Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

[Pd]-Catalyzed *para*-Selective Allylation of Phenols: Access to 4-[(*E*)-3-Aryl/Alkylprop-2-enyl]phenols

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Supporting Information

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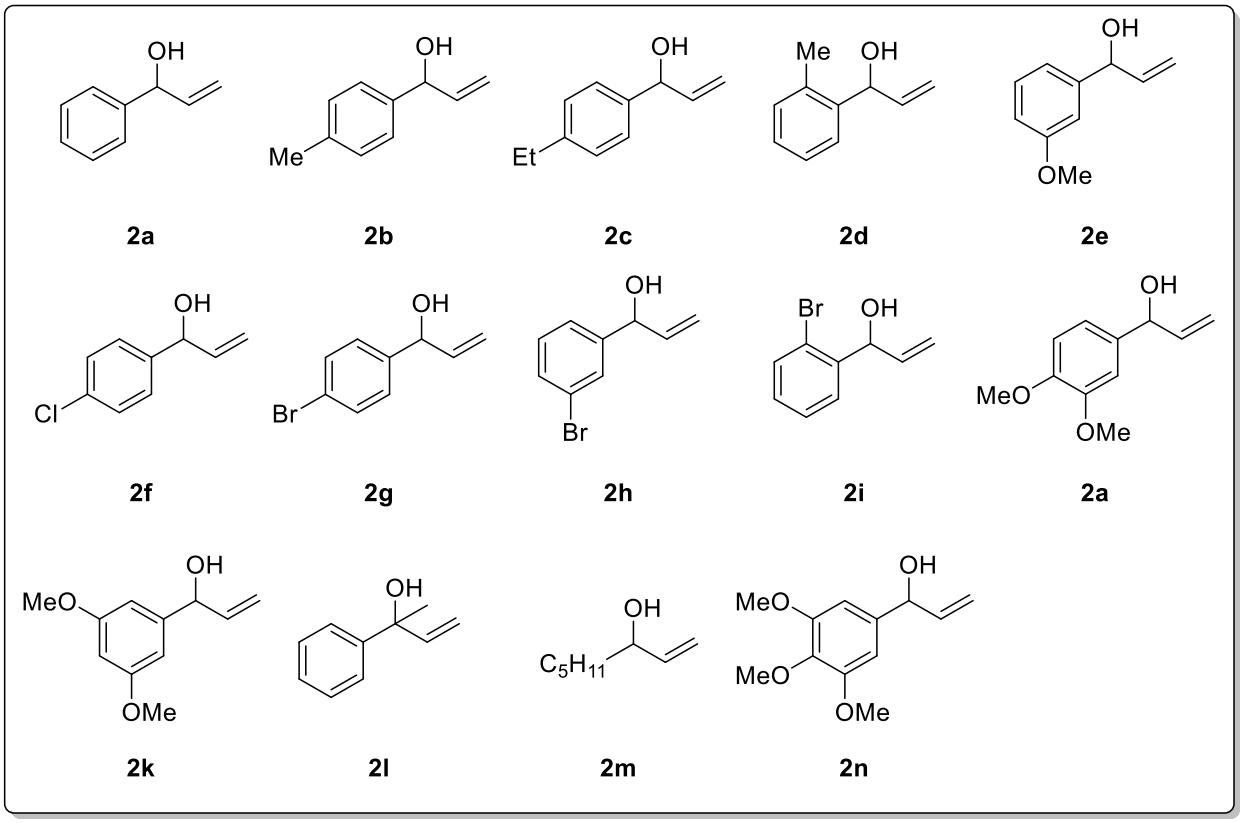
Experimental:

General: IR spectra were recorded on a Bruker Tensor 37 (FTIR) spectrophotometer. ^{1}H NMR spectra were recorded on Bruker Avance 400 (400 MHz) spectrometer at RT in $CDCl_3$; chemical shifts (δ ppm) and coupling constants (Hz) are reported in standard fashion with reference to either internal standard tetramethylsilane (TMS) (δ_H = 0.00 ppm) or $CHCl_3$ (δ_H = 7.25 ppm). ^{13}C{H} NMR spectra were recorded on Bruker Avance 400 (100 MHz) spectrometer at RT in $CDCl_3$; chemical shifts (δ ppm) are reported relative to $CDCl_3$ [δ_C = 77.00 ppm (central line of the triplet)]. In the ^{13}C{H} NMR, the nature of carbons (C, CH, CH_2 and CH_3) was determined by recording the DEPT-135 spectra, and is given in parentheses and noted as s = singlet (for C), d = doublet (for CH), t = triplet (for CH_2) and q = quartet (for CH_3). In the ^{1}H-NMR, the following abbreviations were used throughout: s = singlet, d = doublet, t = triplet, q = quartet, qui =quintet, sept = septet, dd = doublet of doublet, m = multiplet and brs = broad singlet. The assignment of signals was confirmed by ^{1}H, ^{13}C{^{1}H} CPD, and DEPT spectra. High-resolution mass spectra (HR-MS) were recorded on an Agilent 6538 UHD Q-TOF electron spray ionization (ESI) mode. Melting points are recorded using Tempo and Mettler FP1 melting point apparatus in capillary tubes and are uncorrected. All small-scale reactions were carried out using Schlenk tubes open air atmosphere. Reactions were monitored by TLC on silica gel using a combination of hexane and ethyl acetate as eluents. Solvents were distilled prior to use; petroleum ether with a boiling range of 60 to 80 °C was used. All vinyl Grignard reactions were performed in dry THF solvent. Palladium catalyzed reactions were done in DCE. Toluene, DMF, MeCN, Dioxane, THF, and DMSO solvents were purchased from the local chemicals and used as received. Acme's silica gel (60–120 mesh) was used for column chromatography (approximately 20 g per one gram of crude material). The catalysts $Pd(OAc)_2$, $PdCl_2$, $PdCl_2(PPh_3)_2$, $NiCl_2$, $NiBr_2$, and the base K_2CO_3 were purchased from Sigma-Aldrich and used as received. Substituted benzaldehydes, vinylmegnesiumbromide (1M in THF), and all the substituted phenols, 1-naphathol, 2-naphathol were purchased from TCI/local.

The following allylic alcohols were prepared using Grignard reaction of vinylmegnesiumbromide and substituted benzaldehydes in THF solvent at 0 °C to RT.[1]

Table-1S: 1,3-diarylpropanes **2a–n**.



1 (1–3)

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Following final products **3** are reported in the literature.[4–7]

Table-2S: 1,3-diarylpropanes **3aa–ba**.

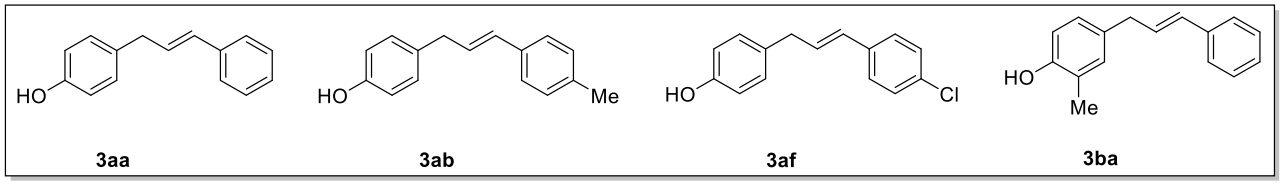
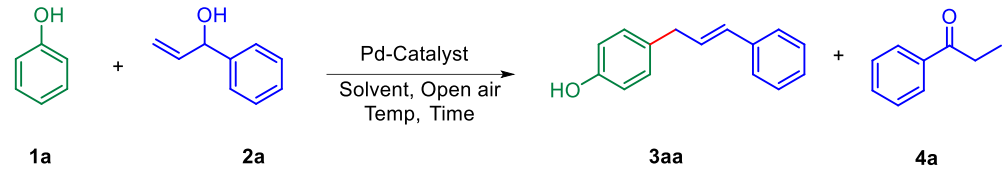


Table 3S. Optimization studies for the formation of **3aa**.[a,b,c,d,e,f]



Entry	Catalyst (10 mol%)	Solvent (2 mL)	Temp (°C)	Time (h)	3aa (%)[b]	4a (%)[b]
1	$Pd(OAc)_2$	toluene	80	12	46	35[c]
2	$Pd(OAc)_2$	toluene	80	12	52	20
3	$Pd(OAc)_2$	DCE	80	12	61	15
4	$Pd(OAc)_2$	MeCN	80	12	56	18
5	$Pd(OAc)_2$	DMF	80	12	26	30
6	$PdCl_2(PPh_3)_2$	DCE	80	12	42	22
7	$PdCl_2(PPh_3)_2$	MeCN	80	12	38	18
8	$PdCl_2$	MeCN	80	12	65	12
9	$PdCl_2$	DCE	80	12	71	8
10	$PdCl_2$	DCE	60	12	75	–
11	**$PdCl_2$**	**DCE**	**60**	**24**	**83**	**<10**
12	$PdCl_2$	toluene	60	24	62	24
13	$PdCl_2$	THF	60	24	56	35
14	$PdCl_2$	dioxane	60	24	51	20
15	$PdCl_2$	DMSO	60	24	42	28
16	$NiCl_2$	DCE	60	24	46	-[d]

17	$NiBr_2$	DCE	60	24	49	- [d]
18	$CuCl_2$	DCE	60	24	44	10
19	$PdCl_2$	DCE	60	24	55	8[e]
20	$PdCl_2$	DCE	60	24	46	20[f]

Reaction Conditions: [a]**1a** (0.2 mmol), **2a** (0.3 mmol), catalyst (10 mol%) under open-air atmosphere at 60 °C, 24 h. [b]Isolated yields of products. [c]K_2CO_3 is used as a base (0.2 mmol). [d]*The ortho*-allylated product was also obtained in around 15% to 20% yields, respectively. [e]The reaction was conducted with 0.3 mmol of **1a** and 0.2 mmol of **2a**. [f]Reaction conducted under N_2 atmosphere.

We started our initial investigations with phenol **1a** and allylic alcohol **2a** as the starting materials and the outcomes are as present in Table 3S. Thus, initially, the reaction of phenol (0.2 mmol) **1a** with allylic alcohol (0.3 mmol) **2a**, base K_2CO_3 (1 equiv), in toluene (2 mL) in the presence of $Pd(OAc)_2$ (10 mol%) at 80 °C for 12 h under open-air conditions, furnished the desired product **3aa** in 36% along with 55% yield of by-product ketone **4a** (Table 3S, entry 1). When performed without base, the yield of **3aa** was increased to 52% with a decreased 20% yield of the ketone **4a** (Table 3S, entry 2), which indicates that the use of base would promote the oxidation of the allylic alcohol **2a**. Switching to solvent 1,2-dichloroethane (DCE), there was a further improvement in the yield of the product **3aa** to 61% and with 15% of **4a** (Table 3S, entry 3). The reaction using other solvents such as acetonitrile (MeCN) and dimethylformamide (DMF), resulted in "**3aa** in 56% and 26%" and "**4a** in 18% and 30%" yields, respectively (Table 3S, entries 4 & 5). Changing the catalyst from $Pd(OAc)_2$ to $PdCl_2(PPh_3)_2$ (10 mol%), in DCE as well as in MeCN at 80 °C for 12 h in the open air, afforded "**3aa** in decreased 42% and 38%" and "**4a** in 22% and 18%" yields, respectively (Table 3S, entries 6 & 7). On the other hand, using $PdCl_2$ (10 mol%) as a catalyst in MeCN (2 mL), at 80 °C for 12 h in the open air, gave **3aa** in 65% yield and 12% yield of **4a** (Table 3S, entry 8). While the reaction under the same reaction conditions but in solvent DCE (2 mL), showed a further increase in the yield of the product **3aa** (71%) along with the decreased 8% yield of **4a** (Table 3S, entry 9). Furthermore, the reaction at a slightly lower temperature (60 °C), furnished the desired products **3aa** in 75% yield (Table 3S, entry 10). To our delight, by increasing the reaction time to 24 h at 60 °C, in DCE (2 mL), the yield of **3aa** was raised to 83% with less than 10% yield of the by-product **4a** (Table 3S, entry 11). On the other hand, the reaction with other solvents like toluene, THF, dioxane, and DMSO, showed no improvement in the yield of the product **3aa** (Table 3S, entries 12 to 15). The reaction using $NiCl_2$

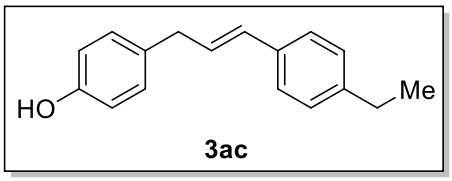
(10 mol%) as a catalyst in DCE (2 mL), at 60 °C for 24 h in open-air, delivered **3aa** in 46% along with 15% of the *ortho*-allylated product (Table 3S, entry 16). When $NiBr_2$ (10 mol%) was employed as a catalyst in DCE (2 mL), at 60 °C for 24 h in the open air, resulting in 49% yield of the product **3aa** and with 20% of the *ortho*-allylated product (Table 3S, entry 17). While the reaction in the presence of catalyst $CuCl_2$ (10 mol%), afforded **3aa** in moderate yields and with 10% of **4a** (Table 3S, entry 18). When the stoichiometric ratio of both the starting materials [i.e., phenol **1a** (0.3 mmol) and allylic alcohol **2a** (0.2 mmol)] is reversed, under the same conditions, **3aa** was isolated in 55% yield (Table 3S, entry 19). Also, the reaction was investigated under nitrogen atmosphere using $PdCl_2$ (10 mol%) in DCE (2 mL), at 60 °C for 24 h, the product **3aa** was obtained in a moderate yield of 46% (Table 3S, entry 20). Overall, the reaction gave the best yields of the product **3aa** in the open- air atmosphere in solvent DCE (2 mL) at 60 °C, for 24 h (Table 3S, entry 11).

Experimental:

GP-1 (General procedure for preparation of allylated phenols 3): To a solution of phenol **1a-k** (19 – 34 mg, 0.2 mmol) and allylic alcohols **2a-n** (40 – 64 mg, 0.3 mmol) in a Schlenk tube in DCE (2 mL), was added $PdCl_2$ (4 mg, 0.02 mmol) at room temperature under open air atmosphere and allowed the reaction mixture to stir at 60 °C for 24 to 36 h. Completion of the reaction was monitored by TLC (5:95 to 15:85 ethyl acetate and hexane). The reaction mixture was quenched with aqueous ammonium chloride and extracted with ethyl acetate (3×15 mL). The combined organic layers were washed with brine solution, dried (Na_2SO_4) and evaporated under reduced pressure. Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate) furnished the 1,3-diarylproperne **3aa-3am** (62–90%) as viscous liquid.

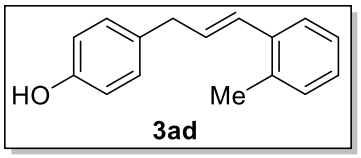
GP-2 (General procedure for preparation of 4): To an oven dried Schlenk tube, were added 1,3-diarylpropene **3** (119 – 120 mg, 0.5 mmol), alkyl/benzyl halide (106 – 147 mg, 0.75 mmol) followed by K_2CO_3 (138 mg, 1 mmol) and then acetone (10 mL) at room temperature under nitrogen atmosphere. The reaction mixture was stirred at room temperature for 3 h. Completion of the reaction was monitored by TLC (5:95 ethyl acetate and hexane). The reaction mixture was quenched with aqueous ammonium chloride and extracted with ethyl acetate (3×15 mL). The

combined organic layers were washed with brine solution, dried (Na_2SO_4) and evaporated under reduced pressure. Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate) furnished the **4da** (92%) and **4cd** (94%) as viscous liquid.



3ac

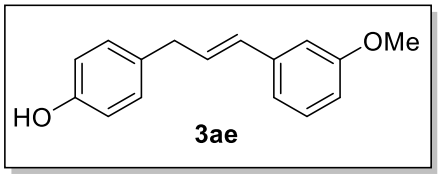
(*E*)-4-(3-(4-Ethylphenyl)allyl)phenol (3ac): GP-1 was carried out with phenol **1a** (19 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2c** (49 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(4-ethylphenyl)allyl)phenol **3ac** (37 mg, 78%) as a pale yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1a**) = 0.70, R_f(**2c**) = 0.40, R_f(**3ac**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3344, 3020, 2920, 1604, 1510, 1442, 1218, 968, 824, 752, 670 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ = 7.31 (d, 2H, *J* = 8.1 Hz, Ar-H), 7.13 (ddd, 4H, *J* = 9.3, 8.0, 5.5 Hz, Ar-H), 6.82 – 6.78 (m, 2H, Ar-H), 6.44 (d, 1H, *J* = 15.7 Hz, C*H*=CH-CH_2), 6.31 (dt, 1H, *J* = 15.7 and 6.8 Hz, CH=C*H*-CH_2), 5.24 (s, 1H, Ar-O*H*), 3.49 (d, 2H, *J* = 6.8 Hz, CH=CH-CH_2), 2.65 (q, 2H, *J* = 7.6 Hz, Ar-CH_2CH_3), 1.25 (t, 3H, *J* = 7.6 Hz, Ar-CH_2CH_3) ppm. $^{13}C\{H\}$-NMR ($CDCl_3$,100 MHz): δ =153.8 (s, Ar-C), 143.2 (s, Ar-C), 134.9 (s, Ar-C), 132.4 (s, Ar-C), 130.6 (d, CH=C*H*-CH_2), 129.7 (d, 2C, 2 × Ar-CH), 128.6 (d, CH=C*H*-CH_2), 128.0 (d, 2C, 2 × Ar-CH), 126.0 (d, 2C, 2 × Ar-CH), 115.3 (d, 2C, 2 × Ar-CH), 38.4 (t, *C*H_2), 28.5 (t, Ar-*C*H_2CH_3), 15.5 (q, Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{17}H_{17}]^+$=[(M+H) + (–H_2O)]$^+$: calculated: 221.1325; found: 221.1321.



3ad

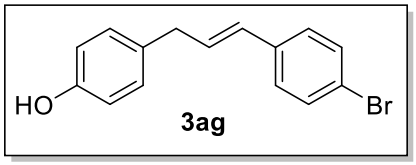
(*E*)-4-(3-(*o*-Tolyl)allyl)phenol (3ad): GP-1 was carried out with phenol **1a** (19 mg, 0.5 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2d** (44.4 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(*o*-tolyl)allyl)phenol **3ad** (36 mg, 81%) as

a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1a**) = 0.70, R_f(**2d**) = 0.40, R_f(**3ad**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3432, 3026, 2894, 2825, 1714, 1640, 1583, 1435, 1251, 1204, 1024, 737, 692 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.42 (dd, 1H, *J* = 6.8, 2.6 Hz, Ar-H), 7.16 – 7.09 (m, 5H, Ar-H), 6.82 – 6.73 (m, 2H, Ar-H), 6.64 (d, 1H, *J* = 15.6 Hz, C*H*=CH-CH_2), 6.20 (dt, 1H, *J* = 15.6 and 6.8 Hz, CH=C*H*-CH_2), 4.84 (s, 1H, Ar-O*H*), 3.50 (d, 2H, *J* = 6.8 Hz, CH=CH-CH_2), 2.34 (s, 3H, Ar-CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ =153.9 (s, Ar-C), 136.6 (s, Ar-C), 135.1 (s, Ar-C), 132.4 (s, Ar-C), 130.9 (d, Ar-CH), 130.2 (d, Ar-CH), 129.7 (d, 2C, 2 × Ar-CH), 128.7 (d, Ar-CH), 127.0 (d, Ar-CH), 126.0 (d, Ar-CH), 125.5 (d, Ar-CH), 115.3 (d, 2C, 2 × Ar-CH), 38.7 (t, CH_2), 19.8 (q, Ar-*C*H_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{20}NO]^+$=$[M+NH_4]^+$: calculated: 242.1539; found: 242.1530.

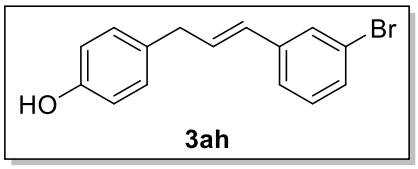


3ae

(*E*)-4-(3-(3-Methoxyphenyl)allyl)phenol (3ae): GP-1 was carried out with phenol **1a** (19 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2e** (49 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 87:13) furnished (*E*)-4-(3-(3-methoxyphenyl)allyl)phenol **3ae** (43 mg, 90%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 90:10), R_f(**1a**) = 0.70, R_f(**2e**) = 0.30, R_f(**3ae**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3486, 3295, 3028, 2893, 1572, 1424, 1322, 1205, 1067, 1013, 743 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.20 (t, 1H, *J* = 7.9 Hz, Ar-H), 7.09 (d, 2H, *J* = 8.5 Hz, Ar-H), 6.95 (d, 1H, *J* = 7.7 Hz, Ar-H), 6.89 (s, 1H, Ar-H), 6.81 – 6.73 (m, 3H, Ar-H), 6.39 (d, 1H, *J* = 15.7 Hz, C*H*=CH-CH_2), 6.32 (dt, 1H, *J* = 15.7 and 6.2 Hz, CH=C*H*-CH_2), 5.06 (s, 1H, Ar-O*H*), 3.80 (s, 3H, Ar-OCH_3), 3.46 (d, 2H, *J* = 6.2 Hz, CH=CH-CH_2) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ =159.7 (s, Ar-C), 153.9 (s, Ar-C), 139.0 (s, Ar-C), 132.2 (s, Ar-C), 130.6 (d, Ar-CH), 130.0 (d, Ar-CH), 129.8 (d, 2C, 2 × Ar-CH), 129.4 (d, Ar-CH), 118.8 (d, Ar-CH), 115.3 (d, 2C, 2 × Ar-CH), 112.8 (d, Ar-CH), 111.3 (d, Ar-CH), 55.2 (s, Ar-O*C*H_3), 38.4 (t, CH_2) ppm. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{17}O_2]^+$=$[M+H]^+$: calculated: 241.1223; found: 241.1221.

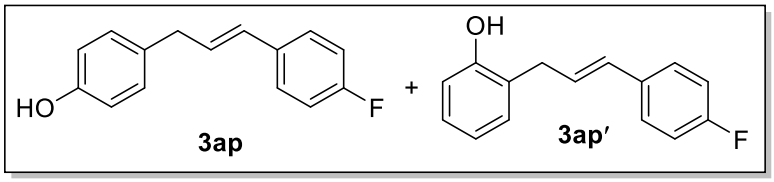


(*E*)-4-(3-(4-Bromophenyl)allyl)phenol (3ag): GP-1 was carried out with phenol **1a** (19 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2g** (64 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 36 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(4-bromophenyl)allyl)phenol **3ag** (49 mg, 85%) as a colorless liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1a**) = 0.70, R_f(**2g**) = 0.40, R_f(**3ag**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3375, 2894, 2819, 1582, 1451, 1422, 1253, 1227, 1075, 1011, 952, 767, 732, 661 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ = 7.40 (d, 2H, *J* = 8.5 Hz, Ar-H), 7.24 – 7.16 (m, 2H, Ar-H), 7.09 (d, 2H, *J* = 8.5 Hz, Ar-H), 6.78 (d, 2H, *J* = 8.4 Hz, Ar-H), 6.34 – 6.31 (m, 2H, C*H*=C*H*-CH_2), 4.96 (s, 1H, Ar-O*H*), 3.45 (d, 2H, *J* = 5.1 Hz, CH=CH-C*H*$_2$) ppm. $^{13}C\{H\}$-NMR ($CDCl_3$,100 MHz): δ = 153.9 (s, Ar-C), 136.4 (s, Ar-C), 131.9 (s, Ar-C), 131.5 (d, 2C, 2 × Ar-CH), 130.5 (d, CH=C*H*-CH_2), 129.8 (d, 2C, 2 × Ar-CH), 129.5 (d, CH=C*H*-CH_2), 127.6 (d, 2C, 2 × Ar-CH), 120.7 (s, Ar-C), 115.3 (d, 2C, 2 × Ar-CH), 38.4 (t, CH_2) ppm. HR-MS (ESI^+) m/z calculated for $[C_{15}H_{14}Br^{79}O]^+$=$[M+H]^+$: 289.0223; found 289.0201, calculated for $[C_{15}H_{14}Br^{81}O]^+$=$[M+H]^+$: calculated: 291.0202; found: 291.0195.



(*E*)-4-(3-(3-Bromophenyl)allyl)phenol (3ah): GP-1 was carried out with phenol **1a** (19 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2h** (64 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 36 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(3-bromophenyl)allyl)phenol **3ah** (43 mg, 75%) as a colorless liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1a**)

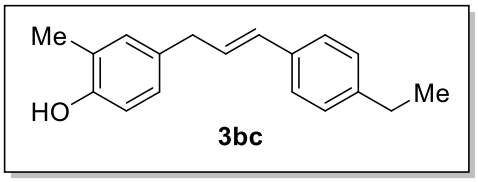
= 0.70, R_f(**2h**) = 0.40, R_f(**3ah**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3299, 3022, 1589, 1495, 1325, 1249, 817, 726 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.62 (t, 1H, *J* = 1.8 Hz, Ar-H), 7.45 (ddd, 1H, *J* = 7.9, 1.9, 1.1 Hz, Ar-H), 7.40 – 7.36 (m, 1H, Ar-H), 7.28 (d, 1H, *J* = 7.8 Hz, Ar-H), 7.24 – 7.19 (m, 2H, Ar-H), 6.95 – 6.86 (m, 2H, Ar-H), 6.47 – 6.46 (m, 2H, C*H*=C*H*-CH_2), 5.03 (s, 1H, Ar-O*H*), 3.60 (d, 2H, *J* = 4.7 Hz, CH=CH-CH_2) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 154.0 (s, Ar-C), 139.7 (s, Ar-C), 131.8 (s, Ar-C), 131.3 (d, Ar-CH), 130.0 (d, Ar-CH), 129.9 (d, Ar-CH), 129.8 (d, 2C, 2 × Ar-CH), 129.3 (d, Ar-CH), 128.9 (d, Ar-CH), 124.7 (d, Ar-CH), 122.7 (s, Ar-C), 115.4 (d, 2C, 2 × Ar-CH), 38.3 (t, CH_2) ppm. HR-MS (ESI^+) m/z calculated for $[C_{15}H_{14}Br^{79}O]^+$=$[M+H]^+$: 289.0223; found 289.0221, calculated for $[C_{15}H_{14}Br^{81}O]^+$=$[M+H]^+$: calculated: 291.0202; found: 291.0208.



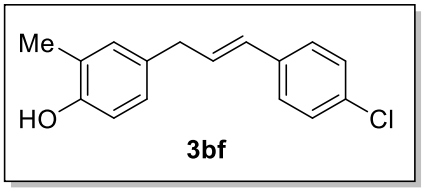
(*E*)-4-(3-(4-Fluorophenyl)allyl)phenol (3ap) & (*E*)-2-(3-(4-Fluorophenyl)allyl)phenol (3ap′): **GP-1** was carried out with phenol **1c** (500 mg, 5.3 mmol), $PdCl_2$ (47 mg, 0.265 mmol) and allylic alcohol **2d** (1.2 g, 7.9 mmol), in DCE (20 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) first the minor regioisomer (*E*)-2-(3-(4-Fluorophenyl)allyl)phenol **3ap′** (60 mg, 5%) as a pale-yellow liquid followed by (*E*)-4-(3-(4-Fluorophenyl)allyl)phenol **3ap** (1.02 g, 85%) as a pale-yellow liquid.

(*E*)-4-(3-(4-Fluorophenyl)allyl)phenol (3ap): [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1a**) = 0.70, R_f(**2p**) = 0.40, R_f(**3ap**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3308, 2996, 1584, 1490, 1424, 1207, 1149, 956, 818 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.34 – 7.27 (m, 2H), 7.09 (d, *J* = 8.5 Hz, 2H), 7.00 – 6.95 (m, 2H), 6.85 – 6.69 (m, 2H), 6.37 (d, *J* = 15.8 Hz, 1H), 6.23 (dt, *J* = 15.8, 6.7 Hz, 1H), 4.79 (s, 1H), 3.45 (d, *J* = 6.7 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ = 163.2, 160.8, 153.9, 133.6, 133.6, 132.2, 129.8, 129.5, 129.4, 129.4, 127.5, 127.5, 115.4, 115.3, 115.2, 38.3 ppm. HR-MS (ESI^+) m/z calculated for $[C_{15}H_{14}FO]^+$=$[M+H]^+$: calculated: 229.1023; found: 229.1039.

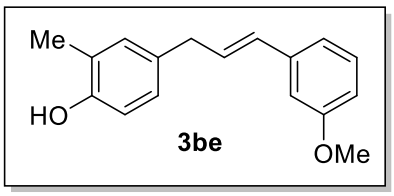
(*E*)-2-(3-(4-Fluorophenyl)allyl)phenol (3ap′): [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1a**) = 0.70, R_f(**2p**) = 0.40, R_f(**3ap**) = 0.60, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3375, 3004, 1579, 1487, 1438, 1316, 1209, 1146, 1084, 958, 828, 743 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.34 – 7.27 (m, 2H), 7.17 – 712 (m, 2H), 7.01 – 6.93 (m, 2H), 6.90 (ddd, *J* = 7.4, 7.4, 1.1 Hz, 1H), 6.84 – 6.80 (m, 1H), 6.45 (d, *J* = 15.9 Hz, 1H), 6.30 (dt, *J* = 15.9, 6.5 Hz, 1H), 4.98 (s, 1H), 3.55 (d, *J* = 6.5 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ = 163.3, 160.9, 153.9, 133.3, 133.2, 130.4, 130.2, 129.6, 127.9, 127.7, 127.7, 127.6, 127.6, 125.6, 121.0, 120.7, 115.7, 115.5, 115.3, 33.9 ppm. HR-MS (ESI^+) m/z calculated for $[C_{15}H_{17}FNO]^+$=$[M+NH_4]^+$: calculated: 246.1289; found: 246.1263.



(*E*)-4-(3-(4-Ethylphenyl)allyl)-2-methylphenol (3bc): GP-1 was carried out with phenol **1b** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2c** (49 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 36 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(4-ethylphenyl)allyl)-2-methylphenol **3bc** (38.3 mg, 76%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1b**) = 0.70, R_f(**2c**) = 0.40, R_f(**3bc**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3486, 3290, 3028, 1571, 1481, 1205, 1012, 958, 743 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.27 (t, 2H, *J* = 9.2 Hz, Ar-H), 7.13 (d, 2H, *J* = 8.1 Hz, Ar-H), 6.99 (s, 1H, Ar-H), 6.94 (dd, 1H, *J* = 8.1, 1.8 Hz, Ar-H), 6.71 (d, 1H, *J* = 8.1 Hz, Ar-H), 6.41 (d, 1H, *J* = 15.7 Hz, C*H*=CH-CH_2), 6.28 (dt, 1H, *J* = 15.7 and 6.8 Hz, CH=C*H*-CH_2), 4.66 (s, 1H, Ar-O*H*), 3.44 (d, 2H, *J* = 6.8 Hz, CH=CH-CH_2), 2.62 (q, 2H, *J* = 7.6 Hz, Ar-C$H_2$$CH_3$), 2.23 (s, 3H, Ar-$CH_3$), 1.22 (t, 3H, *J* = 7.6 Hz, Ar-CH_2CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 152.1 (s, Ar-C), 143.2 (s, Ar-C), 135.0 (s, Ar-C), 132.4 (s, Ar-C), 131.2 (d, Ar-CH), 130.5 (d, Ar-CH), 128.8 (d, Ar-CH), 128.0 (d, 2C, 2 × Ar-CH), 127.1 (d, Ar-CH), 126.1 (d, 2C, 2 × Ar-CH), 123.6 (s, Ar-C), 114.9 (d, Ar-CH), 38.5 (t, CH_2), 28.5 (t, Ar-*C*$H_2$$CH_3$), 15.7 (q, Ar-$CH_3$) 15.6 (q, Ar-$CH_3$) ppm. HR-MS ($ESI^+$) m/z calculated for $[C_{18}H_{21}O]^+$=$[M+H]^+$: calculated: 253.1587; found: 253.1571.

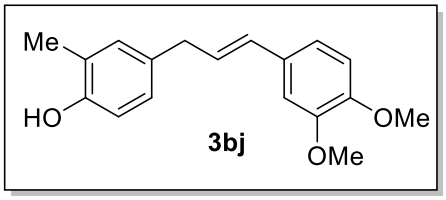


(*E*)-4-(3-(4-Chlorophenyl)allyl)-2-methylphenol (3bf): GP-1 was carried out with phenol **1b** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2f** (51 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(4-chlorophenyl)allyl)-2-methylphenol **3bf** (43 mg, 82%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1b**) = 0.70, R_f(**2f**) = 0.40, R_f(**3bf**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3302, 2994, 2875, 1587, 1494, 1421, 1332, 1210, 1010, 955, 816, 737, 659 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ = 7.28 – 7.21 (m, 4H, Ar-H), 6.96 (s, 1H, Ar-H), 6.91 (dd, 1H, *J* = 8.1, 2.0 Hz, Ar-H), 6.70 (d, 1H, *J* = 8.1 Hz, Ar-H), 6.35 (d, 1H, *J* = 15.7 Hz, C*H*=CH-CH_2), 6.28 (dt, 1H, *J* = 15.7 and 6.2 Hz, CH=C*H*-CH_2), 4.85 (s, 1H, Ar-O*H*), 3.42 (d, 2H, *J* = 6.2 Hz, CH=CH-CH_2), 2.22 (s, 3H, Ar-CH_3) ppm. $^{13}C\{H\}$-NMR ($CDCl_3$,100 MHz): δ = 152.2 (s, Ar-C), 136.0 (s, Ar-C), 132.5 (s, Ar-C), 131.9 (s, Ar-C), 131.2 (d, Ar-CH), 130.6 (d, Ar-CH), 129.4 (d, Ar-CH), 128.6 (d, 2C, 2 × Ar-CH), 127.3 (d, 2C, 2 × Ar-CH), 127.1 (d, Ar-CH), 123.8 (s, Ar-C), 114.9 (d, Ar-CH), 38.4 (t, CH_2), 15.7 (q, Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{16}ClO]^+$=$[M+H]^+$: calculated: 259.0884; found: 259.0873.



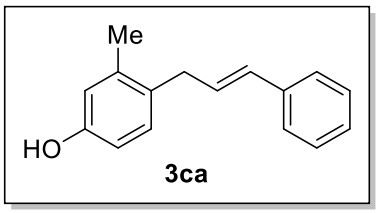
(*E*)-4-(3-(3-Methoxyphenyl)allyl)-2-methylphenol (3be): GP-1 was carried out with phenol **1b** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2e** (49 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 87:13) furnished (*E*)-4-(3-(3-methoxyphenyl)allyl)-2-methylphenol **3be** (44 mg, 86%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 90:10), R_f(**1b**) = 0.70, R_f(**2e**) = 0.40, R_f(**3be**) = 0.50, UV detection].

IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3487, 3026, 2894, 2825, 1640, 1583, 1436, 1254, 1203, 1023, 748, 690 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.24 (t, 1H, *J* = 7.9 Hz, Ar-H), 7.04 – 6.92 (m, 4H, Ar-H), 6.80 (ddd, 1H, *J* = 8.2, 2.6, 0.8 Hz, Ar-H), 6.72 (d, 1H, *J* = 8.1 Hz, Ar-H), 6.43 (d, 1H, *J* = 15.7 Hz, C*H*=CH-CH_2), 6.35 (dt, 1H, *J* = 15.7 and 6.4 Hz, CH=C*H*-CH_2), 4.97 (s, 1H, Ar-O*H*), 3.82 (s, 3H, Ar-OCH_3), 3.47 (d, 2H, *J* = 6.4 Hz, CH=CH-CH_2), 2.26 (s, 3H, Ar-CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 159.7 (s, Ar-C), 152.2 (s, Ar-C), 139.0 (s, Ar-C), 132.1 (s, Ar-C), 131.2 (d, Ar-CH), 130.4 (d, Ar-CH), 130.1 (d, Ar-CH), 129.4 (d, Ar-CH), 127.1 (d, Ar-CH), 123.8 (s, Ar-C), 118.8 (d, Ar-CH), 114.9 (d, Ar-CH), 112.7 (d, Ar-CH), 111.3 (d, Ar-CH), 55.2 (s, Ar-O*C*H_3), 38.4 (t, CH_2), 15.7 (q, Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{17}H_{19}O_2]^+$=$[M+H]^+$: calculated: 255.1380; found: 255.1379.

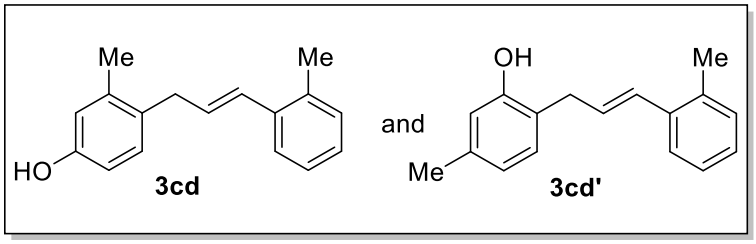


3bj

(*E*)-4-(3-(3,4-Dimethoxyphenyl)allyl)-2-methylphenol (3bj): GP-1 was carried out with phenol **1b** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allyl alcohol **2j** (58 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 85:15) furnished (*E*)-4-(3-(3,4-dimethoxyphenyl)allyl)-2-methylphenol **3bj** (48 mg, 84%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 85:15), R_f(**1b**) = 0.70, R_f(**2j**) = 0.30, R_f(**3bj**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3389, 2897, 2819, 1567, 1485, 1441, 1223, 1110, 992, 805, 724 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 6.99 (d, 1H, *J* = 1.6 Hz, Ar-H), 6.96 – 6.87 (m, 3H, Ar-H), 6.80 (d, 1H, *J* = 8.2 Hz, Ar-H), 6.71 (d, 1H, *J* = 8.1 Hz, Ar-H), 6.36 (d, 1H, *J* = 15.7 Hz, C*H*=CH-CH_2), 6.19 (dt, 1H, *J* = 15.7 and 6.8 Hz, CH=C*H*-CH_2), 4.89 (s, 1H, Ar-O*H*), 3.87 (s, 3H, Ar-O*C*H_3), 3.86 (s, 3H, Ar-O*C*H_3), 3.43 (d, 2H, *J* = 6.8 Hz, CH=CH-CH_2), 2.23 (s, 3H, Ar-CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ =152.2 (s, Ar-C), 148.9 (s, Ar-C), 148.3 (s, Ar-C), 132.3 (s, Ar-C), 131.2 (d, Ar-CH), 130.7 (s, Ar-C), 130.2 (d, Ar-CH), 127.9 (d, Ar-CH), 127.1 (d, Ar-CH), 123.8 (s, Ar-C), 119.1 (d, Ar-CH), 114.9 (d, Ar-CH), 111.1 (d, Ar-CH), 108.6 (d, Ar-CH), 55.9 (s, Ar-O*C*H_3), 55.7 (s, Ar-O*C*H_3), 38.4 (t, CH_2), 15.7 (q, Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{18}H_{21}O_3]^+$=$[M+H]^+$: calculated: 285.1485; found: 285.1490.



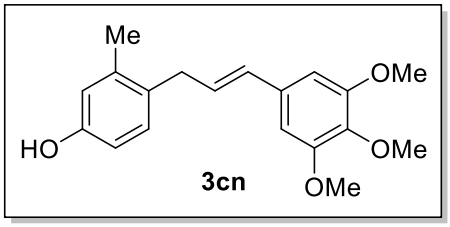
4-Cinnamyl-3-methylphenol (3ca): GP-1 was carried out with phenol **1c** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2a** (40 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished 4-cinnamyl-3-methylphenol **3ca** (35 mg, 77%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1c**) = 0.70, R_f(**2a**) = 0.40, R_f(**3ca**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3487, 3286, 2893, 2822, 1570, 1481, 1446, 1206, 1013, 958, 743 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ = 7.34 (d, 2H, J = 7.1 Hz, Ar-H), 7.29 (t, 2H, J = 7.5 Hz, Ar-H), 7.20 (ddd, 1H, J = 6.3, 2.5, 1.2 Hz, Ar-H), 7.05 (d, 1H, J = 8.1 Hz, Ar-H), 6.76 – 6.57 (m, 2H, Ar-H), 6.45 – 6.22 (m, 2H, C*H*=C*H*-CH_2), 4.89 (br.s, 1H, Ar-O*H*), 3.46 (d, 2H, J = 4.7 Hz, CH=CH-CH_2), 2.28 (s, 3H, Ar-CH_3) ppm. $^{13}C\{H\}$-NMR ($CDCl_3$,100 MHz): δ = 153.9 (s, 2C, 2 × Ar-C), 138.1 (s, Ar-C), 137.6 (s, Ar-C), 130.6 (d, Ar-CH), 130.5 (s, Ar-C), 130.4 (d, Ar-CH), 129.0 (d, Ar-CH), 128.5 (d, 2C, 2 × Ar-CH), 127.0 (d, Ar-CH), 126.1 (d, 2C, 2 × Ar-CH), 117.1 (d, Ar-CH), 112.7 (d, Ar-CH), 36.1 (t, CH_2), 19.6 (q, Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{17}O]^+$=$[M+H]^+$: calculated: 225.1274; found: 225.1271.



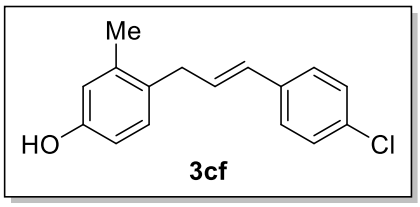
(*E*)-3-Methyl-4-(3-(*o*-tolyl)allyl)phenol (3cd) and (*E*)-5-Methyl-2-(3-(*o*-tolyl)allyl)phenol (3cd′): GP-1 was carried out with phenol **1c** (216 mg, 2 mmol), $PdCl_2$ (17 mg, 0.1 mmol) and allylic alcohol **2d** (444 mg, 3 mmol), in DCE (20 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) first the minor regioisomer (*E*)-5-methyl-2-(3-(*o*-tolyl)allyl)phenol **3cd′** (57 mg, 12%) as a pale-yellow solid followed by (*E*)-3-methyl-4-(3-(*o*-tolyl)allyl)phenol **3cd** (252 mg, 53%) as a pale-yellow liquid.

(*E*)-3-methyl-4-(3-(*o*-tolyl)allyl)phenol (3cd): [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1c**) = 0.70, R_f(**2d**) = 0.40, R_f(**3cd**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3378, 2990, 2890, 1573, 1477, 1437, 1254, 1145, 955, 850, 727 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45 – 7.36 (m, 1H, Ar-H), 7.19 – 7.09 (m, 3H, Ar-H), 7.05 (d, *J* = 8.1 Hz, 1H, Ar-H), 6.72 – 6.59 (m, 2H, Ar-H), 6.55 (d, *J* = 15.6 Hz, 1H, C*H*=C*H*-CH_2), 6.16 (dt, *J* = 15.6 and 6.6 Hz, 1H, C*H*=C*H*-CH_2), 4.78 (d, *J* = 3.1 Hz, 1H, Ar-O*H*), 3.47 (dd, *J* = 6.6 and 1.3 Hz, 2H, CH=CH-CH_2), 2.29 (d, *J* = 3.5 Hz, 6H, 2 × Ar-CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 153.8 (s, Ar-C), 137.9 (s, Ar-C), 136.7 (s, Ar-C), 135.0 (s, Ar-C), 130.6 (s, Ar-C), 130.3 (d, Ar-CH), 130.2 (d, Ar-CH), 130.1 (d, Ar-CH), 128.5 (d, Ar-CH), 126.9 (d, Ar-CH), 126.0 (d, Ar-CH), 125.5 (d, Ar-CH), 117.0 (d, Ar-CH), 112.7 (d, Ar-CH), 36.4 (t, CH_2), 19.7 (q, Ar-CH_3), 19.5 (q, Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{17}H_{19}O]^+$=$[M+H]^+$: calculated: 239.1430; found: 239.1417.

(*E*)-5-Methyl-2-(3-(*o*-tolyl)allyl)phenol (3cd′): (57 mg, 12%) as a pale-yellow solid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1c**) = 0.70, R_f(**2d**) = 0.40, R_f(**3cd′**) = 0.60, UV detection]. Mp: 117 – 119 °C; IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3303, 2993, 2872, 1587, 1494, 1446, 1420, 1210, 1160, 1010, 954, 817, 737 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.44 – 7.35 (m, 1H, Ar-H), 7.16 – 7.09 (m, 3H, Ar-H), 7.04 (d, *J* = 7.6 Hz, 1H, Ar-H), 6.74 – 6.64 (m, 3H, Ar-H), 6.23 (dt, *J* = 15.7 and 6.7 Hz, 1H, CH=C*H*-CH_2), 4.88 (d, *J* = 1.5 Hz, 1H, Ar-O*H*), 3.54 (d, *J* = 6.7 Hz, 2H, CH=CH-CH_2), 2.31 (s, 3H, Ar-CH_3), 2.29 (s, 3H, Ar-CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 153.9 (s, Ar-C), 137.9 (s, Ar-C), 136.2 (s, Ar-C), 135.1 (s, Ar-C), 130.2 (d, Ar-CH), 130.1 (d, Ar-CH), 129.5 (d, Ar-CH), 129.4 (d, Ar-CH), 127.2 (d, Ar-CH), 126.0 (d, Ar-CH), 125.6 (d, Ar-CH), 122.5 (s, Ar-C), 121.7 (d, Ar-CH), 116.4 (d, Ar-CH), 34.2 (t, CH_2), 21.0 (q, Ar-CH_3), 19.8 (q, Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{17}H_{19}O]^+$=$[M+H]^+$: calculated: 239.1430; found: 239.1416.

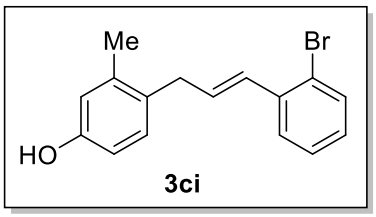


(*E*)-3-Methyl-4-(3-(3,4,5-trimethoxyphenyl)allyl)phenol (3cn): GP-1 was carried out with phenol **1c** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2n** (67 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-3-methyl-4-(3-(3,4,5-trimethoxyphenyl)allyl)phenol **3cn** (45 mg, 71%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1c**) = 0.70, R_f(**2n**) = 0.40, R_f(**3cn**) = 0.50, UV detection]. Mp: 135 – 137 °C; IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3390, 2897, 2820, 1567, 1485, 1441, 1224, 1110, 992, 805, 724 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.04 (d, J = 8.1 Hz, 1H, Ar-H), 6.70 – 6.61 (m, 2H, Ar-H), 6.55 (s, 2H, Ar-H), 6.23 – 6.21 (m, 2H, C*H*=C*H*-CH_2), 4.93 (br.s, 1H, Ar-O*H*), 3.84 (s, 6H, 2 × Ar-OCH_3), 3.82 (s, 3H, Ar-OCH_3), 3.44 (d, J = 4.5 Hz, 2H, CH=CH-C*H*$_2$), 2.26 (s, 3H, Ar-CH_3) ppm. ^{13}C{H}-NMR (100 MHz, $CDCl_3$) δ = 154.1 (s, Ar-C), 153.2 (s, 2C, 2 × Ar-C), 138.0 (s, Ar-C), 137.2 (s, Ar-C), 133.3 (s, Ar-C), 130.4 (d, Ar-CH), 130.3 (d, Ar-CH), 130.2 (s, Ar-C), 128.6 (d, Ar-CH), 117.1 (d, Ar-CH), 112.7 (d, Ar-CH), 103.0 (d, 2C, 2 × Ar-CH), 60.9 (q, Ar-OCH_3), 56.0 (q, 2C, 2 × Ar-OCH_3), 35.9 (t, CH_2), 19.5 (q, Ar-CH_3) ppm. HR-MS (ESI$^+$) m/z calculated for $[C_{19}H_{23}O_4]^+$=$[M+H]^+$: calculated: 315.1591; found: 315.1585.



