Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Consecutive visible-light photoredox decarboxylative couplings of adipic acid active esters with alkynyl sulfones leading to cyclic compounds

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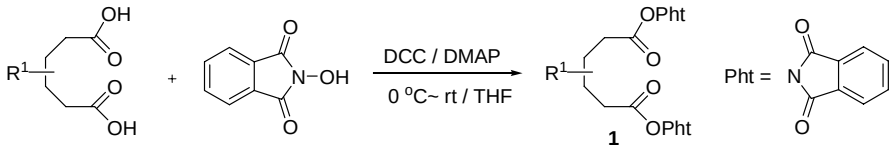
General Procedures

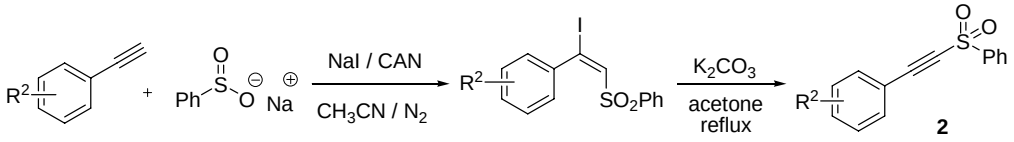
All reactions were carried out in dry solvents and under an atmosphere of argon unless otherwise indicated. Dichloromethane, dichloroethane and MeCN were dried over calcium hydride. Reagents were purchased from commercial sources and used directly without further purification. Substrates adipic acid active esters and alkynyl sulfones were prepared according to the previously reported procedures.[1,2] Reactions were monitored by thin layer chromatography (TLC) and products were obtained by column chromatography on silica gel. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded in $CDCl_3$ using TMS as an internal standard (^{1}H NMR: TMS at 0.00 ppm, $CHCl_3$ at 7.26 ppm; ^{13}C NMR: $CDCl_3$ at 77.16 ppm. Signal patterns are indicated as s, singlet; d, doublet; t, triplet; q, quartet; and m, multiplet.)

General procedure for synthesis of adipic acid active esters



DCC (24 mmol) was added to a stirred solution of adipic acid (10 mmol), *N*-hydroxyphthalimide (24 mmol) and DMAP (2 mmol) in THF (100 mL) at 0℃ under N_2 during 15 min, and stirred overnight at room temperature. After the completion of the reaction, the precipitate was filtered and washed with CH_2Cl_2. The filtrate was evaporated by rotary evaporator, and the residue was purified by silica gel column chromatography (CH_2Cl_2 or CH_2Cl_2/EtOAc) to provide the adipic acid active ester (**1**) as a white solid.

General procedure for synthesis of alkynyl sulfones



To a mixture of arylacetylene (1 mmol), sodium benzenesulfinate (1.2 mmol) and NaI (1.2 mmol) in anhydrous CH_3CN (5 mL) was added a solution of CAN (2.5 mmol) in the same solvent (10 mL) under an argon atmosphere and the reaction was stirred at

room temperature overnight. After the completion of the reaction, the reaction mixture was diluted with H_2O (20 mL), extracted with CH_2Cl_2 (3 × 20 mL). The combined organic phase was washed with sat. $Na_2S_2O_3$ (20 mL), brine (20 mL) and dried over anhydrous Na_2SO_4.

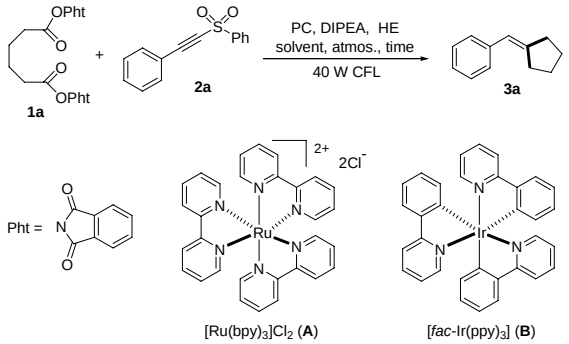
The residue after removing the solvent was refluxed with K_2CO_3 (2 mmol) in anhydrous acetone (5 mL) for about 5 hours. After the completion of the reaction, the reaction mixture after removing the solvent was washed with H_2O (20 mL) and extracted with CH_2Cl_2 (3 × 20 mL). The combined organic phase was washed with brine (20 mL) and dried over anhydrous Na_2SO_4. The solvent was removed in vacuo using a rotary evaporator, and the residue was chromatographed to afford the target product (**2**).

Optimization of conditions

Consecutive photoredox decarboxylative couplings of bis(1,3-dioxoisoindolin-2-yl)hexanedioate (**1a**) with 1-(2-phenylethynylsulfonyl)benzene (**2a**) was selected as the model reaction to optimize conditions including photocatalysts, solvents, atmosphere and time. As shown in Table S1, five solvents were screened using 1.0 mol% $[Ru(bpy)_3]Cl_2$ as the photoredox catalyst, 4.4 equiv of diisopropylethylamine (DIPEA) and 3.0 equiv of Hantzsch ester (HE) (relative to amount of **2a**) as the radical initiators and reductants at room temperature under argon atmosphere for 12 h (entries 1-5), and dichloromethane (DCM) gave the highest yield (entry 1). Yields were changed when time was shortened (entries 6-8), and the reaction provided 90% yield within 4 h. When air replaced argon atmosphere, a 68% yield was afforded (entry 9). No reaction occurred in the absence of photocatalyst (entry 10). When [*fac*-$Ir(ppy)_3$] was used as the photoredox catalyst, only trace amount of product was observed (entry 11). Yield dramatically decreased (only 39% yield) without addition of DIPEA (entry 12). The reaction did not work in the absence of HE (entry 13) or light (entry 14). When diethylamine (entry 15) or K_2CO_3 (entry 16) replaced DIPEA, the poor results were observed. Therefore, the optimal photoredox conditions are as follows: 1.0 mol%

$[Ru(bpy)_3]Cl_2$ as the photocatalyst, 4.4 equiv of DIPEA and 3.0 equiv of HE as the radical initiators and reductants at room temperature under argon atmosphere.

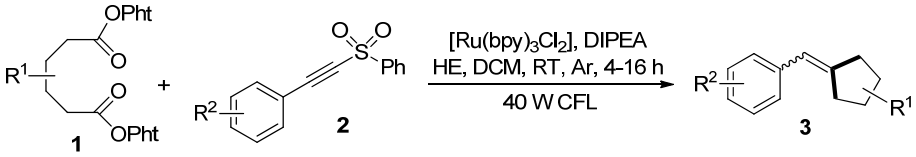
Table S1 Optimization of conditions on consecutive photoredox decarboxylative couplings of bis(1,3-dioxoisoindolin-2-yl)hexanedioate (**1a**) with 1-(2-phenylethynylsulfonyl)benzene (**2a**)[a]



Entry	PC	Solvent	Atmos.	Time (h)	Yield (%)[b]
1	A	DCM	Ar	12	86
2	A	DCE	Ar	12	71
3	A	DMF	Ar	12	76
4	A	DMSO	Ar	12	67
5	A	MeCN	Ar	12	42
6	A	DCM	Ar	8	90
7	**A**	**DCM**	**Ar**	**4**	**90**
8	A	DCM	Ar	2	47
9	A	DCM	air	4	68
10	-	DCM	Ar	4	NR[c]
11	B	DCM	Ar	4	trace
12	A	DCM	Ar	4	39[d]
13	A	DCM	Ar	4	NR[e]
14	A	DCM	Ar	4	NR[f]
15	A	DCM	Ar	4	0[g]
16	A	DCM	Ar	8	21[h]

[a] Reaction conditions: under irradiation of visible light, bis(1,3-dioxoisoindolin-2-yl)hexanedioate (**1a**) (0.3 mmol), 1-(2-phenylethynylsulfonyl)benzene (**2a**) (0.2 mmol), photocatalyst (PC) (2 μmol), diisopropylethylamine (DIPEA) (0.88 mmol), Hantzsch ester (HE) (0.6 mmol), solvent (1.5 mL), temperature (rt, ~25 °C), time (2-12 h) in a sealed Schlenk tube. [b] Isolated yield. [c] No addition of photocatalyst. [d] No addition of DIPEA. [e] No addition of HE. [f] No light. [g] using 0.88 mmol of diethylamine instead of DIPEA. [h] Using 0.88 mmol of K_2CO_3 instead of DIPEA. DCM = dichloromethane. DCE = 1,2-dichloroethane. DMF = *N,N*-dimethylformamide. DMSO = dimethyl sulfoxide. CFL = compact fluorescent light.

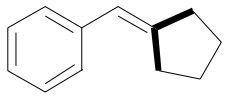
General procedure for synthesis of compound 3a-ac.



$[Ru(bpy)_3]Cl_2$ (2.0 μmol, 1.5 mg), bis(1,3-dioxoisoindolin-2-yl)-substituted adipate (**1**) (0.3mmol), substituted 1-(2-phenylethynylsulfonyl)benzene (**2**) (0.2 mmol), diisopropylethylamine amine (DIPEA) (0.88 mmol, 145 μL), Hantzsch ester (HE) (0.6 mmol, 152 mg), CH_2Cl_2 (1.5 mL) were added to a 25-mL Schlenk tube. The tube was filled with argon and then sealed, and irradiated with a 40 W fluorescent lamp (approximately 2 cm away from the light source). After the complete conversion of the substrates (monitored by TLC), the reaction mixture was concentrated and purified directly by silica gel column chromatography to give the desired product (**3a-ac**).

Characterization data of compounds 3a-ac

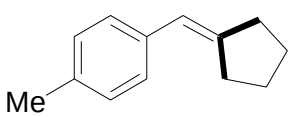
(Cyclopentylidenemethyl)benzene (3a)[3]



3a

Purified by silica gel column chromatography (*n*-hexane) to give compound **3a** as a colourless oil (28.4 mg, 90%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.35 - 7.34 (m, 4H), 7.22 - 7.17 (m, 1H), 6.41 - 6.39 (m, 1H), 2.61 - 2.57 (m, 2H), 2.55 - 2.51 (m, 2H), 1.86 - 1.79 (m, 2H), 1.74 - 1.67 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 147.3, 139.0, 128.3, 128.1, 125.7, 120.9, 36.1, 31.3, 27.4, 25.8; **IR** (cm^{-1}): 2956 (s), 2869 (m), 1654 (w), 1600 (w), 1489 (w), 1447 (m), 1260 (m), 1075 (m), 1027 (m), 802 (m), 692 (s); **EI-MS: [M^+] m/z** 158.3.

1-Cyclopentylidenemethyl-4-methylbenzene (3b)[3]

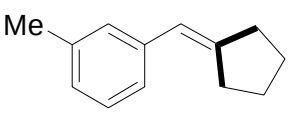


3b

Purified by silica gel column chromatography (*n*-hexane) to give compound **3b** as a colourless oil (25.1 mg, 73%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.19 (d, J = 8.2 Hz, 2H), 7.11 (d, J = 8.2 Hz, 2H), 6.32 - 6.31 (m, 1H), 2.54 - 2.50 (m, 2H), 2.48 - 2.44 (m, 2H), 2.32 (s,

3H), 1.80 - 1.73 (m, 2H), 1.68 - 1.61 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 146.2, 136.2, 135.3, 129.0, 128.0, 120.7, 36.0, 31.3, 27.4, 25.8, 21.2; **IR** (cm^{-1}): 2955 (s), 2870 (s), 1704 (s), 1607 (m), 1514 (m), 1449 (w), 1036 (m), 808 (s), 755 (m); **EI-MS: [M^+] m/z** 172.4.

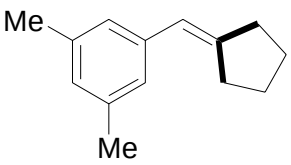
1-Cyclopentylidenemethyl-3-methylbenzene (3c)



3c

Purified by silica gel column chromatography (*n*-hexane) to give compound **3c** as a colourless oil (26 mg, 76%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.23 (t, J = 8.0 Hz, 1H), 7.15 - 7.13 (m, 2H), 7.00 (d, J = 7.8 Hz, 1H), 6.36 - 6.34 (m, 1H), 2.59 - 2.55 (m, 2H), 2.52 - 2.49 (m, 2H), 2.36 (s, 3H), 1.83 - 1.76 (m, 2H), 1.72 - 1.65 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 147.1, 139.0, 137.7, 128.9, 128.2, 126.5, 125.1, 120.9, 36.1, 31.3, 27.4, 25.8, 21.7; **IR** (cm^{-1}): 2954 (s), 2952 (s), 2867 (w), 1603 (w), 1457 (m), 1377 (w), 1260 (m), 1091 (m), 1024 (s), 902 (w), 801 (s), 694 (m); **EI-MS: [M^+] m/z** 172.0.

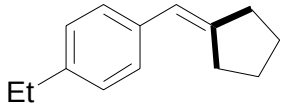
1-Cyclopentylidenemethyl-3,5-dimethylbenzene (3d)



3d

Purified by silica gel column chromatography (*n*-hexane) to give compound **3d** as a colourless oil (26.5 mg, 71%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 6.98 (s, 2H), 6.86 (s, 1H), 6.36 - 6.34 (m, 1H), 2.62 - 2.58 (m, 2H), 2.54 - 2.50 (m, 2H), 2.36 (s, 6H), 1.86 - 1.79 (m, 2H), 1.74 - 1.68 (m , 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 146.9, 139.0, 137.6, 127.5, 126.0, 121.0, 36.0, 31.3, 27.3, 25.8, 21.5; **IR** (cm^{-1}): 2954 (s), 2869 (w), 1700 (w), 1599 (m), 1451 (s), 1295 (w), 1037 (w), 851 (m), 702 (m); **EI-MS: [M^+] m/z** 186.3.

1-Cyclopentylidenemethyl-4-ethylbenzene (3e)

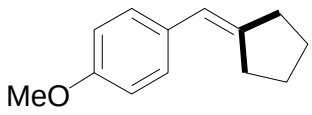


3e

Purified by silica gel column chromatography (*n*-hexane) to give compound **3e** as a colourless oil (25 mg, 67%); **^{1}H**

NMR ($CDCl_3$, 400 MHz) δ 7.26 (d, J = 8.2 Hz, 2H), 7.18 (d, J = 8.2 Hz, 2H), 6.37 - 6.36 (m, 1H), 2.66 (q, J = 7.8 Hz, 2H), 2.57 (t, J = 7.1 Hz, 2H), 2.50 (t, J = 7.8 Hz, 2H), 1.82 - 1.78 (m, 2H), 1.70 - 1.67 (m, 2H), 1.26 (t, J = 7.6 Hz, 3H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 146.3, 141.7, 136.5, 128.0, 127.8, 120.7, 36.0, 31.3, 28.7, 27.4, 25.8, 15.7; **IR** (cm^{-1}): 2959 (s), 2931 (m), 2870 (w), 1704 (w), 1607 (w), 1512 (m), 1453 (m), 1171 (w), 1017 (w), 871 (m), 826 (s); **EI-MS: [M^+] m/z** 186.4.

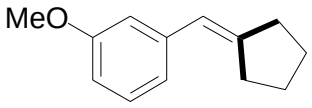
1-Cyclopentylidenemethyl-4-methoxybenzene (3f)[3]



3f

Purified by silica gel column chromatography (*n*-hexane/dichloromethane = 3/1) to give compound **3f** as a colourless oil (36.4 mg, 97%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.26 (d, J = 8.7 Hz, 2H), 6.88 (d, J = 8.7 Hz, 2H), 6.32 - 6.31 (m, 1H), 3.80 (s, 3H), 2.55 - 2.46 (m, 4H), 1.81 - 1.77 (m, 2H), 1.68 - 1.65 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 157.6, 144.9, 131.9, 129.1, 120.2, 113.7, 55.4, 35.9, 31.1, 27.4, 25.8; **IR** (cm^{-1}): 2953 (s), 2835 (w), 1607 (m), 1509 (m), 1462 (w), 1245 (s), 1176 (w), 1035 (s), 867 (w), 824 (m); **EI-MS: [M^+] m/z** 188.0.

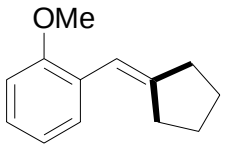
1-Cyclopentylidenemethyl-3-methoxybenzene (3g)



3g

Purified by silica gel column chromatography (*n*-hexane/dichloromethane = 3/1) to give compound **3g** as a colourless oil (25 mg, 66%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.22(t, J = 8.0 Hz, 1H), 6.90 (d, J = 7.8 Hz, 1H), 6.86 (s, 1H), 6.71(dd, J = 8.0 Hz, 1H), 6.33(m, 1H), 3.79 (s, 3H), 2.54 (t, J = 7.1 Hz, 2H), 2.48 (t, J = 7.0 Hz, 2H), 1.79 - 1.74 (m, 2H), 1.69 - 1.64 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 159.6, 147.7, 140.4, 129.2, 120.8, 120.8, 113.6, 111.3, 55.2, 36.1, 31.4, 27.3, 25.8; **IR** (cm^{-1}): 2953 (s), 2872 (w), 1597 (s), 1575 (s), 1489 (m), 1452 (m), 1429 (m), 1257 (s), 1160 (s), 1043 (s), 885 (w), 780 (m), 690 (s); **EI-MS: [M^+] m/z** 188.3.

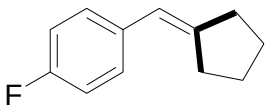
1-Cyclopentylidenemethyl-2-methoxybenzene (3h)



3h

Purified by silica gel column chromatography (*n*-hexane/dichloromethane = 3/1) to give compound **3h** as a colourless oil (30.0 mg, 80%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.38 (d, J = 7.8 Hz, 1H), 7.18 (t, J = 7.8 Hz, 1H), 6.95 (t, J = 7.3 Hz, 1H), 6.88 (d, J = 8.2 Hz, 1H), 6.61 (m, 1H), 3.85 (s, 3H), 2.56 - 2.50 (m, 4H), 1.78 - 1.68 (m, 4H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 156.5, 147.2, 128.8, 127.9, 127.1, 120.2, 115.0, 110.4, 55.5, 35.5, 31.2, 27.1, 25.7; **IR** (cm^{-1}): 2950 (s), 2868 (w), 1597 (w), 1486 (m), 1461 (m), 1434 (w), 1239 (s), 1104 (w), 1030 (m), 747 (s); **EI-MS: [M^+] m/z** 188.3.

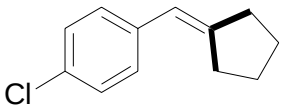
1-Cyclopentylidenemethyl-4-fluorobenzene (3i)[3]



3i

Purified by silica gel column chromatography (*n*-hexane) to give compound **3i** as a colourless oil (18 mg, 51%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.27 - 7.23 (m, 2H), 7.01 - 6.97 (m, 2H), 6.32 - 6.30 (m, 1H), 2.51 - 2.45 (m, 4H), 1.79 - 1.74 (m, 2H), 1.68 - 1.64 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 161.0 (d, $J_{C\text{-}F}$ = 244.4 Hz), 146.8, 135.1 (d, $J_{C\text{-}F}$ = 3.8 Hz), 129.4 (d, $J_{C\text{-}F}$ = 7.7 Hz), 119.8, 115.1 (d, $J_{C\text{-}F}$ = 21.1 Hz), 36.0, 31.2, 27.3, 25.8; **^{19}F NMR ($CDCl_3$, 564 MHz)** δ -116.9; **IR** (cm^{-1}): 2957 (s), 2869 (w), 1602 (m), 1506 (s), 1225 (s), 1158 (w), 868 (m), 822 (m); **EI-MS: [M^+] m/z** 176.3.

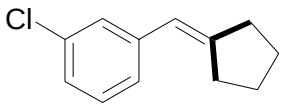
1-Chloro-4-(cyclopentylidenemethyl)benzene (3j)[3]



3j

Purified by silica gel column chromatography (*n*-hexane) to give compound **3j** as a waxy oil (28.3 mg, 74%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.30 - 7.22 (m, 4H), 6.33 - 6.31 (m, 1H), 2.54 - 2.48 (m, 4H), 1.82 - 1.77 (m, 2H), 1.71 - 1.67 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 148.1, 137.4, 131.2, 129.2, 128.4, 119.8, 36.1, 31.3, 27.3, 25.8; **IR** (cm^{-1}): 2956 (s), 2872 (w), 1709 (m), 1593 (w), 1490 (s), 1089 (s), 1013 (m), 820 (s); **EI-MS: [M^+] m/z** 192.2.

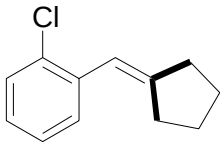
1-Chloro-3-(cyclopentylidenemethyl)benzene (3k)



3k

Purified by silica gel column chromatography (*n*-hexane) to give compound **3k** as a colourless oil (21.5 mg, 56%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.30 (s, 1H), 7.27 - 7.22 (m, 1H), 7.18 - 7.13 (m, 2H), 6.31 - 6.30 (m, 1H), 2.56 - 2.48 (m, 4H), 1.84 - 1.77 (m, 2H), 1.72 - 1.65 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 149.2, 140.8, 134.2, 129.5, 127.9, 126.2, 125.7, 119.8, 36.2, 31.4, 27.3, 25.7; **IR** (cm^{-1}): 2955 (s), 2868 (w), 1652 (w), 1591 (m), 1562 (m), 1474 (m), 1424 (w), 1260 (w), 1079 (m), 1019 (w), 883 (s), 780 (s), 684 (s); **EI-MS: [M^+] m/z** 192.3.

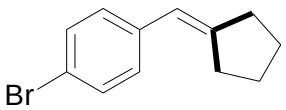
1-Chloro-2-(cyclopentylidenemethyl)benzene (3l)



3l

Purified by silica gel column chromatography (*n*-hexane) to give compound **3l** as a colourless oil (25 mg, 65%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.42 (dd, J = 7.8 Hz, 1H), 7.37 (dd, J = 7.8 Hz, 1H), 7.22 (td, J = 7.6 Hz, 1H), 7.11 (td, J = 7.7 Hz, 1H), 6.60 - 6.58 (m, 1H), 2.54 (t, J = 7.1 Hz, 2H), 2.47 (t, J = 7.1 Hz, 2H), 1.78 - 1.70 (m, 4H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 149.4, 136.9, 133.2, 129.4, 127.1, 126.4, 117.4, 35.4, 31.1, 27.0, 25.7; **IR** (cm^{-1}): 2954 (s), 2868 (w), 1650 (w), 1590 (w), 1467 (m), 1434 (m), 1051 (m), 1035 (m), 745 (s), 691 (m); **EI-MS: [M^+] m/z** 192.3.

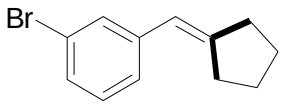
1-Bromo-4-(cyclopentylidenemethyl)benzene (3m)[3]



3m

Purified by silica gel column chromatography (*n*-hexane) to give compound **3m** as a waxy oil (25 mg, 53%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.42 (d, J = 8.7 Hz, 2H), 7.16 (d, J = 8.2 Hz, 2H), 6.30 - 6.29 (m, 1H), 2.52 - 2.46 (m, 4H), 1.81 - 1.76 (m, 2H), 1.71 - 1.66 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 148.4, 137.9, 131.3, 129.6, 119.9 119.3, 36.2, 31.4, 27.3, 25.7; **IR** (cm^{-1}): 2955 (s), 2869 (w), 1705 (m), 1589 (w), 1487 (s), 1071 (m), 1008 (m), 814 (s); **EI-MS: [M^+] m/z** 236.1, 238.1.

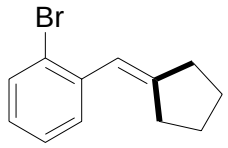
1-Bromo-3-(cyclopentylidenemethyl)benzene (3n)



3n

Purified by silica gel column chromatography (*n*-hexane) to give compound **3n** as a colourless oil (32 mg, 68%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.44 (s, 1H), 7.29 - 7.25 (m, 1H), 7.22 - 7.14 (m, 2H), 6.28 - 6.27 (m, 1H), 2.54 - 2.47 (m, 4H), 1.82 - 1.75 (m, 2H), 1.70 - 1.63 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 149.2, 141.1, 130.8, 129.8, 128.6, 126.6, 122.5, 119.7, 36.2, 31.4, 27.3, 25.7; **IR** (cm^{-1}): 2954 (s), 2867 (w), 1651 (w), 1587 (m), 1556 (m), 1471 (m), 1421 (m), 1072 (m), 879 (s), 777 (s), 683 (s); **EI-MS: [M^+] m/z** 236.3, 238.2.

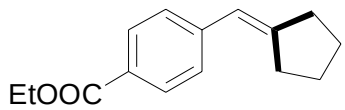
1-Bromo-2-(cyclopentylidenemethyl)benzene (3o)



3o

Purified by silica gel column chromatography (*n*-hexane) to give compound **3o** as a colourless oil (28.2 mg, 60%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.56 (dd, J = 7.8 Hz, 1H), 7.40 (dd, J = 7.8 Hz, 1H), 7.26 (t, J = 7.1 Hz, 1H), 7.03 (td, J = 7.7 Hz, 1H), 6.53 - 6.52 (m, 1H), 2.54 (t, J = 6.6 Hz, 2H), 2.46 - 2.43 (m, 2H), 1.79 - 1.70 (m, 4H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 149.2, 138.6, 132.7, 129.6, 127.37, 127.0, 124.0, 120.0, 35.2, 31.0, 27.0, 25.7; **IR** (cm^{-1}): 2985 (s), 2868 (w), 1649 (w), 1586 (w), 1462 (m), 1431 (m), 1023 (s), 743 (s), 666 (w); **EI-MS: [M^+] m/z** 236.1, 238.2.

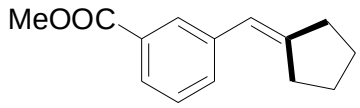
4-(Cyclopentylidenemethyl)benzoic acid ethyl ester (3p)



3p

Purified by silica gel column chromatography (*n*-hexane/dichloromethane = 3/1) to give compound **3p** as a waxy oil (20.5 mg, 45%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.98 (d, J = 8.2 Hz, 2H), 7.34 (d, J = 8.2 Hz, 2H), 6.40 - 6.39 (m, 1H), 4.36 (q, J = 7.2 Hz, 2H), 2.58 - 2.50 (m, 4H), 1.84 - 1.77 (m , 2H), 1.72 - 1.65 (m, 2H), 1.39 (t, J = 7.4 Hz, 3H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 166.8, 150.7, 143.5, 129.6, 127.8, 127.4, 120.5, 60.9, 36.4, 31.7, 27.3, 25.7, 14.5; **IR** (cm^{-1}): 2957 (s), 2871 (w), 1713 (s), 1605 (m), 1366 (w), 1270 (s), 1178 (w), 1101 (s), 1019 (m), 879 (w), 760 (m), 700 (w); **EI-MS: [M^+] m/z** 230.2.

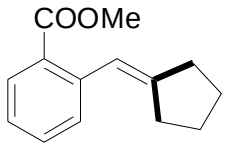
3-(Cyclopentylidenemethyl)benzoic acid methyl ester (3q)



3q

Purified by silica gel column chromatography (*n*-hexane/dichloromethane = 3/1) to give compound **3q** as a colourless oil (28 mg, 65%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.98 (s, 1H), 7.82 (d, J = 7.8 Hz, 1H), 7.47 (d, J = 7.8 Hz, 1H), 7.37 (t, J = 7.8 Hz, 1H), 6.39 - 6.38 (m, 1H), 3.91 (s, 3H), 2.57 (t, J = 7.1 Hz, 2H), 2.50 (t, J = 6.9 Hz, 2H), 1.83 - 1.76 (m, 2H), 1.71 - 1.64 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 167.4, 148.8, 139.2, 132.4, 130.1, 129.1, 128.3, 126.8, 120.0, 51.2, 36.1, 31.4, 27.3, 25.7; **IR** (cm^{-1}): 2953 (s), 2870 (w), 1721 (s), 1603 (w), 1436 (m), 1286 (s), 1202 (m), 1106 (w), 860 (w), 803 (w), 749 (s), 688 (m); **EI-MS: [M^+] m/z** 216.2.

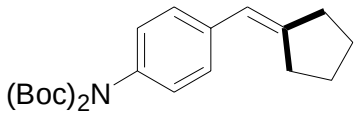
2-(Cyclopentylidenemethyl)benzoic acid methyl ester (3r)



3r

Purified by silica gel column chromatography (*n*-hexane/dichloromethane = 3/1) to give compound **3r** as a colourless oil (22.5 mg, 52%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.85 (d, J = 7.8 Hz, 1H), 7.44 - 7.42 (m, 2H), 7.24 - 7.20 (m, 1H), 6.89 - 6.88 (m , 1H), 3.88 (s, 3H), 2.53 - 2.50 (m, 2H), 2.42 - 2.39 (m, 2H), 1.73 - 1.66 (m, 4H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 168.3, 148.0, 140.2, 131.4, 130.2, 129.7, 128.87, 125.7, 119.6, 52.0, 35.1, 31.0, 27.0, 25.6; **IR** (cm^{-1}): 2951 (s), 2868 (w), 1720 (s), 1598 (w), 1567 (w), 1479 (w), 1432 (m), 1292 (m), 1248 (s), 1120 (m), 1076 (s), 737 (m); **EI-MS: [M^+] m/z** 216.3.

2-(4-Cyclopentylidenemethylphenyl)-1,3-bis(1,1-dimethylethyl)ester (3s)

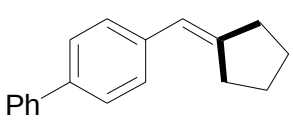


3s

Purified by silica gel column chromatography *n*-hexane to give compound **3s** as a waxy oil (33 mg, 44%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.25 (d, J = 7.79 Hz, 2H), 7.04 (d, J = 8.24 Hz, 2H), 6.33 (s, 1H), 2.54 - 2.45 (m, 4H), 1.80 - 1.73 (m, 2H), 1.68 - 1.61 (m, 2H), 1.39 (s, 18H) ; **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 152.1, 147.8, 138.0, 136.7, 128.2, 127.6, 120.3, 82.6, 36.2, 31.3, 28.0, 27.3, 25.8; **IR** (cm^{-1}): 2958 (s), 2931 (s), 1788 (m), 1749 (s), 1716 (s), 1511 (w), 1457 (w), 1367 (w), 1313 (w), 1273

(m), 1243 (m), 1151 (s), 1112 (s), 1004 (w), 911 (m), 853 (w), 731(s); **EI-MS: [M^+] m/z 373.1**.

