Supporting Information

Pd-Catalyzed Decarboxylative Cross-Coupling Reactions of Epoxides with α,β-Unsaturated Carboxylic Acids

Table of Contents

I. General Information

a). Materials

All the reactions were carried out in oven-dried schlenk tubes under argon atmosphere (purity ≥99.999%). $Pd(PPh_3)_4$ was purchased from Energy Chemical. The following chemicals were purchased and used as received: dppf (Energy Chemical), NaI (dry, anhydrous, Energy Chemical), Cy_2NMe (Energy Chemical). Anhydrous $PhCF_3$ (Acros), dioxane, THF were stored over 4 Å molecular sieves under an argon atmosphere in a septum-capped bottle.

All the other reagents and solvents mentioned in this text were purchased from commercial sources and used without purification.

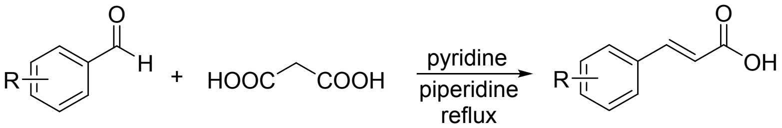
b). Analytical Methods

^1H-NMR, ^{13}C-NMR and ^{19}F-NMR spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature in $CDCl_3$ unless otherwise noted; Data for ^1H-NMR are reported as follows: chemical shift (δ ppm), multiplicity, integration, and coupling constant (Hz). Data for ^{13}C-NMR are reported in terms of chemical shift (δ ppm), multiplicity, and coupling constant (Hz). Gas chromatographic (GC) analysis was acquired on a Shimadzu GC-2014 Series GC System equipped with a flame-ionization detector. GC-MS analysis was performed on Thermo Scientific AS 3000 Series GC-MS System. HRMS analysis was performed on Finnigan LCQ advantage Max Series MS System. HPLC analysis was performed on Waters-Breeze (2487 Dual Absorbance Detector and 1525 Binary HPLC Pump). Chiralpak IC, AD, AS, KM columns were purchased from Daicel Chemical Industries, LTD. Organic solutions were concentrated under reduced pressure on a Buchi rotary evaporator. Flash column chromatographic purification of products was accomplished using forced-flow chromatography on Silica Gel (200-300 mesh).

II. Preparation of Substrates

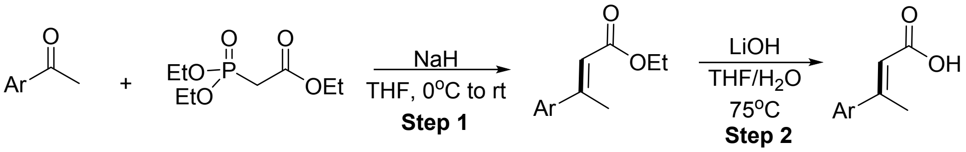
Synthesis of acrylic acids

Method 1:



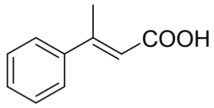
Malonic acid (10 mmol) was added to a stirred solution of benzaldehyde (5 mmol) in pyridine (20 mL) and piperidine (1 mL). The mixture was heated to reflux for 2h. The reaction was neutralized with 1M hydrochloric acid in an ice bath. The aqueous mixture was extracted with ethyl acetate, and the combined organic layer was dried over Na_2SO_4, filtrated and concentrated under vacuum. The residue was purified by flash column chromatography.[1]

Method 2:



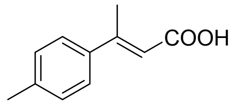
Step1: To a suspension of sodium hydride (60% in mineral oil, 11 mmol) in THF (30 mL) was added dropwise triethyl 2-phosphonopropionate (11 mmol, 1.1 equiv.). The resulting solution was stirred at 0 °C for 1 h and ketone (10 mmol, 1.0 equiv.) in THF (10 mL) was added via syringe. The reaction mixture was stirred at room temperature for 10 h and quenched with saturated aqueous NH_4Cl solution. The layers were separated and the aqueous phase was extracted with diethyl ether. The combined organic layers were washed with saturated aqueous NaCl solution, dried over Na_2SO_4 and concentrated in vacuo. Purification by column chromatography afforded the corresponding acrylate as a colorless oil.

Step2: A 50 mL round-bottom flask was charged with acrylate (10 mmol, 1 equiv.), LiOH (5 equiv.), THF/H_2O (1:1, 0.25 M) sequentially. The reaction flask was subjected to an 80 °C preheated oil bath and stirred 3 h, at which time the resulting mixture was cooled down to room temperature and extracted with diethyl ether. The aqueous phase was acidified with 2 N HCl and extracted with ethyl acetate. The combined organic layers were dried over $MgSO_4$. The volatile compounds were removed in vacuo to afford α-substituted acrylic acids.[2]



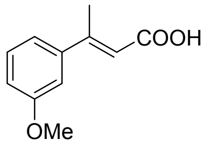
(E)-3-phenylbut-2-enoic acid (CAS: 1199-20-8)

^1H NMR (400 MHz, $CDCl_3$): δ 7.53–7.46 (m, 2H), 7.42–7.38 (m, 3H), 6.19 (q, J = 1.2 Hz, 1H), 2.60 (d, J = 1.2 Hz, 3H).



(E)-3-(p-tolyl)but-2-enoic acid (CAS: 32147-13-0)

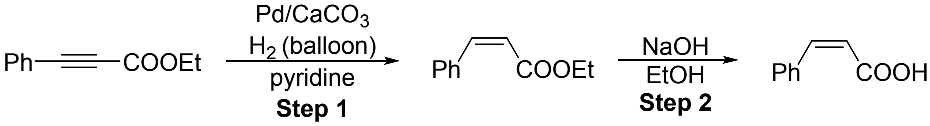
^1H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 8.0 Hz, 2H), 7.20 (d, J = 8.0 Hz, 2H), 6.17 (s, 1H), 2.58 (s, 3H), 2.38 (s, 3H).



(E)-3-(3-methoxyphenyl)but-2-enoic acid (CAS:37730-32-8)

^1H NMR (400 MHz, $CDCl_3$) δ 7.35−7.26 (m, 1H), 7.12−7.06 (m, 1H), 7.02−7.00 (m, 1H), 6.94−6.90 (m, 1H), 6.17 (s, 1H), 3.85 (s, 3H), 2.58 (d, J = 1.3 Hz, 3H).

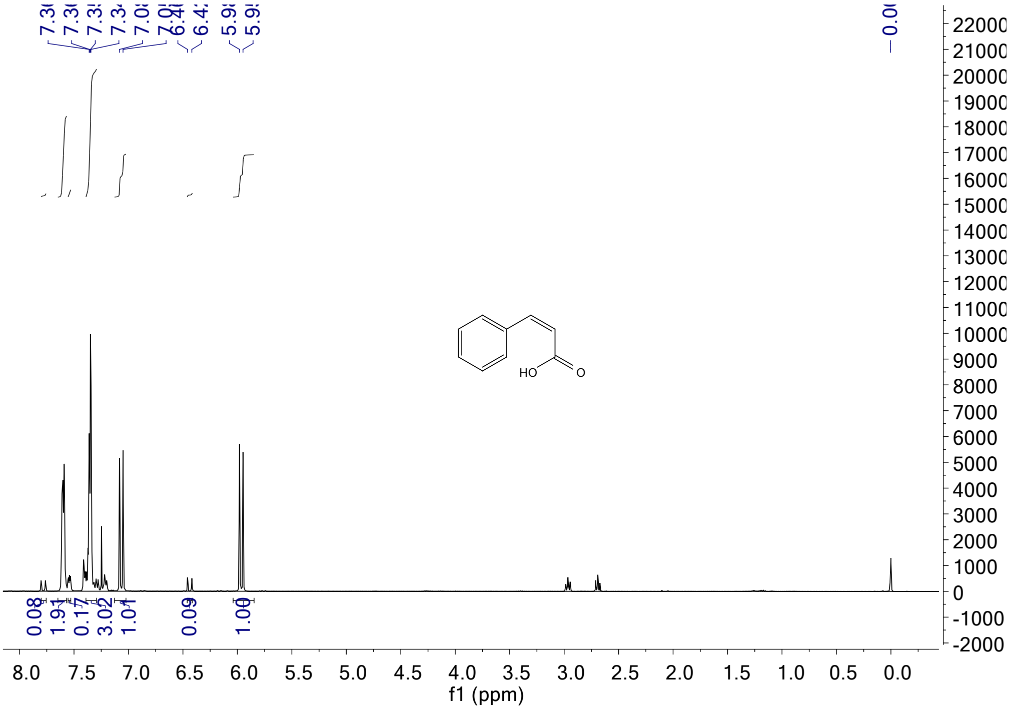
Synthesis of *cis*-cinnamic acid



Step 1. To a stirred solution of ethyl 3-phenylpropiolate (10 mmol) in MeOH (20 mL) were added Lindlar catalyst (5% Pd on $CaCO_3$, poisoned with lead) (1 mmol) and pyridine (10 mmol). The reaction mixture was stirred under H_2 balloon for 4 h at room temperature. The mixture was filtered through a Celite® pad, which was washed with EtOAc (50 mL). The filtrate was washed with 2 N HCl (30 mL) and brinesuccessively, dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo. The residue was used for the next step without further purification.

Step 2. To a stirred solution of ethyl (Z)-3-phenylacrylate in EtOH (20 mL) was added 1 M aqueous NaOH (1.5 eq.) and the reaction mixture was stirred at room temperature for 12 h. The reaction was concentrated in vacuo and extracted with CH_2Cl_2 (30 mL × 3). The organic layer was washed with 5% aqueous citric acid and brine. The extract was dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo to afford crude *cis*-cinnamic acid.[3]

^{1}H NMR of the ***cis*****-cinnamic acid** (400 MHz, $CDCl_3$) δ ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 – 7.57 (m, 2H), 7.42 – 7.32 (m, 3H), 7.07 (d, J = 12.7 Hz, 1H), 5.96 (d, J = 12.7 Hz, 1H). The ^{1}H NMR data is the same as the literature.



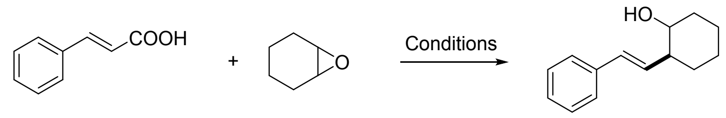
Rferences

1. (a) Li, L.; Chen, M.; Jiang, F.-C., Design, synthesis, and evaluation of 2-piperidone derivatives for the inhibition of β-amyloid aggregation and inflammation mediated neurotoxicity. *Bioorg. Med. Chem.* 2016, **24**, 1853-1865; (b) Tardugno, R.; Giancotti, G.; De Burghgraeve, T.; Delang, L.; Neyts, J.; Leyssen, P.; Brancale, A.; Bassetto, M., Design, synthesis and evaluation against Chikungunya virus of novel small-molecule antiviral agents. *Bioorg. Med. Chem.* 2018, **26**, 869-874.
2. Jiang, B.; Zhao, M.; Li, S.-S.; Xu, Y.-H.; Loh, T.-P., Macrolide Synthesis through Intramolecular Oxidative Cross-Coupling of Alkenes. *Angew. Chem. Int. Ed.,* 2018, **57**, 555 - 559.
3. Du, W.; Gu, Q.; Li, Y.; Lin, Z.; Yang, D., Enantioselective Palladium-Catalyzed Oxidative Cascade Cyclization of Aliphatic Alkenyl Amides. *Org. Lett.*, 2017, **19**, 316-319.

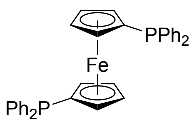
III. General Experimental Procedures

Experimental Procedures for Examples Described in Table 1.

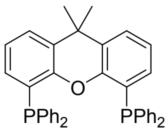
In air, Catalyst (5 mol%), Ligand (6 mol%), cinnamic acid and aditive (30 mol%) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent, Cy_2NMe (1.5 equiv.) and cyclohexene oxide were added in turn by syringe. The resulting reaction mixture was stirred vigorously at the mentioned temperature for the indicated amount of time. Benzophenone was added as internal standard. The product was yielded by GC.



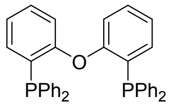
1a **2a** **3a**



dppf



Xantphos



DPEphos

Entry	Catalyst	Ligand	Base	Solvent	Aditive	T(℃)	Yield (trans/cis)
1[a]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	$DMF/PhCF_3$	NaI	100	20(4.7:1)
2[a]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	$PhCF_3$	NaI	100	28(4.5:1)
3[a]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	dioxane	NaI	100	31(3.5:1)
4[a]	$Pd(PPh_3)_4$	Xantphos	Cy_2NMe	$PhCF_3$	NaI	100	10(3:1)
5[a]	$Pd(PPh_3)_4$	Xantphos	Cy_2NMe	dioxane	NaI	100	15(3.2:1)
6[b]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	$PhCF_3$	NaI	100	50(4:1)
7[b]	$Pd(PPh_3)_4$	Dppe	Cy_2NMe	$PhCF_3$	NaI	100	10(4.2:1)
8[b]	$Pd(PPh_3)_4$	dppp	Cy_2NMe	$PhCF_3$	NaI	100	2
9[b]	$Pd(PPh_3)_4$	PCy_3	Cy_2NMe	$PhCF_3$	NaI	100	trace
10[b]	$Pd(PPh_3)_4$	DPEphos	Cy_2NMe	$PhCF_3$	NaI	100	16(4.2:1)
11[b]	$Pd(PPh_3)_4$	dppf	K_3PO_4	$PhCF_3$	NaI	100	8(4:1)
12[b]	$Pd(PPh_3)_4$	dppf	Cs_2CO_3	$PhCF_3$	NaI	100	5(4:1)
13[b]	$Pd(PPh_3)_4$	dppf	$(iPr)_2NEt$	$PhCF_3$	NaI	100	10(4.1:1)
14[b]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	$PhCF_3$	NaI	90	62(5:1)
15[b]	$Pd(OAc)_2$	dppf	Cy_2NMe	$PhCF_3$	NaI	90	23(5:1)
16[b]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	Toluene	NaI	90	46(5.5:1)
17[b]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	DMA	NaI	90	21(4.2:1)
18[b]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	dioxane	NaI	90	65(5:1)
19[b]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	$PhCF_3$	LiBr	90	trace
20[b]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	$PhCF_3$	$BuNI_4$	90	9(3.5:1)
21[d]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	$PhCF_3/DMA$	NaI	90	28(4.3:1)
22[e]	$Pd(PPh_3)_4$	dppf	Cy_2NMe	$PhCF_3$/dioxane	NaI	90	77 (4.2:1)
23[f]	**$Pd(PPh_3)_4$**	**dppf**	**Cy_2NMe**	**$PhCF_3$/dioxane**	**$NaI/Et_3N{\cdot}HCl$**	**90**	**81(3.9:1)**

24	-	dppf	Cy_2NMe	$PhCF_3$/dioxane	$NaI/Et_3N{\cdot}HCl$	90	0
25	$Pd(PPh_3)_4$	-	Cy_2NMe	$PhCF_3$/dioxane	$NaI/Et_3N{\cdot}HCl$	90	trace
26	$Ni(acac)_2$	Bpy	K_3PO_4	DMA	$NaI/Et_3N{\cdot}HCl$	90	trace
27	$Ni(acac)_2$	Bpy	K_2CO_3	DMA	$NaI/Et_3N{\cdot}HCl$	90	trace

[a]**Reaction conditions:** 1a (0.6 mmol), 2a (0.3 mmol), ctalyst (5 mol%), Base (1.5 equiv.), ligand (6 mol%), aditive (30 mol%) in 2 mL solvent at 100 °C for 24 h under Ar atmosphere. [b]**Reaction conditions:** 1a (0.3 mol), 2a (0.6 mol), ctalyst (5 mol%), Base (1.5 equiv.), ligand (6 mol%), aditive (30 mol%) in 2 mL solvent at 100 °C or 90 °C for 24 h under Ar atmosphere. [c]**Reaction conditions:** 1a (0.3 mol), 2a (0.6 mol), ctalyst (5 mol%), Base (1.5 equiv.), ligand (6 mol%), aditive (30 mol%) in 2 mL solvent ($PhCF_3/H_2O$ = 10:1) at 90 °C for 24 h under Ar atmosphere. [d]**Reaction conditions:** 1a (0.3 mol), 2a (0.6 mol), ctalyst (5 mol%), Base (1.5 equiv.), ligand (6 mol%), aditive (30 mol%) in 2 mL solvent ($PhCF_3$/DMA = 10:1) at 90 °C for 24 h under Ar atmosphere. [e]**Reaction conditions:** 1a (0.3 mol), 2a (0.6 mol), ctalyst (5 mol%), Base (1.5 equiv.), ligand (6 mol%), aditive (30 mol%) in 2 mL solvent ($PhCF_3$/dioxane = 10:1) at 90 °C for 24 h under Ar atmosphere. [f]**Reaction conditions:** 1a (0.3 mol), 2a (0.6 mol), ctalyst (5 mol%), Base (1.5 equiv.), ligand (6 mol%), $NaI/Et_3N{\cdot}HCl$ (30 mol%) in 1.5 mL solvent ($PhCF_3$/dioxane = 5:1) at 90 °C for 30 h under Ar atmosphere. The yield was determined by GC (average of two GC runs). Bpy = 2,2'-Bipyridine. The trans/cis ratio was determined by GC.

General procedure：Experimental Procedures for Examples Described in Table 2

In air, $Pd(PPh_3)_4$ (5 mol%), dppf (6 mol%) and α,β-unsaturated carboxylic acids (0.3 mmol) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent (1.5 mL, $PhCF_3$: dioxane = 5:1), Cy_2NMe (1.5 equiv.) were added in turn by syringe, stirred at room temperature for 2 minutes, then NaI (30 mol%), $Et_3N{\cdot}HCl$ (30 mol%) and epoxide (0.6 mmol) were added to a schlenk tube equipped with a stir bar. The resulting reaction mixture was stirred vigorously at 90 °C for 30 h under Ar atmosphere. The resulting solution was then extracted with CH_2Cl_2, dried over Na_2SO_4, and filtered. The resulting solution was then extracted with CH_2Cl_2, dried over Na_2SO_4, and filtered, concentrated, and purified by column chromatography.

Experimental Procedures for Examples Described in Scheme 2

In air, $Pd(PPh_3)_4$ (5 mol%), dppf (6 mol%) and sorbic acid (0.5 mmol), NaI (30 mol%) and $Et_3N{\cdot}HCl$ (30 mol%) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent (2.5 mL, $PhCF_3$: dioxane = 5:1), Cy_2NMe (1.5 equiv.) were added in turn by syringe, stirred at room temperature for 2 minutes, then cyclopentene oxide (3 equiv.) were added to a schlenk tube equipped with a stir bar. The resulting reaction mixture was stirred vigorously at 95 °C for 30 h under Ar atmosphere. The resulting solution was then extracted with CH_2Cl_2, dried over Na_2SO_4, and filtered. The resulting solution was then extracted with CH_2Cl_2, dried over Na_2SO_4, and filtered, concentrated, and purified by column chromatography.

Experimental Procedures for Examples Described in Table 3

In air, $Pd(PPh_3)_4$ (8 mol%), dppf (10 mol%) and α,β-unsaturated carboxylic acids (0.3 mmol) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent (2 mL, $PhCF_3$: dioxane = 5:1), Cy_2NMe (1.5 equiv.) were added in turn by syringe, stirred at room temperature for 2 minutes, then NaI (30 mol%), $Et_3N{\cdot}HCl$ (30 mol%) and epoxide (3 equiv.) were added to a schlenk tube equipped with a stir bar. The resulting reaction mixture was stirred vigorously at 95 ^{o}C for 36 h under Ar atmosphere. The resulting solution was then extracted with CH_2Cl_2, dried over Na_2SO_4, and filtered. The resulting solution was then extracted with CH_2Cl_2, dried over Na_2SO_4, and filtered, concentrated, and purified by column chromatography.

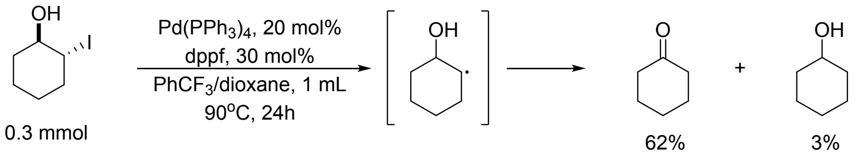
Experimental Procedures for Examples Described in Scheme 3 (acyclic epoxides)

In air, $Pd(PPh_3)_4$ (8 mol%), dppf (10 mol%) and α,β-unsaturated carboxylic acids (0.3 mmol) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent (2 mL, $PhCF_3$: dioxane = 5:1), Cy_2NMe (1.5 equiv.) were added in turn by syringe, stirred at room temperature for 2 minutes, then NaI (30 mol%), $Et_3N{\cdot}HCl$ (30 mol%) and epoxide were added to a schlenk tube equipped with a stir bar. The resulting reaction mixture was stirred vigorously at 90 ^{o}C for 48 h under Ar atmosphere. The resulting solution was then extracted with CH_2Cl_2, dried over Na_2SO_4, and filtered. The resulting solution was then extracted with CH_2Cl_2, dried over Na_2SO_4, and filtered, concentrated, and purified by column chromatography.

Experimental Procedures for Examples Described in Scheme 4

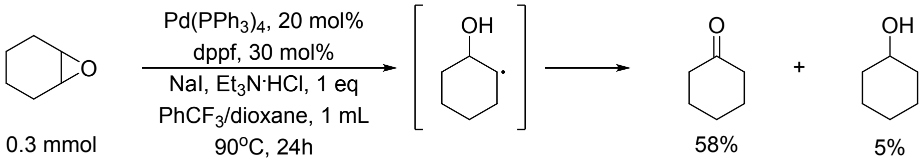
In air, $Pd(PPh_3)_4$ (8 mol%), dppf (10 mol%) and cinnamic acid (0.3 mmol) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent (2 mL, $PhCF_3$: dioxane = 5:1), Cy_2NMe (1.5 equiv.) were added in turn by syringe, stirred at room temperature for 2 minutes, then NaI (30 mol%), $Et_3N{\cdot}HCl$ (30 mol%) and (+)-limonene 1,2-epoxide (3 equiv.) were added to a schlenk tube equipped with a stir bar. The resulting reaction mixture was stirred vigorously at 90 °C for 36 h under Ar atmosphere. The resulting solution was then extracted with CH_2Cl_2, dried over Na_2SO_4, and filtered. The resulting solution was then extracted with CH_2Cl_2, dried over Na_2SO_4, and filtered, concentrated, and purified by column chromatography.

A reaction of in situ formed palladium(0) complex of dppf and 2-iodocyclohexanol

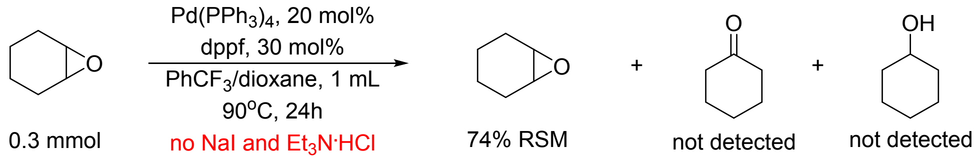


In air, $Pd(PPh_3)_4$ (20 mol%) and dppf (30 mol%) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent (1 mL, $PhCF_3$: dioxane = 5:1) was added, after stirring at room temperature for 10 min, 2-iodocyclohexanol (0.3 mmol) was added. The resulting reaction mixture was stirred vigorously at 90 °C for 24 h. Acetophenone was added as internal standard. The product was yielded by GC and GC-MS.

A reaction of in situ formed palladium(0) complex of dppf and Cyclohexene oxide

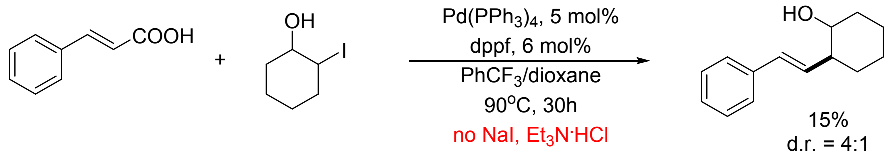


In air, $Pd(PPh_3)_4$ (20 mol%) and dppf (30 mol%) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent (1 mL, $PhCF_3$: dioxane = 5:1), after stirring at room temperature for 10 min, then NaI (1 equiv.), $Et_3N{\cdot}HCl$ (1 equiv.) and cyclohexene oxide (0.3 mmol) were added to a schlenk tube equipped with a stir bar. The resulting reaction mixture was stirred vigorously at 90 °C for 24 h. Acetophenone was added as internal standard. The product was yielded by GC and GC-MS.



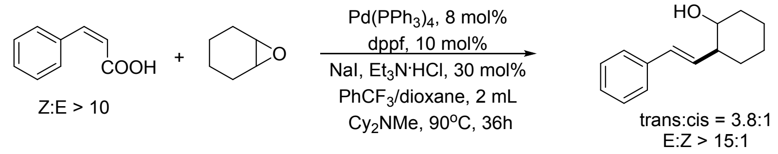
In air, $Pd(PPh_3)_4$ (20 mol%) and dppf (30 mol%) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent (1 mL, $PhCF_3$: dioxane = 5:1), after stirring at room temperature for 10 min, then cyclohexene oxide was added to a schlenk tube equipped with a stir bar. The resulting reaction mixture was stirred vigorously at 90 °C for 24 h. Acetophenone was added as internal standard. The product was yielded by GC and GC-MS.

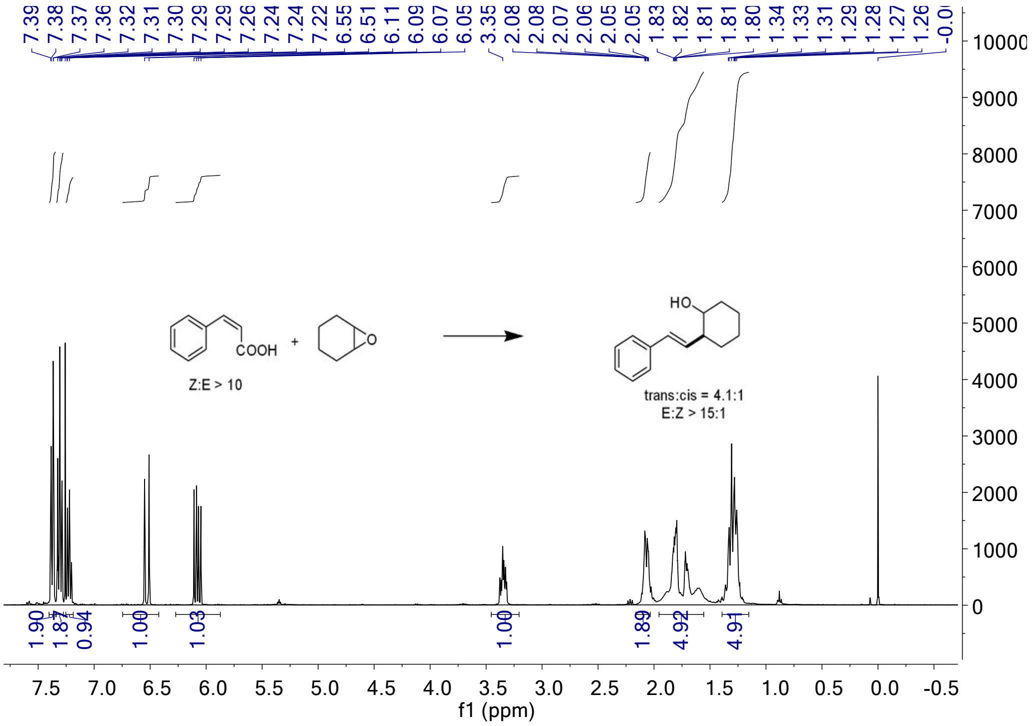
Experiment of iodohydrin

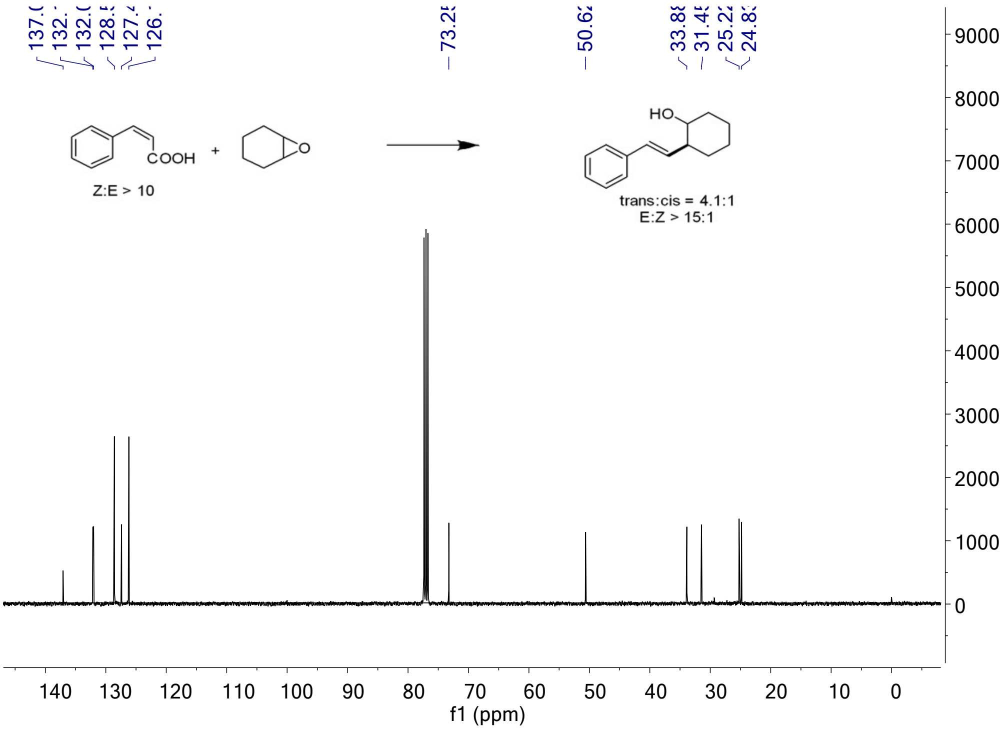


In air, $Pd(PPh_3)_4$ (5 mol%)，dppf (6 mol%), iodohydrin (0.6 mmol) and cinnamic acid (0.3 mmol) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent (1.5 mL, $PhCF_3$: dioxane = 5:1), after stirring at room temperature for 10 min, then cyclohexene oxide was added to a schlenk tube equipped with a stir bar. The resulting reaction mixture was stirred vigorously at 90 °C for 24 h. Acetophenone was added as internal standard. The product was yielded by GC.

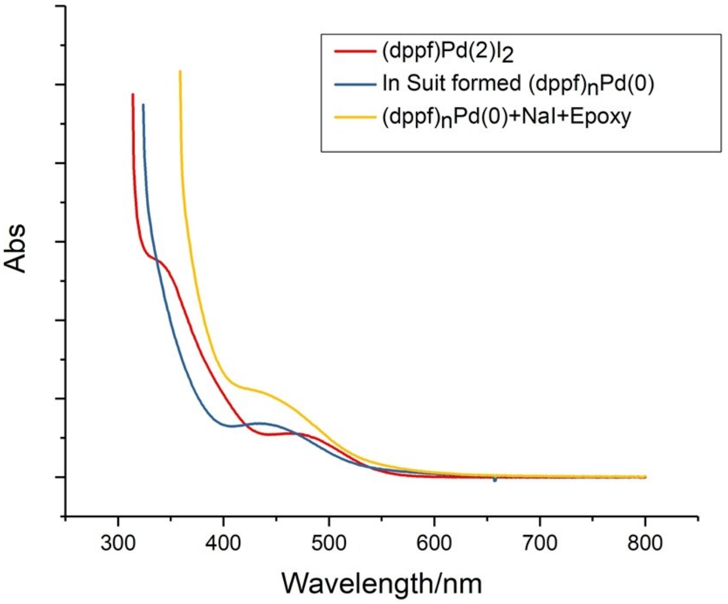
Experiment of cis-cinnamic acid







UV-Vis Absorption Spectroscopy Experimental of Catalyst



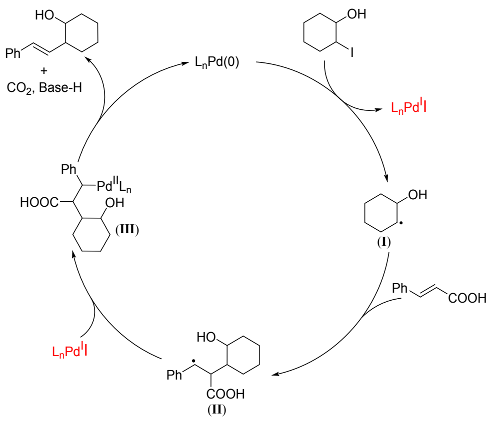
Synthesis of (dppf)Pd(2)I$_2$ complexes

A mixture of PdI$_2$ (0.3 mmol), dppf (0.3 mmol) and dry toluene (0.3 mL) were added to a dry 10 mL Schlenk tube under argon. The reaction mixture was stirred at 120 ^{o}C for 3 h. After cooled down to rt, the crude product was filtered under vacuum in air, and then washed carefully with anhydrous Et_2O (*Ref. Angew. Chem. Int. Ed.* 2015, **54**, 10912-10916).

In situ formed palladium(0) complex of dppf

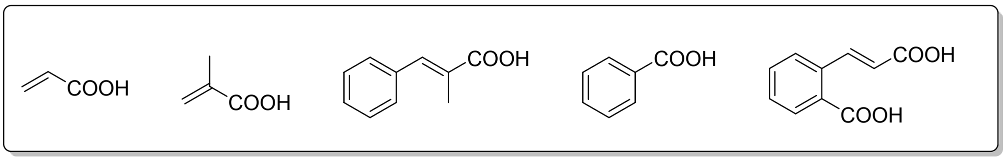
In air, $Pd(PPh_3)_4$ (0.2 mmol) and dppf (0.4 mmol) were added to a schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Solvent (5 mL dioxane), was added, stirring at room temperature for 20 min.

Proposed reaction mechanism

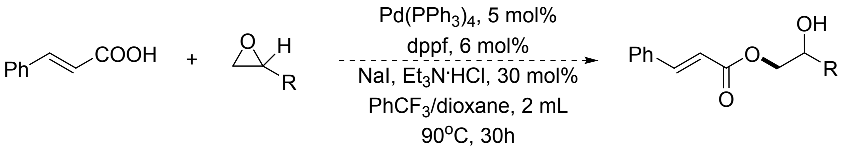


Incompatible substrate

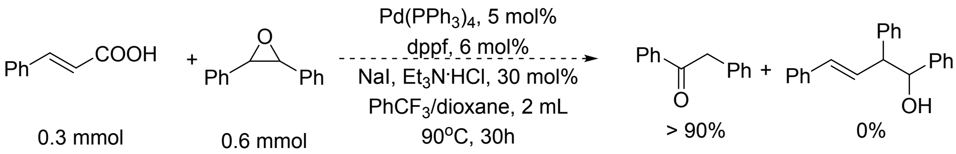
(a)

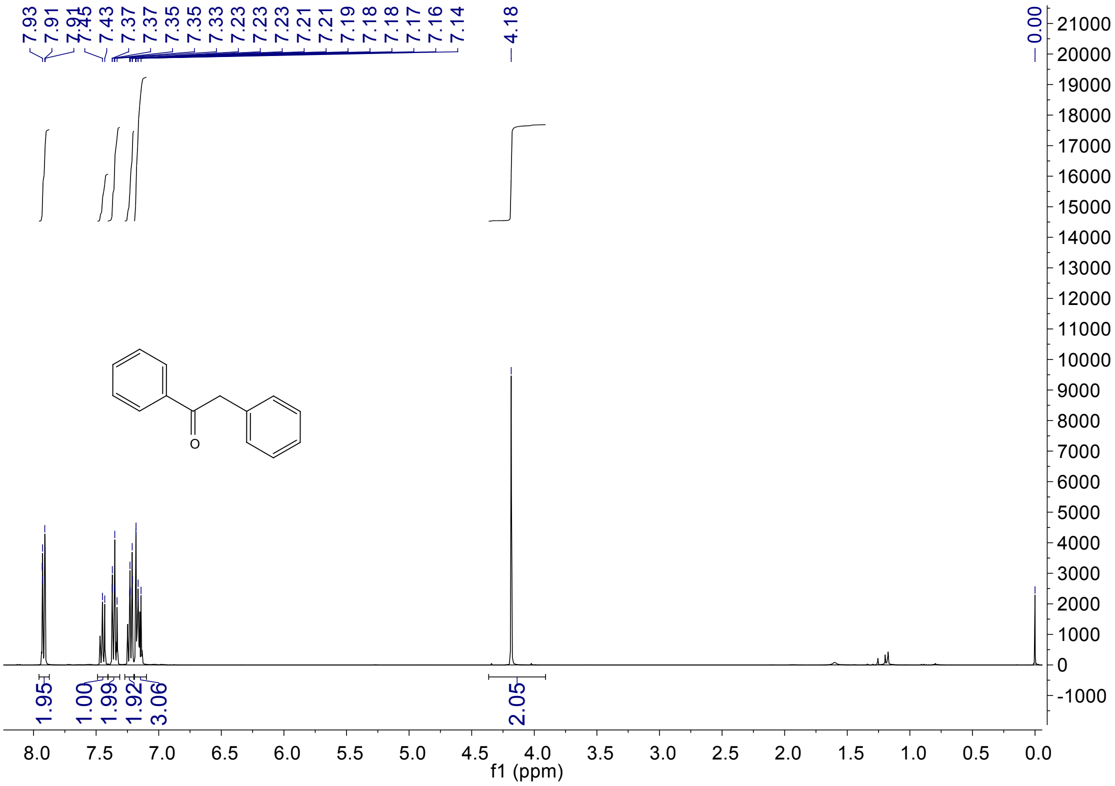


(b)

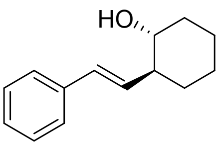


(c)



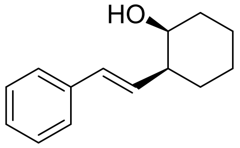


Ⅳ. Substrate Scope and Spectral Data

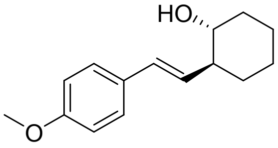


(E)-trans-2-styrylcyclohexan-1-ol

Following general procedure, as a sticky solid. The *trans/cis* ratio was determined to be 3.9:1 by GC and ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.40 – 7.35 (m, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.25 – 7.18 (m, 1H), 6.53 (d, *J* = 15.9 Hz, 1H), 6.08 (dd, *J* = 15.9, 8.9 Hz, 1H), 3.40 – 3.29 (m, 1H), 2.07 (d, *J* = 8.9 Hz, 2H), 1.81 (dd, *J* = 8.8, 2.7 Hz, 2H), 1.74 – 1.63 (m, 2H), 1.30 (dd, *J* = 18.3, 7.7 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.07, 132.19, 131.99, 128.59, 127.41, 126.21, 73.26, 50.62, 33.90, 31.46, 25.23, 24.84. HRMS (APCI) calcd for C14H18NaO (M+Na+): 225.1250; found: 225.1252.

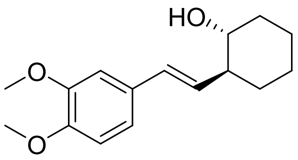


^{1}H NMR of *cis* isomer (400 MHz, $CDCl_3$) δ 7.41 – 7.35 (m, 2H), 7.32 – 7.27 (m, 2H), 7.25 – 7.16 (m, 1H), 6.48 (d, *J* = 15.9 Hz, 1H), 6.35 (dd, *J* = 16.1, 6.9 Hz, 1H), 3.96 – 3.91 (m, 1H), 2.51 – 2.37 (m, 1H), 1.77 – 1.56 (m, 6H), 1.36 – 1.24 (m, 3H).



