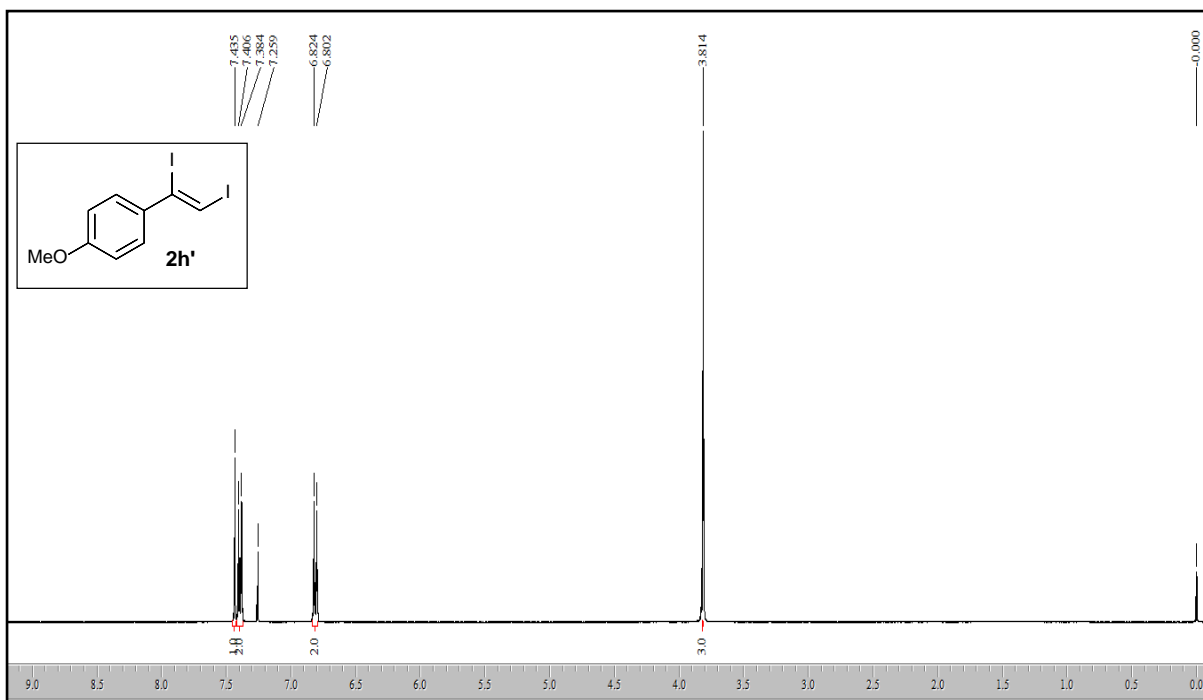


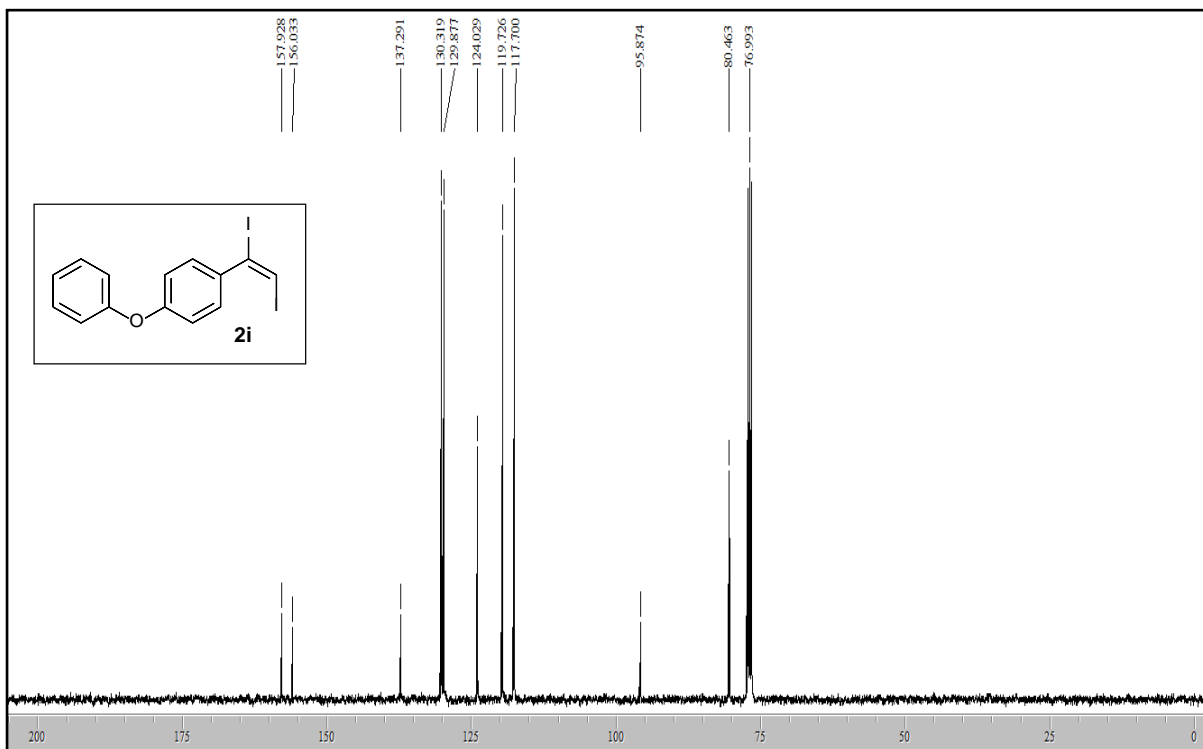
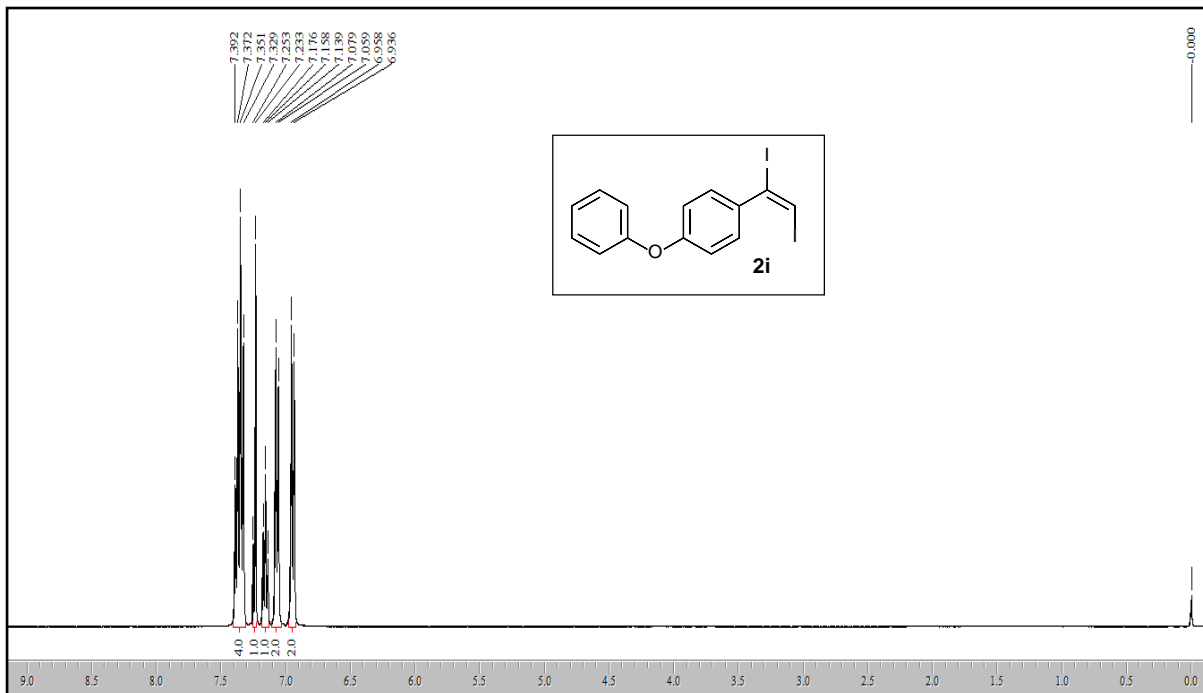


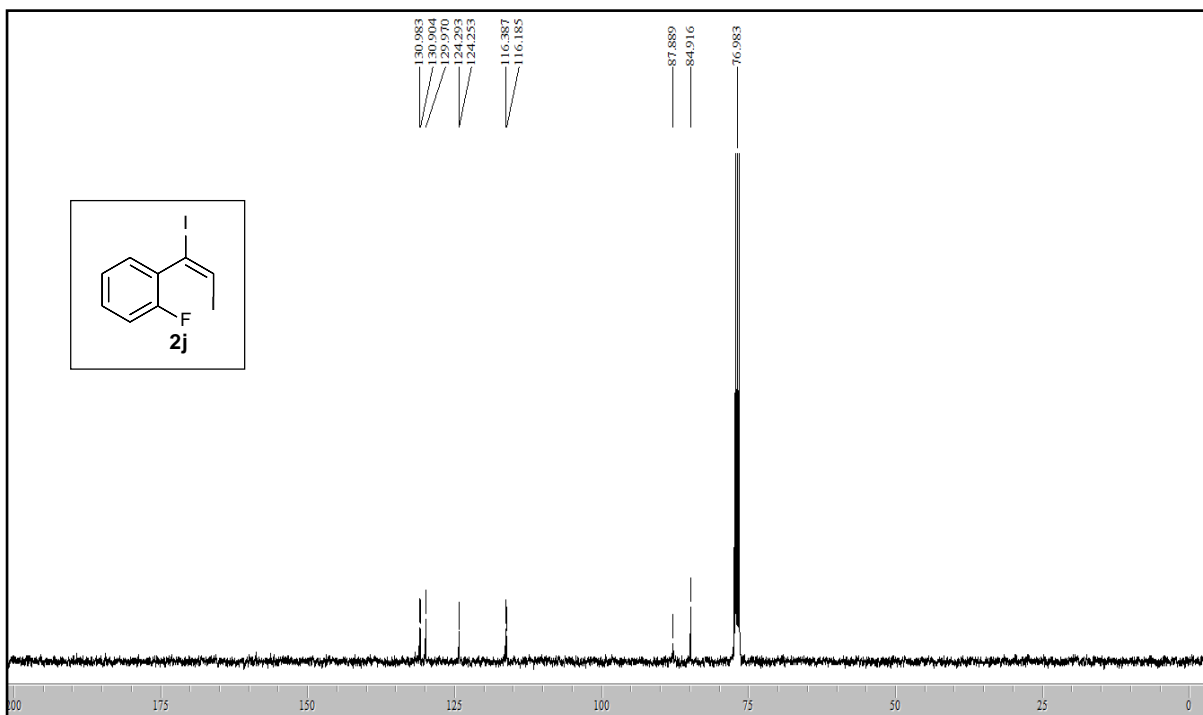
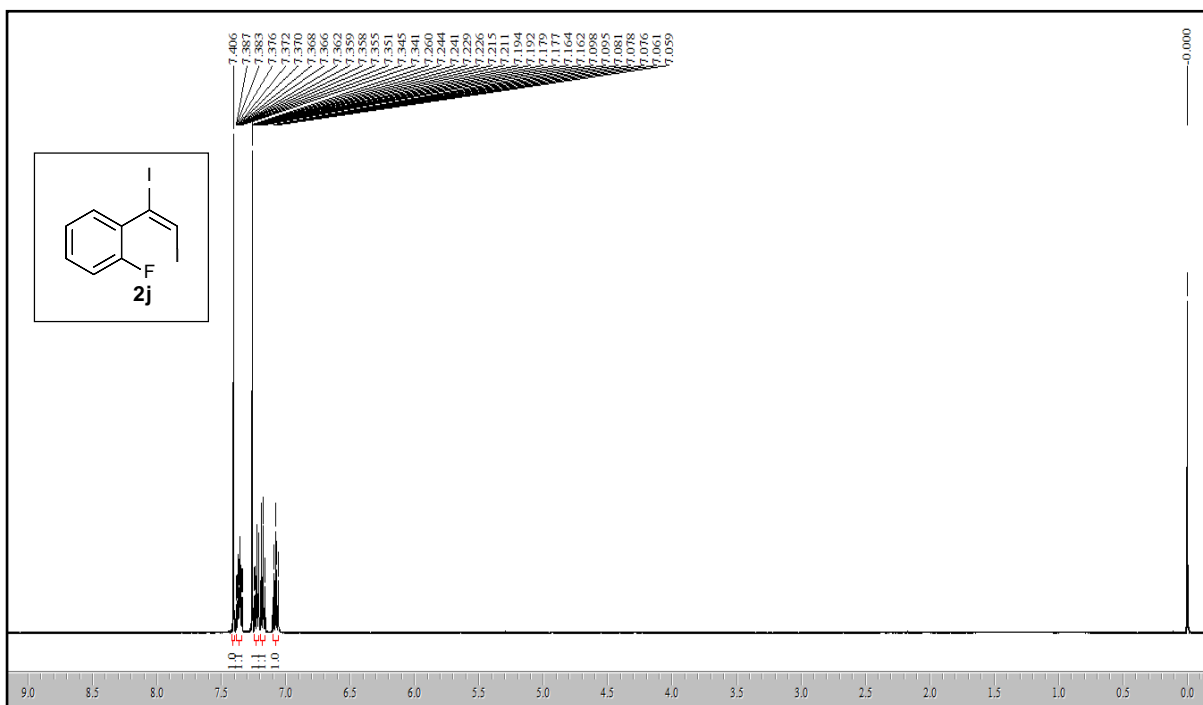