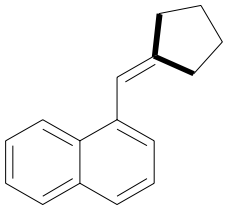
4-(Cyclopentylidenemethyl)biphenyl (3t)



3t

Purified by silica gel column chromatography (*n*-hexane) to give compound **3t** as a waxy oil (24.2 mg, 52%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.63 (d, J = 7.8 Hz, 2H), 7.59 (d, J = 8.2 Hz, 2H), 7.46 (t, J = 7.5 Hz, 2H), 7.41 (d, J = 8.2 Hz, 2H), 7.35 (t, J = 7.3 Hz, 1H), 6.43 - 6.42 (m,1H), 2.64 - 2.60 (m, 2H), 2.56 - 2.52 (m, 2H), 1.87 - 1.80 (m, 2H), 1.74 - 1.68 (m, 2H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 147.7, 141.1, 138.4, 138.1, 128.9, 128.4, 127.2, 127.0, 126.9, 120.5, 36.2, 31.5, 27.4, 25.8; **IR** (cm^{-1}): 2954 (m), 2926 (s), 2855 (w), 1601 (w), 1485 (m), 1461 (w), 1408 (w), 1027 (m), 758 (s), 693 (s); **EI-MS: [M^+] m/z** 234.2.

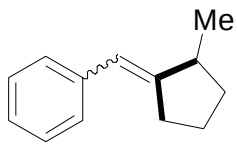
1-(Cyclopentylidenemethyl)naphthalene (3u)



3u

Purified by silica gel column chromatography (*n*-hexane) to give compound **3u** as a waxy oil (27.5 mg, 66%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 8.13 - 8.10 (m, 1H), 7.89 - 7.87 (m, 1H), 7.76 - 7.74 (m, 1H), 7.54 - 7.47 (m, 4H), 6.94 - 6.93 (m, 1H), 2.64 (t, J = 6.9 Hz, 2H), 2.44 (t, J = 7.3 Hz, 2H), 1.80 - 1.73 (m, 4H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 148.9, 136.2, 133.7, 131.8, 128.5, 126.6, 125.8, 125.7, 125.6, 125.5, 124.8, 117.9, 34.9, 31.0, 26.9, 25.9; **IR** (cm^{-1}): 2952 (s), 2866 (w), 1589 (w), 1507 (w), 1452 (w), 1395 (w), 1014 (m), 797 (m), 777 (s); **EI-MS: [M^+] m/z 208.0.**

(2-Methylcyclopentylidenemethyl)benzene (3v)

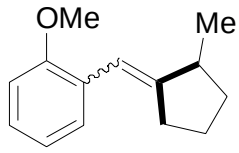


3v

Purified by silica gel column chromatography (*n*-hexane) to give compound **3v** as a colourless oil (29 mg, 84%, Z / E = 2 : 3); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.40 - 7.33 (m, 6.67H), 7.22 - 7.19 (m, 1.62H), 6.36 - 6.35 (m, 1H), 6.30 - 6.29 (m, 0.65H), 3.21 - 3.20 (m, 1H), 2.68 - 2.61 (m, 3H), 2.50 - 2.48 (m, 1H), 1.98 - 1.67 (m, 5H), 1.58 - 1.56 (m, 1H), 1.33 - 1.29 (m, 1H), 1.24 (d, J = 6.9 Hz, 2H), 1.10 (d, J = 6.9Hz, 3H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 152.13, 151.80, 139.05, 138.57, 128.33, 128.28, 128.20, 128.15, 125.82, 125.75, 120.90, 120.23, 41.06, 35.56, 35.43, 35.41, 34.72, 31.69, 24.85, 22.82, 19.51, 19.22; **IR** (cm^{-1}): 2956 (s), 2869 (w), 1713 (m), 1598 (w), 1494 (w), 1448 (m), 1376 (w), 1075 (m), 1029 (w), 751 (w), 695 (s); **EI-MS: [M^+] m/z 172.3.**

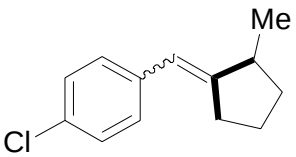
1-Methoxy-2-(2-methylcyclopentylidenemethyl)benzene (3w)



3w

Purified by silica gel column chromatography (*n*-hexane/dichloromethane = 3/1) to give compound **3w** as a colourless oil (27 mg, 67%, Z / E = 2 : 3); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.43 - 7.37 (m, 1.63H), 7.22 - 7.16 (m, 1.38H), 6.97 - 6.92 (m, 1.48H), 6.89 - 6.86 (m, 1.45H), 6.53 - 6.50 (m, 1.26H), 3.86 (s, 2H), 3.85 (s, 3H), 3.11 - 3.06 (m, 1H), 2.66 - 2.56 (m, 3H), 2.53 - 2.46 (m, 1H), 1.97 - 1.88 (m, 1.63H), 1.85 - 1.73 (m, 1.73H), 1.68 - 1.61 (m, 1.65H), 1.51 - 1.45 (m, 1H), 1.32 - 1.24 (m, 0.67H), 1.23 (d, J = 6.9 Hz, 2H), 0.98 (d, J = 6.9 Hz, 3H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 156.65, 156.60, 152.01, 151.74, 128.88, 128.00, 127.71, 127.26, 127.09, 120.35, 120.21, 115.31, 114.35, 110.44, 110.39, 55.57, 40.73, 35.61, 35.43, 35.34, 34.80, 31.48, 24.88, 23.14, 19.85, 19.33; **IR** (cm^{-1}): 2955 (s), 2869 (w), 1715 (w), 1598 (w), 1488 (m), 1462 (m), 1437 (w), 1241 (s), 1107 (w), 1049 (w), 1028 (m), 750 (s); **EI-MS: [M^+] m/z 202.3.**

1-Chloro-4-(2-methylcyclopentylidenemethyl)benzene (3x)

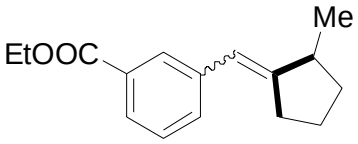


3x

Purified by silica gel column chromatography (*n*-hexane) to give compound **3x** as a waxy oil (23 mg, 56%, Z / E = 2 : 3); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.30 - 7.22 (m, 6.66H), 6.23 (m, 1H), 6.18 - 6.16 (m, 0.65H), 3.10 - 3.07 (m, 1H), 2.60 - 2.50 (m, 3H), 2.45 - 2.36 (m, 1H), 1.94 - 1.83 (m, 2H), 1.72 - 1.61 (m, 3H), 1.54 - 1.48 (m, 1H), 1.28 - 1.20 (m, 1H), 1.17 (d, J = 6.9 Hz, 2H), 1.01 (d, J = 6.9 Hz, 3H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 153.03, 152.67, 137.51, 137.01, 131.30, 131.24, 129.38, 128.44, 128.38, 119.75, 119.13, 41.14, 35.54, 35.52, 35.42, 34.67, 31.70, 24.81, 22.82, 19.40,

19.06; **IR** (cm^{-1}): 2957 (s), 2870 (w), 1709 (w), 1594 (w), 1490 (s), 1458 (w), 1407 (w), 1376 (w), 1090 (s), 1013 (m), 818 (s); **EI-MS: [M^+] m/z 206.2.**

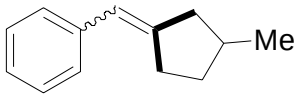
3-(2-Methylcyclopentylidenemethyl)benzoic acid ethyl ester (3y)



3y

Purified by silica gel column chromatography (*n*-hexane/dichloromethane = 3/1) to give compound **3y** as a colourless oil (32 mg, 66%, Z / E = 2 : 3); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 8.01 (m, 1.58H), 7.85 - 7.83 (m, 1.57H), 7.50 - 7.48 (m, 1.60H), 7.39 - 7.34 (m, 1.68H), 6.33 - 6.32 (m, 1H), 6.28 - 6.26 (m, 0.60H), 4.40 - 4.35 (m, 3.34H), 3.17 - 3.14 (m, 1H), 2.67 - 2.55 (m, 3H), 2.48 - 2.39 (m, 1H), 1.96 - 1.84 (m, 2H), 1.79 - 1.72 (m, 1H), 1.68 - 1.61 (m, 1.60H), 1.55 - 1.50 (m, 1H), 1.40 (t, *J* = 7.1 Hz, 5H), 1.30 - 1.22 (m, 1H), 1.19 (d, *J* = 6.9 Hz, 2H), 1.04 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 166.90, 153.52, 153.22, 139.25, 138.76, 132.32, 130.51, 130.46, 129.26, 128.30, 128.25, 126.83, 126.74, 120.03, 119.42, 60.99, 41.12, 35.53, 35.46, 35.42, 34.64, 31.65, 24.78, 22.79, 19.38, 19.09, 14.44; **IR** (cm^{-1}): 2956 (s), 2869 (w), 1718 (s), 1600 (w), 1444 (w), 1367 (w), 1263 (s), 1200 (s), 1105 (w), 1024 (w), 916 (w), 864 (w), 749 (m), 688 (w); **EI-MS: [M^+] m/z 244.2.**

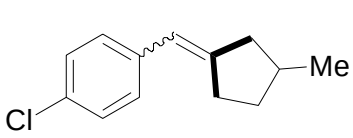
3-Methylcyclopentylidenemethylbenzene (3z)



3z

Purified by silica gel column chromatography (*n*-hexane) to give compound **3z** as a colourless oil (24.4 mg, 71%, Z / E = 1 : 1); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.36 - 7.31 (m, 8H), 7.20 - 7.16 (m, 2H), 6.36 - 6.33 (m, 2H), 2.83 - 2.80 (m, 1H), 2.70 - 2.51 (m, 5H), 2.18 - 1.97 (m, 5H), 1.90 - 1.85 (m, 1H), 1.41 - 1.36 (m, 1H), 1.33 - 1.25 (m, 1H), 1.09 (d, *J* = 6.4 Hz, 3H), 1.06 (d, *J* = 6.0 Hz, 3H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 147.23, 139.00, 138.92, 128.30, 128.28, 128.05, 125.70, 121.11, 121.07, 44.66, 40.16, 35.68, 35.45, 35.36, 33.94, 33.78, 31.05, 20.28, 19.76; **IR** (cm^{-1}): 2951 (s), 2925 (m), 2868 (w), 1600 (w), 1489 (w), 1447 (w), 1429 (w), 1376 (w), 1260 (m), 1071 (w), 1029 (m), 803 (w), 740 (m), 692 (s); **EI-MS: [M^+] m/z 172.0.**

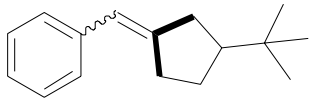
1-Chloro-4-(3-methylcyclopentylidenemethyl)benzene (3aa)



3aa

Purified by silica gel column chromatography (*n*-hexane) to give compound **3aa** as a colourless oil (25.5 mg, 62%, Z / E = 1 : 1); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.27 - 7.19 (m, 8H), 6.26 - 6.25 (m, 2H), 2.76 - 2.46 (m, 6H), 2.13 - 1.1.96 (m, 5H), 1.88 - 1.82 (m, 1H), 1.38 - 1.22 (m, 2H), 1.06 (d, J = 6.4 Hz, 3H), 1.03 (d, J = 6.4 Hz, 3H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 148.07, 137.44, 137.35, 131.18, 129.23, 128.38, 120.04, 119.98, 44.67, 40.15, 35.68, 35.49, 35.28, 33.88, 33.77, 31.07, 20.24, 19.72; **IR** (cm^{-1}): 2951 (s), 2868 (w), 1653 (w), 1450 (s), 1455 (w), 1406 (w), 1376 (w), 1090 (s), 1011 (m), 872 (m), 859 (m), 812 (m); **EI-MS: [M^+] m/z 206.2.**

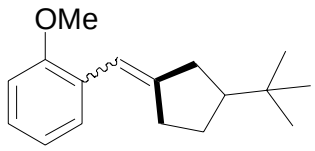
3-*tert*-Butyl-cyclopentylidenemethylbenzene (3ab)



3ab

Purified by silica gel column chromatography (*n*-hexane) to give compound **3ab** as a colourless oil (24 mg, 56%, Z / E = 1 : 1); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.36 - 7.33 (m, 8H), 7.22 - 7.17 (m, 2H), 6.36 - 6.34 (m, 2H), 2.78 - 2.64 (m, 2H), 2.59 - 2.48 (m, 4H), 2.36 - 2.22 (m, 2H), 1.90 - 1.83 (m, 2H), 1.82 - 1.75 (m, 2H), 1.54 - 1.30 (m, 2H), 0.96 (s, 9H), 0.95 (s, 9H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 147.22, 147.08, 138.99, 138.94, 128.33, 128.29, 128.06, 125.67, 121.06, 120.94, 52.45, 50.48, 37.47, 35.96, 32.95, 31.91, 31.68, 31.49, 27.87, 26.68; **IR** (cm^{-1}): 2956 (s), 2867 (w), 1491 (m), 1475 (m), 1394 (w), 1364 (m), 1029 (w), 909 (w), 856 (w), 753 (m), 692 (s); **EI-MS: [M^+] m/z 214.4.**

1-(3-*tert*-Butyl-cyclopentylidenemethyl)-2-methoxybenzene (3ac)

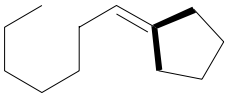


3ac

Purified by silica gel column chromatography (*n*-hexane/dichloromethane = 3/1)to give compound **3ac** as a colourless oil (32 mg, 66%, Z / E = 1 : 1); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.42 - 7.37 (m, 2H), 7.21 - 7.18 (m, 2H), 7.01 - 6.96 (m, 2H), 6.91 - 6.88 (m, 2H), 6.60 - 6.58 (m, 2H), 3.87 (s, 6H), 2.72 - 2.49 (m, 6H), 2.39 - 2.20 (m, 2H), 1.86 - 1.80 (m, 4H), 1.47 - 1.39 (m, 2H), 0.95 (s,

9H), 0.94 (s, 9H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 156.49, 156.45, 147.05, 146.93, 128.80, 127.88, 127.78, 127.04, 120.26, 120.20, 115.09, 114.96, 110.34, 110.31, 55.49, 52.15, 50.53,36.97, 35.38, 32.75, 31.86, 31.71, 31.31, 27.86, 27.83, 26.62; **IR** (cm^{-1}): 2953 (s), 2867 (w), 1596 (w), 1468 (m), 1461 (m), 1434 (w), 1393 (w), 1364 (m), 1292 (w), 1239 (s), 1105 (w), 1031 (m), 747 (s); **EI-MS: [M^+] m/z 244.4.**

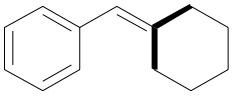
Heptylidene-cyclopentane (5)



5

Purified by silica gel column chromatography *n*-hexane to give compound **5** as a colourless oil (6 mg, 18%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 5.24 - 5.23 (m, 1H), 2.23 - 2.15 (m, 4H), 1.98 - 1.92 (m, 2H), 1.67 - 1.57 (m, 4H), 1.34 - 1.28 (m, 8H), 0.88 (t, J = 6.87 Hz, 3H) ; **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 143.1, 120.5, 33.7, 32.0, 29.9, 29.8, 29.2, 28.7, 26.6, 26.5, 22.8, 14.3; **IR** (cm^{-1}): 2923 (s), 2854 (w), 1460 (m), 1377 (w), 1081 (w), 1026 (m), 802 (m); **EI-MS: [M^+] m/z** 166.1.

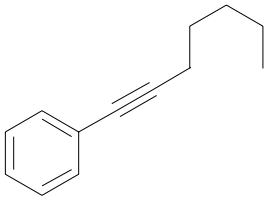
(Cyclohexylidenemethyl)benzene (7)



7

Purified by silica gel column chromatography *n*-hexane to give compound **7** as a colourless oil (10.5 mg, 31%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.29 (t, J = 7.56 Hz, 2H), 7.20 - 7.14 (m, 3H), 6.22 (s, 1H), 2.36 (t, J = 5.95 Hz, 2H), 2.25 (t, J = 5.5 Hz, 2H), 1.66 - 1.52 (m, 6H) ; **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 143.5, 138.5, 129.0, 128.1, 125.9, 122.1, 37.8, 29.6, 28.8, 28.0, 26.8; **IR** (cm^{-1}): 2925 (s), 2853 (w), 1653 (w), 1598 (w), 1489 (w), 1445 (m), 1342 (w), 1238 (w), 1073 (w), 1029 (w), 917 (w), 840 (m), 735 (s), 697 (s); **EI-MS: [M^+] m/z** 172.1.

Hept-1-ynyl-benzene (8)



8

Purified by silica gel column chromatography *n*-hexane to give compound **8** as a colourless oil (4.5 mg, 13%); **^{1}H NMR ($CDCl_3$, 400 MHz)** δ 7.41 - 7.38 (m, 2H), 7.29 - 7.26 (m, 3H),

2.40 (t, J = 7.33 Hz, 2H), 1.65 - 1.55 (m, 2H), 1.47 - 1.33 (m, 4H), 0.92 (t, J = 7.33 Hz, 3H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 131.7, 128.3, 127.6, 124.2, 90.6, 80.6, 31.3, 28.6, 22.4, 19.5, 14.2 ; **IR** (cm^{-1}): 2955 (m), 2925 (s), 2856 (m), 1490 (w), 1460 (m), 1377 (w), 1250 (w), 1116 (s), 754 (s), 691 (s); **EI-MS: [M^+] m/z** 172.2.

Benzene sulfinic acid

^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.61 (br s, 1H), 7.69 – 7.68 (m, 2H), 7.53 – 7.50 (m, 3H); **^{13}C NMR ($CDCl_3$, 100 MHz)** δ 146.0, 132.2, 129.2, 125.1

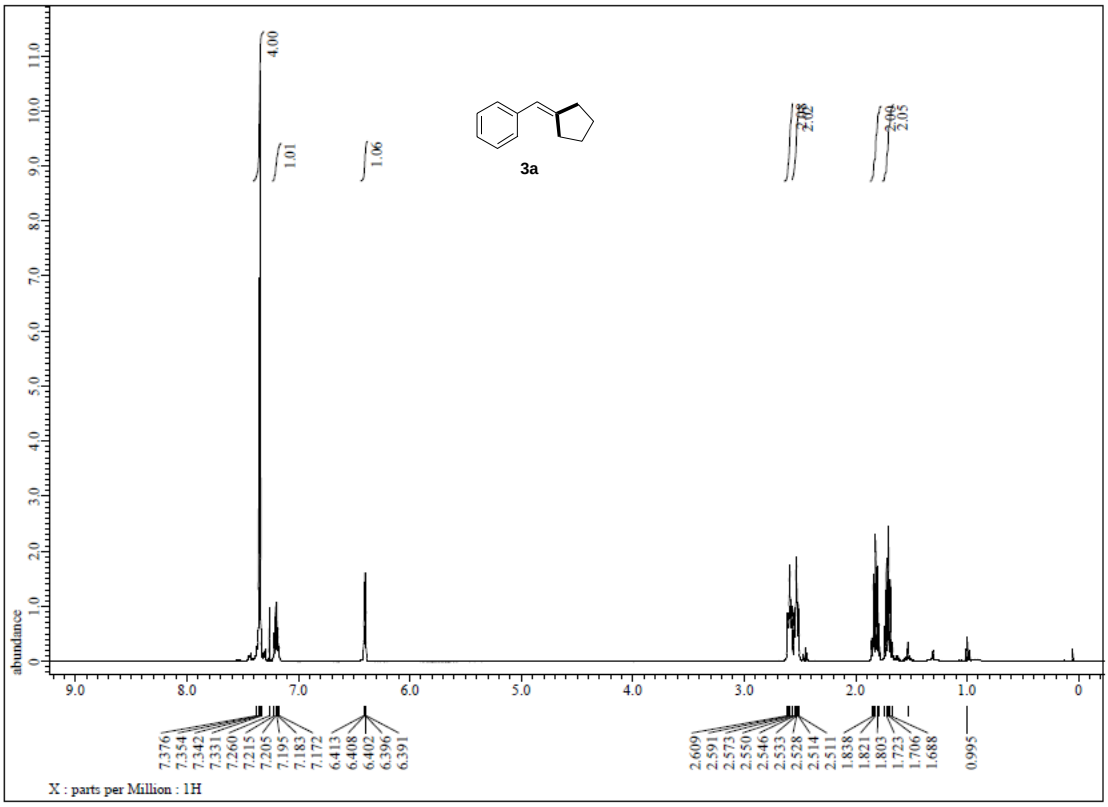
References

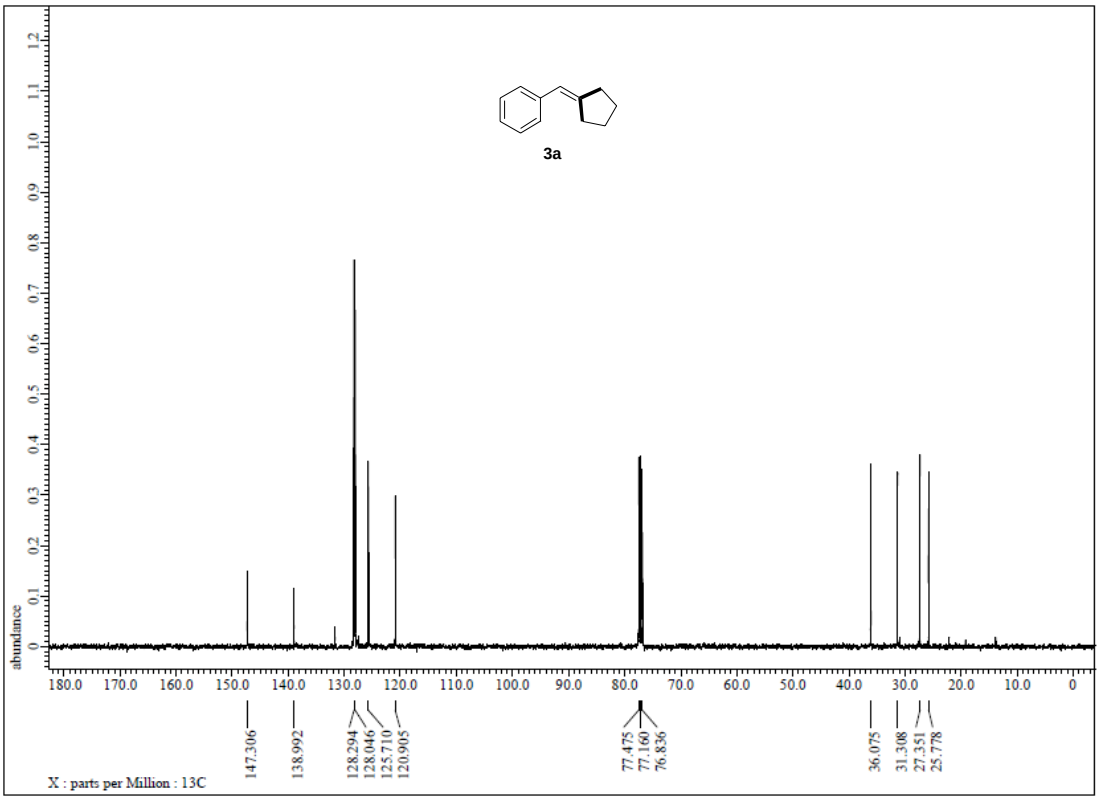
(1) M. Sheikh, S. Takagi and M. Sakai, *Org. Biomol. Chem.,* 2011, **9**, 1244.

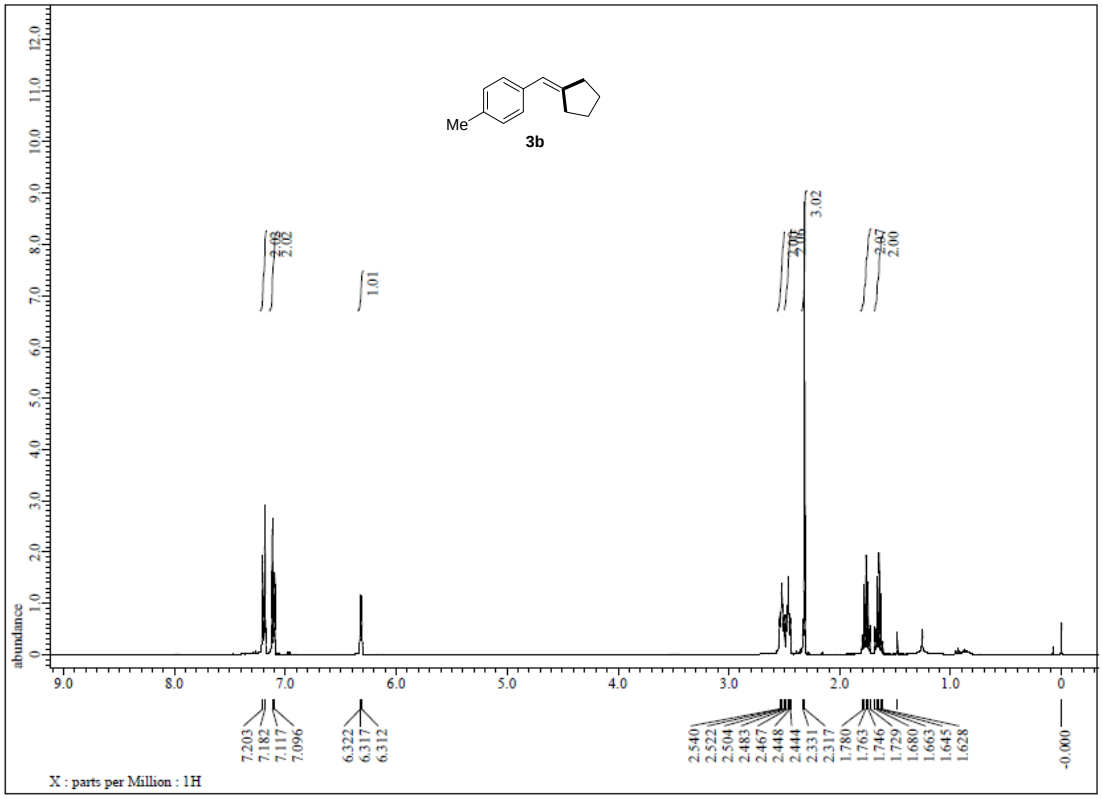
(2) V. Nair, A. Augustine and T. D. Suja, *Synthesis,* 2014, 2259.

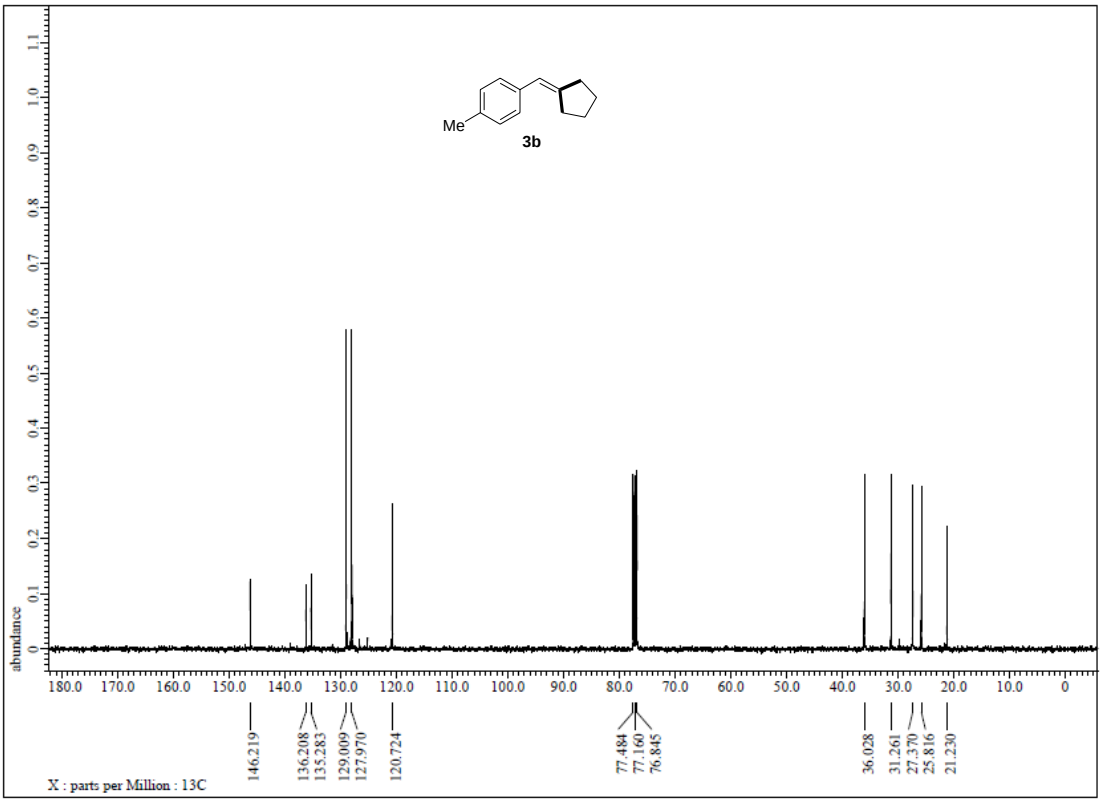
(3) W. Huang, S.-H. Zhao and N. Xu, *Synthesis,* 2015, 359.