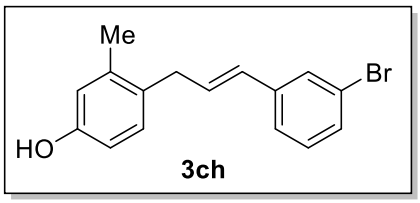
(*E*)-4-(3-(4-Chlorophenyl)allyl)-3-methylphenol (3cf): GP-1 was carried out with phenol **1c** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2f** (51 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(4-chlorophenyl)allyl)-3-methylphenol **3cf** (34 mg, 66%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1c**) = 0.70, R_f(**2f**) = 0.40, R_f(**3cf**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3301, 2994, 2874, 1587, 1494, 1420, 1330, 1210, 1162, 1010, 955, 816, 738, 659

cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.23 (s, 4H, Ar-H), 7.01 (d, 1H, J = 8.1 Hz, Ar-H), 6.70 – 6.59 (m, 2H, Ar-H), 6.35 – 6.21 (m, 2H, C*H*=C*H*-CH_2), 5.15 (br.s, 1H, Ar-O*H*), 3.42 (d, 2H, J = 6.1 Hz, CH=CH-C*H*$_2$), 2.25 (s, 3H, Ar-CH_3) ppm. $^{13}C\{H\}$-NMR ($CDCl_3$,100 MHz): δ = 153.9 (s, Ar-C), 138.0 (s, Ar-C), 136.0 (s, Ar-C), 132.5 (s, Ar-C), 130.4 (d, Ar-CH), 130.1 (s, Ar-C), 129.7 (d, Ar-CH), 129.3 (d, Ar-CH), 128.5 (d, 2C, 2 × Ar-CH), 127.2 (d, 2C, 2 × Ar-CH), 117.1 (d, Ar-CH), 112.7 (d, Ar-CH), 35.9 (t, CH_2), 19.5 (q, Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{16}ClO]^+$=$[M+H]^+$: calculated: 259.0884; found: 259.0870.



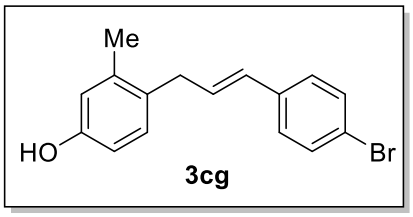
3ci

(*E*)-4-(3-(2-Bromophenyl)allyl)-3-methylphenol (3ci): GP-1 was carried out with phenol **1c** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2i** (64 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(2-bromophenyl)allyl)-3-methylphenol **3ci** (38 mg, 62%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1c**) = 0.70, R_f(**2i**) = 0.40, R_f(**3ci**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3395, 2993, 2889, 1600, 1570, 1453, 1405, 1253, 952, 724, 696 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.51 (dd, J = 8.0, 1.2 Hz, 1H, Ar-H), 7.46 (dd, J = 7.8, 1.6 Hz, 1H, Ar-H), 7.21 (ddd, J = 7.9, 7.4, 0.7 Hz, 1H, Ar-H), 7.05 (ddd, J = 7.5, 4.4, 1.8 Hz, 2H, Ar-H), 6.79 – 6.55 (m, 3H, Ar-H), 6.20 (dt, J = 15.7 and 6.6 Hz, 1H, C*H*=C*H*-CH_2), 4.68 (s, 1H, Ar-O*H*), 3.49 (dd, J = 6.6 and 1.5 Hz, 2H, CH=CH-C*H*$_2$), 2.29 (s, 3H, Ar-CH_3) ppm. $^{13}C\{H\}$-NMR ($CDCl_3$,100 MHz): δ = 154.0 (s, Ar-C), 138.1 (s, Ar-C), 137.4 (s, Ar-C), 132.8 (d, Ar-CH), 132.1 (d, Ar-CH), 130.3 (d, Ar-CH), 130.1 (s, Ar-C), 129.6 (d, Ar-CH), 128.3 (d, Ar-CH), 127.4 (d, Ar-CH), 127.0 (d, Ar-CH), 123.2 (s, Ar-C), 117.1 (d, Ar-CH), 112.7 (d, Ar-CH), 36.2 (t, CH_2), 19.5 (q, Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{16}Br^{79}O]^+$=$[M+H]^+$: calculated: 303.0379; found: 303.0374, calculated for $[C_{16}H_{16}Br^{81}O]^+$=$[M+H]^+$: calculated: 305.0359; found: 305.0357.



3ch

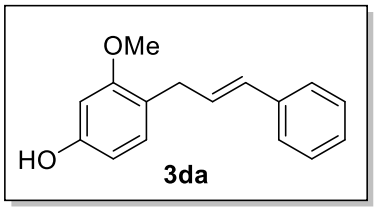
(*E*)-4-(3-(3-Bromophenyl)allyl)-3-methylphenol (3ch): GP-1 was carried out with phenol **1c** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2h** (64 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(3-bromophenyl)allyl)-3-methylphenol **3ch** (40 mg, 66%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1c**) = 0.70, R_f(**2h**) = 0.40, R_f(**3ch**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3391, 2992, 2890, 1569, 1452, 1406, 1263, 1199, 1061, 952, 764, 674 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.46 (t, *J* = 1.8 Hz, 1H, Ar-H), 7.30 (ddd, *J* = 7.8, 1.8, 1.1 Hz, 1H, Ar-H), 7.22 (dd, *J* = 7.7, 1.1 Hz, 1H, Ar-H), 7.13 (t, *J* = 7.8 Hz, 1H, Ar-H), 7.01 (d, *J* = 8.1 Hz, 1H, Ar-H), 6.72 – 6.56 (m, 2H, Ar-H), 6.31 (dt, *J* = 15.8 and 6.1 Hz, 1H, C*H*=C*H*-CH_2), 6.22 (d, *J* = 15.8 Hz, 1H, C*H*=C*H*-CH_2), 4.70 (s, 1H, Ar-O*H*), 3.44 (d, *J* = 6.1 Hz, 2H, CH=CH-CH_2), 2.26 (s, 3H, Ar-CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 154.0 (s, Ar-C), 139.7 (s, Ar-C), 138.0 (s, Ar-C), 130.7 (d, Ar-CH), 130.4 (d, Ar-CH), 130.0 (s, Ar-C), 129.9 (d, Ar-CH), 129.8 (d, Ar-CH), 129.1 (d, Ar-CH), 128.9 (d, Ar-CH), 124.7 (d, Ar-CH), 122.7 (s, Ar-C), 117.1 (d, Ar-CH), 112.8 (d, Ar-CH), 35.9 (t, CH_2), 19.5 (q, Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{16}Br^{79}O]^+$=$[M+H]^+$: calculated: 303.0379; found: 303.0379, calculated for $[C_{16}H_{16}Br^{81}O]^+$=$[M+H]^+$: calculated: 305.0359; found: 305.0363.



3cg

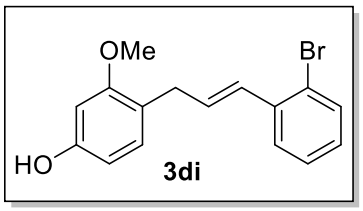
(*E*)-4-(3-(4-Bromophenyl)allyl)-3-methylphenol (3cg): GP-1 was carried out with phenol **1c** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2g** (64 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(4-bromophenyl)allyl)-3-methylphenol **3cg** (35 mg, 58%) as a pale-yellow liquid. [TLC control

(petroleum ether/ethyl acetate 93:07), R_f(**1c**) = 0.70, R_f(**2g**) = 0.40, R_f(**3cg**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3373, 2893, 1579, 1453, 1422, 1078, 1010, 951, 769, 740 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.41 – 7.35 (m, 2H, Ar-H), 7.18 (d, 2H, *J* = 8.5 Hz, Ar-H), 7.02 (d, 1H, *J* = 8.1 Hz, Ar-H), 6.76 – 6.55 (m, 2H, Ar-H), 6.41 – 6.19 (m, 2H, C*H*=C*H*-CH_2), 4.77 (s, 1H, Ar-O*H*), 3.43 (d, 2H, *J* = 5.4 Hz, CH=CH-CH_2), 2.26 (s, 3H, Ar-CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 154.0 (s, Ar-C), 138.0 (s, Ar-C), 136.5 (s, Ar-C), 131.5 (d, 2C, 2 × Ar-CH), 130.4 (d, Ar-CH), 130.1 (s, Ar-C), 129.9 (d, Ar-CH), 129.4 (d, Ar-CH), 127.6 (d, 2C, 2 × Ar-CH), 120.6 (s, Ar-C), 117.1 (d, Ar-CH), 112.7 (d, Ar-CH), 36.0 (t, CH_2), 19.5 (q, Ar-CH_3) ppm. HR-MS (ESI$^+$) m/z calculated for $[C_{16}H_{16}Br^{79}O]^+$=$[M+H]^+$: calculated: 303.0379; found: 303.0405, calculated for $[C_{16}H_{16}Br^{81}O]^+$=$[M+H]^+$: calculated: 305.0359; found: 305.0349.

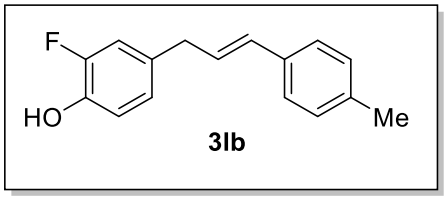


3da

4-Cinnamyl-3-methoxyphenol (3da): GP-1 was carried out with phenol **1d** (25 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2a** (40 mg, 0.75 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished 4-cinnamyl-3-methoxyphenol **3da** (33 mg, 69%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1d**) = 0.70, R_f(**2a**) = 0.40, R_f(**3da**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3377, 2896, 2816, 1583, 1452, 1422, 1256, 1185, 1077, 953, 736 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.35 (dd, *J* = 8.3 and 1.1 Hz, 2H, Ar-H), 7.29 (dd, *J* = 7.1 and 1.7 Hz, 2H, Ar-H), 7.23 – 7.15 (m, 1H, Ar-H), 7.01 (d, *J* = 8.1 Hz, 1H, Ar-H), 6.43 (d, *J* = 2.3 Hz, 1H, Ar-H), 6.44 – 6. 31 (m, 1H, Ar-H and 2H, C*H*=C*H*-CH_2), 4.80 (s, 1H, Ar-O*H*), 3.81 (s, 3H, Ar-OCH_3), 3.45 (d, *J* = 5.7 Hz, 2H, CH=CH-CH_2) ppm. ^{13}C{H}-NMR (100 MHz, $CDCl_3$) δ = 158.3 (s, Ar-C), 155.2 (s, Ar-C), 137.8 (s, Ar-C), 130.4 (d, Ar-CH), 130.3 (d, Ar-CH), 129.3 (d, Ar-CH), 128.4 (d, 2C, 2 × Ar-CH), 126.8 (d, Ar-CH), 126.0 (d, 2C, 2 × Ar-CH), 120.9 (s, Ar-C), 106.6 (d, Ar-CH), 98.9 (d, Ar-CH), 55.4 (q, Ar-OCH_3), 32.7 (t, CH_2) ppm. HR-MS (ESI$^+$) m/z calculated for $[C_{16}H_{17}O_2]^+$=$[M+H]^+$: calculated: 241.1223; found: 241.1227.

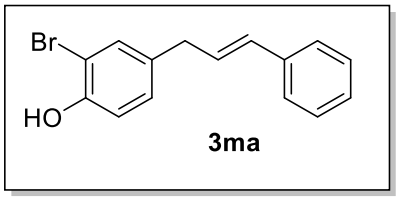


(*E*)-4-(3-(2-Bromophenyl)allyl)-3-methoxyphenol (3di): GP-1 was carried out with phenol **1d** (25 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2i** (64 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(2-bromophenyl)allyl)-3-methoxyphenol **3di** (40 mg, 62%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1d**) = 0.70, R_f(**2i**) = 0.40, R_f(**3di**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3329, 2942, 2831, 1593, 1444, 1021, 656 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.56 – 7.44 (m, 2H, Ar-H), 7.25 – 7.17 (m, 1H, Ar-H), 7.04 (ddd, *J* = 8.1, 6.4 and 2.5 Hz, 2H, Ar-H), 6.75 (d, *J* = 15.6 Hz, 1H, C*H*=CH-CH_2), 6.43 (d, *J* = 2.4 Hz, 1H, Ar-H), 6.37 (dd, *J* = 8.1 and 2.4 Hz, 1H, Ar-H), 6.27 (dt, *J* = 15.6 and 6.9 Hz, 1H, CH=C*H*-CH_2), 4.76 (s, 1H, Ar-O*H*), 3.82 (s, 3H, Ar-OCH_3), 3.49 (dd, *J* = 6.9 and 1.4 Hz, 2H, CH=CH-C*H*$_2$) ppm. ^{13}C{H}-NMR (100 MHz, $CDCl_3$) δ = 158.4 (s, Ar-C), 155.3 (s, Ar-C), 137.6 (s, Ar-C), 132.8 (d, Ar-CH), 132.3 (d, Ar-CH), 130.3 (d, Ar-CH), 129.3 (d, Ar-CH), 128.1 (d, Ar-CH), 127.3 (d, Ar-CH), 126.9 (d, Ar-CH), 123.2 (s, Ar-C), 120.5 (s, Ar-C), 106.7 (d, Ar-CH), 98.9 (d, Ar-CH), 55.4 (q, Ar-OCH_3), 33.0 (t, CH_2) ppm. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{16}Br^{79}O_2]^+$=$[M+H]^+$: calculated: 319.0328; found: 319.0303. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{16}Br^{81}O_2]^+$=$[M+H]^+$: calculated: 321.0308; found: 321.0285.



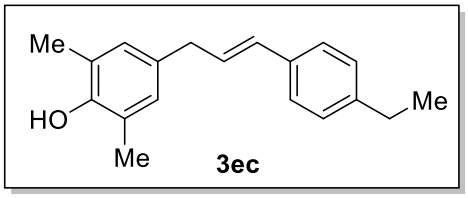
(*E*)-2-Fluoro-4-(3-(*p*-tolyl)allyl)phenol (3lb): GP-1 was carried out with phenol **1l** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allyl alcohol **2b** (44 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 85:15) furnished (*E*)-2-Fluoro-4-(3-(*p*-tolyl)allyl)phenol **3lb** (37 mg, 77%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 85:15),

R_f(**1l**) = 0.80, R_f(**2b**) = 0.50, R_f(**3lb**) = 0.40, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3376, 3004, 1579, 1487, 1439, 1210, 1147, 958, 829, 745 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.31 (d, *J* = 8.1 Hz, 2H), 7.14 (d, *J* = 7.9 Hz, 2H), 6.99 – 6.96 (m, 1H), 6.94 – 6.82 (m, 2H), 6.69 (d, *J* = 16.0 Hz, 1H), 6.35 (dt, *J* = 16.0, 5.9 Hz, 1H), 4.65 (dd, *J* = 5.9, 1.4 Hz, 2H), 2.34 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ = 158.5, 154.7, 137.9, 133.5, 133.2, 129.3, 129.1, 129.1, 127.0, 126.5, 123.1, 115.9, 115.8, 115.7, 115.7, 69.4, 21.2 ppm. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{13}FNa]^+$=[M+Na]–$[H_2O]^+$: calculated: 247.0893; found: 247.0881.



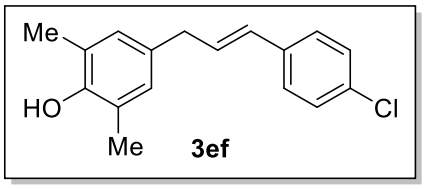
3ma

2-Bromo-4-cinnamylphenol (**3ma**): **GP-1** was carried out with phenol **1m** (35 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allyl alcohol **2a** (40 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 85:15) furnished 2-Bromo-4-cinnamylphenol **3ma** (48 mg, 82%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 85:15), R_f(**1m**) = 0.70, R_f(**2a**) = 0.30, R_f(**3ma**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3302, 3027, 2890, 1685, 1582, 1477, 1432, 1272, 1165, 957, 741, 691 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36 – 7.28 (m, 5H), 7.21 (ddd, *J* = 7.2, 3.9, 1.3 Hz, 1H), 7.08 (dd, *J* = 8.3, 2.0 Hz, 1H), 6.95 (d, *J* = 8.3 Hz, 1H), 6.43 (d, *J* = 15.7 Hz, 1H), 6.29 (dt, *J* = 15.7, 6.8 Hz, 1H), 5.41 (s, 1H), 3.45 (d, *J* = 6.8 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ = 150.6, 137.2, 133.8, 131.8, 131.3, 129.4, 128.6, 128.5, 127.2, 126.1, 115.9, 110.1, 38.1 ppm. HR-MS (ESI^+) m/z calculated for $[C_{15}H_{14}O]^+$=[M+H]–$[Br]^+$: calculated: 210.1039; found: 210.1067.



3ec

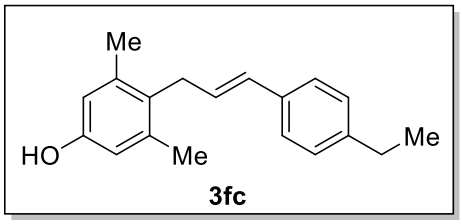
(*E*)-4-(3-(4-Ethylphenyl)allyl)-2,6-dimethylphenol (3ec): GP-1 was carried out with phenol **1e** (25 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2c** (49 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(4-ethylphenyl)allyl)-2,6-dimethylphenol **3ec** (46 mg, 87%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1e**) = 0.70, R_f(**2c**) = 0.40, R_f(**3ec**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3056, 2923, 2852, 1603, 1483, 1445, 1378, 1195, 1076, 1022, 941, 809, 764, 693 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.31 (d, 2H, *J* = 8.1 Hz, Ar-H), 7.15 (d, 2H, *J* = 8.1 Hz, Ar-H), 6.87 (s, 2H, Ar-H), 6.44 (d, 1H, *J* = 15.7 Hz, C*H*=CH-CH_2), 6.30 (dt, 1H, *J* = 15.7 and 6.8 Hz, CH=C*H*-CH_2), 4.55 (s, 1H, Ar-O*H*), 3.43 (d, 2H, *J* = 6.8 Hz, CH=CH-C*H*$_2$), 2.64 (q, 2H, *J* = 7.6 Hz, Ar-C*H*$_2$$CH_3$), 2.24 (s, 6H, 2 × Ar-$CH_3$), 1.24 (t, 3H, *J* = 7.6 Hz, Ar-CH_2C*H*$_3$) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 150.5 (s, Ar-C), 143.2 (s, Ar-C), 135.1 (s, Ar-C), 131.8 (s, Ar-C), 130.4 (d, CH=C*H*-CH_2), 128.9 (d, CH=C*H*-CH_2), 128.7 (d, 2C, 2 × Ar-CH), 128.0 (d, 2C, 2 × Ar-CH), 126.1 (d, 2C, 2 × Ar-CH), 123.0 (s, Ar-C), 38.5 (t, CH_2), 28.5 (t, Ar-*C*H_2CH_3), 15.8 (q, 2 × Ar-CH_3), 15.7 (q, Ar-CH_2*C*H_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{19}H_{21}O]^+$=$[M–H]^+$: calculated: 265.1587; found: 265.1588.



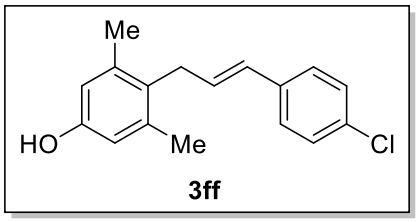
3ef

(*E*)-4-(3-(4-Chlorophenyl)allyl)-2,6-dimethylphenol (3ef): GP-1 was carried out with phenol **1e** (25 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2f** (51 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(4-chlorophenyl)allyl)-2,6-dimethylphenol **3ef** (46 mg, 84%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1e**) = 0.70, R_f(**2f**) = 0.40, R_f(**3ef**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3304, 2923, 2872, 1587, 1494, 1446, 1328, 1210, 1161, 1010, 955, 817, 737, 659 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.27 – 7.22 (m, 4H, Ar-H), 6.83 (s, 2H, Ar-H), 6.40 – 6.24 (m, 2H, C*H*=C*H*-CH_2), 4.53 (s, 1H, Ar-O*H*), 3.40 (d, 2H, *J* = 6.2 Hz, CH=CH-C*H*$_2$), 2.22 (s, 6H, 2 × Ar-CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 150.6 (s, Ar-

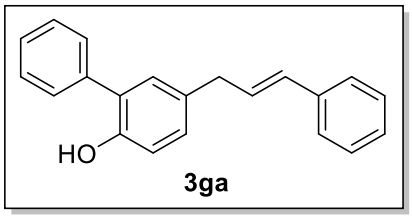
C), 136.1 (s, Ar-C), 132.5 (s, Ar-C), 131.3 (s, Ar-C), 130.7 (d, CH=C*H*-CH_2), 129.2 (d, CH=C*H*-CH_2), 128.7 (d, 2C, 2 × Ar-CH), 128.6 (d, 2C, 2 × Ar-CH), 127.2 (d, 2C, 2 × Ar-CH), 123.1 (s, Ar-C), 38.5 (t, CH_2), 15.9 (q, 2 × Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{17}H_{18}ClO]^+$=$[M+H]^+$: calculated: 273.1041; found: 373.1033.



(*E*)-4-(3-(4-Ethylphenyl)allyl)-3,5-dimethylphenol (3fc): GP-1 was carried out with phenol **1f** (25 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2c** (49 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(4-ethylphenyl)allyl)-3,5-dimethylphenol **3fc** (41 mg, 78%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1f**) = 0.70, R_f(**2c**) = 0.40, R_f(**3fc**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3399, 2993, 2870, 1587, 1494, 1446, 1420, 1326, 1210, 1160, 1010, 955, 817, 738 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ = 7.24 (d, 2H, *J* = 8.2 Hz, Ar-H), 7.12 (d, 2H, *J* = 8.2 Hz, Ar-H), 6.55 (s, 2H, Ar-H), 6.32 – 6.05 (m, 2H, C*H*=C*H*-CH_2), 4.82 (s, 1H, Ar-O*H*), 3.47 (d, 2H, *J* = 4.2 Hz, CH=CH-CH_2), 2.62 (q, 2H, *J* = 7.6 Hz, Ar-CH_2CH_3), 2.29 (s, 6H, 2 × Ar-CH_3), 1.22 (t, 3H, *J* = 7.6 Hz, Ar-CH_2CH_3) ppm. $^{13}C\{H\}$-NMR ($CDCl_3$,100 MHz): δ = 153.3 (s, Ar-C), 143.1 (s, Ar-C), 138.3 (s, Ar-C), 135.1 (s, Ar-C), 129.5 (d, CH=C*H*-CH_2), 128.6 (s, Ar-C), 127.9 (d, 2C, 2 × Ar-CH), 126.9 (d, CH=C*H*-CH_2), 125.9 (d, 2C, 2 × Ar-CH), 114.7 (d, 2C, 2 × Ar-CH), 32.1 (t, CH_2), 28.5 (t, Ar-*C*H_2CH_3), 20.0 (q, 2 × Ar-CH_3), 15.6 (q, Ar-CH_2*C*H_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{19}H_{23}O]^+$=$[M+H]^+$: calculated: 267.1743; found: 267.1739.



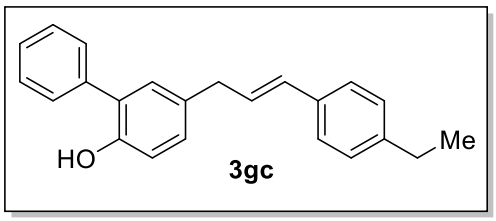
(*E*)-4-(3-(4-Chlorophenyl)allyl)-3,5-dimethylphenol (3ff): GP-1 was carried out with phenol **1f** (25 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2f** (51 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-(4-chlorophenyl)allyl)-3,5-dimethylphenol **3ff** (40 mg, 73%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1f**) = 0.70, R_f(**2f**) = 0.40, R_f(**3ff**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3294, 2990, 1629, 1494, 1449, 1324, 1210, 1159, 1011, 953, 817, 735, 659 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.21 (d, 4H, *J* = 0.7 Hz, Ar-H), 6.54 (s, 2H, Ar-H), 6.24 (dt, 1H, *J* = 15.8 and 5.6 Hz, CH=C*H*-CH_2), 6.13 (dt, 1H, *J* = 15.8 and 1.6 Hz, C*H*=CH-CH_2), 4.62 (s, 1H, Ar-O*H*), 3.45 (dd, 2H, *J* = 5.6 and 1.5 Hz, CH=CH-C*H*$_2$), 2.27 (s, 6H, 2 × Ar-CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$, 100 MHz): δ = 153.4 (s, Ar-C), 138.3 (s, Ar-C), 136.1 (s, Ar-C), 132.4 (s, Ar-C), 128.7 (d, CH=C*H*-CH_2), 128.5 (d, 2C, 2 × Ar-CH), 128.5 (d, CH=C*H*-CH_2), 128.2 (s, Ar-C), 127.2 (d, 2C, 2 × Ar-CH), 114.8 (d, 2C, 2 × Ar-CH), 32.0 (t, CH_2), 20.0 (q, 2 × Ar-CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{17}H_{18}ClO]^+$=$[M+H]^+$: calculated: 273.1041; found: 273.1040.



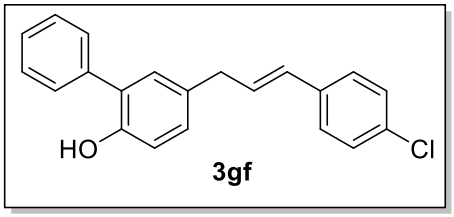
3ga

5-Cinnamyl-[1,1'-biphenyl]-2-ol (3ga): GP-1 was carried out with phenol **1g** (34 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2a** (40 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished 5-cinnamyl-[1,1'-biphenyl]-2-ol **3ga** (43 mg, 76%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1g**) = 0.70, R_f(**2a**) = 0.30, R_f(**3ga**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3303, 2994, 1588, 1494, 1446, 1420, 1209, 1160, 1010, 955, 817, 737, 659 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.49 (d, 4H, *J* = 4.5 Hz, Ar-H), 7.44 – 7.36 (m, 3H, Ar-H), 7.31 (t, 2H, *J* = 7.6 Hz, Ar-H), 7.22 (t, 1H, *J* = 7.3 Hz, Ar-H), 7.18 – 7.11 (m, 2H, Ar-H), 6.95 (d, 1H, *J* = 8.0 Hz, Ar-H), 6.49 (d, 1H, *J* = 15.8 Hz, C*H*=CH-CH_2), 6.38 (dt, 1H, *J* = 15.8 and 6.6 Hz, CH=C*H*-CH_2), 5.18 (s, 1H, Ar-O*H*),

3.54 (d, 2H, J = 6.6 Hz, CH=CH-CH_2) ppm. ^{13}C{H}-NMR($CDCl_3$,100MHz): δ = 150.8 (s, Ar-C), 137.4 (s, Ar-C), 137.1 (s, Ar-C), 132.4 (s, Ar-C), 130.8 (d, Ar-CH), 130.3 (d, Ar-CH), 129.4 (d, Ar-CH), 129.2 (d, Ar-CH), 129.2 (d, 2C, 2 × Ar-CH), 129.0 (d, 2C, 2 × Ar-CH), 128.5 (d, 2C, 2 × Ar-CH), 128.1 (s, Ar-C), 127.8 (d, Ar-CH), 127.1 (d, Ar-CH), 126.1 (d, 2C, 2 × Ar-CH), 115.8 (d, Ar-CH), 32.0 (t, CH_2) ppm. HR-MS (ESI$^+$) m/z calculated for $[C_{21}H_{19}O]^+$=$[M+H]^+$: calculated: 287.1430; found: 287.1422.

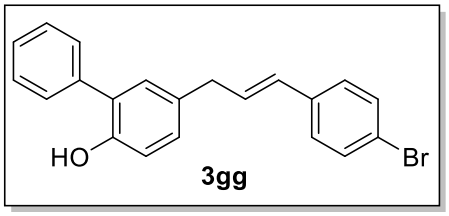


(*E*)-5-(3-(4-Ethylphenyl)allyl)-[1,1'-biphenyl]-2-ol (3gc): GP-1 was carried out with phenol **1g** (34 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2c** (49 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-5-(3-(4-ethylphenyl)allyl)-[1,1'-biphenyl]-2-ol **3gc** (46 mg, 73%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1g**) = 0.70, R_f(**2c**) = 0.30, R_f(**3gc**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3381, 2893, 2823, 1490, 1443, 1248, 1188, 1102, 960, 808 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.54 – 7.44 (m, 4H, Ar-H), 7.42 – 7.36 (m, 1H, Ar-H), 7.29 (d, 2H, J = 8.1 Hz, Ar-H), 7.13 (d, 4H, J = 7.9 Hz, Ar-H), 6.93 (d, 1H, J = 8.1 Hz, Ar-H), 6.45 (d, 1H, J = 15.8 Hz, C*H*=CH-CH_2), 6.32 (dt, 1H, J = 15.8 and 6.8 Hz, CH=C*H*-CH_2), 5.14 (s, 1H, Ar-O*H*), 3.51 (d, 2H, J = 6.8 Hz, CH=CH-CH_2), 2.62 (q, 2H, J = 7.6 Hz, Ar-C$H_2$$CH_3$), 1.22 (t, 3H, J = 7.6 Hz, Ar-CH_2CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 150.7 (s, Ar-C), 143.3 (s, Ar-C), 137.1 (s, Ar-C), 134.9 (s, Ar-C), 132.6 (s, Ar-C), 130.7 (d, Ar-CH), 130.3 (d, Ar-CH), 129.2 (d, Ar-CH), 129.2 (d, 2C, 2 × Ar-CH), 129.1 (d, 2C, 2 × Ar-CH), 128.5 (d, Ar-CH), 128.0 (s, Ar-C), 128.0 (d, 2C, 2 × Ar-CH), 127.8 (d, Ar-CH), 126.1 (d, 2C, 2 × Ar-CH), 115.8 (d, Ar-CH), 38.5 (t, CH_2), 28.5 (t, Ar-*C*$H_2$$CH_3$), 15.6 (q, Ar-$CH_2$*C*$H_3$) ppm. HR-MS (ESI$^+$) m/z calculated for $[C_{23}H_{23}O]^+$=$[M+H]^+$: calculated: 315.1743; found: 315.1744.



3gf

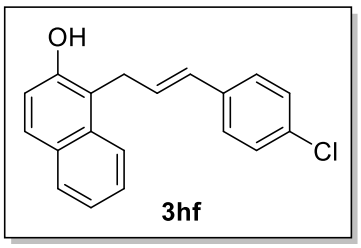
(*E*)-5-(3-(4-Chlorophenyl)allyl)-[1,1'-biphenyl]-2-ol (3gf): GP-1 was carried out with phenol **1g** (34 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2f** (51 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 18 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-5-(3-(4-chlorophenyl)allyl)-[1,1'-biphenyl]-2-ol **3gf** (45 mg, 70%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1g**) = 0.70, R_f(**2f**) = 0.30, R_f(**3gf**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3299, 2994, 1588, 1494, 1446, 1420, 1208, 1160, 1010, 955, 817, 736 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ = 7.45 (d, 4H, *J* = 4.5 Hz, Ar-H), 7.36 (ddd, 1H, *J* = 8.8, 5.1, 3.7 Hz, Ar-H), 7.24 (d, 4H, *J* = 2.9 Hz, Ar-H), 7.14 – 7.07 (m, 2H, Ar-H), 6.92 (d, 1H, *J* = 8.1 Hz, Ar-H), 6.38 (d, 1H, *J* = 15.8 Hz, C*H*=CH-CH_2), 6.31 (dt, 1H, *J* = 15.8 and 6.2 Hz, CH=C*H*-CH_2), 5.17 (s, 1H, Ar-O*H*), 3.48 (d, 2H, *J* = 6.2 Hz, CH=CH-CH_2) ppm. $^{13}C\{H\}$-NMR ($CDCl_3$, 100 MHz): δ = 150.8 (s, Ar-C), 137.1 (s, Ar-C), 135.9 (s, Ar-C), 132.6 (s, Ar-C), 132.1 (s, Ar-C), 130.3 (d, Ar-CH), 130.2 (d, Ar-CH), 129.6 (d, Ar-CH), 129.2 (d, Ar-CH), 129.2 (d, 2C, 2 × Ar-CH), 129.0 (d, 2C, 2 × Ar-CH), 128.6 (d, 2C, 2 × Ar-CH), 128.1 (s, Ar-C), 127.8 (d, Ar-CH), 127.3 (d, 2C, 2 × Ar-CH), 115.9 (d, Ar-CH), 38.5 (t, CH_2) ppm. HR-MS (ESI^+) m/z calculated for $[C_{21}H_{18}ClO]^+$=$[M+H]^+$: calculated: 321.1041; found: 321.1038.



3gg

(*E*)-5-(3-(4-Bromophenyl)allyl)-[1,1'-biphenyl]-2-ol (3gg): GP-1 was carried out with phenol **1g** (34 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2g** (64 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 20 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-5-(3-(4-bromophenyl)allyl)-[1,1'-biphenyl]-2-ol **3gg** (47 mg, 64%) as a pale-yellow liquid. [TLC control

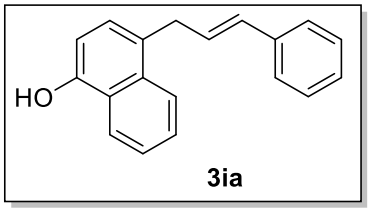
(petroleum ether/ethyl acetate 93:07), R_f(**1g**) = 0.70, R_f(**2g**) = 0.30, R_f(**3gg**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3393, 2895, 2818, 1583, 1452, 1258, 1184, 1076, 1013, 953, 738 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.51 – 7.44 (m, 4H, Ar-H), 7.42 – 7.36 (m, 3H, Ar-H), 7.24 – 7.17 (m, 2H, Ar-H), 7.15 – 7.07 (m, 2H, Ar-H), 6.93 (d, 1H, *J* = 8.1 Hz, Ar-H), 6.45 – 6.25 (m, 2H, C*H*=C*H*-CH_2), 5.14 (s, 1H, Ar-O*H*), 3.50 (d, 2H, *J* = 5.3 Hz, CH=CH-CH_2) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 150.9 (s, Ar-C), 137.1 (s, Ar-C), 136.4 (s, Ar-C), 132.0 (s, Ar-C), 131.5 (d, 2C, 2 × Ar-CH), 130.4 (d, Ar-CH), 130.3 (d, Ar-CH), 129.7 (d, Ar-CH), 129.3 (d, 3C, 3 × Ar-CH), 129.0 (d, 2C, 2 × Ar-CH), 128.1 (s, Ar-C), 127.9 (d, Ar-CH), 127.7 (d, 2C, 2 × Ar-CH), 120.7 (s, Ar-C), 115.9 (d, Ar-CH), 38.5 (t, CH_2) ppm. HR-MS (ESI^+) m/z calculated for $[C_{21}H_{21}Br^{79}NO]^+$=$[M+NH_4]^+$: calculated: 382.0801; found: 382.0799. calculated for $[C_{21}H_{21}Br^{81}NO]^+$=$[M+NH_4]^+$: calculated: 384.0781; found: 384.0808.



3hf

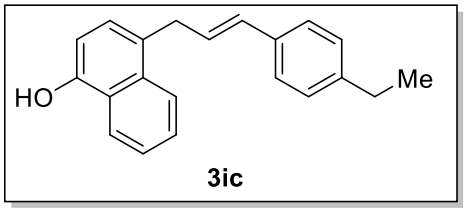
(*E*)-1-(3-(4-Chlorophenyl)allyl)naphthalen-2-ol (3hf): GP-1 was carried out with phenol **1h** (29 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2f** (51 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 16 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-1-(3-(4-chlorophenyl)allyl)naphthalen-2-ol **3hf** (47 mg, 79%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1h**) = 0.70, R_f(**2f**) = 0.40, R_f(**3hf**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3329, 2942, 2831, 1593, 1444, 1021, 656 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.95 (d, 1H, *J* = 8.5 Hz, Ar-H), 7.80 (d, 1H, *J* = 8.1 Hz, Ar-H), 7.69 (d, 1H, *J* = 8.8 Hz, Ar-H), 7.50 (ddd, 1H, *J* = 8.4, 6.8, 1.3 Hz, Ar-H), 7.36 (ddd, 1H, *J* = 8.0, 6.9, 1.0 Hz, Ar-H), 7.19 (s, 4H, Ar-H), 7.09 (d, 1H, *J* = 8.8 Hz, Ar-H), 6.67 – 6.18 (m, 2H, C*H*=C*H*-CH_2), 5.13 (s, 1H, Ar-O*H*), 3.97 (d, 2H, *J* = 4.9 Hz, CH=CH-CH_2) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 151.0 (s, Ar-C), 135.7 (s, Ar-C), 133.2 (s, Ar-C), 132.6 (s, Ar-C), 129.5 (d, Ar-CH), 129.4 (s, Ar-C), 128.6 (d, Ar-CH), 128.5 (d, 2C, 2 × Ar-CH), 128.4 (d, 2C, 2 × Ar-CH), 127.3 (d, 2C, 2 × Ar-CH), 126.7 (d, Ar-CH), 123.3 (d, Ar-CH), 123.0 (d, Ar-CH), 117.8 (d, Ar-CH), 117.8

(s, Ar-C), 28.3 (t, CH_2) ppm. HR-MS (ESI^+) m/z calculated for $[C_{19}H_{15}ClNaO]^+$=$[M+Na]^+$: calculated: 317.0704; found: 317.0728.



3ia

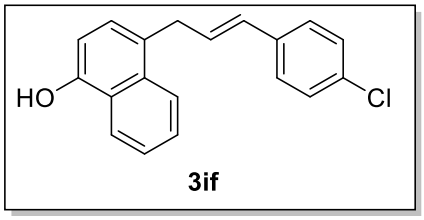
4-Cinnamylnaphthalen-1-ol (3ia): GP-1 was carried out with phenol **1i** (29 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2a** (40 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 14 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished 4-cinnamylnaphthalen-1-ol **3ia** (43 mg, 82%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1i**) = 0.70, R_f(**2a**) = 0.40, R_f(**3ia**) = 0.50, UV detection]. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.19 – 8.11 (m, 1H), 7.94 (d, J = 7.8 Hz, 1H), 7.49 – 7.37 (m, 2H), 7.24 (d, J = 7.6 Hz, 2H), 7.21 – 7.16 (m, 2H), 7.11 (dd, J = 13.2, 7.3 Hz, 2H), 6.67 (d, J = 7.6 Hz, 1H), 6.46 – 6.27 (m, 2H), 3.82 (d, J = 5.1 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.4, 137.5, 133.0, 131.0, 129.3, 128.6, 128.4, 127.0, 126.5, 126.2, 126.0, 124.9, 124.7, 124.1, 122.2, 108.2, 36.0 ppm.



3ic

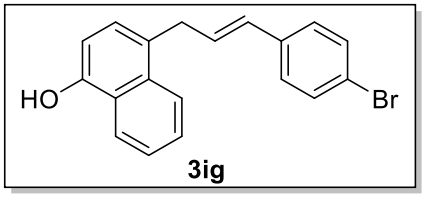
(*E*)-4-(3-(4-ethylphenyl)allyl)naphthalen-1-ol (3ic): GP-1 was carried out with phenol **1i** (29 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2c** (49 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 14 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-2-(3-(4-ethylphenyl)allyl)naphthalen-1-ol **3ic** (38 mg, 65%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1i**) = 0.70, R_f(**2c**) = 0.40, R_f(**3ic**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3382, 2893, 2827, 1490, 1442, 1322, 1248, 1187, 1102, 960, 807 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.31 – 8.13 (m, 1H, Ar-H), 8.02 (dd, 1H, J = 7.2, 2.1

Hz, Ar-H), 7.59 – 7.46 (m, 2H, Ar-H), 7.25 (t, 2H, *J* = 4.1 Hz, Ar-H), 7.20 (d, 1H, *J* = 7.6 Hz, Ar-H), 7.10 (d, 2H, *J* = 8.2 Hz, Ar-H), 6.76 (d, 1H, *J* = 7.6 Hz, Ar-H), 6.57 – 6.22 (m, 2H, C*H*=C*H*-CH_2), 5.28 (s, 1H, Ar-O*H*), 3.89 (d, 2H, *J* = 4.2 Hz, CH=CH-C*H*$_2$), 2.60 (q, 2H, *J* = 7.6 Hz, Ar-C*H*$_2$$CH_3$), 1.20 (t, 3H, *J* = 7.6 Hz, Ar-CH_2C*H*$_3$) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 151.0 (s, Ar-C), 135.7 (s, Ar-C), 133.2 (s, Ar-C), 132.6 (s, Ar-C), 129.5 (d, Ar-CH), 129.4 (s, Ar-C), 128.6 (d, Ar-CH), 128.5 (d, 2C, 2 × Ar-CH), 128.4 (d, 2C, 2 × Ar-CH), 127.3 (d, 2C, 2 × Ar-CH), 126.7 (d, Ar-CH), 123.3 (d, Ar-CH), 123.0 (d, Ar-CH), 117.8 (d, Ar-CH), 117.8 (s, Ar-C), 28.3 (t, CH_2) ppm. HR-MS (ESI$^+$) m/z calculated for $[C_{21}H_{21}O]^+$=$[M+H]^+$: calculated: 289.1587; found: 289.1579.

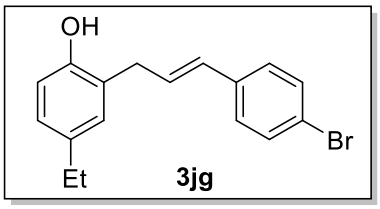


3if

(*E*)-4-(3-(4-Chlorophenyl)allyl)naphthalen-1-ol (3if): GP-1 was carried out with phenol **1i** (29 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2f** (51 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 18 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-2-(3-(4-chlorophenyl)allyl)naphthalen-1-ol **3if** (37 mg, 63%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1i**) = 0.70, R_f(**2f**) = 0.40, R_f(**3if**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3312, 2991, 1594, 1580, 1494, 1449, 1420, 1210, 1158, 1010, 953, 817, 735, 659 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.13 (dd, 1H, *J* = 4.7, 4.1 Hz, Ar-H), 7.78 (dd, 1H, *J* = 6.1, 3.0 Hz, Ar-H), 7.51 – 7.39 (m, 2H, Ar-H), 7.35 – 7.05 (m, 6H, Ar-H), 6.46 (d, 1H, *J* = 15.7 Hz, C*H*=CH-CH_2), 6.37 (dt, 1H, *J* = 15.7 and 5.9 Hz, CH=C*H*-CH_2), 5.46 (d, 1H, *J* = 2.4 Hz, Ar-O*H*), 3.69 (d, 2H, *J* = 5.9 Hz, CH=CH-C*H*$_2$) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 149.3 (s, Ar-C), 135.2 (s, Ar-C), 133.8 (s, Ar-C), 133.1 (s, Ar-C), 130.5 (d, Ar-CH), 128.7 (d, 2C, 2 × Ar-CH), 128.3 (d, Ar-CH), 128.2 (d, Ar-CH), 127.6 (d, Ar-CH), 127.4 (d, 2C, 2 × Ar-CH), 125.8 (d, Ar-CH), 125.4 (d, Ar-CH), 124.7 (s, Ar-C), 121.1 (d, Ar-CH), 120.6 (d, Ar-CH), 118.1 (s, Ar-C), 34.4 (t, CH_2) ppm. HR-MS (ESI$^+$) m/z calculated for $[C_{19}H_{16}ClO]^+$=$[M+H]^+$: calculated: 295.0884; found: 295.0878.

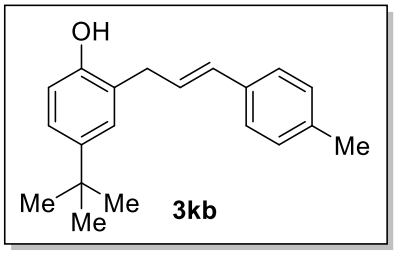


(*E*)-4-(3-(4-Bromophenyl)allyl)naphthalen-1-ol (3ig): GP-1 was carried out with phenol **1i** (29 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2g** (64 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 20 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-2-(3-(4-bromophenyl)allyl)naphthalen-1-ol **3ig** (42 mg, 61%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1i**) = 0.70, R_f(**2g**) = 0.40, R_f(**3ig**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3304, 2994, 1587, 1494, 1421, 1330, 1211, 1163, 1011, 955, 817, 738, 660 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.23 – 8.13 (m, 1H, Ar-H), 7.91 – 7.78 (m, 1H, Ar-H), 7.48 (ddd, 3H, *J* = 14.1, 7.2, 5.2 Hz, Ar-H), 7.44 – 7.39 (m, 2H, Ar-H), 7.28 (d, 1H, *J* = 8.3 Hz, Ar-H) 7.22 – 7.17 (m, 2H, Ar-H), 6.54 – 6.36 (m, 2H, C*H*=C*H*-CH_2), 5.49 (s, 1H, Ar-O*H*), 3.70 (d, 2H, *J* = 5.2 Hz, CH=CH-CH_2) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 149.2 (s, Ar-C), 135.6 (s, Ar-C), 133.7 (s, Ar-C), 131.6 (d, 2C, 2 × Ar-CH), 130.5 (d, Ar-CH), 128.3 (d, Ar-CH), 128.3 (d, Ar-CH), 127.7 (d, 2C, 2 × Ar-CH), 127.6 (d, Ar-CH), 125.8 (d, Ar-CH), 125.4 (d, Ar-CH), 124.7 (s, Ar-C), 121.2 (s, Ar-C), 121.1 (d, Ar-CH), 120.6 (d, Ar-CH), 118.1 (s, Ar-C), 34.4 (t, CH_2) ppm. HR-MS (ESI^+) m/z calculated for $[C_{19}H_{16}Br^{79}O]^+$=$[M+H]^+$: calculated: 339.0379; found: 339.0365, calculated for $[C_{19}H_{16}Br^{81}O]^+$=$[M+H]^+$: calculated: 341.0359; found: 341.0374.