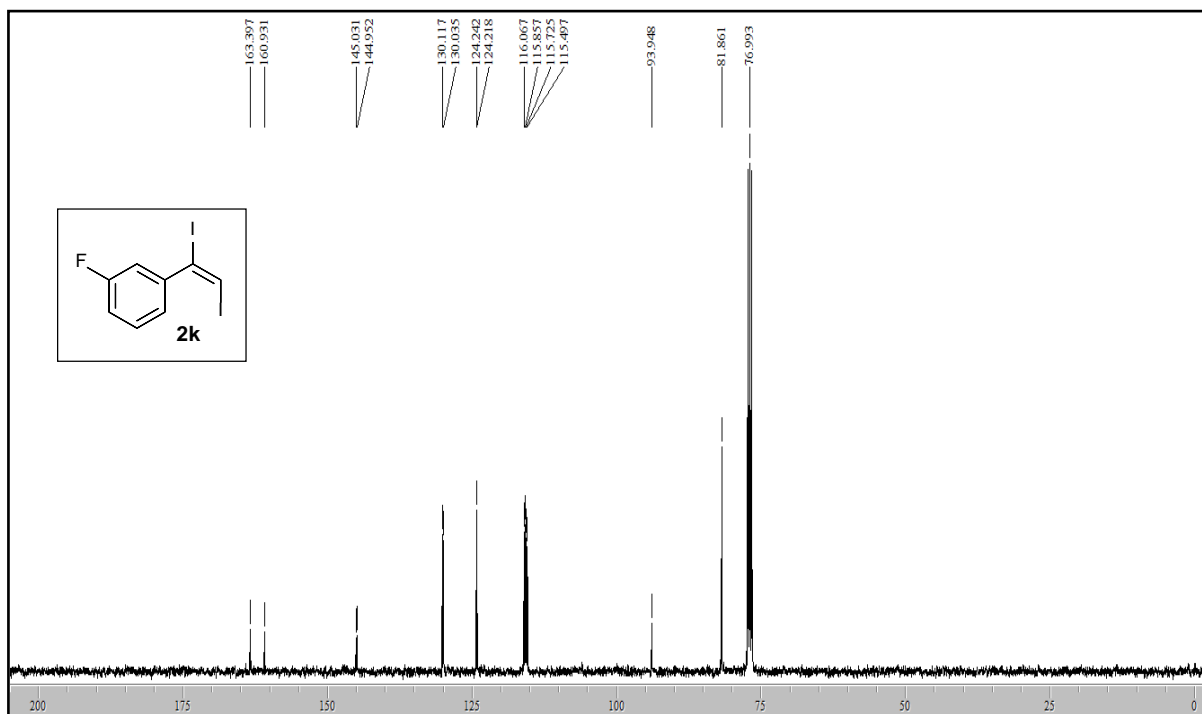
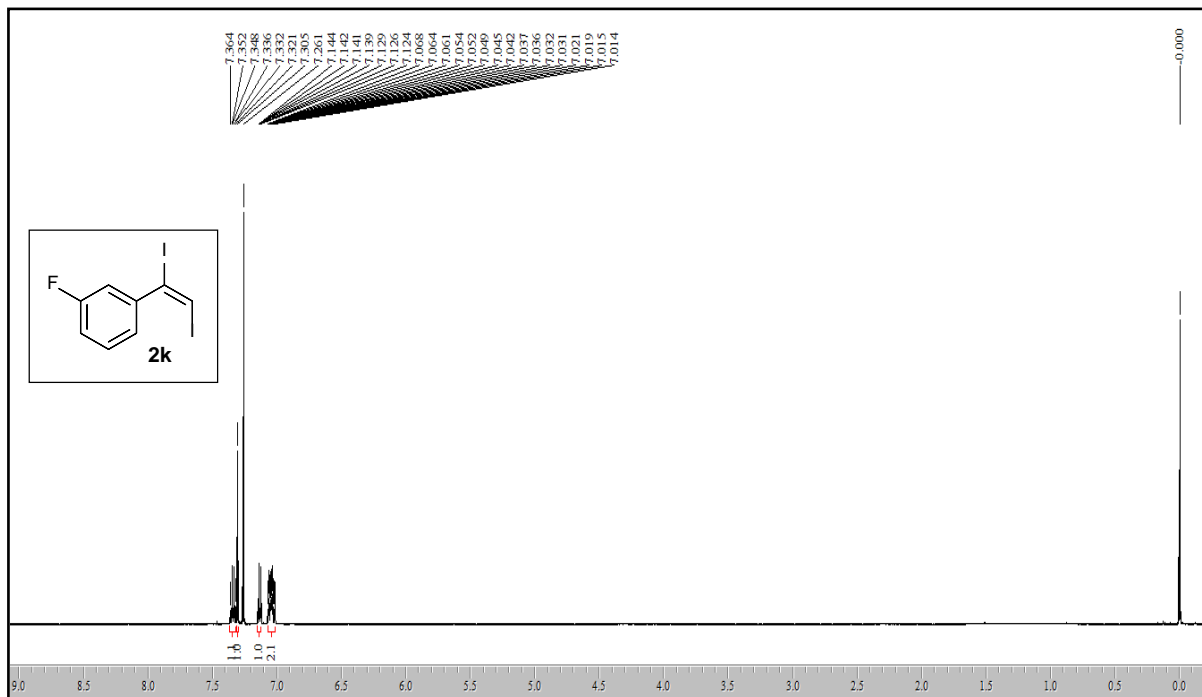


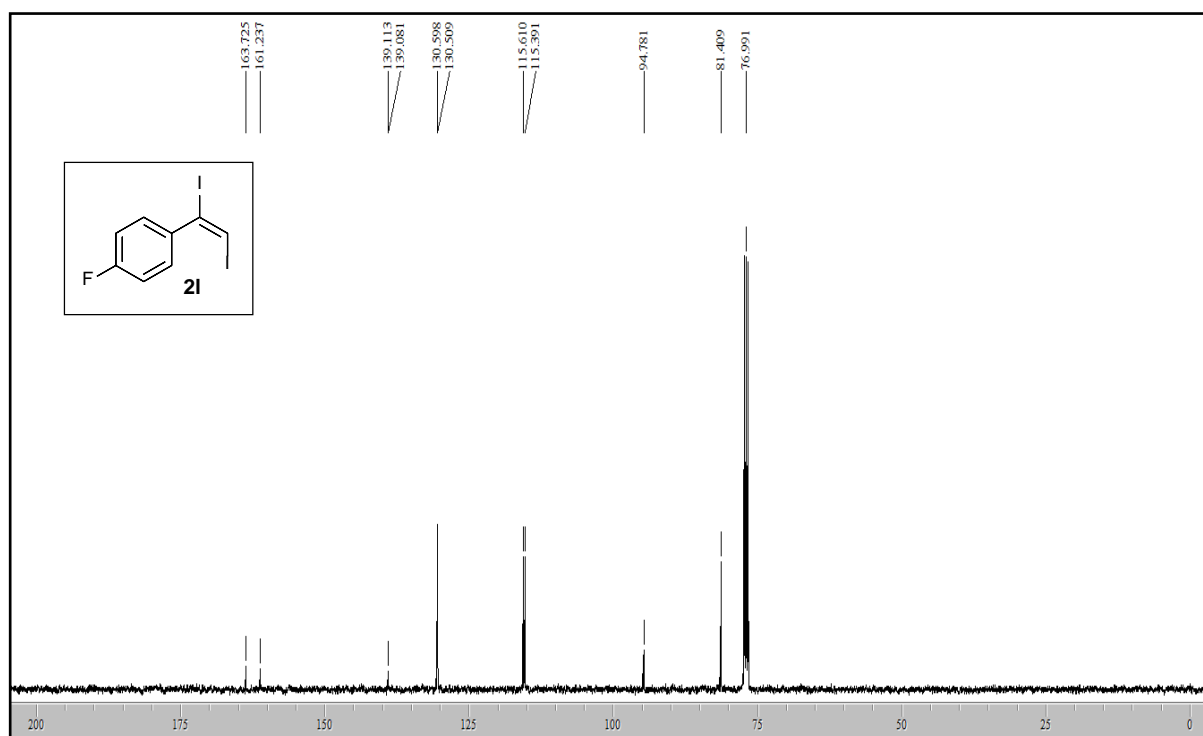
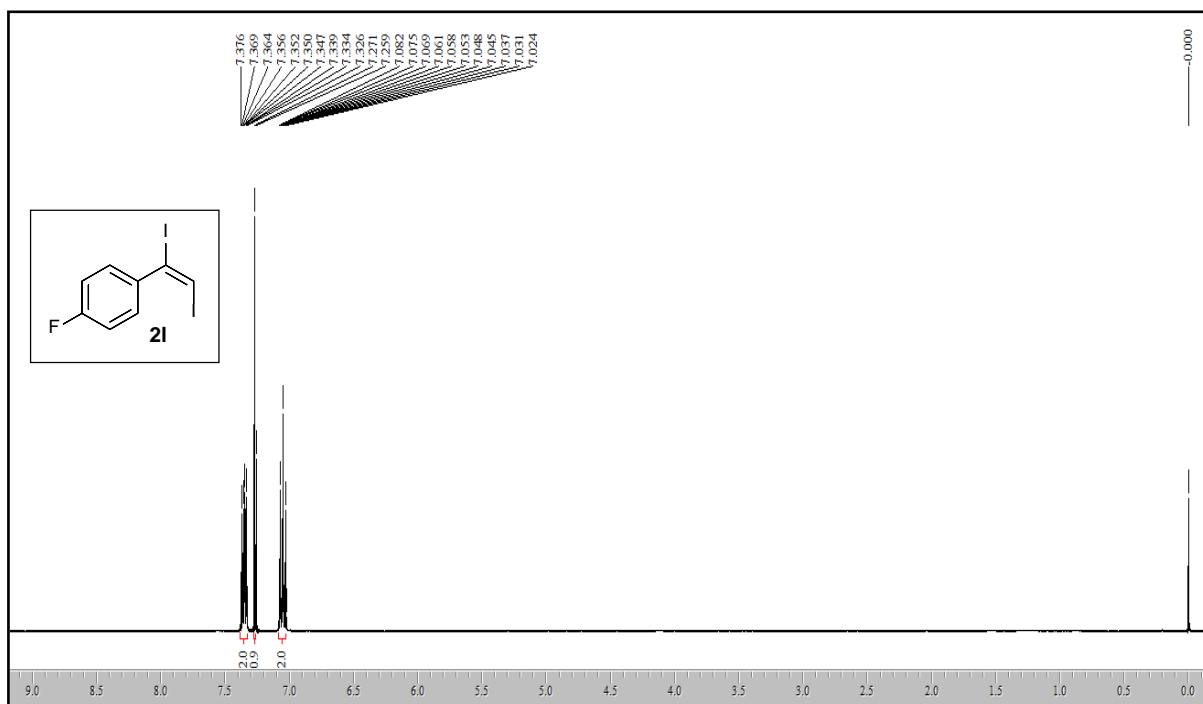


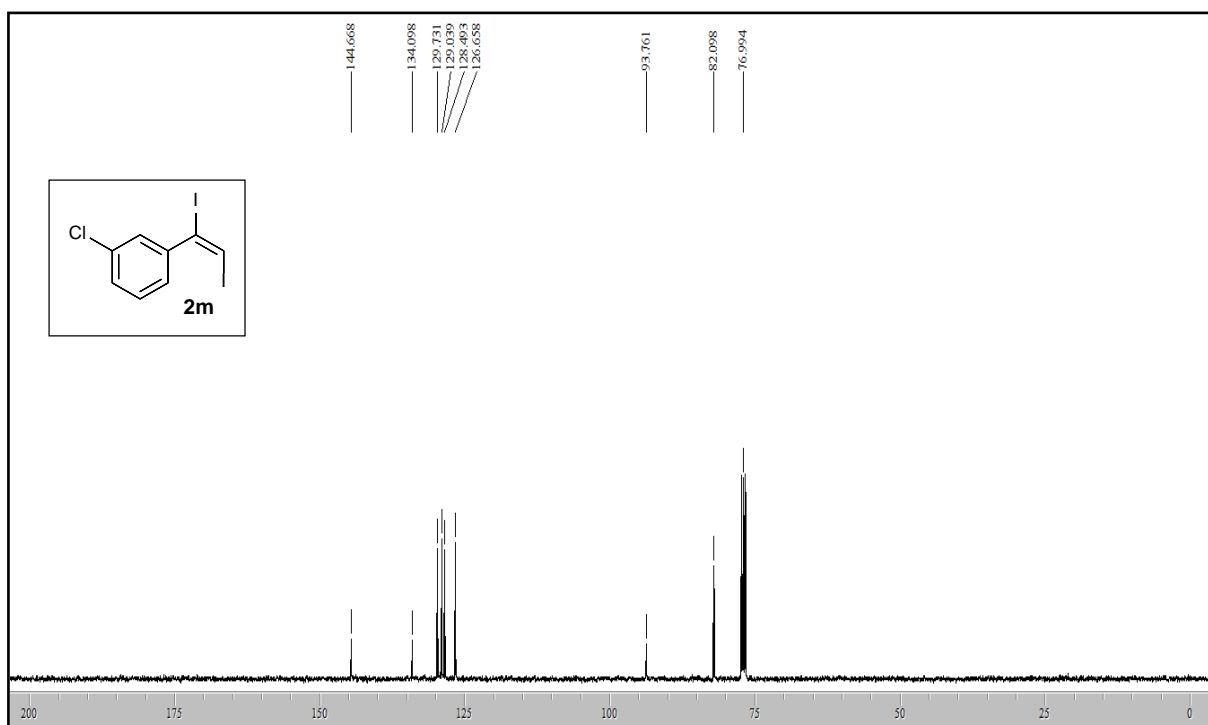
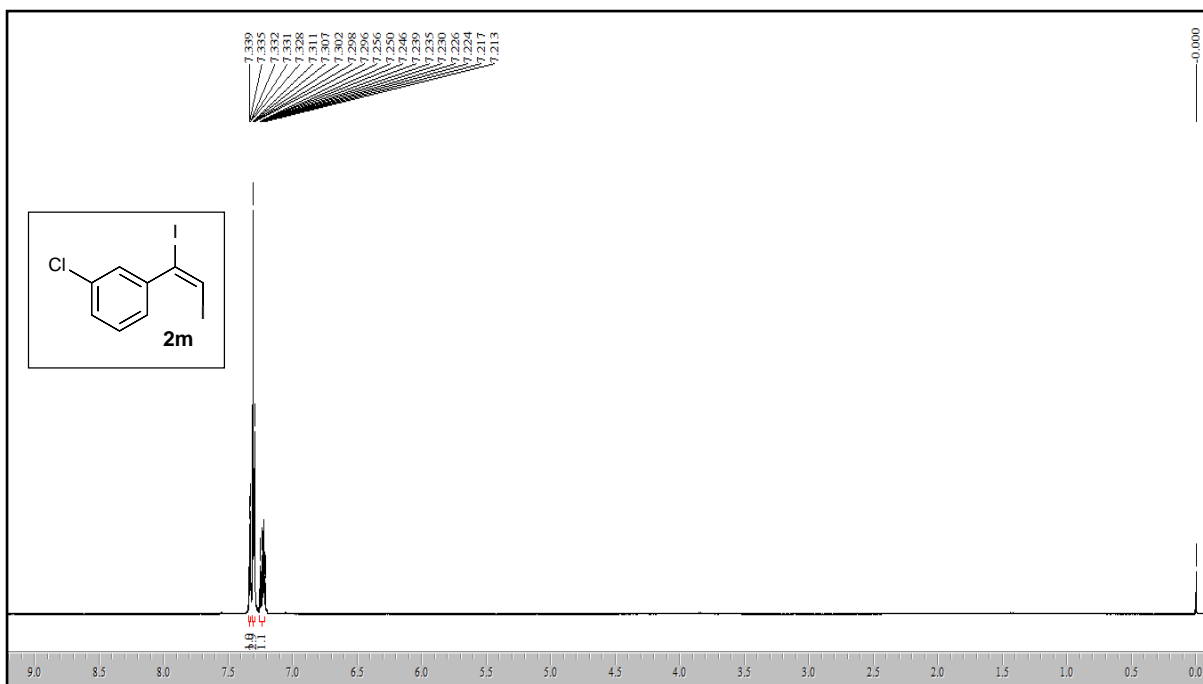


