Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information for

Pd-Catalyzed Ring-Opening Cross-Coupling of Cyclopropenes with Aryl Iodides

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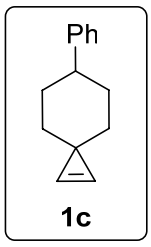
1. General information

All the palladium-catalyzed reactions were carried out in oven-dried glassware sealed with rubber septa under nitrogen condition. All solvents were distilled under nitrogen atmosphere prior to use. Toluene, dioxane and THF were dried over Na with benzophenone-ketyl intermediate as indicator. MeCN, DCE was dried over calcium hydride. $Pd(OAc)_2$, dppf, Cs_2CO_3 and other metal salts were commercially available. Purification of products was accomplished by flash chromatography on silica gel (200-300 mesh, from Qingdao, China). NMR spectra were measured on a Bruker ARX 400 (^{1}H at 400 MHz, ^{13}C at 100 MHz) magnetic resonance spectrometer. Chemical shifts are reported in *ppm* using tetramethylsilane as internal standard (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet). Infrared spectra were recorded on a Nicolet Avatar 330 Fourier transform spectrometer (FT-IR) and are reported in wave numbers (cm^{-1}). MS data were obtained on an Agilent 5975C inert 350 EI mass spectrometer (GC-MS). HRMS data were obtained on a VG ZAB-HS mass spectrometer, Brucker Apex IV FTMS spectrometer. Compounds described in the literature were characterized by comparison of their ^{1}H, and/or ^{13}C NMR spectra to the previously reported data.

2. Preparation of cyclopropenes

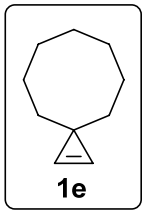
The corresponding cyclopropenes of **1a**,[1] **1b**,[1] **1d**,[2] **1f**,[3] **1h**,[1] **1i**[1] and **1j**[4] were known and prepared *via* literature procedures. **1c**, **1e** and **1g** were prepared *via* similar procedures shown in the literature above.[1-4]



1c

6-Phenylspiro[2.5]oct-1-ene (1c)

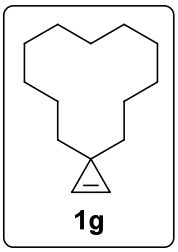
A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.47 (s, 1H), 7.24-7.31 (m, 4H), 7.16-7.20 (m, 1H), 2.59 (tt, *J* = 3.4 Hz, *J* = 12.1 Hz, 1H), 2.04 (dt, *J* = 3.6, 13.2 Hz, 2H), 1.86 (m, 2H), 1.65 (ddd, *J* = 3.6, 12.8, 12.9 Hz, 2H), 1.06 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.9, 128.2, 126.8, 125.7, 123.9, 123.1, 44.6, 39.5, 33.8, 23.1; IR (FT-IR): 3026 (w), 2923 (m), 2838 (w), 1681 (w), 1627 (w), 1492 (w), 1443 (w), 1250 (w), 1158 (w), 1065 (w), 1006 (w), 928 (w), 753 (m), 698 (s) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 184 (12), 169 (6), 156 (44), 141 (15), 129 (14), 115 (16), 104 (100), 91 (26), 78 (18), 65 (12), 57 (3), 51 (6). HRMS (EI) *m*/*e* calcd for $C_{14}H_{16}$ (M^+) 184.1247, found 184.1259.


1e

Spiro[2.7]dec-1-ene (1e)

A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (s, 2H), 1.46-1.66 (m, 14H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 121.3, 40.5, 26.9, 26.3, 24.5, 23.0; IR (FT-IR): 2917 (s), 2850 (m), 1633 (w), 1486 (w), 1445 (m), 1005 (m), 729 (w), 701 (w)

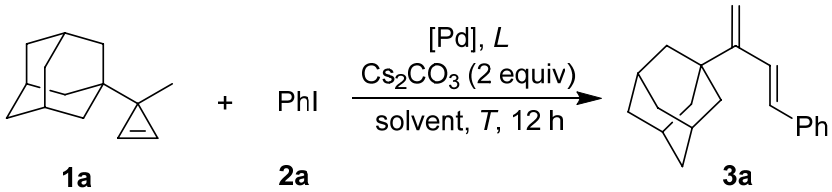
cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 136 (2), 121 (10), 107 (15), 93 (55), 82 (10), 79 (100), 67 (90), 63 (5), 53 (30), 50 (10). HRMS (EI) *m*/*e* calcd for $C_{10}H_{16}$ (M^+) 136.1247, found 136.1262.



Spiro[2.11]tetradec-1-ene (1g)

A colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.29 (s, 2H), 1.33-1.46 (m, 22H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 121.0, 35.2, 24.5, 24.0, 23.9, 23.8, 22.8, 22.2; IR (FT-IR): 2927 (s), 2849 (m), 1636 (w), 1469 (m), 1445 (m), 1007 (m), 741 (w), 723 (w), 699 (m) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 192 (30), 135 (10), 121 (13), 105 (13), 93 (50), 79 (100), 67 (55), 55 (40). HRMS (EI) *m*/*e* calcd for $C_{14}H_{24}$ (M^+) 192.1873, found 192.1888.

3. Optimization of reaction conditions



entry	[Pd] (mol %)	*L* (mol%)	solvent	temp. (°C)	conv. of **2a** (%)	**3a**, %[b]
1	$Pd(PPh_3)_4$ (5)	dppf (10)	DCE	70	72	30
2	$Pd(OAc)_2$ (5)	dppf (10)	DCE	70	76	45
3	$Pd(OAc)_2$ (5)	dppb (10)	DCE	70	68	40
4	$Pd(OAc)_2$ (5)	Xphos (10)	DCE	70	30	5
5	$Pd(OAc)_2$ (5)	Sphos (10)	DCE	70	36	11
6	$Pd(OAc)_2$ (5)	dppf (10)	MeCN	70	64	46
7	$Pd(OAc)_2$ (5)	dppf (10)	MeCN	60	58	50
8	$Pd(OAc)_2$ (5)	dppf (10)	MeCN	80	52	34
9[c]	$Pd(OAc)_2$ (5)	dppf (10)	MeCN	60	78	63
10[c]	$Pd(OAc)_2$ (8)	dppf (16)	MeCN	60	100	85

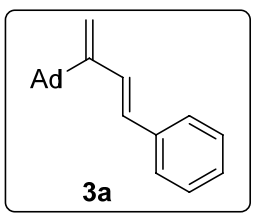
[a]If not otherwise noted, the reaction conditions are as following: a solution of **1a** (0.1 mmol), **2a** (0.1 mmol), $Pd(OAc)_2$ (5 mol% or 8 mol%), dppf (10 mol% or 16 mol%), Cs_2CO_3 (0.2 mmol) in solvent (0.3 mL) was stirred for 12h. [b]Isolated yield. [c]**1a** (0.15 mmol) was used.

At the outset, we have investigated the Pd-catalyzed cross-coupling of cyclopropene **1a** with phenyliodide **2a**. The cyclopropene **1a**, which bears an adamantyl substituent, was chosen as the carbene precursor mainly because of the simplicity in the product isolation and identification. In the initial attempts, we were delighted to find that in the presence of 5 mol% $Pd(PPh_3)_4$, 10 mol% dppf, and 2 equiv of Cs_2CO_3, the expected 1,3-diene **3a** was obtained in 30% yield (Table 1, entry 1). By screening the palladium catalyst, we found that $Pd(OAc)_2$ could afford slightly higher yield (entry 2). Phosphine ligands were then tested with $Pd(OAc)_2$ as the catalyst, however, the reaction could not be improved (entries 3-5). The yield could be slightly improved by using MeCN as the solvent (entry 6). The reaction was found favorable under low temperature (entries 7, 8). Because the conversion of **2a** is not complete in all the cases, we further increased the ratio of cyclopropene substrate *versus* iodide from 1:1 to 1.5:1, and found both conversion of **2a** and the yield of **3a** could be improved (entry 9). Finally, the complete conversion of **2a** was achieved by increasing the loading of $Pd(OAc)_2$ from 5 mol% to 10 mol%, and dppf from 8 mol% to 16 mol%, respectively, affording the product **3a** in 85% yield (entry 10). It is noteworthy that in all the cases only one stereoisomer was observed. The configuration of the formed double bond of **3a** was confirmed to be *E* through ^{1}H NMR spectrum.

4. General procedure for the Pd-catalyzed reaction

Under an argon atmosphere, $Pd(OAc)_2$ (5.4 mg, 0.024 mmol, 8 mol%), dppf (26.6 mg, 0.048 mmol, 16 mol%), Cs_2CO_3 (195.6 mg, 0.6 mmol, 2 equiv) and aryl iodide (0.3 mmol, 1 equiv) were successively added to a flame-dried 10 mL Schlenk tube. The reaction flask was degassed three times with nitrogen and dry MeCN (1.0 mL) was added using a syringe. Note that the aryl iodide in a liquid form was added to the reaction tube by syringe after being added in the solvent. The mixture was stirred at rt under argon for 5 min. Then cyclopropene (0.45 mmol, 1.5 equiv) was added by syringe. The reaction was heated at 60 ºC with stirring for 12 h, then cooled to room temperature. The reaction mixture was filtered through a short column of silica gel (eluent ether). Solvent was then removed in *vacuo* to leave a crude mixture, which is purified by silica gel column chromatography to afford pure 1,3-diene product.

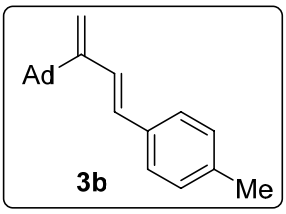
5. Characterization data



1-((*E*)-4-phenylbuta-1,3-dien-2-yl)adamantane (3a)

79%; a white solid (m.p. = 49-50 ^{o}C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.3 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.21 (t, *J* = 7.3 Hz, 1H), 6.87 (d, *J* = 15.8 Hz, 1H), 6.75 (d, *J* = 15.8 Hz, 1H), 5.20 (m, 1H), 4.80 (d, *J* = 1.2 Hz, 1H), 2.03 (m, 3H), 1.67-1.75 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.5, 137.7, 129.7, 128.5, 128.1, 127.2, 126.4, 107.1, 41.3, 37.1, 36.8,

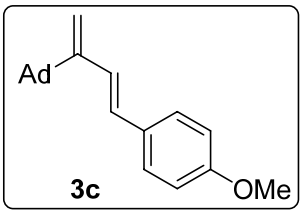
28.6; IR (FT-IR): 3026 (w), 2901 (s), 2847 (m), 1632 (w), 1600 (w), 1494 (m), 1344 (m), 1308 (w), 1203 (w), 1102 (w), 1073 (w), 1028 (w), 957 (m), 895 (m), 853 (w), 815 (w), 751 (s), 692 (s), 671 (w) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 264 (11), 178 (3), 165 (4), 135 (100), 128 (48), 115 (7), 107 (12), 93 (21), 79 (22), 67 (8), 55 (11). HRMS (ESI) *m*/*e* calcd for $C_{20}H_{25}$ ($M+H^+$) 265.1951, found 265.1954.



1-((*E*)-4-(*p*-tolyl)buta-1,3-dien-2-yl)adamantane (3b)

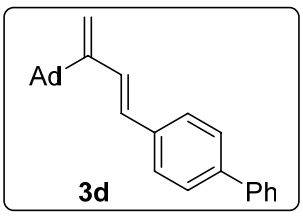
81%; a white solid (m.p. = 69-70 °C); ^1H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.1 Hz, 2H), 7.12 (d, *J* = 8.1 Hz, 2H), 6.81 (d, *J* = 15.8 Hz, 1H), 6.72 (d, *J* = 15.8 Hz, 1H), 5.19 (m, 1H), 4.78 (d, *J* = 1.4 Hz, 1H), 2.33 (s, 3H), 2.03 (m, 3H), 1.67-1.75 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 137.0, 135.0, 129.5, 129.2, 127.1, 126.3, 106.7, 41.3, 37.1, 36.9, 28.6, 21.1; IR (FT-IR): 3022 (w), 2902 (s), 2847 (m), 1632 (w), 1601 (w), 1510 (m), 1499 (m), 1344 (w), 1307 (w), 1102 (w), 1074 (w), 957 (m), 889 (m), 859 (m), 802 (s), 761 (w), 713 (w), 694 (w) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 278 (9), 219 (1), 207 (2), 178 (3), 165 (9), 152 (5), 142 (60), 135 (12), 128 (13), 115 (27), 107 (13), 93 (28), 67 (9), 55 (5). HRMS (ESI) *m*/*e* calcd for $C_{21}H_{27}$ ($M+H^+$) 279.2107, found 279.2111.

1-((*E*)-4-phenylbuta-1,3-dien-2-yl)adamantane (3c)



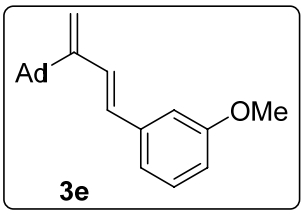
85%; a white solid (m.p. = 76-77 °C); ^1H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 8.8 Hz, 2H), 6.71 (m, 2H), 5.17 (d, *J* = 1.1 Hz, 1H), 4.77 (d, *J* = 1.5 Hz, 1H), 3.81 (s, 3H), 2.03 (m, 3H), 1.67-1.75 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 156.7, 130.6, 129.1, 127.6, 126.0, 114.0, 106.4, 55.3, 41.4, 37.1, 36.9, 28.7; IR (FT-IR): 2902 (m), 2847 (m), 1632 (w),1605 (m), 1575 (w), 1509 (s), 1452 (w), 1343 (w), 1284 (w), 1248 (s), 1214 (w), 1173 (m), 1108 (w), 1036 (m), 957 (m), 889 (m), 857 (w), 768 (w), 704 (w), 699 (w) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 294 (18), 253 (1), 214 (9), 206 (8), 199 (10), 171 (7), 159 (100), 152 (4), 144 (21), 135 (78), 128 (18), 115 (27), 107 (13), 93 (27), 79 (29), 67 (11), 55 (7). HRMS (ESI) *m*/*e* calcd for $C_{21}H_{27}O$ ($M+H^+$) 295.2066, found 295.2063.

1-((*E*)-4-([1,1'-biphenyl]-4-yl)buta-1,3-dien-2-yl)adamantane (3d)

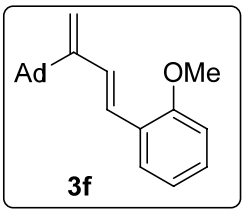


78%; a white solid (m.p. = 155-156 °C); ^1H NMR (400 MHz, $CDCl_3$) δ 7.55-7.61 (m, 4H), 7.48-7.50 (m, 2H), 7.41-7.45 (m, 2H), 7.31-7.35 (m, 1H), 6.91 (d, *J* = 15.8 Hz, 1H), 6.79 (d, *J* = 15.8 Hz, 1H), 5.23 (m, 1H), 4.82 (d, *J* = 1.3 Hz, 1H), 2.04 (m, 3H), 1.68-1.77 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.5, 140.7, 140.0, 136.8, 129.2, 128.7, 128.3, 127.2, 126.9, 126.8, 107.2, 41.4, 37.2, 36.8, 28.6; IR (FT-IR): 3085 (w),

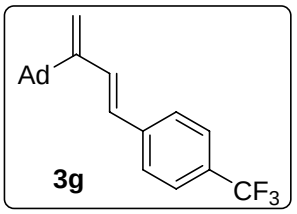
3025 (w), 2905 (s), 2846 (m), 1632 (w), 1601 (w), 1485 (m), 1447 (m), 1407 (w), 1343 (w), 1320 (w), 1100 (w), 1076 (w), 1005 (w), 959 (m), 909 (m), 886 (m), 823 (m), 767 (s), 761 (s), 724 (m), 699 (m), 690 (m), 673 (w) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 340 (9), 281 (1), 228 (2), 215 (4), 204 (83), 189 (6), 178 (9), 165 (10), 152 (6), 135 (100), 126 (2), 107 (13), 93 (25), 79 (25), 67 (9), 55 (6). HRMS (ESI) *m*/*e* calcd for $C_{26}H_{29}$ ($M+H^+$) 341.2264, found 341.2270.



1-((*E*)-4-(3-methoxyphenyl)buta-1,3-dien-2-yl)adamantane (3e)
86%; a colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, *J* = 7.9 Hz, 1H), 7.02 (d, *J* = 7.7 Hz, 1H), 6.95 (m, 1H), 6.86 (d, *J* = 16.0 Hz, 1H), 6.78 (m, 1H), 6.72 (d, *J* = 15.7 Hz, 1H), 5.20 (m, 1H), 4.81 (d, *J* = 1.3 Hz, 1H), 3.83 (s, 3H), 2.03 (m, 3H), 1.67-1.74 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 156.5, 139.2, 129.5, 129.4, 128.5, 119.1, 112.7, 111.9, 107.2, 55.2, 41.3, 37.1, 36.8, 28.6; IR (FT-IR): 2902(s), 2847(m), 1598(m), 1578(m), 1487(m), 1464(m), 1343(w), 1287(m), 1245(m), 1154(m), 1102(w), 1050(m), 955(m), 937(w), 892(m), 815(w), 778(m), 689(m) cm^{-1}; MS (EI, 70 eV): m/z (%) = 294 (18), 205 (2), 178 (3), 165 (6), 159 (48), 144 (13), 135 (100), 128 (16), 115 (21), 107 (14), 93 (25), 79 (27), 67 (9), 55 (6). HRMS (ESI) *m*/*e* calcd for $C_{21}H_{27}O$ ($M+H^+$) 295.2056, found 295.2060.



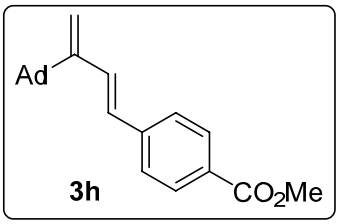
1-((*E*)-4-(2-methoxyphenyl)buta-1,3-dien-2-yl)adamantane (3f)
83%; a white solid (m.p. = 50-51 °C); ^1H NMR (400 MHz, $CDCl_3$) δ 7.49 (dd, *J* = 1.4 Hz, *J* = 7.6 Hz, 1H), 7.20 (m, 1H), 7.10 (d, *J* = 15.9 Hz, 1H), 6.93 (t, *J* = 7.46 Hz, 1H), 6.88 (d, J = 2.6 Hz, 1H), 6.85 (d, *J* = 4.9 Hz, 1H), 5.23 (m, 1H), 4.79 (d, *J* = 1.3 Hz, 1H), 3.85 (s, 3H), 2.03 (m, 3H), 1.67-1.76 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.9, 156.7, 128.5, 128.2, 126.8, 126.6, 124.2, 120.6, 110.9, 106.8, 55.5, 41.4, 37.1, 36.9, 28.7; IR (FT-IR): 2901 (m), 2847 (m), 1629 (w), 1596 (w), 1487 (m), 1462 (m), 1344 (w), 1292 (w), 1242 (s), 1176 (w), 1106 (m), 1074 (w), 1029 (m), 974 (m), 968 (m), 889 (m), 783 (w), 747 (s) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 294 (17), 207 (3), 178 (45), 159 (100), 152 (61), 144 (20), 135 (72), 128 (19), 115 (25), 107 (13), 93 (28), 79 (28), 67 (10), 55 (8). HRMS (ESI) *m*/*e* calcd for $C_{21}H_{27}O$ ($M+H^+$) 295.2056, found 295.2062.



1-((*E*)-4-phenylbuta-1,3-dien-2-yl)adamantane (3g)
63%; a white solid (m.p. = 83-84 °C); ^1H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.3 Hz, 2H), 7.50 (d, *J* = 8.3 Hz, 2H), 6.96 (d, *J* = 15.8 Hz, 1H), 6.76 (d, *J* = 15.8 Hz, 1H), 5.24 (m, 1H), 4.86 (d, *J* = 1.1 Hz, 1H), 2.04 (m, 3H), 1.67-1.77 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 141.2, 130.8, 129.0 (q, *J* = 32.3 Hz), 128.3, 126.5, 125.5 (q, *J* = 3.8 Hz), 124.3 (q, *J* = 272.2 Hz), 108.2, 41.3, 37.2, 36.8, 28.6; IR (FT-IR): 2903 (m), 2849 (m), 1614 (w), 1449 (w),

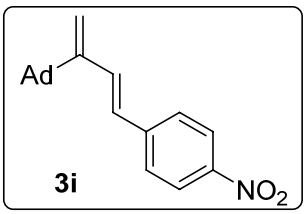
1413 (w), 1324 (s), 1164 (m), 1124 (s), 1066 (s), 1061 (w), 949 (w), 899 (w), 867 (w), 824 (m), 680 (w) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 332 (13), 313 (5), 305 (1), 235 (1), 209 (1), 196 (5), 177 (7), 159 (5), 135 (100), 127 (3), 107 (9), 93 (19), 79 (20), 67 (7), 55 (5). HRMS (ESI) *m*/*e* calcd for $C_{21}H_{23}F_3$ ($M+H^+$) 333.1825, found 333.1830.

methyl 4-((*E*)-3-((3s)-adamantan-1-yl)buta-1,3-dien-1-yl)benzoate (3h)



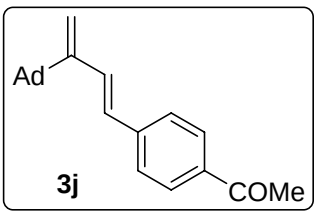
73%; a yellow solid (m.p. = 91-92 °C); ^1H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.3 Hz, 2H), 6.98 (d, *J* = 15.8 Hz, 1H), 6.77 (d, *J* = 15.8 Hz, 1H), 5.24 (m, 1H), 4.86 (d, *J* = 1.1 Hz, 1H), 3.91 (s, 3H), 2.04 (m, 3H), 1.68-1.77 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9, 156.2, 142.2, 130.8, 129.9, 128.7, 128.6, 126.2, 108.1, 52.0, 41.3, 37.2, 36.8, 28.6; IR (FT-IR): 2903 (m), 2848 (m), 1721 (s), 1606 (m), 1434 (m), 1411 (w), 1276 (s), 1177 (m), 1108 (m), 1016 (w), 954 (w), 898 (w), 873 (w), 767 (m), 701 (m) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 322 (8), 291 (3), 207 (3), 186 (12), 165 (5), 155 (5), 135 (100), 127 (6), 115 (5), 107 (10), 93 (20), 79 (19), 67 (7), 59 (5). HRMS (ESI) *m*/*e* calcd for $C_{22}H_{27}O_2$ ($M+H^+$) 323.2006, found 323.2009.

1-((*E*)-4-(4-nitrophenyl)buta-1,3-dien-2-yl)adamantane (3i)



59%; a yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ 8.17-8.19 (m, 2H), 7.52-7.54 (m, 2H), 7.05 (d, *J* = 16.2 Hz, 1H), 6.80 (d, *J* = 15.8 Hz, 1H), 5.28 (m, 1H), 4.91 (d, *J* = 1.0 Hz, 1H), 2.05 (m, 3H), 1.68-1.79 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8, 146.6, 144.1, 133.0, 127.6, 126.8, 124.0, 109.1, 41.3, 37.2, 36.7, 28.5; IR (FT-IR): 2904 (w), 2848 (w), 1594 (w), 1514 (m), 1449 (w), 1340 (s), 1109 (w), 953 (w), 873 (w), 862 (w), 836 (w), 750 (w) cm^{-1}; MS (EI, 70eV): *m*/*z* (%) = 309 (15), 178 (7), 165 (11), 152 (9), 135 (100), 128 (33), 115 (15), 107 (12), 79 (32), 67 (12), 55 (9). HRMS (ESI) *m*/*e* calcd for $C_{20}H_{23}NO_2$ ($M+H^+$) 310.1802, found 310.1801.

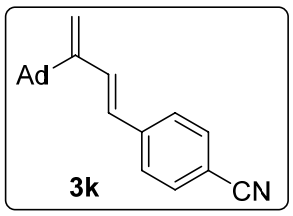
1-(4-((*E*)-3-((3s)-adamantan-1-yl)buta-1,3-dien-1-yl)phenyl)ethan-1-one (3j)



52%; a white solid (m.p. = 125-126 °C); ^1H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.00 (d, *J* = 15.8 Hz, 1H), 6.78 (d, *J* = 15.8 Hz, 1H), 5.25 (m, 1H), 4.87 (d, *J* = 1.1 Hz, 1H), 2.59 (s, 3H), 2.04 (m, 3H), 1.68-1.77 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.4, 156.1, 142.4, 135.7, 131.1, 128.7, 128.6, 126.4, 108.2, 41.3, 37.2, 36.8, 28.6, 26.5; IR (FT-IR): 2903 (m), 2846 (m), 1670 (s), 1631 (w), 1599 (m), 1445 (m), 1411 (m), 1360 (m), 1344 (w), 1305 (w), 1274 (m), 1181 (w), 1113 (w), 1075 (w), 955 (m), 897 (m), 870 (m), 830 (m), 815 (m), 735 (w), 697 (w) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 306 (13), 206 (6), 170 (7), 155 (6), 135 (100), 115 (1), 107 (11), 93

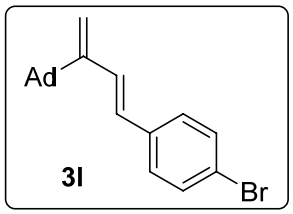
(21), 79 (23), 67 (8), 55 (5). HRMS (ESI) *m*/*e* calcd for $C_{22}H_{27}O$ ($M+H^+$) 307.20564, found 307.20536.

4-((*E*)-3-((3s)-adamantan-1-yl)buta-1,3-dien-1-yl)benzonitrile (3k)



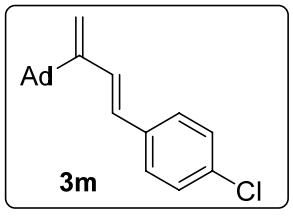
66%; a white solid (m.p. = 123-124 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 8.3 Hz, 2H), 6.99 (d, *J* = 15.9 Hz, 1H), 6.74 (d, *J* = 15.8 Hz, 1H), 5.25 (m, 1H), 4.89 (d, *J* = 1.0 Hz, 1H), 2.04 (m, 3H), 1.67-1.77 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.9, 142.2, 132.3, 132.1, 128.0, 126.8, 119.0, 110.3, 108.7, 41.3, 37.2, 36.7, 28.6; IR (FT-IR): 2903 (s), 2848 (m), 2221 (m), 1633 (w), 1602 (m), 1505 (w), 1451 (m), 1410 (w), 1344 (w), 1309 (w), 1175 (w), 1103 (w), 1074 (w), 974 (m), 949 (w), 894 (w), 870 (w), 825 (m), 815 (m), 738 (w), 667 (m) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 289 (15), 203 (3), 190 (4), 166 (4), 153 (10), 135 (100), 127 (13), 116 (6), 107 (8), 93 (17), 79 (18), 67 (7), 55 (4). HRMS (ESI) *m*/*e* calcd for $C_{21}H_{24}N$ ($M+H^+$) 290.1903, found 290.1909.

1-((*E*)-4-(4-bromophenyl)buta-1,3-dien-2-yl)adamantane (3l)



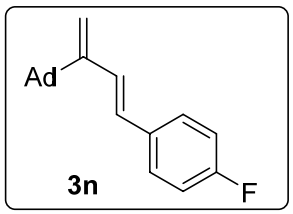
69%; a white solid (m.p. = 91-92 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 15.8 Hz, 1H), 6.68 (d, *J* = 15.8 Hz, 1H), 5.20 (m, 1H), 4.82 (d, *J* = 1.3 Hz, 1H), 2.03 (m, 3H), 1.67-1.76 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 136.6, 131.6, 128.9, 128.4, 127.9, 120.9, 107.5, 41.3, 37.1, 36.8, 28.6; IR (FT-IR): 2903 (s), 2847 (m), 1632 (w), 1602 (w), 1486 (m), 1448 (m), 1399 (w), 1344 (w), 1309 (w), 1257 (w), 1101 (w), 1071 (m), 1008 (m), 958 (m), 897 (m), 861 (m), 843 (w), 806 (s), 735 (w), 687 (w) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 344 (5), 342 (5), 281 (5), 207 (21), 191 (6), 178 (7), 165 (8), 162 (10), 135 (100), 107 (13), 93 (24), 79 (21), 67 (8), 55 (5). HRMS (ESI) *m*/*e* calcd for $C_{20}H_{24}Br$ ($M+H^+$) 343.1056, found 343.1061.

1-((*E*)-4-(4-chlorophenyl)buta-1,3-dien-2-yl)adamantane (3m)



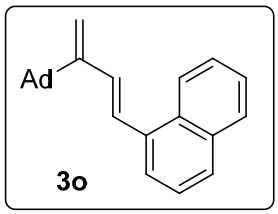
80%; a white solid (m.p. = 85-86 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 15.8 Hz, 1H), 6.69 (d, *J* = 15.8 Hz, 1H), 5.20 (m, 1H), 4.82 (d, *J* = 1.2 Hz, 1H), 2.03 (m, 3H), 1.67-1.76 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 136.2, 132.7, 128.8, 128.6, 128.4, 127.6, 107.5, 41.3, 37.1, 36.8, 28.6; IR (FT-IR): 2902 (s), 2847 (m), 1632 (w), 1592 (w), 1490 (m), 1449 (w), 1308 (w), 1091 (m), 1012 (m), 936 (m), 896 (m), 861 (m), 809 (s), 761 (w), 726 (w), 681 (w) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 298 (7), 222 (3), 178 (4), 162 (14), 135 (100), 115 (5), 107 (11), 93 (20), 79 (20), 67 (7), 55 (7). HRMS (ESI) *m*/*e* calcd for $C_{20}H_{24}Cl$ ($M+H^+$) 299.1561, found 299.1564.

1-((*E*)-4-(4-fluorophenyl)buta-1,3-dien-2-yl)adamantane (3n)



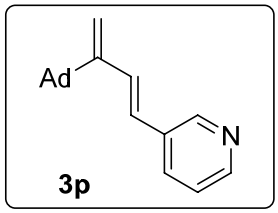
75%; a white solid (m.p. = 44-45 ^{o}C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.39 (m, 2H), 6.98-7.02 (m, 2H), 6.78 (d, *J* = 15.8 Hz, 1H), 6.70 (d, *J* = 15.8 Hz, 1H), 5.18 (m, 1H), 4.80 (d, *J* = 1.3 Hz, 1H), 2.03 (m, 3H), 1.67-1.76 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.1 (d, *J* = 246.5 Hz), 156.4, 133.9 (d, *J* = 3.3 Hz), 128.4, 128.0 (d, *J* = 2.3 Hz), 127.9 (d, *J* = 8.0 Hz), 115.4 (d, *J* = 21.8 Hz), 107.1, 41.3, 37.1, 36.8, 28.6; IR (FT-IR): 2903 (m), 2846 (m), 1681 (w), 1632 (w), 1601 (m), 1507 (s), 1451 (m), 1308 (w), 1231 (m), 1157 (m), 1095 (w), 1074 (w), 1013 (w), 958 (m), 897 (m), 861 (m), 821 (m), 790 (m), 736 (w), 708 (w) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 282 (10), 209 (2), 196 (4), 183 (5), 159 (36), 135 (100), 127 (3), 107 (12), 93 (20), 79 (19), 67 (7), 55 (5). HRMS (ESI) *m*/*e* calcd for $C_{20}H_{24}F$ ($M+H^+$) 283.1857, found 283.1860.

1-((*E*)-4-(naphthalen-1-yl)buta-1,3-dien-2-yl)adamantane (3o)



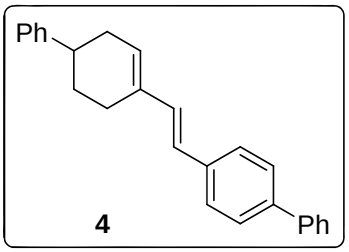
76%; a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, *J* = 7.9 Hz, 1H), 7.84 (d, *J* = 7.7 Hz, 1H), 7.76 (d, *J* = 8.2 Hz, 1H), 7.63 (d, *J* = 7.1 Hz, 1H), 7.43-7.52 (m, 4H), 6.89 (d, *J* = 15.4 Hz, 1H), 5.32 (m, 1H), 4.88 (m, 1H), 2.04 (m, 3H), 1.68-1.80 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.0, 135.5, 133.6, 131.4, 131.3, 128.4, 127.6, 126.7, 125.9, 125.7, 125.6, 123.9, 123.7, 107.4, 41.4, 37.2, 36.8, 28.6; IR (FT-IR): 2901 (m), 2847 (m), 1599 (w), 1507(w), 1448 (w), 1395 (w), 1344 (w), 1302 (w), 1251 (w), 1167 (w), 1102 (w), 1073 (w), 955 (m), 895 (m), 794 (w), 773 (s), 731 (w) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 314 (13), 263 (2), 241 (2), 229 (3), 216 (5), 202 (7), 189 (6), 178 (100), 166 (12), 152 (15), 136 (93), 127 (6), 116 (5), 107 (13), 95 (30), 79 (24), 67 (8), 55 (7). HRMS (ESI) *m*/*e* calcd for $C_{24}H_{27}$ ($M+H^+$) 315.2107, found 315.2110.

