

Supporting Information

Ni-Catalyzed Regio- and Stereoselective Addition of Arylboronic Acids to Terminal Alkynes with Directing Group Tether

*Madala Hari Babu, Gadi Ranjith Kuma, Ruchir Kant and Maddi Sridhar Reddy**

Table of Contents

General Information.....	S2
Optimization Studies.....	S2-S4
Experimental Procedures and data of the compounds.....	S5-S30
X-Ray Crystallography data.....	.S31-S32
References.....	S32-S34
Copy of ¹ H and ¹³ C NMR Spectra.....	S35-S91

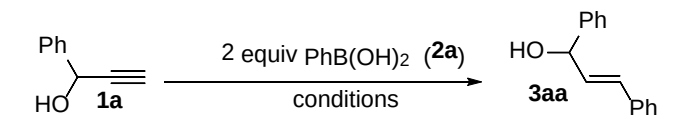
General Information: All reagents and solvents were purchased from commercial sources and used without purification. NMR spectra were recorded with a 400 or 500 MHz spectrometer for ^1H NMR, 100 or 125 MHz for ^{13}C NMR spectroscopy. Chemical shifts are reported relative to the residual signals of deuterated solvent CDCl_3 for ^1H and ^{13}C NMR spectroscopy. Multiplicities are reported as follows: singlet (s), doublet (d), doublet of doublets (dd), doublet of triplets (dt), triplet (t), quartet (q), multiplet (m). HRMS were recorded by using Q-TOF mass spectrometer. Column chromatography was performed with silica gel (100–200 mesh) as the stationary phase. All reactions were monitored by using TLC. The purity and characterization of compounds were further established by using HRMS.

Optimization Studies

Optimization studies were conducted on the conversion of **1a** to **3aa** (Table 1). Initially, we chose few reported conditions which were used for hydro/carboarylation of internal alkynes. We reasoned that this would unveil, apart from guiding us to achieve new conditions for our intended transformation, why most of the reported conditions were not tested on terminal alkynes. Thus, when we employed Marinelli's conditions^{1a} (using in situ generated $\text{Pd}(0)$) on **1a** in presence of 2 equivalents of $\text{PhB}(\text{OH})_2$ (entry 1), as predicted, no desired product was detected (entry 1). Direct use of $\text{Pd}(0)$ source $\text{Pd}(\text{PPh}_3)_4$ ^{1b} produced the desired product but in traces in spite of complete consumption of the starting material (entry 2). Use of in situ generated $\text{Ni}(\text{I})$ catalyst ($\text{Ni}(\text{acac})_2$ with PPh_3 and Cs_2CO_3)^{1c} resulted the formation of **3aa** in 12% (entry 3). In both cases only a single isomer with *syn* addition was detected. Note that the earlier usage of same nickel catalyst led to *anti*-carbometallation. Considering the economic viability compared to the palladium catalysts, we chose $\text{Ni}(\text{acac})_2$ for further standardization. No product was detected when we switched to the other solvents like DMF, DMSO, DMA, CH_3CN , DME along with $\text{Ni}(\text{acac})_2$ (entries 4-8). Use of other nickel catalysts like $\text{Ni}(\text{OAc})_2$, NiCl_2 and $\text{Ni}(\text{COD})_2$ along with PPh_3 and

Cs₂CO₃ showed no discernible effect (entries 9-10) indicating that the only Ni(acac)₂ in dioxane is capable of performing the title reaction.

Table 1. Optimization studies.^a

					
S. No	catalyst	ligand	additive	solvent (M)	yield (%)
1 ^b	Pd(OAc) ₂	PCy ₃	AcOH	EtOH (0.2)	--
2 ^c	Pd(PPh ₃) ₄	--	AcOH	1,4-dioxane (0.2)	8
3	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	1,4-dioxane (0.2)	12
4	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	DMF (0.2)	--
5	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	DMSO (0.2)	--
6	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	DMA (0.2)	--
7	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	CH ₃ CN (0.2)	--
8	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	DME (0.2)	--
9	Ni(OAc) ₂	PPh ₃	Cs ₂ CO ₃	1,4-dioxane (0.2)	--
10	NiCl ₂	PPh ₃	Cs ₂ CO ₃	1,4-dioxane (0.2)	--
11	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	1,4-dioxane (0.1)	18
12	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	1,4-dioxane/AcOH (0.2) ^d	--
13	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	1,4-dioxane/EtOH (0.2) ^d	40
14	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	1,4-dioxane/EtOH (0.1) ^d	57
15	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	EtOH (0.2)	--
16	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	1,4-dioxane/MeOH (0.1) ^d	27
17	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	1,4-dioxane/ <i>t</i> BuOH (0.1) ^d	--
18	Ni(acac) ₂	PPh ₃	Cs ₂ CO ₃	1,4-dioxane/EtOH (0.1) ^{d,e}	84

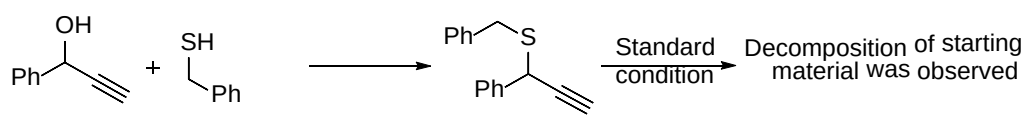
^aReaction conditions: 10 mol % catalyst, 10 mol% ligand, 20 mol% additive at 90 °C for 8-12h. ^b5 mol% Pd(OAc)₂, 10 mol% PCy₃ and 1.3 equiv AcOH at 60 °C for 6 h. ^c3 mol% Pd(PPh₃)₄, 10 mol% AcOH at 80 °C. ^ddioxane to co-solvent in 4:1 ratio. ^eportion wise addition of substrates to the stirred catalytic system over 4 h.

Accounting the carbophilic nature of organo nickel intermediates we decided to perform the reaction under low concentrations. We indeed observed the formation, with Ni(acac)₂, of a polymer like unidentified highly polar product as the major byproduct. Thus the reaction was done in 0.1M solution to avoid any such intermolecular reaction but the effort led to only a slight increase of the yield (entry 11). We then realised that a quick protodienickelation using protic environment may solve the problem. We thus used AcOH as a co-solvent (entry 12). This in fact proved to be detrimental to the reaction resulting the total recovery of the starting material. Pleasingly, addition of EtOH as the co-solvent increased the yield of **3aa** to 40% (entry 13). Dilution of the reaction to 0.1M further increased the yield to 57% (entry 14). However, replacement of EtOH with MeOH or

*t*BuOH, or use of EtOH as the sole solvent proved to be inappropriate to the reaction (entries 15-17). Delightedly, slow addition of substrate together with phenylboronic acid to the preformed catalyst (Ni(acac)₂, PPh₃ and Cs₂CO₃) in dioxane/EtOH system cleanly formed the product in 84% yield (entry 18). We reasoned that such a slow addition not only halted the polymerization of the substrate but also eliminated any dimerization of the reagent.

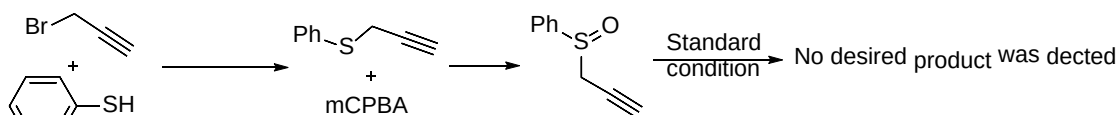
Additional Experiments:

Scheme 1.



Chem. Commun., **2006**, 3352-3354

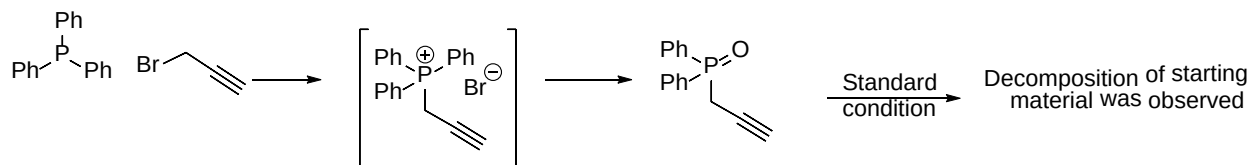
Scheme 2.



Tetrahedron Lett., 2015, **56**, 3144

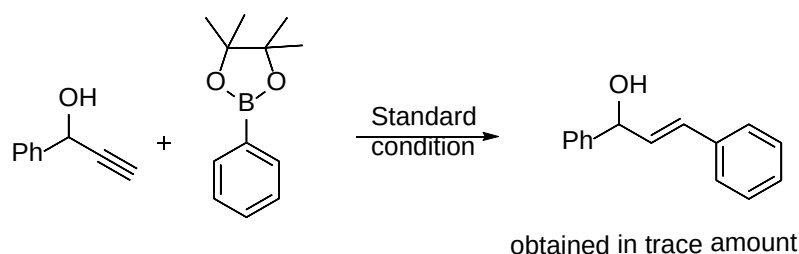
J. Med. Chem., 2016, **59**, 10692

Scheme 3.

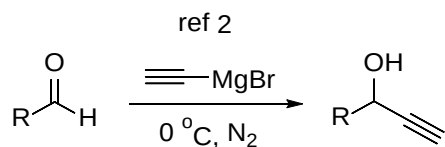


J. Heterocyclic Chem., 2015, **52**, 1876

Scheme 4.

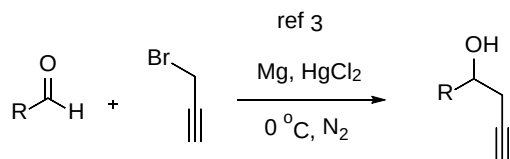


General procedure A for the synthesis of terminal Propargyl alcohols (1a, 1c, 1e-f):



To an ice-cold solution of aldehyde in anhydrous THF (5 mL/0.2 mmol) under nitrogen atmosphere was added ethynylmagnesium bromide (1.5 eq, 0.5 M in THF), and the reaction mixture was stirred for 2–4 h (monitored by TLC analysis) at 0 °C. Reaction mixture was diluted with saturated aq. NH_4Cl (10 mL) and extracted with ethyl acetate (3×10 mL). The combined organic layers were washed with brine (10 mL) and dried over Na_2SO_4 . Evaporation of the solvent and purification of the crude mixture by flash column chromatography (9:1, hexane: EtOAc) gave the corresponding propargylic alcohols in 80–95% yields.

General procedure B for the synthesis of terminal homo propargyl alcohols (1b, 1d):

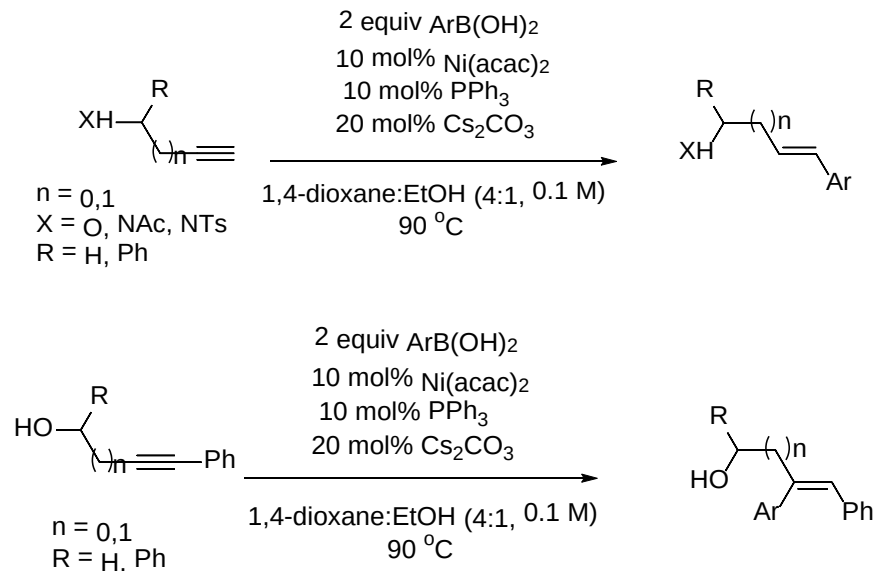


A 100 mL two-necked flask was charged with Mg (321 mg, 13.2 mmol, 1.32 equiv), HgCl_2 (2.7 mg, 0.10 mmol, 0.01 equiv) and dry diethyl ether (30 mL) and to this mixture was added propargyl bromide in a dropwise (2.86 g, 12.0 mmol, 1.20 equiv) manner. When the solution became homogeneous, the aldehyde (10.0 mmol, 1.00 equiv) was added again in dropwise manner. The reaction was quenched with a saturated NH_4Cl solution (30 mL) when TLC (hexanes/EtOAc: 5/1)

indicated that the benzaldehyde was completely consumed. The aqueous and organic layers were separated; the aqueous layer was extracted with diethyl ether (3×20 mL). The combined organic layers were dried over Na₂SO₄, concentrated under vacuum and purified by column chromatography (hexanes/EtOAc: 5/1).

The synthesis of other starting materials **1g**⁴, **1h**⁵, **1j-l**⁶, **1m**⁷, **1n**⁴, **1q**⁸, **1r**⁹, **1t**¹⁰, **5a**¹¹, **5b**¹⁰, **5c**¹¹, **5d**¹⁰, **5e**¹² were accomplished by following the reported procedures.

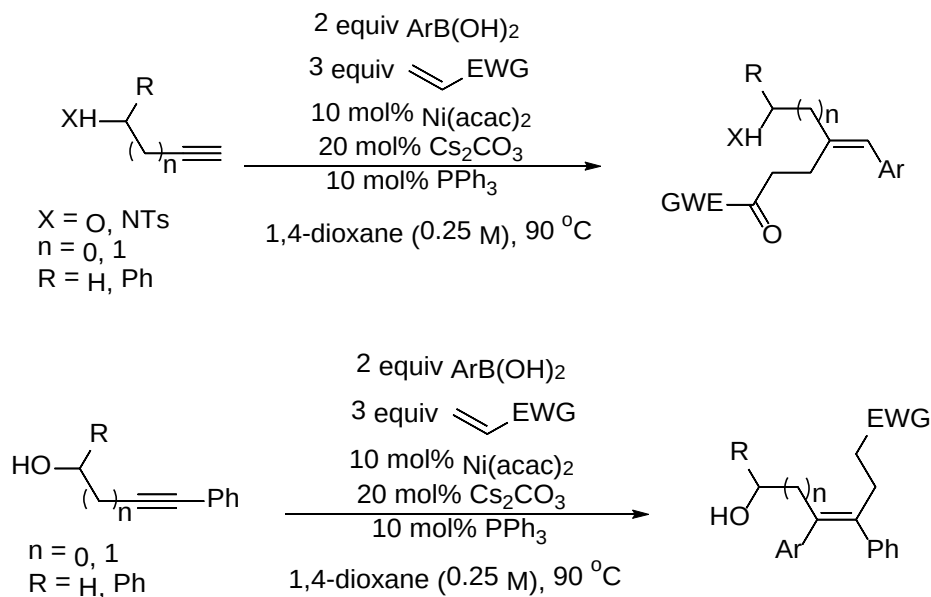
Typical procedure C for the synthesis of di and tri substituted olefins (3aa-aj, 3ba, 3bc-bf, 3bi-bj, 3ca-ra, 6a-g):



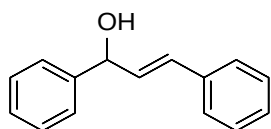
A mixture of 1 mmol alkyne (**1** or **5**) and 1 mmol boronic acid (**2**) in 5 ml of dioxane:EtOH (4:1) was added dropwise (for a period of 4h) to the stirred solution of Ni(acac)₂ (0.1 equiv, 25.8 mg), PPh₃ (0.1 equiv, 26.2 mg), Cs₂CO₃ (0.2 equiv, 65 mg) and boronic acid (1 equiv) in 5 ml of dioxane:EtOH (4:1) at 90 °C under N₂ atmosphere in 50 ml round bottom flask and the resultant mixture was stirred for 4-8 hr at the same temperature. After the completion of the reaction as indicated by TLC, the reaction mass was diluted with water (10 mL) and extracted with ethyl acetate (2x10 mL). The combined organic layers were washed with brine (20 mL) and were dried over anhydrous Na₂SO₄. After removal of solvent under reduced pressure the crude was purified

by column chromatography with silica gel (100-200 mesh) using 5-20% EtOAc/hexanes to get the required product.

General procedure D for the synthesis of tri and tetra substituted olefins with directing group tether (4a-k, 7a-7h):

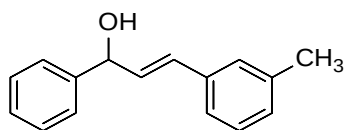


To a stirred solution of alkyne partner (**1** or **5**, 1 mmol), Ni(acac)₂ (0.1 equiv, 25.8 mg), PPh₃ (0.1 equiv, 26.2 mg), Cs₂CO₃ (0.2 equiv, 65 mg) and boronic acid (2 equiv) in 4 ml of dioxane in 50 ml round bottom flask, the electrophile (EVK or acrylonitrile, 3 equivalents) was added at rt under N₂ atmosphere. The reaction mixture was heated to 90 °C and allowed to stir at the same temperature for 4-8 hrs. After the completion of the reaction by TLC, the reaction mass was diluted with water (10 mL) and was extracted with ethyl acetate (2x10 mL). The combined organic layers were washed with brine (20 mL), and were dried over anhydrous Na₂SO₄. After removal of solvent under reduced pressure the crude was purified by column chromatography with silica gel (100-200 mesh) using 5-20% EtOAc/hexanes to get the required product.

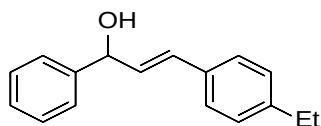


(E)-1,3-diphenylprop-2-en-1-ol (3aa)¹³: 0.176 g of **3aa** was obtained from 0.132 g (1 mmol) of **1a** following general procedure **C**. Yield 84%; white solid; *R_f* = 0.5 (SiO₂, 20% EtOAc/hexanes);

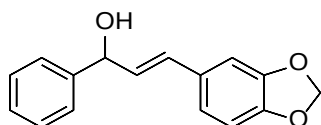
^1H NMR (400 MHz, CDCl_3) δ 7.46-7.44 (m, 2H), 7.41-7.36 (m, 4H), 7.34-7.29 (m, 3H), 7.27-7.23 (m, 1H), 6.70 (d, J = 15.8 Hz, 1H), 6.40 (dd, J = 15.8, 6.5 Hz, 1H), 5.39 (d, J = 6.5 Hz, 1H), 2.10 (bs, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 142.8, 136.6, 131.6, 130.6, 128.6, 128.6, 127.8, 126.7, 126.4, 75.1; IR (KBr) ν 3599, 3408, 3021, 1602, 2401, 1521, 1424, 1215, 670 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{15}\text{H}_{13}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+$ 193.1017, found 193.1011.



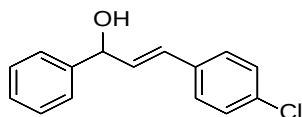
(E)-1-phenyl-3-(m-tolyl)prop-2-en-1-ol (3ab)¹⁴: 0.184 g of **3ab** was obtained from 0.132 g (1 mmol) of **1a** following general procedure **C**. Yield 81%; pale yellow oil; R_f = 0.5 (SiO_2 , 20% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.45-7.36 (m, 4H), 7.32-7.30 (m, 1H), 7.21-7.19 (m, 3H), 7.07-7.05 (m, 1H), 6.66 (dd, J = 15.8, 1.0 Hz, 1H), 6.38 (dd, J = 15.8, 6.5 Hz, 1H), 5.38 (d, J = 6.5 Hz, 1H), 2.33 (s, 3H), 2.07 (bs, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 142.9, 138.2, 136.5, 131.4, 130.7, 128.7, 128.5, 127.8, 127.4, 126.4, 123.9, 75.2, 21.4; IR (KBr) ν 3401, 3020, 2400, 1602, 1525, 1476, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{16}\text{H}_{15}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+$ 207.1174, found 207.1165.



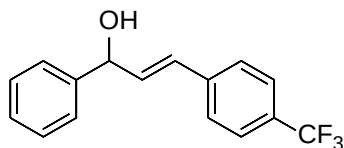
(E)-3-(4-ethylphenyl)-1-phenylprop-2-en-1-ol (3ac): 0.188 g of **3ac** was obtained from 0.132 g (1 mmol) of **1a** following general procedure **C**. Yield 79%; yellow oil; R_f = 0.5 (SiO_2 , 20% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.45-7.43 (m, 2H), 7.40-7.36 (m, 2H), 7.33-7.28 (m, 3H), 7.15 (d, J = 8.0 Hz, 2H), 6.67 (d, J = 15.7 Hz, 1H), 6.35 (dd, J = 15.7, 6.4 Hz, 1H), 5.38 (d, J = 6.4 Hz, 1H), 2.64 (q, J = 7.3 Hz, 2H), 2.13 (s, 1H), 1.24 (t, J = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.1, 143.0, 134.1, 130.7, 130.6, 128.6, 127.8, 127.8, 126.7, 126.4, 75.3, 28.7, 15.6; IR (KBr) ν 3400, 3020, 2400, 1603, 1523, 1384, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{17}\text{H}_{17}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+$ 221.1330, found 221.1321.



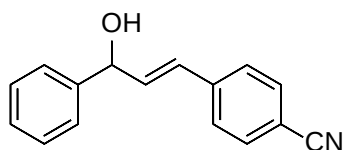
(E)-3-(1,3-dihydroisobenzofuran-5-yl)-1-phenylprop-2-en-1-ol (3ae)¹⁶: 0.180 g of **3ae** was obtained from 0.132 g (1 mmol) of **1a** following general procedure **C**. Yield 71%; brown oil; R_f = 0.4 (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.43-7.35 (m, 5H), 6.91 (s, 1H), 6.83-6.73 (m, 2H), 6.59 (d, J = 15.7 Hz, 1H), 6.21 (dd, J = 15.7, 6.6 Hz, 1H), 5.94 (s, 2H), 5.35 (d, J = 6.6 Hz, 1H), 2.04 (bs, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 148.1, 147.5, 143.0, 131.1, 130.4, 129.9, 128.7, 127.8, 126.4, 121.4, 108.3, 106.0, 101.1, 75.2; IR (KBr) ν 3400, 3020, 2400, 1603, 1525, 1423, 1384, 1215, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₆H₁₃O₂[(M + H)-H₂O]⁺ 237.0916, found 237.0911.



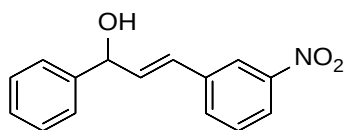
(E)-3-(4-chlorophenyl)-1-phenylprop-2-en-1-ol (3af)¹⁷: 0.192 g of **3af** was obtained from 0.132 g (1 mmol) of **1a** following general procedure **C**. Yield 79%; brown oil; R_f = 0.4 (SiO₂, 10% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.43-7.36 (m, 4H), 7.33-7.26 (m, 5H), 6.64 (dd, J = 15.9, 0.9 Hz, 1H), 6.36 (dd, J = 15.9, 6.2 Hz, 1H), 5.38 (d, J = 6.2 Hz, 1H), 2.07 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 142.7, 135.1, 133.5, 132.2, 129.3, 128.8, 128.8, 128.0, 127.9, 126.4, 75.1; IR (KBr) ν 3401, 3020, 2400, 1602, 1520, 1423, 1384, 1216, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₅H₁₂Cl[(M + H)-H₂O]⁺ 227.0628, found 227.0628.



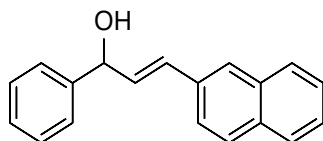
(E)-1-phenyl-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-ol (3ag)¹⁸: 0.214 g of **3ag** was obtained from 0.132 g (1 mmol) of **1a** following general procedure **C**. Yield 77%; yellow solid; R_f = 0.4 (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.56 (d, J = 8.3 Hz, 2H), 7.48-7.37 (m, 6H), 7.32 (t, J = 6.8 Hz, 1H), 6.73 (d, J = 15.8 Hz, 1H), 6.48 (dd, J = 15.8, 5.8 Hz, 1H), 5.41 (d, J = 5.8 Hz, 1H), 2.21 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 142.5, 140.2, 134.2, 129.6 (q, J = 32.8 Hz), 128.9, 128.8, 128.1, 126.8, 126.5, 125.6 (q, J = 3.8 Hz), 124.3 (d, J = 271.0 Hz), 74.9; IR (KBr) ν 3685, 3401, 3020, 2400, 1602, 1520, 1423, 1215, 669 cm⁻¹.



(E)-4-(3-hydroxy-3-phenylprop-1-en-1-yl)benzonitrile (3ah)¹⁵: 0.164 g of **3ah** was obtained from 0.132 g (1 mmol) of **1a** following general procedure **C**. Yield 70%; yellow solid; R_f = 0.4 (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.56 (d, J = 8.3 Hz, 2H), 7.44-7.36 (m, 6H), 7.33-7.30 (m, 1H), 6.71 (d, J = 15.8 Hz, 1H), 6.49 (dd, J = 15.8, 5.9 Hz, 1H), 5.40 (d, J = 5.9 Hz, 1H), 2.42 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 142.2, 141.3, 135.6, 132.4, 128.8, 128.3, 128.1, 127.1, 126.4, 118.9, 110.8, 74.6; IR (KBr) ν 3419, 1650, 1384, 1218, 1069, 771 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₆H₁₃NO[(M + H)-H₂O]⁺ 236.1075, found 236.1076.

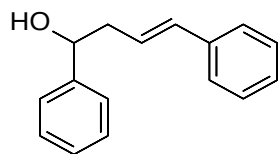


(E)-3-(3-nitrophenyl)-1-phenylprop-2-en-1-ol (3ai)¹⁹: 0.175 g of **3ai** was obtained from 0.132 g (1 mmol) of **1a** following general procedure **C**. Yield 69%; brown oil; R_f = 0.3 (SiO₂, 30% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 8.22 (t, J = 1.9 Hz, 1H), 8.07 (ddd, J = 8.2, 2.2, 0.9 Hz, 1H), 7.67 (d, J = 7.7 Hz, 1H), 7.49-7.30 (m, 6H), 6.76 (dd, J = 15.9, 1.0 Hz, 1H), 6.52 (dd, J = 15.9, 5.8 Hz, 1H), 5.43 (dd, J = 5.8, 1.0 Hz, 1H), 2.04 (bs, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 148.6, 142.2, 138.5, 134.8, 132.4, 129.5, 128.8, 128.1, 127.7, 126.4, 122.2, 121.1, 74.6; IR (KBr) ν 3401, 3021, 2401, 1602, 1526, 1425, 1215, 671 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₅H₁₂NO₂[(M + H)-H₂O]⁺ 238.0868, found 238.0861.

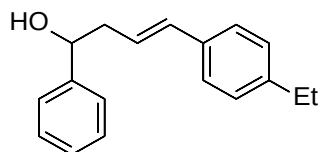


(E)-3-(naphthalen-1-yl)-1-phenylprop-2-en-1-ol (3aj)²⁰: 0.166 g of **3aj** was obtained from 0.132 g (1 mmol) of **1a** following general procedure **C**. Yield 64%; yellow solid; R_f = 0.4 (SiO₂, 10% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.80-7.75 (m, 4H), 7.59 (dd, J = 7.5, 1.7 Hz, 1H), 7.49-7.38 (m, 6H), 7.34-7.31 (m, 1H), 6.85 (d, J = 15.7, 1H), 6.52 (dd, J = 15.7, 6.5 Hz, 1H), 5.45 (d, J = 6.5 Hz, 1H), 2.05 (bs, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 142.8, 134.0, 133.5, 133.0,

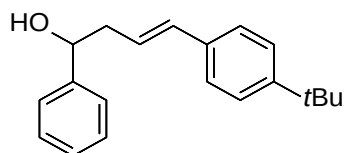
132.0, 130.6, 128.6, 128.2, 128.0, 127.7, 126.7, 126.4, 126.3, 125.9, 123.7, 75.1; IR (KBr) ν 3400, 3020, 2400, 1602, 1523, 1475, 1384, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{19}\text{H}_{15}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+$ 243.1174, found 243.1169.



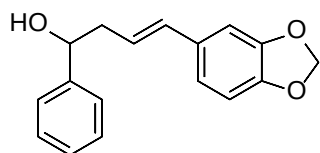
(E)-1,4-diphenylbut-3-en-1-ol (3ba)²¹: 0.136 g of **3ba** was obtained from 0.146 g (1 mmol) of **1b** following general procedure **C**. Yield 61%; white solid, R_f = 0.5 (SiO_2 , 10% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.34 (m, 6H), 7.30 (t, J = 7.5 Hz, 3H), 7.22 (t, J = 7.2 Hz, 1H), 6.51 (d, J = 16.0 Hz, 1H), 6.21 (dt, J = 16.0, 7.2 Hz, 1H), 4.82 (t, J = 6.2 Hz, 1H), 2.67 (t, J = 7.2 Hz, 2H), 2.08 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.0, 137.3, 133.4, 128.6, 128.5, 127.6, 127.3, 126.2, 126.0, 125.9, 73.8, 43.1; IR (KBr) ν 3401, 3020, 2400, 1601, 1514, 1422, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{16}\text{H}_{15}\text{O}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+$ 207.1174, found 207.1169.



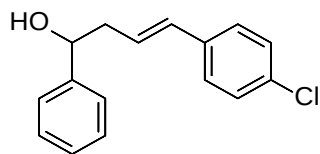
(E)-4-(4-ethylphenyl)-1-phenylbut-3-en-1-ol (3bc): 0.158 g of **3bc** was obtained from 0.146 g (1 mmol) of **1b** following general procedure **C**. Yield 63%; yellow solid, mp 58-60 $^\circ\text{C}$; R_f = 0.5 (SiO_2 , 10% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.40-7.34 (m, 4H), 7.31-7.25 (m, 3H), 7.13 (d, J = 7.9 Hz, 1H), 6.48 (d, J = 15.9 Hz, 1H), 6.15 (dt, J = 15.9, 7.3 Hz, 1H), 4.80 (t, J = 5.1 Hz, 1H), 2.68-2.60 (m, 4H), 2.07 (d, J = 2.8 Hz, 1H), 1.22 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.0, 143.6, 134.8, 133.4, 128.5, 128.1, 127.6, 126.2, 125.9, 124.9, 73.8, 43.2, 28.6, 15.6; IR (KBr) ν 3401, 3020, 2400, 1601, 1523, 1476, 1423, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{18}\text{H}_{19}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+$ 235.1487, found 235.1482.



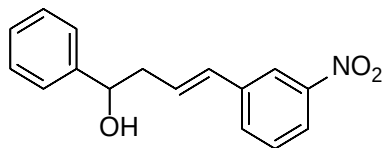
(E)-4-(4-(tert-butyl)phenyl)-1-phenylbut-3-en-1-ol (3bd): 0.168 g of **3bd** was obtained from 0.146 g (1 mmol) of **1b** following general procedure C. Yield 60%; pale yellow oil; R_f = 0.5 (SiO₂, 10% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.38-7.34 (m, 5H), 7.31-7.28 (m, 4H), 6.49 (d, J = 15.9 Hz, 1H), 6.15 (dt, J = 15.9, 7.3 Hz, 1H), 4.82-4.78 (m, 1H), 2.68-2.63 (m, 2H), 2.06 (d, J = 3.2 Hz, 1H), 1.31 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 151.0, 143.0, 133.8, 130.9, 130.4, 128.6, 127.8, 126.4, 125.6, 125.5, 75.2, 34.6, 31.3; IR (KBr) ν 3410, 3020, 2400, 1602, 1524, 1423, 1215, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₀H₂₃[(M + H)-H₂O]⁺ 263.1800, found 263.1792.



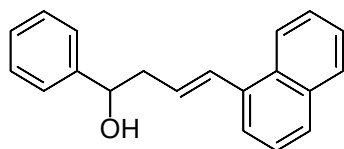
(E)-4-(1,3-dihydroisobenzofuran-5-yl)-1-phenylbut-3-en-1-ol (3be): 0.163 g of **3be** was obtained from 0.146 g (1 mmol) of **1b** following general procedure C. Yield 61%; white solid, mp 64-66 °C; R_f = 0.5 (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.40-7.34 (m, 4H), 7.31-7.27 (m, 1H), 6.88 (d, J = 1.3 Hz, 1H), 6.78-6.72 (m, 2H), 6.41 (d, J = 15.8 Hz, 1H), 6.02 (dt, J = 15.8, 7.2 Hz, 1H), 5.93 (s, 2H), 4.79 (dd, J = 7.2, 5.5 Hz, 1H), 2.65-2.61 (m, 2H), 2.07 (bs, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 148.0, 147.0, 144.0, 133.0, 131.8, 128.5, 127.7, 125.9, 124.2, 120.8, 108.3, 105.6, 101.1, 73.9, 43.0; IR (KBr) ν 3685, 3401, 3020, 2400, 1602, 1523, 1476, 1424, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₇H₁₅O₂[(M + H)-H₂O]⁺ 251.1072, found 251.1072.



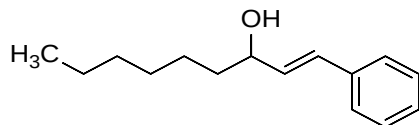
(E)-4-(4-chlorophenyl)-1-phenylbut-3-en-1-ol (3bf): 0.147 g of **3bf** was obtained from 0.146 g (1 mmol) of **1b** following general procedure C. Yield 57%; white solid, mp 80-82 °C; R_f = 0.4 (SiO₂, 10% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.38-7.35 (m, 3H), 7.33-7.27 (m, 2H), 7.59 (s, 4H), 6.44 (d, J = 16.0 Hz, 1H), 6.19 (dt, J = 16.0, 7.3 Hz, 1H), 4.82 (d, J = 6.2 Hz, 1H), 2.68-2.64 (m, 2H), 2.03 (bs, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 143.9, 135.8, 133.0, 132.2, 128.8, 128.6, 127.8, 127.4, 126.8, 125.9, 73.9, 43.0; IR (KBr) ν 3410, 3020, 2400, 1601, 1475, 1215, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₆H₁₄Cl[(M + H)-H₂O]⁺ 241.0784, found 241.0770.



(E)-4-(3-nitrophenyl)-1-phenylbut-3-en-1-ol (3bi): 0.145 g of **3bi** was obtained from 0.146 g (1 mmol) of **1b** following general procedure C. Yield 54%; colorless oil; R_f = 0.30 (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 8.18 (t, J = 1.9 Hz, 1H), 8.06-8.04 (m, 1H), 7.62 (d, J = 7.9 Hz, 1H), 7.45 (t, J = 7.9 Hz, 1H), 7.39-7.36 (m, 4H), 7.33-7.28 (m, 1H), 6.53 (d, J = 15.8 Hz, 1H), 6.39 (dt, J = 15.8, 7.1 Hz, 1H), 4.87-4.84 (m, 1H), 2.73-2.69 (m, 2H), 2.00 (d, J = 2.7 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 148.7, 143.8, 139.2, 132.1, 131.0, 129.8, 129.5, 128.7, 128.0, 125.9, 121.9, 120.8, 73.9, 42.9; IR (KBr) ν 3685, 3435, 3020, 1602, 1529, 1352, 1020, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₆H₁₅NNaO₃[M + Na]⁺ 292.0950, found 292.2941.

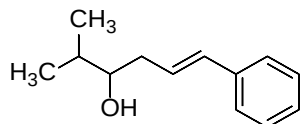


(E)-4-(naphthalen-1-yl)-1-phenylbut-3-en-1-ol (3bj): 0.170 g of **3bj** was obtained from 0.146 g (1 mmol) of **1b** following general procedure C. Yield 62%; pale yellow solid; mp 110-112 °C; R_f = 0.4 (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.80-7.75 (m, 3H), 7.69 (s, 1H), 7.56 (d, J = 8.4 Hz, 1H), 7.47-7.36 (m, 6H), 7.32-7.29 (m, 1H), 6.67 (d, J = 15.8 Hz, 1H), 6.34 (dt, J = 15.8, 7.4 Hz, 1H), 4.86 (t, J = 6.3 Hz, 1H), 2.73 (t, J = 6.8 Hz, 2H), 2.10 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 144.0, 134.8, 133.7, 133.6, 133.0, 128.6, 128.2, 128.0, 127.7, 126.5, 126.3, 126.0, 125.9, 125.8, 123.6, 73.9, 43.3; IR (KBr) ν 3684, 3435, 3019, 2400, 1600, 1521, 1025, 625 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₀H₁₈NaO[M + Na]⁺ 297.1255, found 297.1251.

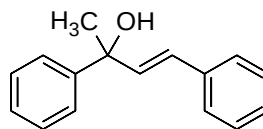


(E)-1-phenylnon-1-en-3-ol (3ca)²²: 0.167 g of **3ca** was obtained from 0.140 g (1 mmol) of **1c** following general procedure C. Yield 77%; pale yellow oil; R_f = 0.5 (SiO₂, 10% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.39 (d, J = 7.9 Hz, 2H), 7.31 (t, J = 7.9 Hz, 2H), 7.23 (t, J = 7.6 Hz, 1H), 6.57 (d, J = 15.9 Hz, 1H), 6.22 (dd, J = 15.9, 6.8 Hz, 1H), 4.27 (q, J = 6.8 Hz, 1H), 1.67-

1.57 (m, 3H), 1.42-1.30 (m, 8H), 0.89 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 136.9, 132.7, 130.2, 128.6, 127.6, 126.5, 73.1, 37.5, 31.9, 29.3, 25.5, 22.7, 14.1; IR (KBr) ν 3401, 3019, 2929, 2400, 1600, 1384, 1215, 669 cm^{-1} .

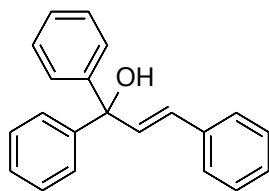


(E)-2-methyl-6-phenylhex-5-en-3-ol (3da)²³: 0.199 g of **3da** was obtained from 0.112 g (1 mmol) of **1d** following general procedure **C**. Yield 63%; yellow oil; $R_f = 0.5$ (SiO_2 , 10% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.36 (d, $J = 7.1$ Hz, 2H), 7.30 (t, $J = 7.6$ Hz, 2H), 7.21 (t, $J = 7.1$ Hz, 1H), 6.49 (d, $J = 16.0$ Hz, 1H), 6.25 (dt, $J = 16.0, 6.7$ Hz, 1H), 3.51-3.46 (m, 1H), 2.49-2.43 (m, 1H), 2.33-2.26 (m, 1H), 1.78-1.70 (m, 1H), 0.98 (d, $J = 2.8$ Hz, 3H), 0.96 (d, $J = 2.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 137.4, 133.1, 128.6, 127.3, 127.0, 126.2, 76.0, 38.2, 33.3, 18.9,



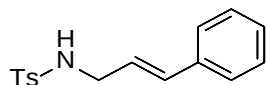
17.6; IR (KBr) ν 3401, 3018, 1384, 1215, 1068, 669 cm^{-1} .

(E)-2,4-diphenylbut-3-en-2-ol (3ea)²⁴: 0.181 g of **3ea** was obtained from 0.146 g (1 mmol) of **1e** following general procedure **C**. Yield 81%; white solid; $R_f = 0.6$ (SiO_2 , 10% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.54-7.52 (m, 2H), 7.41-7.22 (m, 8H), 6.66 (d, $J = 16.1$ Hz, 1H), 6.52 (d, $J = 16.1$ Hz, 1H), 2.03 (s, 1H), 1.78 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 146.7, 136.8, 136.4, 128.6, 128.4, 127.8, 127.7, 127.1, 126.6, 125.3, 74.7, 29.9; IR (KBr) ν 3400, 3020, 2400, 1602, 1523, 1422, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{16}\text{H}_{15}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+$ 207.1174, found 207.1165.

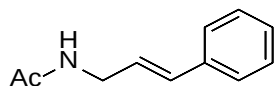


(E)-1,1,3-triphenylprop-2-en-1-ol (3fa)²⁵: 0.220 g of **3fa** was obtained from 0.208 g (1 mmol) of **1f** following general procedure **C**. Yield 77%; yellow solid; $R_f = 0.4$ (SiO_2 , 20% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.46-7.42 (m, 6H), 7.37-7.23 (m, 9H), 6.84 (d, $J = 15.7$ Hz, 1H),

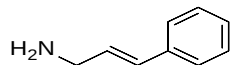
6.66 (d, $J = 15.7$ Hz, 1H), 2.44 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 146.1, 136.7, 135.3, 129.3, 128.7, 128.3, 127.8, 127.4, 127.0, 126.8, 79.5; IR (KBr) ν 3401, 3020, 2400, 1637, 1523, 1423, 1384, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{21}\text{H}_{17}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+$ 269.1330, found 269.1320.



N-cinnamyl-4-methylbenzenesulfonamide (3ga)²⁶: 0.200 g of **3ga** was obtained from 0.209 g (1 mmol) of **1g** following general procedure **C**. Yield 70%; white solid; $R_f = 0.5$ (SiO_2 , 30% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.78 (dt, $J = 8.2, 1.6$ Hz, 2H), 7.31-7.22 (m, 2H), 6.43 (d, $J = 15.9$ Hz, 1H), 6.01 (dt, $J = 15.9, 6.3$ Hz, 1H), 4.60 (t, $J = 5.9$ Hz, 1H), 3.75 (td, $J = 6.3, 1.4$ Hz, 1H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.6, 137.2, 136.2, 133.1, 129.8, 128.6, 128.0, 127.3, 126.5, 124.2, 45.5, 21.5; IR (KBr) ν 3399, 3020, 2922, 2400, 1523, 1423, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{16}\text{H}_{17}\text{NNaO}_2\text{S}[\text{M} + \text{Na}]^+$ 310.0878, found 310.0880.

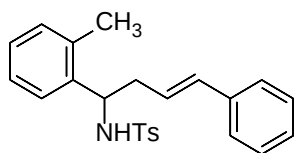


N-cinnamylacetamide (3ha)²⁷: 0.126 g of **3ha** was obtained from 0.097 g (1 mmol) of **1h** following general procedure **C**. Yield 72%; white solid; $R_f = 0.3$ (SiO_2 , 10% MeOH/DCM); ^1H NMR (400 MHz, CDCl_3) δ 7.35-7.28 (m, 4H), 7.25-7.21 (m, 1H), 6.51 (d, $J = 15.9$ Hz, 1H), 6.18 (dt, $J = 15.9, 6.3$ Hz, 1H), 5.75 (bs, 1H), 4.02 (td, $J = 6.0, 1.4$ Hz, 2H), 2.01 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.1, 36.6, 132.2, 128.6, 127.8, 126.4, 125.6, 41.7, 23.3; IR (KBr) ν 3448, 3020, 2400, 1667, 1519, 1476, 1423, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{11}\text{H}_{13}\text{NO}[\text{M} + \text{H}]^+$ 176.1075, found 176.1070.

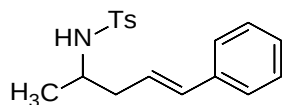


(E)-3-phenylprop-2-en-1-amine (3ia)²⁸: 0.081 g of **3ia** was obtained from 0.055 g (1 mmol) of **1i** following general procedure **C**. Yield 61%; white solid; $R_f = 0.5$ (SiO_2 , 10% MeOH/DCM); ^1H NMR (400 MHz, CDCl_3) δ 7.37-7.35 (m, 2H), 7.30 (t, $J = 7.6$ Hz, 2H), 7.21 (tt, $J = 7.6, 1.2$ Hz, 1H), 6.50 (d, $J = 15.8$ Hz, 1H), 6.31 (dt, $J = 15.8, 5.9$ Hz, 1H), 3.48 (s, 2H), 1.90 (bs, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 137.2, 130.9, 129.8, 128.6, 127.4, 126.3, 44.2; IR (KBr) ν 3686, 3401,

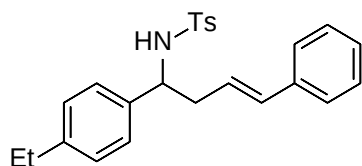
3020, 2400, 1601, 1523, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_9\text{H}_{12}\text{N}[\text{M} + \text{H}]^+$ 134.0970, found 134.0957.



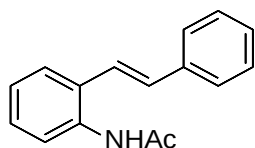
(E)-4-methyl-N-(4-phenyl-1-(o-tolyl)but-3-en-1-yl)benzenesulfonamide (3ja): 0.277 g of **3ja** was obtained from 0.313 g (1 mmol) of **1j** following general procedure C. Yield 71%; white solid, mp 122-124°C; R_f = 0.2 (SiO_2 , 20% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.53 (d, J = 8.2 Hz, 2H), 7.30-7.26 (m, 2H), 7.23-7.20 (m, 3H), 7.13-7.11 (m, 1H), 7.07-7.02 (m, 5H), 6.37 (d, J = 15.8 Hz, 1H) 5.84 (dt, J = 15.8, 7.2 Hz, 1H), 5.14 (d, J = 6.5 Hz, 1H), 4.75 (q, J = 6.8 Hz, 1H), 2.85-2.54 (m, 2H), 2.32 (s, 3H), 2.22 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.1, 138.9, 137.5, 136.9, 134.6, 134.0, 130.5, 129.4, 128.6, 127.6, 127.2, 127.1, 126.3, 126.3, 126.0, 124.7, 53.7, 40.6, 21.5, 19.2; IR (KBr) ν 3685, 3377, 3020, 1521, 1422, 1159, 1093, 929 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{24}\text{H}_{25}\text{NNaO}_2\text{S}[\text{M} + \text{Na}]^+$ 414.1504, found 414.1513.



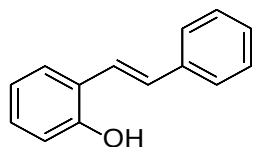
(E)-4-methyl-N-(5-phenylpent-4-en-2-yl)benzenesulfonamide (3ka): 0.211 g of **3ka** was obtained from 0.237 g (1 mmol) of **1k** following general procedure C. Yield 67%; colorless oil, R_f = 0.5 (SiO_2 , 20% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.73 (d, J = 8.2 Hz, 2H), 7.28 (t, J = 7.5 Hz, 2H), 7.22-7.20 (m, 5H), 6.31 (d, J = 15.6 Hz, 1H), 5.84 (dt, J = 15.6, 7.5 Hz, 1H), 4.55 (d, J = 7.5 Hz, 1H), 3.43 (sep, J = 6.7 Hz, 1H), 2.38-2.23 (m, 2H), 1.15 (d, J = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.3, 138.0, 137.0, 133.6, 129.7, 128.5, 127.4, 127.1, 126.2, 125.1, 49.8, 40.7, 21.7, 21.6; IR (KBr) ν 3401, 3019, 2400, 1607, 1512, 1422, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{18}\text{H}_{22}\text{NO}_2\text{S}[\text{M} + \text{H}]^+$ 316.1371, found 316.1373.



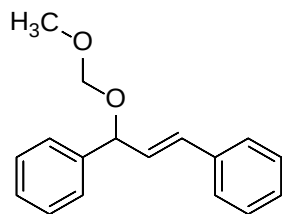
(E)-N-(1-(4-ethylphenyl)-4-phenylbut-3-en-1-yl)-4-methylbenzenesulfonamide (3la): 0.283 g of **3la** was obtained from 0.327 g (1 mmol) of **1l** following general procedure **C**. Yield 70%; white solid, mp 102-104 °C; $R_f = 0.5$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.57 (d, $J = 8.1$ Hz, 2H), 7.29-7.26 (m, 2H), 7.23-7.20 (m, 3H), 7.07-7.00 (m, 6H), 6.37 (d, $J = 15.9$ Hz, 1H), 5.87 (dt, $J = 15.9, 6.9$ Hz, 1H), 5.29 (d, $J = 6.9$ Hz, 1H), 4.42 (q, $J = 6.8$ Hz, 2H), 2.62-2.56 (m, 4H), 2.33 (s, 3H), 1.20 (t, $J = 6.8$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 143.4, 142.9, 137.9, 137.6, 137.0, 133.8, 129.3, 128.5, 127.9, 127.4, 127.2, 126.6, 126.3, 124.9, 57.6, 41.2, 28.5, 21.5, 15.6; IR (KBr) ν 3392, 2922, 1604, 1508, 1383, 1226, 1069, 769 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₅H₂₇NNaO₂S[M + Na]⁺ 428.1660, found 428.1655.



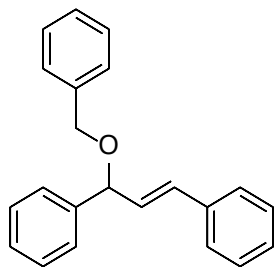
(E)-N-(2-styrylphenyl)acetamide(3ma)²⁹: 0.130 g of **3ma** was obtained from 0.159 g (1 mmol) of **1m** following general procedure **C**. Yield 55%; white solid; $R_f = 0.5$ (SiO₂, 10% MeOH/DCM); ¹H NMR (400 MHz, CDCl₃) δ 7.79 (d, $J = 8.1$ Hz, 1H), 7.51 (t, $J = 8.1$ Hz, 3H), 7.38 (t, $J = 7.4$ Hz, 2H), 7.31-7.24 (m, 3H), 7.19 (d, $J = 7.4$ Hz, 1H), 7.13 (d, $J = 16.4$ Hz, 1H), 6.99 (d, $J = 16.4$ Hz, 1H), 2.22 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 168.8, 137.1, 135.7, 134.7, 132.4, 130.6, 128.8, 128.4, 128.2, 126.8, 125.7, 124.5, 123.6, 24.3; IR (KBr) ν 3427, 3020, 2400, 1687, 1602, 1520, 1476, 756, 669 cm⁻¹.