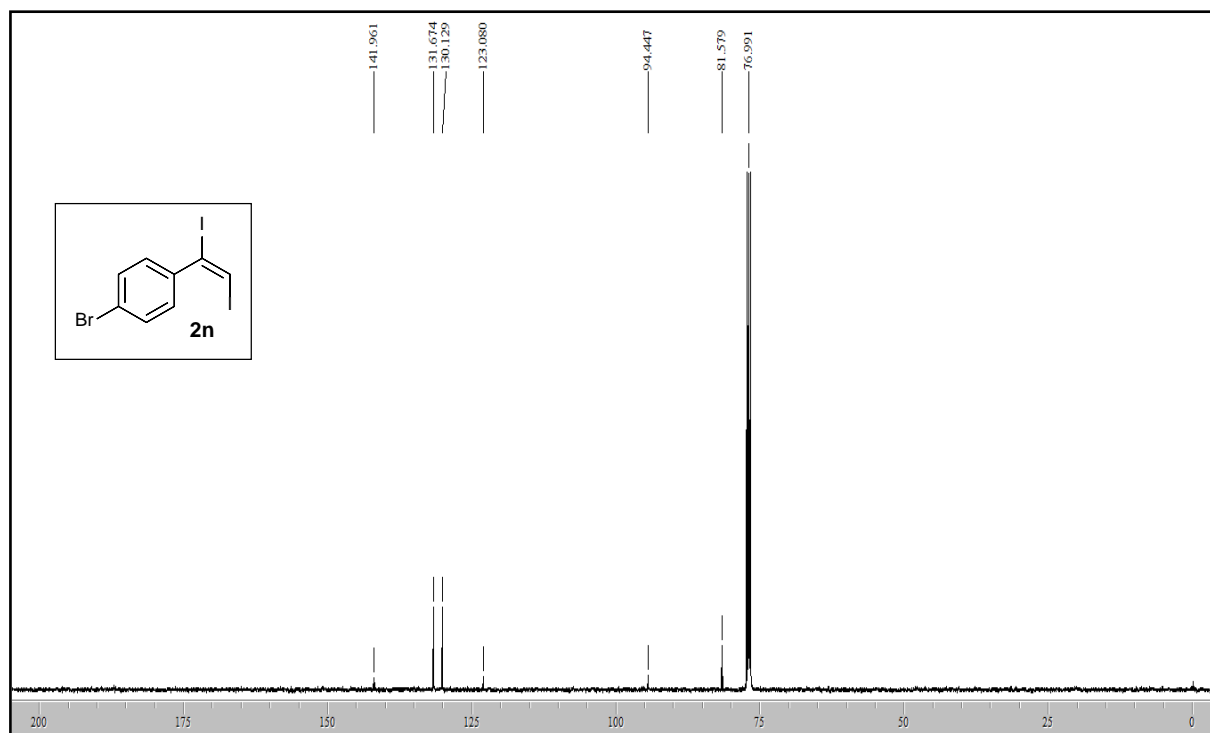
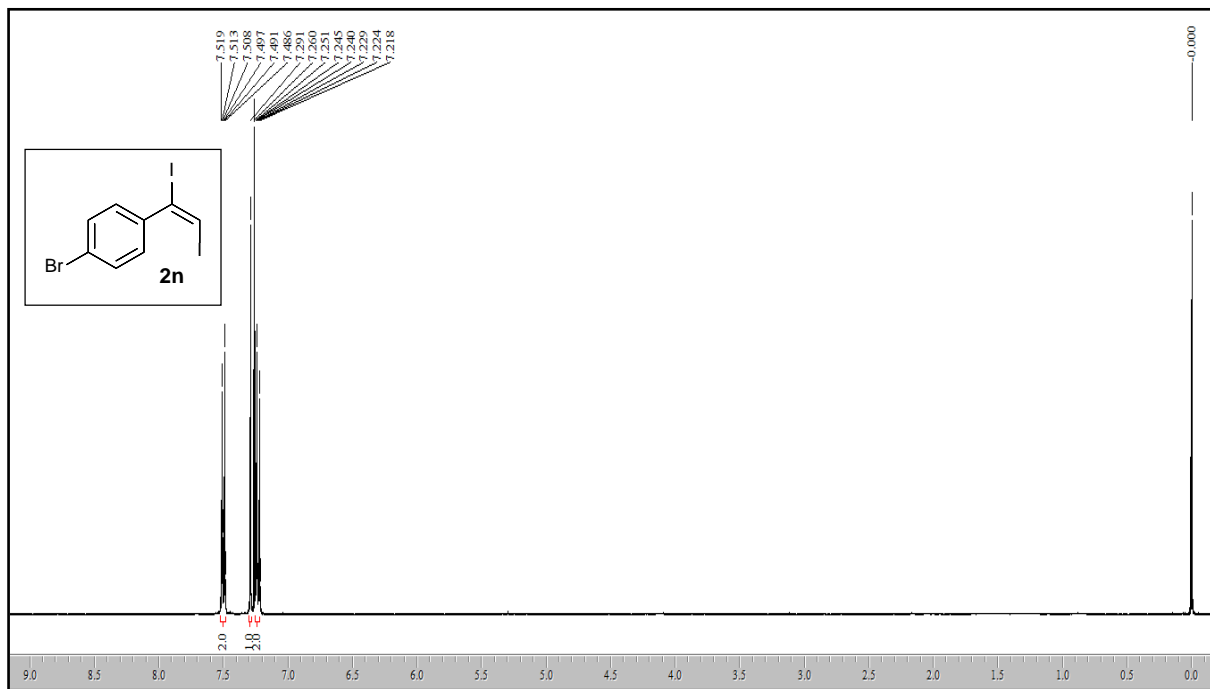


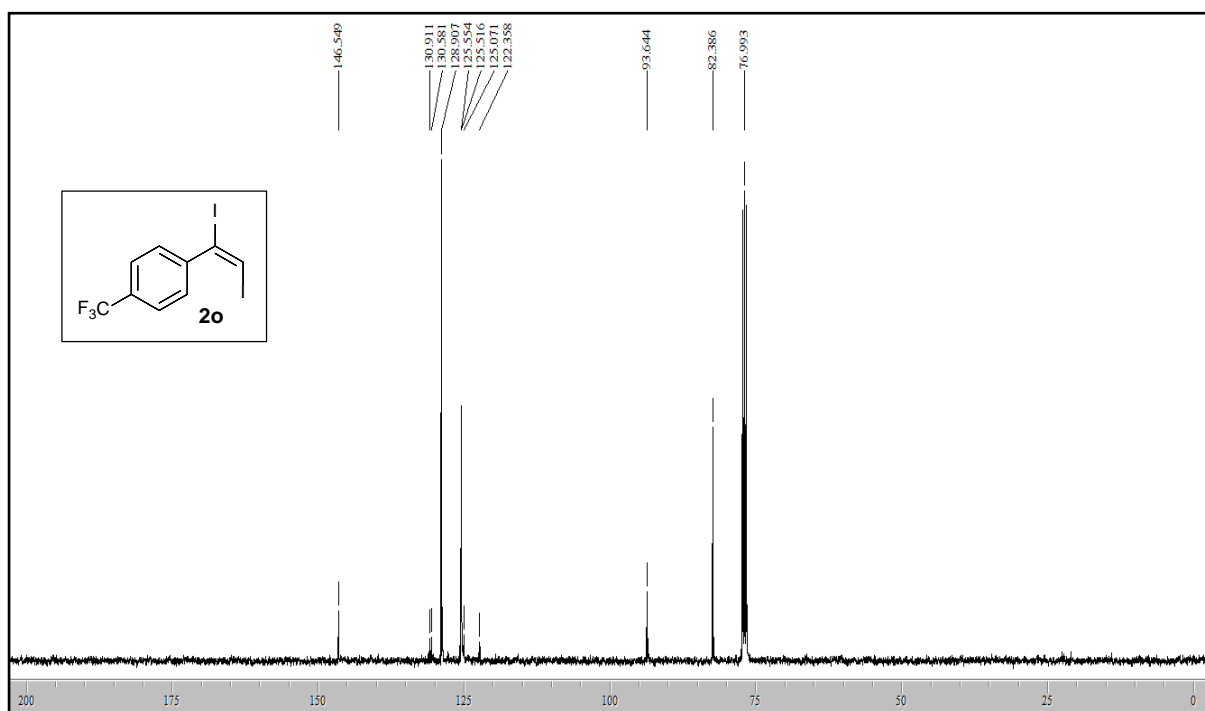


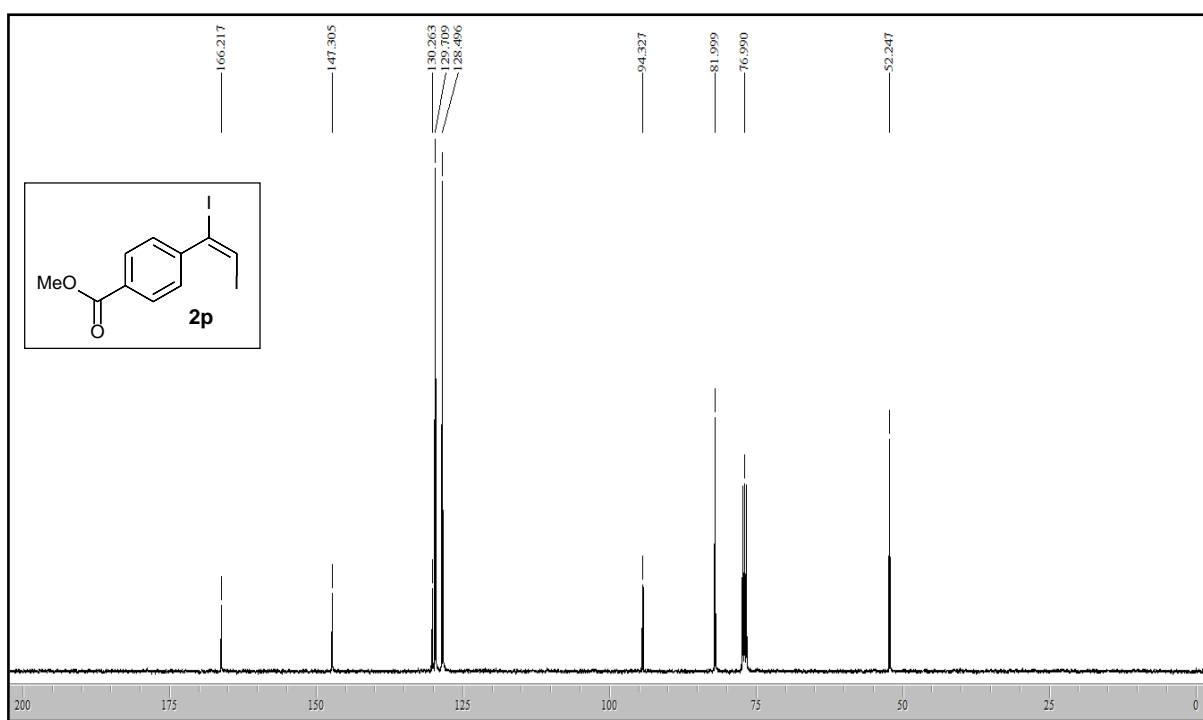
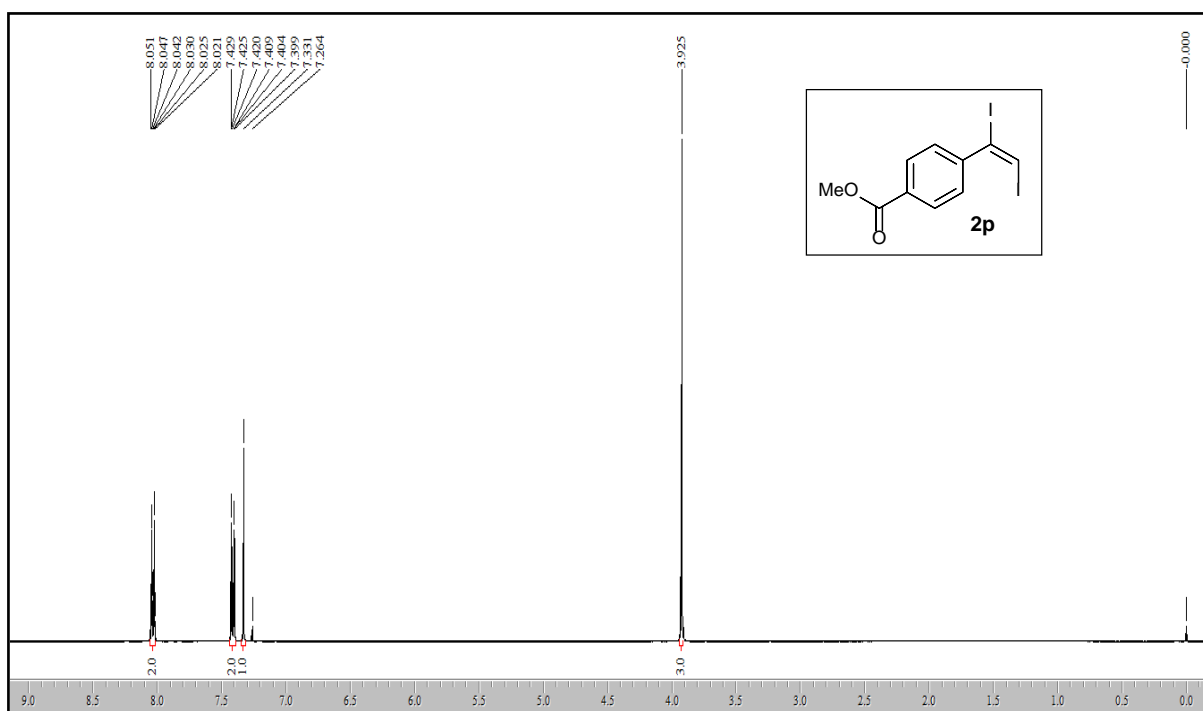