(E)-trans-2-(4-methoxystyryl)cyclohexan-1-ol

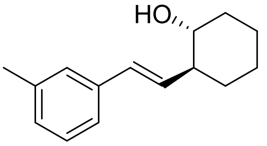
Following general procedure, as a sticky solid. The *trans/cis* ratio was determined to be 3.7:1 by GC and ^{1}H NMR. ^{1}H NMR of mixed products (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.46 (d, *J* = 15.9 Hz, 1H), 5.92 (dd, *J* = 15.9, 8.8 Hz, 1H), 3.80 (s, 3H), 3.31 (td, *J* = 9.9, 4.3 Hz, 1H), 2.13 – 1.94 (m, 3H), 1.86 – 1.67 (m, 3H), 1.41 – 1.18 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.24, 159.05, 136.08, 131.41, 131.30, 129.89, 128.68, 127.33, 113.97, 113.63, 83.67, 73.28, 55.35, 55.33, 50.64, 48.12, 33.84, 31.56, 30.54, 26.56, 25.61, 25.28, 24.86, 24.17. HRMS (APCI) calcd for C15H20NaO2 (M+Na+): 255.1356; found: 255.1359.



(E)- trans-2-(3,4-dimethoxystyryl)cyclohexan-1-ol

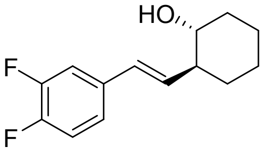
Following general procedure, as a sticky solid. The *trans/cis* ratio was determined to be 5:1

by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, CDCl3) δ 6.95 – 6.88 (m, 2H), 6.81 (d, J = 8.2 Hz, 1H), 6.46 (d, J = 15.8 Hz, 1H), 5.93 (dd, J = 15.8, 8.8 Hz, 1H), 3.91 (s, 3H), 3.88 (s, 3H), 3.33 (td, J = 9.9, 4.3 Hz, 1H), 2.19 – 1.98 (m, 2H), 1.94 (m, 1H), 1.86 – 1.69 (m, 3H), 1.37 – 1.22 (m, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 149.02, 148.65, 131.67, 130.17, 130.13, 119.32, 111.10, 108.51, 73.30, 55.95, 55.84, 50.61, 33.89, 31.57, 25.28, 24.85. HRMS (APCI) calcd for C16H22NaO3 (M+Na+): 285.1461; found: 285.1465.



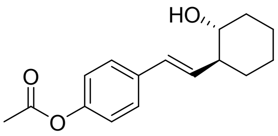
(E)-trans-2-(3-methylstyryl)cyclohexan-1-ol

Following general procedure, as a sticky solid. The *trans/cis* ratio was determined to be 4.8:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, CDCl$_3$) δ 7.22 – 7.14 (m, 3H), 7.07 – 7.01 (m, 1H), 6.48 (d, *J* = 15.9 Hz, 1H), 6.05 (dd, *J* = 15.9, 8.8 Hz, 1H), 3.32 (td, *J* = 9.9, 4.4 Hz, 1H), 2.33 (s, 3H), 2.12 – 1.94 (m, 3H), 1.86 – 1.62 (m, 3H), 1.28 (dt, *J* = 8.8, 8.2 Hz, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 138.13, 137.02, 132.07, 131.97, 128.50, 128.20, 126.89, 123.41, 73.26, 50.62, 33.88, 31.47, 25.25, 24.86, 21.44. HRMS (APCI) calcd for C15H20NaO (M+Na+): 239.1406; found: 239.1405.



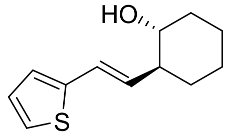
(E)-trans-2-(3,4-difluorostyryl)cyclohexan-1-ol

Following general procedure, as a sticky solid. The *trans/cis* ratio was determined to be 6:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, CDCl$_3$) δ 7.18 (ddd, *J* = 11.3, 7.5, 1.6 Hz, 1H), 7.10 – 7.01 (m, 2H), 6.42 (d, *J* = 15.9 Hz, 1H), 6.03 (dd, *J* = 15.9, 8.7 Hz, 1H), 3.35 (td, *J* = 10.0, 4.4 Hz, 1H), 2.13 – 1.99 (m, 2H), 1.86 – 1.66 (m, 4H), 1.28 (ddd, *J* = 15.7, 12.1, 5.6 Hz, 4H). ^{19}F NMR (377 MHz, CDCl$_3$) δ -138.05 (d, *J* = 21.1 Hz), -139.58 (d, *J* = 21.1 Hz). ^{13}C NMR (101 MHz, CDCl$_3$) δ 150.47 (dd, *J* = 247.4, 12.9 Hz), 149.61 (dd, *J* = 248.3, 12.9 Hz), 134.42 (dd, *J* = 5.8, 4.0 Hz), 133.51 (d, *J* = 2.2 Hz), 129.71, 122.36 (dd, *J* = 6.1, 3.4 Hz), 117.24 (d, *J* = 17.3 Hz), 114.44 (d, *J* = 17.5 Hz), 73.30, 50.32, 34.17, 31.37, 25.13, 24.78. HRMS (APCI) calcd for C14H16F2NaO (M+Na+): 261.1061; found: 261.1062.



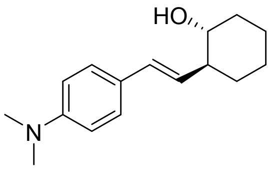
(E)-trans-4-(2-(2-hydroxycyclohexyl)vinyl)phenyl acetate

Following general procedure, as a sticky solid. The *trans/cis* ratio was determined to be 6:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.41 – 7.33 (m, 2H), 7.08 – 6.99 (m, 2H), 6.50 (d, *J* = 15.9 Hz, 1H), 6.03 (dd, *J* = 15.9, 8.8 Hz, 1H), 3.34 (td, *J* = 9.9, 4.3 Hz, 1H), 2.29 (s, 3H), 2.06 (dd, *J* = 8.6, 3.5 Hz, 2H), 1.78 (dd, *J* = 22.9, 17.3 Hz, 4H), 1.40 – 1.22 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.53, 149.86, 134.91, 132.49, 130.96, 127.12, 121.69, 73.25, 50.56, 33.93, 31.42, 25.20, 24.82, 21.17. HRMS (APCI) calcd for C16H20NaO3 (M+Na+):283.1305; found: 283.1311.



(E)-trans-2-(2-(thiophen-2-yl)vinyl)cyclohexan-1-ol

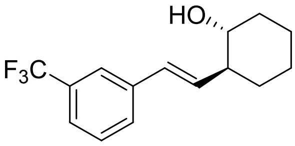
Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 5:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.11 (d, *J* = 5.0 Hz, 1H), 6.93 (dt, *J* = 9.5, 3.2 Hz, 2H), 6.63 (d, *J* = 15.7 Hz, 1H), 5.91 (dd, *J* = 15.7, 8.7 Hz, 1H), 3.31 (td, *J* = 9.9, 4.3 Hz, 1H), 2.14 – 1.88 (m, 3H), 1.89 – 1.60 (m, 3H), 1.48 – 1.13 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.38, 132.02, 127.32, 125.16, 125.04, 123.73, 73.24, 50.40, 33.96, 31.37, 25.19, 24.80. HRMS (APCI) calcd for C12H16NaOS (M+Na+): 231.0814; found: 231.0815.



(E)-trans-2-(4-(dimethylamino)styryl)cyclohexan-1-ol

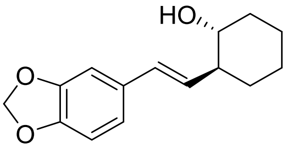
Following general procedure, as a pale-yellow solid. The *trans/cis* ratio was determined to be 5:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.36 – 7.06 (m, 2H), 6.79 – 6.50 (m, 2H), 6.42 (d, *J* = 15.8 Hz, 1H), 5.82 (dd, *J* = 15.8, 8.9 Hz, 1H), 3.27 (td, *J* = 10.0, 4.3 Hz, 1H), 2.92 (s, 6H), 2.06 – 1.91 (m, 2H), 1.87 – 1.59 (m, 4H), 1.44 – 1.13 (m, 4H). ^{13}C

NMR (101 MHz, $CDCl_3$) δ 150.02, 131.95, 127.70, 127.12, 125.71, 112.60, 73.34, 50.74, 40.64, 33.76, 31.70, 25.38, 24.91. HRMS (APCI) calcd for C16H23NNaO (M+Na+): 268.1672; found: 268.1675.



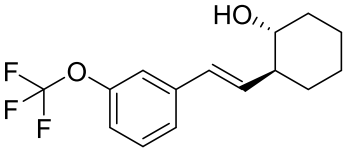
(E)-trans-2-(3-(trifluoromethyl)styryl)cyclohexan-1-ol

Following general procedure, as a pale-yellow solid. The *trans/cis* ratio was determined to be 4.2:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.55 – 7.34 (m, 4H), 6.54 (d, *J* = 15.9 Hz, 1H), 6.19 (dd, *J* = 15.9, 8.7 Hz, 1H), 3.37 (td, *J* = 9.9, 4.4 Hz, 1H), 2.17 – 1.99 (m, 2H), 1.93 – 1.65 (m, 4H), 1.36 – 1.11 (m, 4H). ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.74. ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.91, 134.44, 130.94 (q, *J* = 32.1 Hz), 130.36, 129.44, 128.22, 124.16 (q, *J* = 272.3 Hz), 123.83 (q, *J* = 3.8 Hz), 122.72 (q, *J* = 3.8 Hz), 73.30, 50.43, 34.19, 31.35, 25.13, 24.78. HRMS (APCI) calcd for C15H17F3NaO (M+Na+): 293.1124; found: 293.1128.



(E)-trans-2-(2-(benzo[d][1,3]dioxol-5-yl)vinyl)cyclohexan-1-ol

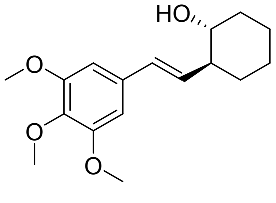
Following general procedure, as a pale-yellow solid. The *trans/cis* ratio was determined to be 5.6:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 6.92 (dd, *J* = 4.5, 1.5 Hz, 1H), 6.81 – 6.70 (m, 2H), 6.42 (d, *J* = 15.8 Hz, 1H), 5.97 – 5.84 (m, 3H), 3.31 (td, *J* = 10.0, 4.3 Hz, 1H), 2.10 – 1.90 (m, 3H), 1.83 – 1.64 (m, 3H), 1.37 – 1.13 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.00, 146.99, 131.61, 131.47, 130.39, 120.74, 108.26, 105.52, 101.04, 73.29, 50.47, 33.91, 31.52, 25.24, 24.83. HRMS (APCI) calcd for C15H18NaO3 (M+Na+): 269.1148; found: 269.1153.



(E)-trans-2-(3-(trifluoromethoxy)styryl)cyclohexan-1-ol

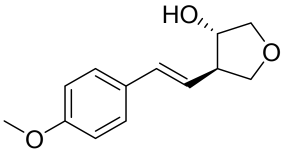
Following general procedure, as a pale-yellow solid. The *trans/cis* ratio was determined to be

5:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, CDCl$_3$) δ 7.30 (dd, *J* = 13.5, 7.9 Hz, 2H), 7.21 (s, 1H), 7.07 (d, *J* = 7.7 Hz, 1H), 6.50 (d, *J* = 15.9 Hz, 1H), 6.14 (dd, *J* = 15.9, 8.7 Hz, 1H), 3.37 (td, *J* = 9.9, 4.4 Hz, 1H), 2.13 – 2.00 (m, 2H), 1.78 (dd, *J* = 26.9, 11.1 Hz, 4H), 1.28 (dt, *J* = 12.1, 5.3 Hz, 4H). ^{19}F NMR (377 MHz, CDCl$_3$) δ -57.67. ^{13}C NMR (101 MHz, CDCl$_3$) δ 149.59, 139.29, 134.23, 130.44, 129.84, 129.63 (q, *J* = 284.3 Hz), 124.67, 119.59, 118.44, 73.27, 50.44, 34.13, 31.35, 25.14, 24.79. HRMS (APCI) calcd for C15H17F3NaO2 (M+Na+): 309.1073; found: 309.1079.



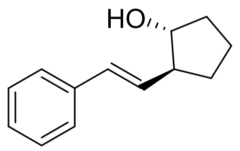
(E)-trans-2-(3,4,5-trimethoxystyryl)cyclohexan-1-ol

Following general procedure, as a pale-yellow solid. The *trans/cis* ratio was determined to be 6:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, CDCl$_3$) δ 6.60 (s, 2H), 6.44 (d, *J* = 15.8 Hz, 1H), 6.00 (dd, *J* = 15.8, 8.8 Hz, 1H), 3.88 (s, 6H), 3.84 (s, 3H), 3.35 (td, *J* = 9.9, 4.3 Hz, 1H), 2.12 – 2.01 (m, 2H), 1.86 – 1.68 (m, 4H), 1.38 – 1.21 (m, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 153.31, 137.61, 132.85, 131.77, 131.72, 103.17, 73.28, 60.96, 56.09, 50.50, 33.99, 31.50, 25.22, 24.83. (APCI) calcd for C17H24NaO4 (M+Na+): 315.1567; found: 315.1573.



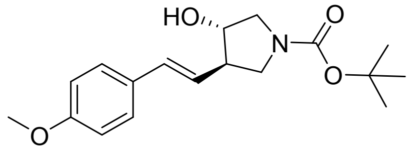
(E)-trans-4-(4-methoxystyryl)tetrahydrofuran-3-ol

Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 3.5:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, CDCl$_3$) δ 7.31 – 7.24 (m, 2H), 6.88 – 6.79 (m, 2H), 6.45 (d, *J* = 15.6 Hz, 1H), 5.93 (dd, *J* = 15.8, 8.7 Hz, 1H), 4.25 (dd, J = 8.6, 3.6 Hz, 1H), 4.16 (dd, J = 8.7, 7.0 Hz, 1H), 4.06 – 3.94 (m, 2H), 3.90 – 3.82 (m, 1H), 3.80 (s, 3H), 3.71 (ddd, J = 14.6, 9.2, 4.6 Hz, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 159.26, 159.16, 133.37, 131.34, 129.64, 127.43, 127.34, 125.74, 121.71, 114.03, 75.82, 74.34, 72.04, 70.56, 55.33, 52.25. HRMS (APCI) calcd for C13H16NaO3 (M+Na+): 243.0992; found: 243.0998.



(E)-trans-2-styrylcyclopentan-1-ol

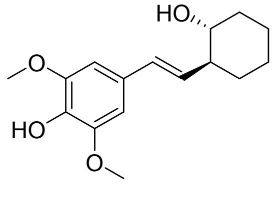
Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 7:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.38 – 7.33 (m, 2H), 7.29 (dd, J = 10.3, 4.8 Hz, 2H), 7.21 (dd, J = 5.6, 1.7 Hz, 1H), 6.46 (d, J = 15.8 Hz, 1H), 6.13 (dd, J = 15.8, 8.3 Hz, 1H), 4.04 – 3.86 (m, 1H), 2.57 – 2.41 (m, 1H), 2.12 – 1.91 (m, 2H), 1.79 – 1.44 (m, 5H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.38, 132.30, 130.48, 128.55, 127.16, 126.09, 78.67, 52.38, 33.61, 30.11, 21.29. HRMS (APCI) calcd for C13H16NaO (M+Na+): 211.1093; found: 211.1096.



trans-tert-butyl (E)-3-hydroxy-4-(4-methoxystyryl)pyrrolidine-1-carboxylate

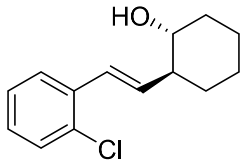
Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 4.5:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.31 – 7.24 (m, 2H), 6.88 – 6.80 (m, 1H), 6.45 (d, J = 15.6 Hz, 1H), 5.90 (dd, J = 15.9, 8.2 Hz, 1H), 4.18 – 4.06 (m, 1H), 3.80 (s, 3H), 3.75 – 3.61 (m, 2H), 3.54 – 3.17 (m, 4H), 2.89 – 2.74 (m, 1H), 1.47 (s, 9H). ^{13}C NMR (101 MHz, CDCl3) δ 159.20, 154.65, 131.76, 129.97, 129.53, 127.46, 127.40, 125.39, 114.00, 79.61, 75.15, 74.48, 73.82, 73.33, 72.62, 55.30, 52.24, 51.97, 50.01, 49.42, 28.52.

HRMS (APCI) calcd for C18H25NNaO4 (M+Na+): 342.1676; found: 342.1679.



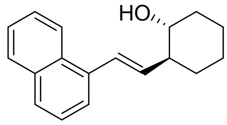
(E)-trans-4-(2-(2-hydroxycyclohexyl)vinyl)-2,6-dimethoxyphenol

Following general procedure, as a pale-yellow solid. The *trans/cis* ratio was determined to be 4:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.15 (s, 1H), 6.60 (s, 1H), 6.42 (d, J = 15.8 Hz, 1H), 5.93 (dd, J = 15.8, 8.8 Hz, 1H), 5.63 (s, 1H), 3.97 (s, 3H), 3.90 (s, 3H), 3.40 – 3.29 (m, 1H), 2.09 – 1.99 (m, 3H), 1.87 – 1.67 (m, 3H), 1.34 – 1.20 (m, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 147.39, 147.13, 134.46, 131.96, 130.14, 106.71, 102.98, 73.37, 56.47, 56.28, 50.51, 33.90, 31.57, 25.25, 24.84. HRMS (APCI) calcd for C16H22NaO4 (M+Na+): 301.1410; found: 301.1416.



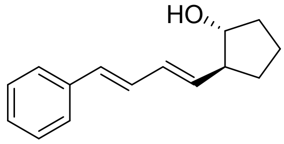
(E)-trans-2-(2-chlorostyryl)cyclohexan-1-ol

Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 4.5:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, CDCl$_3$) δ 7.53 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.44 – 7.30 (m, 2H), 7.24 – 7.13 (m, 1H), 6.90 (d, *J* = 15.9 Hz, 1H), 6.09 (dd, *J* = 15.9, 8.7 Hz, 1H), 3.36 (td, *J* = 9.9, 4.5 Hz, 1H), 2.20 – 2.05 (m, 3H), 1.92 – 1.79 (m, 3H), 1.39 – 1.24 (m, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 139.23, 135.25, 132.81, 129.69, 128.37, 128.04, 126.80, 126.71, 73.21, 50.57, 34.03, 31.35, 25.15, 24.81. HRMS (APCI) calcd for C14H17ClNaO (M+Na+): 259.0860; found: 259.0861.



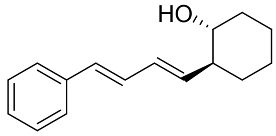
(E)-trans-2-(2-(naphthalen-1-yl)vinyl)cyclohexan-1-ol

Following general procedure, as a pale-yellow solid. The *trans/cis* ratio was determined to be 7:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, CDCl$_3$) δ 8.15 – 8.10 (m, 1H), 7.90 – 7.80 (m, 1H), 7.76 (d, J = 8.2 Hz, 1H), 7.60 (m, 1H), 7.54 – 7.46 (m, 2H), 7.44 (dd, J = 13.3, 5.7 Hz, 1H), 7.28 (d, J = 15.6 Hz, 1H), 6.10 (dd, J = 15.6, 8.8 Hz, 1H), 3.40 (td, J = 9.6, 4.6 Hz, 1H), 2.20 (dt, J = 9.5, 6.7 Hz, 1H), 2.15 – 1.97 (m, 2H), 1.95 – 1.66 (m, 3H), 1.46 – 1.29 (m, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 135.55, 134.85, 133.65, 131.06, 129.15, 128.56, 127.80, 126.05, 125.78, 125.62, 123.77, 123.67, 73.29, 50.94, 33.97, 31.52, 25.26, 24.87. HRMS (APCI) calcd for C18H20NaO (M+Na+): 275.1406; found: 275.1408.



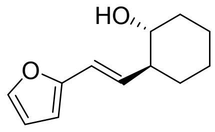
trans-2-((1E,3E)-4-phenylbuta-1,3-dien-1-yl)cyclopentan-1-ol

Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 6.1:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.43 – 7.17 (m, 5H), 6.76 (dd, *J* = 15.7, 10.4 Hz, 1H), 6.48 (d, *J* = 15.7 Hz, 1H), 6.30 (dd, *J* = 15.1, 10.4 Hz, 1H), 5.74 (dd, *J* = 15.1, 8.3 Hz, 1H), 3.92 (q, *J* = 6.9 Hz, 1H), 2.51 – 2.34 (m, 1H), 2.10 – 1.94 (m, 2H), 1.84 – 1.58 (m, 4H), 1.52 – 1.41 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.48, 136.87, 131.13, 130.96, 128.98, 128.59, 127.29, 126.22, 78.78, 52.21, 33.64, 30.11, 21.27. HRMS (APCI) calcd for C15H18NaO (M+Na+): 237.1250; found: 237.1253.



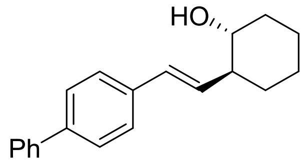
trans-2-((1E,3E)-4-phenylbuta-1,3-dien-1-yl)cyclohexan-1-ol

Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 5:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.4 Hz, 2H), 7.34 – 7.28 (m, 2H), 7.21 (t, *J* = 7.2 Hz, 1H), 6.77 (dd, *J* = 15.7, 10.4 Hz, 1H), 6.50 (d, *J* = 15.7 Hz, 1H), 6.35 (dd, *J* = 15.1, 10.4 Hz, 1H), 5.69 (dd, *J* = 15.2, 8.9 Hz, 1H), 3.30 (td, *J* = 9.8, 4.5 Hz, 1H), 2.10 – 1.94 (m, 1H), 1.83 – 1.66 (m, 4H), 1.38 – 1.18 (m, 5H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.34, 136.71, 132.49, 131.49, 128.76, 128.62, 127.42, 126.27, 73.38, 50.36, 33.96, 31.42, 25.19, 24.81. HRMS (APCI) calcd for C16H20NaO (M+Na+): 251.1406; found: 251.1411.



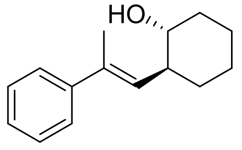
(E)-trans-2-(2-(furan-2-yl)vinyl)cyclohexan-1-ol

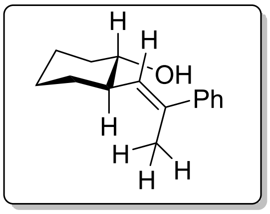
Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 4:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.32 (d, *J* = 1.8 Hz, 1H), 6.38 – 6.30 (m, 2H), 6.19 (d, *J* = 3.3 Hz, 1H), 6.02 (dd, *J* = 15.9, 8.9 Hz, 1H), 3.38 – 3.27 (m, 1H), 2.09 – 1.95 (m, 2H), 1.84 – 1.67 (m, 4H), 1.34 – 1.23 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.63, 141.59, 131.02, 120.38, 111.23, 107.13, 73.32, 50.36, 33.93, 31.41, 25.17, 24.79. HRMS (APCI) calcd for C12H16NaO2 (M+Na+): 215.1043; found 215.1049.



(E)-trans-2-(2-([1,1'-biphenyl]-4-yl)vinyl)cyclohexan-1-ol

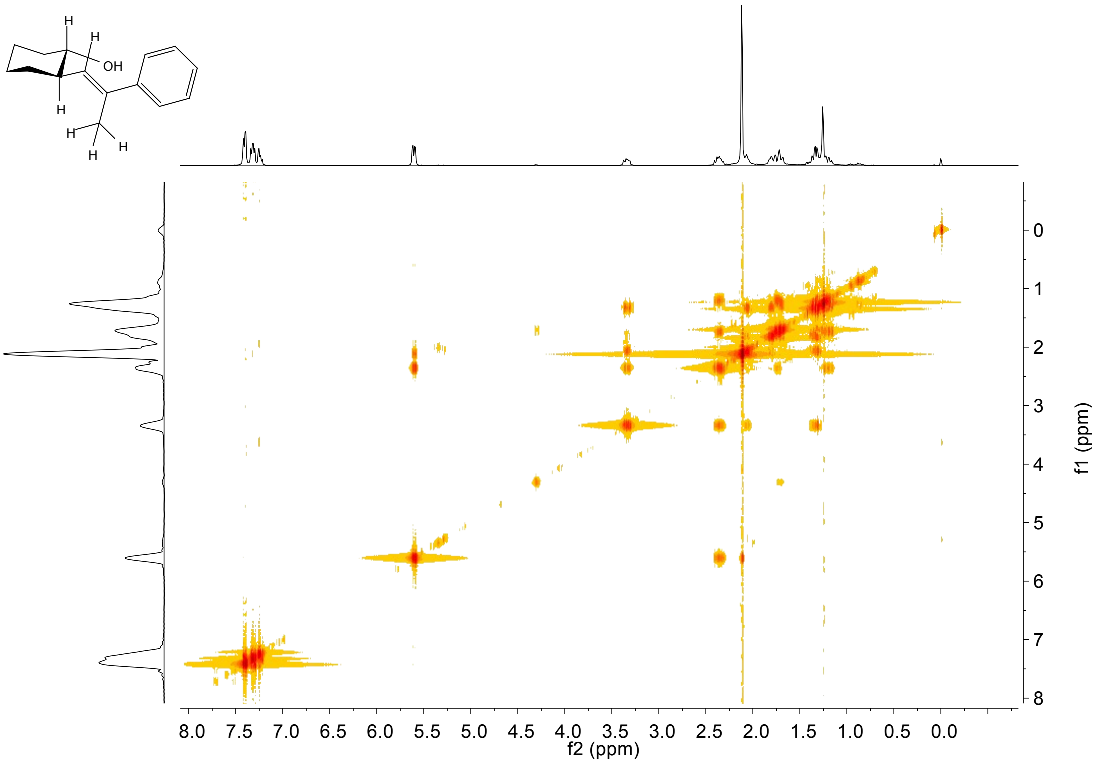
Following general procedure, as a pale-yellow solid. The *trans/cis* ratio was determined to be 6:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, CDCl$_3$) δ 7.56 – 7.46 (m, 4H), 7.41 – 7.31 (m, 4H), 7.27 (t, *J* = 7.4 Hz, 1H), 6.50 (d, *J* = 15.9 Hz, 1H), 6.05 (dd, *J* = 15.9, 8.8 Hz, 1H), 3.29 (td, *J* = 9.9, 4.3 Hz, 1H), 2.02 (dd, *J* = 6.9, 4.0 Hz, 2H), 1.90 – 1.61 (m, 4H), 1.33 – 1.16 (m, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 140.73, 140.17, 136.10, 132.33, 131.56, 128.80, 127.30, 127.28, 126.94, 126.62, 73.29, 50.70, 33.90, 31.47, 25.23, 24.84. HRMS (APCI) calcd for C20H22NaO (M+Na+): 301.1563; found: 301.1567.

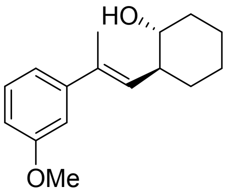




(E)-2-(2-phenylprop-1-en-1-yl)cyclohexan-1-ol

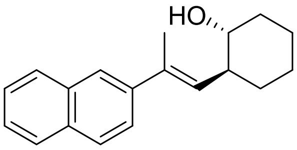
Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 4.3:1 by ^{1}H NMR. The assignment of onfiguration in the major isomer was based on the coupling constant at 2.36 ppm typical of an axial proton in a cyclohexane chair system and H-H COSY spectra. ^{1}H NMR of the *trans* isomer (400 MHz, CDCl$_3$) δ 7.41 (d, *J* = 7.9 Hz, 2H), 7.32 (t, *J* = 7.5 Hz, 2H), 7.25 (d, *J* = 6.3 Hz, 1H), 5.61 (d, *J* = 9.6 Hz, 1H), 3.34 (td, *J* = 9.6, 4.2 Hz, 1H), 2.36 (dtd, J = 11.1, 9.6, 3.7 Hz, 1H), 2.12 (d, *J* = 1.3 Hz, 3H), 2.06 (d, *J* = 3.9 Hz, 1H), 1.86 – 1.65 (m, 4H), 1.40 – 1.14 (m, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 143.37, 137.73, 130.32, 128.26, 126.99, 125.71, 74.42, 46.41, 33.71, 31.31, 25.27, 24.86, 16.51. HRMS (APCI) calcd for C15H20NaO (M+Na+): 239.1406; found: 239.1410.





(E)-2-(2-(3-methoxyphenyl)prop-1-en-1-yl)cyclohexan-1-ol

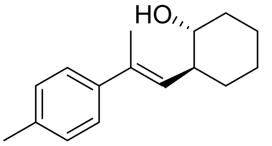
Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 5:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.6 Hz, 1H), 7.00 (ddd, *J* = 7.7, 1.7, 0.9 Hz, 1H), 6.95 – 6.92 (m, 1H), 6.80 (ddd, *J* = 8.2, 2.6, 0.8 Hz, 1H), 5.61 (dd, *J* = 9.7, 1.3 Hz, 1H), 3.82 (s, 3H), 3.41 – 3.27 (m, 1H), 2.40 – 2.29 (m, 1H), 2.10 (d, *J* = 1.3 Hz, 3H), 2.08 – 2.05 (m, 1H), 1.85 – 1.64 (m, 4H), 1.39 – 1.18 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.56, 144.99, 137.55, 130.54, 129.18, 118.30, 112.10, 111.82, 74.38, 55.28, 46.38, 33.74, 31.28, 25.25, 24.85, 16.58. HRMS (APCI) calcd for C16H22NaO2 (M+Na+): 269.1512; found: 269.1519.



(E)-2-(2-(naphthalen-2-yl)prop-1-en-1-yl)cyclohexan-1-ol

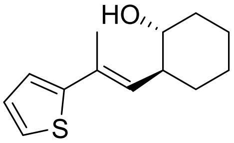
Following general procedure, as a pale-yellow solid. The *trans/cis* ratio was determined to be

4:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.85 – 7.73 (m, 4H), 7.60 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.51 – 7.38 (m, 2H), 5.77 (dd, *J* = 9.6, 1.3 Hz, 1H), 3.39 (td, *J* = 9.9, 4.4 Hz, 1H), 2.50 – 2.35 (m, 1H), 2.23 (d, *J* = 1.3 Hz, 3H), 2.10 (dd, *J* = 8.6, 3.8 Hz, 1H), 1.90 – 1.67 (m, 4H), 1.42 – 1.26 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.52, 137.51, 133.45, 132.57, 130.99, 128.08, 127.69, 127.51, 126.14, 125.65, 124.27, 124.24, 74.47, 46.55, 33.82, 31.37, 25.29, 24.88, 16.55. HRMS (APCI) calcd for C19H22NaO (M+Na+): 289.1563; found: 289.1571.



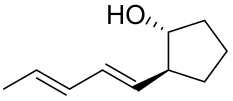
(E)-2-(2-(p-tolyl)prop-1-en-1-yl)cyclohexan-1-ol

Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 4:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.34 – 7.28 (m, 2H), 7.13 (d, *J* = 7.9 Hz, 2H), 5.57 (dd, *J* = 9.7, 1.3 Hz, 1H), 3.33 (td, *J* = 9.8, 4.3 Hz, 1H), 2.39 – 2.30 (m, 4H), 2.10 (d, *J* = 1.3 Hz, 3H), 2.08 – 2.05 (m, 1H), 1.84 – 1.67 (m, 4H), 1.36 – 1.22 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.50, 137.58, 136.71, 129.46, 128.92, 125.56, 74.43, 46.39, 33.67, 31.34, 25.29, 24.86, 21.03, 16.50. HRMS (APCI) calcd for C16H22NaO (M+Na+): 253.1563; found: 253.1566.



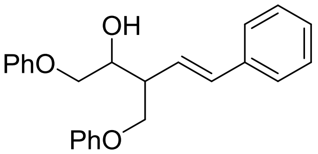
(E)-2-(2-(thiophen-2-yl)prop-1-en-1-yl)cyclohexan-1-ol

Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 5:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.12 (dd, *J* = 5.0, 1.2 Hz, 1H), 7.00 (dd, *J* = 3.6, 1.2 Hz, 1H), 6.98 – 6.96 (m, 1H), 5.74 (dd, *J* = 9.6, 1.3 Hz, 1H), 3.39 – 3.27 (m, 1H), 2.39 – 2.26 (m, 1H), 2.12 (d, *J* = 1.3 Hz, 3H), 2.08 – 2.05 (m, 1H), 1.81 – 1.67 (m, 4H), 1.35 – 1.20 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.42, 131.46, 128.94, 127.29, 123.32, 122.66, 74.22, 46.18, 33.74, 31.30, 25.20, 24.82, 16.46. HRMS (APCI) calcd for C13H18NaOS (M+Na+): 245.0971; found: 245.0976.



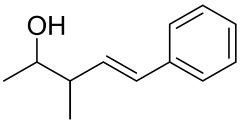
2-((1E,3E)-penta-1,3-dien-1-yl)cyclopentan-1-ol

Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 6.7:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 6.15 – 5.90 (m, 2H), 5.69 – 5.53 (m, 1H), 5.50 – 5.39 (m, 1H), 3.90 – 3.79 (m, 1H), 2.41 – 2.23 (m, 1H), 2.03 – 1.86 (m, 2H), 1.82 – 1.52 (m, 7H), 1.47 – 1.36 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 133.18, 131.36, 130.98, 127.94, 78.74, 51.95, 33.46, 30.06, 21.19, 18.02. HRMS (APCI) calcd for C10H16NaO (M+Na+): 175.1093; found: 175.1097.



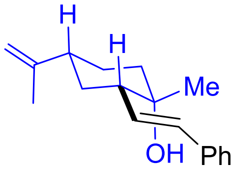
(E)-1-phenoxy-3-(phenoxymethyl)-5-phenylpent-4-en-2-ol

Following general procedure, as a pale-yellow solid. The *d.r.* ratio was determined to be 2.1:1 by ^{1}H NMR. ^{1}H NMR of the mixed products (400 MHz, $CDCl_3$) δ 7.40 – 7.26 (m, 8H), 7.01 – 6.87 (m, 7H), 6.60 (d, *J* = 15.9, 1H), 6.31 (dd, *J* = 15.9, 9.2 Hz, 1H), 4.55 – 4.24 (m, 4H), 4.16 – 4.08 (m, 1H), 4.06 – 3.98 (m, 1H), 3.03 – 2.92 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.76, 158.53, 140.17, 136.89, 133.41, 129.61, 129.55, 128.59, 127.64, 126.77, 126.34, 121.22, 121.00, 114.76, 114.68, 114.61, 70.56, 69.14, 68.54, 46.29. HRMS (APCI) calcd for C24H24NaO3 (M+Na+): 383.1618; found: 383.1613.



(E)-3-methyl-5-phenylpent-4-en-2-ol

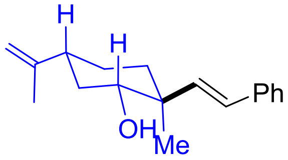
Following general procedure, as a pale-yellow liquid. The *d.r.* ratio was determined to be 1.3:1 by ^{1}H NMR. ^{1}H NMR of the mixted products (400 MHz, $CDCl_3$) δ 7.40 – 7.35 (m, 2H), 7.33 – 7.27 (m, 2H), 7.24 – 7.18 (m, 1H), 6.52 – 6.41 (m, 1H), 6.22 – 6.06 (m, 1H), 3.84 – 3.47 (m, 1H), 2.53 – 2.16 (m, 1H), 1.63 (br, 1H), 1.25 – 1.16 (m, 3H), 1.14 – 1.08 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.40, 137.17, 132.11, 131.64, 130.76, 128.56, 128.54, 127.33, 127.21, 126.17, 126.12, 71.35, 71.27, 45.46, 44.41, 20.34, 20.24, 16.58, 15.51. HRMS (APCI) calcd for C12H16NaO (M+Na+): 199.1093; found: 199.1099.



major

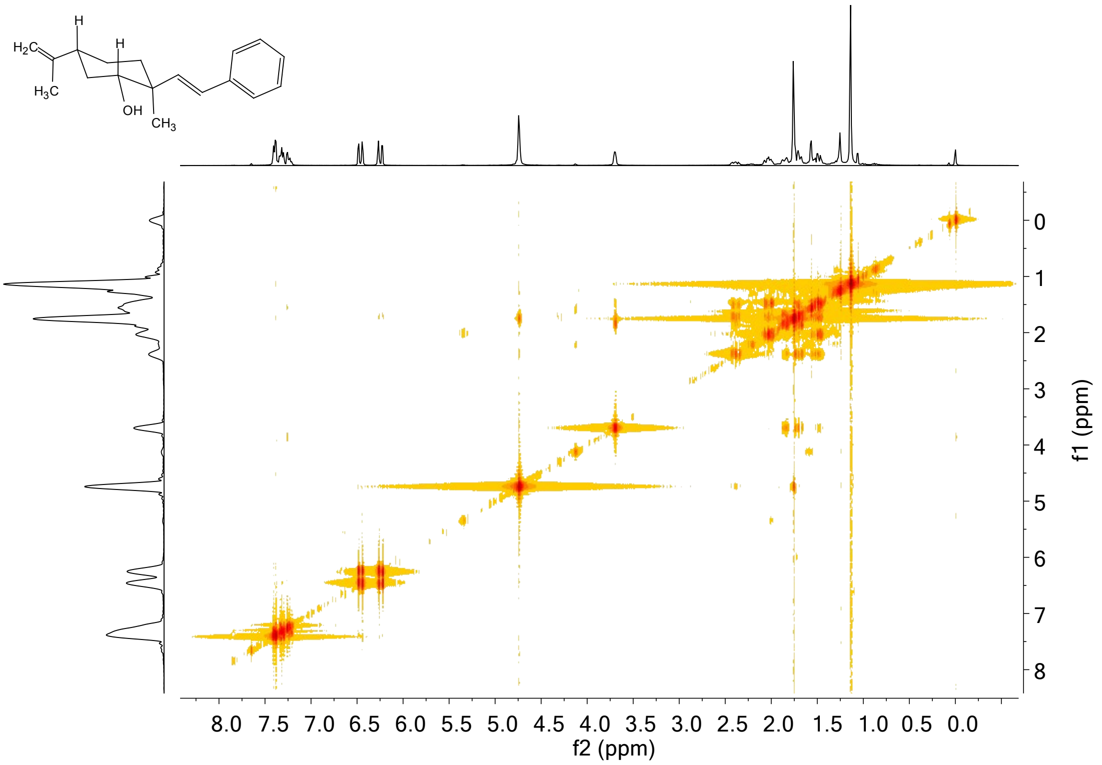
(1S,2S,4R)-1-methyl-4-(prop-1-en-2-yl)-2-((E)-styryl)cyclohexan-1-ol

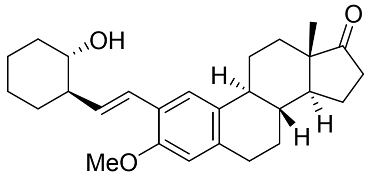
Following general procedure, as a pale-yellow liquid. The *cis/trans* ratio was determined to be 3.5:1 by ^{1}H NMR and GC. The assignment of onfiguration in the major isomer was based on ^{1}H NMR. NMR datas were the same as literature. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (dd, *J* = 8.3, 0.9 Hz, 2H), 7.33 – 7.27 (m, 2H), 7.22 (d, *J* = 7.3 Hz, 1H), 6.43 (d, *J* = 16.1 Hz, 1H), 6.34 (dd, *J* = 16.0, 8.1 Hz, 1H), 4.74 (dt, *J* = 1.8, 0.9 Hz, 1H), 4.72 – 4.70 (m, 1H), 2.22 – 2.14 (m, 1H), 2.10 – 1.96 (m, 1H), 1.81 (dt, *J* = 13.2, 2.9 Hz, 1H), 1.76 – 1.74 (m, 3H), 1.64 – 1.57 (m, 4H), 1.52 – 1.47 (m, 1H), 1.31 (br, 1H), 1.23 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.95, 137.55, 131.35, 131.00, 128.53, 127.13, 126.12, 108.62, 70.44, 50.29, 44.79, 39.87, 33.29, 29.52, 26.79, 20.94. HRMS (APCI) calcd for C18H24NaO (M+Na+): 279.1719; found: 279.1726.