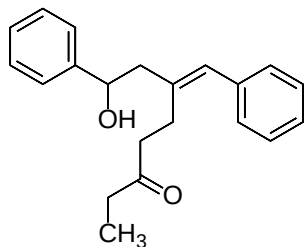
(E)-2-styrylphenol (3na)³⁰: 0.133 g of **3na** was obtained from 0.118 g (1 mmol) of **1n** following general procedure **C**. Yield 68%; white solid; $R_f = 0.4$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.54 (dt, $J = 7.7, 1.7$ Hz, 3H), 7.40-7.35 (m, 3H), 7.29-7.25 (m, 1H), 7.18-7.11 (m, 2H), 6.98-6.94 (m, 1H), 6.81 (dd, $J = 8.0, 0.9$ Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 153.1, 137.7, 130.2, 128.7, 127.7, 127.3, 126.6, 124.8, 123.1, 121.2, 116.0; IR (KBr) ν 3400, 3020, 2400, 1603, 1522, 1476, 1384, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₄H₁₂O[M + H]⁺ 197.0966, found 197.0958.



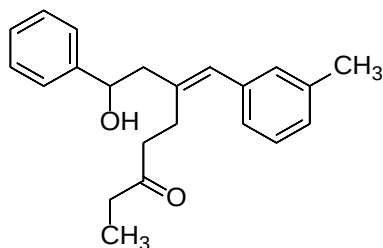
(E)-(3-(methoxymethoxy)prop-1-ene-1,3-diyl)dibenzene (3qa): 0.193 g of **3qa** was obtained from 0.176 g (1 mmol) of **1q** following general procedure **C**; Yield 76%; pale yellow oil; $R_f = 0.4$ (SiO₂, 5% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.44-7.35 (m, 6H), 7.32-7.28 (m, 3H), 7.25-7.21 (m, 1H), 6.66 (d, $J = 15.9$ Hz, 1H), 6.30 (dd, $J = 15.9, 7.2$ Hz, 1H), 5.28 (d, $J = 7.2$ Hz, 1H), 4.81 (d, $J = 6.8$ Hz, 1H), 4.66 (d, $J = 6.8$ Hz, 1H), 3.42 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 140.9, 136.6, 131.9, 129.6, 128.6, 127.8, 127.5, 127.1, 126.7, 93.7, 78.0, 67.2, 55.6; IR (KBr) ν 3401, 3020, 2400, 1726, 1638, 1423, 1215, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₇H₁₈NaO₂[M + Na]⁺ 277.1204, found 277.1205.



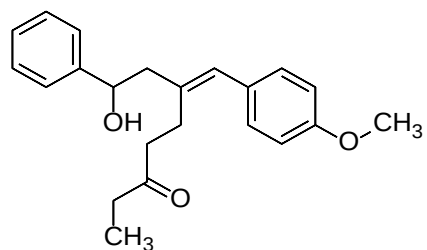
(E)-(3-(benzyloxy)prop-1-ene-1,3-diyl)dibenzene (3ra)³¹: 0.213 g of **3ra** was obtained from 0.222 g (1 mmol) of **1r** following general procedure **C**. Yield 71%; brown oil; $R_f = 0.5$ (SiO₂, 5% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.45 (d, $J = 7.0$ Hz, 2H), 7.41-7.37 (m, 8H), 7.33-7.29 (m, 4H), 7.25 (d, $J = 7.0$ Hz, 1H), 6.65 (d, $J = 15.9$ Hz, 1H), 6.36 (dd, $J = 15.9, 7.1$ Hz, 1H), 5.03 (d, $J = 7.1$ Hz, 1H), 4.60 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 141.2, 138.5, 136.7, 133.1, 131.7, 130.4, 129.8, 128.6, 128.5, 128.3, 127.8, 127.6, 127.1, 126.7, 81.7, 70.2; IR (KBr) ν 3685, 3020, 2400, 1601, 1521, 1476, 1423, 1215, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₂H₂₃NaO[M + Na]⁺ 323.1412, found 323.1415.



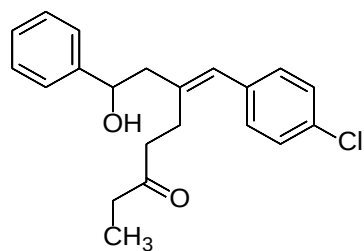
(E)-6-benzylidene-8-hydroxy-8-phenyloctan-3-one (4a): 0.215 g of **4a** was obtained from 0.146 g (1 mmol) of **1b** following general procedure **D**. Yield 70%; pale yellow oil; $R_f = 0.3$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.41 (d, $J = 7.2$ Hz, 2H), 7.36 (t, $J = 7.7$ Hz, 2H), 7.33-7.27 (m, 3H), 7.21 (t, $J = 7.4$ Hz, 1H), 7.17 (d, $J = 7.7$ Hz, 2H), 6.39 (s, 1H), 4.90 (dd, $J = 8.2, 5.0$ Hz, 1H), 2.66-2.62 (m, 1H), 2.57-2.49 (m, 5H), 2.37-2.33 (m, 3H), 1.01 (t, $J = 7.3$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.9, 144.2, 138.0, 137.5, 129.7, 128.5, 128.4, 128.2, 127.5, 126.5, 125.8, 72.5, 47.5, 40.7, 35.7, 25.0, 7.7; IR (KBr) ν 3401, 3020, 2400, 1654, 1511, 1420, 1215, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₁H₂₃O[(M + H)-H₂O]⁺ 291.1749, found 291.1748.



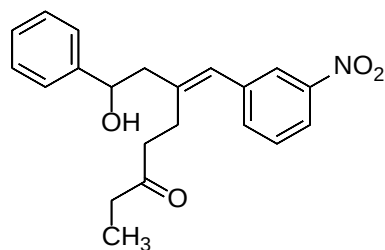
(E)-8-hydroxy-6-(3-methylbenzylidene)-8-phenyloctan-3-one (4b): 0.232 g of **4b** was obtained from 0.146 g (1 mmol) of **1b** following general procedure **D**. Yield 72%; pale yellow oil; $R_f = 0.3$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.41-7.34 (m, 2H), 7.30-7.28 (m, 2H), 7.22-7.18 (m, 1H), 7.04-6.98 (m, 3H), 6.36 (s, 1H), 4.89 (dd, $J = 8.0, 4.5$ Hz, 1H), 2.68-2.62 (m, 1H), 2.57-2.52 (m, 5H), 2.36-2.33 (m, 6H), 1.02 (t, $J = 7.2$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.9, 144.2, 137.9, 137.9, 137.4, 130.0, 129.4, 128.5, 128.3, 127.7, 127.5, 125.9, 125.6, 72.5, 47.8, 40.9, 35.9, 25.1, 21.5, 7.9; IR (KBr) ν 3685, 3401, 3020, 1710, 1524, 1215, 929, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₂H₂₆NaO₂[M + Na]⁺ 345.1819, found 345.1830.



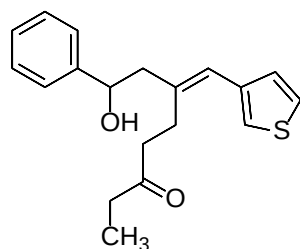
(E)-8-hydroxy-6-(4-methoxybenzylidene)-8-phenyloctan-3-one (4c): 0.233 g of **4c** was obtained from 0.146 g (1 mmol) of **1b** following general procedure **D**. Yield 69%; pale yellow oil; $R_f = 0.2$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.41-7.34 (m, 4H), 7.31-7.27 (m, 1H), 7.11 (d, $J = 8.6$ Hz, 2H), 6.85 (d, $J = 8.6$ Hz, 2H), 6.32 (s, 1H), 4.87 (dd, $J = 8.7, 4.5$ Hz, 1H), 3.80 (s, 3H), 2.68-2.62 (m, 1H), 2.56-2.50 (m, 4H), 2.41-2.33 (m, 3H), 2.19 (s, 1H), 1.02 (t, $J = 7.2$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 211.0, 158.3, 144.2, 136.6, 130.0, 129.8, 129.4, 128.5, 127.6, 125.9, 113.8, 72.4, 55.3, 48.0, 40.8, 35.9, 25.0, 7.8; IR (KBr) ν 3685, 3436, 3020, 1711, 1607, 1510, 1033, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₂H₂₆NaO₃[M + Na]⁺ 361.1780, found 361.1780.



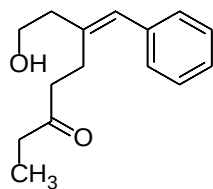
(E)-6-(4-chlorobenzylidene)-8-hydroxy-8-phenyloctan-3-one (4d): 0.198 g of **4d** was obtained from 0.146 g (1 mmol) of **1b** following general procedure **D**. Yield 58%; pale yellow gum; $R_f = 0.3$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.40-7.33 (m, 4H), 7.30-7.25 (m, 3H), 7.08 (d, $J = 8.3$ Hz, 2H), 6.29 (s, 1H), 4.88 (t, $J = 6.4$ Hz, 1H), 2.61-2.56 (m, 1H), 2.53-2.49 (m, 5H), 2.38-2.31 (m, 3H), 1.01 (t, $J = 7.3$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.7, 144.1, 138.9, 136.6, 132.4, 129.9, 128.5, 128.5, 127.7, 125.9, 72.7, 47.5, 40.6, 35.9, 25.0, 7.8; IR (KBr) ν 3685, 3020, 1712, 1602, 1521, 1215, 849, 627 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₁H₂₃ClNaO₂[M + Na]⁺ 365.1284, found 365.1277.



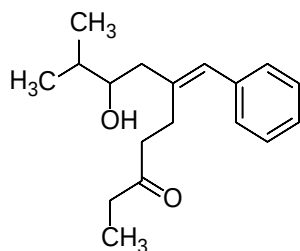
(E)-8-hydroxy-6-(3-nitrobenzylidene)-8-phenyloctan-3-one (4e): 0.190 g of **4e** was obtained from 0.146 g (1 mmol) of **1b** following general procedure **D**. Yield 54%; pale yellow oil; R_f = 0.2 (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 8.08-8.05 (m, 1H), 8.00(s, 1H), 7.48-7.47 (m, 2H), 7.41-7.35 (m, 4H), 7.32-7.29 (m, 1H), 6.36 (s, 1H), 4.93 (t, J = 6.5 Hz, 1H), 2.58-2.54 (m, 5H), 2.52-2.46 (m, 1H), 2.40 (q, J = 7.3 Hz, 2H), 2.21 (s, 1H), 1.03 (t, J = 7.3 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.4, 148.3, 144.0, 141.3, 139.3, 134.7, 129.3, 128.6, 127.9, 127.3, 125.9, 123.4, 121.5, 72.9, 47.1, 40.4, 36.1, 25.1, 7.8; IR (KBr) ν 3401, 3020, 2400, 1627, 1384, 1215, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₁H₂₂NO₃[(M + H)-H₂O]⁺ 336.1600, found 336.1592.



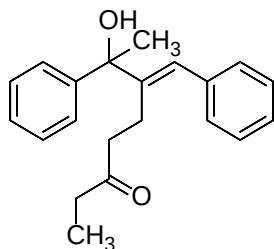
(E)-8-hydroxy-8-phenyl-6-(thiophen-3-ylmethylene)octan-3-one (4f): 0.185 g of **4f** was obtained from 0.146 g (1 mmol) of **1b** following general procedure **D**. Yield 59%; pale yellow oil; R_f = 0.3 (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.40-7.34 (m, 4H), 7.29-7.26 (m, 2H), 7.11-7.10 (m, 1H), 7.02 (dd, J = 5.0, 1.0 Hz, 1H), 6.32 (s, 1H), 4.86 (dd, J = 8.8, 4.3 Hz, 1H), 2.73-2.68 (m, 1H), 2.60-2.44 (m, 5H), 2.40 (q, J = 7.3 Hz, 2H), 2.28 (s, 1H), 1.04 (t, J = 7.3 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.9, 144.1, 138.2, 137.6, 128.5, 128.4, 127.7, 125.9, 125.3, 123.9, 122.5, 72.4, 48.3, 40.5, 36.0, 25.6, 7.9; IR (KBr) ν 3401, 3020, 2400, 1606, 1525, 1423, 1215, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₉H₂₁OS[(M + H)-H₂O]⁺ 297.1313, found 297.1311.



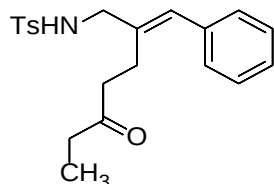
(E)-6-benzylidene-8-hydroxyoctan-3-one (4g): 0.141 g of **4g** was obtained from 0.072 g (1 mmol) of **1t** following general procedure **D**. Yield 61%; pale yellow oil; $R_f = 0.4$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.31 (t, $J = 7.5$ Hz, 2H), 7.19 (t, $J = 7.5$ Hz, 3H), 6.39 (s, 1H), 3.79 (t, $J = 6.4$ Hz, 2H), 2.54 (s, 4H), 2.41 (td, $J = 6.4, 0.8$ Hz, 2H), 2.35 (q, $J = 7.3$ Hz, 2H), 1.79 (s, 1H), 1.00 (t, $J = 7.3$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 211.1, 138.1, 137.5, 128.4, 128.3, 128.2, 126.4, 60.9, 40.6, 40.2, 35.7, 24.9, 7.7; IR (KBr) ν 3410, 3021, 1638, 1530, 1215, 670 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₅H₂₁O₂[M + H]⁺ 233.1542, found 233.1537.



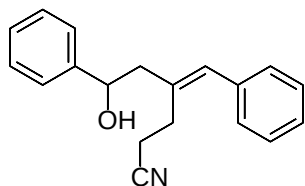
(E)-6-benzylidene-8-hydroxy-9-methyldecan-3-one (4h): 0.178 g of **4h** was obtained from 0.112 g (1 mmol) of **1d** following general procedure **D**. Yield 65%; pale yellow oil; $R_f = 0.4$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.33-7.28 (m, 2H), 7.22-7.18 (m, 3H), 6.41 (s, 1H), 3.58-3.54 (m, 1H), 2.70-2.63 (m, 1H), 2.55-2.51 (m, 2H), 2.49-2.41 (m, 2H), 2.35 (q, $J = 7.3$ Hz, 2H), 2.14-2.08 (m, 1H), 1.87 (bs, 1H), 1.78-1.70 (m, 1H), 1.02-0.96 (m, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 210.8, 139.1, 137.6, 129.2, 128.6, 128.4, 126.6, 74.0, 42.5, 40.8, 35.9, 33.6, 25.0, 18.9, 17.5, 7.8; IR (KBr) ν 3686, 3435, 3020, 2345, 1523, 1423, 1021, 849 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₈H₂₇O₂[M + ,H]⁺ 275.2011, found 275.2021.



(E)-6-benzylidene-7-hydroxy-7-phenyloctan-3-one (4i): 0.157 g of **4i** was obtained from 0.146 g (1 mmol) of **1e** following general procedure **D**. Yield 51%; colorless oil; $R_f = 0.2$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.53-7.51 (m, 2H), 7.35-7.31 (m, 4H), 7.27-7.22 (m, 4H), 6.79 (s, 1H), 3.47 (s, 1H), 2.53-2.46 (m, 1H), 2.37-2.23 (m, 3H), 2.08 (q, $J = 7.3$ Hz, 2H), 1.76 (s, 3H), 0.88 (t, $J = 7.3$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 212.6, 129.2, 128.5, 128.5, 128.3, 128.2, 127.0, 126.9, 125.9, 125.7, 125.2, 93.6, 41.1, 35.6, 29.9, 21.9, 7.8; IR (KBr) ν 3685, 3435, 3019, 2400, 1522, 1423, 1215, 626 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₁H₂₄NaO₂ [M + Na]⁺ 331.1674, found 331.1678.

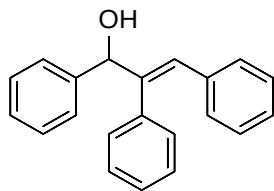


(E)-N-(2-benzylidene-5-oxoheptyl)-4-methylbenzenesulfonamide (4j): 0.193 g of **4j** was obtained from 0.209 g (1 mmol) of **1g** following general procedure **D**. Yield 52%; pale yellow oil; $R_f = 0.3$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.77 (d, $J = 8.2$ Hz, 2H), 7.31-7.27 (m, 4H), 7.23-7.19 (m, 1H), 7.08 (d, $J = 7.3$ Hz, 2H), 6.37 (s, 1H), 4.93 (t, $J = 5.6$ Hz, 1H), 3.64-3.62 (m, 2H), 2.51-2.45 (m, 4H), 2.41 (s, 3H), 2.33 (q, $J = 7.3$ Hz, 2H), 0.99 (t, $J = 7.3$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.9, 143.6, 137.2, 136.6, 136.2, 129.8, 129.6, 128.5, 128.4, 127.3, 127.1, 49.6, 40.3, 35.8, 22.8, 21.6, 7.8; IR (KBr) ν 3394, 3020, 2346, 1711, 1600, 1521, 1476, 1161, 1019 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₁H₂₆NO₃S[M + H]⁺ 372.1633, found 372.1630.

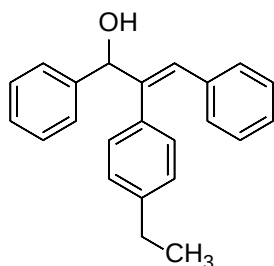


(E)-4-benzylidene-6-hydroxy-6-phenylhexanenitrile (4k): 0.171 g of **4k** was obtained from 0.146 g (1 mmol) of **1b** following general procedure **D**. Yield 62%; pale yellow oil; $R_f = 0.3$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.41-7.38 (m, 4H), 7.36-7.29 (m, 3H), 7.27-7.25 (m, 1H), 7.17-7.15 (m, 2H), 6.51 (s, 1H), 4.90 (t, $J = 6.1$ Hz, 1H), 2.71-2.64 (m, 1H), 2.62-2.60 (m, 2H), 2.56-2.49 (m, 1H), 2.45-2.41 (m, 2H), 2.22 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ

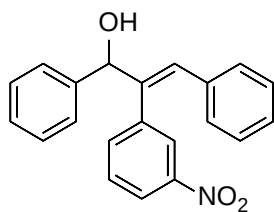
143.9, 136.9, 135.4, 131.9, 128.7, 128.5, 127.9, 127.1, 125.8, 119.4, 73.4, 46.3, 26.9, 15.9; IR (KBr) ν 3685, 3435, 3020, 2400, 2347, 1521, 1264, 1025, 928 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{19}\text{H}_{19}\text{NNaO}[\text{M} + \text{Na}]^+$ 300.1364, found 300.1357.



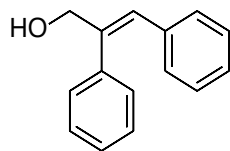
(E)-1,2,3-triphenylprop-2-en-1-ol (6a)³²: 0.191 g of **6a** was obtained from 0.208 g (1 mmol) of **5a** following general procedure C. Yield 67%; white solid; R_f = 0.4 (SiO_2 , 10% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.35-7.26 (m, 5H), 7.23-7.21 (m, 3H), 7.10-7.08 (m, 3H), 6.97-6.93 (m, 4H), 6.88 (s, 1H), 5.56 (s, 1H), 2.18 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.0, 141.8, 138.1, 136.5, 129.5, 129.4, 128.6, 128.4, 128.0, 127.8, 127.5, 127.3, 127.0, 126.9, 79.3; IR (KBr) ν 3401, 3019, 2926, 1600, 1492, 1447, 1384, 1215, 756 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{21}\text{H}_{17}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+$ 269.1330, found 269.1324.



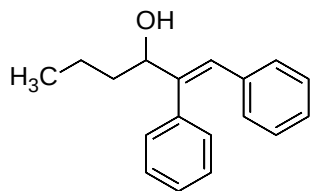
(E)-2-(4-ethylphenyl)-1,3-diphenylprop-2-en-1-ol (6b): 0.210 g of **6b** was obtained from 0.208 g (1 mmol) of **5a** following general procedure C. Yield 67%; white solid, mp 82-84 $^\circ\text{C}$; R_f = 0.5 (SiO_2 , 10% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.36-7.23 (m, 5H), 7.09-7.05 (m, 3H), 7.03 (d, J = 8.1 Hz, 2H), 6.96-6.94 (m, 2H), 6.84 (d, J = 8.1 Hz, 2H), 6.80 (s, 1H), 5.53 (s, 1H), 2.59 (q, J = 7.5 Hz, 2H), 2.12 (s, 1H), 1.19 (t, J = 7.5 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.0, 143.5, 142.0, 136.7, 135.2, 129.4, 129.4, 128.4, 128.1, 128.0, 127.7, 127.3, 126.9, 126.9, 79.2, 28.6, 15.4; IR (KBr) ν 3679, 3401, 3020, 2926, 2400, 1600, 1522, 1215, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{23}\text{H}_{21}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+$ 297.1643, found 297.1634.



(E)-2-(3-nitrophenyl)-1,3-diphenylprop-2-en-1-ol (6c): 0.218 g of **6c** was obtained from 0.208 g (1 mmol) of **5a** following general procedure **C**. Yield 66%; yellow solid, mp 104-106 °C; R_f = 0.3 (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 8.08-8.05 (m, 1H), 7.87 (t, J = 1.9 Hz, 1H), 7.34-7.27 (m, 6H), 7.22-7.20 (m, 1H), 7.12-7.11 (m, 3H), 7.02 (s, 1H), 6.94-6.91 (m, 2H), 5.60 (s, 1H), 2.39 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 148.2, 141.6, 141.0, 140.0, 136.3, 135.5, 129.4, 129.2, 129.2, 128.6, 128.3, 128.2, 127.5, 126.7, 124.6, 122.3, 78.9; IR (KBr) ν 3680, 3401, 3020, 2400, 1650, 1523, 1215, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₁H₁₆NO₂[(M + H)-H₂O]⁺ 314.1181, found 314.1173.

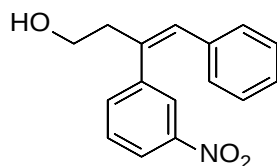


(E)-2,3-diphenylprop-2-en-1-ol (6d)³³: 0.128 g of **6d** was obtained from 0.132 g (1 mmol) of **5b** following general procedure **C**. Yield 61%; pale yellow oil; R_f = 0.5 (SiO₂, 10% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.36-7.29 (m, 3H), 7.24-7.22 (m, 2H), 7.13-7.09 (m, 2H), 7.01-6.99 (m, 2H), 6.70 (s, 1H), 4.47 (s, 2H), 1.73 (bs, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 141.6, 138.6, 136.5, 129.3, 128.9, 128.8, 128.0, 127.6, 126.9, 126.6, 68.6; IR (KBr) ν 3686, 3435, 3019, 2400, 1524, 1421, 1215, 1026, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₅H₁₃[(M + H)-H₂O]⁺ 193.1017, found 193.1003.

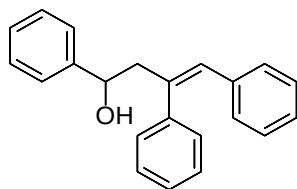


(E)-1,2-diphenylhex-1-en-3-ol (6e)³⁴: 0.176 g of **6e** was obtained from 0.174 g (1 mmol) of **5c** following general procedure **C**. Yield 70%; white solid; R_f = 0.4 (SiO₂, 10% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.36-7.30 (m, 3H), 7.20-7.078 (m, 2H), 7.10-7.07 (m, 3H), 6.94-6.91

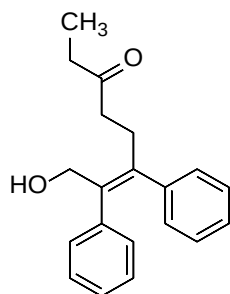
(m, 2H), 6.66 (s, 1H), 4.47 (s, 1H), 1.76 (s, 1H), 1.55-1.35 (m, 4H), 0.91 (t, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 145.1, 138.6, 136.7, 129.4, 129.3, 128.7, 128.0, 127.4, 126.9, 126.8, 77.3, 37.8, 19.1, 14.0; IR (KBr) ν 3401, 3020, 2927, 2400, 1619, 1384, 1215 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{18}\text{H}_{19}[(\text{M} + \text{H}) - \text{H}_2\text{O}]^+ 235.1487$, found 235.1483.



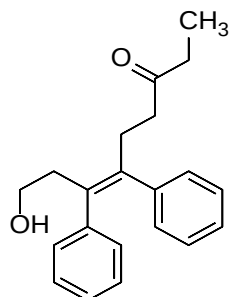
(Z)-3-(3-nitrophenyl)-4-phenylbut-3-en-1-ol (6f): 0.139 g of **6f** was obtained from 0.146 g (1 mmol) of **5d** following general procedure C. Yield 52%; pale yellow oil; $R_f = 0.3$ (SiO_2 , 20% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 8.13-8.08 (m, 2H), 7.50-7.41 (m, 2H), 7.12-7.10 (m, 3H), 6.91-6.89 (m, 2H), 6.68 (s, 1H), 3.70 (t, $J = 6.4$ Hz, 2H), 2.82 (t, $J = 6.4$, 1.0 Hz, 2H), 1.44 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 148.6, 142.5, 136.8, 136.1, 135.4, 130.8, 129.6, 129.1, 128.2, 127.2, 123.6, 122.3, 60.5, 42.9; IR (KBr) ν 3400, 3019, 2924, 1615, 1529, 1349, 668 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{16}\text{H}_{15}\text{NNaO}_3[\text{M} + \text{Na}]^+ 292.0950$, found 292.0940.



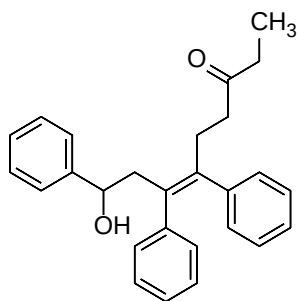
(Z)-1,3,4-triphenylbut-3-en-1-ol (6g): 0.213 g of **6g** was obtained from 0.222 g (1 mmol) of **5e** following general procedure C. Yield 71%; white solid; $R_f = 0.5$ (SiO_2 , 20% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3) δ 7.36-7.31 (m, 6H), 7.30-7.25 (m, 2H), 7.23-7.21 (m, 2H), 7.09-7.07 (m, 3H), 6.93-6.91 (m, 2H), 6.50 (s, 1H), 4.67-4.63 (m, 1H), 2.98-2.85 (m, 2H), 2.08 (d, $J = 2.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.0, 140.3, 139.1, 136.9, 129.9, 129.2, 128.8, 128.8, 128.4, 127.9, 127.6, 127.4, 126.6, 126.0, 71.5, 51.0; IR (KBr) ν 3401, 3020, 2400, 1603, 1521, 1476, 1424, 669 cm^{-1} ; HRMS (ESI-TOF) calcd for $\text{C}_{22}\text{H}_{20}\text{NaO}[\text{M} + \text{Na}]^+ 323.1412$, found 323.1415.



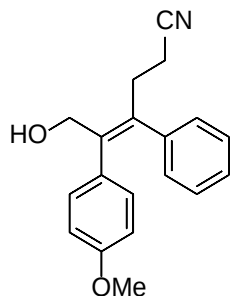
(E)-8-hydroxy-6,7-diphenyloct-6-en-3-one (7a): 0.165 g of **7a** was obtained from 0.132 g (1 mmol) of **5b** following general procedure **D**. Yield 56%; colorless oil, $R_f = 0.3$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.42-7.36 (m, 4H), 7.32-7.28 (m, 4H), 7.26-7.24 (m, 2H), 4.14 (s, 2H), 2.51 (t, $J = 7.4$ Hz, 2H), 2.25 (t, $J = 8.2$ Hz, 2H), 2.16 (q, $J = 7.3$ Hz, 2H), 1.33 (bs, 1H), 0.88 (t, $J = 7.3$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.4, 140.2, 140.1, 139.9, 138.0, 128.8, 128.6, 128.3, 127.2, 64.4, 40.7, 35.6, 29.4, 7.6; IR (KBr) ν 3686, 3434, 3020, 2400, 1521, 1476, 1019, 849 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₀H₂₂NaO₂ [M + Na]⁺ 317.1517, found 317.1516.



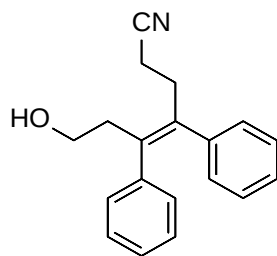
(Z)-9-hydroxy-6,7-diphenylnon-6-en-3-one (7b): 0.178 g of **7b** was obtained from 0.146 g (1 mmol) of **5d** following general procedure **D**. Yield 58%; brown oil; $R_f = 0.4$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.39 (t, $J = 7.2$ Hz, 4H), 7.30 (t, $J = 7.7$ Hz, 2H), 7.25 (d, $J = 7.2$ Hz, 4H), 3.44 (t, $J = 6.6$ Hz, 2H), 2.50-2.45 (m, 4H), 2.27-2.23 (m, 2H), 2.18 (q, $J = 7.2$ Hz, 2H), 1.66 (s, 1H), 0.90 (t, $J = 7.2$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.9, 141.6, 141.4, 139.4, 135.5, 128.9, 128.5, 128.5, 126.9, 126.9, 60.9, 40.9, 38.6, 35.7, 29.9, 7.7; IR (KBr) ν 3400, 3020, 2400, 1524, 1476, 1424, 1215, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₁H₂₅O₂[M + H]⁺ 309.1855, found 309.1852.



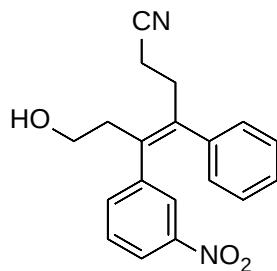
(Z)-9-hydroxy-6,7,9-triphenylnon-6-en-3-one (7c): 0.234 g of **7c** was obtained from 0.222 g (1 mmol) of **5e** following general procedure **D**. Yield 61%; pale yellow oil; $R_f = 0.4$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.38 (t, $J = 7.3$ Hz, 2H), 7.31-7.22 (m, 6H), 7.19-7.15 (m, 3H), 7.07 (d, $J = 7.3$ Hz, 2H), 7.00 (d, $J = 6.8$ Hz, 2H), 4.39 (dd, $J = 8.7, 5.5$ Hz, 1H), 2.72-2.68 (m, 1H), 2.55-2.51 (m, 1H), 2.50-2.39 (m, 2H), 2.19 (t, $J = 7.4$ Hz, 2H), 2.11 (q, $J = 7.3$ Hz, 2H), 0.85 (t, $J = 7.3$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 210.9, 144.2, 141.5, 141.1, 139.9, 135.3, 129.1, 128.7, 128.5, 128.3, 128.2, 127.3, 126.9, 126.7, 125.9, 72.1, 45.2, 40.7, 35.6, 29.9, 7.6; IR (KBr) ν 3401, 3020, 2400, 1654, 1515, 1384, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₇H₂₆O[(M + H)-H₂O]⁺ 367.2062, found 367.2067.



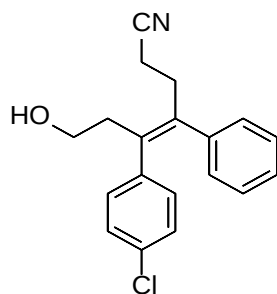
(E)-6-hydroxy-5-(4-methoxyphenyl)-4-phenylhex-4-enenitrileone (7d): 0.187 g of **7d** was obtained from 0.132 g (1 mmol) of **5b** following general procedure **D**. Yield 64%; white solid, mp 78-80 °C; $R_f = 0.4$ (SiO₂, 30% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.41 (t, $J = 7.4$ Hz, 2H), 7.34 (d, $J = 7.3$ Hz, 2H), 7.27 (d, $J = 8.4$ Hz, 4H), 6.98 (d, $J = 8.4$ Hz, 2H), 4.17 (s, 2H), 3.85 (s, 3H), 2.61 (d, $J = 7.2$ Hz, 2H), 2.14 (d, $J = 7.2$ Hz, 2H), 1.57 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 159.1, 140.4, 138.9, 137.0, 131.1, 130.1, 128.8, 128.6, 127.8, 119.1, 114.3, 64.4, 55.4, 30.8, 16.0; IR (KBr) ν 3401, 3020, 2927, 2400, 1602, 1524, 1476 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₉H₂₀NO₂[(M + H)]⁺ 294.1494, found 294.1490.



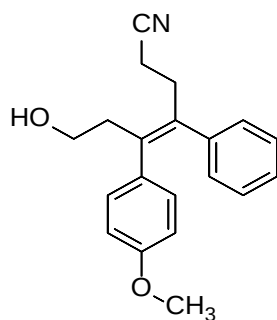
(Z)-7-hydroxy-4,5-diphenylhept-4-enenitrile (7e): 0.210 g of **7e** was obtained from 0.146 g (1 mmol) of **5d** following general procedure **D**. Yield 76%; white solid, mp 94-96 °C; R_f = 0.2 (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.43-7.40 (m, 4H), 7.34-7.33 (m, 2H), 7.29-7.27 (m, 4H), 3.45 (t, J = 6.5 Hz, 2H), 2.54 (t, J = 7.2 Hz, 2H), 2.49 (t, J = 6.5 Hz, 2H), 2.12 (t, J = 7.2 Hz, 2H), 1.11 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 140.8, 139.8, 138.5, 136.6, 128.9, 128.8, 128.6, 127.5, 127.3, 119.4, 60.7, 38.7, 31.0, 16.1; IR (KBr) ν 3401, 3019, 2400, 1600, 1511, 1396, 1215, 929 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₉H₂₀NO[M + H]⁺ 278.1545, found 278.1550.



(Z)-7-hydroxy-5-(3-nitrophenyl)-4-phenylhept-4-enenitrile (7f): 0.234 g of **7f** was obtained from 0.146 g (1 mmol) of **5d** following general procedure **D**. Yield 56%; white solid, mp 132-134 °C; R_f = 0.4 (SiO₂, 30% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 8.22 (m, 2H), 7.72-7.70 (m, 1H), 7.62 (t, J = 7.7 Hz, 1H), 7.44 (t, J = 7.7 Hz, 2H), 7.38-7.34 (m, 1H), 7.31-7.28 (m, 2H), 3.44 (t, J = 6.6 Hz, 2H), 2.51 (q, J = 6.8 Hz, 4H), 2.14 (t, J = 6.9 Hz, 2H), 1.59 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 148.5, 142.6, 138.9, 138.2, 136.6, 135.3, 129.8, 129.0, 128.7, 127.9, 123.6, 122.4, 119.1, 60.1, 38.4, 31.0, 16.0; IR (KBr) ν 3401, 3019, 2400, 1600, 1531, 1475, 1424, 669 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₉H₁₉N₂O₃[M + H]⁺ 323.1396, found 323.1389.



(Z)-5-(4-chlorophenyl)-7-hydroxy-4-phenylhept-4-enenitrile (7g): 0.193 g of **7g** was obtained from 0.146 g (1 mmol) of **5d** following general procedure **D**. Yield 62%; white semi solid; $R_f = 0.3$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.43-7.38 (m, 4H), 7.35-7.31 (m, 1H), 7.28-7.23 (m, 4H), 3.44 (t, $J = 6.6$ Hz, 2H), 2.51 (t, $J = 7.0$ Hz, 2H), 2.46 (t, $J = 6.6$ Hz, 2H), 2.12 (t, $J = 7.0$ Hz, 2H), 1.11 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 139.4, 139.2, 137.5, 133.2, 130.1, 128.9, 128.9, 127.6, 119.2, 60.4, 38.6, 30.9, 16.0; IR (KBr) ν 3686, 3400, 3020, 1602, 1476, 1423, 1023, 929 cm⁻¹; HRMS (ESI-TOF) calcd for C₁₉H₁₉ClO[M + H]⁺312.1155, found 312.1146.



(Z)-7-hydroxy-5-(4-methoxyphenyl)-4-phenylhept-4-enenitrile (7h): 0.212 g of **7h** was obtained from 0.146 g (1 mmol) of **5d** following general procedure **D**. Yield 69%; white semi solid; $R_f = 0.2$ (SiO₂, 20% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃) δ 7.42-7.38 (m, 2H), 7.34-7.30 (m, 1H), 7.27-7.25 (m, 2H), 7.21-7.18 (m, 2H), 6.96-6.93 (m, 2H), 3.84 (s, 3H), 3.45 (t, $J = 6.4$ Hz, 2H), 2.55 (t, $J = 7.2$ Hz, 2H), 2.46 (t, $J = 6.4$ Hz, 2H), 2.12 (t, $J = 7.2$ Hz, 2H), 1.09 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 158.8, 140.0, 138.1, 136.6, 132.9, 129.7, 128.9, 127.4, 119.4, 114.2, 60.8, 55.4, 38.8, 31.0, 16.1; IR (KBr) ν 3685, 3401, 3020, 2928, 2400, 1606, 1424, 1045, 929 cm⁻¹; HRMS (ESI-TOF) calcd for C₂₀H₂₂NO₂[M + H]⁺308.1651, found 308.1641.

X-ray Crystallography-

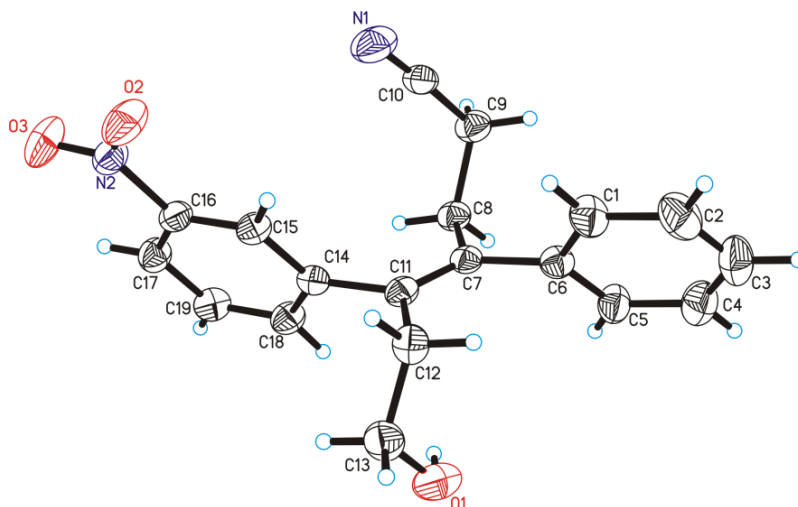


Figure 1. ORTEP diagram drawn with 30% ellipsoid probability for non-H atoms of the crystal structure of compound **7f** determined at 293 K.

Crystallization: Crystals of compound **7f** was grown from the solvent dichloromethane by slow evaporation method.

X-Ray Data Collection and Structure Refinement Details:

Good quality single crystals of size 0.25 x 0.15 x 0.10 mm of compound **7f** respectively, was selected under a polarizing microscope and were mounted on a loop for data collection. Single crystal X-ray data for compound **7f** was collected on the Rigaku Kappa 3 circle diffractometer equipped with the AFC12 goniometer and enhanced sensitivity (HG) Saturn724+ CCD detector in the 4x4 bin mode using the monochromated Mo-K α radiation generated from the microfocus sealed tube MicroMax-003 X-ray generator equipped with specially designed confocal multilayer optics. Data collections were performed using ω -scans of 0.5 $^\circ$ steps at 293(2) K for first three compounds and at 100(2) K for fourth compound. Cell determinations, data collections and data reductions were performed using the Rigaku CrystalClear-SM Expert 2.1 b24³⁵ software. Structure solutions and refinements were performed by using SHELXTL-NT³⁶. Refinement of coordinates and anisotropic thermal parameters of non-hydrogen atoms were carried out by the full-matrix least-squares method. The hydrogen atoms attached to carbon atoms were generated with idealized geometries and isotropically refined using a riding model.

Table 1 Crystal data and structure refinement details for **7f**.

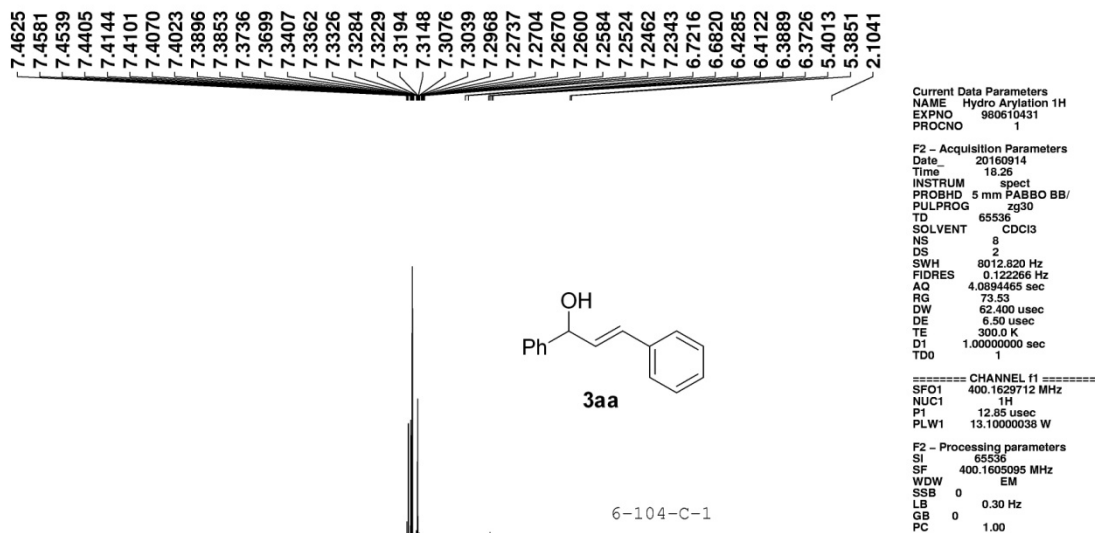
Compound	****
Empirical formula	C ₁₉ H ₁₈ N ₂ O ₃
Formula weight	322.35
Crystal System	Monoclinic
Space group	P2 ₁ /c
<i>a</i> (Å)	16.456(13)
<i>b</i> (Å)	6.808(5)
<i>c</i> (Å)	15.922(13)
α (°)	90.00
β (°)	111.027(12)
γ (°)	90.00
<i>V</i> (Å ³)	1665(2)
<i>Z</i>	4
<i>D</i> _c (g/cm ³)	1.286
<i>F</i> ₀₀₀	680
μ (mm ⁻¹)	0.088
$\theta_{\max}$ (°)	25.35
Total reflections	12506
Unique reflections	3034
Reflections [<i>I</i> > 2σ(<i>I</i>)]	1232
Parameters	218
<i>R</i> _{int}	0.0943
Goodness-of-fit	0.943
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)]	0.0714
<i>wR</i> (<i>F</i> ² , all data)	0.2478
CCDC No.	1533434