(*E*)-2-(3-(4-Bromophenyl)allyl)-4-ethylphenol (3jg): GP-1 was carried out with phenol **1j** (25 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2g** (64 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-2-(3-(4-

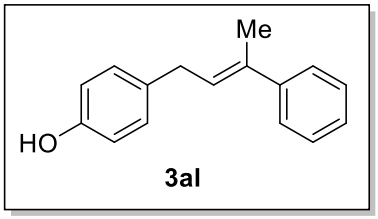
bromophenyl)allyl)-4-ethylphenol **3jg** (48 mg, 75%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1j**) = 0.70, R_f(**2g**) = 0.40, R_f(**3jg**) = 0.50, UV detection]. Mp: 123 – 125 °C; IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3303, 2993, 2873, 2805, 1587, 1494, 1421, 1331, 1212, 1162, 1011, 955, 817, 738, 660 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.42 – 7.36 (m, 2H, Ar-H), 7.23 – 7.18 (m, 2H, Ar-H), 7.01 – 6.93 (m, 2H, Ar-H), 6.73 (d, 1H, *J* = 8.5 Hz, Ar-H), 6.47 – 6.33 (m, 2H, C*H*=C*H*-CH_2), 4.81 (s, 1H, Ar-O*H*), 3.52 (d, 2H, *J* = 5.2 Hz, CH=CH-CH_2), 2.57 (q, 2H, *J* = 7.6 Hz, Ar-C$H_2$$CH_3$), 1.20 (t, 3H, *J* = 7.6 Hz, Ar-CH_2CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 151.7 (s, Ar-C), 136.8 (s, Ar-C), 136.1 (s, Ar-C), 131.5 (d, 2C, 2 × Ar-CH), 130.0 (d, Ar-CH), 129.9 (d, Ar-CH), 129.1 (d, Ar-CH), 127.7 (d, 2C, 2 × Ar-CH), 127.0 (d, Ar-CH), 125.2 (s, Ar-C), 120.9 (s, Ar-C), 115.6 (d, Ar-CH), 34.1 (t, CH_2), 28.0 (t, Ar-C$H_2$$CH_3$), 15.9 (q, Ar-$CH_2CH_3$) ppm. HR-MS ($ESI^+$) m/z calculated for $[C_{17}H_{18}Br^{79}O]^+$=$[M+H]^+$: calculated: 317.0536; found: 317.0492, calculated for $[C_{17}H_{18}Br^{81}O]^+$=$[M+H]^+$: calculated: 319.0515; found: 319.0510.



3kb

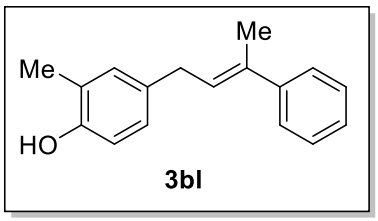
(*E*)-4-(*tert*-Butyl)-2-(3-(*p*-tolyl)allyl)phenol (3kb): GP-1 was carried out with phenol **1k** (30 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2b** (44 mg, 0.3 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(*tert*-butyl)-2-(3-(*p*-tolyl)allyl)phenol **3kb** (46 mg, 82%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1k**) = 0.70, R_f(**2b**) = 0.40, R_f(**3kb**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3308, 2993, 2872, 1587, 1494, 1446, 1420, 1210, 1160, 1010, 955, 817, 737 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.31 – 7.26 (m, 2H, Ar-H), 7.20 – 7.16 (m, 2H, Ar-H), 7.12 (d, *J* = 8.1 Hz, 2H, Ar-H), 6.77 (dd, *J* = 7.4, 1.4 Hz, 1H, Ar-H), 6.53 (d, *J* = 15.8 Hz, 1H, C*H*=CH-CH_2), 6.36 (dt, *J* = 15.8 and 6.6 Hz, 1H, CH=C*H*-CH_2), 4.97 (s, 1H, Ar-O*H*), 3.58 (dd, *J* = 6.6 and 0.9 Hz, 2H, CH=CH-CH_2), 2.34 (s, 3H, Ar-CH_3), 1.32 (s, 9H, Ar- 3 × CH_3) ppm. ^{13}C{H}-NMR ($CDCl_3$,100 MHz): δ = 151.9 (s, Ar-C), 143.7 (s, Ar-C), 137.0 (s, Ar-C), 134.3 (s, Ar-C),

131.2 (d, Ar-CH), 129.2 (d, 2C, 2 × Ar-CH), 127.3 (d, Ar-CH), 127.1 (d, Ar-CH), 126.1 (d, 2C, 2 × Ar-CH), 124.9 (s, Ar-C), 124.6 (d, Ar-CH), 115.3 (d, Ar-CH), 34.8 (t, CH_2), 34.1 (s, $C(CH_3)_3$), 31.6 (q, 3 × Ar-CH_3), 21.1 (q, Ar-*C*H_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{20}H_{25}O]^+$=$[M+H]^+$: calculated: 281.1900; found: 281.1894.



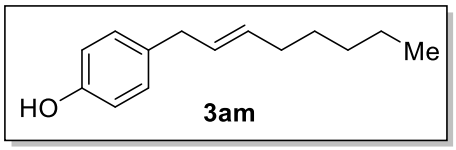
3al

(*E*)-4-(3-Phenylbut-2-en-1-yl)phenol (3al): GP-1 was carried out with phenol **1a** (19 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2l** (44.4 mg, 0.3 mmol), in DCE (2 mL) solvent, at rt for 12 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(3-phenylbut-2-en-1-yl)phenol **3al** (26 mg, 58%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1a**) = 0.70, R_f(**2l**) = 0.40, R_f(**3al**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3379, 2893, 2827, 1590, 1489, 1443, 1248, 1186, 1101, 1022, 901, 807 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45 – 7.39 (m, 2H, Ar-H), 7.36 – 7.28 (m, 2H, Ar-H), 7.23 (ddd, *J* = 6.3, 3.9, 1.3 Hz, 1H, Ar-H), 7.15 – 7.08 (m, 2H, Ar-H), 6.81 – 6.74 (m, 2H, Ar-H), 5.95 (ddd, *J* = 8.8, 6.0, 1.4 Hz, 1H, CH=C*H*-CH_2), 4.71 (s, 1H, Ar-O*H*), 3.50 (d, *J* = 7.4 Hz, 2H, CH=CH-CH_2), 2.14 (dt, *J* = 1.4, 0.7 Hz, 3H, CH_3) ppm. ^{13}C{H}- NMR (100 MHz, $CDCl_3$) δ=153.7 (s, Ar-C), 143.7 (s, Ar-C), 135.4 (s, Ar-C), 133.2 (s, Ar-C), 129.5 (d, 2 × Ar-CH), 128.2 (d, 2 × Ar-CH), 127.1 (d, Ar-CH), 126.7 (d, Ar-CH), 125.7 (d, 2 × Ar-CH), 115.3 (d, 2 × Ar-CH), 34.0 (t, CH_2), 15.9 (q, CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{16}H_{14}K]^+$=$[(M+K) – (H_2O)]^+$: calculated: 245.0727; found: 245.0730.



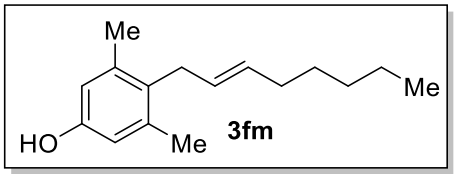
3bl

(*E*)-2-Methyl-4-(3-phenylbut-2-en-1-yl)phenol (3bl): GP-1 was carried out with phenol **1b** (22 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2l** (44.4 mg, 0.3 mmol), in DCE (2 mL) solvent, at rt for 12 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-2-methyl-4-(3-phenylbut-2-en-1-yl)phenol **3al** (27 mg, 57%) as a pale-yellow liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1b**) = 0.70, R_f(**2l**) = 0.40, R_f(**3bl**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3380, 2889, 2822, 1580, 1481, 1440, 1252, 1187, 1025, 911, 827 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41 (dd, *J* = 8.4, 1.1 Hz, 2H, Ar-H), 7.30 (dd, *J* = 8.1, 7.0 Hz, 2H, Ar-H), 7.25 – 7.19 (m, 1H, Ar-H), 6.98 (s, 1H, Ar-H), 6.93 (d, *J* = 8.1 Hz, 1H, Ar-H), 6.69 (d, *J* = 8.1 Hz, 1H, Ar-H), 5.99 – 5.88 (m, 1H, CH=C*H*-CH_2), 4.66 (d, *J* = 3.8 Hz, 1H, Ar-O*H*), 3.46 (d, *J* = 7.4 Hz, 2H, CH=CH-CH_2), 2.22 (s, 3H, Ar-CH_3), 2.13 (s, 3H, CH_3) ppm. ^{13}C{H}- NMR (100 MHz, $CDCl_3$) δ=151.9 (s, Ar-C), 143.6 (s, Ar-C), 135.1 (s, Ar-C), 133.1 (s, Ar-C), 131.0 (d, Ar-CH), 128.1 (d, 2 × Ar-CH), 127.3 (d, Ar-CH), 126.8 (d, Ar-CH), 126.6 (d, Ar-CH), 125.7 (d, 2 × Ar-CH), 123.7 (s, Ar-C), 114.8 (d, Ar-CH), 34.0 (t, CH_2), 15.9 (q, Ar-CH_3), 15.8 (q, CH_3) ppm. HR-MS (ESI^+) m/z calculated for $[C_{17}H_{16}K]^+$=[(M+K) – $(H_2O)]^+$: calculated: 259.0883; found: 259.0860.

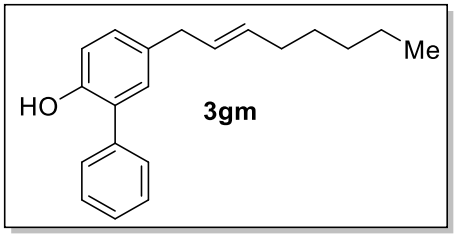


(*E*)-4-(Oct-2-en-1-yl)phenol (3am): GP-1 was carried out with phenol **1a** (19 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2m** (64 mg, 0.5 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-4-(oct-2-en-1-yl)phenol **3am** (26 mg, 63%) as a colorless liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1a**) = 0.70, R_f(**2m**) = 0.40, R_f(**3am**) = 0.50, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3301, 2893, 2821, 1588, 1495, 1429, 1344, 1213, 814 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.05 (d, *J* = 8.6 Hz, 2H, Ar-H), 6.75 (d, *J* = 8.6 Hz, 2H, Ar-H), 5.83 – 5.30 (m, 2H, CH=C*H*-CH_2), 4.63 (s, 1H, Ar-O*H*), 3.25 (d, *J* = 5.5 Hz, 2H, CH=CH-CH_2), 2.08 – 1.92 (m, 2H, CH_2), 1.33 – 1.26 (m, 6H, 3 × CH_2), 0.88 (m, 3H, CH_3) ppm. ^{13}C{H}-NMR (100 MHz, $CDCl_3$) δ = 131.9, 129.5, 129.0, 115.1, 38.1, 32.4,

31.4, 29.2, 22.5, 14.1, 1.0 ppm. HR-MS (ESI$^+$) m/z calculated for $[C_{14}H_{21}O]^+$=$[M+H]^+$: calculated: 205.1587; found: 205.1595.

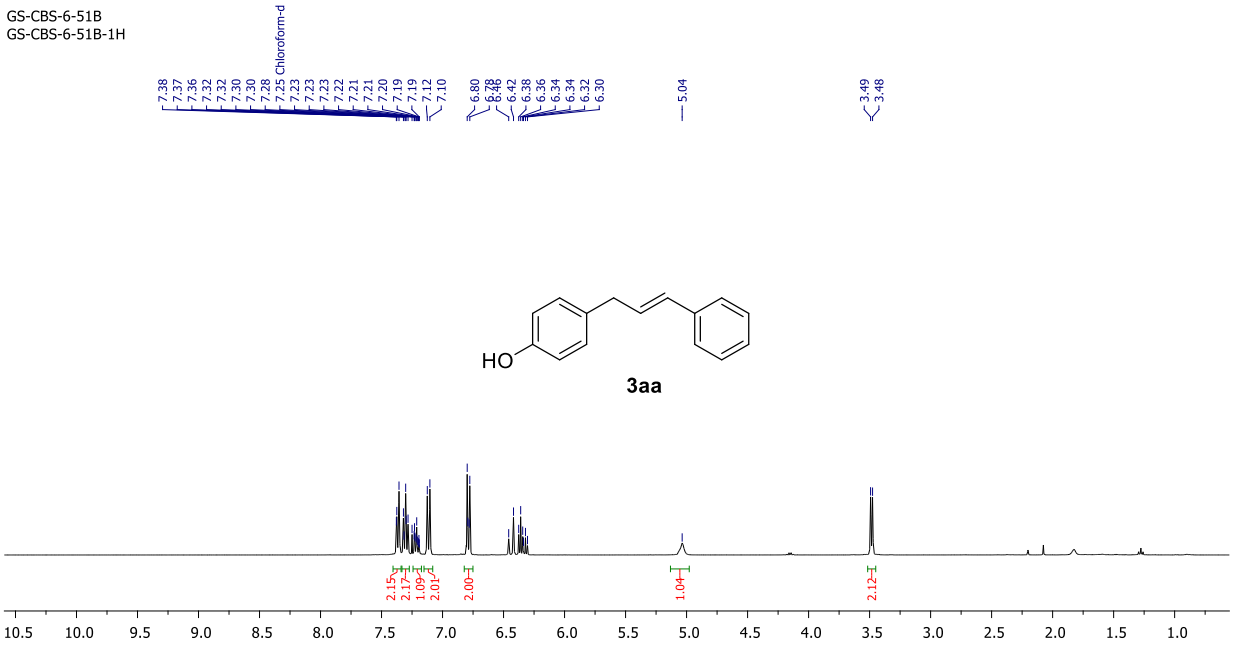


(*E*)-3,5-Dimethyl-4-(oct-2-en-1-yl)phenol (3fm): GP-1 was carried out with phenol **1a** (24 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2m** (64 mg, 0.5 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 95:05) furnished (*E*)-3,5-dimethyl-4-(oct-2-en-1-yl)phenol **3fm** (31 mg, 66%) as a colorless liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f(**1f**) = 0.70, R_f(**2m**) = 0.40, R_f(**3fm**) = 0.70, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}): ν_{max}=3318, 2894, 2830, 1581, 1445, 1293, 1137, 1016, 958, 828 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.50 (s, 2H), 5.44 – 5.37 (m, 1H), 5.29 – 5.21 (m, 1H), 4.59 (s, 1H), 3.22 (dd, *J* = 5.7, 1.3 Hz, 2H), 2.23 (s, 6H), 1.96 – 1.90 (m, 2H), 1.31 – 1.23 (m, 6H), 0.86 (t, *J* = 6.8 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ = 153.1, 138.1(2C), 130.8, 129.5, 126.8, 114.6(2C), 32.5, 31.8, 31.4, 29.2, 22.5, 19.9(2C), 14.1 ppm. HR-MS (ESI$^+$) m/z calculated for $[C_{16}H_{24}NaO]^+$=$[M+Na]^+$: calculated: 255.1719; found: 255.1920.

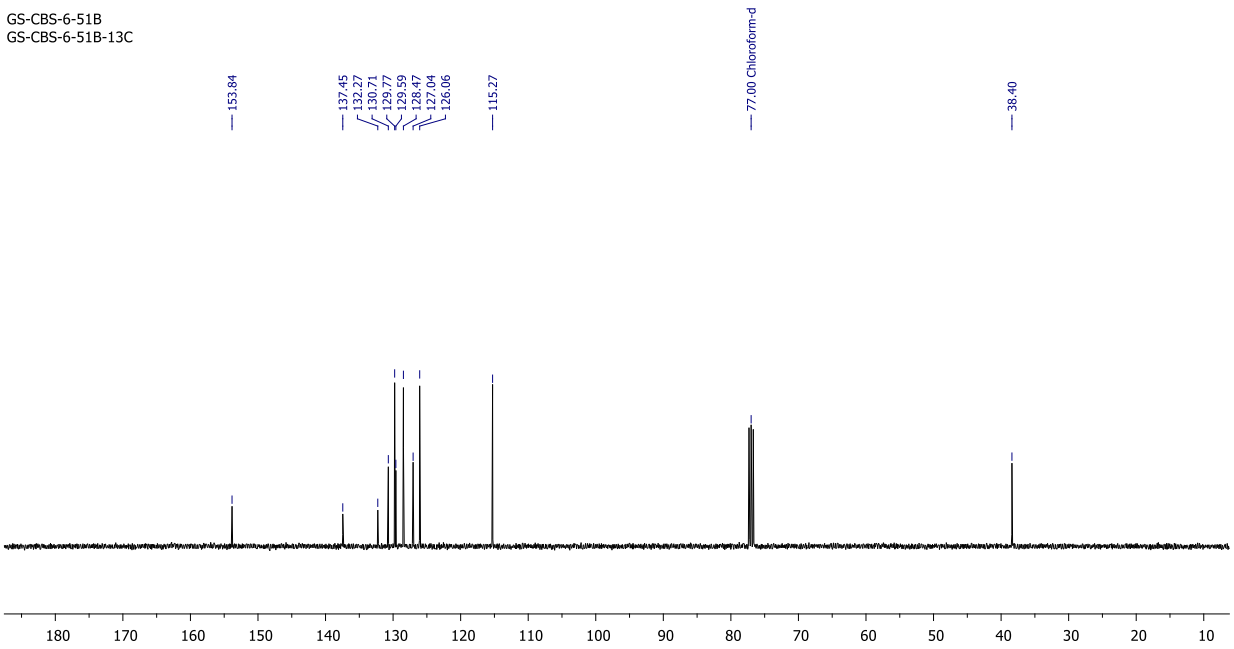


(*E*)-5-(Oct-2-en-1-yl)-[1,1'-biphenyl]-2-ol (3gm): GP-1 was carried out with phenol **1g** (34 mg, 0.2 mmol), $PdCl_2$ (4 mg, 0.02 mmol) and allylic alcohol **2m** (64 mg, 0.5 mmol), in DCE (2 mL) solvent, at 60 °C for 24 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 100:0 to 90:10) furnished (*E*)-5-(oct-2-en-1-yl)-[1,1'-biphenyl]-2-ol **3gm** (40 mg, 72%) as a colorless liquid. [TLC control (petroleum ether/ethyl acetate 93:07), R_f(**1g**) = 0.70, R_f(**2m**) = 0.40, R_f(**39m**) = 0.60, UV detection]. IR (MIR-ATR, 4000–600 cm^{-1}):

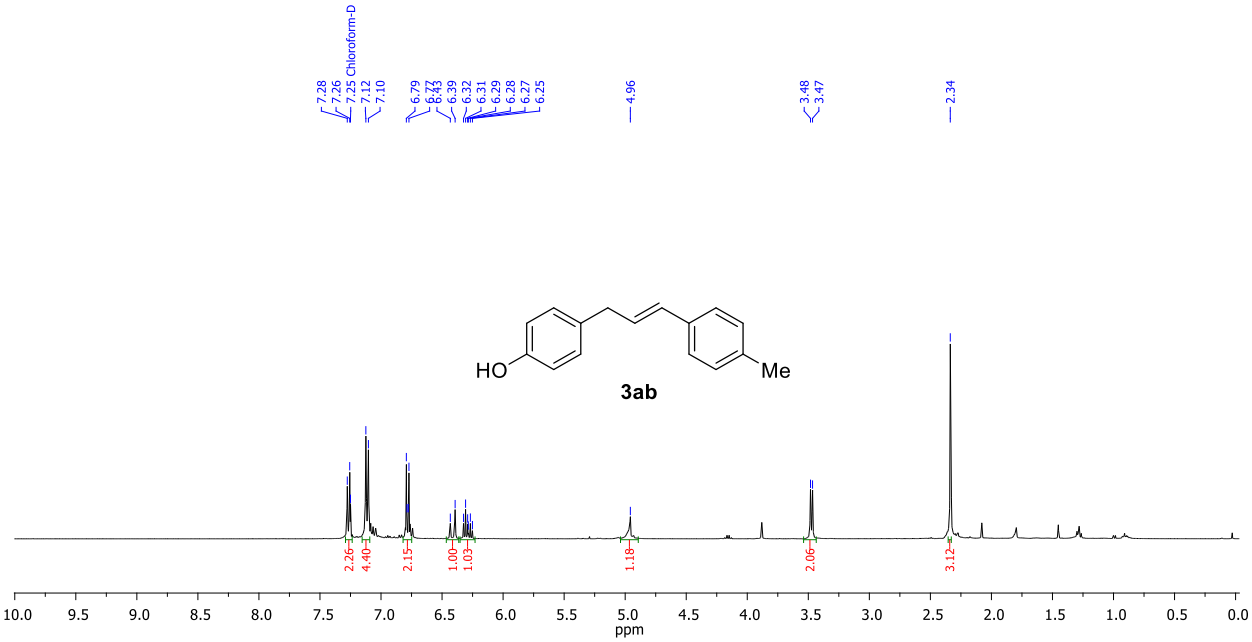
ν_{max}=3317, 2924, 2894, 2829, 1580, 1447, 1294, 1136, 1015, 960, 827 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.52 – 7.43 (m, 4H), 7.42 – 7.35 (m, 1H), 7.11 – 7.00 (m, 2H), 6.90 (d, *J* = 8.0 Hz, 1H), 5.60 – 5.43 (m, 2H), 5.10 (s, 1H), 3.28 (d, *J* = 5.6 Hz, 2H), 2.03 – 1.98 (m, 2H), 1.38 – 1.27 (m, 6H), 0.85 (t, *J* = 7.0 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ = 150.5, 137.3, 133.4, 132.0, 130.1, 129.2, 129.1(2C), 128.9, 127.8, 127.8, 115.7, 38.2, 32.5, 31.4, 29.1, 22.5, 14.1 ppm. HR-MS (ESI^+) m/z calculated for $[C_{20}H_{28}NO]^+$=$[M+NH_4]^+$: calculated: 298.2165; found: 298.2184.



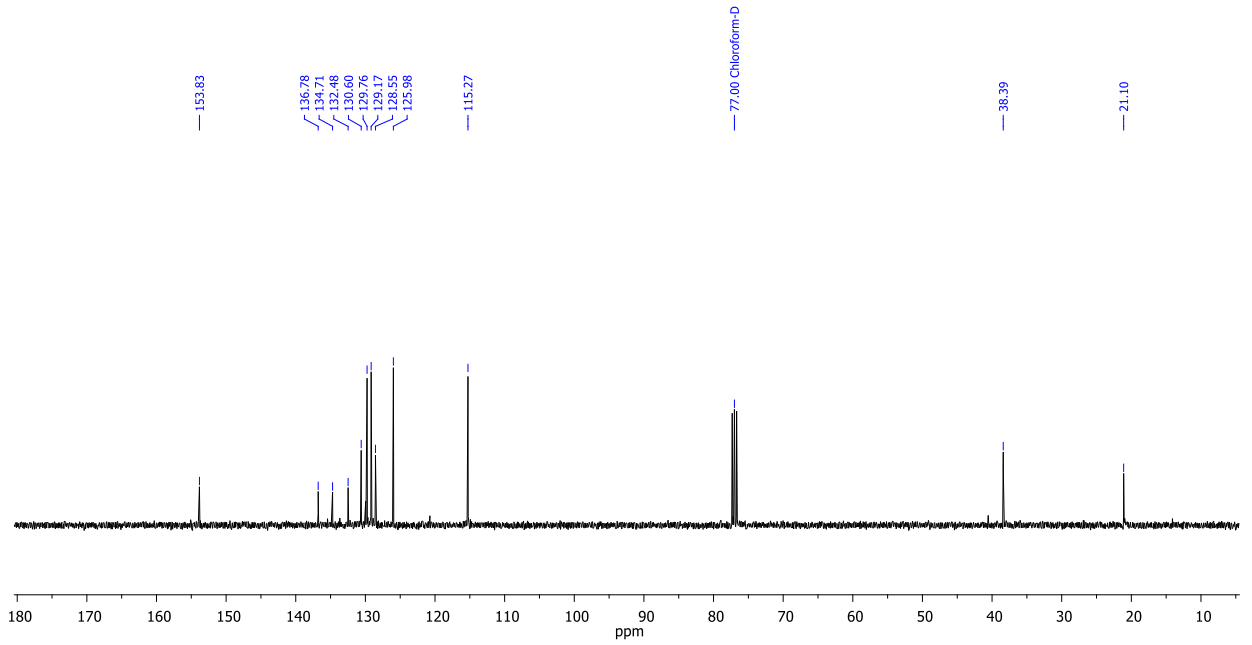
^{1}H NMR (400 MHz) spectrum of **3aa** in $CDCl_3$



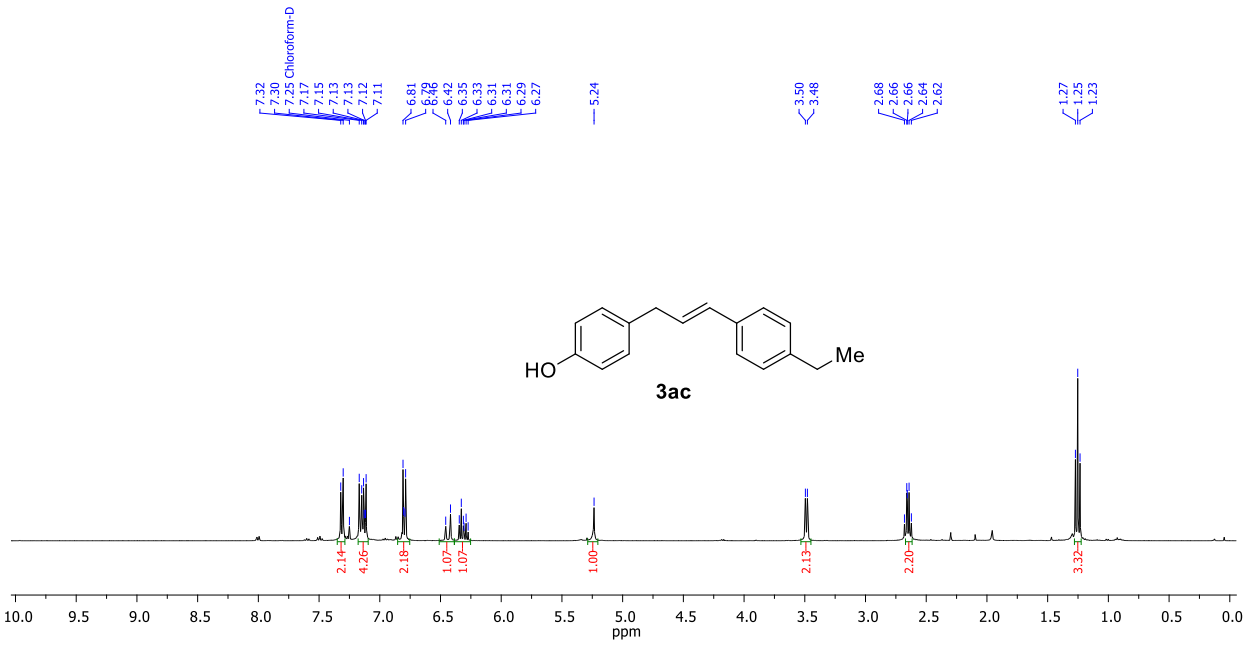
^{13}C NMR (100 MHz) spectrum of **3aa** in $CDCl_3$



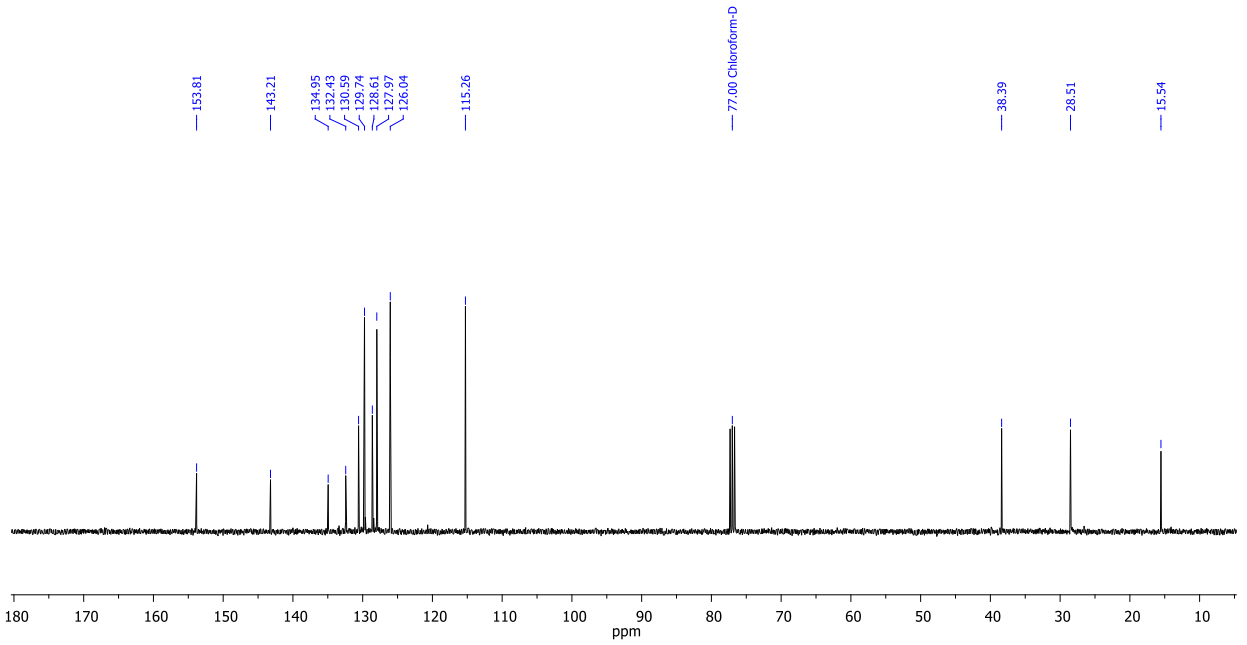
^{1}H NMR (400 MHz) spectrum of **3ab** in $CDCl_3$



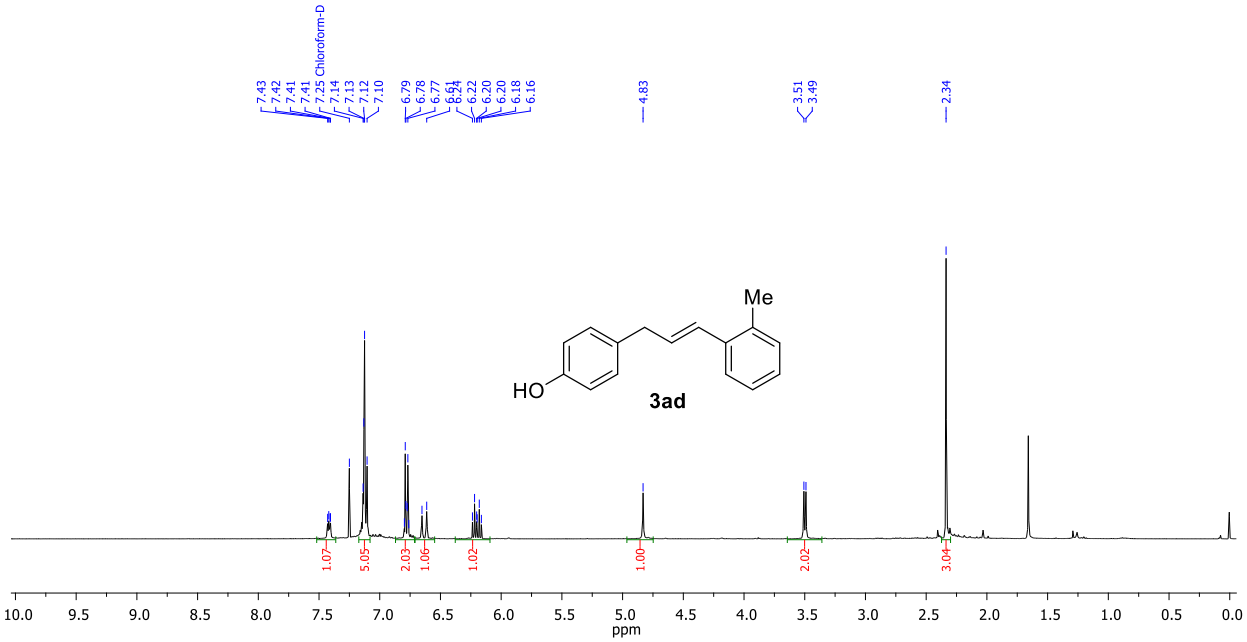
^{13}C NMR (100 MHz) spectrum of **3ab** in $CDCl_3$



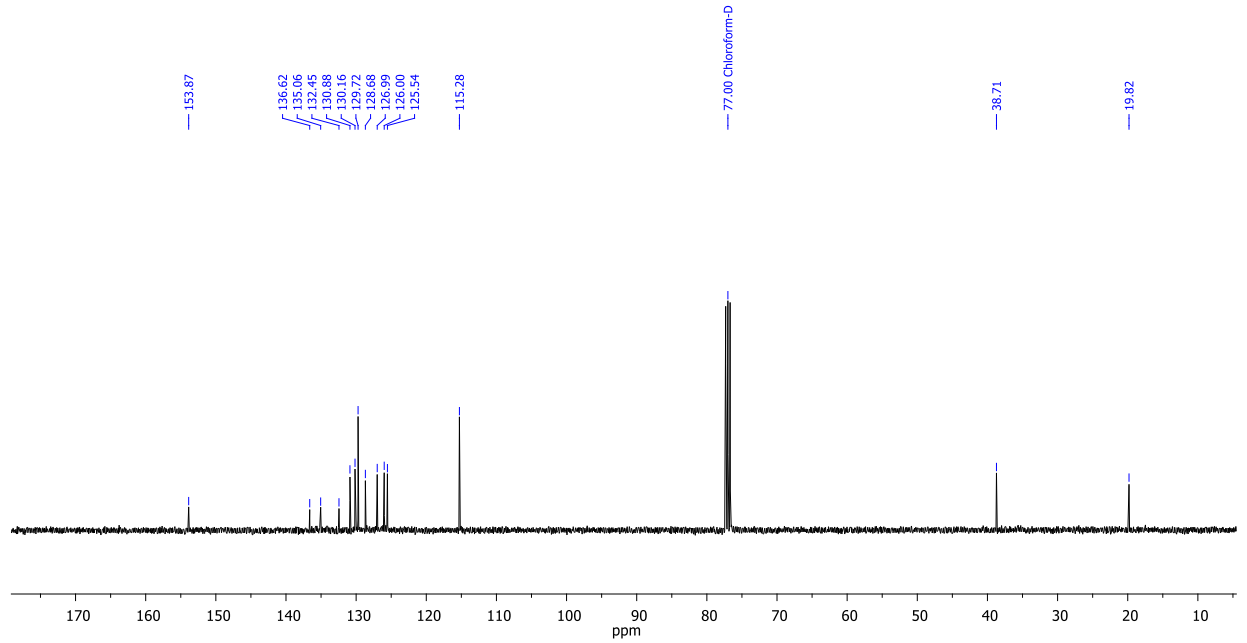
^{1}H NMR (400 MHz) spectrum of **3ac** in $CDCl_3$



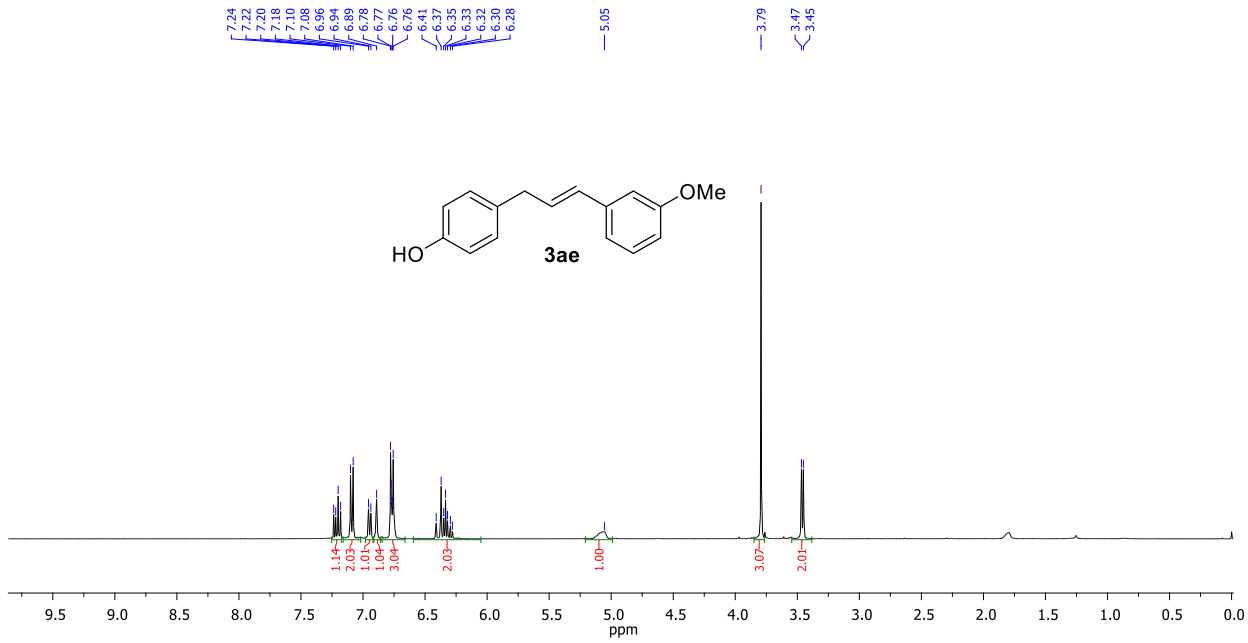
^{13}C NMR (100 MHz) spectrum of **3ac** in $CDCl_3$



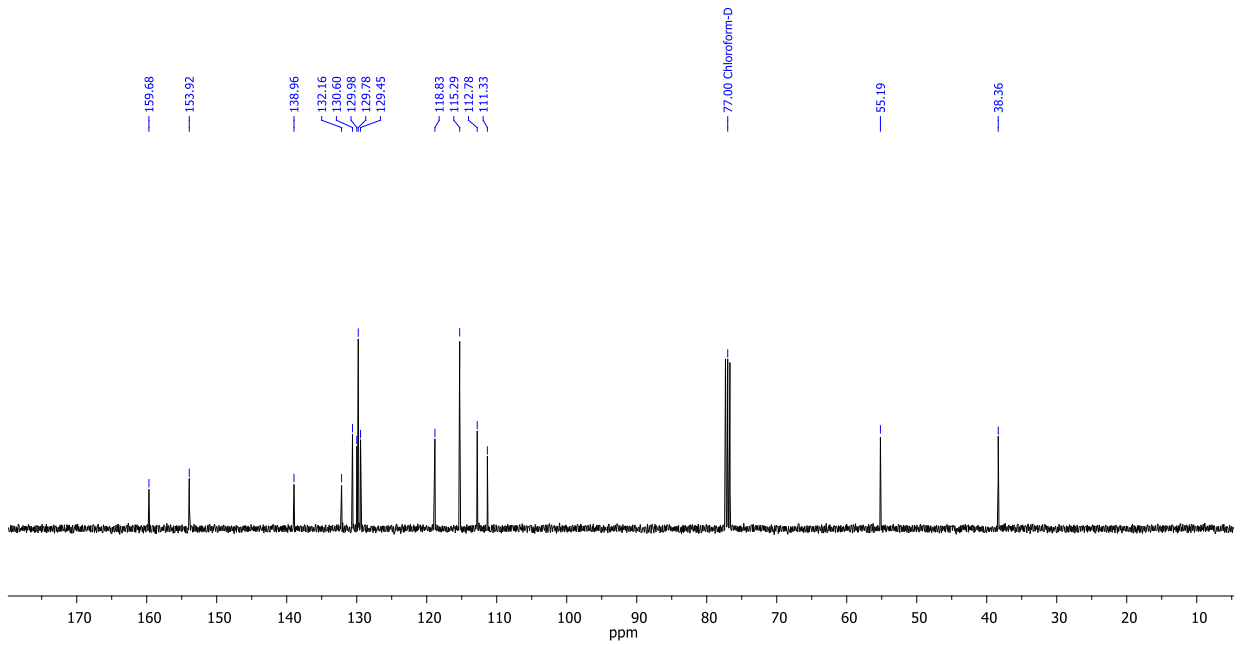
^{1}H NMR (400 MHz) spectrum of **3ad** in $CDCl_3$



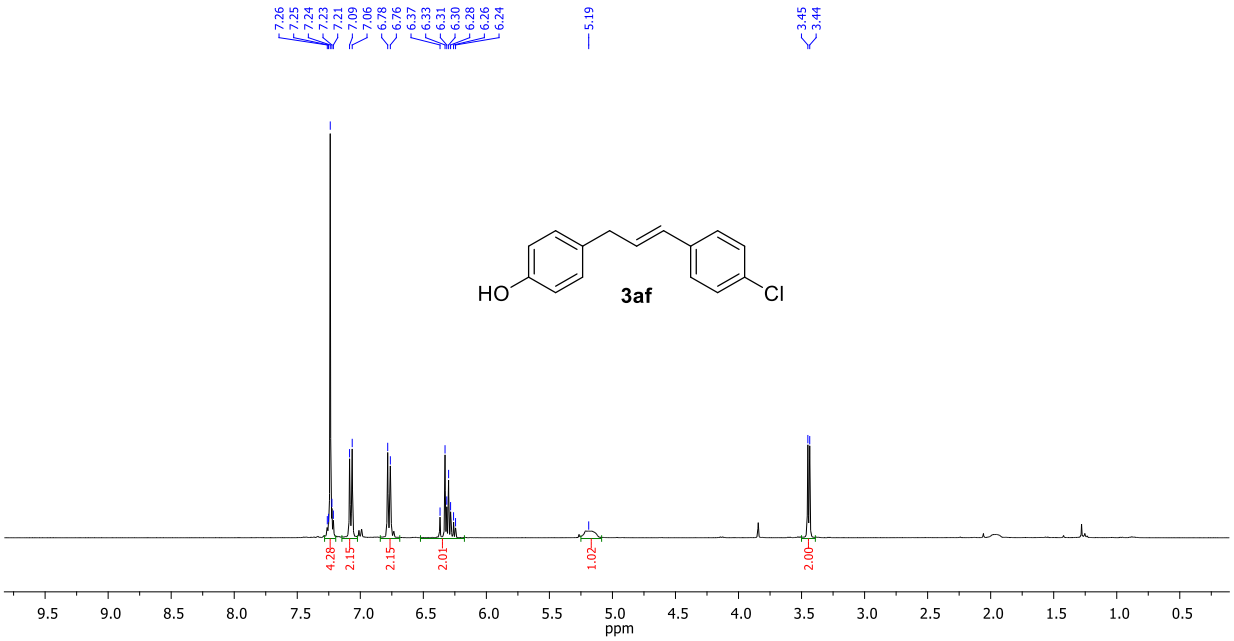
^{13}C NMR (100 MHz) spectrum of **3ad** in $CDCl_3$



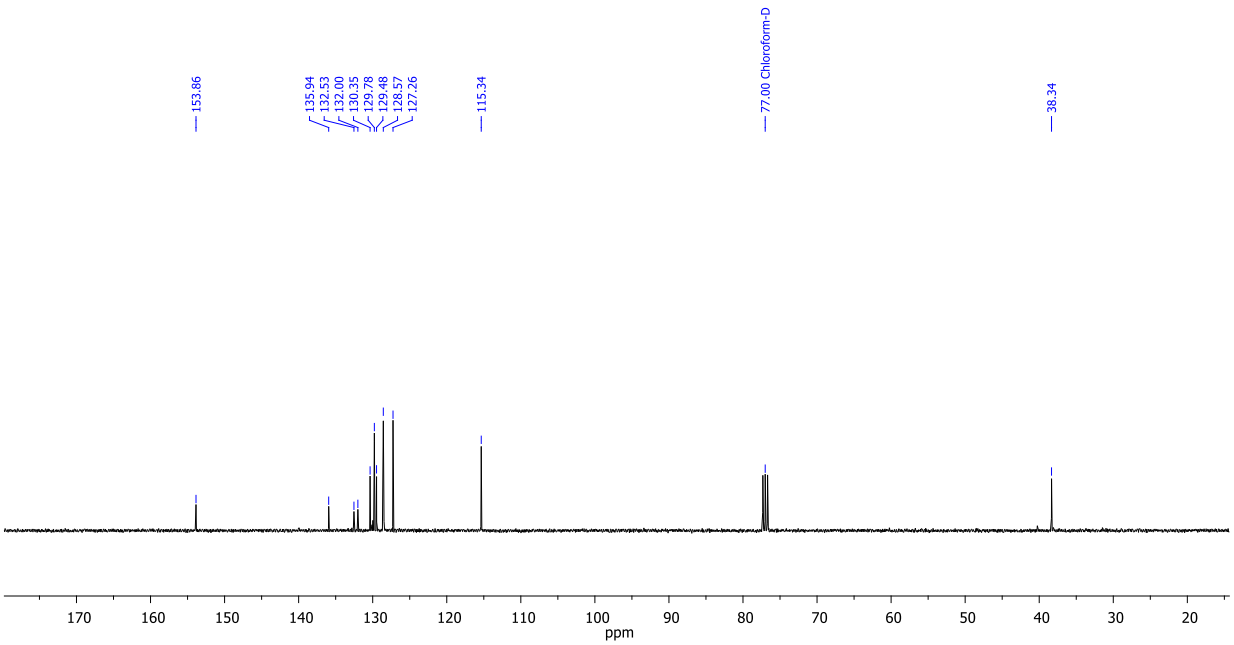
^{1}H NMR (400 MHz) spectrum of **3ae** in $CDCl_3$



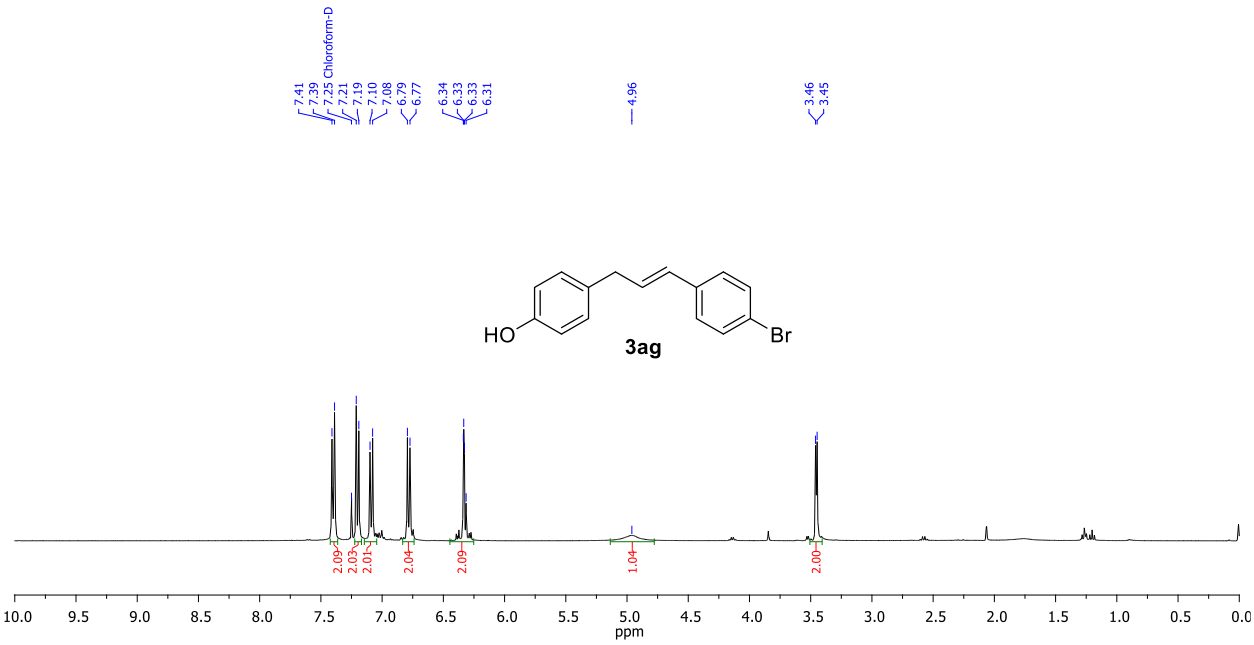
^{13}C NMR (100 MHz) spectrum of **3ae** in $CDCl_3$