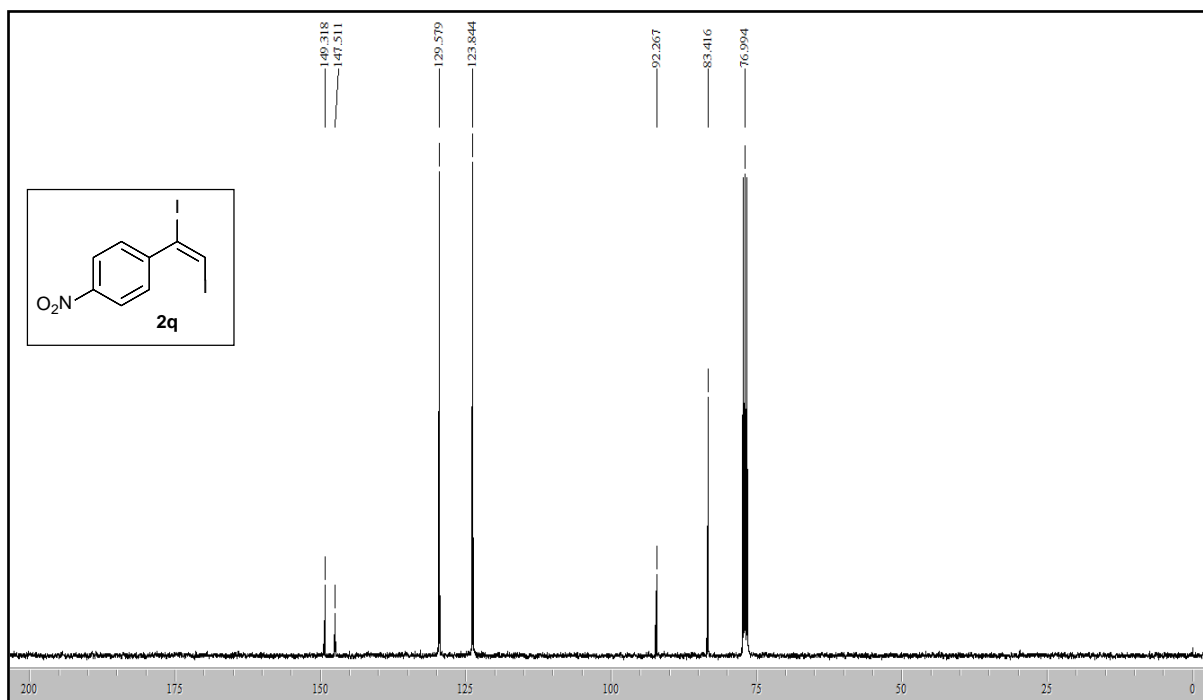
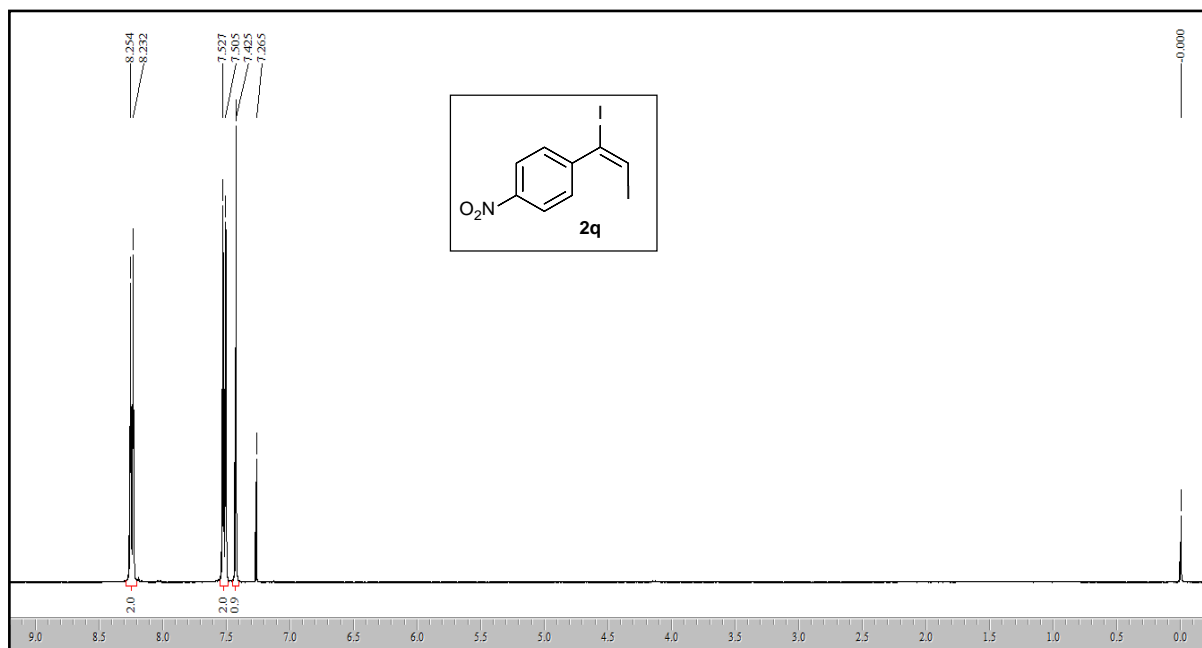


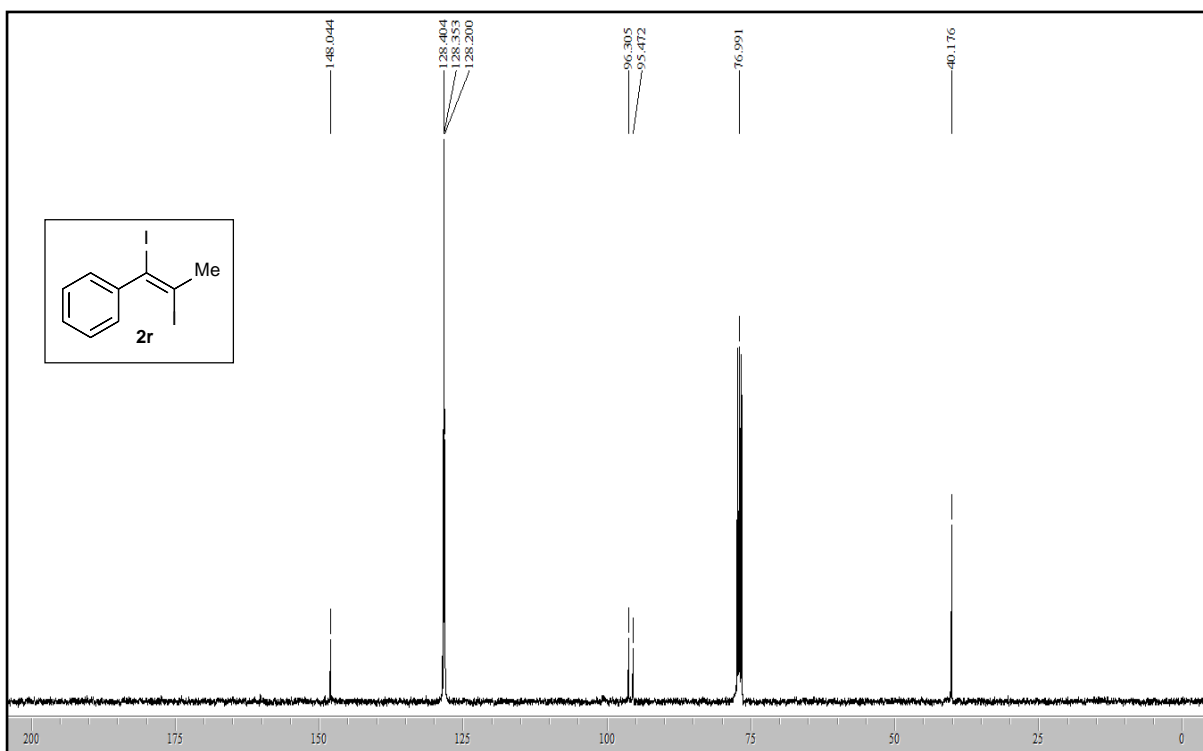
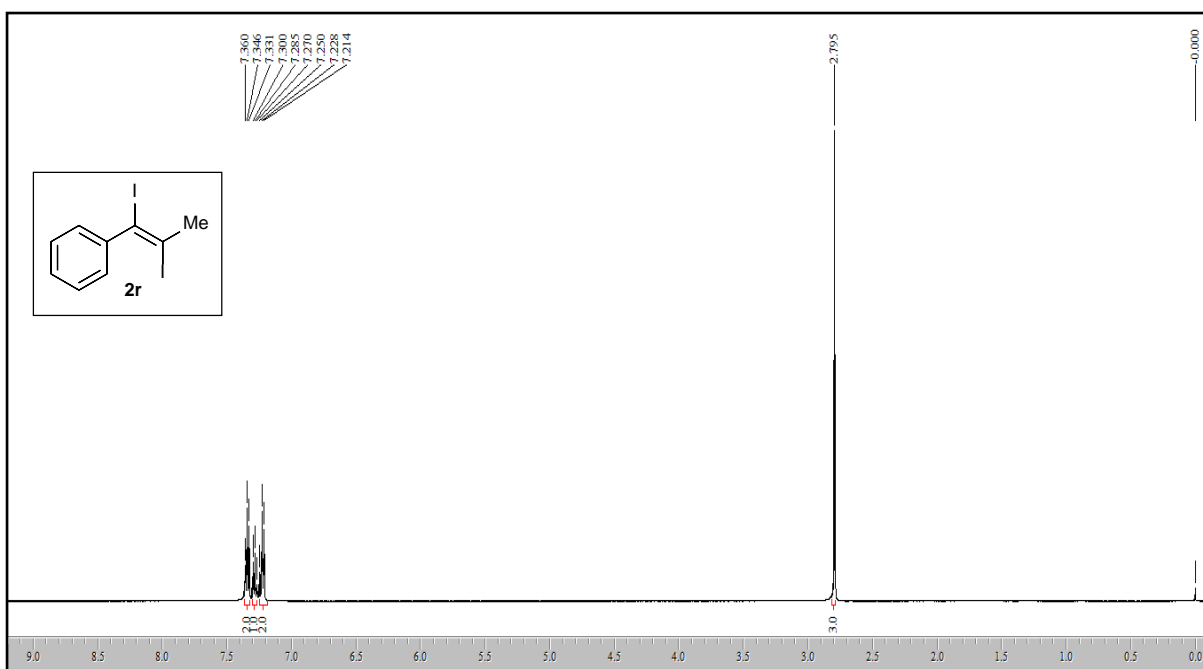


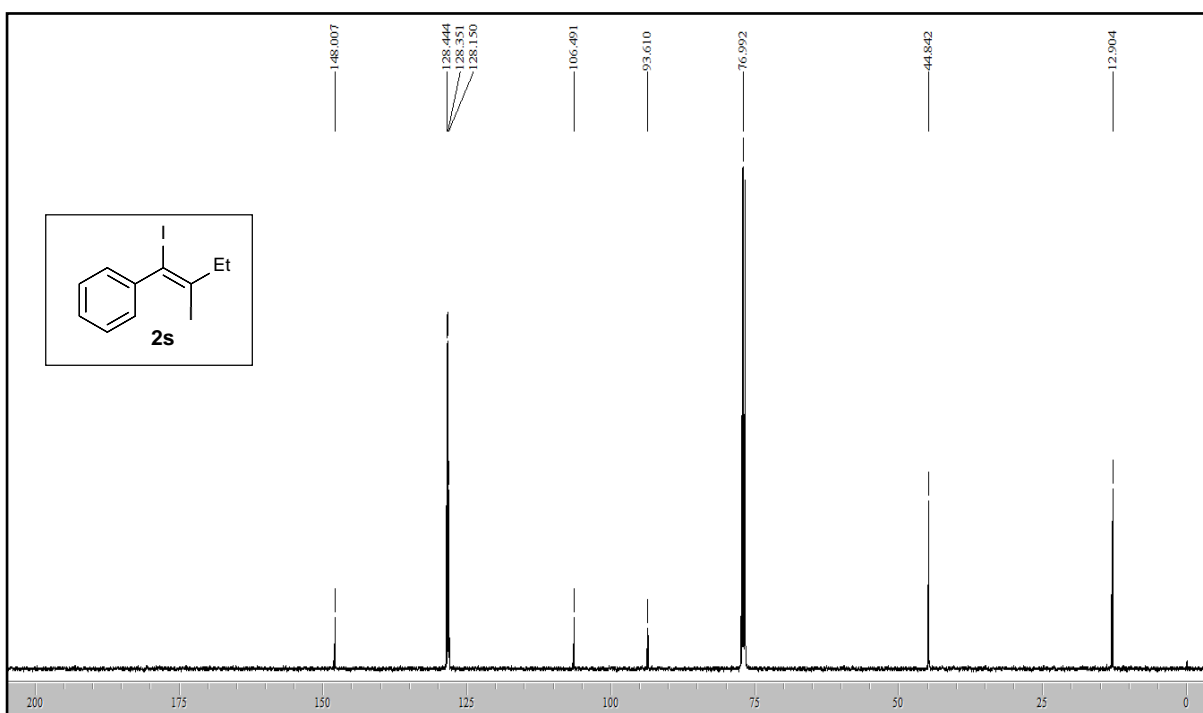
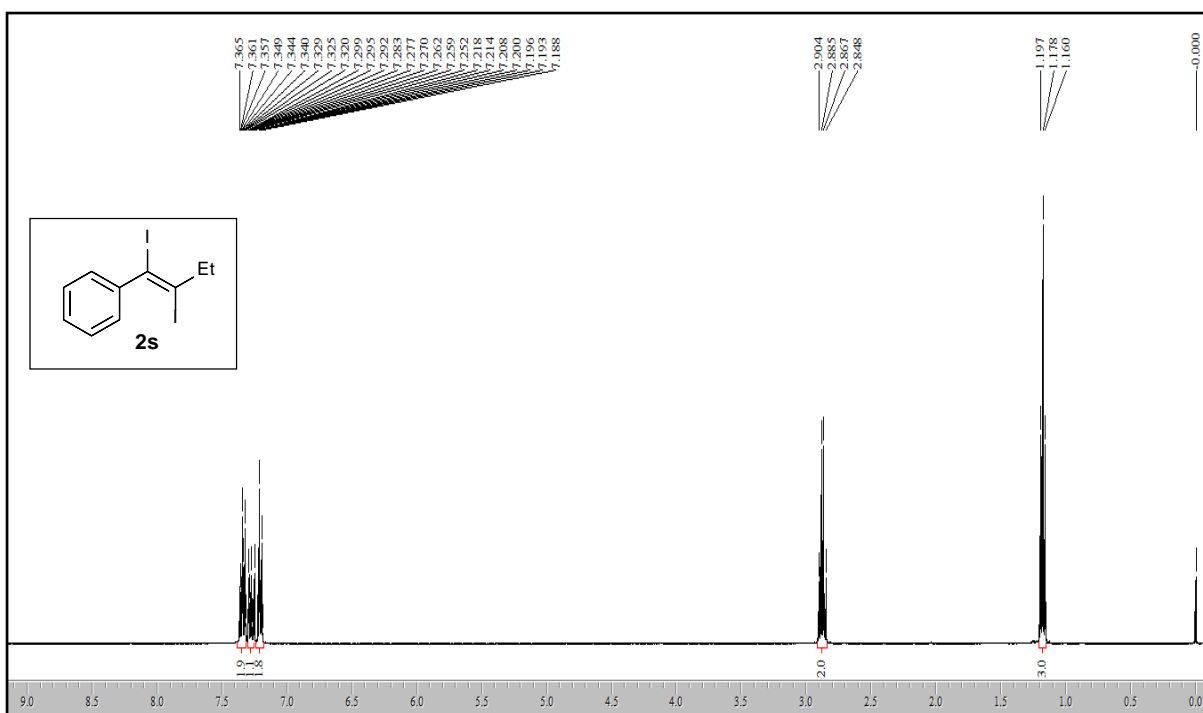