3-((*E*)-3-((3s)-adamantan-1-yl)buta-1,3-dien-1-yl)pyridine (3p)



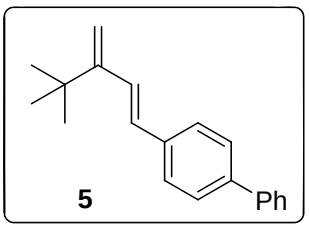
72%; a yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 1.8 Hz, 1H), 8.45 (dd, *J* = 1.3, 4.7 Hz, 1H), 7.73 (dt, *J* = 1.9, 7.9 Hz, 1H), 7.24 (m, 1H), 6.93 (d, *J* = 16.2 Hz, 1H), 6.72 (d, *J* = 15.8 Hz, 1H), 5.23 (m, 1H), 4.86 (d, *J* = 1.2 Hz, 1H), 2.04 (m, 3H), 1.67-1.77 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 148.4, 148.2, 133.3, 132.7, 130.5, 126.0, 123.4, 108.1, 41.3, 37.1, 36.8, 28.6; IR (FT-IR): 2902 (s), 2848 (s), 1633 (w), 1567 (w), 1479 (w), 1451 (w), 1414 (w), 1342 (w), 1306 (w), 1103 (w), 1074 (w), 1023 (w), 959 (m), 897 (m), 856 (w), 796 (m), 708 (s) cm^{-1}; MS (EI, 70eV): *m*/*z* (%) = 265 (27), 264 (27), 207 (9), 180 (8), 167 (11), 156 (10), 135 (100), 130 (48), 117 (18), 107 (23), 93 (41), 77 (50), 67 (19), 55 (16). HRMS (ESI) *m*/*e* calcd for $C_{19}H_{24}N$ ($M+H^+$) 266.1903, found 266.1902.

(*E*)-4-(2-(1,2,3,6-tetrahydro-[1,1'-biphenyl]-4-yl)vinyl)-1,1'-biphenyl (4)



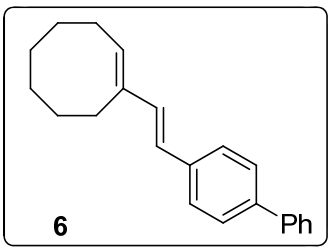
67%; a white solid (m.p. = 112-113 ^{o}C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.62 (m, 4H), 7.41-7.50 (m, 4H), 7.20-7.35 (m, 6H), 6.88 (d, *J* = 16.2 Hz, 1H), 6.51 (d, *J* = 16.2 Hz, 1H), 6.00 (m, 1H), 2.83-2.90 (m, 1H), 2.37-2.56 (m, 4H), 2.08-2.13 (m, 1H), 1.85-1.89 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.7, 140.8, 139.6, 136.9, 135.7, 132.0, 130.1, 128.7, 128.4, 127.2, 126.9, 126.8, 126.8, 126.6, 126.1, 124.9, 40.2, 34.2, 29.6, 25.1; IR (FT-IR): 3027 (w), 2923 (m), 2825 (w), 1601 (w), 1486 (w), 1450 (w), 1076 (w), 1027 (w), 965 (w), 909 (w), 834 (w), 760 (s), 697 (s) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 336 (99), 232 (100), 217 (53), 204 (43), 191 (24), 178 (11), 167 (19), 152 (16), 141 (16), 128 (12), 115 (16), 104 (17), 91 (33), 77 (11), 65 (4), 51 (4). HRMS (ESI) *m*/*e* calcd for $C_{26}H_{25}$ (M+H^{+}) 337.1951, found 337.1948.

(*E*)-4-(4,4-dimethyl-3-methylenepent-1-en-1-yl)-1,1'-biphenyl (5)



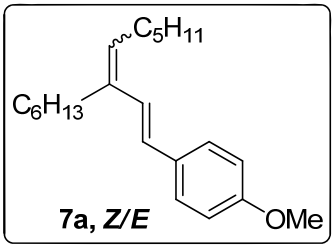
91%; a white solid (m.p. = 105-106 ^{o}C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.61 (m, 4H), 7.41-7.50 (m, 4H), 7.31-7.35 (m, 1H), 6.88 (d, *J* = 15.8 Hz, 1H), 6.81 (d, *J* = 15.8 Hz, 1H), 5.22 (s, 1H), 4.90 (d, *J* = 1.0 Hz, 1H), 1.16 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.0, 140.7, 140.1, 136.7, 129.1, 128.8, 128.8, 127.2, 126.9, 126.9, 107.3, 35.4, 29.5; IR (FT-IR): 2964 (w), 1486 (w), 1363 (w), 1151 (w), 965 (w), 907 (s), 826 (w), 764 (m), 732 (s) cm^{-1}; MS (EI, 70 eV): *m*/*z* (%) = 262 (9), 205 (100), 190 (12), 178 (15), 165 (17), 152 (13), 128 (9), 115 (7), 77 (9), 57 (11). HRMS (ESI) *m*/*e* calcd for $C_{26}H_{25}$ (M+H^{+}) 263.1794, found 263.1791.

4-((*E*)-2-((*E*)-cyclooct-1-en-1-yl)vinyl)-1,1'-biphenyl (6)



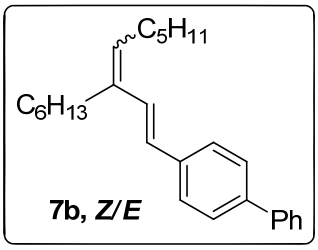
84%; a white solid (m.p. = 109-110 ^{o}C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54-7.65 (m, 4H), 7.40-7.48 (m, 4H), 7.30-7.35 (m, 1H), 6.79 (d, *J* = 16.1 Hz, 1H), 6.51 (d, *J* = 16.1 Hz, 1H), 5.89 (t, *J* = 8.3 Hz, 1H), 2.51-2.54 (m, 2H), 2.24-2.29 (m, 2H), 1.47-1.66 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.8, 139.4, 137.1, 133.9, 132.4, 128.8, 128.7, 127.2, 127.1, 126.8, 126.5, 124.7, 30.4, 28.6, 27.4, 27.0, 26.0, 24.4; IR (FT-IR): 2917 (m), 2848 (w), 1487 (w), 1448 (w), 1408 (w), 969 (m), 909 (w), 842 (m), 761 (s), 733 (m), 689 (m) cm^{-1}; MS (EI, 70eV): *m*/*z* (%) = 288 (82), 260 (77), 245 (30), 231 (35), 217 (93), 205 (100), 189 (40), 178 (55), 165 (73), 152 (59), 141 (16), 128 (22), 115 (35), 91 (30), 77 (41), 67 (26), 55 (25). HRMS (ESI) *m*/*e* calcd for $C_{22}H_{25}$ (M+H^{+}) 289.1951, found 289.1954.

1-(3-hexylnona-1,3-dien-1-yl)-4-methoxybenzene (7a)



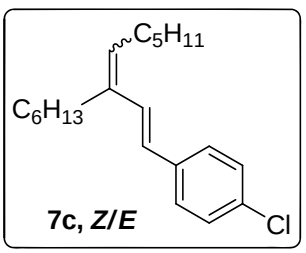
93% (1:1.2); a yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.38 (m, 4.4H), 6.97 (d, *J* = 16.2 Hz, 1.2H), 6.83-6.88 (m, 4.4H), 6.49-6.60 (m, 2.2H), 6.40 (d, *J* = 16.2 Hz, 1H), 5.54 (t, *J* = 7.5 Hz, 1H), 5.40 (t, *J* = 7.5 Hz, 1.2H), 3.80-3.81 (m, 6.6H), 2.13-2.32 (m, 8.8H), 1.25-1.42 (m, 30.8H), 0.88-0.91 (m, 13.2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.9, 158.6, 138.5, 136.3, 133.3, 131.1, 131.0, 130.9, 130.7, 127.4, 127.1, 126.9, 124.5, 123.5, 114.0, 114.0, 55.2, 55.2, 34.0, 31.7, 31.6, 31.5, 29.7, 29.7, 29.6, 29.4, 29.3, 29.1, 29.0, 28.3, 27.5, 26.8, 22.6, 22.6, 22.6, 14.0, 14.0, 14.0; IR (FT-IR): 2955 (m), 2924 (m), 1605 (m), 1509 (s), 1464 (m), 1377 (w), 1302 (w), 1247 (s), 1173 (m), 1039 (m), 959 (m), 820 (m), 721 (w) cm^{-1}; MS (EI, 70 eV): ***isomer a***, *m*/*z* (%) = 314 (57), 257 (36), 243 (27), 229 (99), 185 (13), 173 (48), 159 (79), 144 (22), 129 (16), 121 (100), 91 (17), 77 (13), 55 (15); ***isomer b***, *m*/*z* (%) = 314 (61, 257 (25), 243 (26), 229 (100), 185 (16), 173 (52), 159 (81), 144 (24), 129 (17), 121 (93), 91 (19), 77 (12), 55 (20). HRMS (ESI) *m*/*e* calcd for $C_{22}H_{35}O$ ($M+H^+$) 315.2682, found 315.2691.

4-(3-hexylnona-1,3-dien-1-yl)-1,1'-biphenyl (7b)



90% (1:1.1); a yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.62 (m, 16.8H), 7.30-7.35 (m, 2.1H), 7.15 (d, *J* = 16.2 Hz, 1.1H), 6.75 (d, *J* = 16.2 Hz, 1H), 6.60 (d, *J* = 16.2 Hz, 1.1H), 6.48 (d, *J* = 16.2 Hz, 1H), 5.62 (t, *J* = 7.4 Hz, 1H), 5.47 (t, J = 7.4 Hz, 1.1H), 2.17-2.35 (m, 8.4H), 1.32-1.52 (m, 29.4H), 0.88-0.91 (m, 12.6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.8, 140.8, 139.8, 139.4, 138.5, 137.3, 137.2, 136.3, 134.6, 133.2, 131.9, 128.7, 127.2, 127.1, 127.1, 127.0, 126.8, 126.8, 126.7, 126.5, 125.5, 124.6, 34.0, 31.7, 31.6, 31.5, 29.7, 29.6, 29.4, 29.3, 29.1, 29.0, 28.4, 27.6, 26.8, 22.6, 22.6, 14.1, 14.0, 14.0; IR (FT-IR): 2956 (s), 2927 (s), 2857 (m), 1487 (m), 1466 (w), 1408 (w), 1378 (w), 1075 (w), 961 (m), 866 (w), 824 (w), 761 (s), 723 (m), 696 (s) cm^{-1}; MS (EI, 70eV): ***isomer a***, *m*/*z* (%) = 360 (40), 303 (20), 289 (13), 275 (60), 219 (37), 205 (100), 191 (27), 178 (27), 167 (93), 152 (19), 141 (6), 128 (6), 115 (7), 91 (9), 77 (8), 67 (6), 55 (17); ***isomer b***, *m*/*z* (%) = 360 (41), 303 (9), 289 (14), 275 (53), 219 (32), 205 (100), 191 (25), 178 (26), 167 (70), 152 (16), 141 (6), 128 (6), 115 (7), 91 (9), 77 (8), 67 (6), 55 (16). HRMS (ESI) *m*/*e* calcd for $C_{27}H_{37}$ ($M+H^+$) 361.2890, found 361.2890.

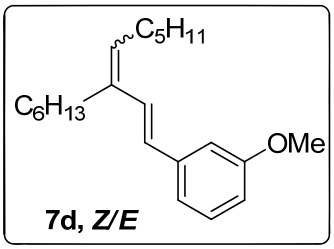
1-chloro-4-(3-hexylnona-1,3-dien-1-yl)benzene (7c)



73% (1:1.2); a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.36 (m, 8.8H), 7.07 (d, *J* = 16.2 Hz, 1.2H), 6.66 (d, *J* = 16.2 Hz, 1.2H), 6.50 (d, *J* = 16.2 Hz, 1H), 6.38 (d, *J* = 16.2 Hz, 1H), 5.60 (t, *J* = 7.4 Hz, 1.2H), 5.47 (t, *J* = 7.4 Hz, 1H), 2.14-2.32 (m, 8.8H), 1.31-1.49 (m, 30.8H), 0.88-0.92 (m, 13.2H); ^{13}C NMR (100 MHz,

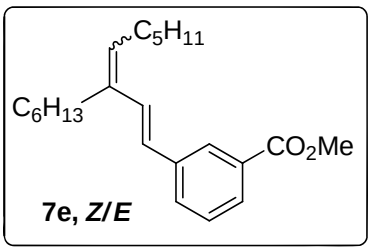
$CDCl_3$) δ 138.3, 136.7, 136.6, 136.0, 135.1, 133.7, 132.5, 132.3, 132.2, 128.6, 128.6, 127.4, 127.2, 126.1, 125.9, 123.7, 33.9, 31.7, 31.6, 31.5, 29.6, 29.6, 29.3, 29.2, 29.0, 28.9, 28.4, 27.6, 26.8, 22.6, 22.5, 14.0, 14.0, 14.0; IR (FT-IR): 2956 (s), 2927 (s), 2857 (m), 1626 (w), 1490 (s), 1457 (m), 1404 (w), 1378 (w), 1092 (s), 1012 (m), 960 (s), 864 (w), 843 (w), 811 (m), 725 (w) cm^{-1}; MS (EI, 70 eV): ***isomer a***, *m*/*z* (%) = 318 (49), 263 (6), 261 (19), 247 (7), 233 (79), 221 (18), 206 (7), 191 (20), 177 (78), 163 (55), 153 (38), 143 (62), 125 (100), 115 (35), 103 (7), 91 (13), 77 (14), 67 (11), 55 (24); ***isomer b***, *m*/*z* (%) = 318 (51), 283 (5), 261 (15), 247 (7), 233 (74), 222 (22), 206 (7), 191 (21), 177 (77), 163 (60), 153 (51), 143 (62), 125 (100), 115 (38), 102 (7), 91 (9), 77 (10), 67 (10), 55 (26). HRMS (ESI) *m*/*e* calcd for $C_{21}H_{32}Cl$ (M^+) 319.2187, found 319.2186.

1-(3-hexylnona-1,3-dien-1-yl)-3-methoxybenzene (7d)



87% (1:1.2); a colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.19-7.25 (m, 2.2H), 6.94-7.11 (m, 5.4H), 6.67-6.78 (m, 3.4H), 6.53 (d, *J* = 16.2 Hz, 1.2H), 6.42 (d, *J* = 16.2 Hz, 1H), 5.60 (t, *J* = 7.4 Hz, 1H), 5.45 (t, *J* = 7.4 Hz, 1.2H), 3.82-3.83 (m, 6.6H), 2.14-2.33 (m, 8.8H), 1.26-1.50 (m, 30.8H), 0.90 (m, 13.2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 139.7, 139.6, 138.4, 136.2, 134.6, 133.4, 131.9, 129.4, 129.4, 127.3, 125.7, 125.0, 119.0, 118.8, 112.5, 111.7, 111.2, 55.2, 55.1, 33.9, 31.7, 31.6, 31.5, 29.6, 29.6, 29.4, 29.2, 29.0, 28.9, 28.4, 27.5, 26.8, 22.6, 22.5, 14.0, 14.0; IR (FT-IR): 2956 (s), 2927 (s), 2857 (m), 1597 (m), 1578 (m), 1489 (w), 1465 (m), 1432 (w), 1378 (w), 1285 (m), 1270 (m), 1156 (m), 1051 (m), 961 (m), 865 (w), 771 (m), 725 (w), 689 (m) cm^{-1}; MS (EI, 70eV): ***isomer a***, *m*/*z* (%) = 314 (46), 257 (15), 243 (59), 229 (76), 187 (7), 173 (44), 159 (100), 144 (26), 129 (17), 121 (54), 91 (22), 77 (10), 67 (7), 55 (20); ***isomer b***, *m*/*z* (%) = 314 (46), 257 (8), 243 (57), 229 (67), 185 (9), 173 (39), 159 (100), 144 (28), 129 (18), 121 (46), 91 (19), 77 (13), 67 (7), 55 (20). HRMS (ESI) *m*/*e* calcd for $C_{22}H_{35}O$ ($M+H^+$) 315.2682, found 315.2684.

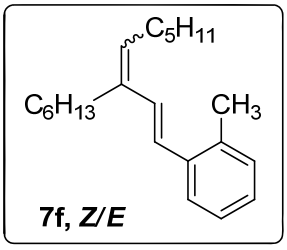
Methyl 3-(3-hexylnona-1,3-dien-1-yl)benzoate (7e)



86% (1:1.6); a yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ 8.07-8.09 (m, 2.6H), 7.83-7.88 (m, 2.6H), 7.57-7.63 (m, 2.6H), 7.34-7.41 (m, 2.6H), 7.16 (d, J = 16.2 Hz, 1.6H), 6.77 (d, *J* = 16.2 Hz, 1H), 6.59 (d, *J* = 16.2 Hz, 1.6H), 6.47 (d, *J* = 16.2 Hz, 1H), 5.64 (t, *J* = 7.4 Hz, 1H), 5.49 (t, *J* = 7.4 Hz, 1.6H), 3.92-3.93 (m, 7.8H), 2.15-2.34 (m, 10.4H), 1.31-1.50 (m, 36.4H), 0.88-0.92 (m, 15.6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 167.1, 138.5, 138.5, 138.3, 136.0, 135.4, 134.3, 132.6, 130.5, 130.4, 130.3, 130.2, 128.5, 128.5, 127.9, 127.6,

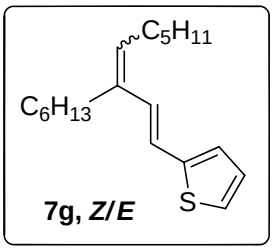
127.5, 127.2, 126.5, 124.0, 52.1, 52.0, 33.9, 31.7, 31.7, 31.6, 31.5, 29.6, 29.3, 29.2, 29.0, 28.9, 28.4, 27.6, 26.8, 22.6, 22.5, 14.0, 14.0; IR (FT-IR): 2953 (m), 2927 (m), 2856 (m), 1726 (s), 1581 (w), 1438 (m), 1289 (s), 1255 (m), 1203 (m), 1107 (m), 959 (m), 750 (m), 686 (m) cm^{-1}; MS (EI, 70 eV): ***isomer a***, *m*/*z* (%) = 342 (49), 331 (7), 311 (14), 285 (27), 271 (17), 257 (100), 239 (17), 215 (18), 201 (82), 187 (29), 169 (28), 155 (50), 143 (92), 128 (56), 115 (43), 105 (16), 91 (35), 81(17), 67 (18), 55 (30); ***isomer b***, *m*/*z* (%) = 342 (58), 311 (20), 285 (15), 271 (18), 257 (100), 239 (20), 215 (16), 201 (78), 187 (29), 169 (32), 155 (50), 143 (92), 128 (62), 115 (43), 105 (15), 91 (31), 81(13), 67 (13), 55 (30). HRMS (ESI) *m*/*e* calcd for $C_{23}H_{35}O_2$ ($M+H^+$) 343.263, found 343.263.

1-(3-hexylnona-1,3-dien-1-yl)-2-methylbenzene (7f)



85% (1:1.3); a colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.47-7.51 (m, 2.3H), 7.10-7.19 (m, 6.9H), 6.96 (d, *J* = 16.1 Hz, 1.3H), 6.78 (d, J = 16.1 Hz, 1.3H), 6.67 (d, *J* = 16.1 Hz, 1H), 6.57 (d, *J* = 16.1 Hz, 1H), 5.59 (t, *J* = 7.4 Hz, 1H), 5.45 (t, *J* = 7.5 Hz, 1.3H), 2.16-2.36 (m, 16.1H), 1.32-1.44 (m, 32.2H), 0.88-0.91 (m, 13.8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.8, 137.3, 137.1, 136.6, 135.4, 135.2, 134.2, 134.2, 131.6, 130.2, 130.2, 127.0, 126.6, 126.6, 126.0, 126.0, 125.4, 125.2, 124.9, 122.9, 34.2, 31.7, 31.6, 31.5, 29.7, 29.6, 29.4, 29.3, 29.0, 28.3, 27.5, 26.9, 22.6, 22.6, 22.5, 19.8, 19.8, 14.0, 14.0; IR (FT-IR): 2956 (s), 2926 (s), 2857 (m), 1599 (w), 1483 (w) 1460 (m), 1378 (w), 1051 (w), 961 (s), 748 (s), 717 (w) cm^{-1}; MS (EI, 70eV): ***isomer a***, *m*/*z* (%) = 298 (26), 241 (8), 227 (14), 213 (42), 171 (6), 157 (36), 143 (100), 129 (31), 128 (32), 115 (24), 105 (42), 91 (12), 79 (8), 67 (4), 55 (11); ***isomer b***, *m*/*z* (%) = 298 (29), 241 (7), 227 (14), 213 (42), 183 (7), 157 (35), 143 (100), 129 (29), 115 (19), 105 (47), 91 (12), 77 (8), 67 (4), 55 (11). HRMS (ESI) *m*/*e* calcd for $C_{22}H_{35}$ ($M+H^+$) 299.2733, found 299.2733.

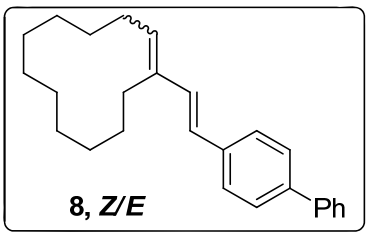
2-(3-hexylnona-1,3-dien-1-yl)thiophene (7g)



62% (1:1.2); a yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.09-7.14 (m, 2.2H), 6.90-6.97 (m, 5.4H), 6.68 (d, *J* = 16.0 Hz, 1H), 6.58 (d, *J* = 16.0 Hz, 1.2H), 6.52 (d, *J* = 16.0 Hz, 1.2H), 5.56 (t, *J* = 7.4 Hz, 1.2H), 5.43 (t, *J* = 7.5 Hz, 1H), 2.12-2.29 (m, 8.8H), 1.30-1.41 (m, 30.8H), 0.88-0.92 (m, 13.2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.9, 143.8, 138.1, 135.9, 134.4, 133.1, 131.8, 127.4, 127.3, 125.4, 125.1, 124.6, 123.6, 123.2, 120.7, 118.4, 33.8, 31.7, 31.7, 31.6, 31.4, 29.6, 29.6, 29.3, 29.2, 29.0, 28.9, 28.4, 27.5, 26.7, 22.6, 22.6, 22.5, 22.5, 14.0, 14.0, 14.0; IR (FT-IR): 2956 (m), 2926 (m), 2857 (m), 1466 (w), 1378 (w), 1202 (w), 1043 (w), 948 (m), 854 (w), 818 (w), 770 (w), 752 (w), 690 (s)

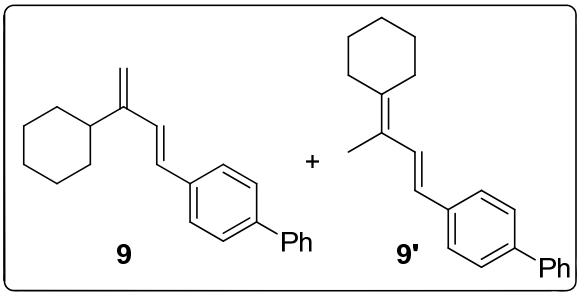
cm^{-1}; MS (EI, 70 eV): ***isomer a***, *m*/*z* (%) = 290 (82), 233 (29), 219 (29), 205 (94), 194 (7), 175 (8), 161 (31), 149 (86), 135 (100), 128 (32), 115 (49), 97 (98), 85 (8), 77 (21), 65 (11), 55 (24); ***isomer b***, *m*/*z* (%) = 290 (81), 233 (28), 219 (32), 205 (94), 191 (7), 178 (8), 161 (31), 149 (86), 135 (100), 128 (32), 115 (49), 97 (98), 85 (8), 77 (21), 65 (11), 55 (24). HRMS (ESI) *m*/*e* calcd for $C_{19}H_{31}S$ ($M+H^+$) 291.2141, found 291.2141.

1-((1E)-2-(cyclododec-1-en-1-yl)vinyl)-4,1'-biphenyl (8)



78% (1:0.4); a white solid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.51-7.63 (m, 5.6H), 7.41-7.48 (m, 5.6H), 7.30-7.36 (m, 1.4H), 7.19 (d, *J* = 16.2 Hz, 0.4H), 6.75 (d, *J* = 16.2 Hz, 1H), 6.65 (d, *J* = 16.2 Hz, 0.4H), 6.52 (d, *J* = 16.2 Hz, 1H), 5.61-5.68 (m, 1.4H), 2.33-2.45 (m, 3.6H), 2.19-2.25 (m, 2H), 1.25-1.65 (m, 22.4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.8, 140.1, 139.8, 139.4, 138.2, 137.3, 137.2, 135.3, 135.1, 134.5, 133.3, 128.8, 128.7, 127.5, 127.2, 127.2, 127.1, 127.0, 126.9, 126.8, 126.7, 126.4, 125.3, 125.1, 33.7, 29.7, 27.3, 26.9, 26.6, 26.5, 26.4, 25.7, 25.6, 25.6, 25.4, 25.3, 25.1, 25.0, 25.0, 23.7, 23.7, 23.5, 23.1, 22.3; IR (FT-IR): 2927 (s), 2850 (m), 1487 (w), 1466 (w), 1408 (w), 968 (m), 908 (m), 831 (m), 760 (s), 734 (s), 692 (s) cm^{-1}; MS (EI, 70eV): ***isomer a***, *m*/*z* (%) = 344 (81), 273 (8), 259 (10), 245 (15), 231 (29), 217 (68), 205 (100), 191 (37), 178 (40), 167 (58), 152 (29), 141 (13), 128 (13), 115 (16), 91 (20), 77 (21), 67 (18), 55 (41); ***isomer b***, *m*/*z* (%) = 344 (70), 245 (13), 231 (28), 217 (70), 205 (99), 191 (43), 180 (49), 167 (100), 152 (48), 127 (13), 115 (30), 91 (28), 77 (26), 67 (24), 55 (59). HRMS (ESI) *m*/*e* calcd for $C_{19}H_{31}S$ ($M+H^+$) 345.2577, found 345.2581.

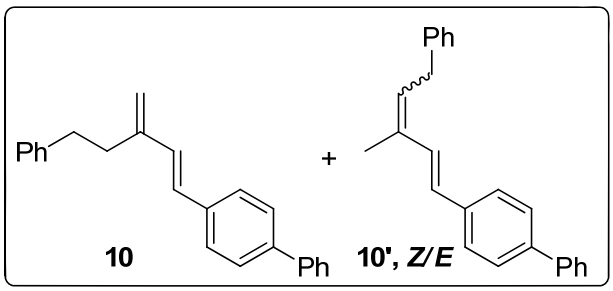
(*E*)-4-(3-cyclohexylbuta-1,3-dien-1-yl)-1,1'-biphenyl (9)
(*E*)-4-(3-cyclohexylidenebut-1-en-1-yl)-1,1'-biphenyl (9')



91% (**9** : **9'** > 1 : 0.05); a white solid (m.p. = 85-86 °C); ^1H NMR (400 MHz, $CDCl_3$) δ 7.55-7.62 (m, 4H), 7.49-7.51 (m, 2H), 7.41-7.45 (m, 2H), 7.31-7.35 (m, 1H), 6.80 (d, *J* = 16.3 Hz, 1H), 6.66 (d, *J* = 16.3 Hz, 1H), 5.15 (s, 1H), 5.05 (s, 1H), 2.33-2.39 (m, 1H), 1.74-1.92 (m, 4H), 1.35-1.45 (m, 2H),1.19-1.30 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.9, 140.7, 139.9, 136.6, 131.2, 128.7, 127.2, 126.9, 126.8, 126.6, 113.4, 39.8, 33.1, 26.9, 26.5; IR (FT-IR): 2923 (m), 2852 (w), 1487 (w), 1449 (w), 965 (w), 866 (m), 827 (w), 764 (s), 722 (w), 692 (m) cm^{-1}; MS (EI, 70eV): ***isomer a***, m/z (%) = 288(13), 205(100), 189(10), 178(14), 165(15), 152(13), 127(8), 117(7), 91(7), 77(7), 67(7), 55(13); ***isomer b***, m/z (%) = 288(10), 215(14), 205(100), 191(16), 178(18), 165(24), 152(19), 127(8), 115(11), 91(14), 77(16), 67(17), 55(22). HRMS (ESI) *m*/*e*

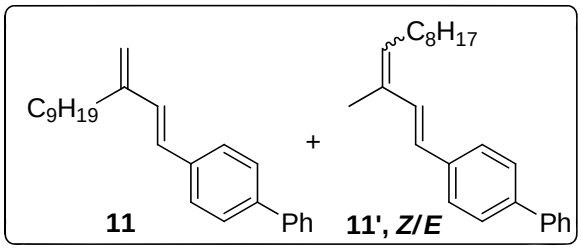
calcd for $C_{24}H_{23}$ ($M+H^+$) 289.19508, found 289.19485.

(*E*)-4-(3-methylene-5-phenylpent-1-en-1-yl)-1,1'-biphenyl (10)
4-(3-methyl-5-phenylpenta-1,3-dien-1-yl)-1,1'-biphenyl (10'):



56% (**10** : **10'** = 1 : (0.15:0.15)); a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.62 (m, 10.4H), 7.21-7.35 (m, 7.8H), 6.88 (d, *J* = 16.3 Hz, 1.3H), 6.54-6.70 (m, 1.3H), 5.84 (t, *J* = 7.5 Hz, 0.15H), 5.65 (t, *J* = 7.9 Hz, 0.15H), 5.19 (s, 1H), 5.09 (d, *J* = 0.7 Hz, 1H), 3.65 (d, *J* = 7.7 Hz, 0.3H), 3.57 (d, *J* = 7.6 Hz, 0.3H), 2.88-2.92 (m, 2H), 2.64-2.68 (m, 2H), 2.01-2.02 (m, 0.45H), 2.00 (s, 0.45H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.5, 142.1, 140.6, 140.2, 136.4, 130.9, 128.7, 128.5, 128.4, 128.3, 128.2, 128.1, 127.6, 127.2, 126.8, 126.8, 125.9, 116.6, 34.7, 33.9; IR (FT-IR): 3027 (w), 2925 (w), 1601 (w), 1486 (w), 1453 (w), 1409 (w), 1076 (w), 1029 (w), 1006 (w), 966 (w), 909 (w), 868 (w), 828 (w), 763 (s), 727 (m), 697 (s) cm^{-1}; MS (EI, 70eV): ***isomer a***, *m*/*z* (%) = 310 (70), 295 (24), 280 (28), 265 (10), 253 (7), 232 (17), 219 (77), 205 (90), 191 (22), 178 (32), 165 (47), 152 (48), 143 (27), 128 (35), 115 (38), 105 (20), 91 (100), 77 (27), 65 (22), 51 (17); ***isomer b***, *m*/*z* (%) = 310 (23), 295 (11), 273 (51), 259 (69), 230 (74), 219 (29), 205 (29), 191 (13), 183 (100), 167 (41), 152 (60), 141 (13), 128 (17), 115 (33), 108 (49), 91 (49), 77 (30), 65 (11), 51 (30); ***isomer c***, *m*/*z* (%) = 310 (88), 295 (31), 280 (20), 232 (27), 219 (100), 204 (49), 191 (25), 178 (32), 165 (41), 152 (47), 143 (37), 128 (37), 115 (41), 105 (13), 91 (86), 77 (31), 65 (16), 51 (18). HRMS (ESI) *m*/*e* calcd for $C_{24}H_{23}$ ($M+H^+$) 311.1794, found 311.1787.