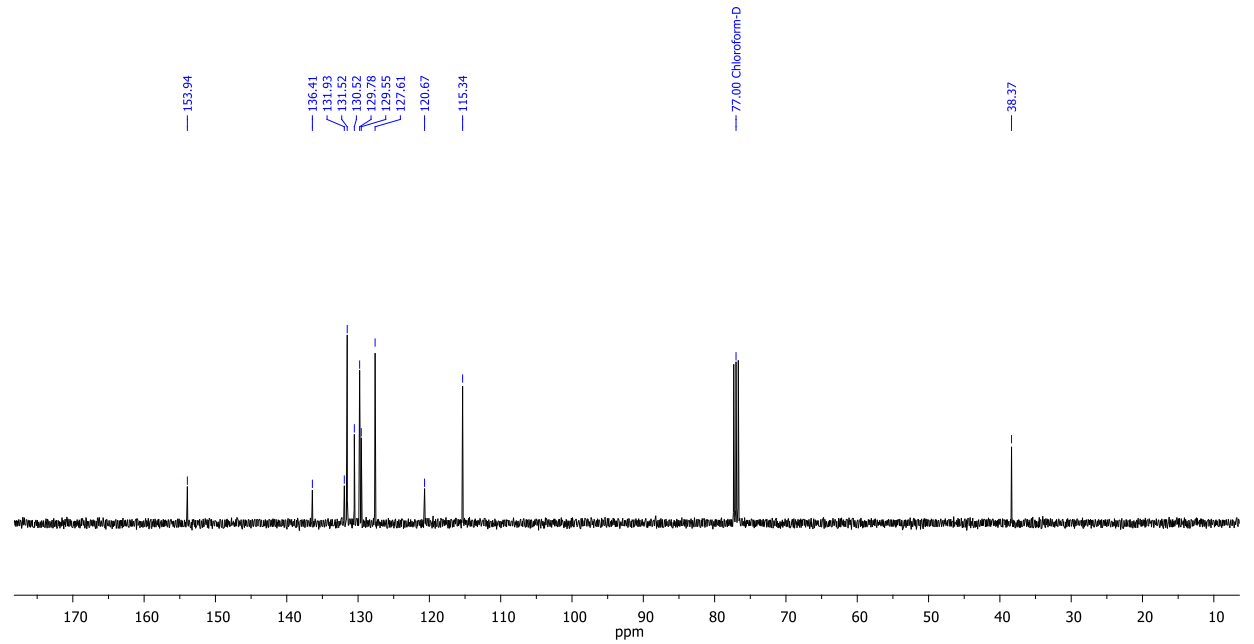
^{1}H NMR (400 MHz) spectrum of **3af** in $CDCl_3$



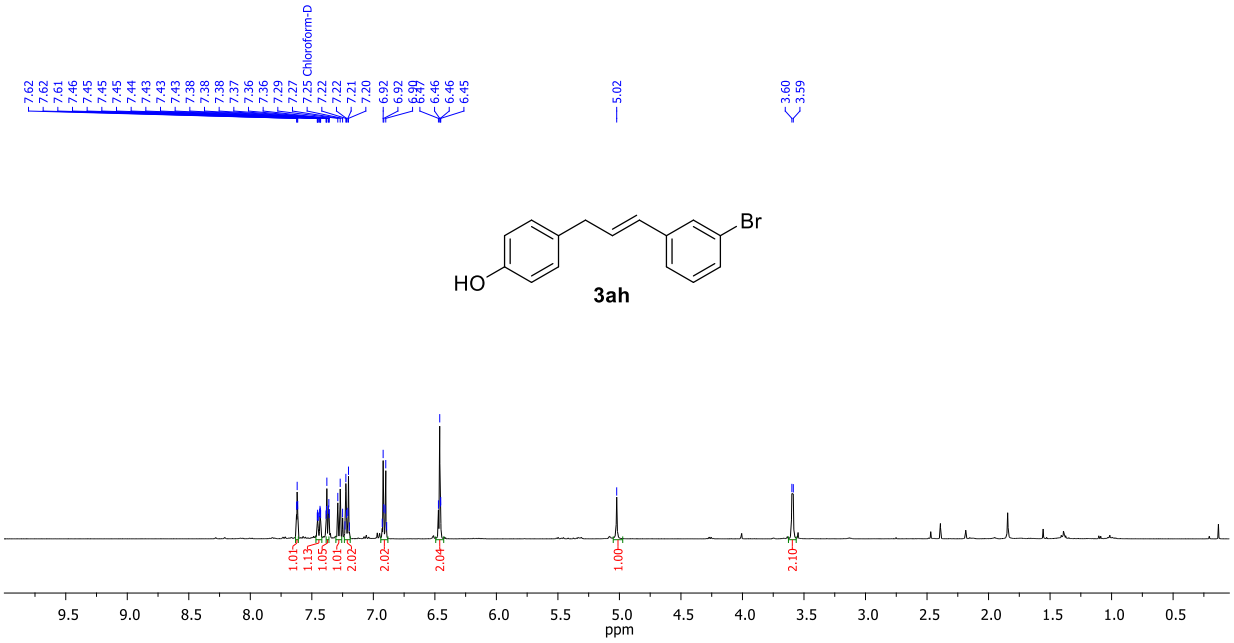
^{13}C NMR (100 MHz) spectrum of **3af** in $CDCl_3$



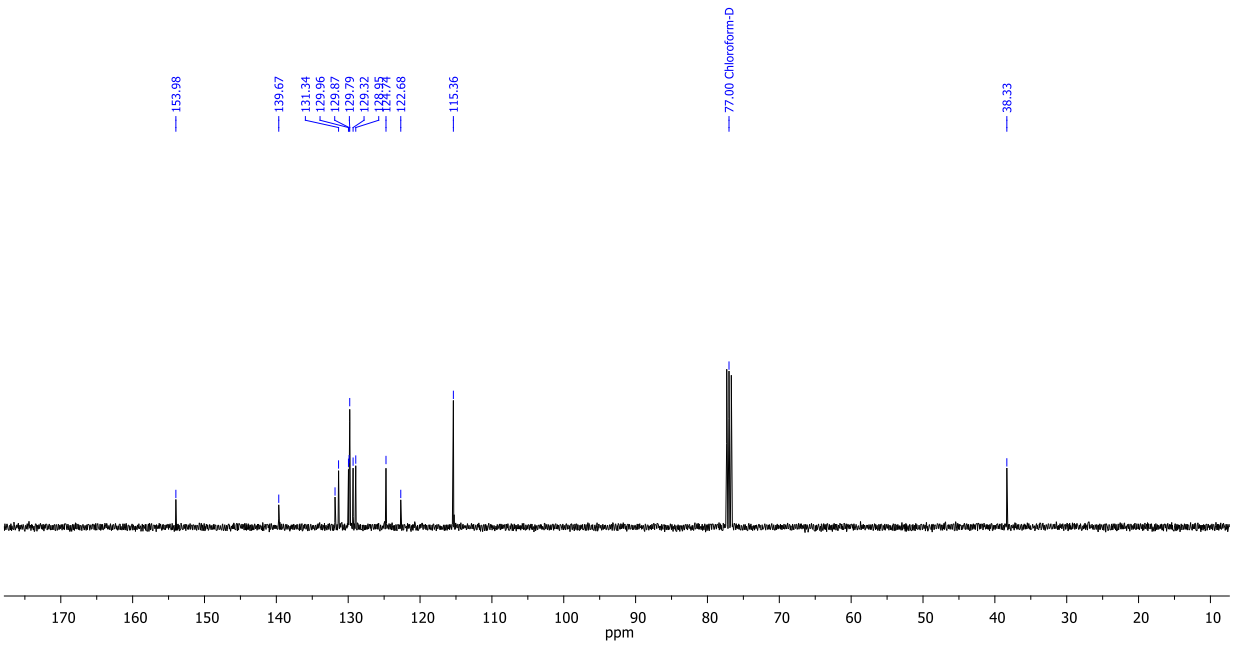
^{1}H NMR (400 MHz) spectrum of **3ag** in $CDCl_3$



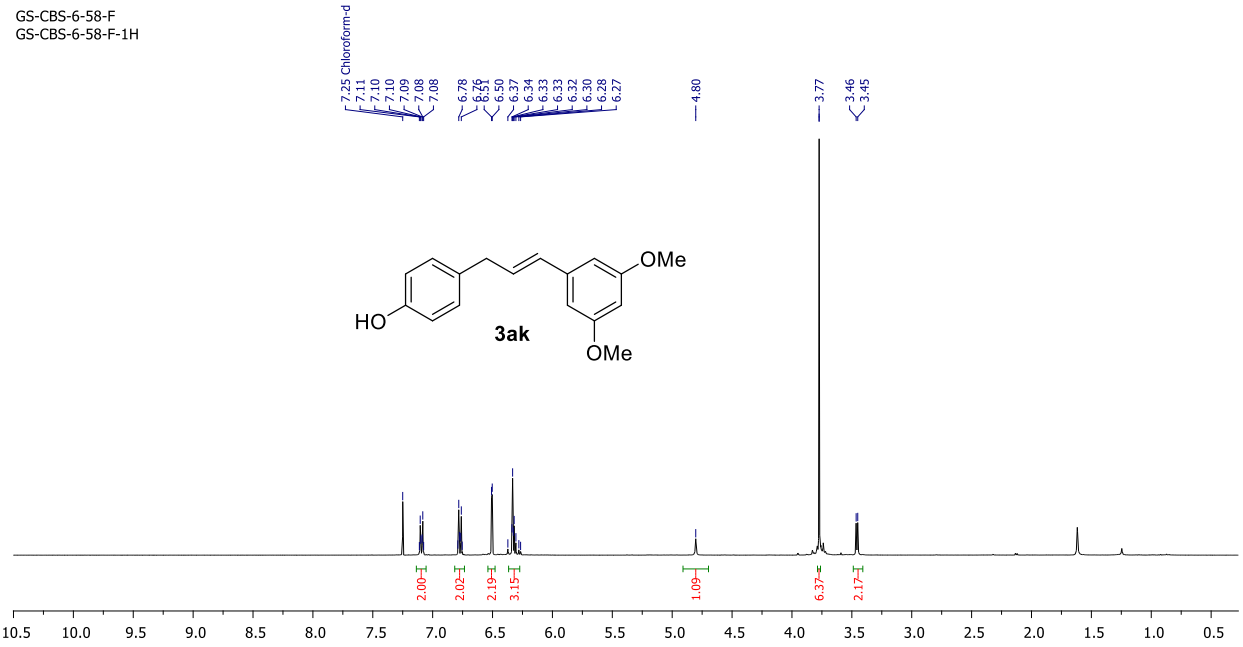
^{13}C NMR (100 MHz) spectrum of **3ag** in $CDCl_3$



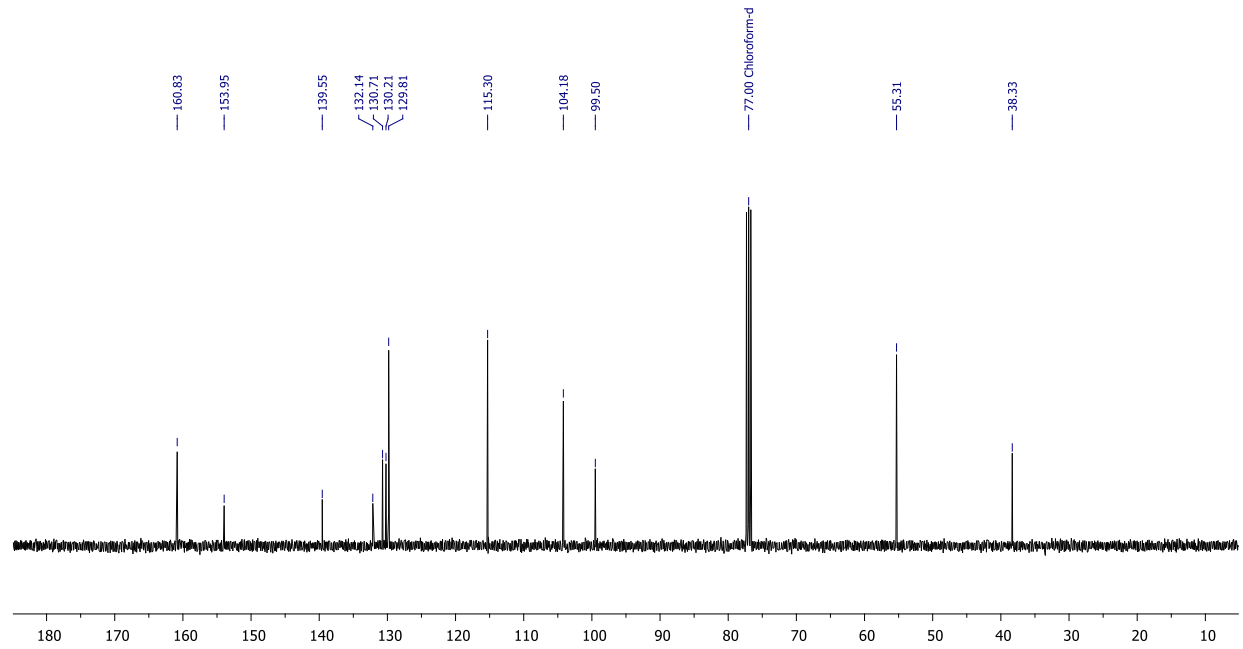
^{1}H NMR (400 MHz) spectrum of **3ah** in $CDCl_3$



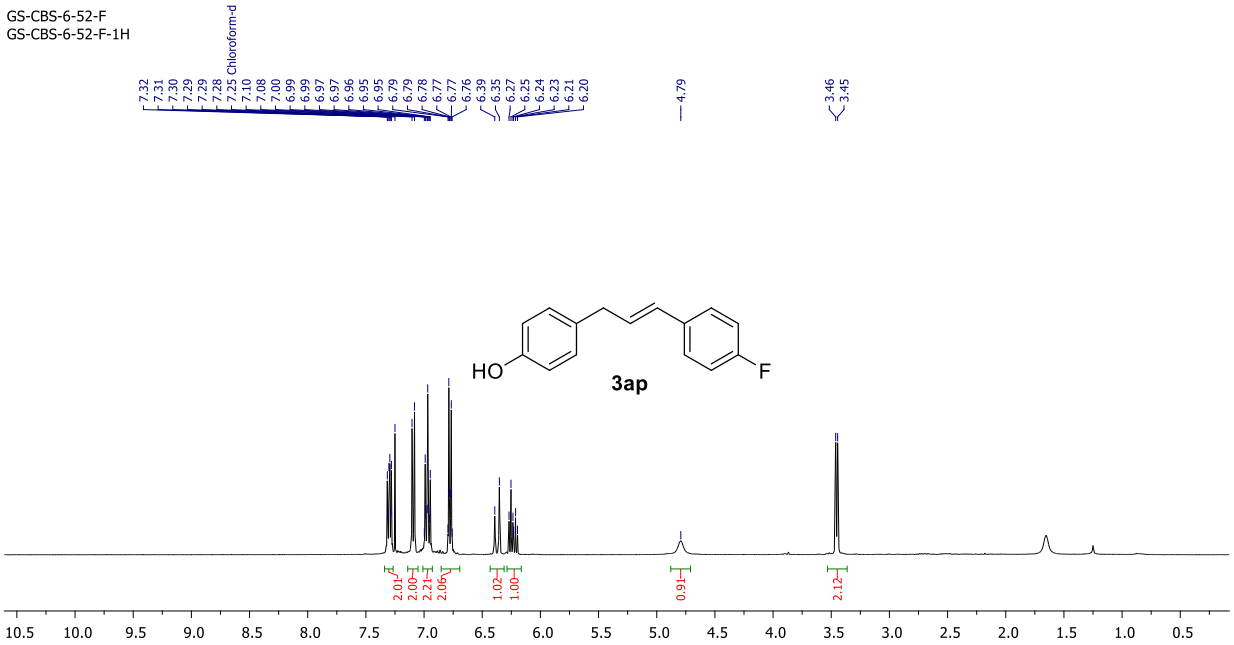
^{13}C NMR (100 MHz) spectrum of **3ah** in $CDCl_3$



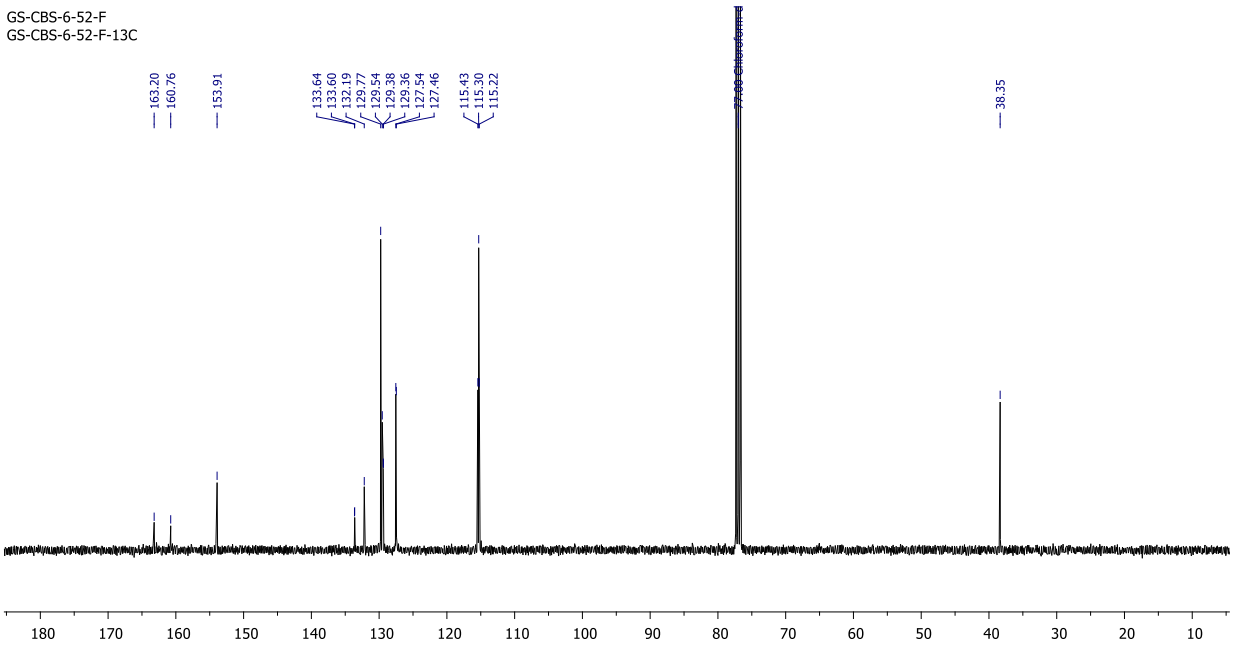
^{1}H NMR (400 MHz) spectrum of **3ak** in $CDCl_3$



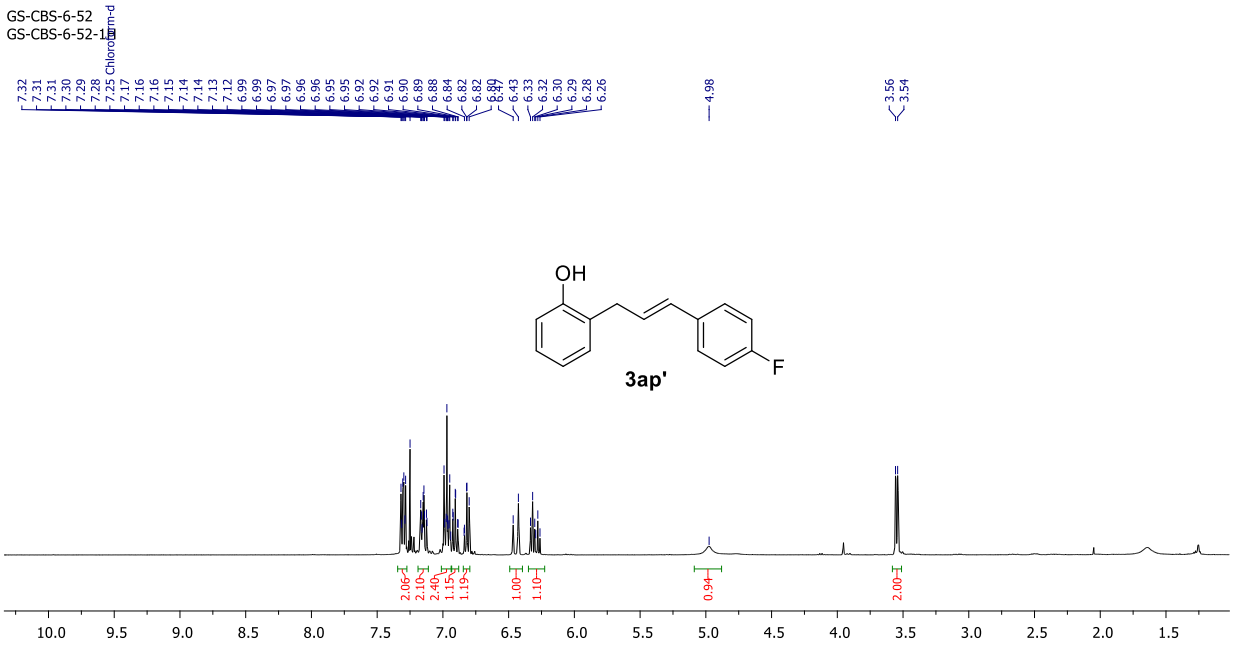
^{13}C NMR (100 MHz) spectrum of **3ak** in $CDCl_3$



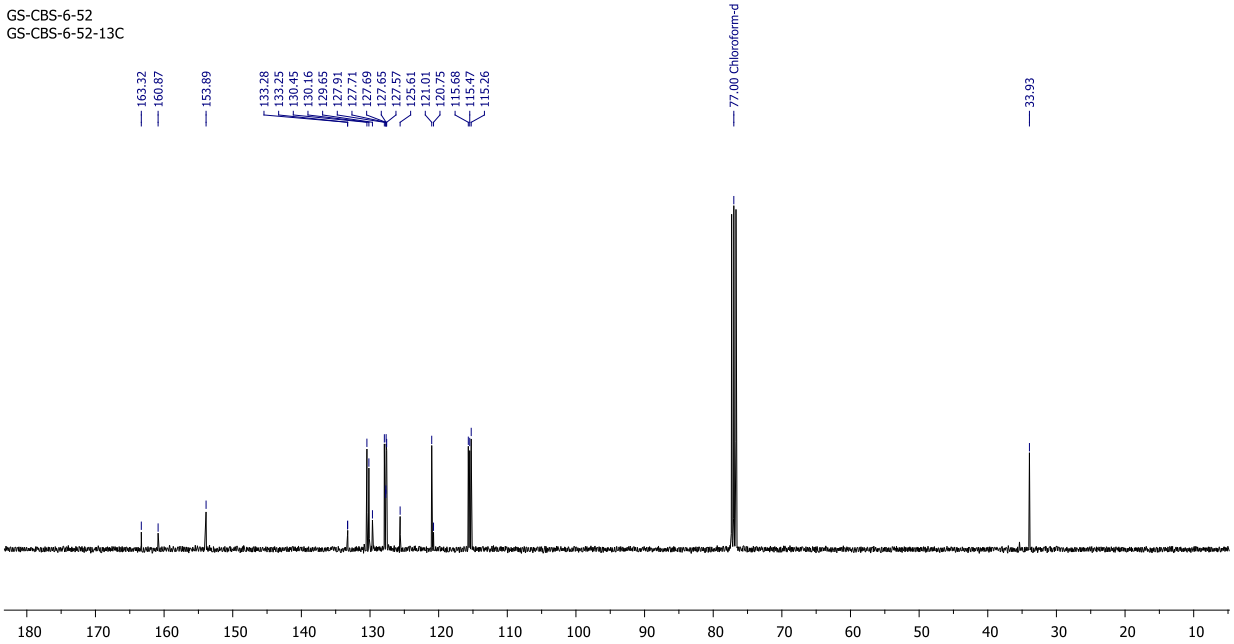
^{1}H NMR (400 MHz) spectrum of **3ap** in $CDCl_3$



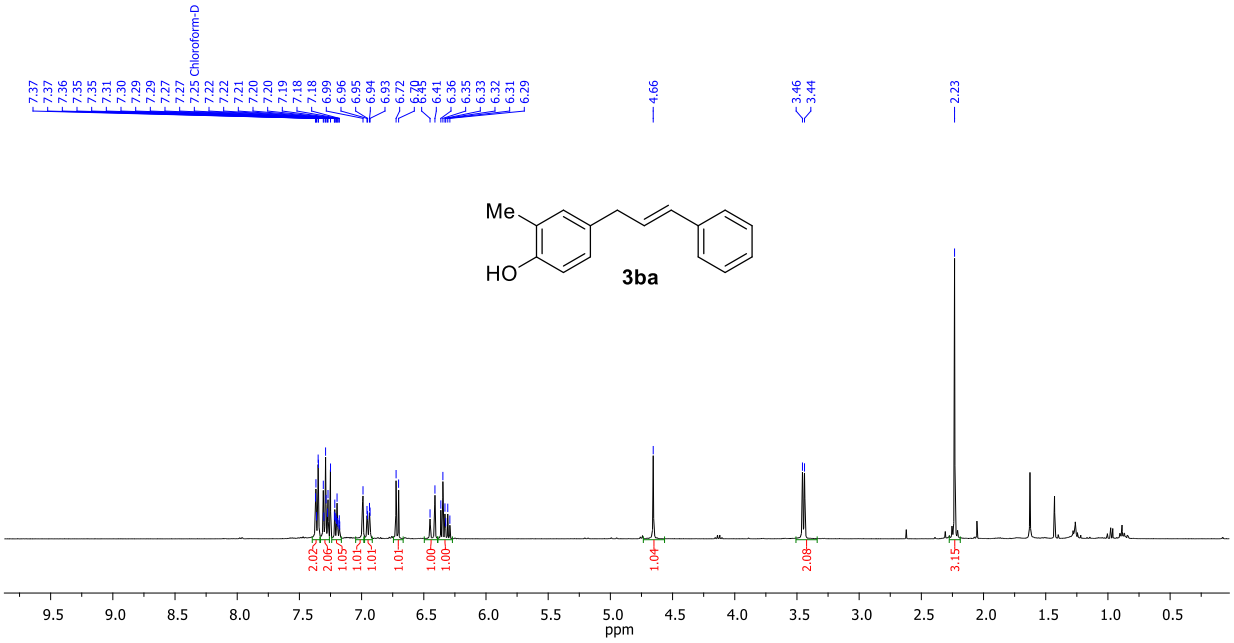
^{13}C NMR (100 MHz) spectrum of **3ap** in $CDCl_3$



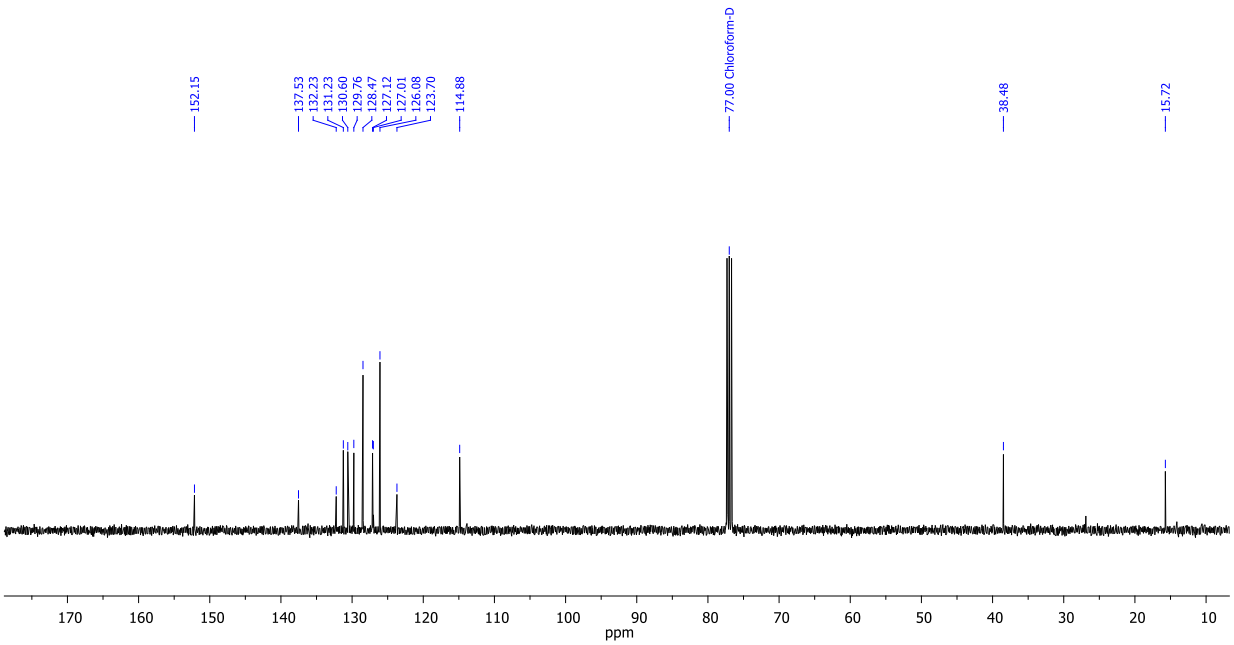
^{1}H NMR (400 MHz) spectrum of **3ap'** in $CDCl_3$



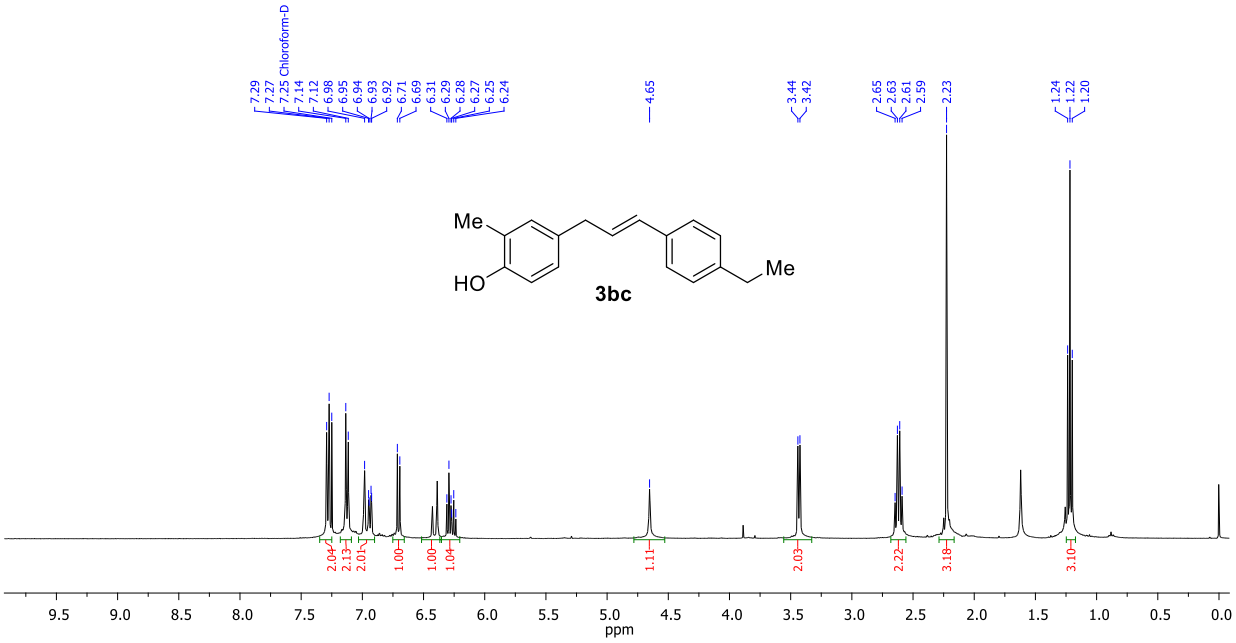
^{13}C NMR (100 MHz) spectrum of **3ap'** in $CDCl_3$



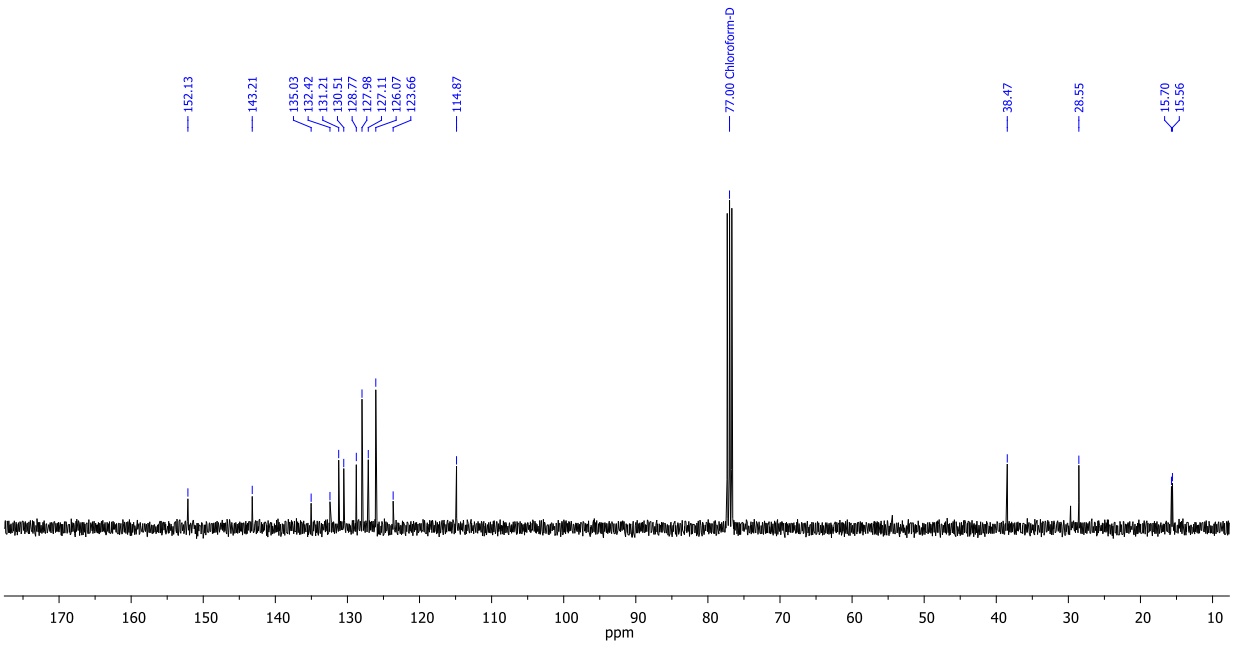
^{1}H NMR (400 MHz) spectrum of **3ba** in $CDCl_3$



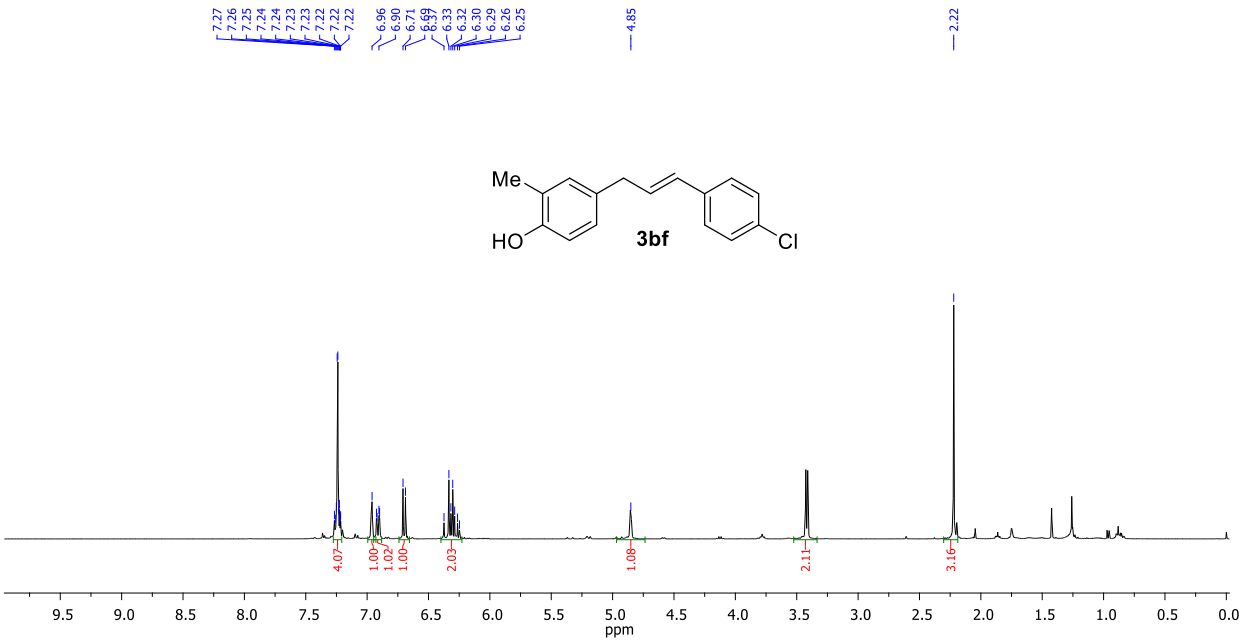
^{13}C NMR (100 MHz) spectrum of **3ba** in $CDCl_3$



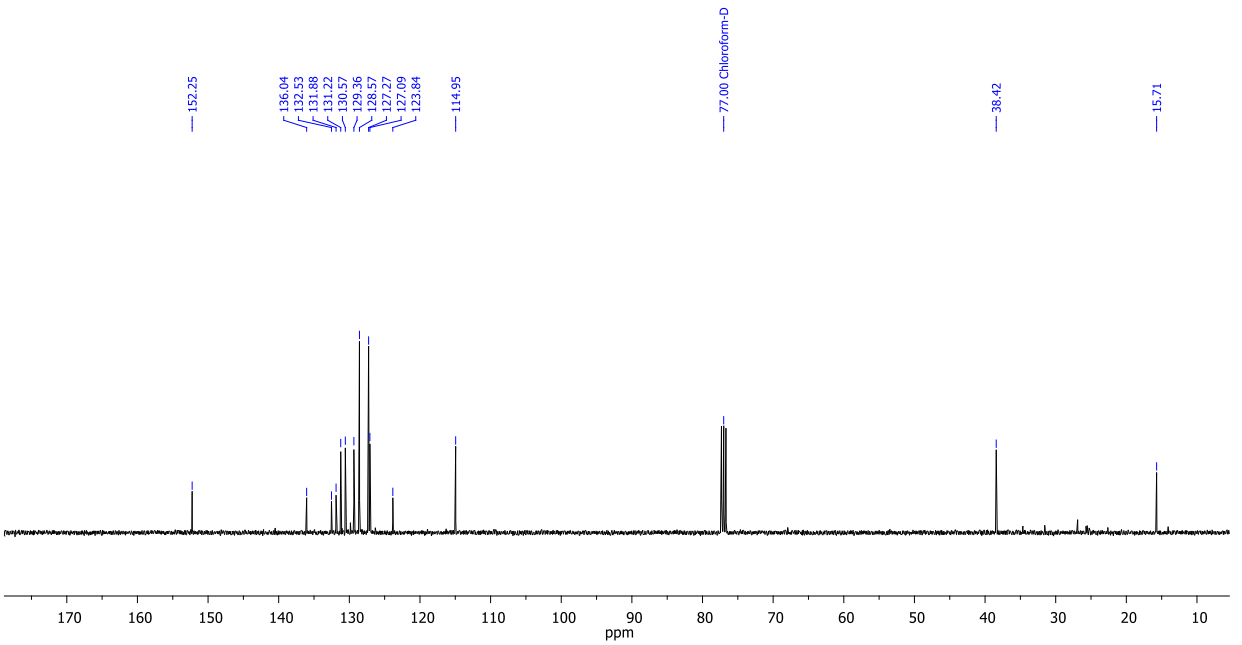
^{1}H NMR (400 MHz) spectrum of **3bc** in $CDCl_3$



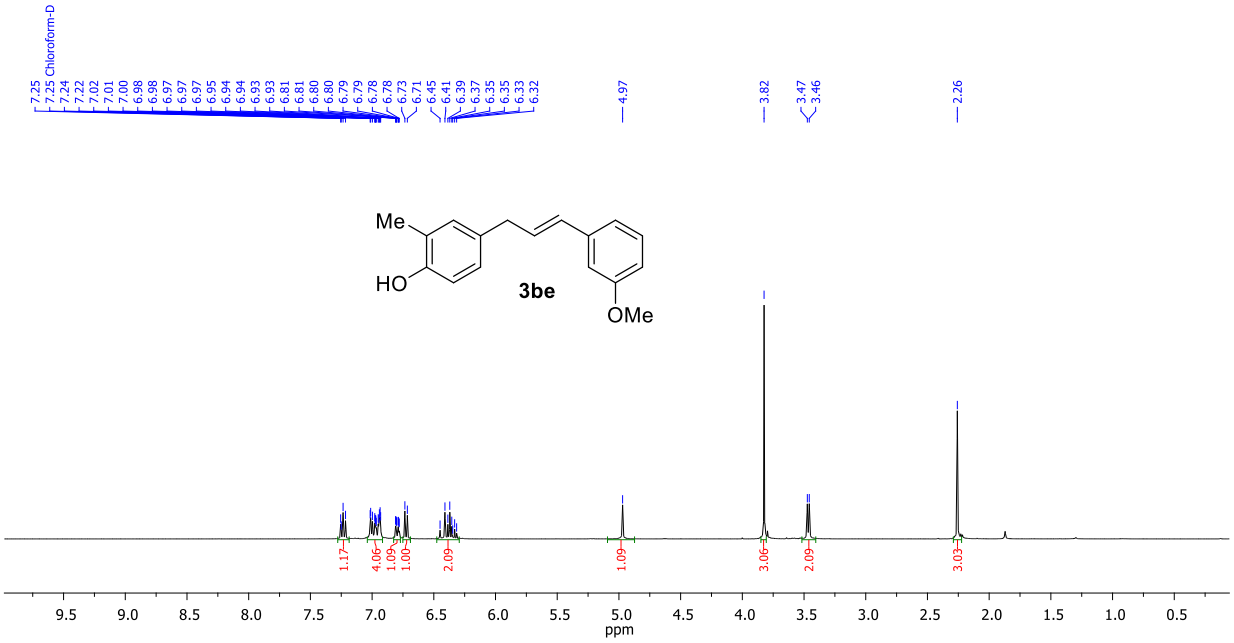
^{13}C NMR (100 MHz) spectrum of **3bc** in $CDCl_3$



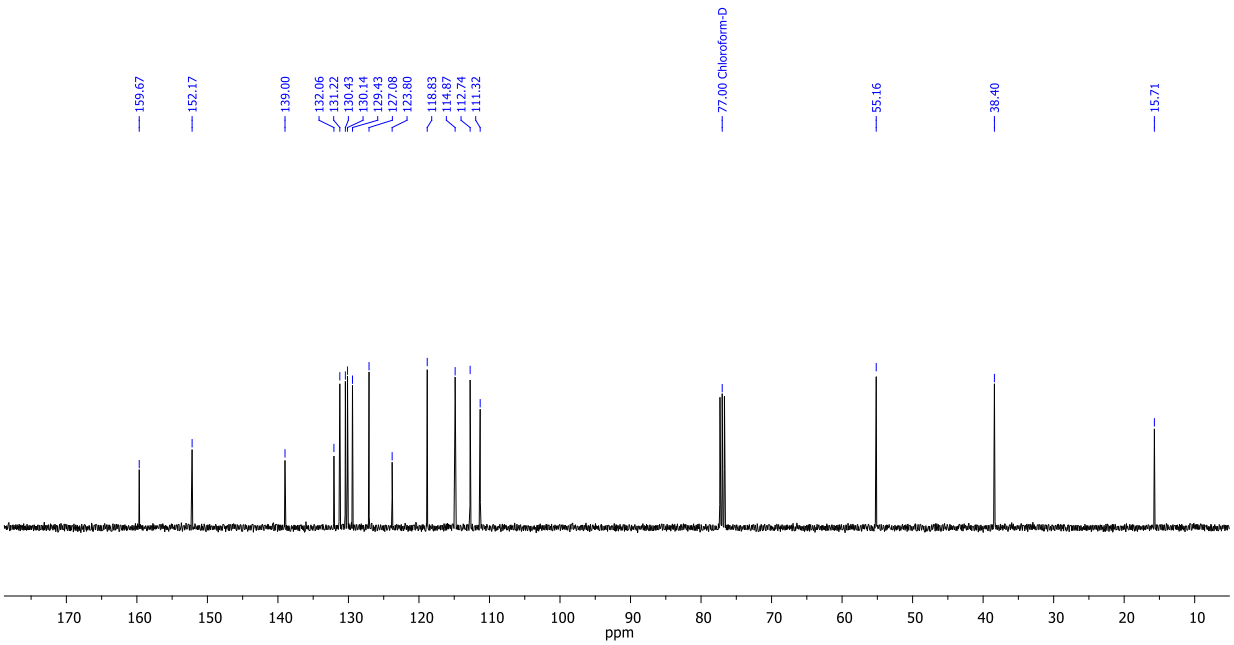
^{1}H NMR (400 MHz) spectrum of **3bf** in $CDCl_3$



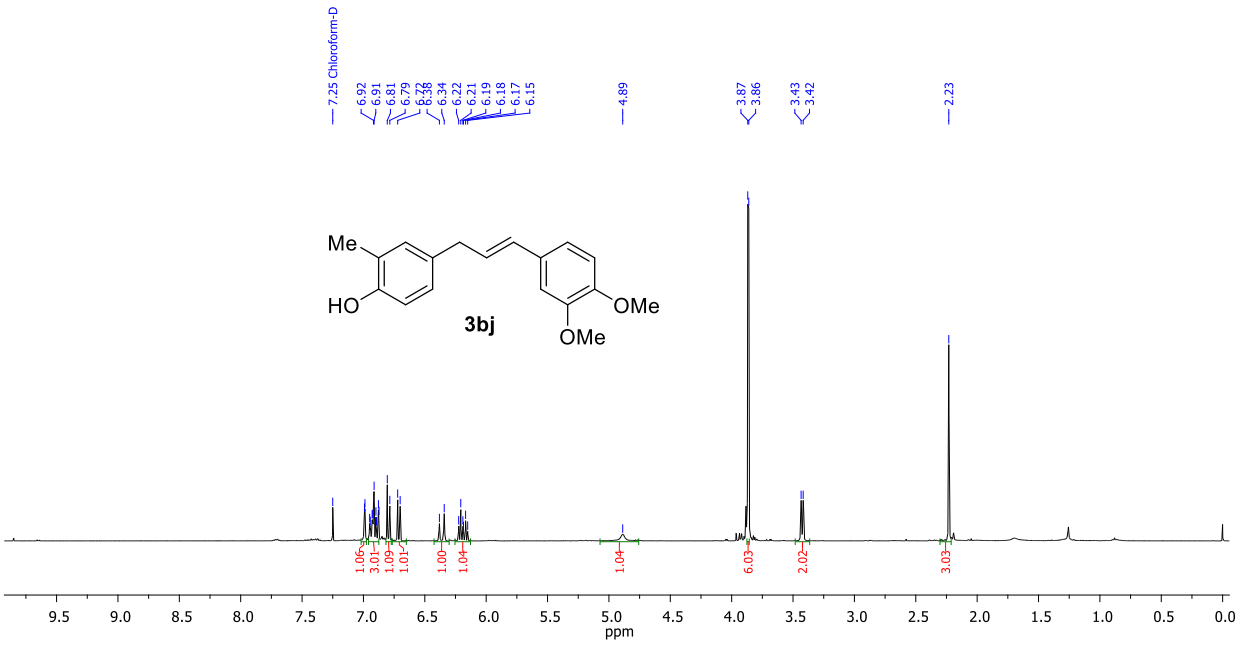
^{13}C NMR (100 MHz) spectrum of **3bf** in $CDCl_3$