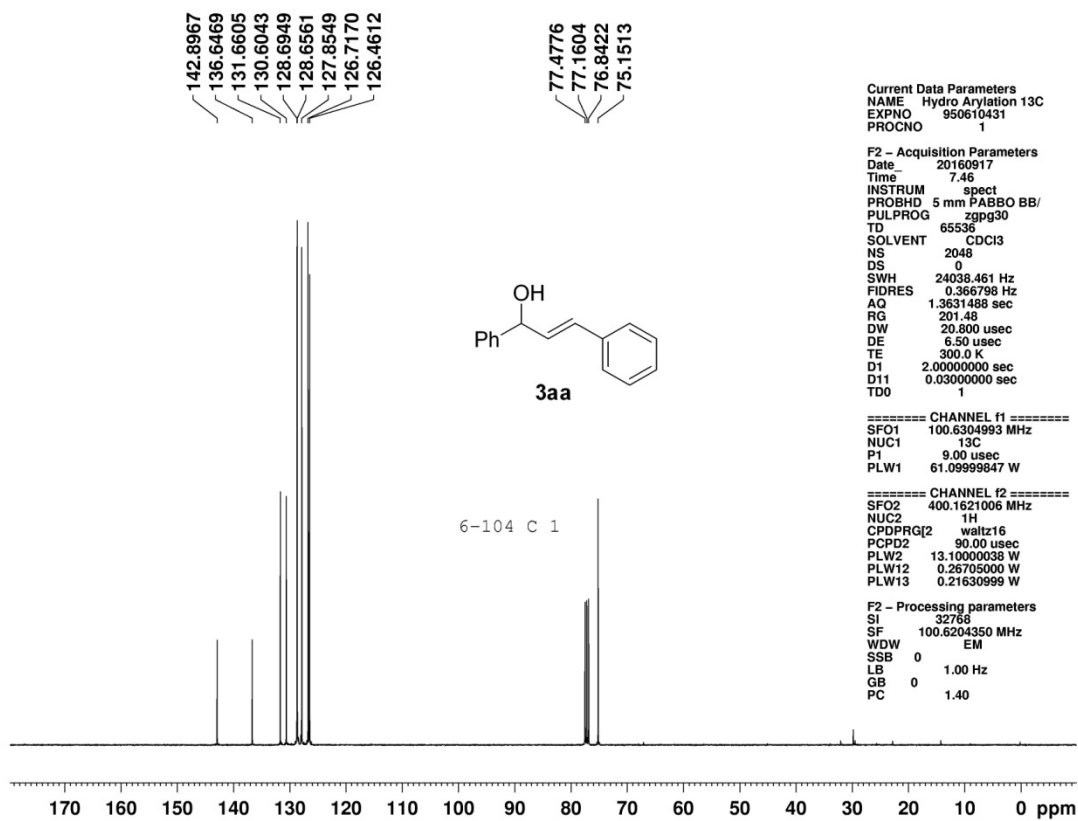
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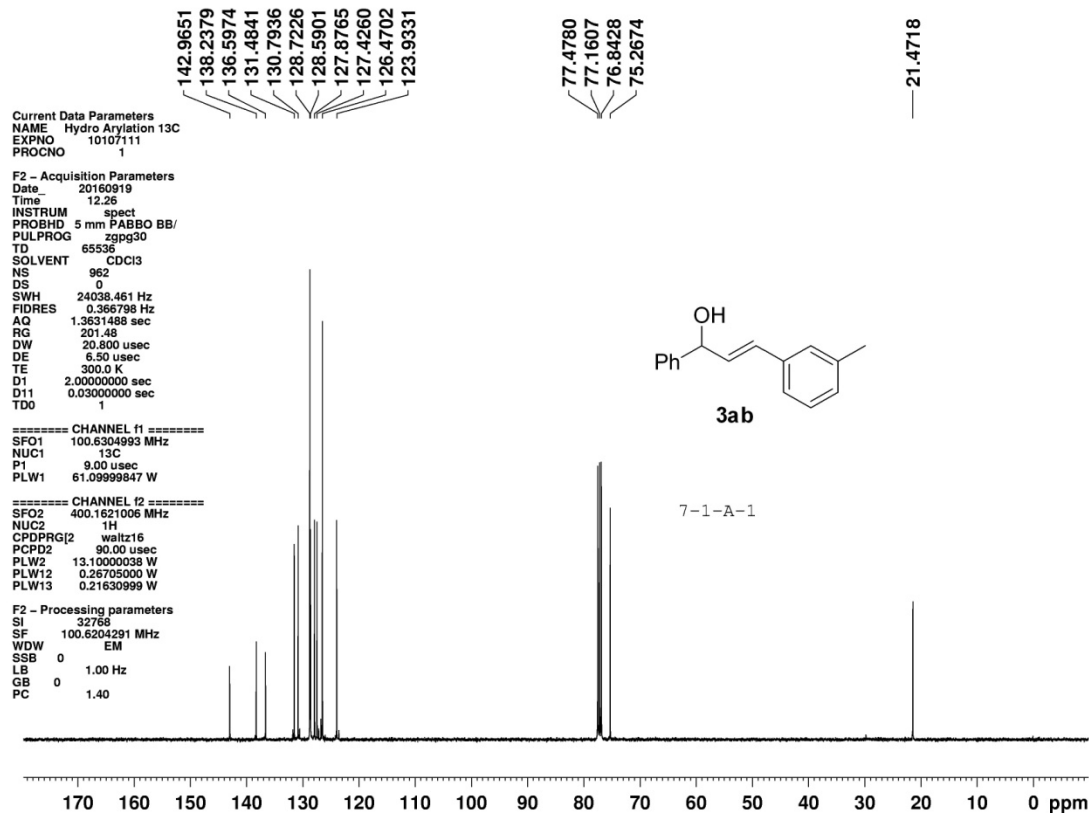
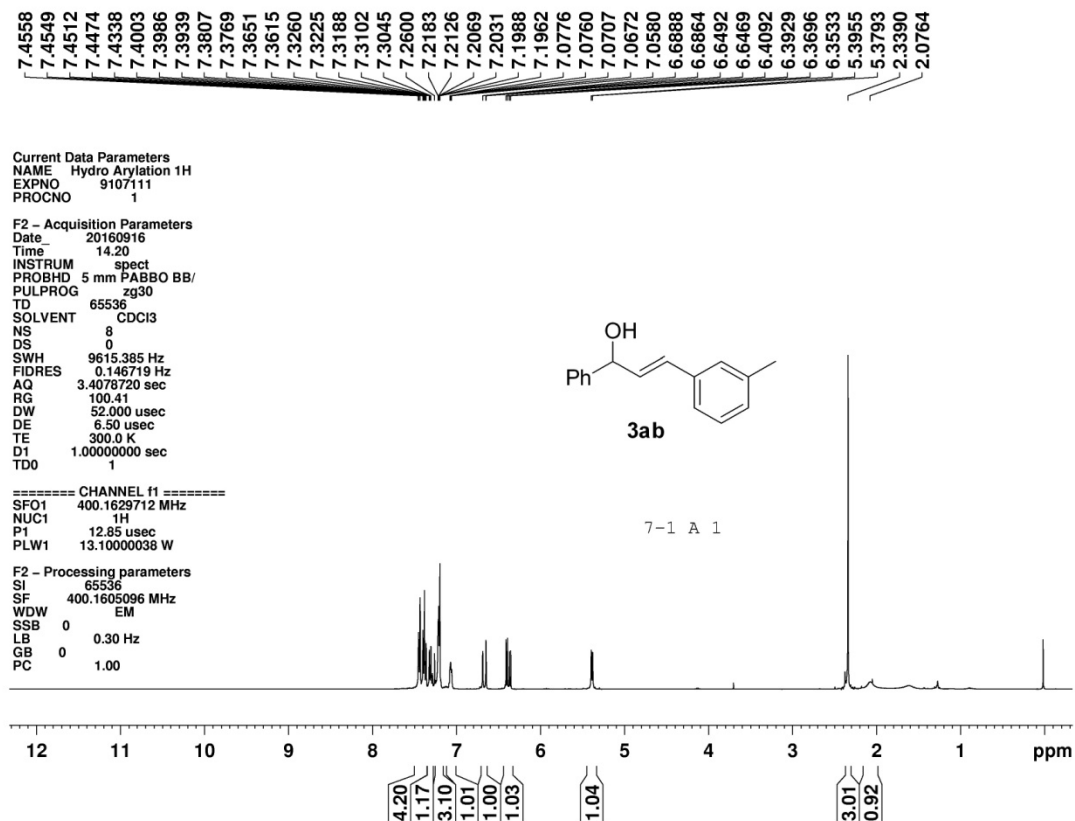
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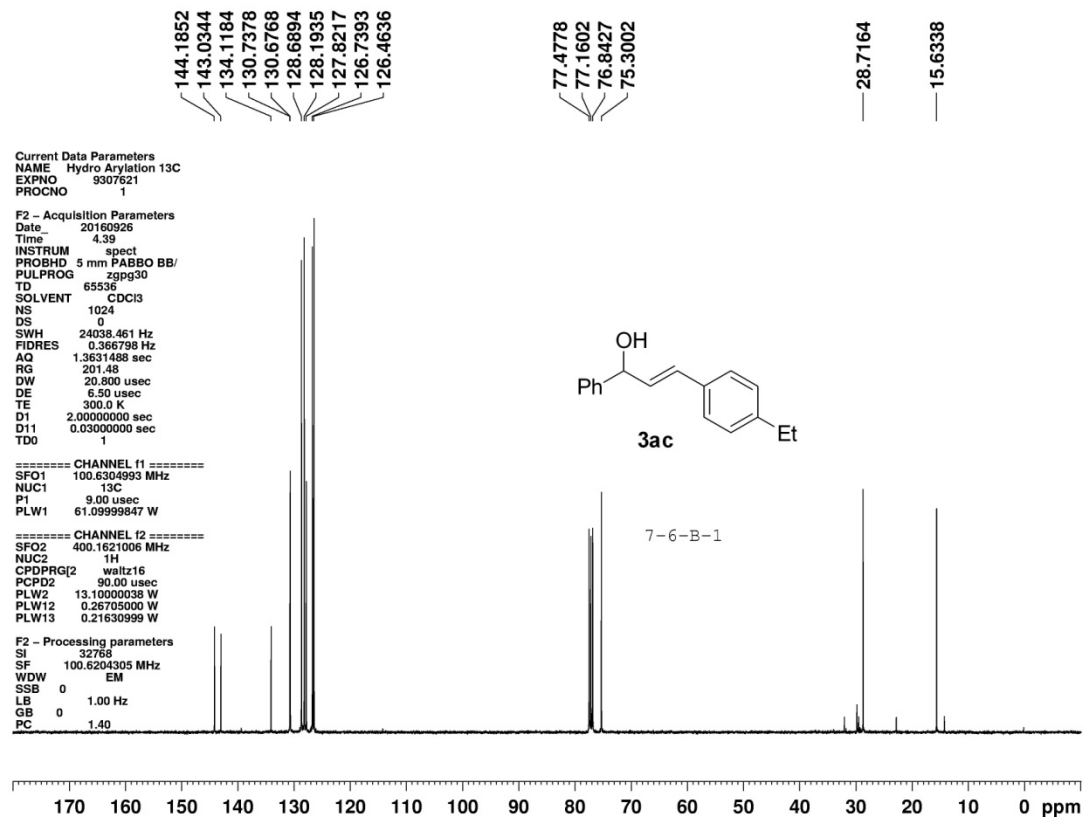
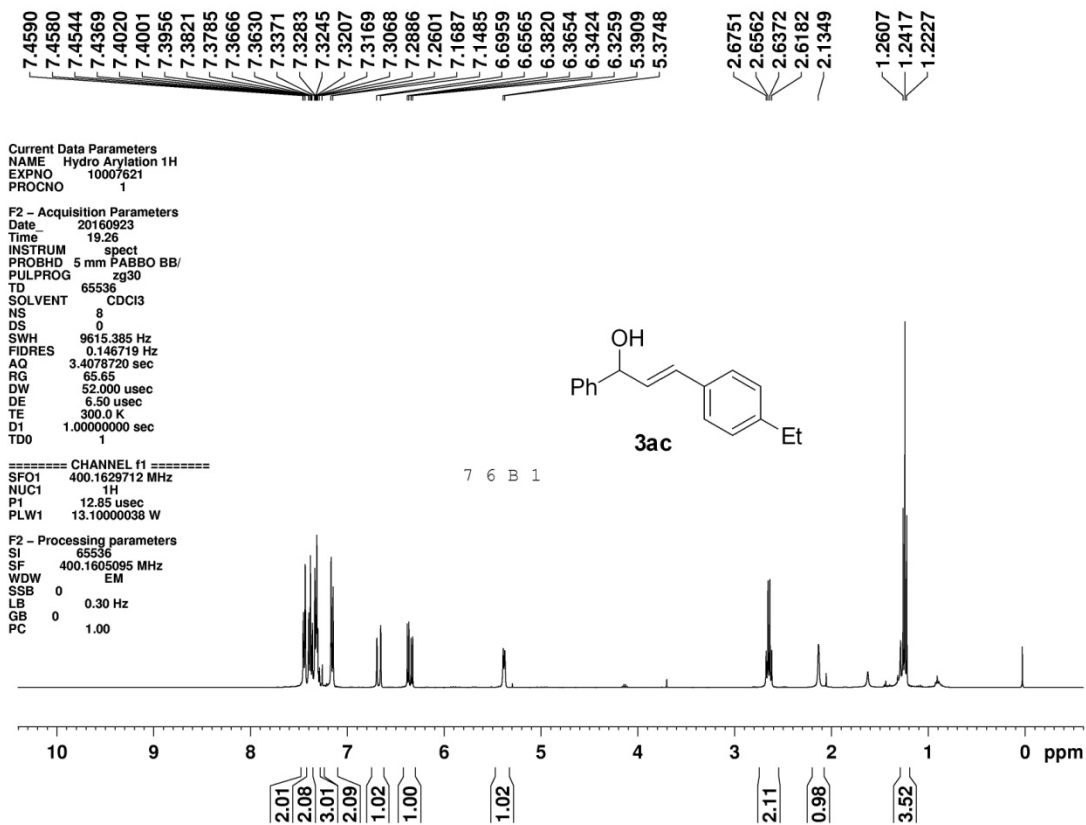
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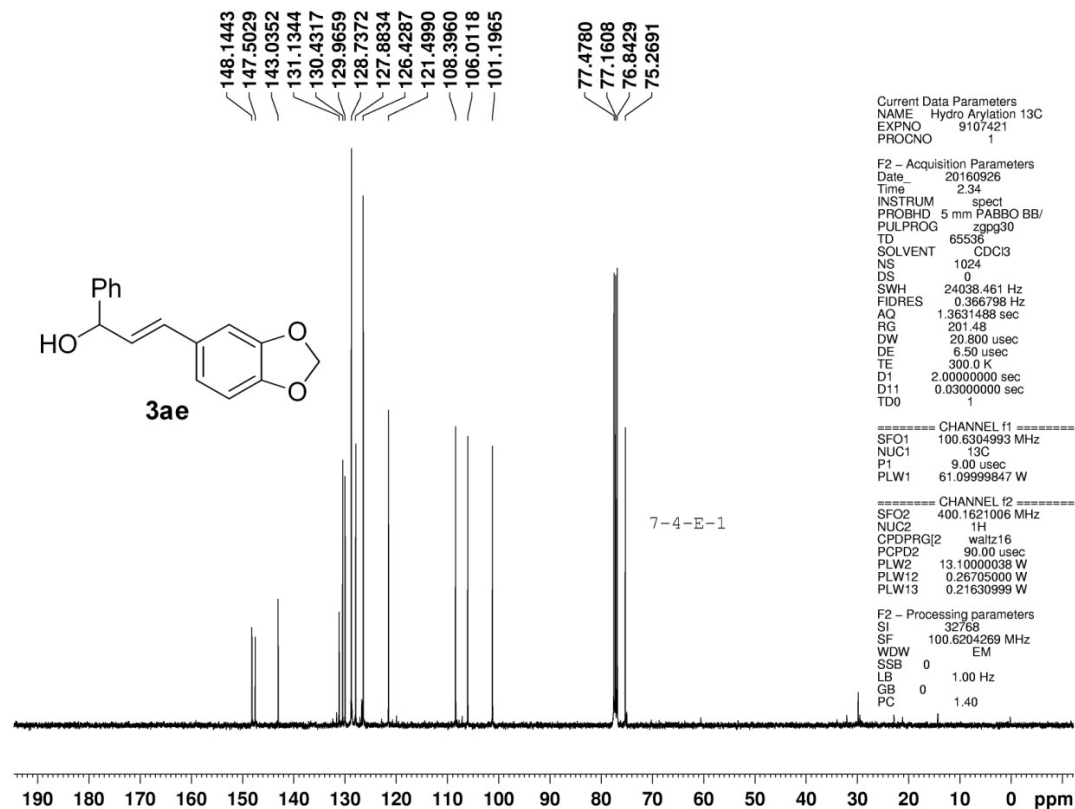
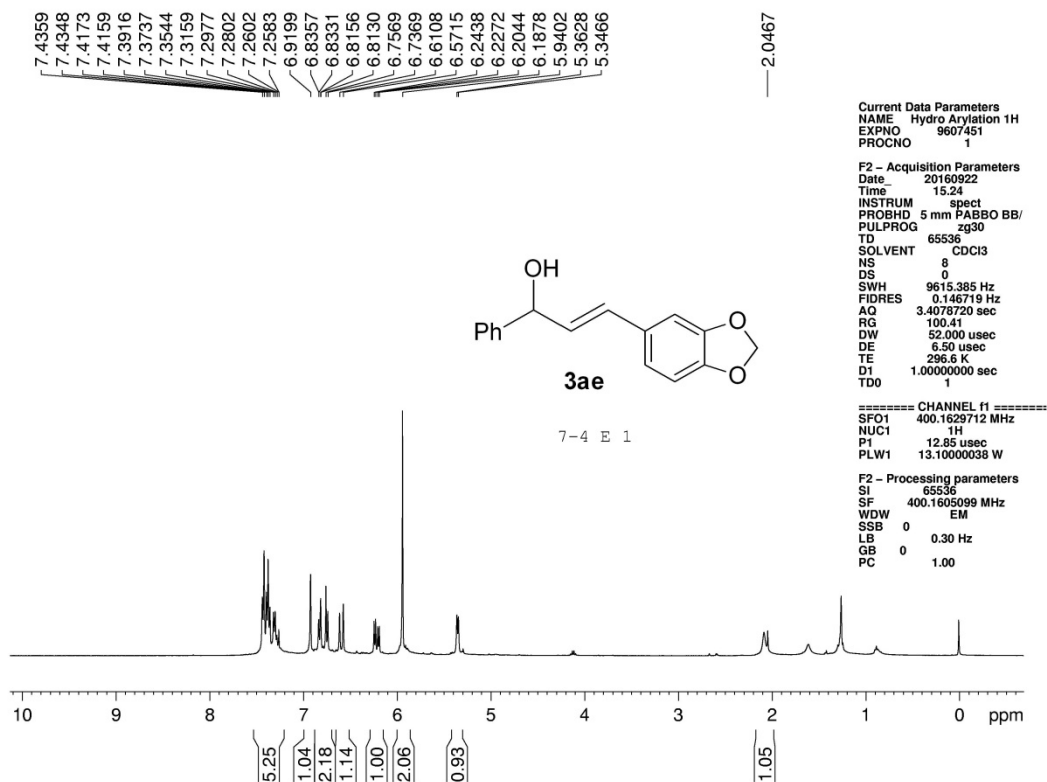
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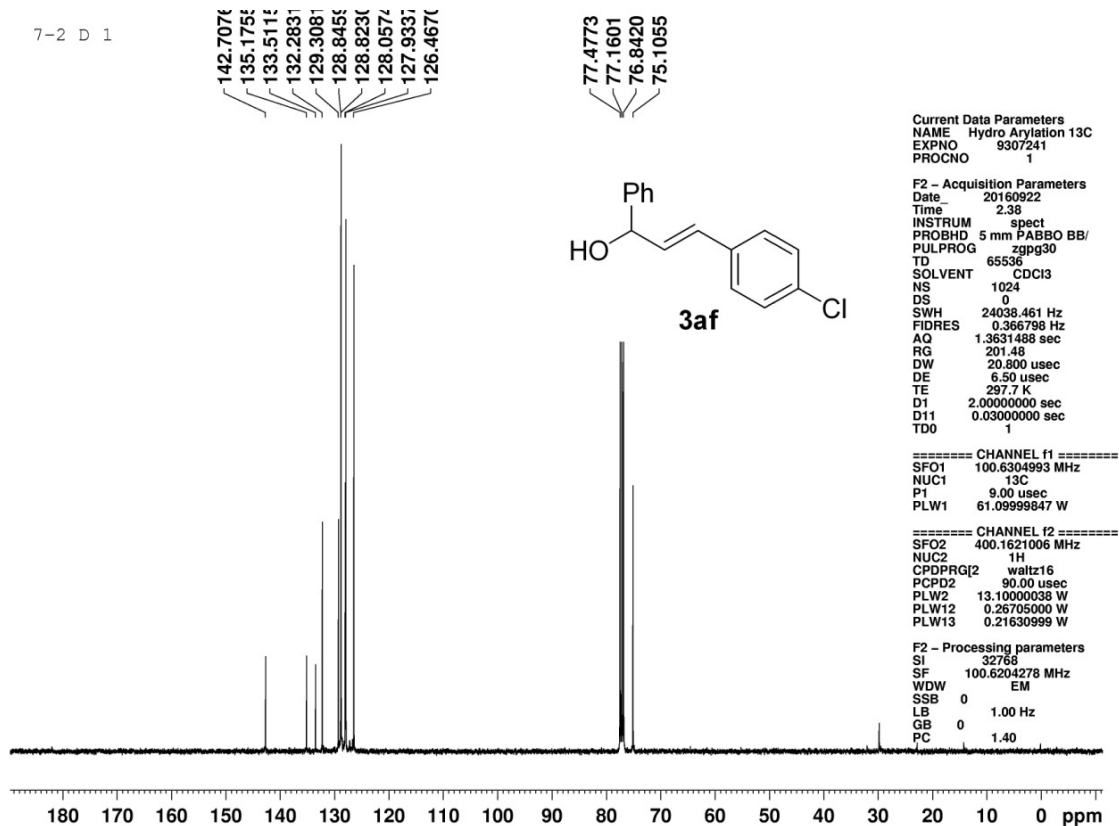
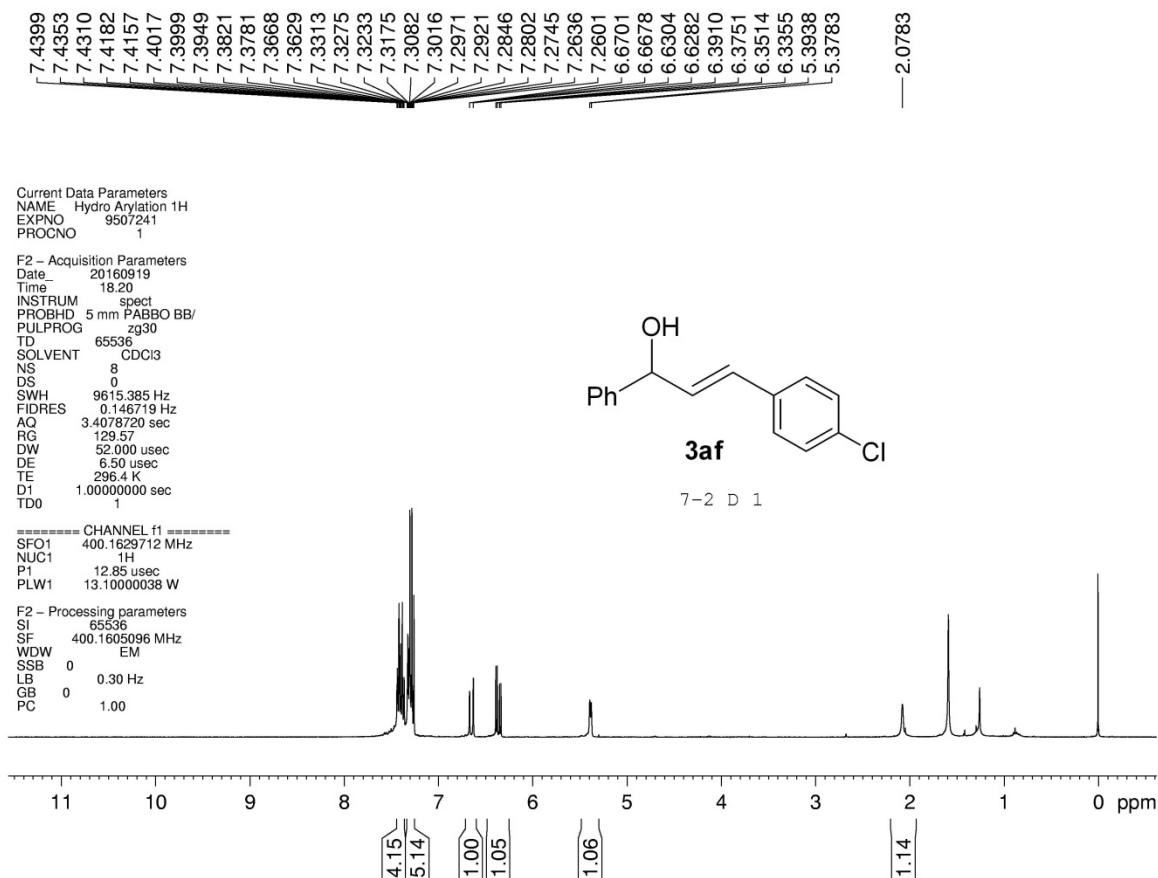


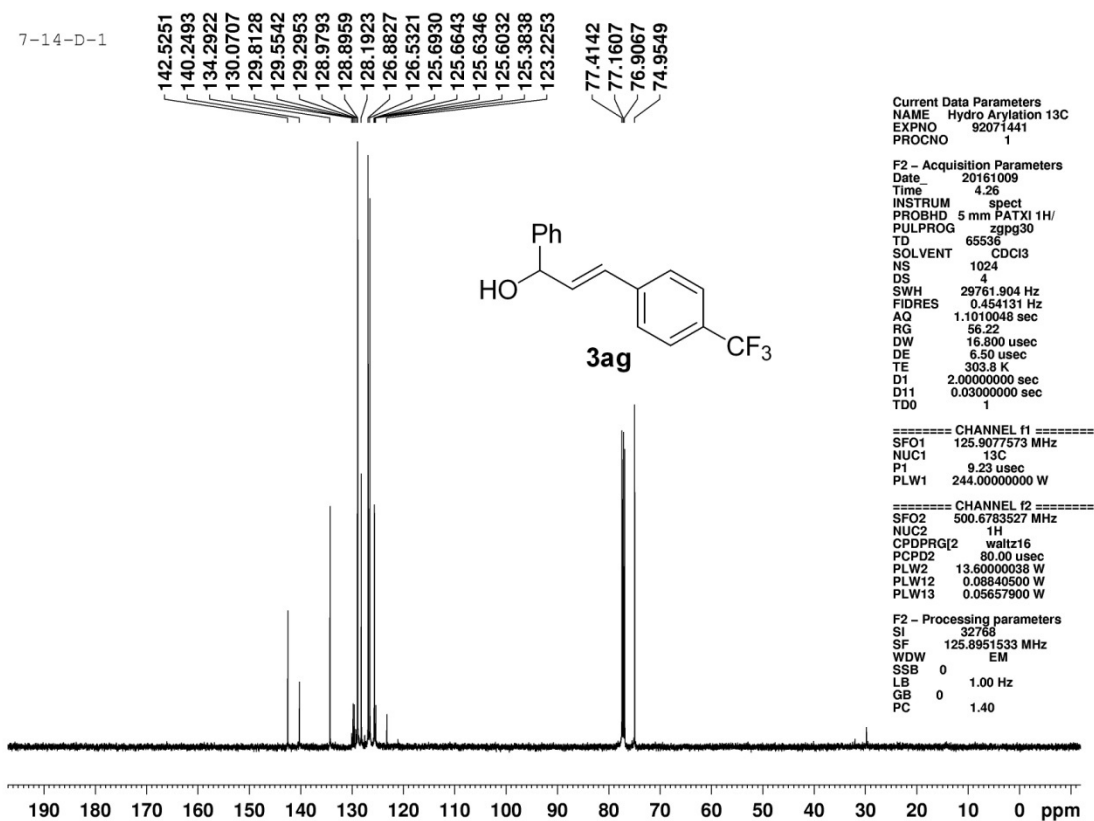
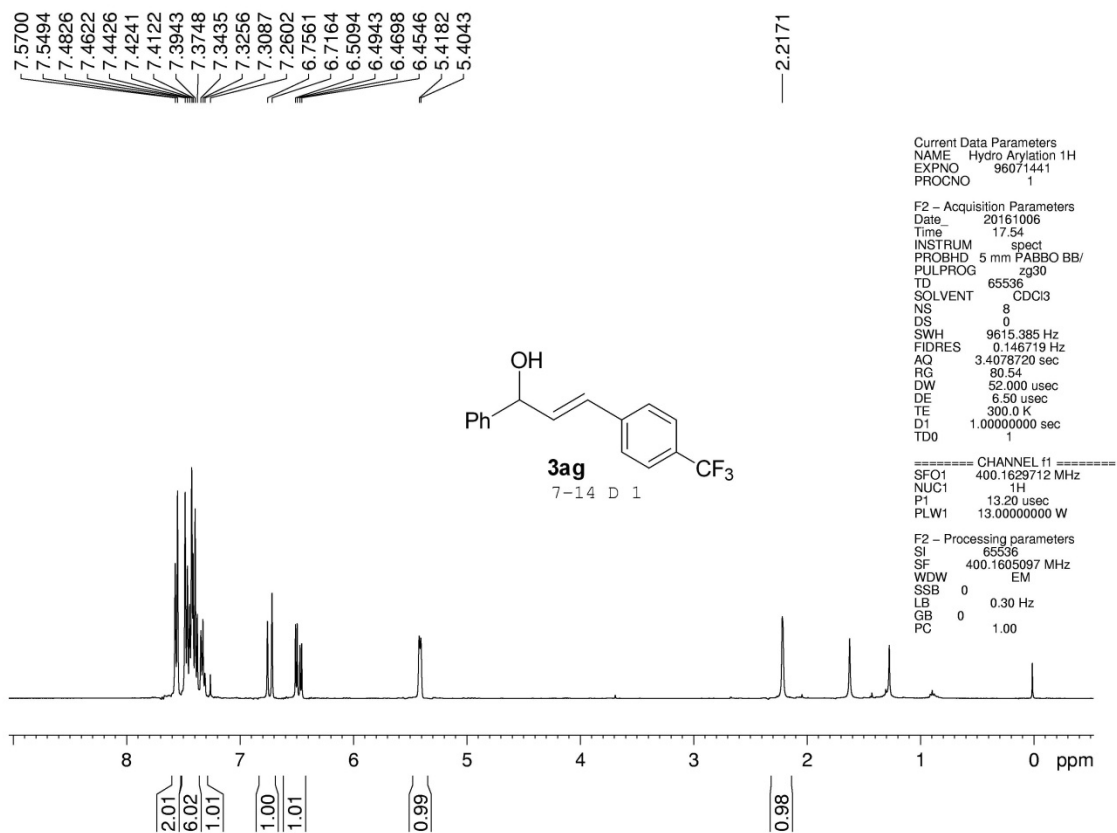


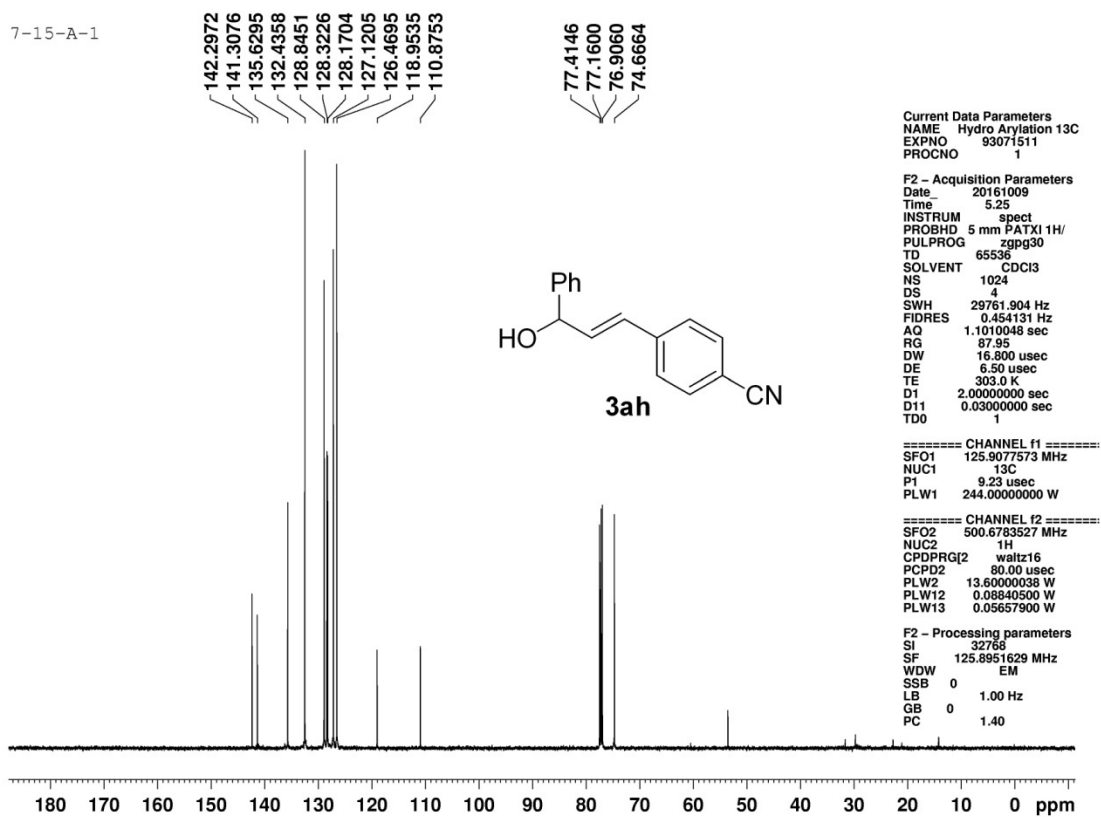
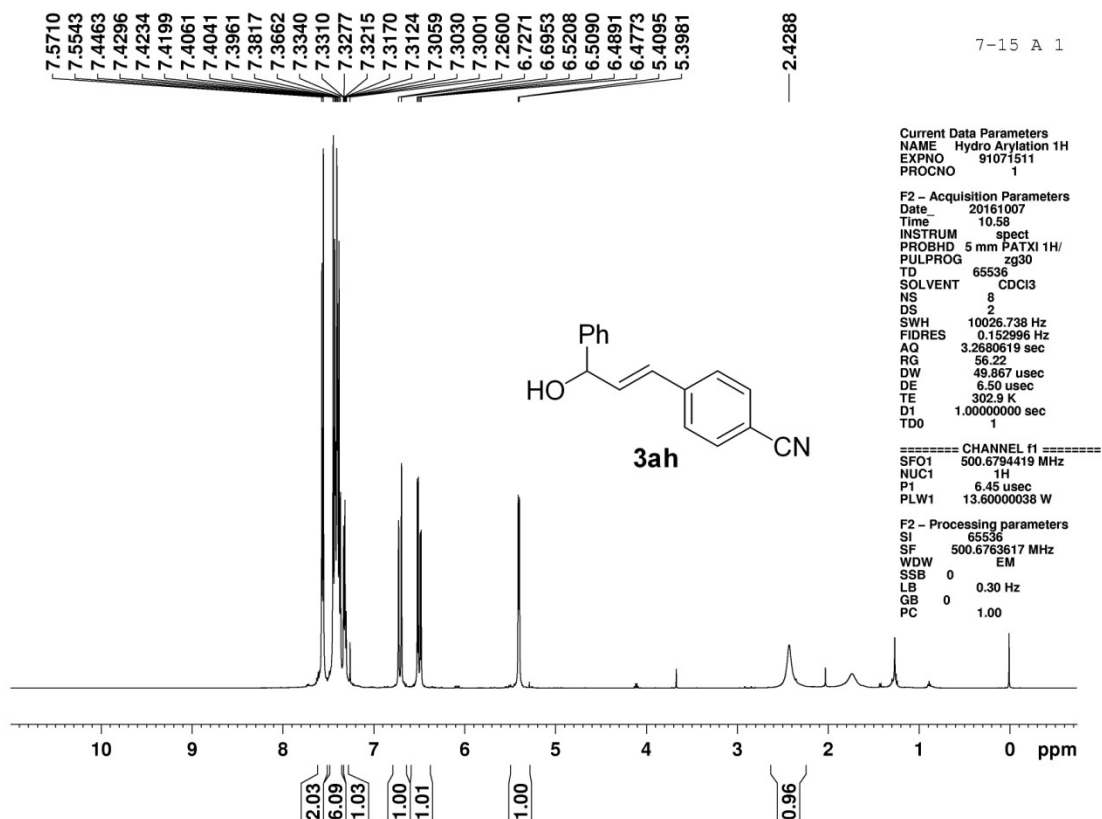


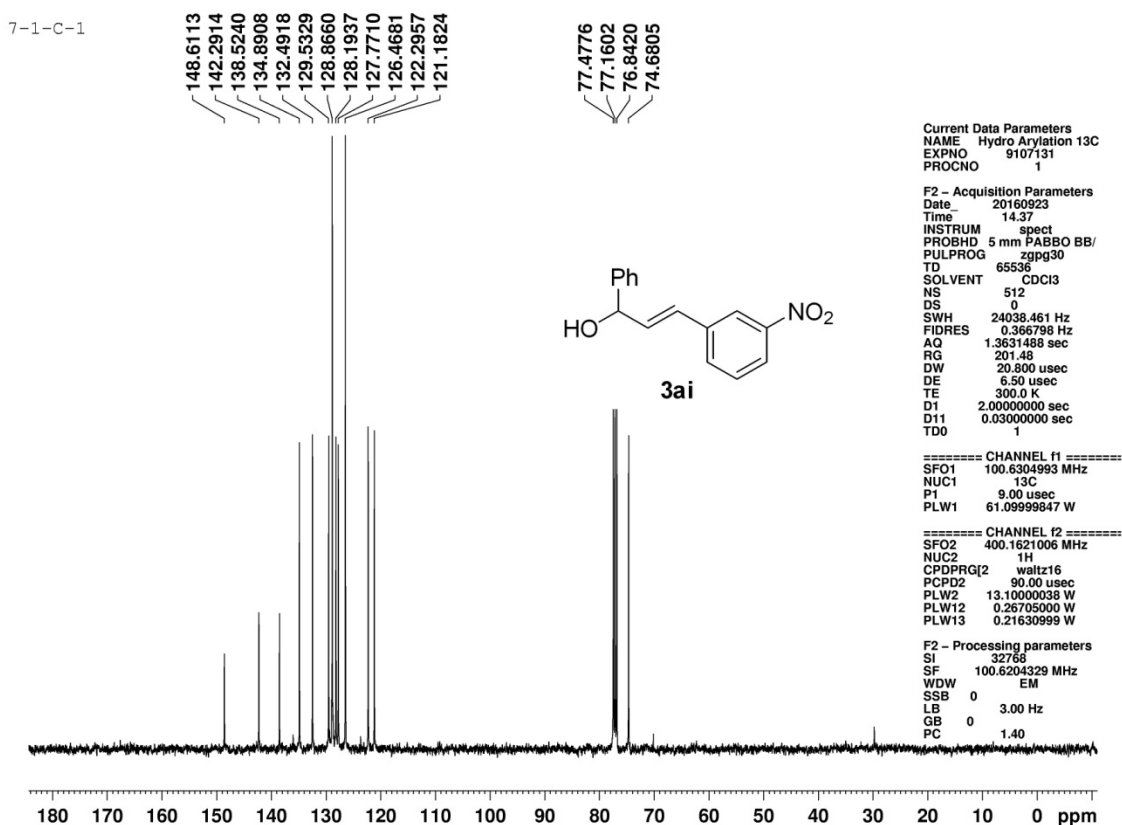
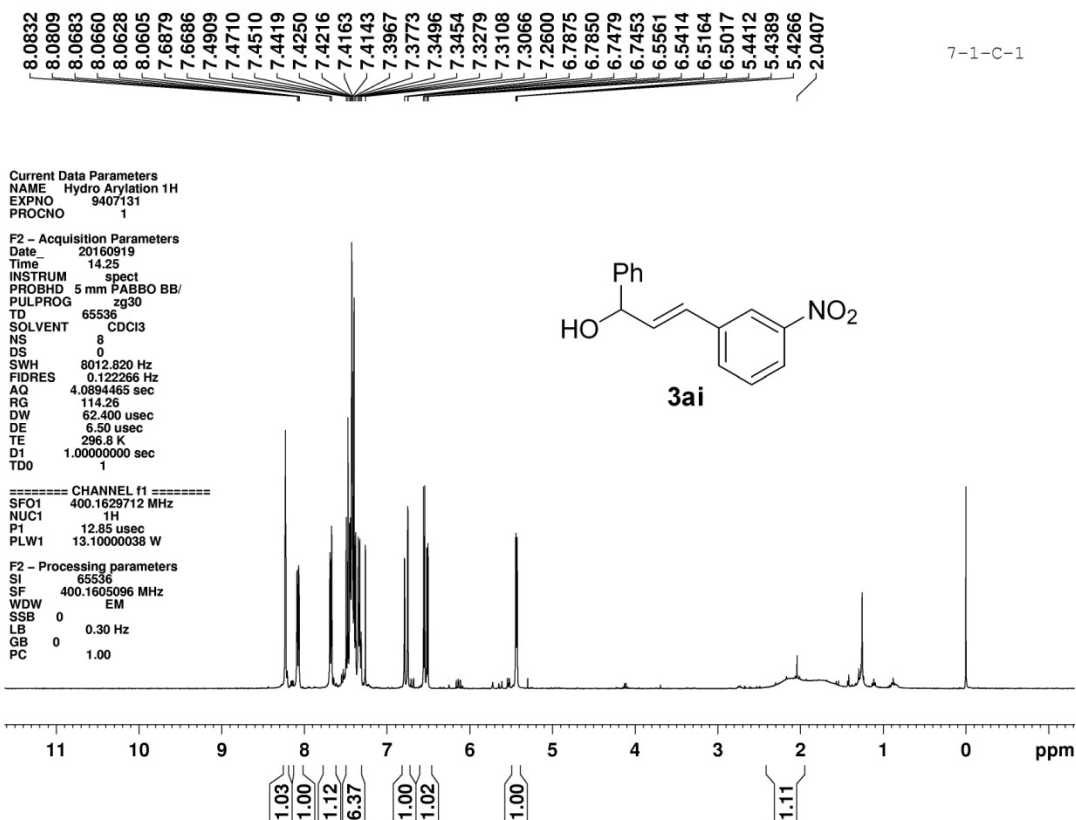


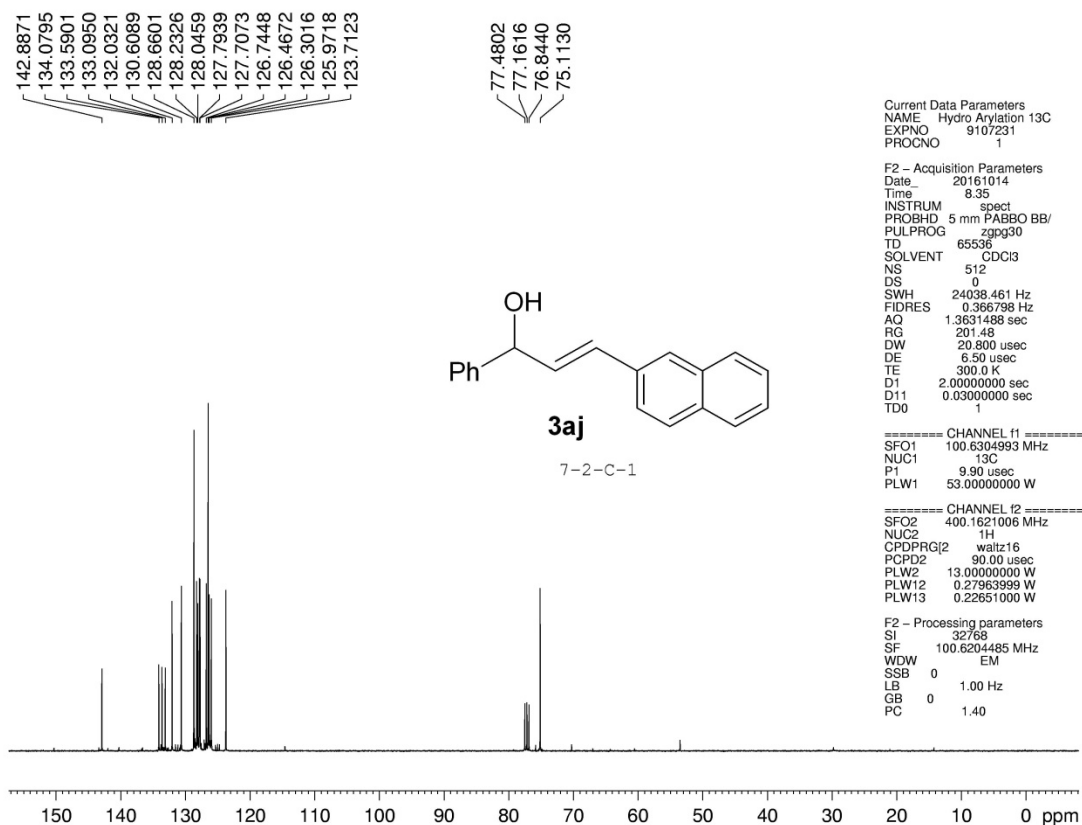
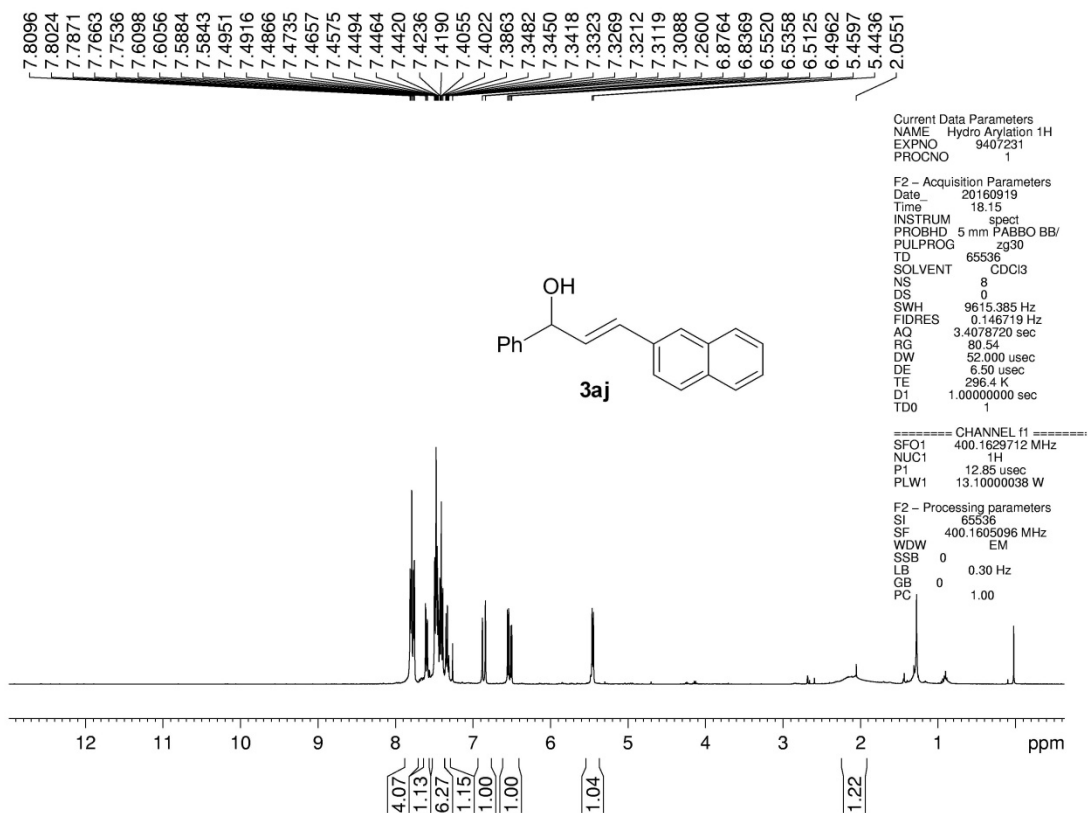


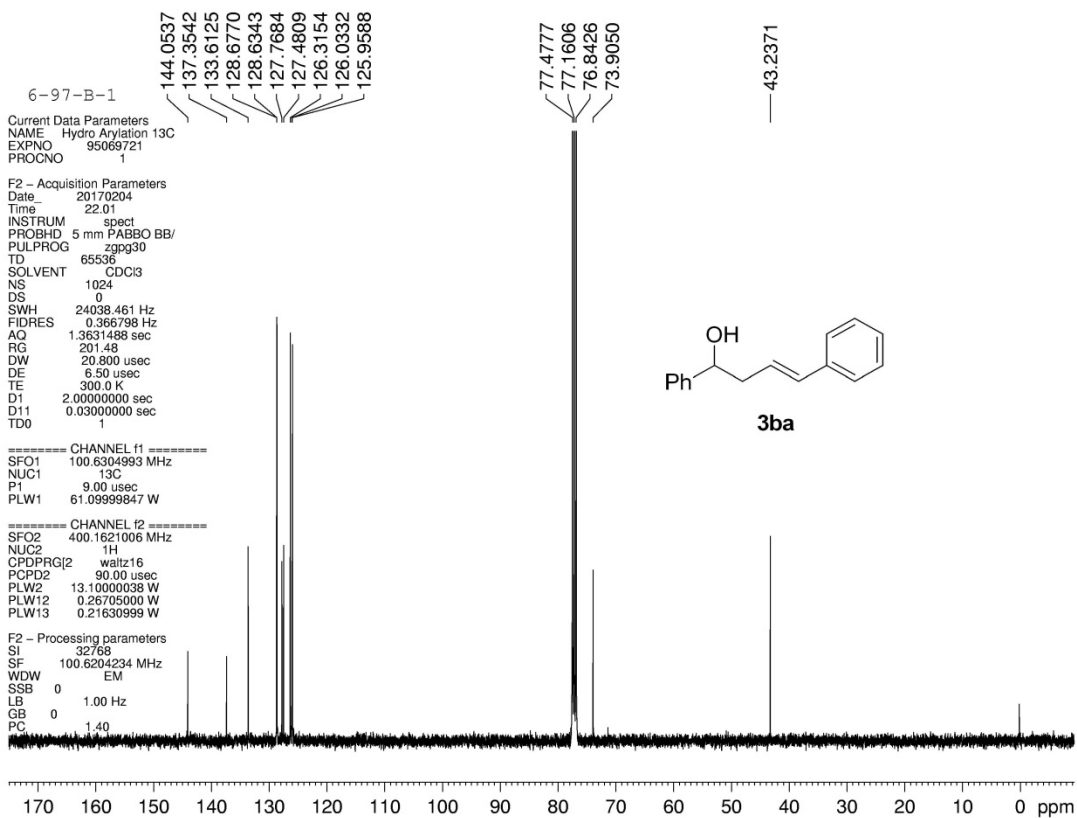
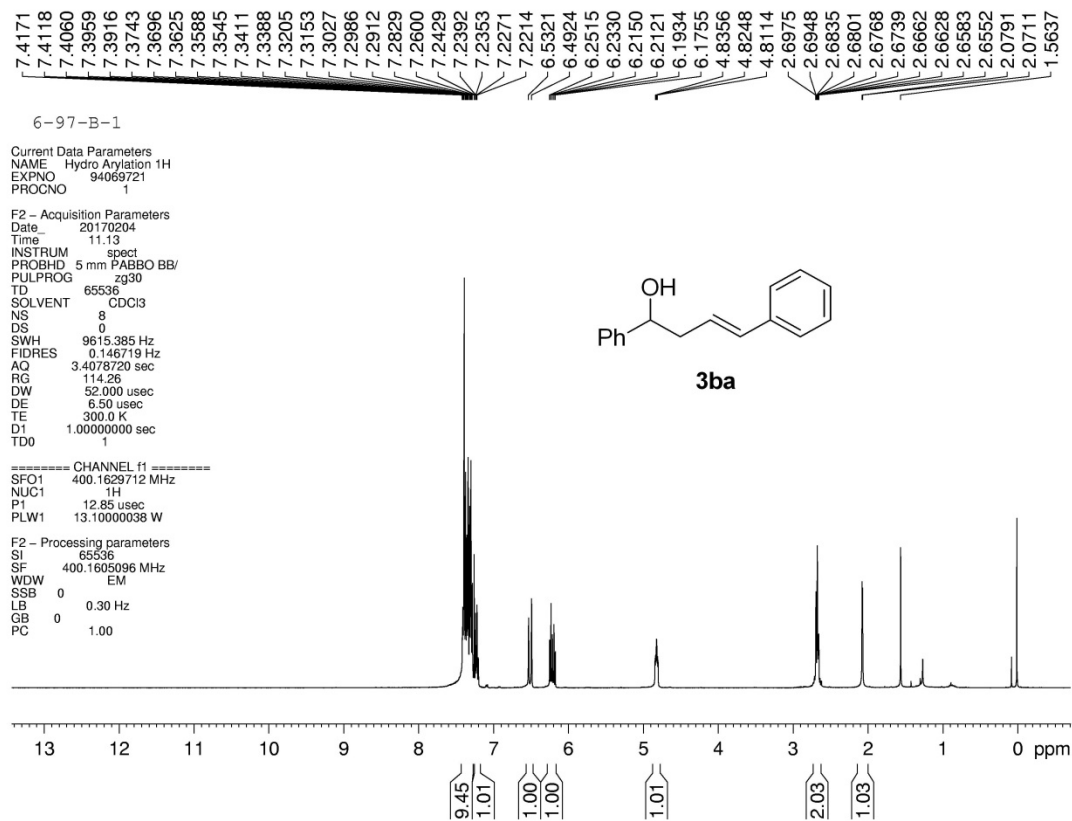


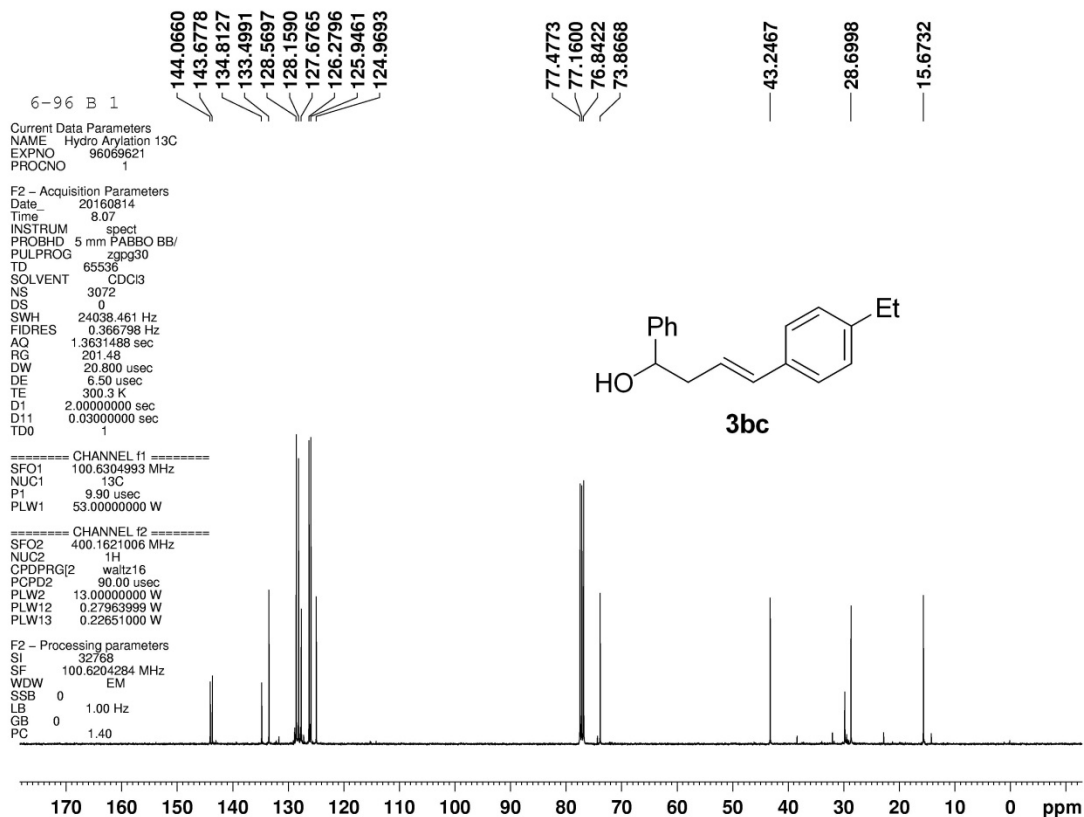
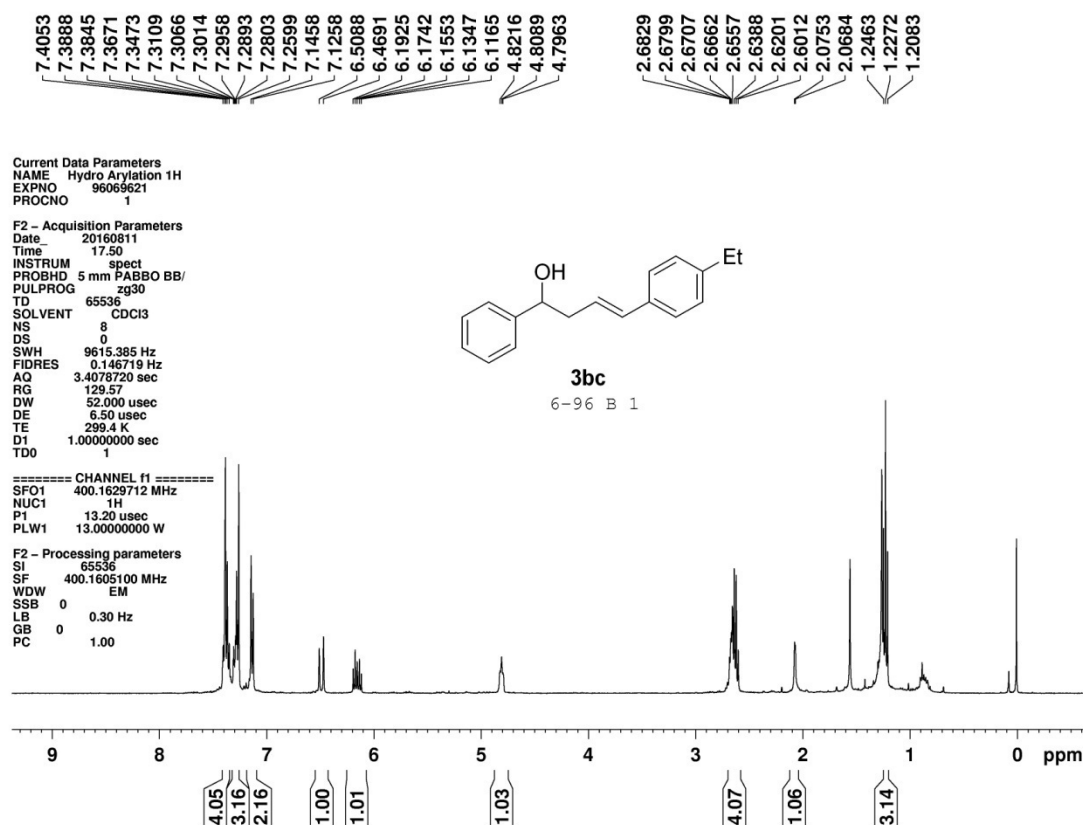