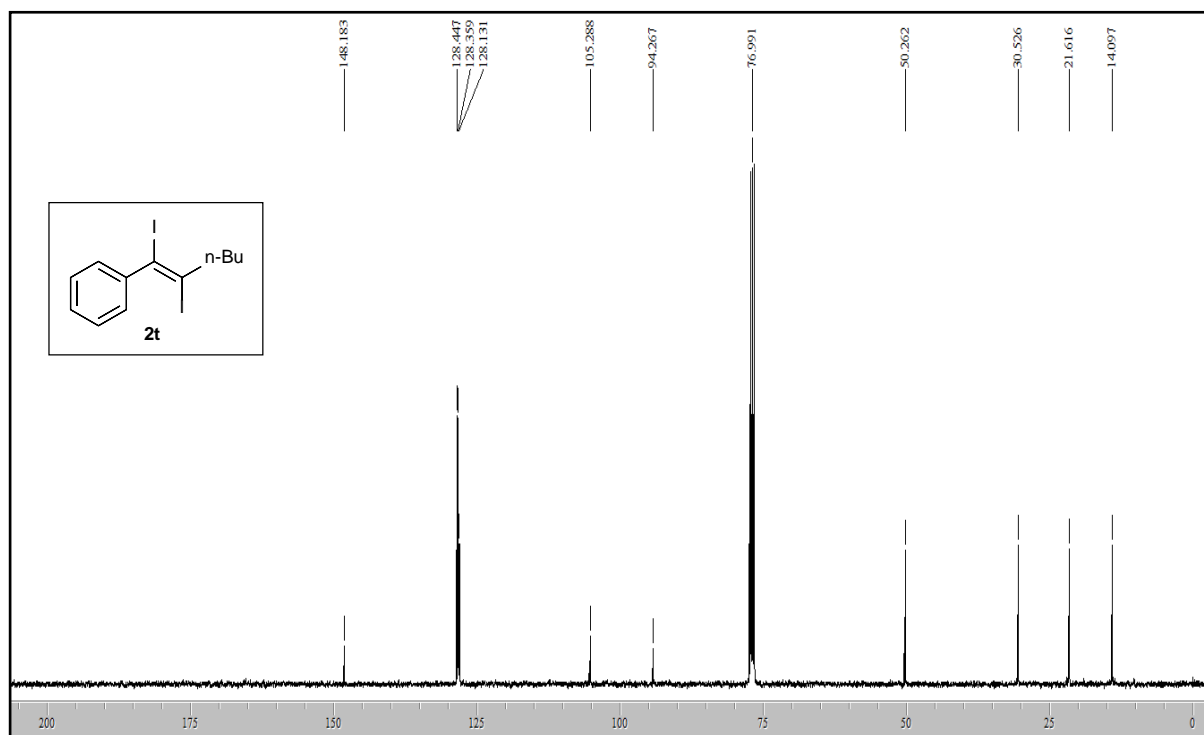
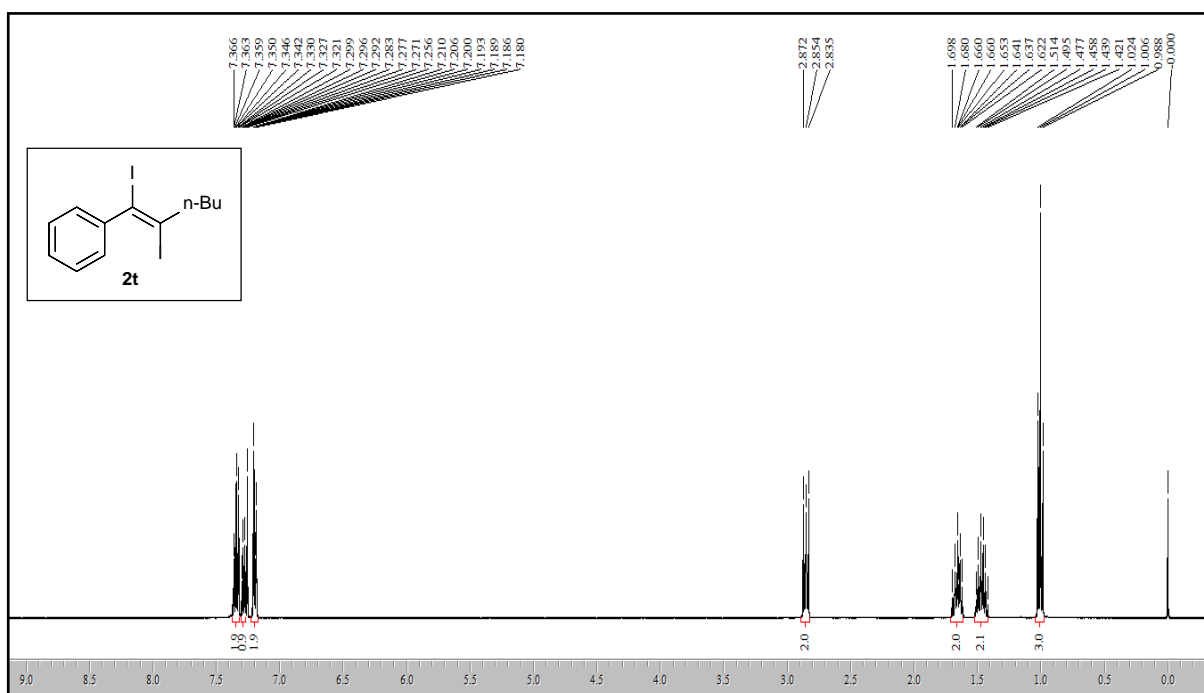


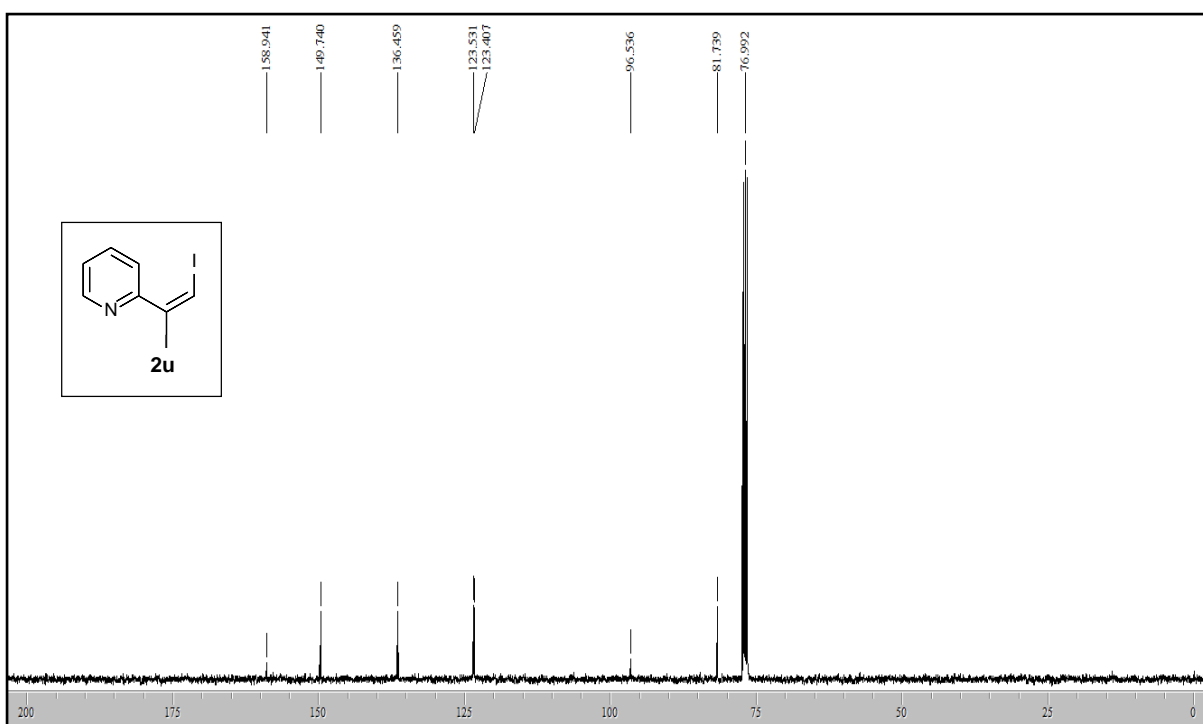
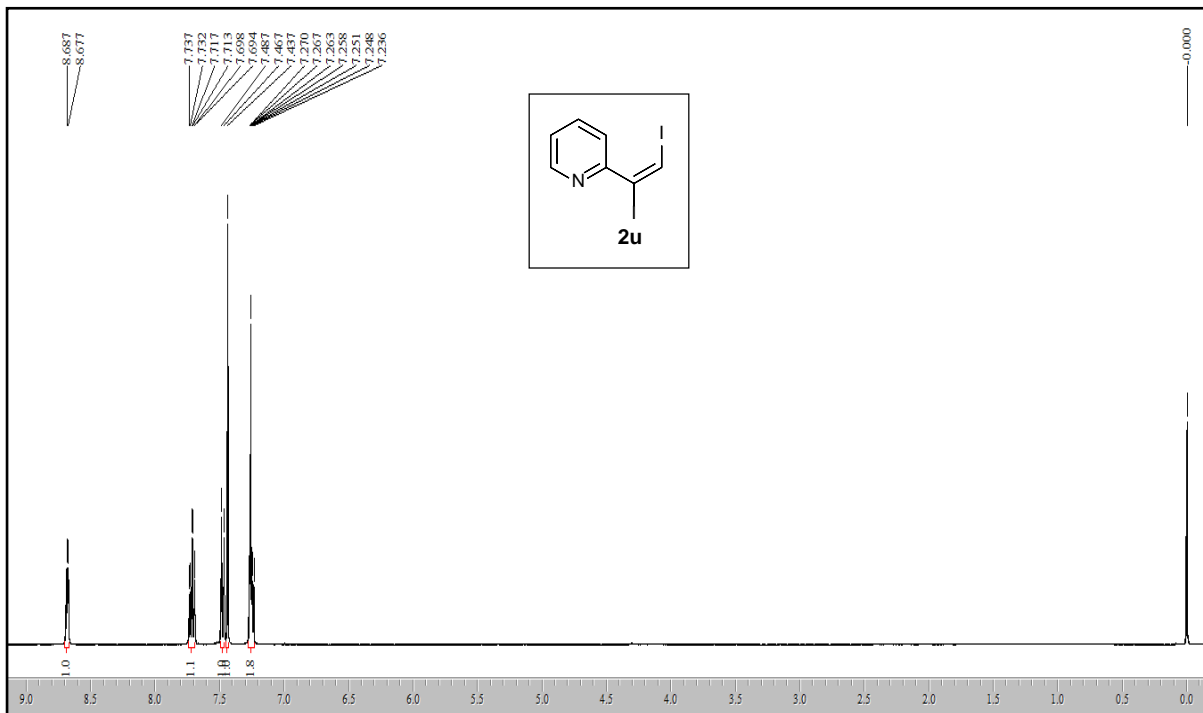