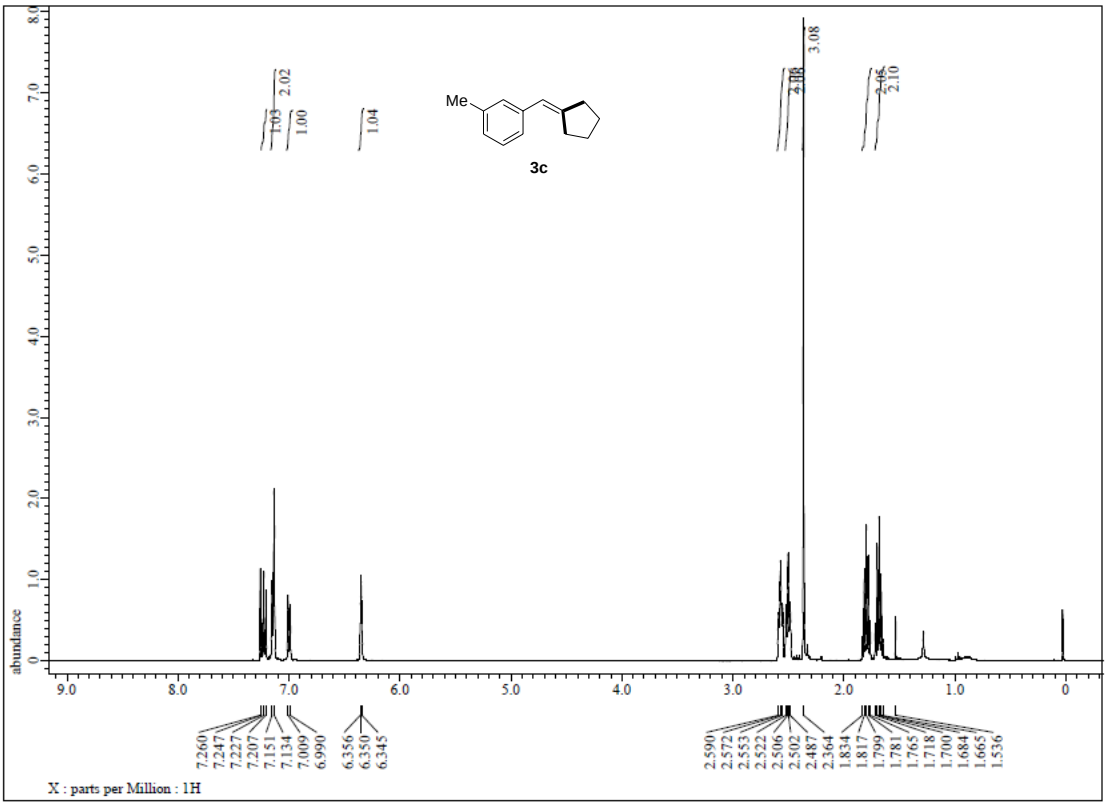
The ^{1}H and ^{13}C NMR spectra of compounds 3a-ac, 5, 7 and 8

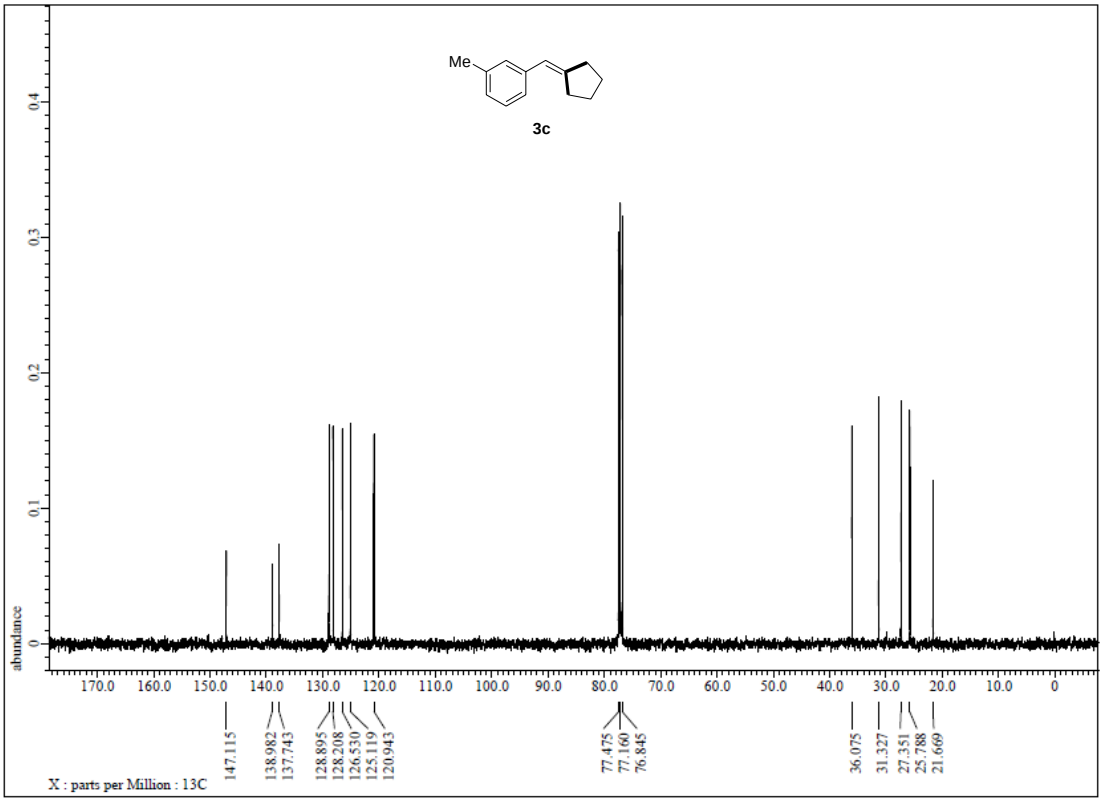


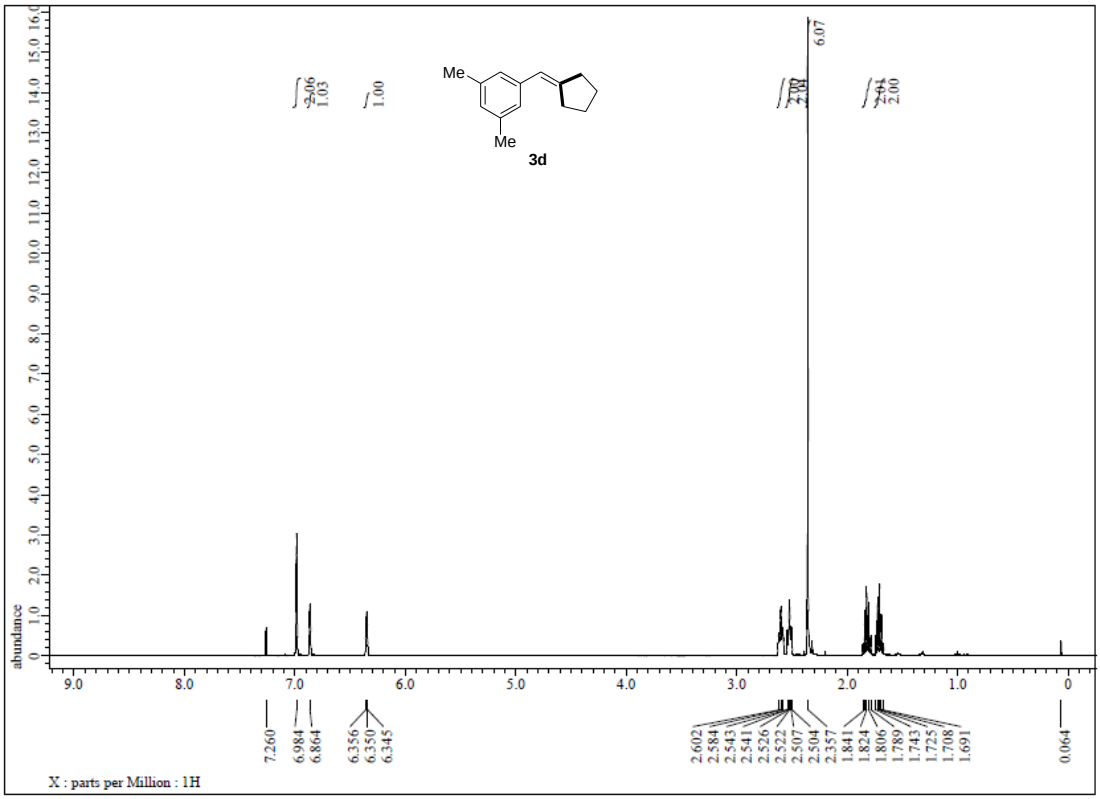


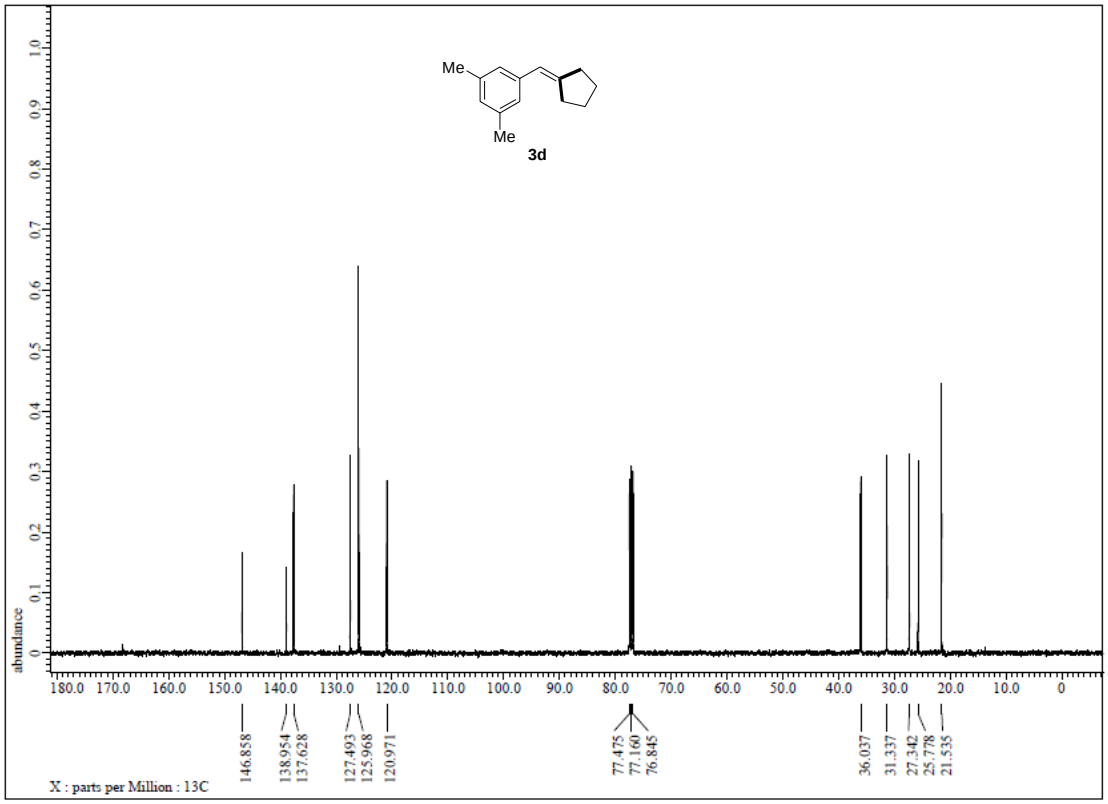

Me
3b
abundance
X : parts per Million : 1H

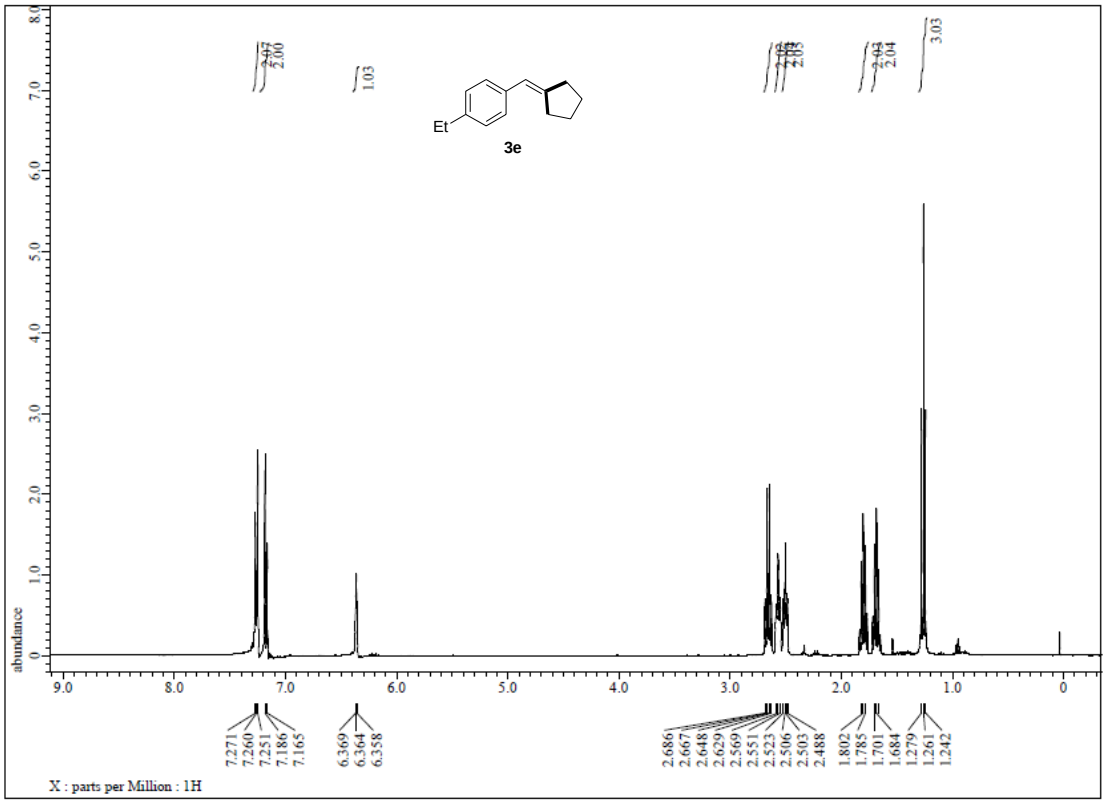

Me
3b
abundance
X : parts per Million : 13C

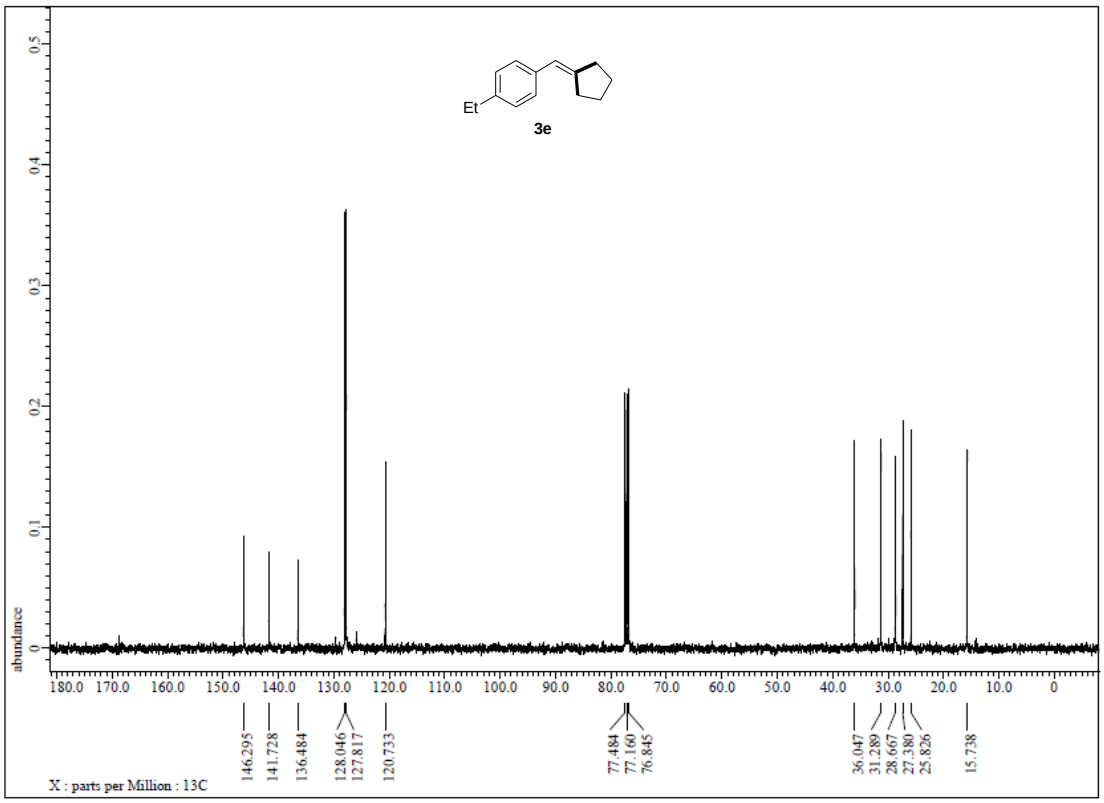

Me
3c
abundance
X : parts per Million : 1H

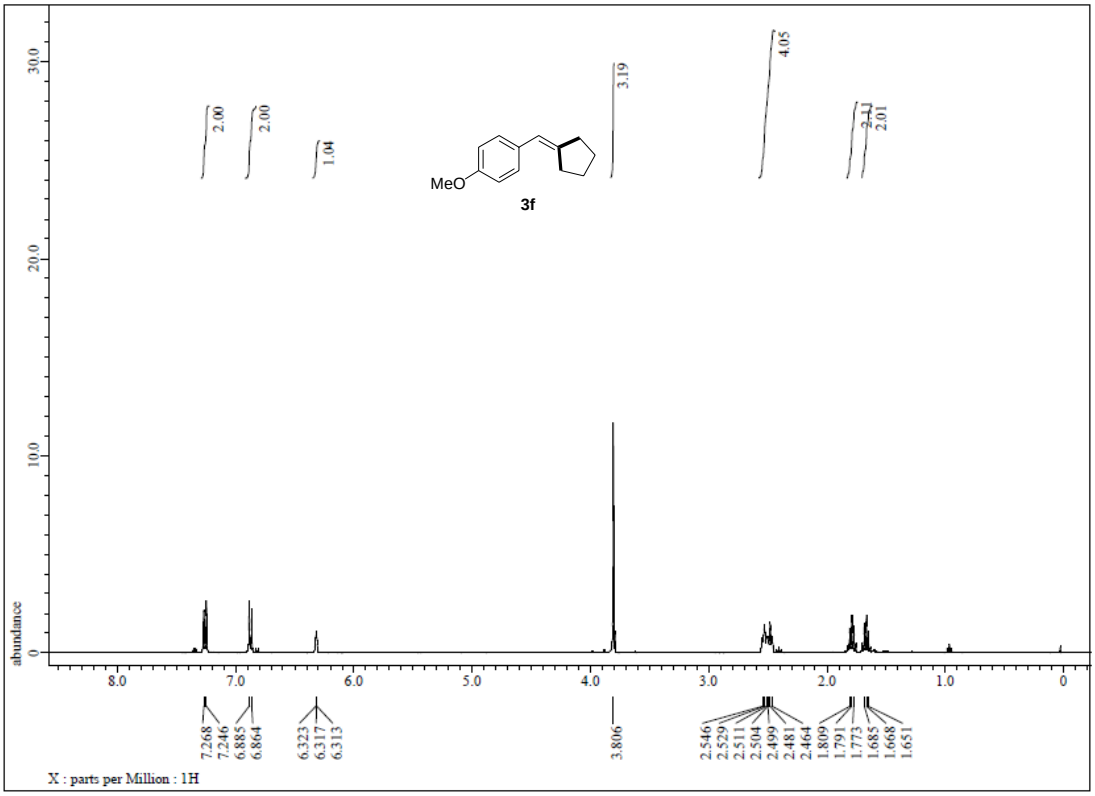

Me
3c
abundance
X : parts per Million : 13C

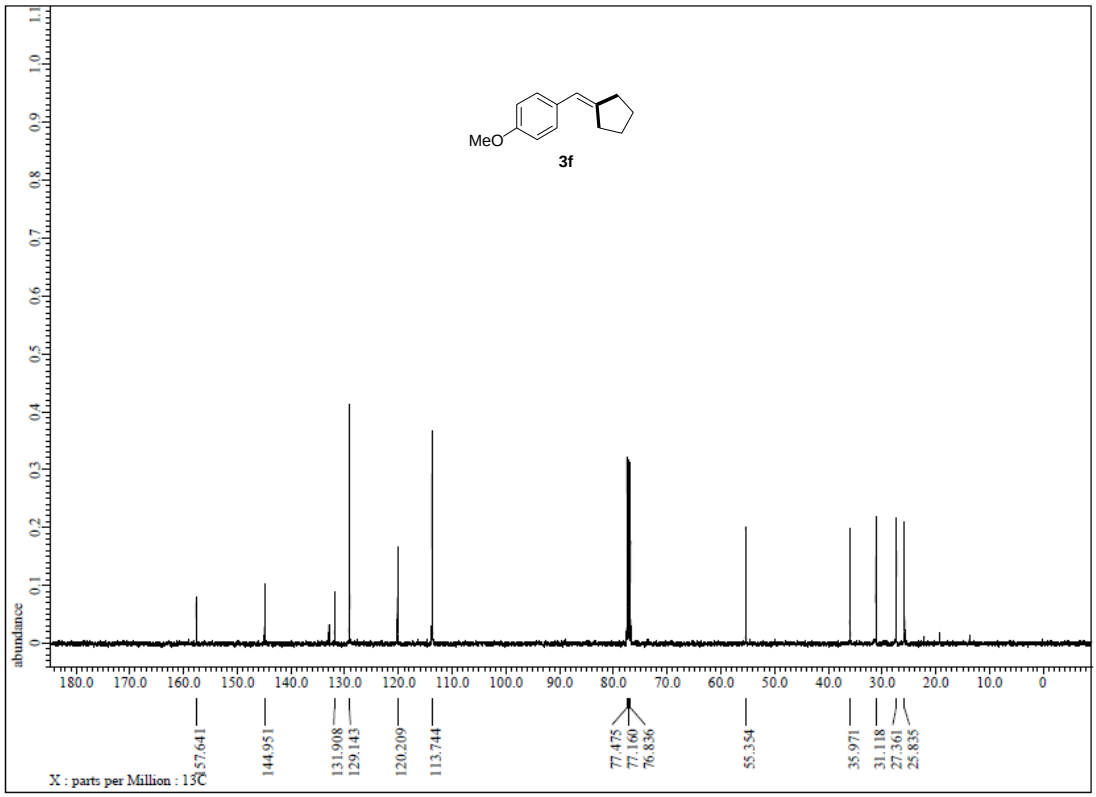

Me
Me
3d
abundance
X : parts per Million : 1H

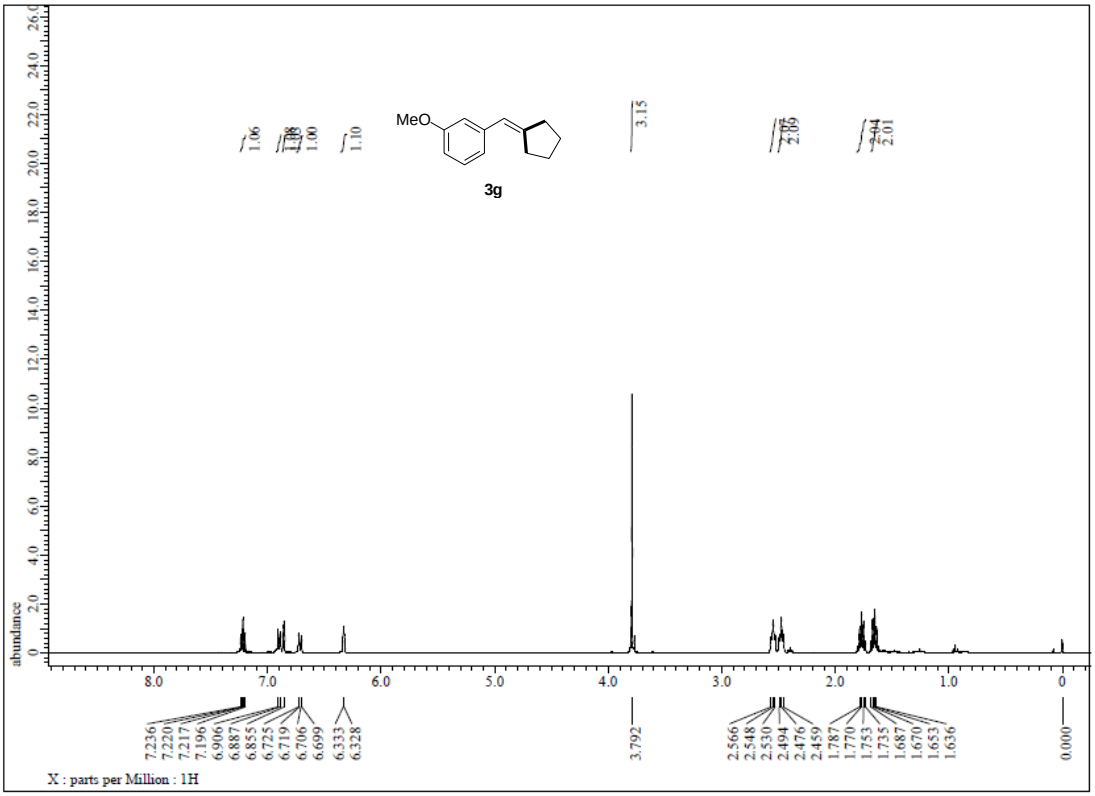

Me
Me
3d
abundance
X : parts per Million : 13C

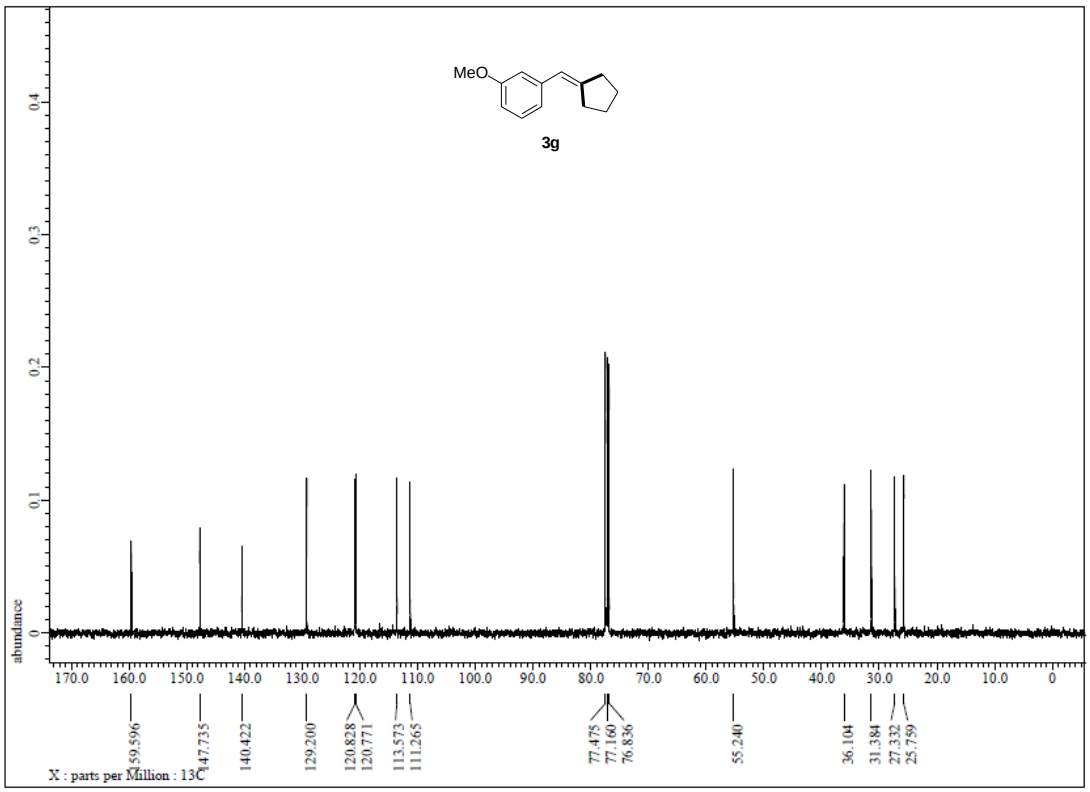

Et
3e
abundance
X : parts per Million : 1H

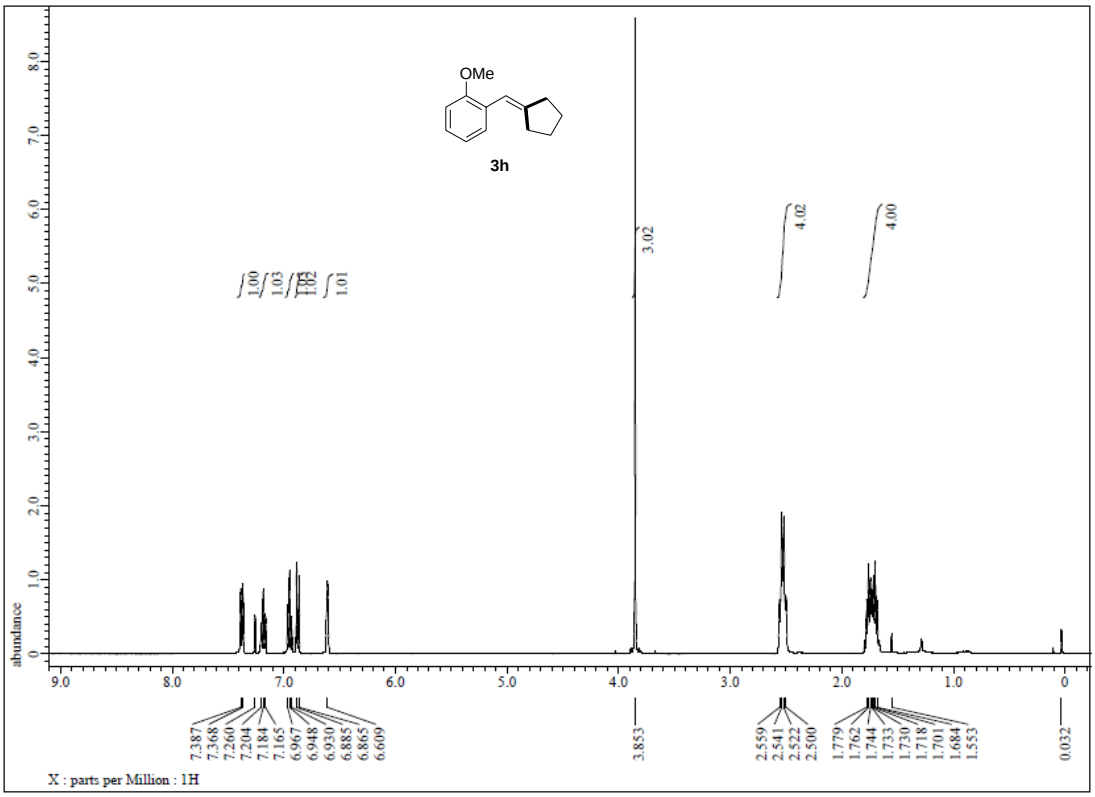

Et
3e
abundance
X : parts per Million : 13C

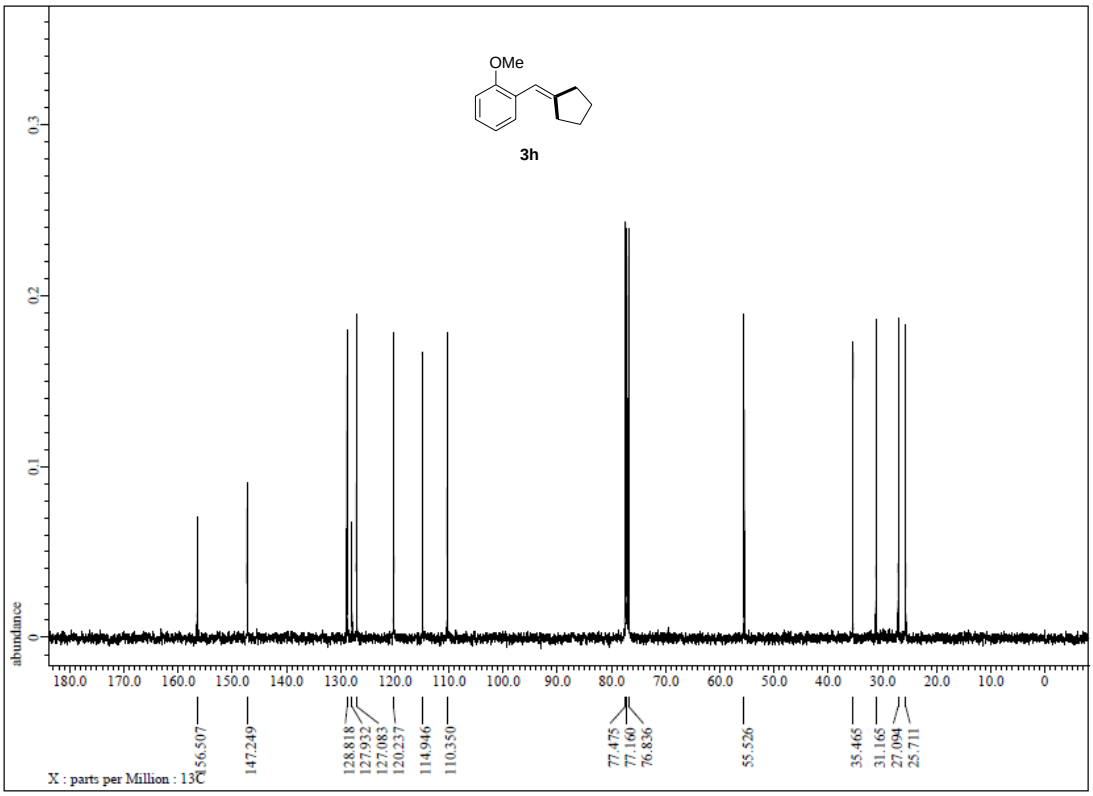

MeO
3f
2.00
2.00
1.04
3.19
4.05
2.11
2.01
abundance
X : parts per Million : 1H
7.268
7.246
6.885
6.864
6.323
6.317
6.313
3.806
2.546
2.529
2.511
2.504
2.499
2.481
2.464
1.809
1.791
1.773
1.685
1.668
1.651