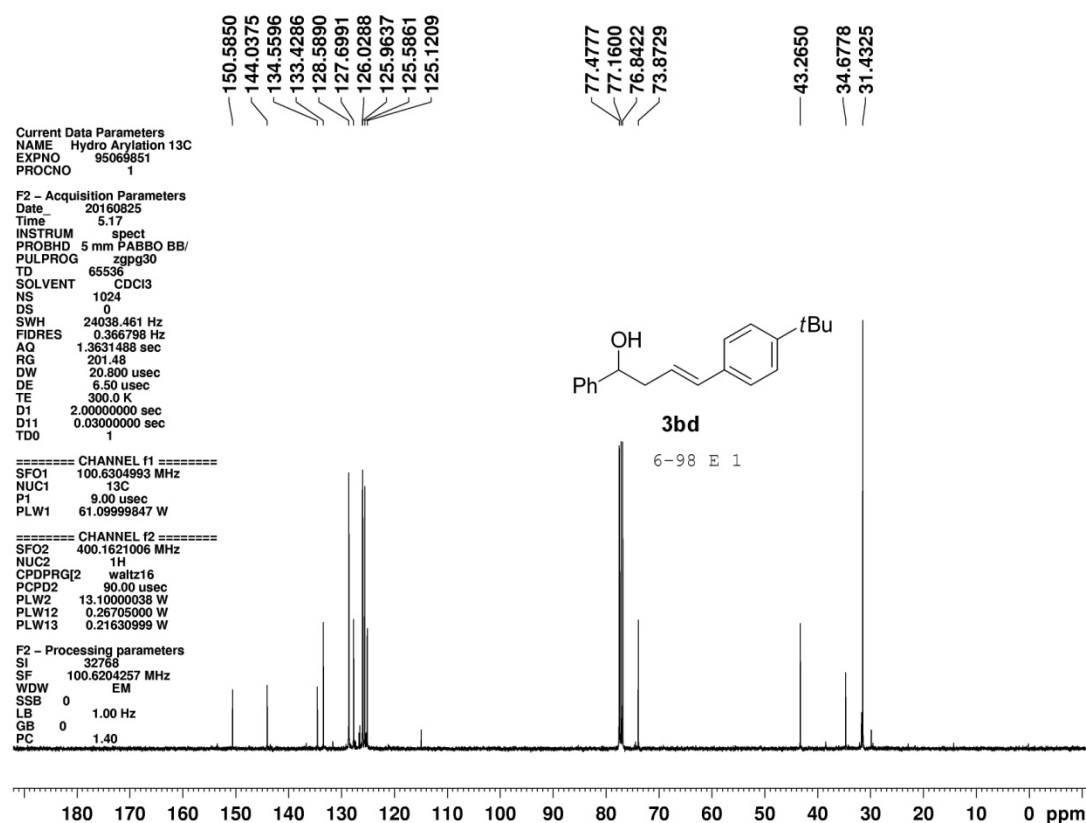
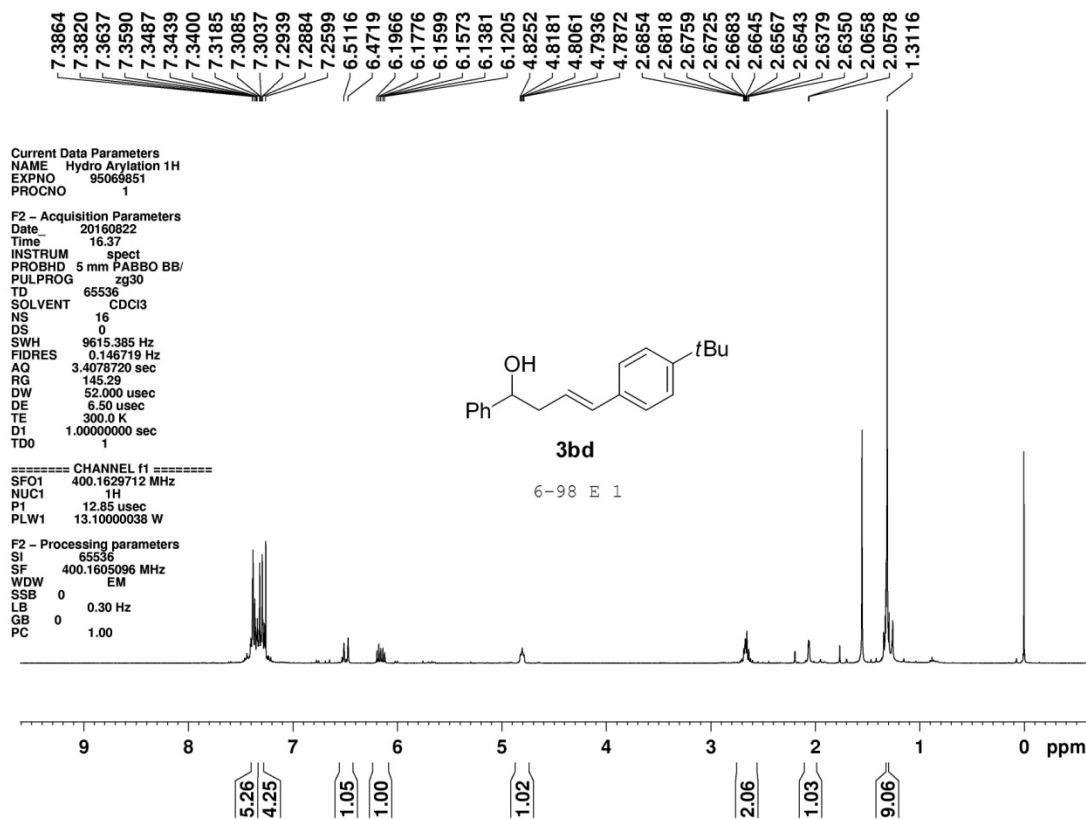


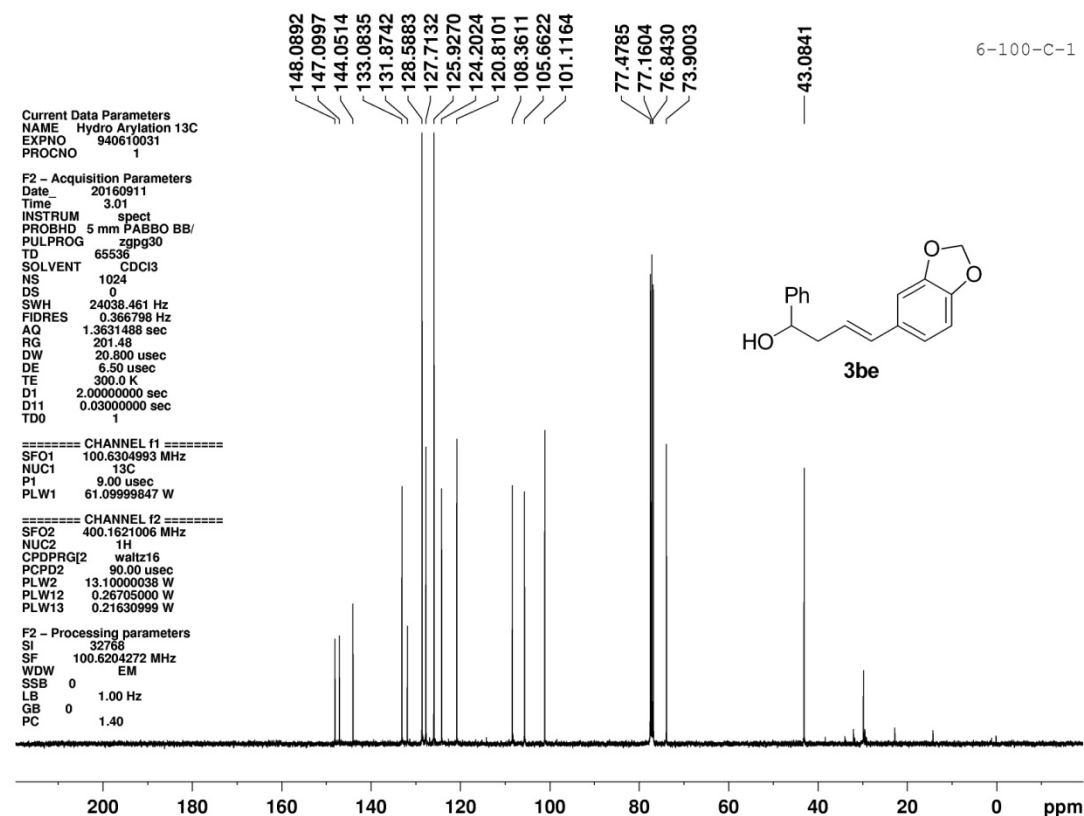
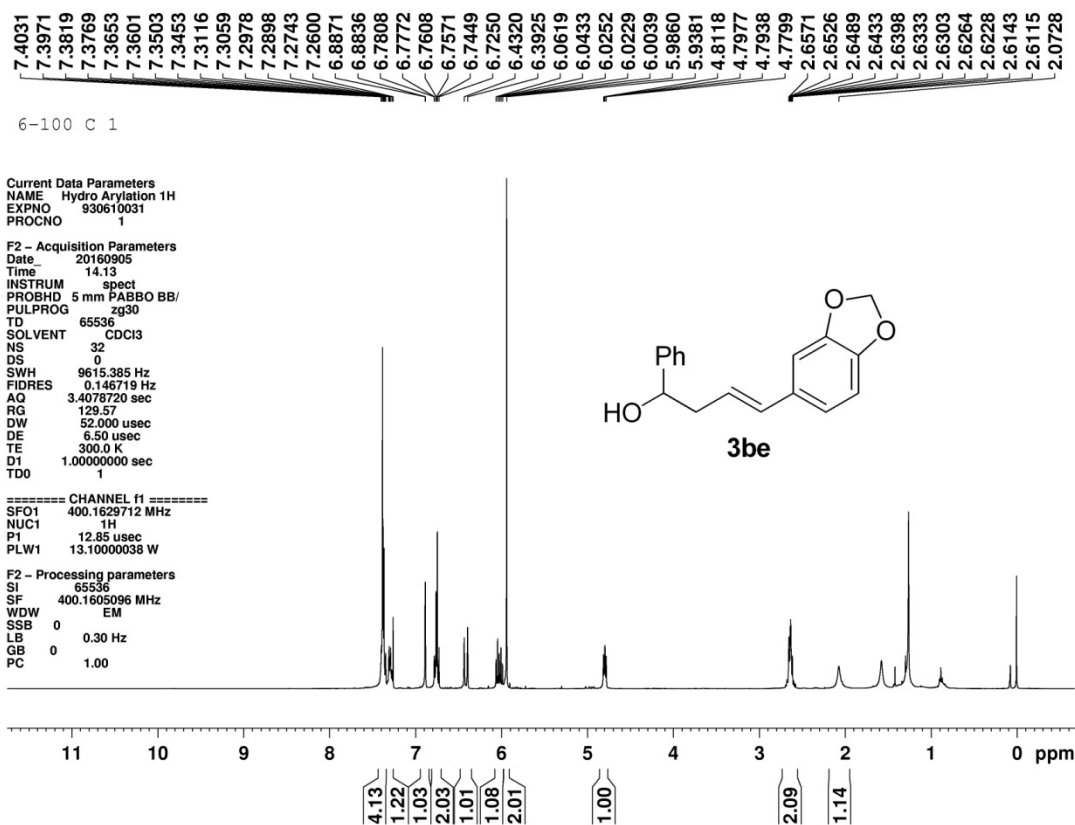


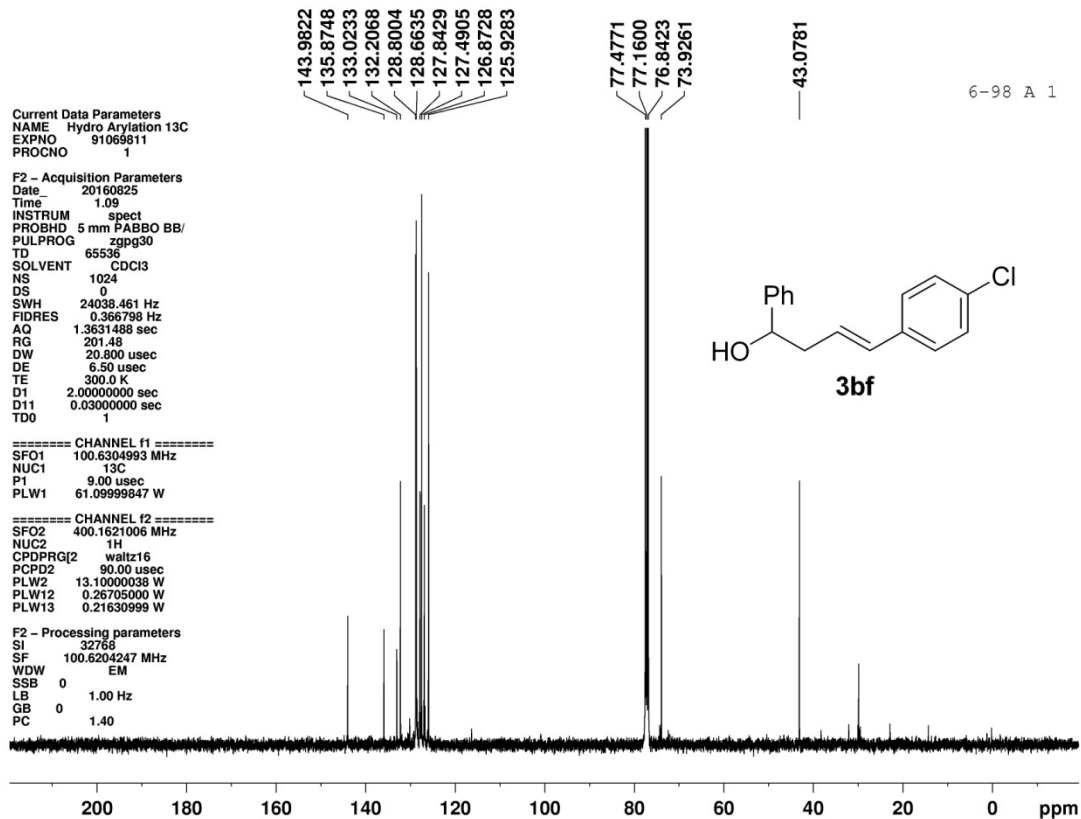
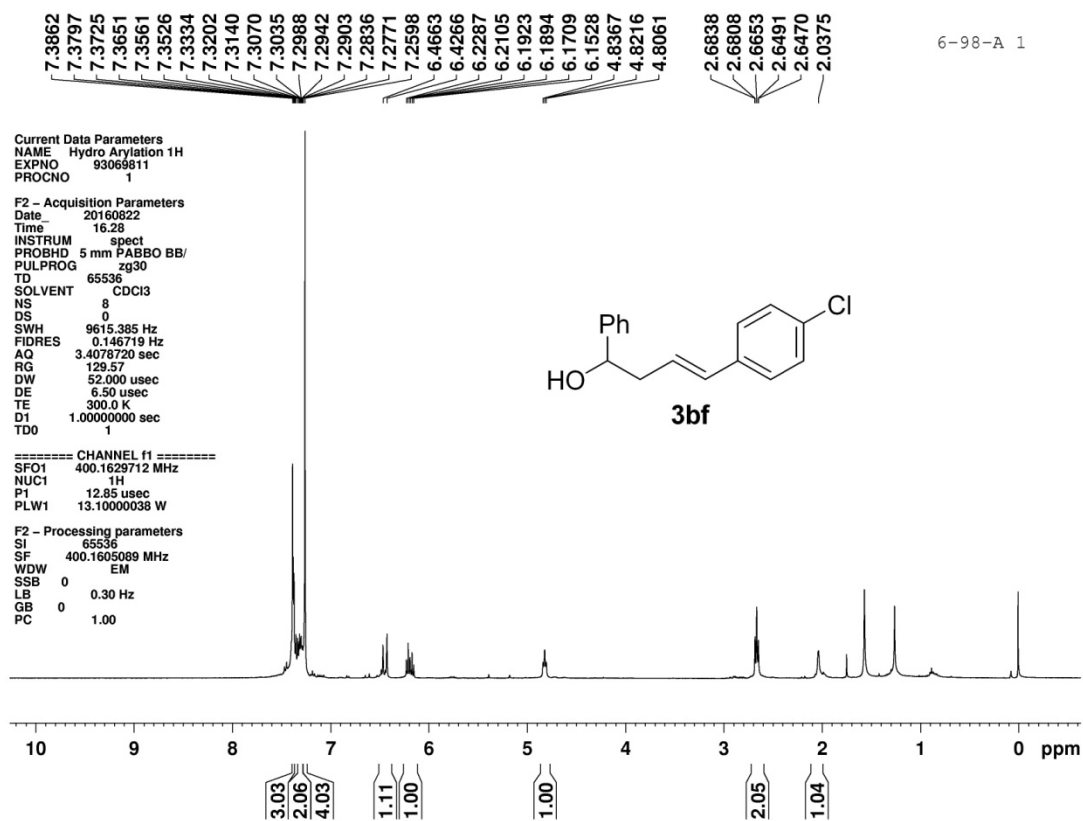


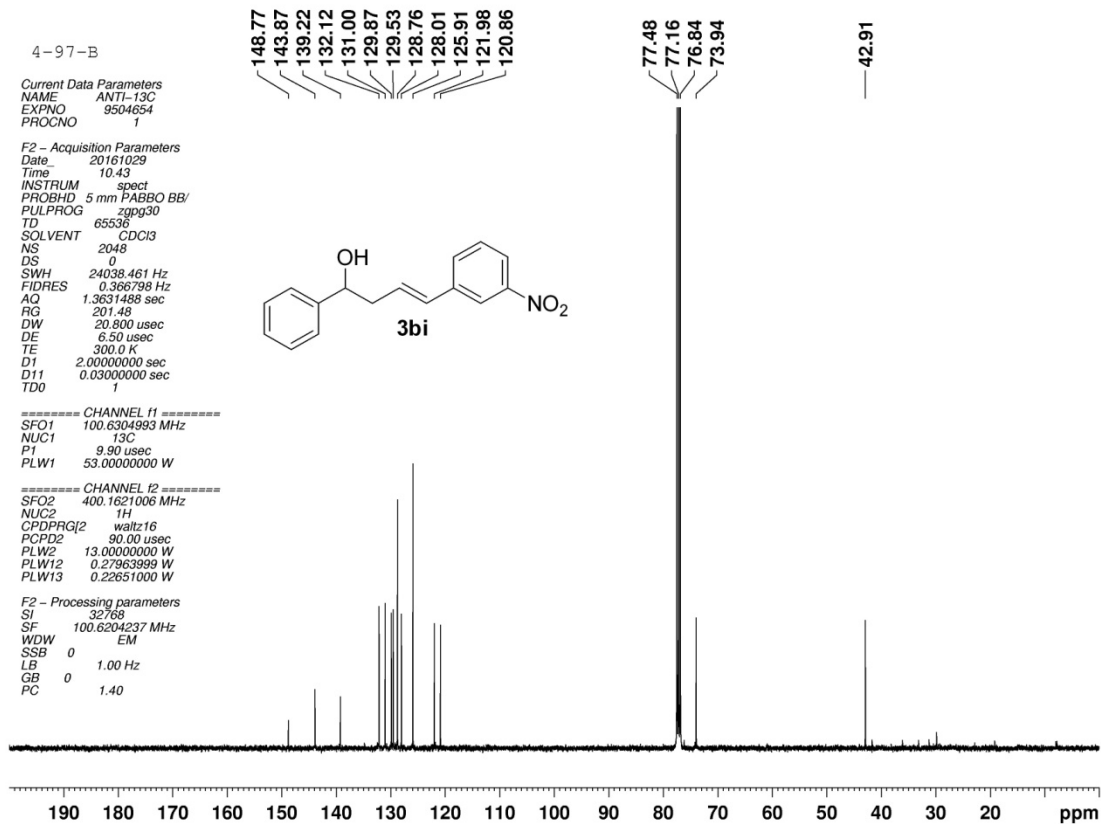
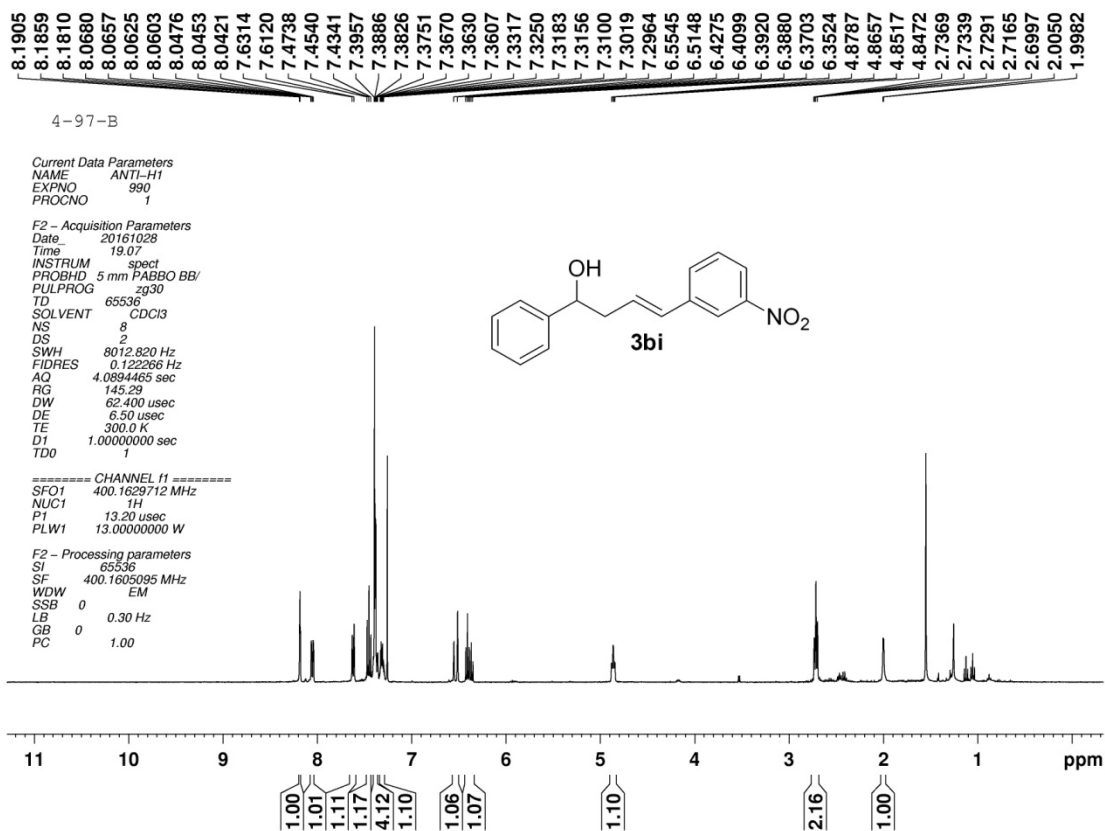


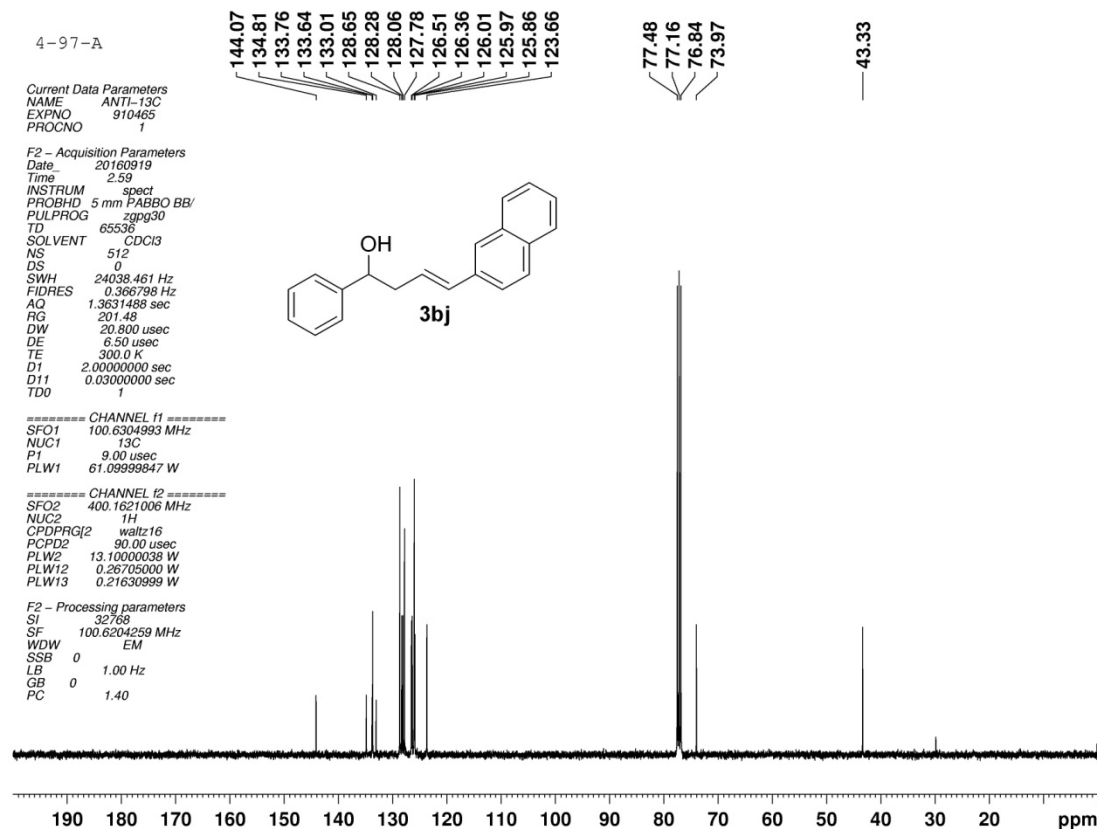
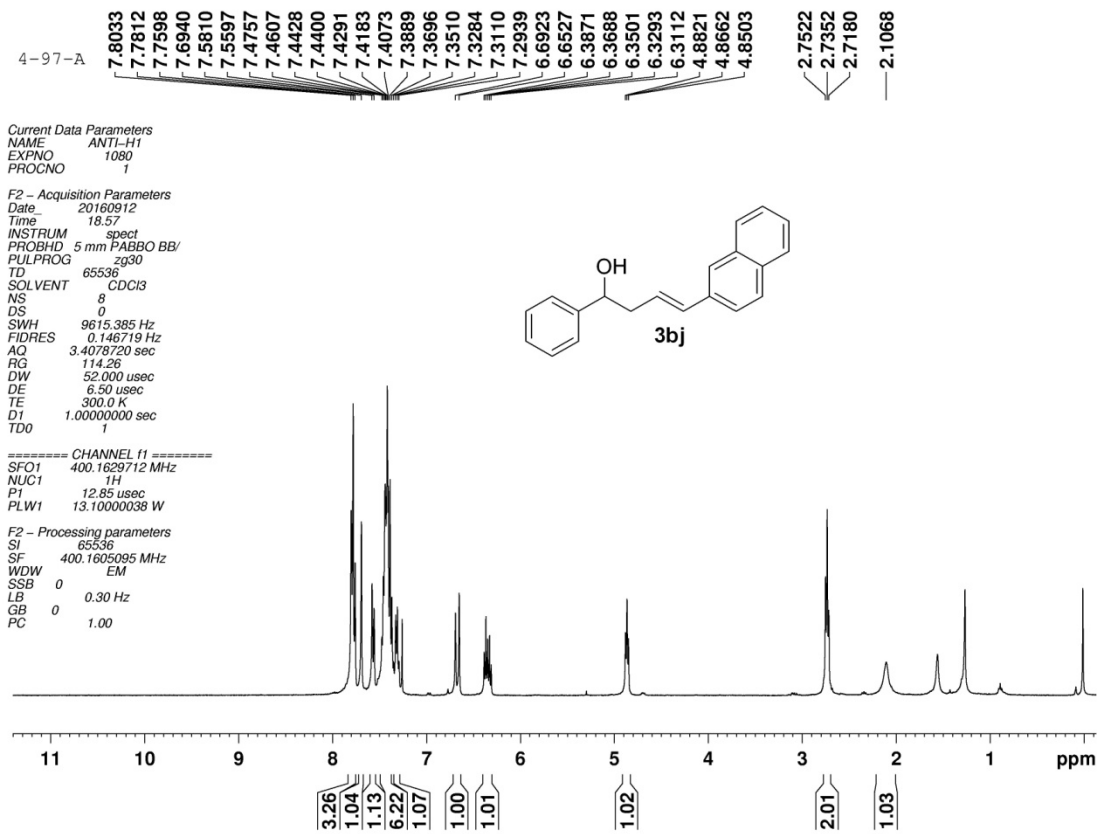


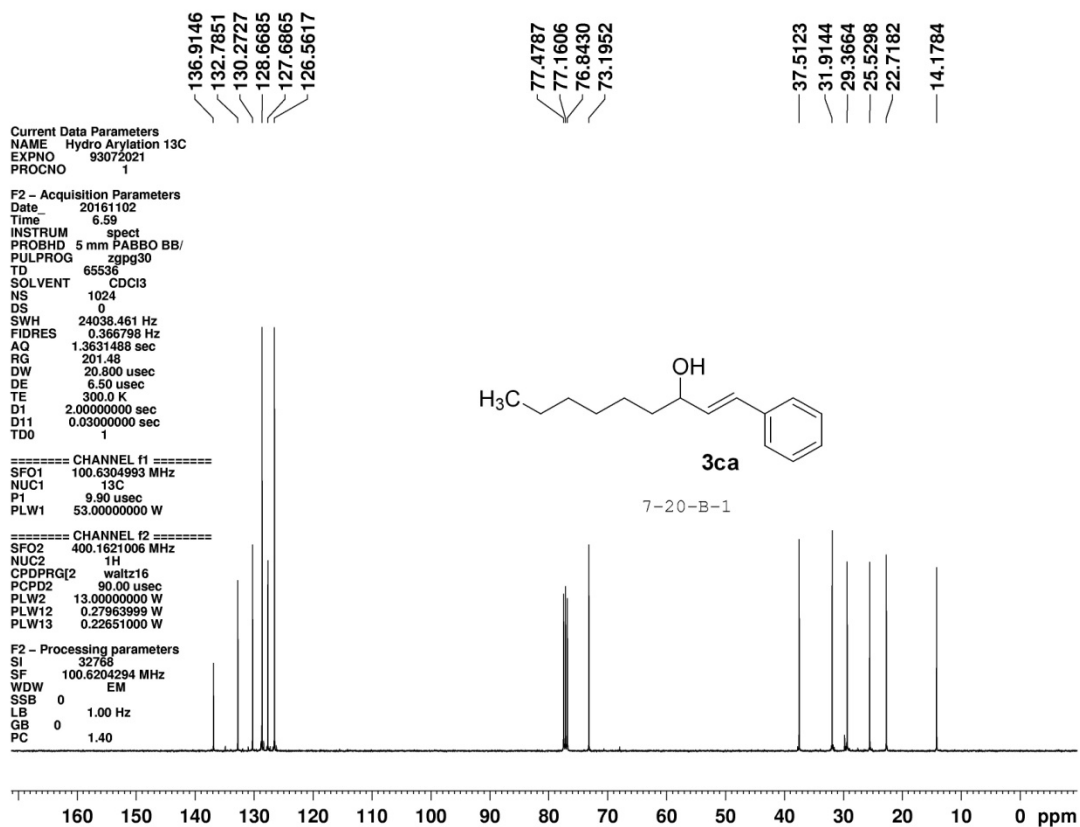
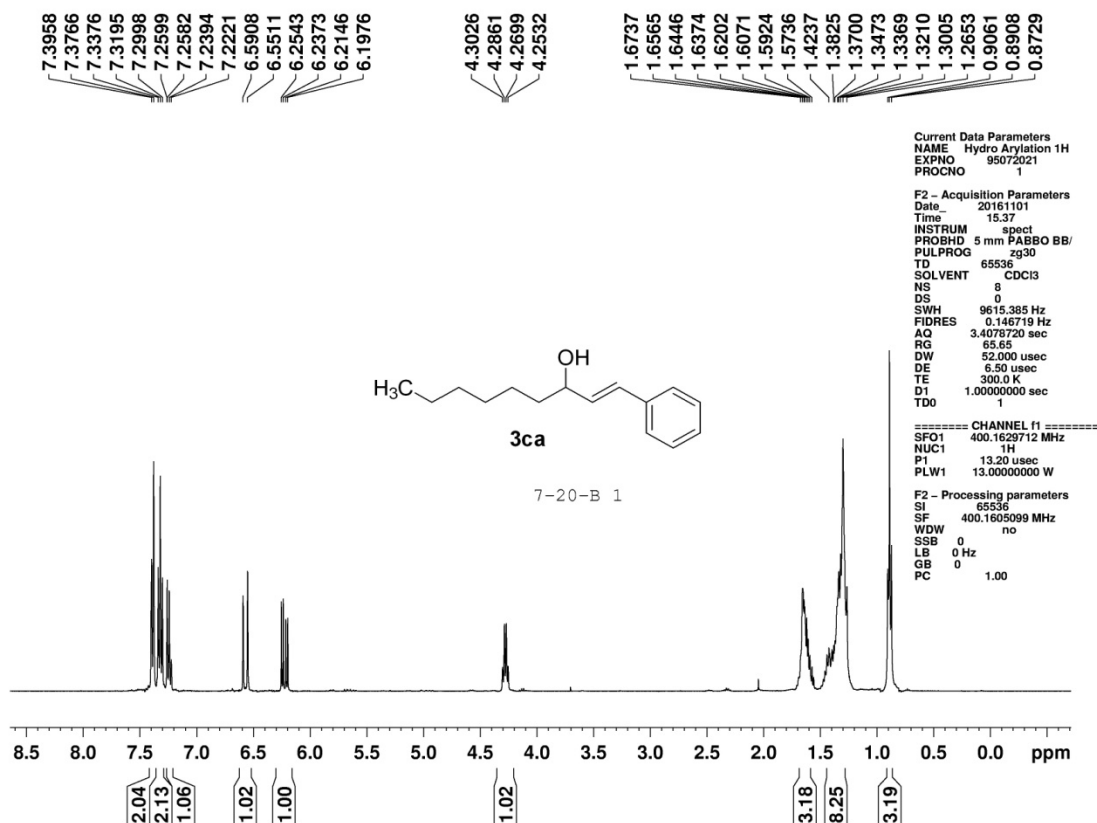


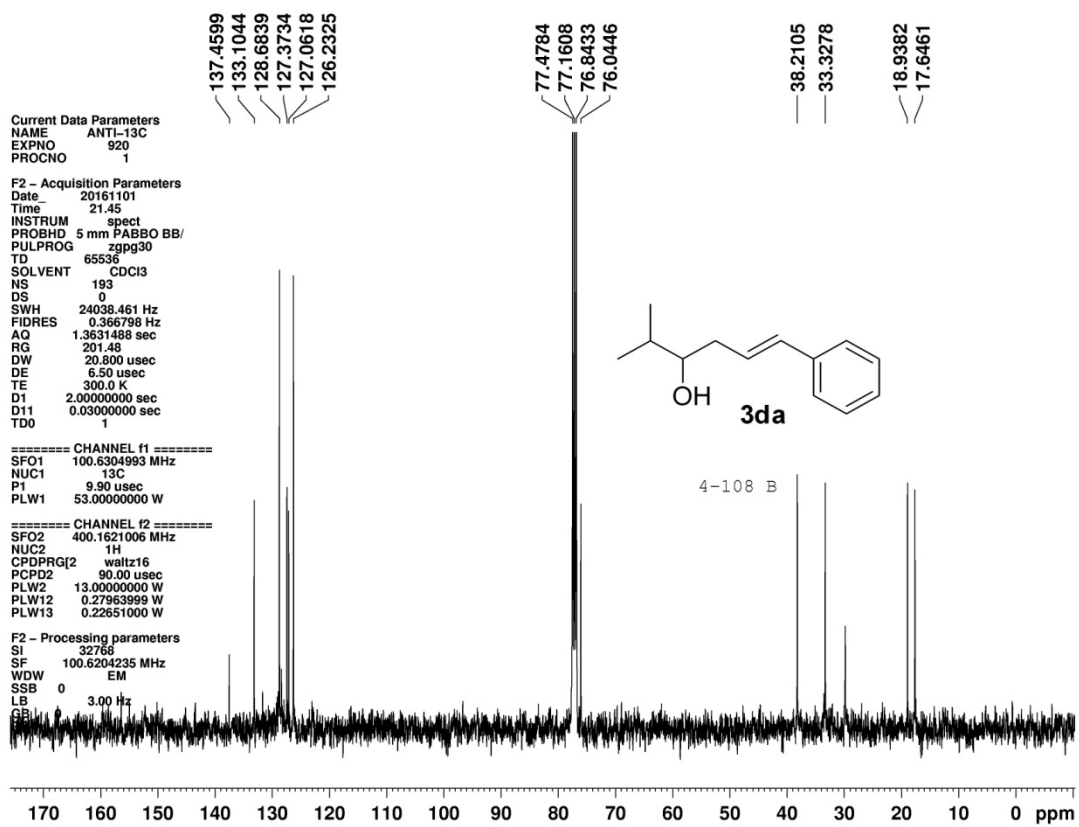
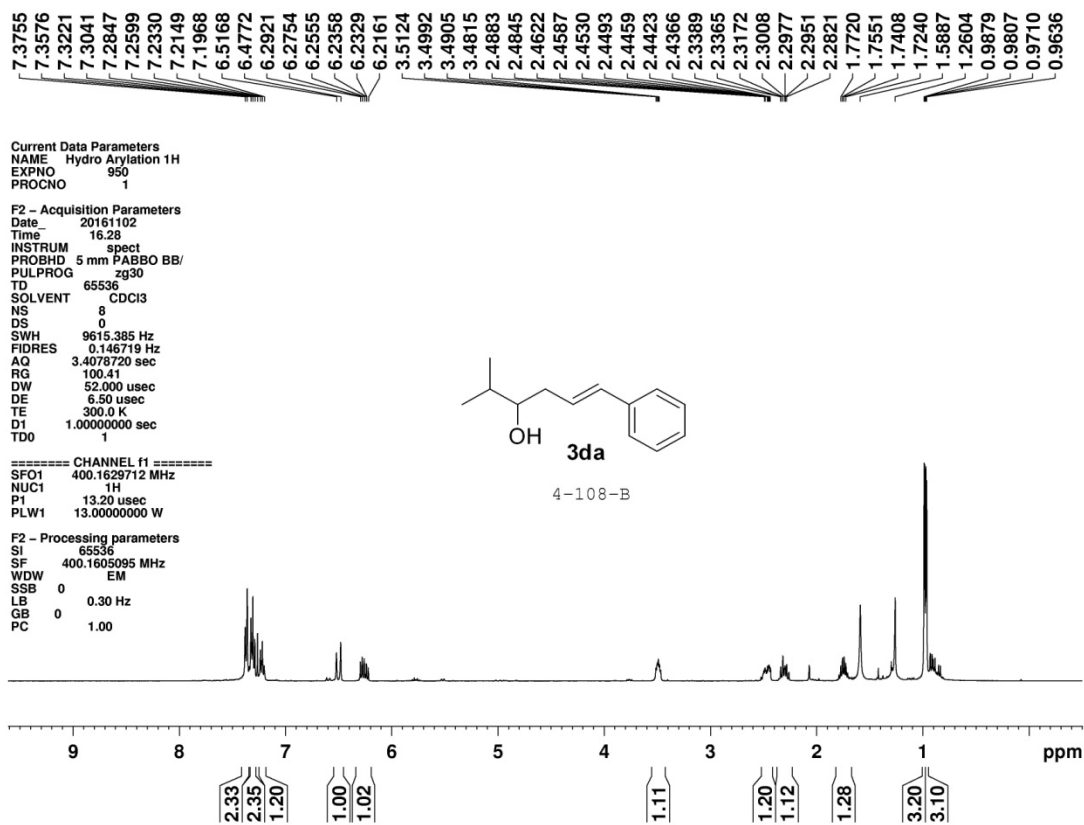


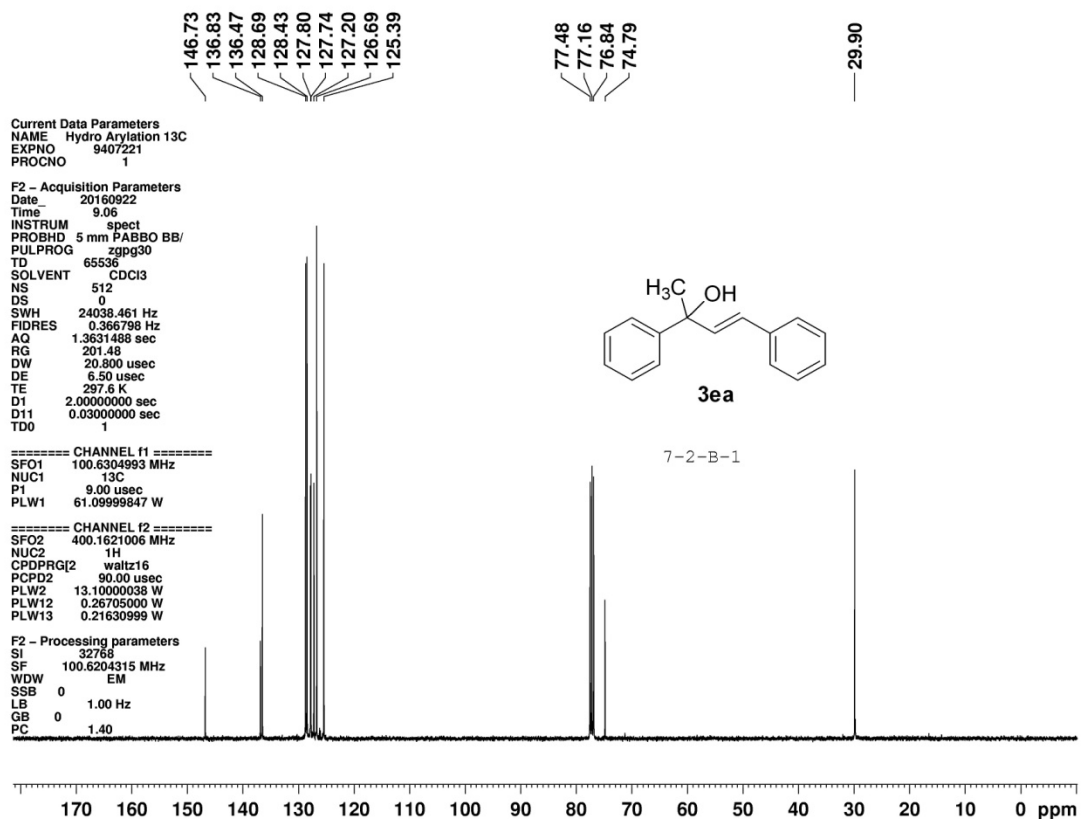
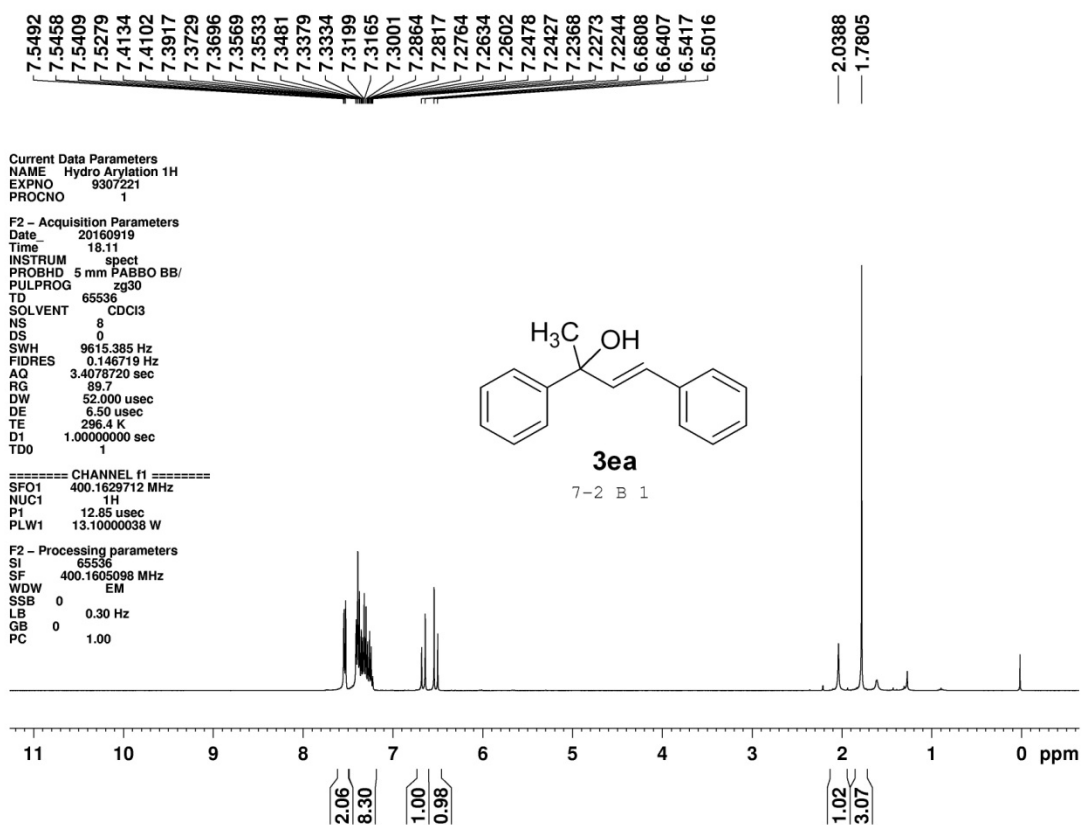