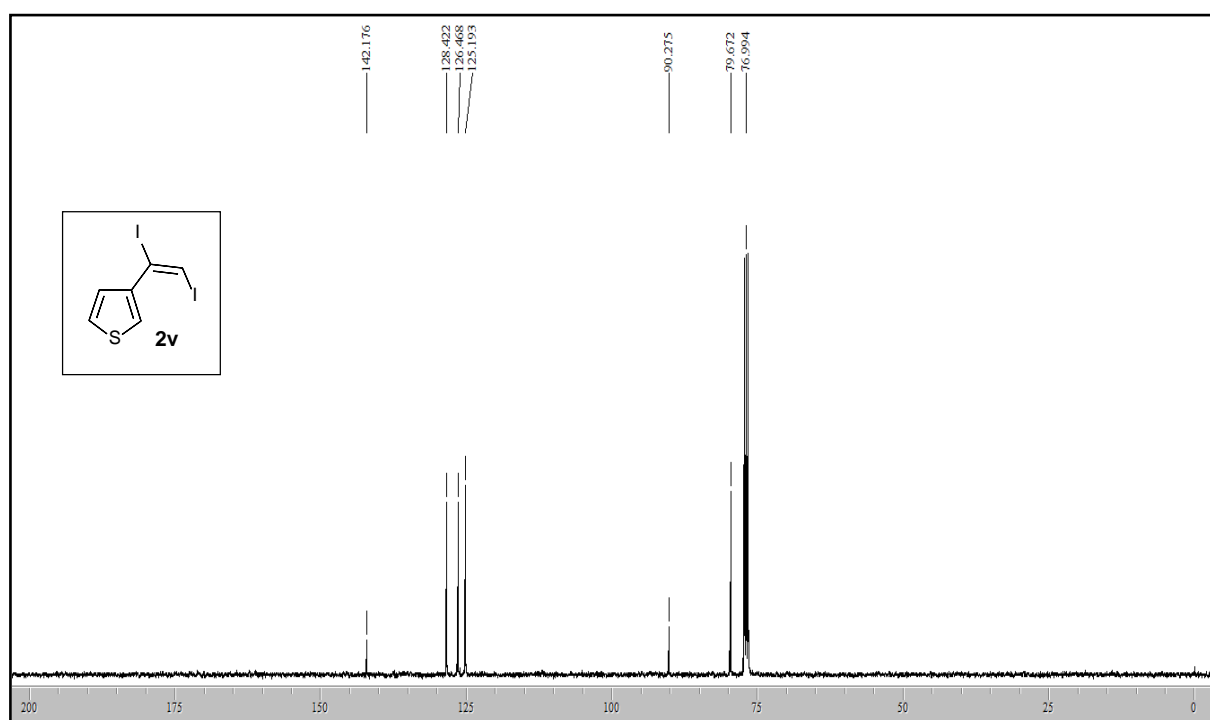
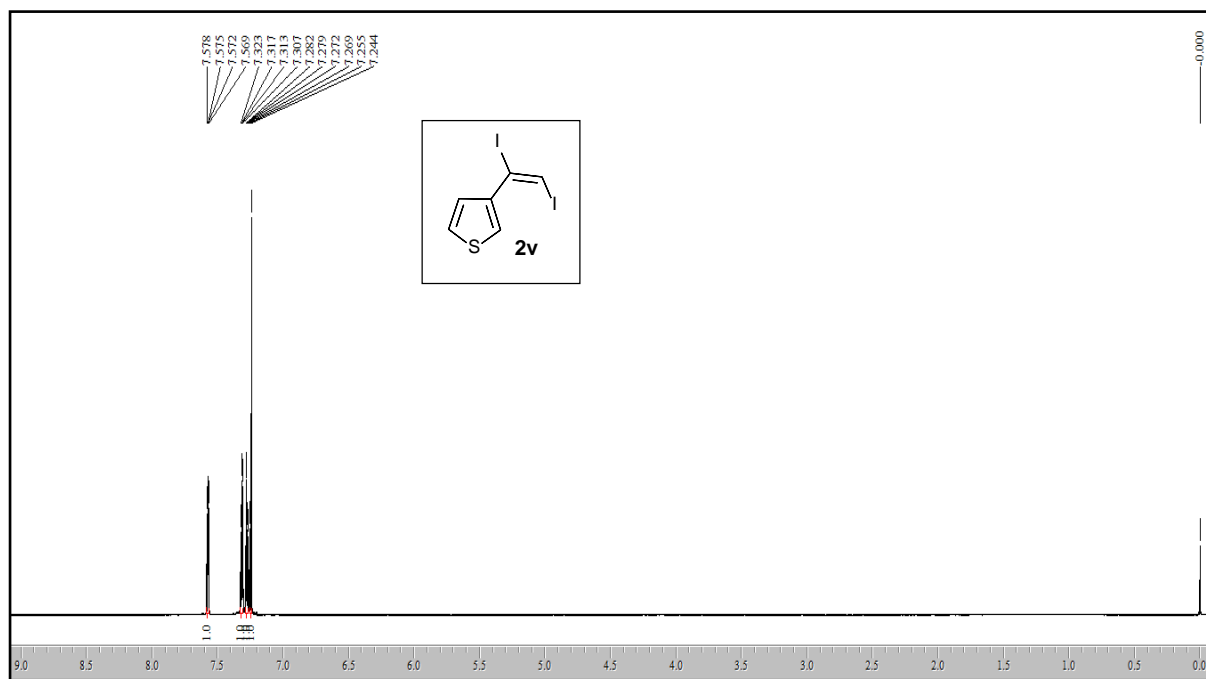


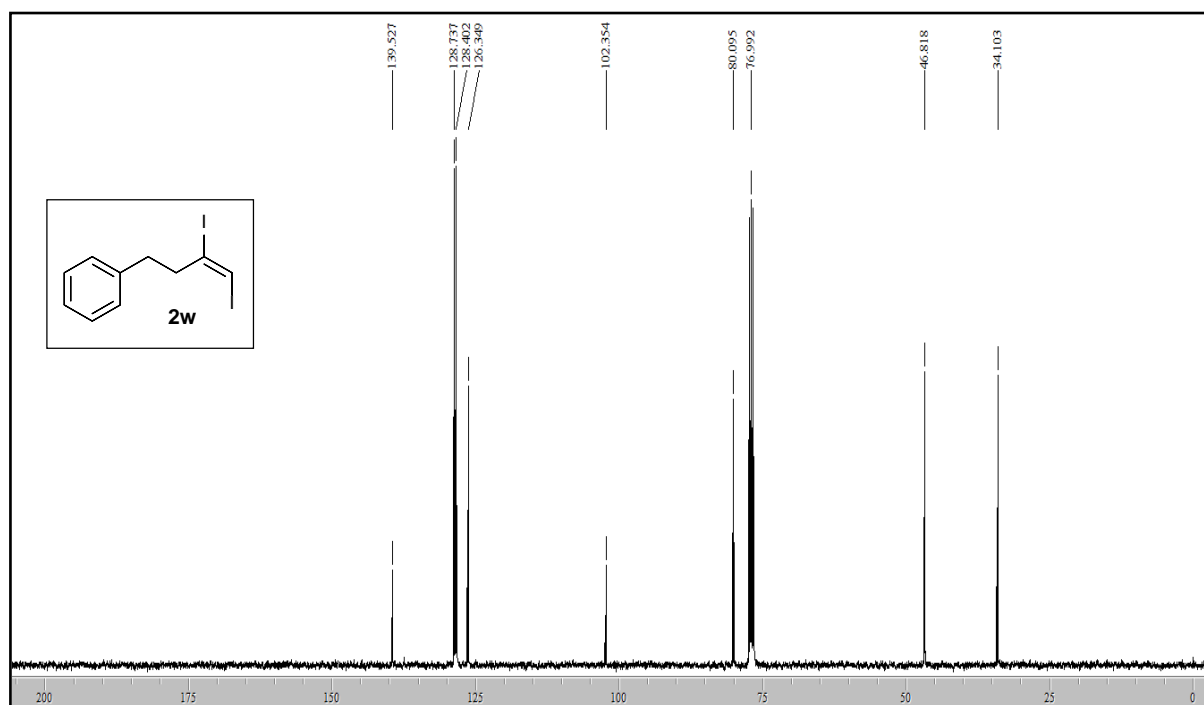
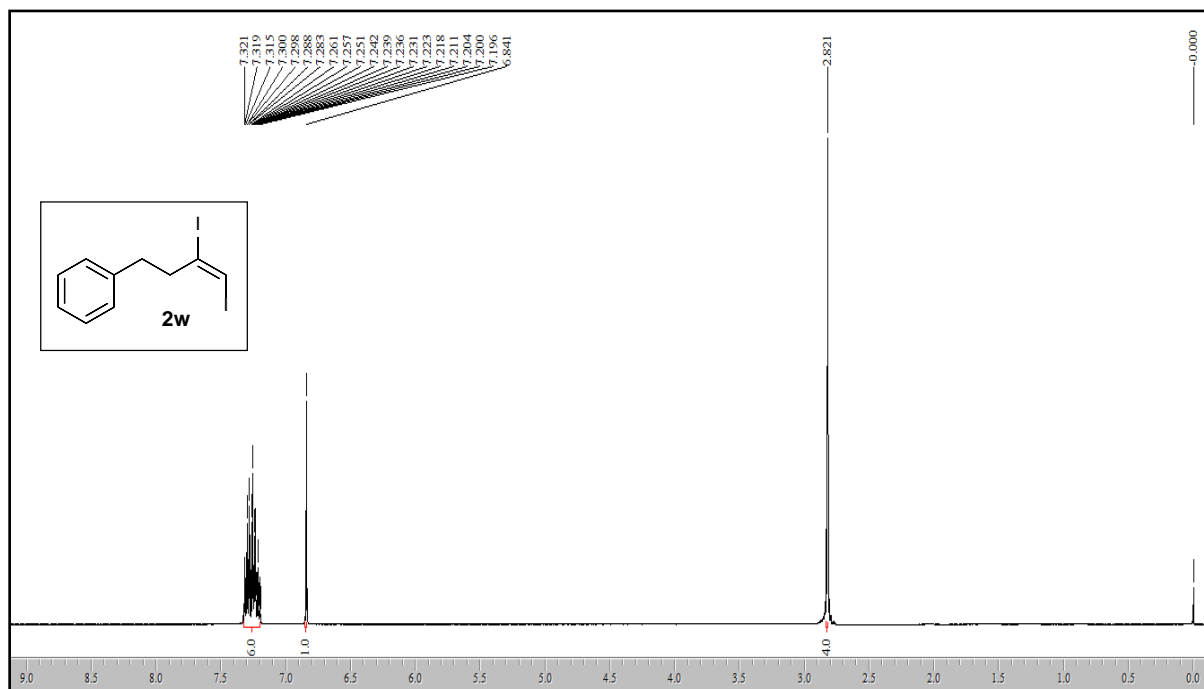


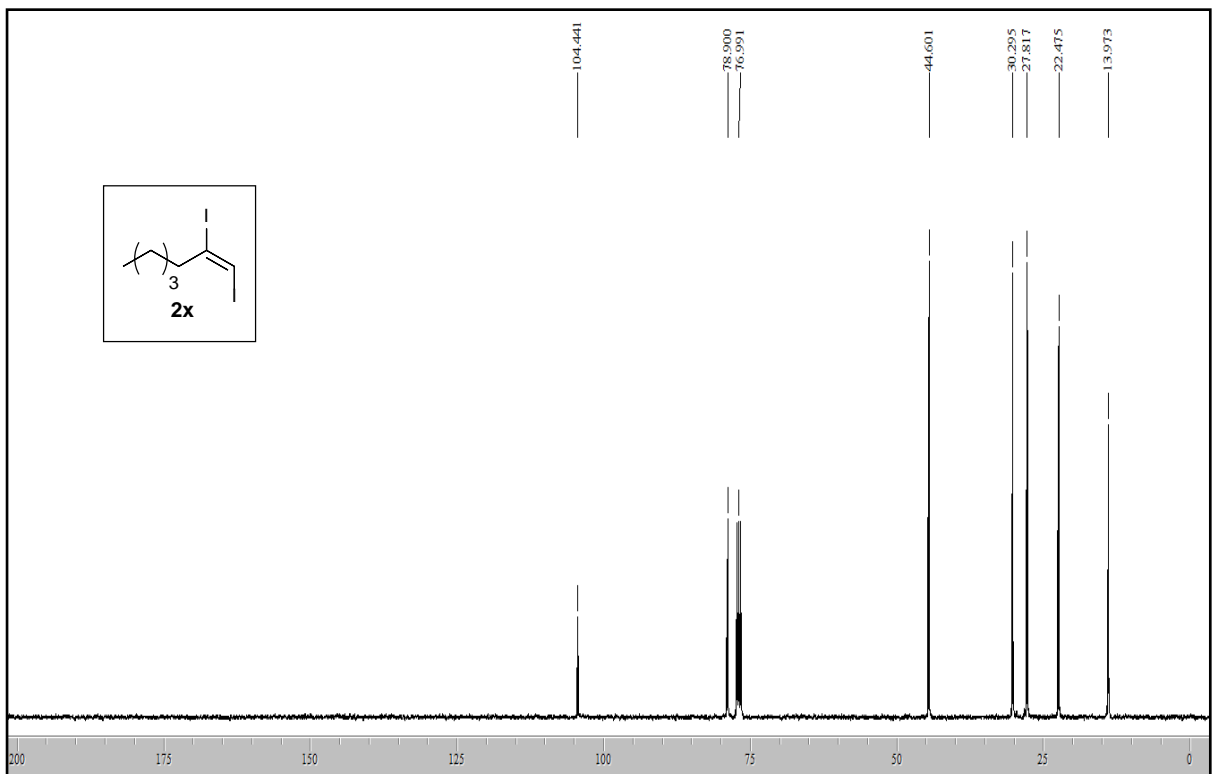
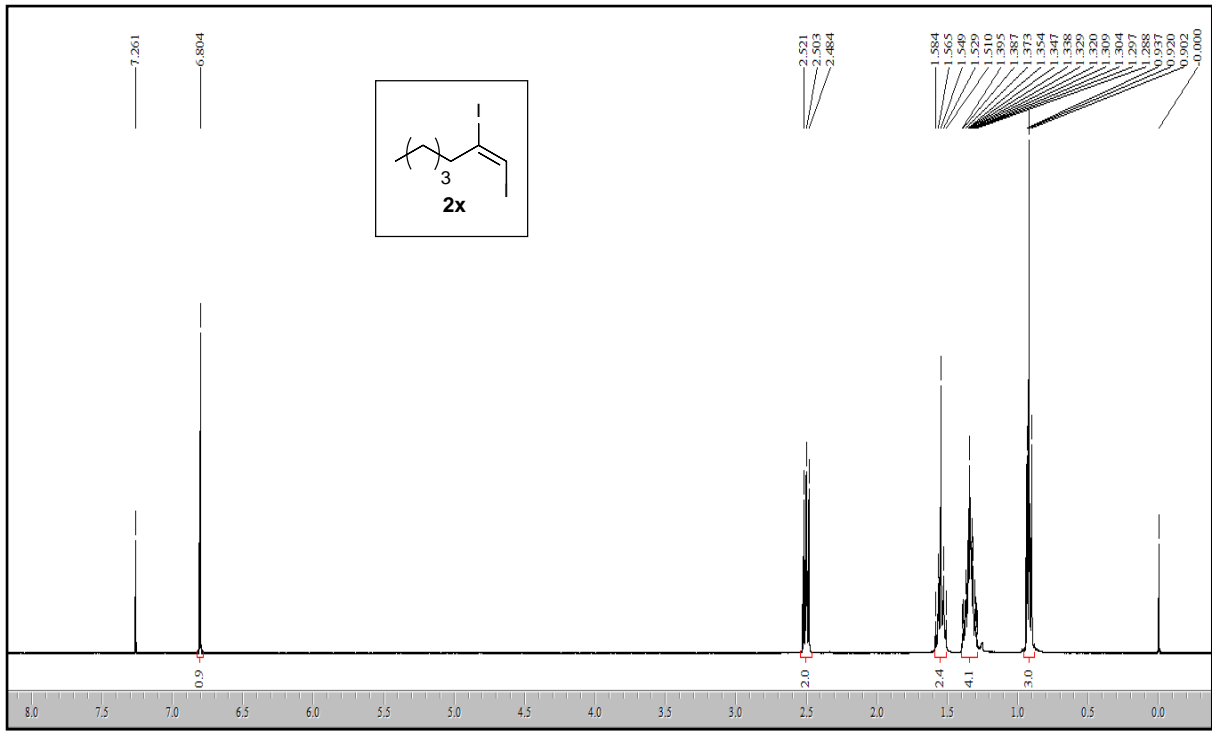