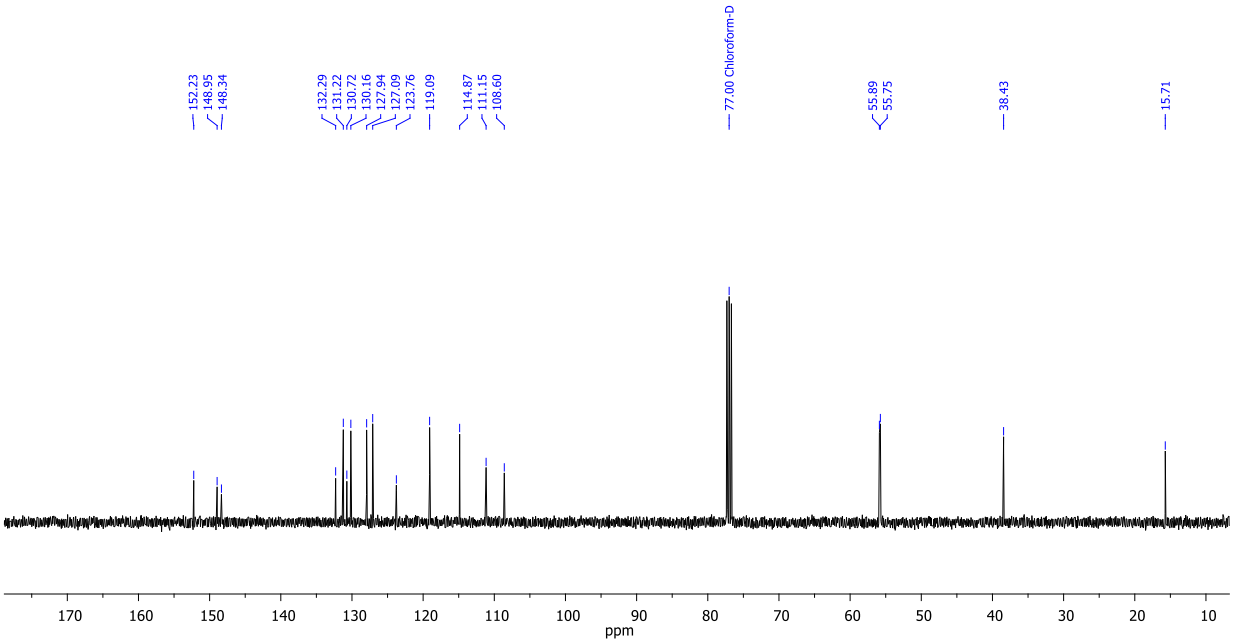
^{1}H NMR (400 MHz) spectrum of **3be** in $CDCl_3$



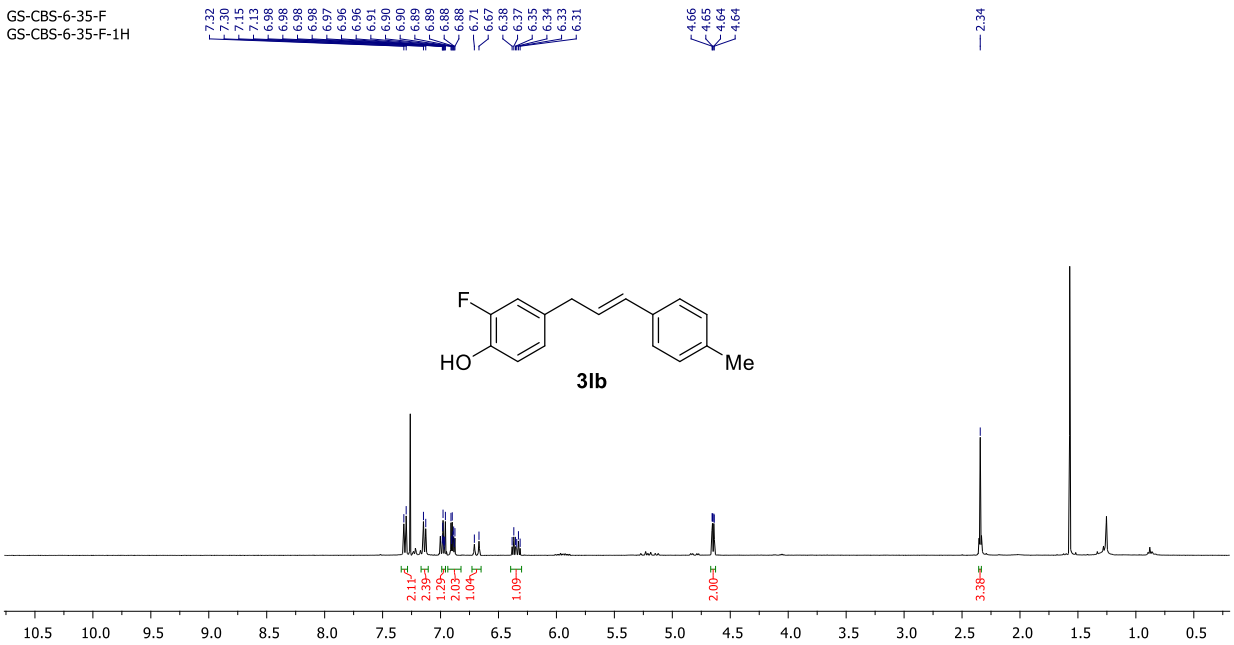
^{13}C NMR (100 MHz) spectrum of **3be** in $CDCl_3$



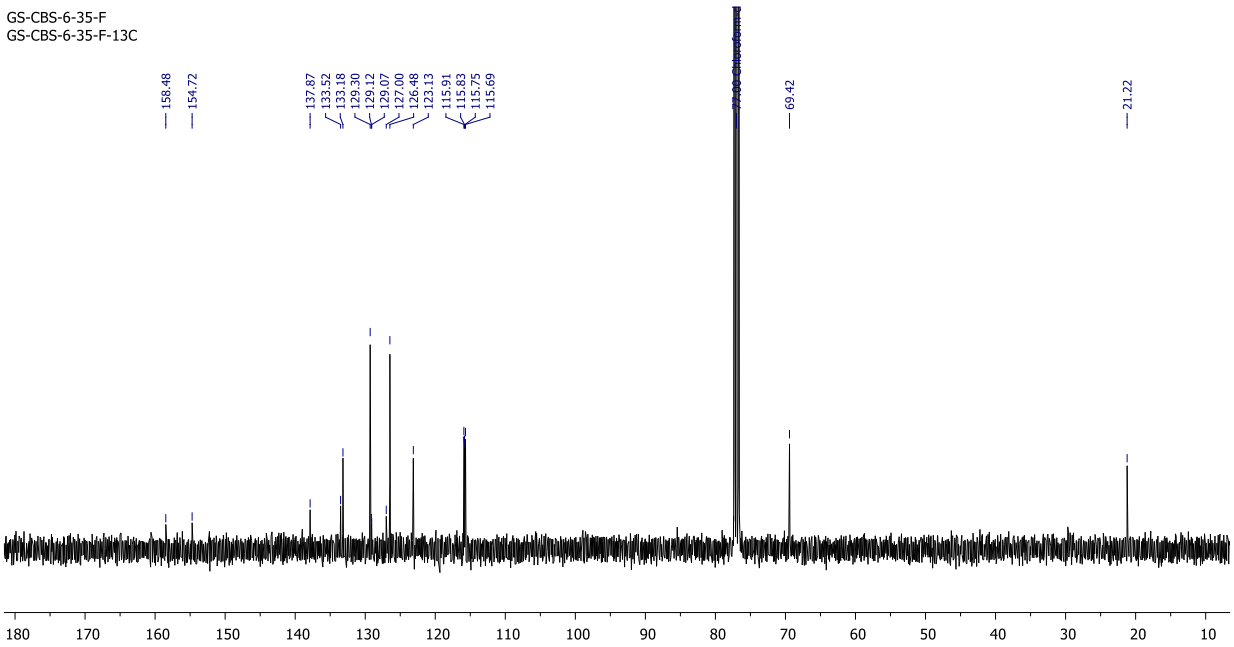
^{1}H NMR (400 MHz) spectrum of **3bj** in $CDCl_3$



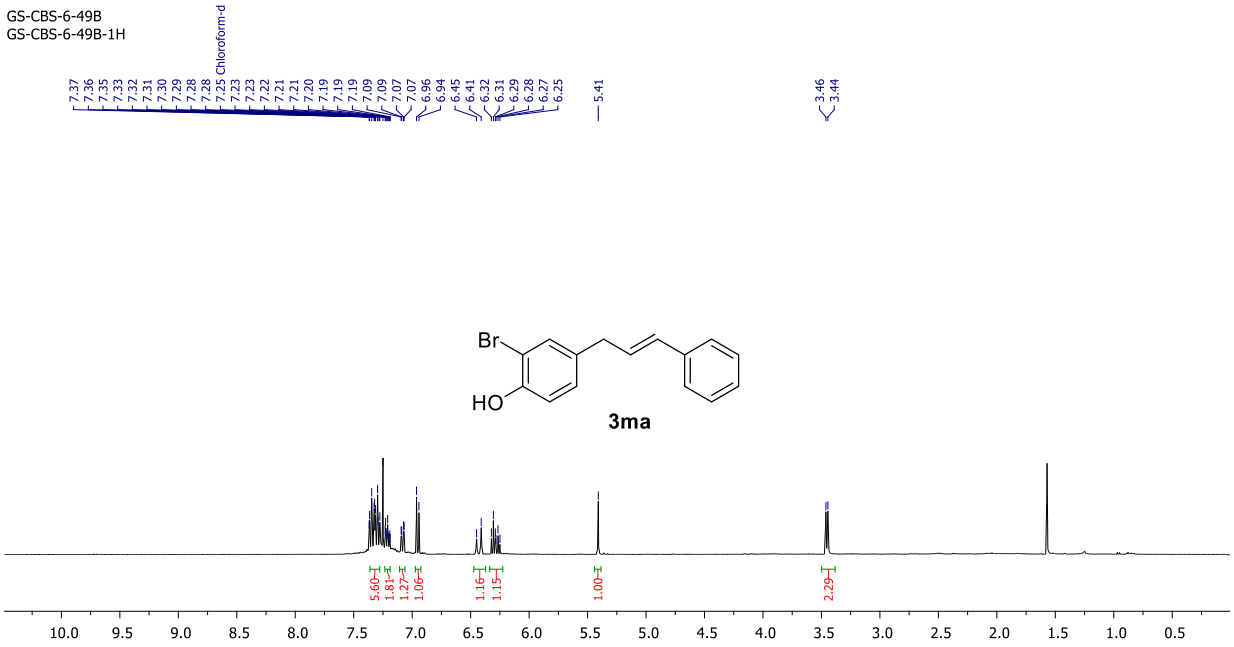
^{13}C NMR (100 MHz) spectrum of **3bj** in $CDCl_3$



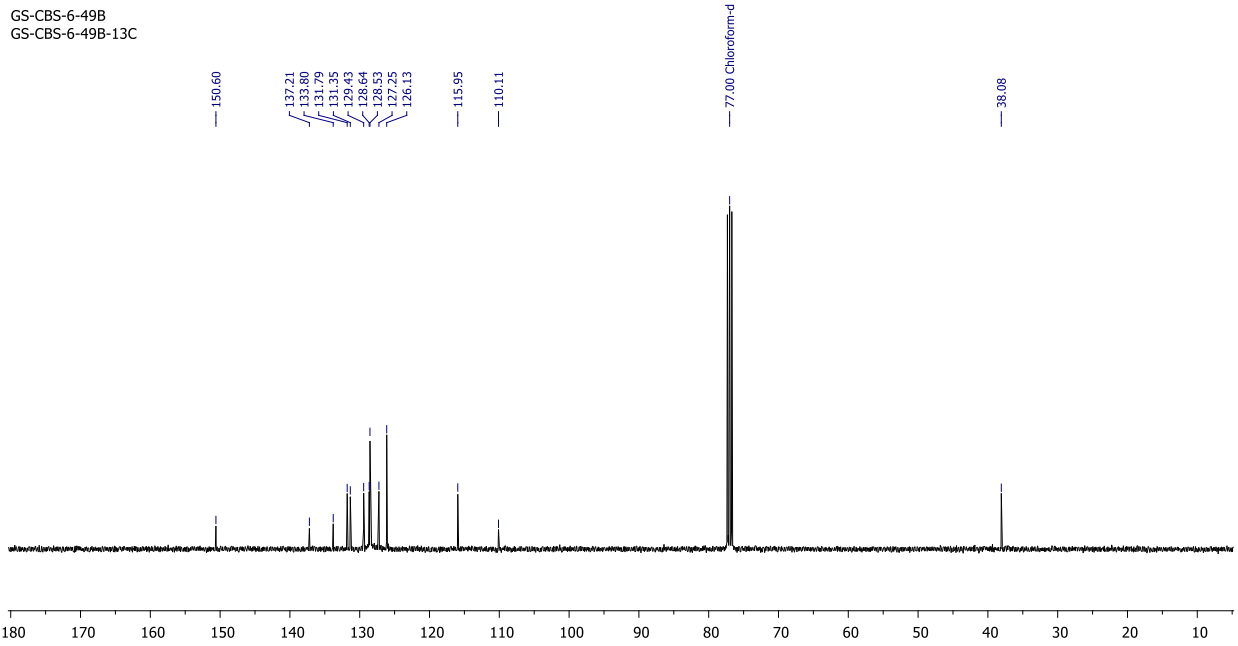
^{1}H NMR (400 MHz) spectrum of **3lb** in $CDCl_3$



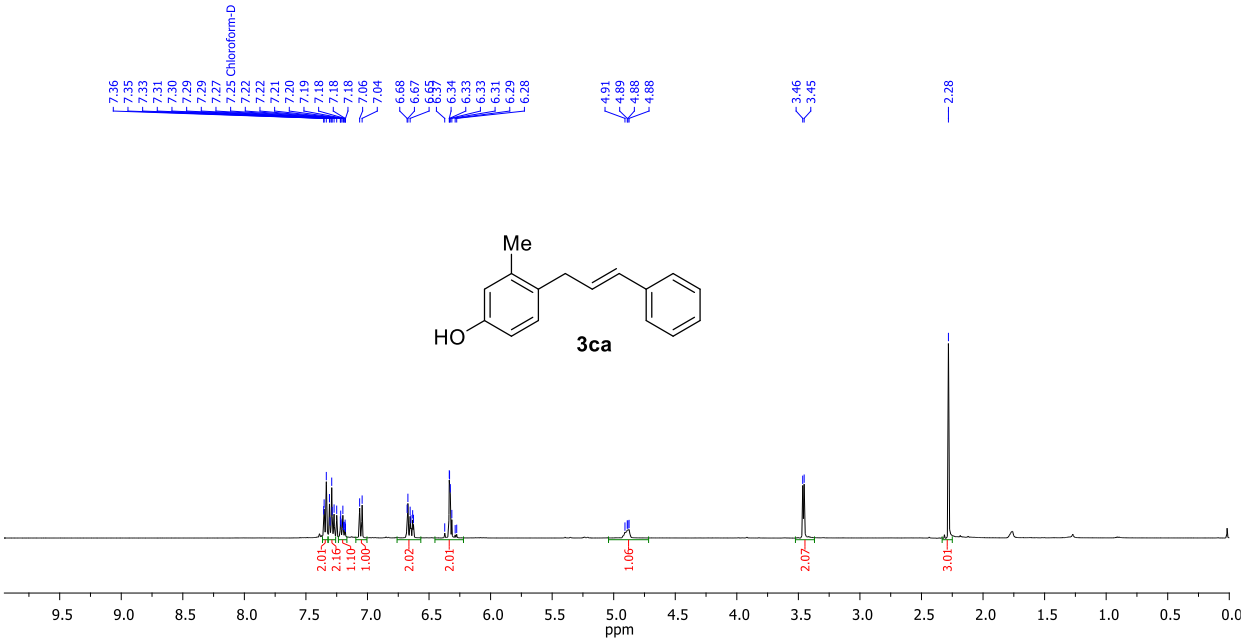
^{13}C NMR (100 MHz) spectrum of **3lb** in $CDCl_3$



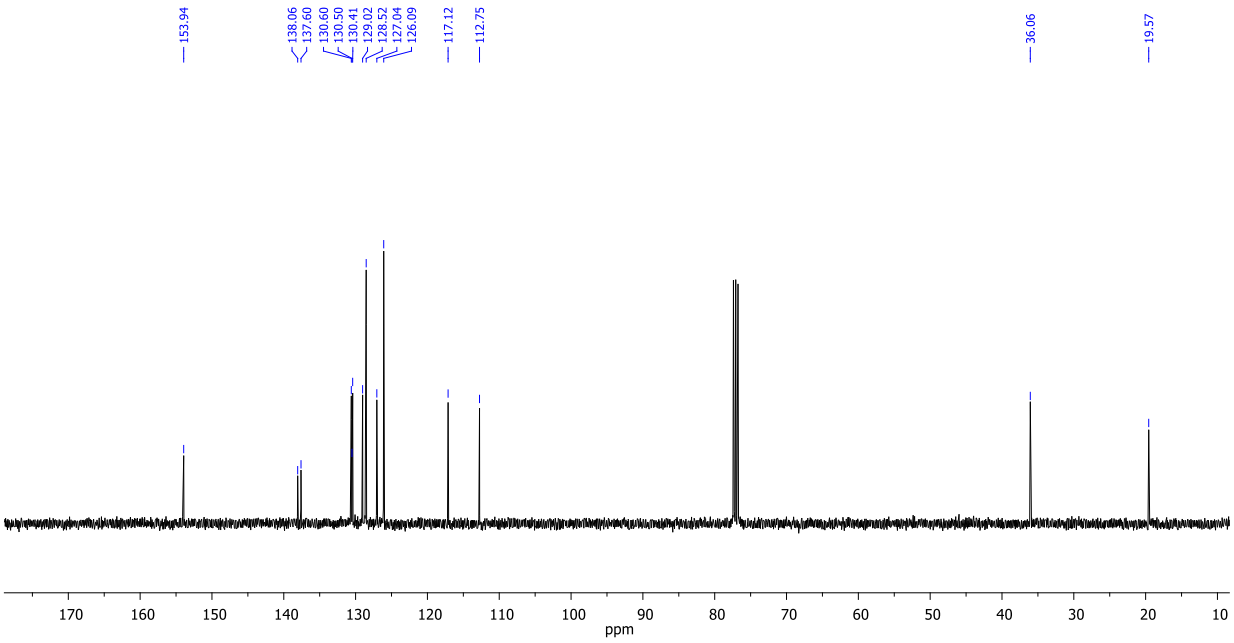
^{1}H NMR (400 MHz) spectrum of **3ma** in $CDCl_3$



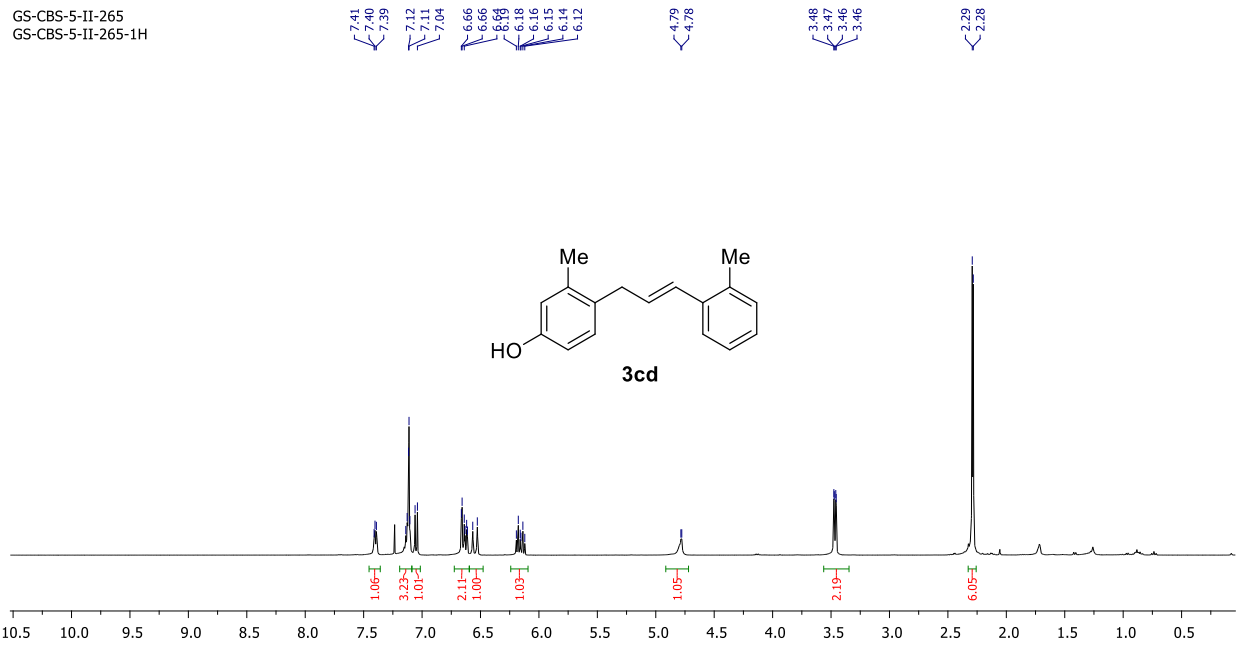
^{13}C NMR (100 MHz) spectrum of **3ma** in $CDCl_3$



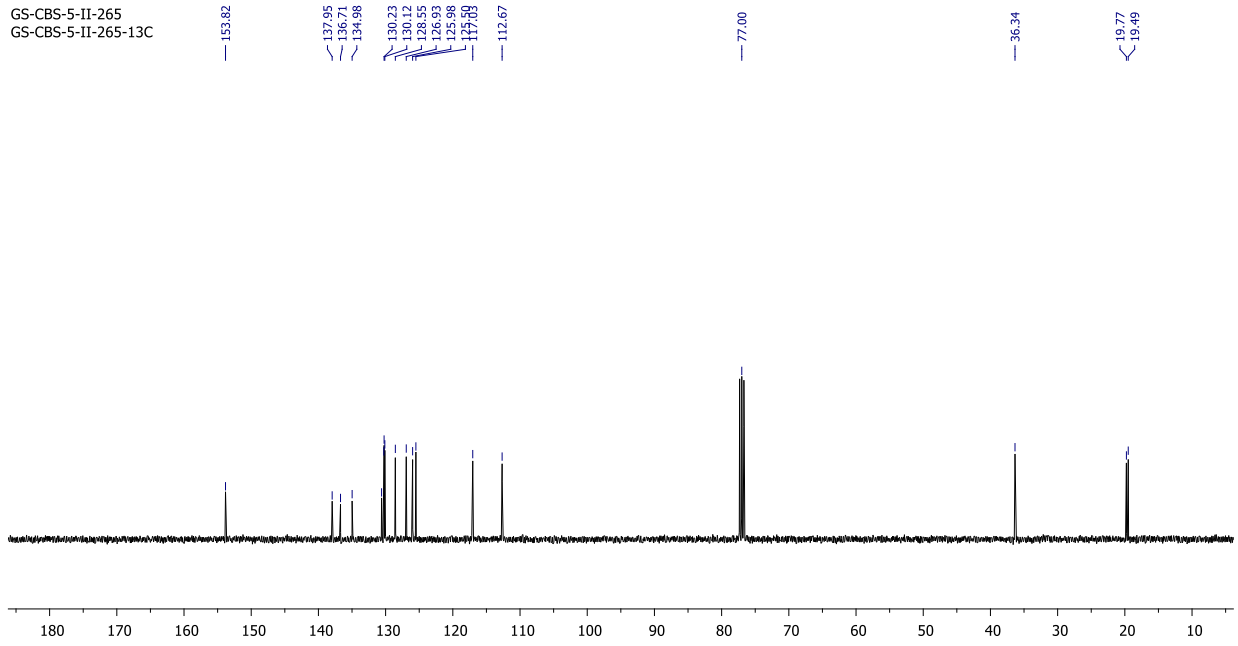
^{1}H NMR (400 MHz) spectrum of **3ca** in $CDCl_3$



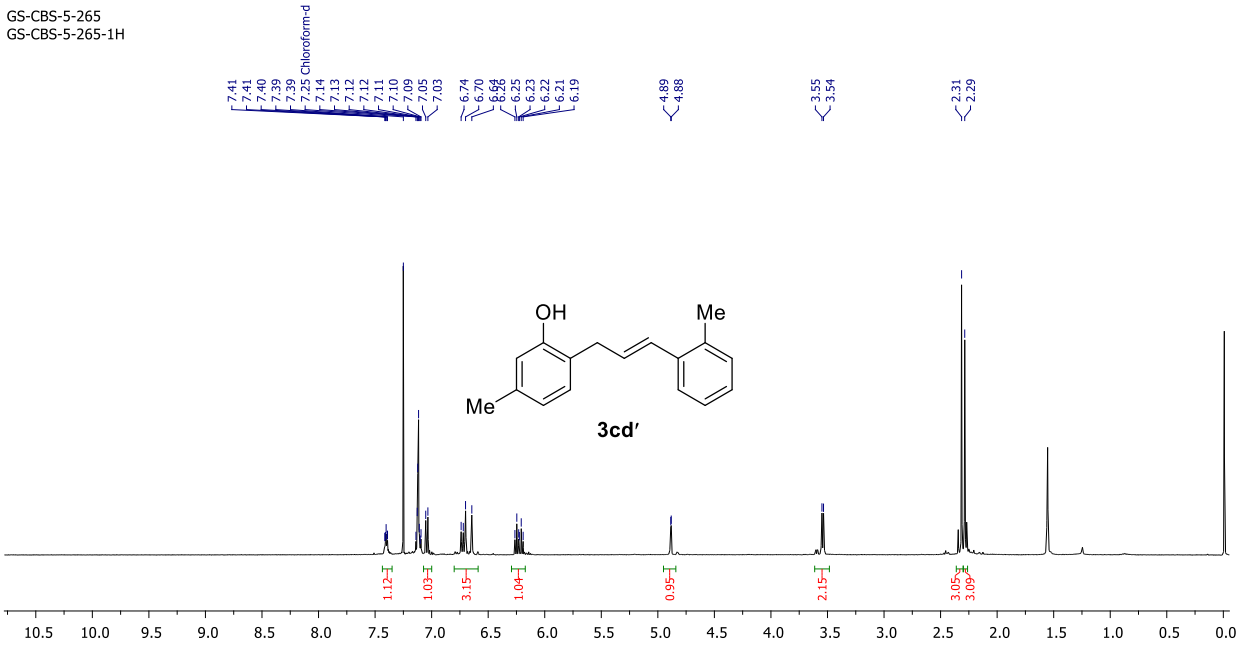
^{13}C NMR (100 MHz) spectrum of **3ca** in $CDCl_3$



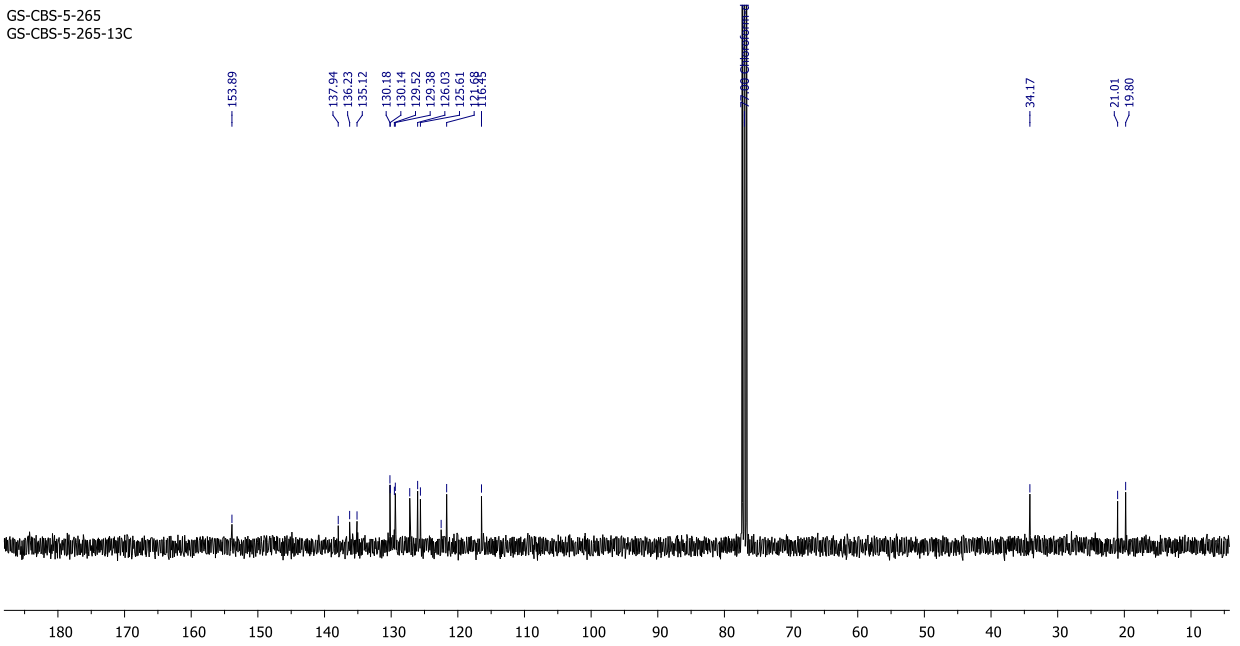
^{1}H NMR (400 MHz) spectrum of **3cd** in $CDCl_3$



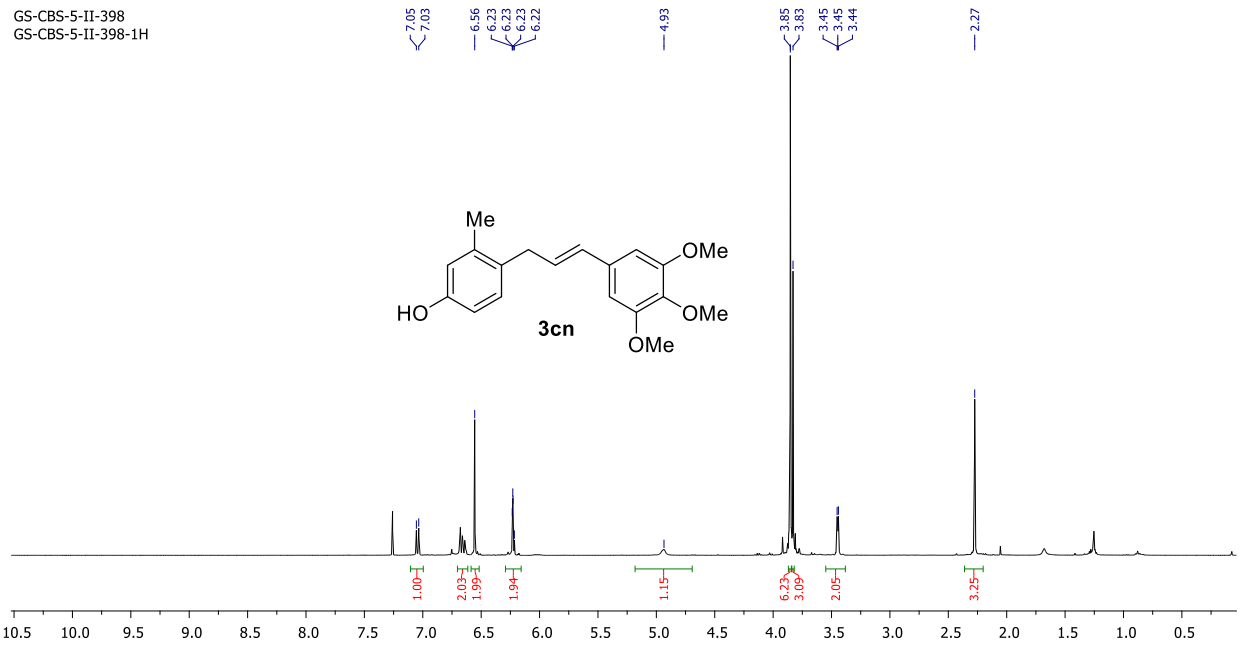
^{13}C NMR (100 MHz) spectrum of **3cd** in $CDCl_3$



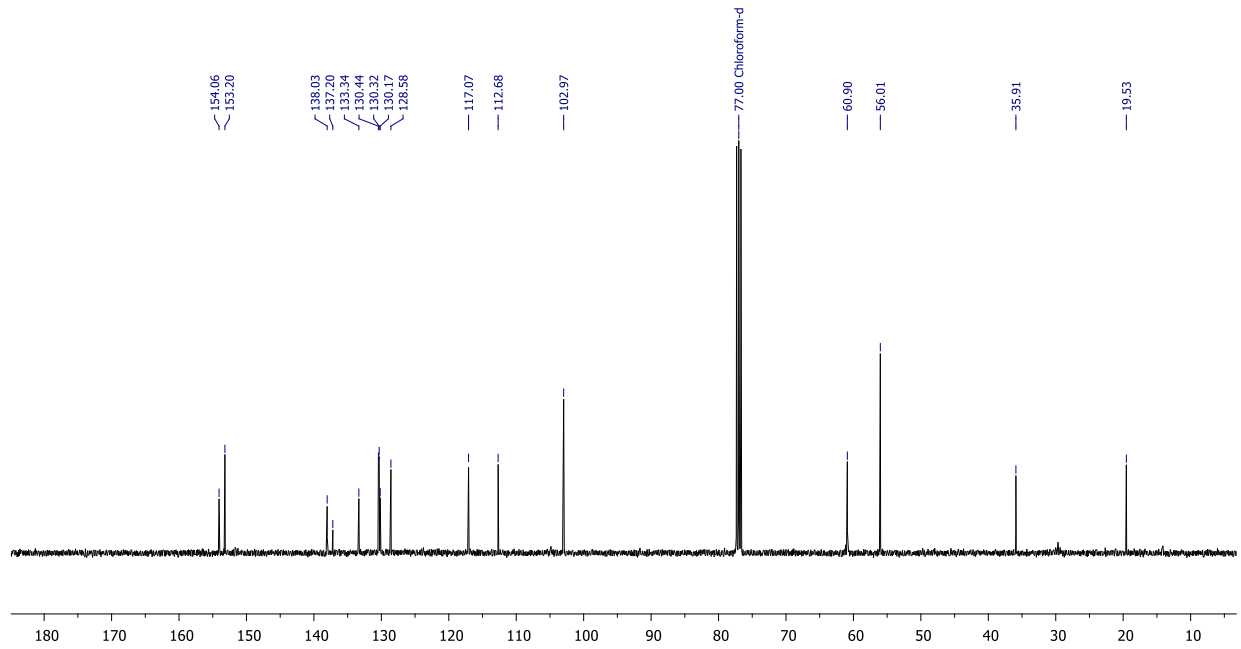
^{1}H NMR (400 MHz) spectrum of **3cd′** in $CDCl_3$



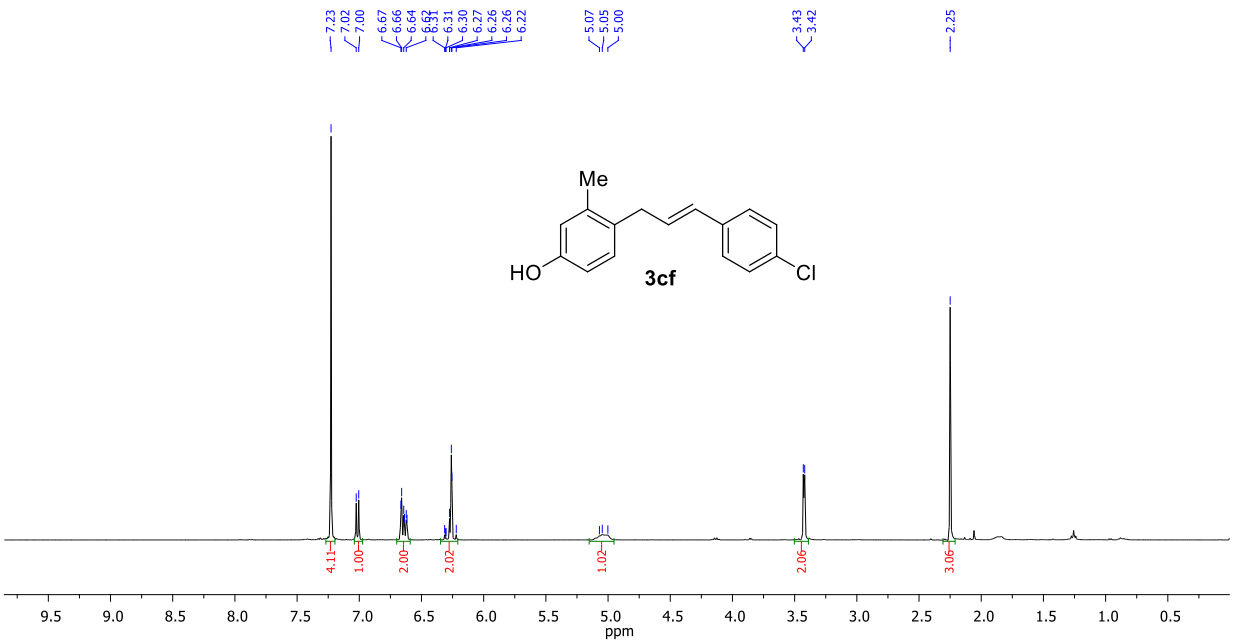
^{13}C NMR (100 MHz) spectrum of **3cd′** in $CDCl_3$



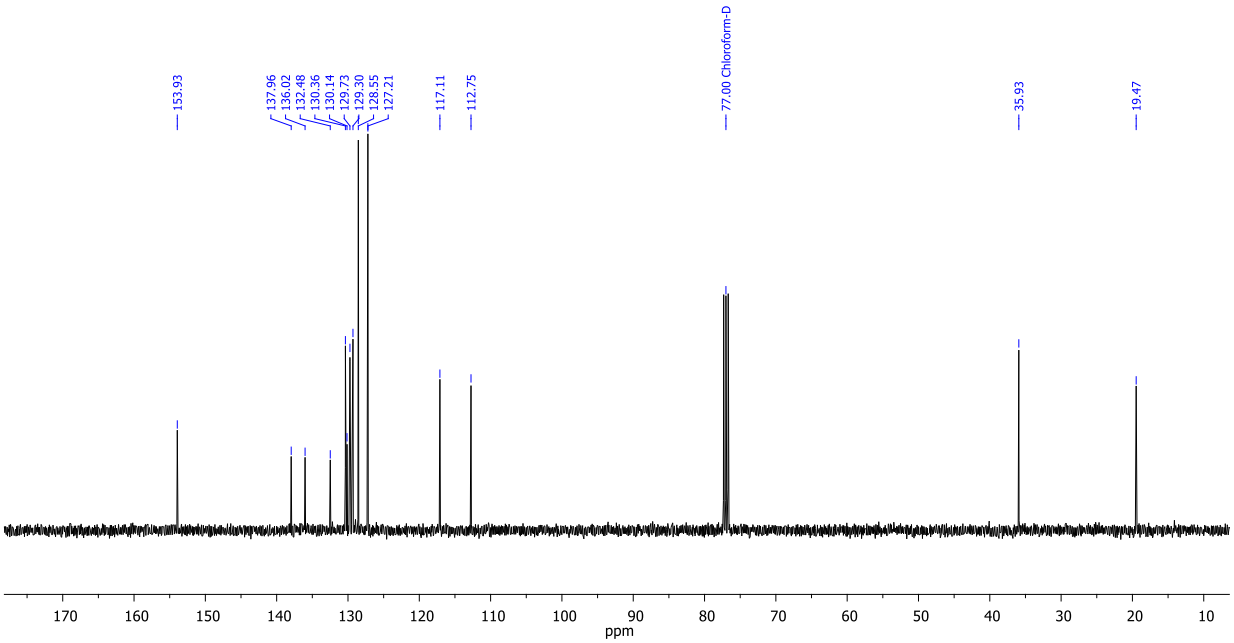
^{1}H NMR (400 MHz) spectrum of **3cn** in $CDCl_3$



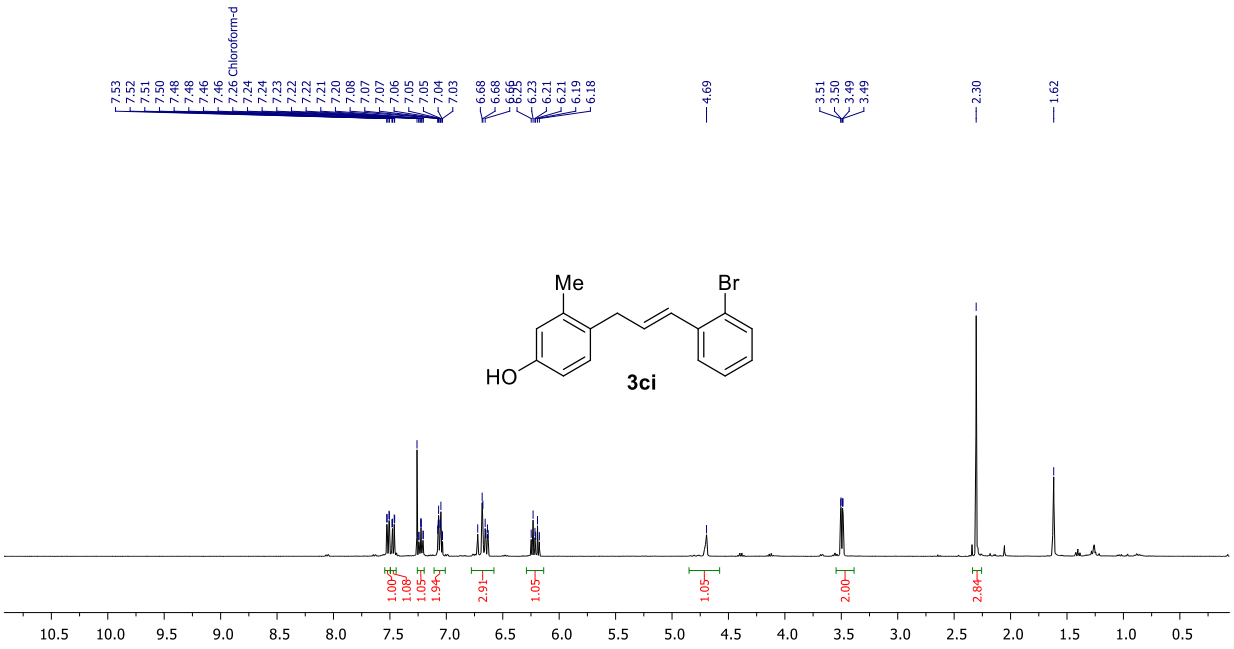
^{13}C NMR (100 MHz) spectrum of **3cn** in $CDCl_3$



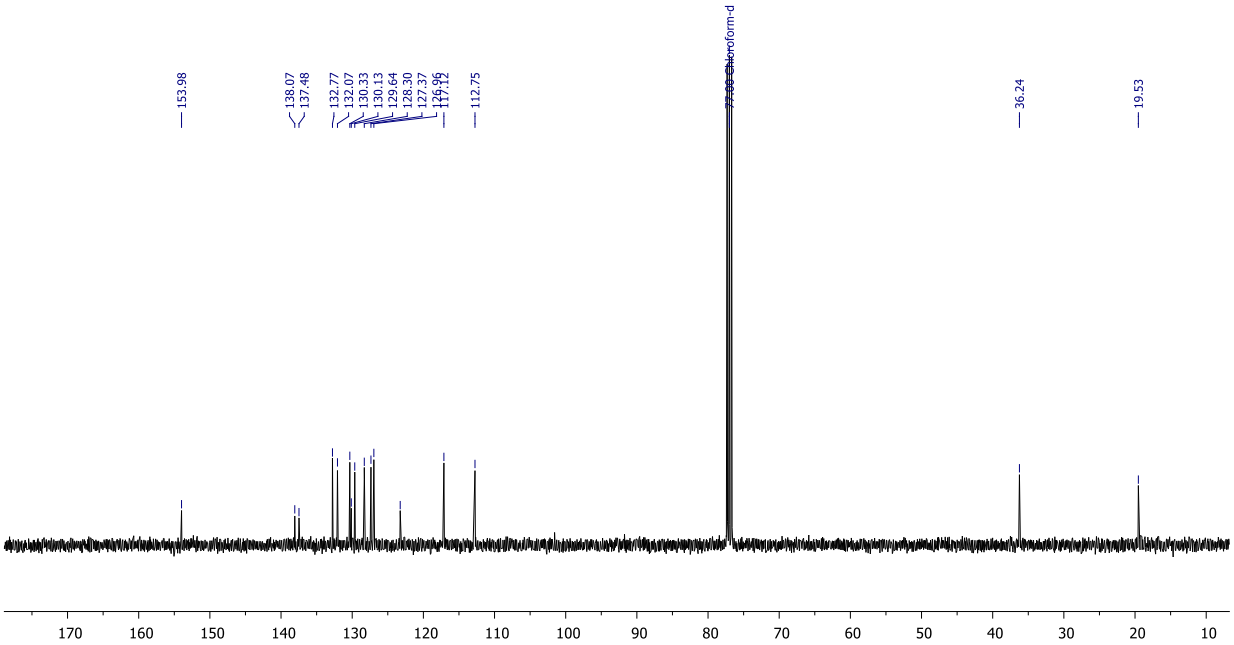
^{1}H NMR (400 MHz) spectrum of **3cf** in $CDCl_3$



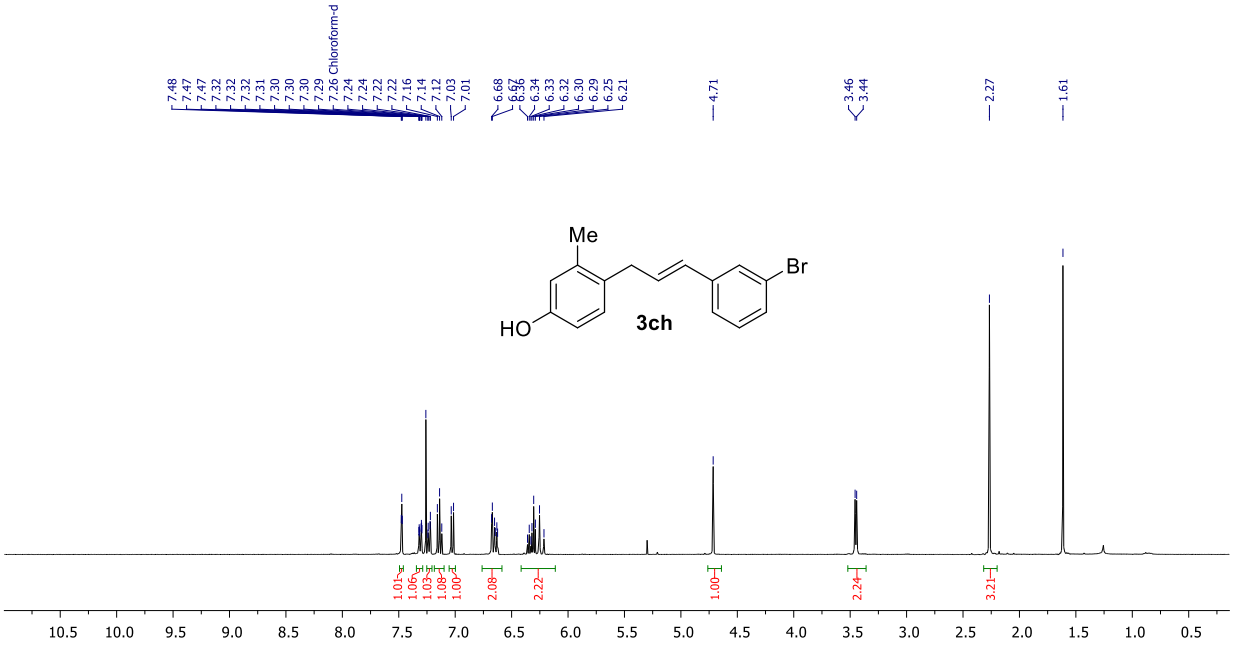
^{13}C NMR (100 MHz) spectrum of **3cf** in $CDCl_3$