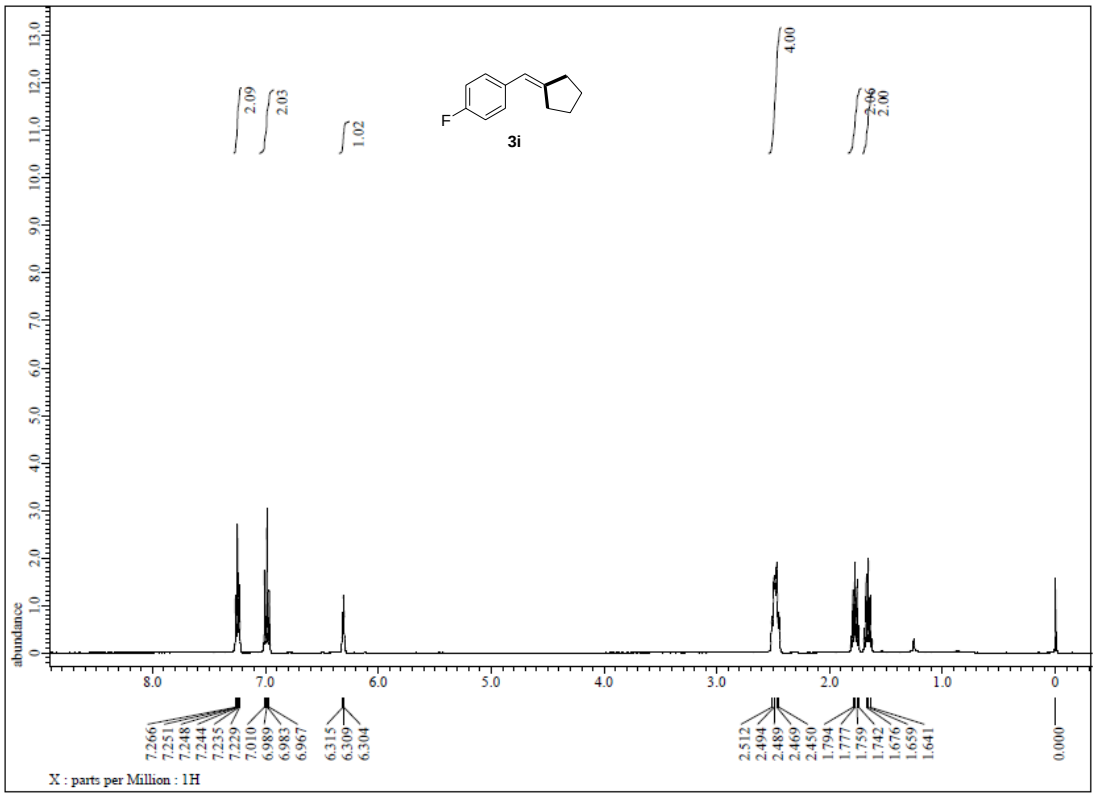

MeO
3f
abundance
X : parts per Million : 13C
157.641
144.951
131.908
129.143
120.209
113.744
77.475
77.160
76.836
55.354
35.971
31.118
27.361
25.835

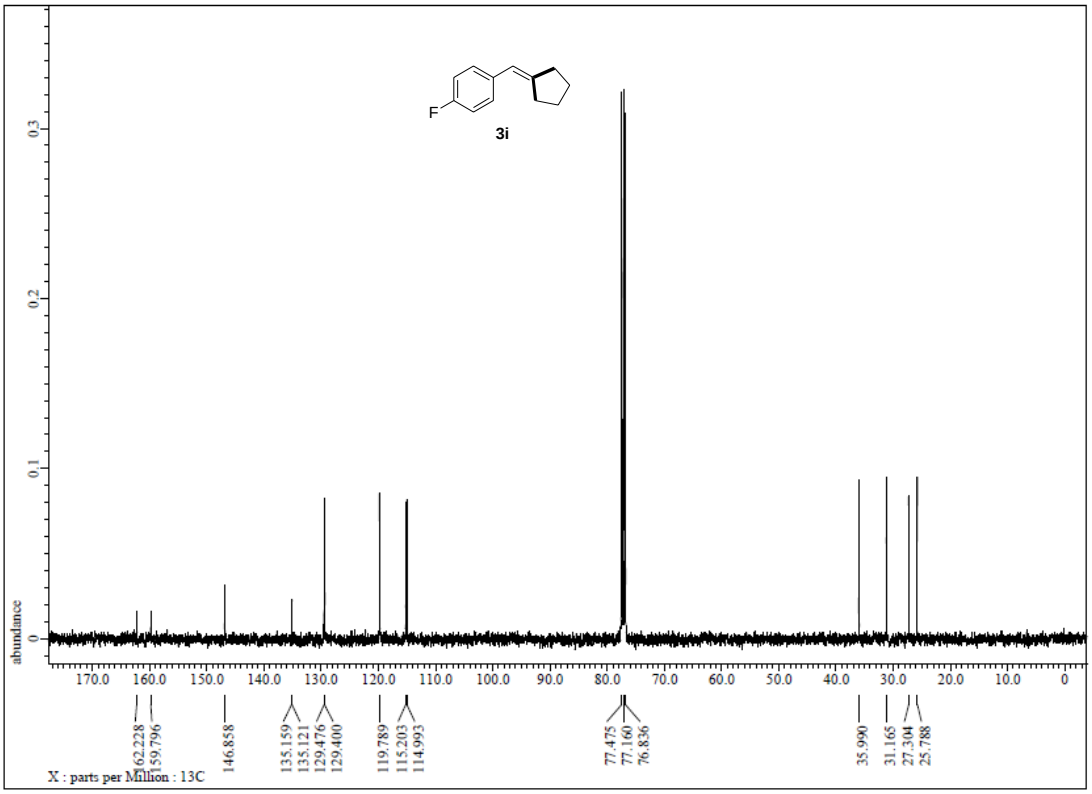

MeO
3g
abundance
X : parts per Million : 1H

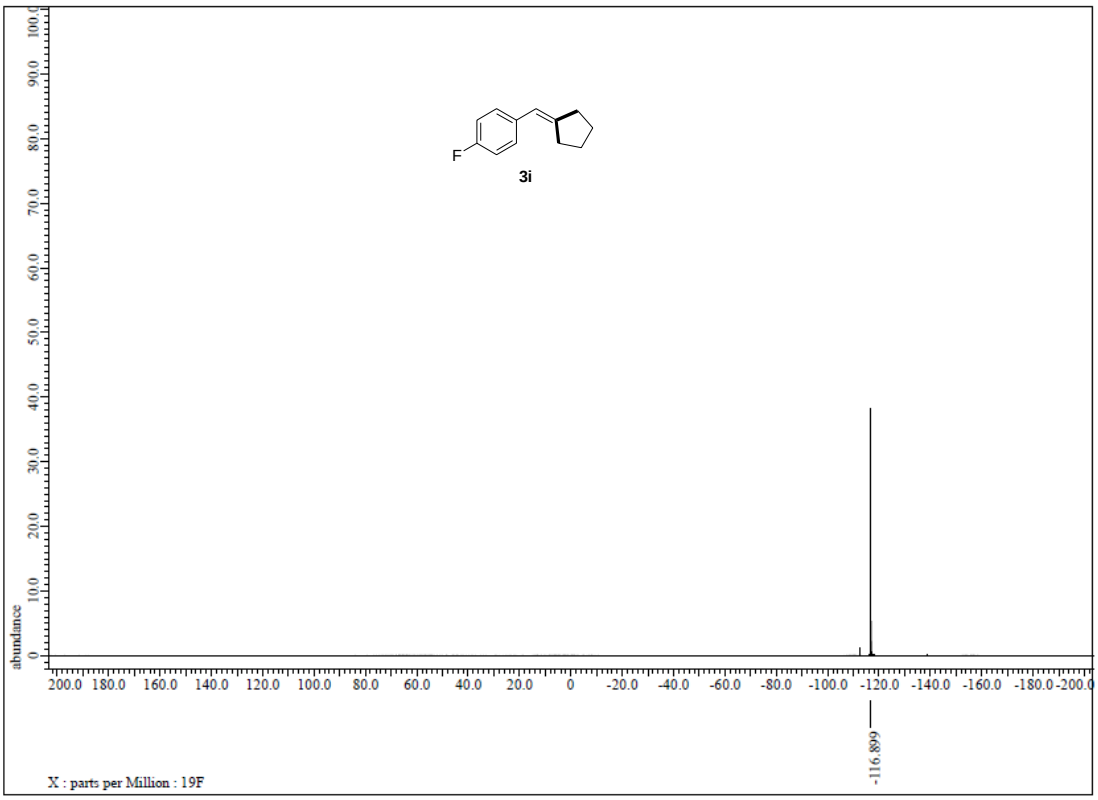

MeO
3g
abundance
X : parts per Million : 13C

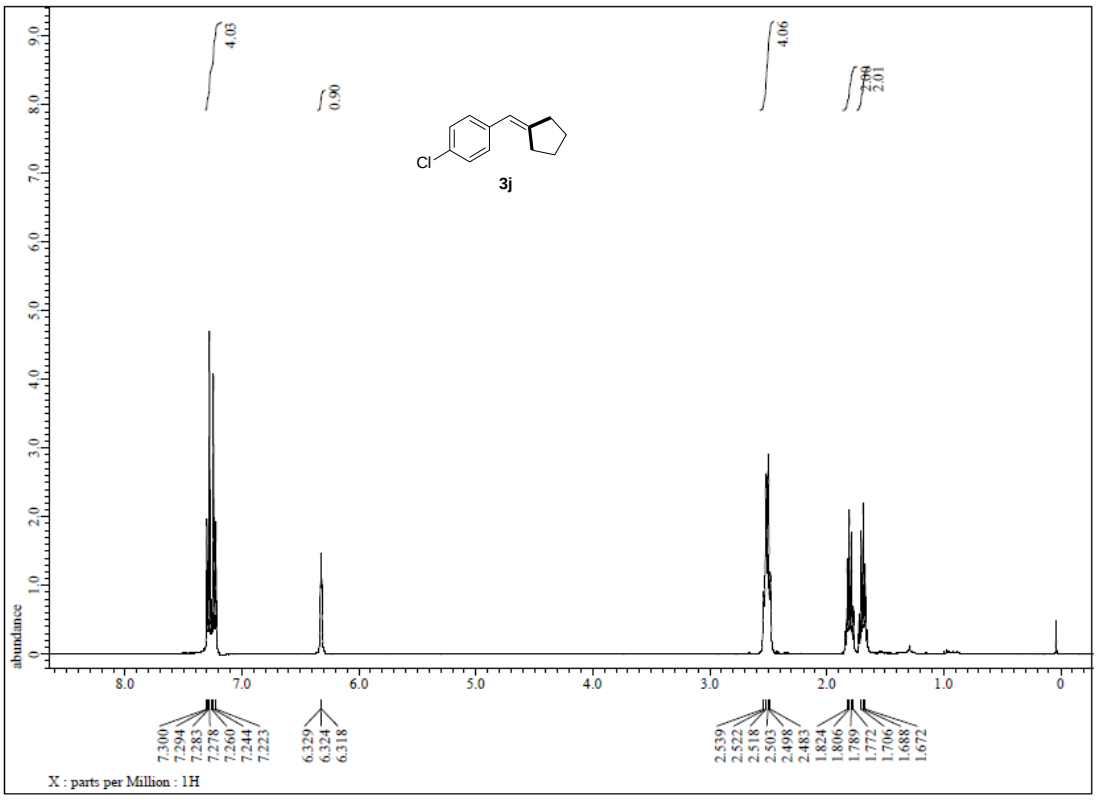

OMe
3h
abundance
X : parts per Million : 1H

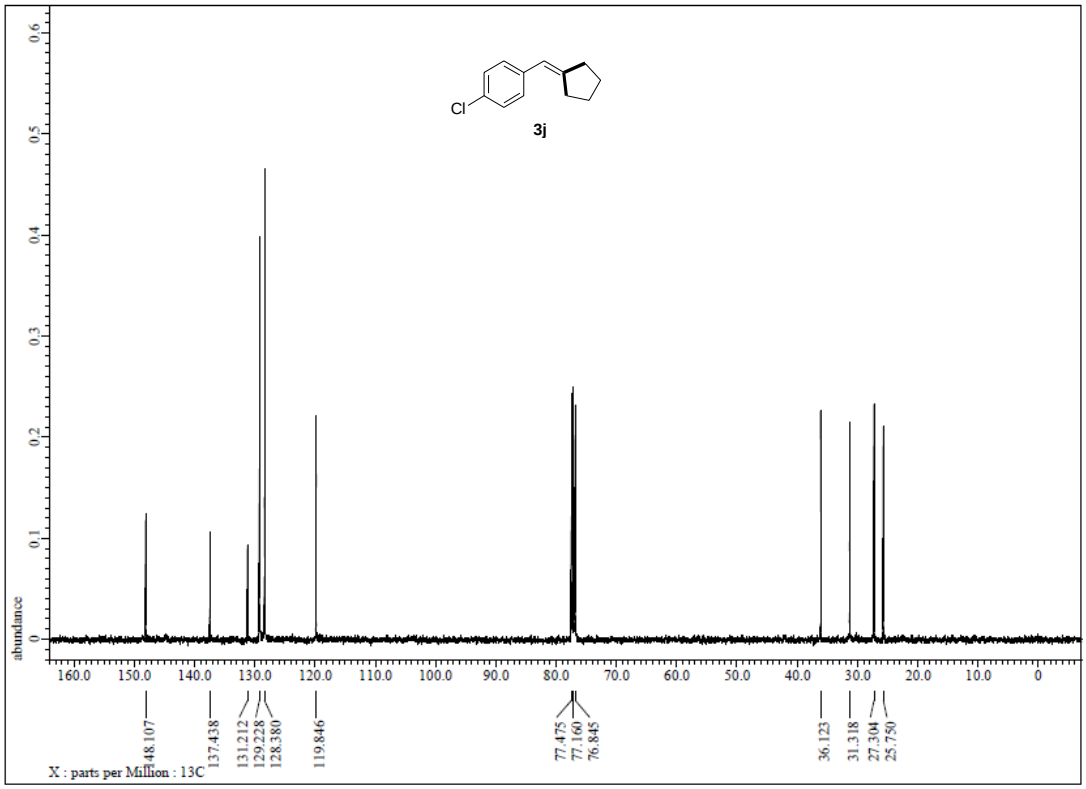

OMe
3h
abundance
X : parts per Million : 13C


3i
F
2.09
2.03
1.02
4.00
2.06
2.00
abundance
7.266
7.251
7.248
7.244
7.235
7.229
7.010
6.989
6.983
6.967
6.315
6.309
6.304
2.512
2.494
2.489
2.469
2.450
1.794
1.777
1.759
1.742
1.676
1.659
1.641
0.000
X : parts per Million : 1H


3i
F
abundance
162.228
159.796
146.858
135.159
135.121
129.476
129.400
119.789
115.203
114.993
77.475
77.160
76.836
35.990
31.165
27.304
25.788
X : parts per Million : 13C


F
3i
abundance
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0 -20.0 -40.0 -60.0 -80.0 -100.0 -120.0 -140.0 -160.0 -180.0 -200.0
-116.899
X : parts per Million : 19F


4.03
0.90
4.06
2.09
2.01
Cl
3j
abundance
8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.300
7.294
7.283
7.278
7.260
7.244
7.223
6.329
6.324
6.318
2.539
2.522
2.518
2.503
2.498
2.483
1.824
1.806
1.789
1.772
1.706
1.688
1.672
X : parts per Million : 1H


Cl
3j
abundance
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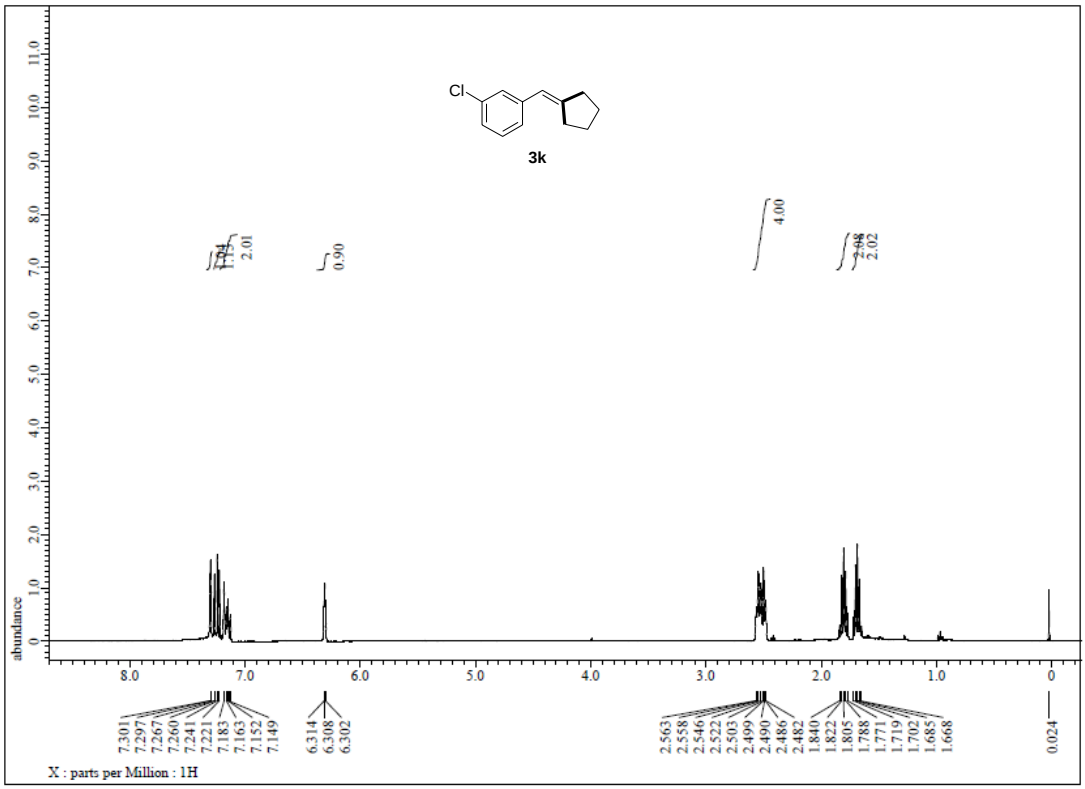
31.318
27.304
25.750
X : parts per Million : 13C


Cl
3k
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0.90
4.00
2.02
abundance
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7.297
7.267
7.260
7.241
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7.163
7.152
7.149
6.314
6.308
6.302
2.563
2.558
2.546
2.522
2.503
2.499
2.490
2.486
2.482
1.840
1.822
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1.788
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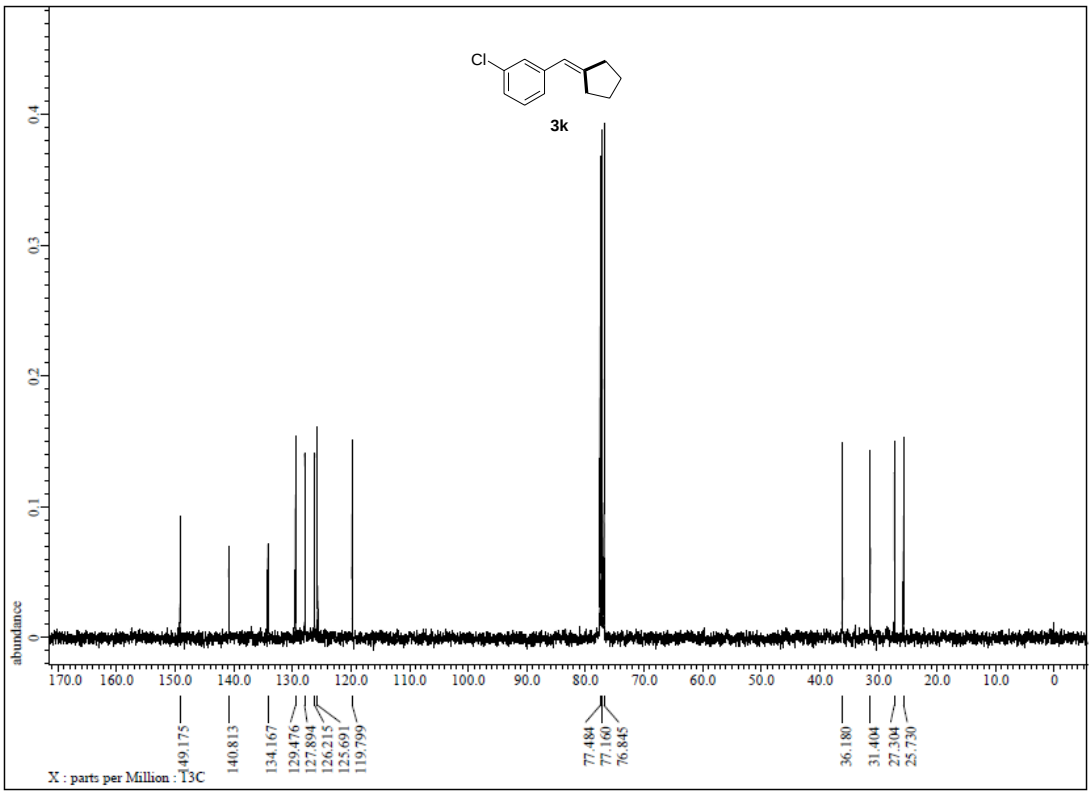
1.719
1.702
1.685
1.668
0.024
X : parts per Million : 1H


Cl
3k
abundance
X : parts per Million : 13C
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140.813
134.167
129.476
127.894
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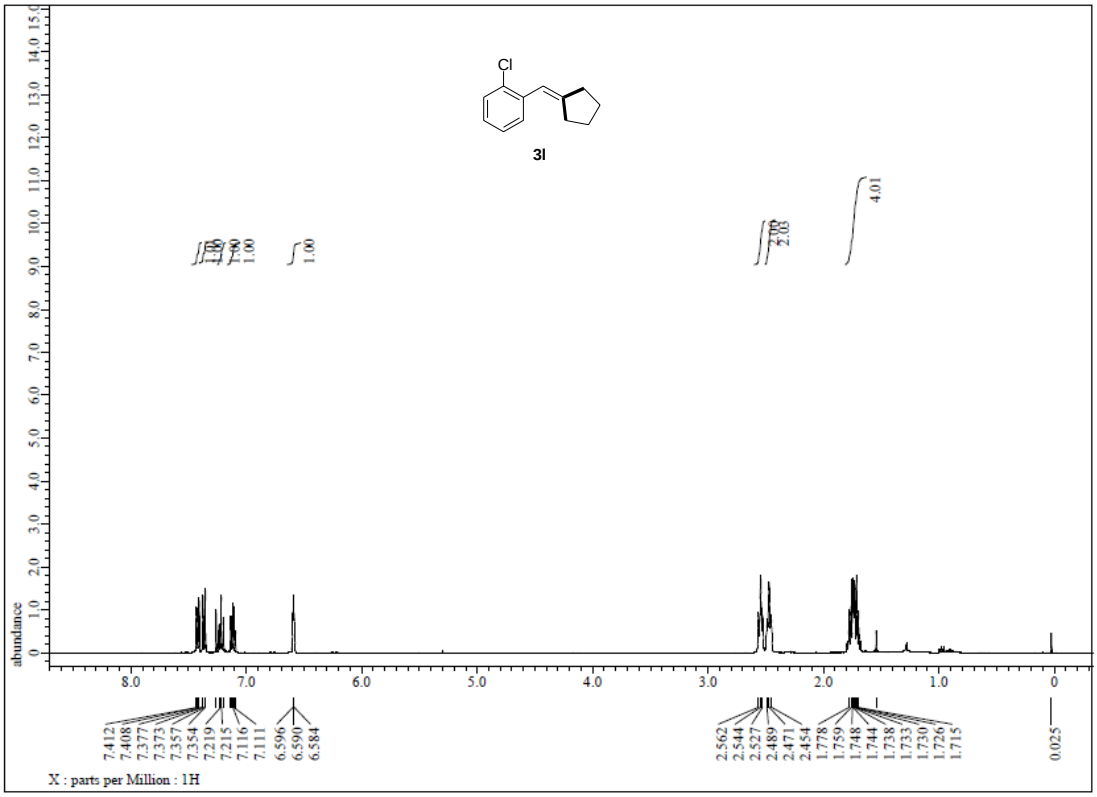
125.691
119.799
77.484
77.160
76.845
36.180
31.404
27.304
25.730


Cl
3l
abundance
X : parts per Million : 1H
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7.377
7.373
7.357
7.354
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7.215
7.116
7.111
6.596
6.590
6.584
2.562
2.544
2.527
2.489
2.471
2.454
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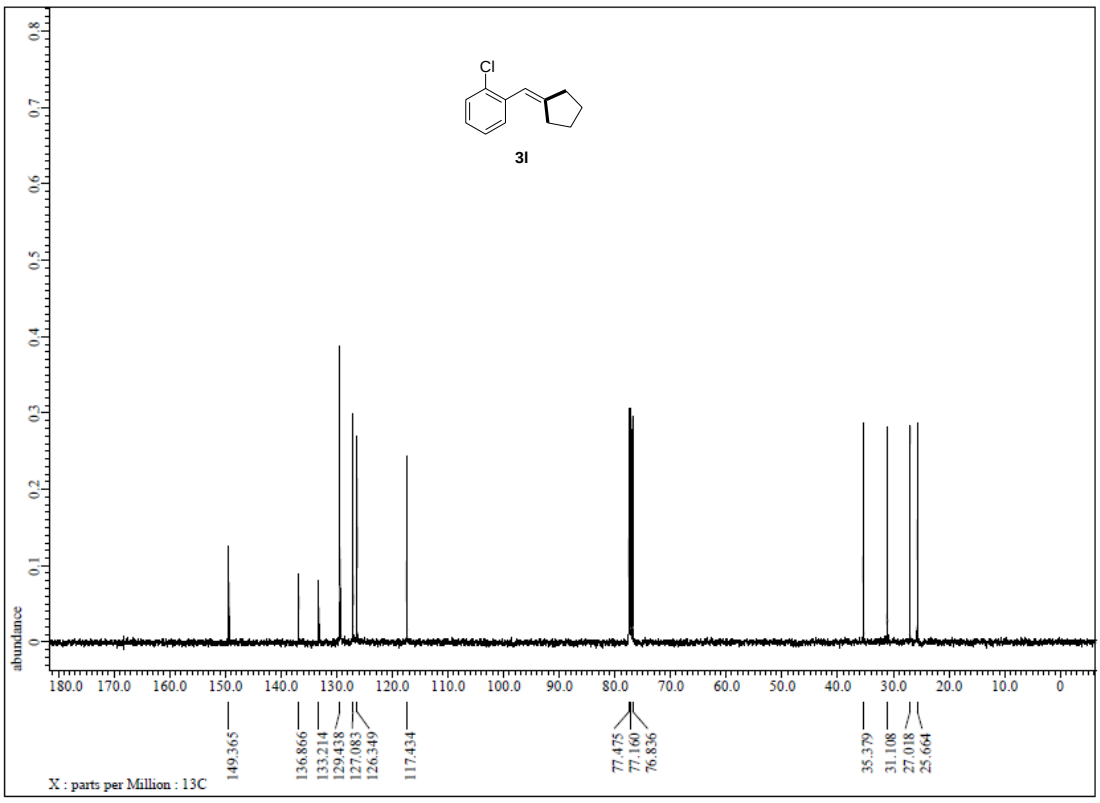
1.748
1.744
1.738
1.733
1.730
1.726
1.715
0.025
4.01