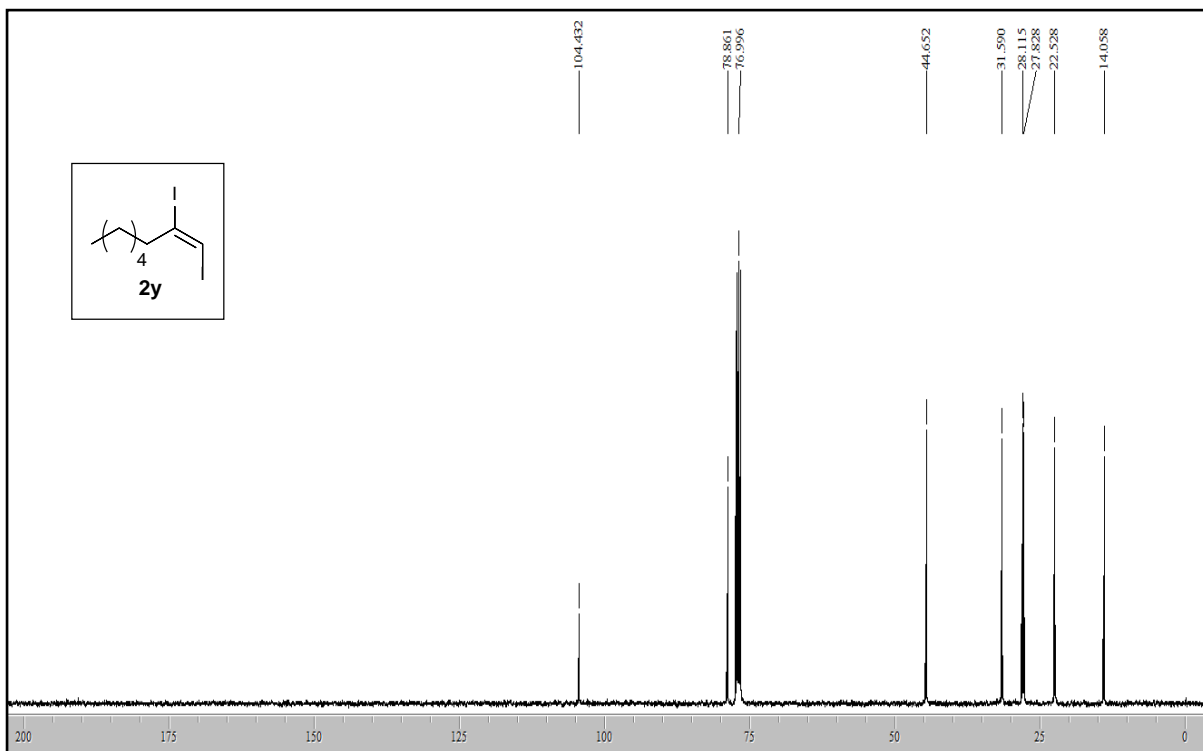
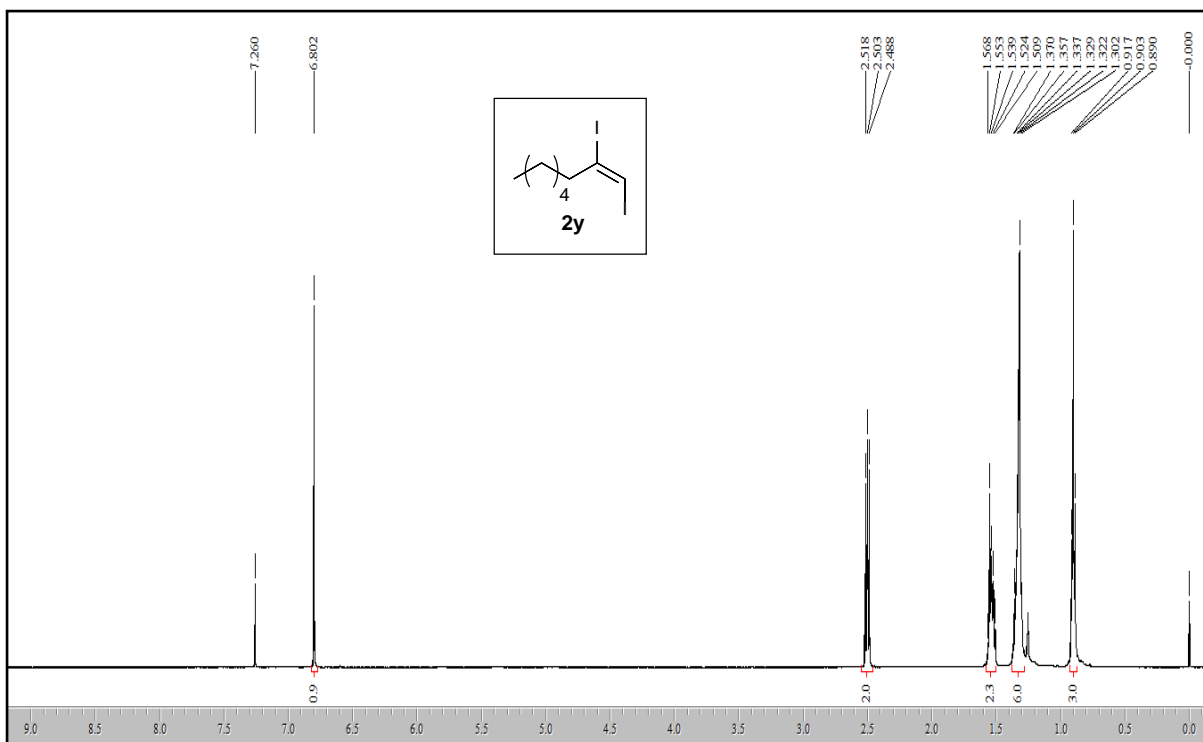


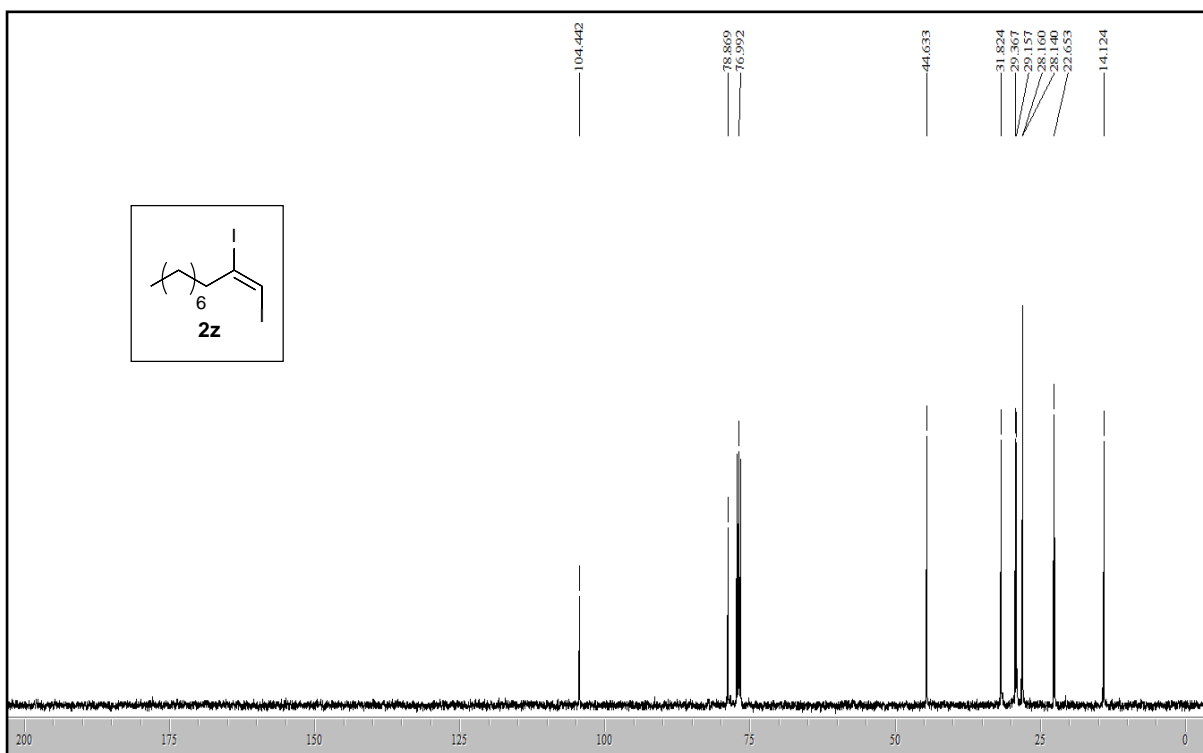
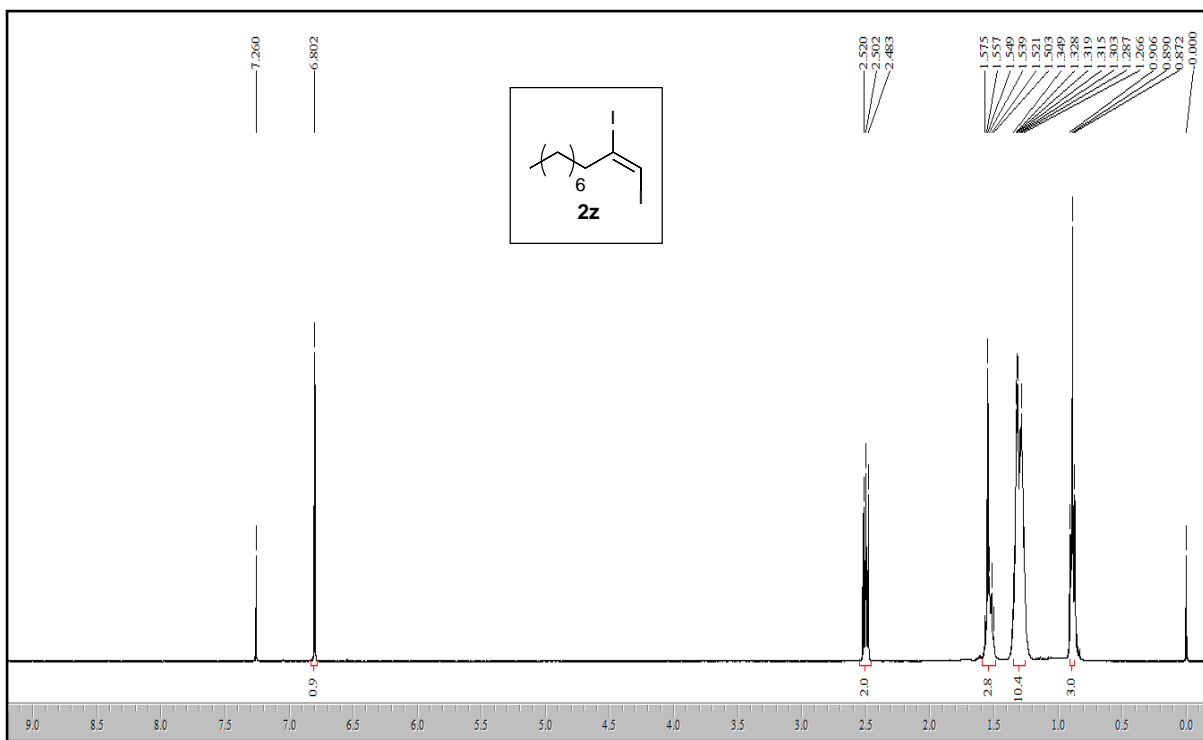