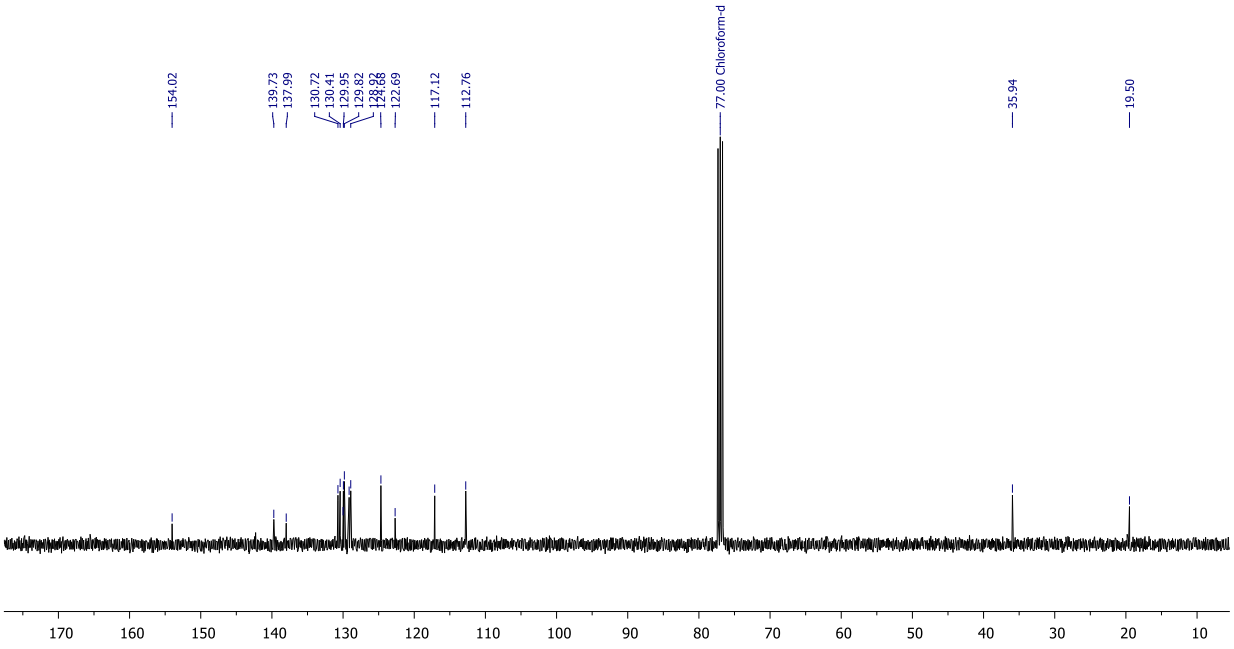
^{1}H NMR (400 MHz) spectrum of **3ci** in $CDCl_3$



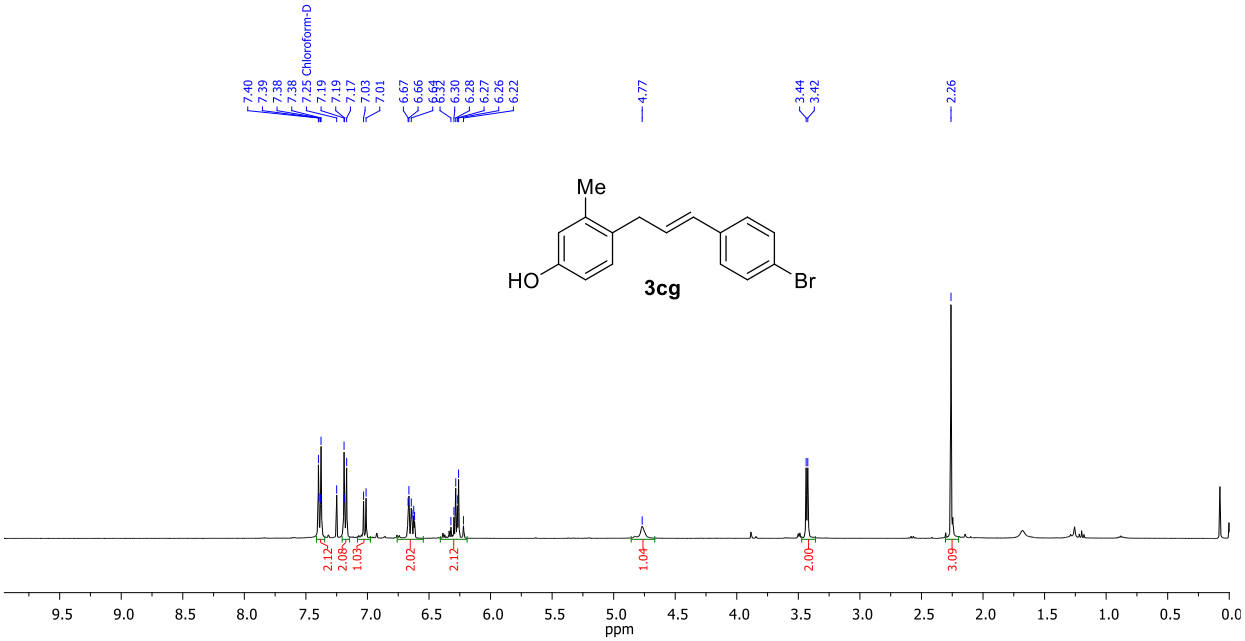
^{13}C NMR (100 MHz) spectrum of **3ci** in $CDCl_3$



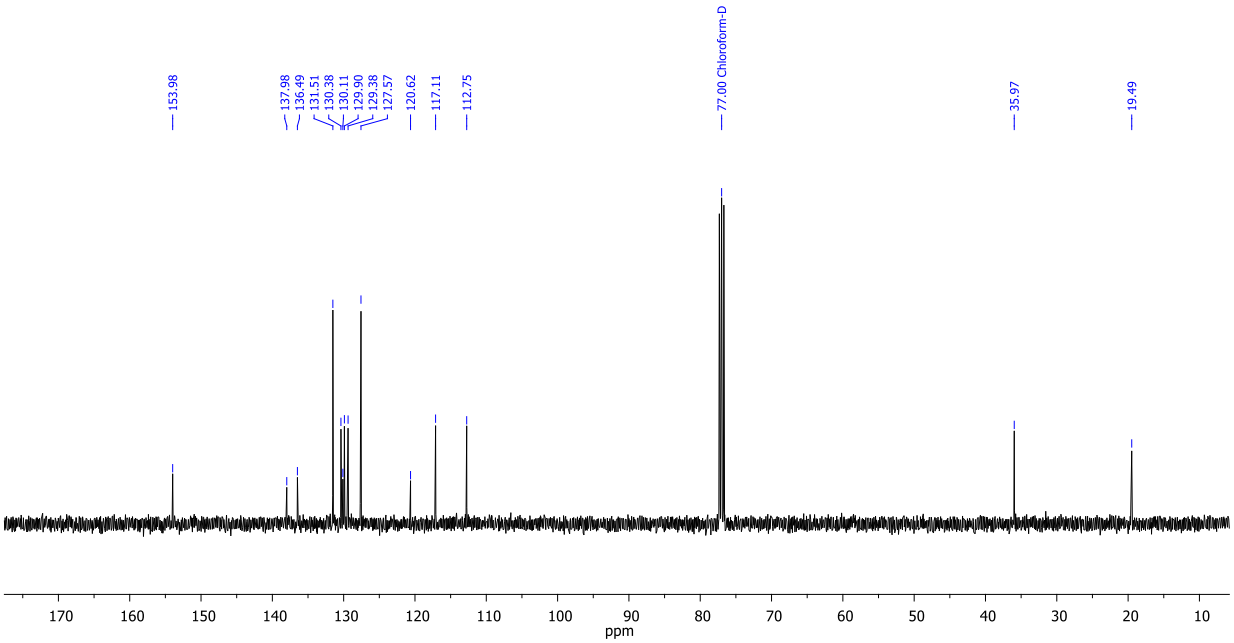
^{1}H NMR (400 MHz) spectrum of **3ch** in $CDCl_3$



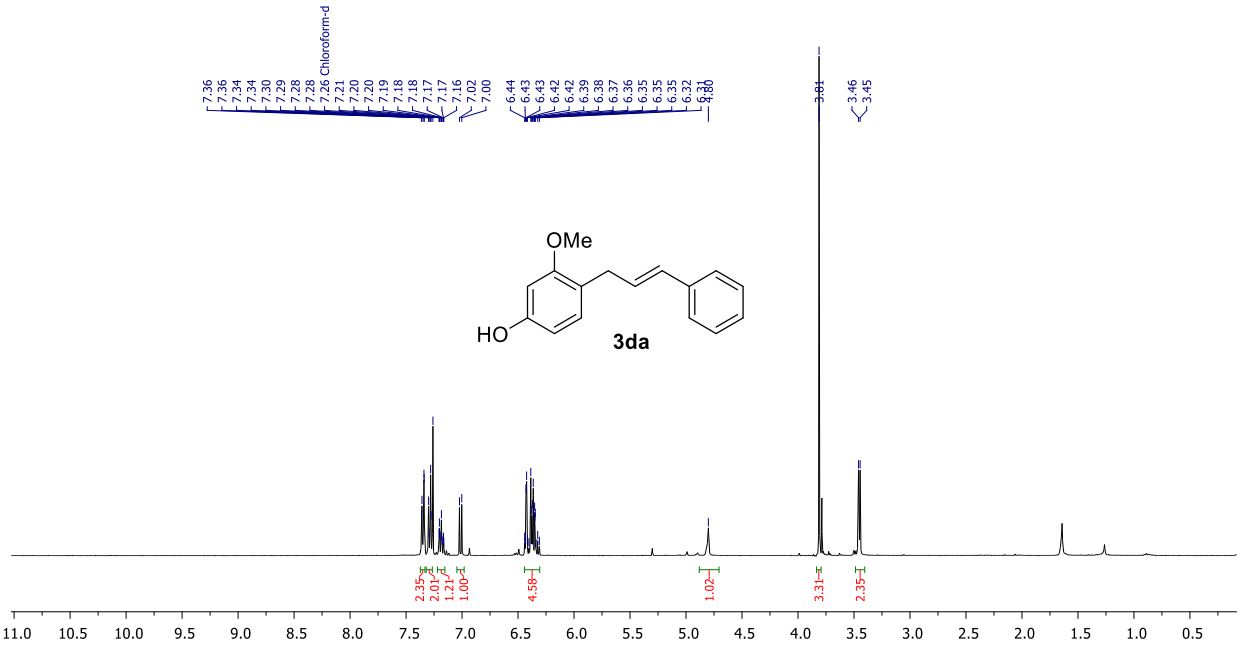
^{13}C NMR (100 MHz) spectrum of **3ch** in $CDCl_3$



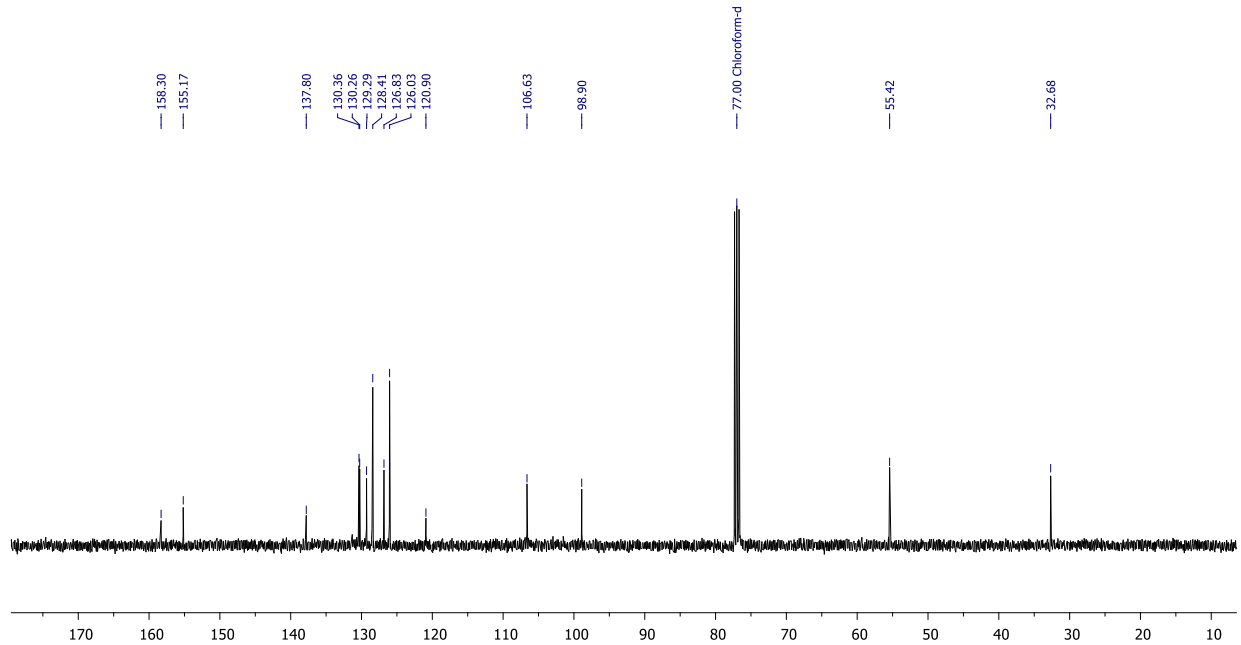
^{1}H NMR (400 MHz) spectrum of **3cg** in $CDCl_3$



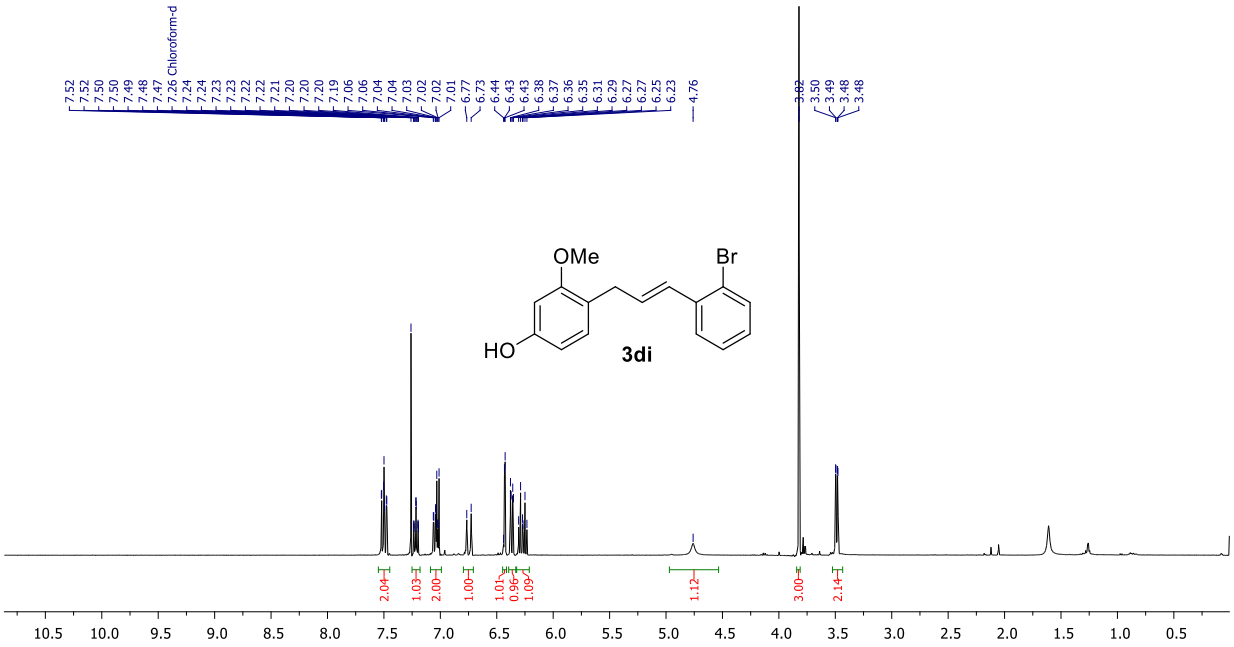
^{13}C NMR (100 MHz) spectrum of **3cg** in $CDCl_3$



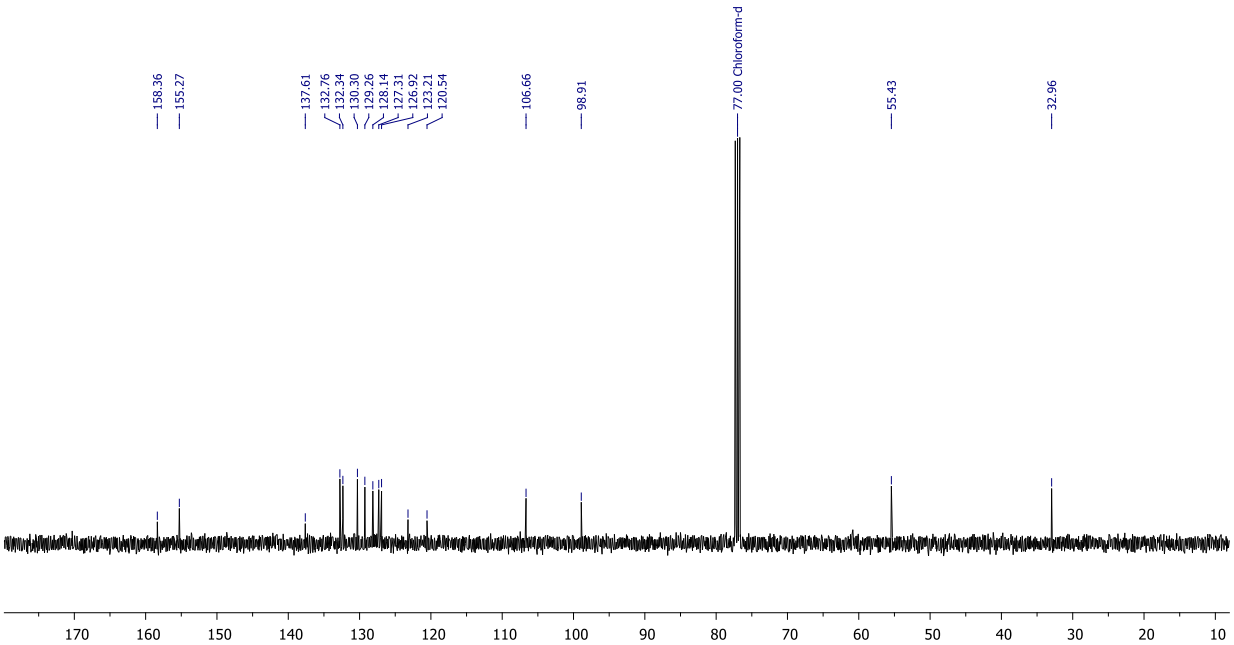
^{1}H NMR (400 MHz) spectrum of **3da** in $CDCl_3$



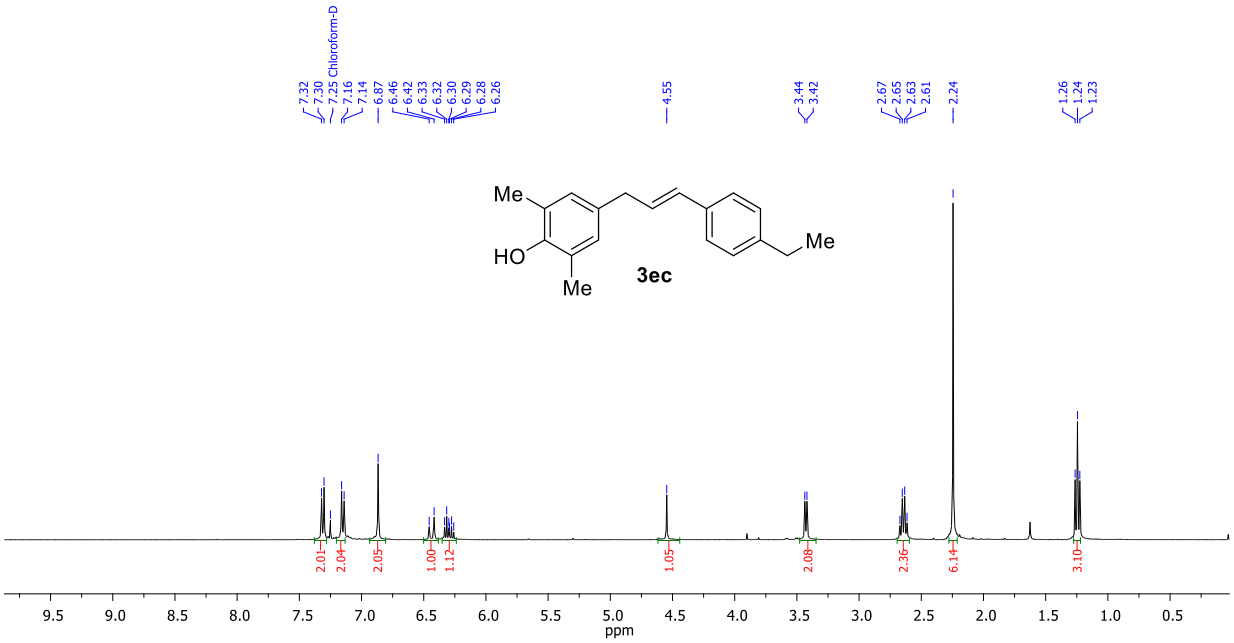
^{13}C NMR (100 MHz) spectrum of **3da** in $CDCl_3$



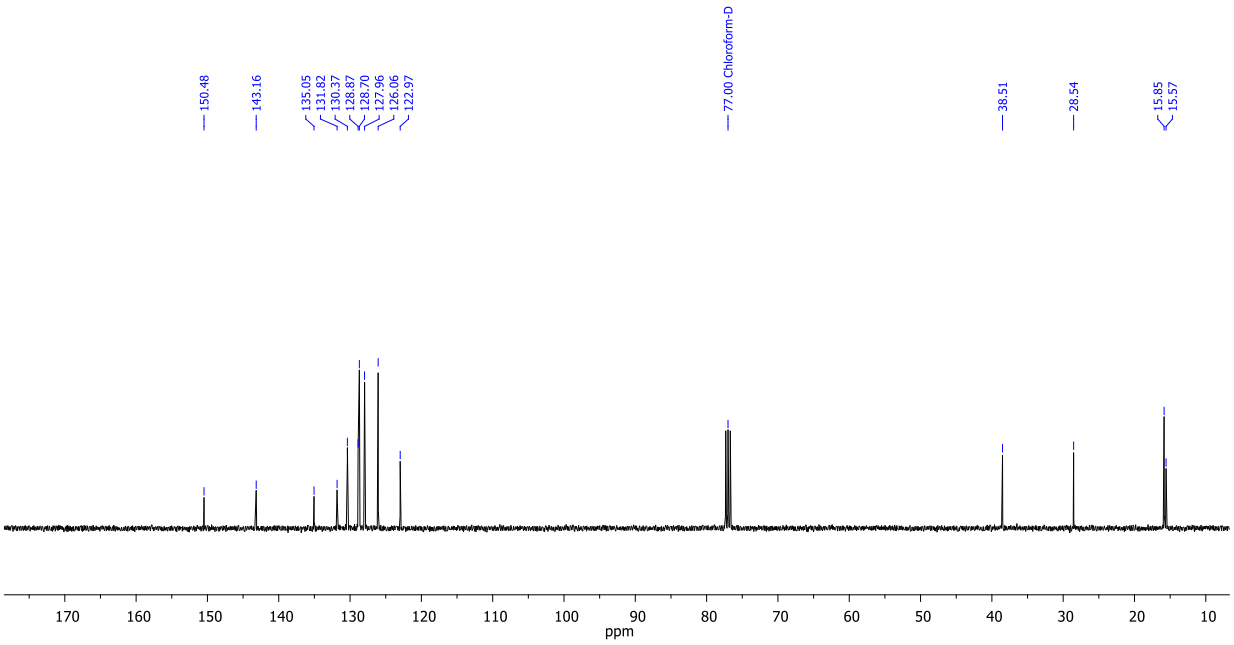
^{1}H NMR (400 MHz) spectrum of **3di** in $CDCl_3$



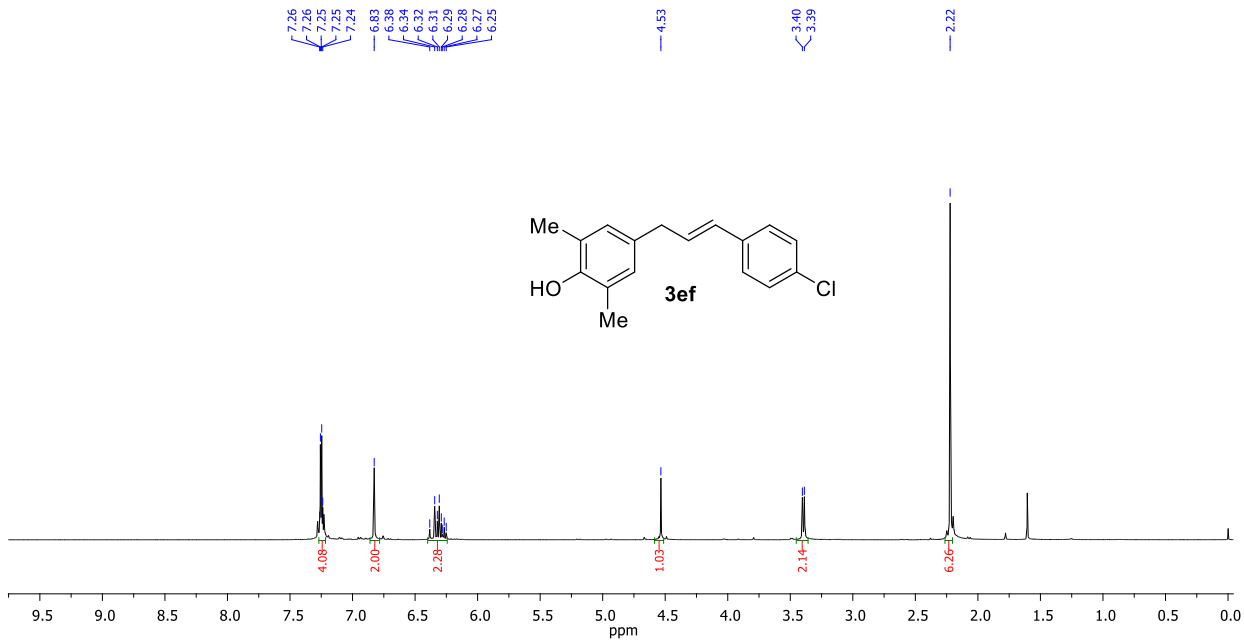
^{13}C NMR (100 MHz) spectrum of **3di** in $CDCl_3$



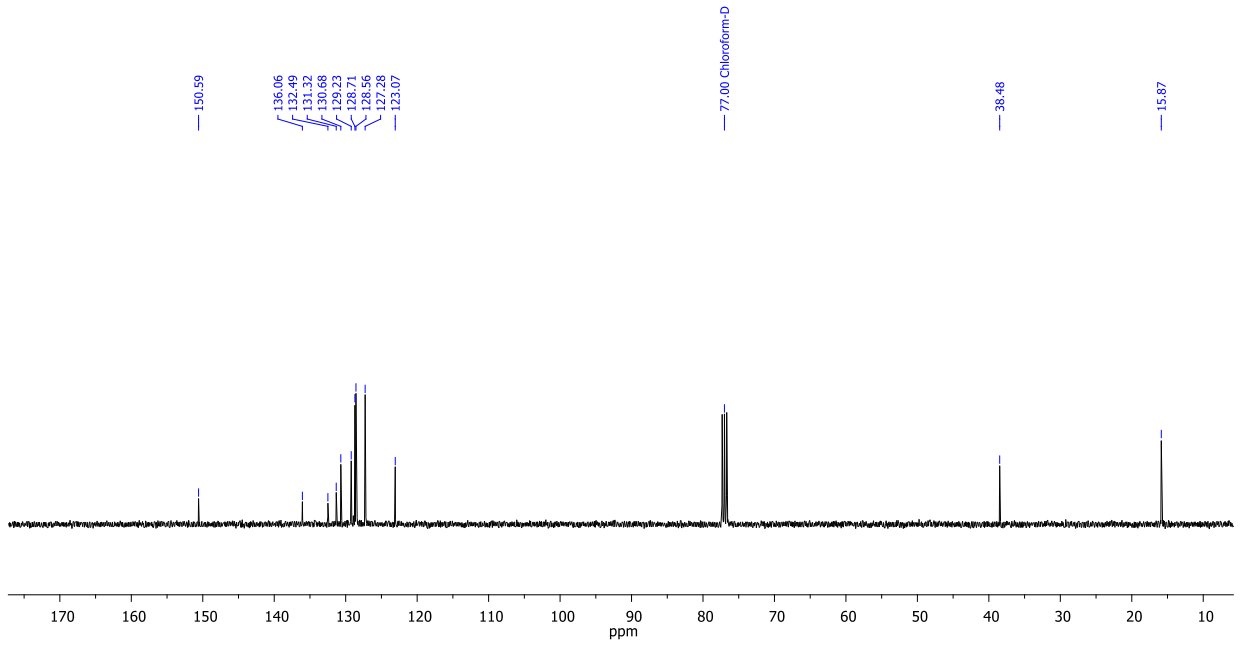
^{1}H NMR (400 MHz) spectrum of **3ec** in $CDCl_3$



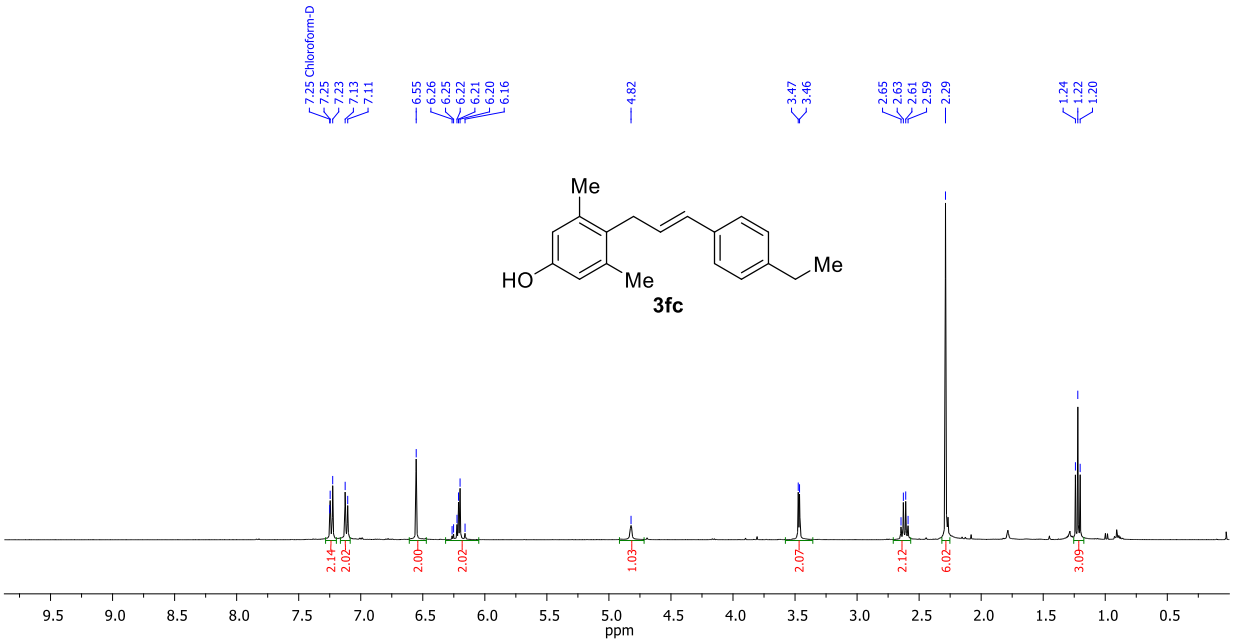
^{13}C NMR (100 MHz) spectrum of **3ec** in $CDCl_3$



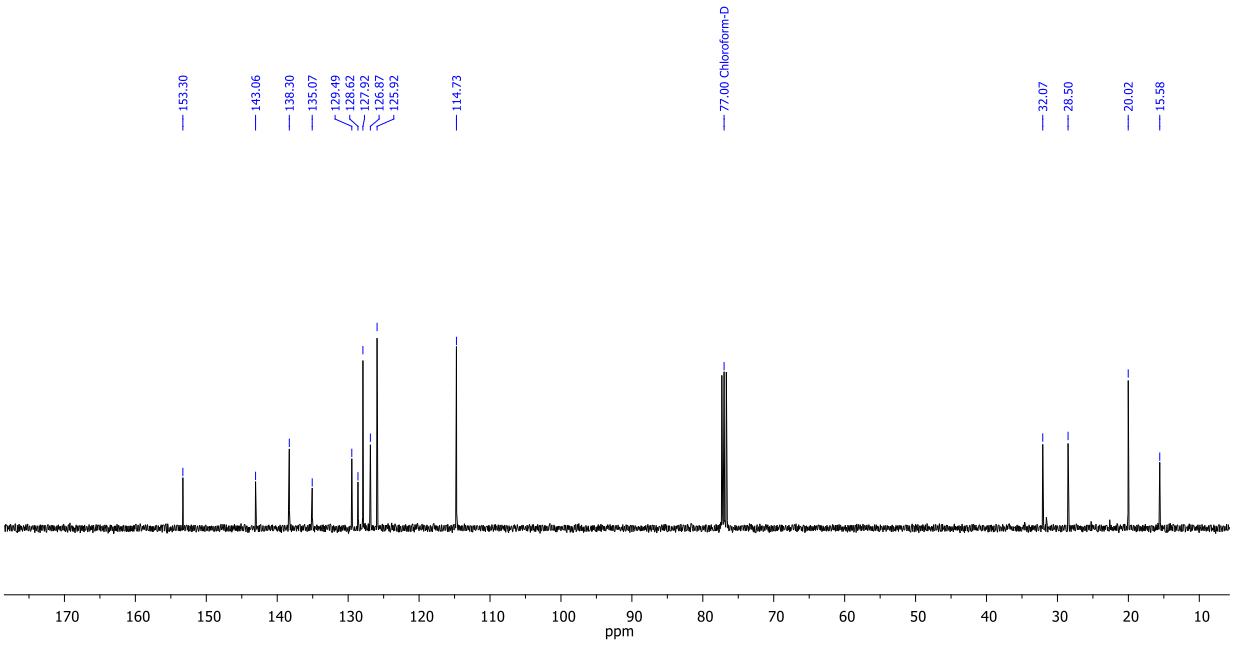
^{1}H NMR (400 MHz) spectrum of **3ef** in $CDCl_3$



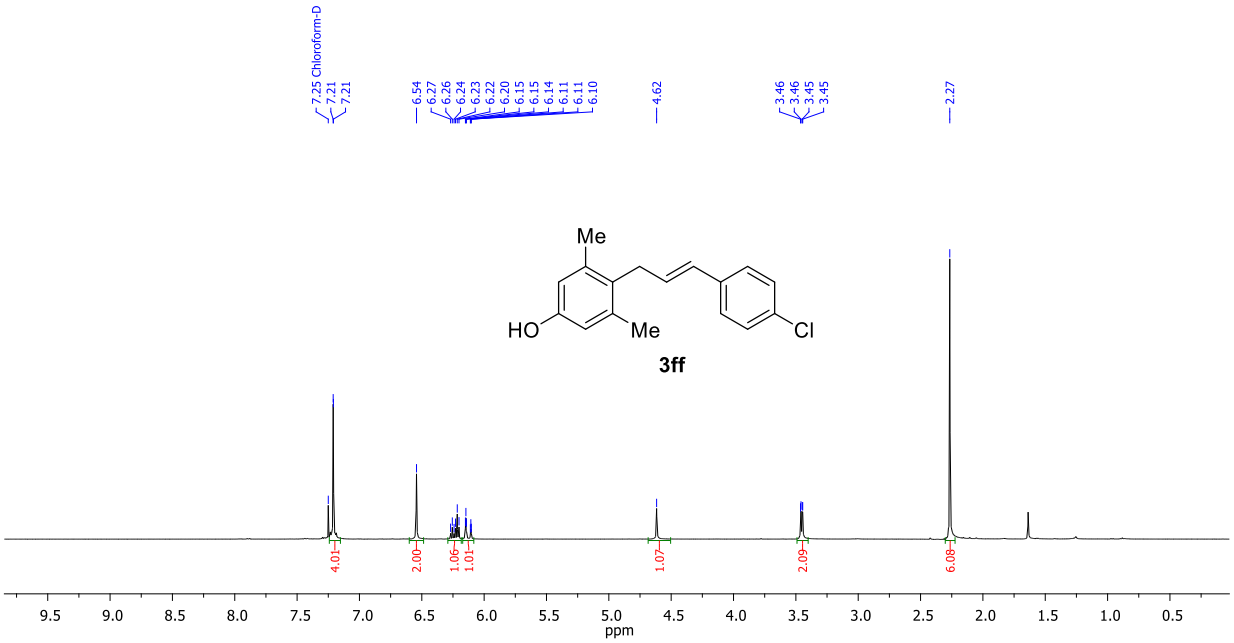
^{13}C NMR (100 MHz) spectrum of **3ef** in $CDCl_3$



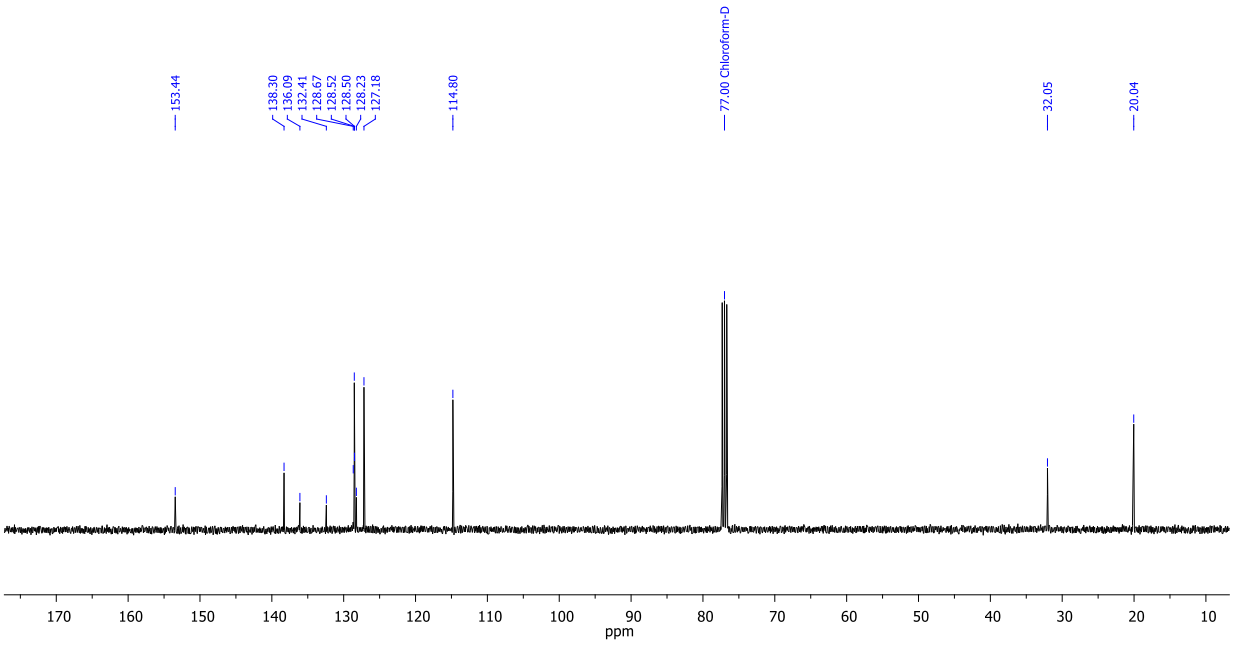
^{1}H NMR (400 MHz) spectrum of **3fc** in $CDCl_3$



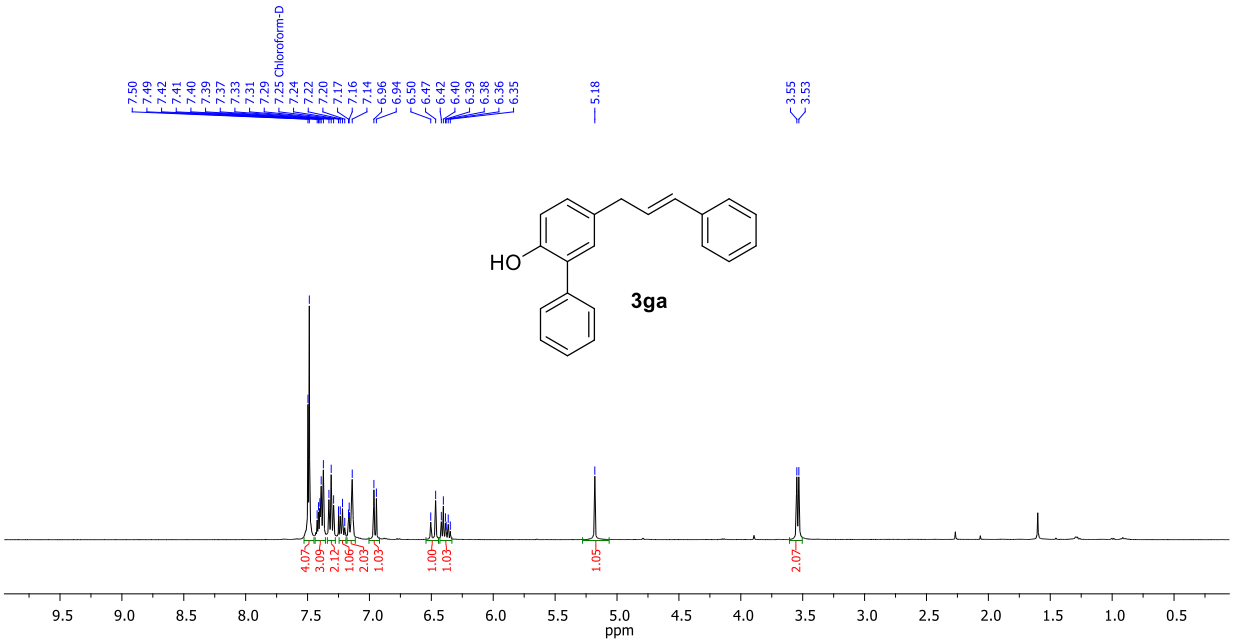
^{13}C NMR (100 MHz) spectrum of **3fc** in $CDCl_3$



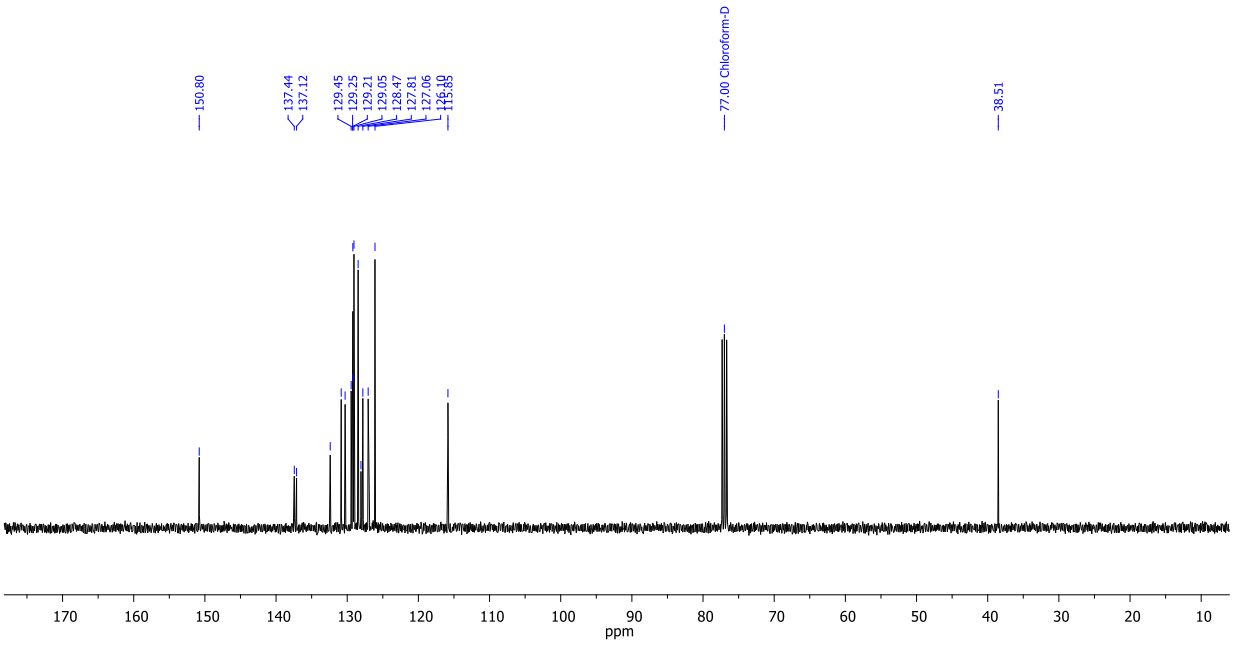
^{1}H NMR (400 MHz) spectrum of **3ff** in $CDCl_3$



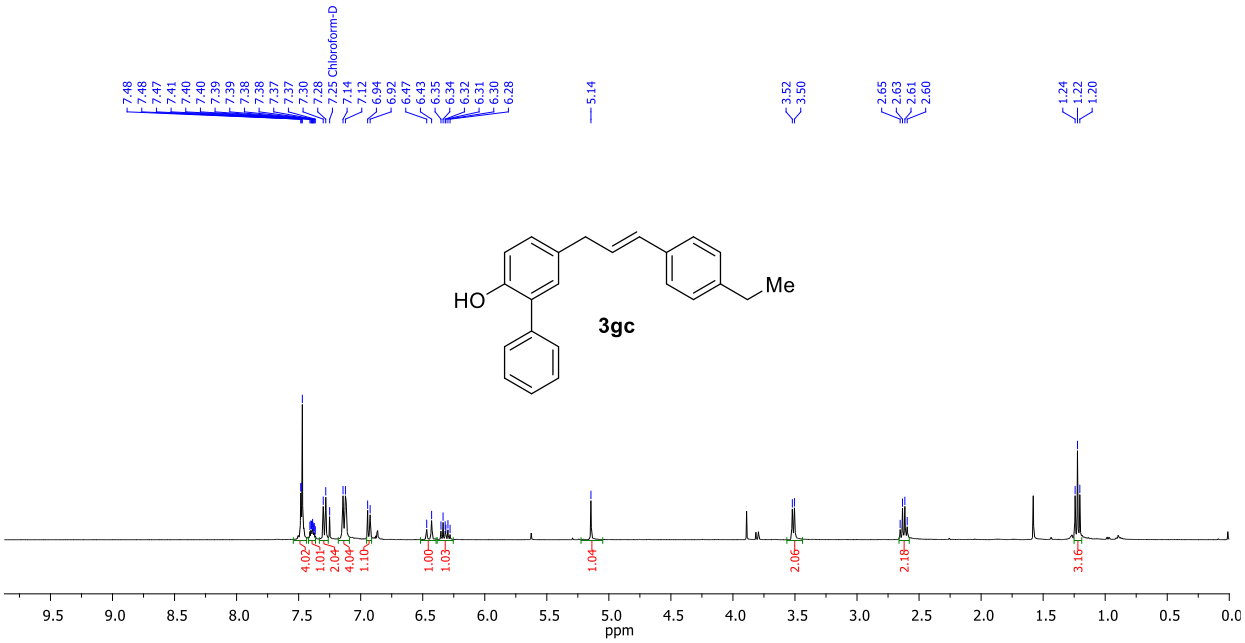
^{13}C NMR (100 MHz) spectrum of **3ff** in $CDCl_3$