Cl
3l
abundance
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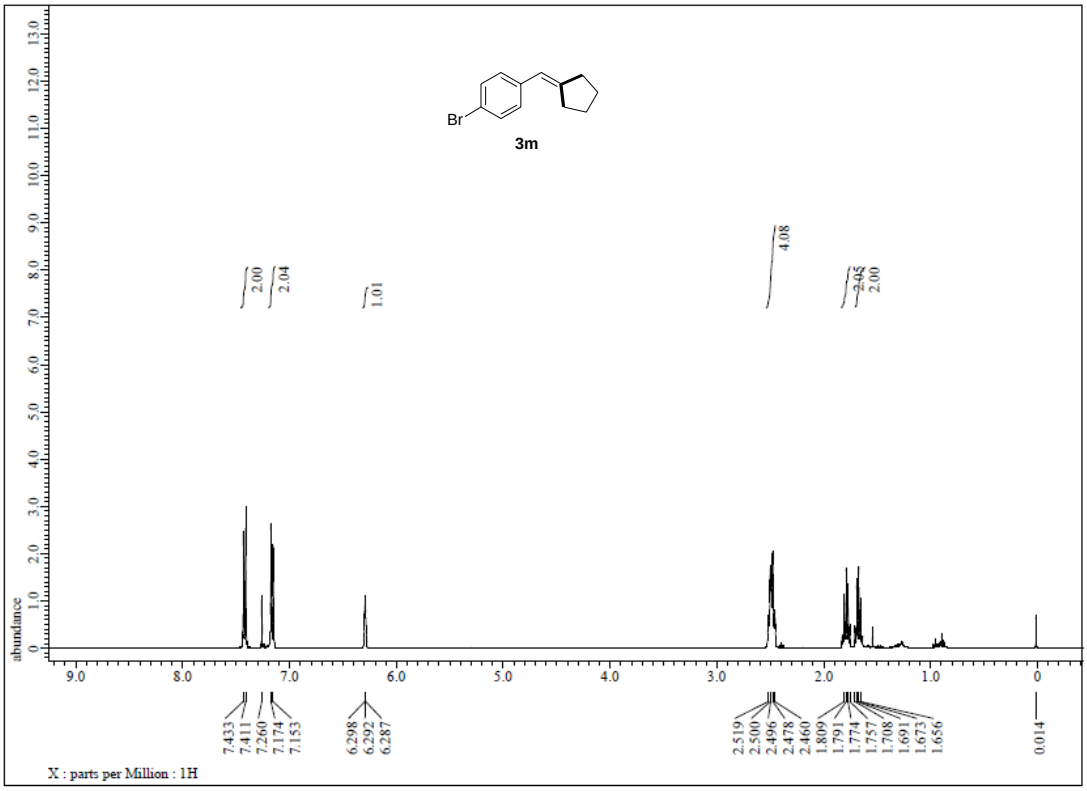
31.108
27.018
25.664
X : parts per Million : 13C


Br
3m
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2.04
1.01
4.08
2.00
abundance
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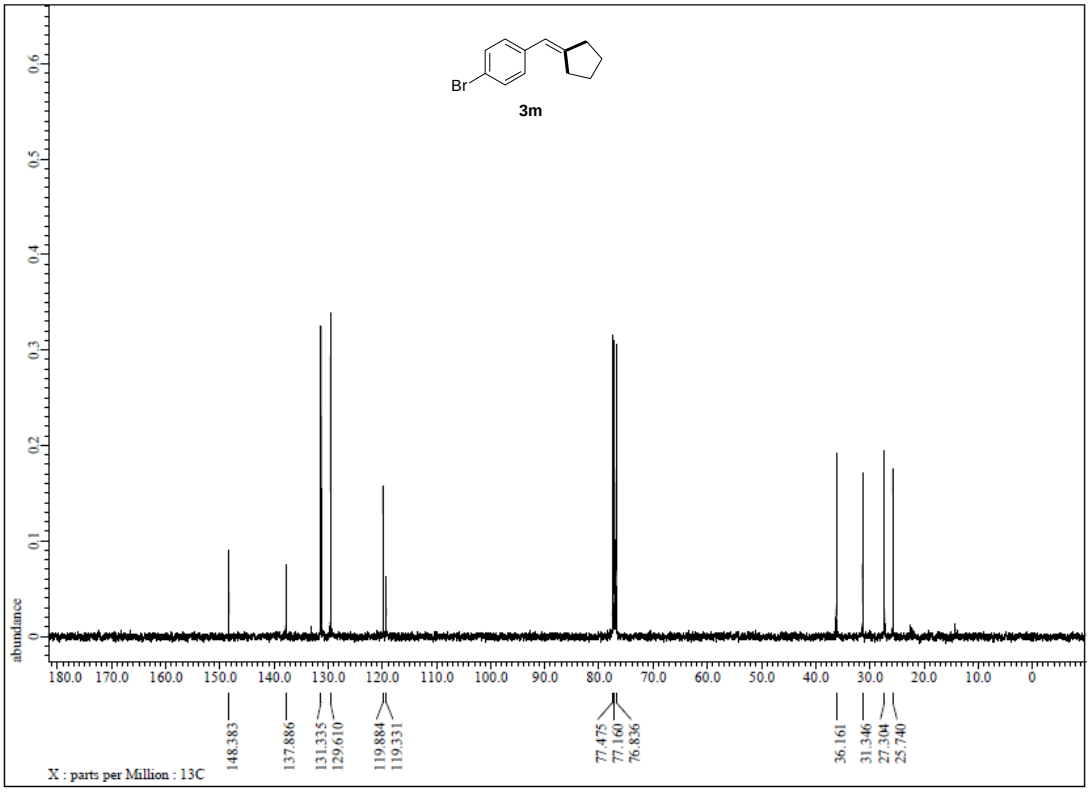
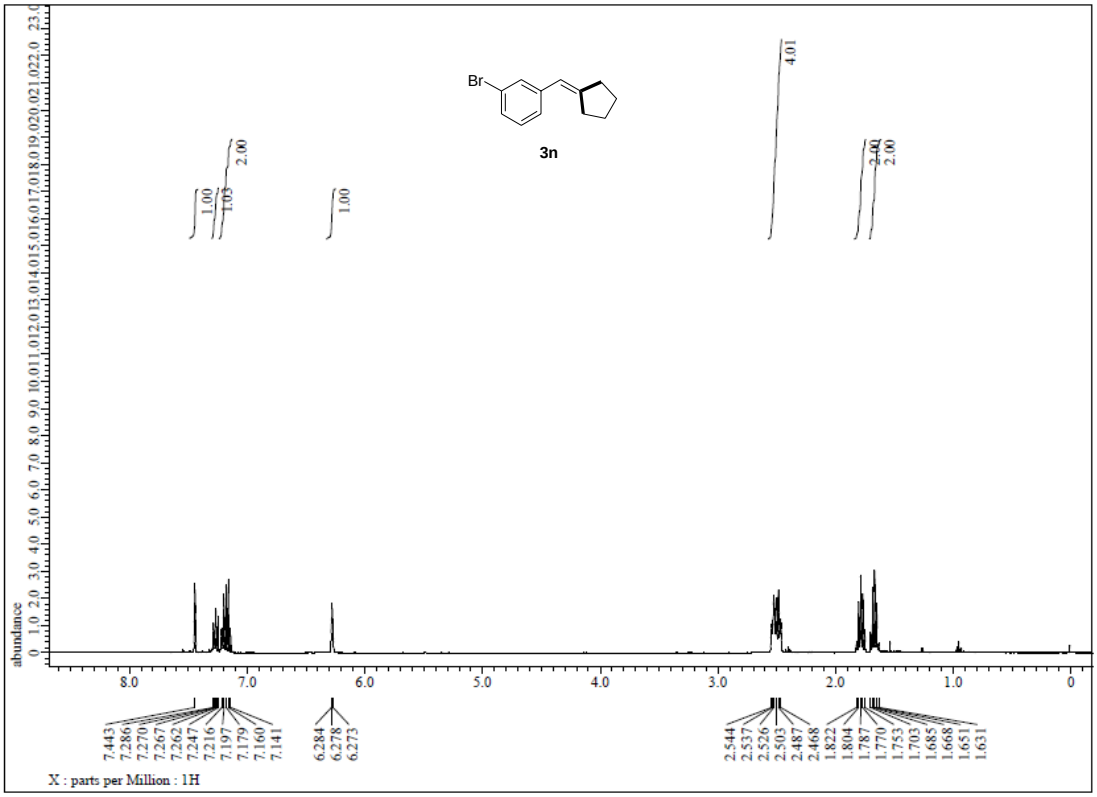
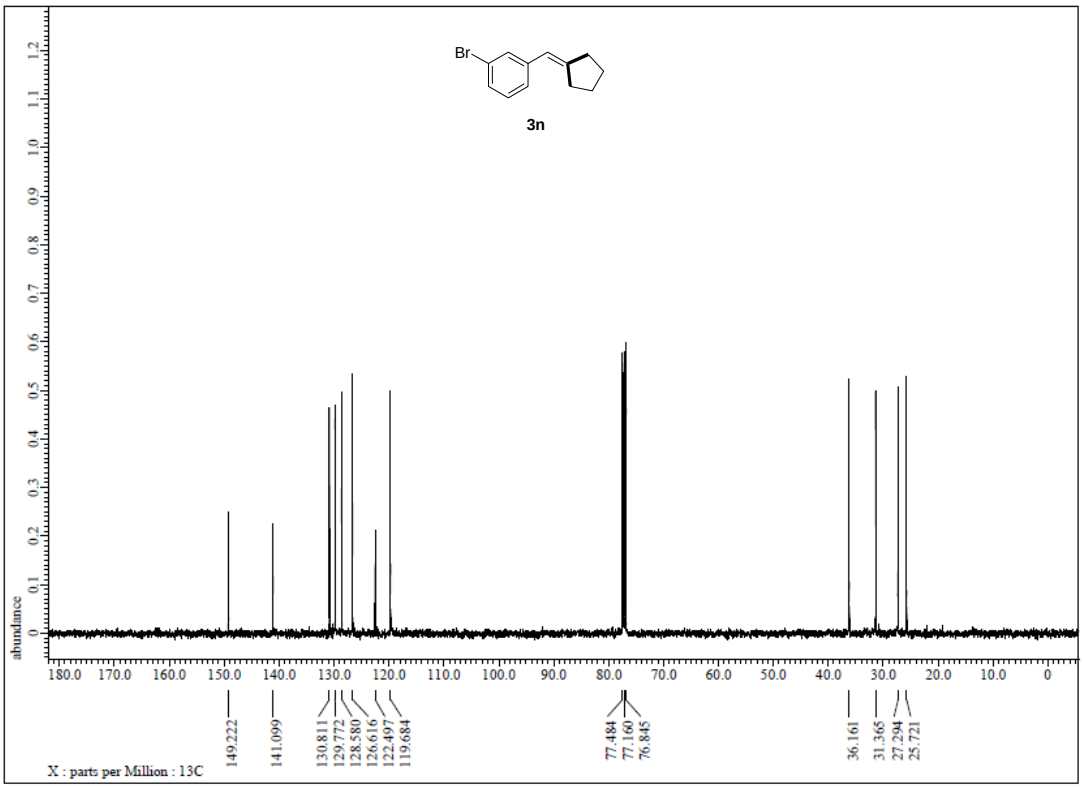
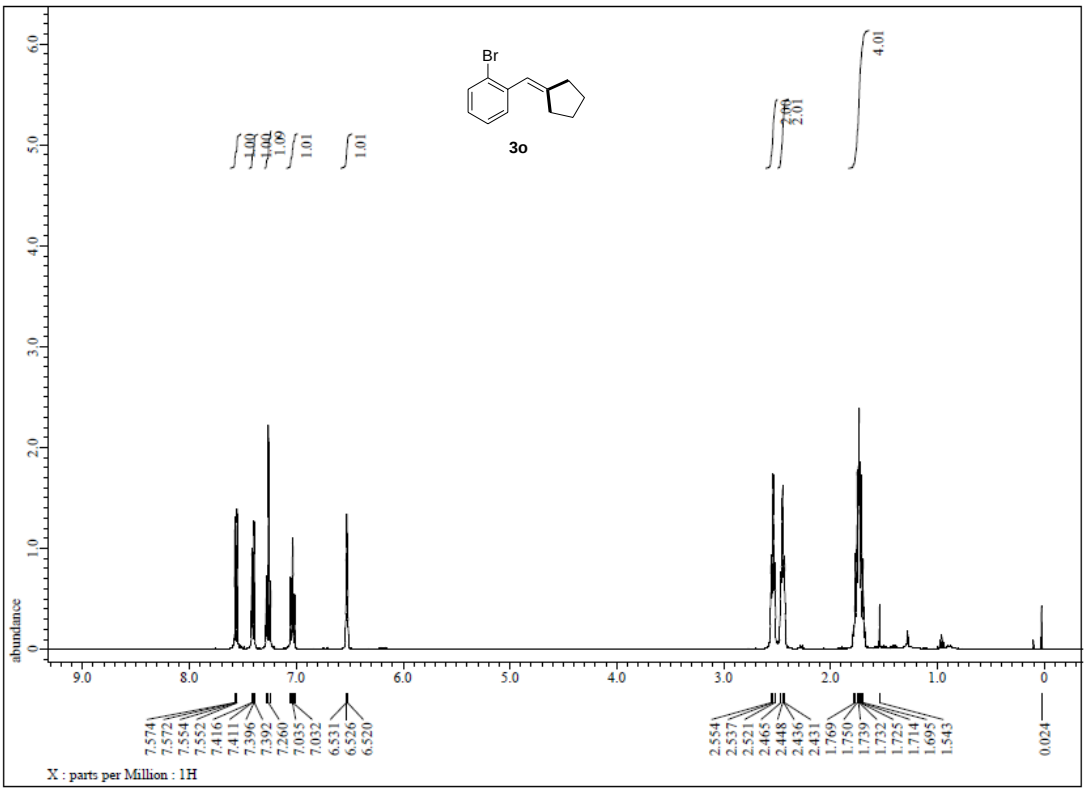
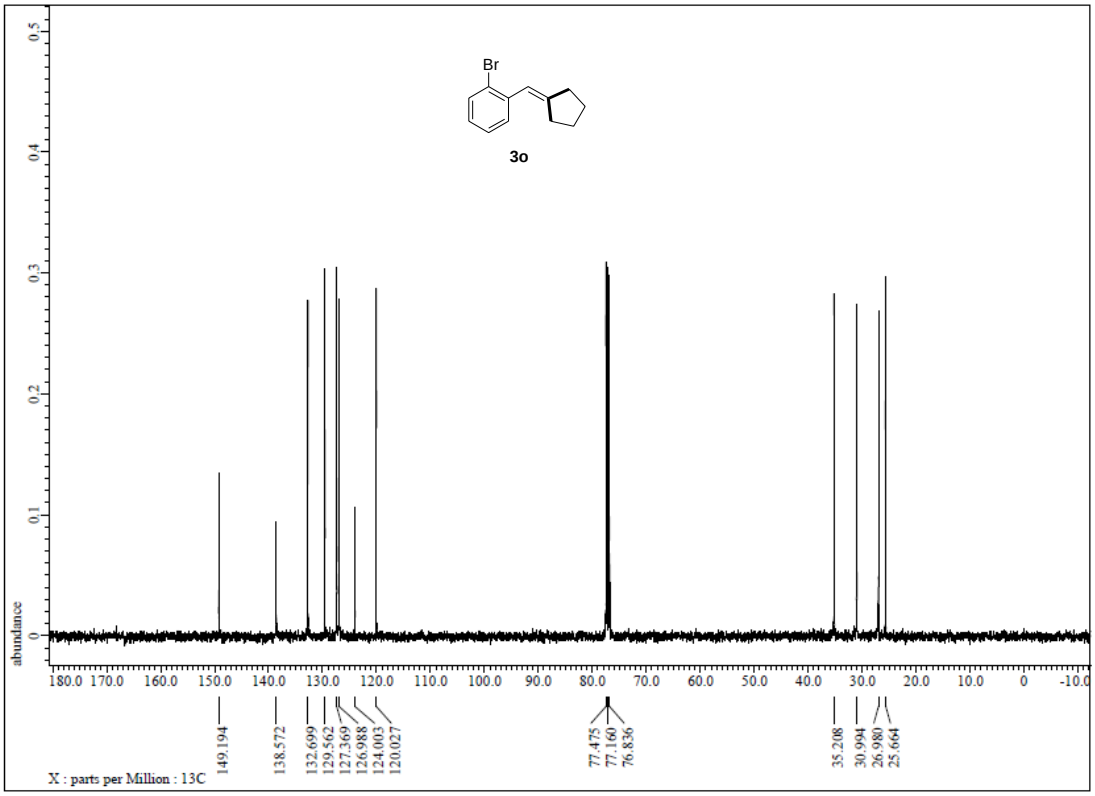
7.260
7.174
7.153
6.298
6.292
6.287
2.519
2.500
2.496
2.478
2.460
1.809
1.791
1.774
1.757
1.708
1.691
1.673
1.656
0.014
X : parts per Million : 1H

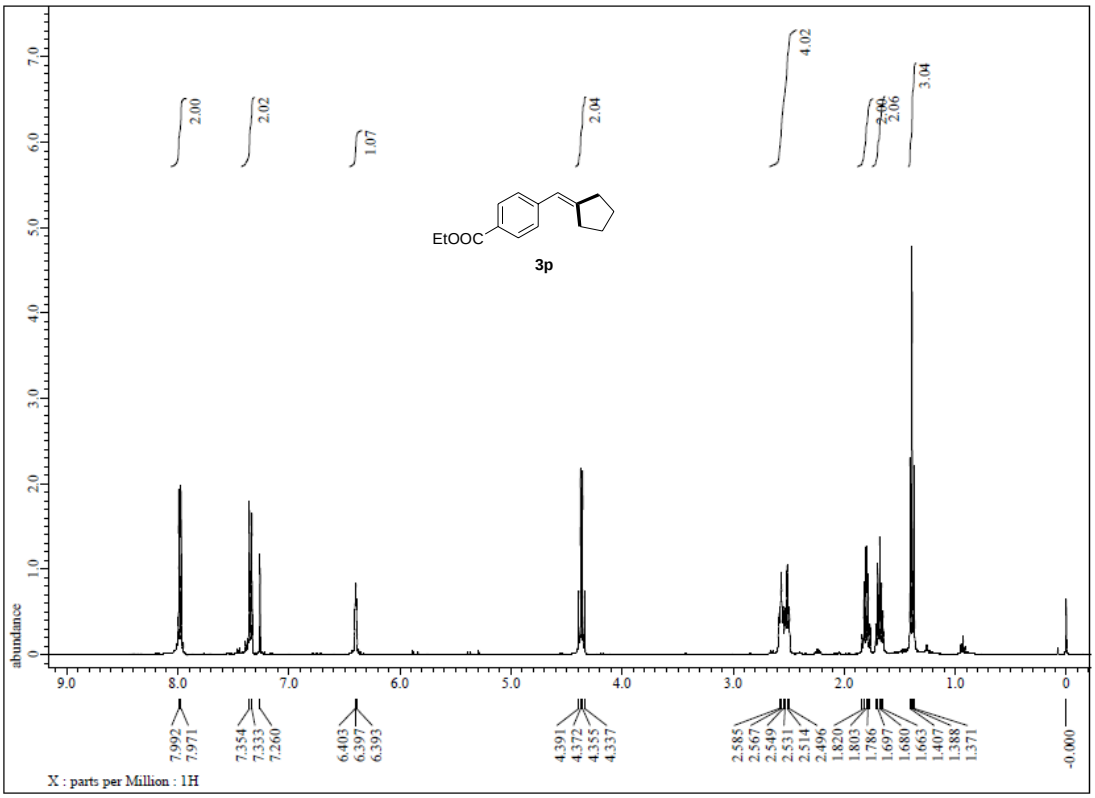

Br
3m
abundance
148.383
137.886
131.335
129.610
119.884
119.331
77.475
77.160
76.836
36.161
31.346
27.304
25.740
X : parts per Million : 13C

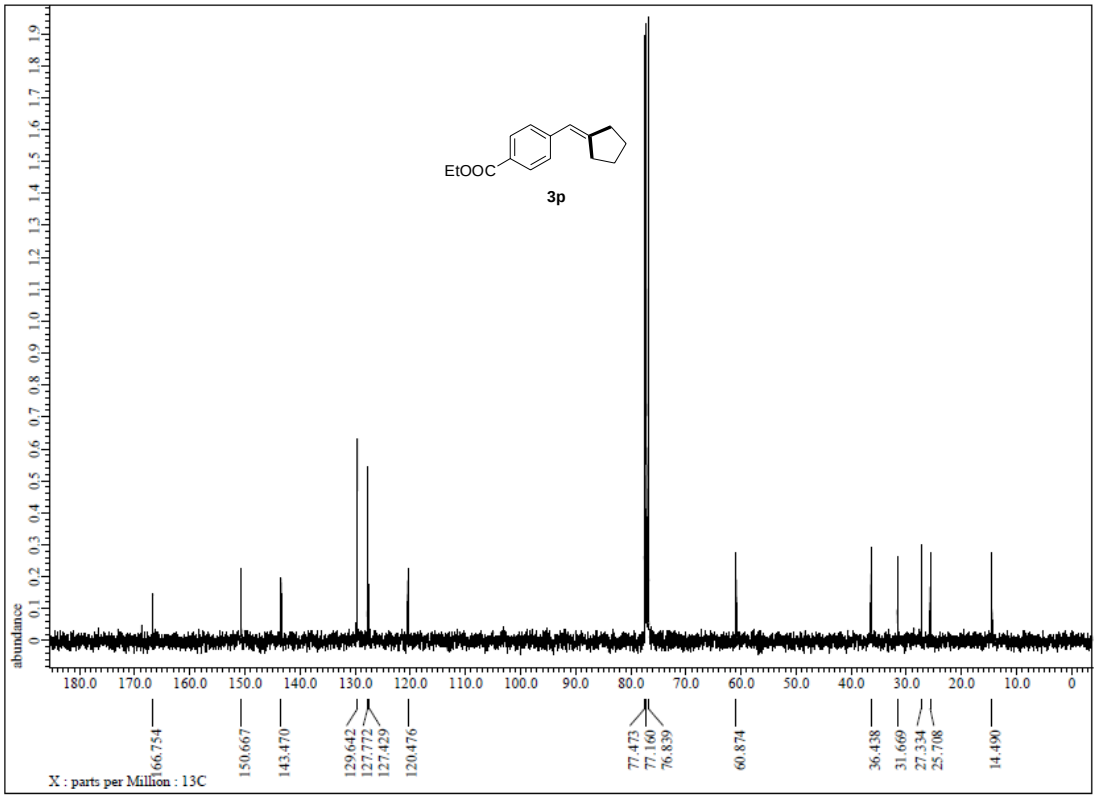

Br
3n
1.00
1.03
2.00
1.00
4.01
2.00
2.00
abundance
7.443
7.286
7.270
7.267
7.262
7.247
7.216
7.197
7.179
7.160
7.141
6.284
6.278
6.273
2.544
2.537
2.526
2.503
2.487
2.468
1.822
1.804
1.787
1.770
1.753
1.703
1.685
1.668
1.651
1.631
X : parts per Million : 1H

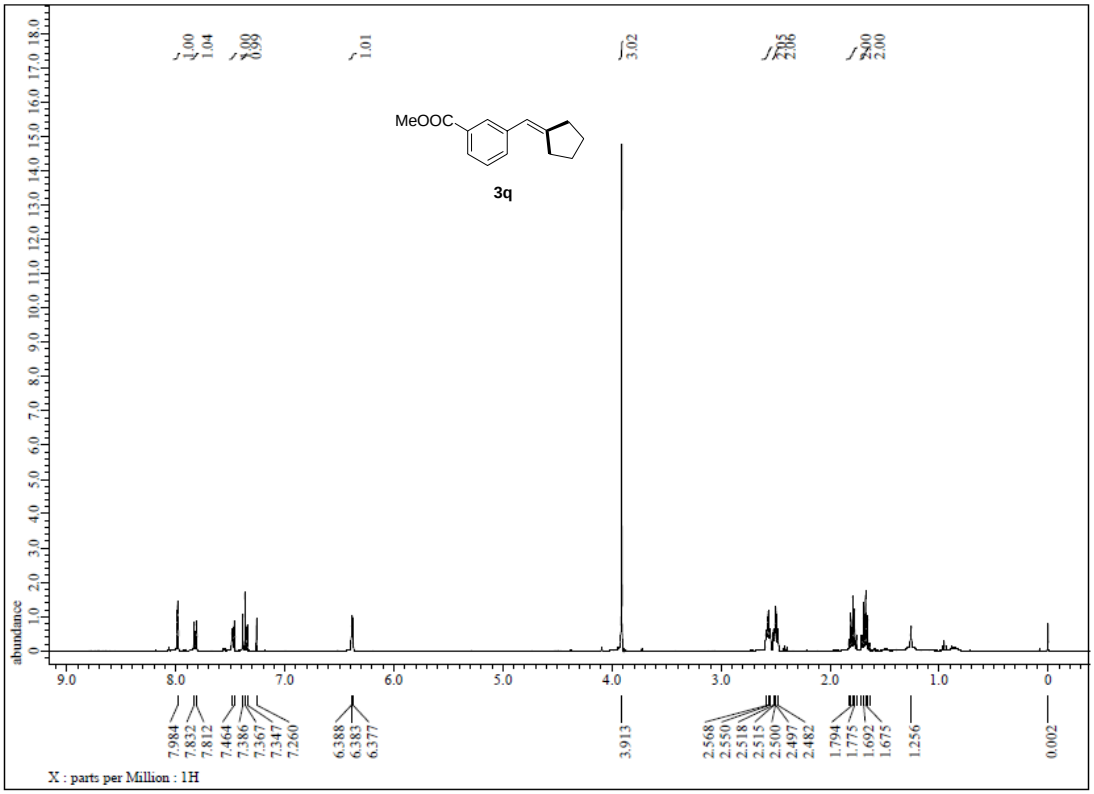

Br
3n
abundance
X : parts per Million : 13C
149.222
141.099
130.811
129.772
128.580
126.616
122.497
119.684
77.484
77.160
76.845
36.161
31.365
27.294
25.721

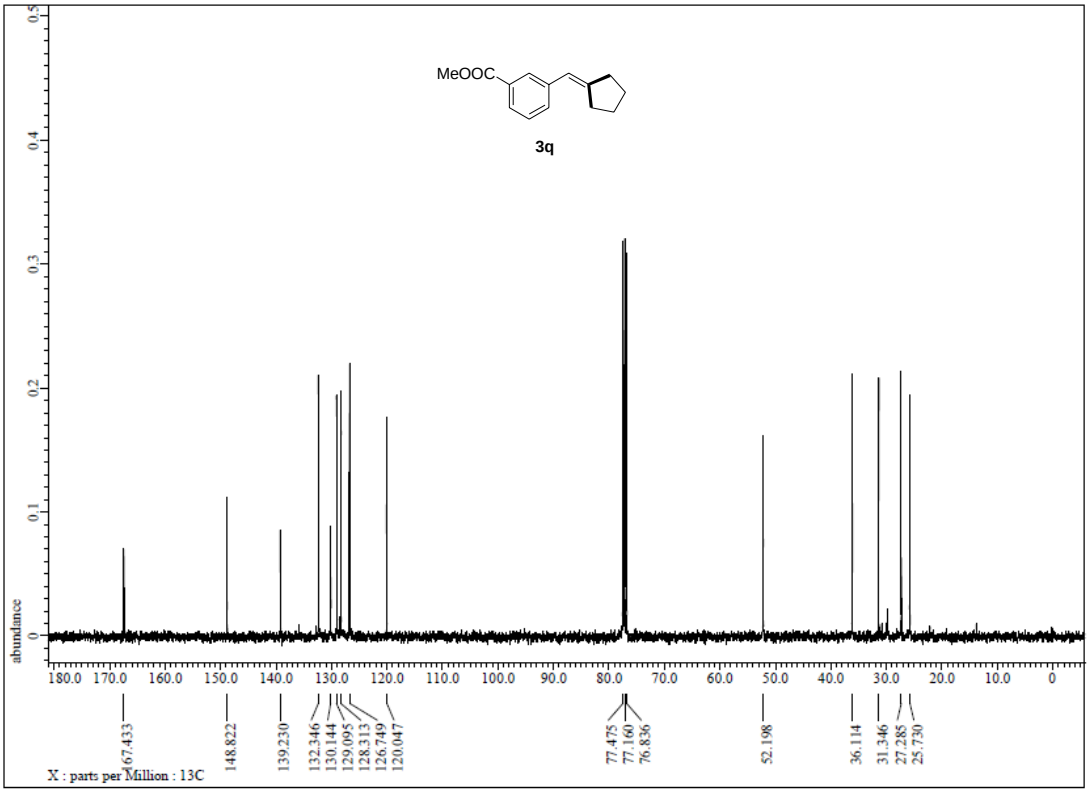

Br
3o
1.00
1.00
1.09
1.01
1.01
2.00
2.01
4.01
abundance
X : parts per Million : 1H
7.574
7.572
7.554
7.552
7.416
7.411
7.396
7.392
7.260
7.035
7.032
6.531
6.526
6.520
2.554
2.537
2.521
2.465
2.448
2.436
2.431
1.769
1.750
1.739
1.732
1.725
1.714
1.695
1.543
0.024

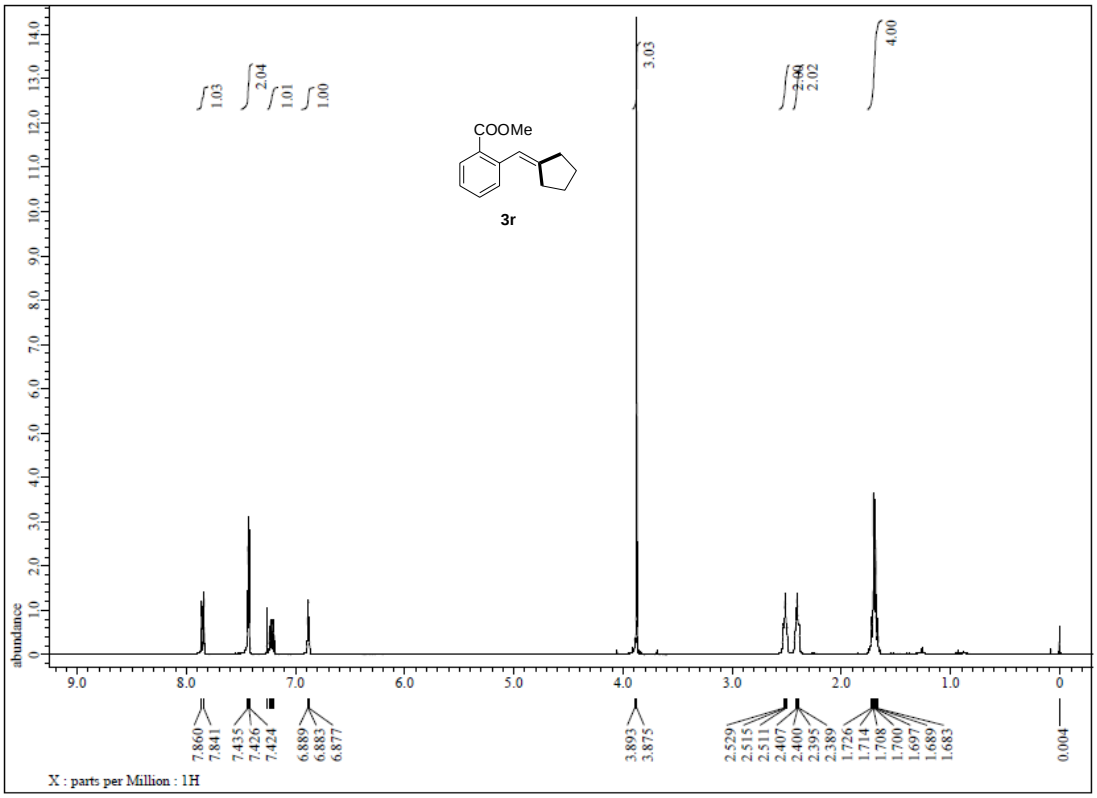

Br
3o
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
149.194
138.572
132.699
129.562
127.369
126.988
124.003
120.027
77.475
77.160
76.836
35.208
30.994
26.980
25.664
X : parts per Million : 13C