(*E*)-4-(3-methylenedodec-1-en-1-yl)-1,1'-biphenyl (11)
4-(3-methyldodeca-1,3-dien-1-yl)-1,1'-biphenyl (11')



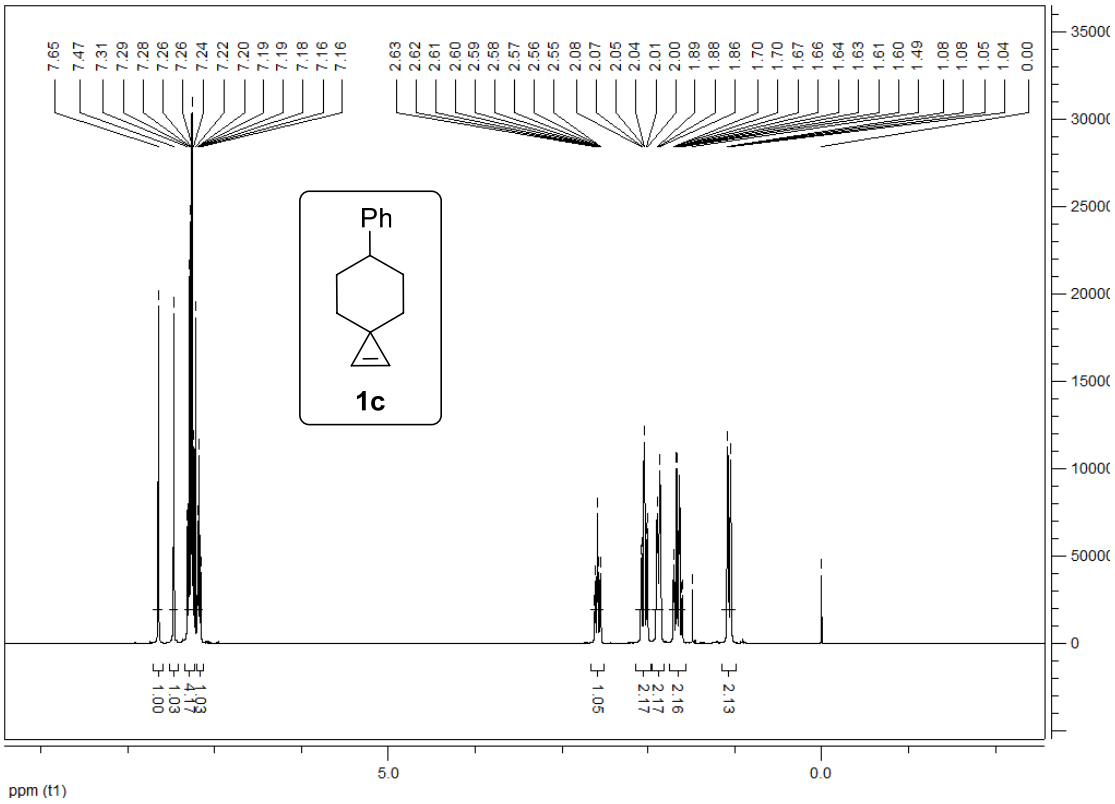
81% (**11** : **11'** = 1 : (0.18:0.16)); a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.61 (m, 10.72H), 7.31-7.35 (m, 1.34H), 6.84 (d, *J* = 16.3 Hz, 1.34H), 6.46-6.63 (m, 1.34H), 5.66 (t, *J* = 7.4 Hz, 0.18H), 5.48 (t, *J* = 7.4 Hz, 0.16H), 5.15 (s, 1H), 5.07 (s, 1H), 2.32-2.35 (m, 2H), 2.25-2.29 (m, 0.32H), 2.17-2.22 (m, 0.36H), 1.95 (m, 0.48H), 1.87 (s, 0.54H), 1.26-1.28 (m, 18.08H), 0.87-0.90 (m, 4.02H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.4, 140.7, 140.0, 137.2, 136.5, 134.7, 134.2, 132.6, 131.9, 131.2, 129.8, 128.7, 128.6, 127.5, 127.4, 127.2, 127.2, 127.1, 126.9, 126.8, 126.8, 126.7, 126.6, 126.5, 126.2, 124.9, 116.1, 77.3, 77.0, 76.6, 32.1, 31.9, 29.9, 29.7, 29.6, 29.6, 29.5, 29.5, 29.4, 29.3, 29.3, 28.5, 28.4, 27.6, 22.6, 20.5, 14.1, 12.3;

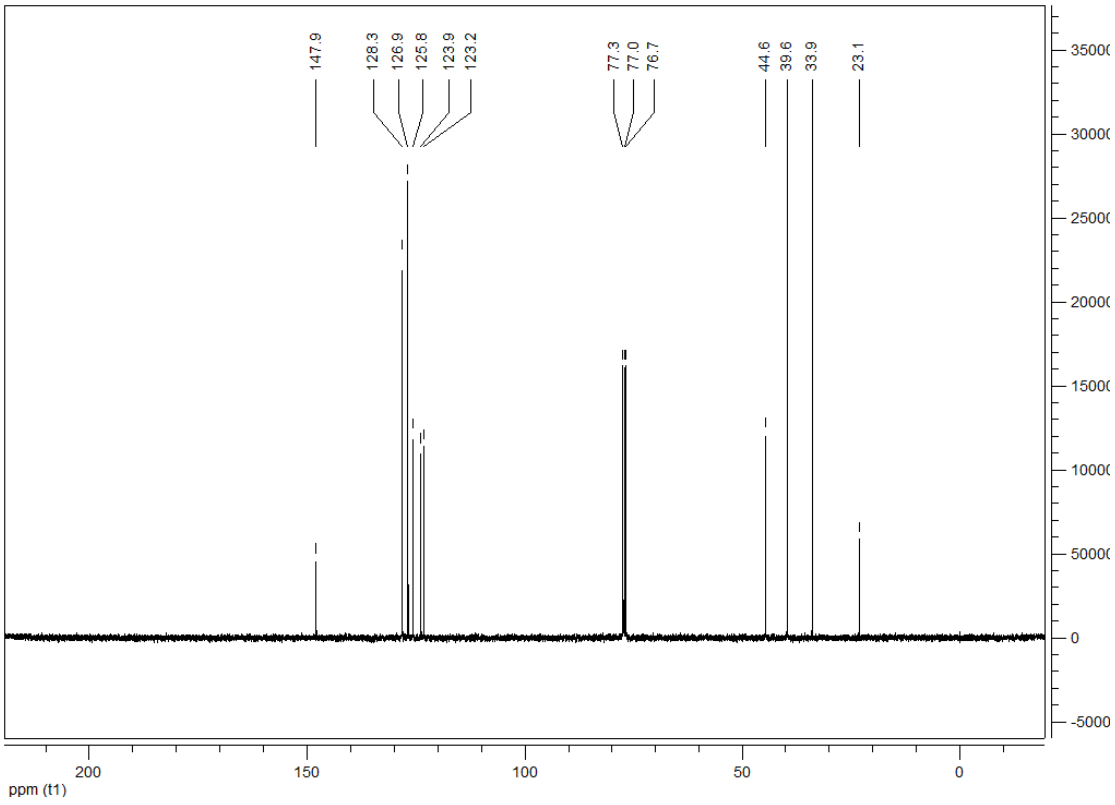
IR (FT-IR): 2922 (m), 2851 (w), 1487 (w), 1467 (w), 963 (w), 908 (m), 867 (m), 827 (w), 760 (m), 734 (s) cm^{-1}; MS (EI, 70eV): ***isomer a***, *m*/*z* (%) = 332 (52), 312 (7), 233 (61), 219 (100), 205 (52), 191 (21), 178 (15), 167 (41), 152 (13), 128 (7), 115 (9), 91 (11), 77 (9), 55 (9); ***isomer b***, *m*/*z* (%) = 332 (33), 233 (28), 219 (54), 205 (100), 191 (13), 178 (12), 167 (28), 152 (10), 128 (6), 115 (6), 91 (10), 77 (7), 55 (9); ***isomer c***, *m*/*z* (%) = 332 (48), 317 (19), 233 (45), 219 (100), 205 (56), 191 (21), 178 (13), 167 (31), 152 (10), 128 (6), 115 (6), 91 (10), 77 (9), 55 (10). HRMS (ESI) *m*/*e* calcd for $C_{25}H_{33}$ ($M+H^+$) 333.2577, found 333.2569.

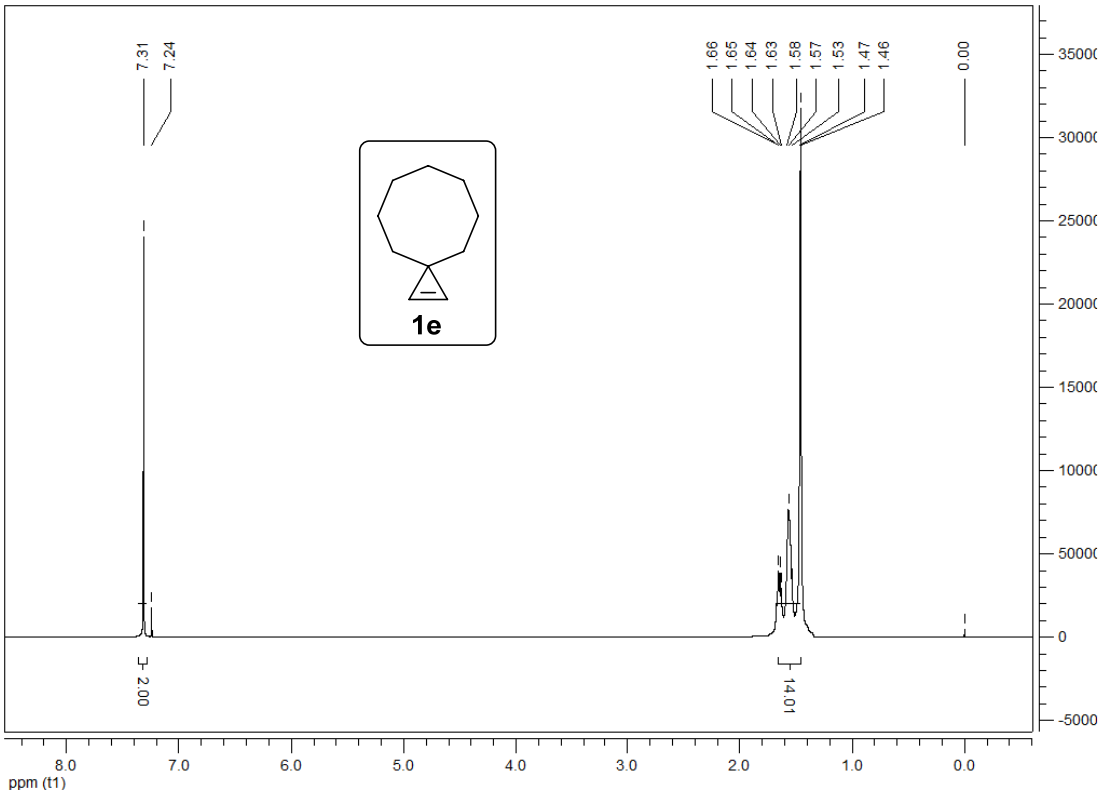
6. References

1) Young, P. C.; Hadfield, M. S.; Arrowsmith, L.; Macleod, K. M.; Mudd, R. J.; Jordan-Hore, J. A.; Lee, A.- L. *Org. Lett*. **2012**, *14*, 898.
2) Giudici, R. E.; Hoveyda, A. H. *J. Am. Chem. Soc*. **2007**, *129*, 3824.
3) Mudd, R. J.; Young, P. C.; Jordan-Hore, J. A.; Rosair, G. M.; Lee, A.-L. *J. Org. Chem*. **2012**, *77*, 7633.
4) Bauer, J. T.; Hadfield, M. S.; Lee, A. –L. *Chem. Commun*. **2008**, 6405.