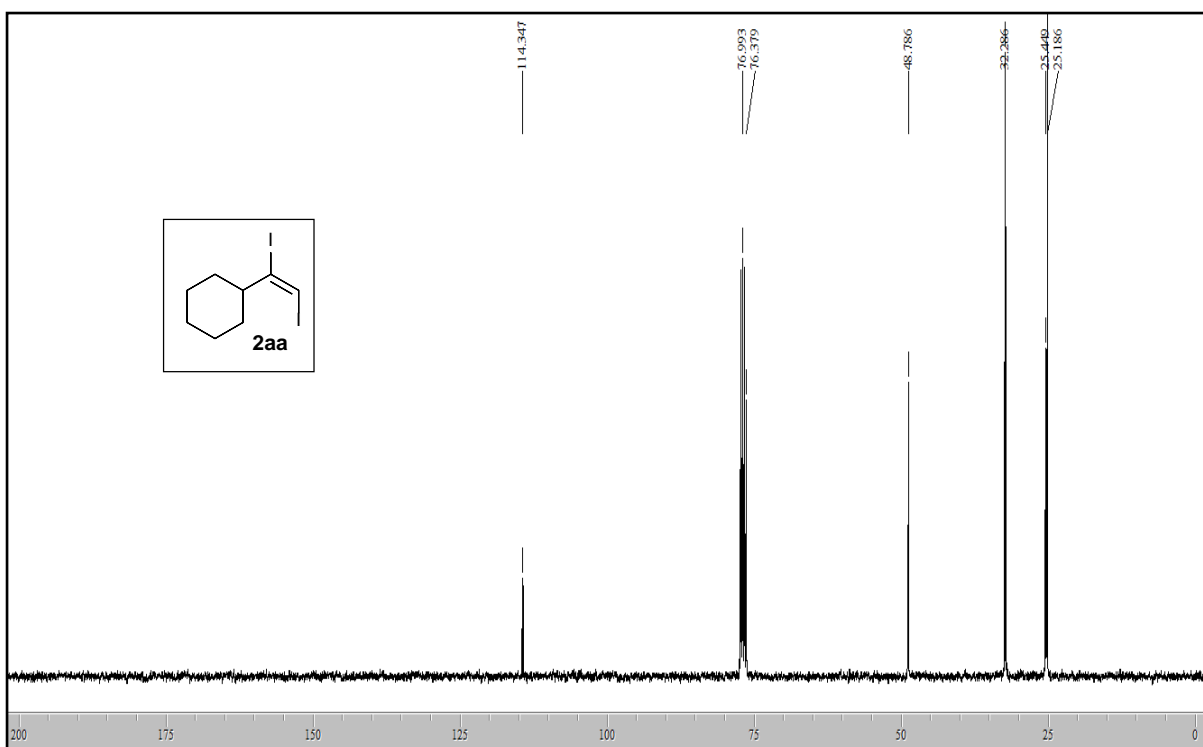
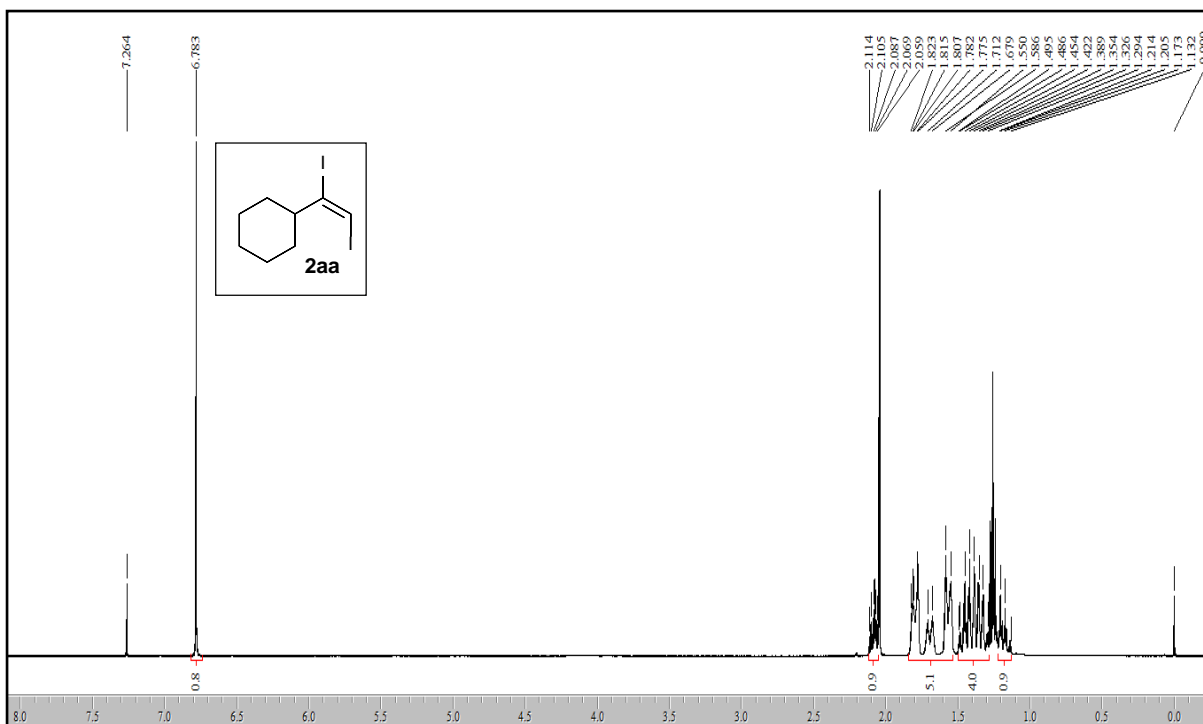


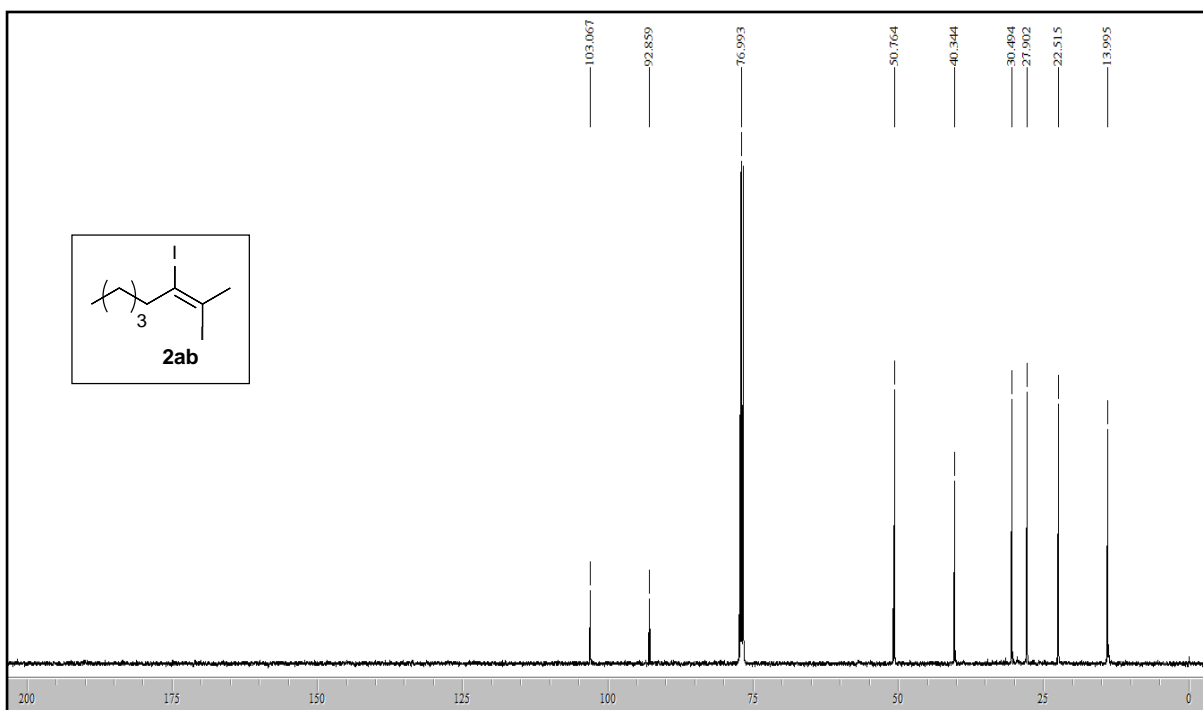
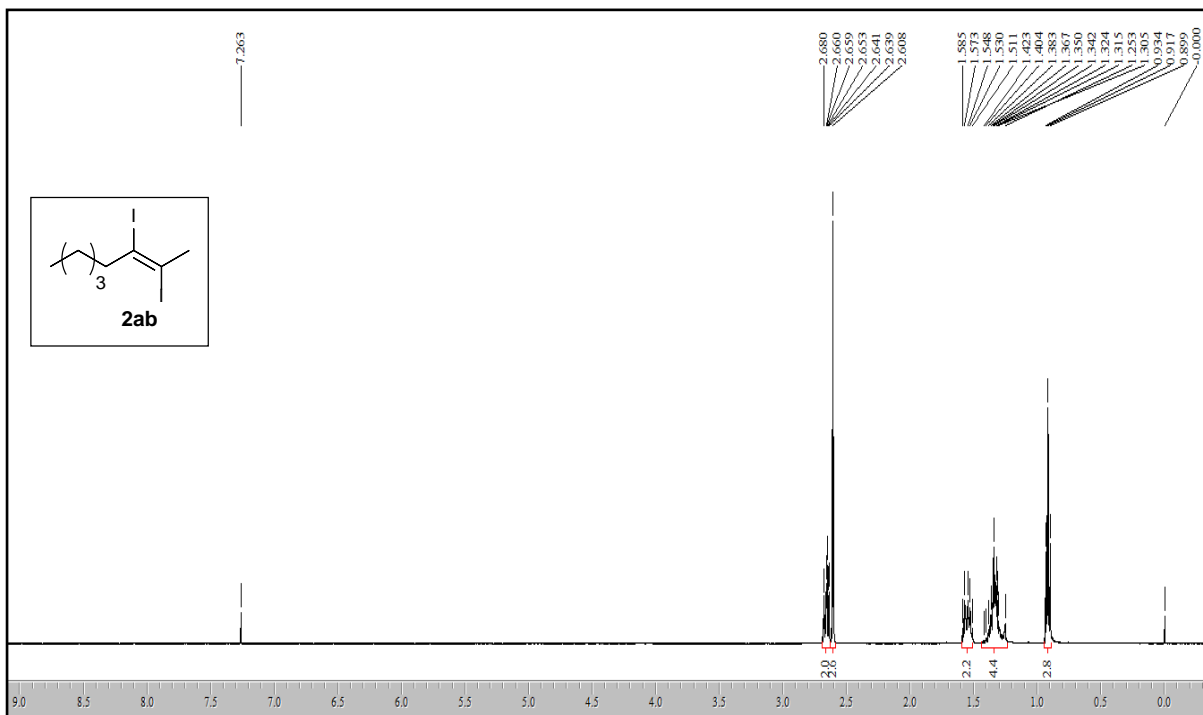


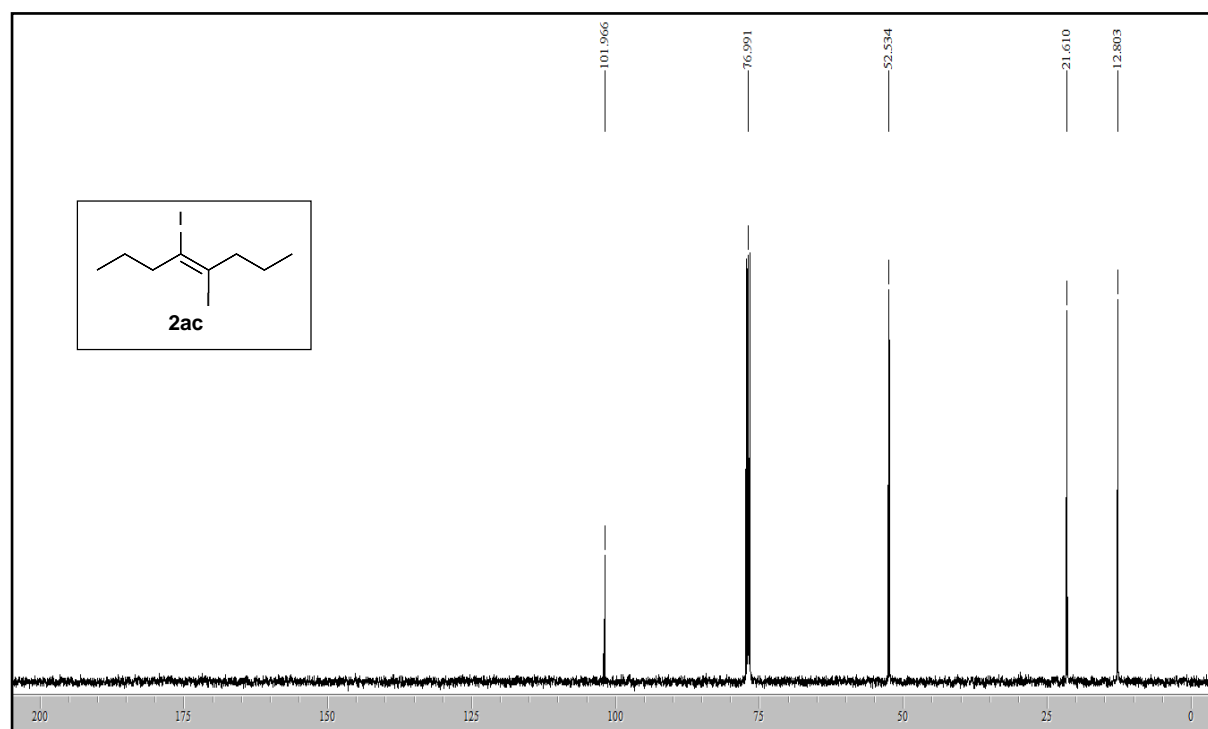
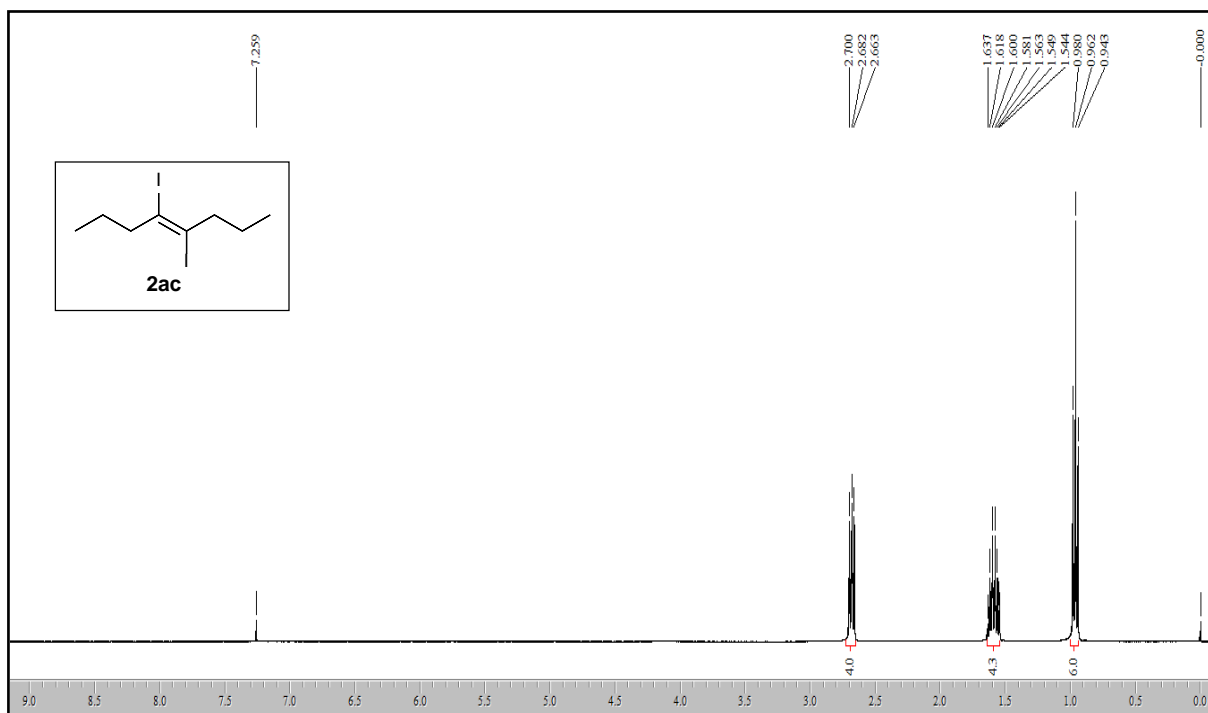












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