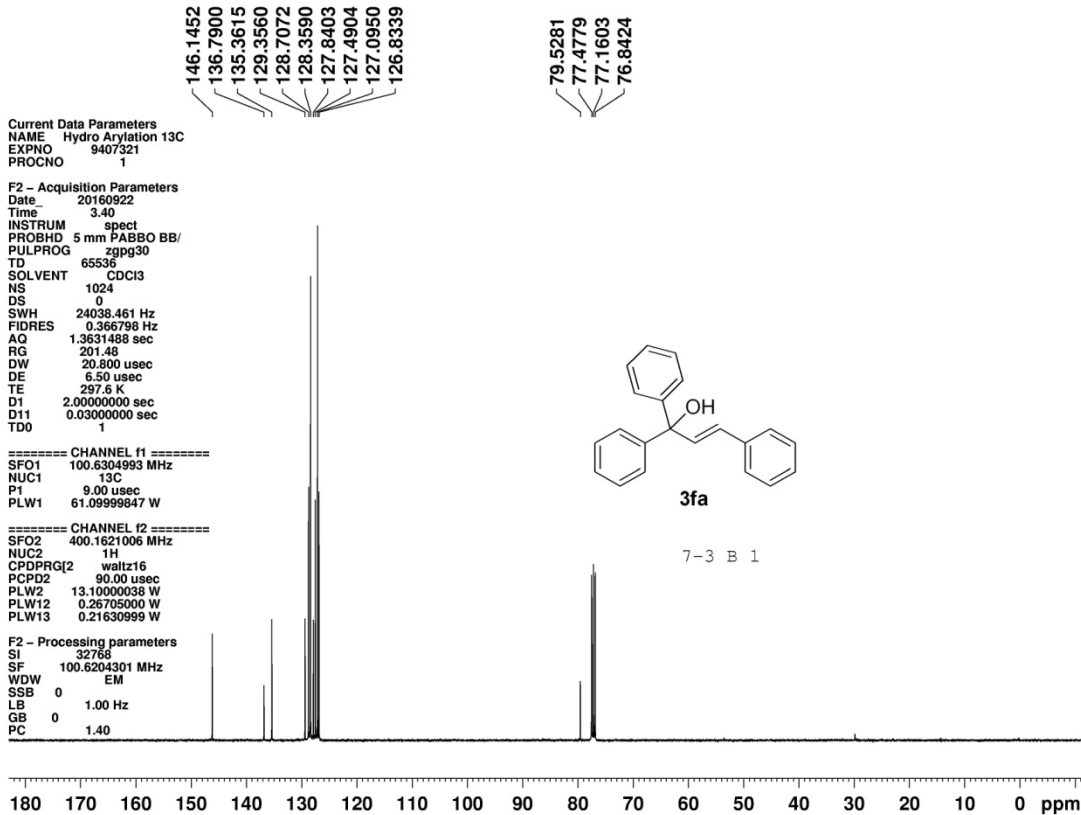
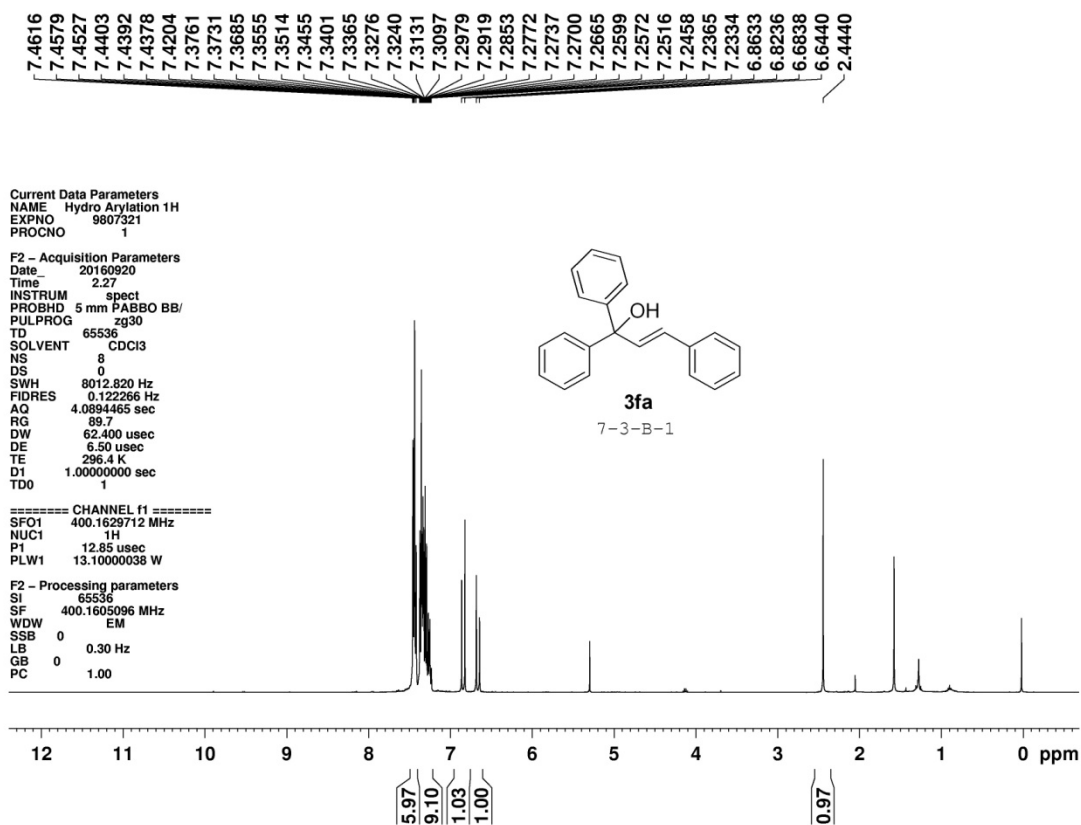


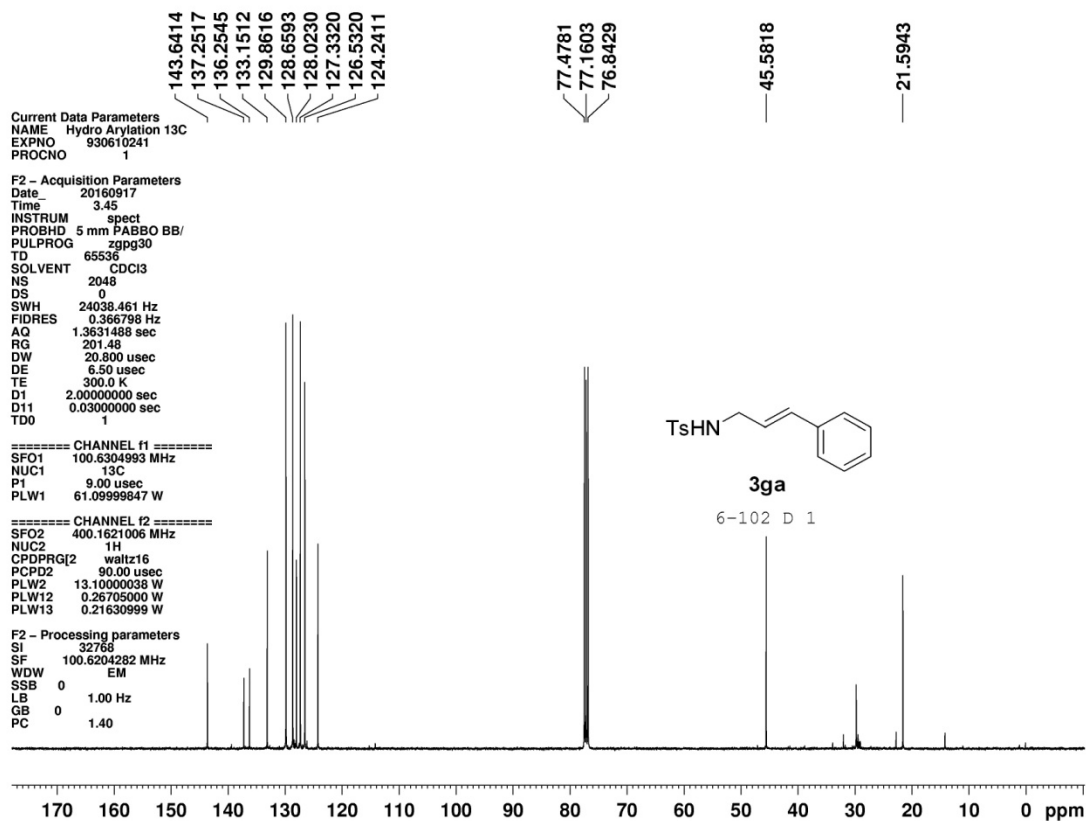
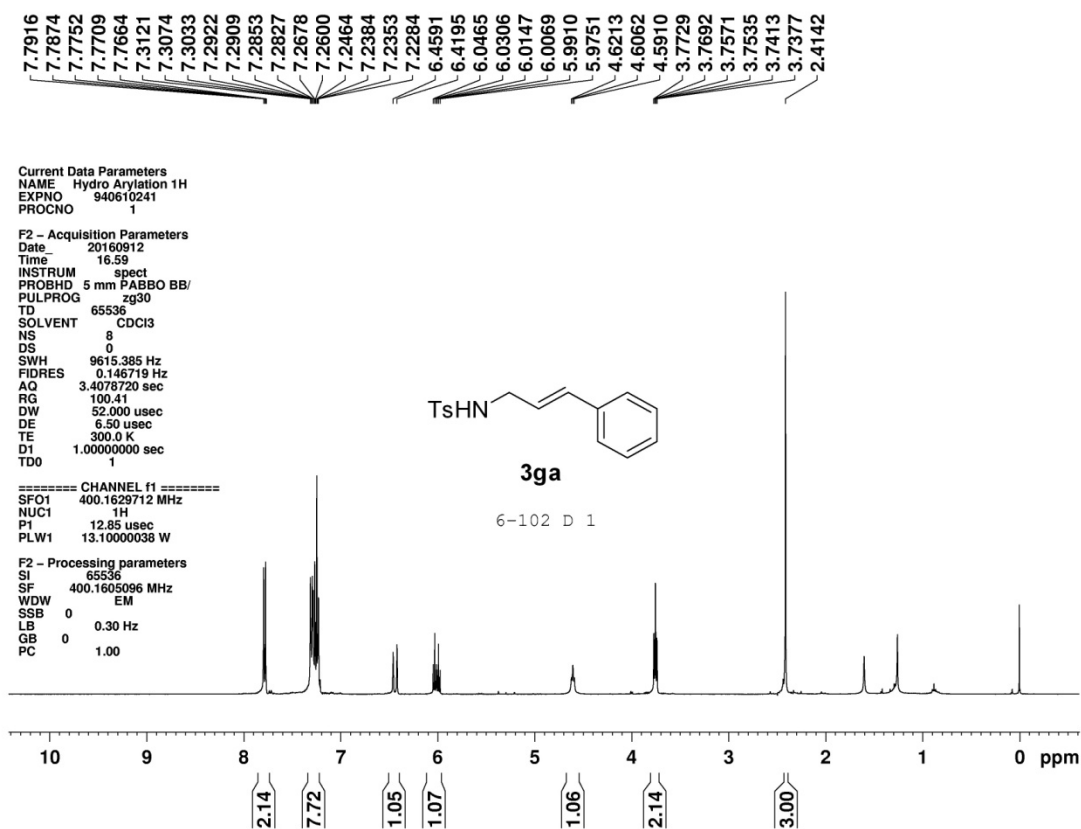


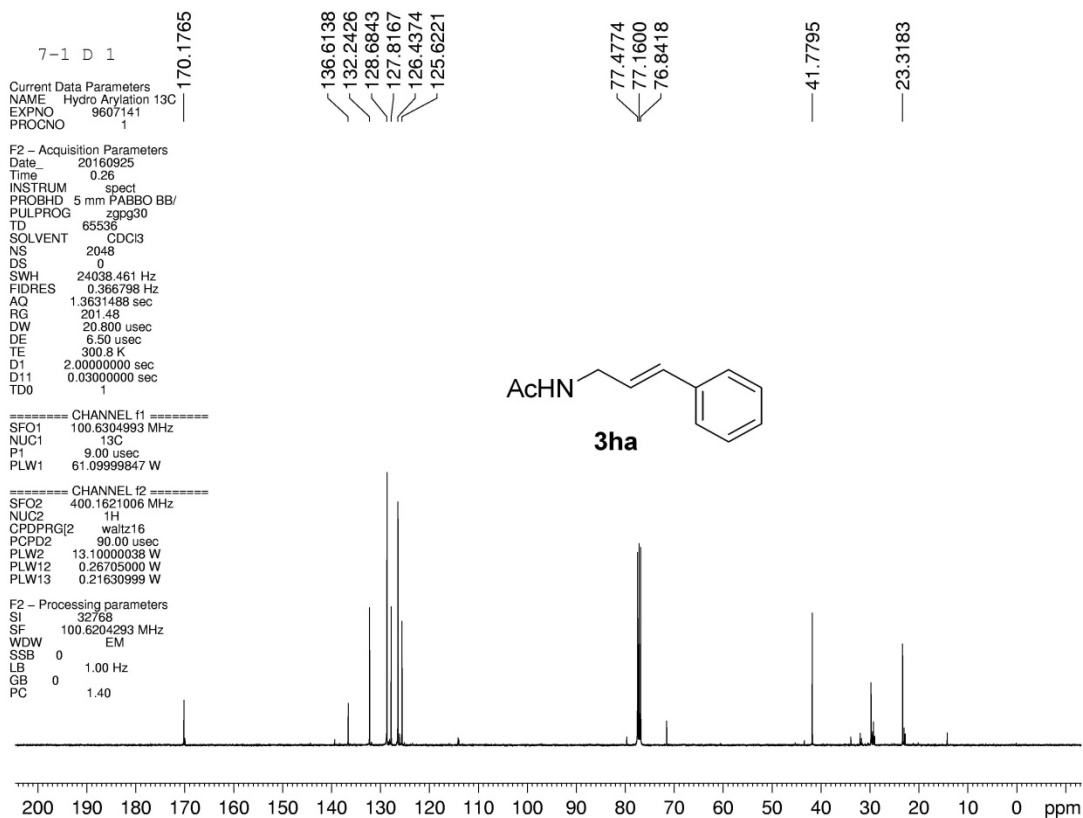
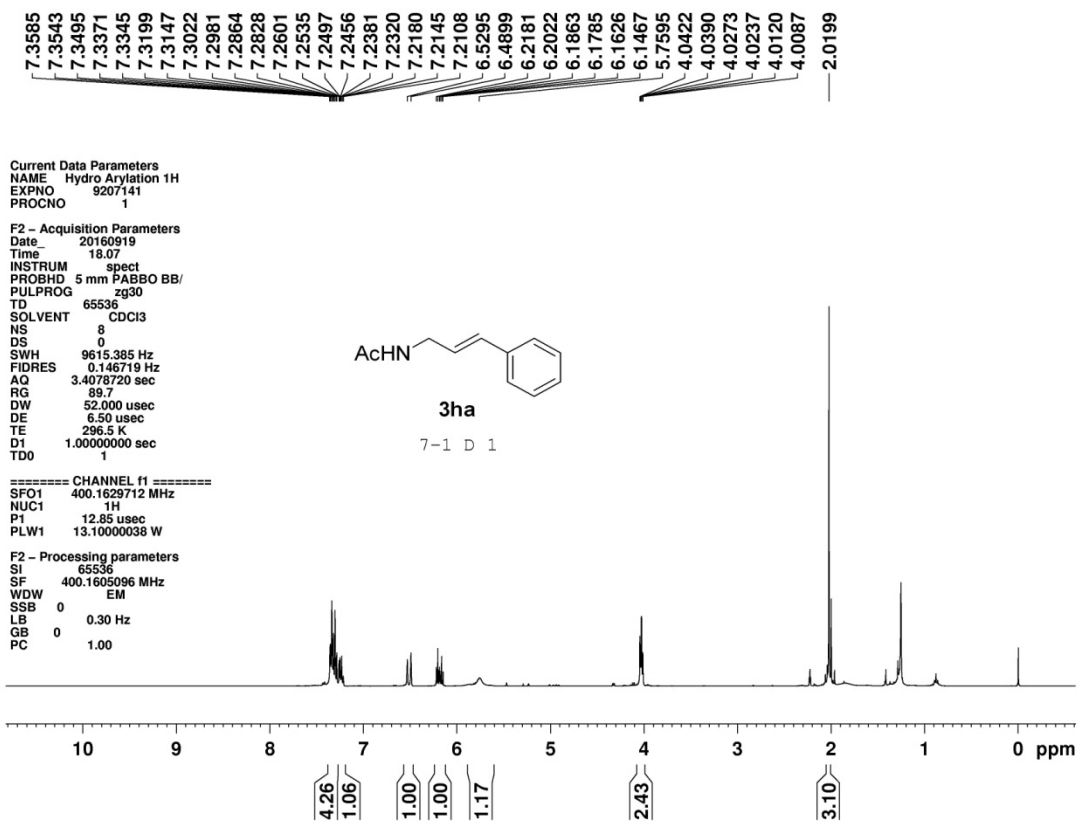


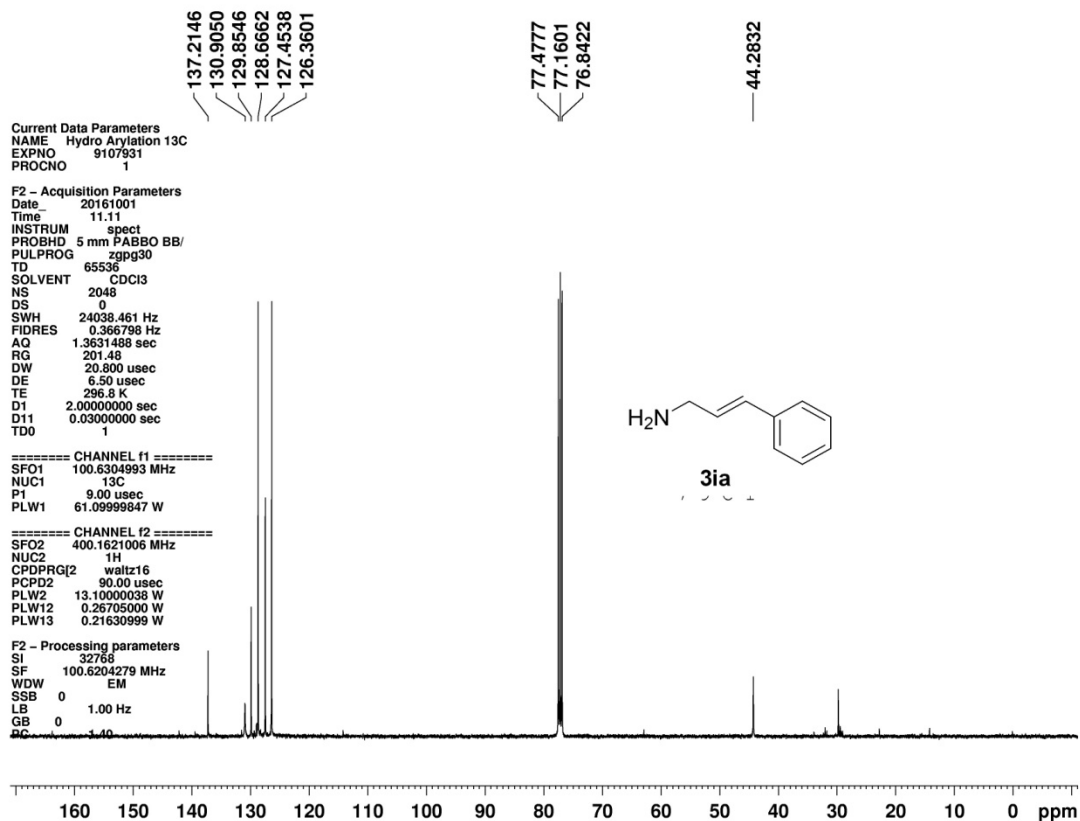
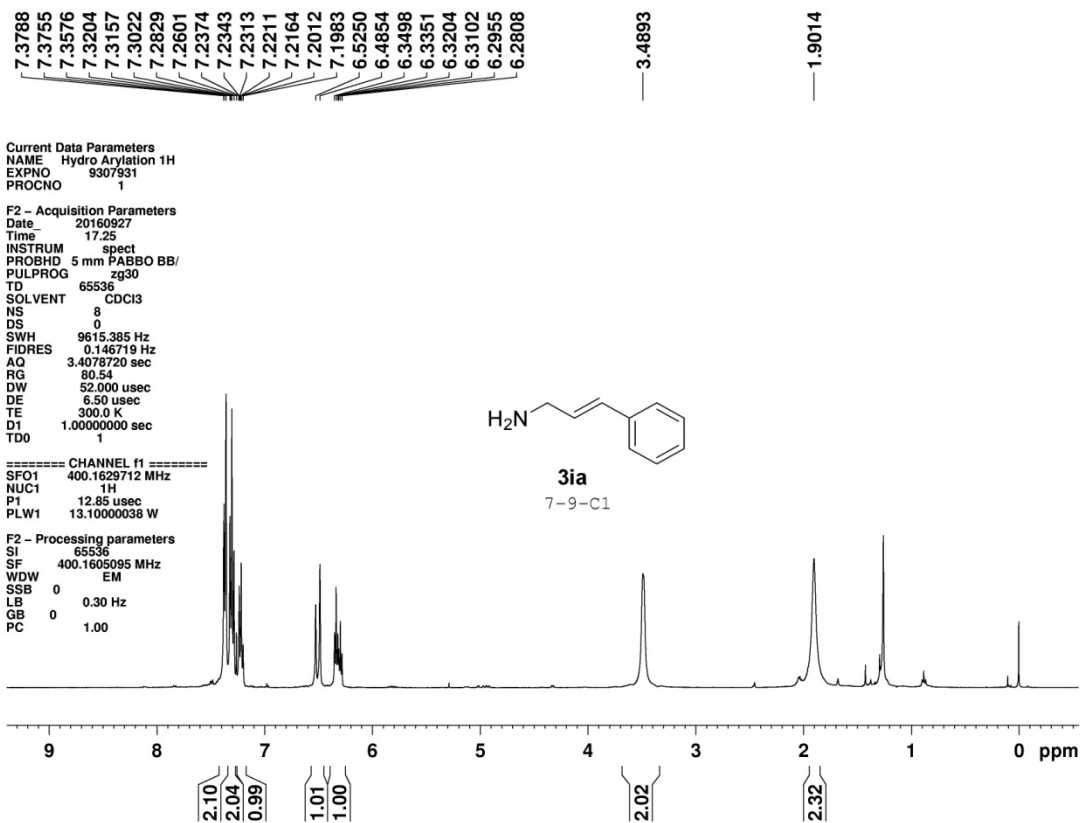


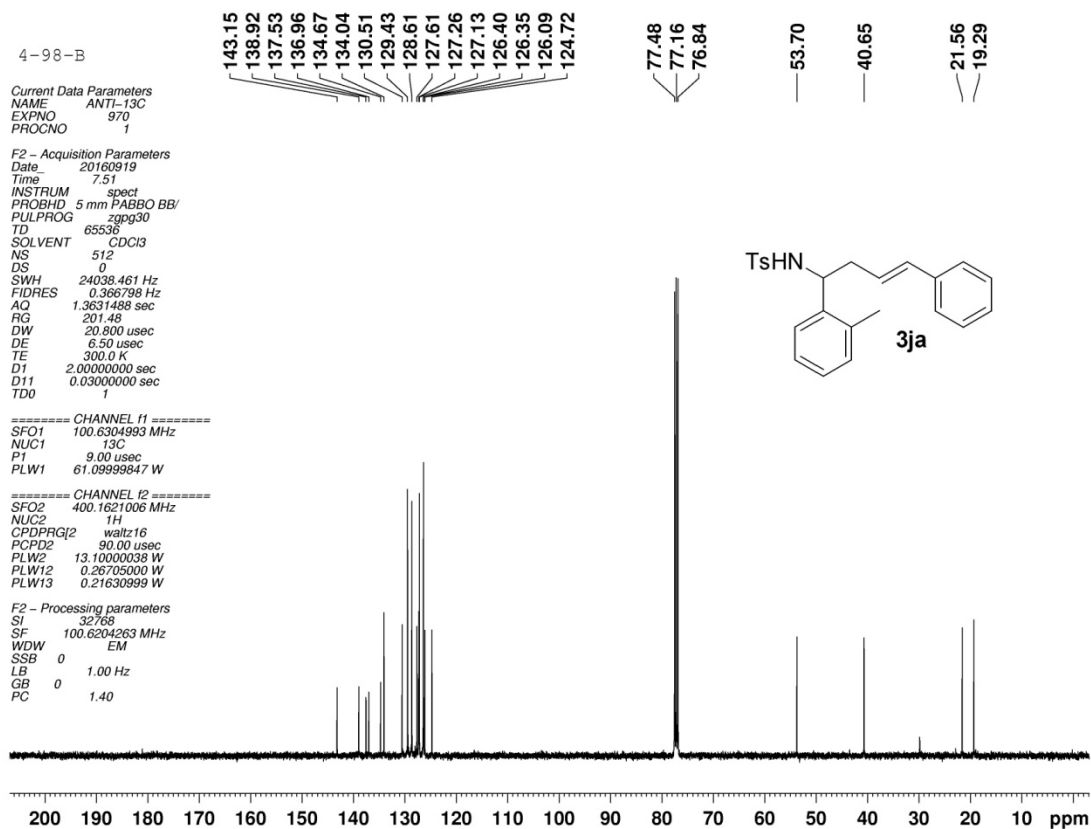
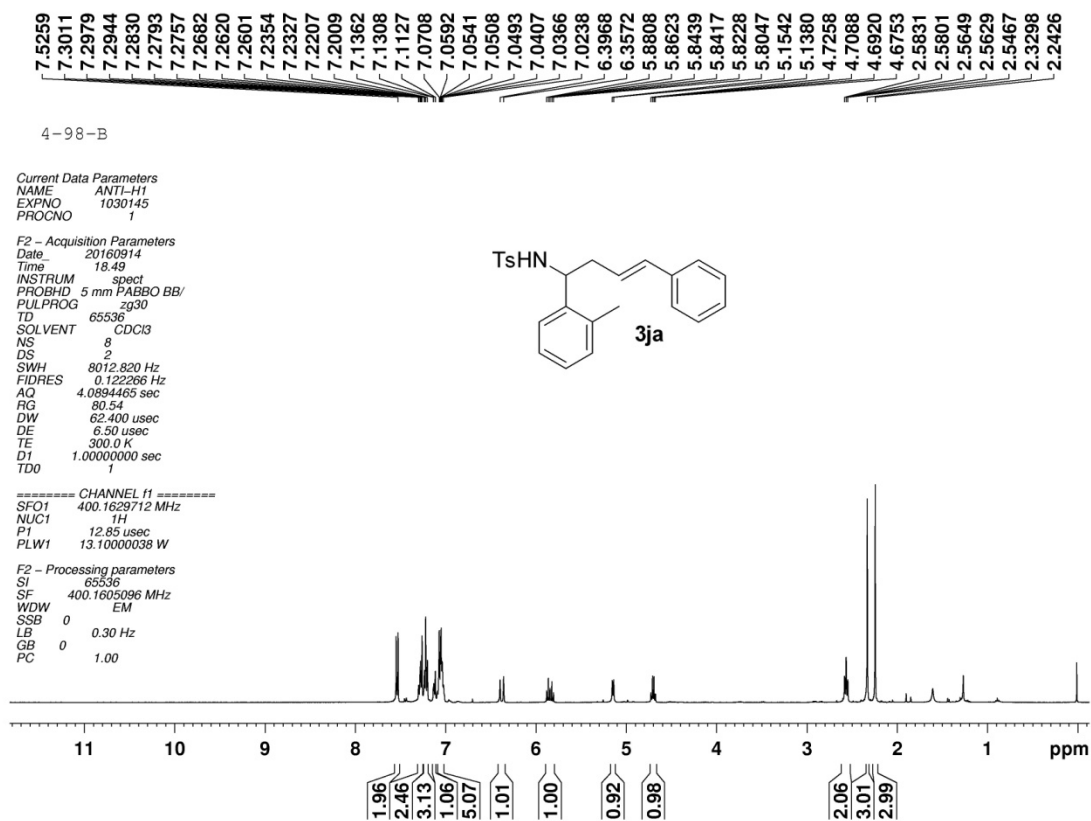


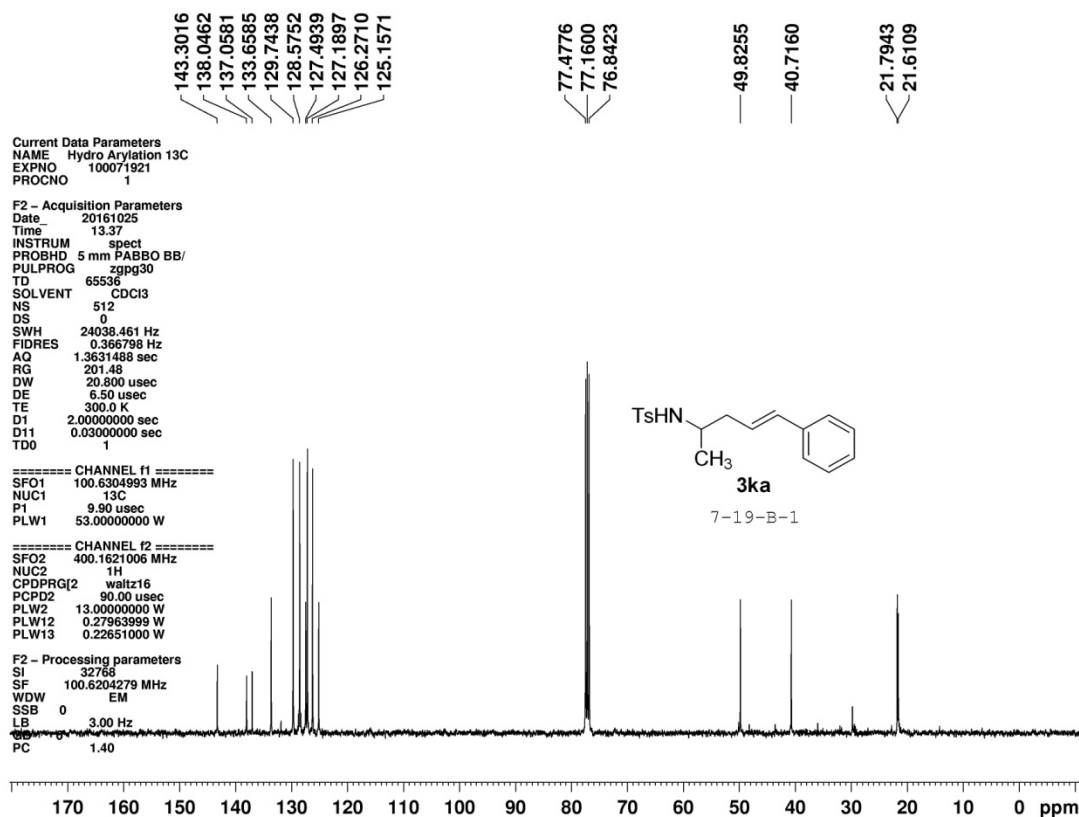
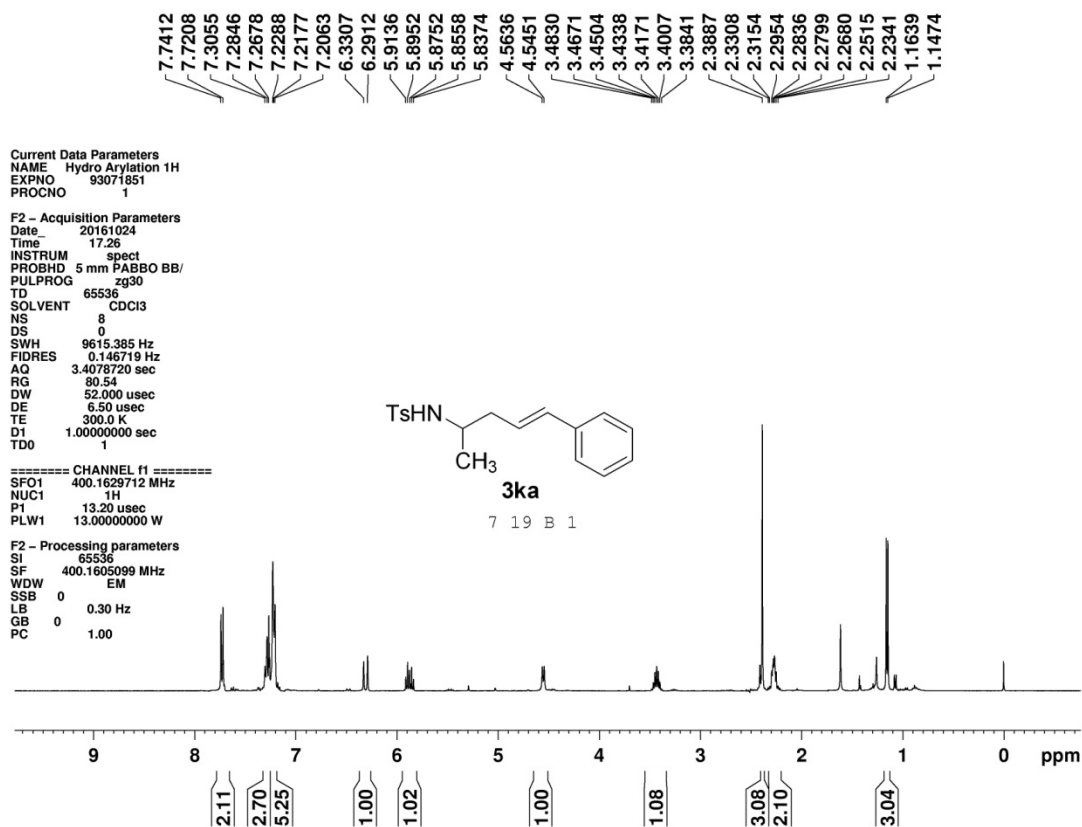


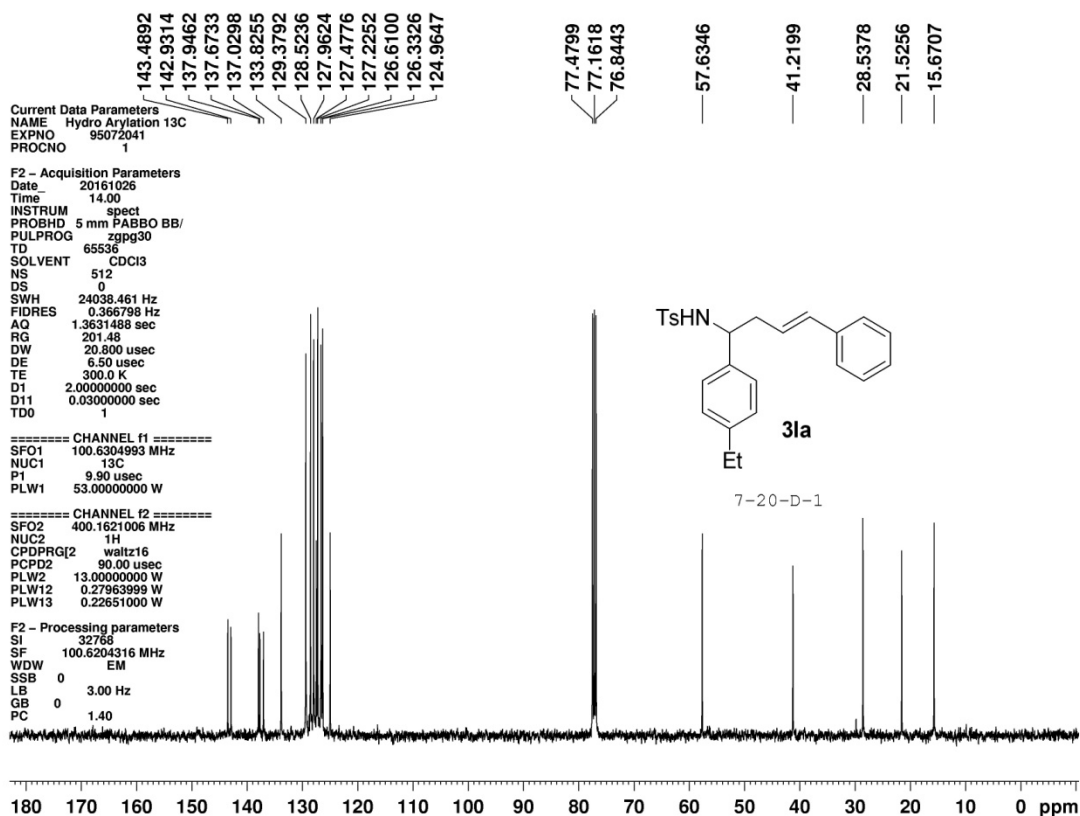
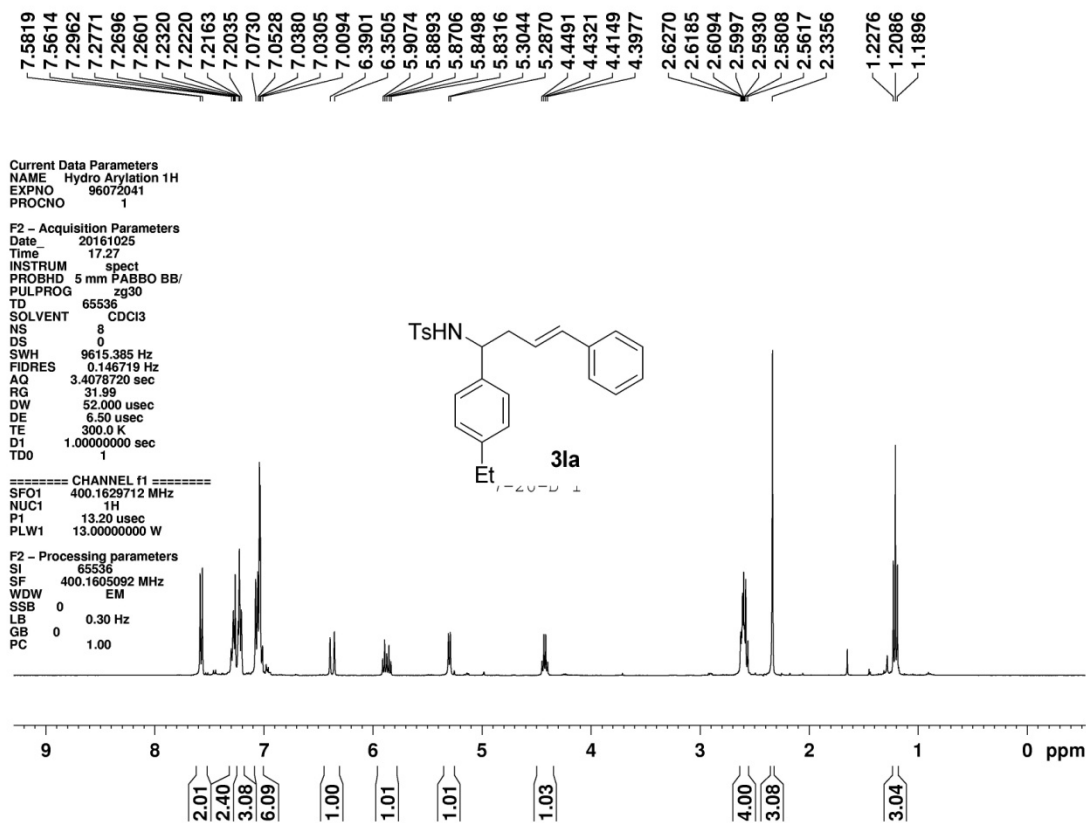


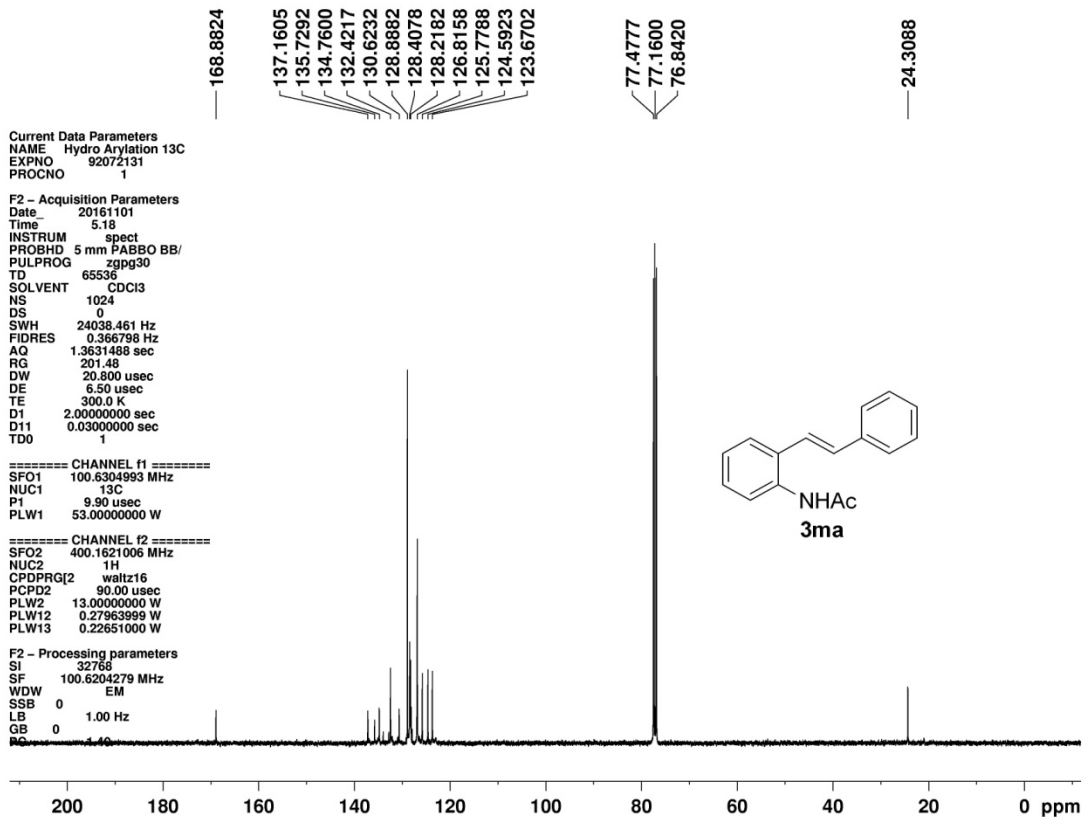
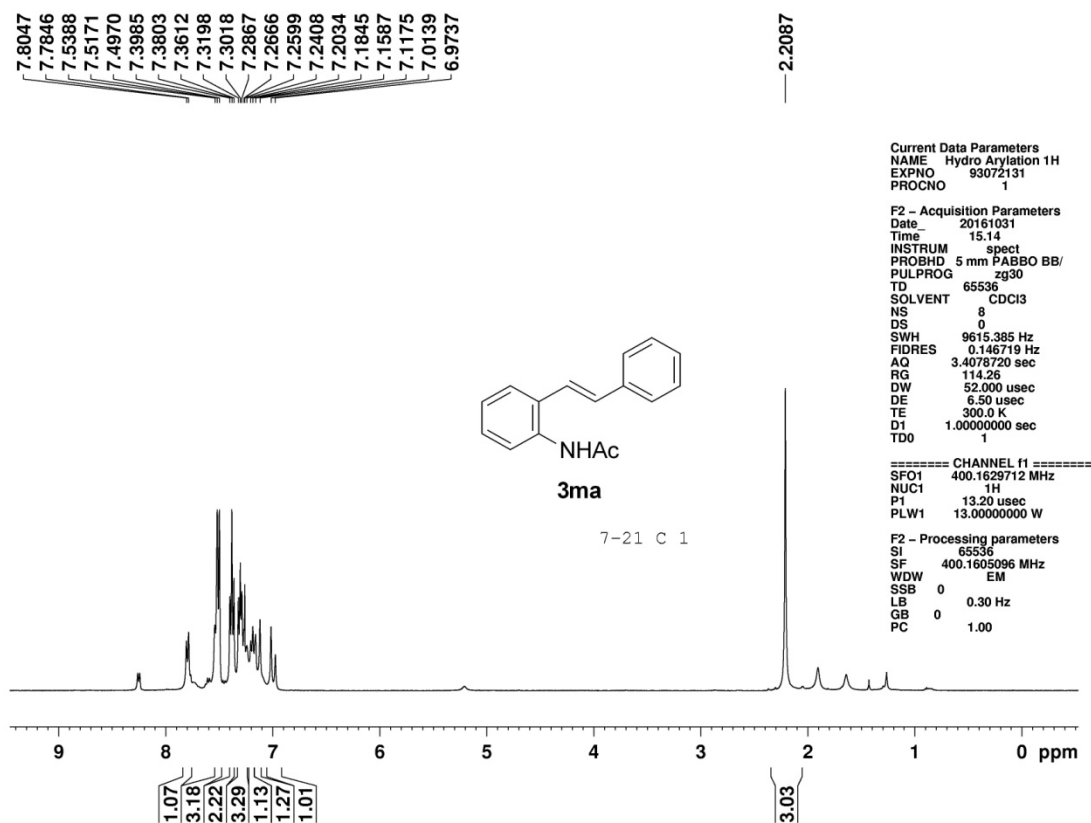


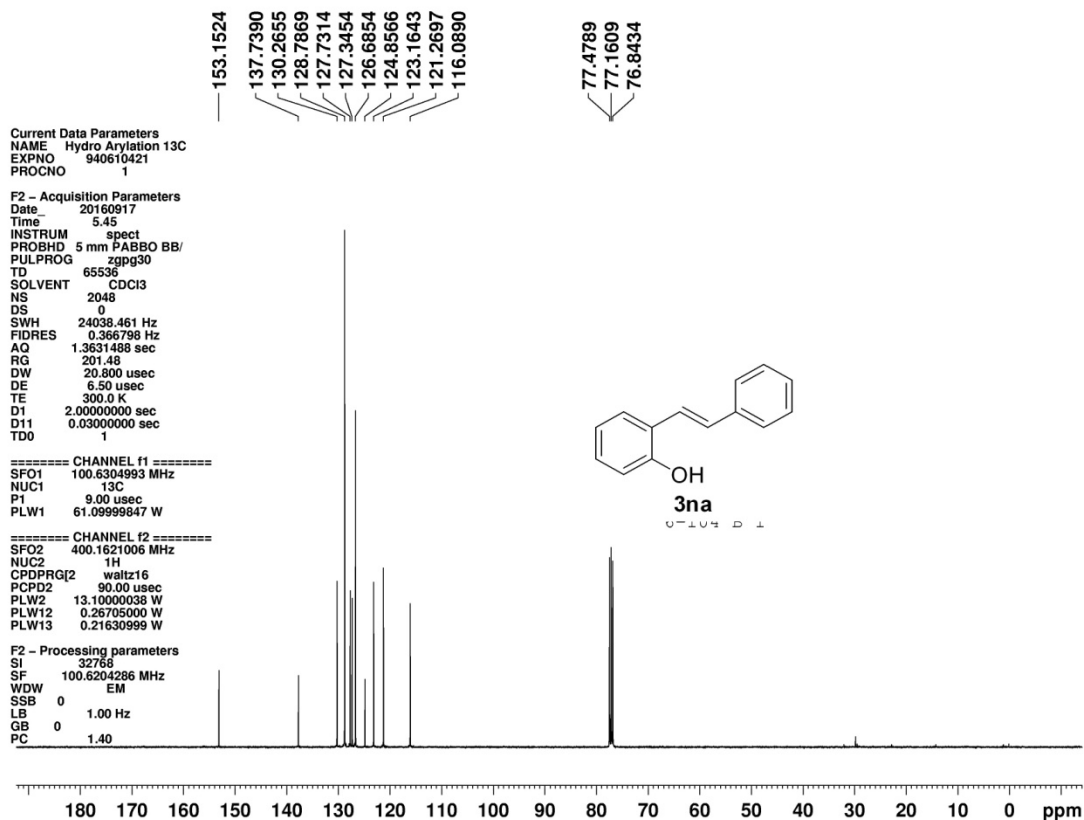
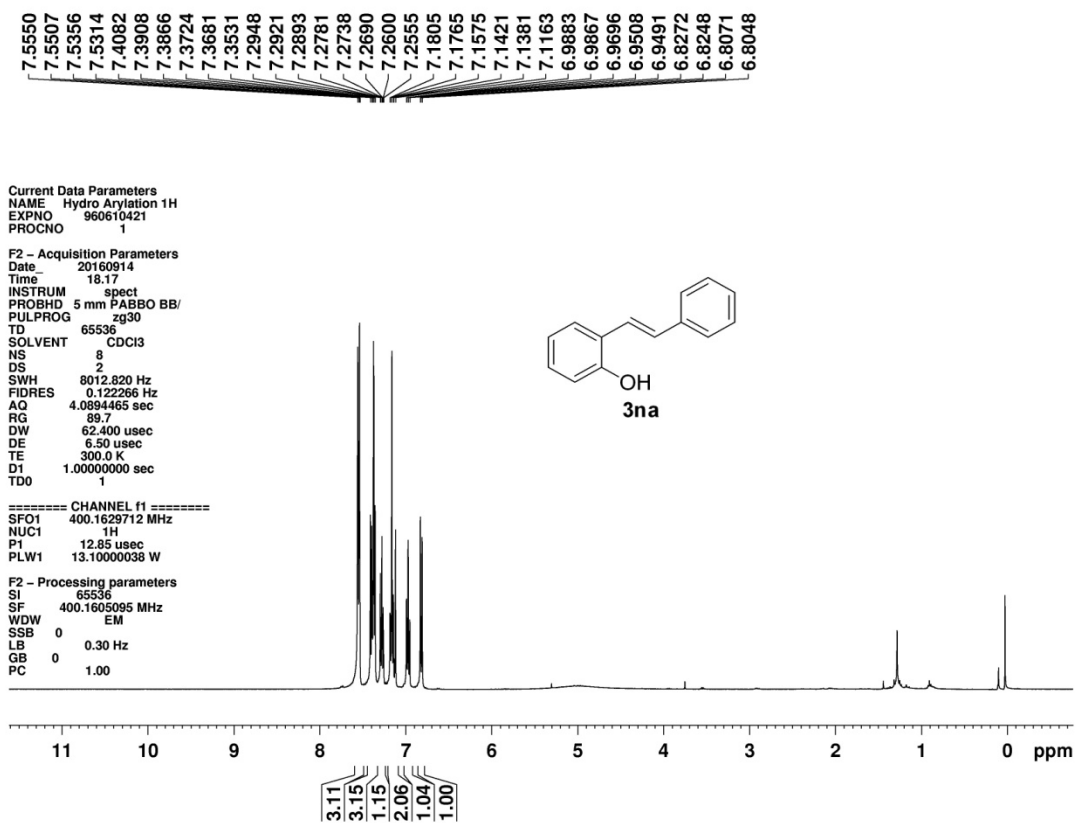