7. ^{1}H and ^{13}C NMR



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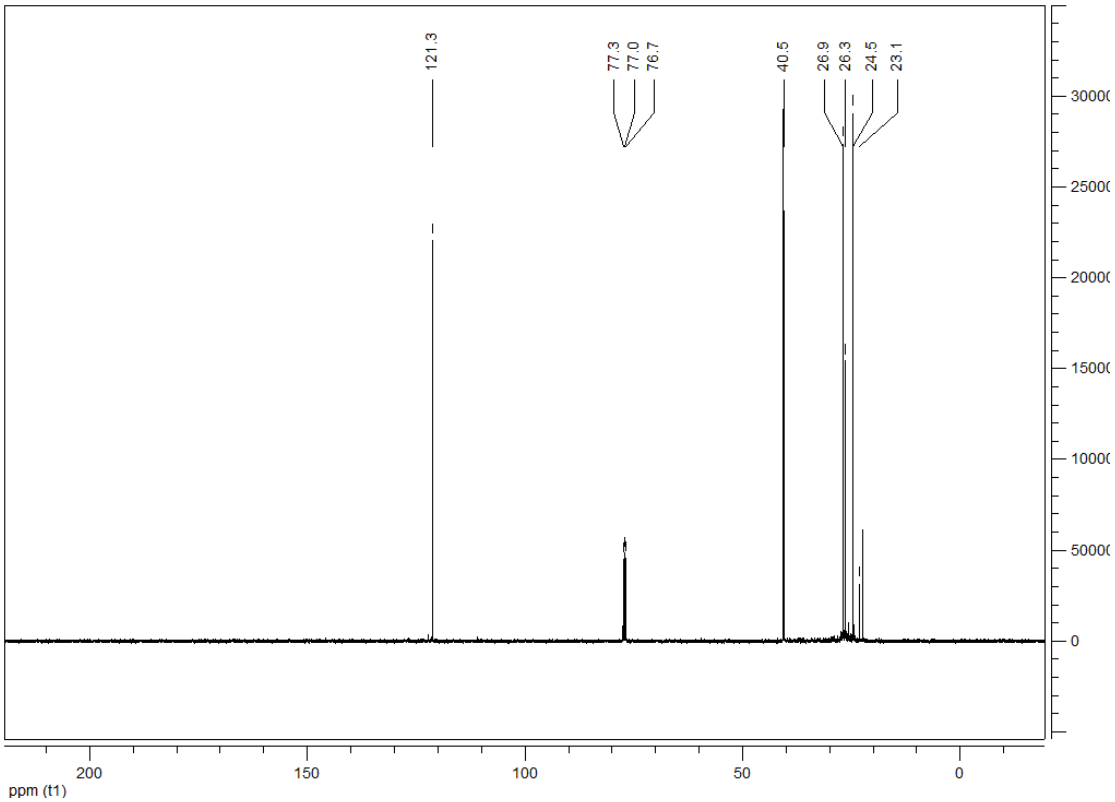
ppm (t1)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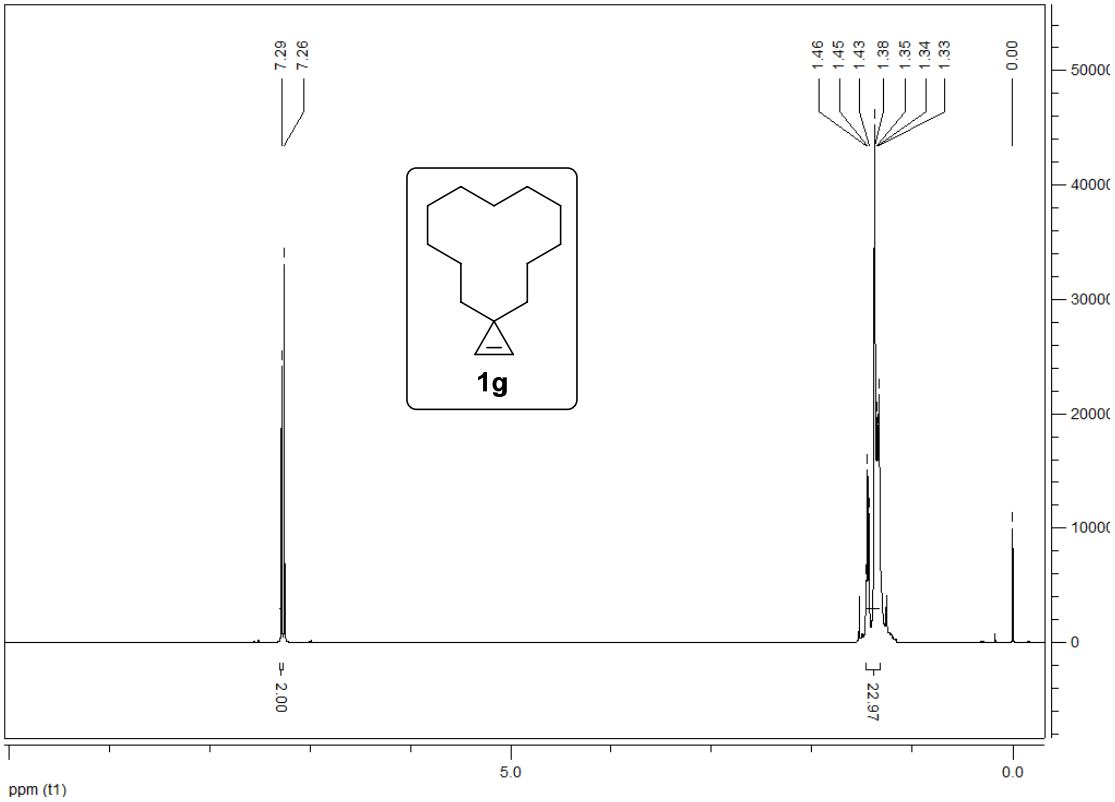
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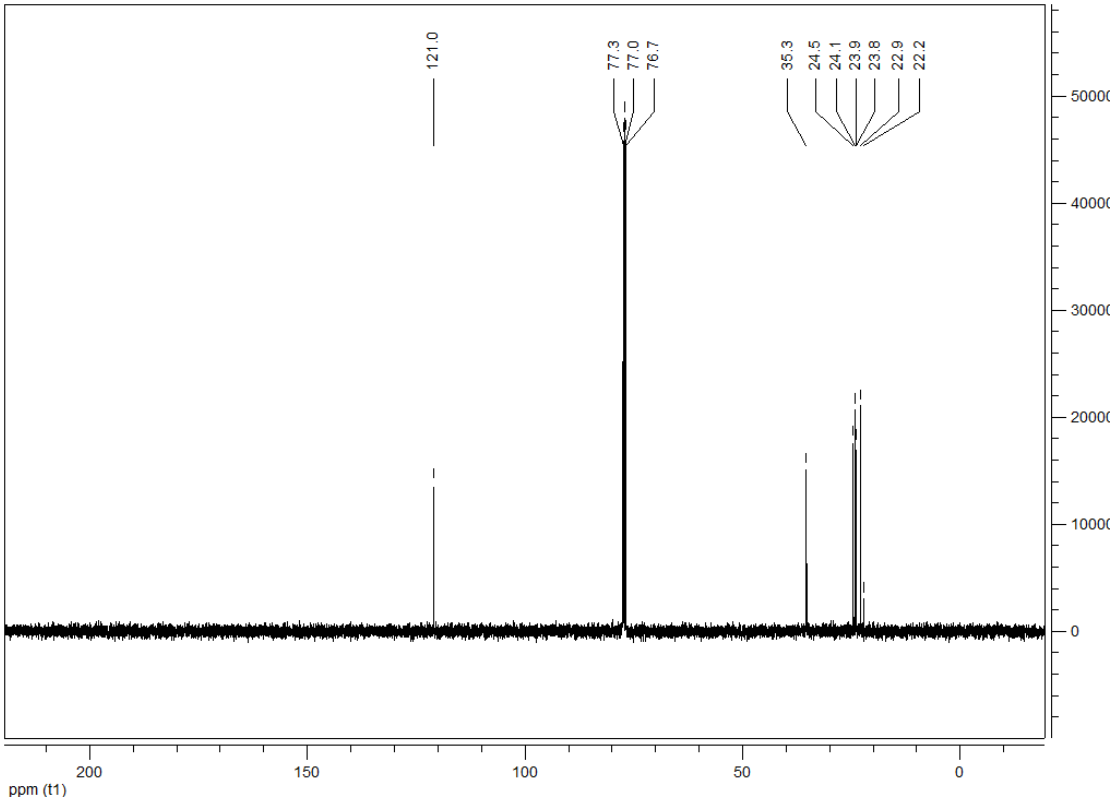
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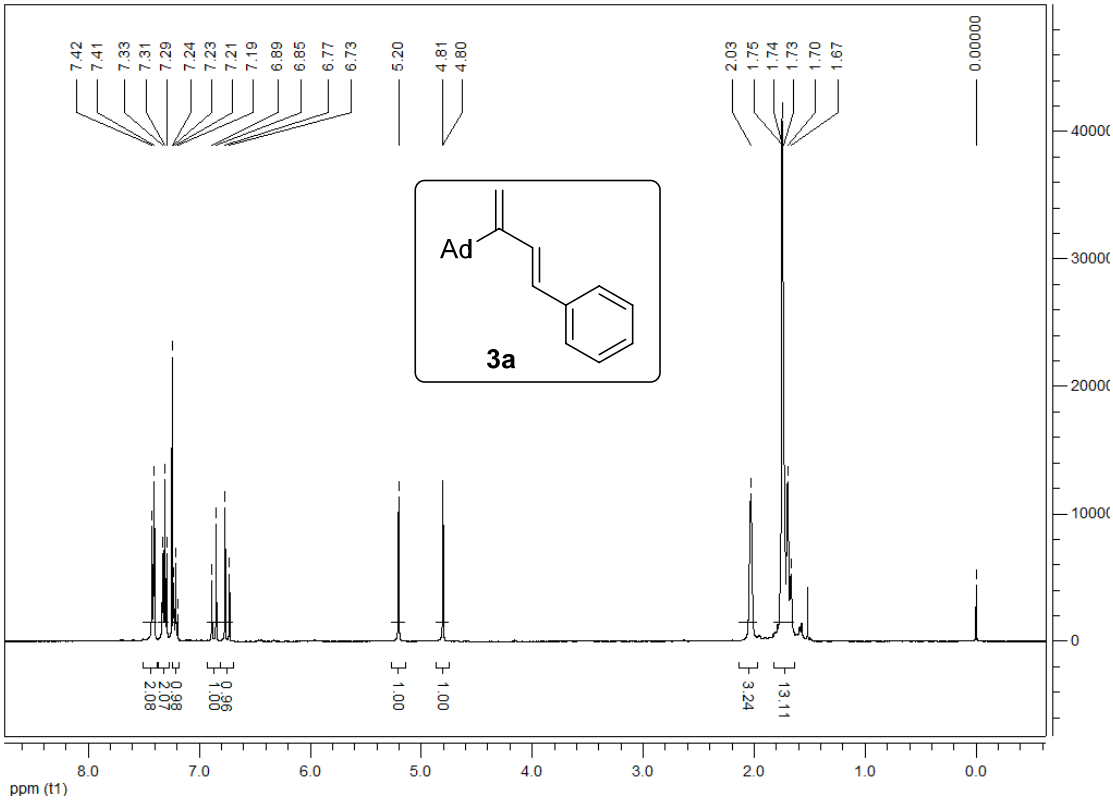
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7.23
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6.77
6.73
5.20
4.81
4.80
2.03
1.75
1.74
1.73
1.70
1.67
0.00000
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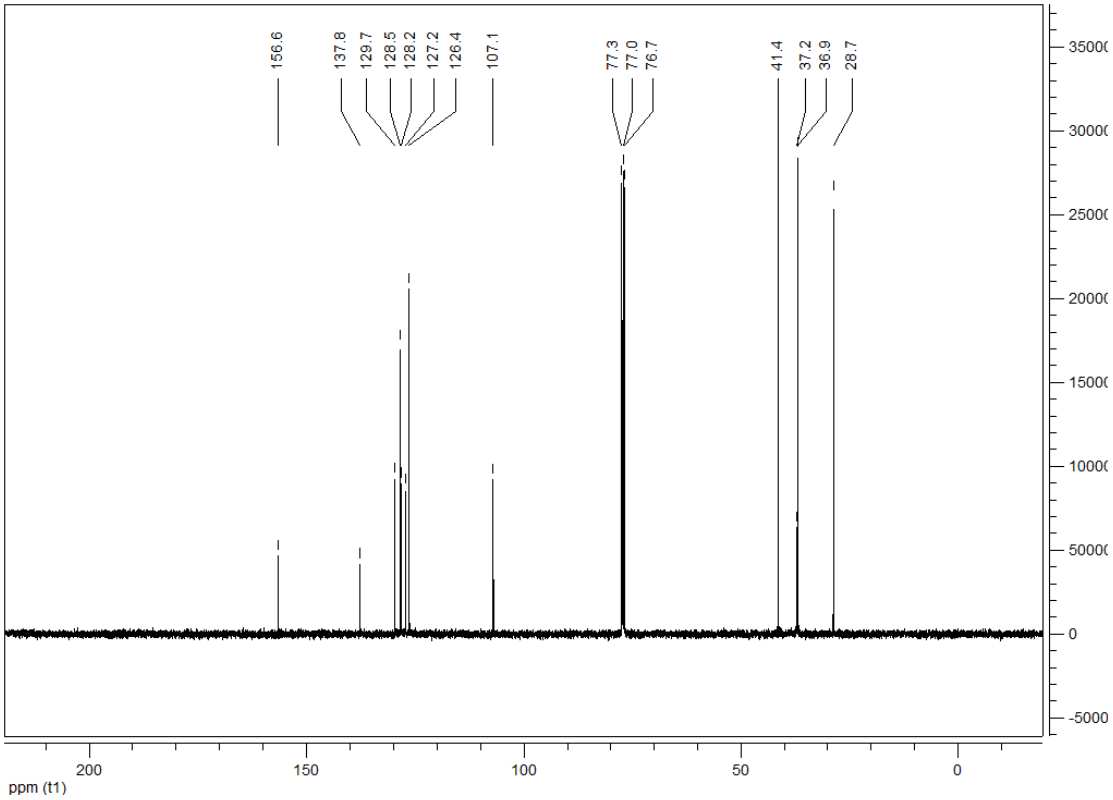
ppm (t1)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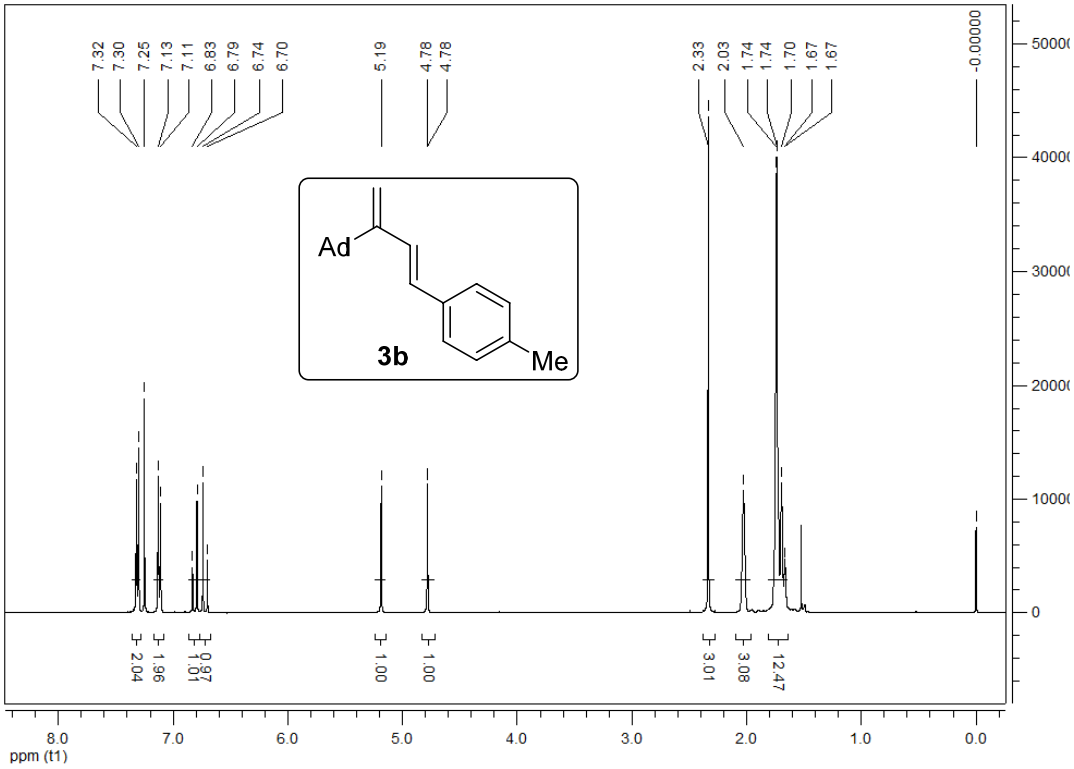
ppm (t1)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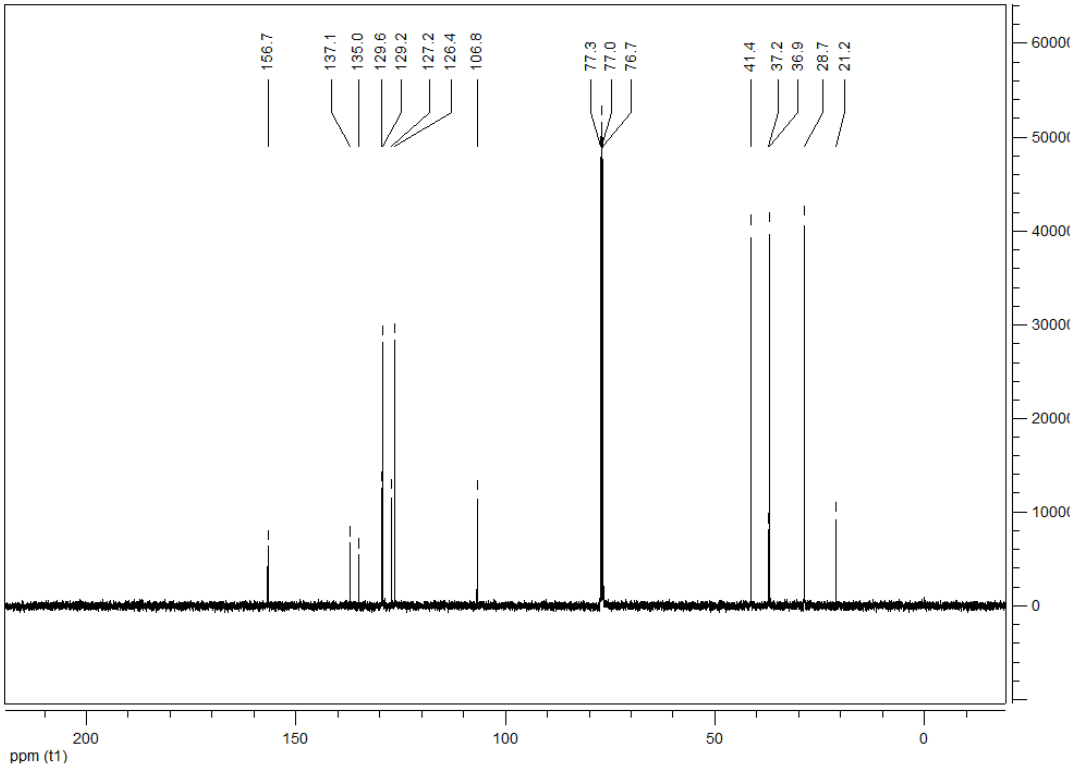
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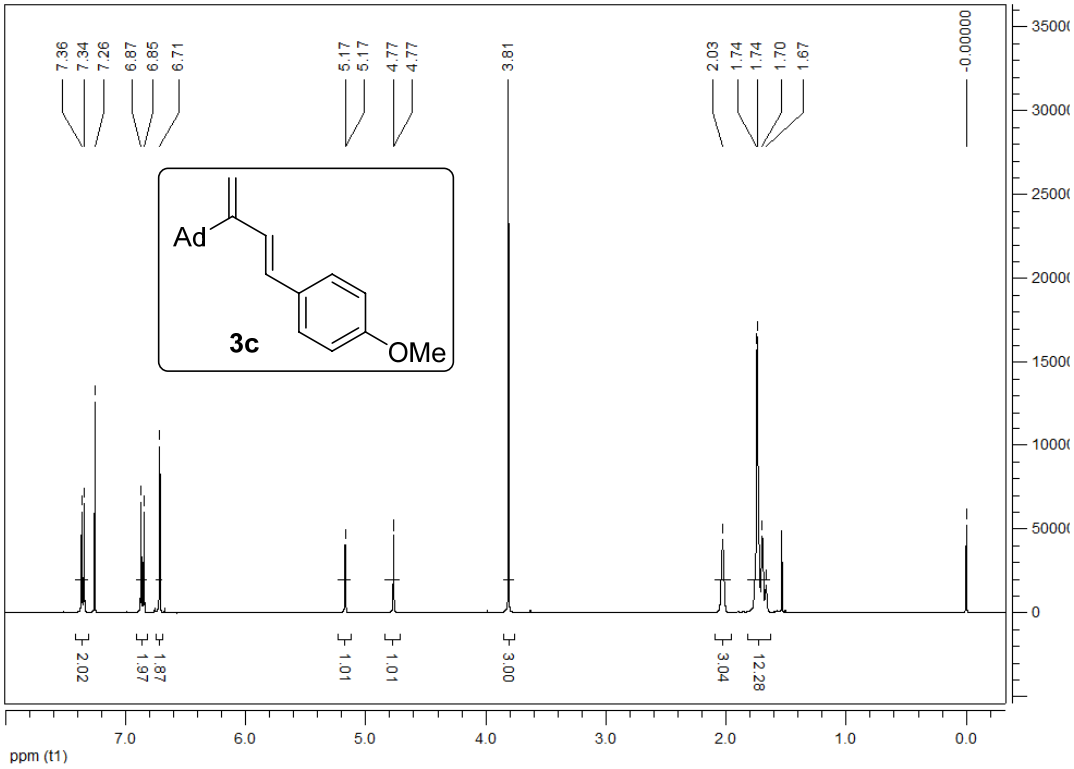
ppm (t1)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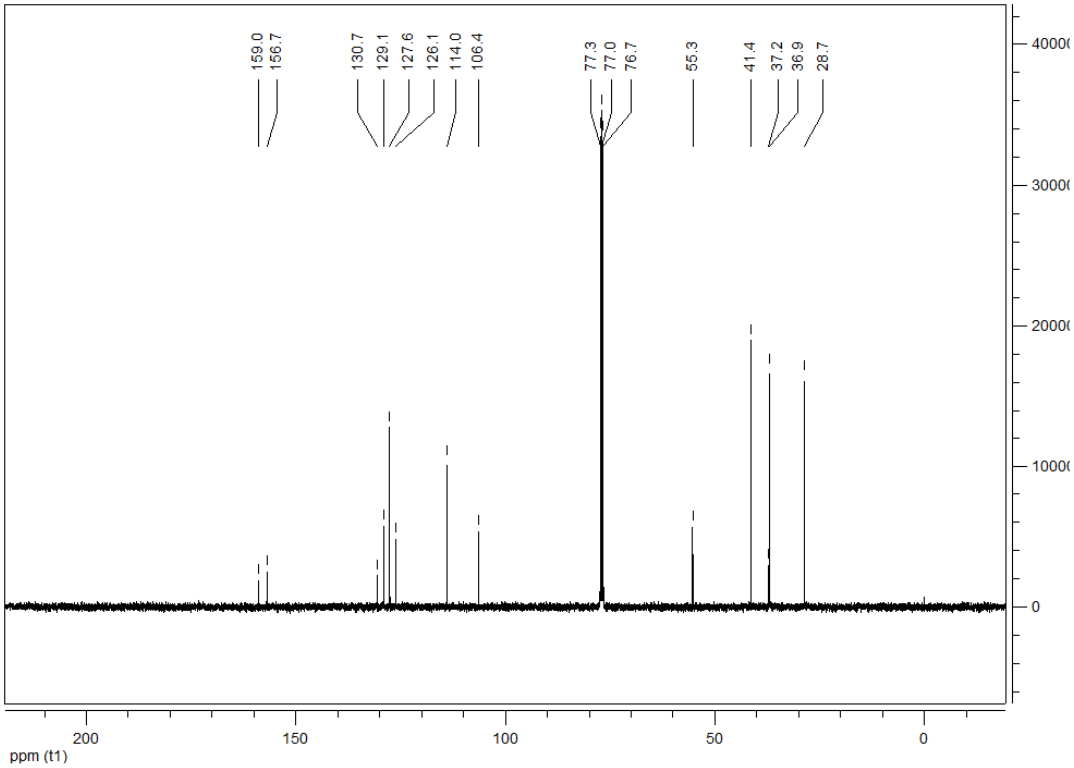
ppm (t1)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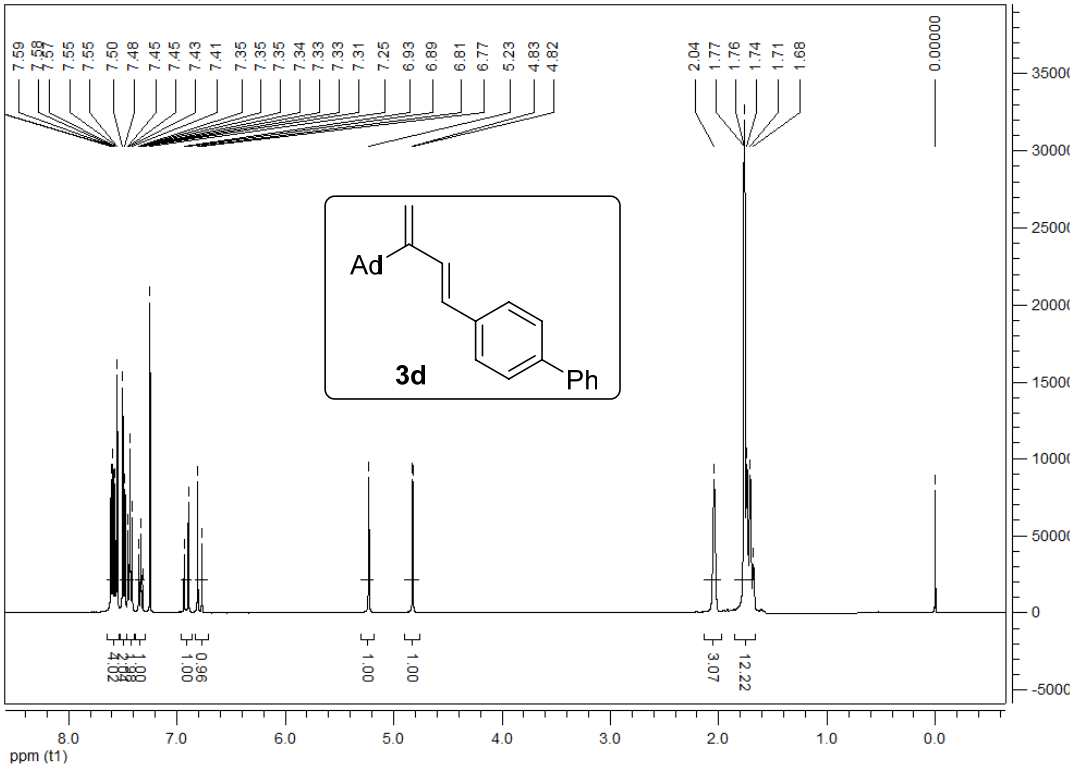
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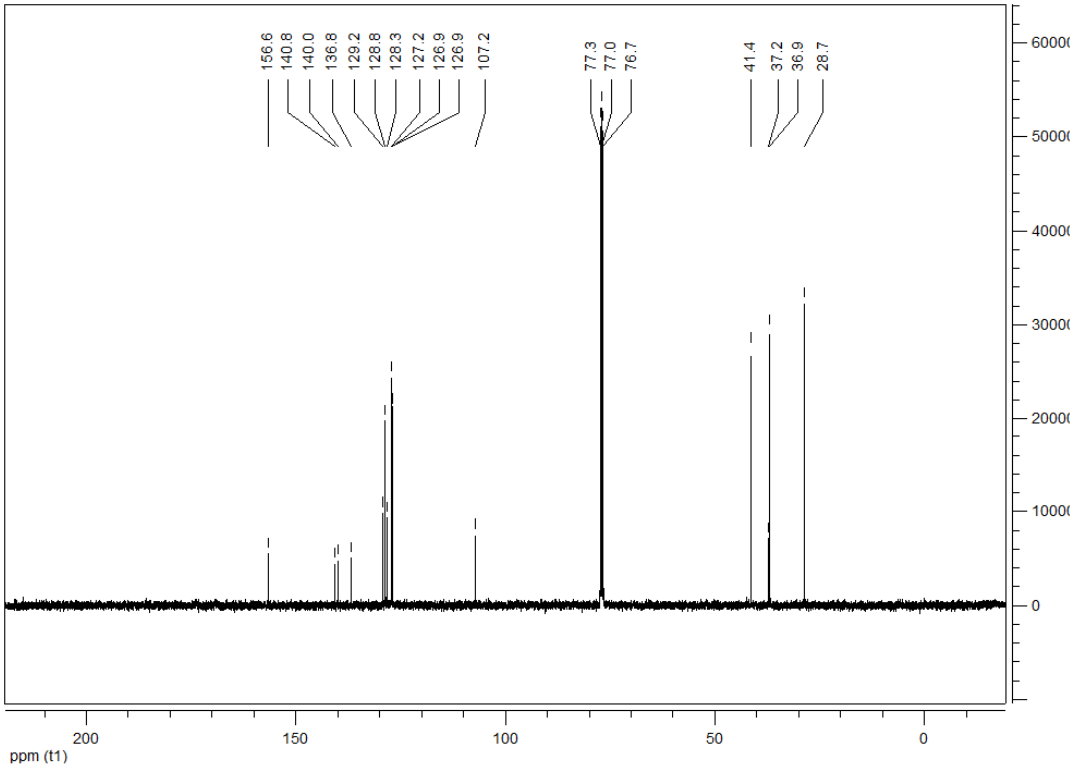
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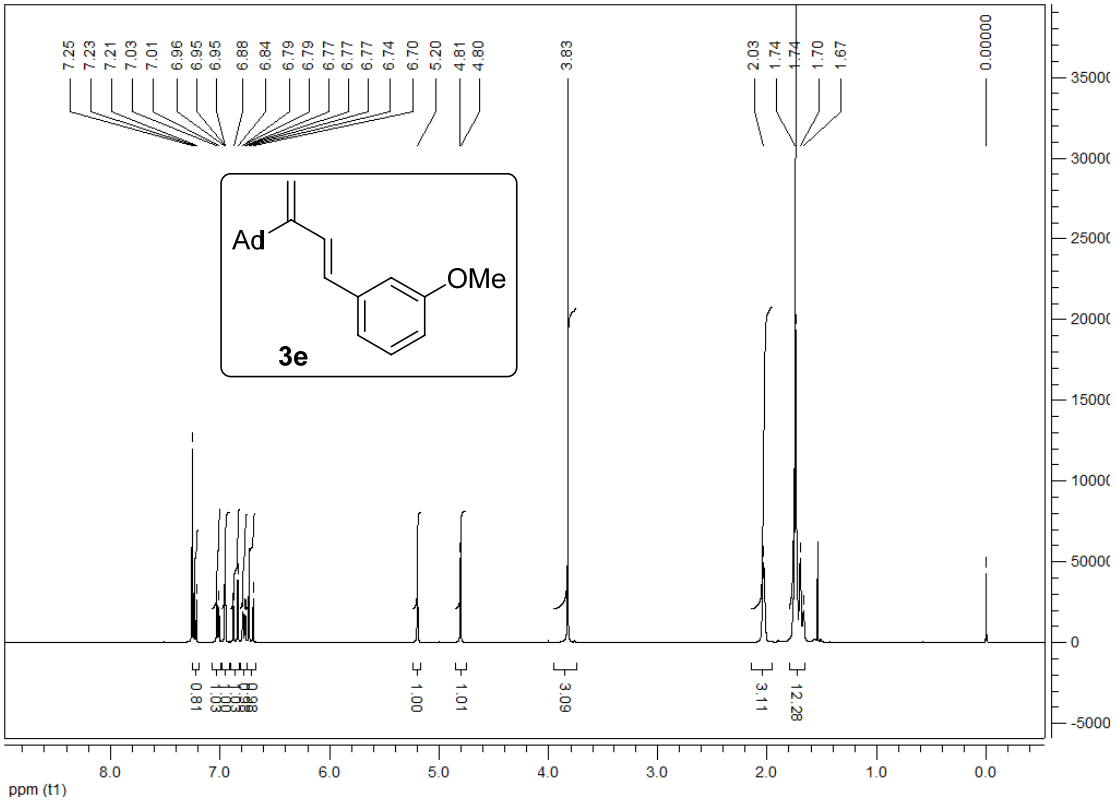
ppm (t1)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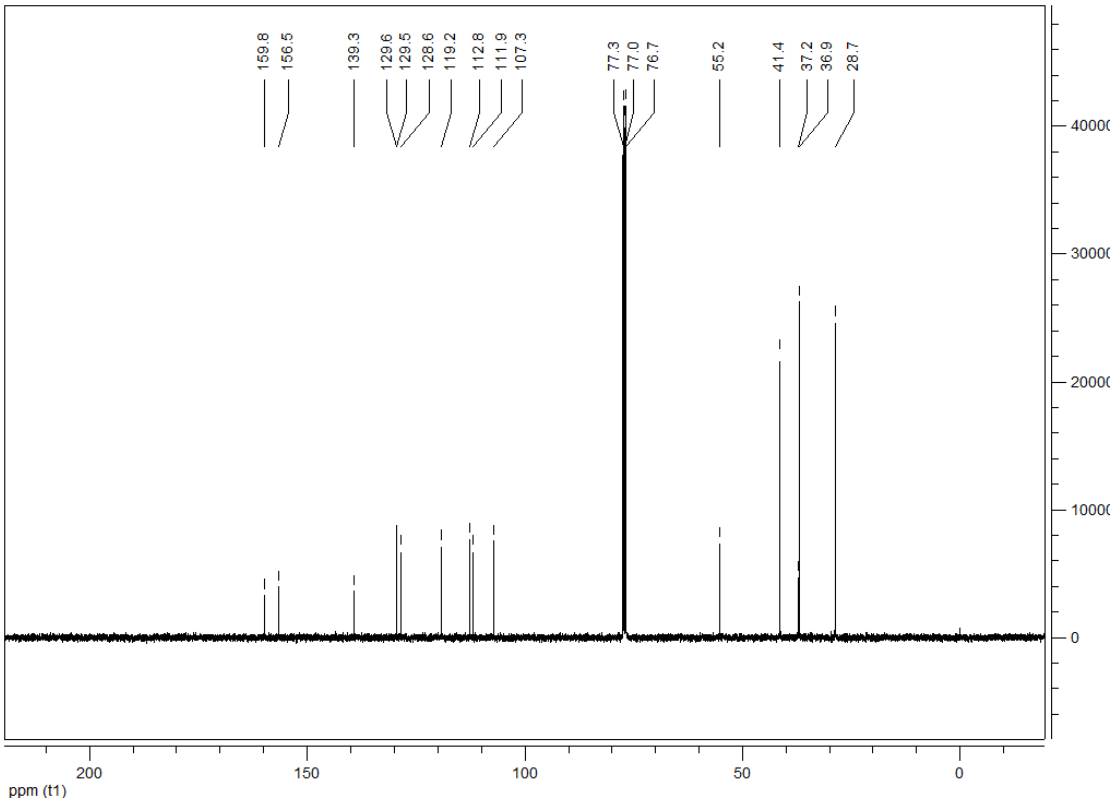
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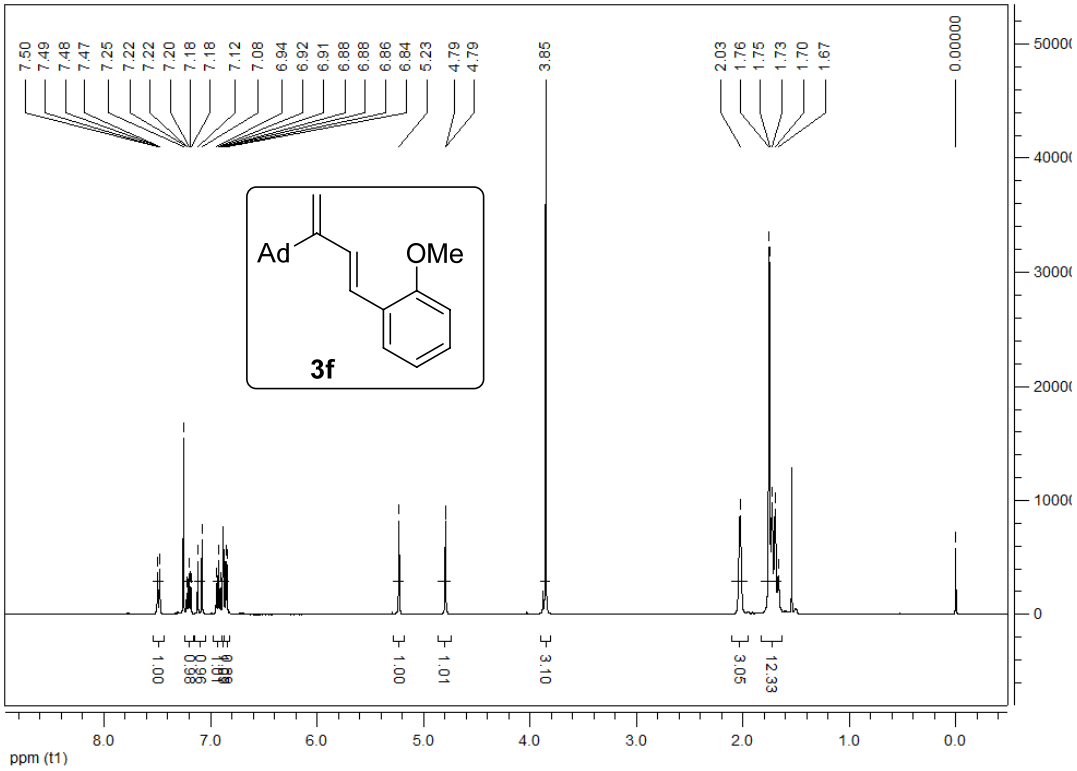
ppm (t1)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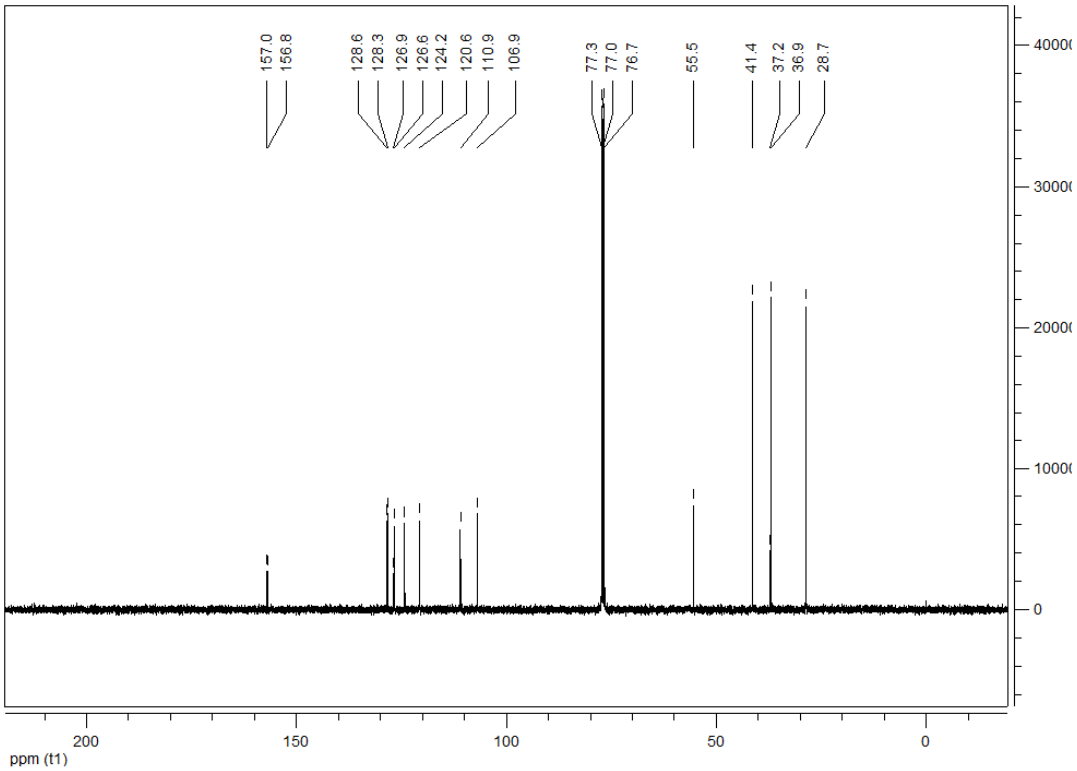
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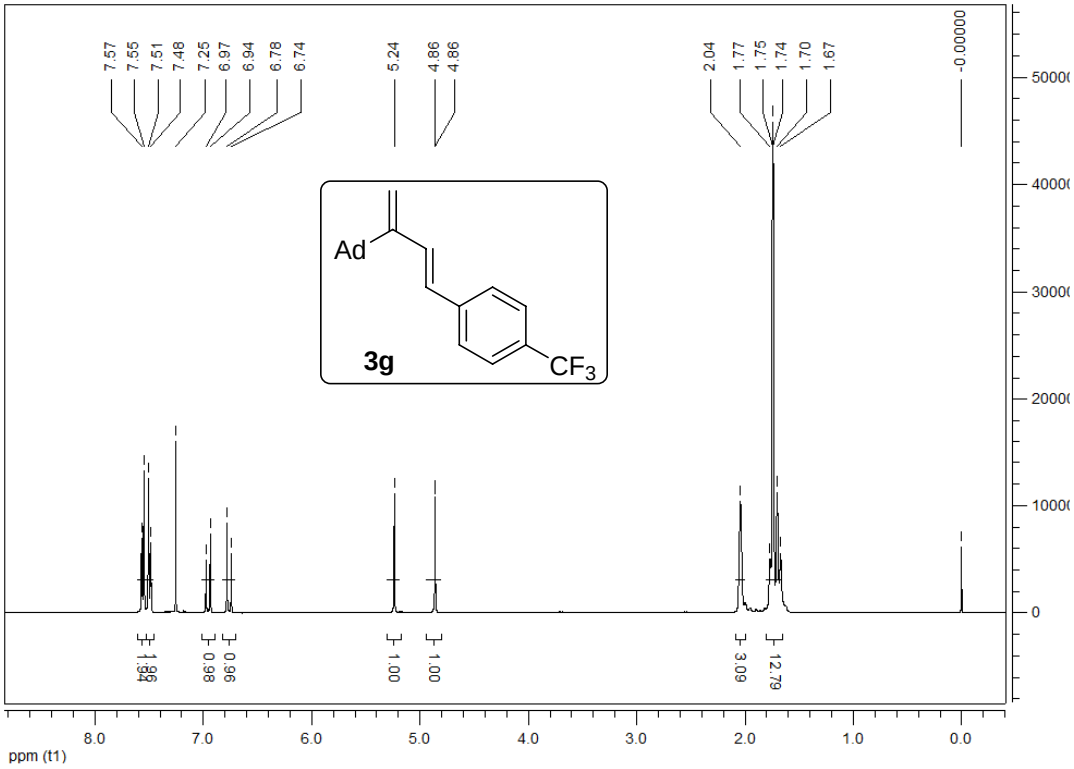
ppm (t1)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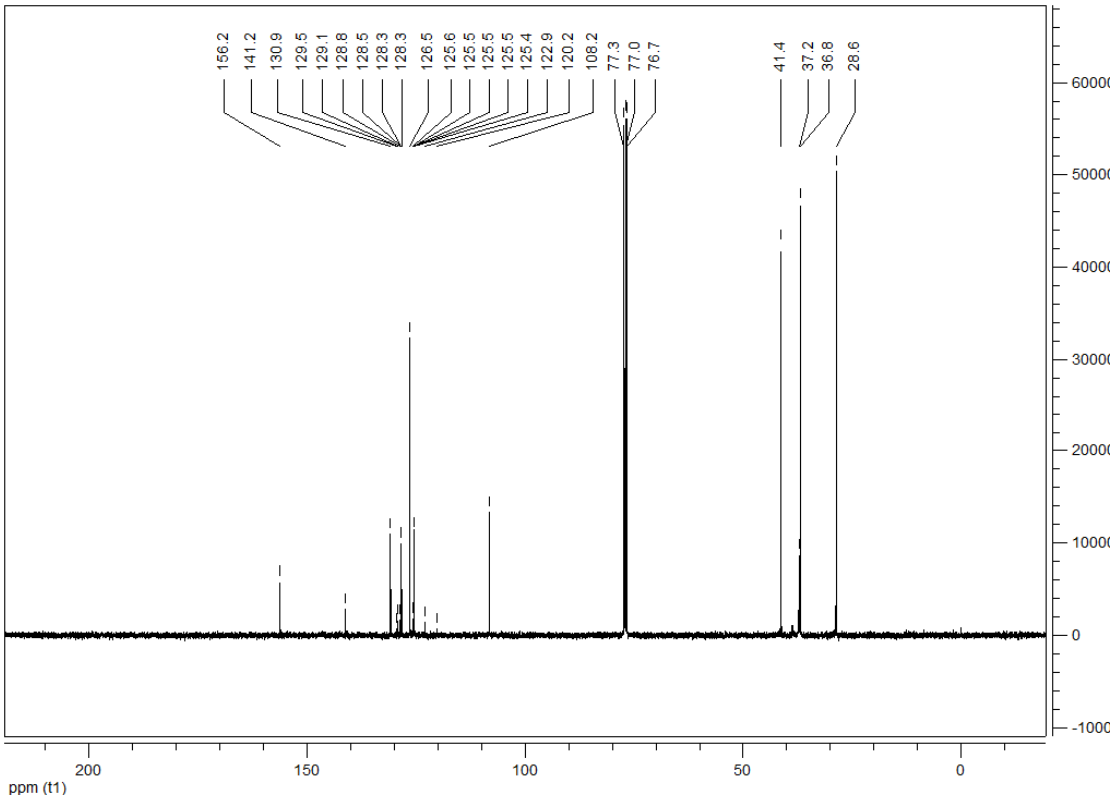
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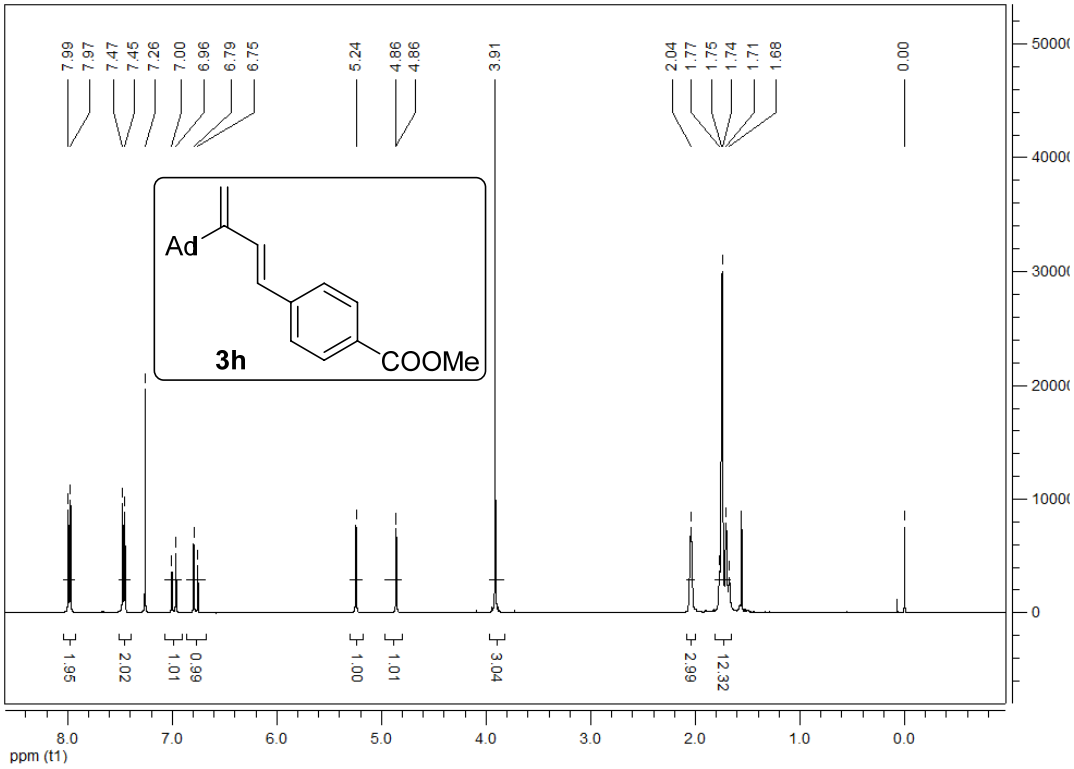
ppm (t1)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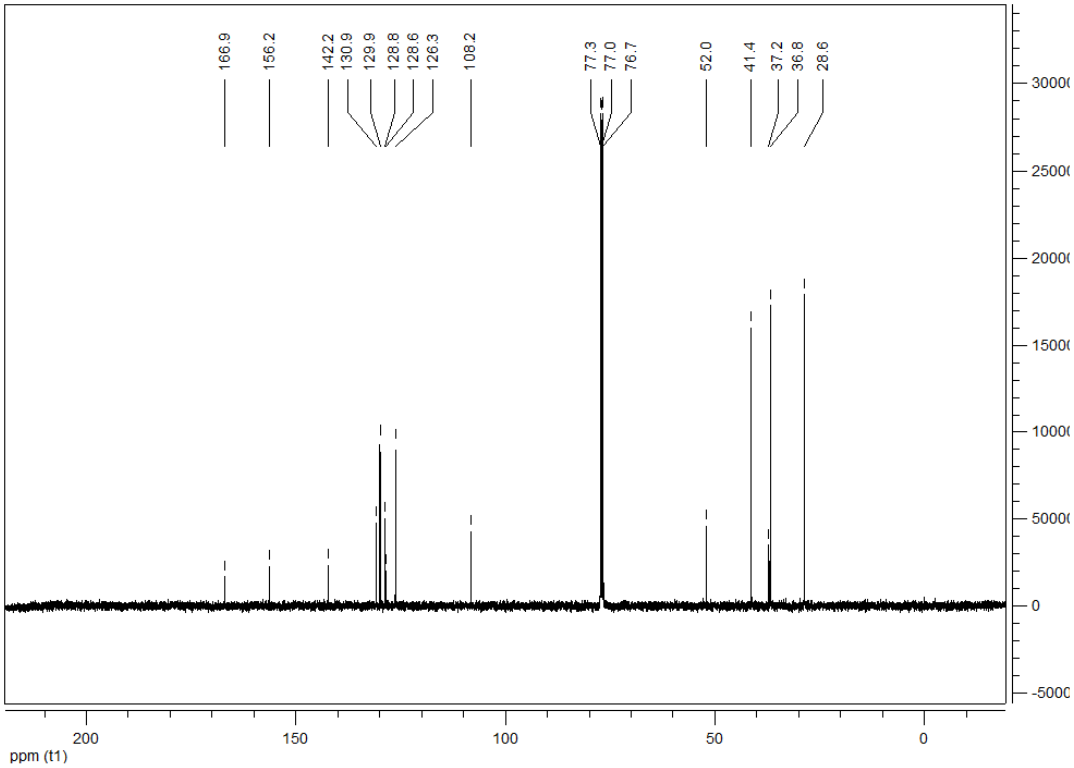
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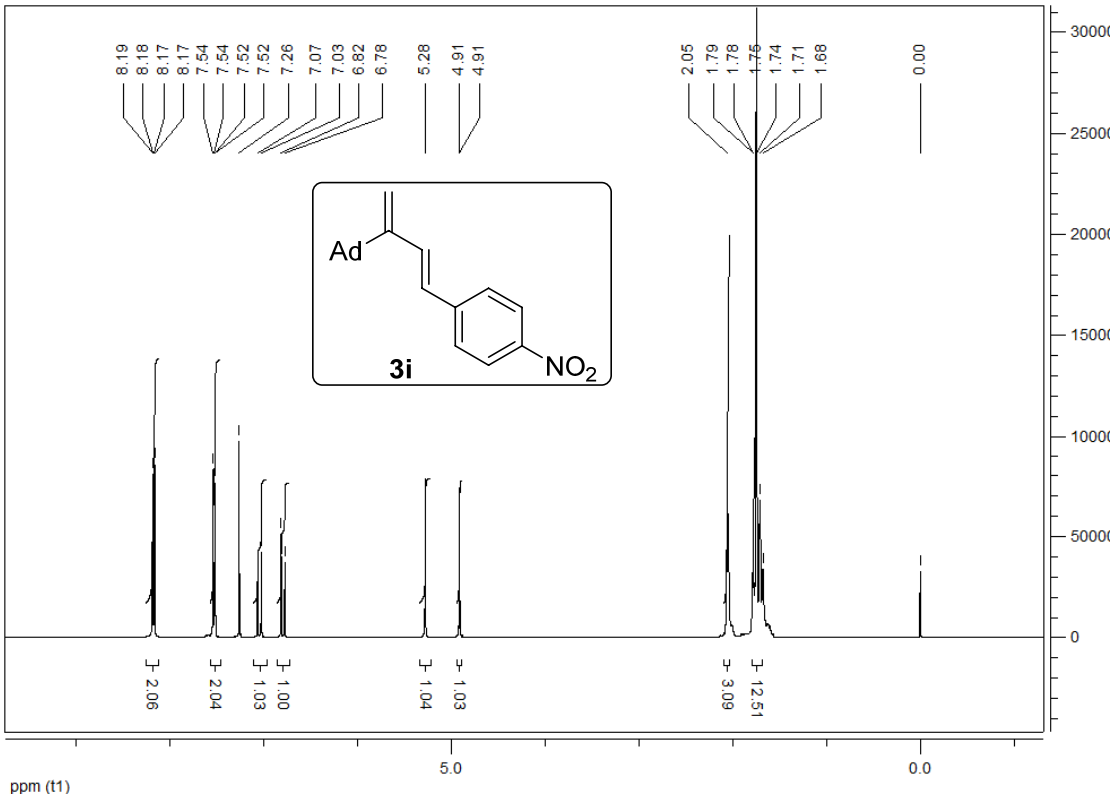
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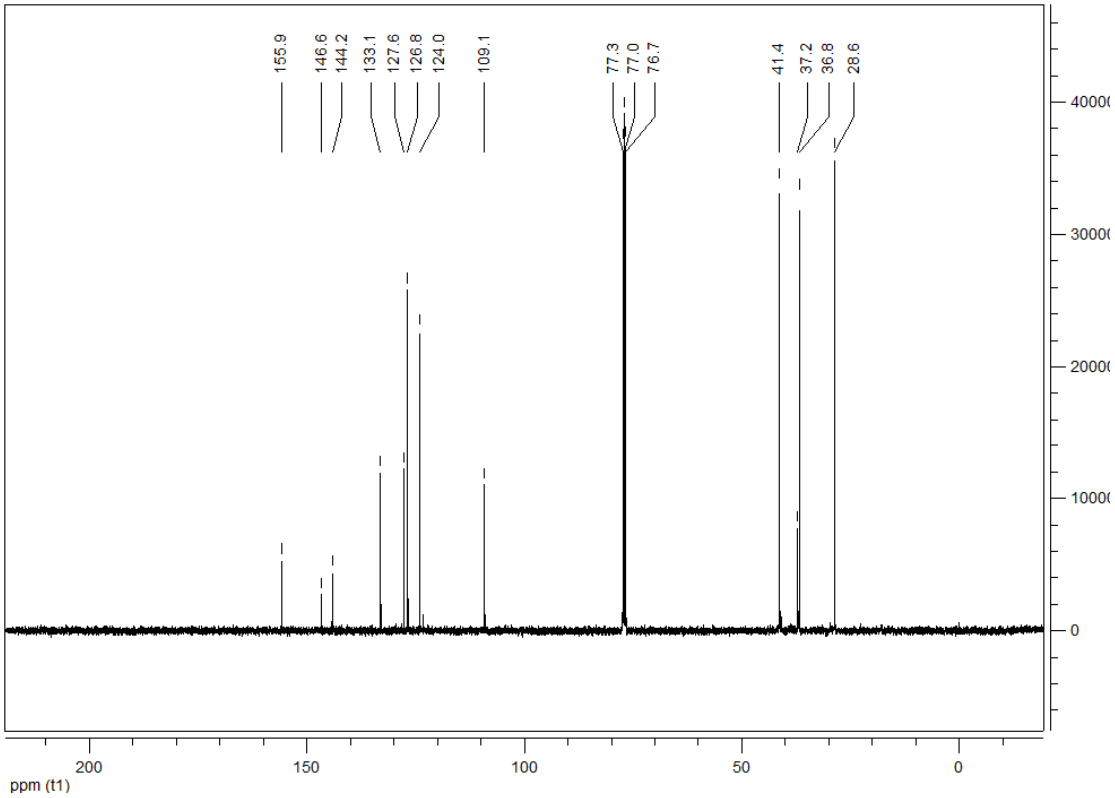
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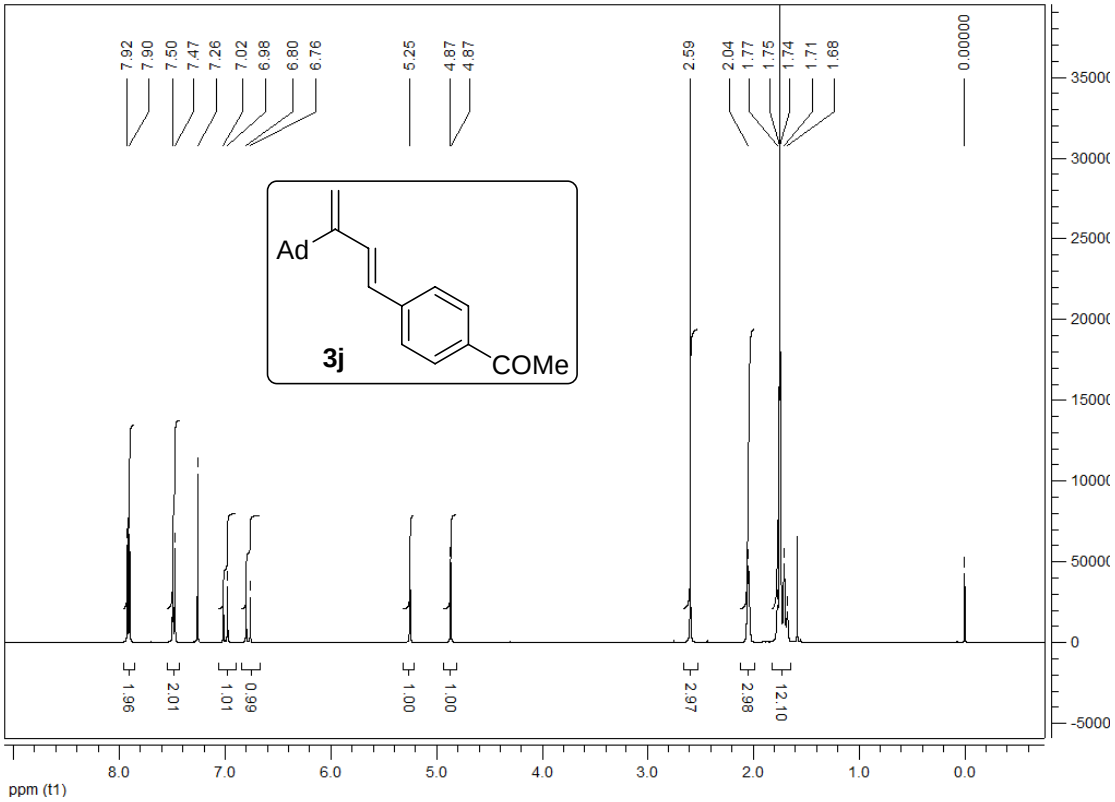
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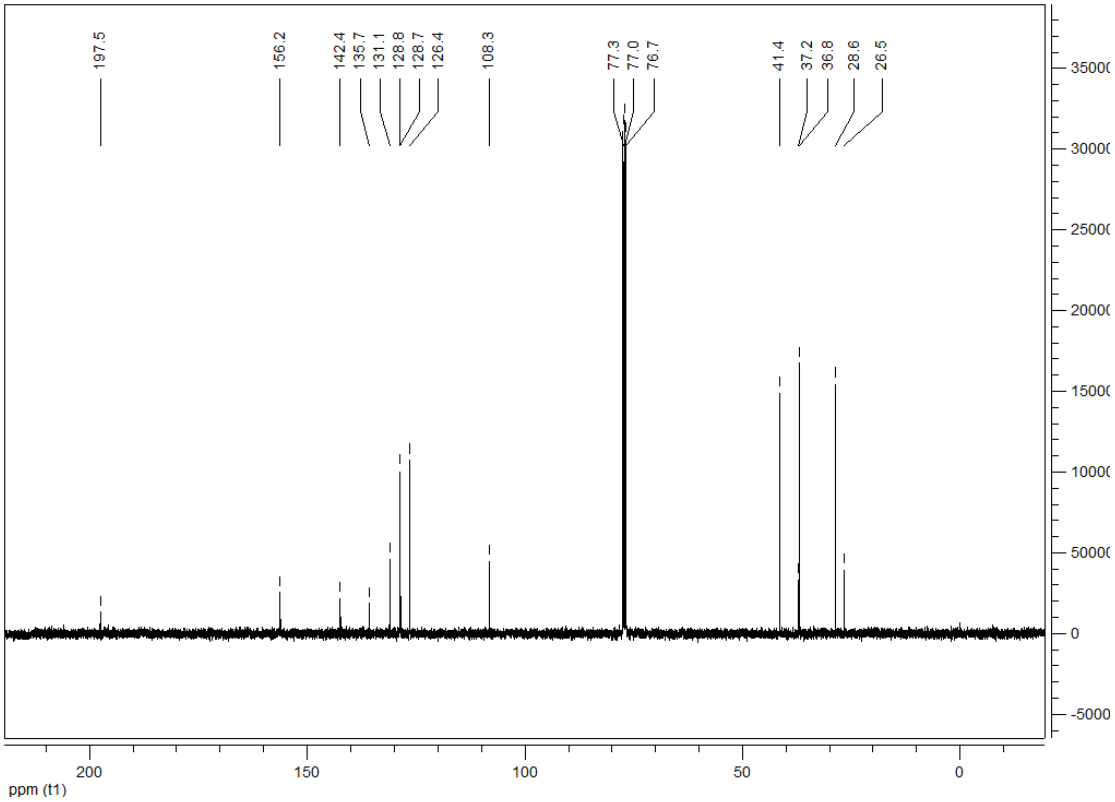
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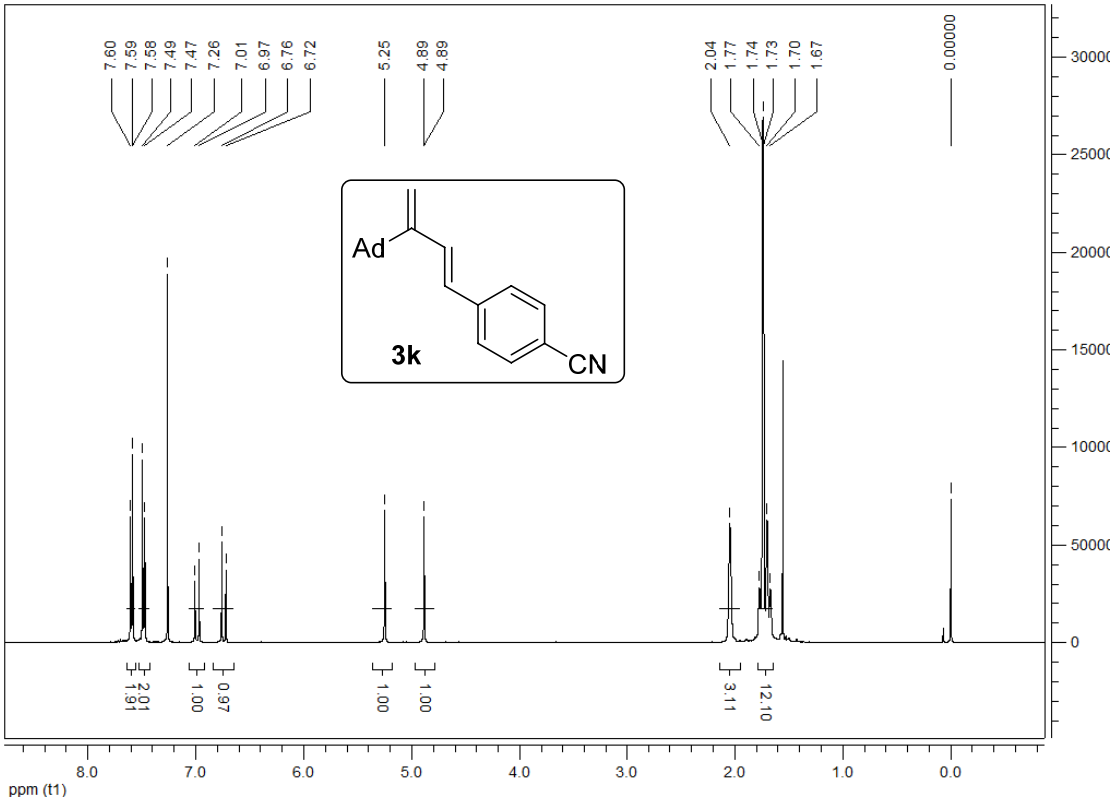
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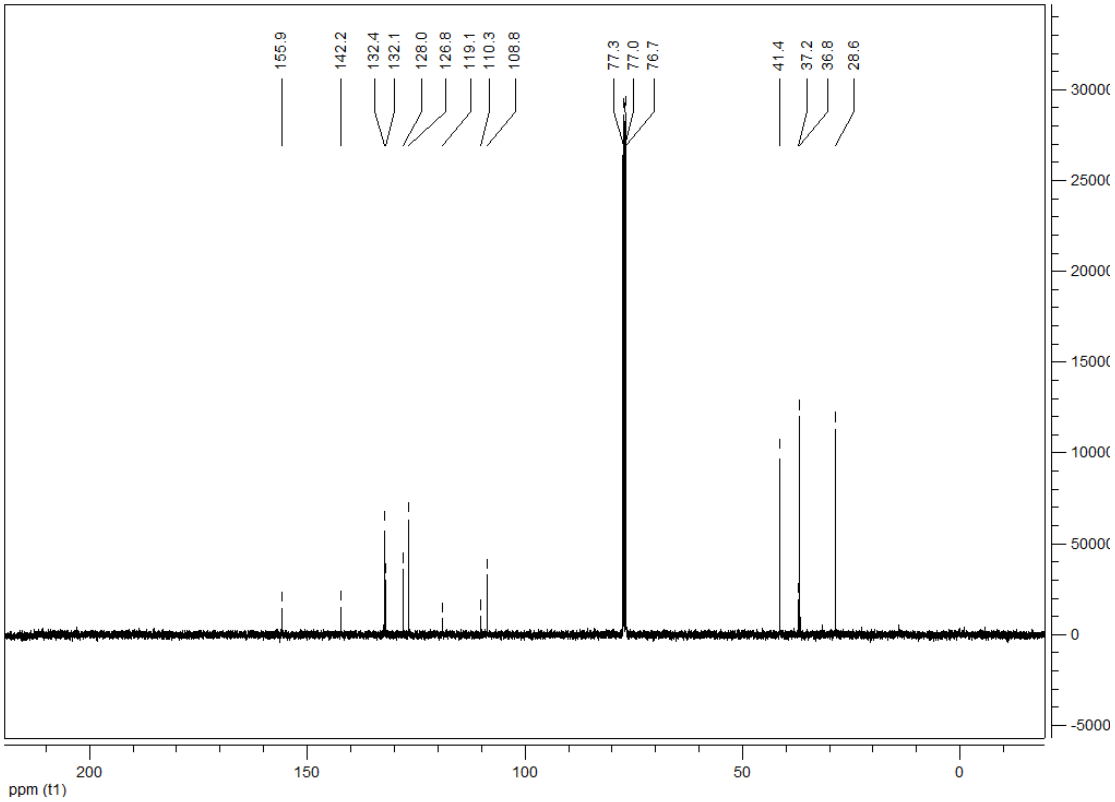
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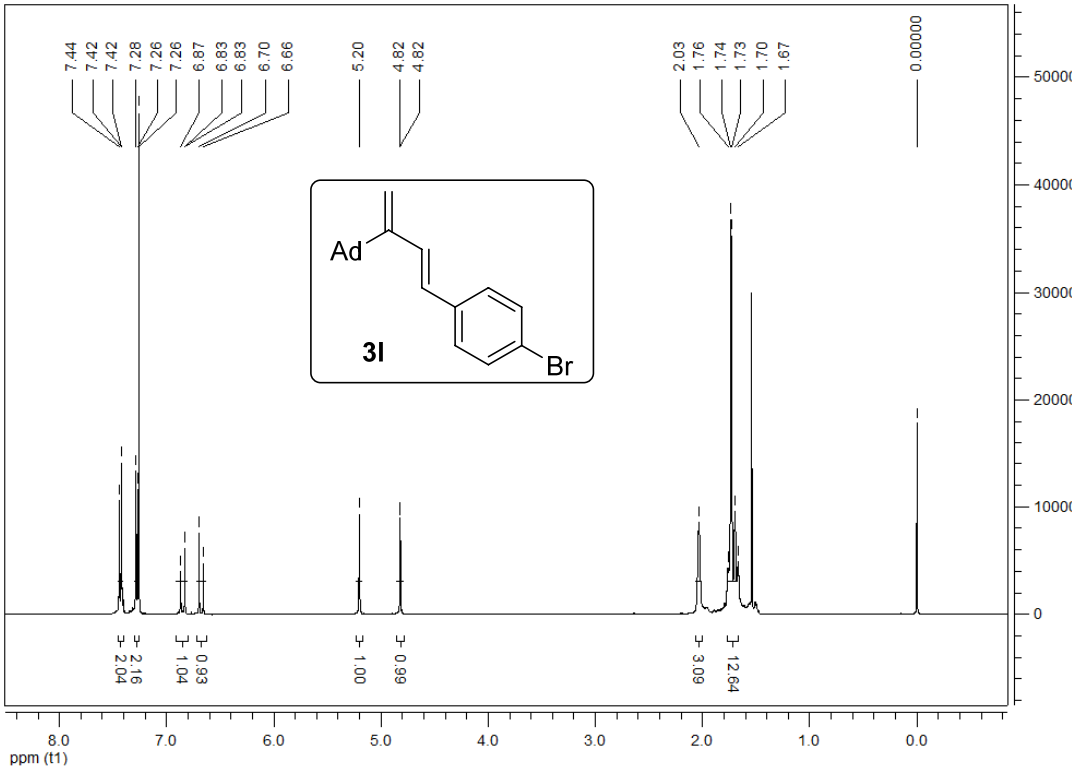
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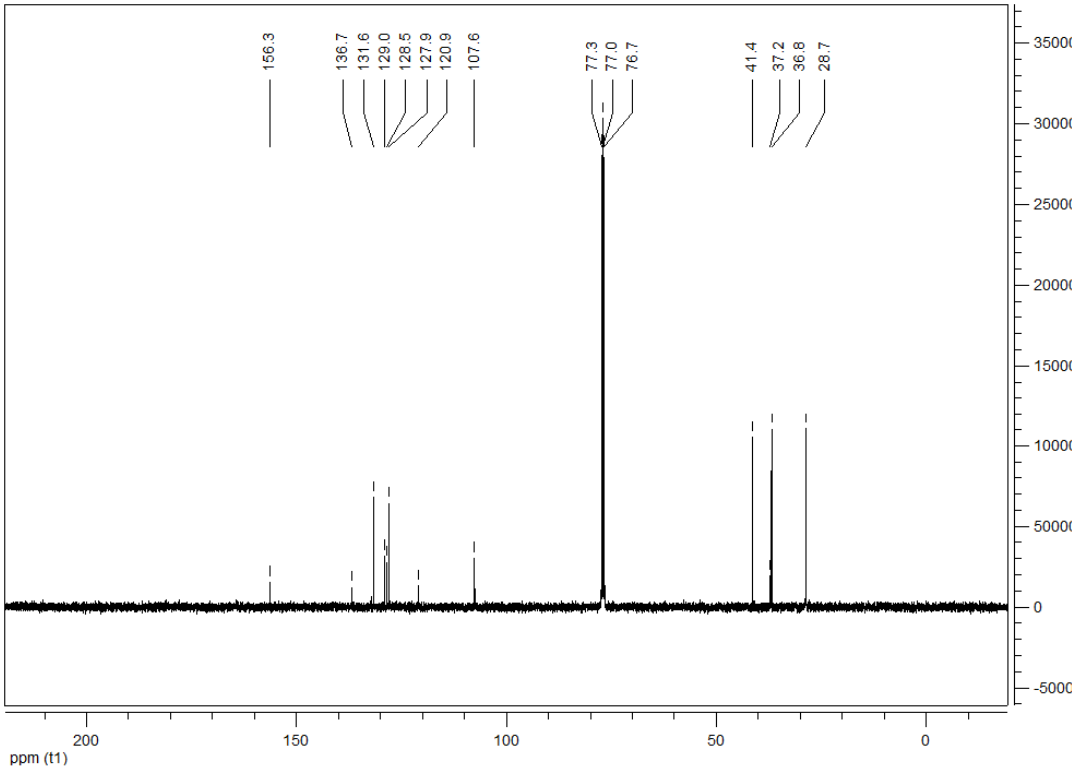
12.64
ppm (t1)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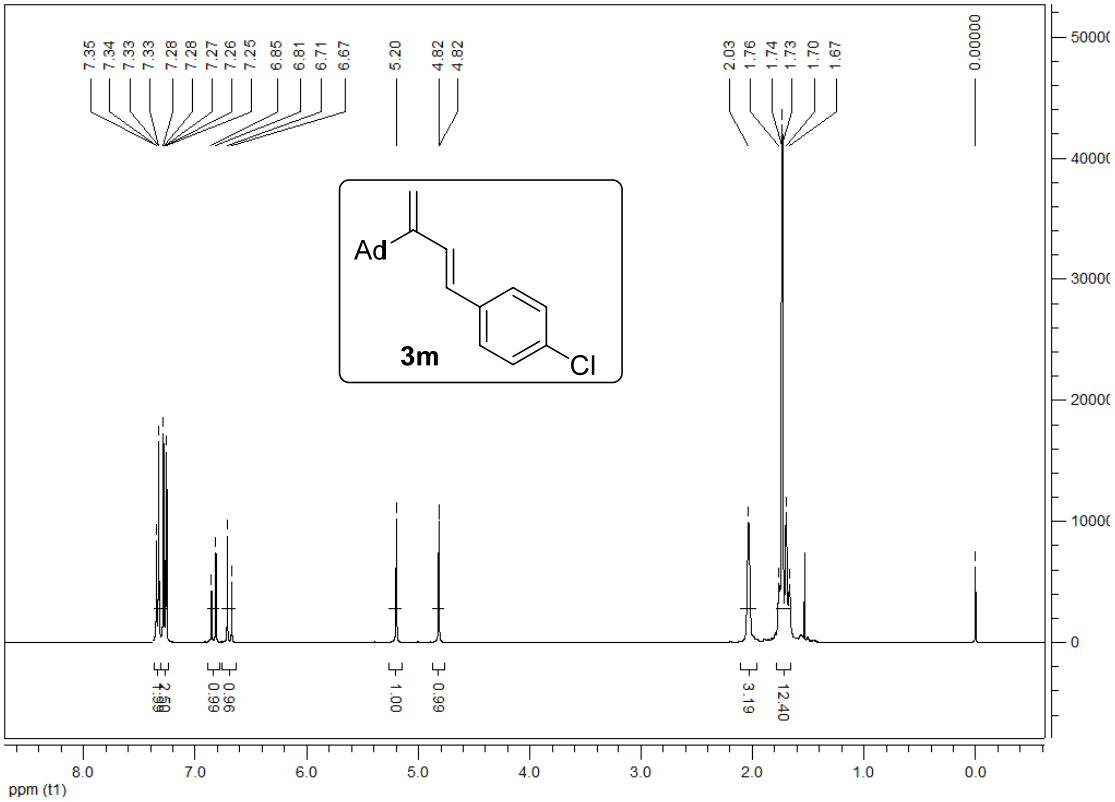
77.3
77.0
76.7
41.4
37.2
36.8
28.7
ppm (t1)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7.34
7.33
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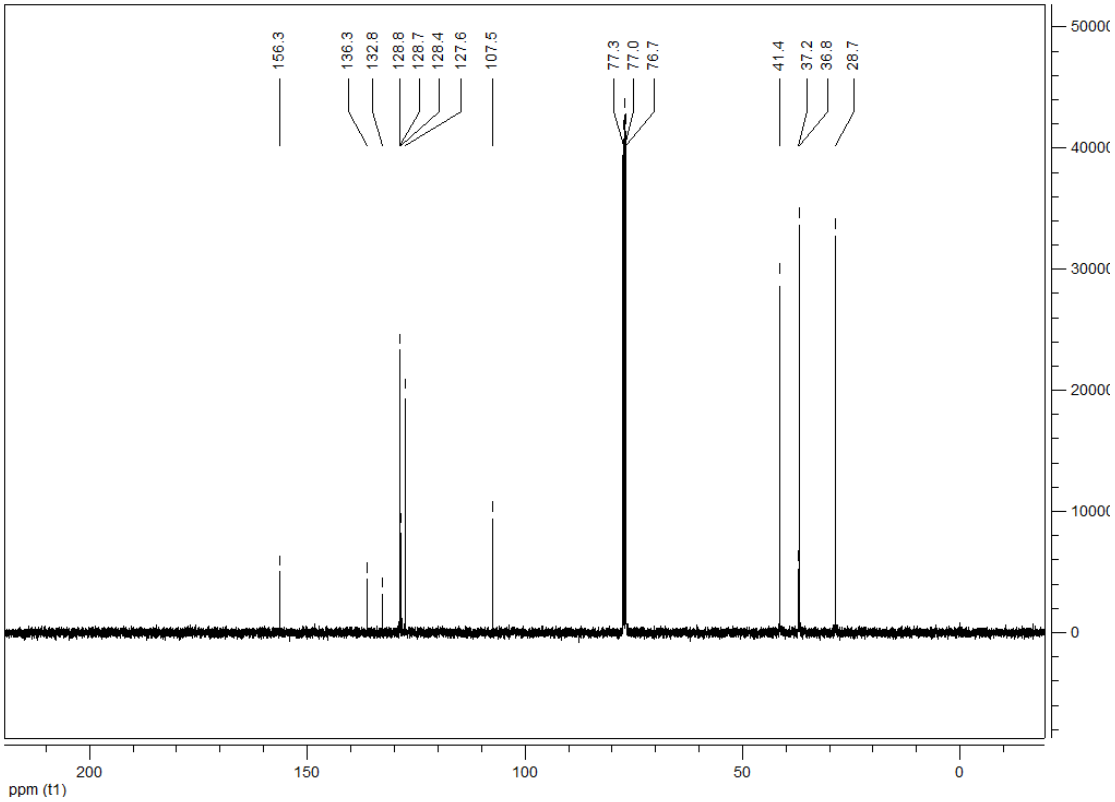
7.27
7.26
7.25
6.85
6.81
6.71
6.67
5.20
4.82
4.82
2.03
1.76
1.74
1.73
1.70
1.67
0.00000
Ad
3m
Cl
1.00
0.99
3.19
1.40
ppm (t1)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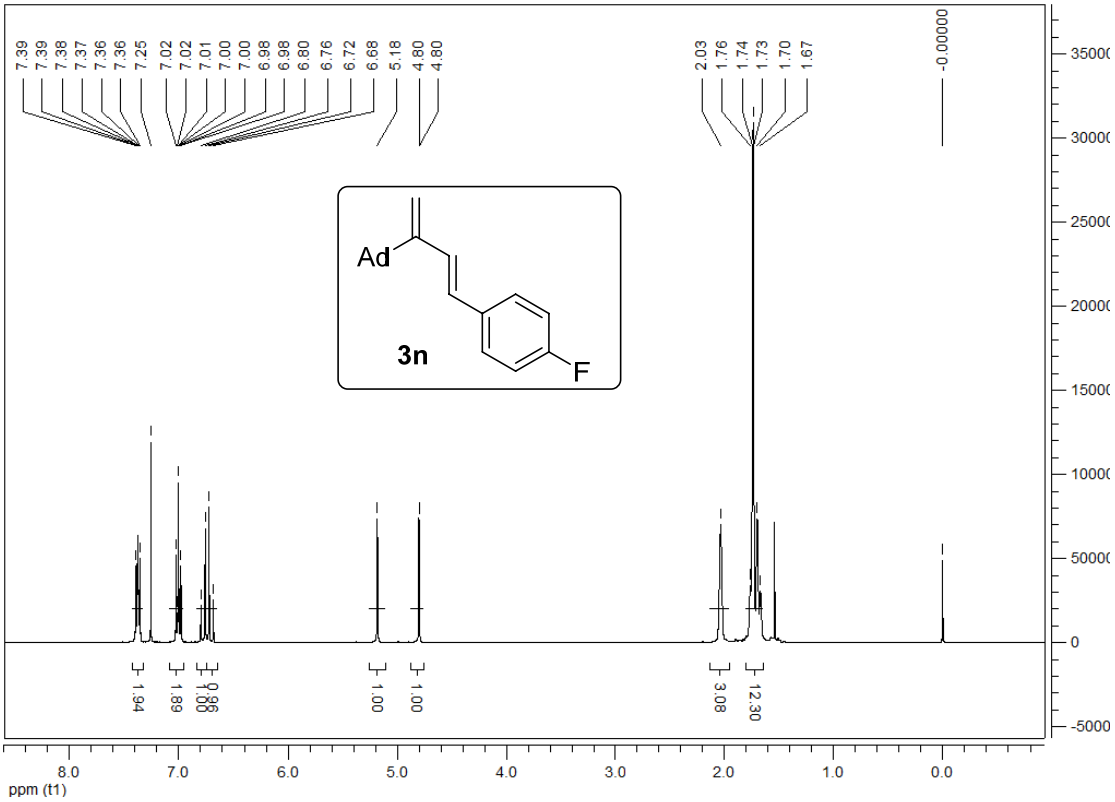
136.3
132.8
128.8
128.7
128.4
127.6
107.5
77.3
77.0
76.7
41.4
37.2
36.8
28.7
ppm (t1)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7.37
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7.36
7.25
7.02
7.02
7.01
7.00
7.00
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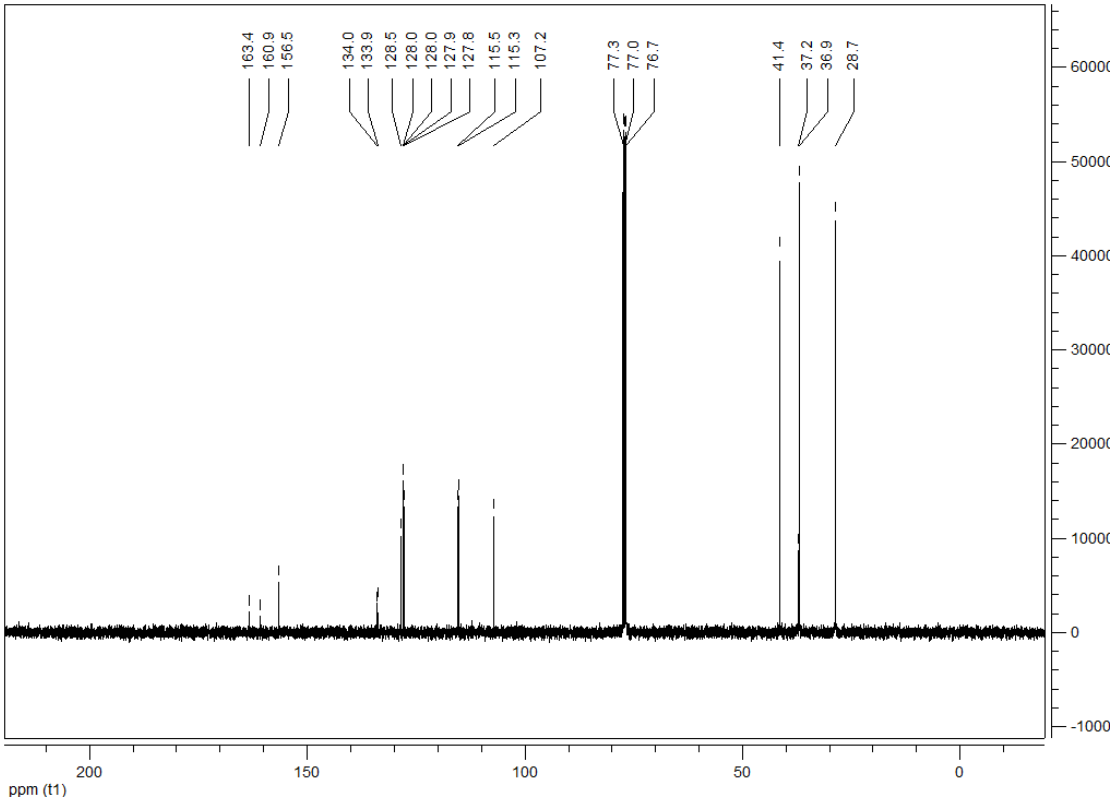
6.98
6.80
6.76
6.72
6.68
5.18
4.80
4.80
2.03
1.76
1.74
1.73
1.70
1.67
-0.00000
Ad
3n
F
1.94
1.89
0.96
1.00
1.00
3.08
2.30
ppm (t1)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156.5
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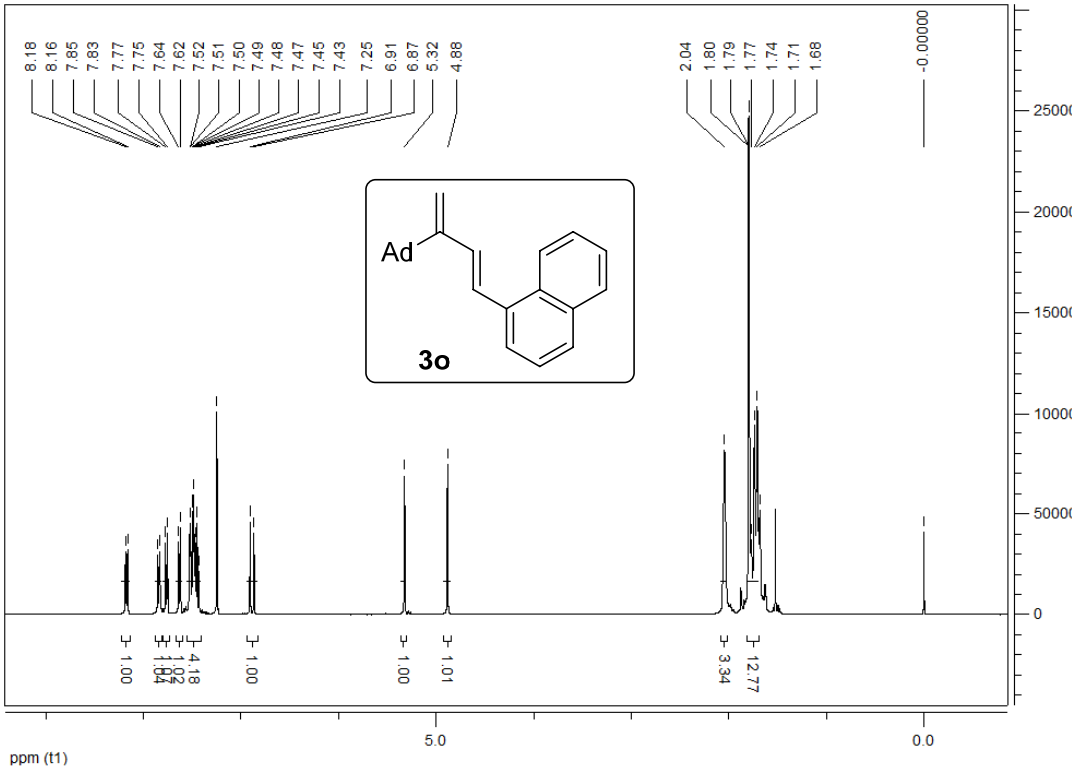
133.9
128.5
128.0
128.0
127.9
127.8
115.5
115.3
107.2
77.3
77.0
76.7
41.4
37.2
36.9
28.7
ppm (t1)