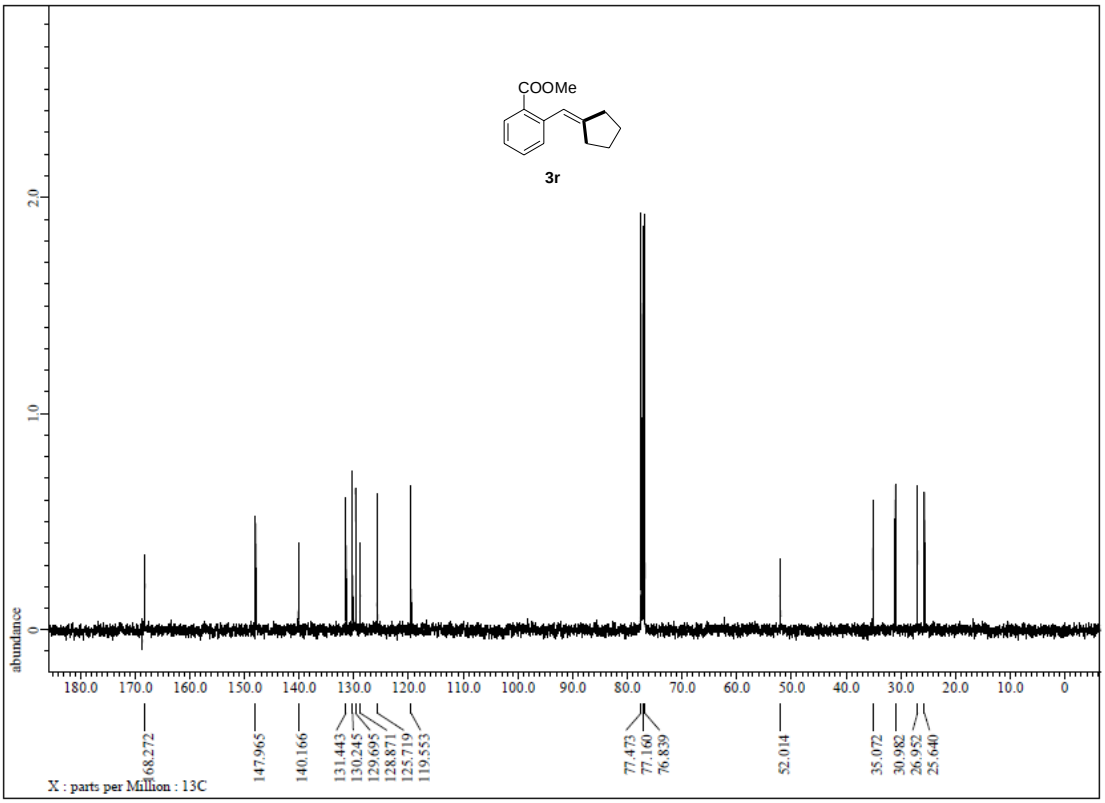

2.00
2.02
1.07
2.04
4.02
3.04
EtOOC
3p
abundance
9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.992
7.971
7.354
7.333
7.260
6.403
6.397
6.393
4.391
4.372
4.355
4.337
2.585
2.567
2.549
2.531
2.514
2.496
1.820
1.803
1.786
1.697
1.680
1.663
1.407
1.388
1.371
-0.000
X : parts per Million : 1H

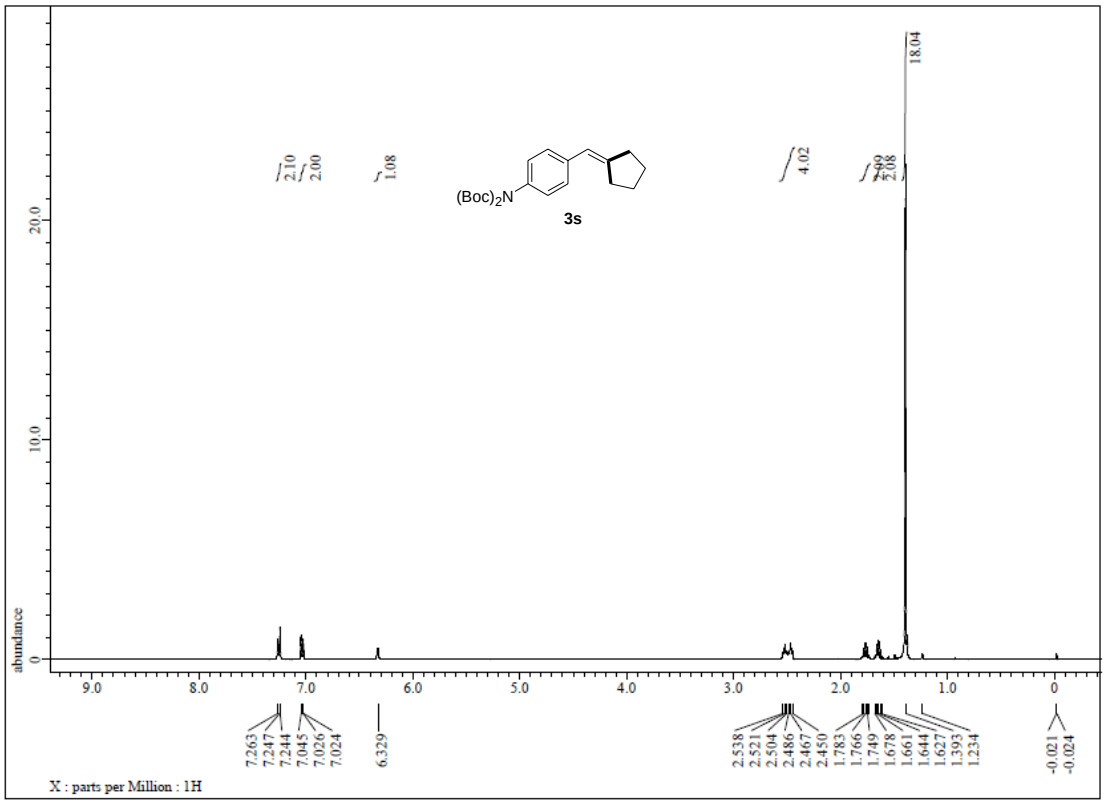

EtOOC
3p
abundance
X : parts per Million : 13C
166.754
150.667
143.470
129.642
127.772
127.429
120.476
77.473
77.160
76.839
60.874
36.438
31.669
27.334
25.708
14.490

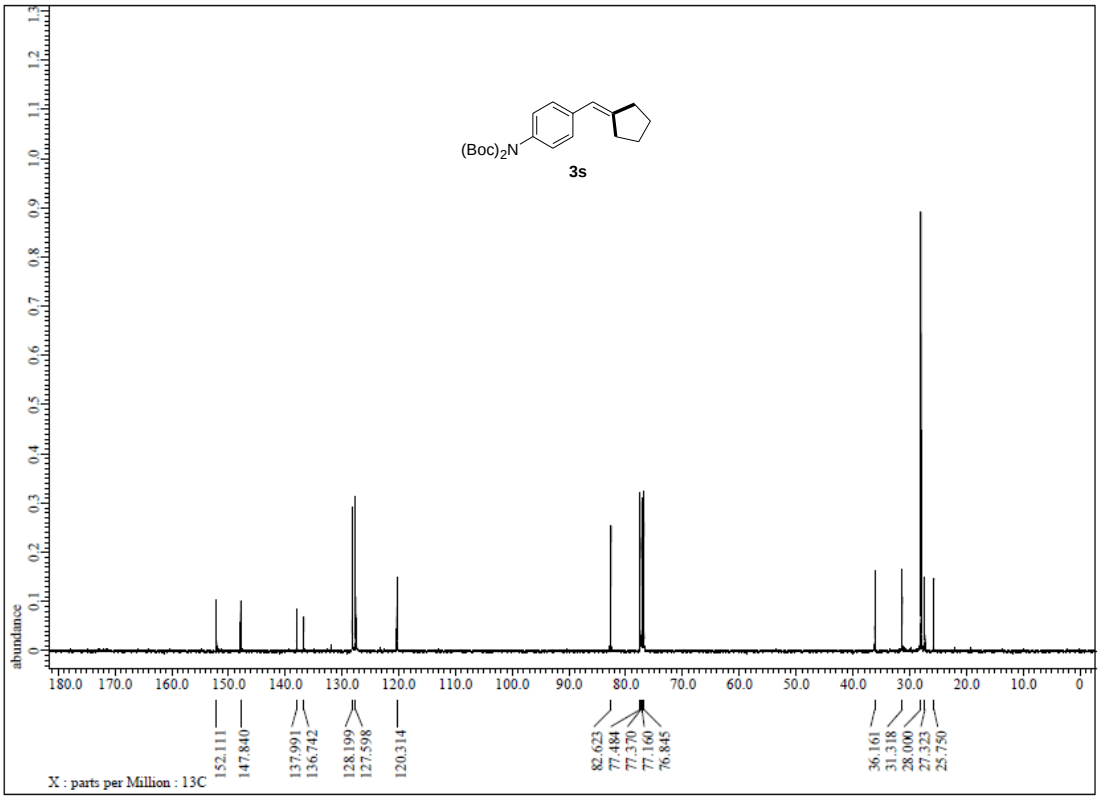

MeOOC
3q
abundance
X : parts per Million : 1H
7.984
7.832
7.812
7.464
7.386
7.367
7.347
7.260
6.388
6.383
6.377
3.913
2.568
2.550
2.518
2.515
2.500
2.497
2.482
1.794
1.775
1.692
1.675
1.256
0.002

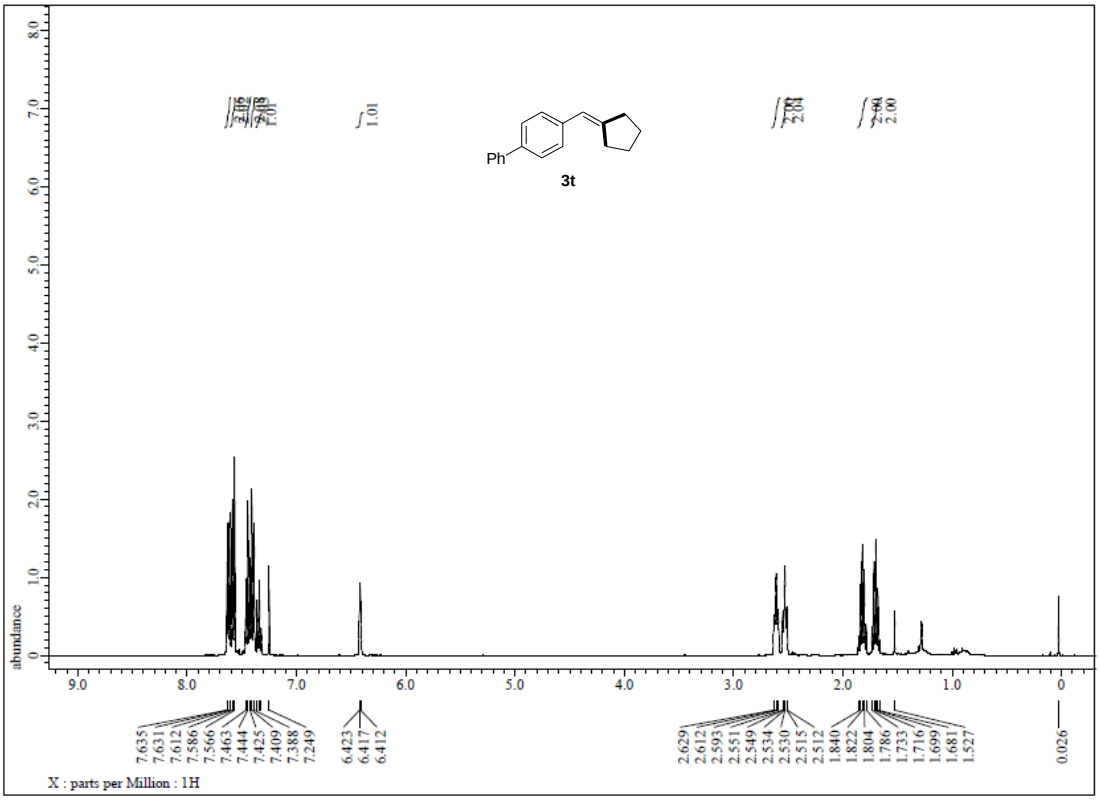

MeOOC
3q
abundance
X : parts per Million : 13C
67.433
148.822
139.230
132.346
130.144
129.095
128.313
126.749
120.047
77.475
77.160
76.836
52.198
36.114
31.346
27.285
25.730

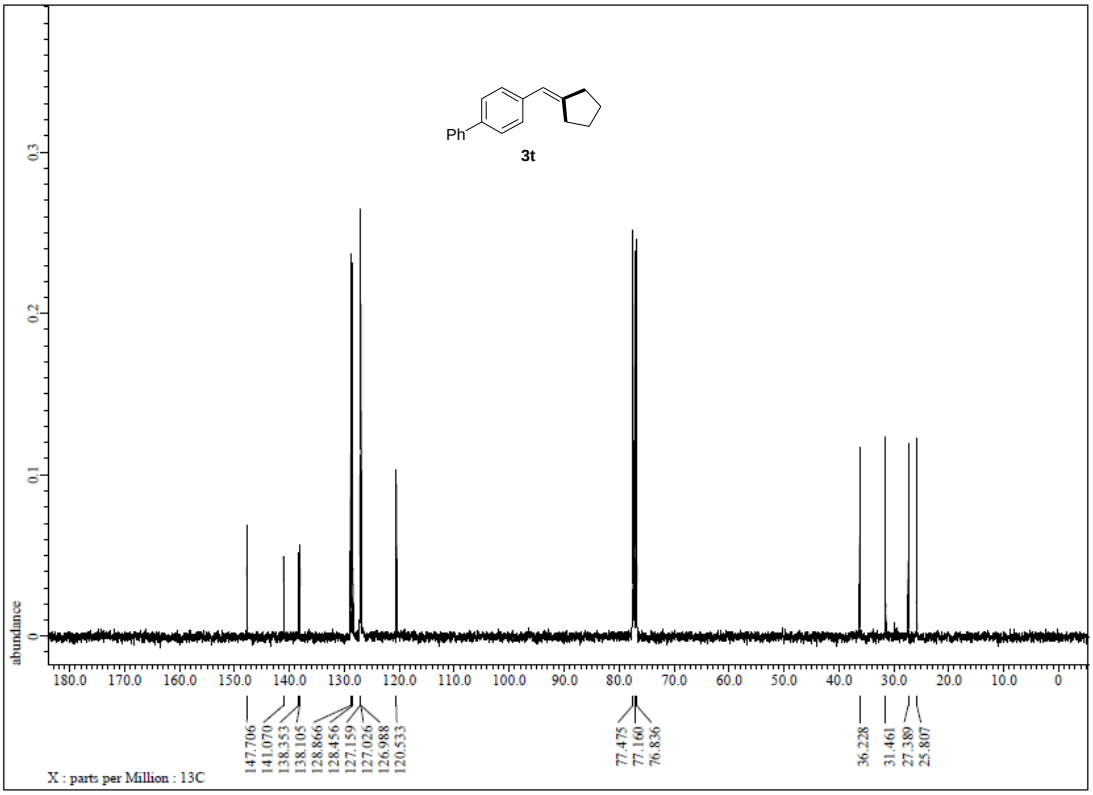

1.03
2.04
1.01
1.00
3.03
2.00
2.02
4.00
COOMe
3r
abundance
X : parts per Million : 1H
7.860
7.841
7.435
7.426
7.424
6.889
6.883
6.877
3.893
3.875
2.529
2.515
2.511
2.407
2.400
2.395
2.389
1.726
1.714
1.708
1.700
1.697
1.689
1.683
0.004

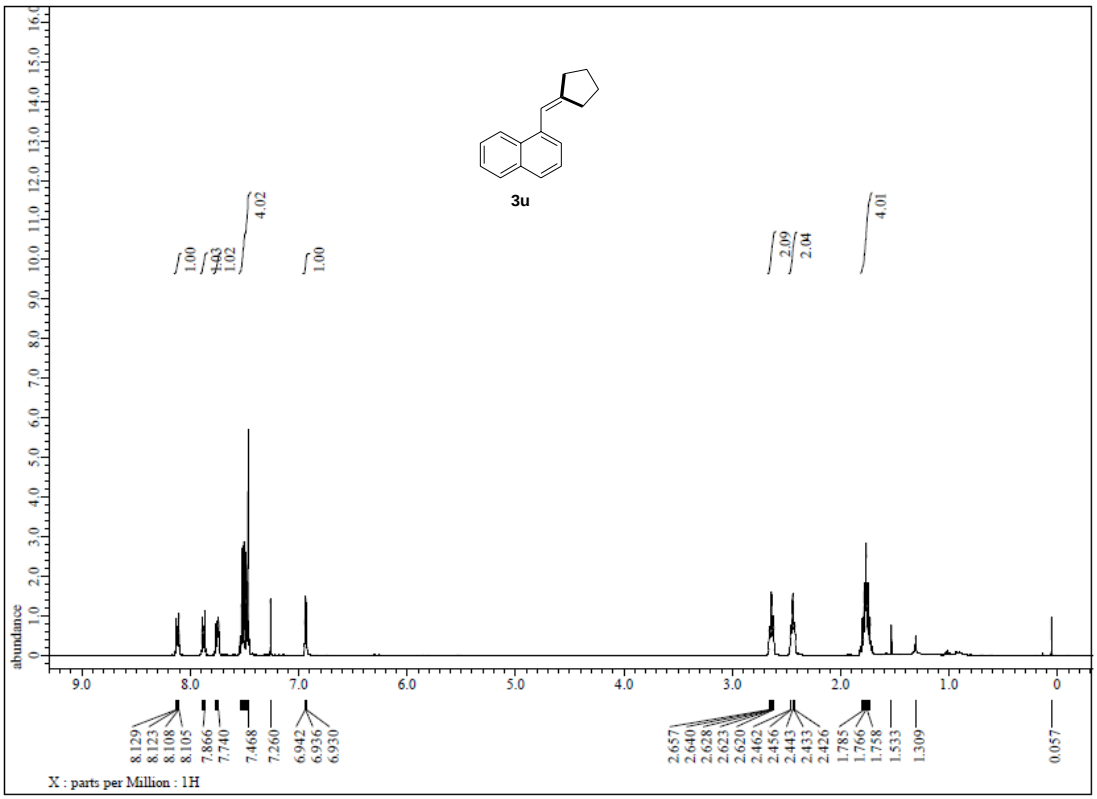

COOMe
3r
abundance
X : parts per Million : 13C
168.272
147.965
140.166
131.443
130.245
129.695
128.871
125.719
119.553
77.473
77.160
76.839
52.014
35.072
30.982
26.952
25.640

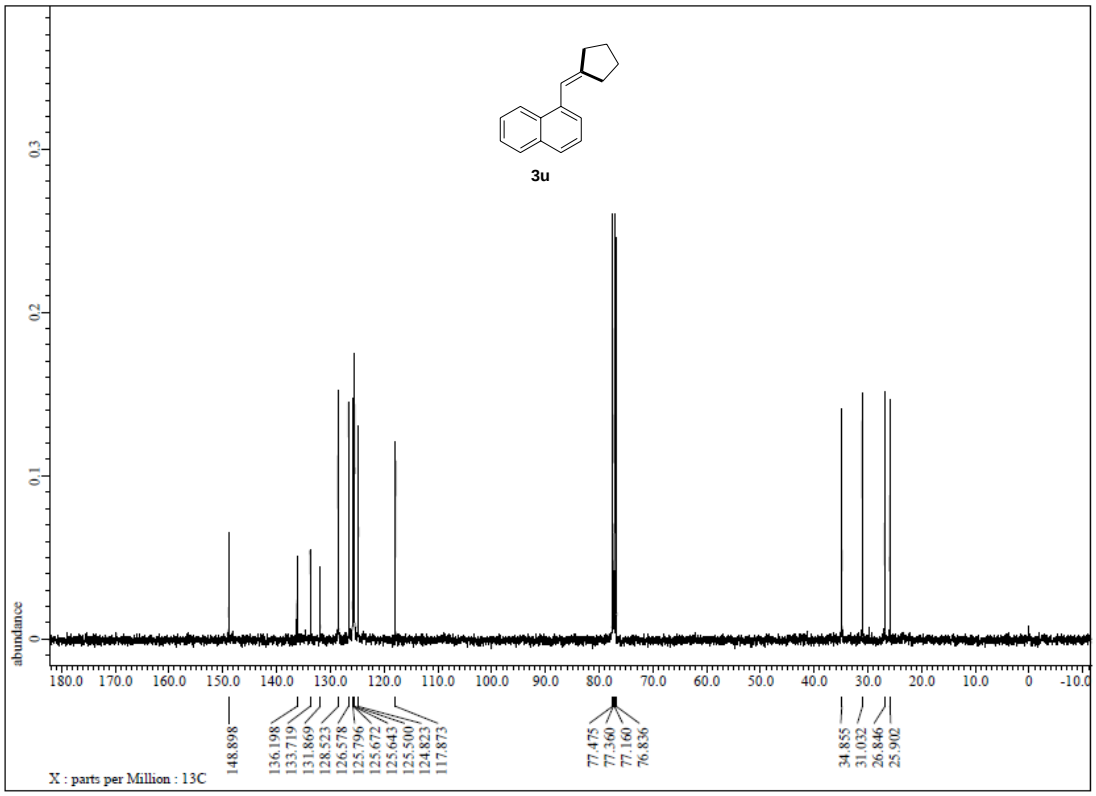

(Boc)2N
3s
abundance
X : parts per Million : 1H
18.04
4.02
2.10
2.00
1.08
7.263
7.247
7.244
7.045
7.026
7.024
6.329
2.538
2.521
2.504
2.486
2.467
2.450
1.783
1.766
1.749
1.678
1.661
1.644
1.627
1.393
1.234
-0.021
-0.024

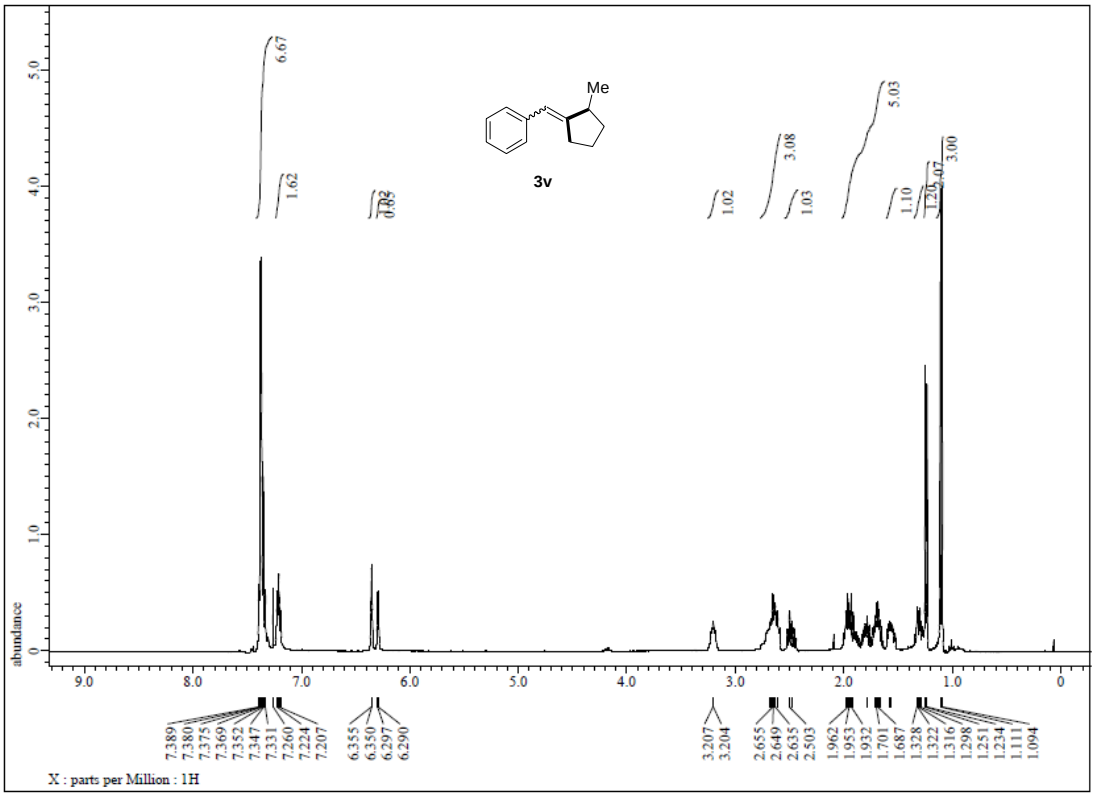

(Boc)2N
3s
abundance
X : parts per Million : 13C
152.111
147.840
137.991
136.742
128.199
127.598
120.314
82.623
77.484
77.370
77.160
76.845
36.161
31.318
28.000
27.323
25.750

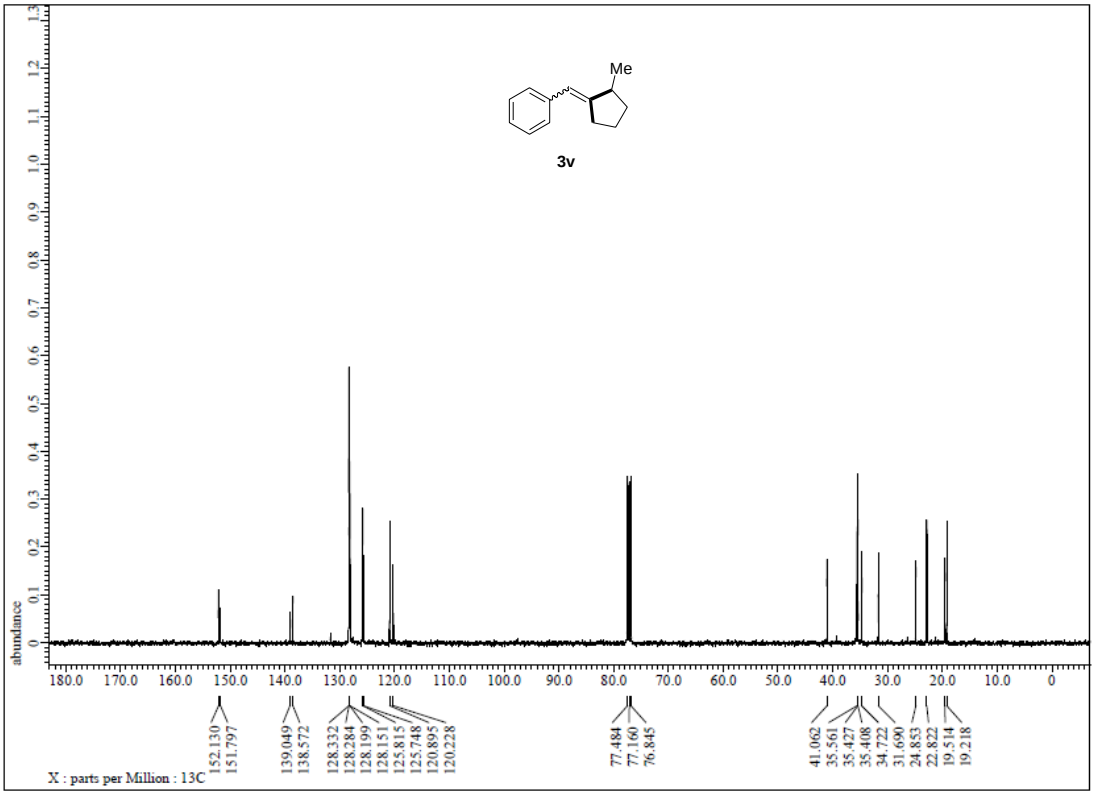

Ph
3t
1.01
abundance
X : parts per Million : 1H
7.635
7.631
7.612
7.586
7.566
7.463
7.444
7.425
7.409
7.388
7.249
6.423
6.417
6.412
2.629
2.612
2.593
2.551
2.549
2.534
2.530
2.515
2.512
1.840
1.822
1.804
1.786
1.733
1.716
1.699
1.681
1.527
0.026

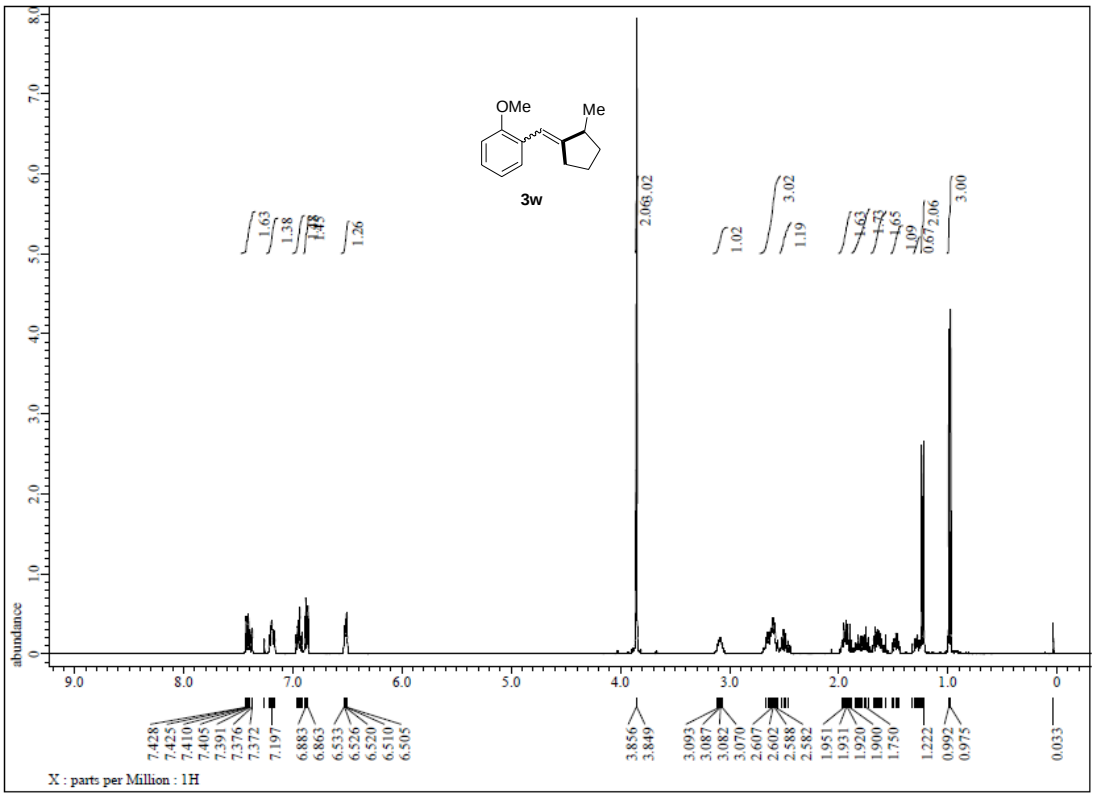

Ph
3t
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
147.706
141.070
138.353
138.105
128.866
128.456
127.159
127.026
126.988
120.533
77.475
77.160
76.836
36.228
31.461
27.389
25.807
X : parts per Million : 13C