(1R,2S,5R)-2-methyl-5-(prop-1-en-2-yl)-2-((E)-styryl)cyclohexan-1-ol

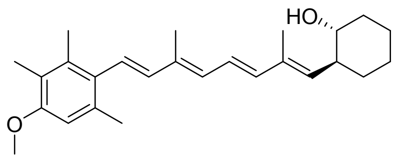
Following general procedure, as a pale-yellow liquid. The assignment of onfiguration in the major isomer was based on H-H COSY spectra. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (dt, *J* = 2.7, 1.8 Hz, 2H), 7.36 – 7.30 (m, 2H), 7.24 (dd, *J* = 5.0, 3.6 Hz, 1H), 6.46 (d, *J* = 16.5 Hz, 1H), 6.25 (d, *J* = 16.5 Hz, 1H), 4.75 – 4.68 (m, 2H), 3.70 (m, 1H), 2.44 – 2.32 (m, 1H), 2.07 – 1.96 (m, 1H), 1.89 – 1.81 (m, 1H), 1.76 (s, 3H), 1.72 – 1.69 (m, 1H), 1.65 – 1.59 (m, 2H), 1.54 – 1.45 (m, 2H), 1.14 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.02, 138.15, 137.18, 129.11, 128.62, 127.44, 126.20, 108.59, 73.28, 40.63, 37.75, 32.75, 29.50, 26.40, 23.16, 21.14. HRMS (APCI) calcd for C18H24NaO (M+Na+): 279.1719; found: 279.1721.





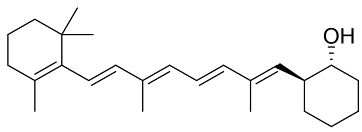
(8R,9S,13S,14S)-2-((E)-2-(2-hydroxycyclohexyl)vinyl)-3-methoxy-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one

Following general procedure, as a pale-yellow solid. The *trans/cis* ratio was determined to be 6.7:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 7.28 (s, 1H), 6.74 (dd, *J* = 16.0, 3.1 Hz, 1H), 6.51 (s, 1H), 5.92 (dd, *J* = 16.0, 8.9 Hz, 1H), 3.73 (s, 3H), 3.29 – 3.22 (m, 1H), 2.82 (dd, *J* = 9.8, 4.7 Hz, 2H), 2.49 – 2.34 (m, 2H), 2.17 (t, *J* = 8.4 Hz, 1H), 2.10 – 2.05 (m, 1H), 1.99 (ddd, *J* = 13.8, 7.8, 4.1 Hz, 6H), 1.72 (d, *J* = 9.7 Hz, 3H), 1.62 (d, *J* = 6.6 Hz, 1H), 1.47 (ddd, *J* = 19.9, 16.8, 7.1 Hz, 5H), 1.29 – 1.16 (m, 4H), 0.84 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 220.96, 154.62, 136.93, 136.91, 131.73, 131.64, 126.97, 126.92, 123.58, 123.55, 123.48, 123.36, 111.22, 73.16, 55.50, 51.13, 50.40, 48.03, 43.94, 38.42, 35.90, 33.74, 33.71, 31.58, 31.54, 29.69, 26.59, 26.03, 25.31, 24.88, 21.61, 13.87. HRMS (APCI) calcd for C27H36NaO3 (M+Na+): 431.2557; found: 431.2562.



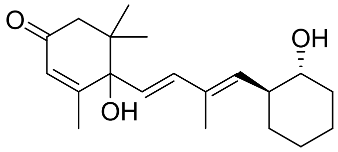
2-((1E,3E,5E,7E)-8-(4-methoxy-2,3,6-trimethylphenyl)-2,6-dimethylocta-1,3,5,7-tetraen-1-yl)cyclohexan-1-ol

This compound is unstable and easily decomposed. Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 4:1 by ^{1}H NMR. ^{1}H NMR of the 3.8:1 mixed products (400 MHz, $CDCl_3$) δ 6.76 – 6.48 (m, 4H), 6.38 – 5.99 (m, 2H), 5.44 – 5.01 (m, 1H), 3.73 (d, *J* = 1.4 Hz, 3H), 3.26 – 3.10 (m, 1H), 2.27 – 2.21 (m, 3H), 2.20 – 2.13 (m, 3H), 2.11 – 2.06 (m, 3H), 2.01 – 1.94 (m, 3H), 1.93 – 1.87 (m, 1H), 1.85 – 1.81 (m, 1H), 1.78 – 1.68 (m, 2H), 1.66 – 1.56 (m, 2H), 1.29 – 0.73 (m, 7H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.84, 156.01, 155.97, 138.62, 138.58, 137.62, 136.89, 136.76, 135.97, 135.94, 135.52, 135.30, 135.10, 133.92, 133.89, 133.28, 131.49, 131.18, 130.29, 130.23, 130.00, 126.66, 126.38, 126.30, 123.87, 122.68, 122.64, 109.93, 109.90, 74.33, 74.17, 55.55, 46.11, 46.07, 44.96, 33.73, 33.70, 31.70, 31.35, 26.94, 25.23, 25.20, 24.83, 21.46, 20.92, 20.68, 17.56, 17.49, 13.24, 12.81, 12.79, 11.90. HRMS (APCI) calcd for C26H36NaO2 (M+Na+): 403.2608; found: 403.2616.



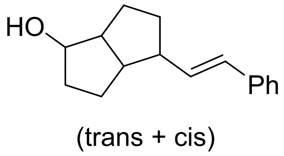
2-((1E,3E,5E,7E)-2,6-dimethyl-8-(2,6,6-trimethylcyclohex-1-en-1-yl)octa-1,3,5,7-tetraen-1-yl)cyclohexan-1-ol

This compound is unstable and easily decomposed. Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 2.9:1 by ^{1}H NMR. ^{1}H NMR of 3:1 mixed products (400 MHz, $CDCl_3$) δ 6.70 – 6.51 (m, 1H), 6.41 – 5.94 (m, 4H), 5.47 – 5.10 (m, 1H), 3.34 – 3.16 (m, 1H), 2.37 – 2.26 (m, 1H), 2.08 – 1.99 (m, 3H), 1.95 (m, 3H), 1.91 – 1.85 (m, 3H), 1.78 (d, *J* = 2.4 Hz, 1H), 1.73 – 1.58 (m, 7H), 1.51 – 1.44 (m, 3H), 1.37 – 1.23 (m, 3H), 1.14 (d, *J* = 10.5 Hz, 1H), 1.03 (d, *J* = 4.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.87, 137.75, 137.68, 137.01, 136.96, 136.26, 135.65, 134.65, 134.51, 134.26, 132.85, 130.23, 130.02, 129.40, 129.18, 128.74, 128.21, 126.76, 126.55, 126.37, 124.04, 122.90, 74.33, 74.16, 46.09, 44.93, 42.00, 39.63, 39.53, 34.27, 34.23, 33.68, 33.08, 33.04, 31.35, 29.00, 28.97, 27.03, 25.20, 24.82, 21.86, 21.75, 20.92, 20.74, 19.29, 13.25, 13.23, 12.75, 12.72. HRMS (APCI) calcd for C25H38NaO (M+Na+): 377.2815; found: 377.2823.



4-hydroxy-4-((1E,3E)-4-(2-hydroxycyclohexyl)-3-methylbuta-1,3-dien-1-yl)-3,5,5-trimethylcyclohex-2-en-1-one

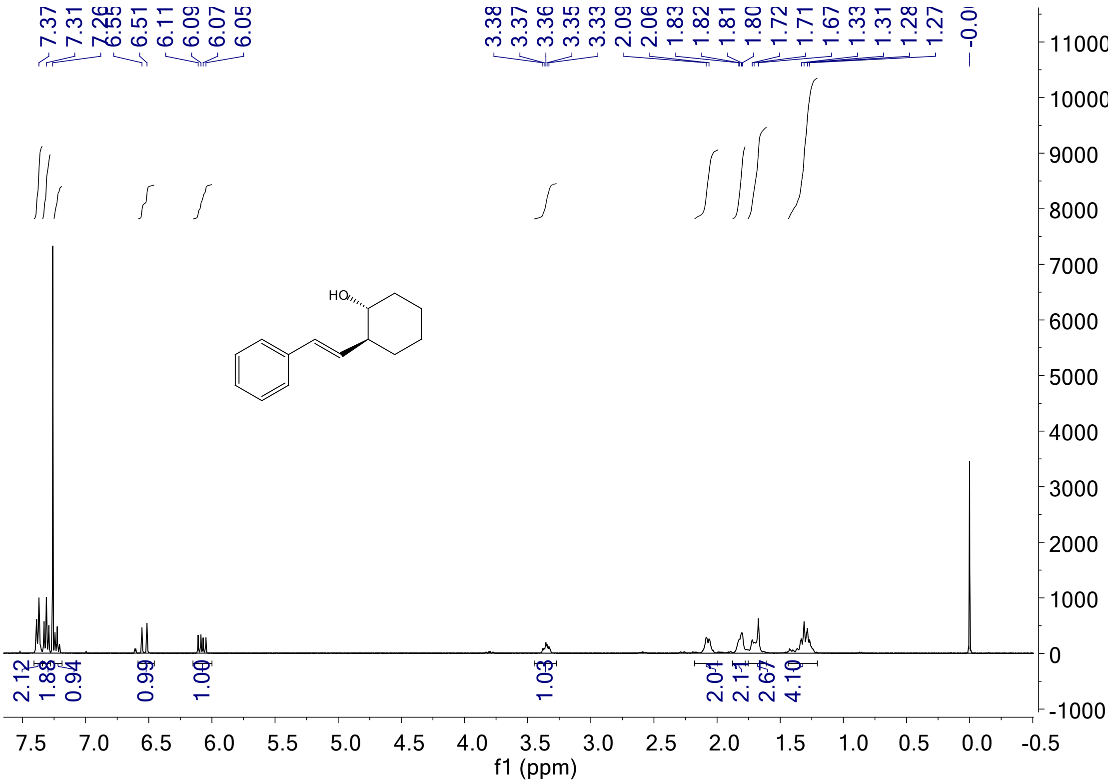
Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 4:1 by ^{1}H NMR. ^{1}H NMR of the *trans* isomer (400 MHz, $CDCl_3$) δ 6.30 (d, *J* = 15.7 Hz, 1H), 5.95 – 5.83 (m, 1H), 5.65 (d, *J* = 15.8 Hz, 1H), 5.37 (d, *J* = 9.5 Hz, 1H), 3.29 (td, *J* = 9.7, 4.5 Hz, 1H), 2.47 (d, *J* = 17.2 Hz, 1H), 2.31 – 2.19 (m, 2H), 2.03 – 1.98 (m, 1H), 1.92 (d, *J* = 1.3 Hz, 3H), 1.90 – 1.87 (m, 2H), 1.82 (d, *J* = 1.1 Hz, 3H), 1.70 – 1.64 (m, 4H), 1.30 – 1.26 (m, 3H), 1.10 (s, 3H), 1.03 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.27, 163.20, 136.39, 135.39, 134.76, 126.80, 126.77, 79.67, 74.28, 49.80, 45.89, 41.61, 33.96, 31.26, 25.33, 25.11, 24.76, 24.20, 23.00, 19.25, 13.33. HRMS (APCI) calcd for C20H31O3 (M+H+): 319.2268; found: 319.2273.

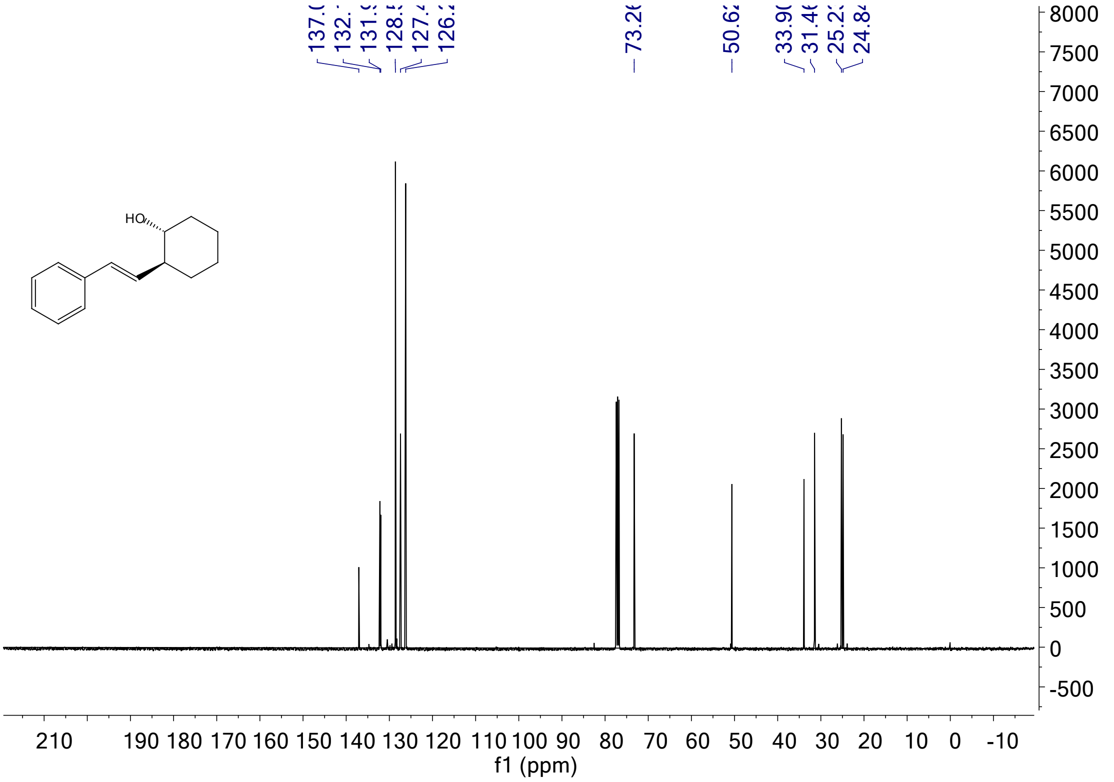


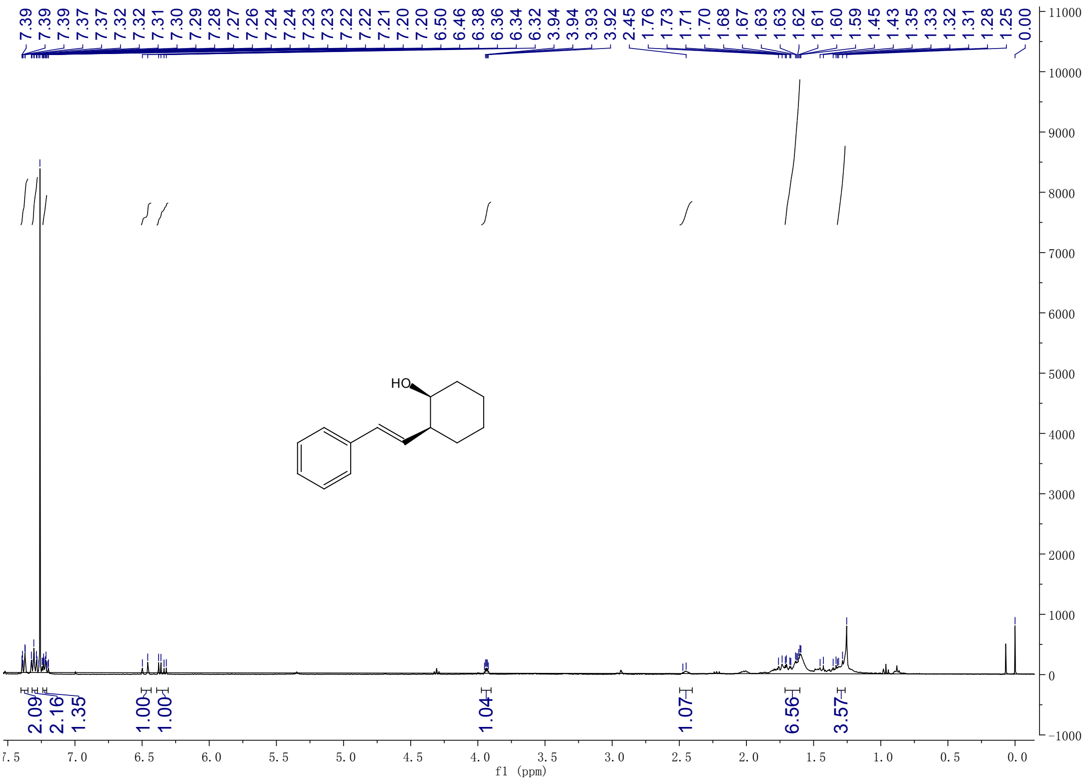
(E)-4-styryloctahydropentalen-1-ol

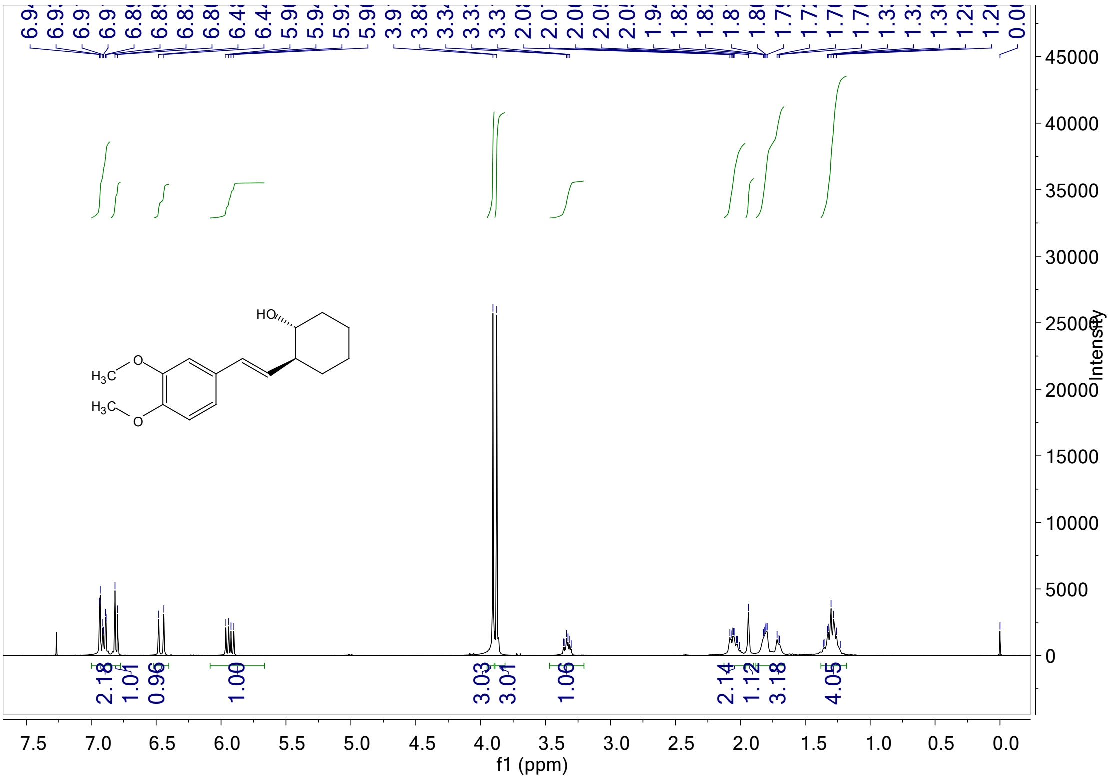
Following general procedure, as a pale-yellow liquid. The *trans/cis* ratio was determined to be 2.8:1 by ^{1}H NMR. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.33 (m, 2H), 7.32 – 7.27 (m, 2H), 7.22 – 7.16 (m, 1H), 6.37 (d, *J* = 15.8 Hz, 1H), 6.18 (dd, *J* = 15.8, 7.9 Hz, 1H), 4.03 – 3.88 (m, 1H), 2.43 – 2.34 (m, 2H), 2.13 – 1.93 (m, 3H), 1.85 – 1.75 (m, 2H), 1.69 – 1.62 (m, 1H), 1.52 – 1.44 (m, 3H), 1.20 – 1.11 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.73, 134.49, 128.49, 128.13, 126.86, 125.98, 79.63, 53.23, 51.33, 48.81, 34.72, 33.38, 30.96, 28.21. HRMS (APCI) calcd for C16H20NaO (M+Na+): 251.1406; found: 251.1411.

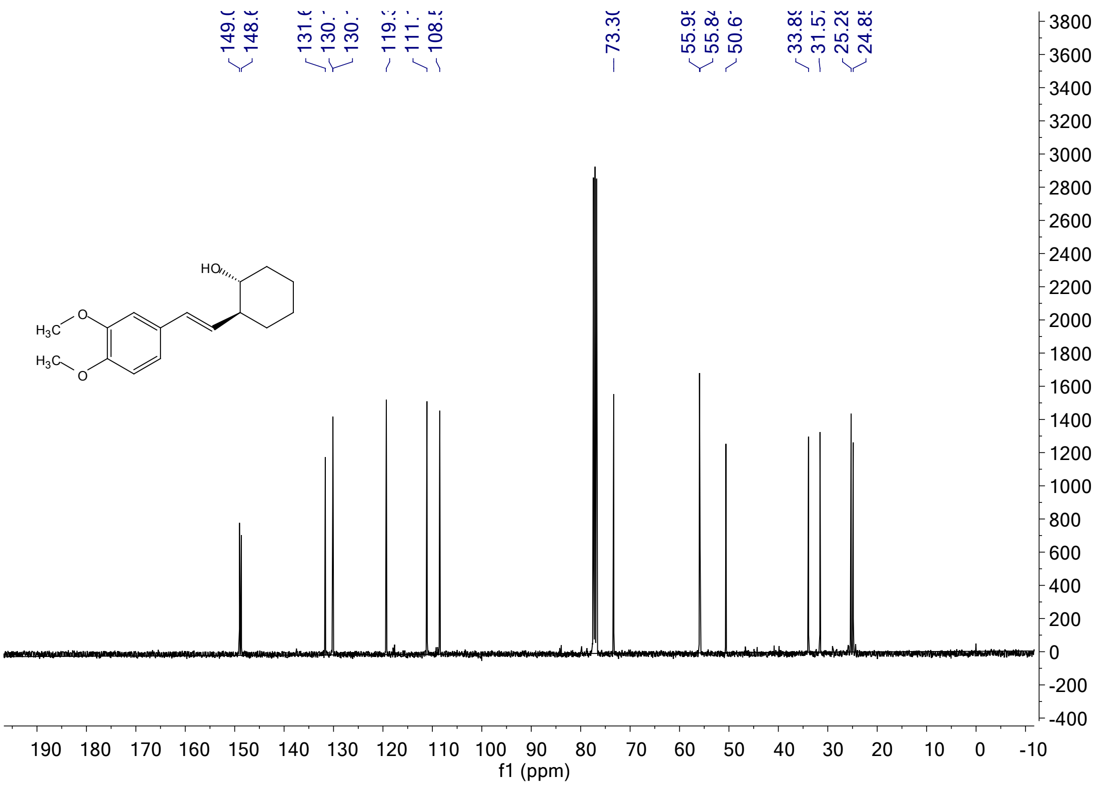
Ⅴ. NMR Spectra

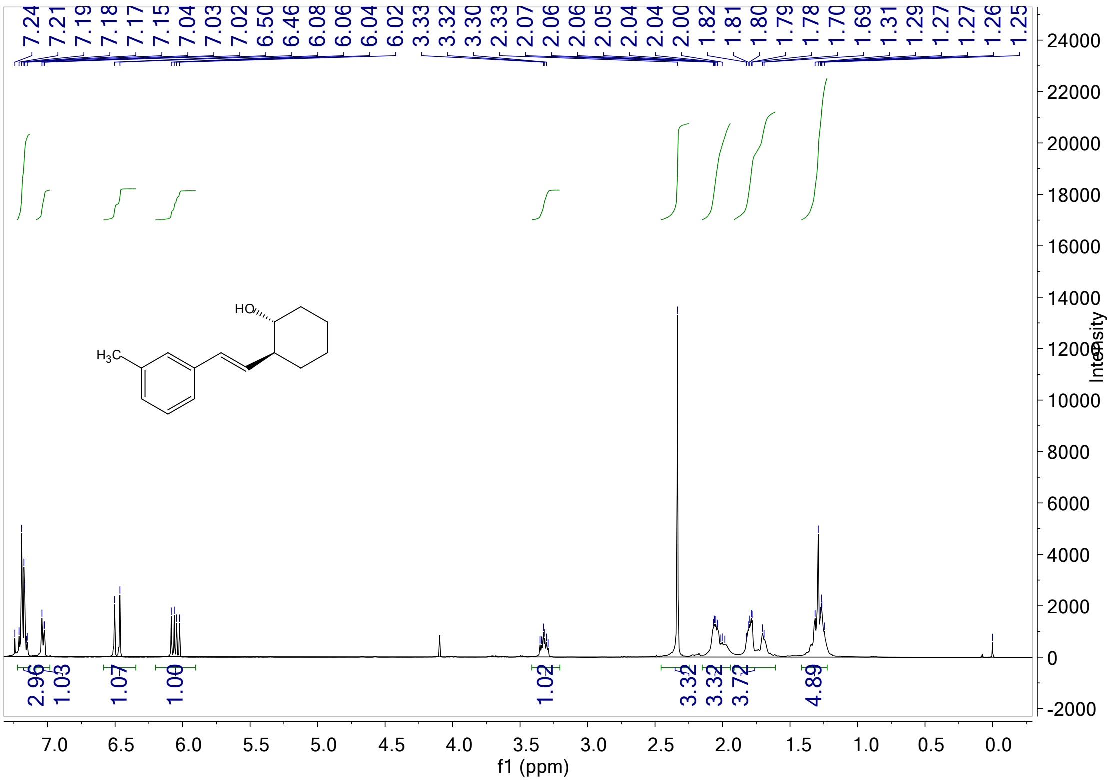


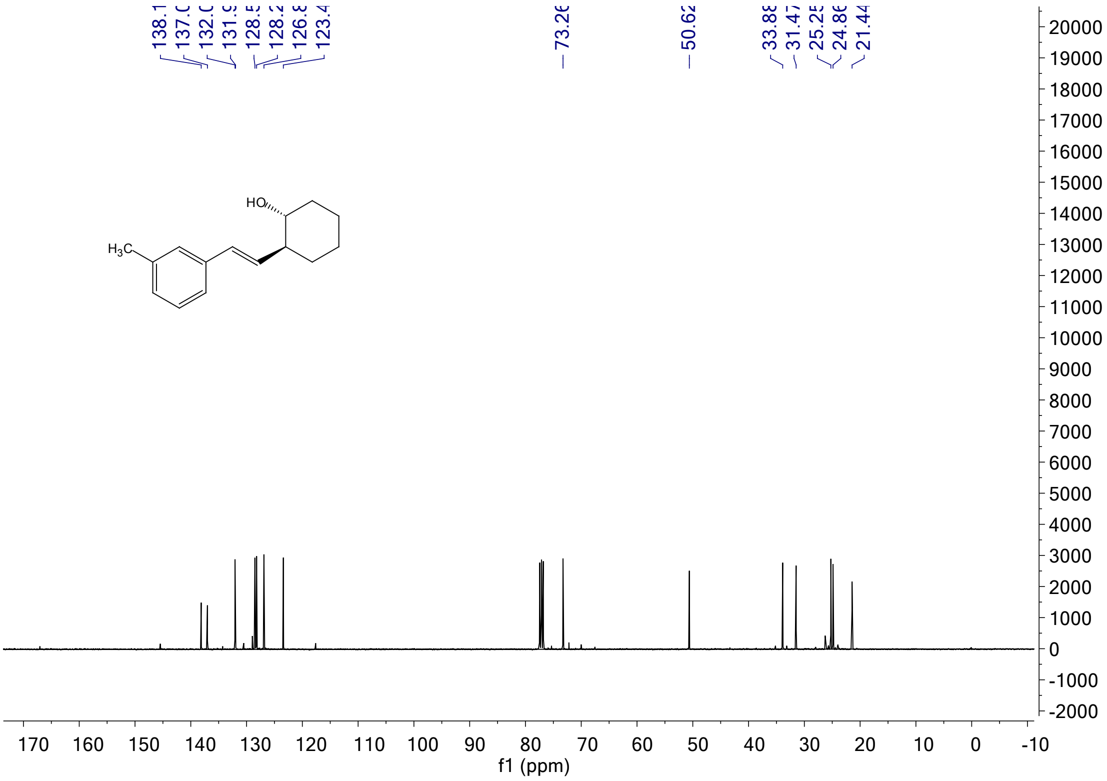


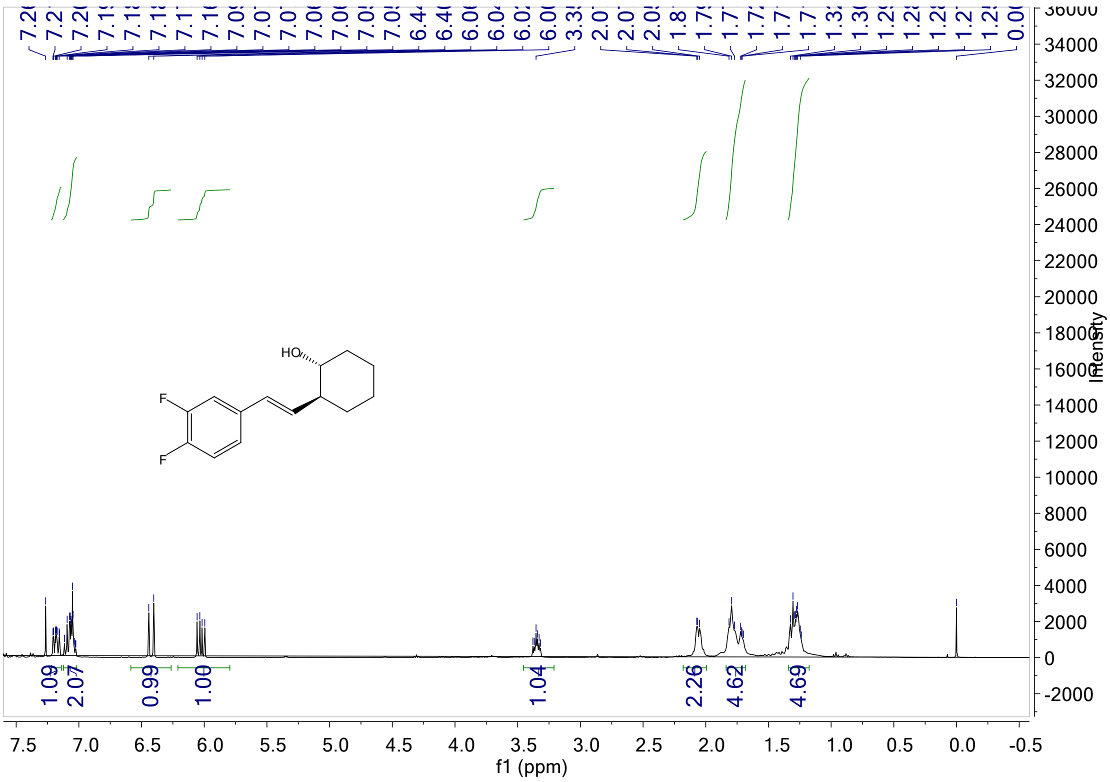

7.39
7.39
7.39
7.37
7.32
7.32
7.31
7.30
7.29
7.28
7.27
7.26
7.24
7.24
7.23
7.23
7.22
7.22
7.21
7.20
7.20
6.50
6.46
6.38
6.36
6.34
6.32
3.94
3.94
3.93
3.92
2.45
1.76
1.73
1.71
1.70
1.68
1.67
1.63
1.63
1.62
1.61
1.60
1.59
1.45
1.43
1.35
1.33
1.32
1.31
1.28
1.25
0.00
HO
2.09
2.16
1.35
1.00
1.00
1.04
1.07
6.56
3.57
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

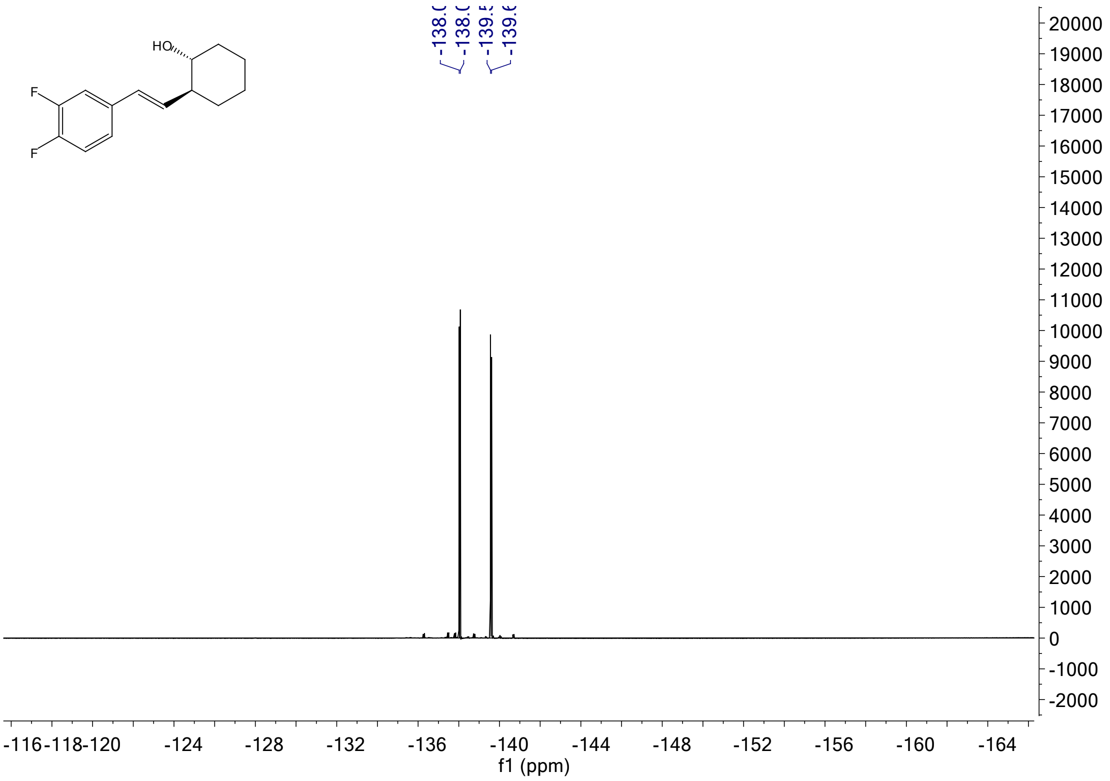

6.94
6.9
6.9
6.9
6.89
6.89
6.82
6.80
6.48
6.4
5.96
5.94
5.92
5.90
3.9
3.88
3.34
3.3
3.3
2.08
2.07
2.06
2.0
2.0
1.94
1.82
1.82
1.8
1.80
1.79
1.72
1.70
1.70
1.3
1.32
1.30
1.28
1.20
0.00
HO
H_3C
O
H_3C
O
2.18
1.01
0.96
1.00
3.03
3.01
1.06
2.14
1.12
3.18
4.05
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
Intensity
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

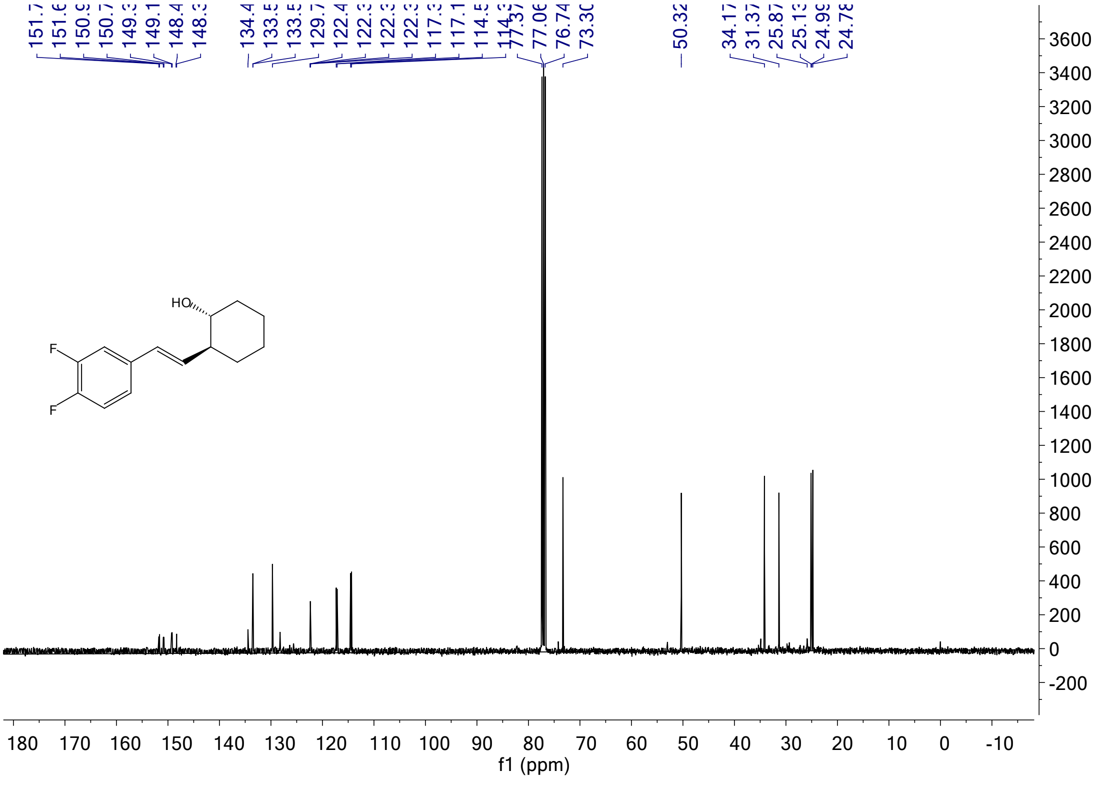

149.0
148.6
131.6
130.
130.
119.3
111.
108.5
73.30
55.95
55.84
50.6
33.89
31.57
25.28
24.85
HO
H3C
O
H3C
O
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
-400
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

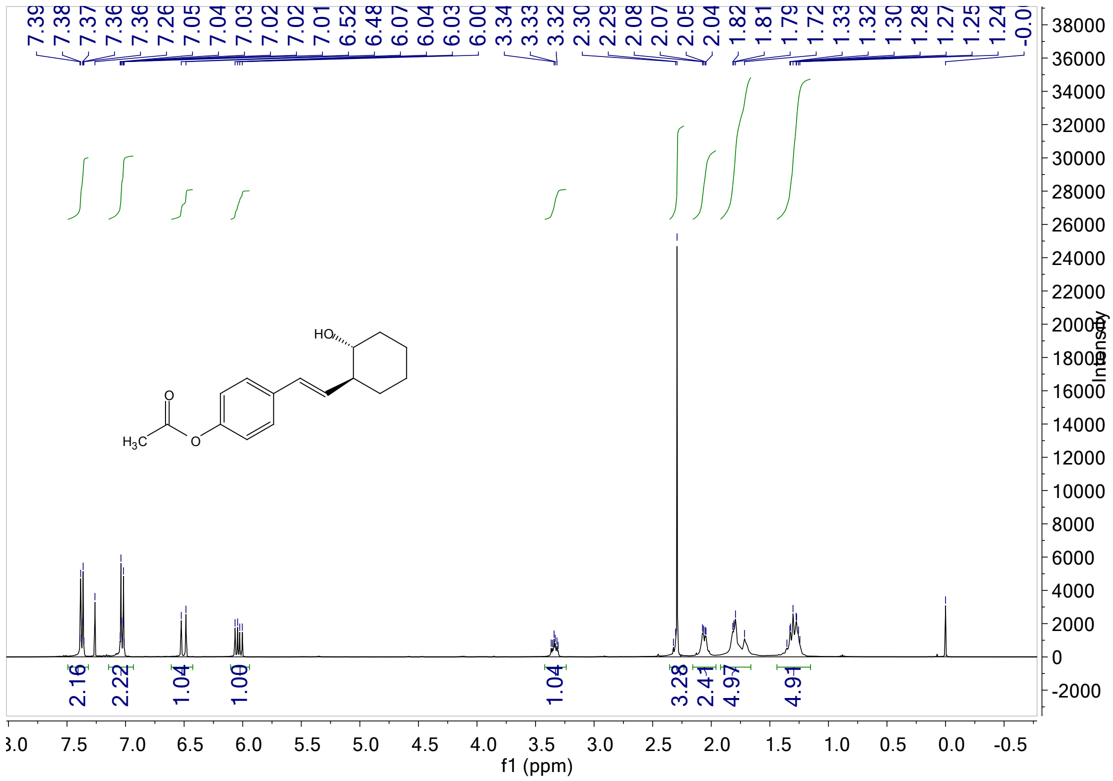

7.24
7.21
7.19
7.18
7.17
7.15
7.04
7.03
7.02
6.50
6.46
6.08
6.06
6.04
6.02
3.33
3.32
3.30
2.33
2.07
2.06
2.06
2.05
2.04
2.04
2.00
1.82
1.81
1.80
1.79
1.78
1.70
1.69
1.31
1.29
1.27
1.27
1.26
1.25
HO
H3C
2.96
1.03
1.07
1.00
1.02
3.32
3.32
3.72
4.89
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
Intensity
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