8.18
8.16
7.85
7.83
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7.75
7.64
7.62
7.52
7.51
7.50
7.49
7.48
7.47
7.45
7.43
7.25
6.91
6.87
5.32
4.88
2.04
1.80
1.79
1.77
1.74
1.71
1.68
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3o
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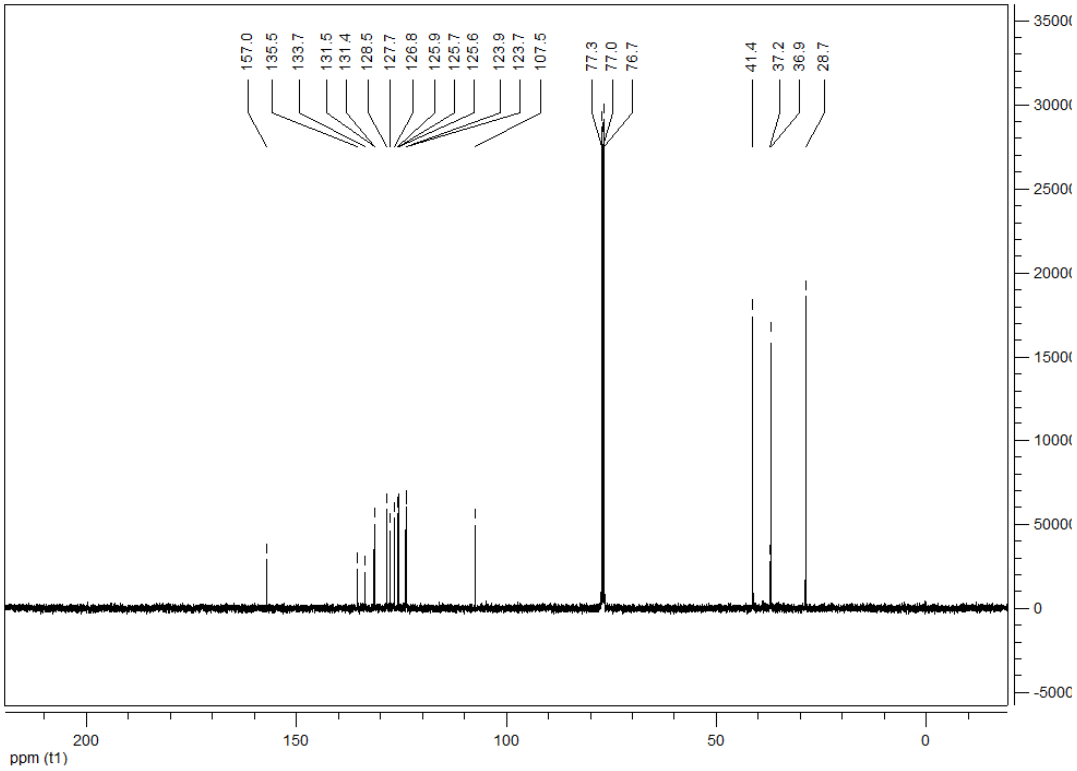
1.00
1.00
1.01
3.34
12.77
5.0
0.0
ppm (t1)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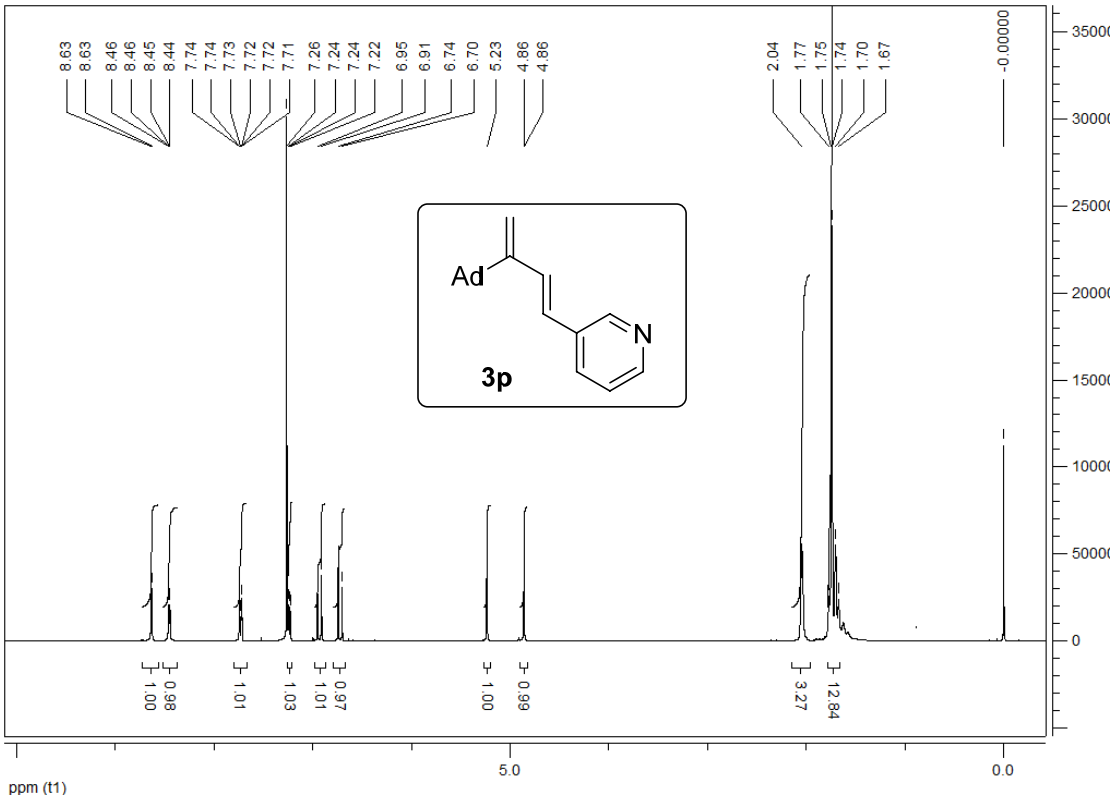
135.5
133.7
131.5
131.4
128.5
127.7
126.8
125.9
125.7
125.6
123.9
123.7
107.5
77.3
77.0
76.7
41.4
37.2
36.9
28.7
200
150
100
50
0
ppm (t1)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8.63
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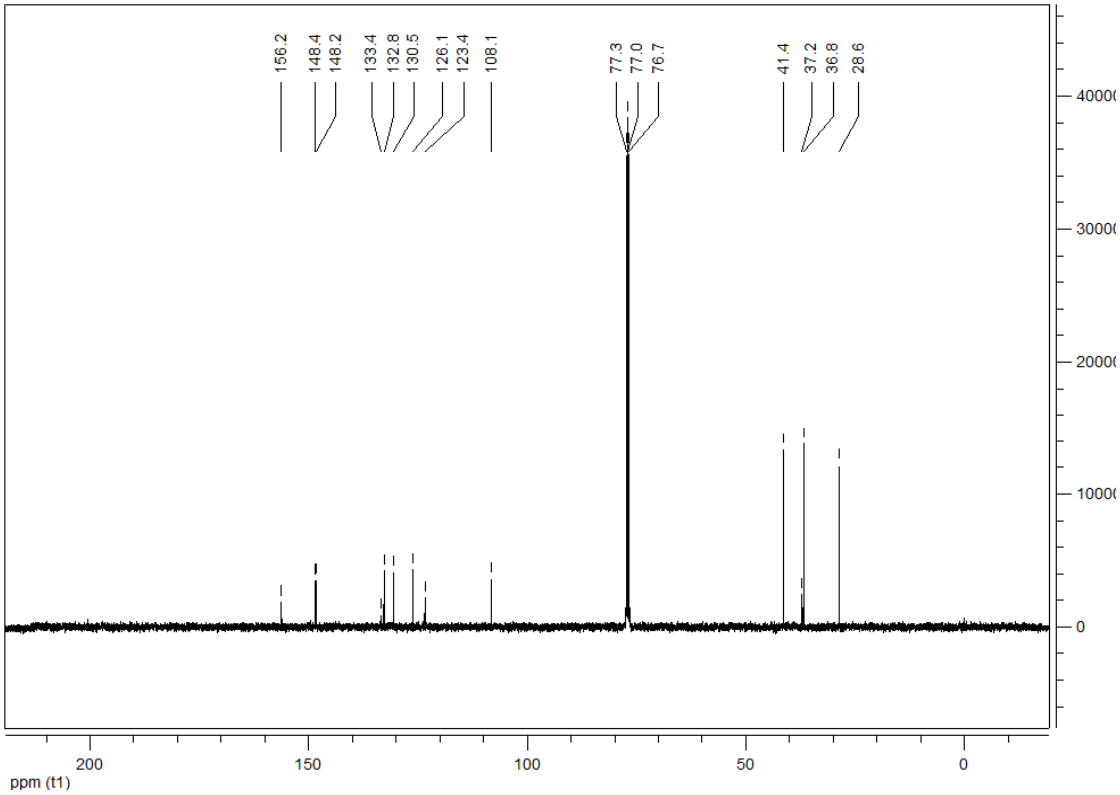
8.45
8.44
7.74
7.74
7.73
7.72
7.72
7.71
7.26
7.24
7.24
7.22
6.95
6.91
6.74
6.70
5.23
4.86
4.86
2.04
1.77
1.75
1.74
1.70
1.67
-0.00000
Ad
N
3p
1.00
0.98
1.01
1.03
1.01
0.97
1.00
0.99
3.27
12.84
5.0
0.0
ppm (t1)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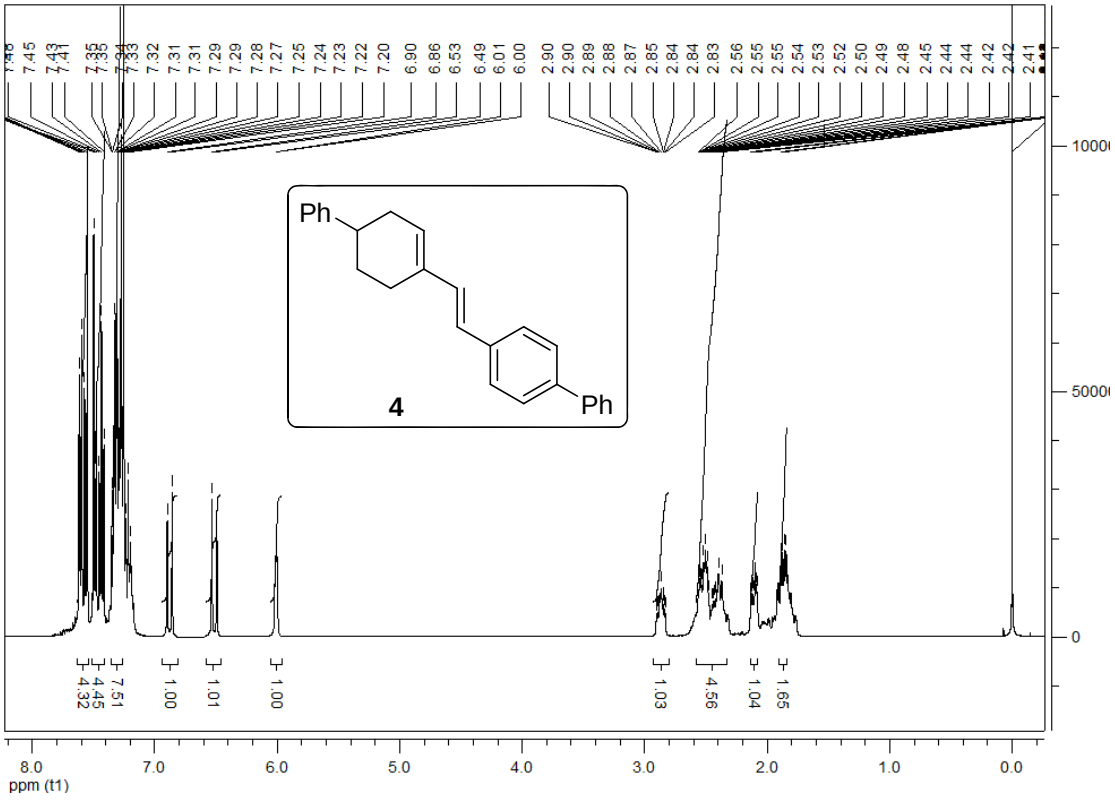
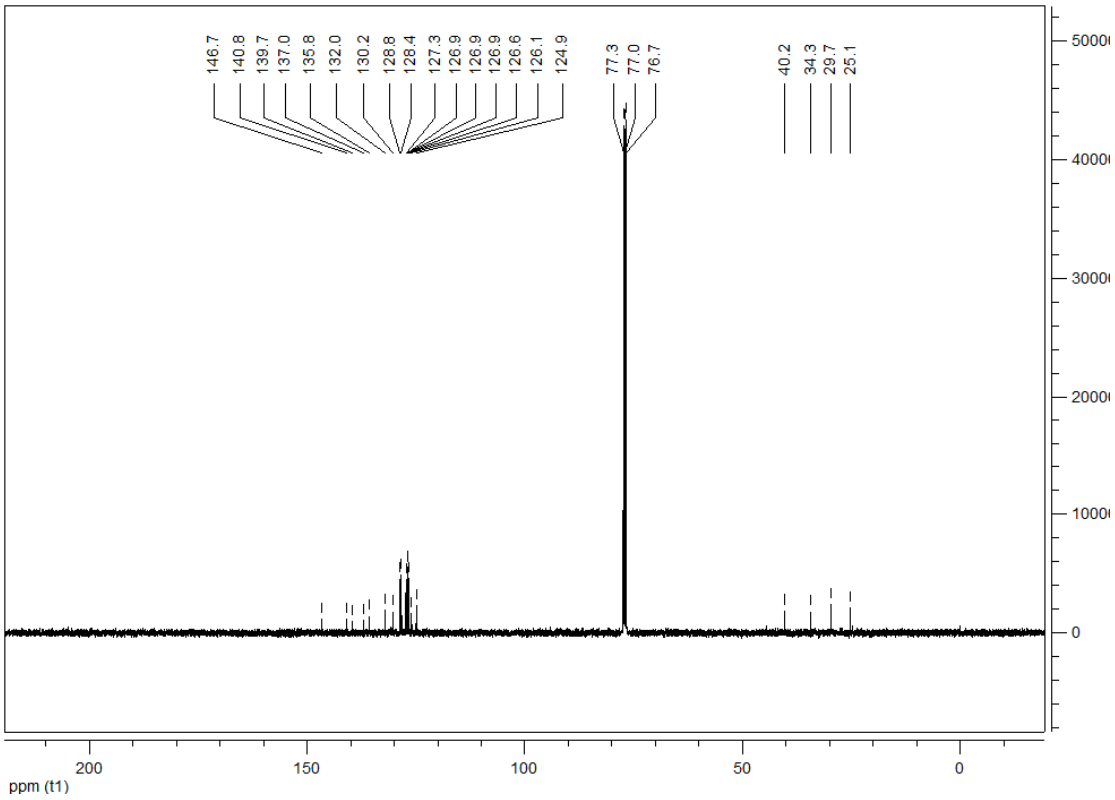
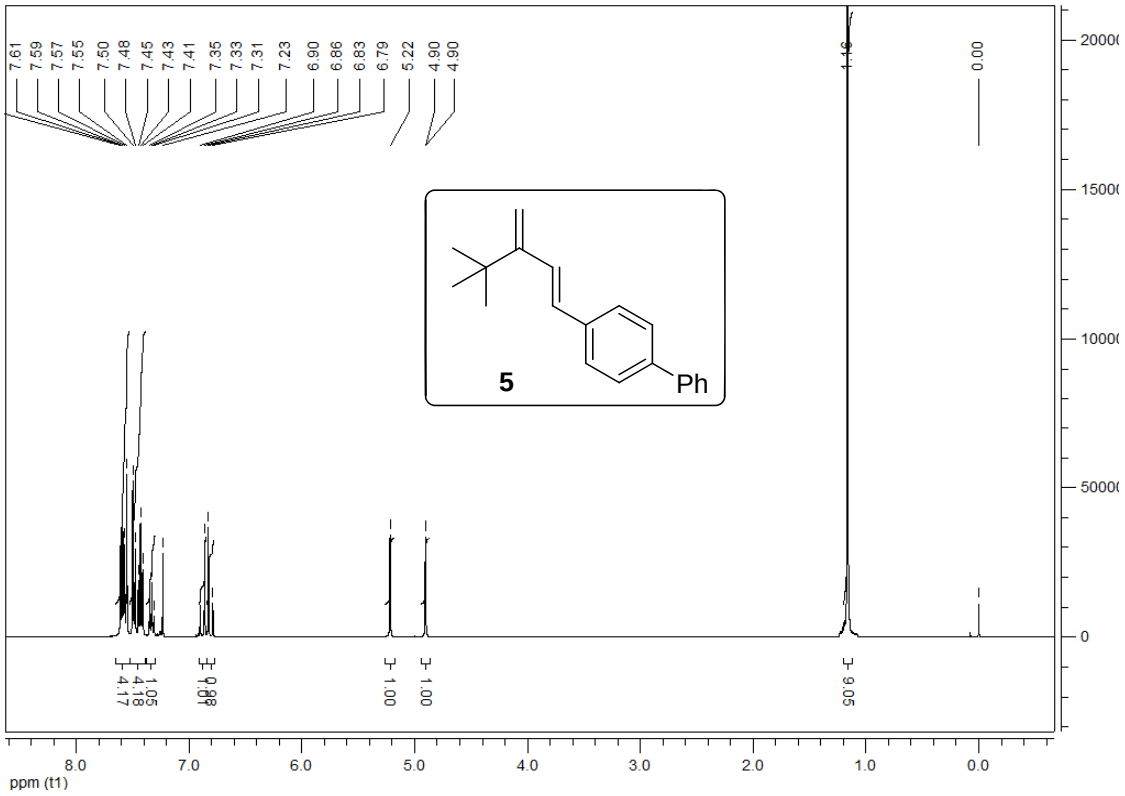
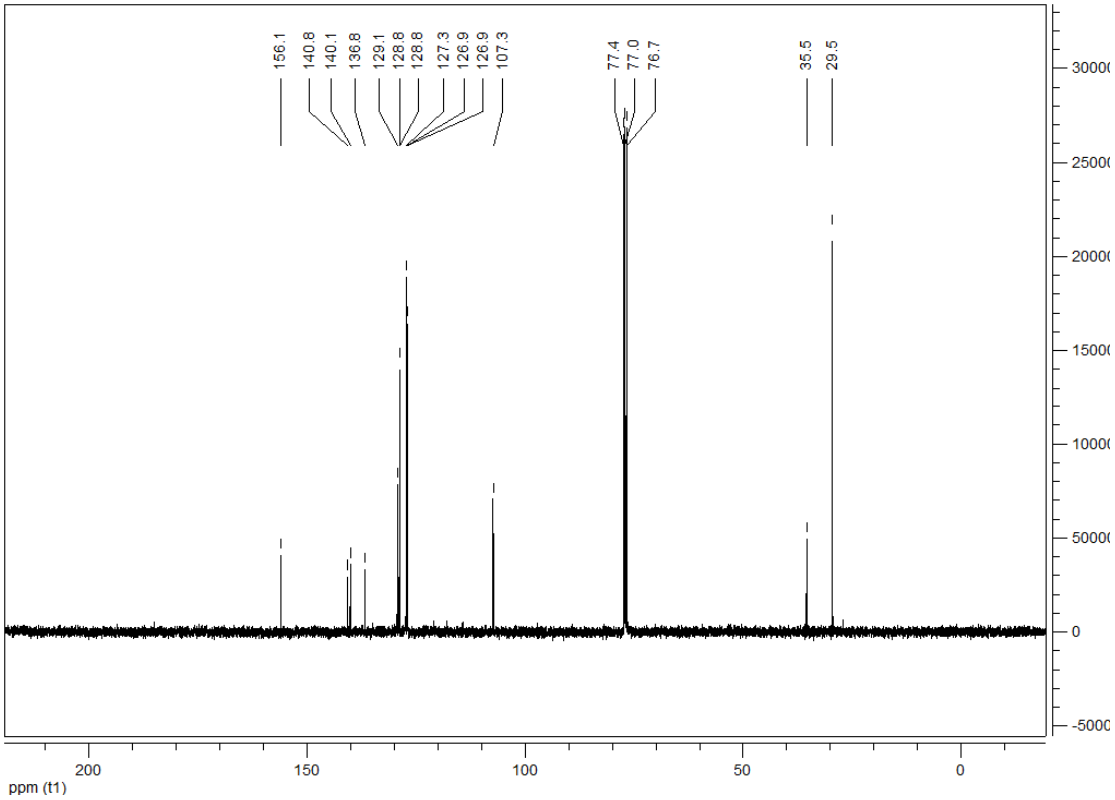
148.2
133.4
132.8
130.5
126.1
123.4
108.1
77.3
77.0
76.7
41.4
37.2
36.8
28.6
200
150
100
50
0
ppm (t1)