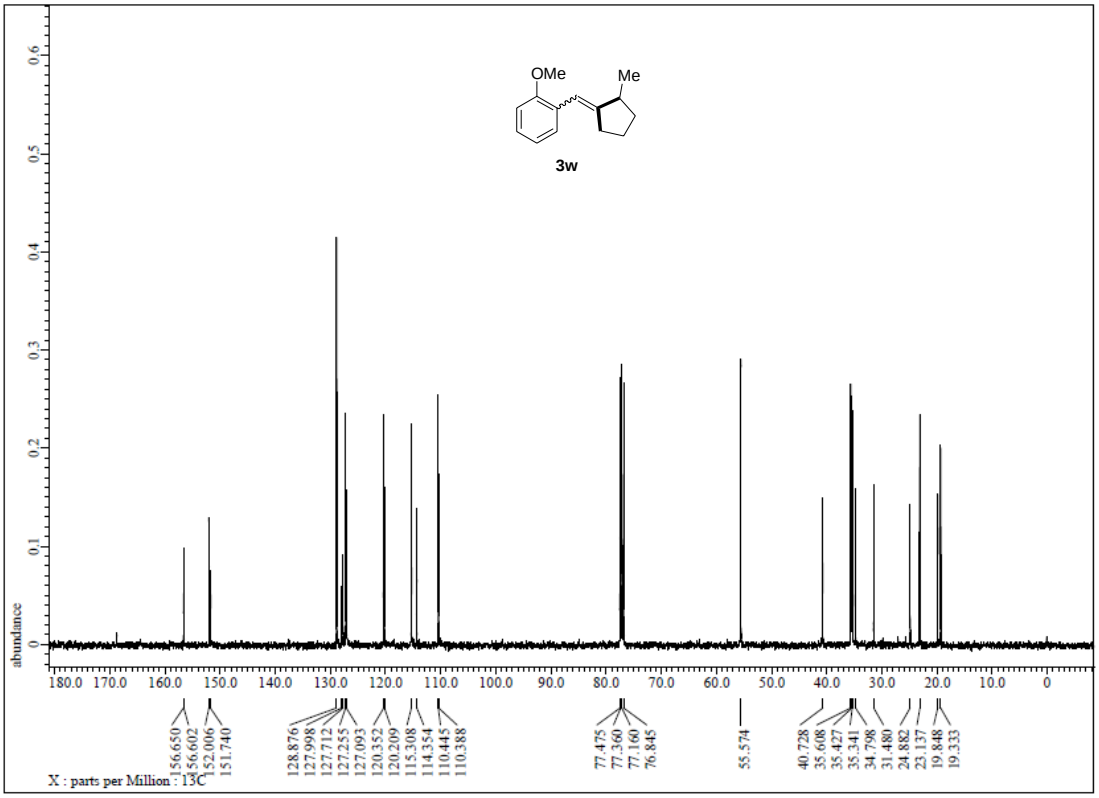

3u
1.00
1.03
1.02
4.02
1.00
2.09
2.04
4.01
abundance
9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
8.129
8.123
8.108
8.105
7.866
7.740
7.468
7.260
6.942
6.936
6.930
2.657
2.640
2.628
2.623
2.620
2.462
2.456
2.443
2.433
2.426
1.785
1.766
1.758
1.533
1.309
0.057
X : parts per Million : 1H

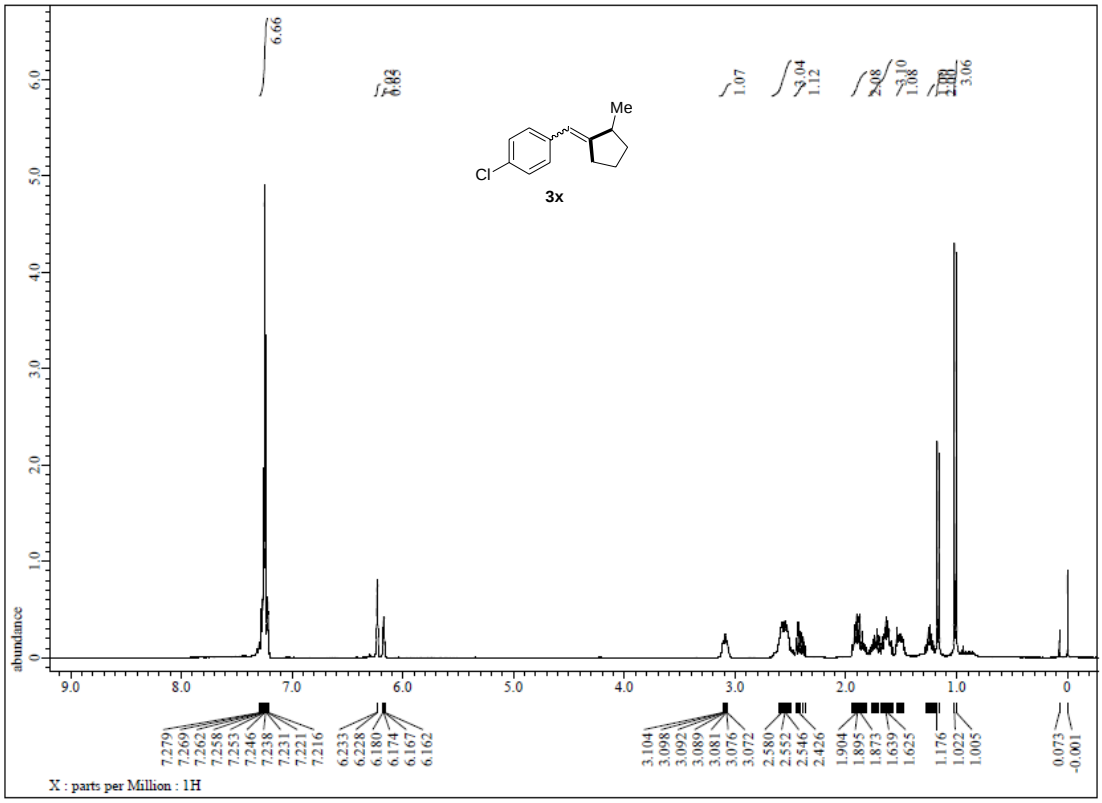

3u
abundance
X : parts per Million : 13C
148.898
136.198
133.719
131.869
128.523
126.578
125.796
125.672
125.643
125.500
124.823
117.873
77.475
77.360
77.160
76.836
34.855
31.032
26.846
25.902

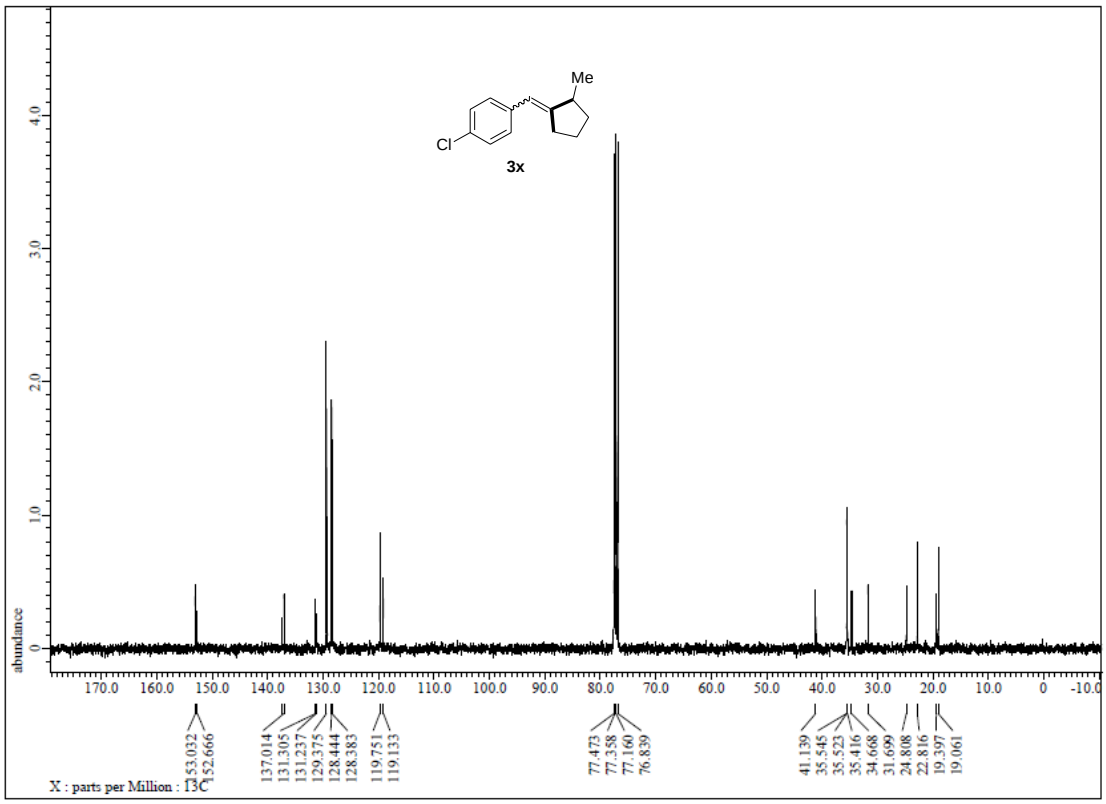

Me
3v
6.67
1.62
1.02
3.08
1.03
5.03
1.10
3.00
abundance
X : parts per Million : 1H
7.389
7.380
7.375
7.369
7.352
7.347
7.331
7.260
7.224
7.207
6.355
6.350
6.297
6.290
3.207
3.204
2.655
2.649
2.635
2.503
1.962
1.953
1.932
1.701
1.687
1.328
1.322
1.316
1.298
1.251
1.234
1.111
1.094