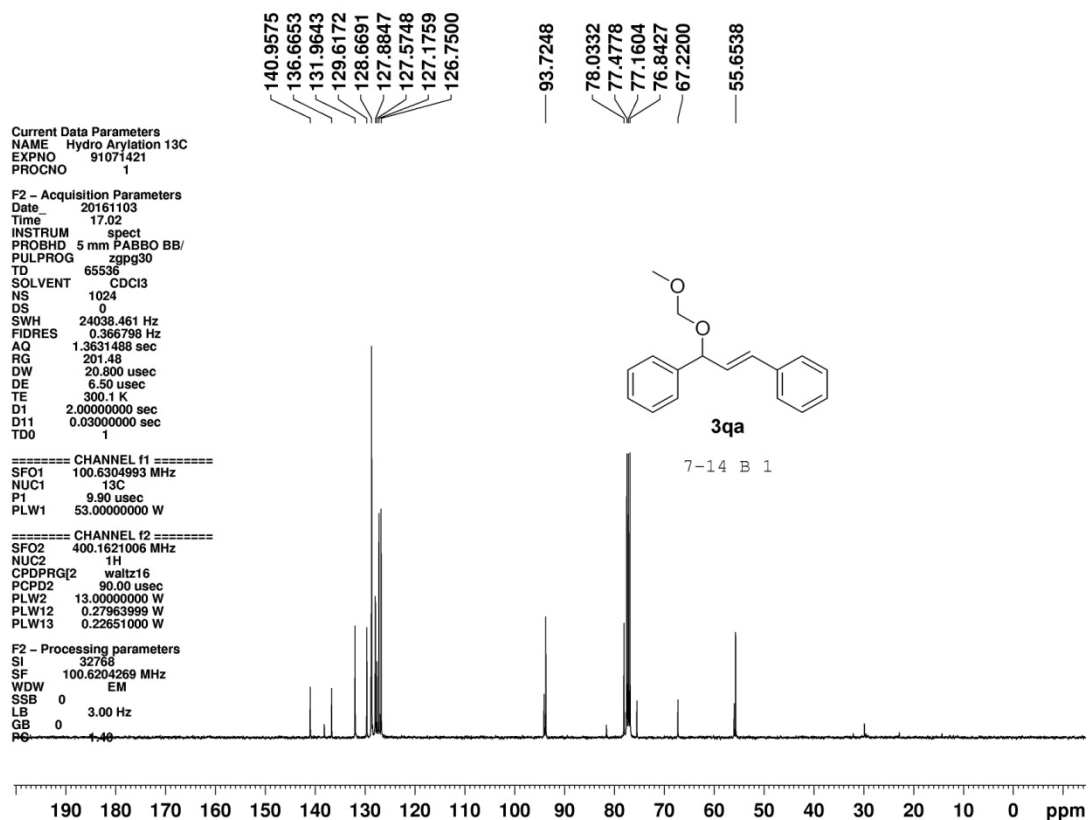
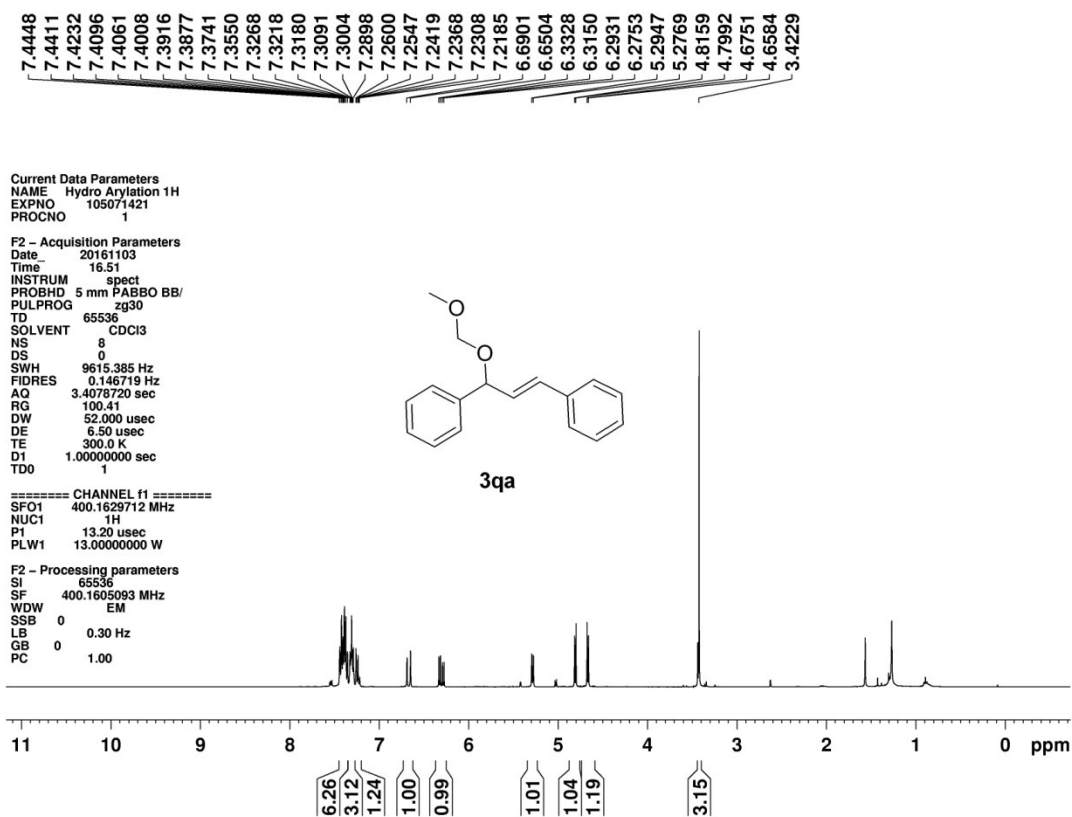








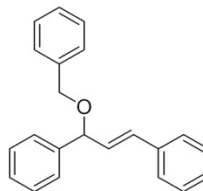




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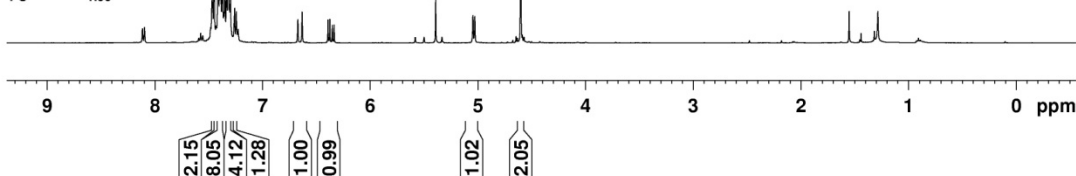
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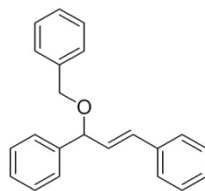
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7-20-C 1

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7-20-C 1

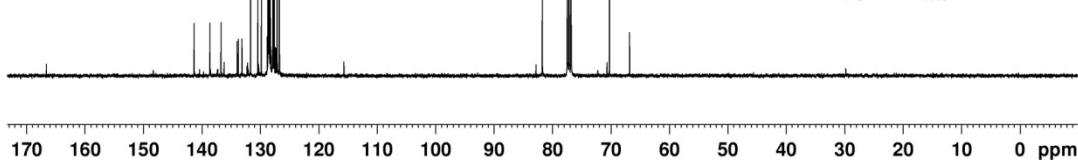
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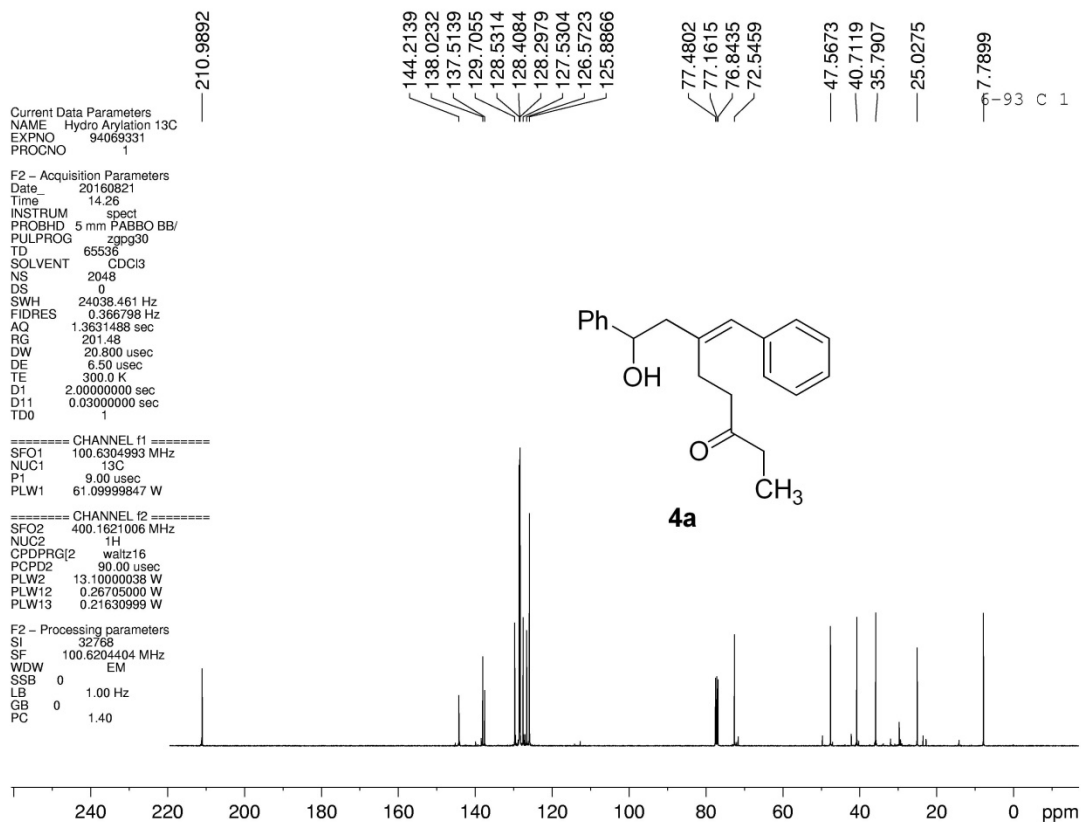
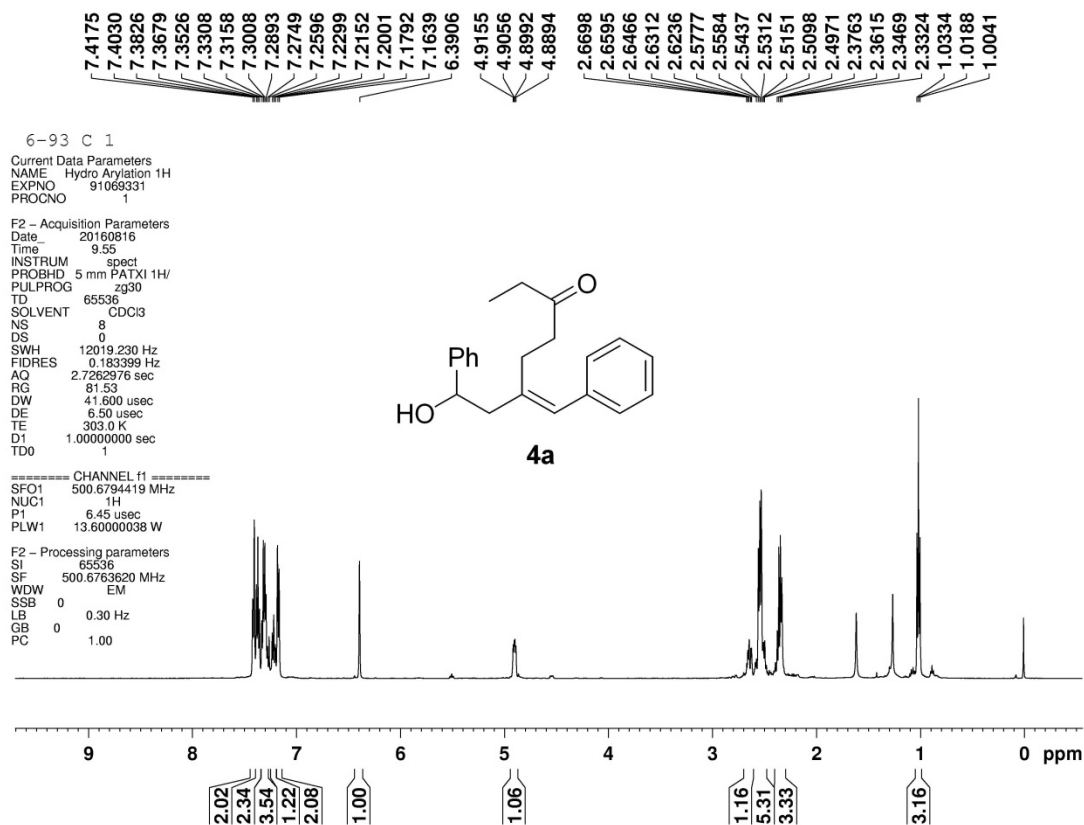
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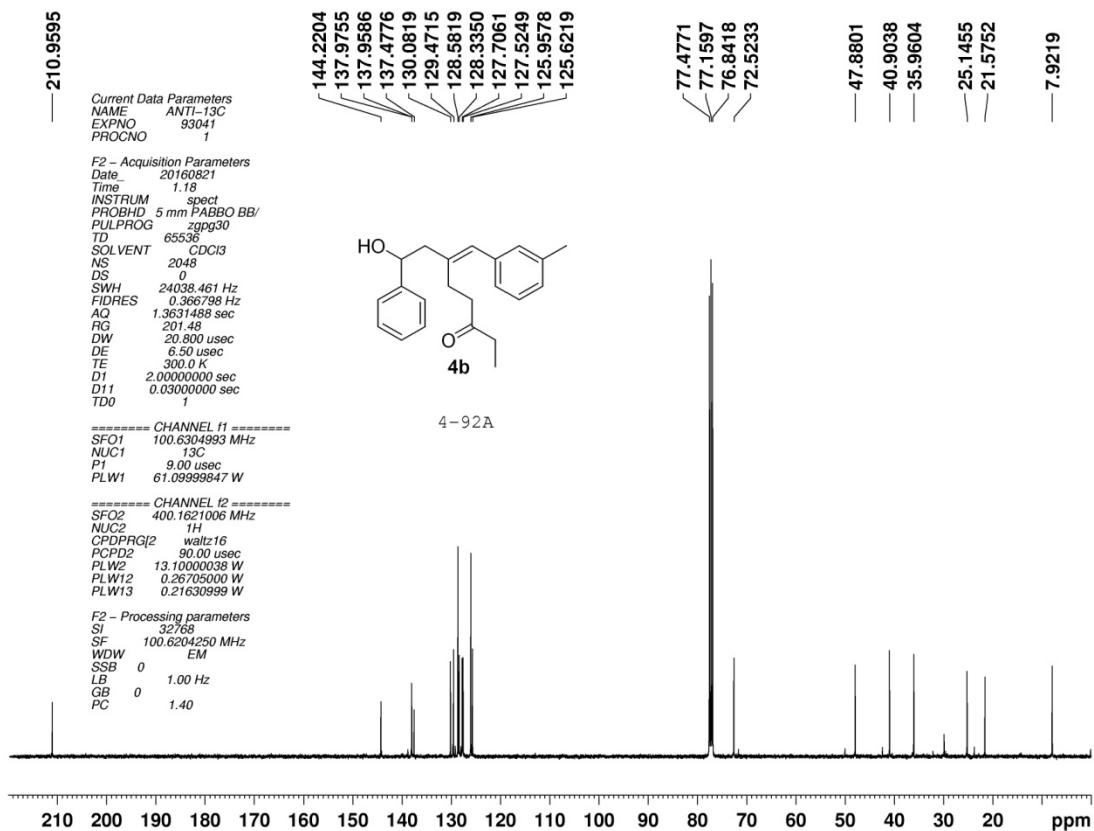
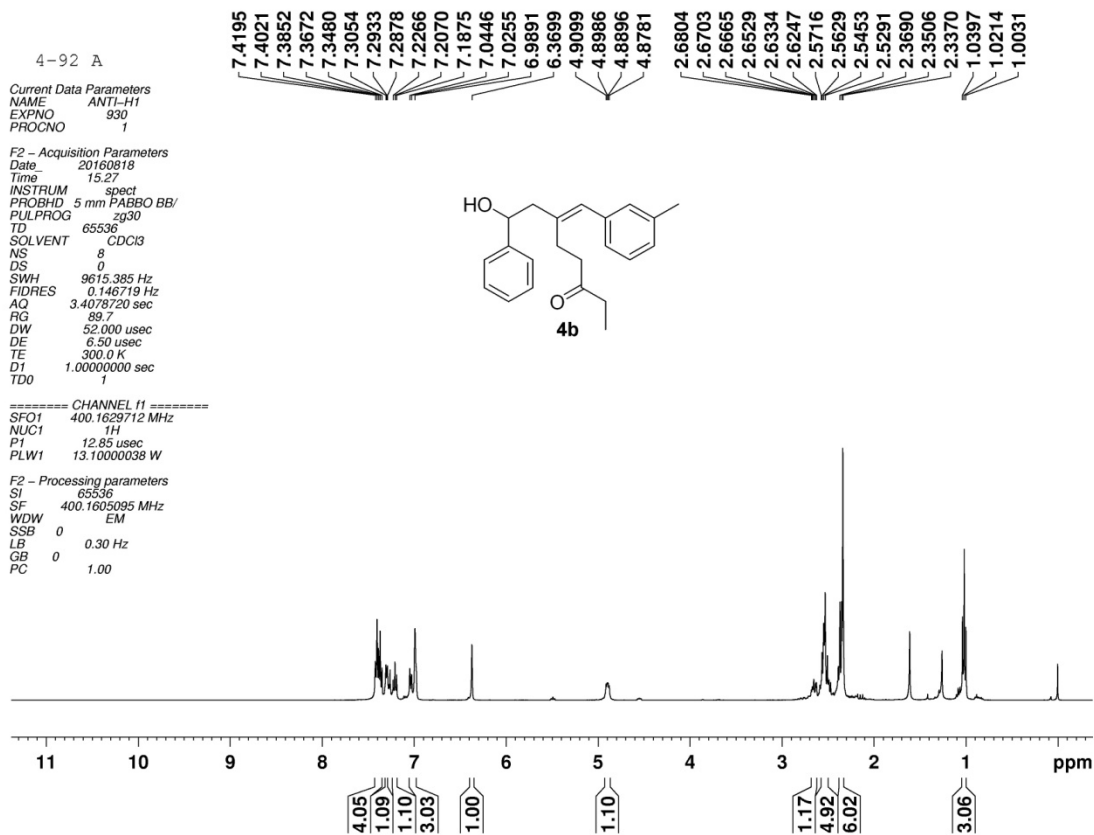
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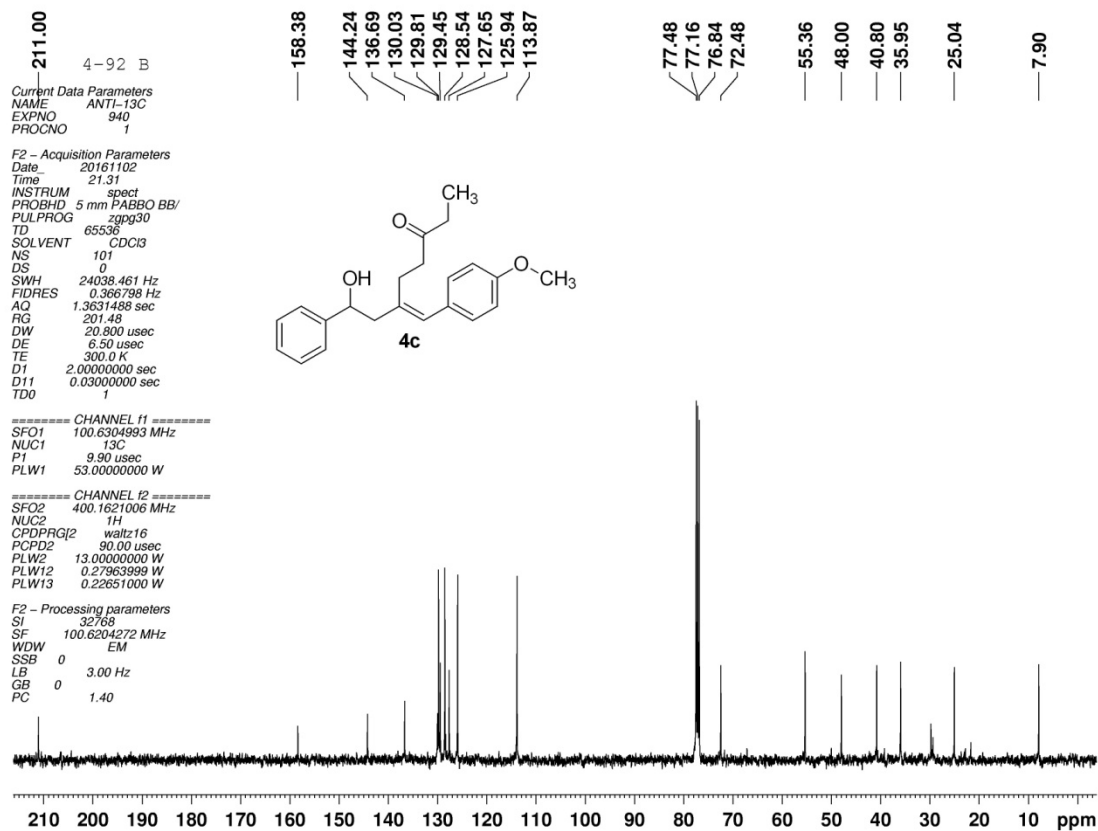
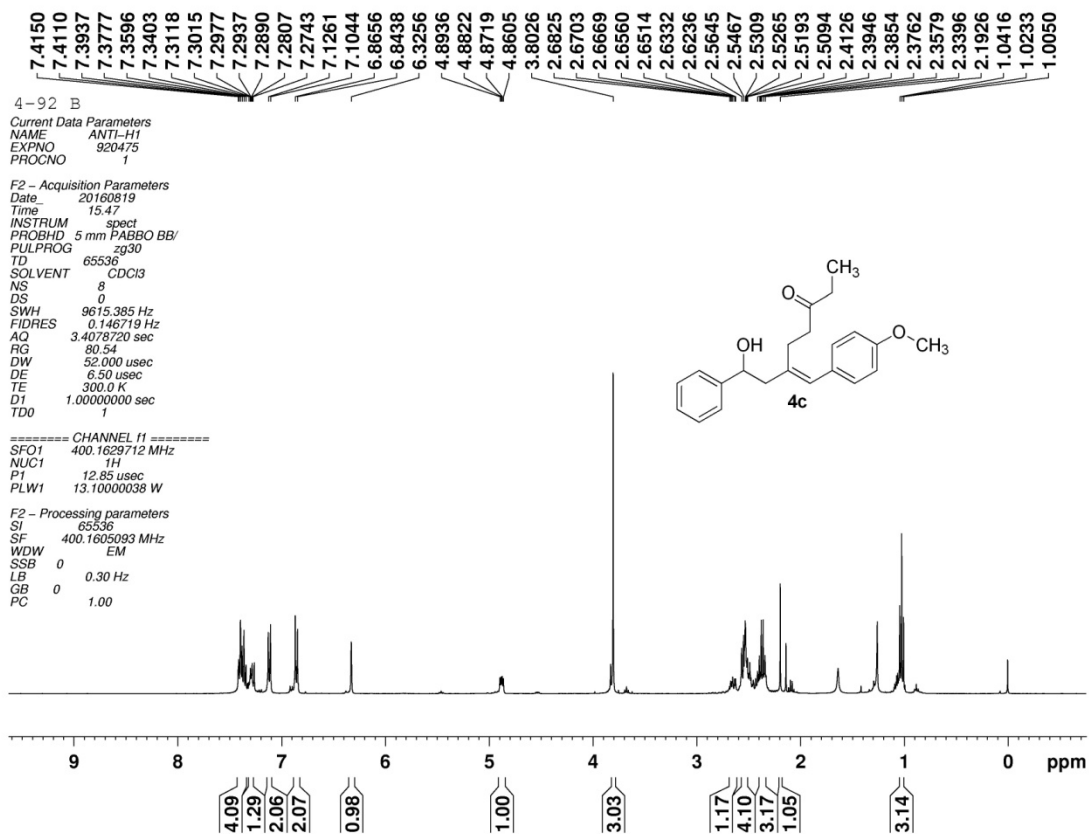
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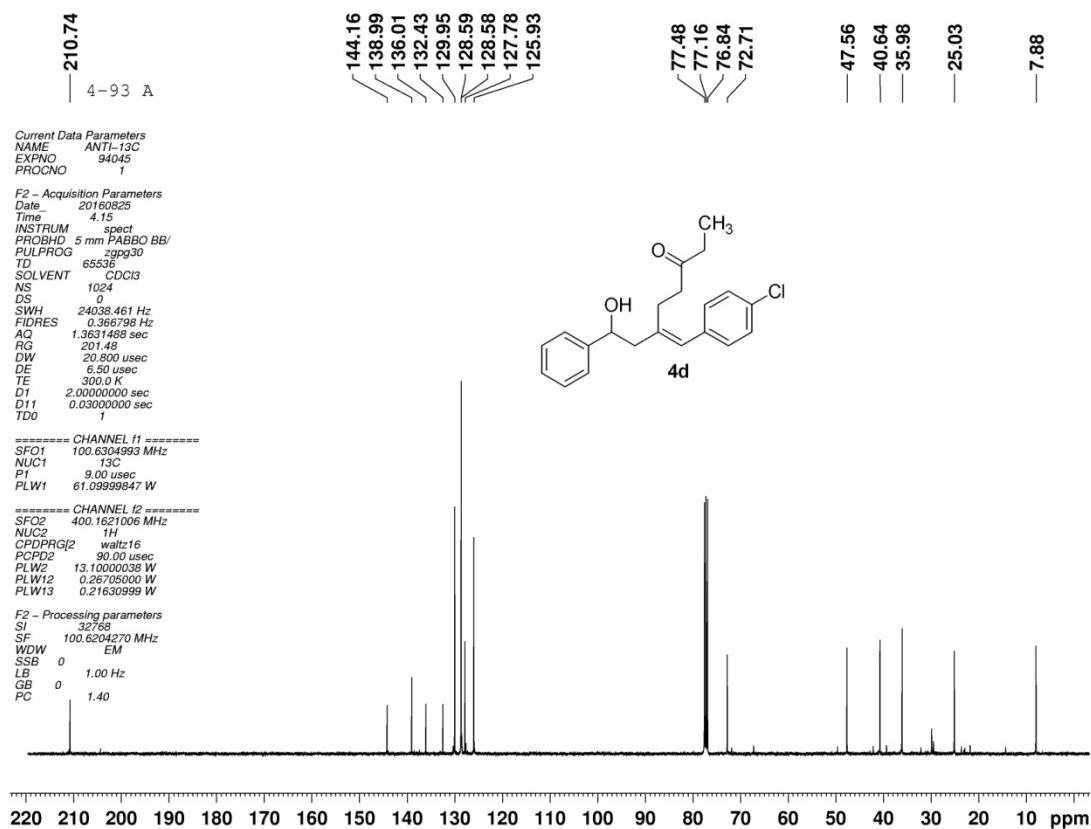
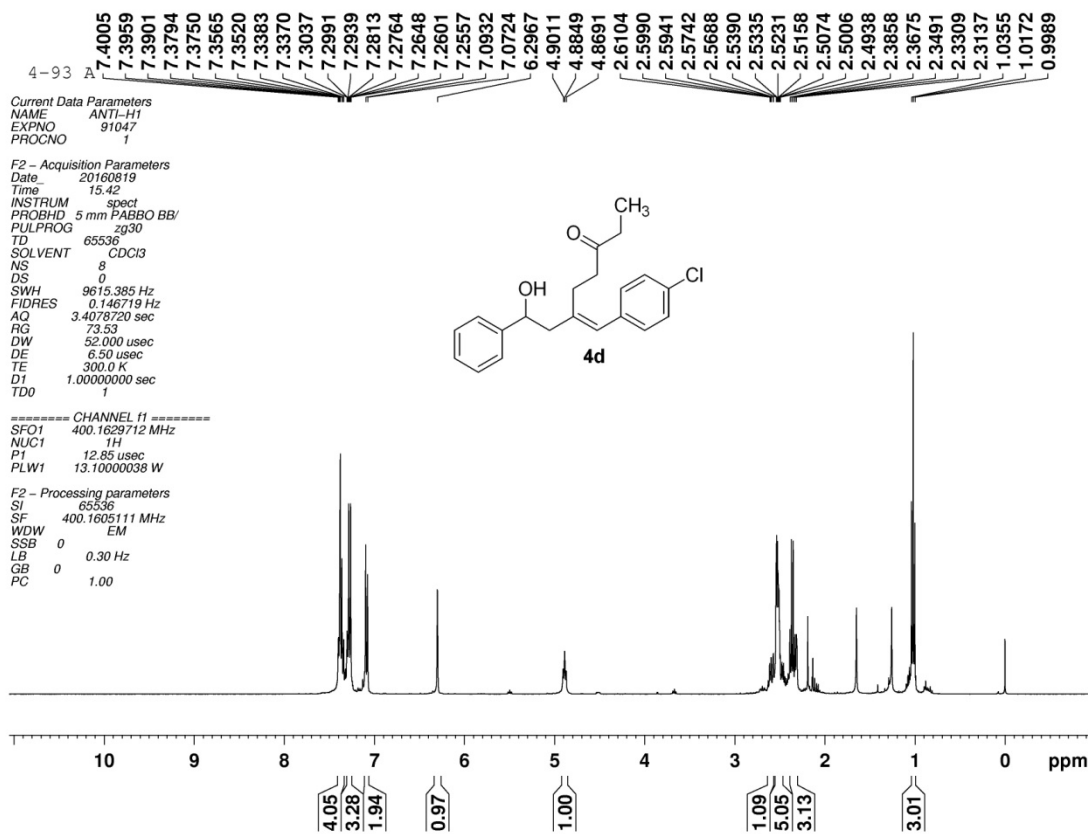
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WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

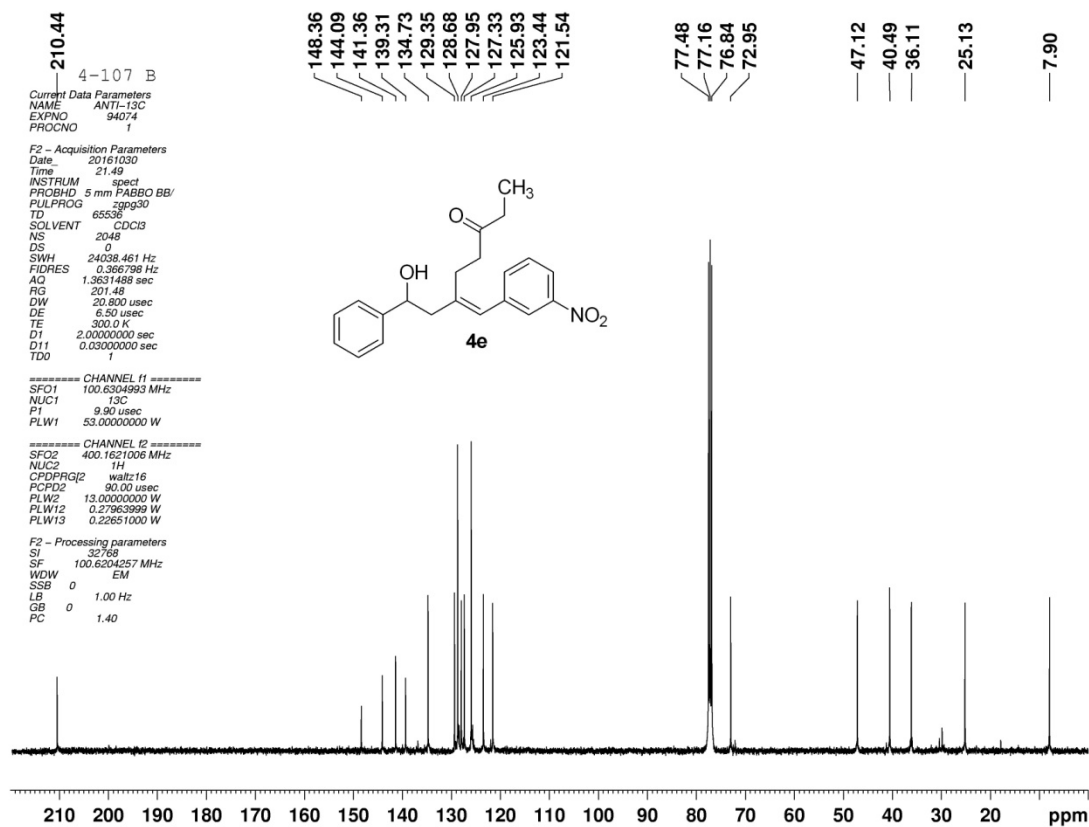
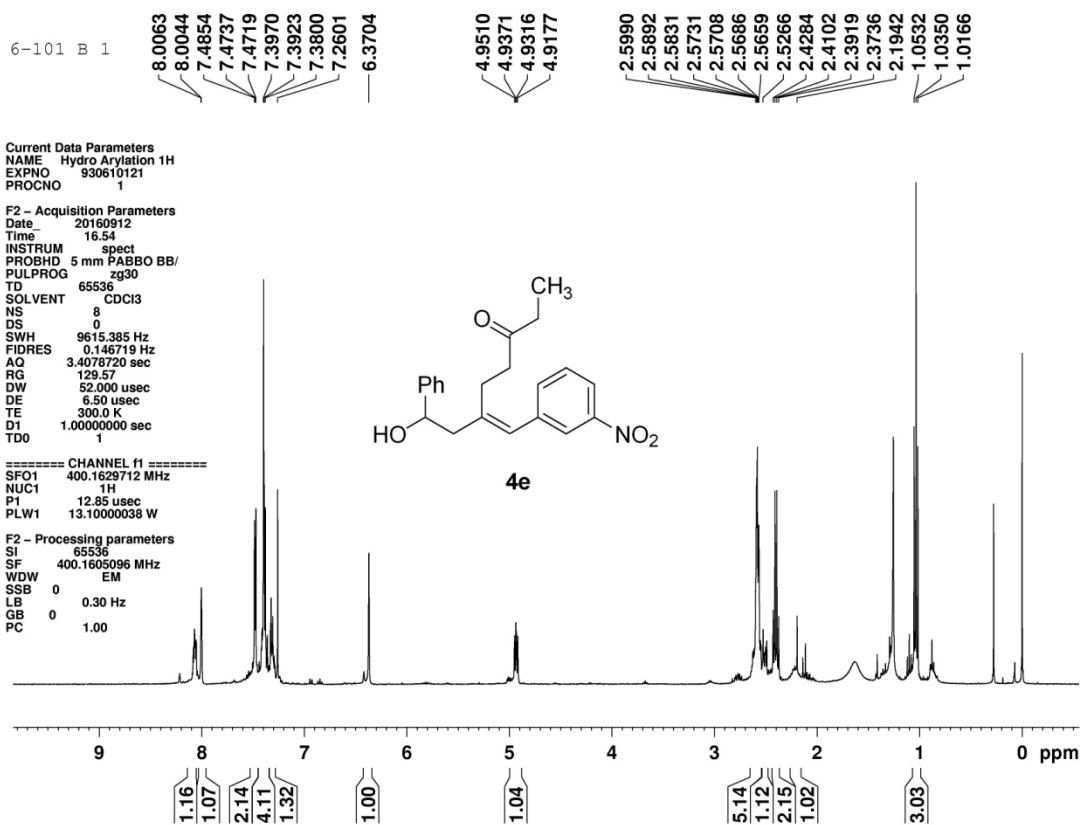


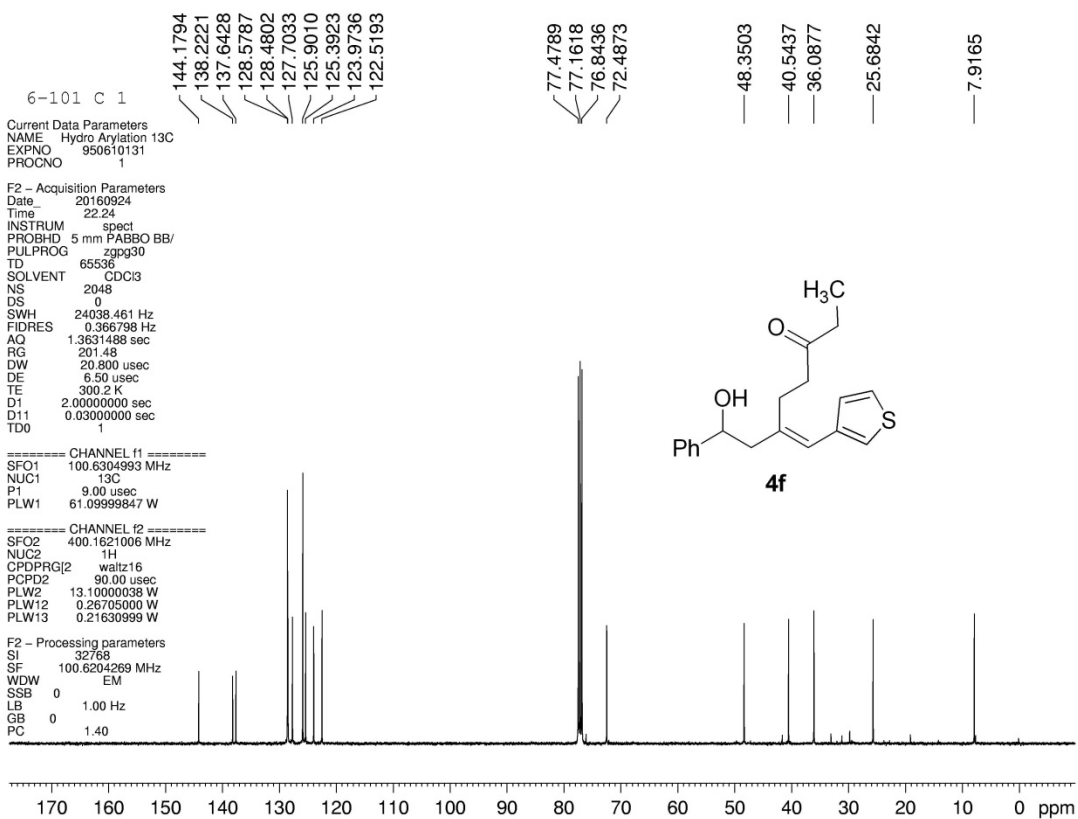
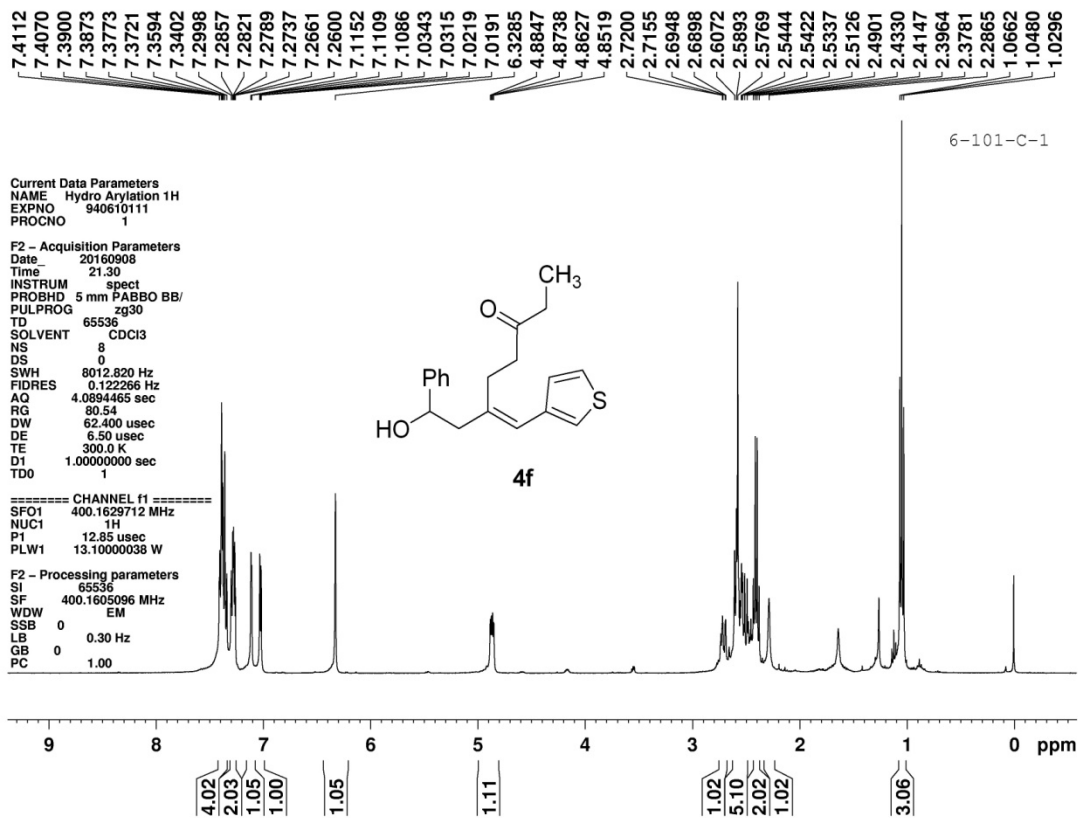


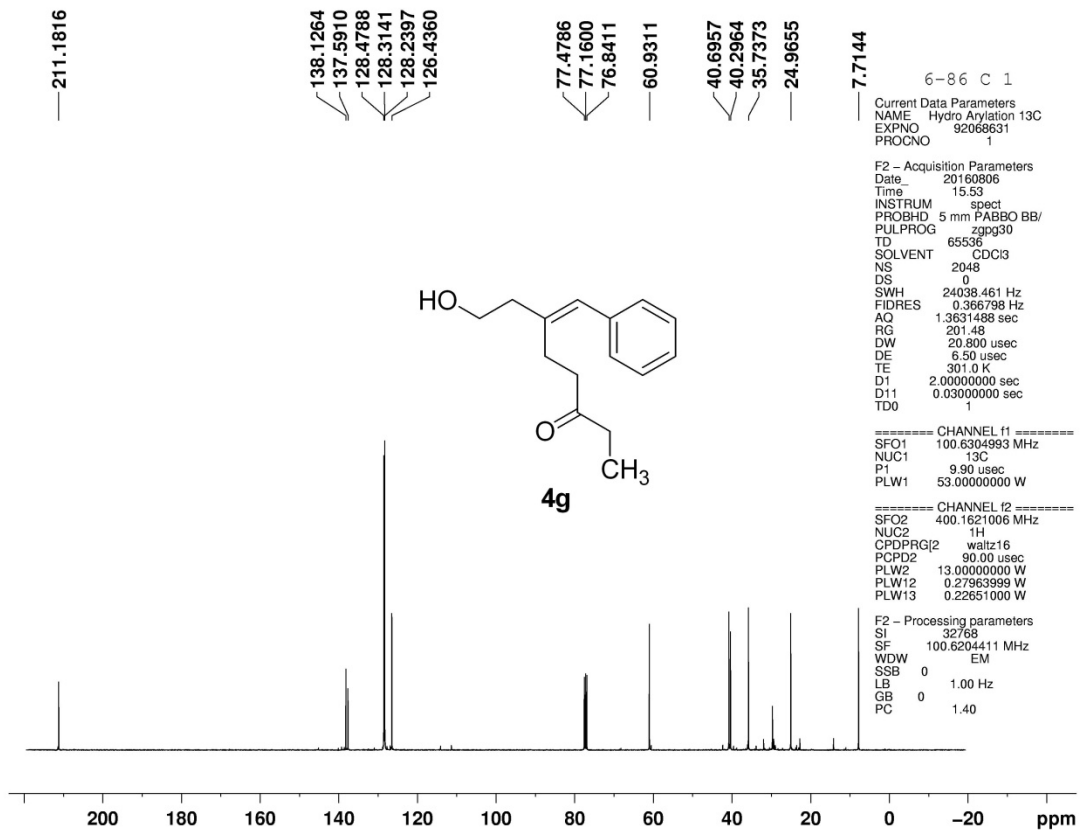
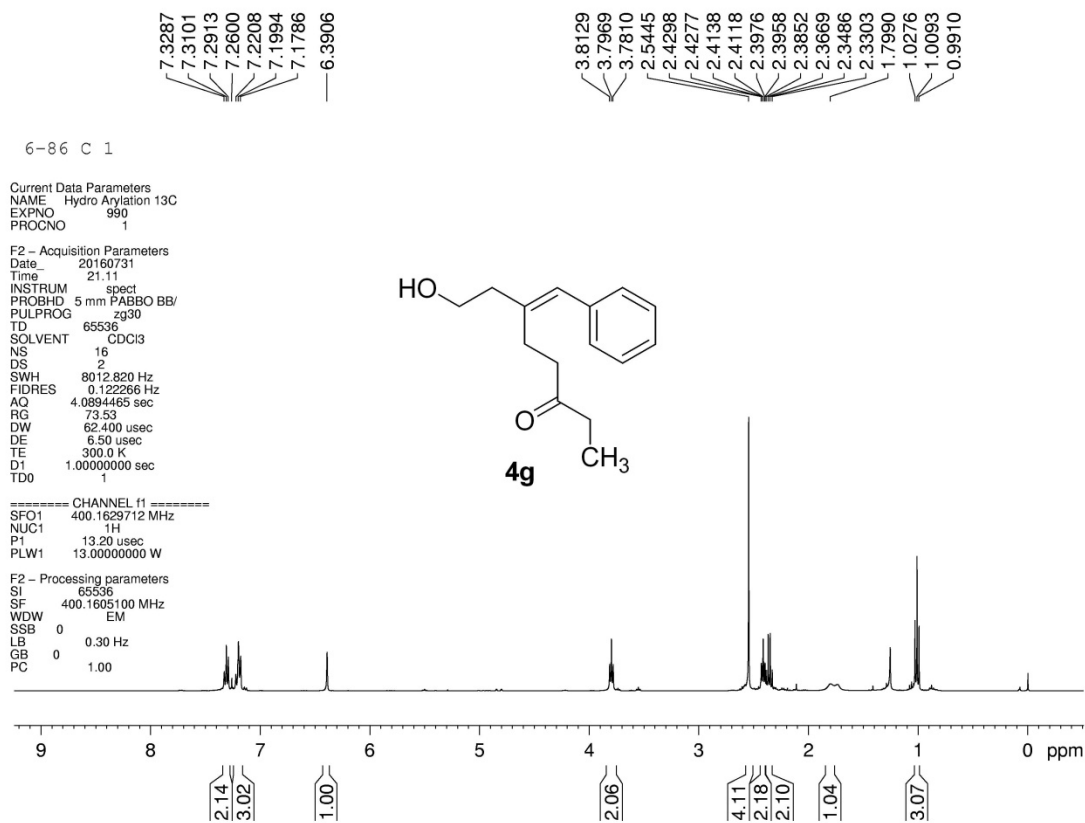


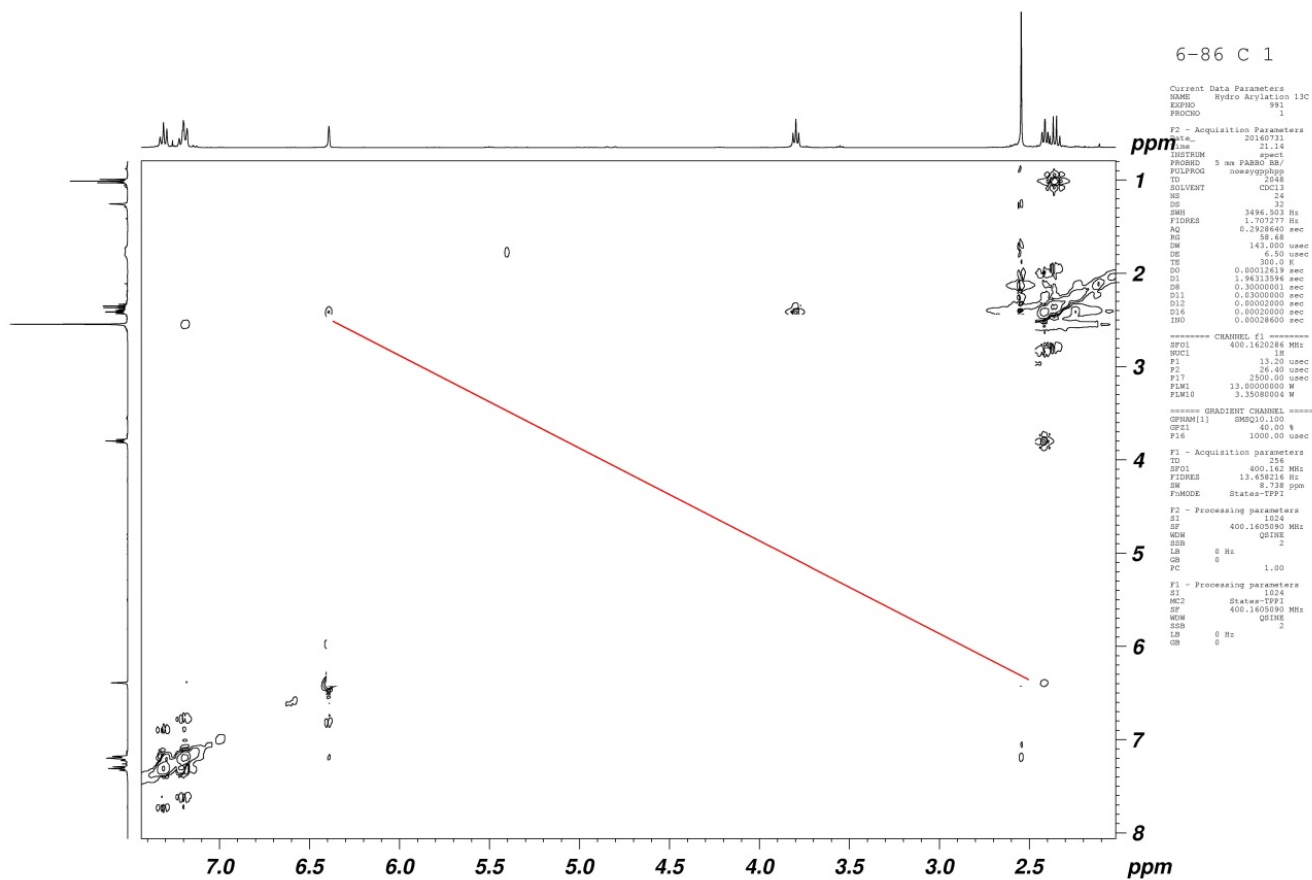
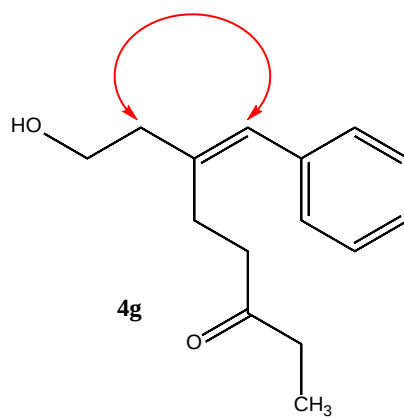