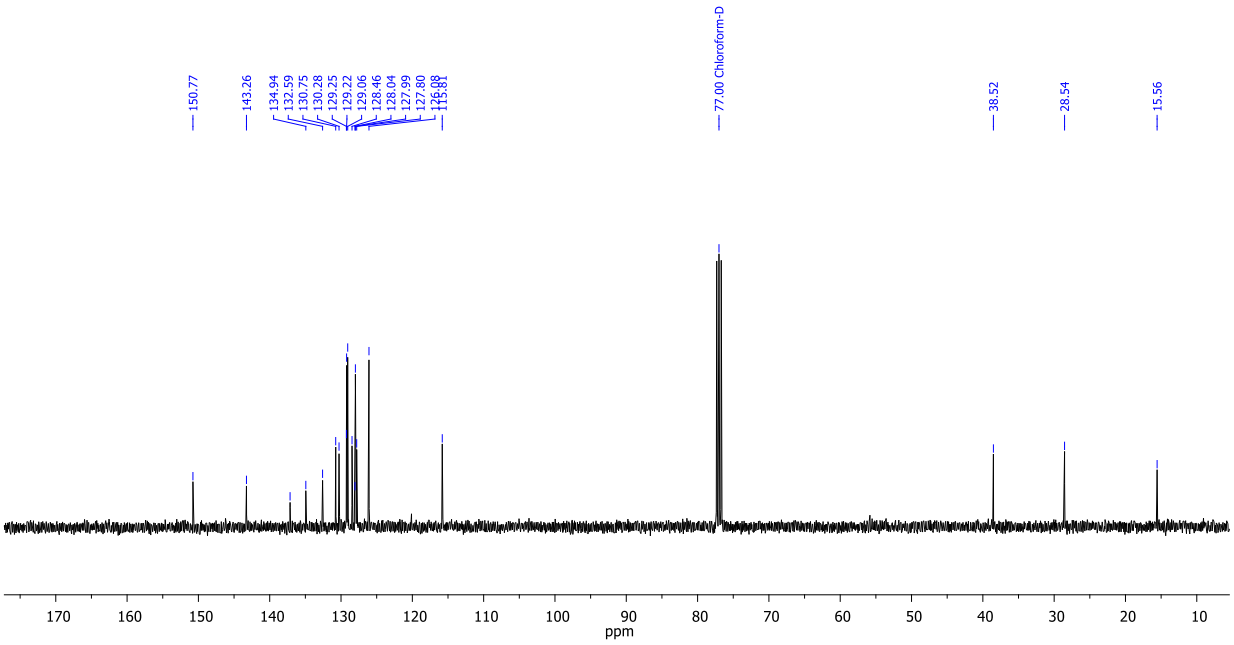
^{1}H NMR (400 MHz) spectrum of **3ga** in $CDCl_3$



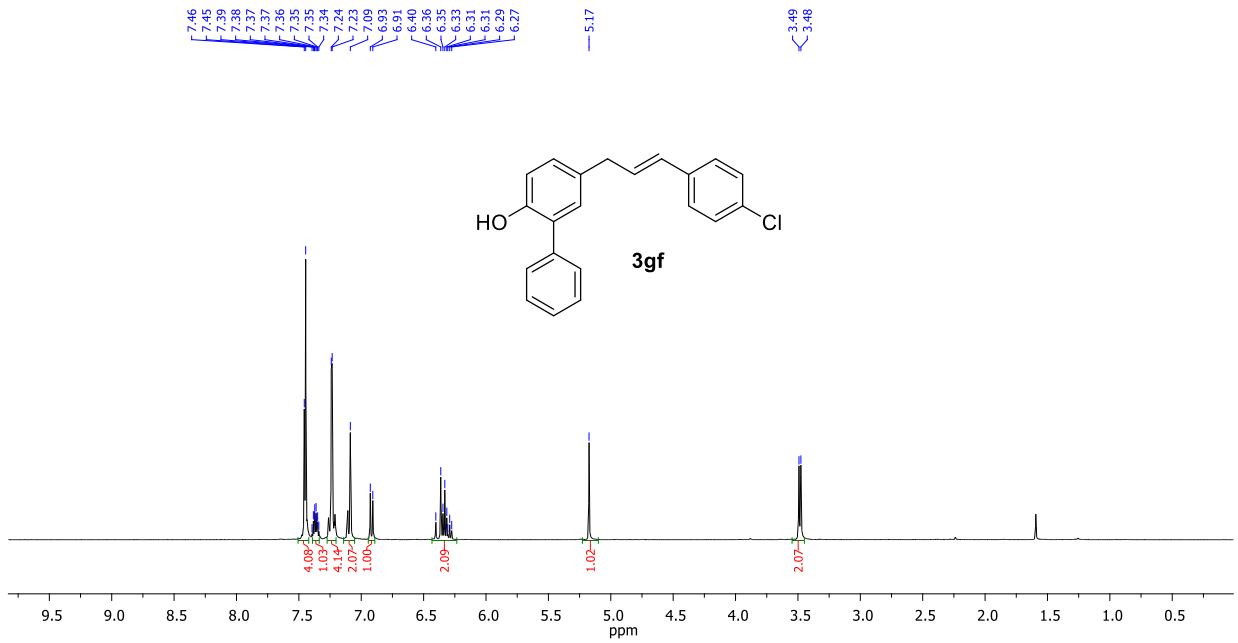
^{13}C NMR (100 MHz) spectrum of **3ga** in $CDCl_3$



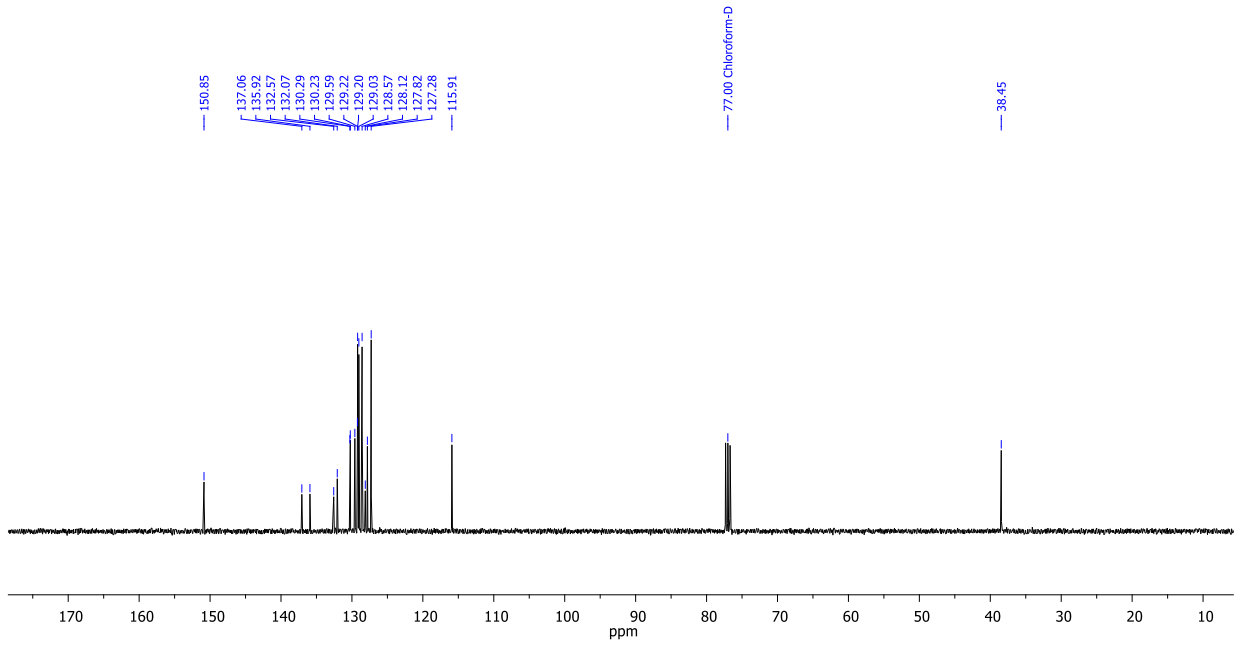
^{1}H NMR (400 MHz) spectrum of **3gc** in $CDCl_3$



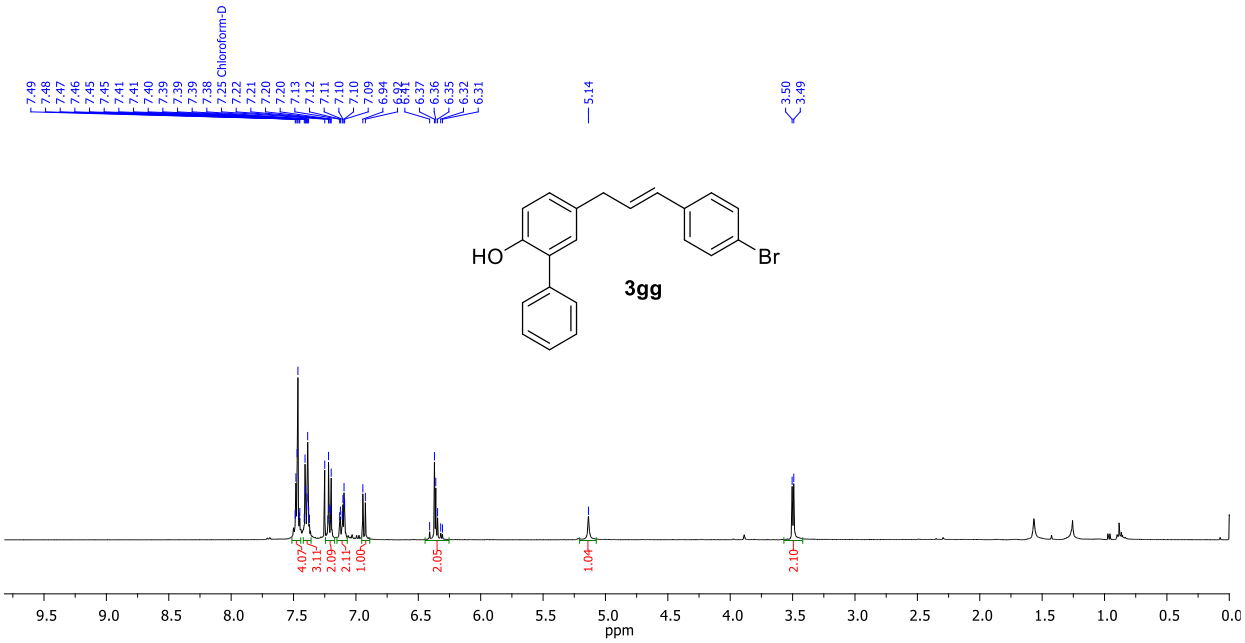
^{13}C NMR (100 MHz) spectrum of **3gc** in $CDCl_3$



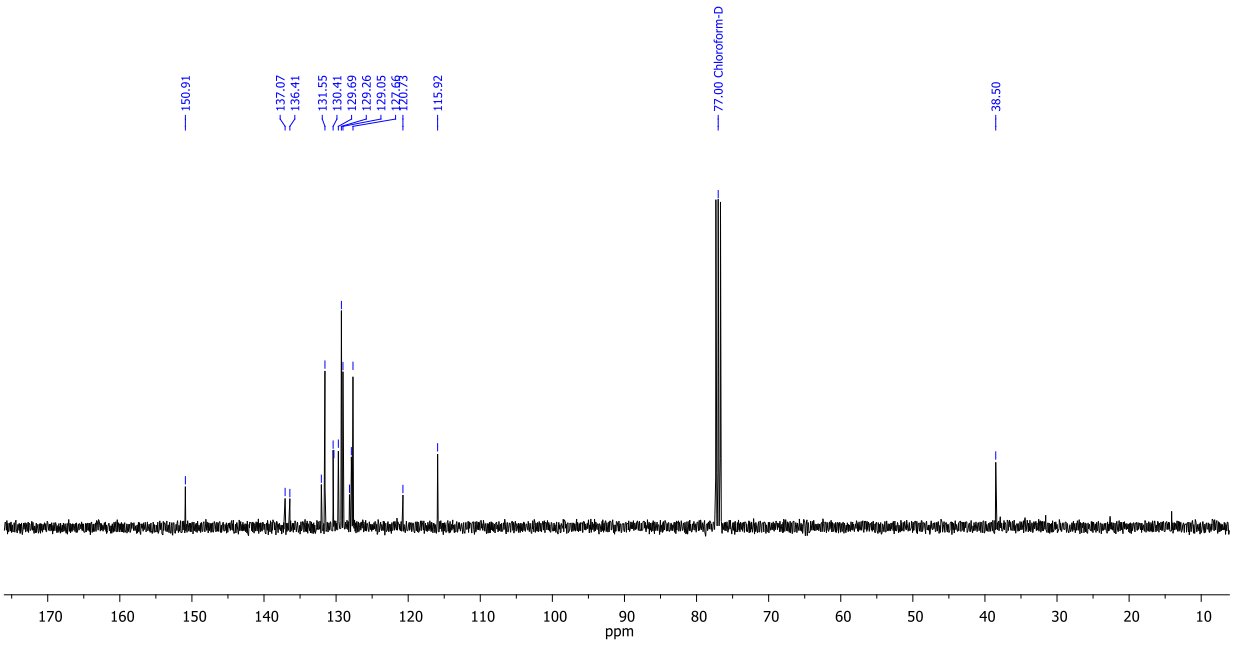
^{1}H NMR (400 MHz) spectrum of **3gf** in $CDCl_3$



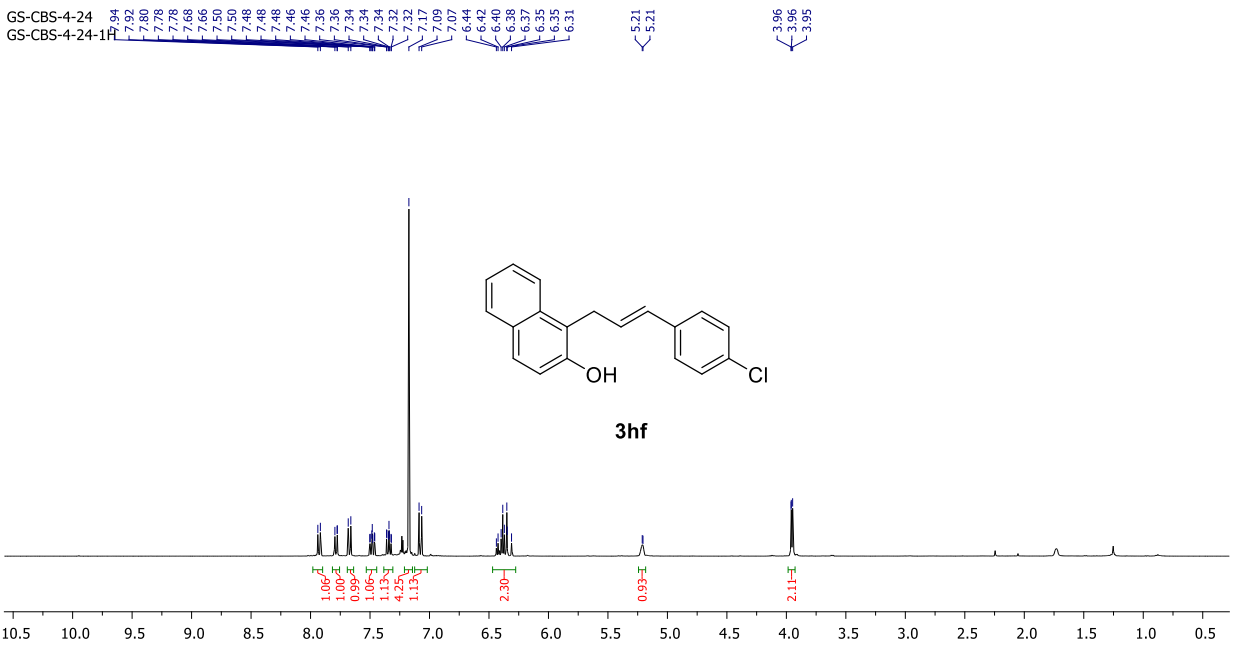
^{13}C NMR (100 MHz) spectrum of **3gf** in $CDCl_3$



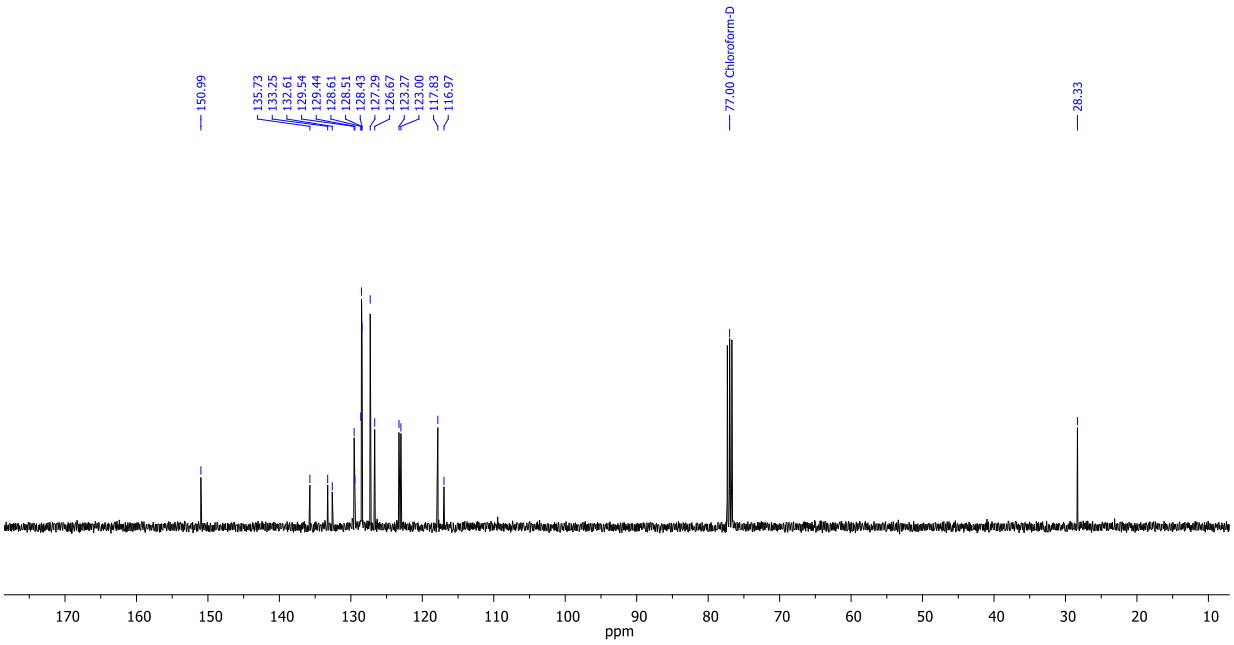
^{1}H NMR (400 MHz) spectrum of **3gg** in $CDCl_3$



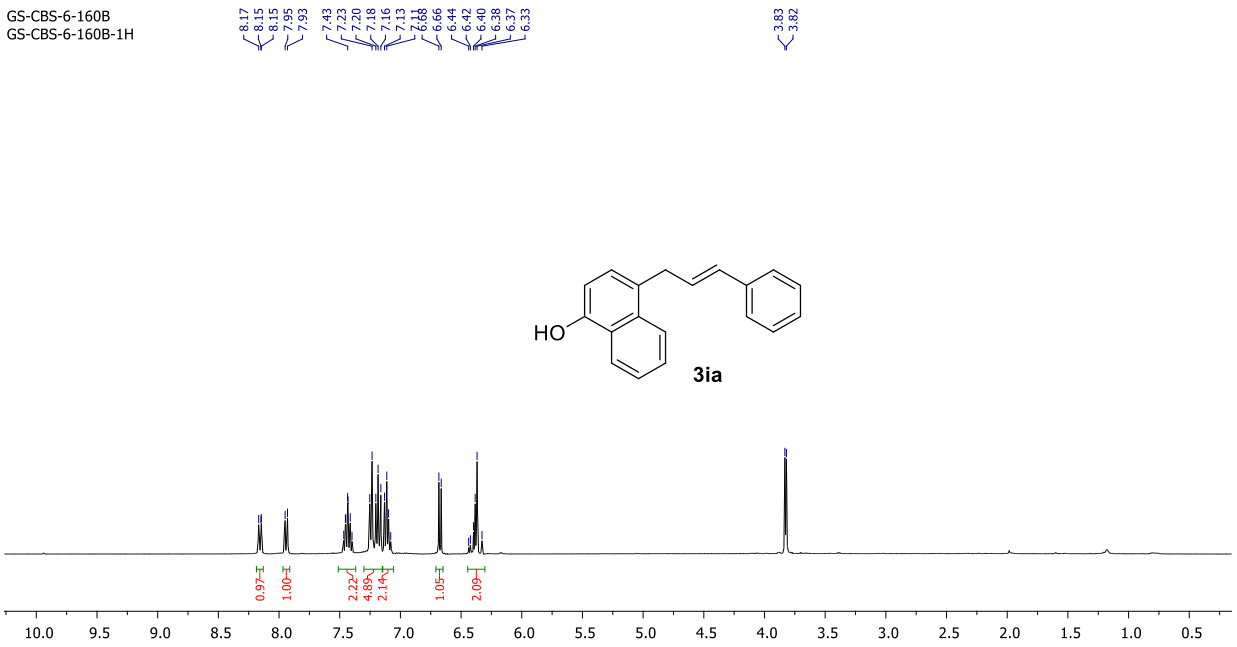
^{13}C NMR (100 MHz) spectrum of **3gg** in $CDCl_3$



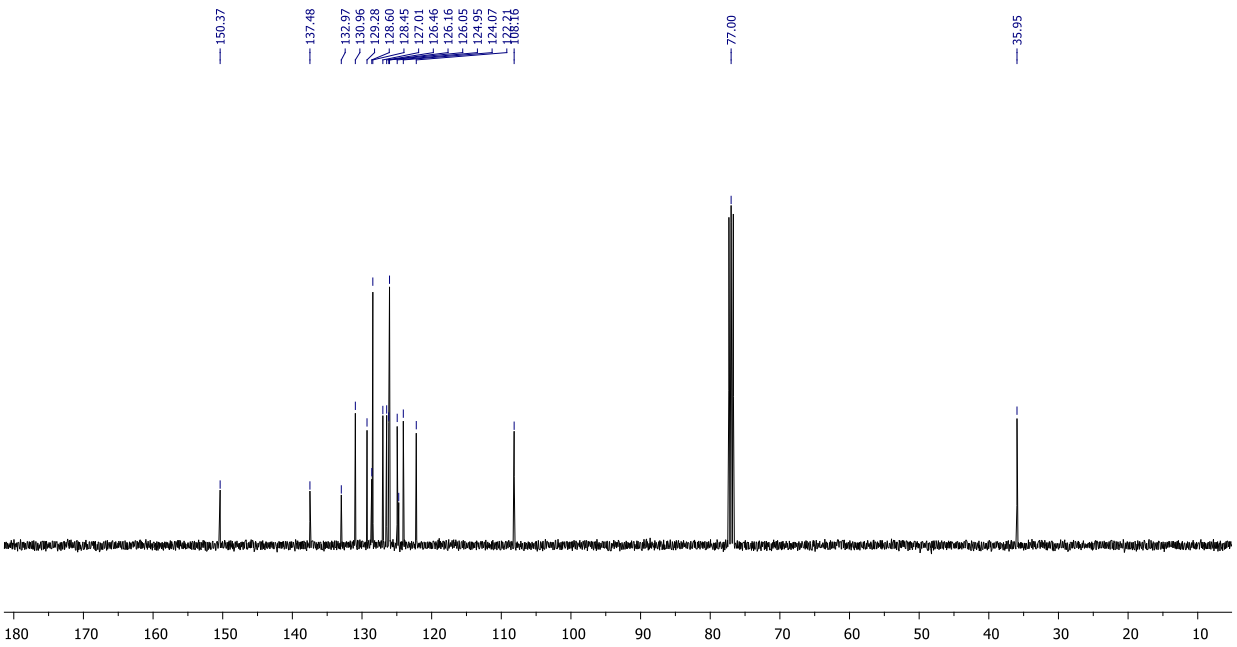
^{1}H NMR (400 MHz) spectrum of **3hf** in $CDCl_3$



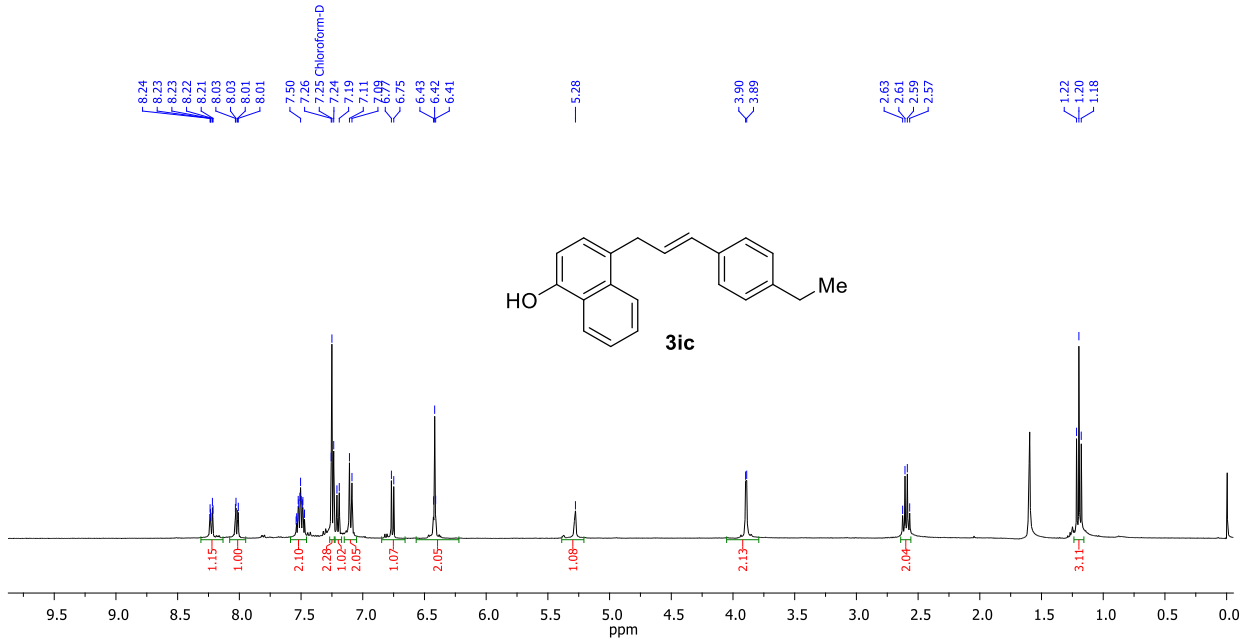
^{13}C NMR (100 MHz) spectrum of **3hf** in $CDCl_3$



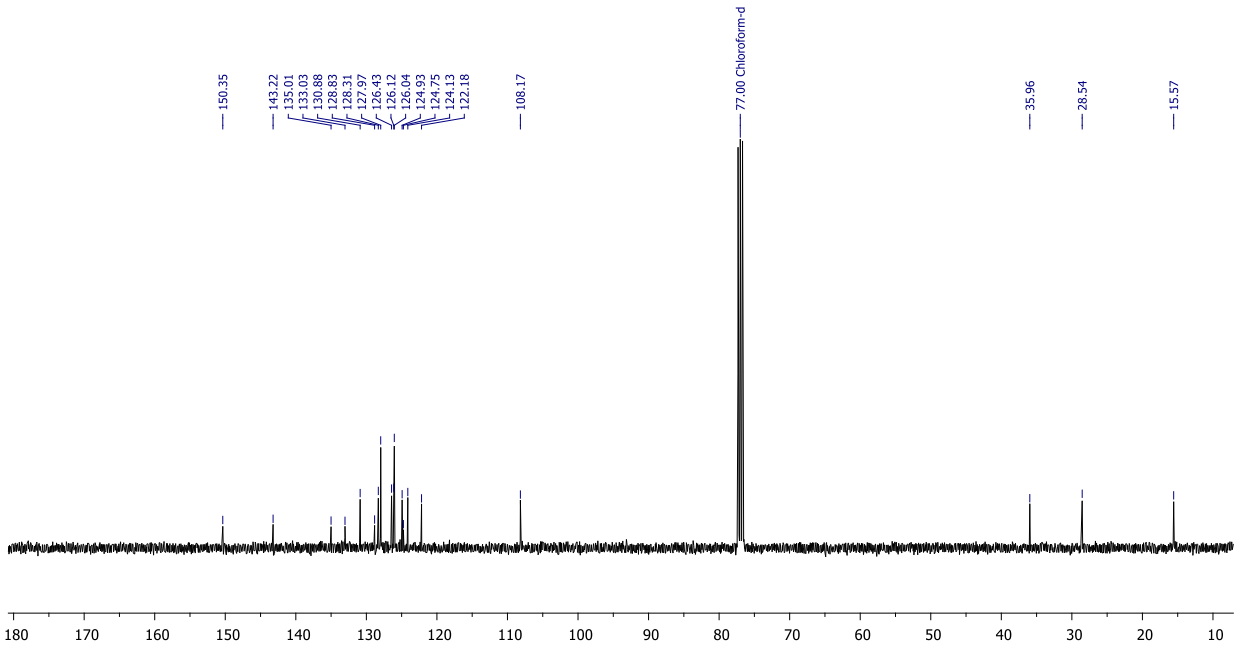
^{1}H NMR (400 MHz) spectrum of **3ia** in $CDCl_3$



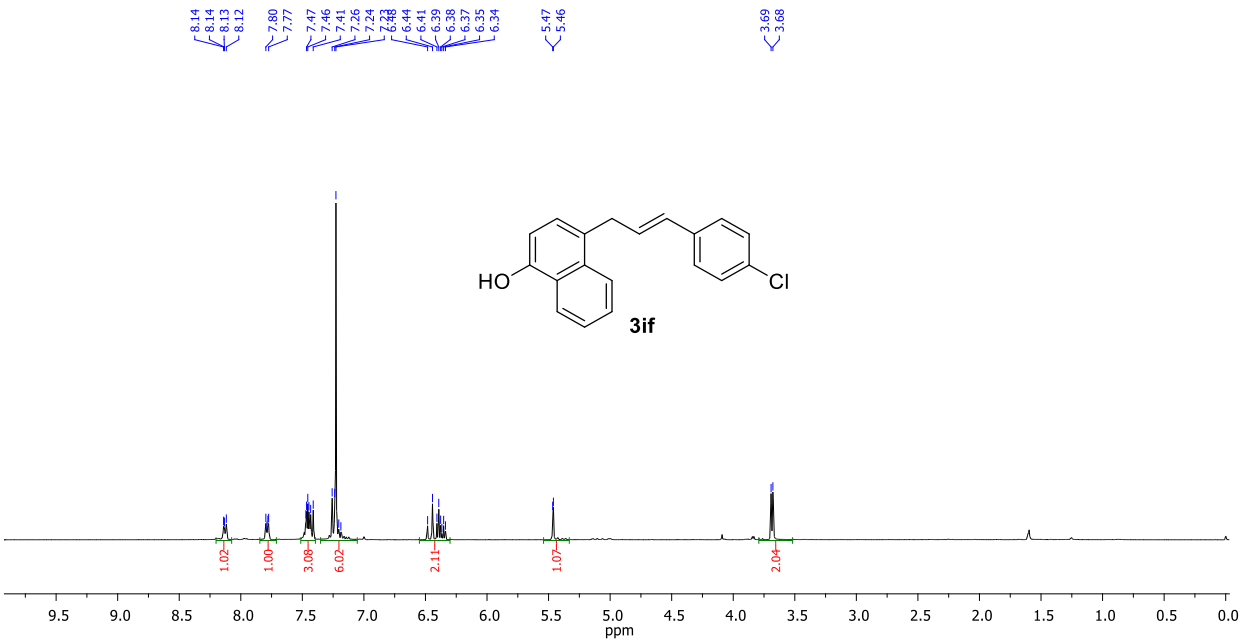
^{13}C NMR (100 MHz) spectrum of **3ia** in $CDCl_3$



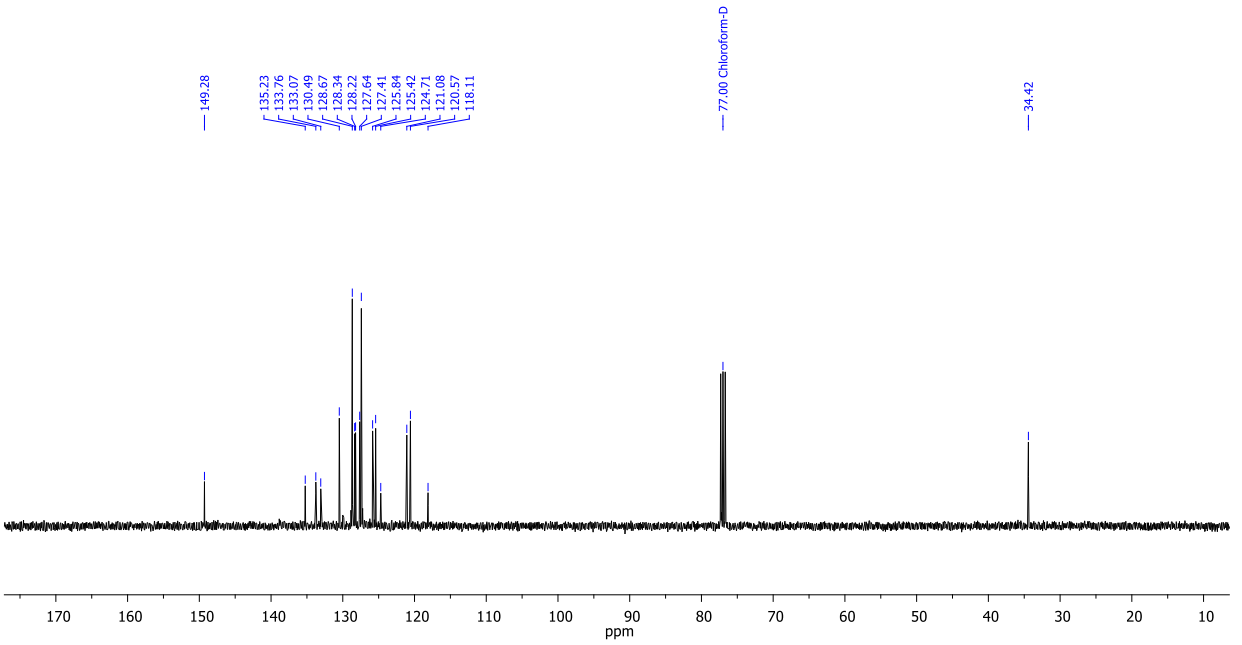
^{1}H NMR (400 MHz) spectrum of **3ic** in $CDCl_3$



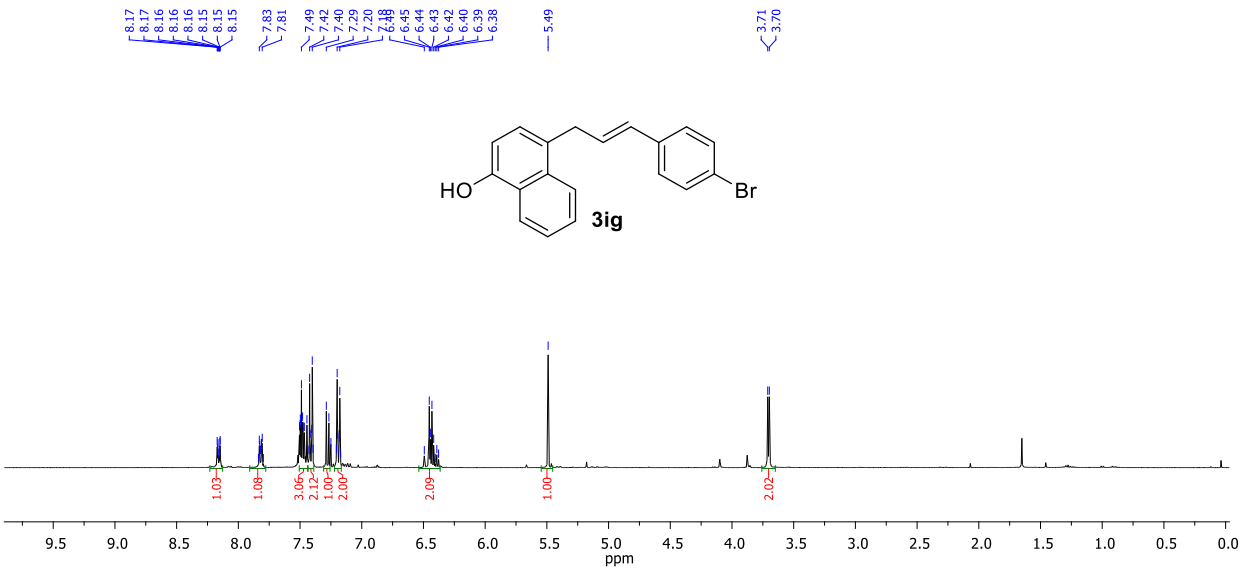
^{13}C NMR (100 MHz) spectrum of **3ic** in $CDCl_3$



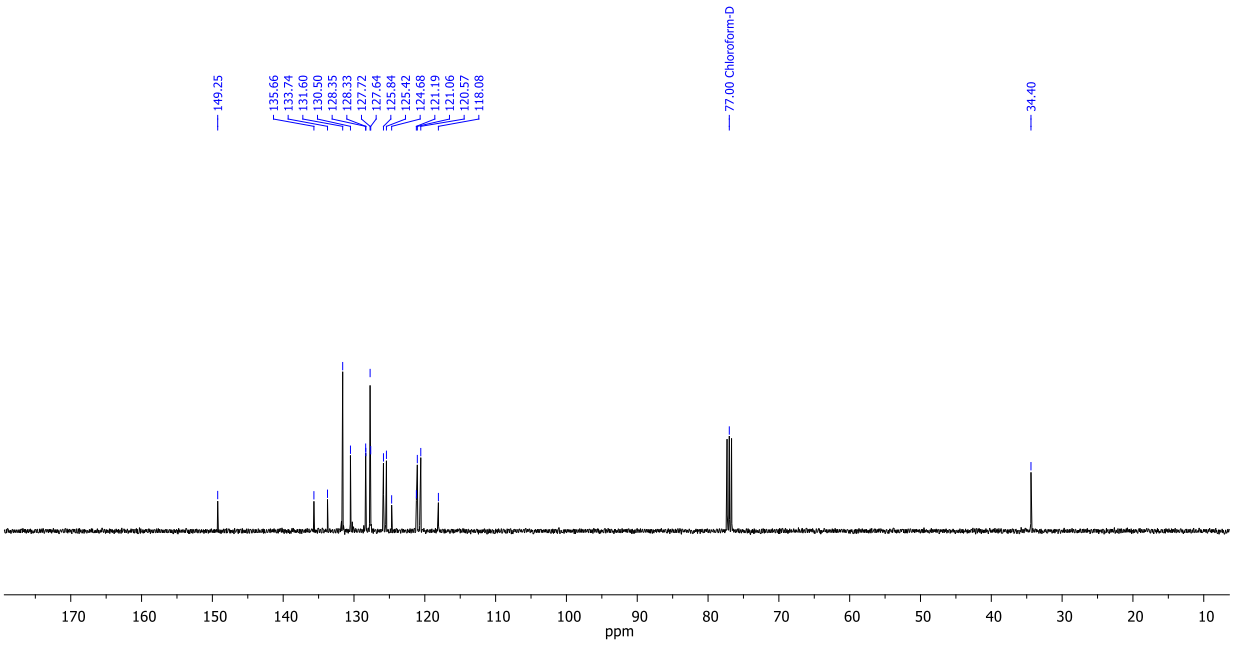
^{1}H NMR (400 MHz) spectrum of **3if** in $CDCl_3$



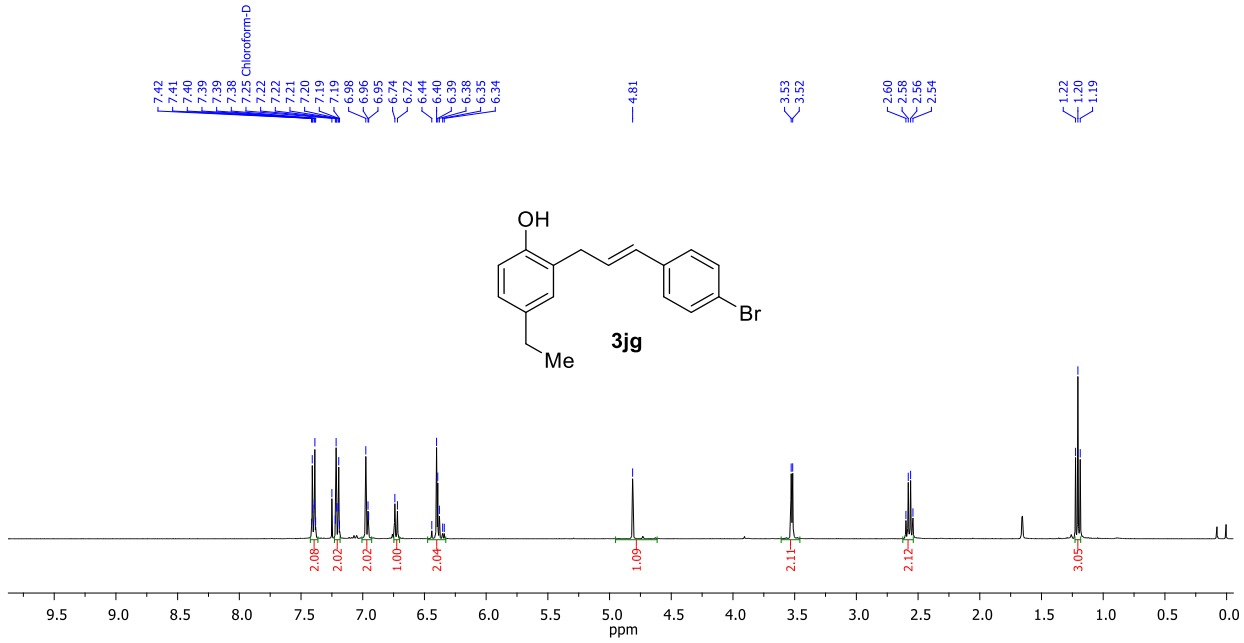
^{13}C NMR (100 MHz) spectrum of **3if** in $CDCl_3$



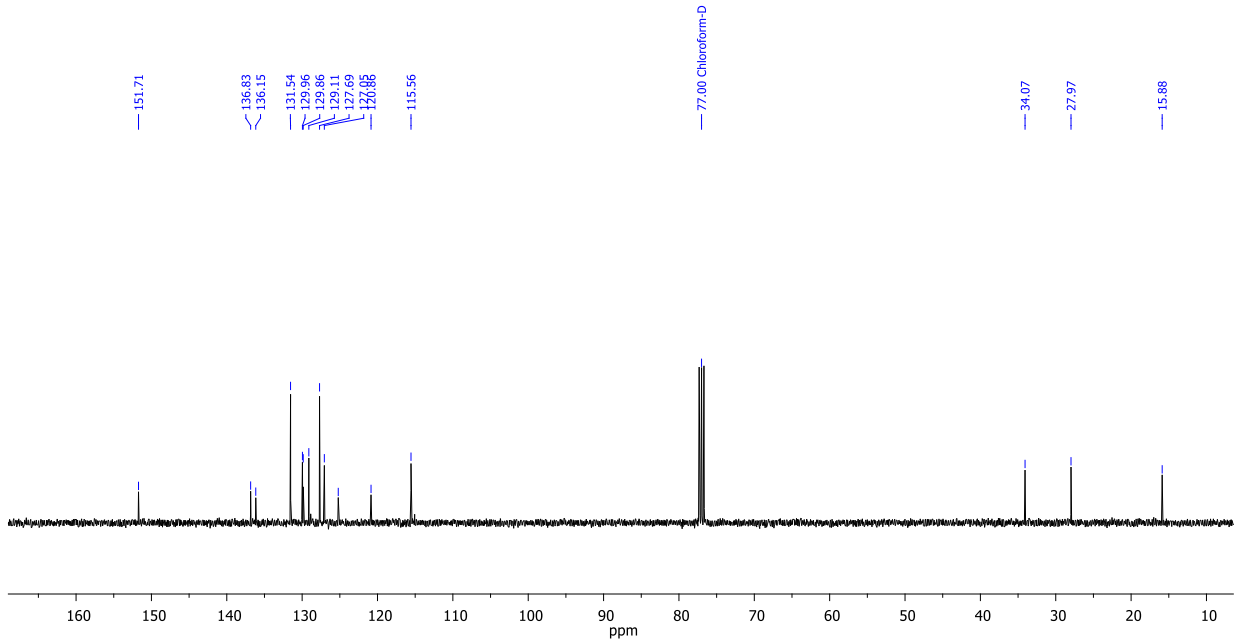
^{1}H NMR (400 MHz) spectrum of **3ig** in $CDCl_3$



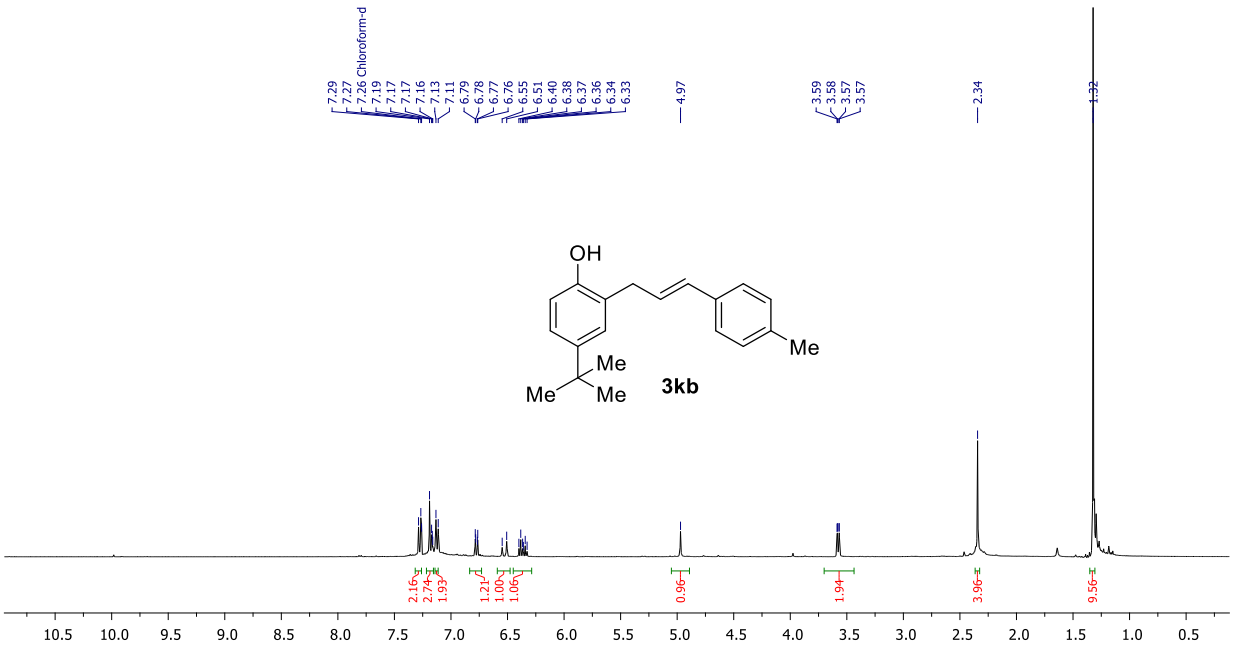
^{13}C NMR (100 MHz) spectrum of **3ig** in $CDCl_3$