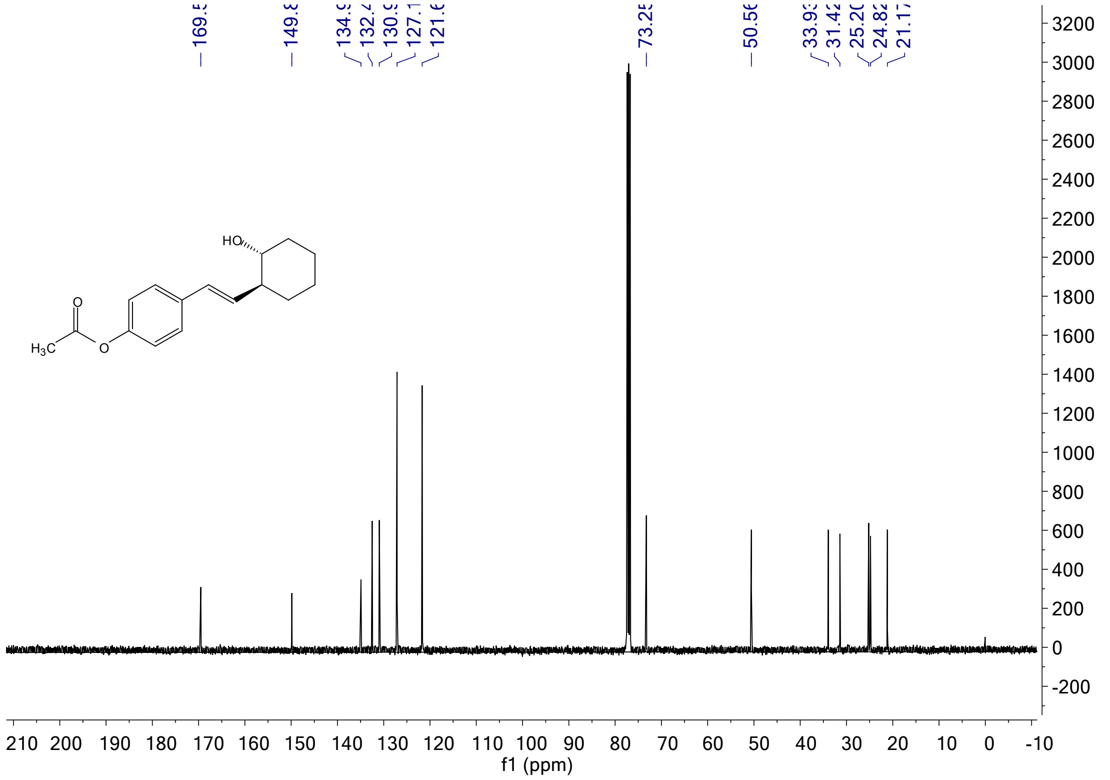

138.1
137.0
132.0
131.9
128.5
128.2
126.8
123.4
73.26
50.62
33.88
31.47
25.25
24.86
21.44
HO
H_3C
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

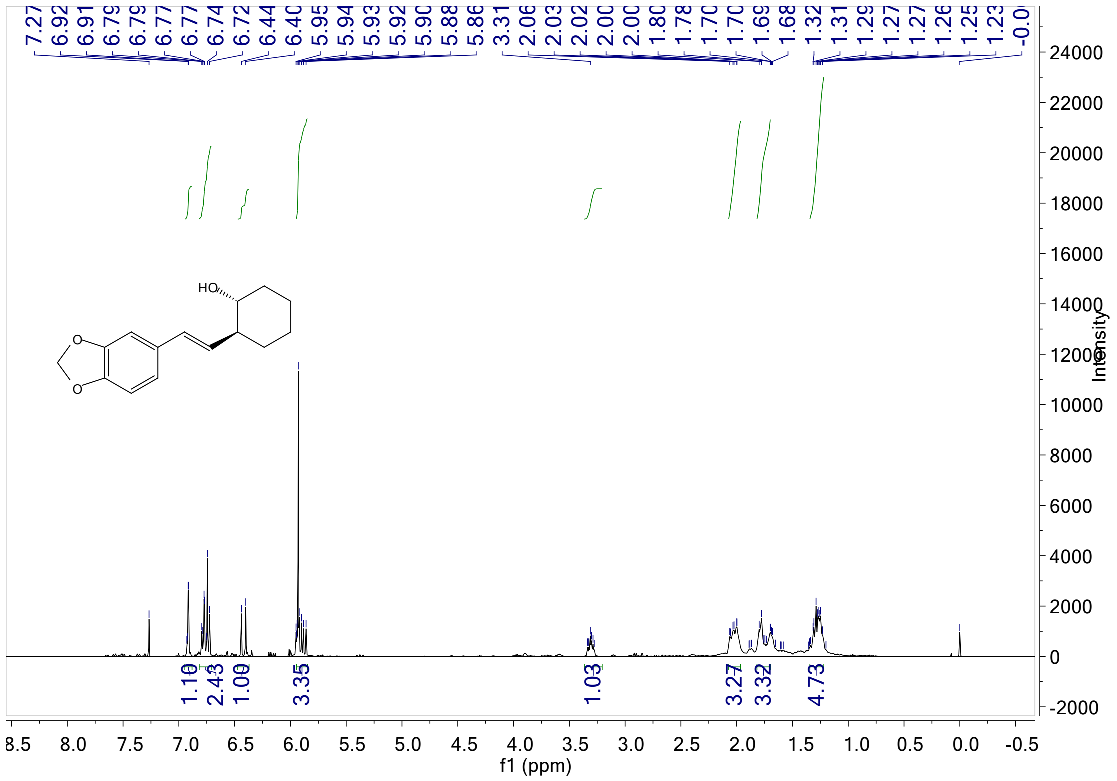

7.2
7.2
7.2
7.1
7.1
7.1
7.1
7.1
7.0
7.0
7.0
7.0
7.0
7.0
7.0
6.4
6.4
6.0
6.0
6.0
6.0
3.3
2.0
2.0
2.0
1.8
1.7
1.7
1.7
1.7
1.7
1.3
1.3
1.2
1.2
1.2
1.2
1.2
0.0
HO
F
F
1.09
2.07
0.99
1.00
1.04
2.26
4.62
4.69
36000
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
Intensity
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

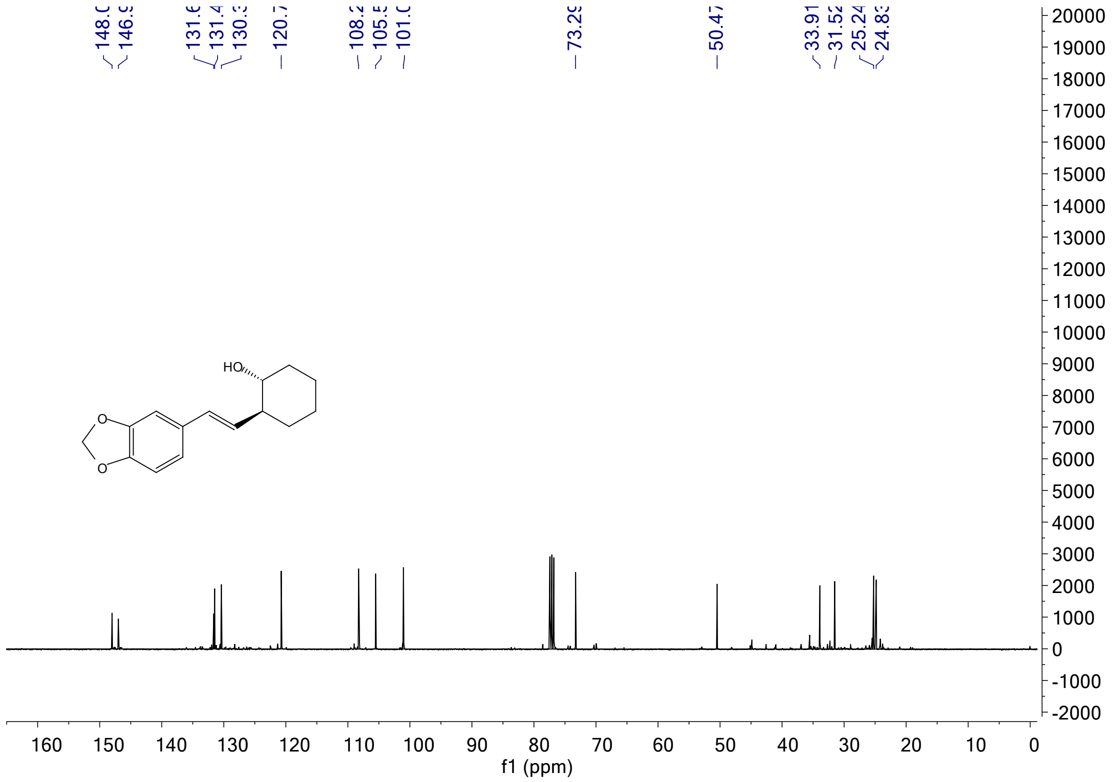

-138.
-138.
-139.
-139.
HO
F
F
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
-116 -118 -120 -124 -128 -132 -136 -140 -144 -148 -152 -156 -160 -164
f1 (ppm)

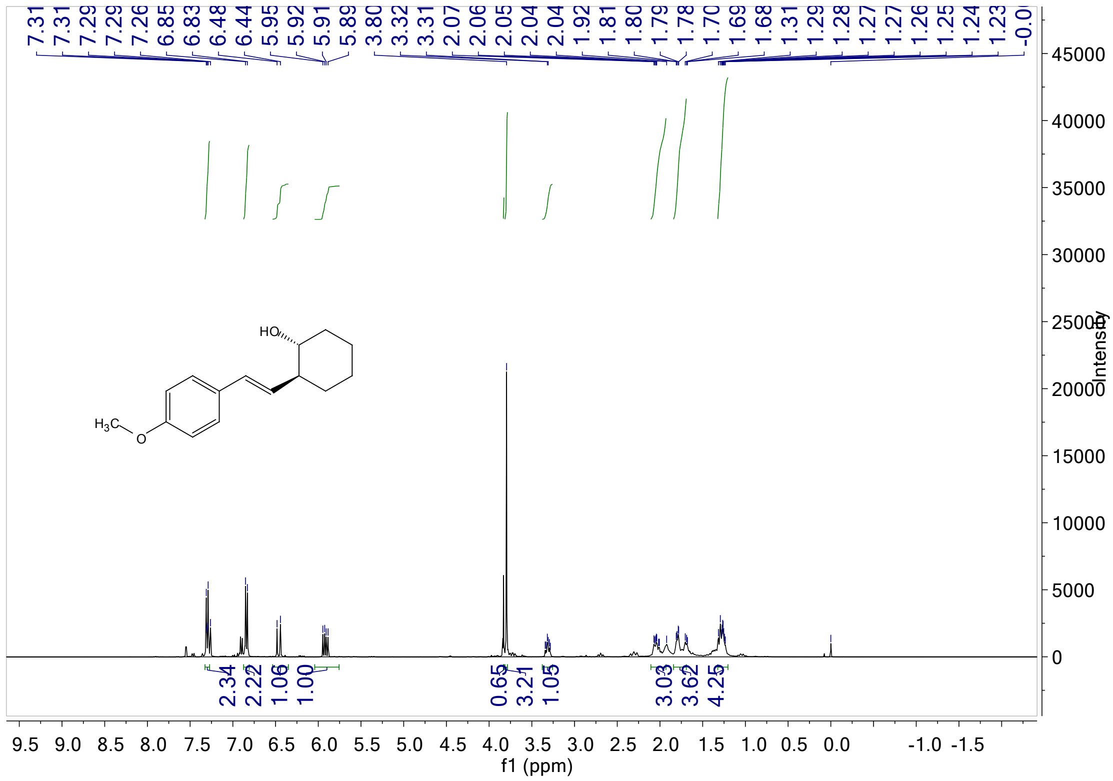

151.7
151.6
150.9
150.7
149.3
149.1
148.4
148.3
134.4
133.5
133.5
129.7
122.4
122.3
122.3
122.3
117.3
117.1
114.5
114.3
77.37
77.06
76.74
73.30
50.32
34.17
31.37
25.87
25.13
24.99
24.78
HO
F
F
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

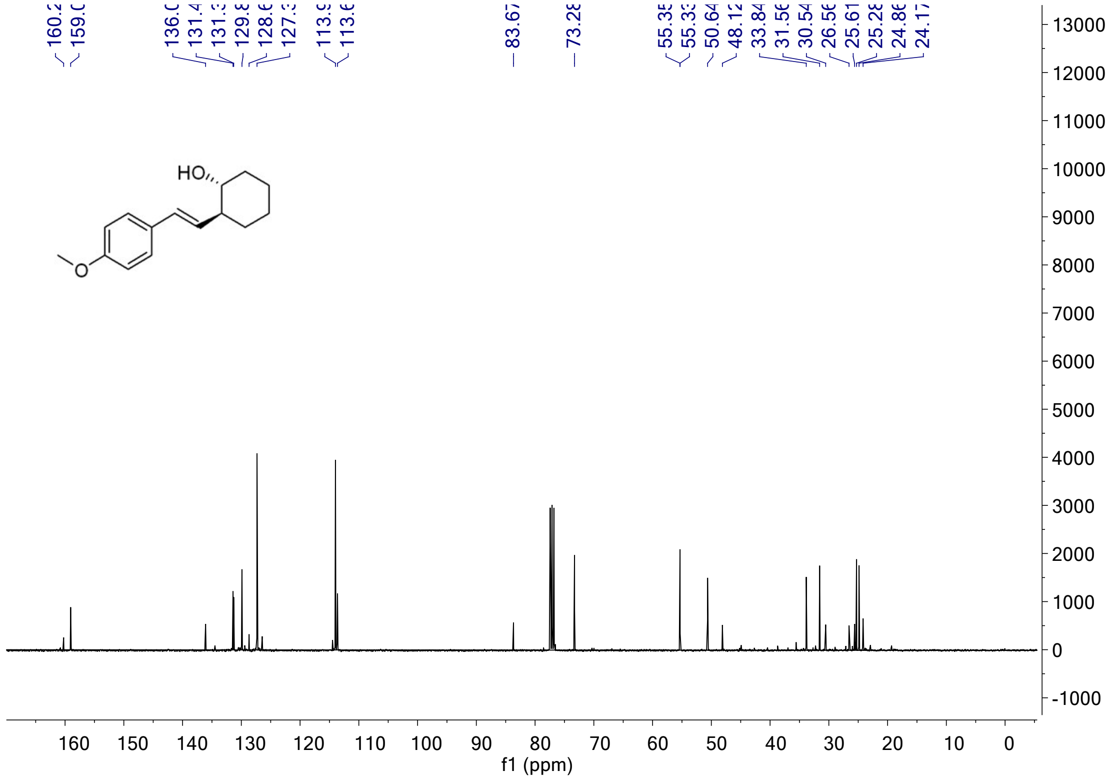

7.39
7.38
7.37
7.36
7.36
7.26
7.05
7.04
7.03
7.02
7.02
7.01
6.52
6.48
6.07
6.04
6.03
6.00
3.34
3.33
3.32
2.30
2.29
2.08
2.07
2.05
2.04
1.82
1.81
1.79
1.72
1.33
1.32
1.30
1.28
1.27
1.25
1.24
-0.0
2.16
2.22
1.04
1.00
1.04
3.28
2.41
4.97
4.91
HO
H3C
O
O
f1 (ppm)
Intensity

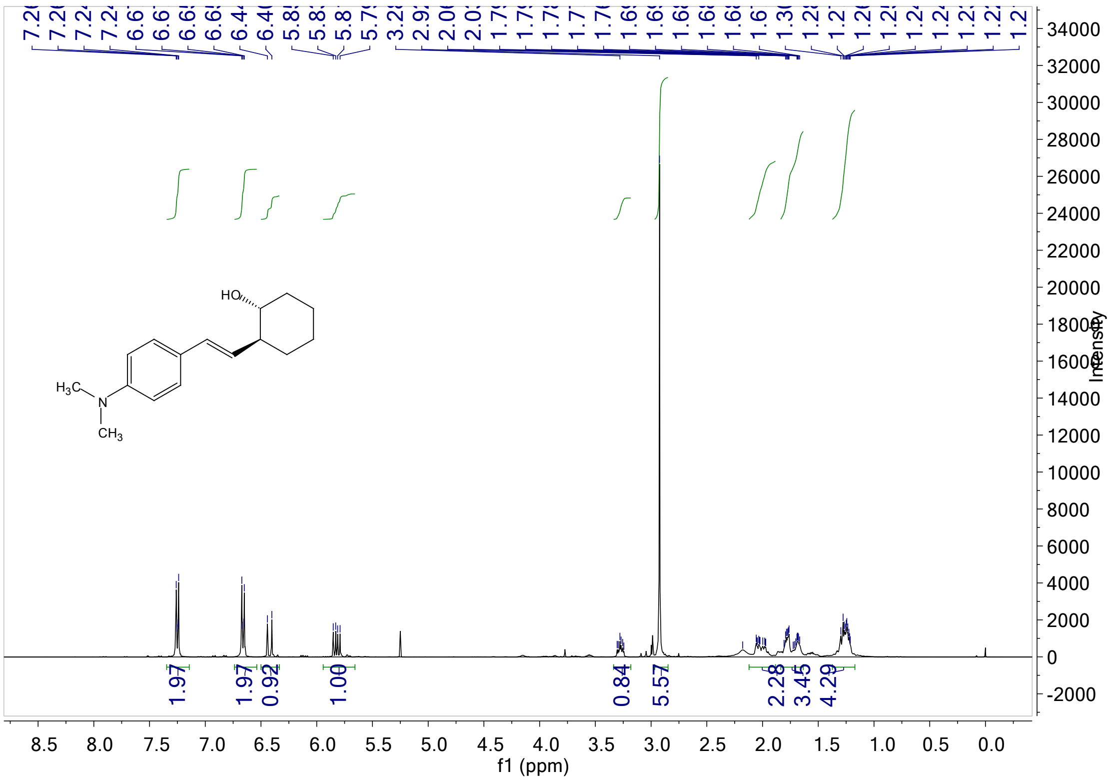

169.5
149.8
134.9
132.4
130.9
127.1
121.6
73.25
50.56
33.93
31.42
25.20
24.82
21.17
HO
H3C
O
O
f1 (ppm)

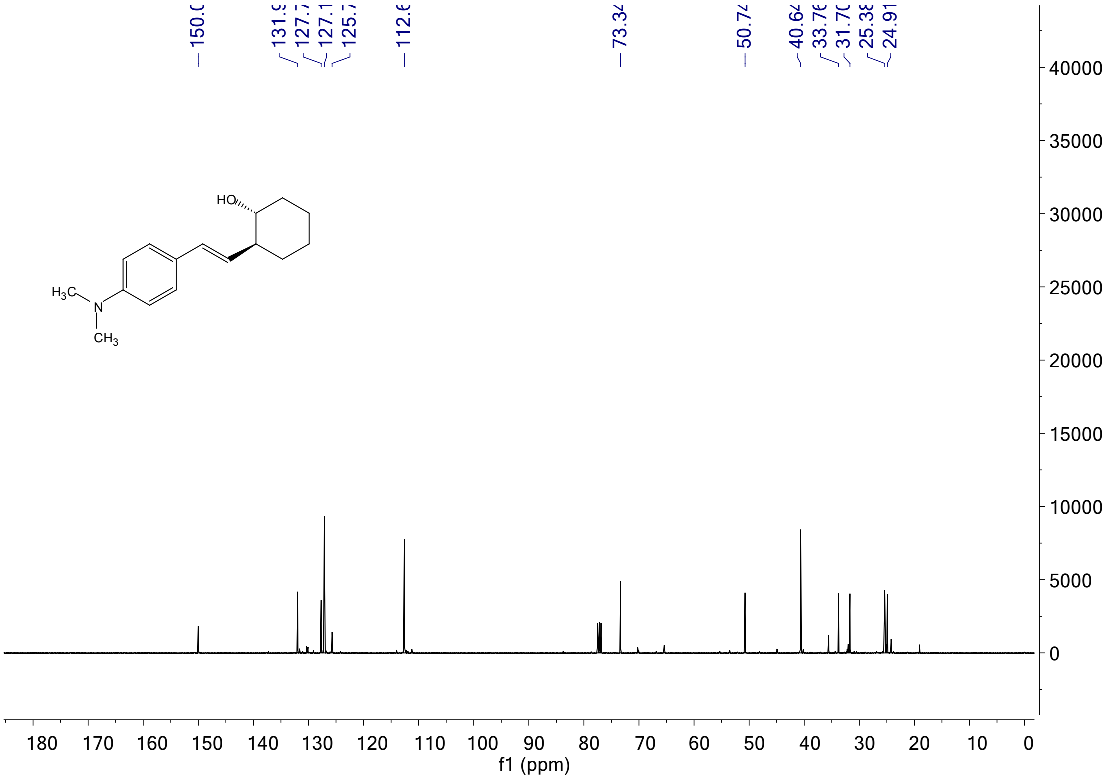

7.27
6.92
6.91
6.79
6.79
6.77
6.77
6.74
6.72
6.44
6.40
5.95
5.94
5.93
5.92
5.90
5.88
5.86
3.31
2.06
2.03
2.02
2.00
2.00
1.80
1.78
1.70
1.70
1.69
1.68
1.32
1.31
1.29
1.27
1.27
1.26
1.25
1.23
-0.0
HO
O
O
1.10
2.43
1.00
3.35
1.03
3.27
3.32
4.73
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
Intensity
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

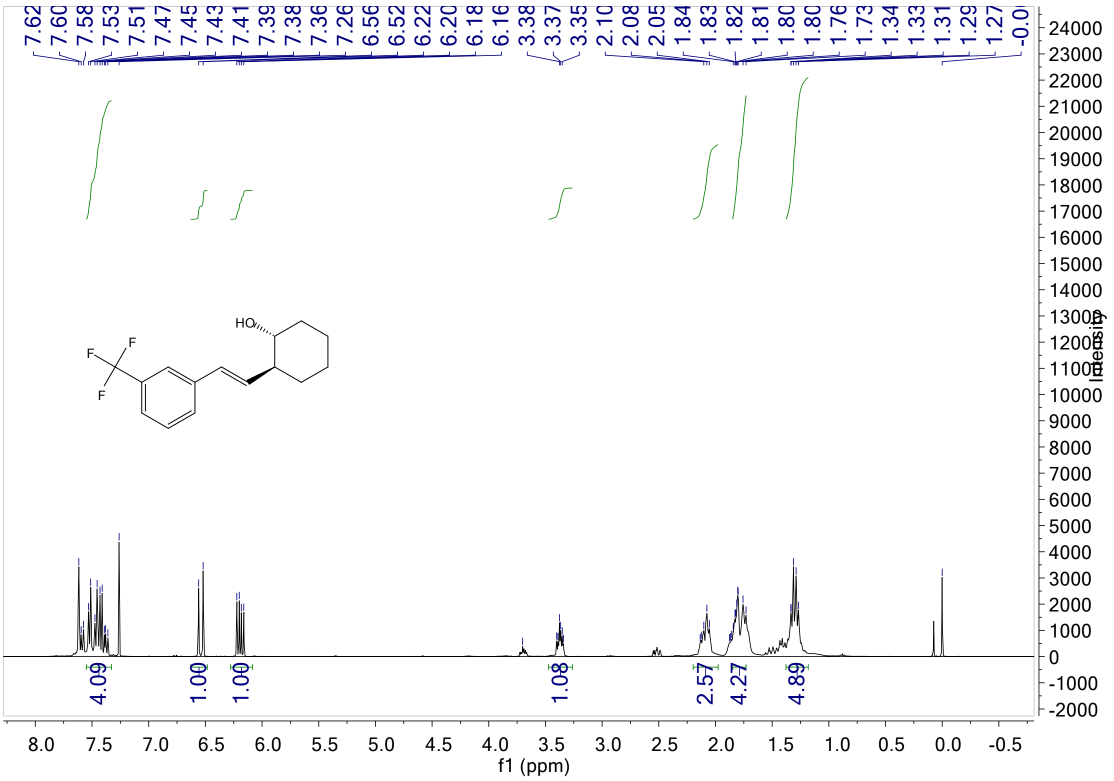

148.
146.
131.
131.
130.
120.
108.
105.
101.
73.29
50.47
33.91
31.52
25.24
24.83
HO
O
O
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.31
7.31
7.29
7.29
7.26
6.85
6.83
6.48
6.44
5.95
5.92
5.91
5.89
3.80
3.32
3.31
2.07
2.06
2.05
2.04
2.04
1.92
1.81
1.80
1.79
1.78
1.70
1.69
1.68
1.31
1.29
1.28
1.27
1.27
1.26
1.25
1.24
1.23
-0.0
HO
H3C
O
2.34
2.22
1.06
1.00
0.65
3.21
1.05
3.03
3.62
4.25
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
Intensity
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -1.0 -1.5
f1 (ppm)

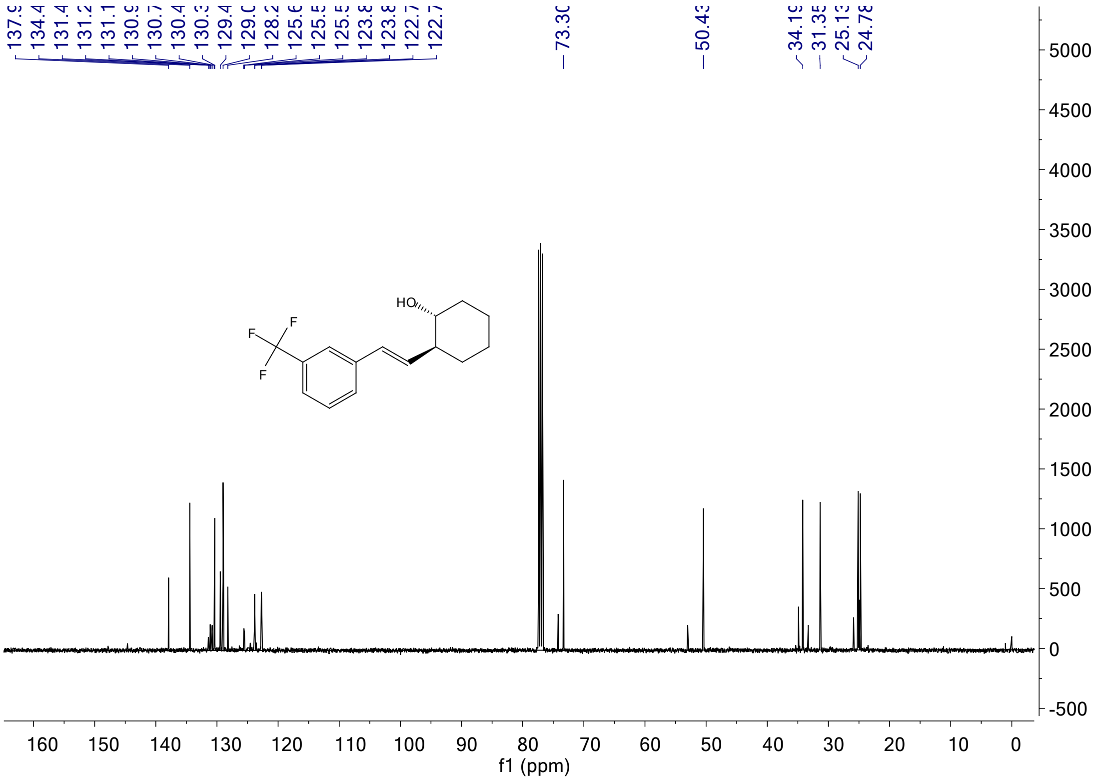

160.2
159.0
136.0
131.4
131.3
129.8
128.6
127.3
113.9
113.6
83.67
73.28
55.35
55.33
50.64
48.12
33.84
31.56
30.54
26.56
25.61
25.28
24.86
24.17
HO
O
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

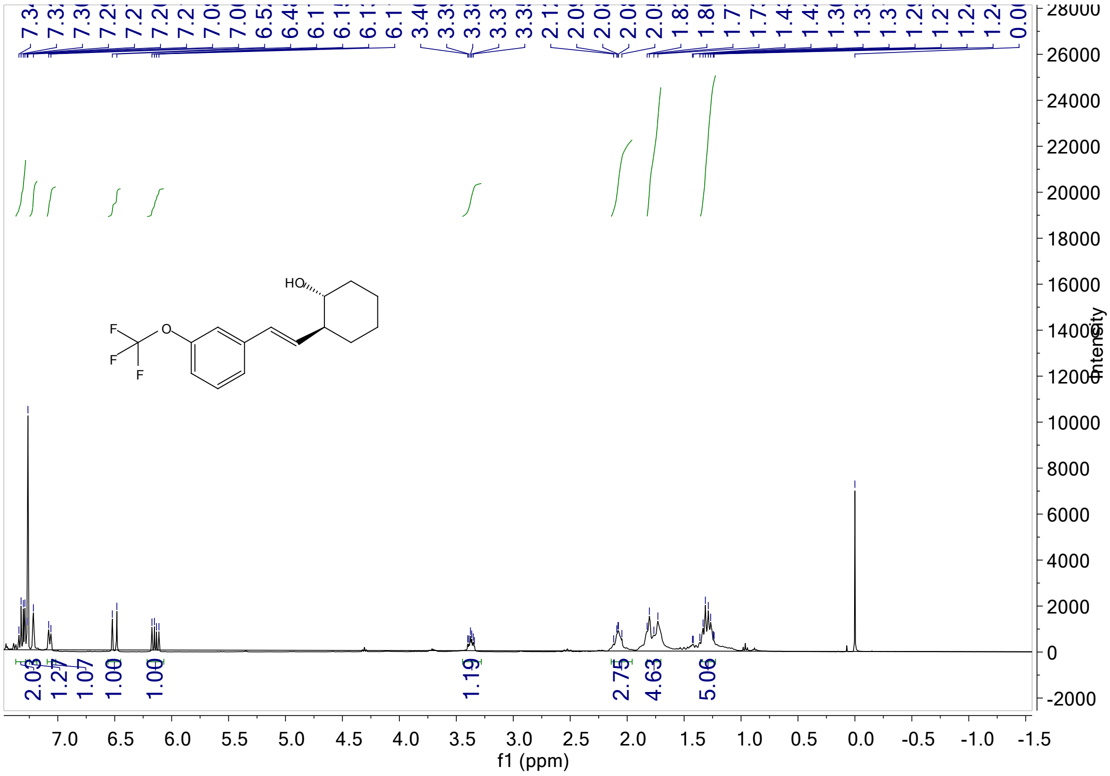

HO
H3C
N
CH3
1.97
1.97
0.92
1.00
0.84
5.57
2.28
3.45
4.29
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
Intensity

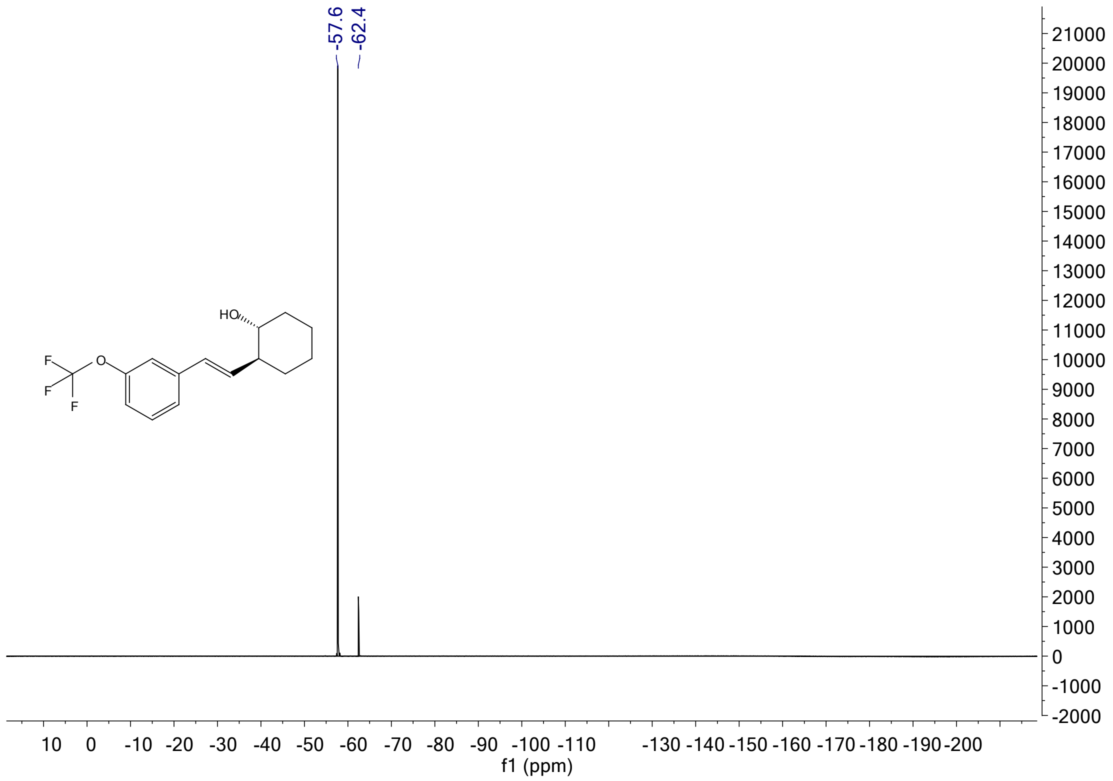

73.34
50.74
40.64
24.91
HO
H3C
N
CH3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
40000
35000
30000
25000
20000
15000
10000
5000
0

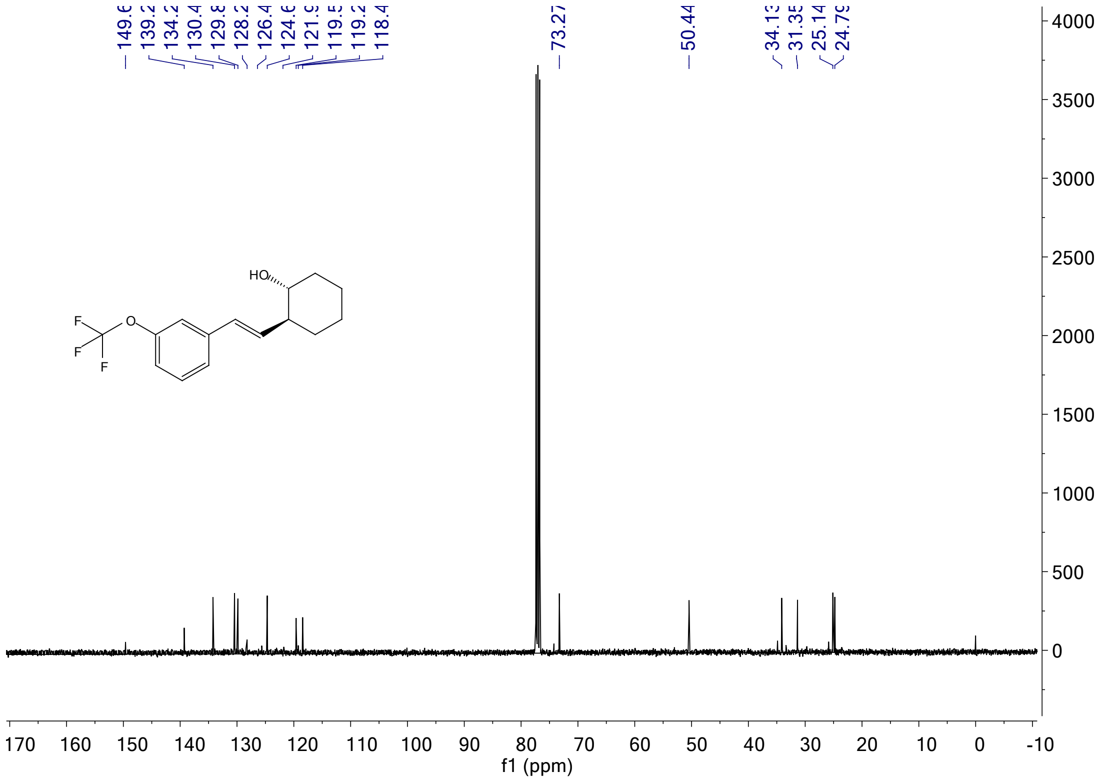

7.62
7.60
7.58
7.53
7.51
7.47
7.45
7.43
7.41
7.39
7.38
7.36
7.26
6.56
6.52
6.22
6.20
6.18
6.16
3.38
3.37
3.35
2.10
2.08
2.05
1.84
1.83
1.82
1.81
1.80
1.80
1.76
1.73
1.34
1.33
1.31
1.29
1.27
-0.0
HO
F
F
F
4.09
1.00
1.00
1.08
2.57
4.27
4.89
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
24000
23000
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
Intensity