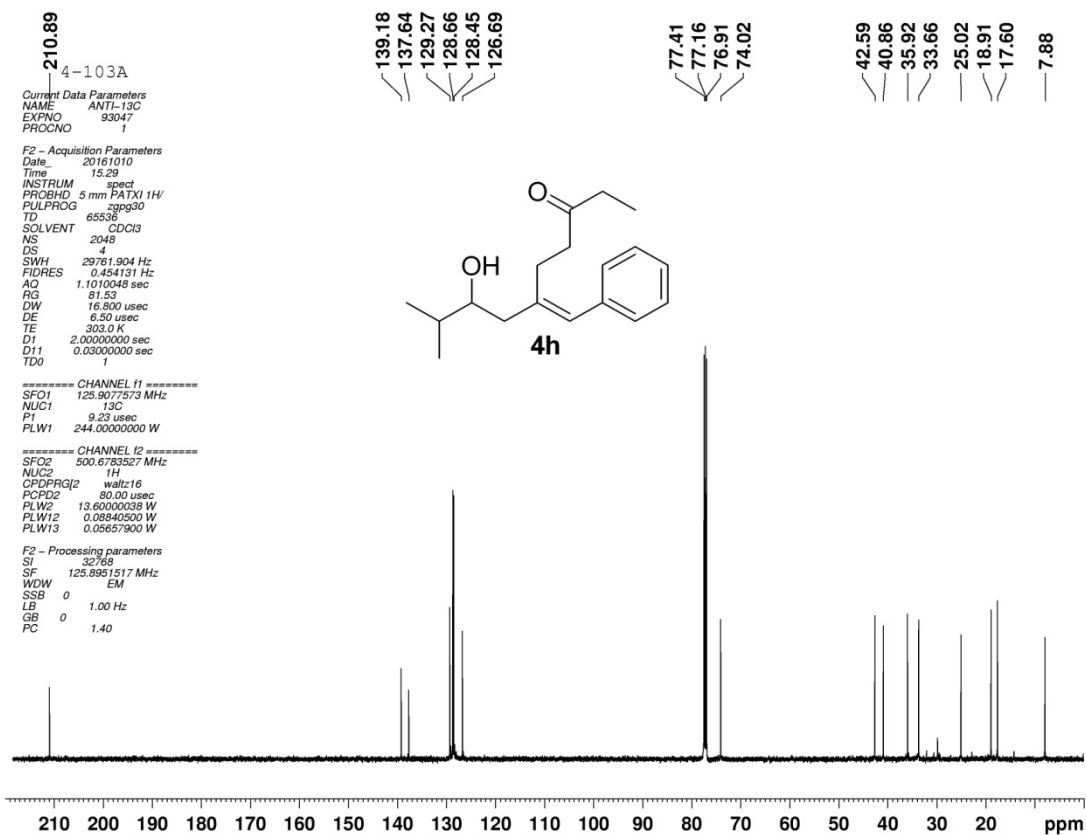
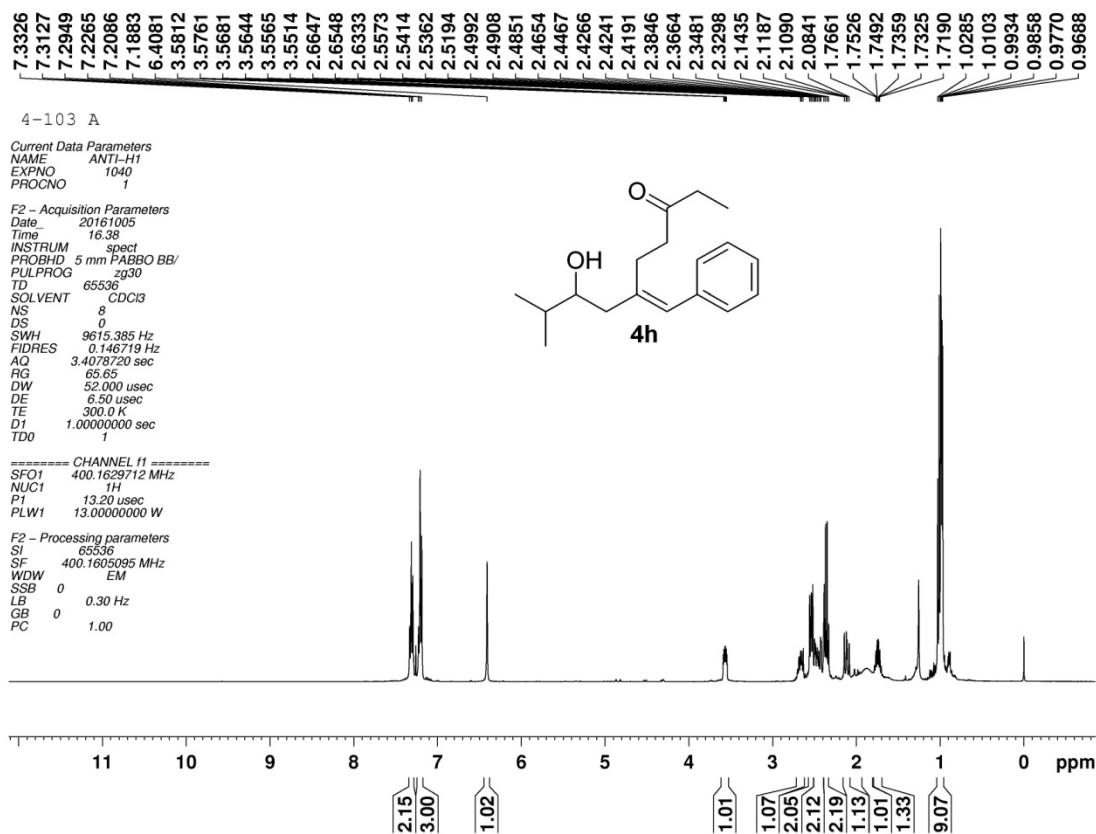


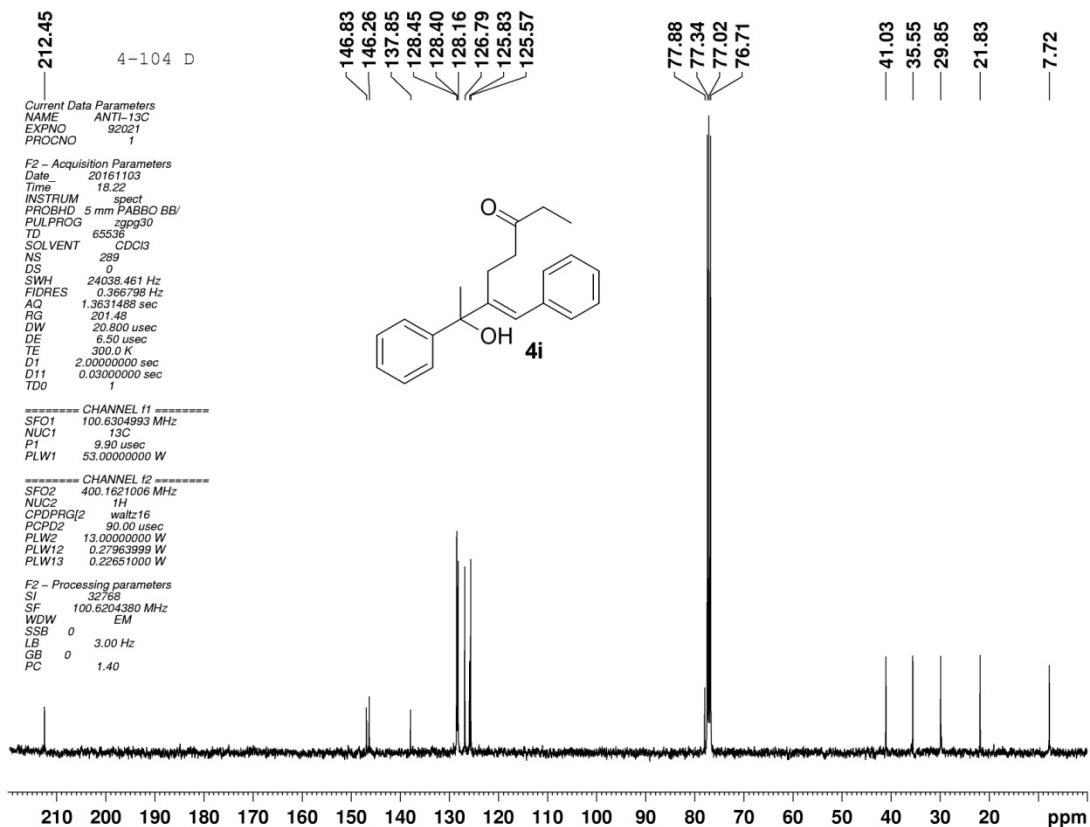
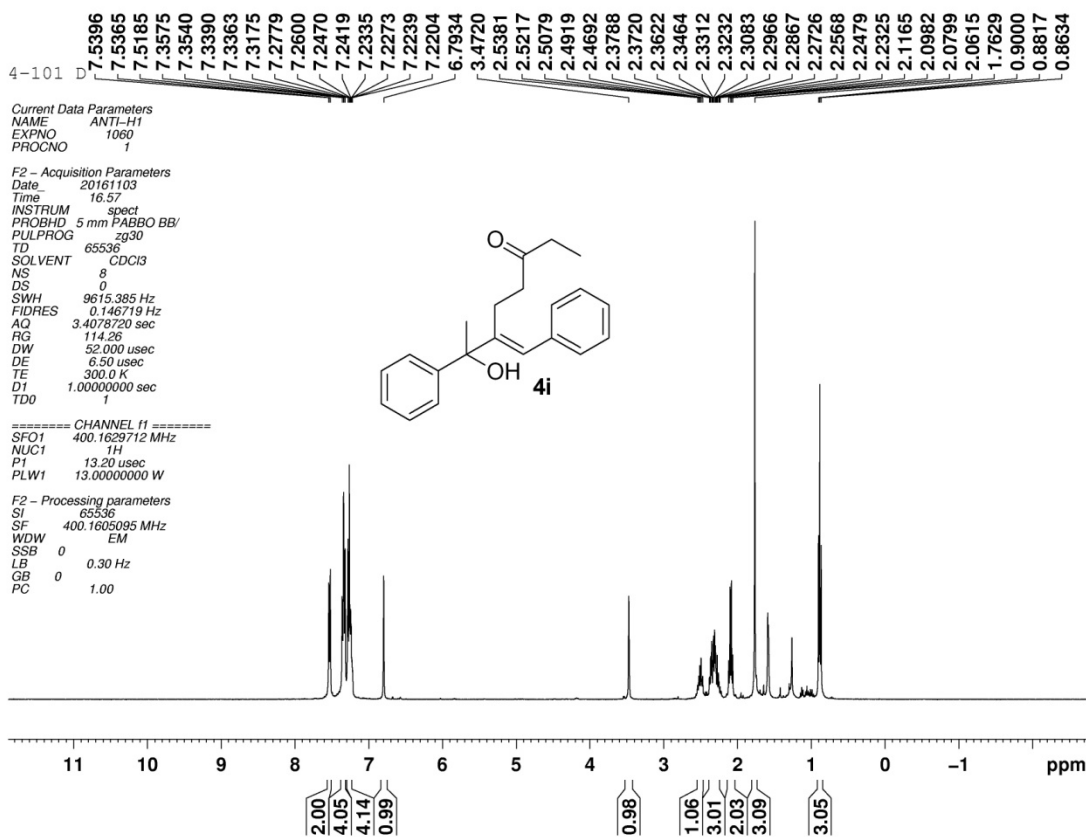


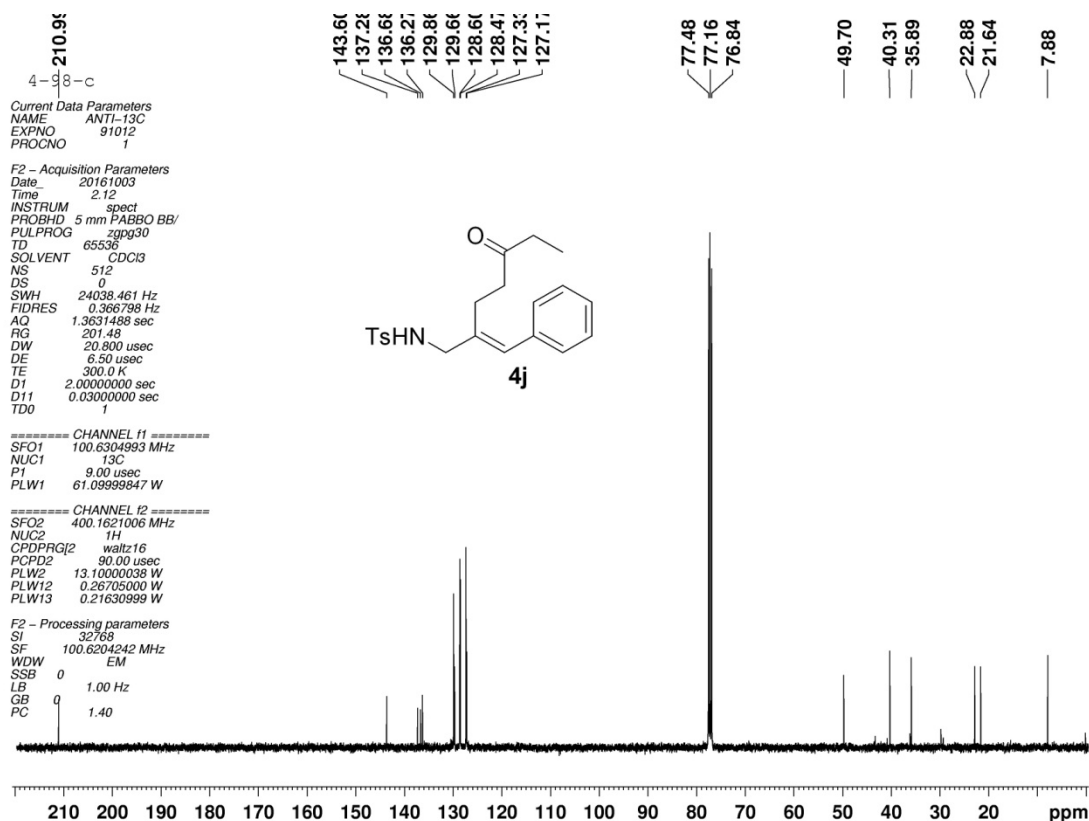
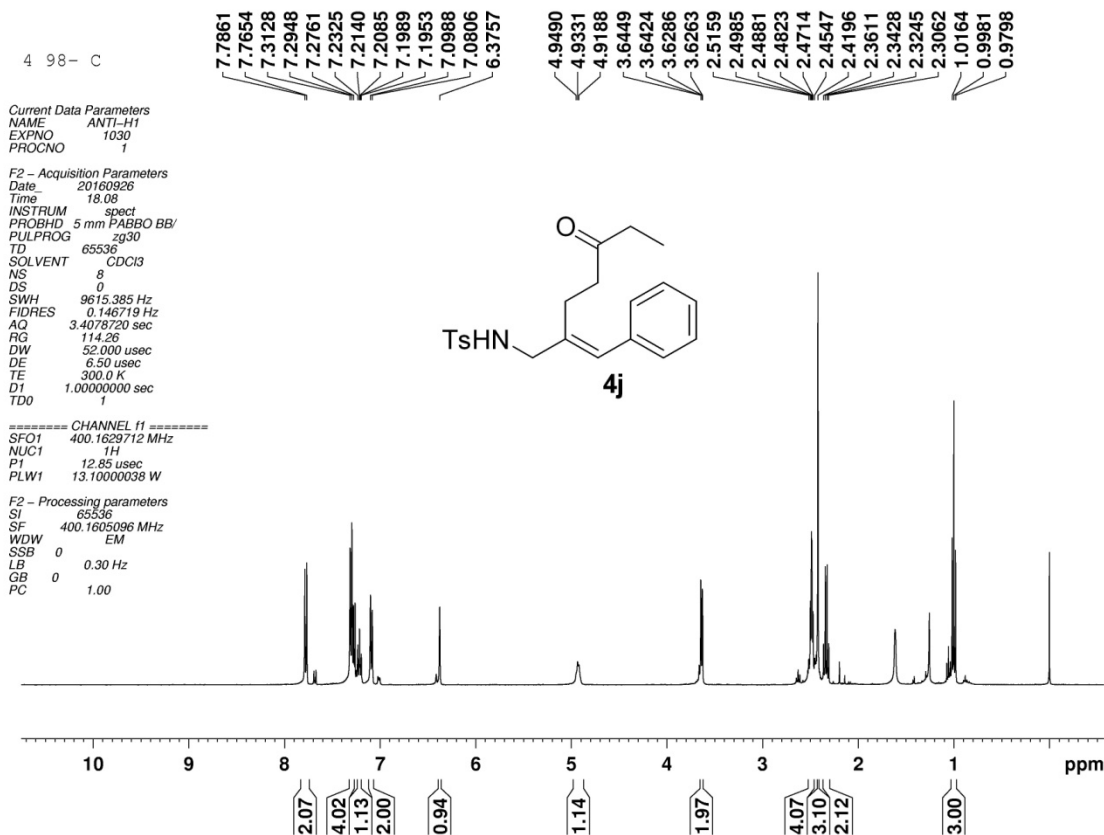


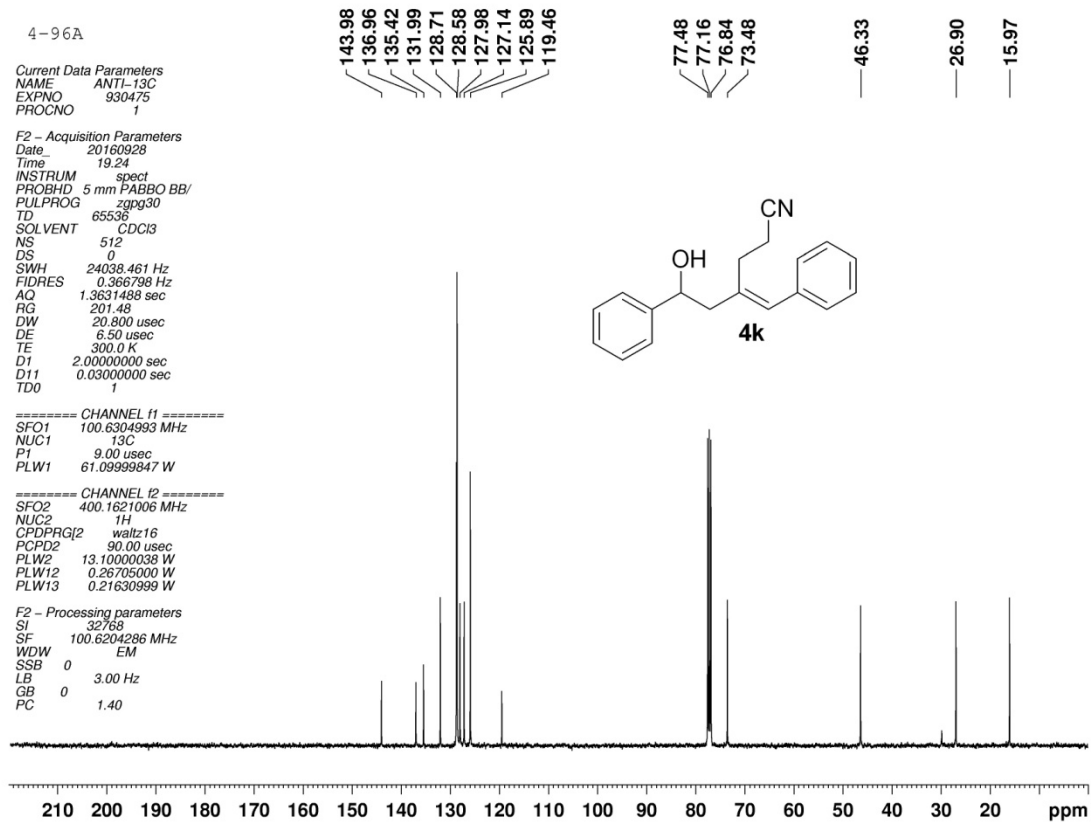
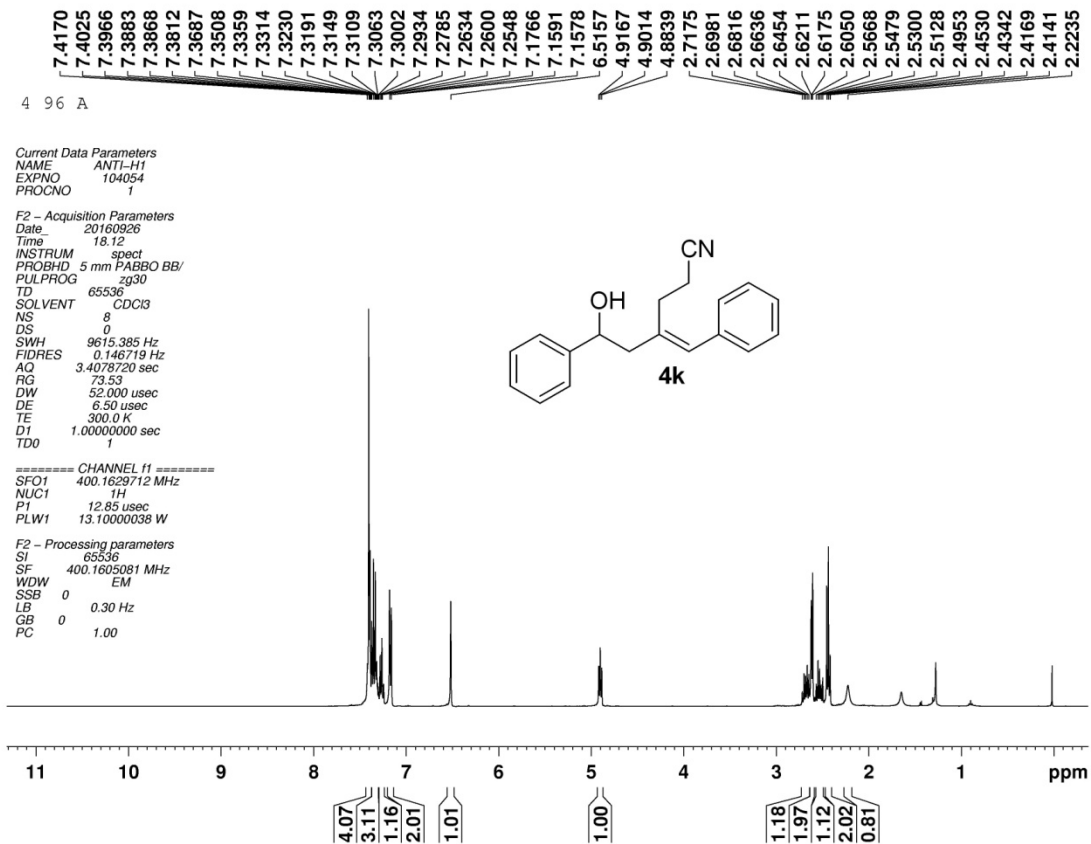


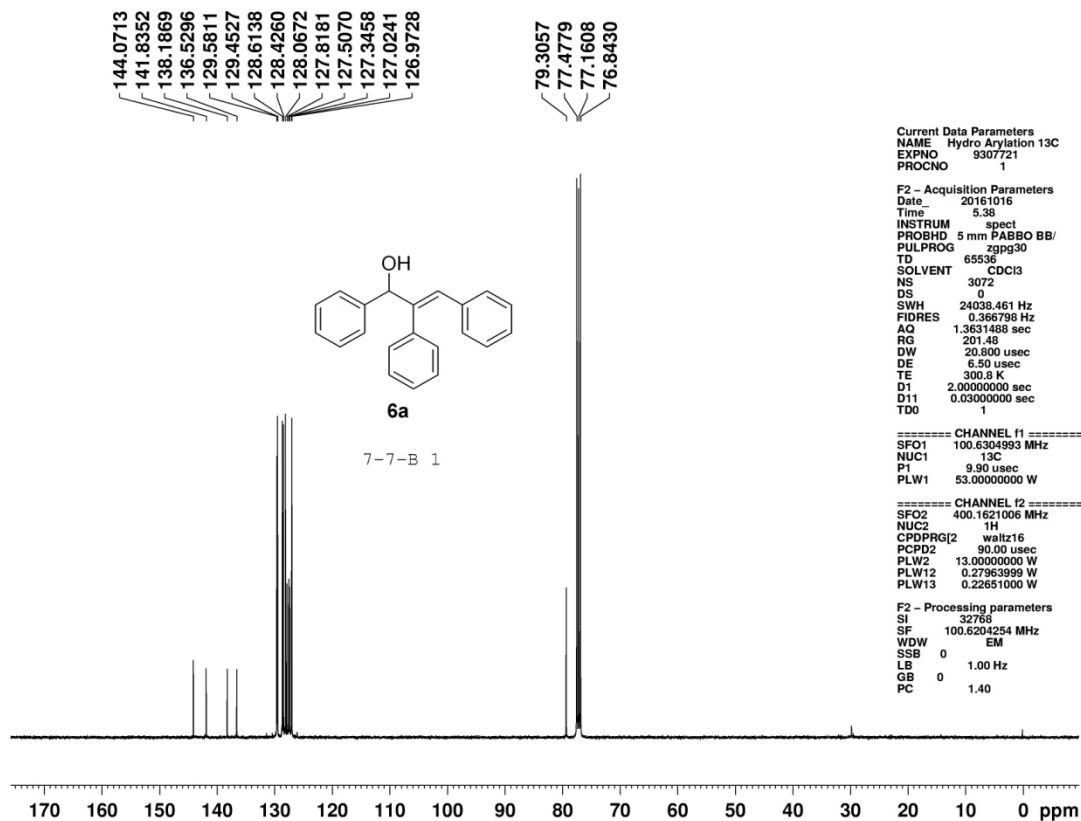
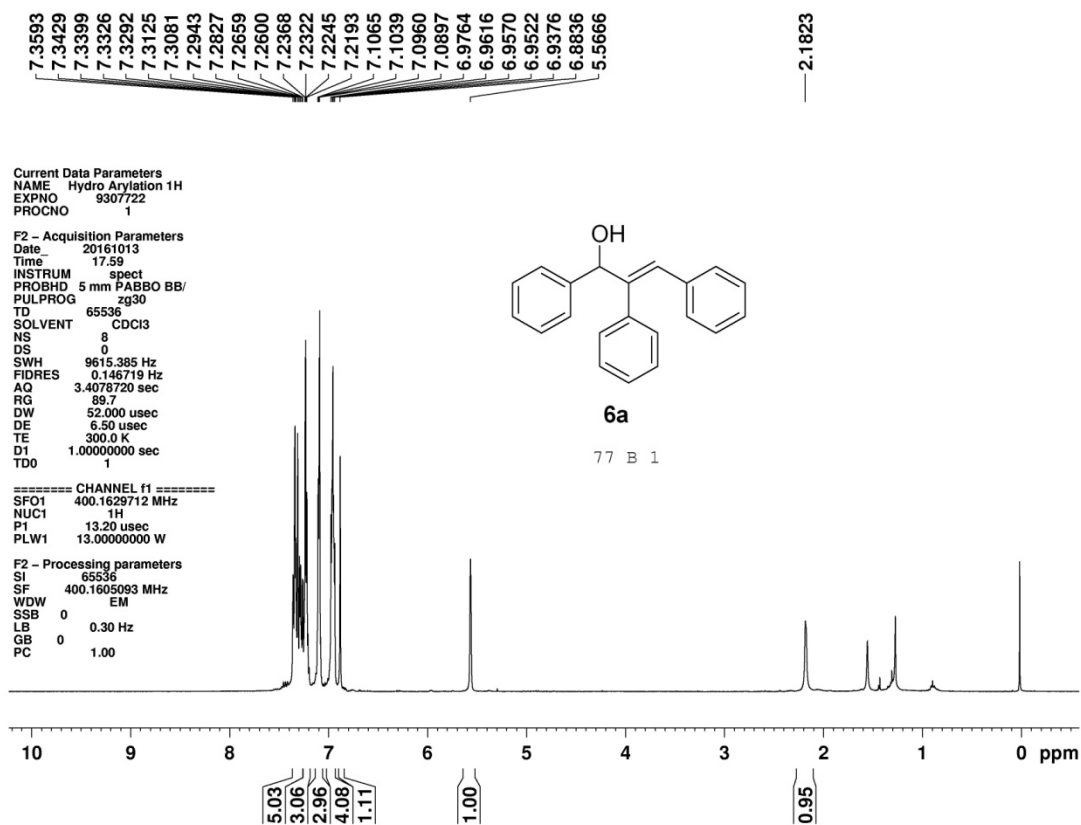


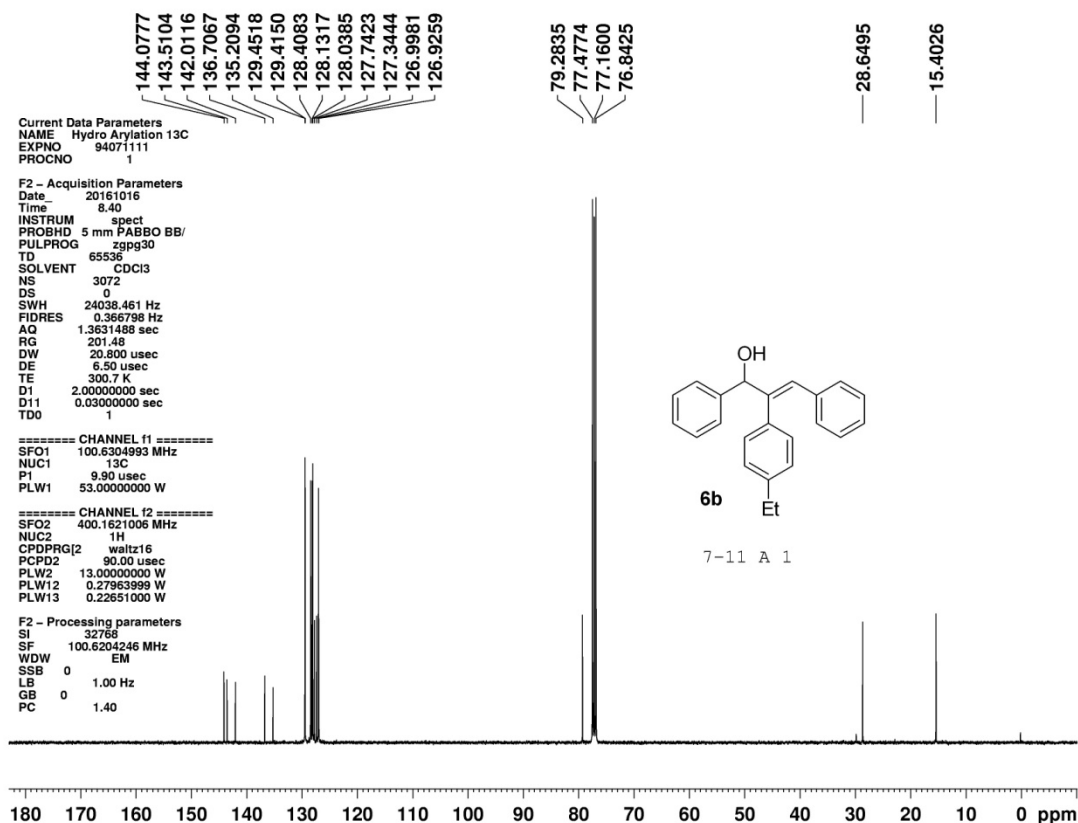
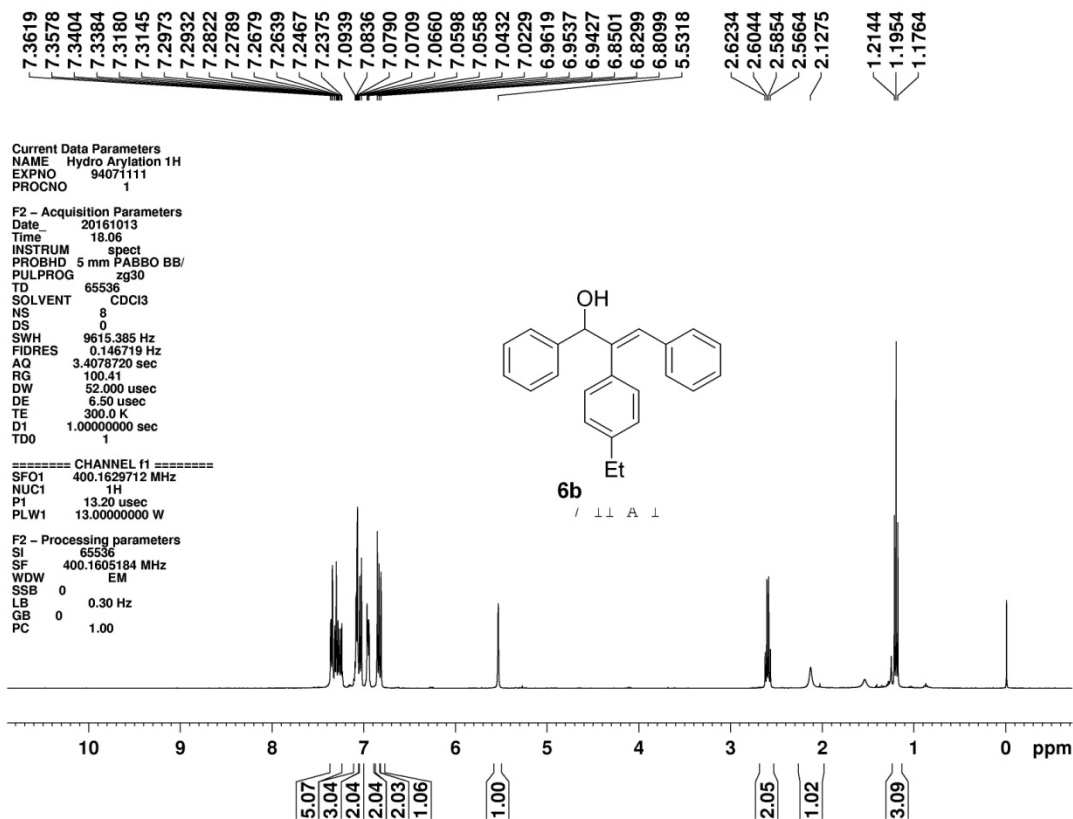


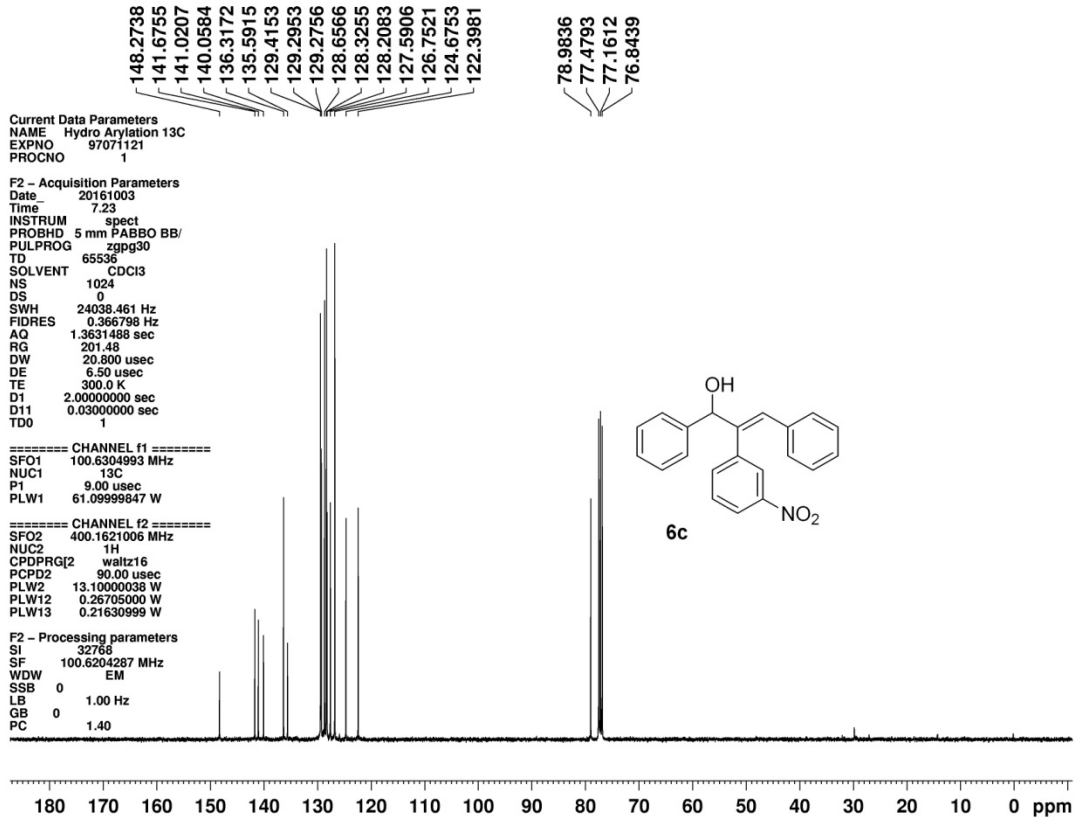
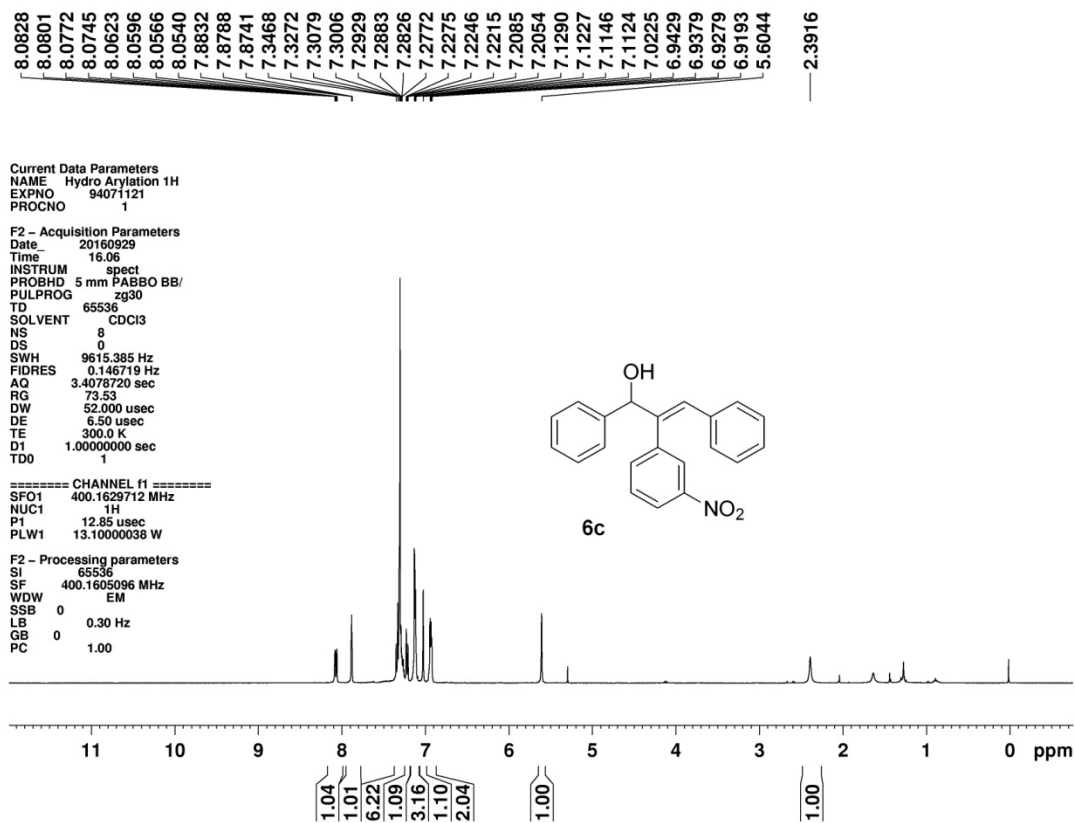


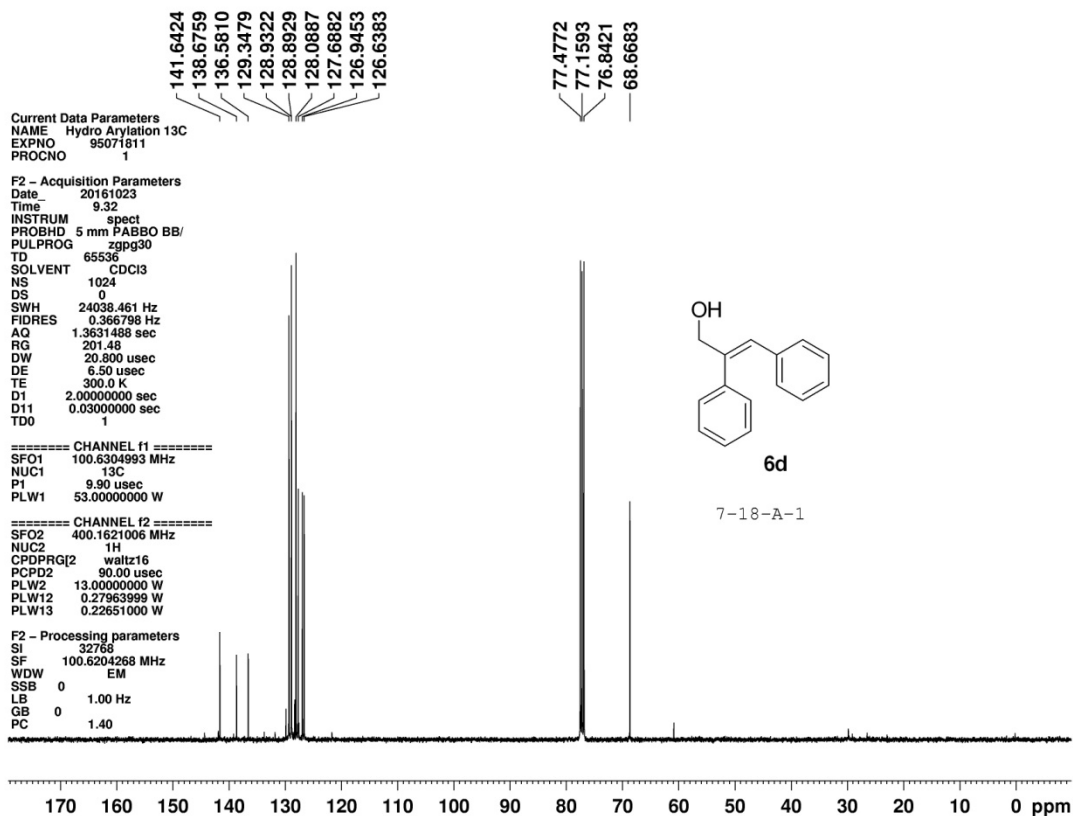
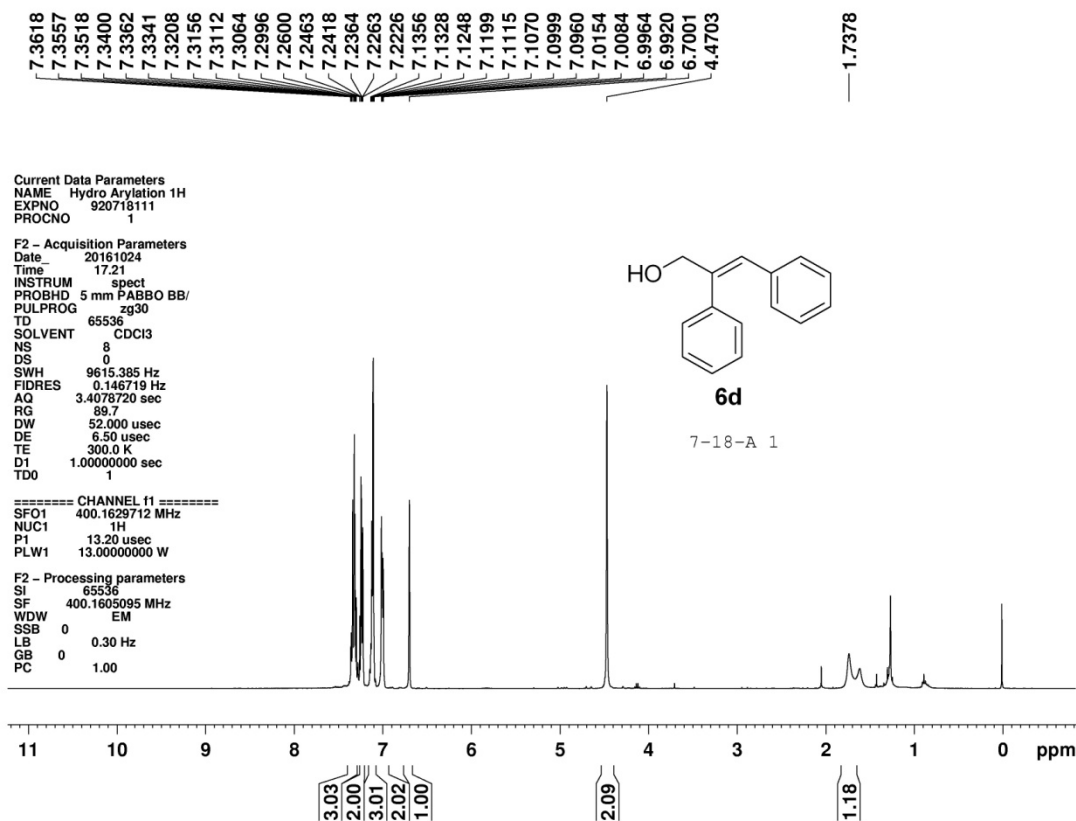


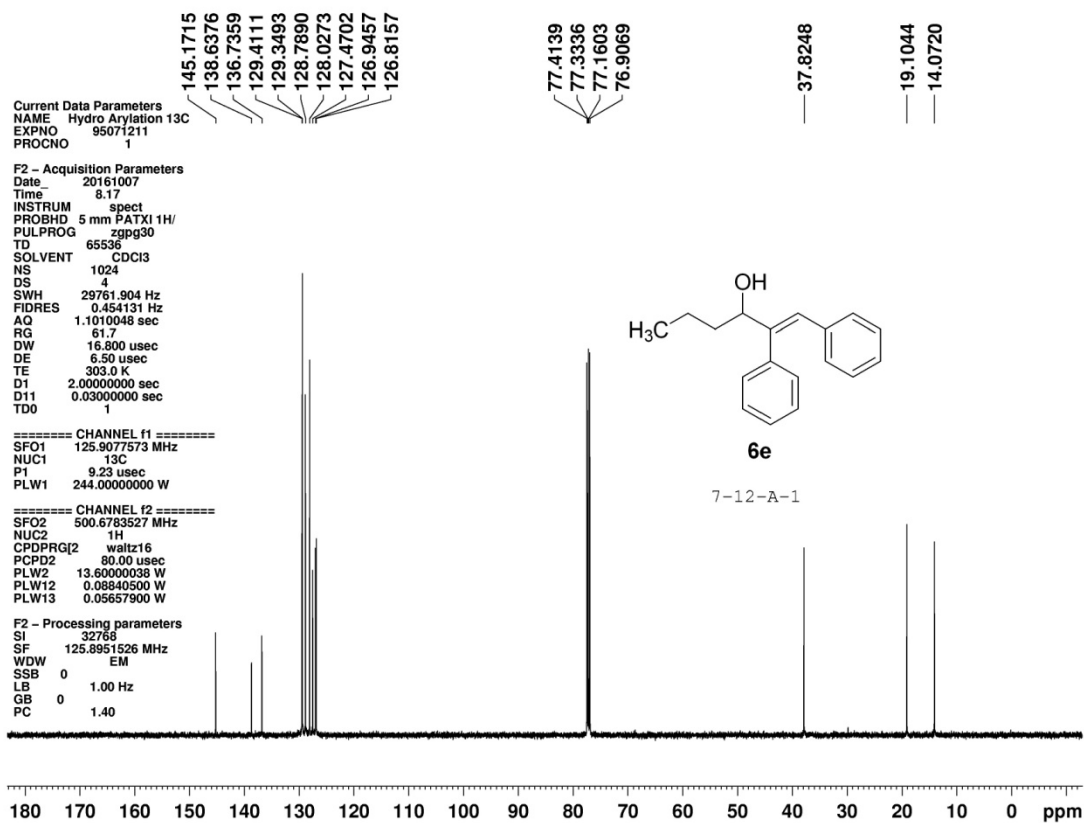
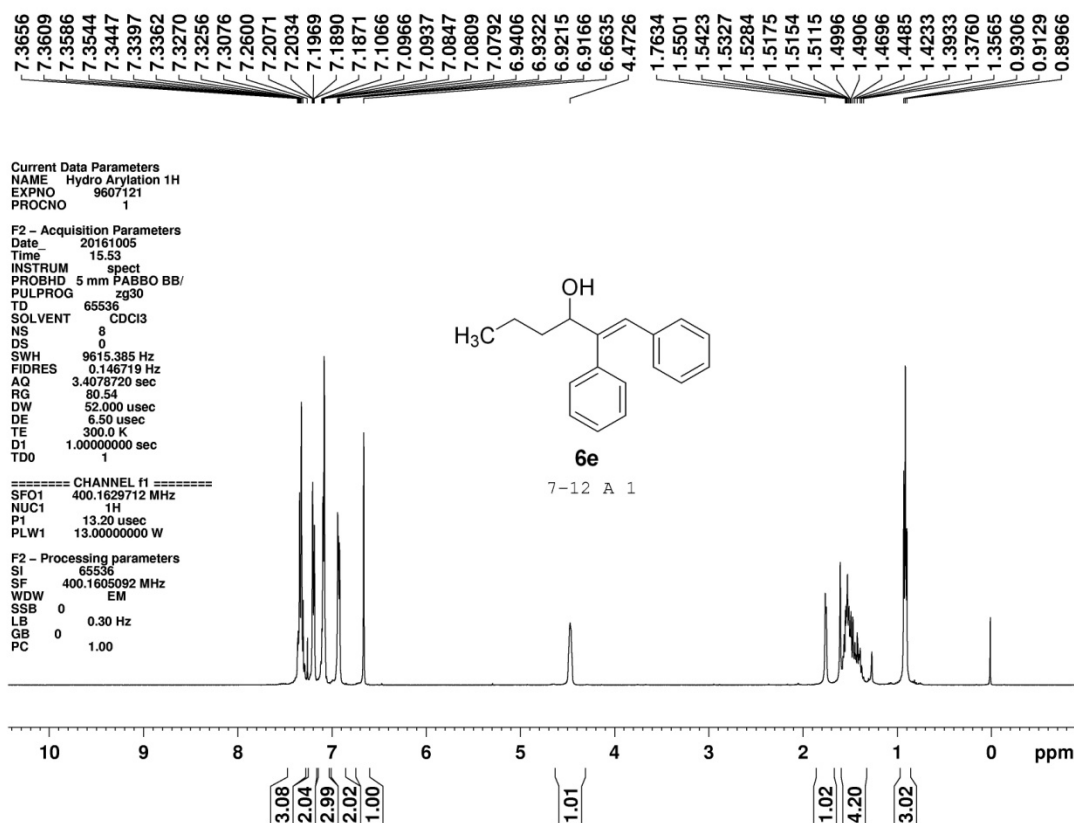