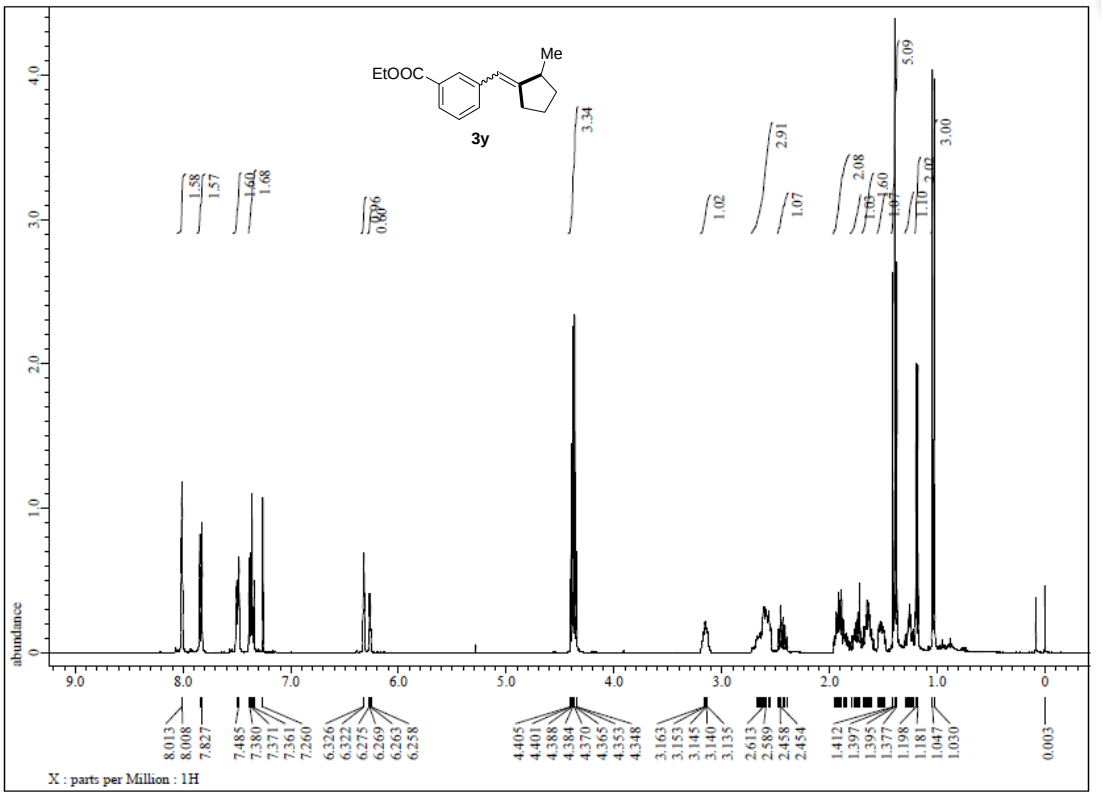

Me
3v
abundance
X : parts per Million : 13C

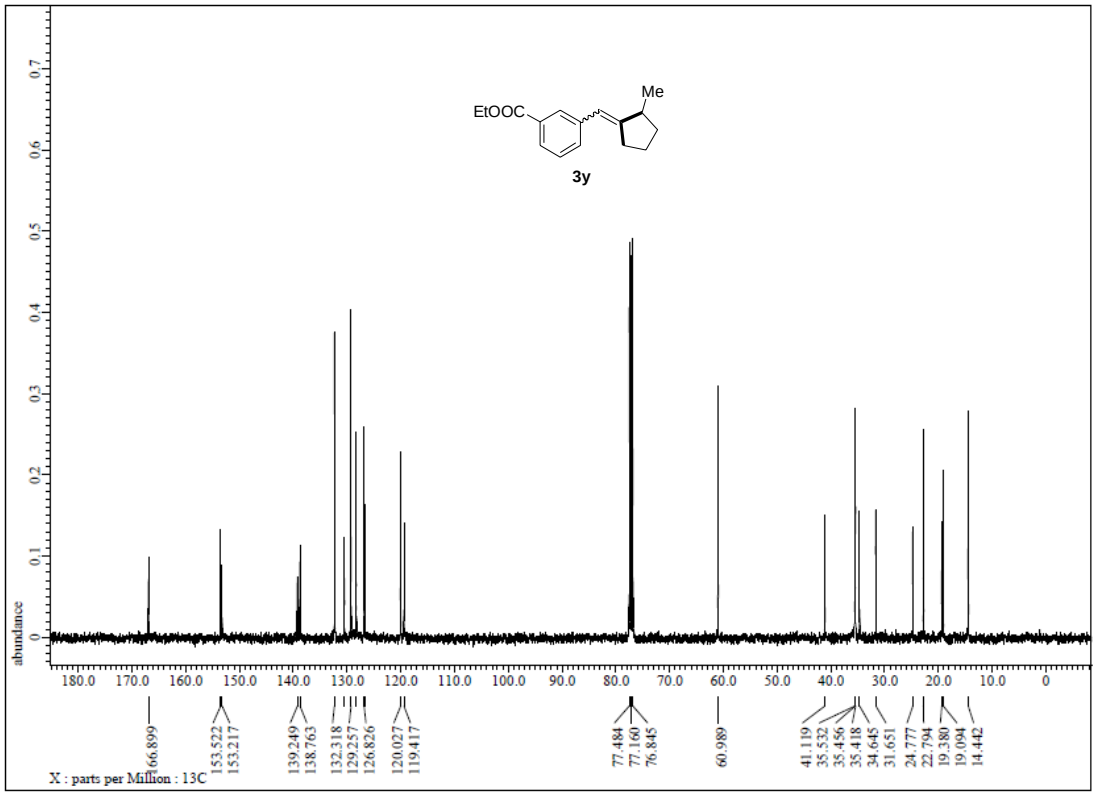

OMe
Me
3w
abundance
X : parts per Million : 1H

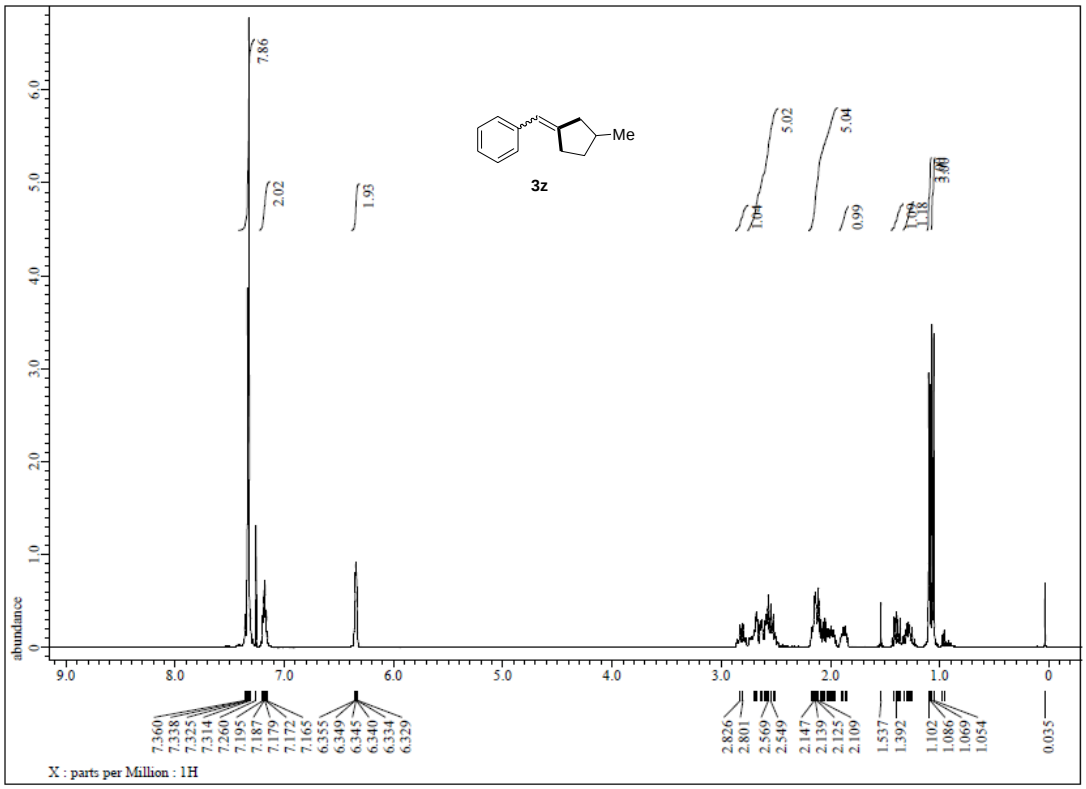

OMe
Me
3w
abundance
X : parts per Million : 13C

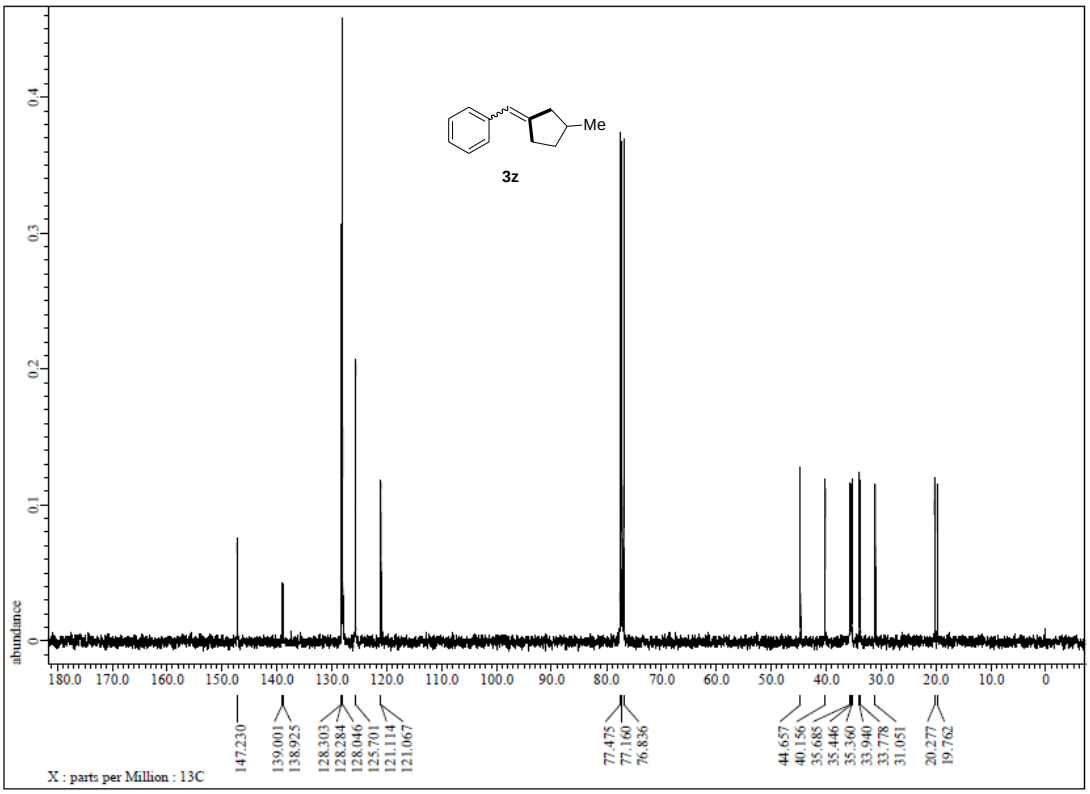

Me
Cl
3x
abundance
X : parts per Million : 1H

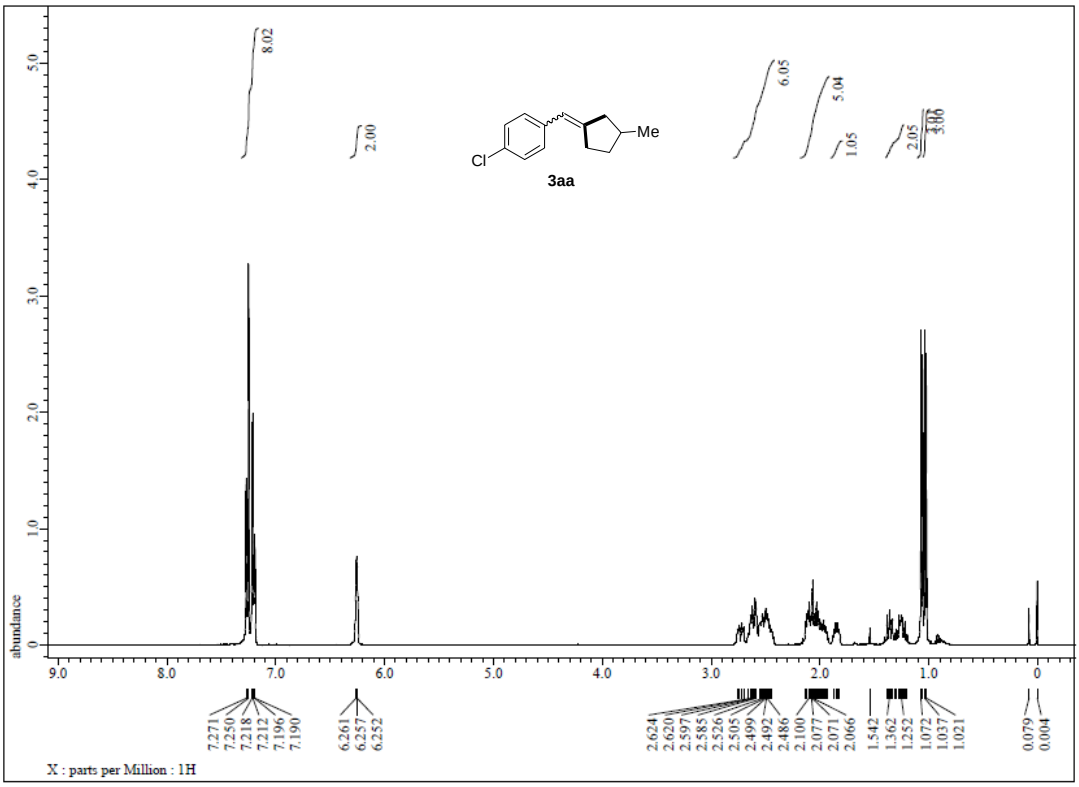

Me
Cl
3x
abundance
X : parts per Million : 13C

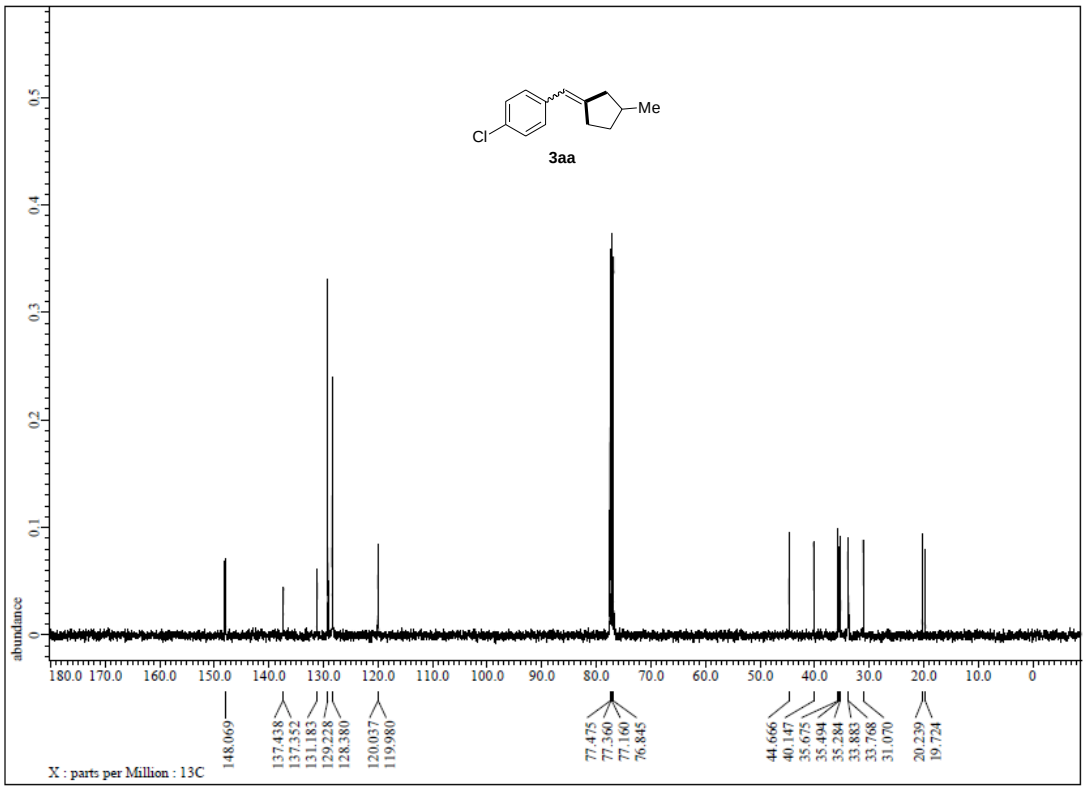

EtOOC
Me
3y
abundance
X : parts per Million : 1H

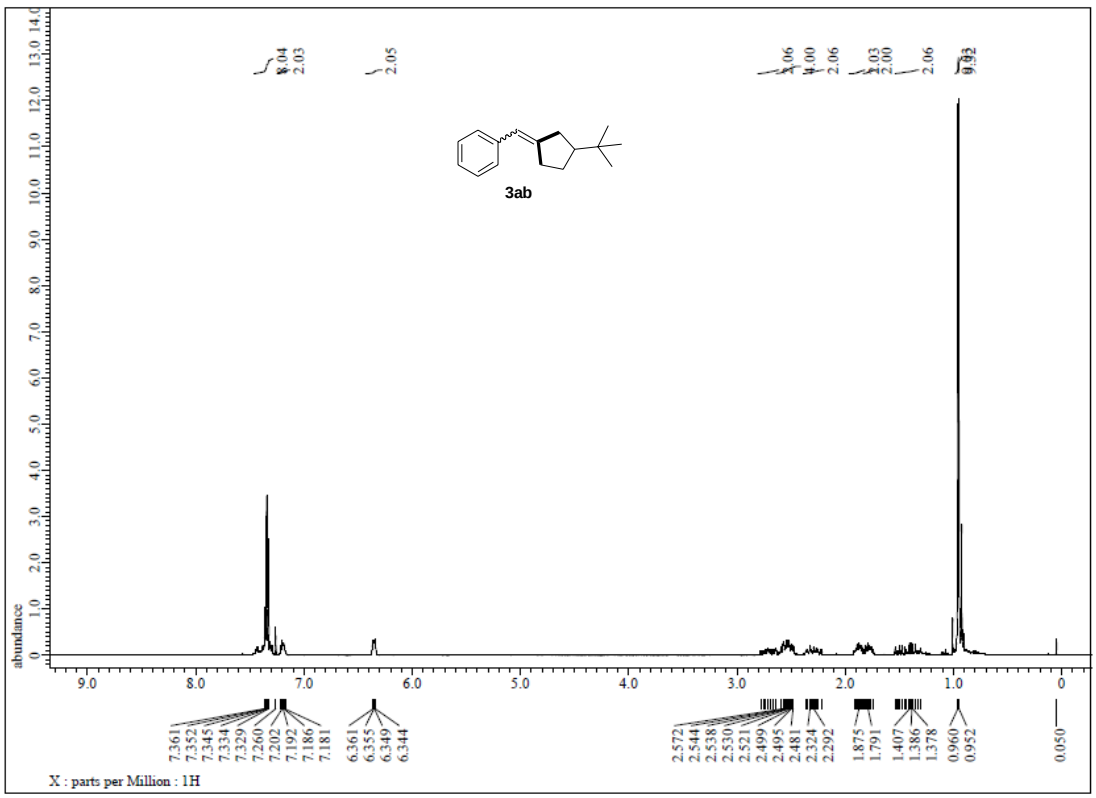

EtOOC
Me
3y
abundance
X : parts per Million : 13C

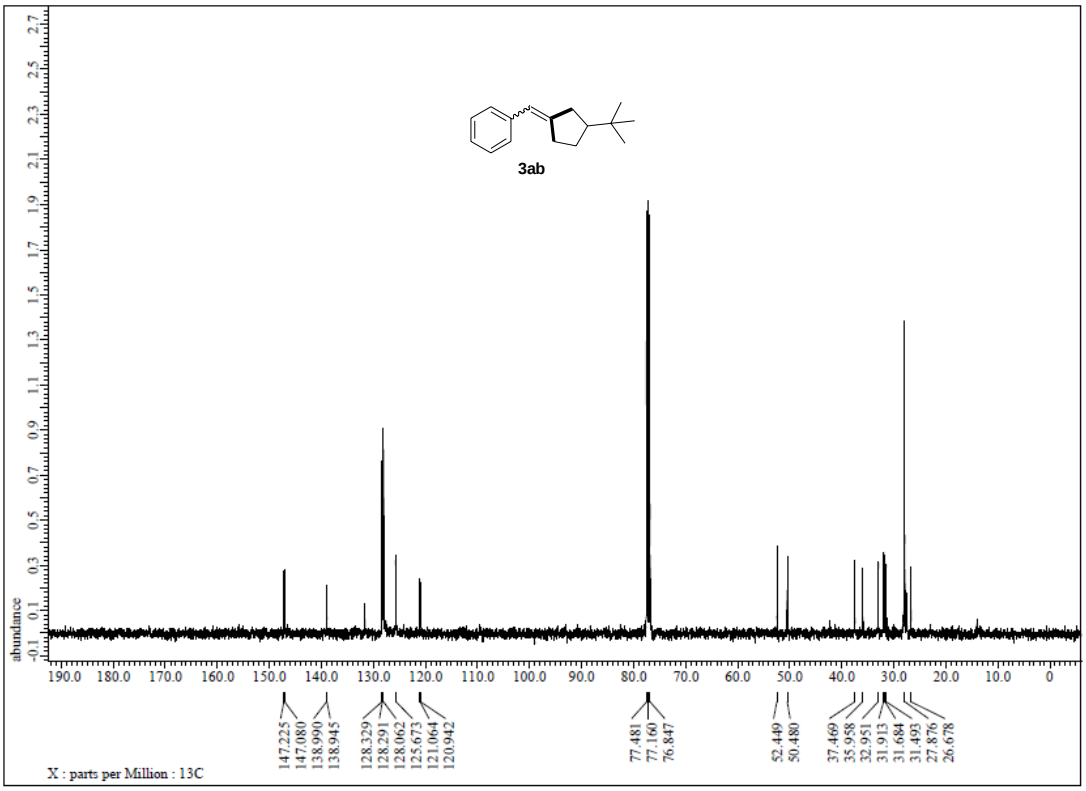

Me
3z
abundance
X : parts per Million : 1H

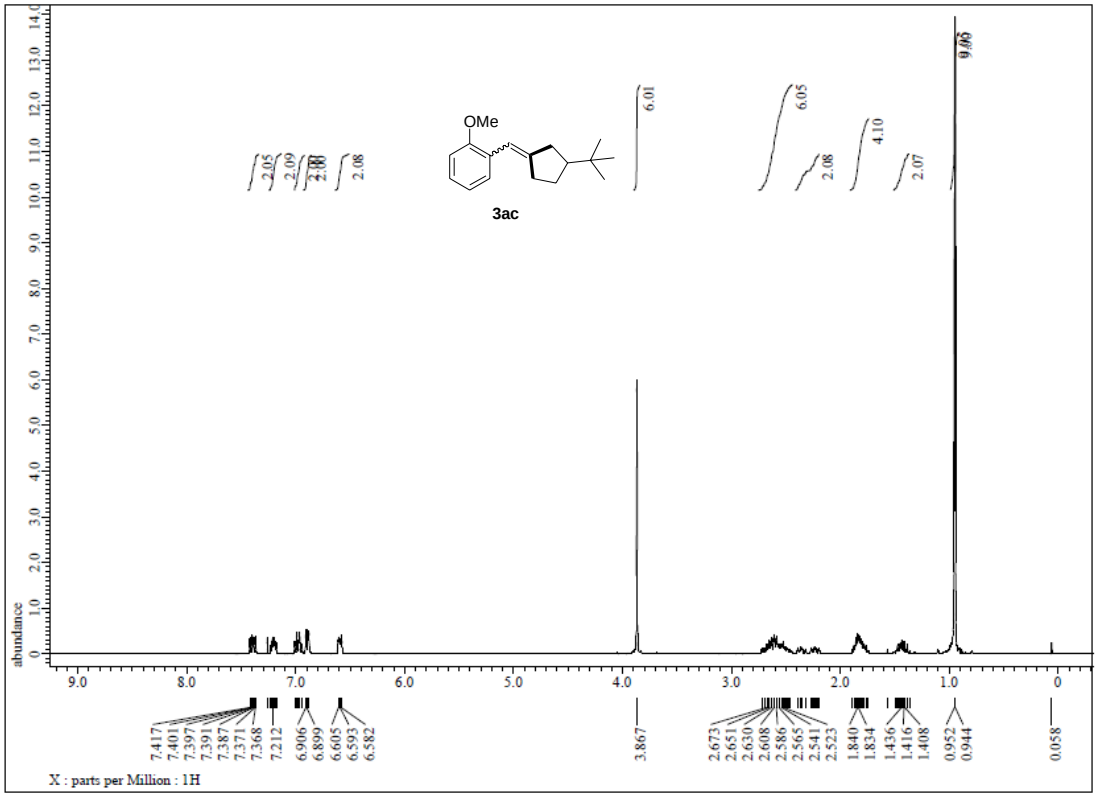

Me
3z
abundance
X : parts per Million : 13C

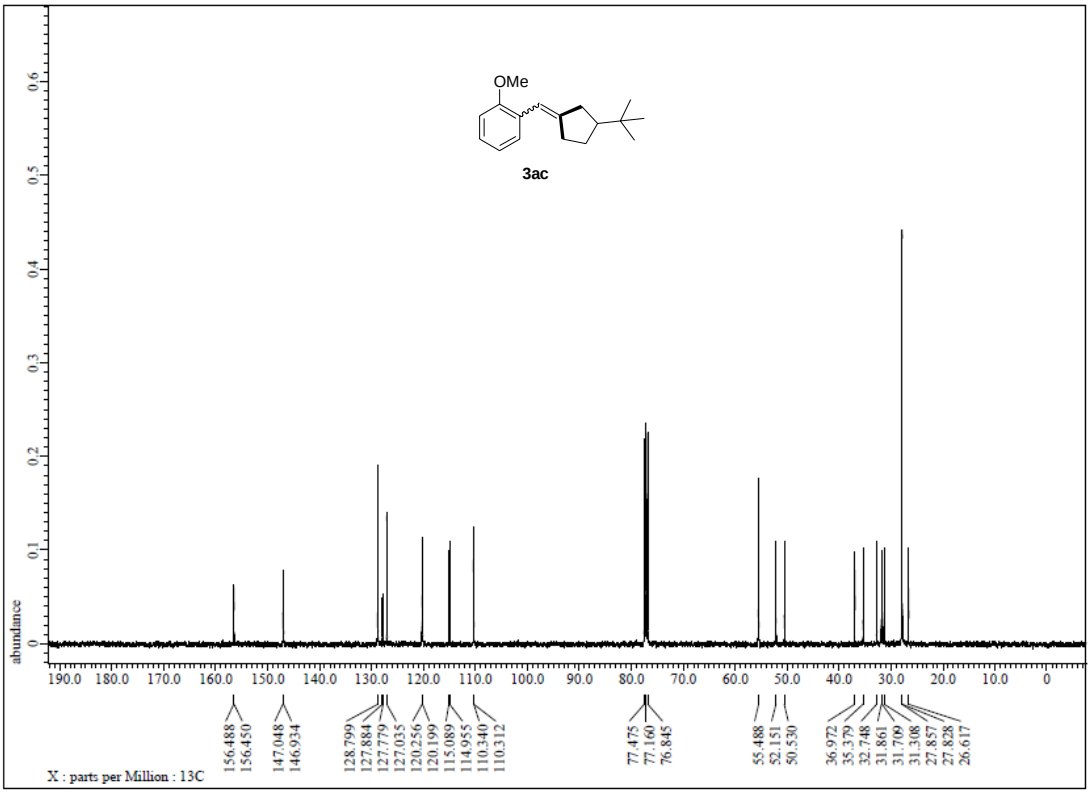

8.02
2.00
6.05
5.04
1.05
Cl
Me
3aa
abundance
X : parts per Million : 1H

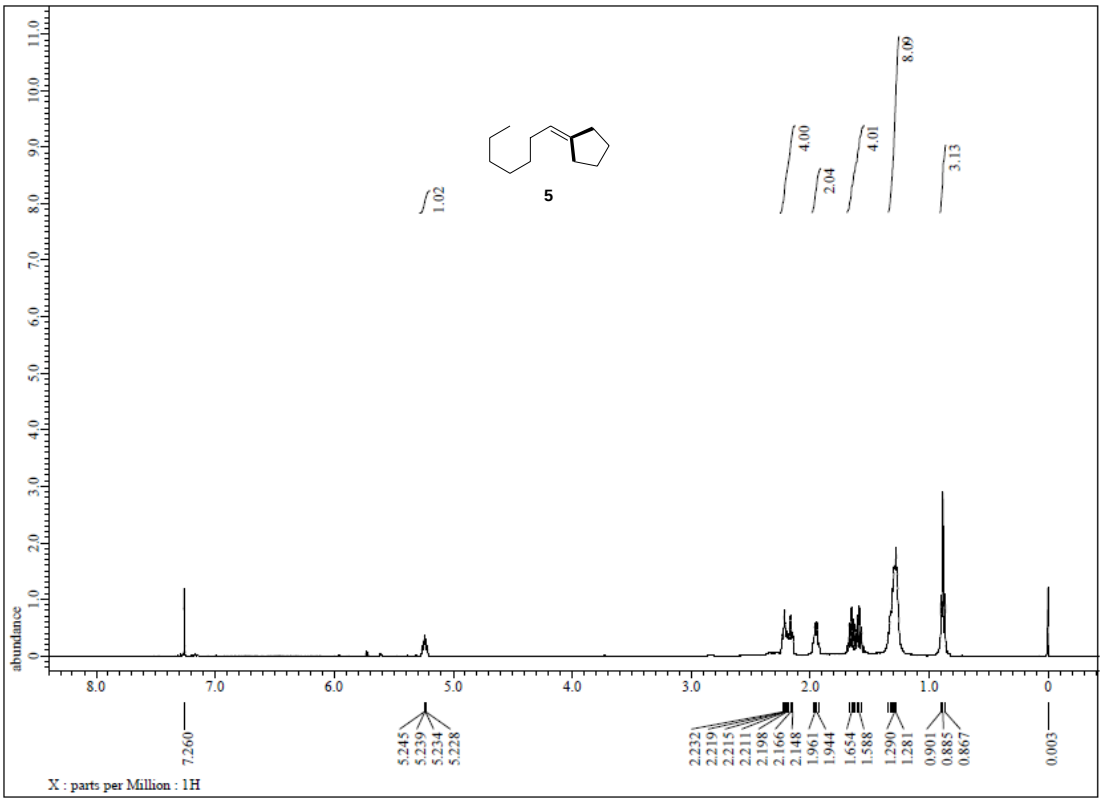

Cl
Me
3aa
abundance
X : parts per Million : 13C

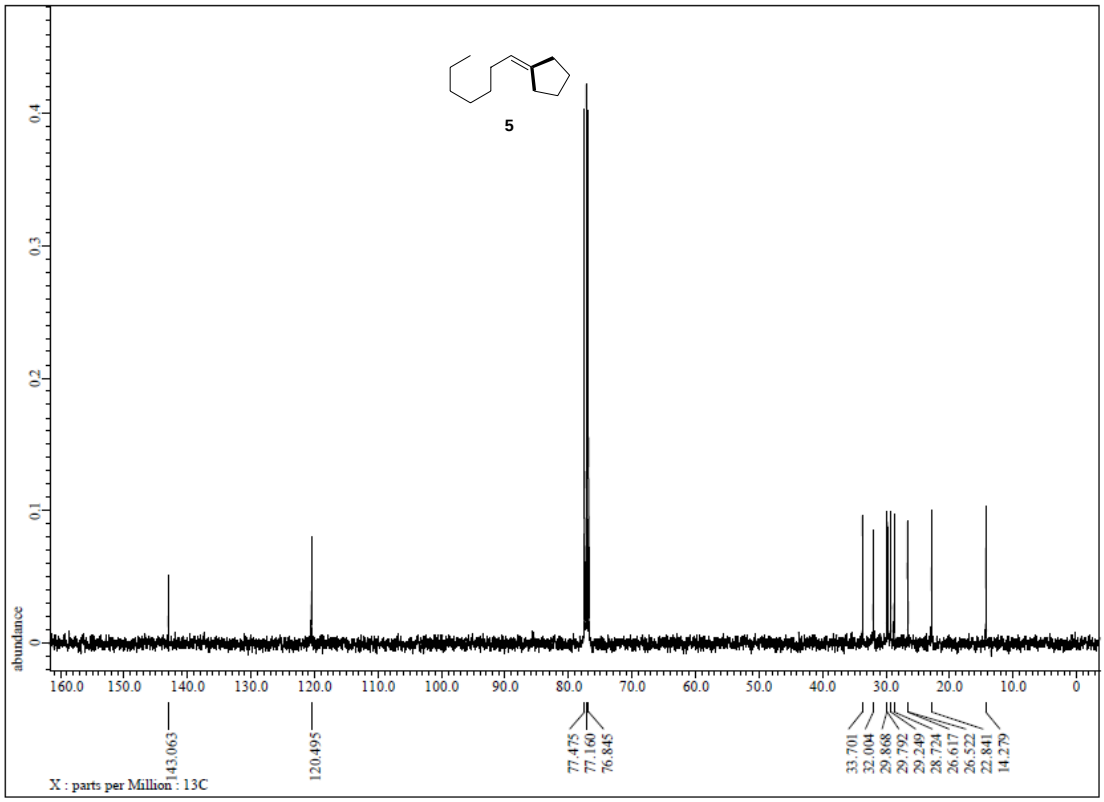

3ab
abundance
X : parts per Million : 1H

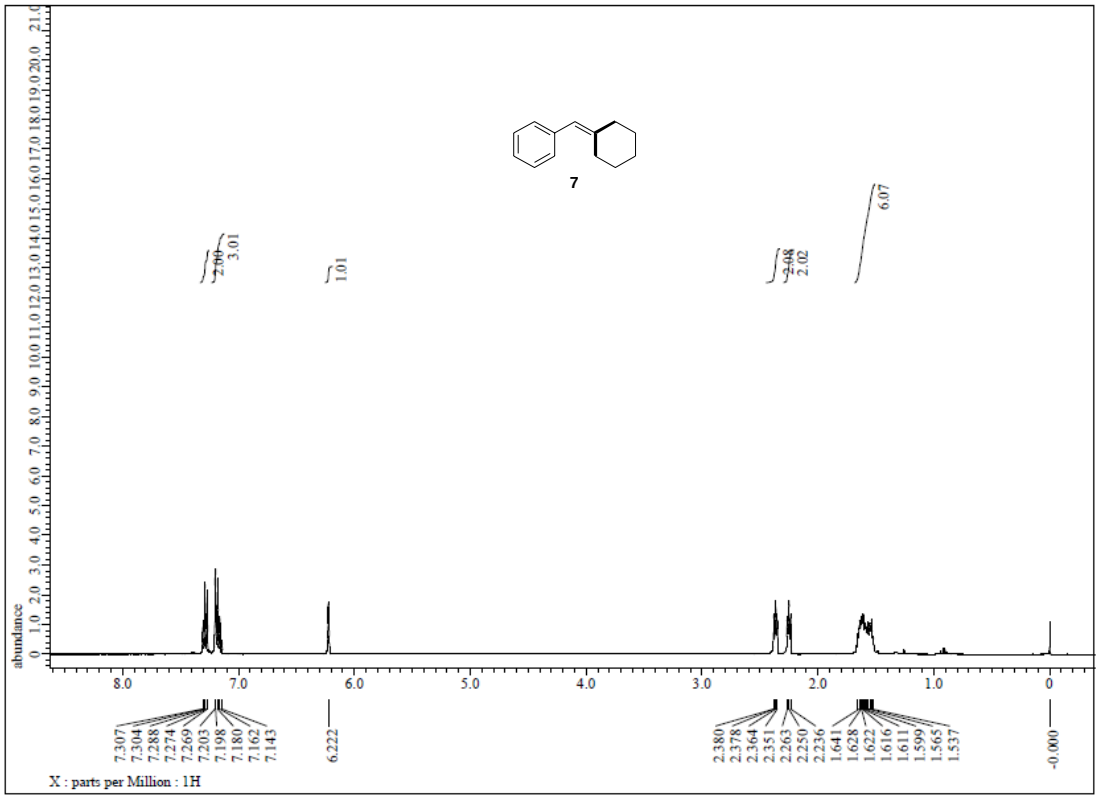

3ab
abundance
X : parts per Million : 13C
147.225
147.080
138.990
138.945
128.329
128.291
128.062
125.673
121.064
120.942
77.481
77.160
76.847
52.449
50.480
37.469
35.958
32.951
31.913
31.684
31.493
27.876
26.678

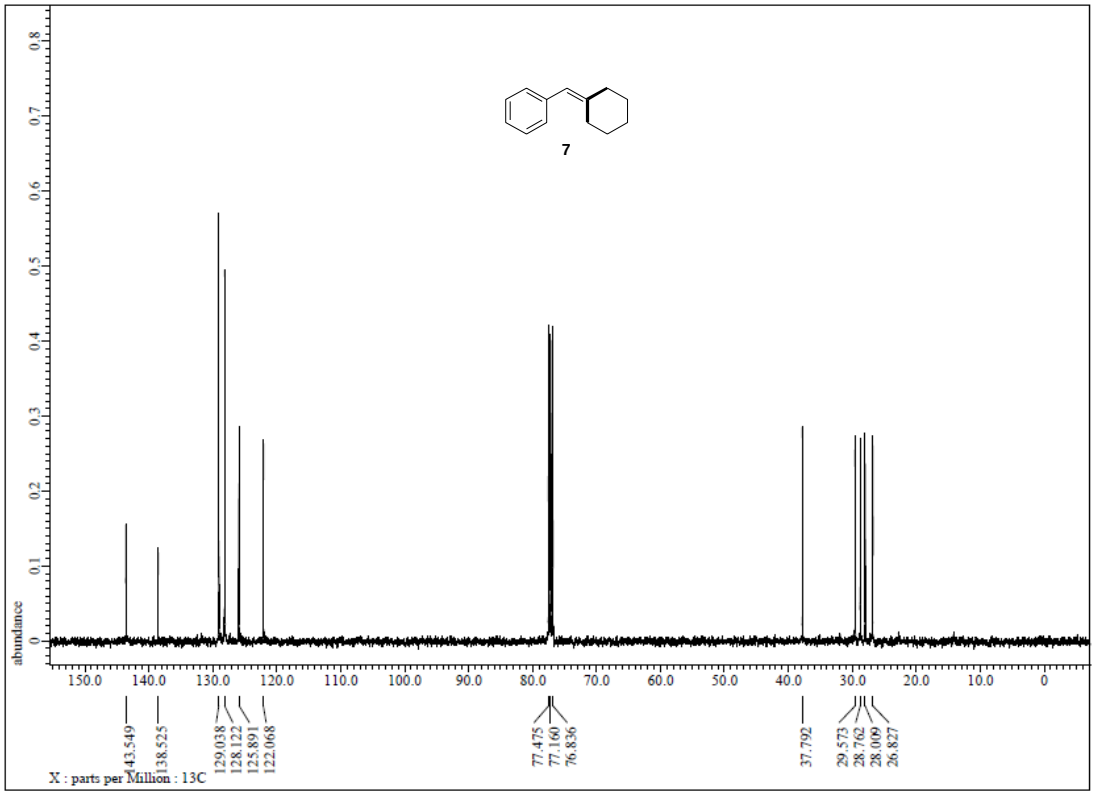

OMe
3ac
6.01
6.05
2.08
4.10
2.07
abundance
X : parts per Million : 1H
7.417
7.401
7.397
7.391
7.387
7.371
7.368
7.212
6.906
6.899
6.605
6.593
6.582
3.867
2.673
2.651
2.630
2.608
2.586
2.565
2.541
2.523
1.840
1.834
1.436
1.416
1.408
0.952
0.944
0.058

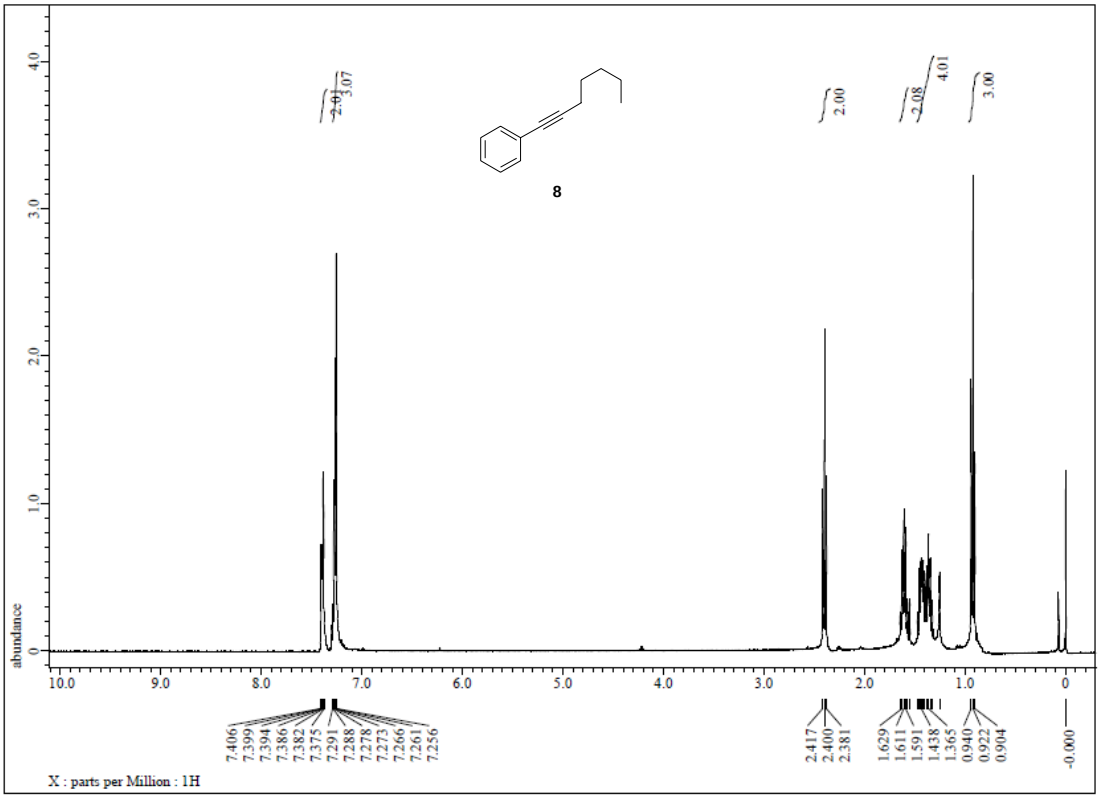

OMe
3ac
abundance
X : parts per Million : 13C

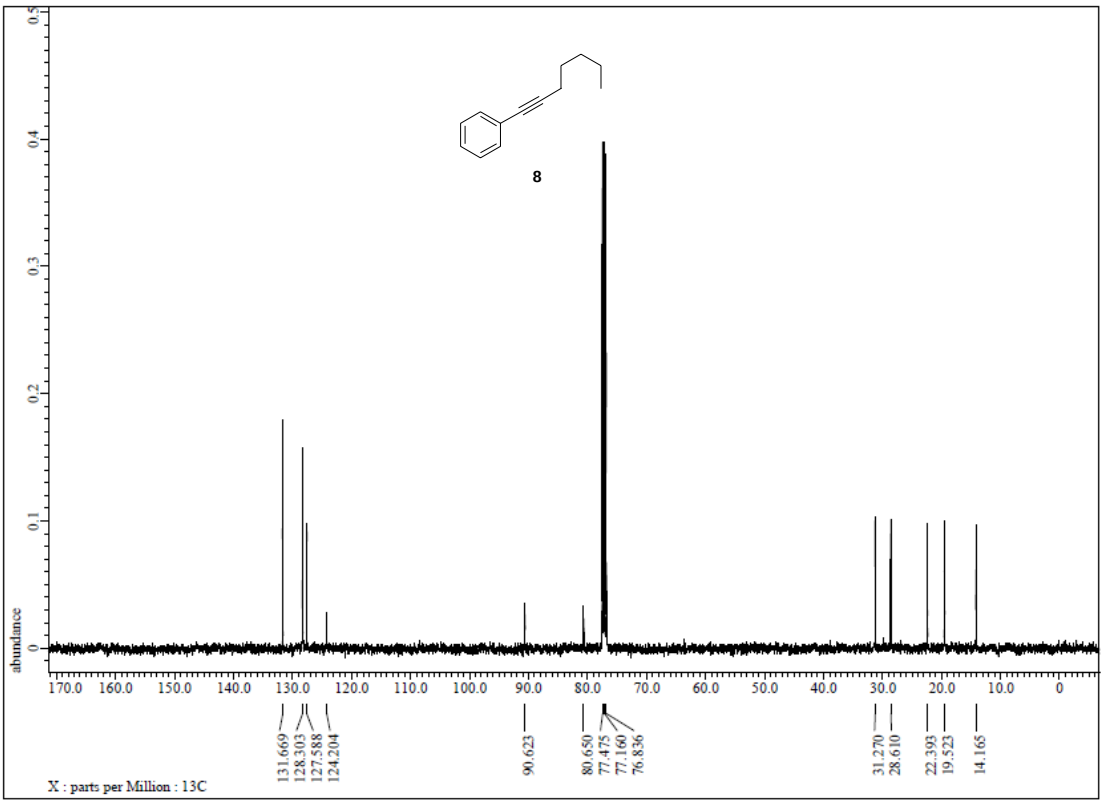

5
1.02
4.00
2.04
4.01
8.09
3.13
abundance
X : parts per Million : 1H

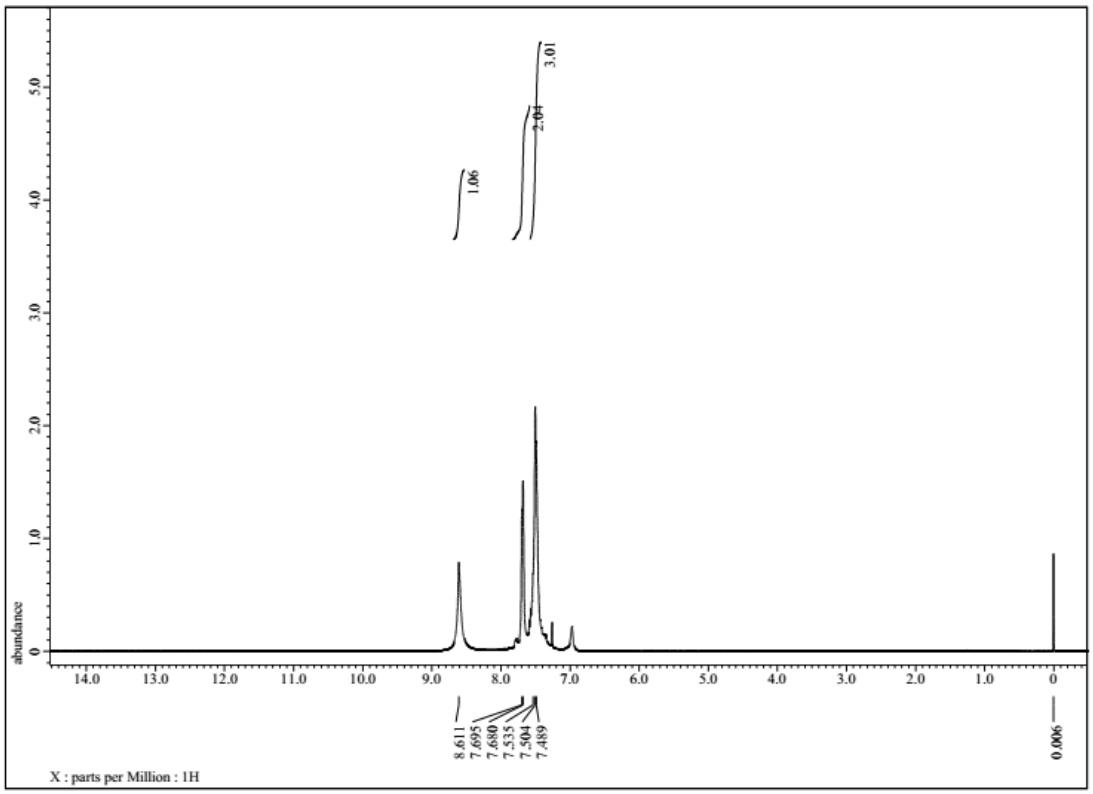

5
abundance
X : parts per Million : 13C
143.063
120.495
77.475
77.160
76.845
33.701
32.004
29.868
29.792
29.249
28.724
26.617
26.522
22.841
14.279


7
abundance
X : parts per Million : 1H
3.01
2.00
1.01
2.02
6.07
7.307
7.304
7.288
7.274
7.269
7.203
7.198
7.180
7.162
7.143
6.222
2.380
2.378
2.364
2.351
2.263
2.250
2.236
1.641
1.628
1.622
1.616
1.611
1.599
1.565
1.537
-0.000


7
abundance
143.549
138.525
129.038
128.122
125.891
122.068
77.475
77.160
76.836
37.792
29.573
28.762
28.009
26.827
X : parts per Million : 13C


8
2.01
3.07
2.00
2.08
4.01
3.00
abundance
7.406
7.399
7.394
7.386
7.382
7.375
7.291
7.288
7.278
7.273
7.266
7.261
7.256
2.417
2.400
2.381
1.629
1.611
1.591
1.438
1.365
0.940
0.922
0.904
-0.000
X : parts per Million : 1H


8
abundance
X : parts per Million : 13C
131.669
128.303
127.588
124.204
90.623
80.650
77.475
77.160
76.836
31.270
28.610
22.393
19.523
14.165


abundance
X : parts per Million : 1H
1.06
2.04
3.01
8.611
7.695
7.680
7.535
7.504
7.489
0.006