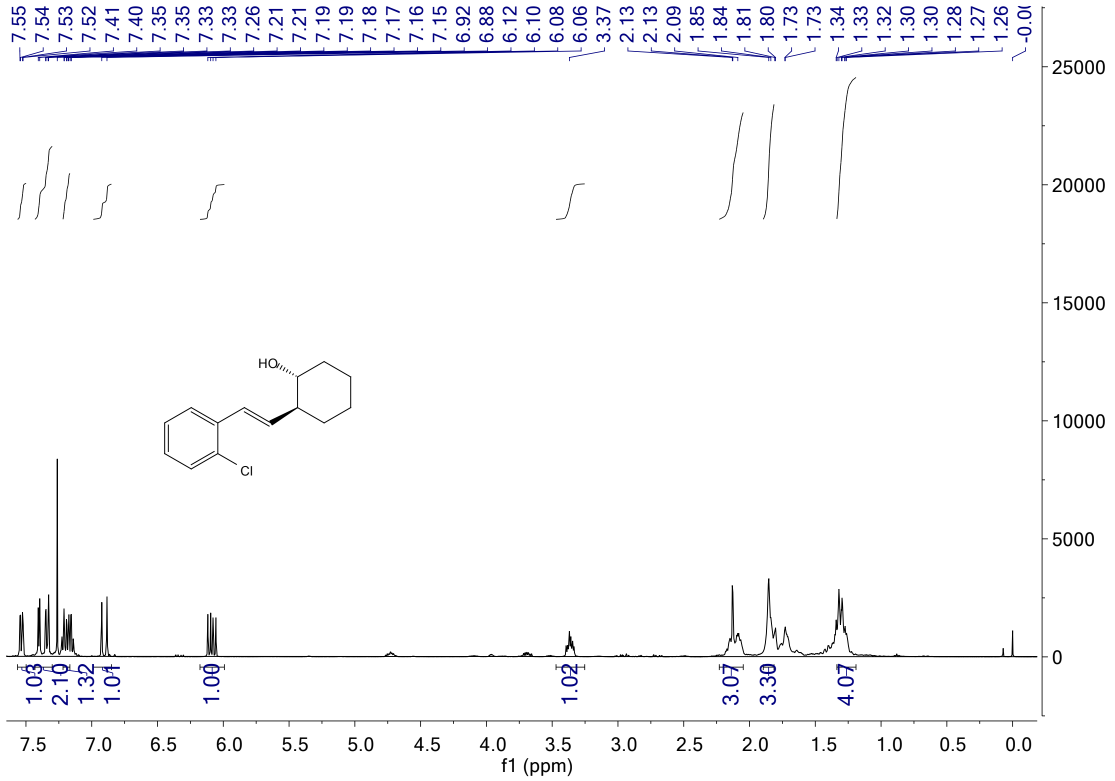

-62.7
HO
F
F
F
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000

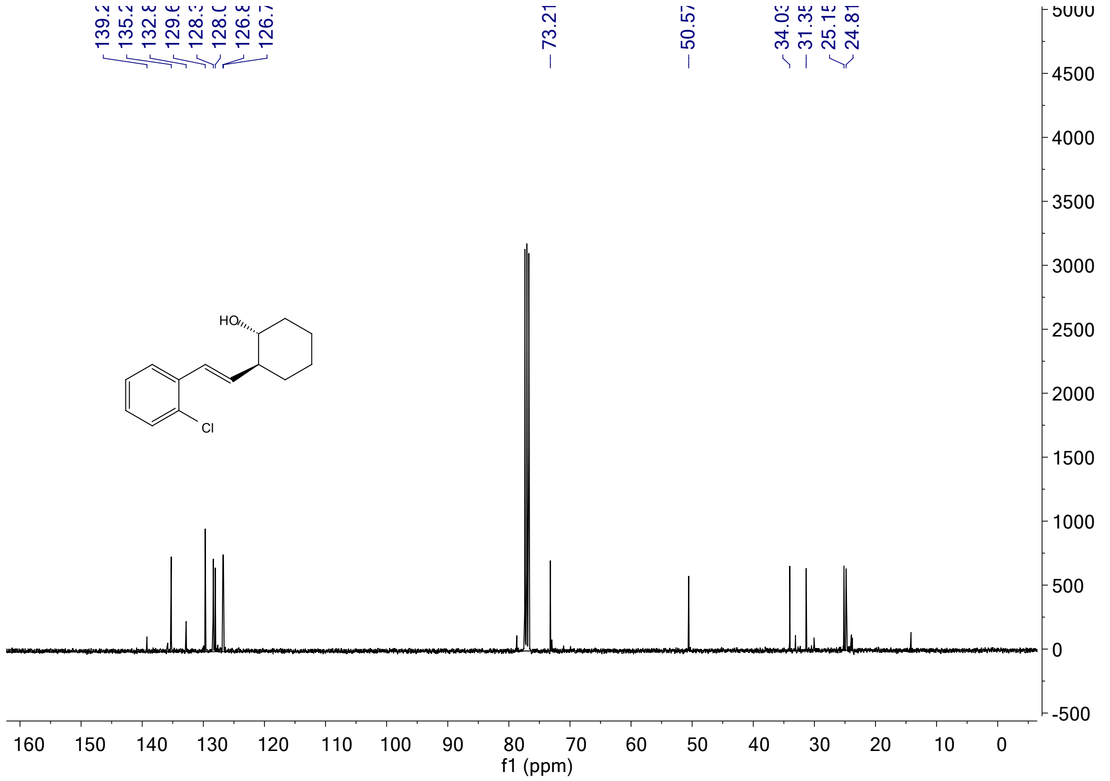

137.9
134.4
131.4
131.2
131.1
130.9
130.7
130.4
130.3
129.4
129.0
128.2
125.6
125.5
125.5
123.8
123.8
122.7
122.7
73.30
50.43
34.19
31.35
25.13
24.78
HO
F
F
F
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

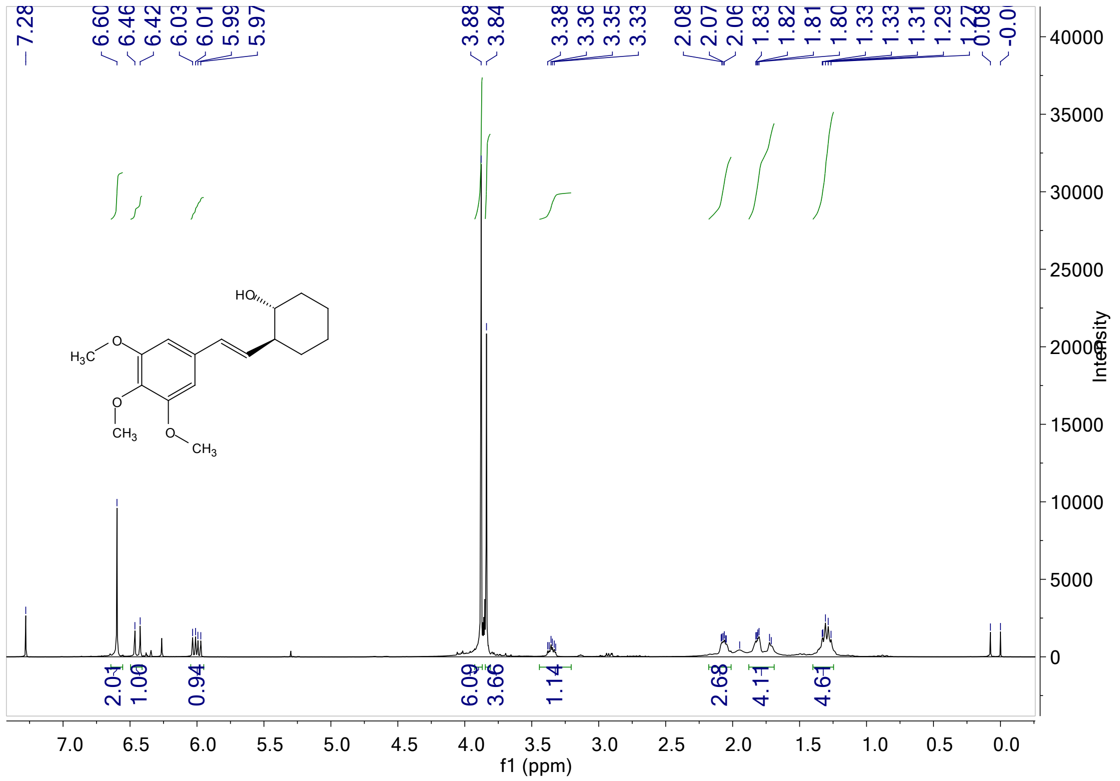

HO
O
F
F
F
2.03
1.27
1.07
1.00
1.00
1.19
2.75
4.63
5.06
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
Intensity
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

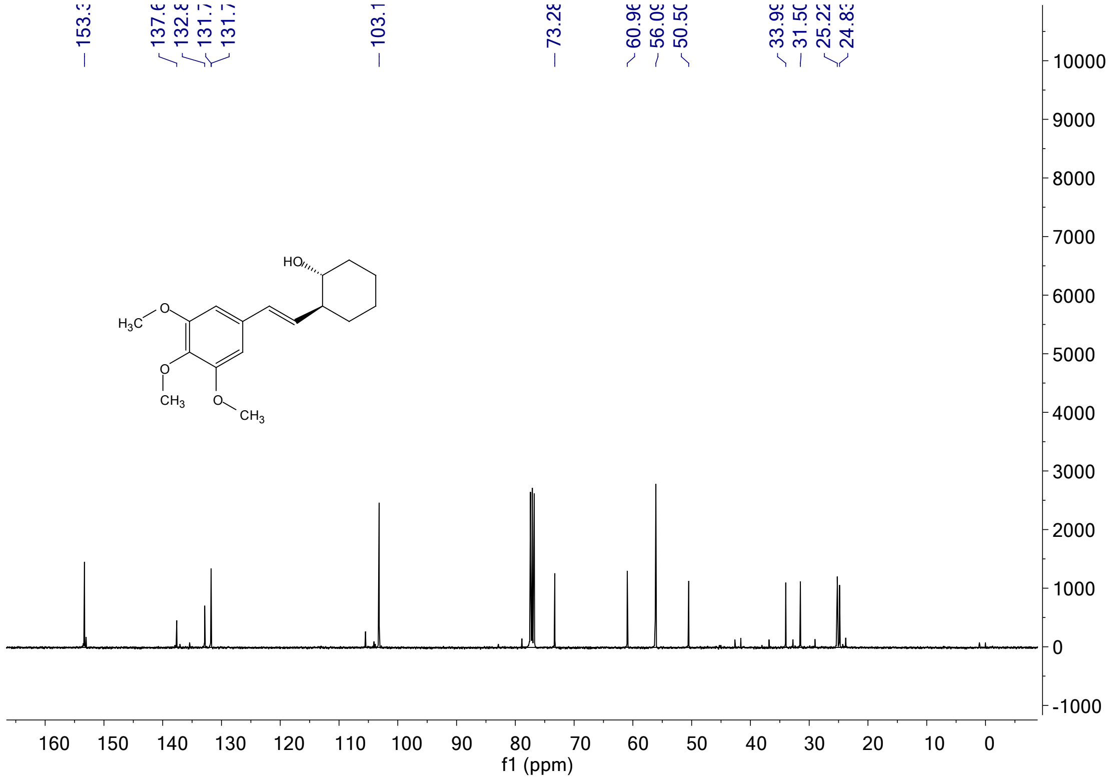

-57.6
-62.4
HO
F
F
F
O
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

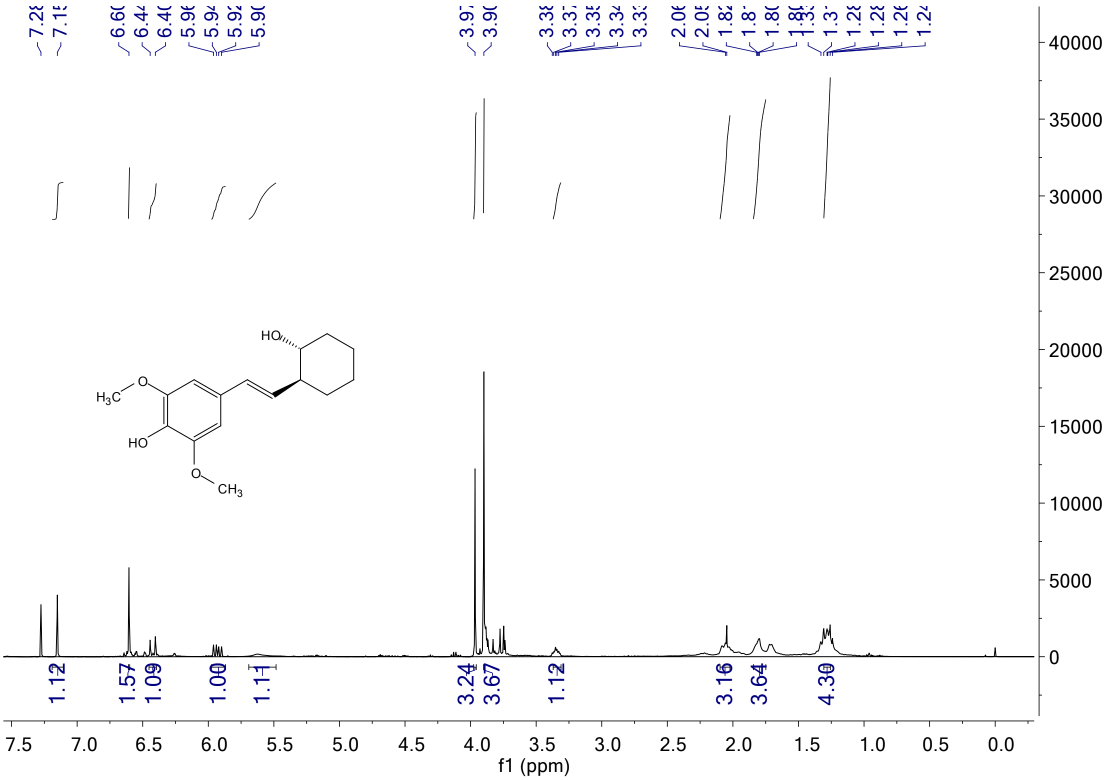

149.6
139.2
134.2
130.4
129.8
128.2
126.4
124.6
121.9
119.5
119.2
118.4
73.27
50.44
34.13
31.35
25.14
24.79
HO
F
F
F
O
4000
3500
3000
2500
2000
1500
1000
500
0
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

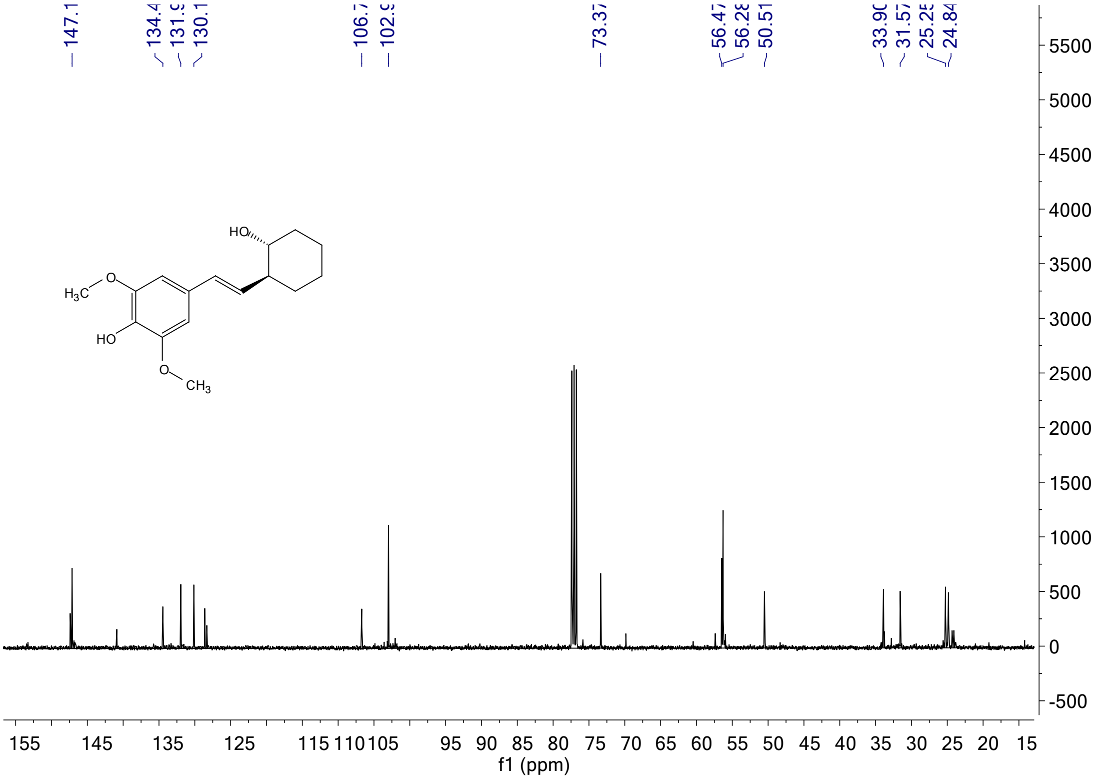

7.55
7.54
7.53
7.52
7.41
7.40
7.35
7.35
7.33
7.33
7.26
7.21
7.21
7.19
7.19
7.18
7.17
7.16
7.15
6.92
6.88
6.12
6.10
6.08
6.06
3.37
2.13
2.13
2.09
1.85
1.84
1.81
1.80
1.73
1.73
1.34
1.33
1.32
1.30
1.30
1.28
1.27
1.26
-0.00
HO
Cl
1.03
2.10
1.32
1.04
1.00
1.02
3.07
3.30
4.07
25000
20000
15000
10000
5000
0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

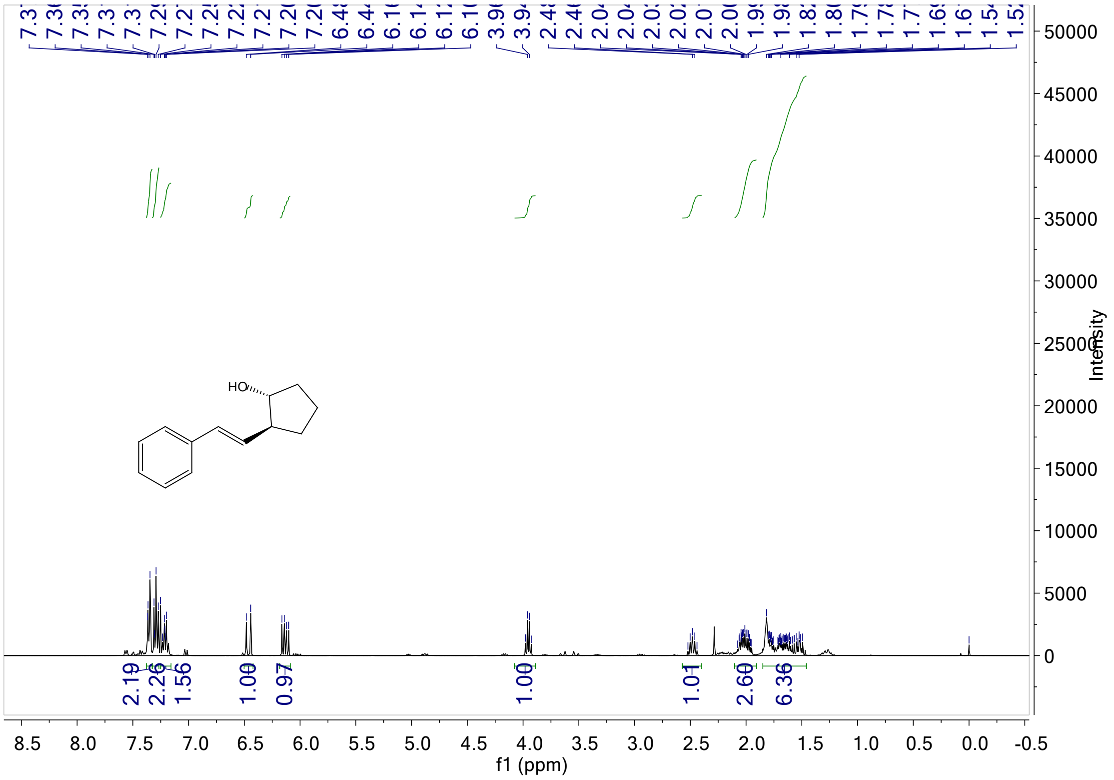

139.2
135.2
132.8
129.6
128.3
128.0
126.8
126.7
73.21
50.57
34.03
31.35
25.15
24.81
HO
Cl
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

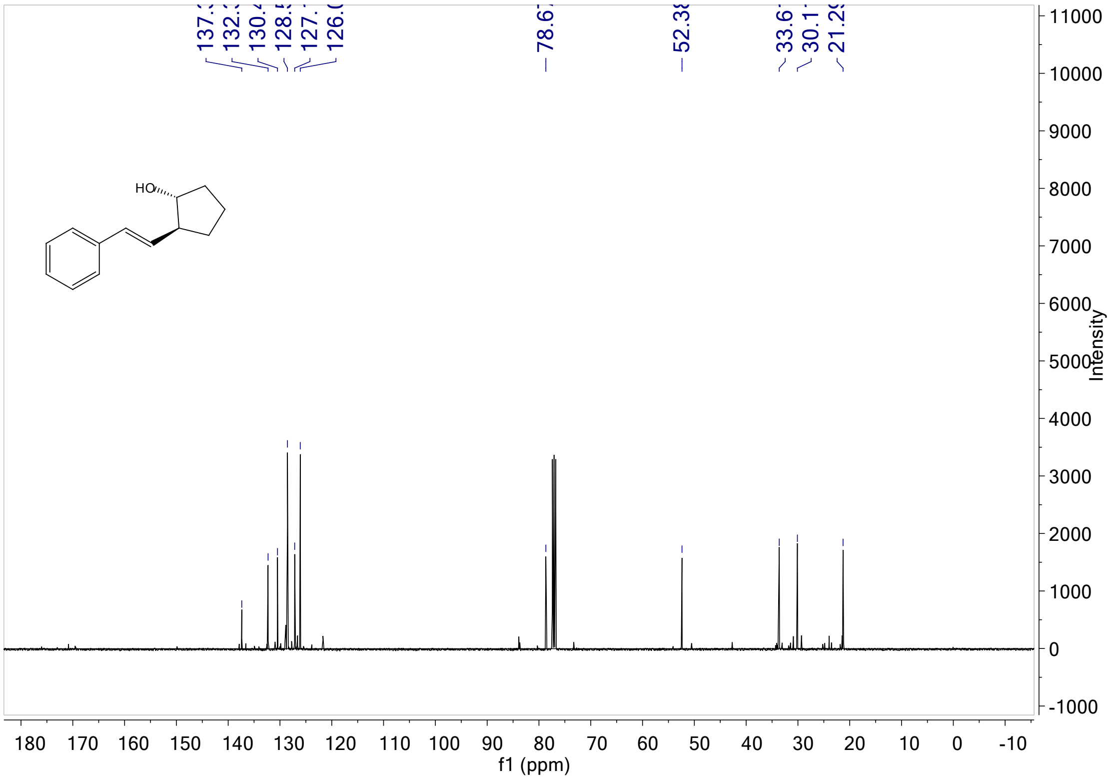

7.28
6.60
6.46
6.42
6.03
6.01
5.99
5.97
3.88
3.84
3.38
3.36
3.35
3.33
2.08
2.07
2.06
1.83
1.82
1.81
1.80
1.33
1.33
1.31
1.29
1.27
0.08
-0.0
2.01
1.00
0.94
6.09
3.66
1.14
2.68
4.11
4.61
HO
H_3C
O
O
CH_3
O
CH_3
40000
35000
30000
25000
20000
15000
10000
5000
0
Intensity
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

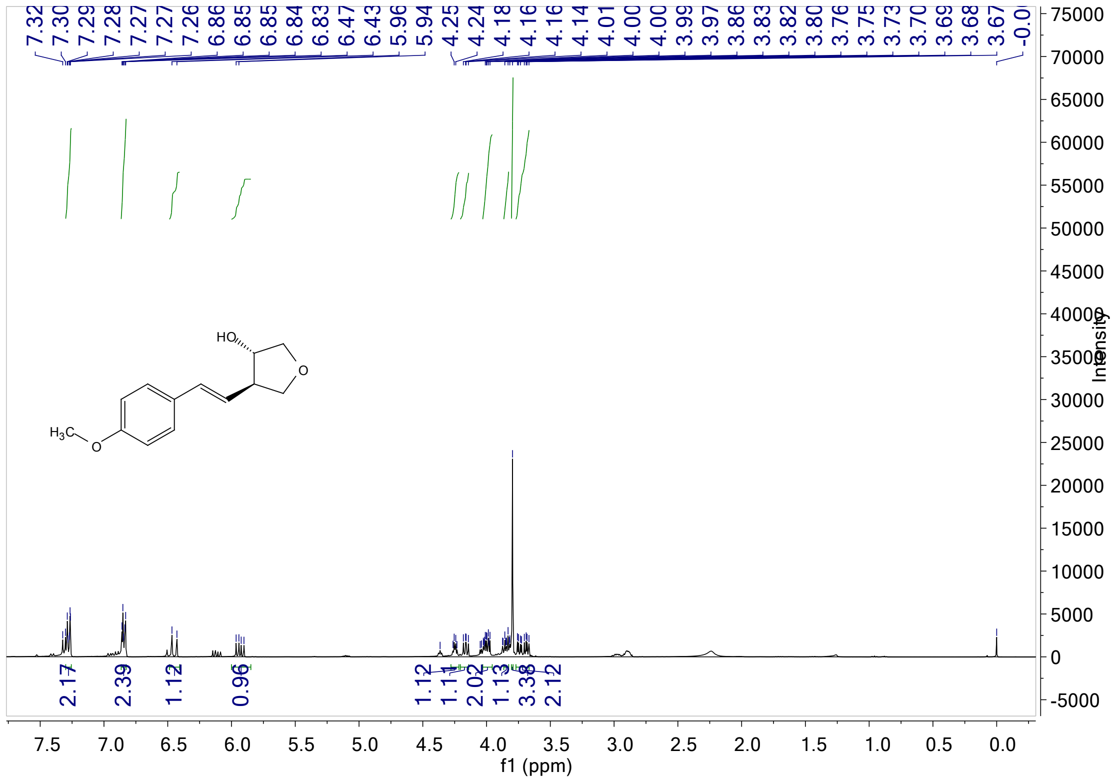

153.3
137.6
132.8
131.7
131.7
103.1
73.28
60.96
56.09
50.50
33.99
31.50
25.22
24.83
HO
H_3C
O
O
CH_3
O
CH_3
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

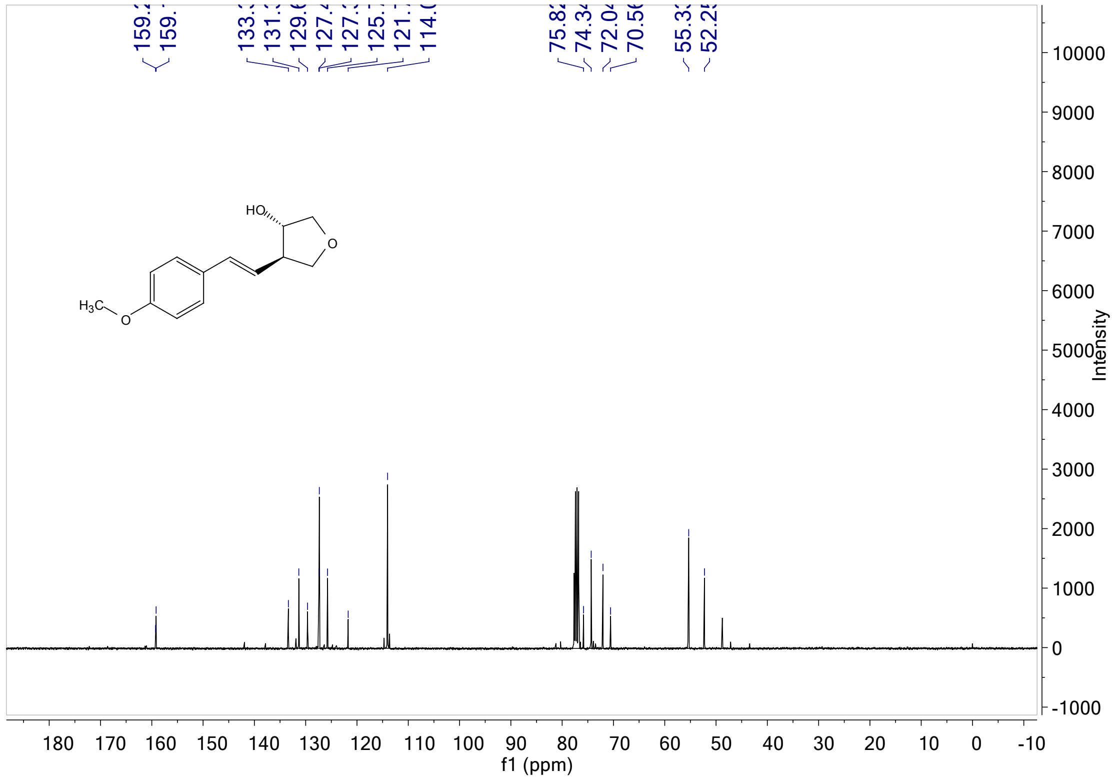

7.2
7.1
6.6
6.4
6.4
5.9
5.9
5.9
5.9
3.9
3.9
3.3
3.3
3.3
3.3
3.3
2.0
2.0
1.8
1.8
1.8
1.8
1.3
1.3
1.2
1.2
1.2
1.2
HO
H3C
O
HO
O
CH3
1.12
1.57
1.09
1.00
1.11
3.24
3.67
1.12
3.16
3.64
4.30
40000
35000
30000
25000
20000
15000
10000
5000
0
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

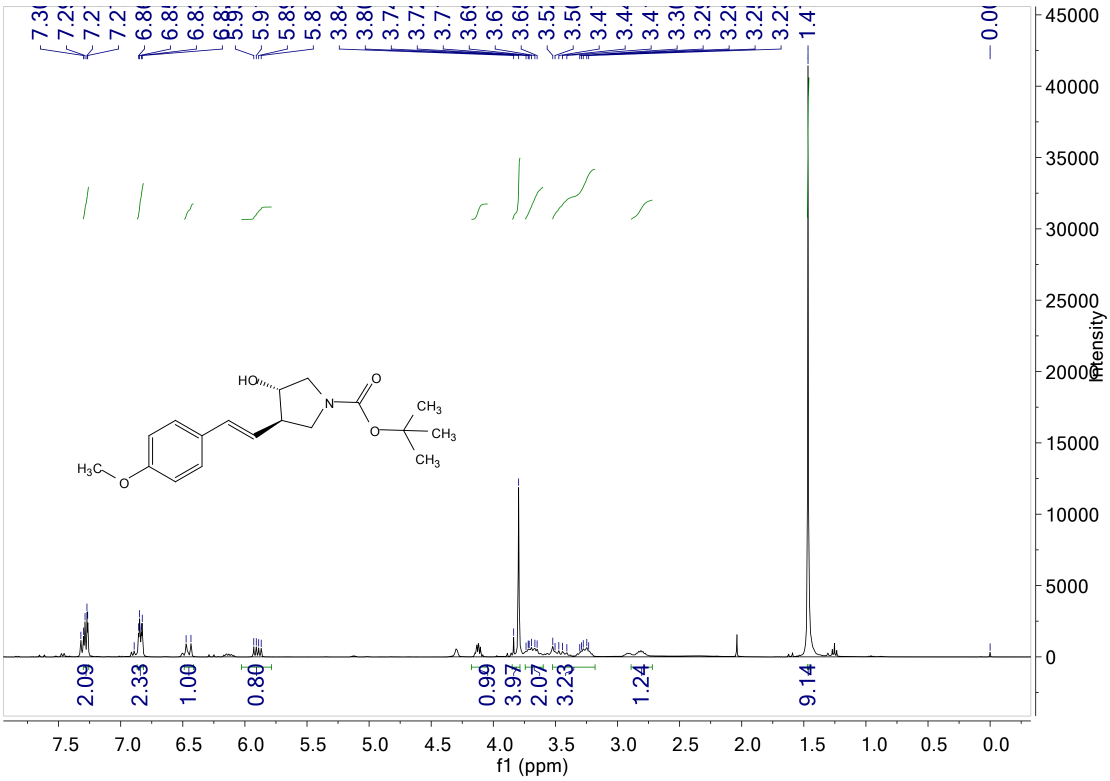

147.1
134.4
131.9
130.1
106.7
102.9
73.37
56.47
56.28
50.51
33.90
31.57
25.25
24.84
HO
H3C
O
HO
O
CH3
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
155 145 135 125 115 110 105 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15
f1 (ppm)

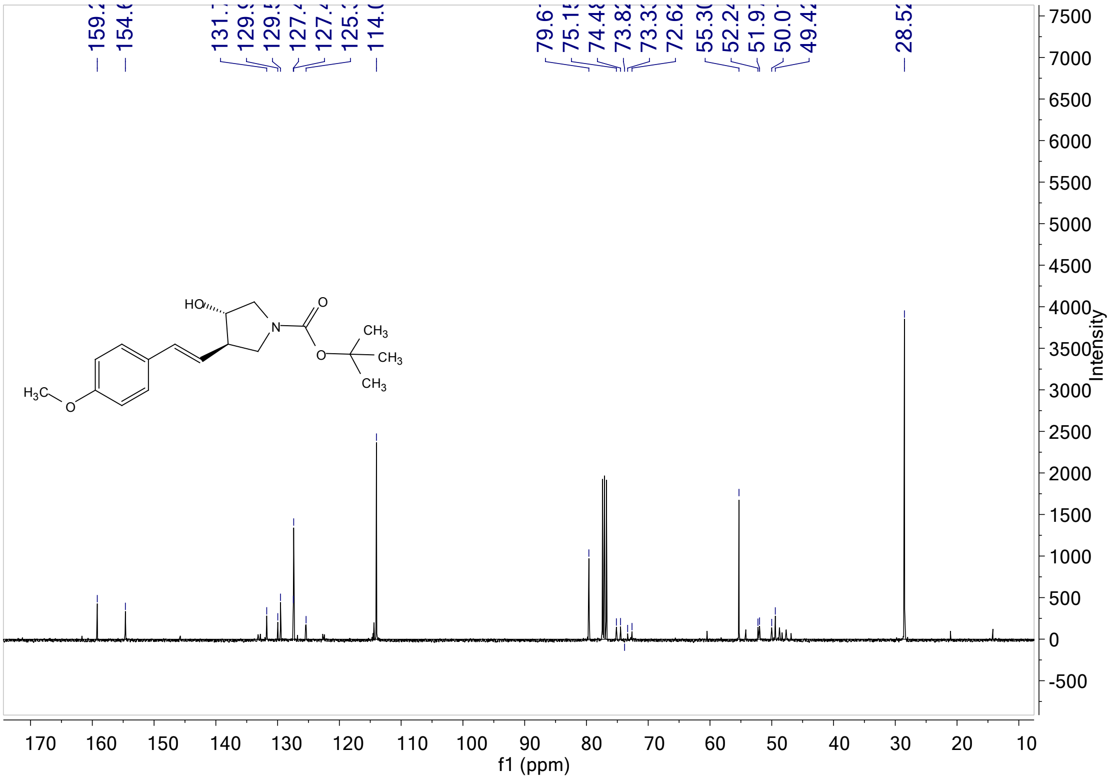

7.3
7.30
7.3
7.3
7.3
7.29
7.2
7.2
7.2
7.2
7.2
7.2
6.4
6.4
6.1
6.1
6.1
6.1
3.96
3.94
2.4
2.40
2.0
2.0
2.0
2.0
2.0
2.00
1.9
1.98
1.8
1.80
1.7
1.7
1.7
1.6
1.6
1.5
1.5
HO
2.19
2.26
1.56
1.00
0.97
1.00
1.01
2.60
6.36
f1 (ppm)
Intensity
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

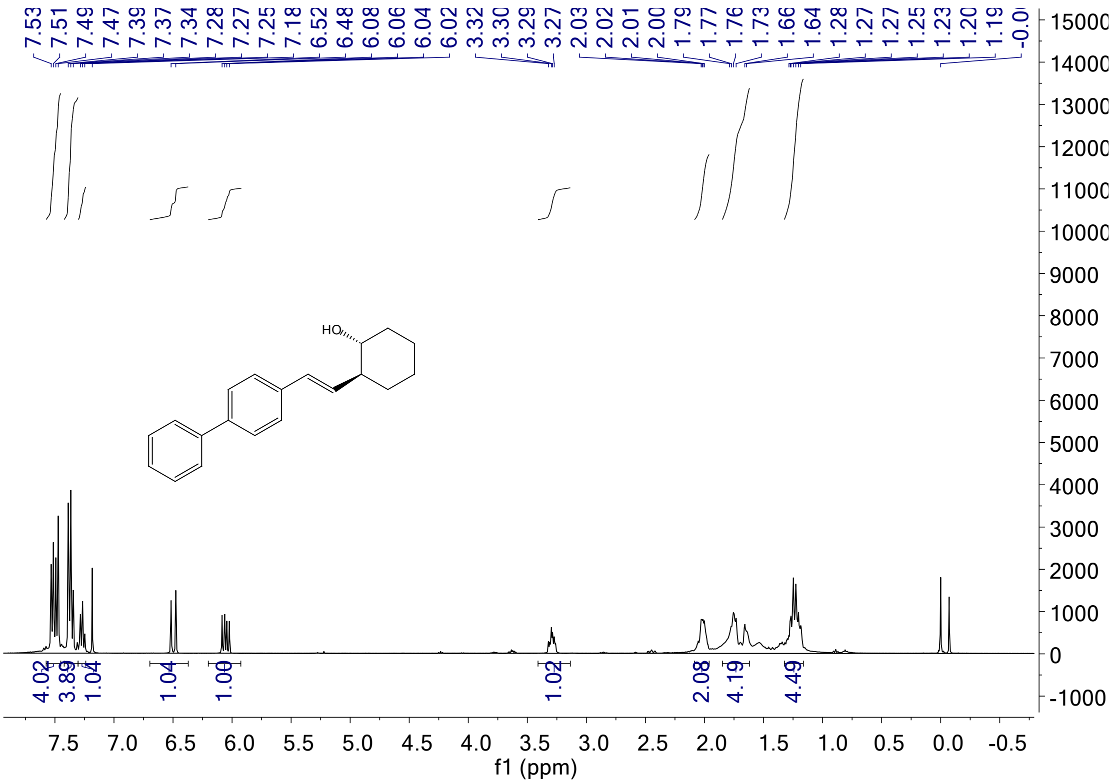

137.
132.
130.
128.
127.
126.
78.6
52.3
33.6
30.1
21.2
HO
f1 (ppm)
Intensity
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

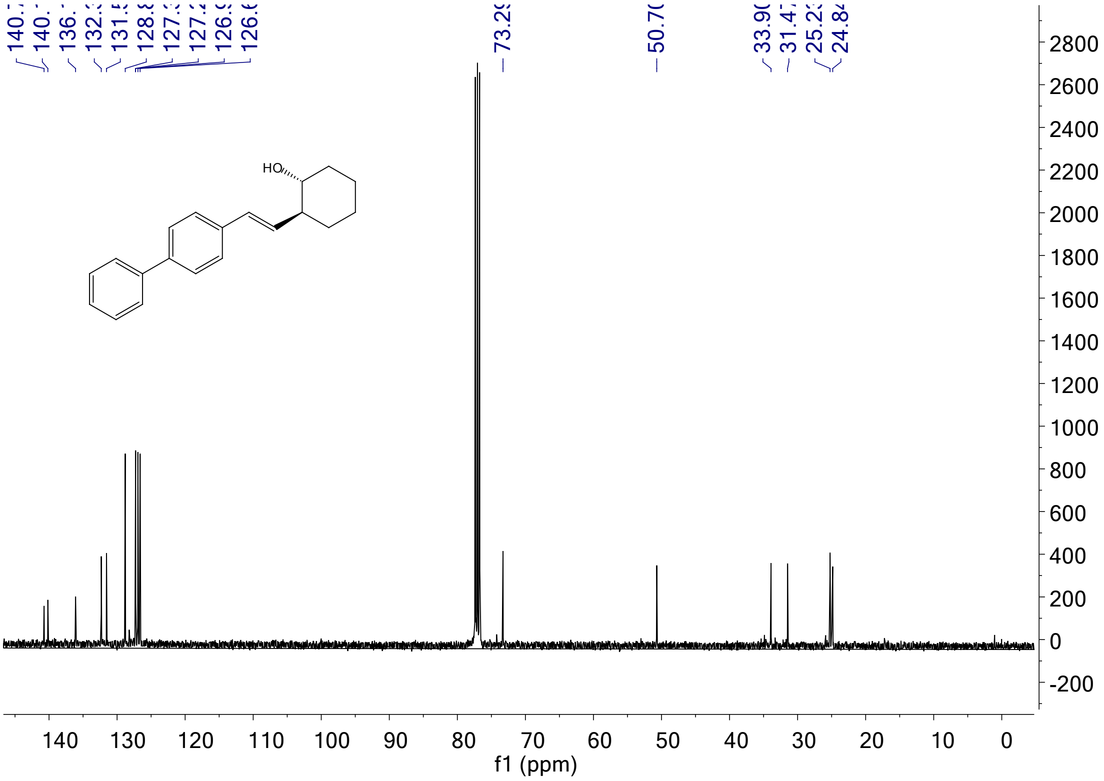

7.32
7.30
7.29
7.28
7.27
7.27
7.26
6.86
6.85
6.85
6.84
6.83
6.47
6.43
5.96
5.94
4.25
4.24
4.18
4.16
4.16
4.14
4.01
4.00
4.00
3.99
3.97
3.86
3.83
3.82
3.80
3.76
3.75
3.73
3.70
3.69
3.68
3.67
-0.0
2.17
2.39
1.12
0.96
1.12
1.14
2.02
1.13
3.38
2.12
HO
O
H3C
O
f1 (ppm)
Intensity