Ph
4
Ph
ppm (t1)


ppm (t1)


7.61
7.59
7.57
7.55
7.50
7.48
7.45
7.43
7.41
7.35
7.33
7.31
7.23
6.90
6.86
6.83
6.79
5.22
4.90
4.90
1.16
0.00
5
Ph
4.17
1.05
1.47
1.18
0.88
1.00
1.00
9.06
ppm (t1)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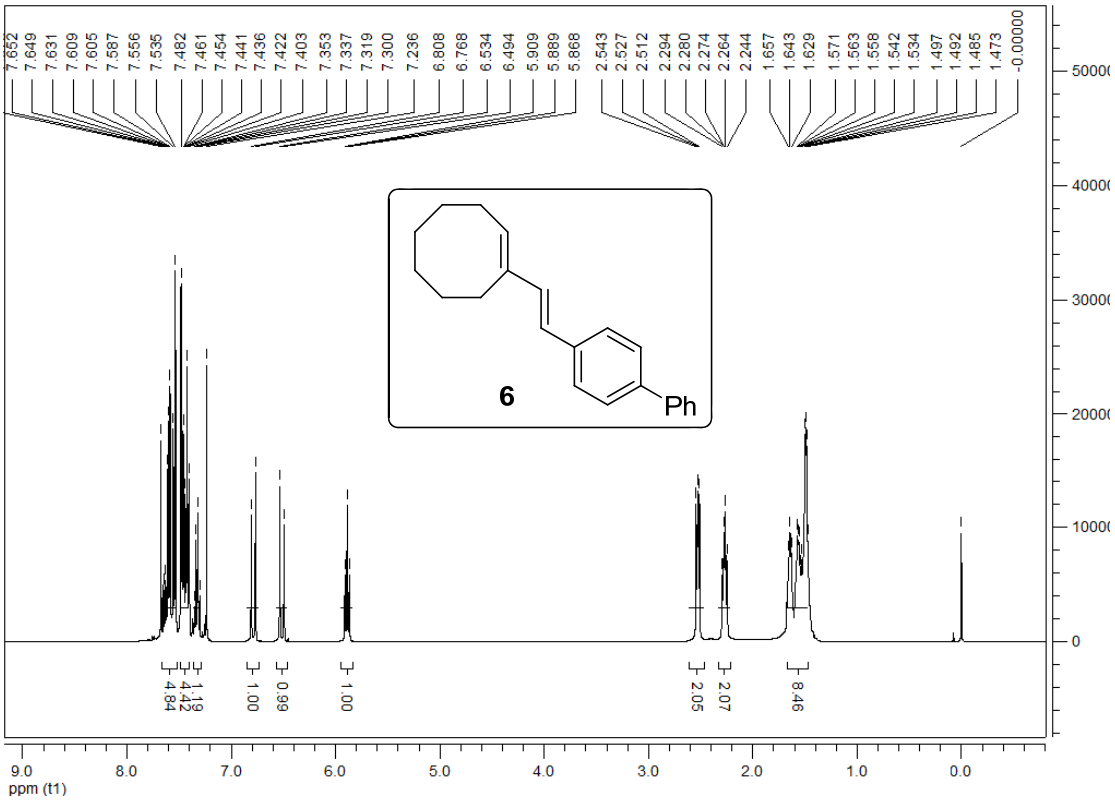
140.8
140.1
136.8
129.1
128.8
128.8
127.3
126.9
126.9
107.3
77.4
77.0
76.7
35.5
29.5
ppm (t1)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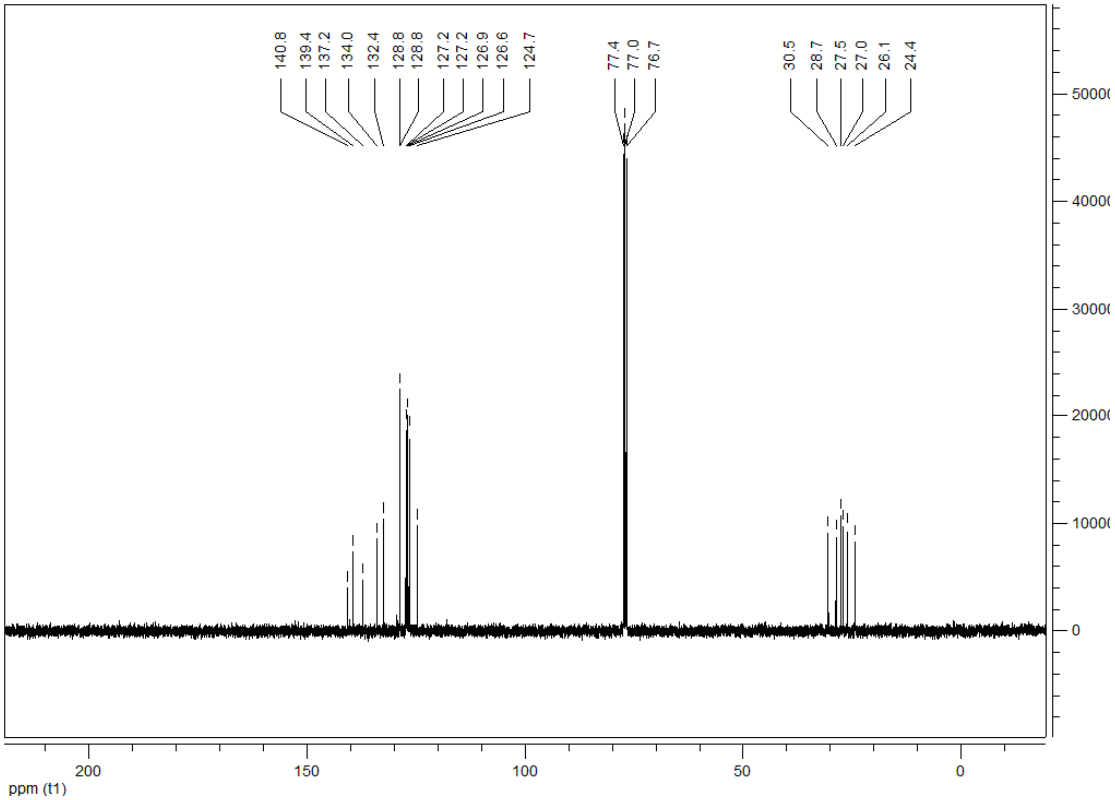
7.587
7.556
7.535
7.482
7.461
7.454
7.441
7.436
7.422
7.403
7.353
7.337
7.319
7.300
7.236
6.808
6.768
6.534
6.494
5.909
5.889
5.868
2.543
2.527
2.512
2.294
2.280
2.274
2.264
2.244
1.657
1.643
1.629
1.571
1.563
1.558
1.542
1.534
1.497
1.492
1.485
1.473
-0.00000
6
Ph
4.84
4.19
4.12
1.00
0.99
1.00
2.05
2.07
8.46
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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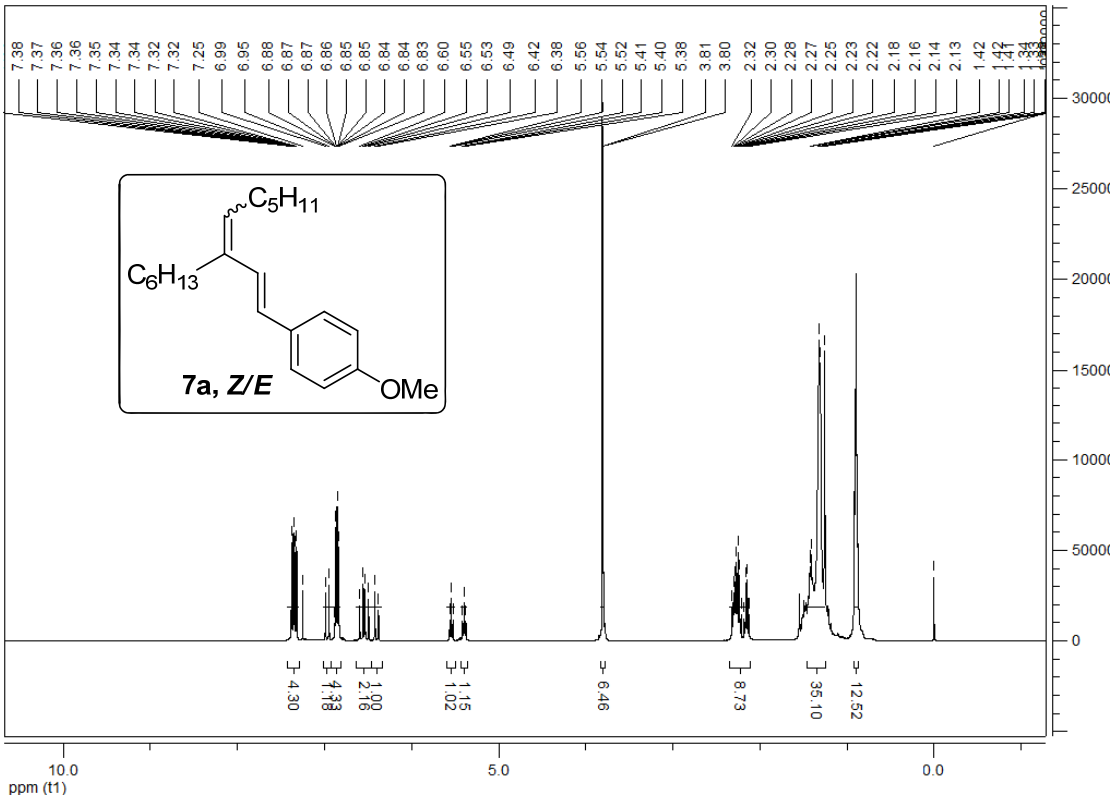
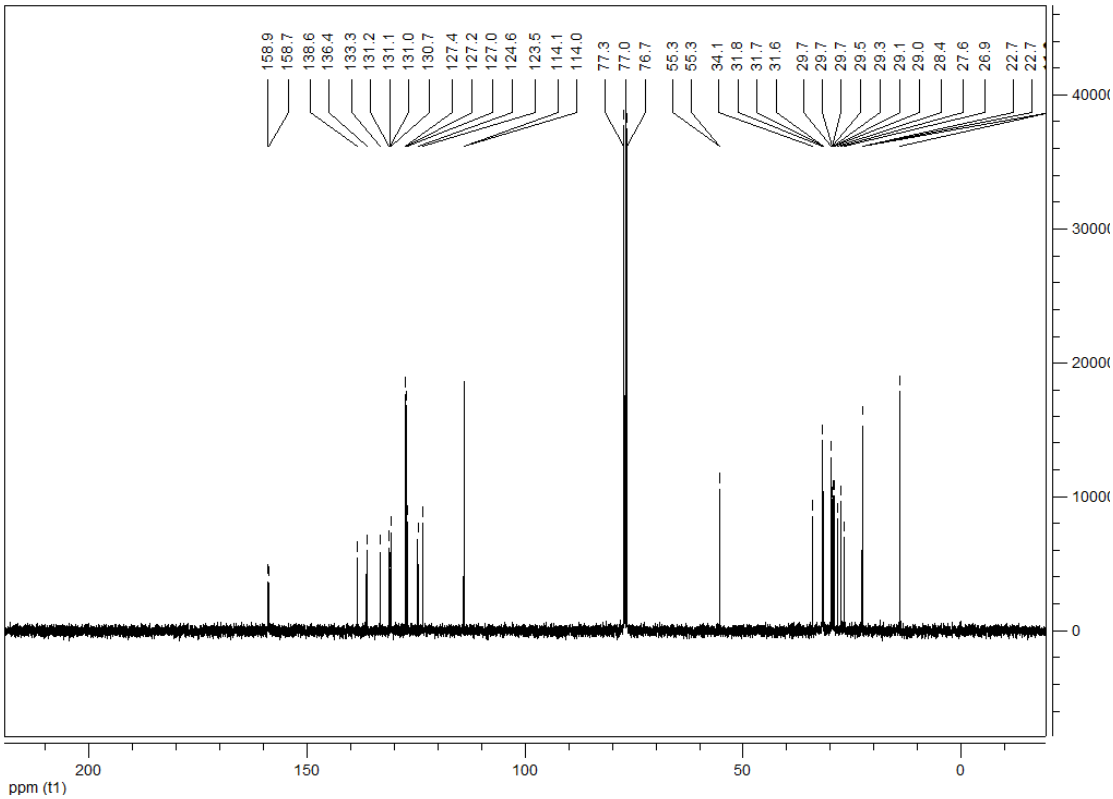
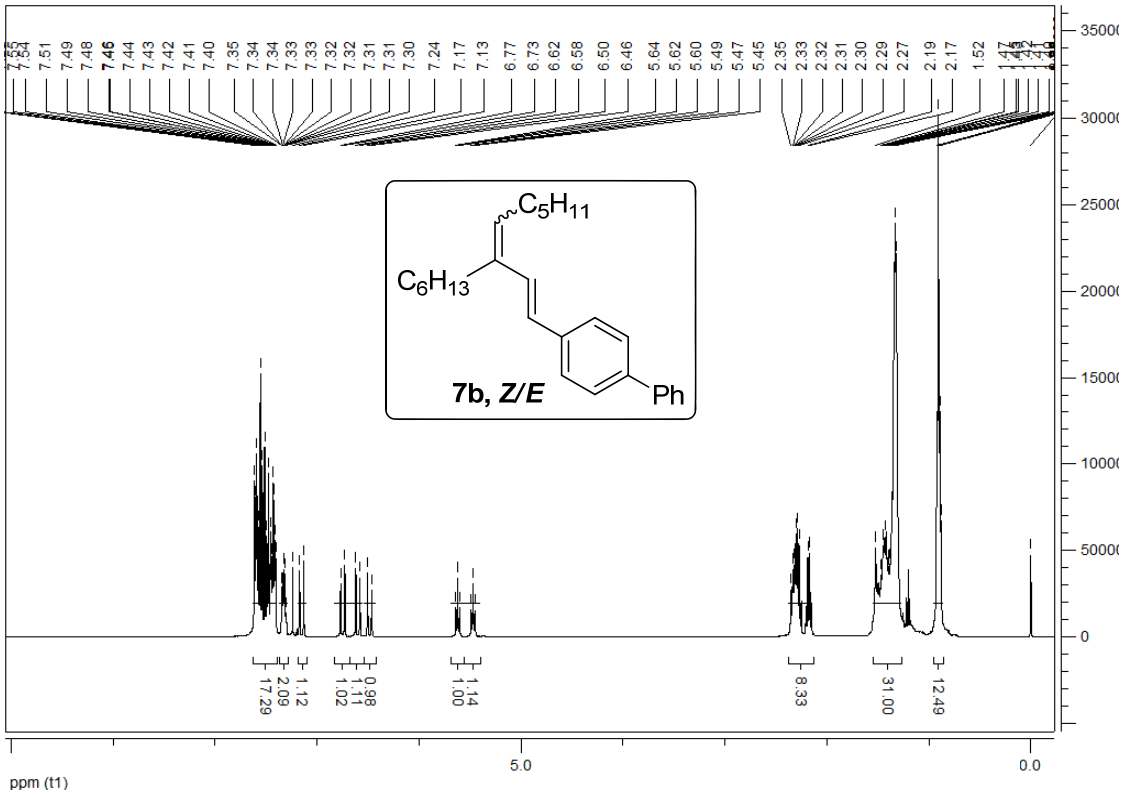
137.2
134.0
132.4
128.8
128.8
127.2
127.2
126.9
126.6
124.7
77.4
77.0
76.7
30.5
28.7
27.5
27.0
26.1
24.4
200
150
100
50
0
ppm (t1)