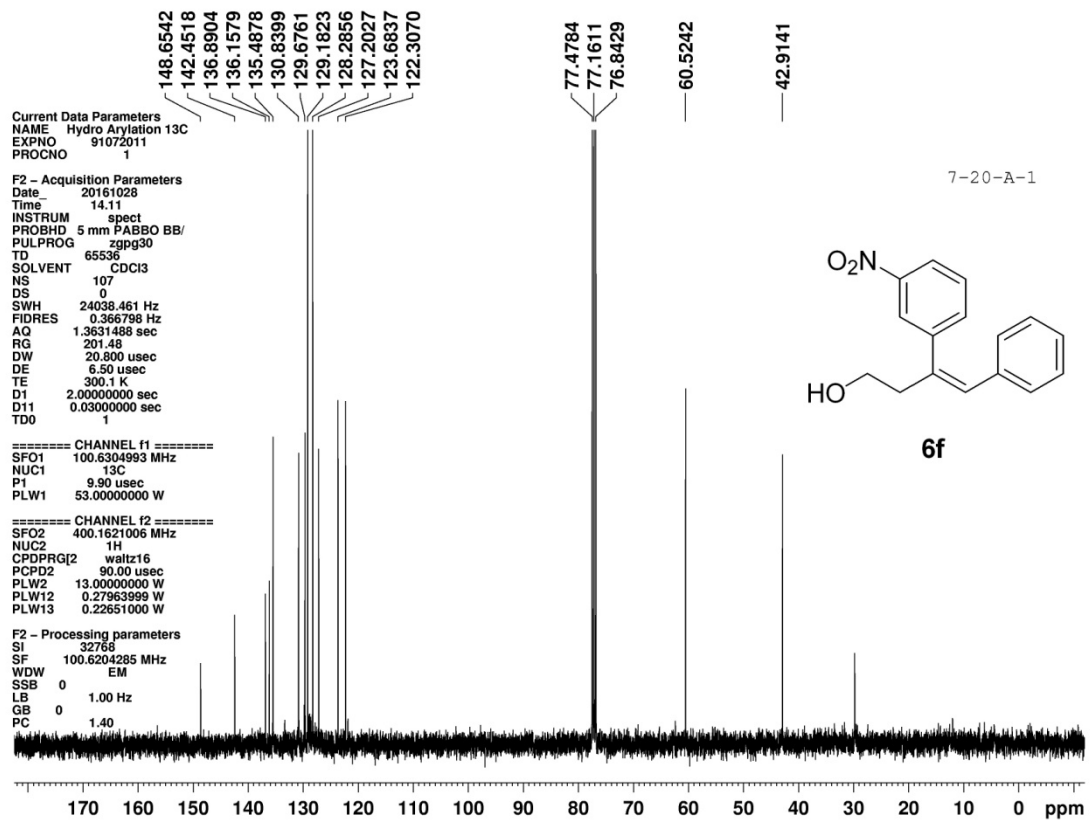
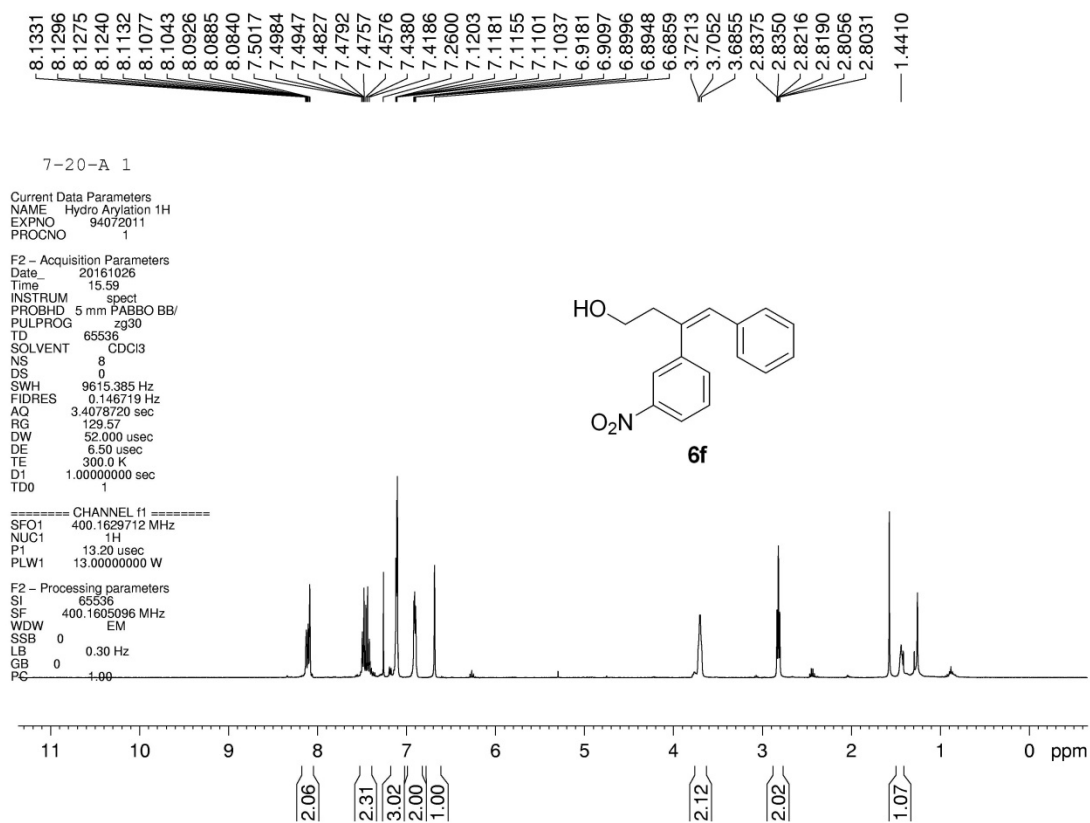


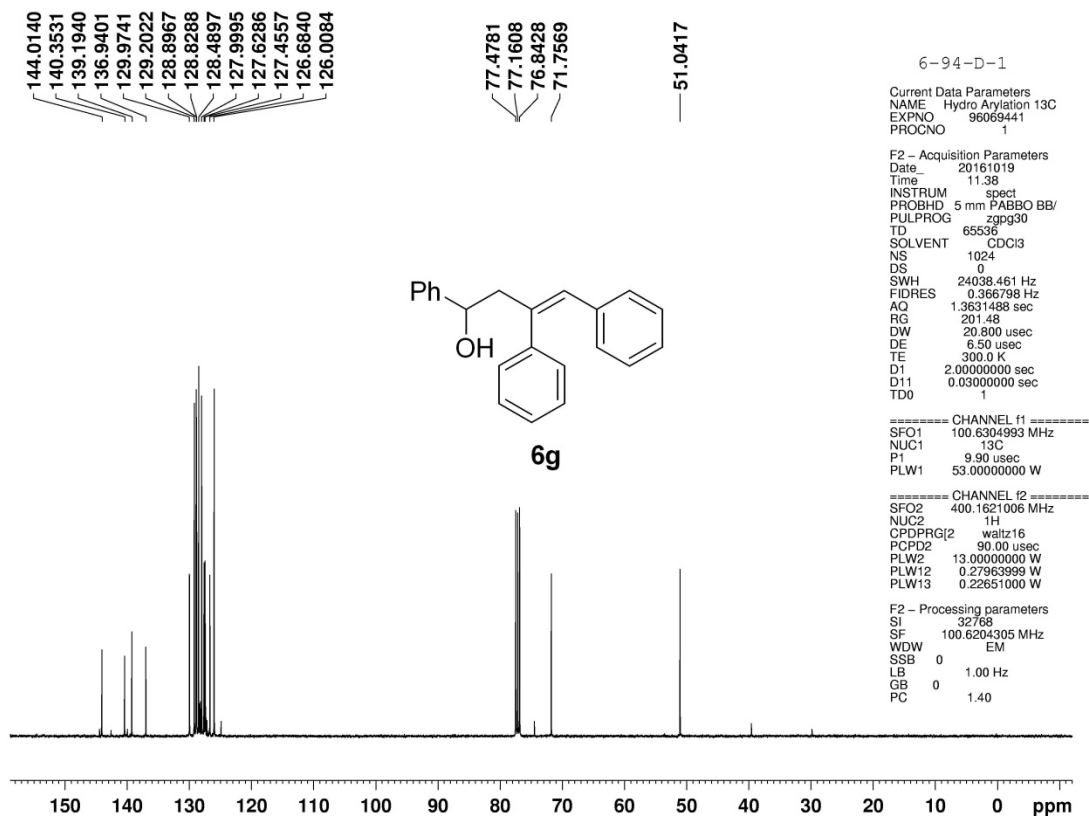
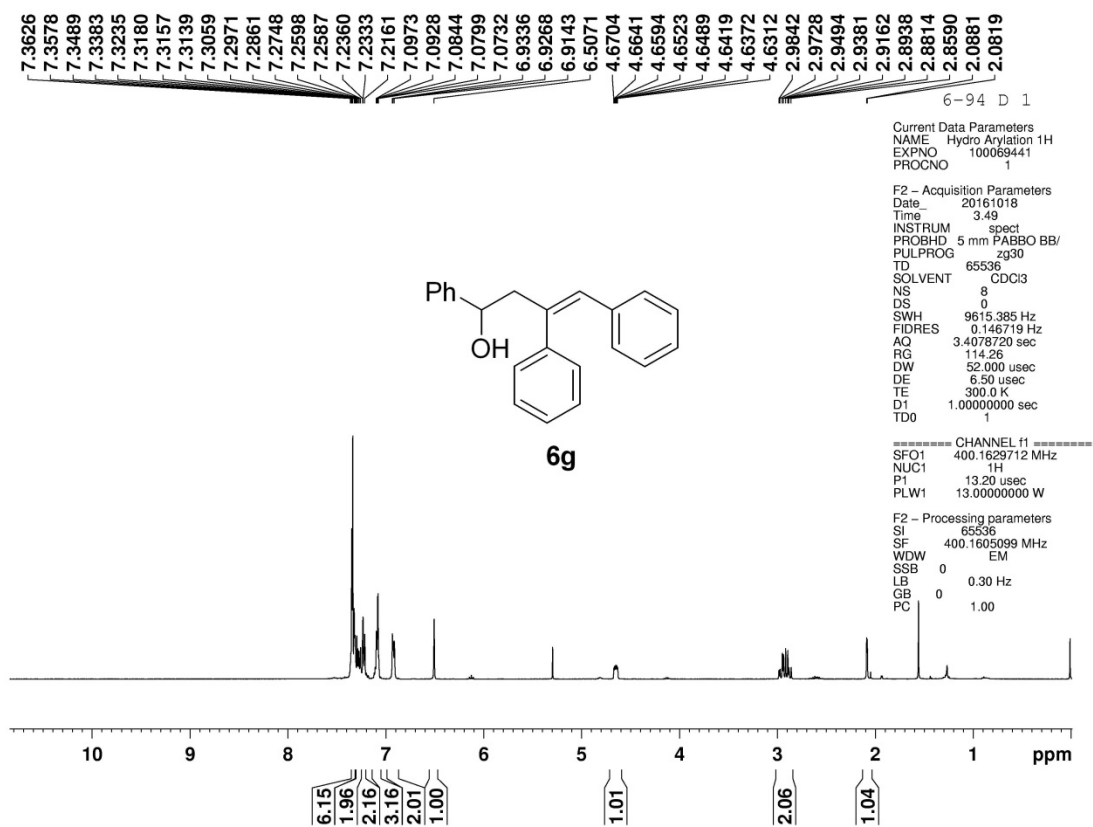


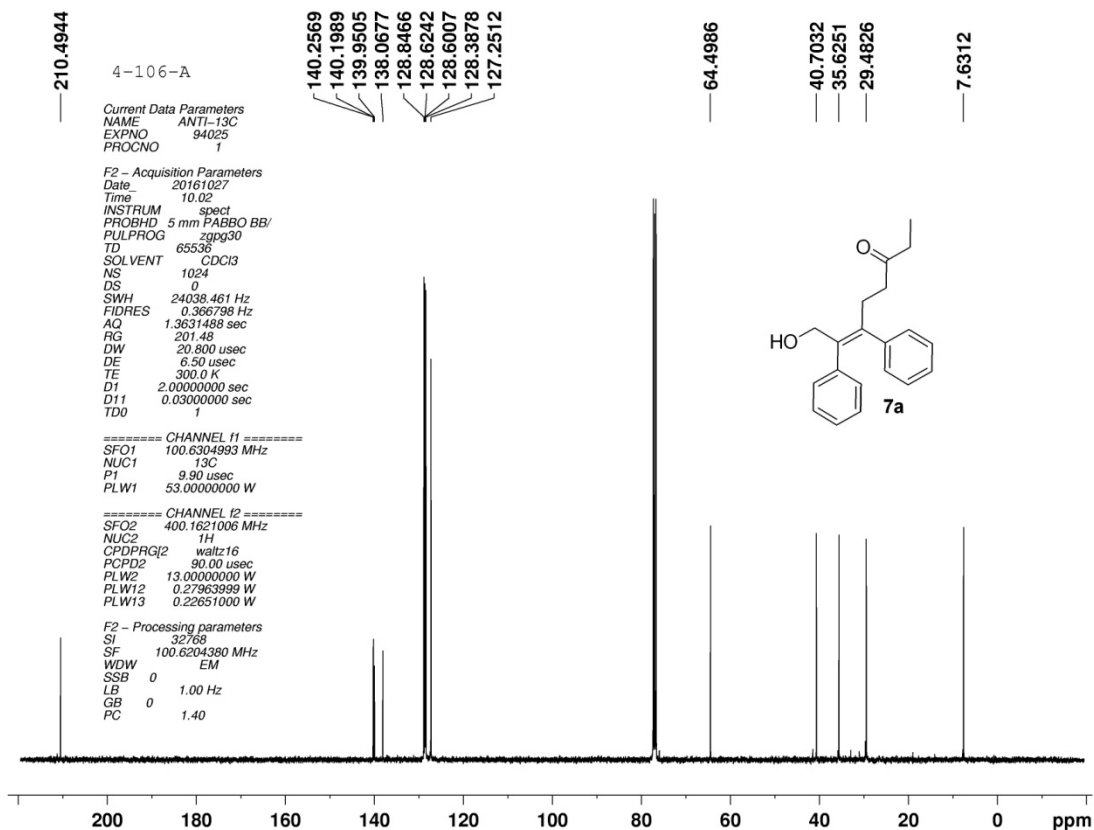
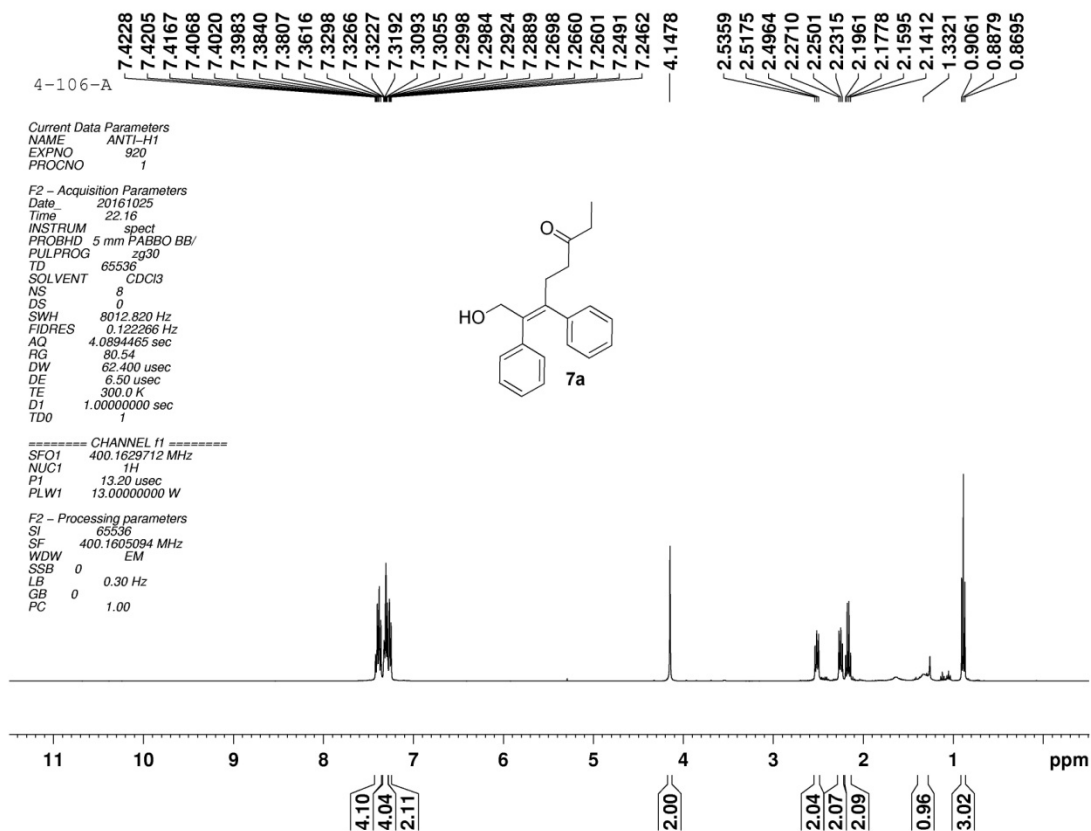


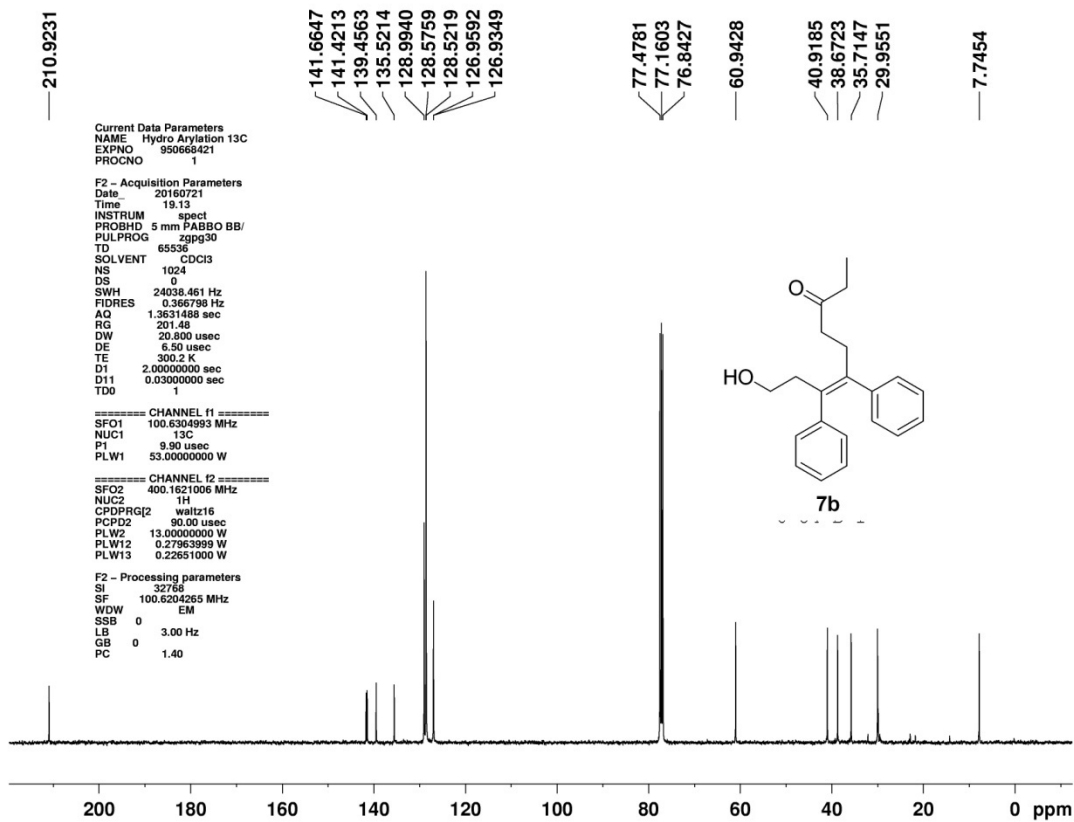
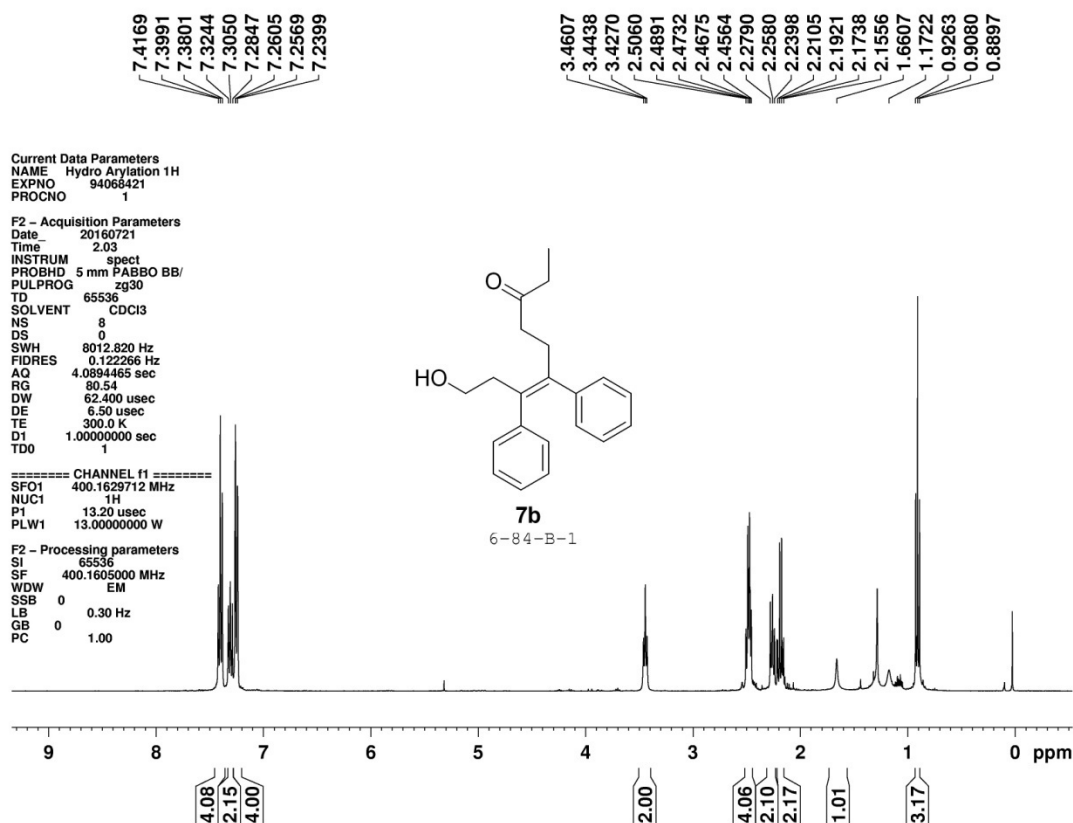


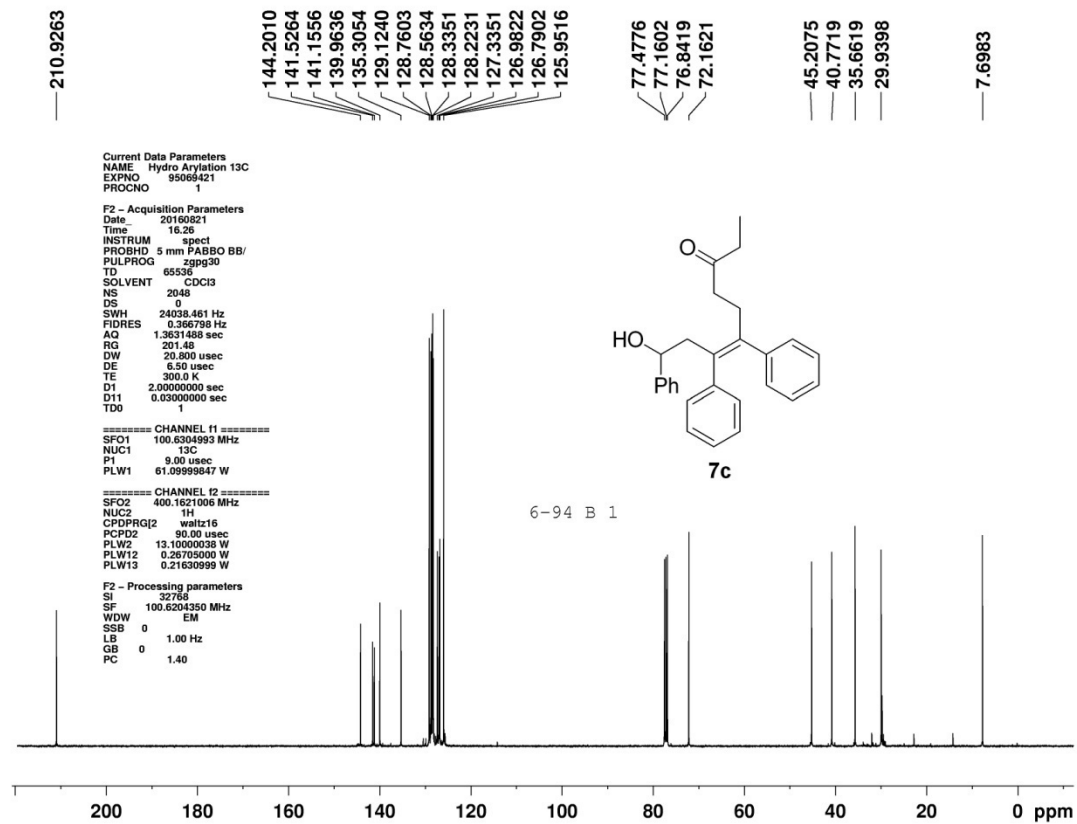
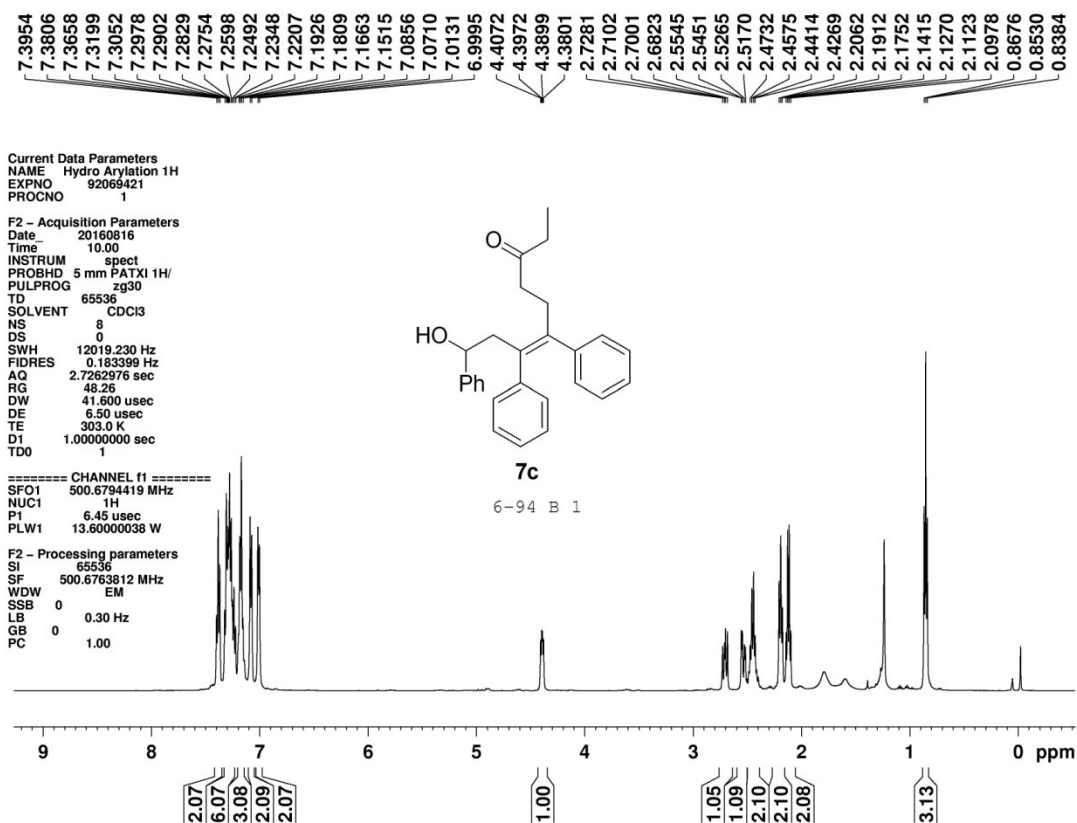


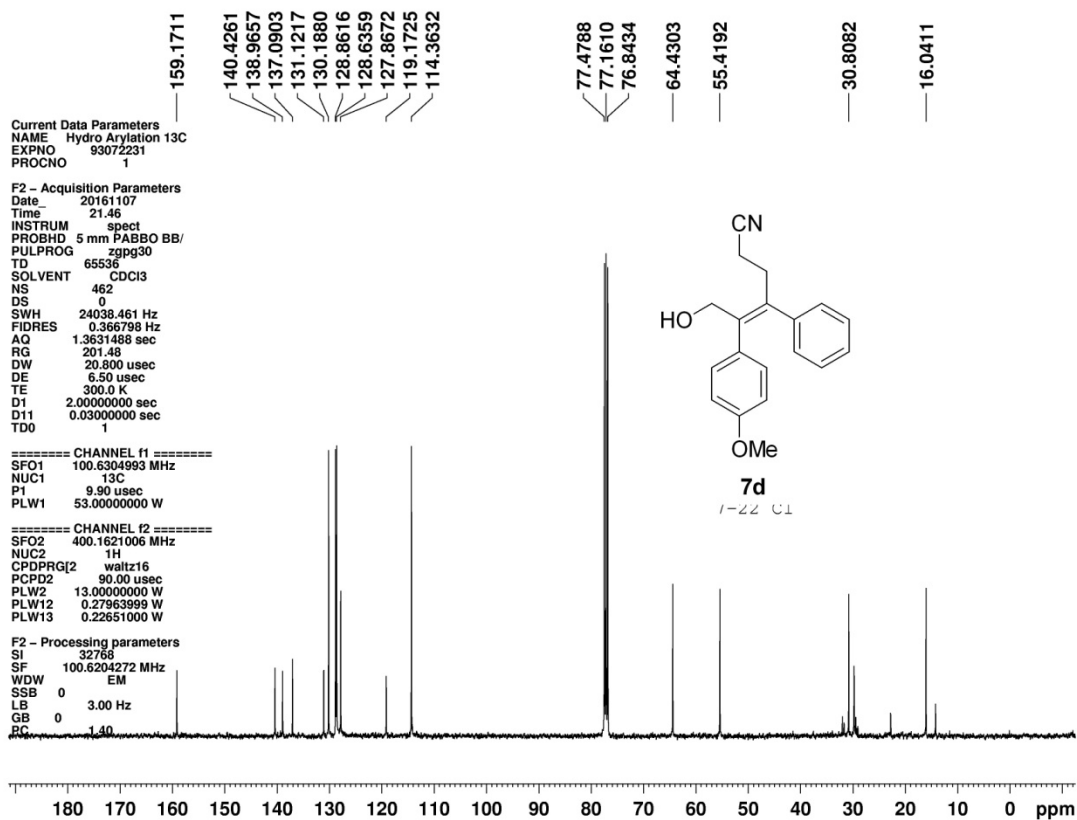
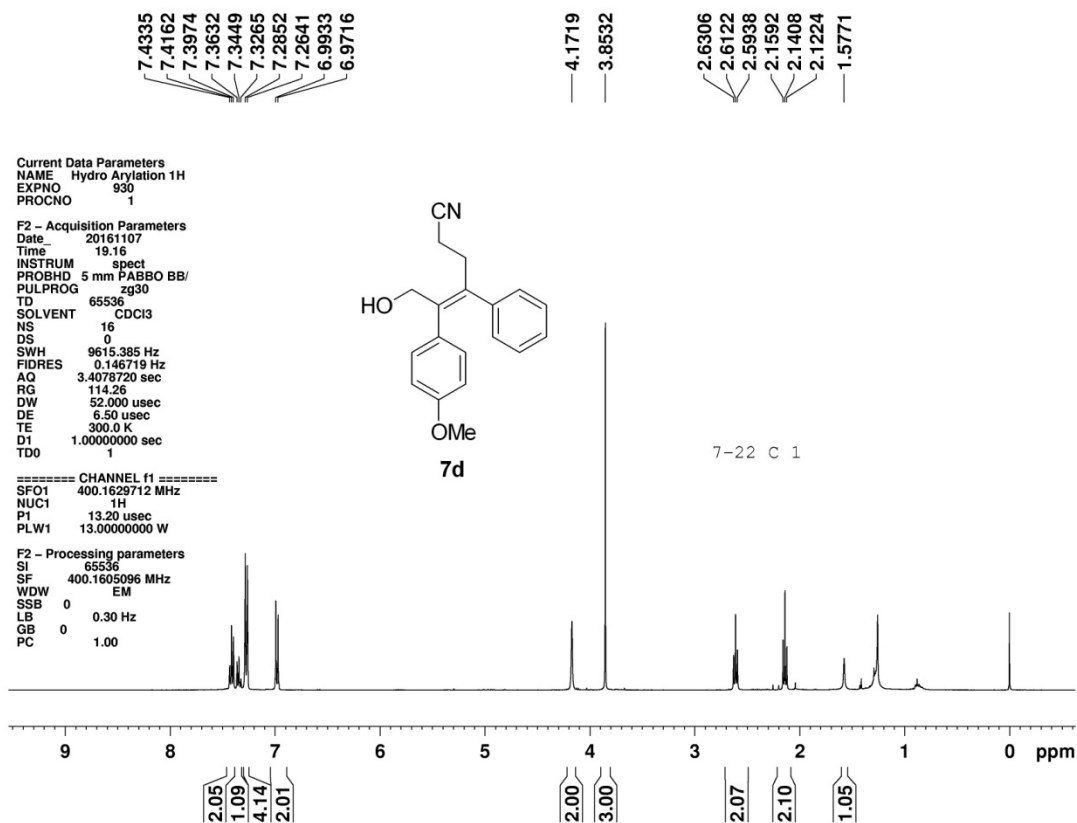












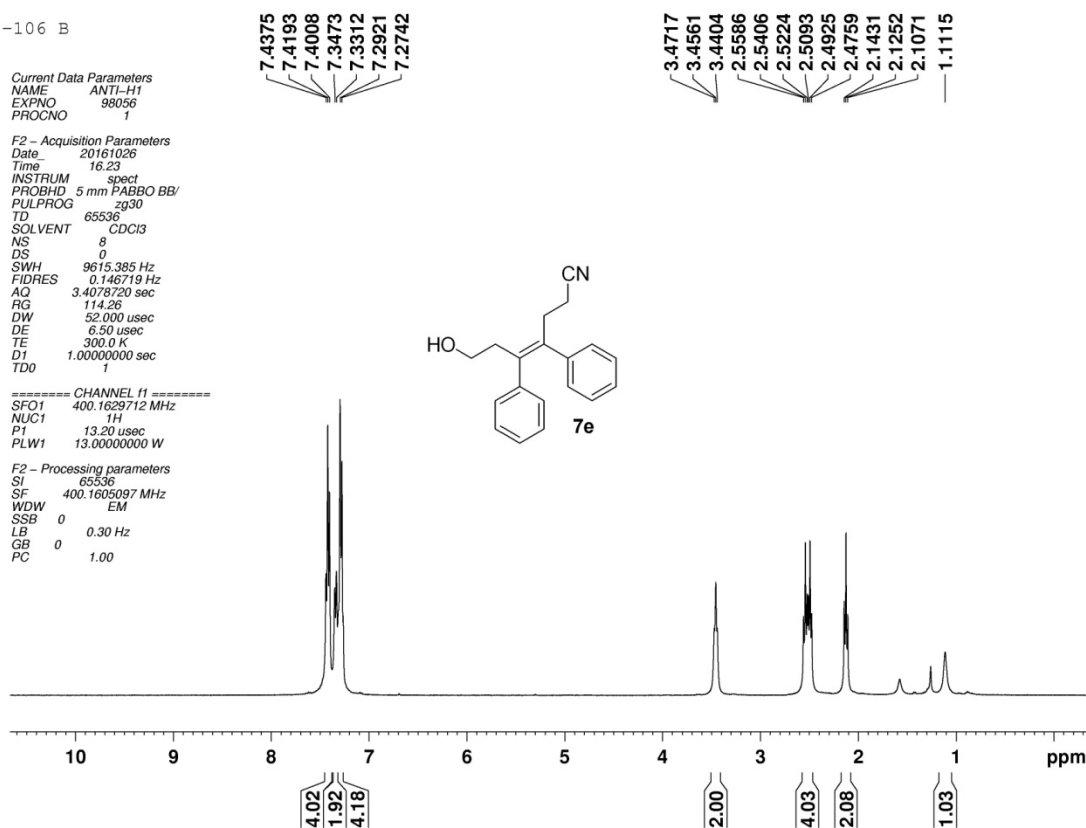
4-106 B

Current Data Parameters
 NAME ANTI-H1
 EXPNO 98056
 PROCNO 1

F2 - Acquisition Parameters
 Date 20161026
 Time 16.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCI3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 114.26
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605097 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



4-106-B

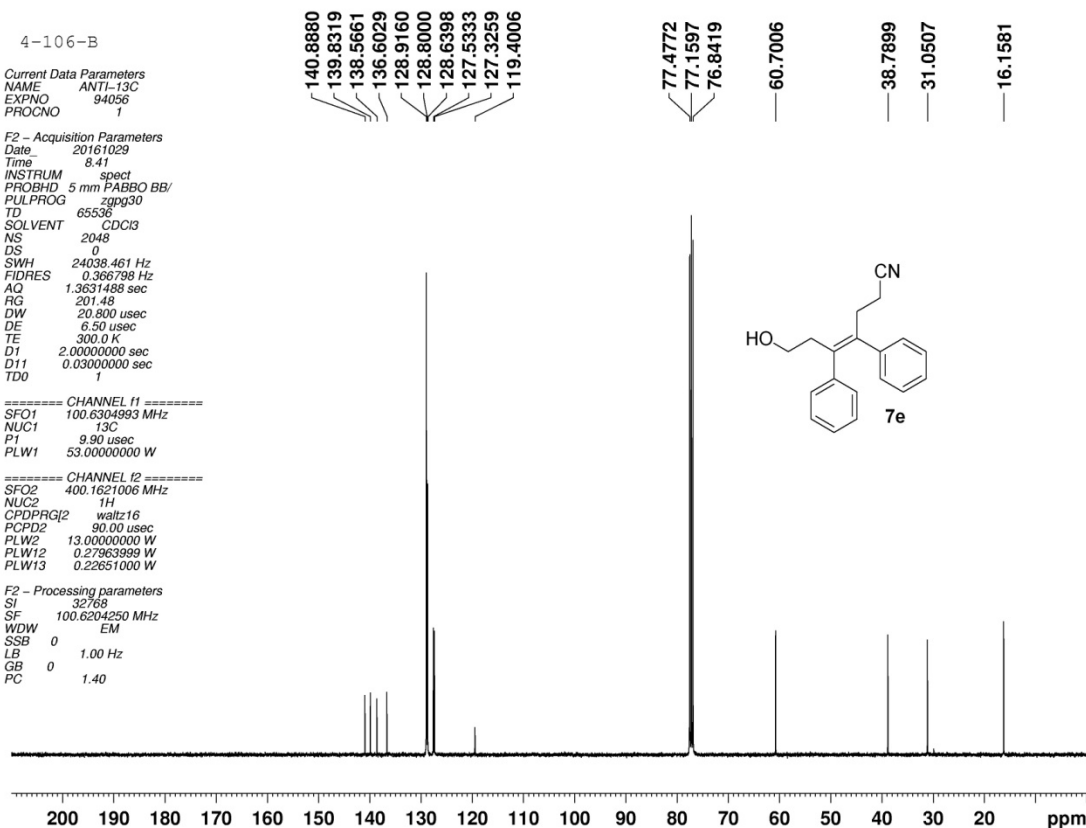
Current Data Parameters
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 EXPNO 94056
 PROCNO 1

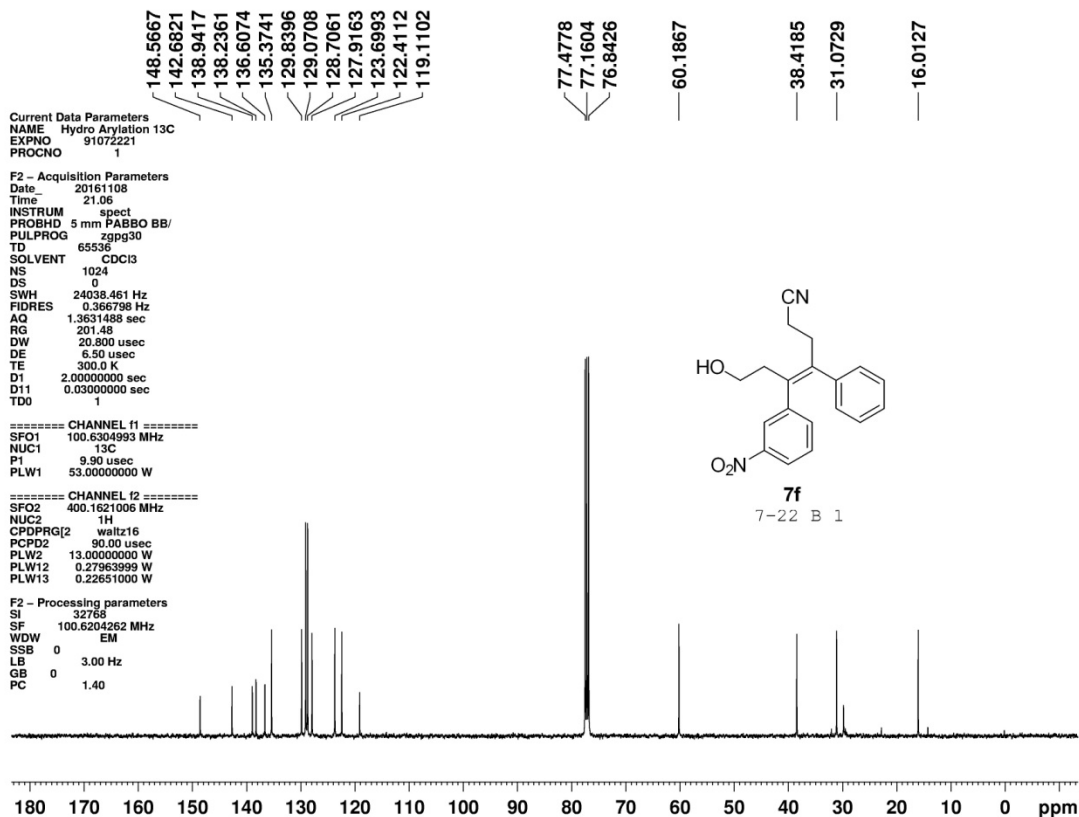
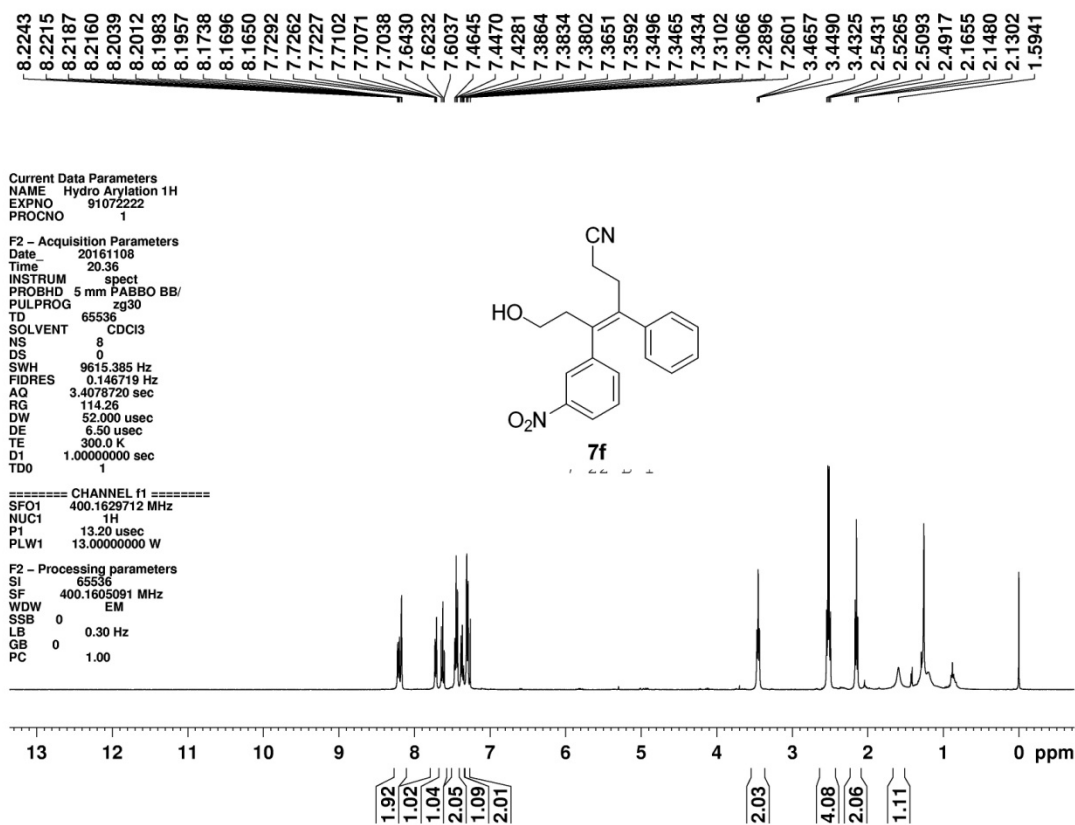
F2 - Acquisition Parameters
 Date 20161029
 Time 8.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCI3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204250 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





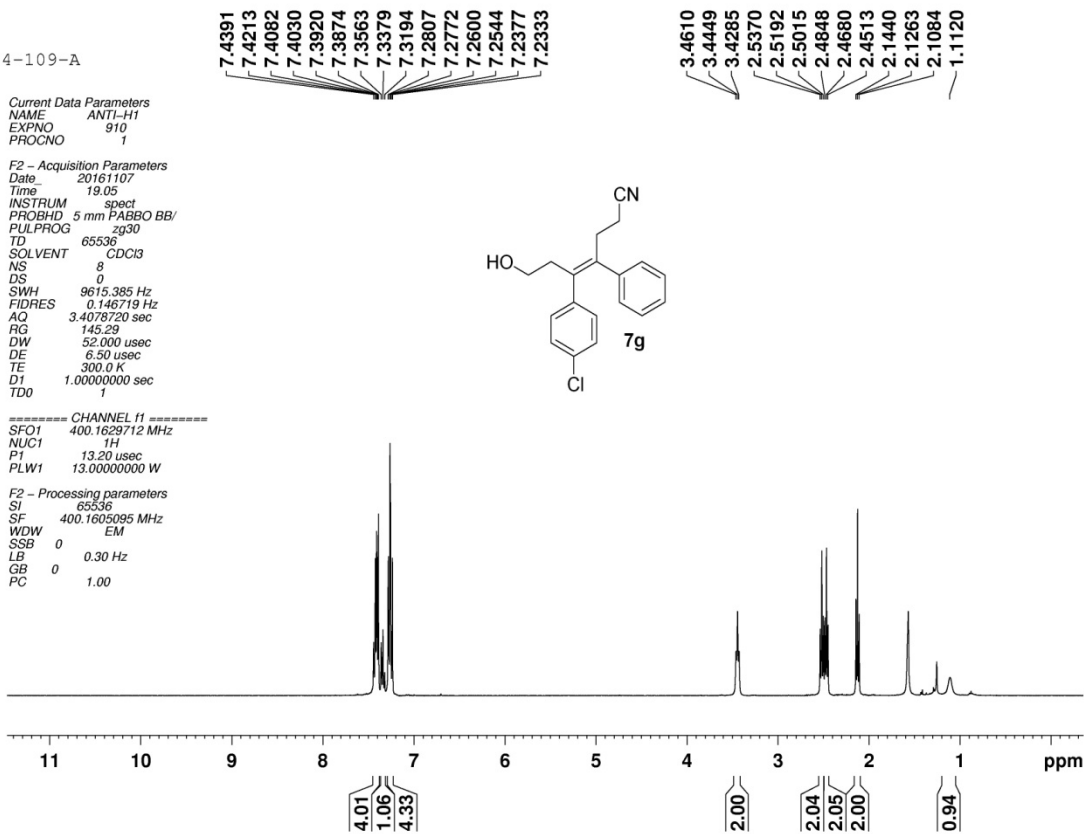
4-109-A

Current Data Parameters
 NAME ANTI-H1
 EXPNO 910
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161107
 Time 19.05
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 145.29
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



4-109 A

Current Data Parameters
 NAME ANTI-13C
 EXPNO 910
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161107
 Time 20.28
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204253 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