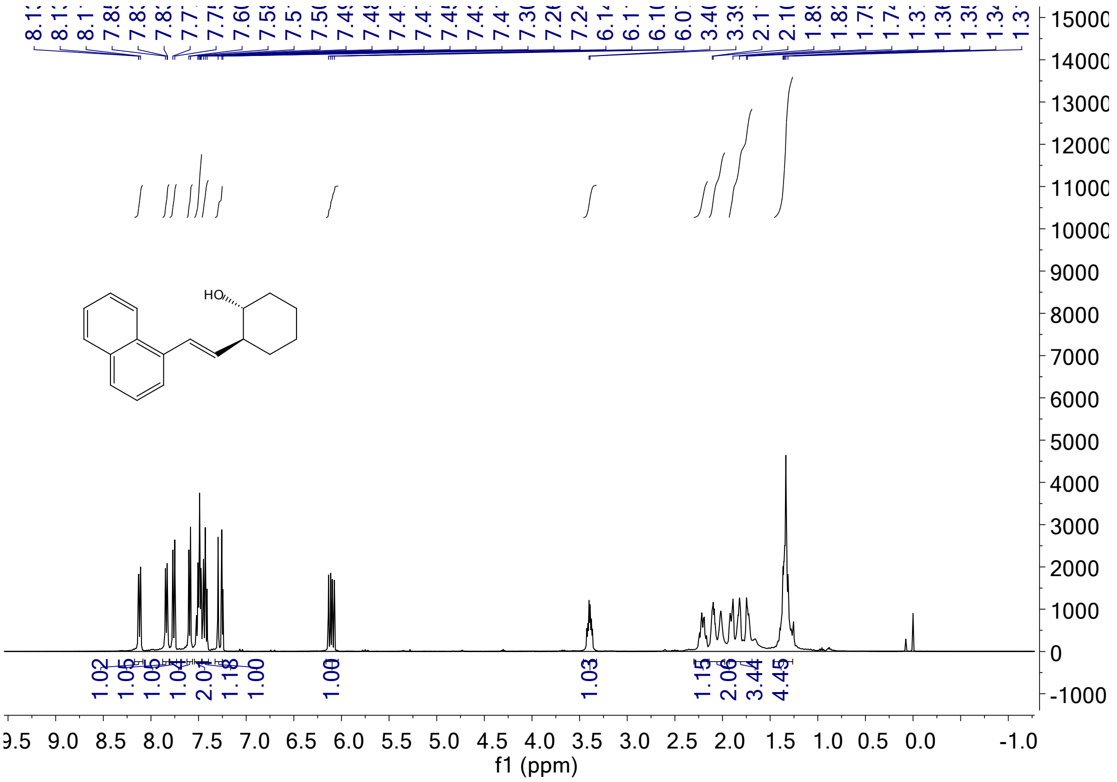

159.2
159.
133.
131.
129.
127.4
127.
125.
121.
114.0
75.82
74.34
72.04
70.56
55.3
52.25
HO
O
H3C
O
f1 (ppm)
Intensity

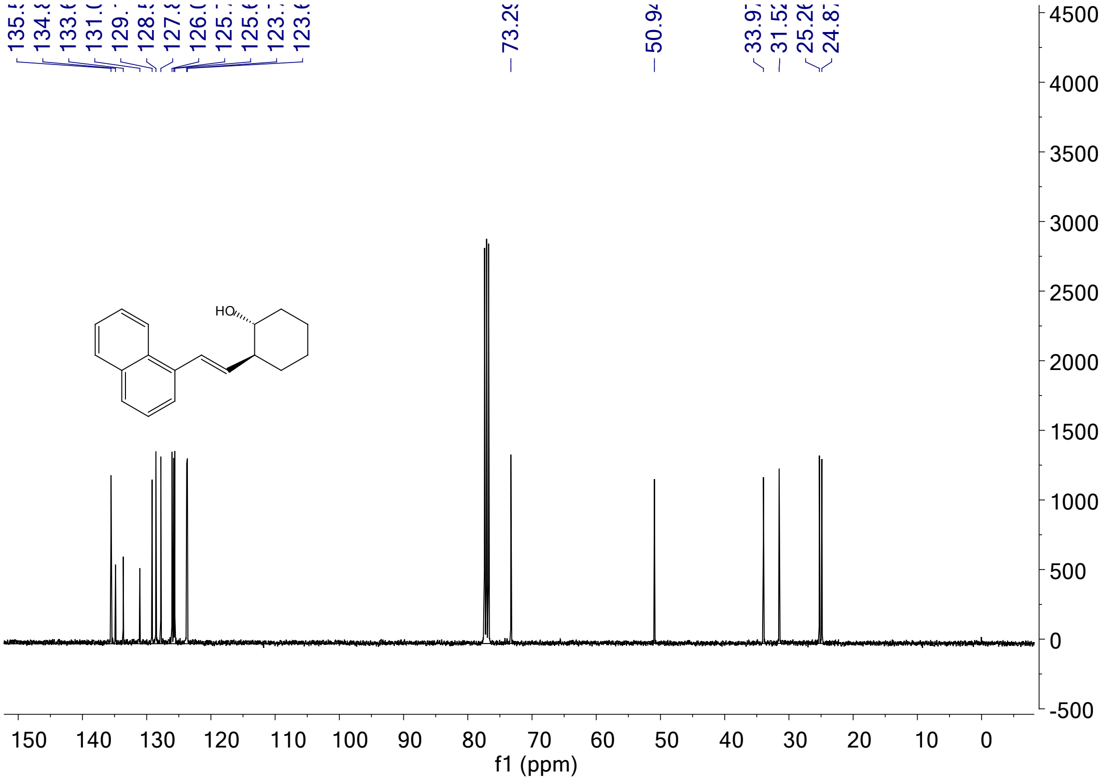

HO
N
O
O
CH3
CH3
CH3
H3C
O
2.09
2.33
1.00
0.80
0.99
3.97
2.07
3.23
1.24
9.14
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
Intensity

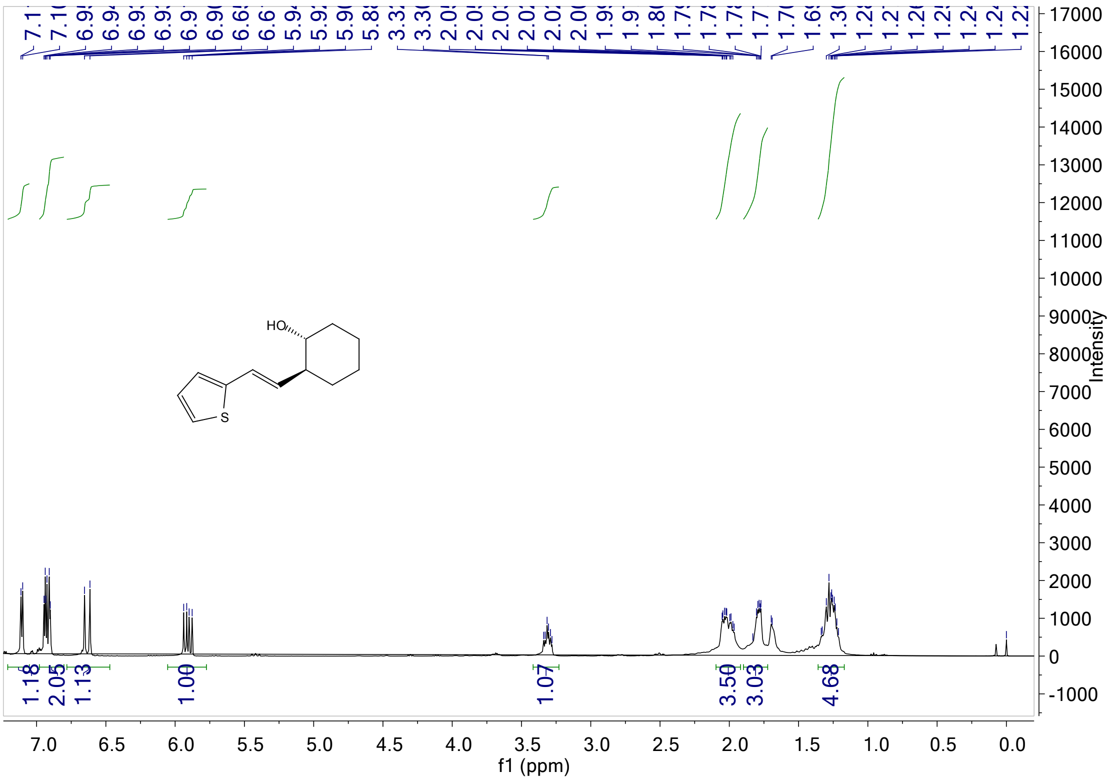

HO
N
O
O
CH3
CH3
CH3
H3C
O
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
Intensity

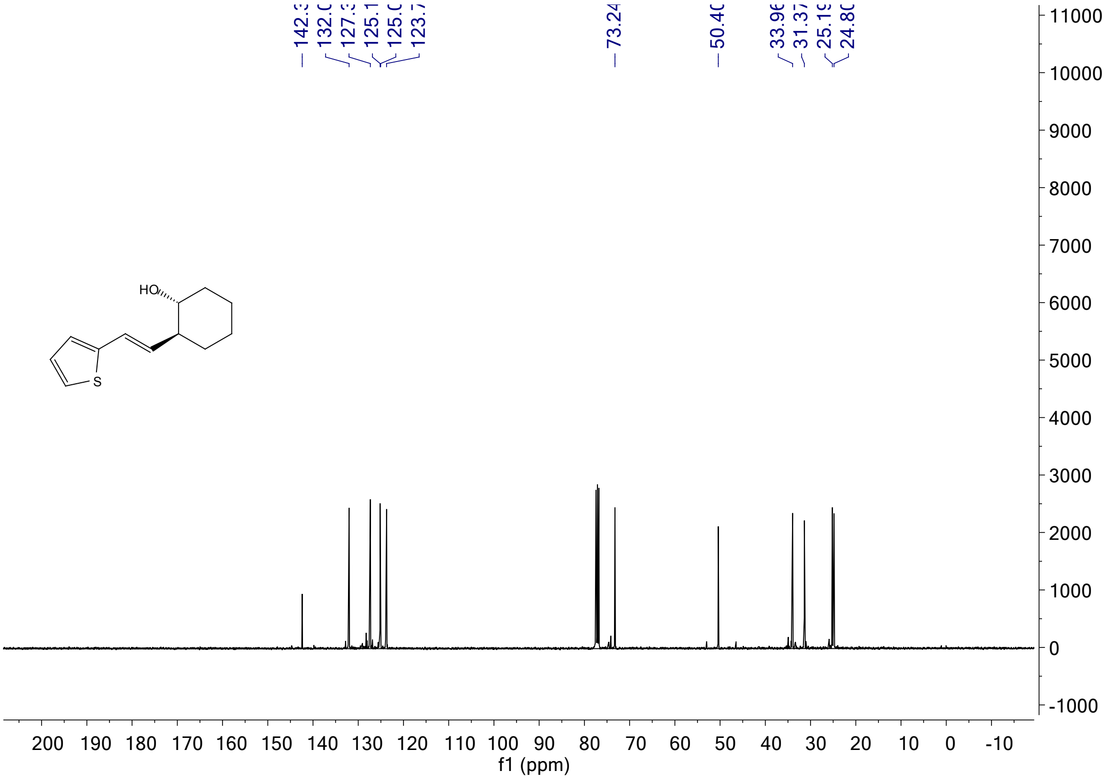

7.53
7.51
7.49
7.47
7.39
7.37
7.34
7.28
7.27
7.25
7.18
6.52
6.48
6.08
6.06
6.04
6.02
3.32
3.30
3.29
3.27
2.03
2.02
2.01
2.00
1.79
1.77
1.76
1.73
1.66
1.64
1.28
1.27
1.27
1.25
1.23
1.20
1.19
HO
4.02
3.89
1.04
1.04
1.00
1.02
2.08
4.19
4.49
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

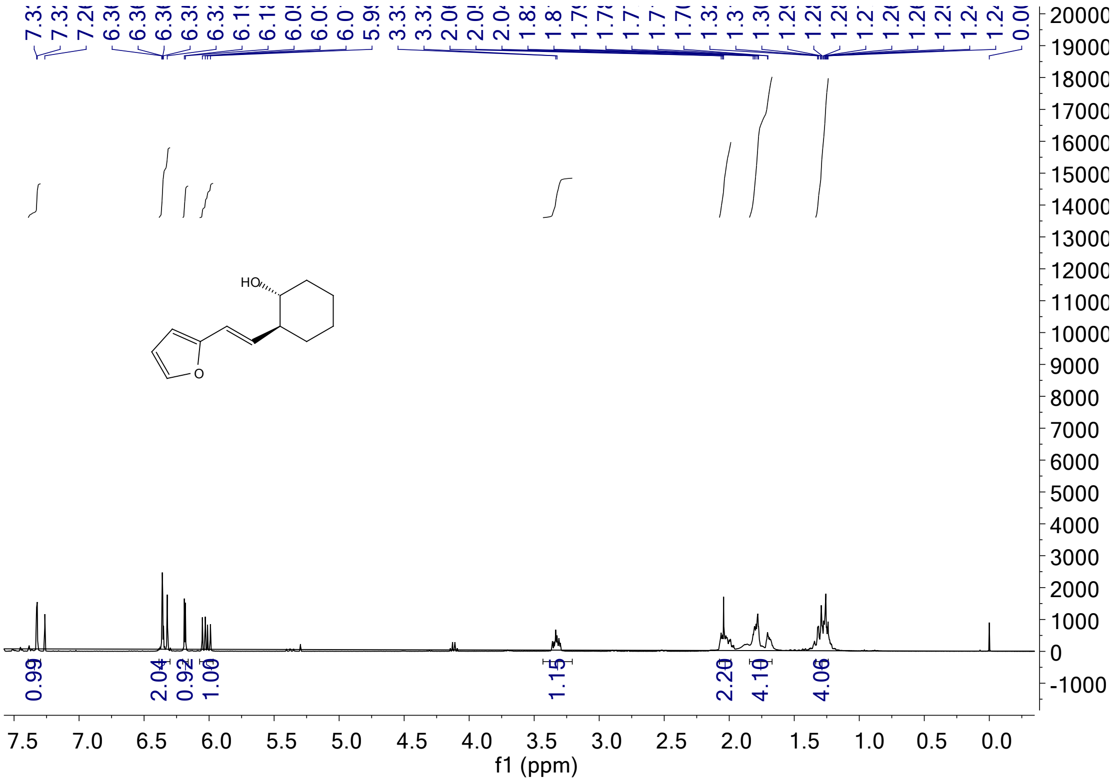

-73.2
-50.7
HO
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200

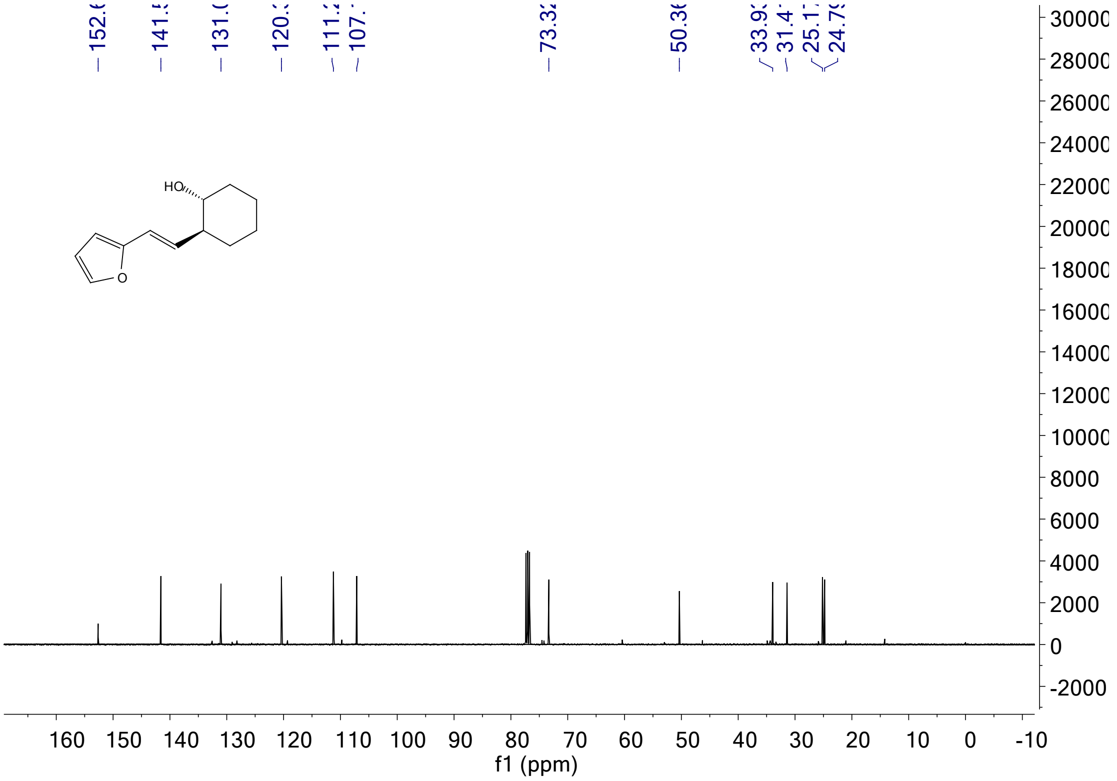

HO
1.02
1.05
1.05
1.04
2.07
1.18
1.00
1.00
1.03
1.15
2.06
3.44
4.45
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-1.0
f1 (ppm)

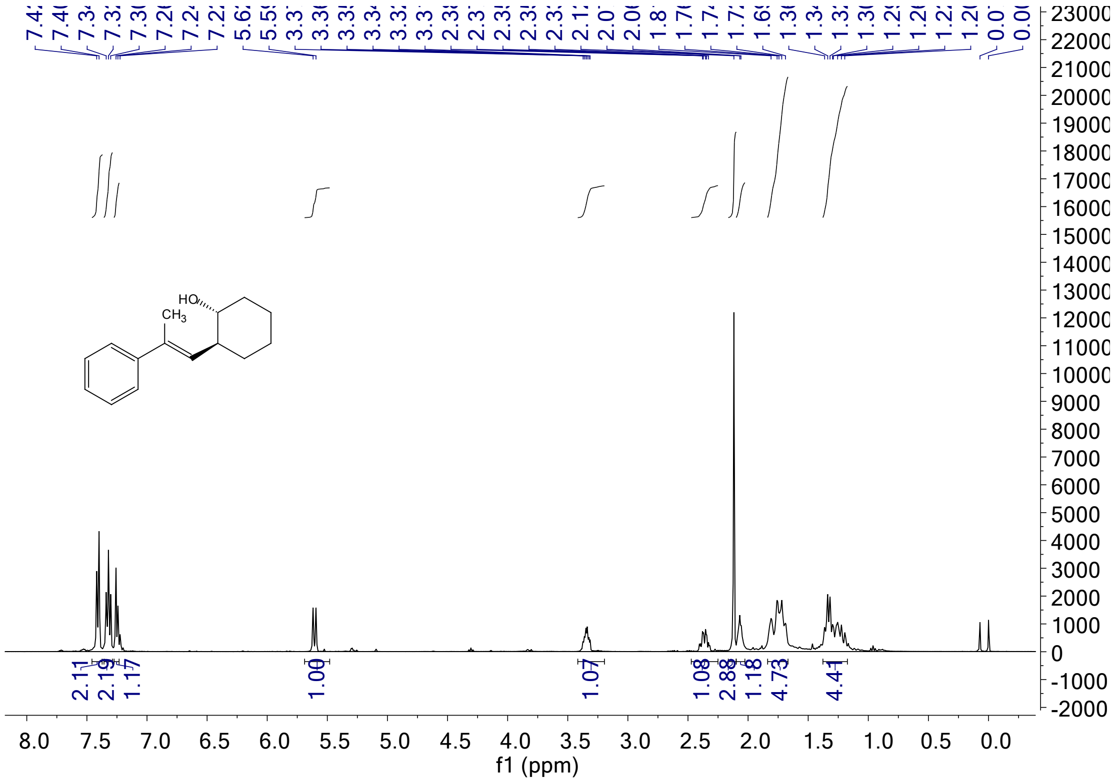

73.29
50.94
33.97
31.52
25.26
24.87
HO
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

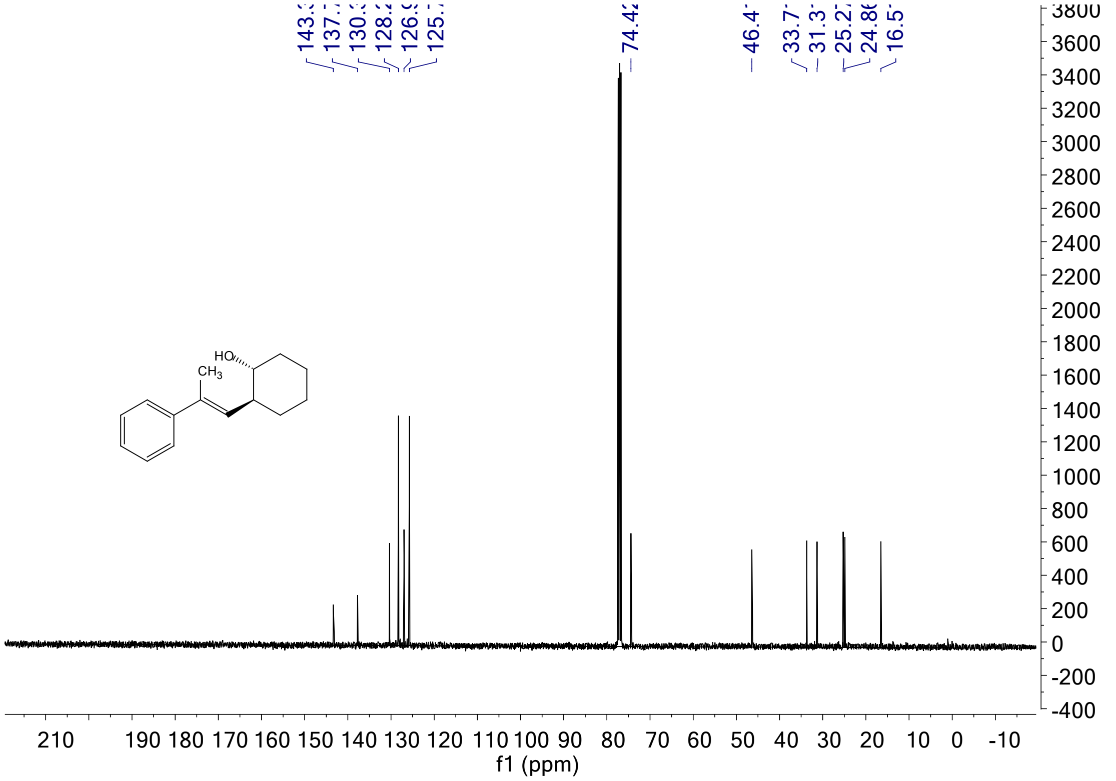

HO
S
1.18
2.05
1.13
1.00
1.07
3.50
3.03
4.68
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
Intensity
17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000

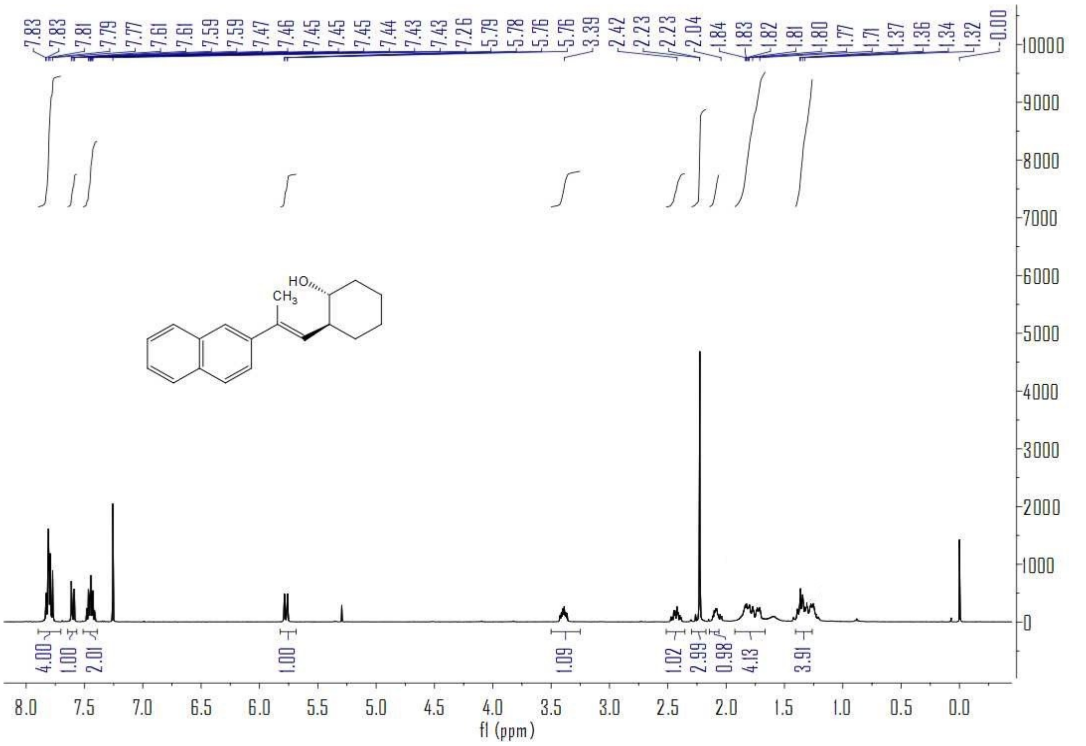

142.3
132.0
127.3
125.1
125.0
123.7
73.24
50.40
33.96
31.37
25.19
24.80
HO
S
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000


HO
O
0.99
2.04
0.92
1.00
1.15
2.20
4.10
4.06
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

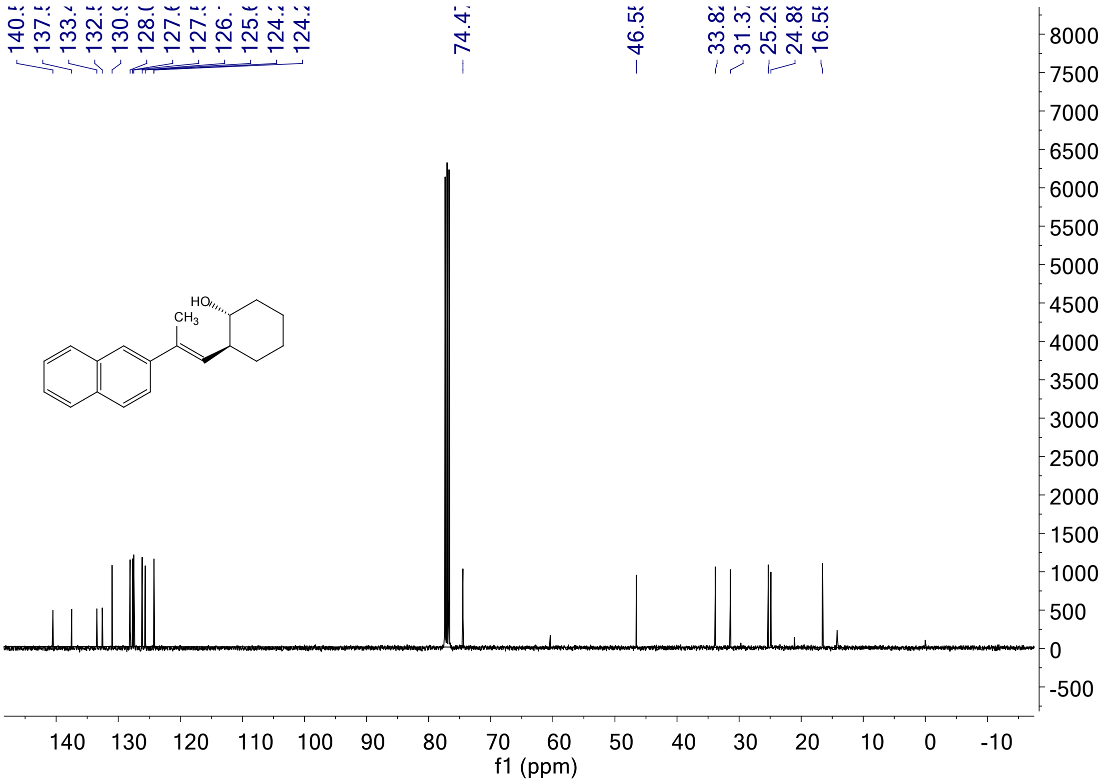

HO
O
73.32
50.36
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


HO
CH3
2.11
2.19
1.17
1.00
1.07
1.08
2.88
1.18
4.73
4.41
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

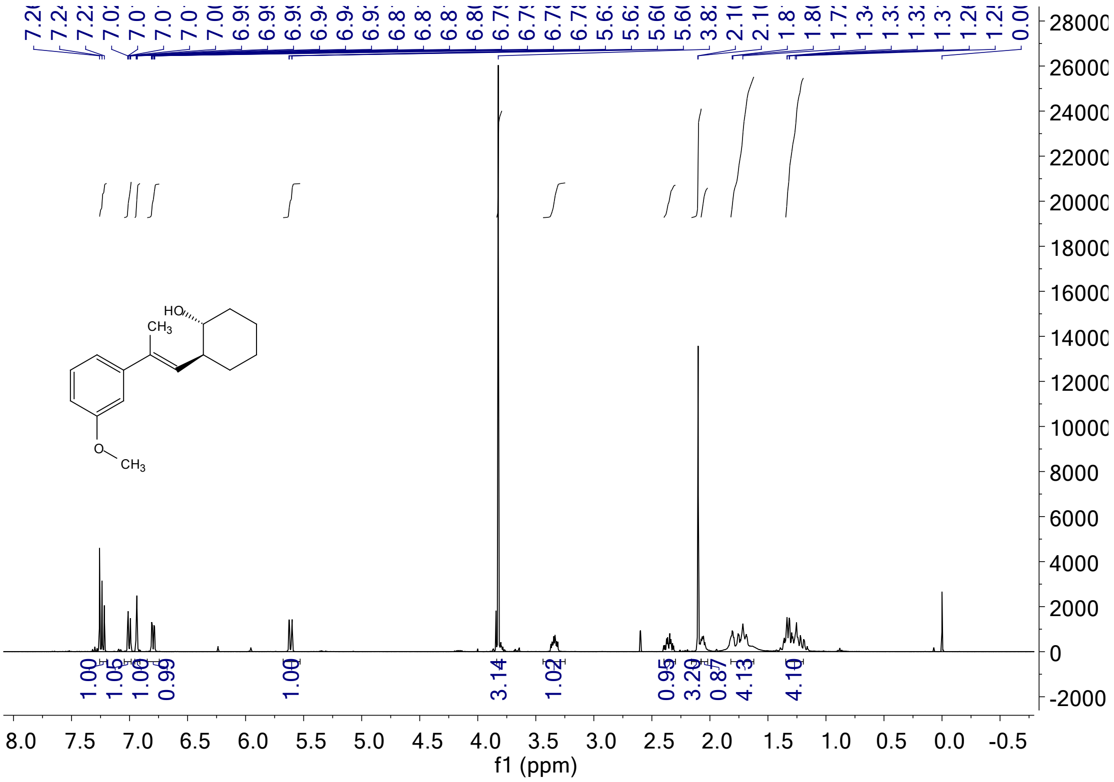

74.42
HO
CH3
210
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

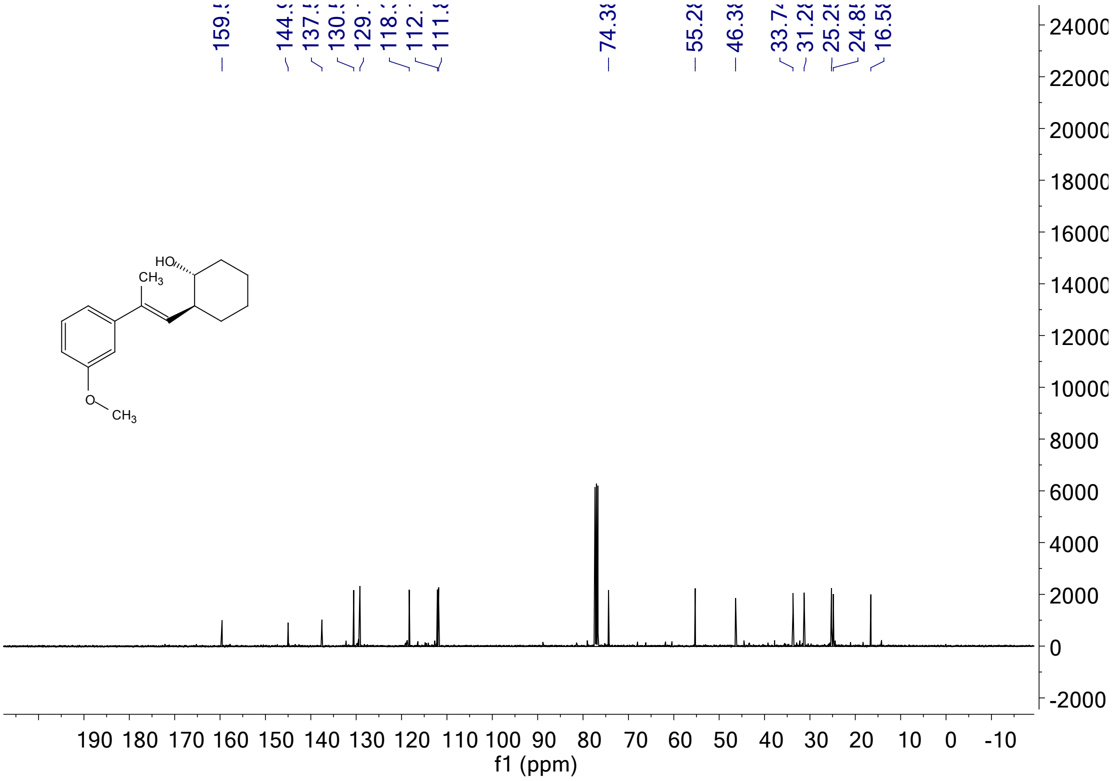


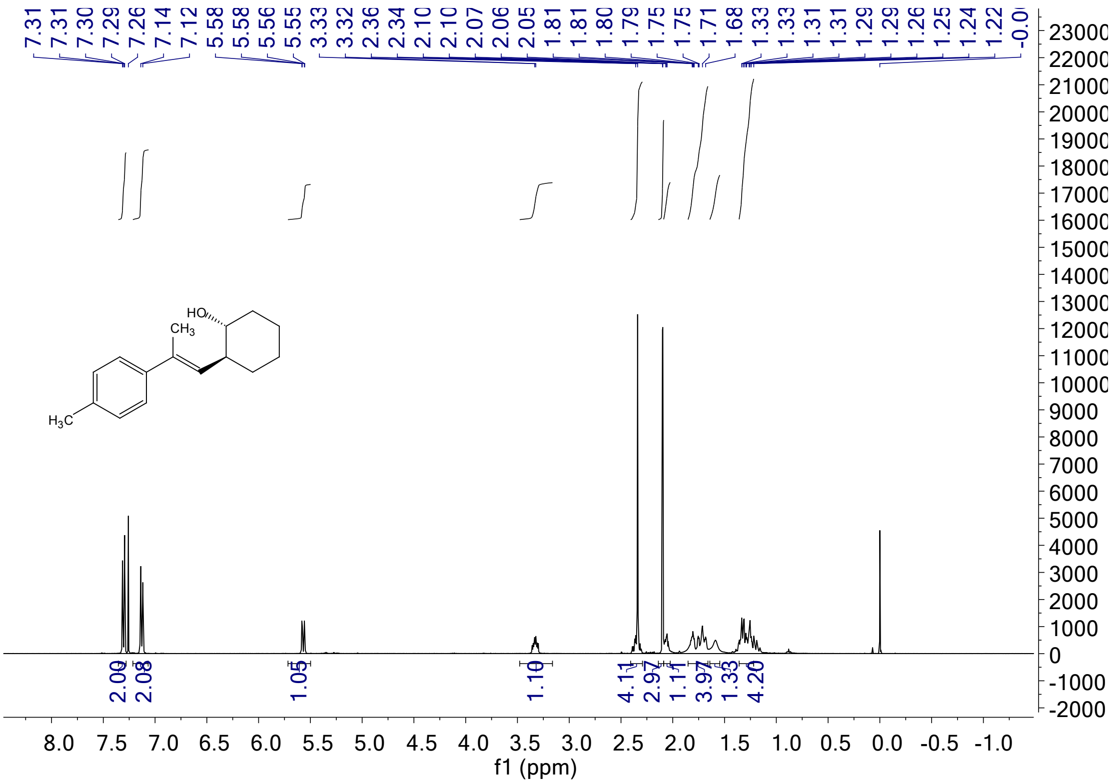
7.83
7.83
7.81
7.79
7.77
7.61
7.61
7.59
7.59
7.47
7.46
7.45
7.45
7.45
7.44
7.43
7.43
7.26
5.79
5.78
5.76
5.76
3.39
2.42
2.23
2.23
2.04
1.84
1.83
1.82
1.81
1.80
1.77
1.71
1.37
1.36
1.34
1.32
-0.00
HO
CH3
4.00
1.00
2.01
1.00
1.09
1.02
2.99
0.98
4.13
3.91
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

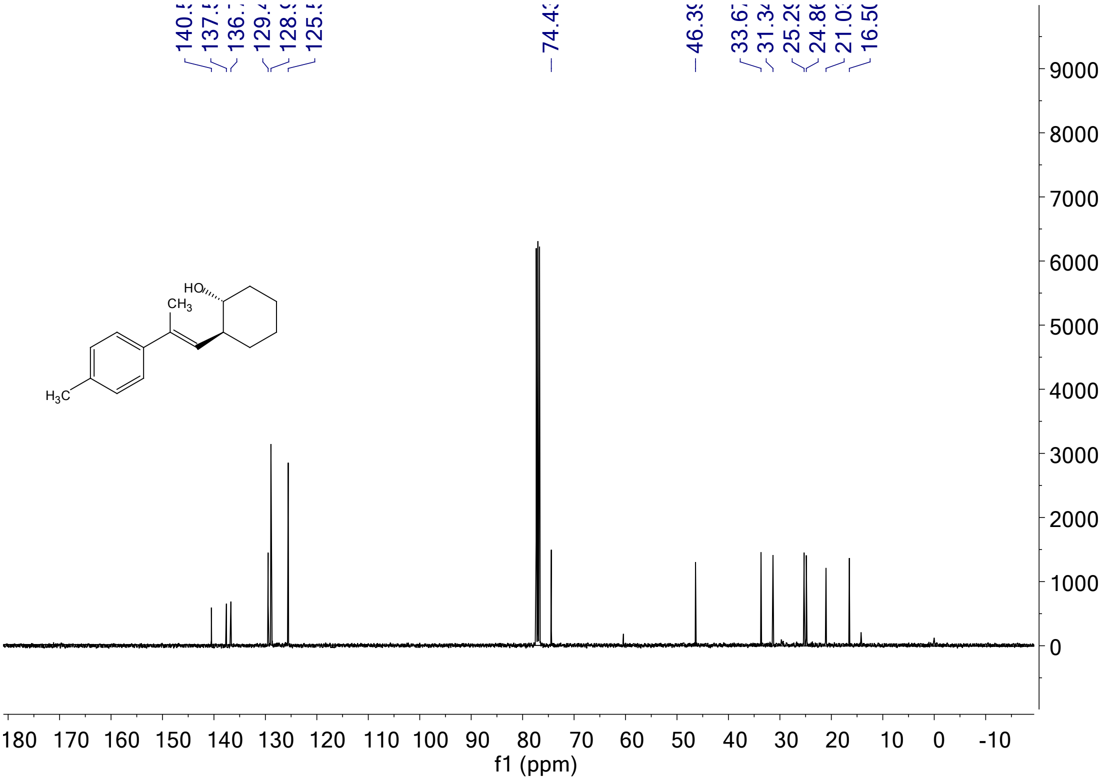


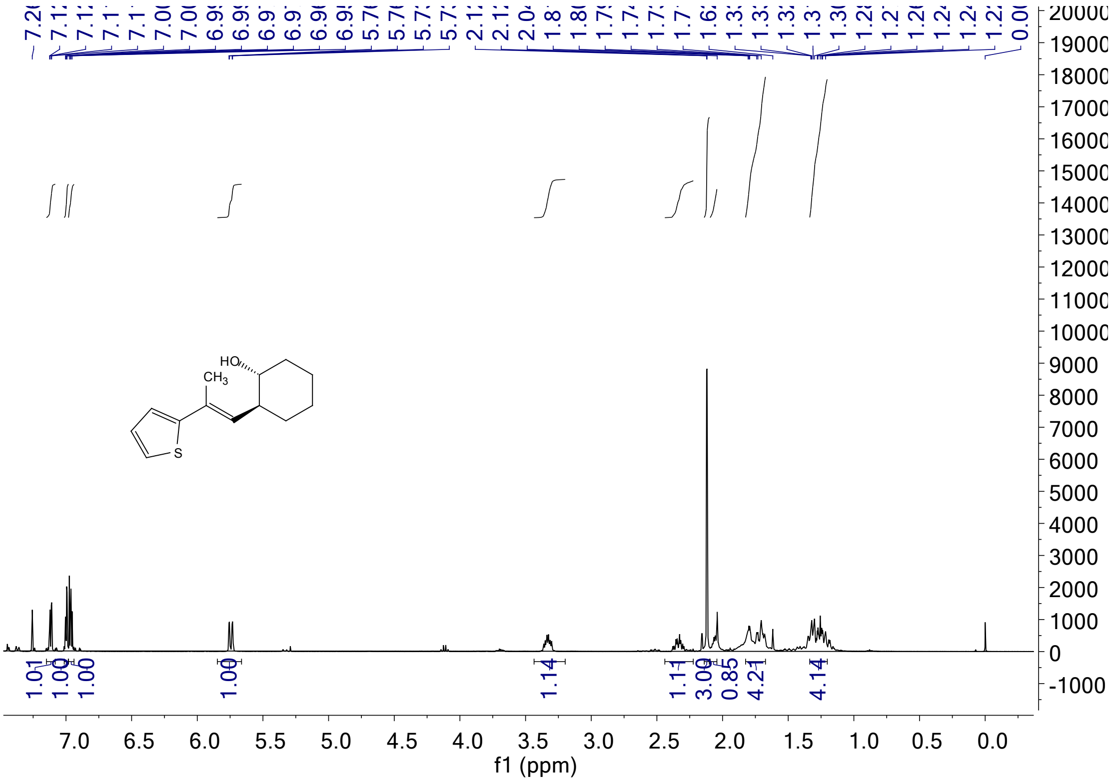
140.
137.
133.
132.
130.
128.
127.
127.
126.
125.
124.
124.
74.4
46.5
33.8
31.3
25.2
24.8
16.5
HO
CH3
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