7.38 7.37 7.36 7.36 7.35 7.34 7.34 7.32 7.32 7.25 6.99 6.95 6.88 6.87 6.87 6.86 6.85 6.85 6.84 6.84 6.83 6.60 6.55 6.53 6.49 6.42 6.38 5.56 5.54 5.52 5.41 5.40 5.38 3.81 3.80 2.32 2.30 2.28 2.27 2.25 2.23 2.22 2.18 2.16 2.14 2.13 1.42
C5H11
C6H13
7a, Z/E
OMe
4.30 4.33 2.16 1.00 1.02 1.15 6.46 8.73 35.10 12.52
10.0 5.0 0.0
ppm (t1)


158.9 158.7 138.6 136.4 133.3 131.2 131.1 131.0 130.7 127.4 127.2 127.0 124.6 123.5 114.1 114.0 77.3 77.0 76.7 55.3 55.3 34.1 31.8 31.7 31.6 29.7 29.7 29.7 29.5 29.3 29.1 29.0 28.4 27.6 26.9 22.7 22.7
200 150 100 50 0
ppm (t1)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7.34
7.34
7.33
7.33
7.32
7.32
7.31
7.31
7.30
7.24
7.17
7.13
6.77
6.73
6.62
6.58
6.50
6.46
5.64
5.62
5.60
5.49
5.47
5.45
2.35
2.33
2.32
2.31
2.30
2.29
2.27
2.19
2.17
1.52
1.47
1.43
1.42
1.40
C5H11
C6H13
7b, Z/E
Ph
17.29
2.09
2.12
1.02
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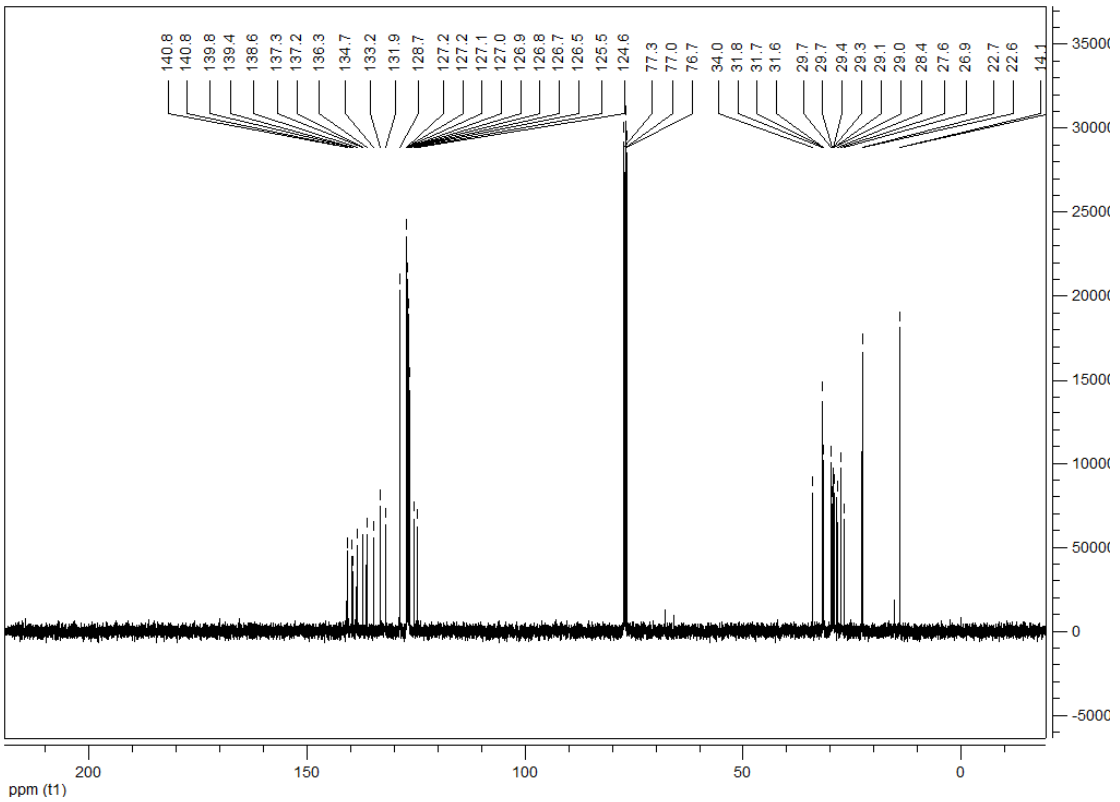
1.00
1.14
8.33
31.00
12.49
5.0
0.0
ppm (t1)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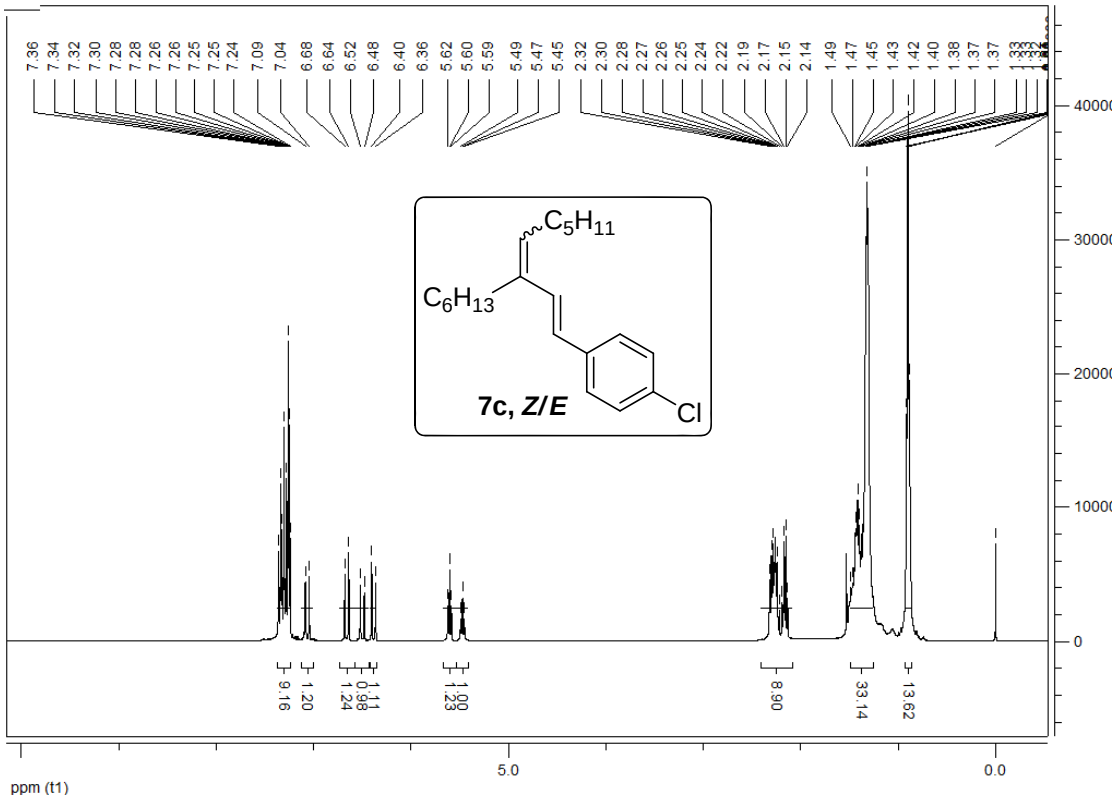
136.3
134.7
133.2
131.9
128.7
127.2
127.2
127.1
127.0
126.9
126.8
126.7
126.5
125.5
124.6
77.3
77.0
76.7
34.0
31.8
31.7
31.6
29.7
29.7
29.4
29.3
29.1
29.0
28.4
27.6
26.9
22.7
22.6
14.1
200
150
100
50
0
ppm (t1)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7.26
7.26
7.25
7.25
7.24
7.09
7.04
6.68
6.64
6.52
6.48
6.40
6.36
5.62
5.60
5.59
5.49
5.47
5.45
2.32
2.30
2.28
2.27
2.26
2.25
2.24
2.22
2.19
2.17
2.15
2.14
1.49
1.47
1.45
1.43
1.42
1.40
1.38
1.37
1.37
C5H11
C6H13
Cl
7c, Z/E
6.19
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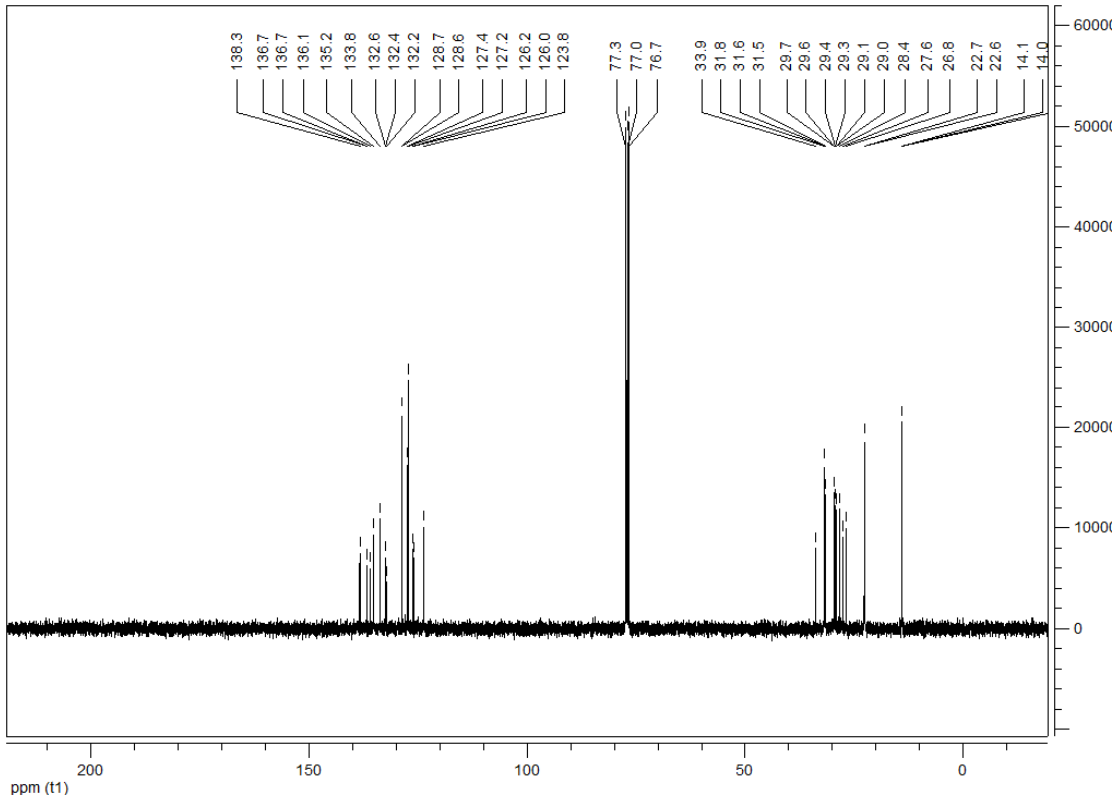
1.24
0.98
1.11
1.23
1.00
8.90
13.14
13.62
ppm (t1)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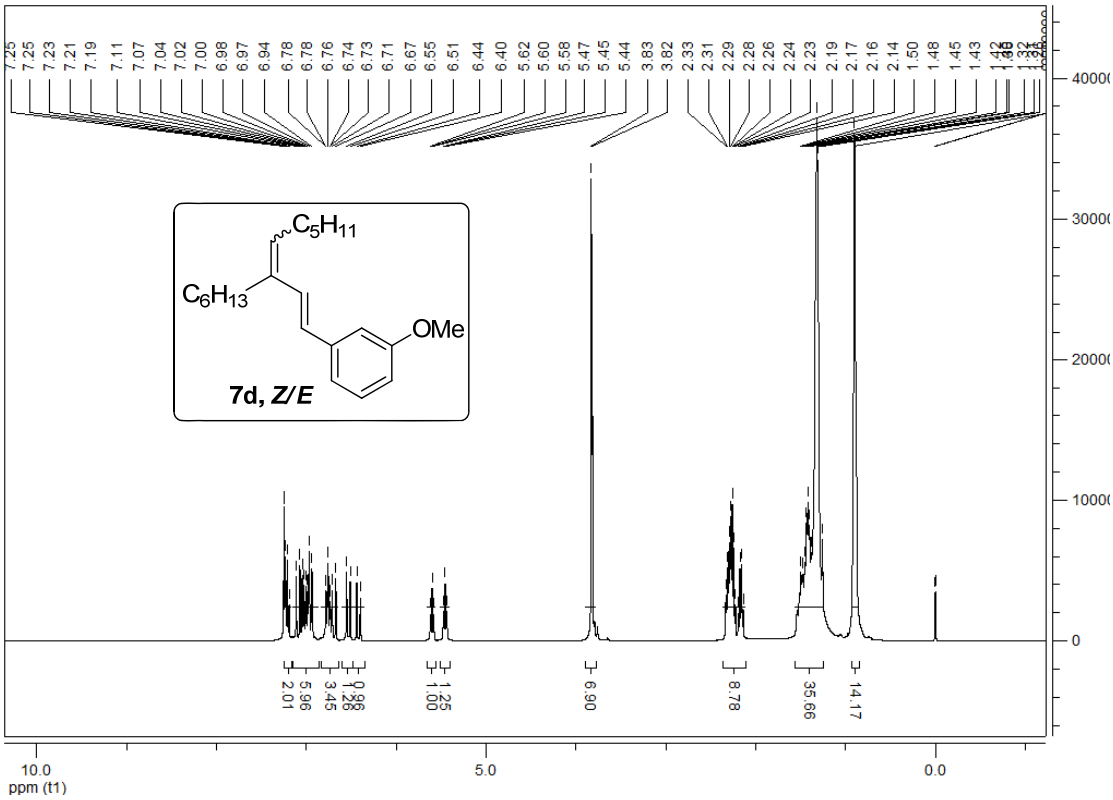
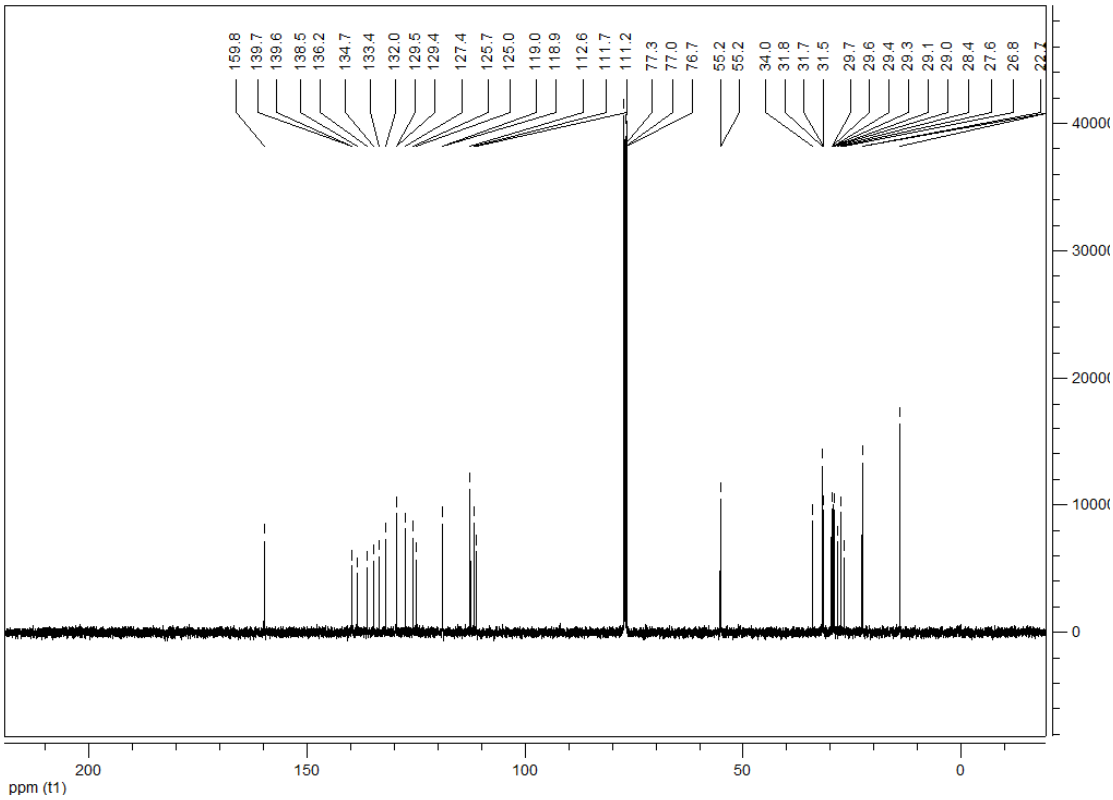
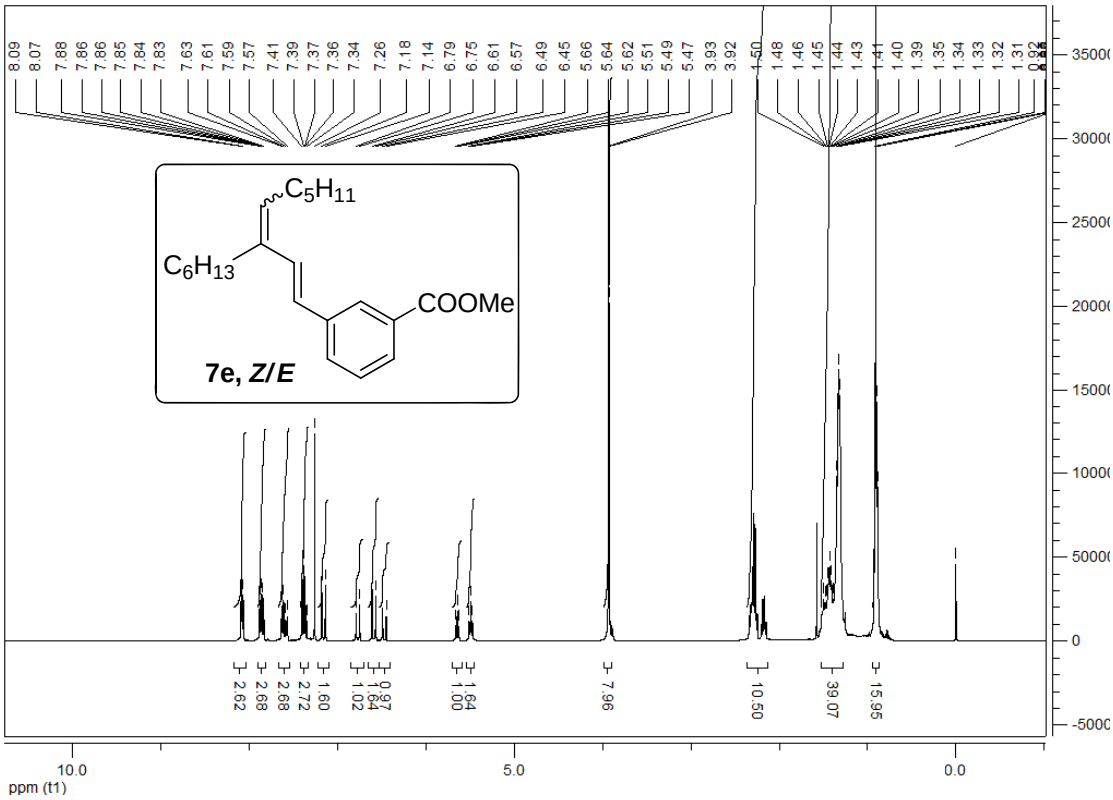
136.7
136.7
136.1
135.2
133.8
132.6
132.4
132.2
128.7
128.6
127.4
127.2
126.2
126.0
123.8
77.3
77.0
76.7
33.9
31.8
31.6
31.5
29.7
29.6
29.4
29.3
29.1
29.0
28.4
27.6
26.8
22.7
22.6
14.1
14.0
ppm (t1)


C5H11
C6H13
OMe
7d, Z/E
10.0
5.0
0.0
ppm (t1)


200
150
100
50
0
ppm (t1)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7.83
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7.59
7.57
7.41
7.39
7.37
7.36
7.34
7.26
7.18
7.14
6.79
6.75
6.61
6.57
6.49
6.45
5.66
5.64
5.62
5.51
5.49
5.47
3.93
3.92
1.50
1.48
1.46
1.45
1.44
1.43
1.41
1.40
1.39
1.35
1.34
1.33
1.32
1.31
C5H11
C6H13
COOMe
7e, Z/E
2.62
2.68
2.68
2.72
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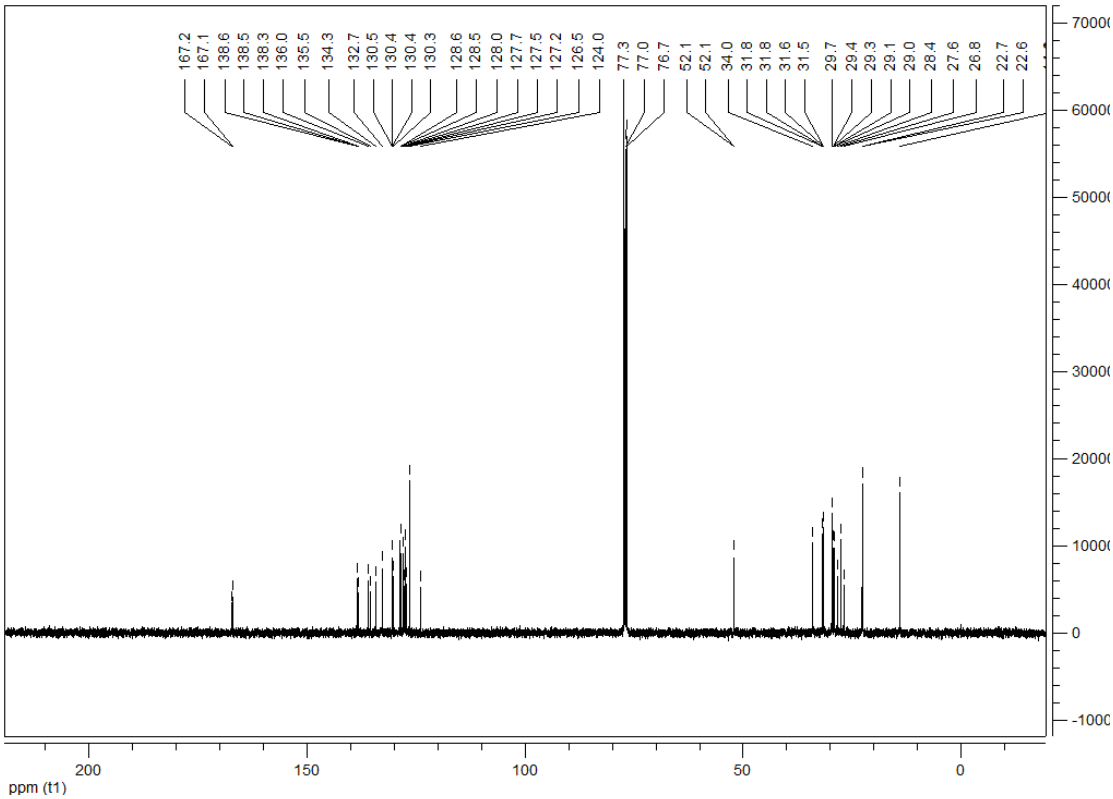
1.02
0.97
1.00
1.64
7.96
10.50
39.07
15.95
ppm (t1)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167.1
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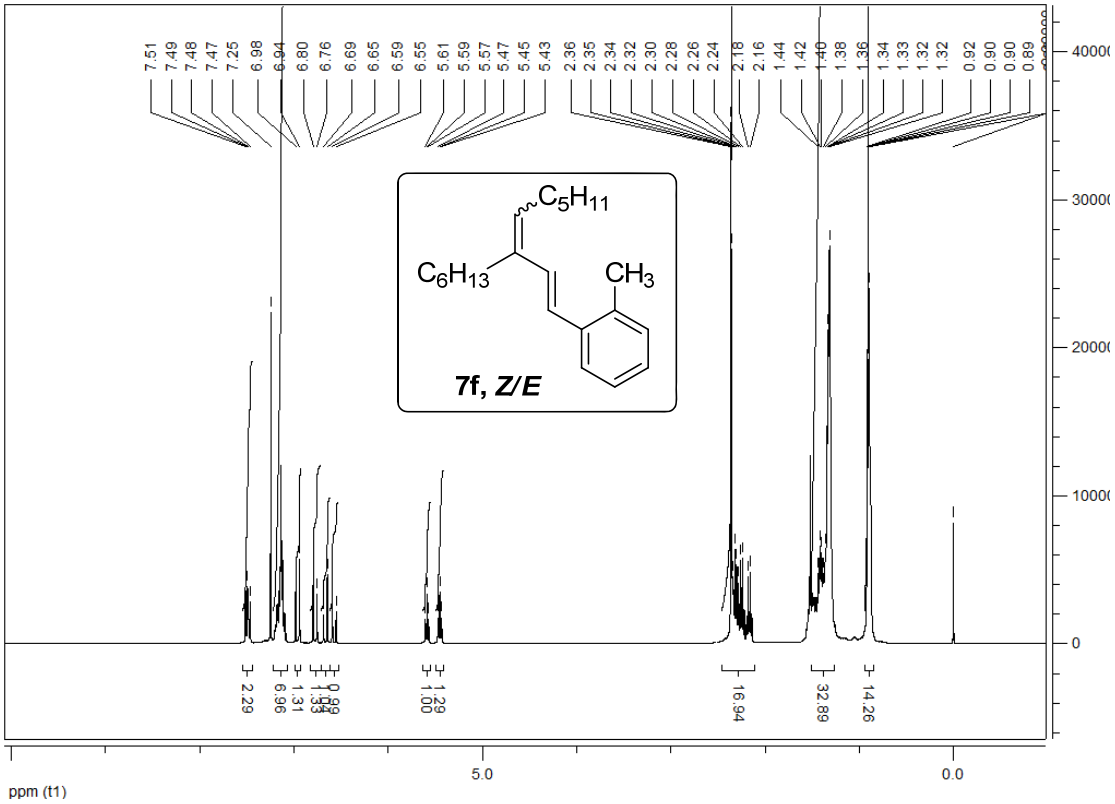
134.3
132.7
130.5
130.4
130.4
130.3
128.6
128.5
128.0
127.7
127.5
127.2
126.5
124.0
77.3
77.0
76.7
52.1
52.1
34.0
31.8
31.8
31.6
31.5
29.7
29.4
29.3
29.1
29.0
28.4
27.6
26.8
22.7
22.6
ppm (t1)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7.49
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6.80
6.76
6.69
6.65
6.59
6.55
5.61
5.59
5.57
5.47
5.45
5.43
2.36
2.35
2.34
2.32
2.30
2.28
2.26
2.24
2.18
2.16
1.44
1.42
1.40
1.38
1.36
1.34
1.33
1.32
0.92
0.90
0.90
0.89
C5H11
C6H13
CH3
7f, Z/E
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9.96
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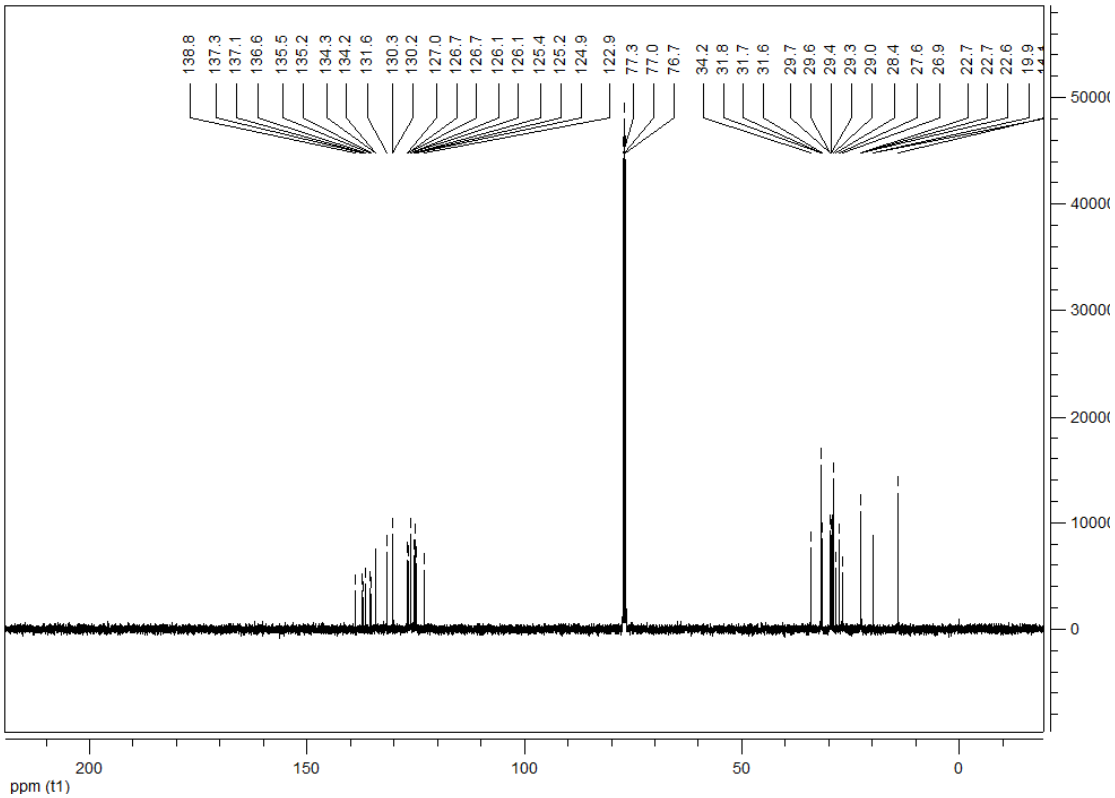
1.33
1.00
1.29
19.64
2.89
14.26
5.0
0.0
ppm (t1)