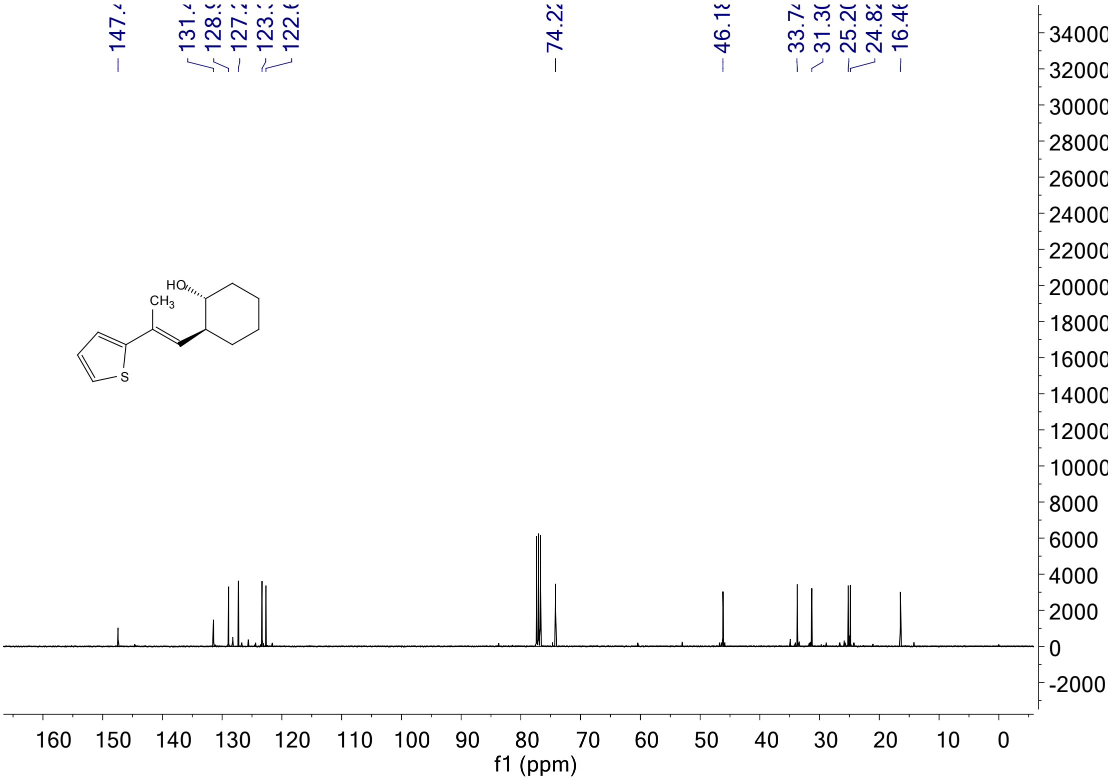

HO
CH3
O
CH3
1.00
1.05
1.00
0.99
1.00
3.14
1.02
0.95
3.20
0.87
4.13
4.10
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

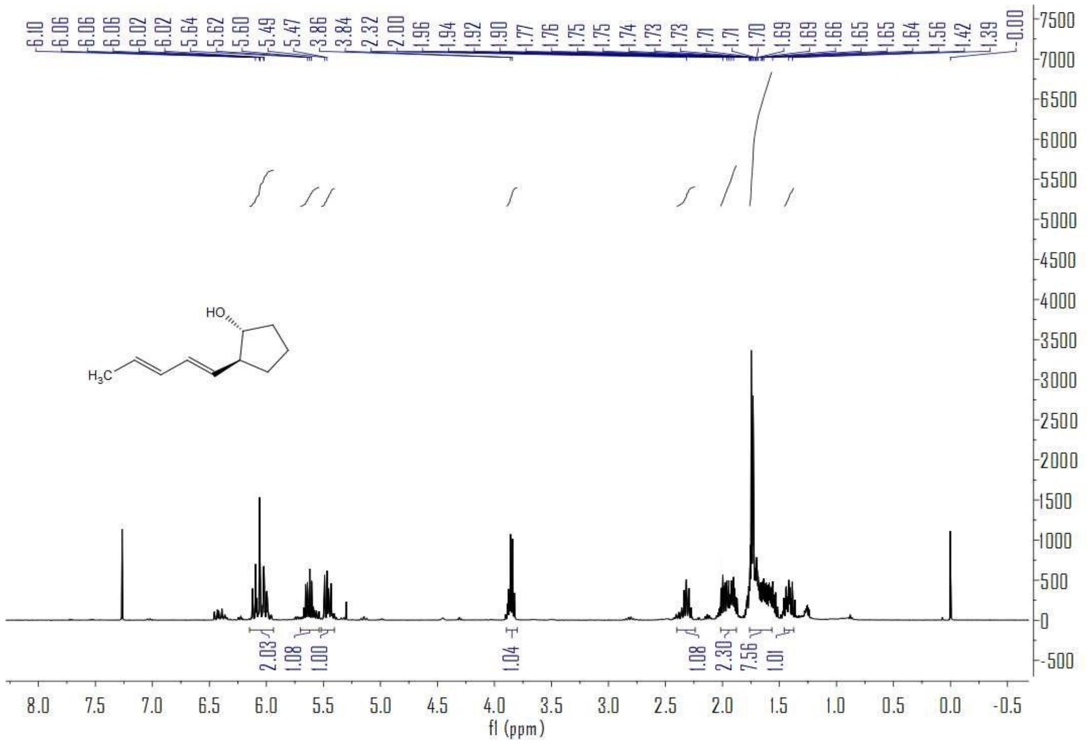

HO
CH3
O
CH3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

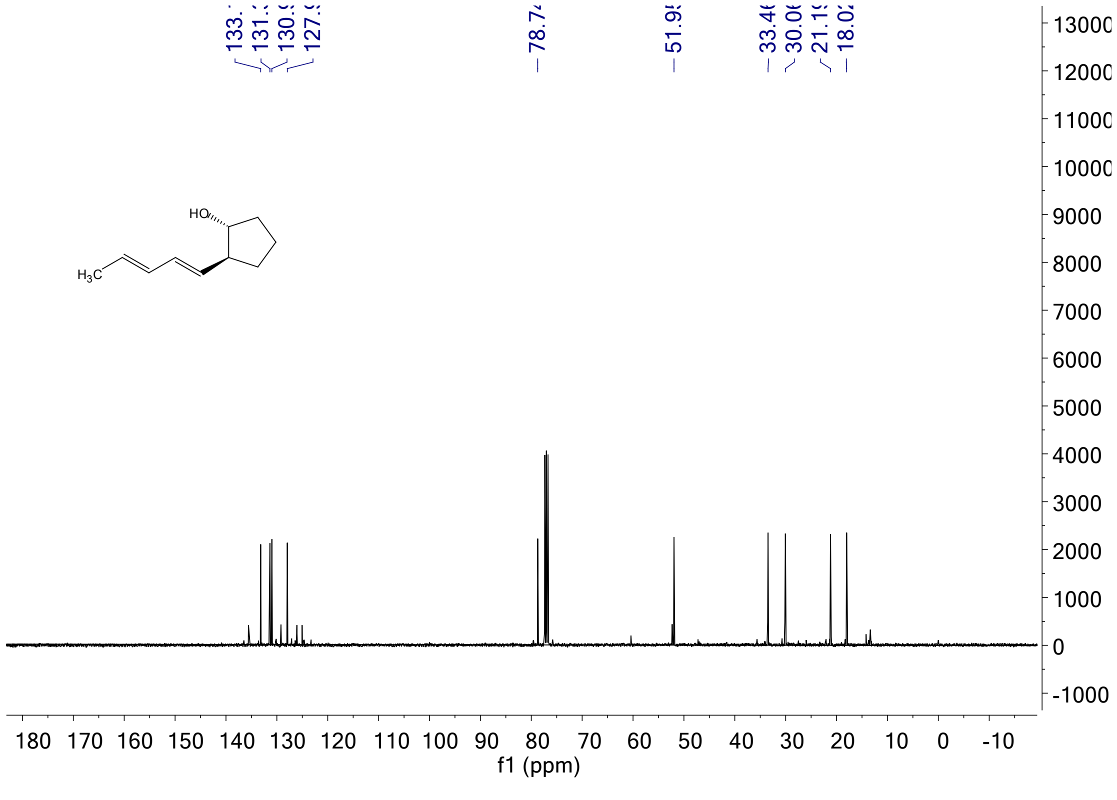

7.31
7.31
7.30
7.29
7.26
7.14
7.12
5.58
5.58
5.56
5.55
3.33
3.32
2.36
2.34
2.10
2.10
2.07
2.06
2.05
1.81
1.81
1.80
1.79
1.75
1.75
1.71
1.68
1.33
1.33
1.31
1.31
1.29
1.29
1.26
1.25
1.24
1.22
-0.0
HO
CH3
H3C
2.00
2.08
1.05
1.10
4.1
2.97
1.1
3.97
1.33
4.20
23000
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

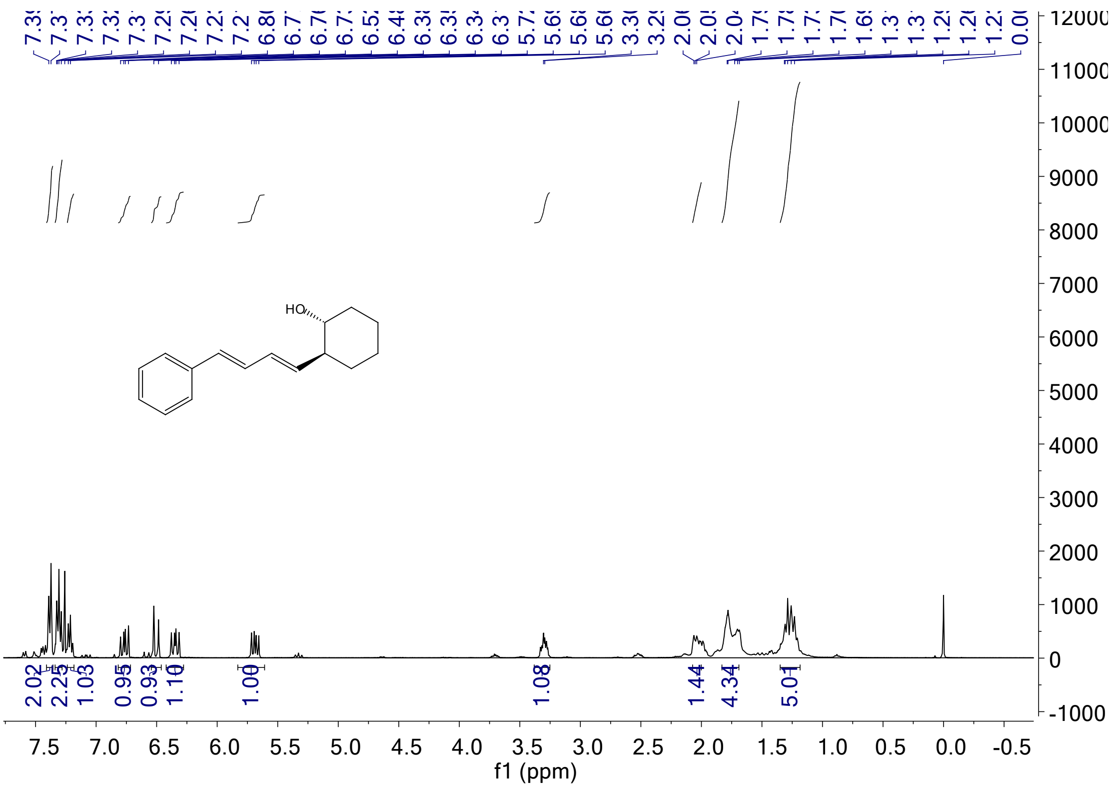

140.
137.
136.
129.
128.
125.
74.4
46.3
33.6
31.3
25.2
24.8
21.0
16.5
HO
CH3
H3C
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

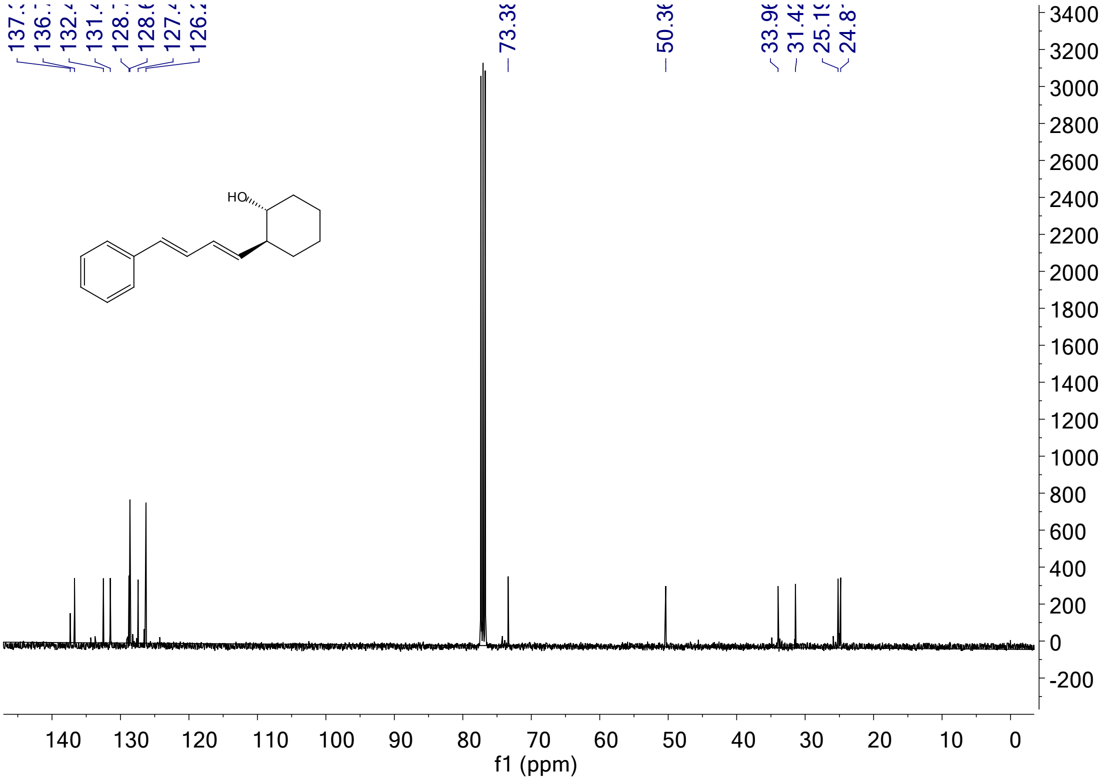

HO
CH3
S
1.01
1.00
1.00
1.00
1.14
1.11
3.00
0.85
4.21
4.14
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

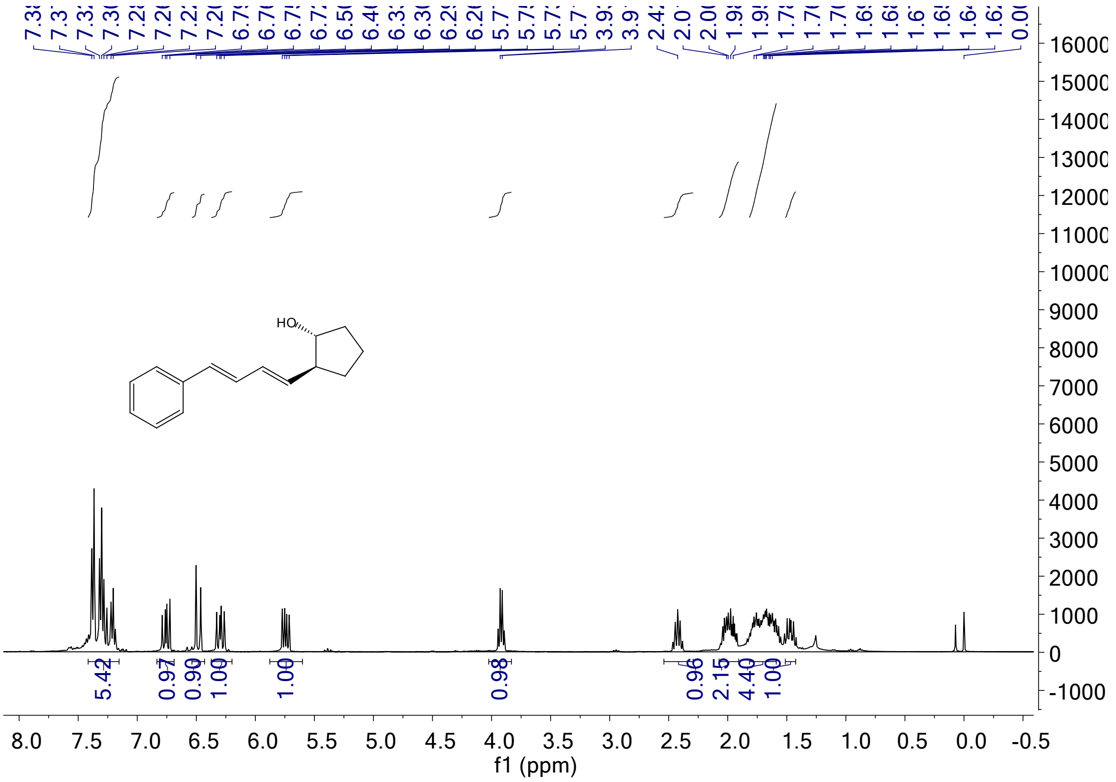

147.4
131.4
128.9
127.2
123.3
122.6
74.22
46.18
33.74
31.30
25.20
24.82
16.46
HO
CH3
S
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000

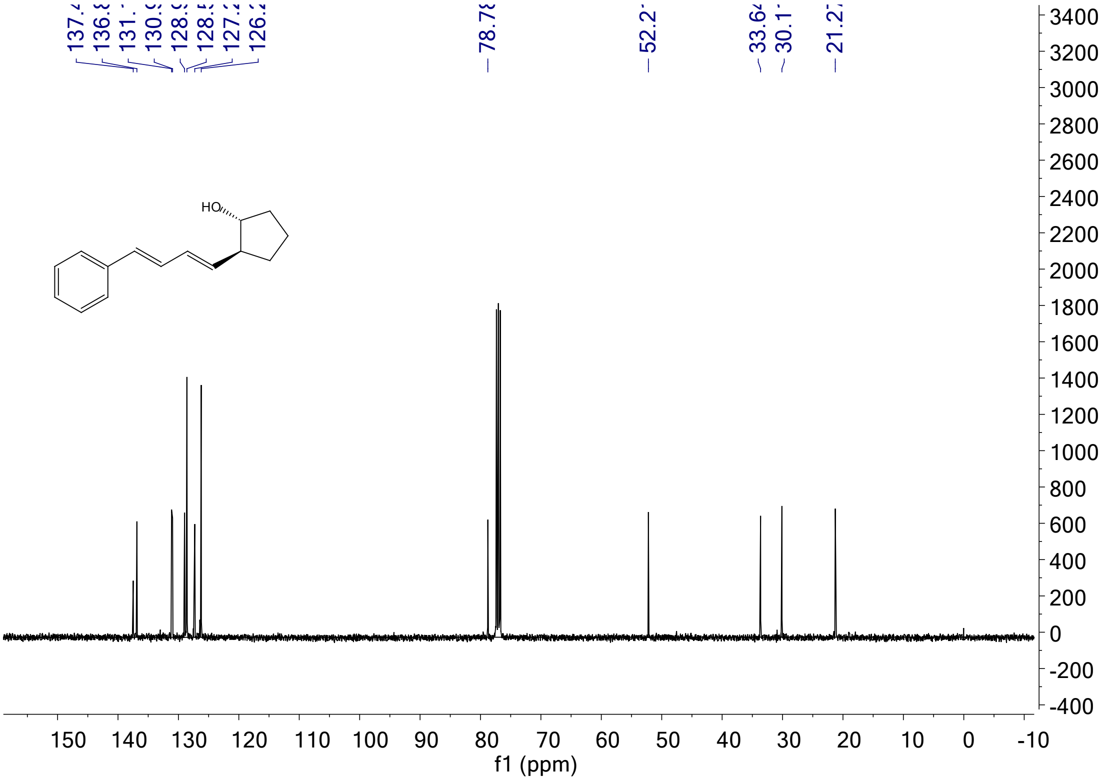

6.10
6.06
6.06
6.06
6.02
6.02
5.64
5.62
5.60
5.49
5.47
3.86
3.84
2.32
2.00
1.96
1.94
1.92
1.90
1.77
1.76
1.75
1.75
1.74
1.73
1.73
1.71
1.71
1.70
1.69
1.69
1.66
1.65
1.65
1.64
1.56
1.42
1.39
0.00
HO
H3C
2.03
1.08
1.00
1.04
1.08
2.30
7.56
1.01
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

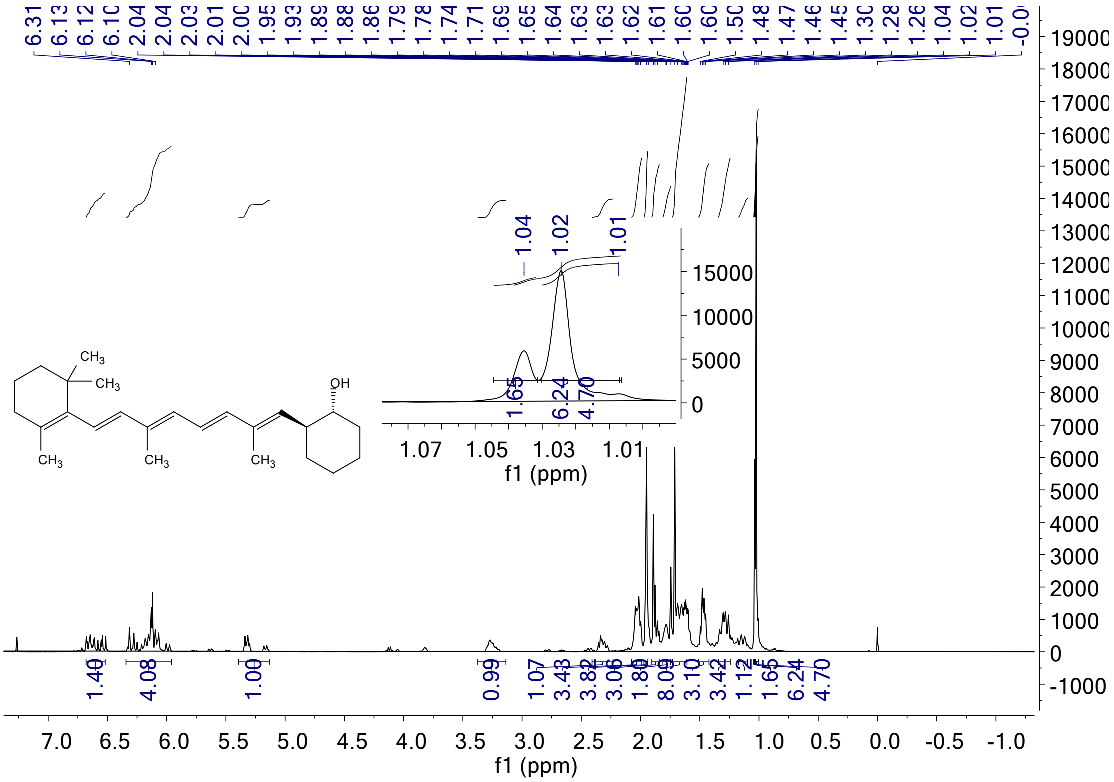

133.
131.
130.
127.
78.7
51.9
33.4
30.0
21.1
18.0
HO
H3C
1300
1200
1100
1000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

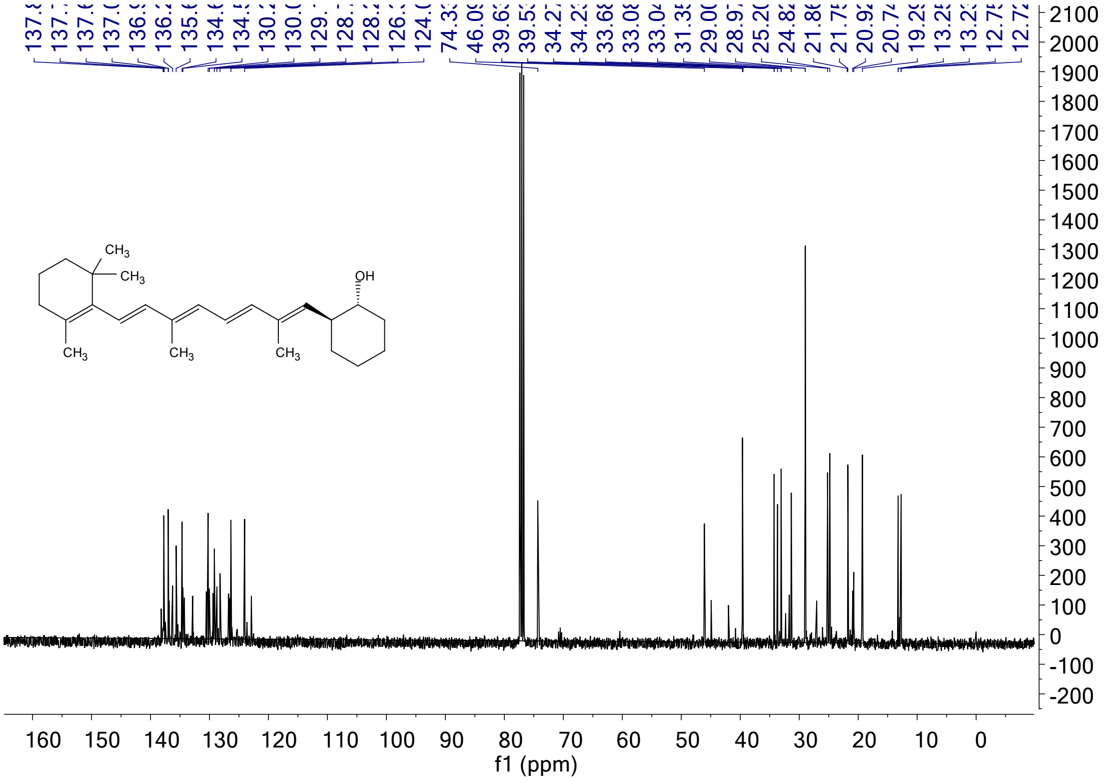

7.3
7.3
7.3
7.3
7.3
7.2
7.2
7.2
7.2
6.8
6.7
6.7
6.7
6.5
6.4
6.3
6.3
6.3
6.3
5.7
5.6
5.6
3.3
3.2
2.0
2.0
2.0
1.7
1.7
1.7
1.7
1.6
1.3
1.3
1.2
1.2
1.2
0.0
HO
2.02
2.25
1.03
0.95
0.93
1.10
1.00
1.08
1.44
4.34
5.01
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000

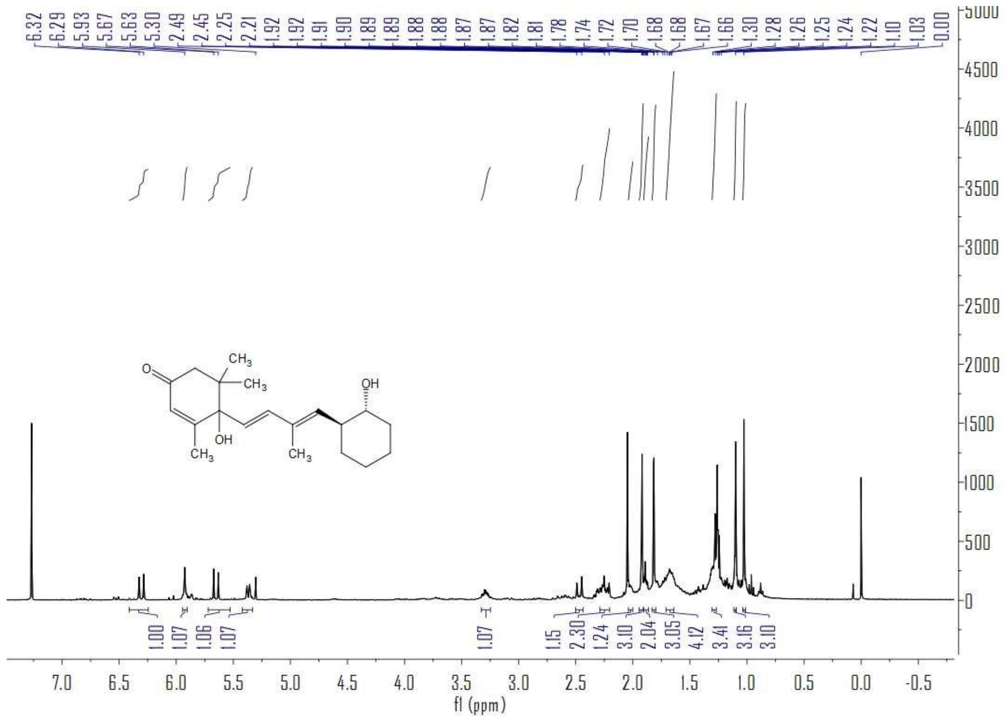

137.
136.
132.4
131.4
128.
128.
127.4
126.2
73.38
50.3
33.9
31.42
25.1
24.8
HO
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
3400 3200 3000 2800 2600 2400 2200 2000 1800 1600 1400 1200 1000 800 600 400 200 0 -200

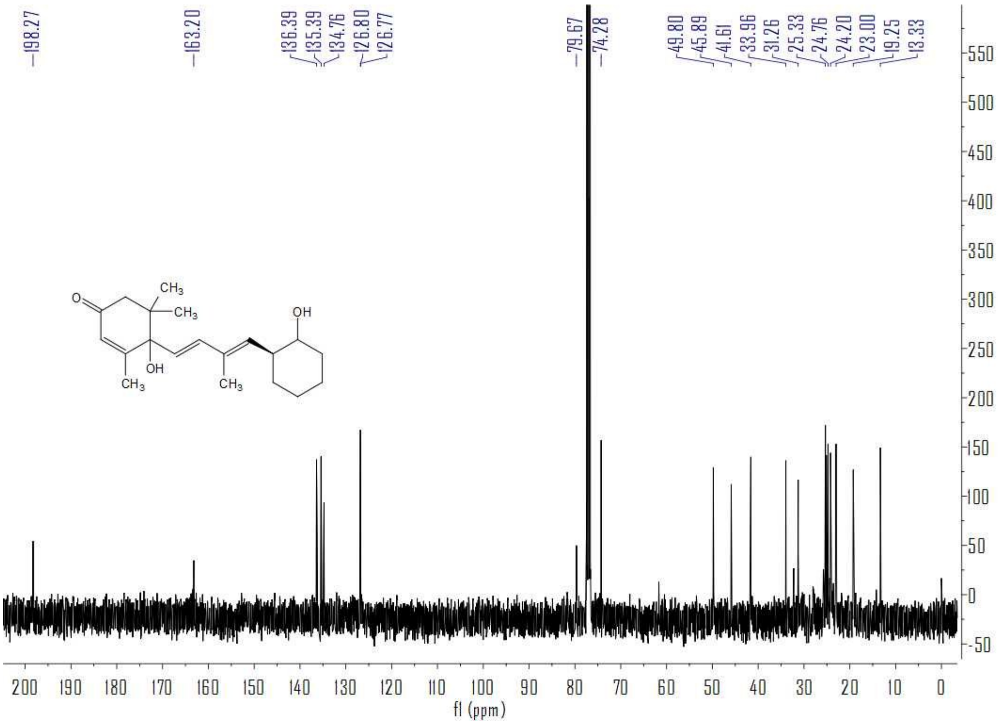

7.3
7.3
7.3
7.3
7.2
7.2
7.2
7.2
6.7
6.7
6.7
6.7
6.5
6.4
6.3
6.3
6.2
6.2
5.7
5.7
5.7
5.7
3.9
3.9
2.4
2.0
2.0
1.9
1.9
1.7
1.7
1.7
1.6
1.6
1.6
1.6
1.6
1.6
0.0
HO
5.42
0.97
0.90
1.00
1.00
0.98
0.96
2.15
4.40
1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000

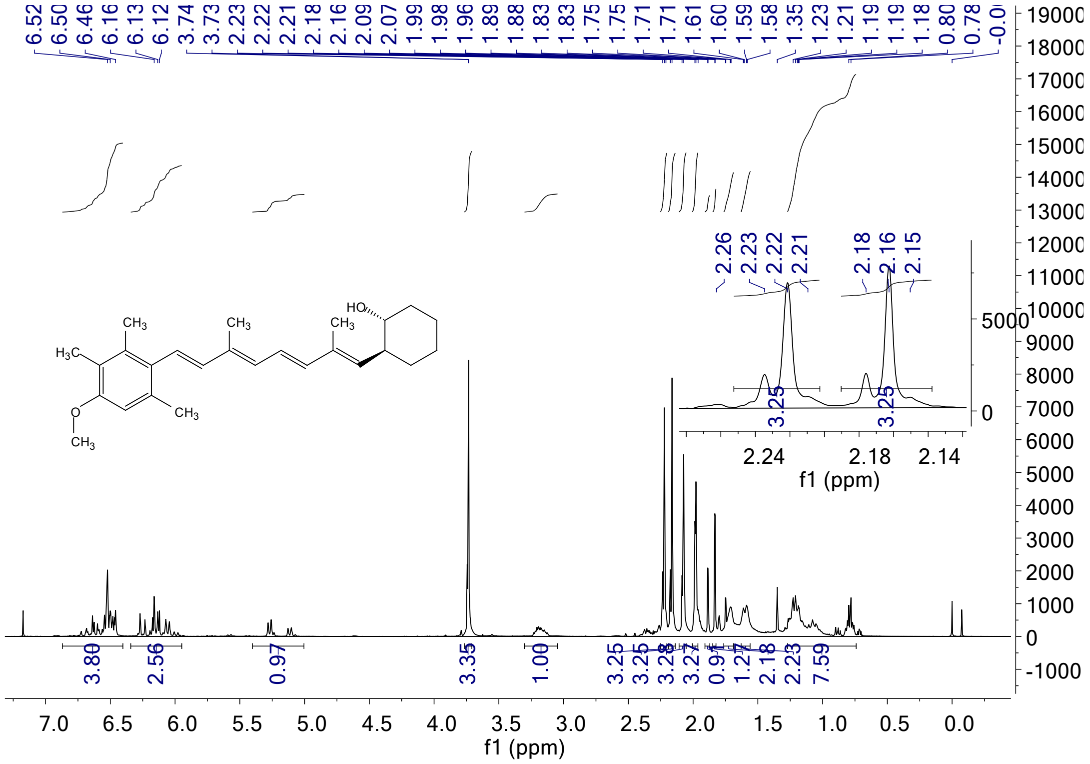

137.
136.
131.
130.
128.
128.
127.
126.
78.7
52.2
33.64
30.1
21.2
HO
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
3400 3200 3000 2800 2600 2400 2200 2000 1800 1600 1400 1200 1000 800 600 400 200 0 -200 -400

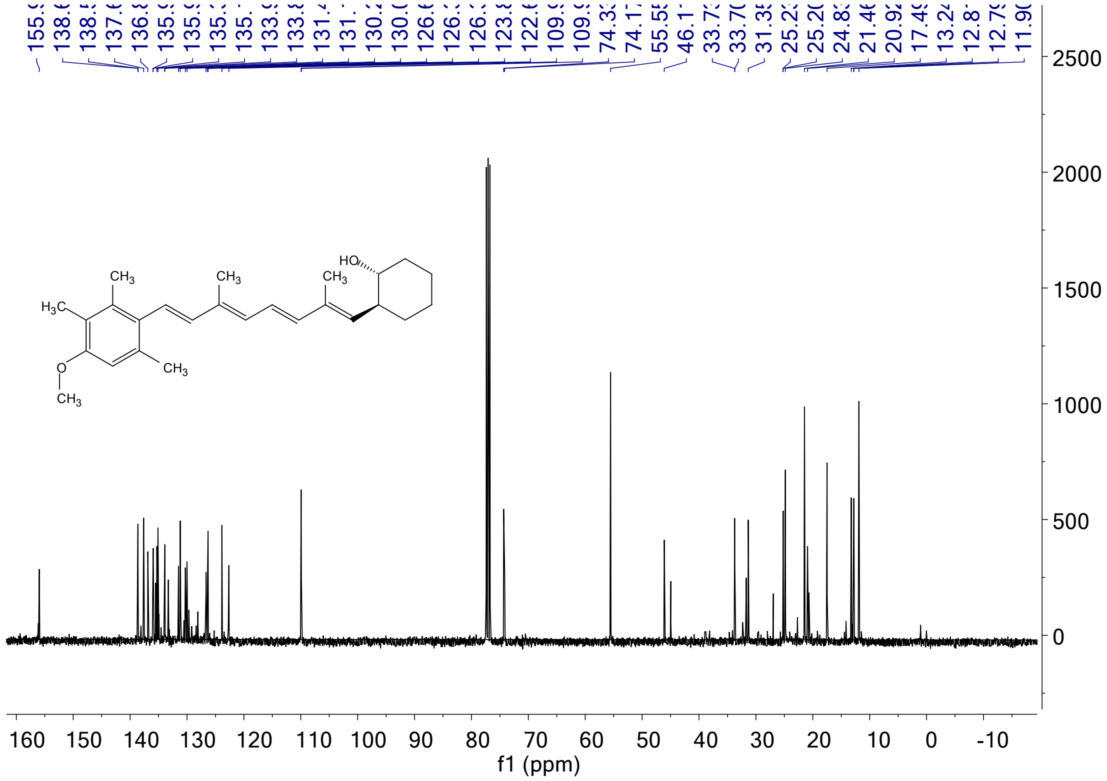

6.31
6.13
6.12
6.10
2.04
2.04
2.03
2.01
2.00
1.95
1.93
1.89
1.88
1.86
1.79
1.78
1.74
1.71
1.69
1.65
1.64
1.63
1.63
1.62
1.61
1.60
1.60
1.50
1.48
1.47
1.46
1.45
1.30
1.28
1.26
1.04
1.02
1.01
-0.0
CH3
CH3
OH
CH3
CH3
CH3
1.04
1.02
1.01
1.65
6.24
4.70
1.07
1.05
1.03
1.01
f1 (ppm)
15000
10000
5000
0
1.40
4.08
1.00
0.99
1.07
3.43
3.82
3.06
1.80
8.09
3.10
3.42
1.12
1.65
6.24
4.70
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