138.8
137.3
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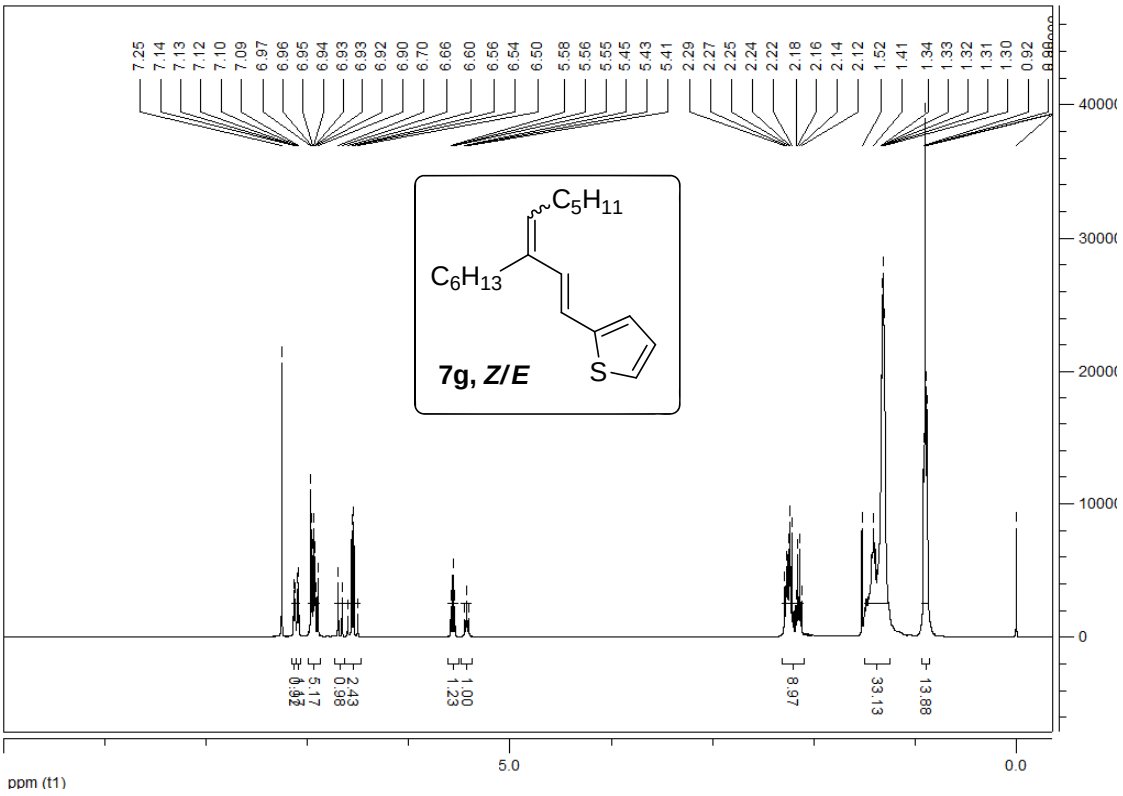
136.6
135.5
135.2
134.3
134.2
131.6
130.3
130.2
127.0
126.7
126.7
126.1
126.1
125.4
125.2
124.9
122.9
77.3
77.0
76.7
34.2
31.8
31.7
31.6
29.7
29.6
29.4
29.3
29.0
28.4
27.6
26.9
22.7
22.7
22.6
19.9
200
150
100
50
0
ppm (t1)


7.25
7.14
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7.12
7.10
7.09
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6.95
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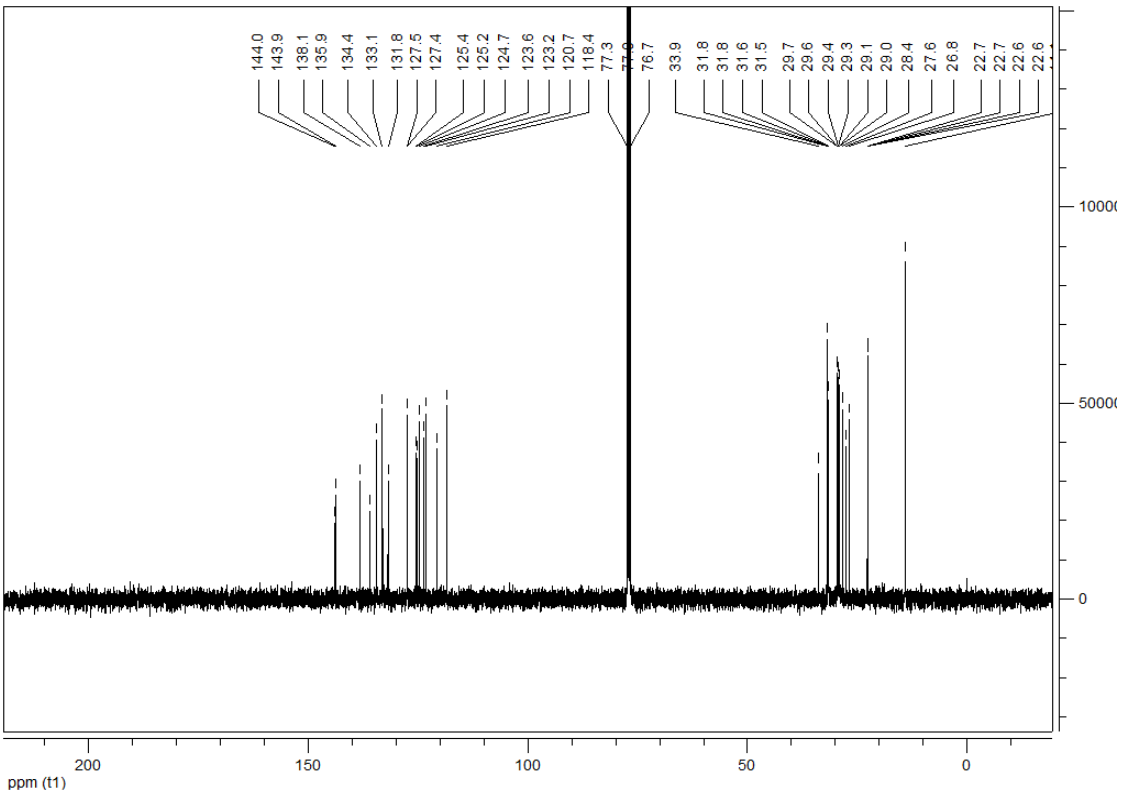
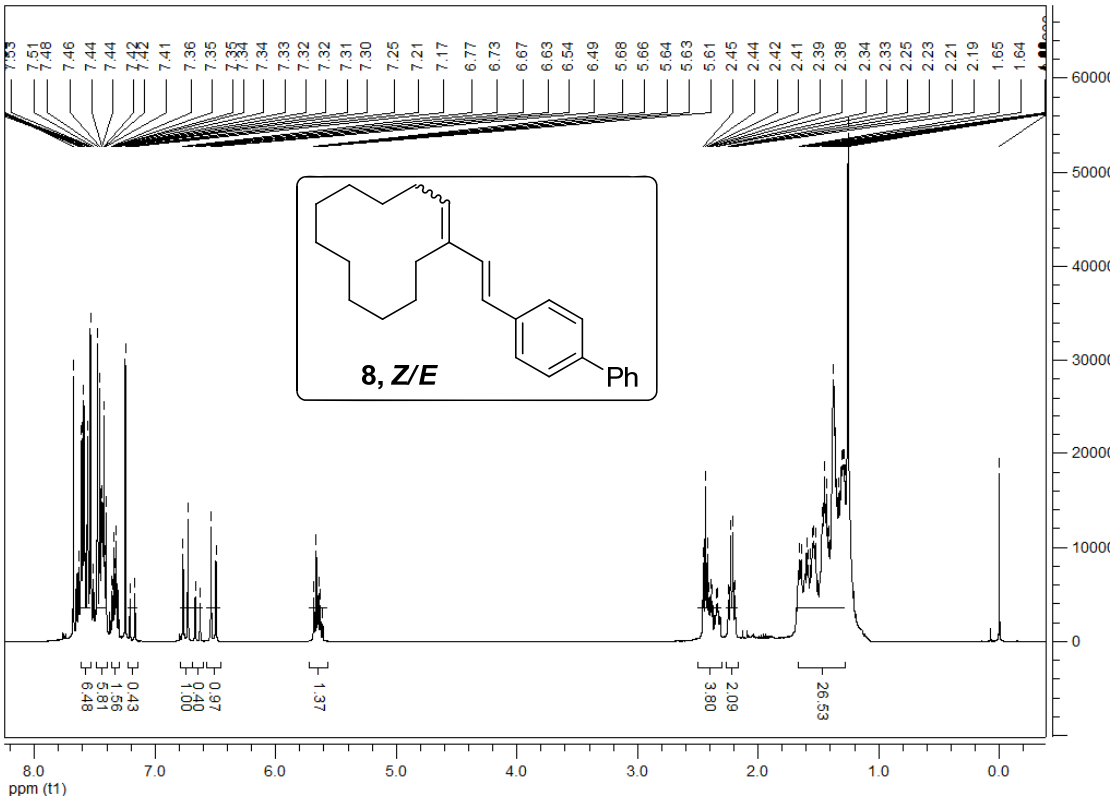
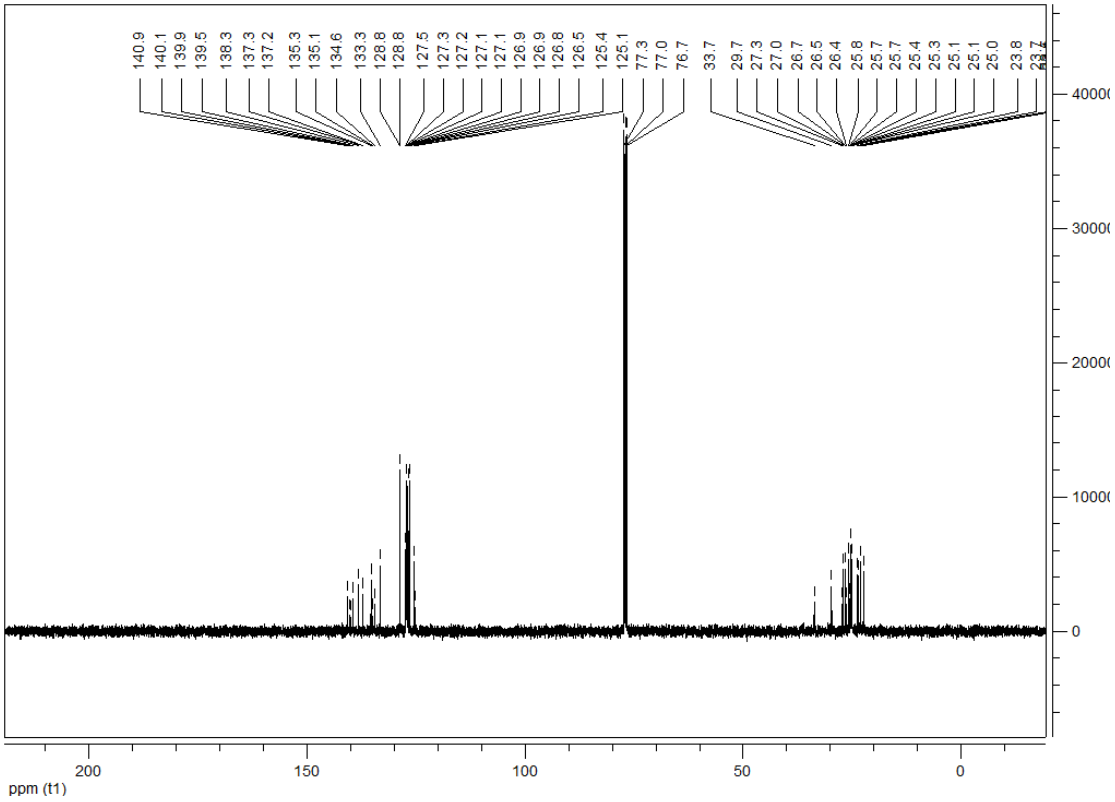
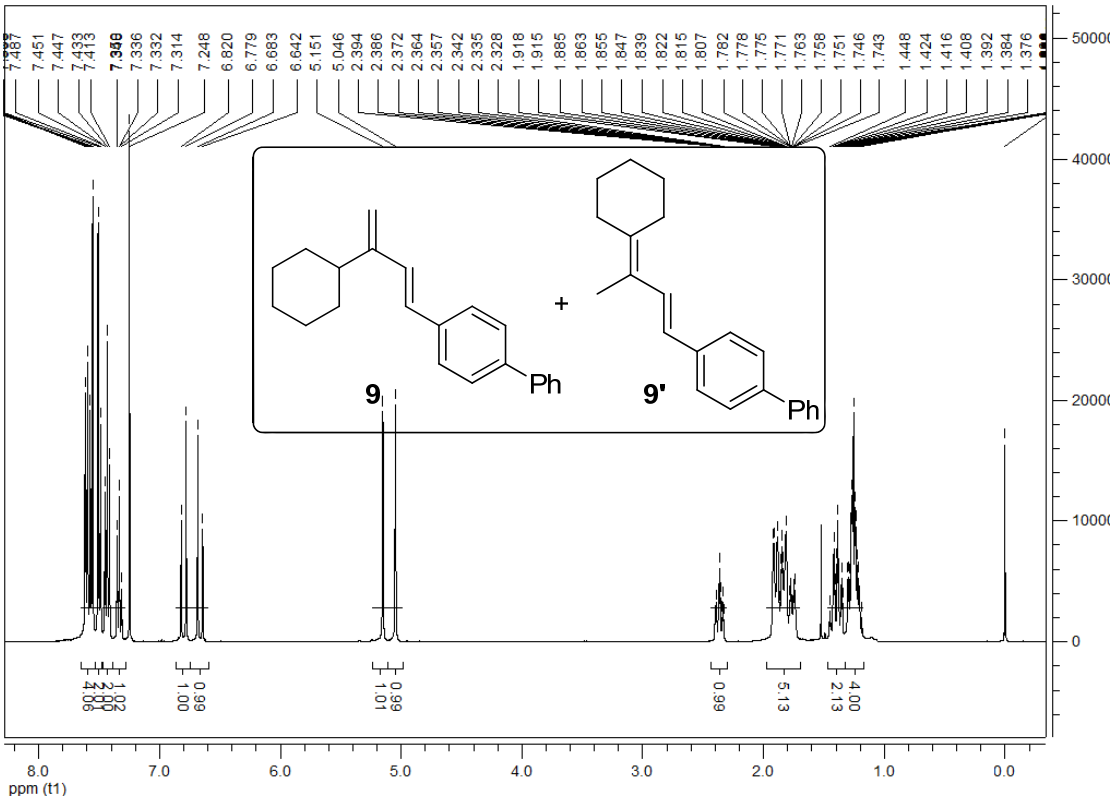
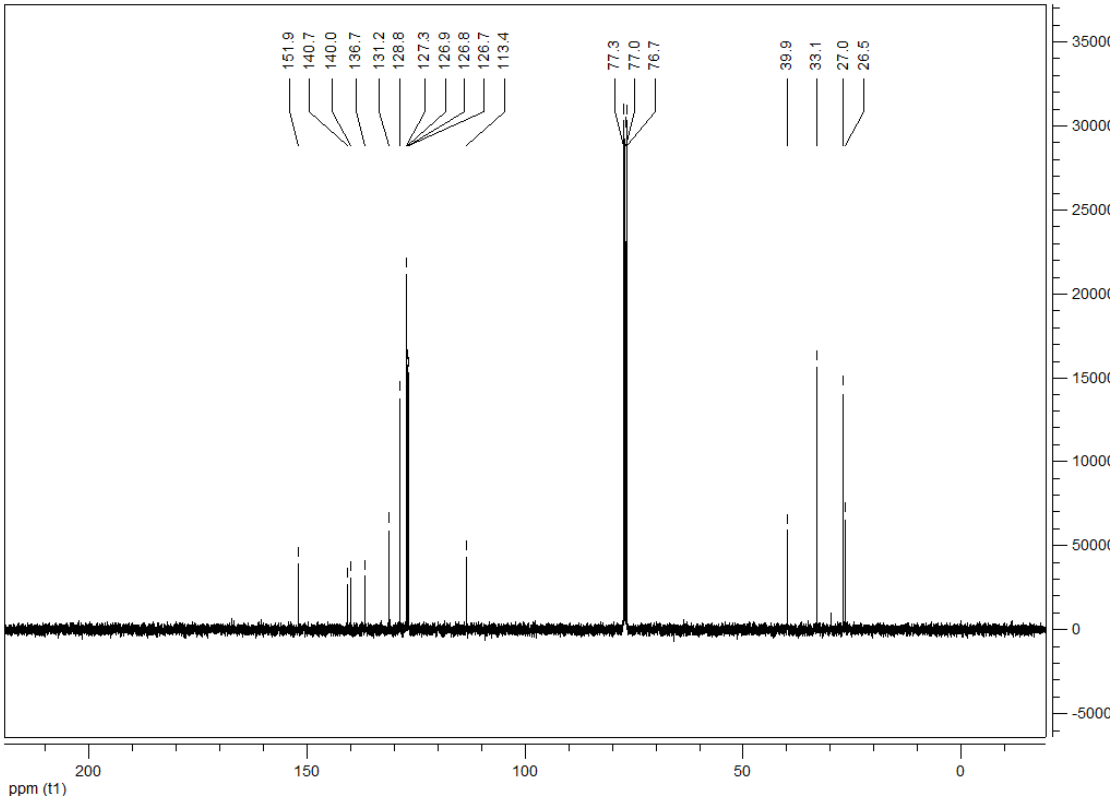
6.90
6.70
6.66
6.60
6.56
6.54
6.50
5.58
5.56
5.55
5.45
5.43
5.41
2.29
2.27
2.25
2.24
2.22
2.18
2.16
2.14
2.12
1.52
1.41
1.34
1.33
1.32
1.31
1.30
0.92
C_5H_{11}
C_6H_{13}
S
7g, *Z*/*E*
0.92
5.17
6.98
2.43
1.23
1.00
8.97
3.13
13.88
4000
3000
2000
1000
0
5.0
0.0
ppm (t1)

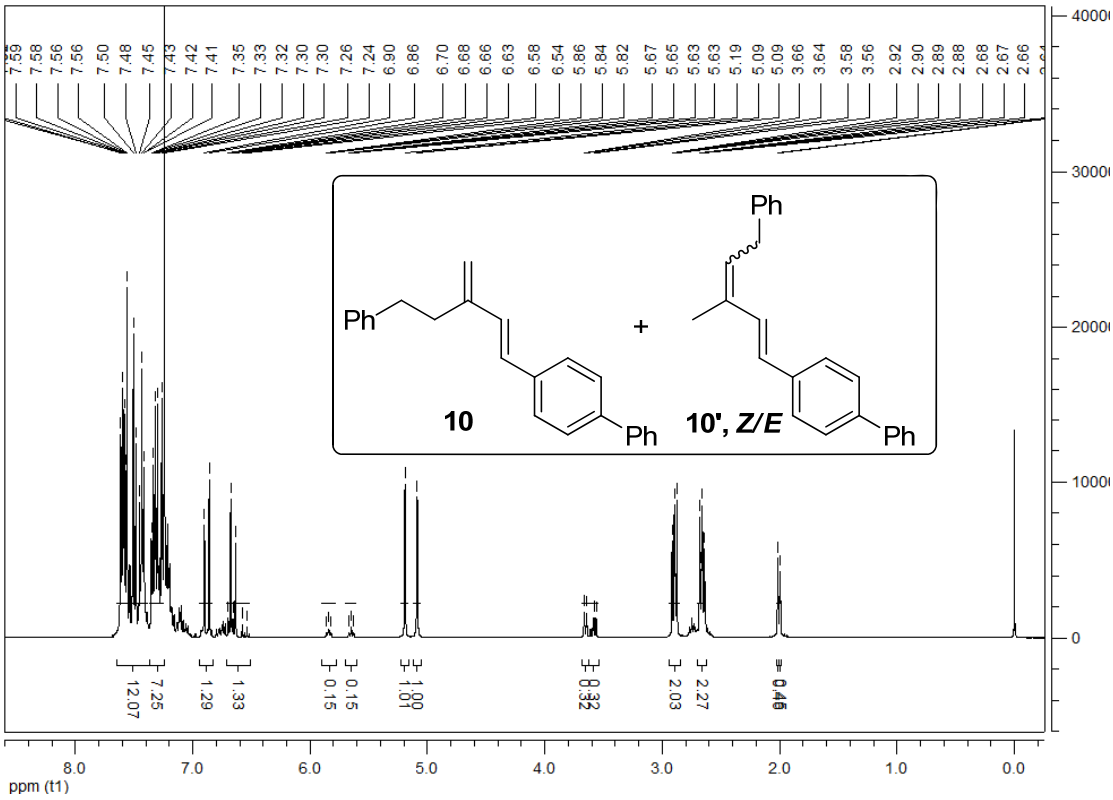

144.0
143.9
138.1
135.9
134.4
133.1
131.8
127.5
127.4
125.4
125.2
124.7
123.6
123.2
120.7
118.4
77.3
77.0
76.7
33.9
31.8
31.8
31.6
31.5
29.7
29.6
29.4
29.3
29.1
29.0
28.4
27.6
26.8
22.7
22.7
22.6
22.6
1000
5000
0
200
150
100
50
0
ppm (t1)

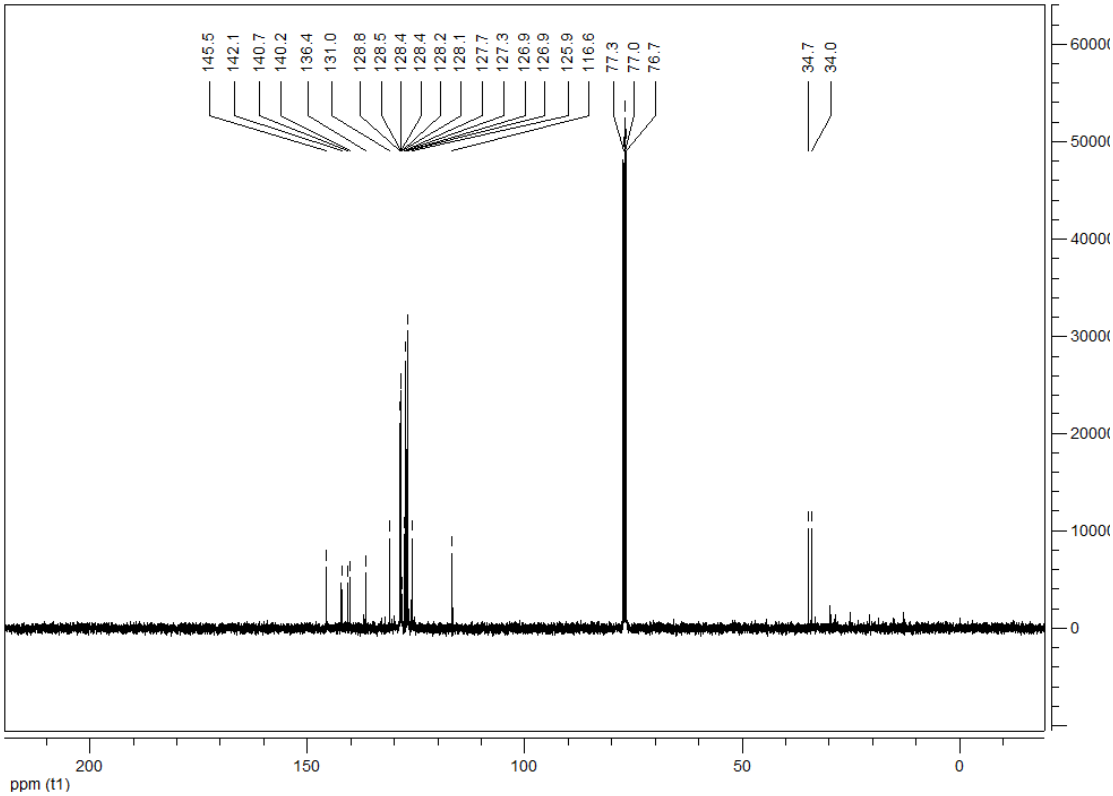

8, Z/E
Ph
ppm (t1)

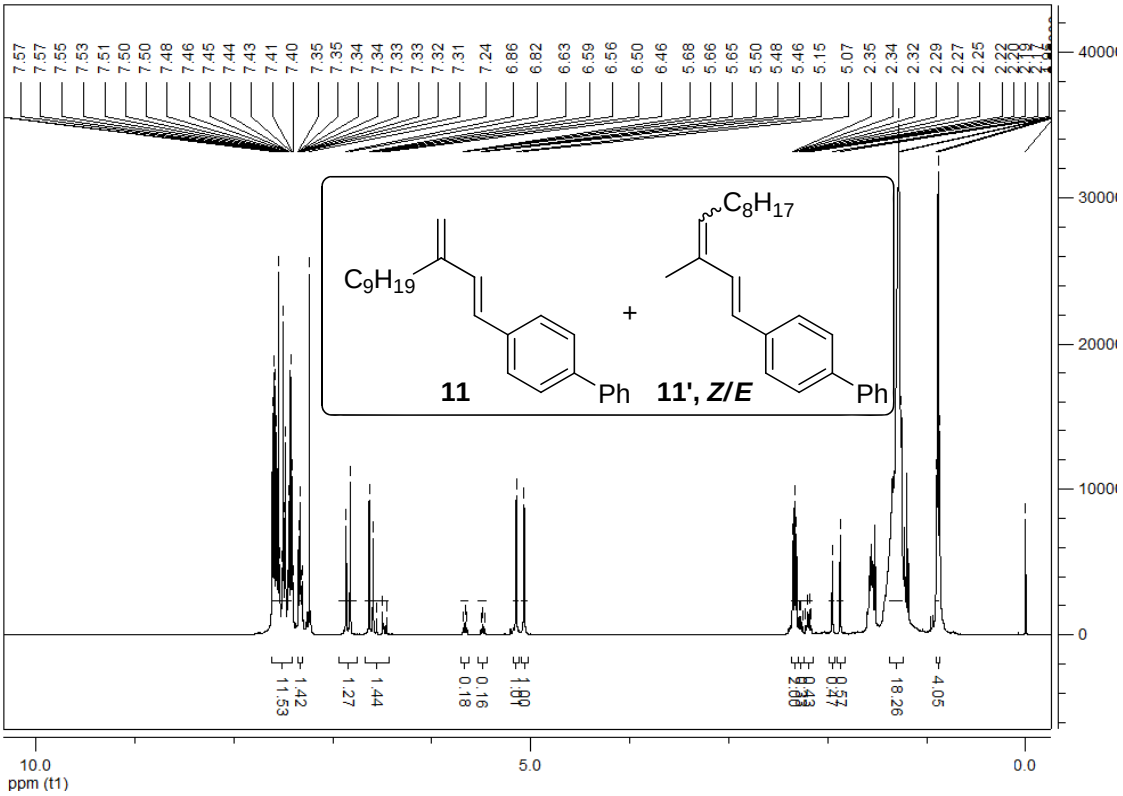

ppm (t1)

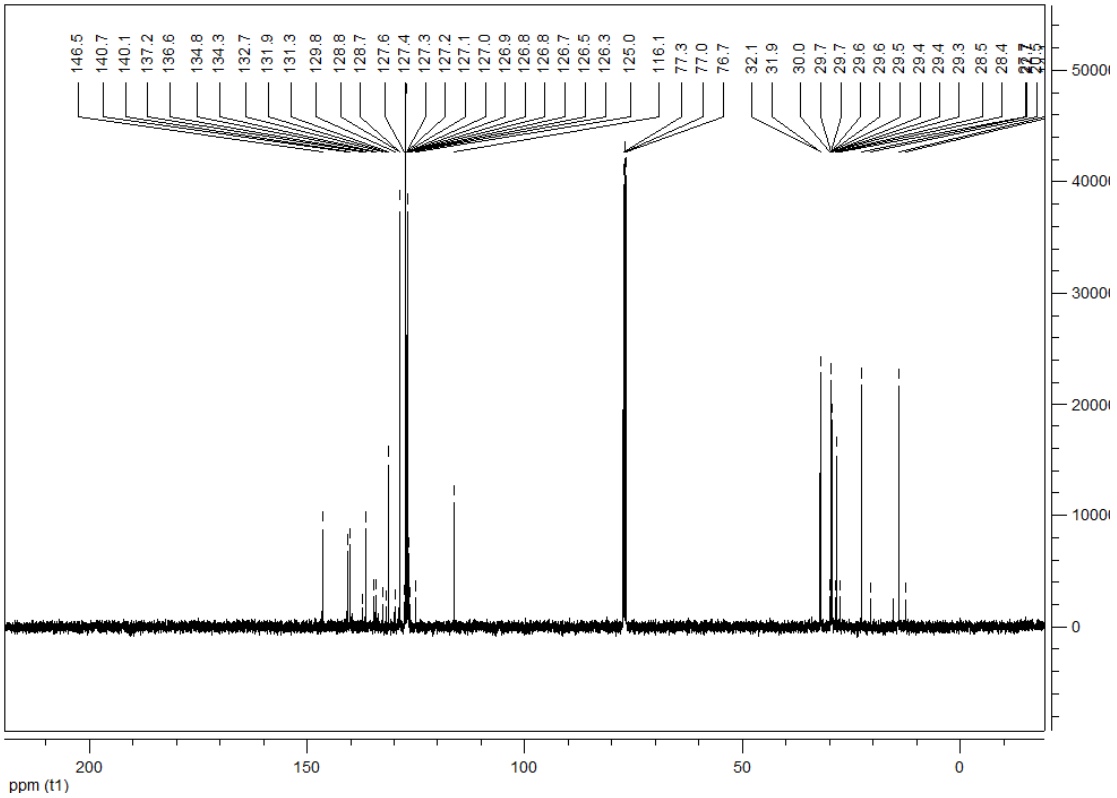

9
Ph
+
9'
Ph
ppm (t1)


151.9
140.7
140.0
136.7
131.2
128.8
127.3
126.9
126.8
126.7
113.4
77.3
77.0
76.7
39.9
33.1
27.0
26.5
ppm (t1)


Ph
Ph
10
+
Ph
Ph
10', Z/E
ppm (t1)


ppm (t1)


11
11', Z/E
Ph
Ph
10.0
5.0
0.0
ppm (t1)


200
150
100
50
0
ppm (t1)