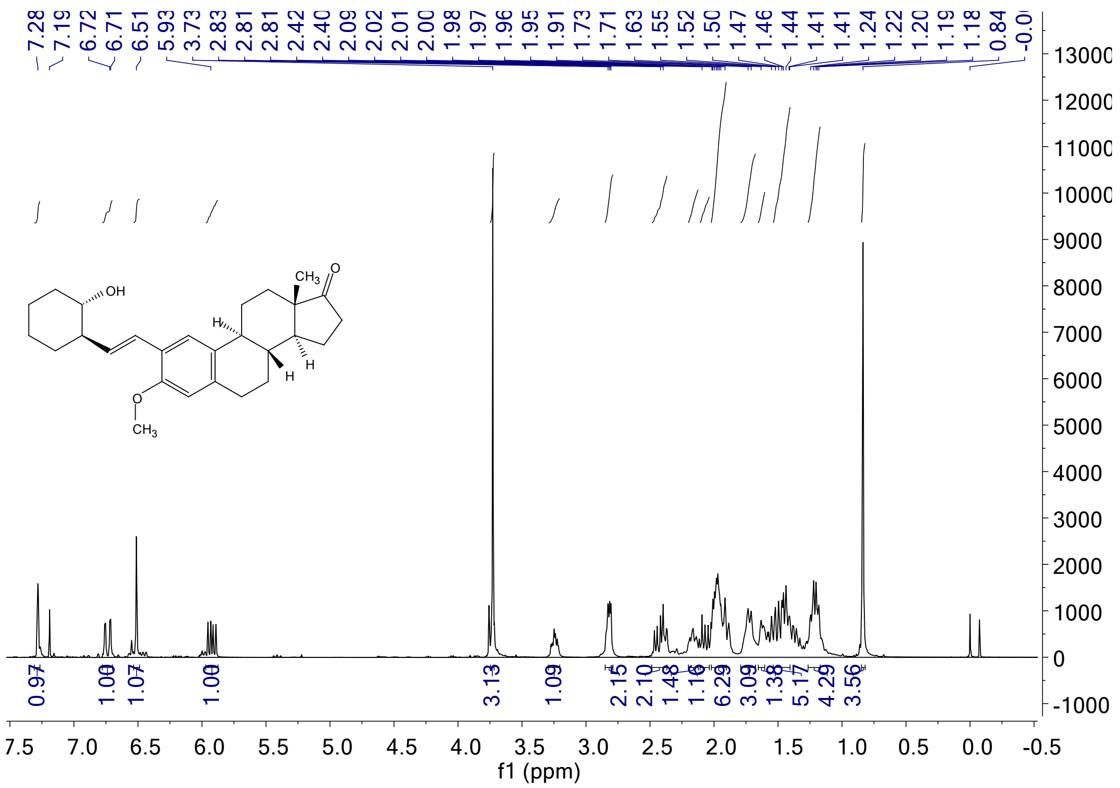

137.
137.
137.
137.
136.
136.
135.
134.
134.
130.
130.
129.
128.
128.
126.
124.
74.33
46.09
39.63
39.53
34.27
34.23
33.68
33.08
33.04
31.39
29.00
28.97
25.20
24.82
21.86
21.75
20.92
20.74
19.29
13.25
13.23
12.75
12.72
CH3
CH3
OH
CH3
CH3
CH3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
-200

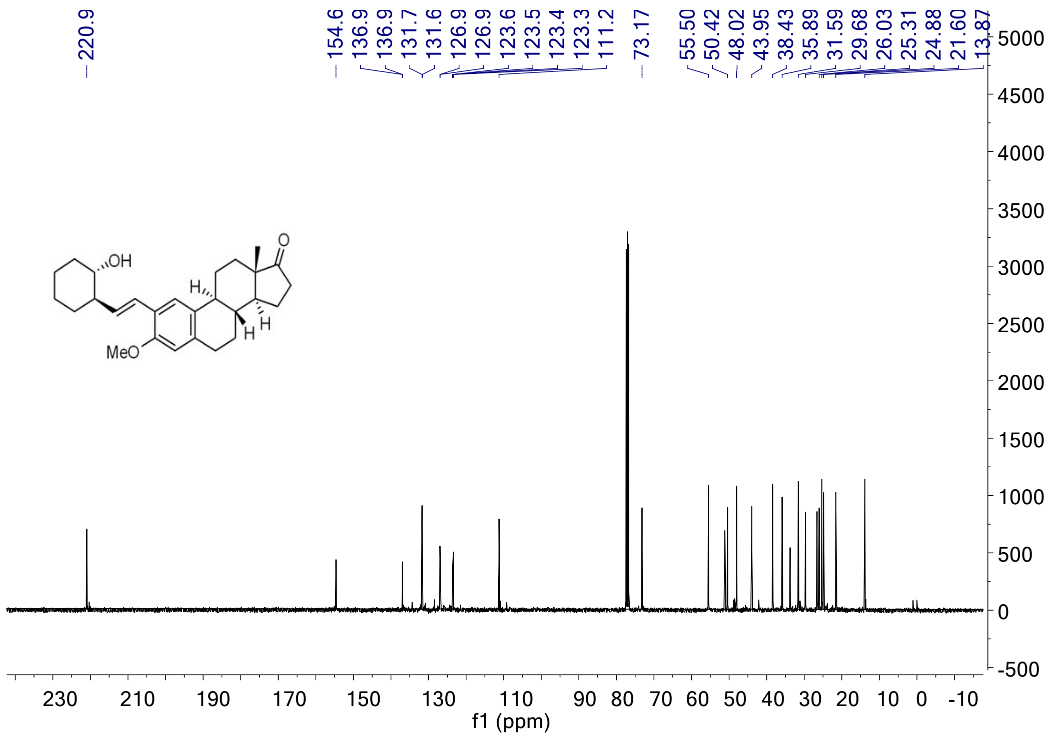

6.32
6.29
5.93
5.67
5.63
5.30
2.49
2.45
2.25
2.21
1.92
1.92
1.91
1.90
1.89
1.89
1.88
1.88
1.87
1.87
1.82
1.81
1.78
1.74
1.72
1.70
1.68
1.68
1.67
1.66
1.30
1.28
1.26
1.25
1.24
1.22
1.10
1.03
0.00
1.00
1.07
1.06
1.07
1.07
1.15
2.30
1.24
3.10
2.04
3.05
4.12
3.41
3.16
3.10
O
CH3
CH3
OH
OH
CH3
CH3
fl (ppm)

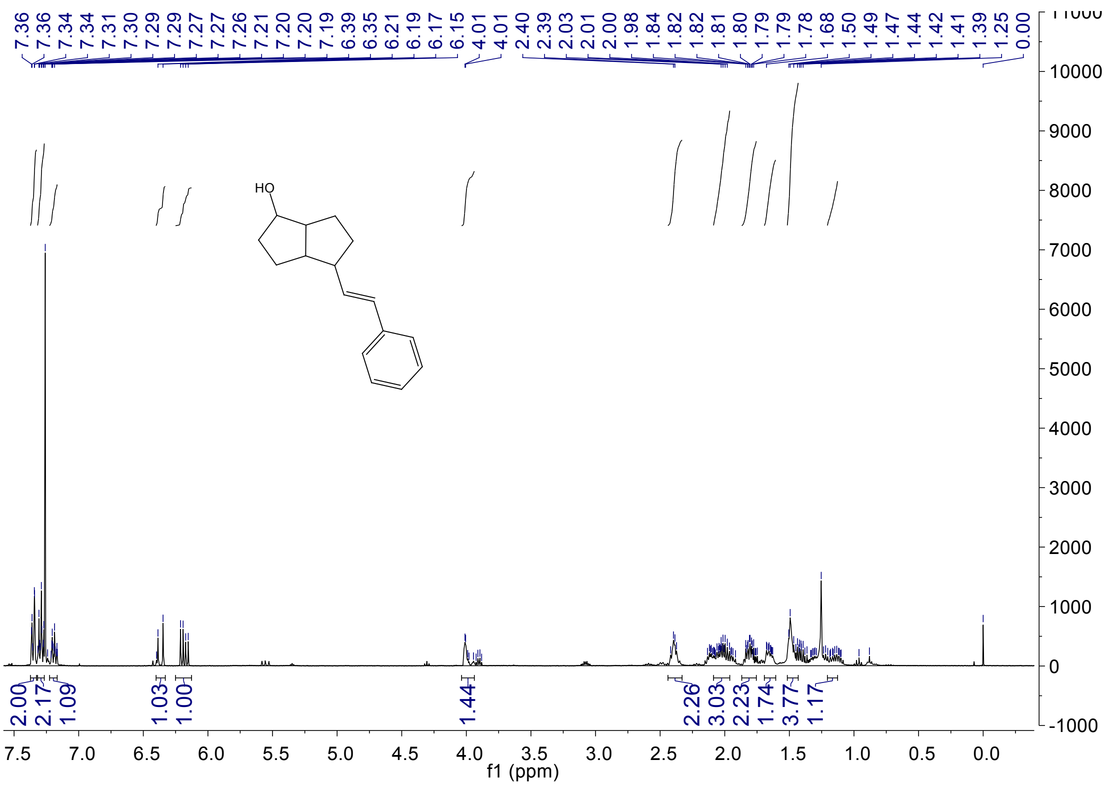

198.27
163.20
136.39
135.39
134.76
126.80
126.77
79.67
74.28
49.80
45.89
41.61
33.96
31.26
25.33
24.76
24.20
23.00
19.25
13.33
O
CH3
CH3
OH
OH
CH3
CH3
fl (ppm)

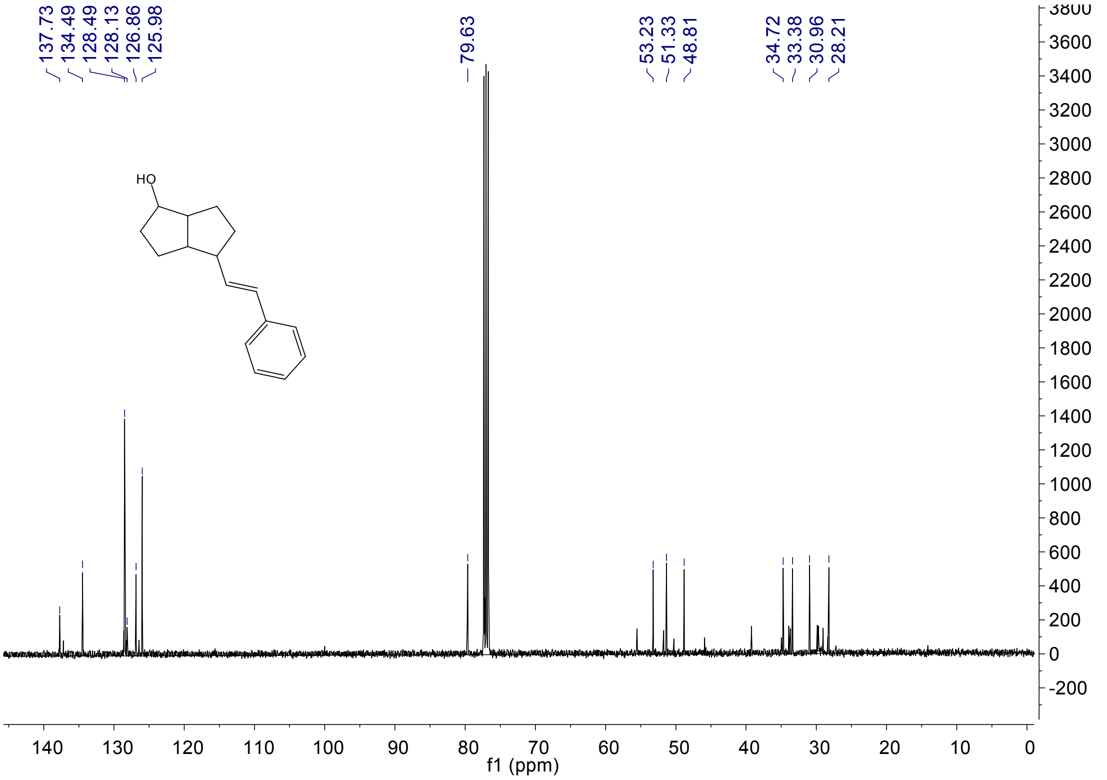

6.52
6.50
6.46
6.16
6.13
6.12
3.74
3.73
2.23
2.22
2.21
2.18
2.16
2.09
2.07
1.99
1.98
1.96
1.89
1.88
1.83
1.83
1.75
1.75
1.71
1.71
1.61
1.60
1.59
1.58
1.35
1.23
1.21
1.19
1.19
1.18
0.80
0.78
-0.0
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
2.26
2.23
2.22
2.21
2.18
2.16
2.15
5000
0
3.25
3.25
2.24
2.18
2.14
f1 (ppm)
HO
CH3
CH3
CH3
H3C
CH3
O
CH3
3.80
2.56
0.97
3.35
1.00
3.25
3.25
3.28
3.27
0.97
1.27
2.18
2.23
7.59
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

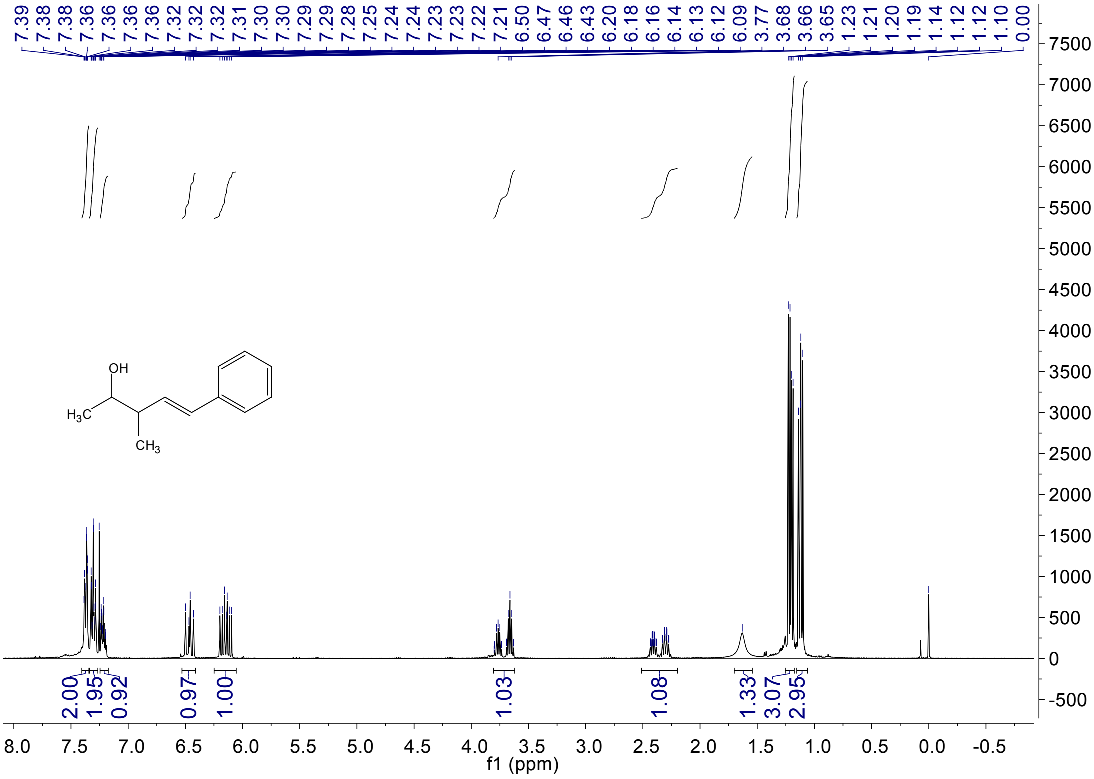

155.9
138.6
138.5
137.6
136.8
135.9
135.9
135.3
135.1
133.9
133.8
131.4
131.1
130.2
130.0
126.6
126.3
126.3
123.8
122.6
109.9
109.9
74.33
74.17
55.55
46.17
33.73
33.70
31.35
25.23
25.20
24.83
21.46
20.92
17.49
13.24
12.87
12.79
11.90
2500
2000
1500
1000
500
0
HO
CH3
CH3
CH3
H3C
CH3
O
CH3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

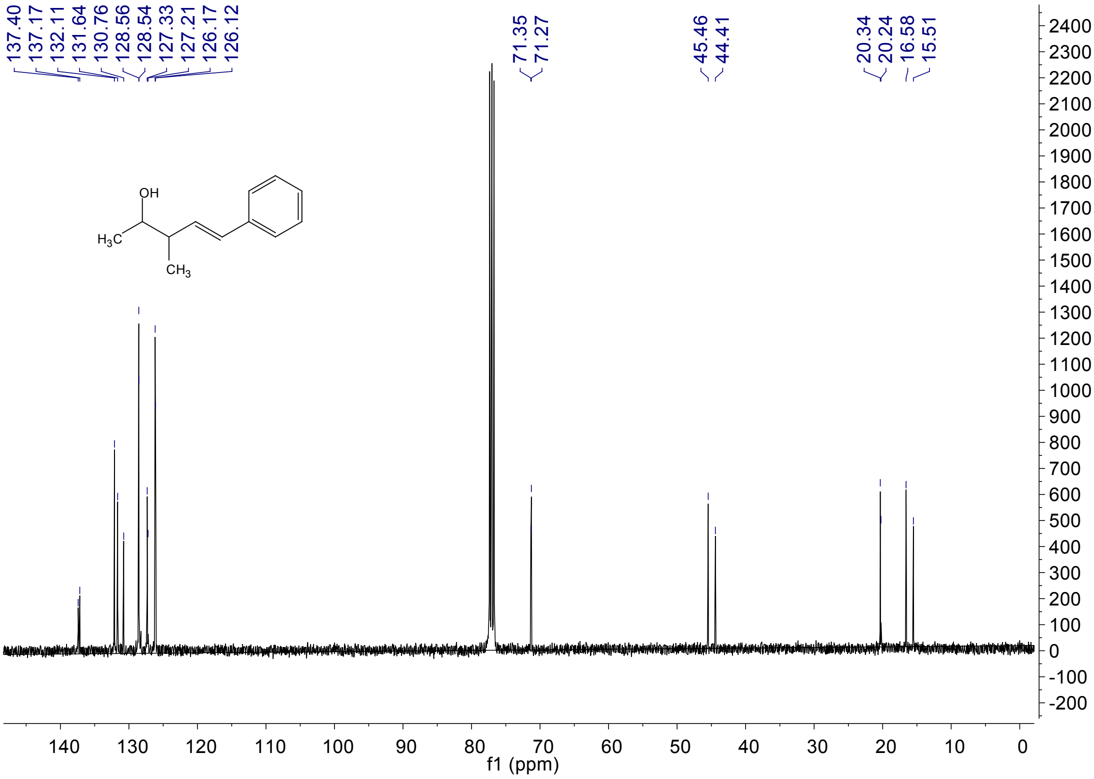

7.28
7.19
6.72
6.71
6.51
5.93
3.73
2.83
2.81
2.81
2.42
2.40
2.09
2.02
2.01
2.00
1.98
1.97
1.96
1.95
1.91
1.73
1.71
1.63
1.55
1.52
1.50
1.47
1.46
1.44
1.41
1.41
1.24
1.22
1.20
1.19
1.18
0.84
-0.0
CH3
O
OH
H
H
H
O
CH3
0.97
1.00
1.07
1.00
3.13
1.09
2.15
2.10
1.48
1.16
6.29
3.09
1.38
5.17
4.29
3.56
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

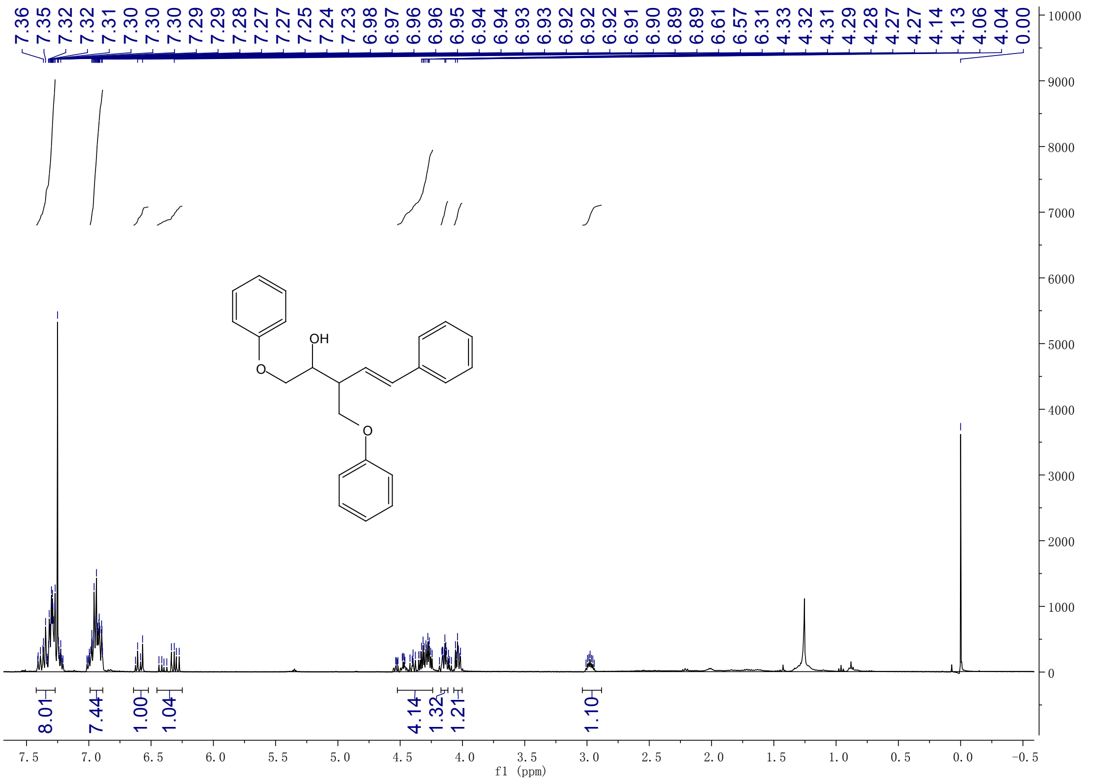

220.9
154.6
136.9
136.9
131.7
131.6
126.9
126.9
123.6
123.5
123.4
123.3
111.2
73.17
55.50
50.42
48.02
43.95
38.43
35.89
31.59
29.68
26.03
25.31
24.88
21.60
13.87
OH
O
H
H
H
MeO
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

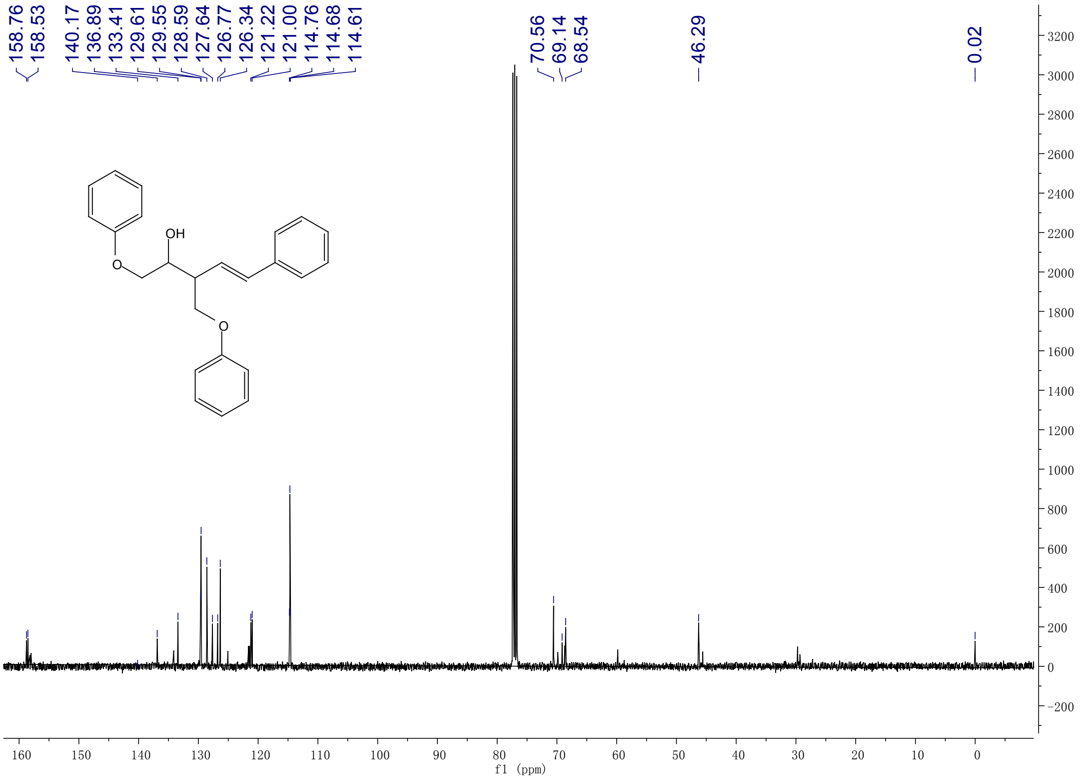

7.36
7.36
7.34
7.34
7.31
7.30
7.29
7.29
7.27
7.26
7.21
7.20
7.20
7.19
6.39
6.35
6.21
6.19
6.17
6.15
4.01
4.01
2.40
2.39
2.03
2.01
2.00
1.98
1.84
1.82
1.82
1.81
1.80
1.79
1.79
1.78
1.68
1.50
1.49
1.47
1.44
1.42
1.41
1.39
1.25
0.00
HO
2.00
2.17
1.09
1.03
1.00
1.44
2.26
3.03
2.23
1.74
3.77
1.17
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

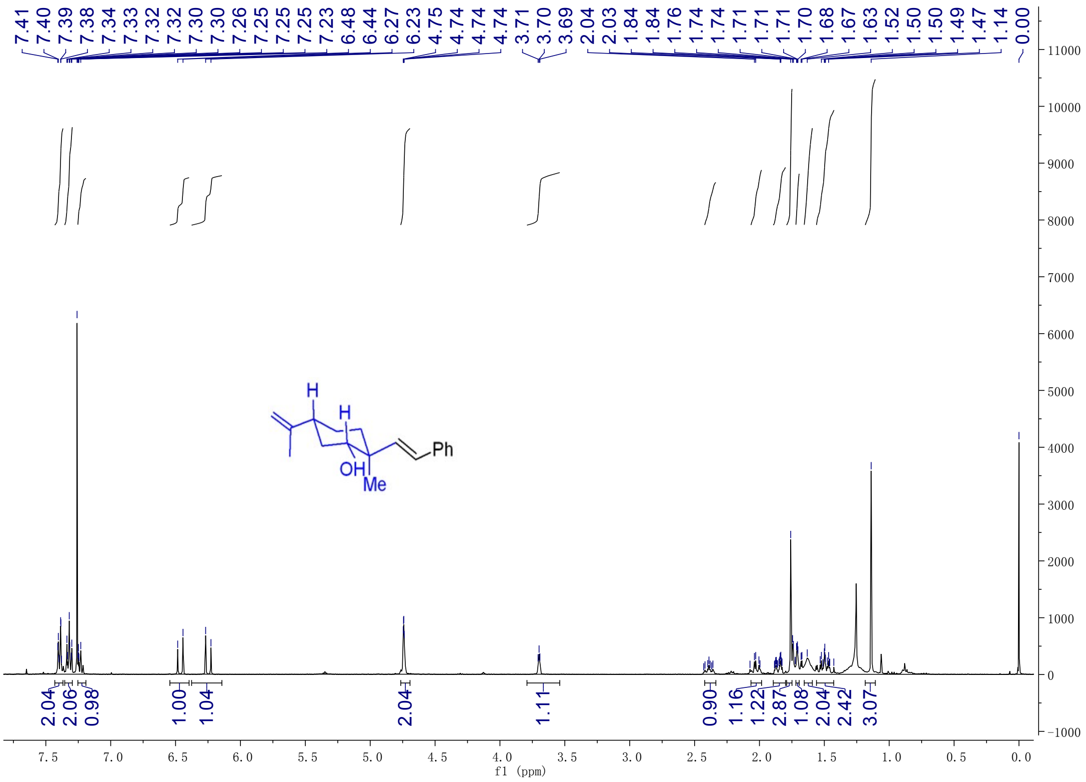

137.73
134.49
128.49
128.13
126.86
125.98
79.63
53.23
51.33
48.81
34.72
33.38
30.96
28.21
HO
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

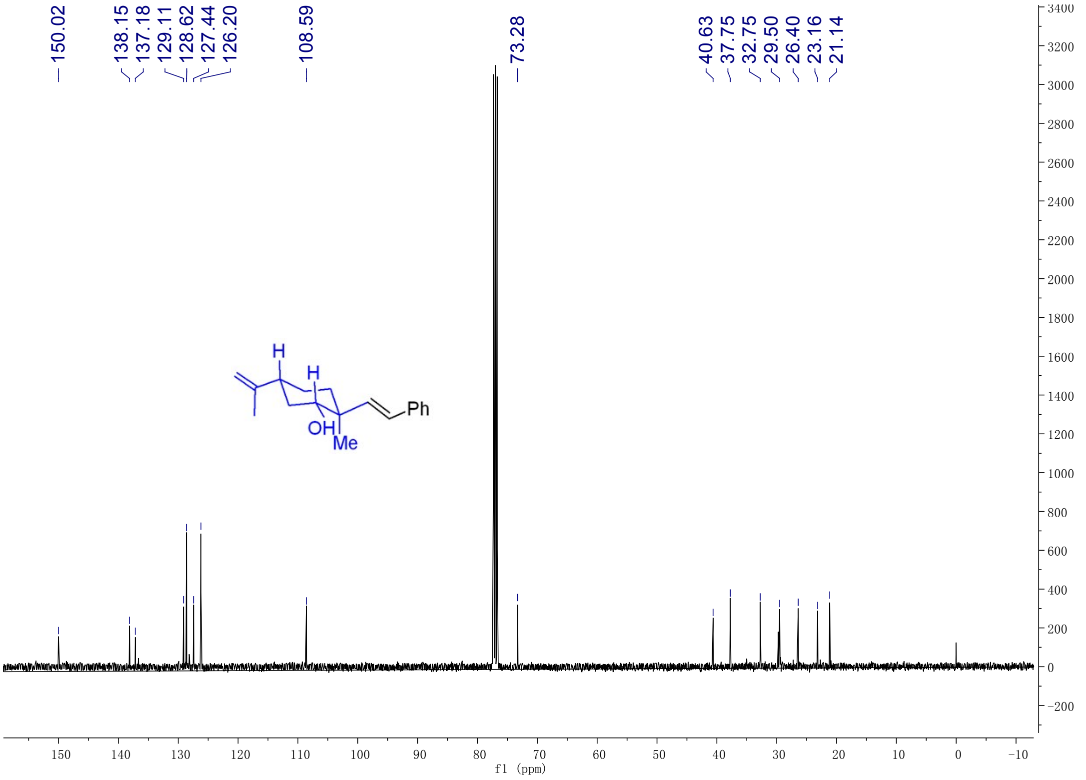

7.39
7.38
7.38
7.36
7.36
7.36
7.32
7.32
7.32
7.31
7.30
7.30
7.29
7.29
7.28
7.25
7.24
7.24
7.23
7.23
7.22
7.21
6.50
6.47
6.46
6.43
6.20
6.18
6.16
6.14
6.13
6.12
6.09
3.77
3.68
3.66
3.65
1.23
1.21
1.20
1.19
1.14
1.12
1.12
1.10
0.00
OH
H3C
CH3
2.00
1.95
0.92
0.97
1.00
1.03
1.08
1.33
3.07
2.95
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500

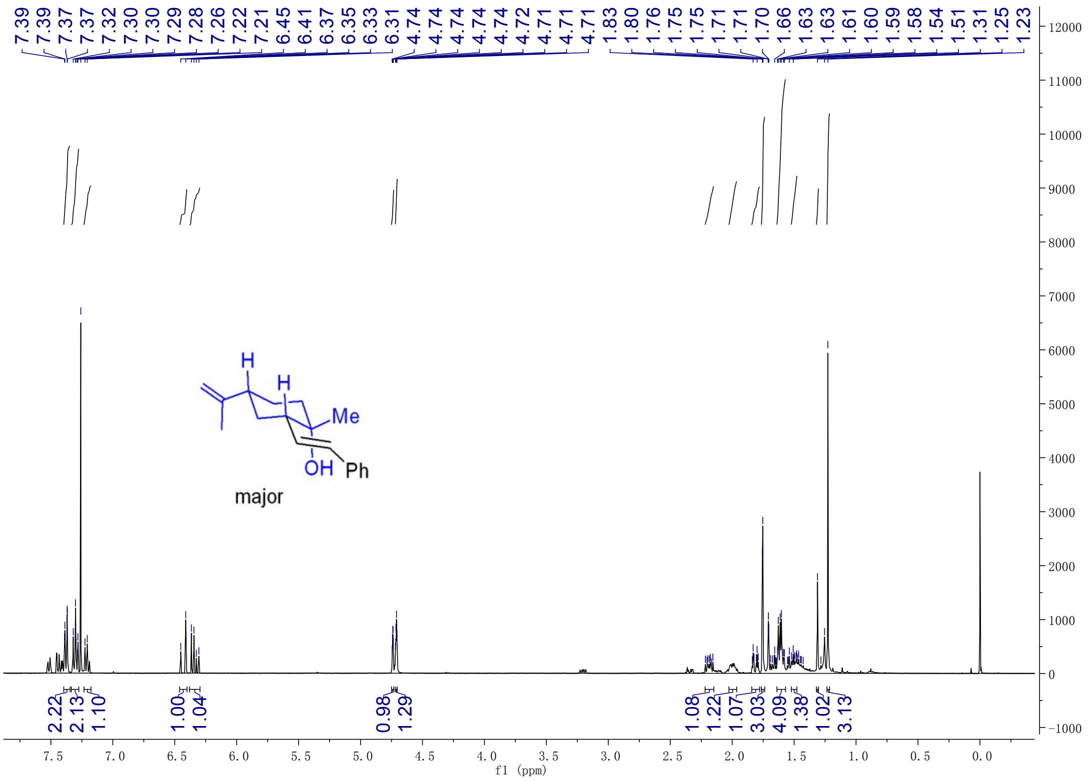

137.40
137.17
132.11
131.64
130.76
128.56
128.54
127.33
127.21
126.17
126.12
71.35
71.27
45.46
44.41
20.34
20.24
16.58
15.51
OH
H3C
CH3
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
2400
2300
2200
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
-200

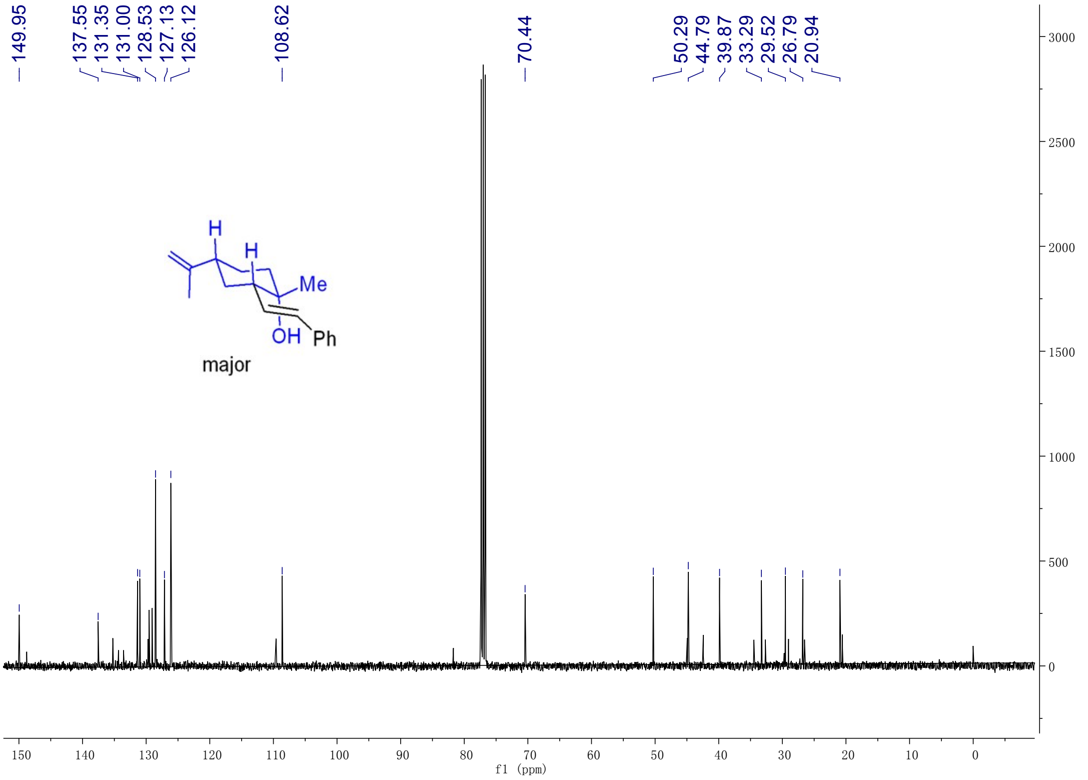

7.36
7.35
7.32
7.32
7.31
7.30
7.30
7.30
7.29
7.29
7.28
7.27
7.27
7.25
7.24
7.23
6.98
6.97
6.96
6.96
6.95
6.94
6.94
6.93
6.93
6.92
6.92
6.92
6.91
6.90
6.89
6.89
6.61
6.57
6.31
4.33
4.32
4.31
4.29
4.28
4.27
4.27
4.14
4.13
4.06
4.04
0.00
OH
O
O
8.01
7.44
1.00
1.04
4.14
1.32
1.21
1.10
f1 (ppm)


158.76
158.53
140.17
136.89
133.41
129.61
129.55
128.59
127.64
126.77
126.34
121.22
121.00
114.76
114.68
114.61
70.56
69.14
68.54
46.29
-0.02
OH
O
O
f1 (ppm)


7.41
7.40
7.39
7.38
7.34
7.33
7.32
7.32
7.30
7.26
7.25
7.25
7.25
7.23
6.48
6.44
6.27
6.23
4.75
4.74
4.74
4.74
3.71
3.70
3.69
2.04
2.03
1.84
1.76
1.74
1.74
1.71
1.71
1.71
1.70
1.68
1.67
1.63
1.52
1.50
1.50
1.49
1.47
1.14
0.00
H
H
Ph
OH
Me
2.04
2.06
0.98
1.00
1.04
2.04
1.11
0.90
1.16
1.22
2.87
1.08
2.04
2.42
3.07
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


150.02
138.15
137.18
129.11
128.62
127.44
126.20
108.59
73.28
40.63
37.75
32.75
29.50
26.40
23.16
21.14
H
H
Ph
OH
Me
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.39
7.39
7.37
7.37
7.32
7.30
7.30
7.29
7.28
7.26
7.22
7.21
6.45
6.41
6.37
6.35
6.33
6.31
4.74
4.74
4.74
4.74
4.72
4.71
4.71
4.71
1.83
1.80
1.76
1.75
1.75
1.71
1.71
1.70
1.66
1.63
1.63
1.61
1.60
1.59
1.58
1.54
1.51
1.31
1.25
1.23
H
H
Me
OH
Ph
major
2.22
2.13
1.10
1.00
1.04
0.98
1.29
1.08
1.22
1.07
3.03
4.09
1.38
1.02
3.13
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000


149.95
137.55
131.35
131.00
128.53
127.13
126.12
108.62
70.44
50.29
44.79
39.87
33.29
29.52
26.79
20.94
H
H
Me
OH
Ph
major
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
3000
2500
2000
1500
1000
500
0