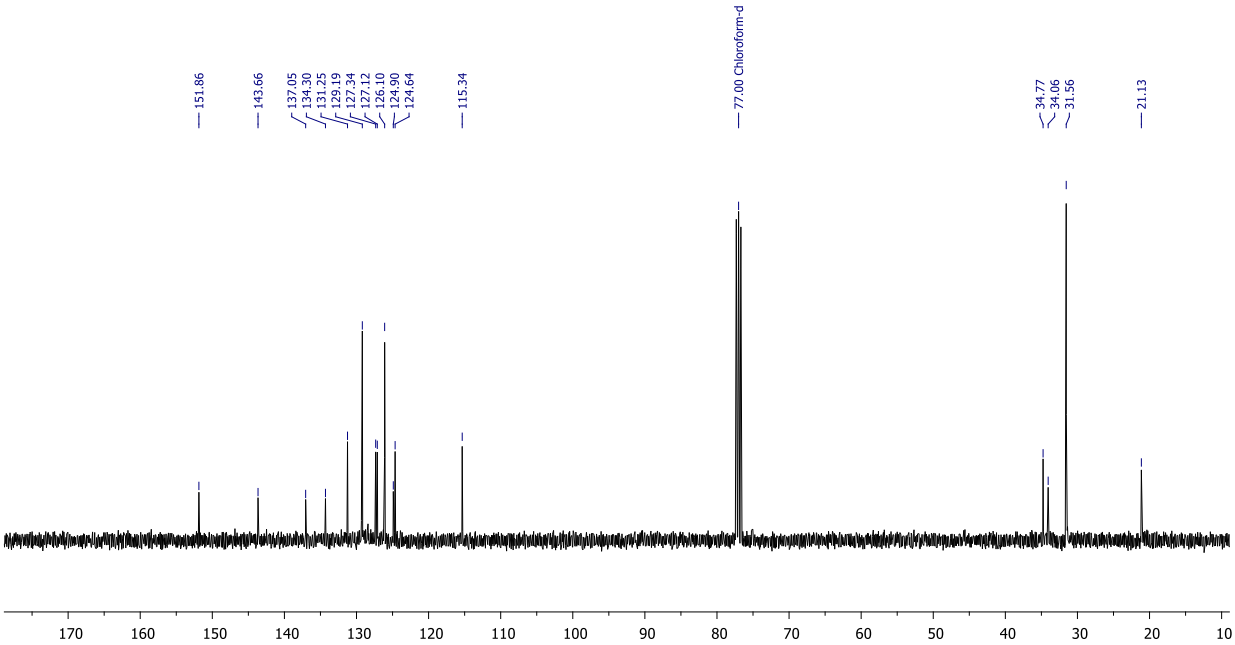
^{1}H NMR (400 MHz) spectrum of **3jg** in $CDCl_3$



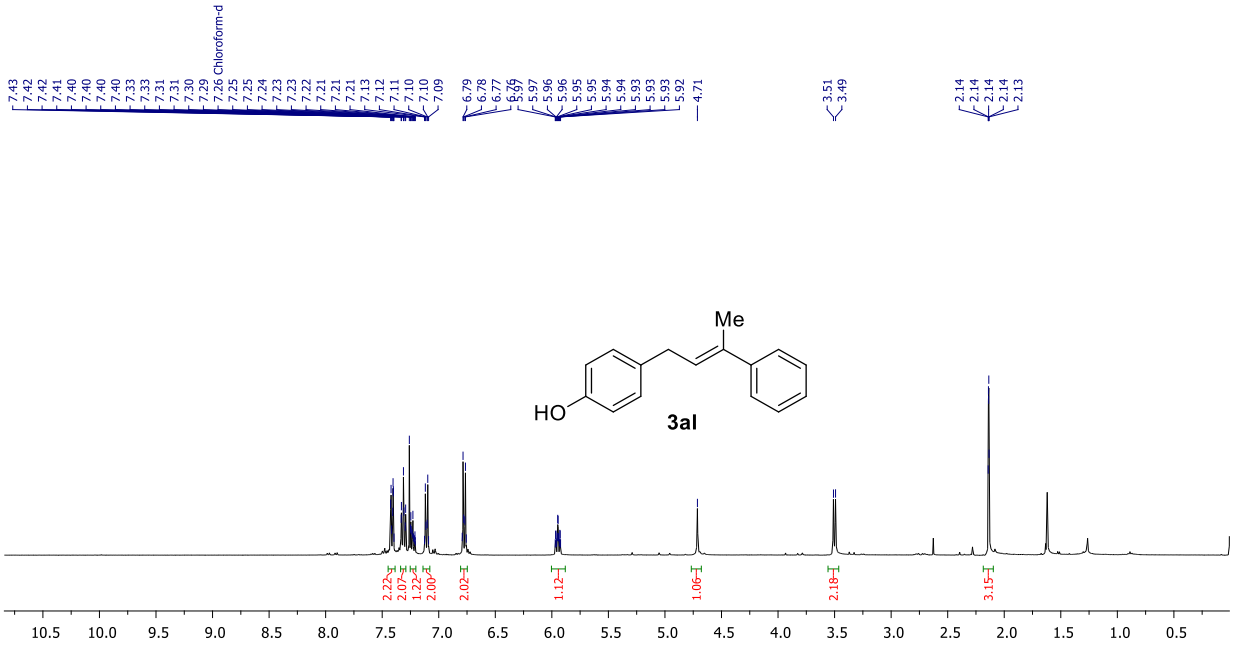
^{13}C NMR (100 MHz) spectrum of **3jg** in $CDCl_3$



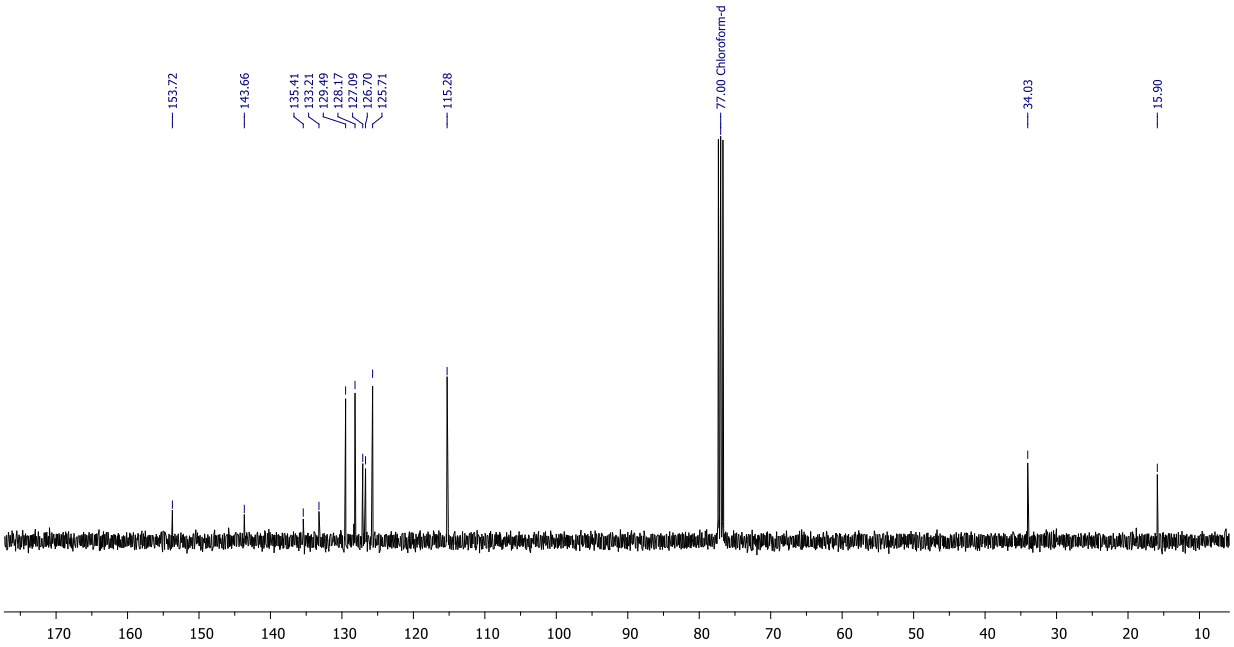
^{1}H NMR (400 MHz) spectrum of **3kb** in $CDCl_3$



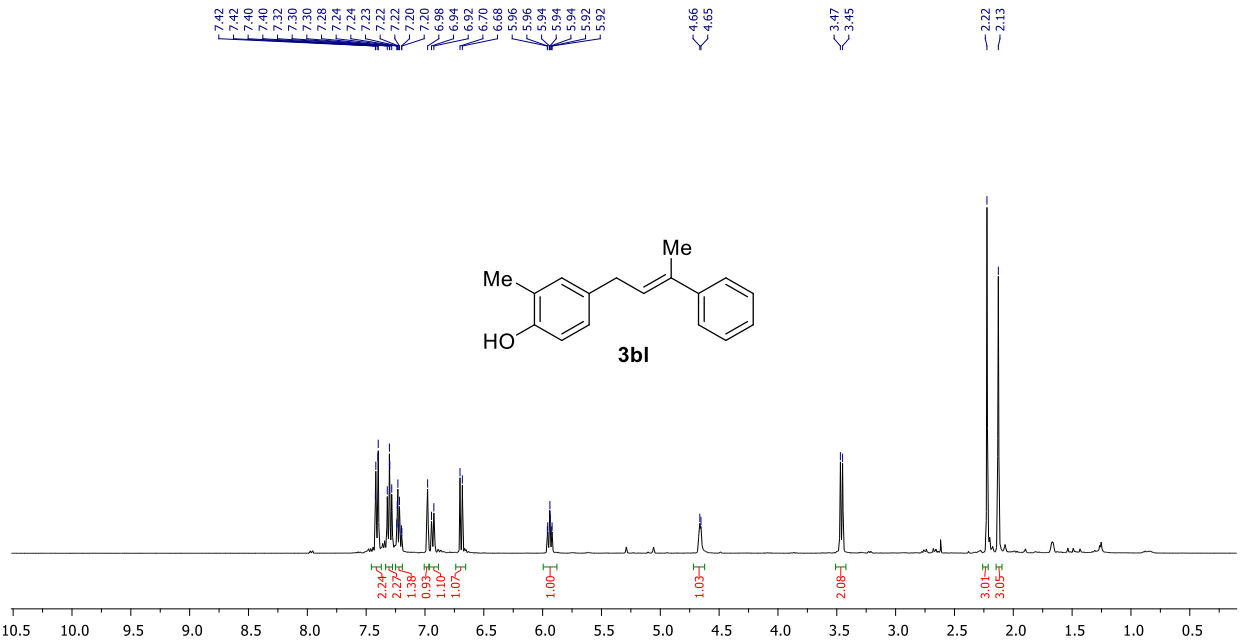
^{13}C NMR (100 MHz) spectrum of **3kb** in $CDCl_3$



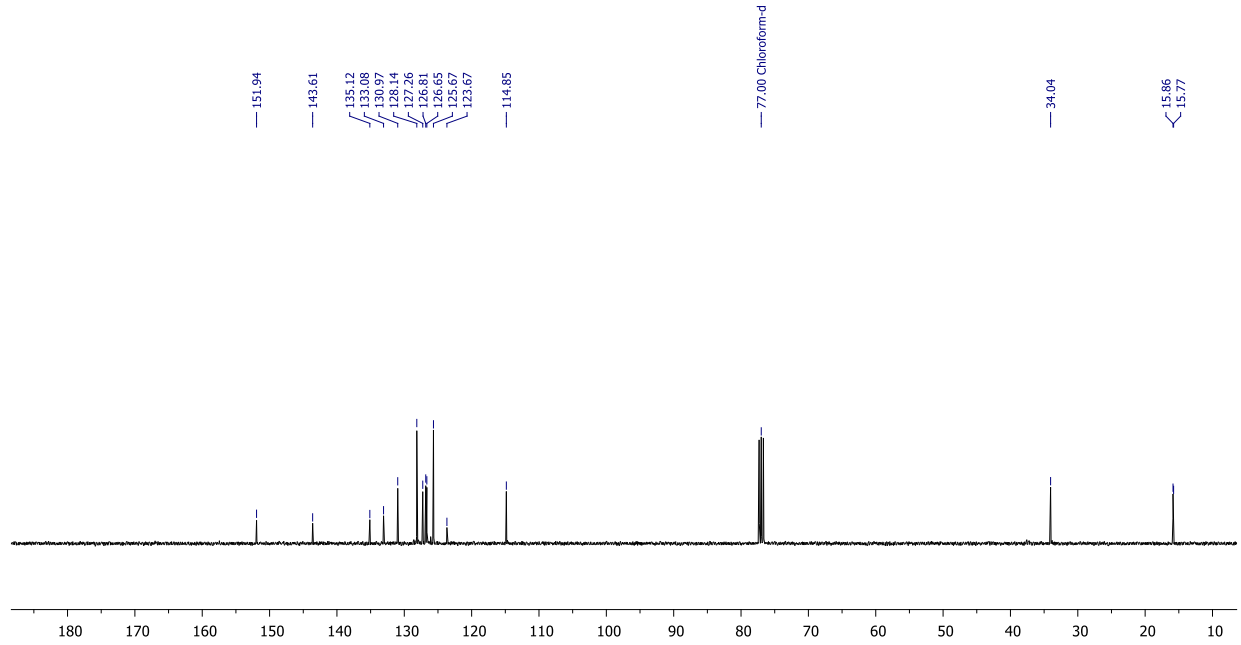
^{1}H NMR (400 MHz) spectrum of **3al** in $CDCl_3$



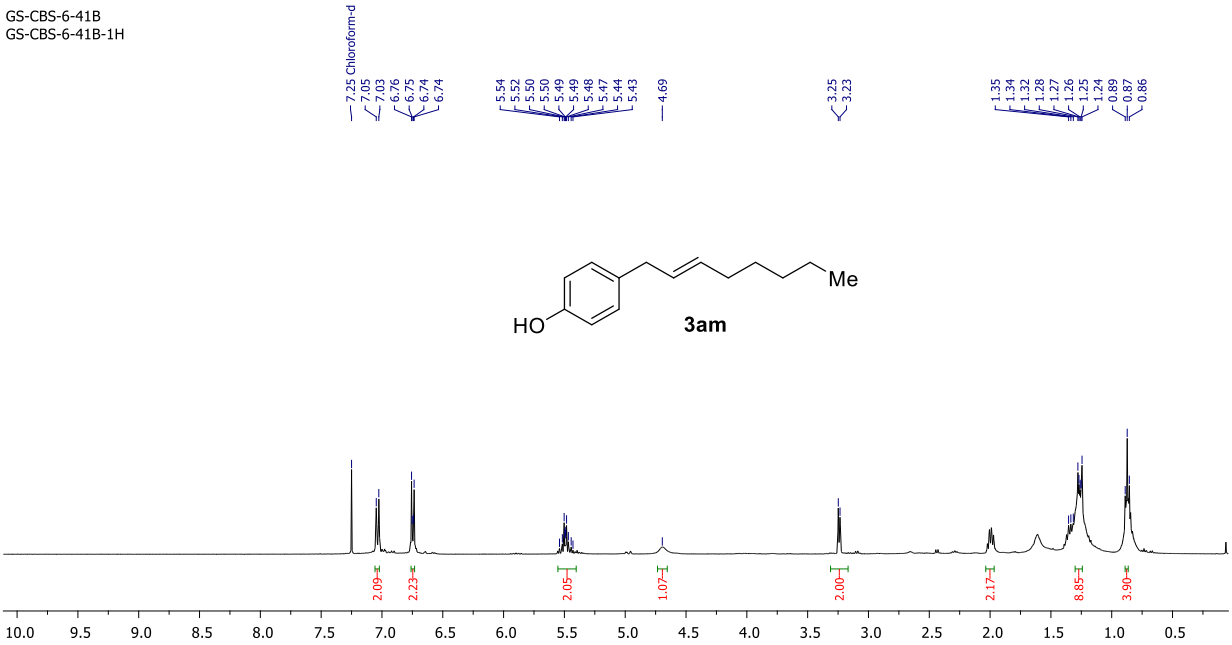
^{13}C NMR (100 MHz) spectrum of **3al** in $CDCl_3$



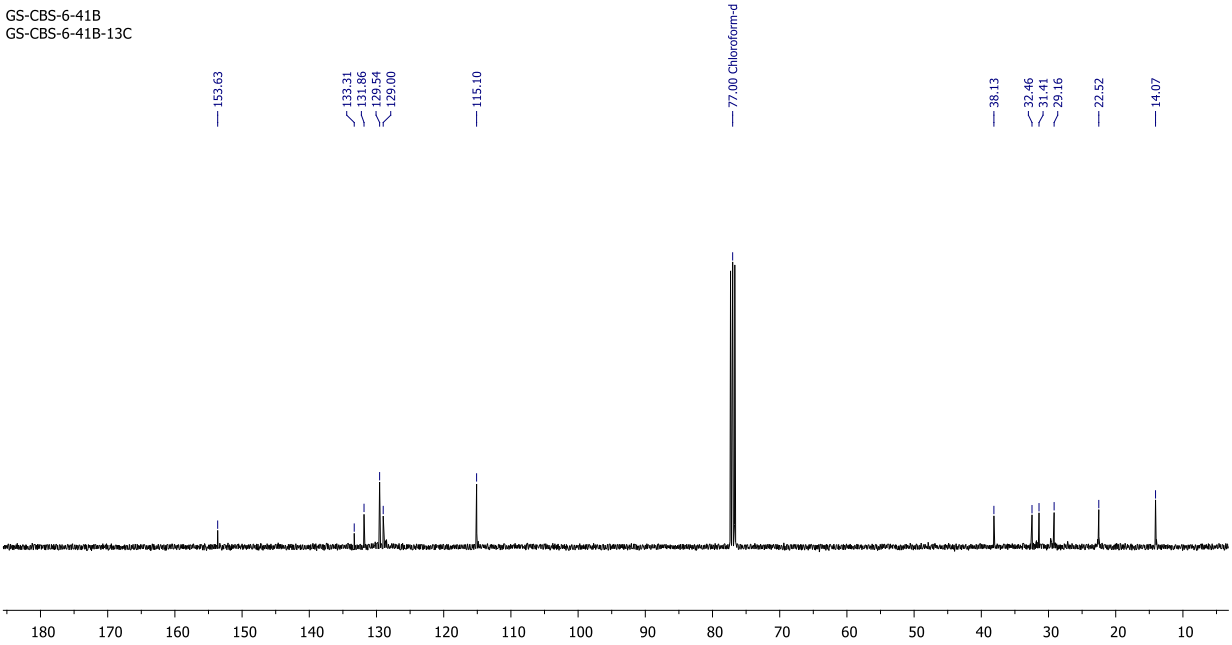
^{1}H NMR (400 MHz) spectrum of **3bl** in $CDCl_3$.



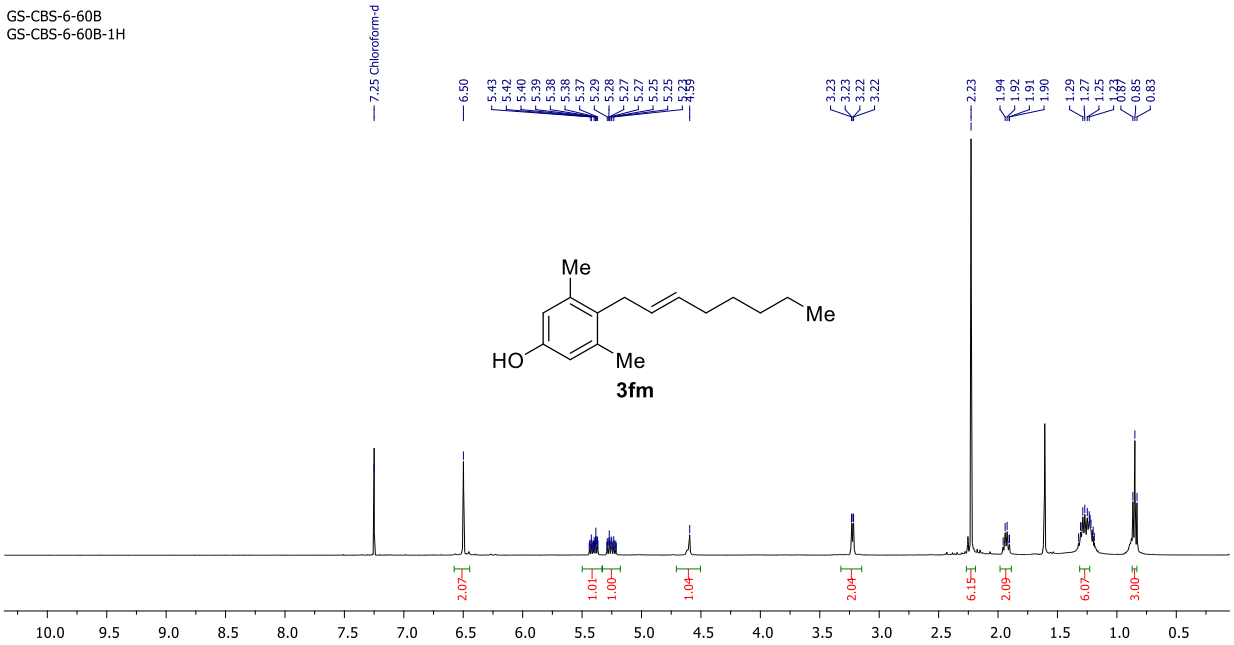
^{13}C NMR (100 MHz) spectrum of **3bl** in $CDCl_3$



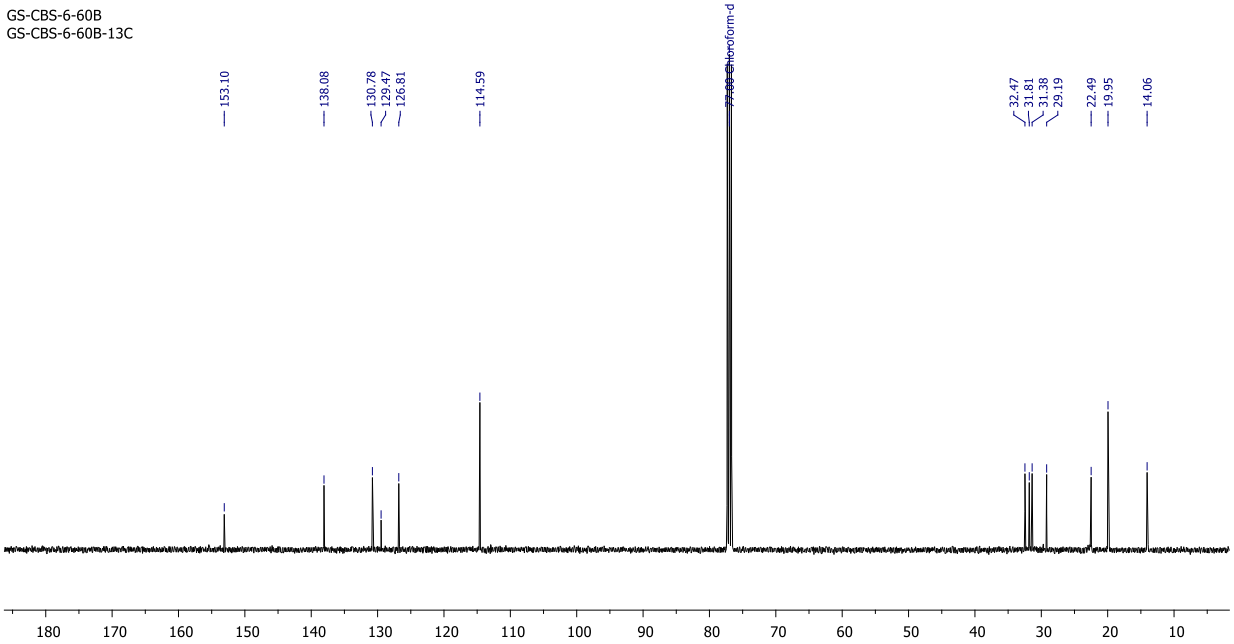
^{1}H NMR (400 MHz) spectra of product **3am** in $CDCl_3$.



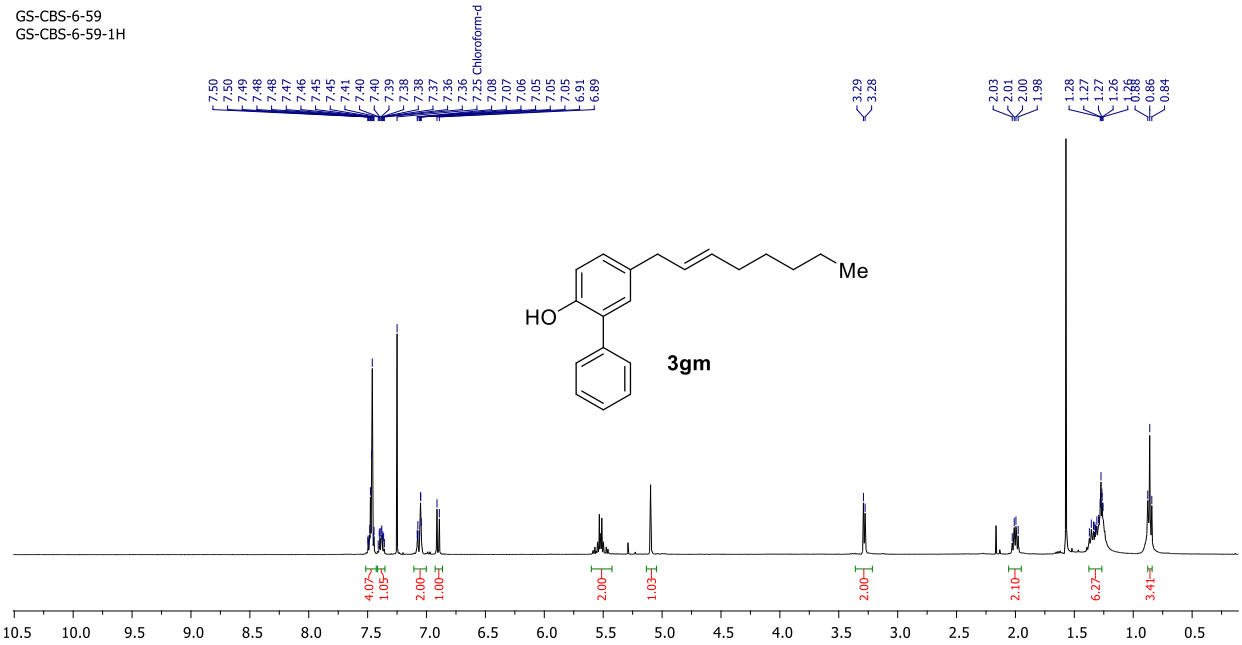
^{13}C NMR (100 MHz) spectra of product **3am** in $CDCl_3$.



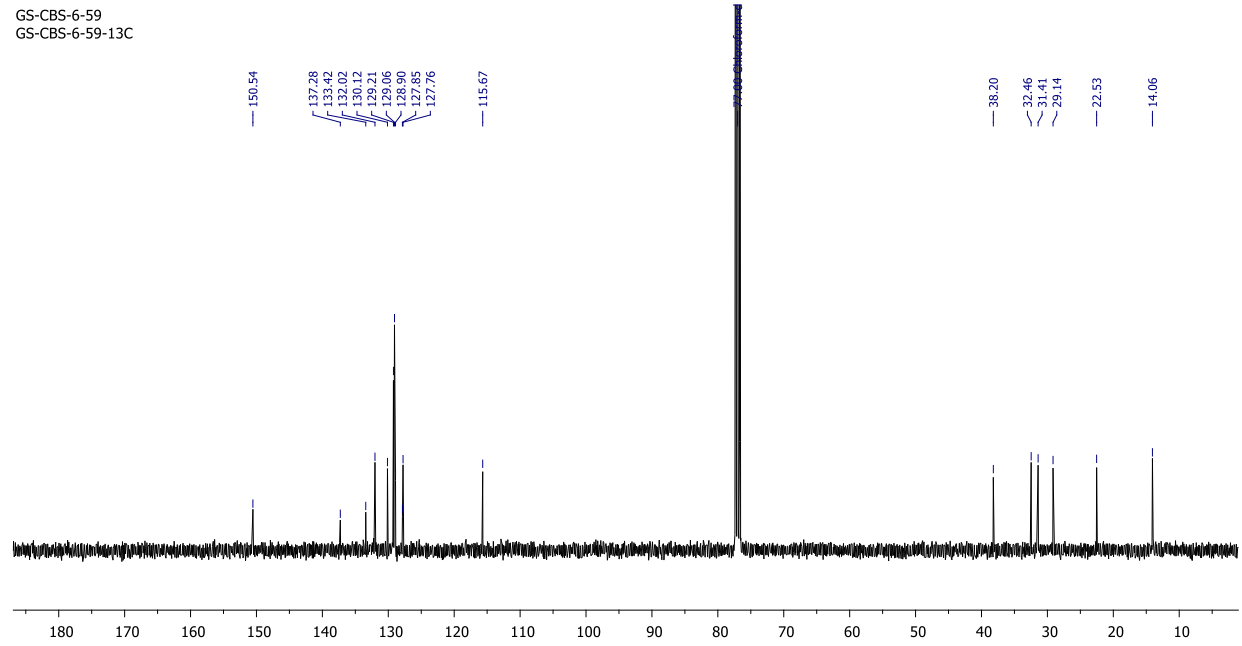
^{1}H NMR (400 MHz) spectra of product **3fm** in $CDCl_3$.



^{13}C NMR (100 MHz) spectra of product **3fm** in $CDCl_3$.



^{1}H NMR (400 MHz) spectra of product **3gm** in $CDCl_3$.



^{13}C NMR (100 MHz) spectra of product **3gm** in $CDCl_3$.

mo_GSCBS5_265_2_0ma

Table 1 Crystal data and structure refinement for mo_GSCBS5_265_2_0ma.

Identification code	mo_GSCBS5_265_2_0ma
Empirical formula	$C_{34}H_{36}O_2$
Formula weight	476.63
Temperature/K	273.15
Crystal system	triclinic
Space group	P-1
a/Å	4.787(7)
b/Å	13.87(2)
c/Å	21.66(2)
α/°	72.77(5)
β/°	90
γ/°	90
Volume/Å^3	1373(3)
Z	2
ρ_{calc}g/cm^3	1.153
μ/mm^{-1}	0.070
F(000)	512.0
Crystal size/mm^3	0.24 × 0.21 × 0.12
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.938 to 53.72
Index ranges	$-6 \leq h \leq 5$, $-17 \leq k \leq 12$, $-23 \leq l \leq 26$
Reflections collected	7877
Independent reflections	5234 [R_{int} = 0.2286, R_{sigma} = 0.8537]
Data/restraints/parameters	5234/0/332
Goodness-of-fit on F^2	0.838
Final R indexes [I>=2σ (I)]	R_1 = 0.1342, wR_2 = 0.2736
Final R indexes [all data]	R_1 = 0.5151, wR_2 = 0.5038
Largest diff. peak/hole / e Å^{-3}	0.20/-0.25

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for mo_GSCBS5_265_2_0ma. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O2	1777(17)	7526(8)	5321(4)	76(3)
O1	6755(17)	7474(7)	4677(4)	72(3)
C1	5850(30)	7072(11)	4208(6)	57(4)
C18	860(30)	7931(12)	5804(7)	57(4)
C20	1070(30)	7937(13)	6857(7)	75(5)
C3	6040(30)	7049(11)	3126(6)	67(4)
C4	4100(30)	6266(11)	3266(7)	68(4)
C2	6970(20)	7458(12)	3605(6)	65(4)
C23	-960(30)	8728(13)	5672(7)	67(5)
C5	3090(20)	5868(11)	3868(7)	64(4)
C6	4000(20)	6264(11)	4355(6)	62(4)
C19	2040(30)	7533(12)	6389(7)	64(4)
C24	4120(20)	6696(11)	6563(6)	64(4)
C22	-1890(30)	9140(12)	6129(8)	64(4)
C7	9070(20)	8308(11)	3438(6)	69(4)
C26	5450(30)	5766(11)	7686(7)	71(5)
C9	10380(30)	9285(13)	2314(9)	83(5)
C25	3720(30)	5904(12)	7225(8)	81(5)
C21	-880(30)	8717(14)	6751(8)	84(5)
C8	8690(30)	9067(12)	2795(9)	83(5)
C17	990(20)	5010(10)	4051(6)	76(5)
C10	10160(40)	10039(17)	1670(10)	89(6)
C34	-3990(30)	9997(12)	5975(7)	90(5)
C27	5140(30)	4989(13)	8298(7)	74(5)
C32	3470(40)	4133(17)	8377(9)	107(6)
C28	6370(40)	5052(17)	8838(10)	108(7)
C11	11530(40)	9930(20)	1158(13)	120(7)
C15	8380(50)	10860(20)	1644(9)	119(8)
C12	11110(40)	10730(20)	554(11)	128(8)
C29	6160(50)	4330(20)	9426(12)	152(10)
C30	4430(60)	3500(20)	9478(11)	153(11)
C13	9320(60)	11510(20)	564(13)	150(9)
C14	7870(50)	11580(20)	1087(14)	147(9)
C31	2980(40)	3408(19)	8935(13)	154(9)
C33	8300(30)	5919(16)	8846(8)	138(8)
C16	13360(40)	9094(16)	1162(8)	127(7)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for mo_GSCBS5_265_2_0ma. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O2	64(7)	108(9)	54(6)	-18(6)	1(5)	-5(6)
O1	65(7)	97(8)	59(6)	-33(6)	15(5)	-23(5)
C1	56(9)	90(13)	30(8)	-27(8)	1(7)	2(8)
C18	46(9)	68(12)	59(11)	-23(9)	11(8)	-12(8)
C20	45(10)	95(15)	71(12)	-2(10)	5(8)	-17(9)
C3	79(11)	72(12)	41(9)	-4(8)	8(7)	2(9)
C4	64(10)	66(12)	74(11)	-22(9)	17(8)	-20(8)
C2	45(9)	115(14)	40(9)	-31(9)	4(7)	7(9)
C23	32(9)	90(14)	69(11)	-9(10)	-9(8)	-11(9)
C5	40(8)	85(12)	62(10)	-12(9)	-1(7)	-12(8)
C6	40(8)	87(13)	57(9)	-21(9)	18(7)	-22(8)
C19	59(10)	85(13)	52(10)	-25(9)	2(8)	7(9)
C24	39(8)	87(13)	74(11)	-35(9)	6(7)	-7(8)
C22	37(9)	76(13)	82(12)	-30(10)	17(9)	-17(8)
C7	49(9)	90(13)	64(10)	-16(9)	9(7)	-12(8)
C26	86(12)	76(13)	50(10)	-18(9)	15(8)	-20(9)
C9	74(12)	75(14)	103(15)	-29(12)	-5(10)	-1(9)
C25	59(11)	103(15)	88(12)	-37(11)	15(9)	-17(9)
C21	53(11)	122(17)	84(13)	-44(11)	-6(9)	-19(10)
C8	73(12)	66(13)	109(15)	-25(11)	-9(10)	-21(9)
C17	46(9)	74(12)	110(12)	-29(9)	21(8)	-10(8)
C10	70(13)	87(18)	102(17)	-16(14)	11(12)	-14(11)
C34	64(11)	113(16)	96(12)	-37(11)	11(8)	-8(10)
C27	104(13)	67(14)	57(11)	-29(10)	-10(10)	14(10)
C32	120(16)	91(17)	99(16)	-13(13)	30(11)	9(13)
C28	106(15)	130(20)	69(14)	0(14)	-17(11)	29(13)
C11	89(16)	160(20)	110(20)	-41(19)	20(14)	-2(14)
C15	140(20)	130(20)	69(14)	-7(15)	-34(13)	-23(16)
C12	117(19)	160(30)	104(19)	-34(19)	8(13)	-23(16)
C29	180(30)	160(30)	110(20)	-30(20)	-50(17)	32(19)
C30	180(30)	170(30)	64(16)	29(17)	4(16)	100(20)
C13	170(30)	120(20)	140(30)	-2(19)	20(19)	-28(18)
C14	130(20)	180(30)	120(20)	-40(20)	7(18)	-5(16)
C31	150(20)	150(20)	130(20)	18(19)	22(17)	-2(15)
C33	109(15)	180(20)	142(18)	-68(16)	-28(13)	-8(15)
C16	113(15)	170(20)	111(15)	-62(15)	55(11)	-29(15)

Table 4 Bond Lengths for mo_GSCBS5_265_2_0ma.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O2	C18	1.398(14)	C7	C8	1.489(17)
O1	C1	1.368(12)	C26	C25	1.268(14)
C1	C2	1.366(15)	C26	C27	1.446(19)
C1	C6	1.390(16)	C9	C8	1.279(18)
C18	C23	1.367(17)	C9	C10	1.48(2)
C18	C19	1.347(16)	C10	C11	1.34(2)
C20	C19	1.378(17)	C10	C15	1.41(3)
C20	C21	1.398(19)	C27	C32	1.40(2)
C3	C4	1.389(16)	C27	C28	1.336(19)
C3	C2	1.395(16)	C32	C31	1.34(2)
C4	C5	1.348(15)	C28	C29	1.37(3)
C2	C7	1.512(17)	C28	C33	1.52(2)
C23	C22	1.357(18)	C11	C12	1.46(3)
C5	C6	1.395(16)	C11	C16	1.45(3)
C5	C17	1.517(17)	C15	C14	1.34(2)
C19	C24	1.488(18)	C12	C13	1.39(3)
C24	C25	1.538(18)	C29	C30	1.39(3)
C22	C21	1.388(17)	C30	C31	1.40(3)
C22	C34	1.516(19)	C13	C14	1.35(3)

Table 5 Bond Angles for mo_GSCBS5_265_2_0ma.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	C1	C6	120.6(12)	C25	C26	C27	123.5(15)
C2	C1	O1	117.1(13)	C8	C9	C10	130.8(18)
C2	C1	C6	122.1(12)	C26	C25	C24	123.7(14)
C23	C18	O2	121.5(14)	C22	C21	C20	118.5(16)
C19	C18	O2	115.3(15)	C9	C8	C7	128.0(17)
C19	C18	C23	123.1(14)	C11	C10	C9	122(2)
C19	C20	C21	124.6(15)	C11	C10	C15	124(2)
C4	C3	C2	120.9(13)	C15	C10	C9	115(2)
C5	C4	C3	121.5(13)	C32	C27	C26	123.2(15)
C1	C2	C3	117.1(13)	C28	C27	C26	122.1(18)
C1	C2	C7	123.1(12)	C28	C27	C32	114.7(18)
C3	C2	C7	119.8(13)	C31	C32	C27	126(2)
C22	C23	C18	123.1(14)	C27	C28	C29	124(2)
C4	C5	C6	118.6(13)	C27	C28	C33	122.3(19)
C4	C5	C17	123.8(13)	C29	C28	C33	113(2)
C6	C5	C17	117.6(13)	C10	C11	C12	116(2)

Table 5 Bond Angles for mo_GSCBS5_265_2_0ma.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	C6	C5	119.7(12)	C10	C11	C16	125(3)
C18	C19	C20	114.0(15)	C16	C11	C12	119(3)
C18	C19	C24	126.2(15)	C14	C15	C10	122(2)
C20	C19	C24	119.7(14)	C13	C12	C11	118(2)
C19	C24	C25	116.3(12)	C28	C29	C30	119(2)
C23	C22	C21	116.4(16)	C29	C30	C31	120(2)
C23	C22	C34	122.1(15)	C14	C13	C12	125(3)
C21	C22	C34	121.5(17)	C15	C14	C13	116(3)
C8	C7	C2	115.3(11)	C32	C31	C30	116(2)

Table 6 Torsion Angles for mo_GSCBS5_265_2_0ma.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O2	C18	C23	C22	180.0(12)	C26	C27	C28	C33	−3(3)
O2	C18	C19	C20	−179.3(12)	C9	C10	C11	C12	−179.9(15)
O2	C18	C19	C24	−2(2)	C9	C10	C11	C16	0(3)
O1	C1	C2	C3	−179.8(12)	C9	C10	C15	C14	−177.2(16)
O1	C1	C2	C7	−1.3(19)	C25	C26	C27	C32	21(3)
O1	C1	C6	C5	179.7(12)	C25	C26	C27	C28	−157.2(17)
C1	C2	C7	C8	−140.3(14)	C21	C20	C19	C18	−2(2)
C18	C23	C22	C21	1(2)	C21	C20	C19	C24	−179.5(13)
C18	C23	C22	C34	−177.6(12)	C8	C9	C10	C11	−155.2(19)
C18	C19	C24	C25	−138.8(14)	C8	C9	C10	C15	24(3)
C20	C19	C24	C25	38.1(19)	C17	C5	C6	C1	−178.0(12)
C3	C4	C5	C6	1(2)	C10	C9	C8	C7	−178.6(15)
C3	C4	C5	C17	−179.7(13)	C10	C11	C12	C13	−1(3)
C3	C2	C7	C8	38(2)	C10	C15	C14	C13	−4(3)
C4	C3	C2	C1	−2(2)	C34	C22	C21	C20	−179.3(13)
C4	C3	C2	C7	180.0(13)	C27	C26	C25	C24	−177.0(14)

Table 6 Torsion Angles for mo_GSCBS5_265_2_0ma.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C4	C5	C6	C1	2(2)	C27	C32	C31	C30	-4(3)
C2	C1	C6	C5	-4(2)	C27	C28	C29	C30	-4(4)
C2	C3	C4	C5	-1(2)	C32	C27	C28	C29	3(3)
C2	C7	C8	C9	−117.5(17)	C32	C27	C28	C33	178.9(16)
C23	C18	C19	C20	6(2)	C28	C27	C32	C31	1(3)
C23	C18	C19	C24	−177.4(14)	C28	C29	C30	C31	1(4)
C23	C22	C21	C20	2(2)	C11	C10	C15	C14	2(3)
C6	C1	C2	C3	4.0(19)	C11	C12	C13	C14	-1(4)
C6	C1	C2	C7	−177.6(13)	C15	C10	C11	C12	1(3)
C19	C18	C23	C22	-5(2)	C15	C10	C11	C16	−179.3(19)
C19	C20	C21	C22	-2(2)	C12	C13	C14	C15	4(4)
C19	C24	C25	C26	−114.9(16)	C29	C30	C31	C32	2(4)
C26	C27	C32	C31	−176.7(19)	C33	C28	C29	C30	179(2)
C26	C27	C28	C29	−178.9(19)	C16	C11	C12	C13	179(2)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for mo_GSCBS5_265_2_0ma.

Atom	*x*	*y*	*z*	U(eq)
H2	582.98	7616.77	5037.94	115
H1	5648.58	7330.38	4979.73	107
H20	1771.94	7672.02	7274.15	90
H3	6715.27	7303.44	2706.47	80
H4	3497.16	6012.01	2937.32	81
H23	-1577.68	8999.75	5249.04	81
H6	3362.77	5988.87	4776.88	74
H24A	5963.71	6987.77	6551.41	77
H24B	4063.2	6351.43	6232.82	77
H7A	8984.89	8653.86	3767.29	83
H7B	10928.03	8019.75	3455.3	83
H26	7003.54	6186.66	7628.9	85
H9	12009.39	8904.89	2380.89	100
H25	2127.12	5503.47	7290.71	98

Table 7 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for mo_GSCBS5_265_2_0ma.

Atom	*x*	*y*	*z*	U(eq)
H21	-1494.14	8948.08	7089.52	100
H8	7027.72	9428.45	2734.3	99
H17A	-846.93	5278.95	4068.37	114
H17B	1462.94	4556.98	4466.61	114
H17C	1025.6	4652.31	3733.64	114
H34A	-3835.55	10379.84	5527.6	135
H34B	-3624.9	10427.76	6240.17	135
H34C	-5846.06	9725.6	6059.5	135
H32	2606.69	4058.82	8008.49	128
H15	7530.62	10897.42	2021.91	143
H12	12020.91	10724.18	175.98	153
H29	7164.89	4383.24	9781.07	182
H30	4224.86	3005.61	9873.43	184
H13	9083.69	12031.62	180.87	179
H14	6606.85	12099.85	1062.65	176
H31	1757.8	2877.56	8960.1	185
H33A	9126.93	6194.08	8425.9	206
H33B	9751.49	5674.73	9159.91	206
H33C	7255.89	6434.78	8955.74	206
H16A	14908.92	9082.76	1443.94	190
H16B	14027.71	9175.64	731.58	190
H16C	12339.03	8471.8	1311.45	190

Experimental

Single crystals of $C_{34}H_{36}O_2$ **[mo_GSCBS5_265_2_0ma]** were **[DCM +Hexane]**. A suitable crystal was selected and **[IN ACRYOLOOP WITH MINERAL OIL]** on a **Bruker APEX-II CCD** diffractometer. The crystal was kept at 273.15 K during data collection. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [mo_GSCBS5_265_2_0ma]

Crystal Data for $C_{34}H_{36}O_2$ (M =476.63 g/mol): triclinic, space group P-1 (no. 2), a = 4.787(7) Å, b = 13.87(2) Å, c = 21.66(2) Å, α = 72.77(5)°, β = 90°, γ = 90°, V = 1373(3) Å^3, Z = 2, T = 273.15 K, μ(MoKα) = 0.070 mm^{-1}, *Dcalc* = 1.153 g/cm^3, 7877 reflections measured (3.938° ≤ 2Θ ≤ 53.72°), 5234 unique (R_{int} = 0.2286, R_{sigma} = 0.8537) which were used in all calculations. The final R_1 was 0.1342 (I > 2σ(I)) and wR_2 was 0.5038 (all data).

mo_C19H22O4_1_0m (1)

Table 1 Crystal data and structure refinement for mo_C19H22O4_1_0m (1).

Identification code	mo_C19H22O4_1_0m (1)
Empirical formula	$C_{19}H_{22}O_4$
Formula weight	314.36
Temperature/K	298
Crystal system	triclinic
Space group	P-1
a/Å	8.4709(3)
b/Å	9.9830(3)
c/Å	11.1522(4)
α/°	72.4800(10)
β/°	75.8640(10)
γ/°	69.7340(10)
Volume/Å^3	833.28(5)
Z	2
ρ_{calc}g/cm^3	1.253
μ/mm^{-1}	0.087
F(000)	336.0
Crystal size/mm^3	0.09 × 0.08 × 0.06
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.878 to 54.278
Index ranges	-10 ≤ h ≤ 10, -12 ≤ k ≤ 12, -14 ≤ l ≤ 14
Reflections collected	19020
Independent reflections	3672 [R_{int} = 0.0474, R_{sigma} = 0.0344]
Data/restraints/parameters	3672/0/213
Goodness-of-fit on F^2	1.039
Final R indexes [I>=2σ (I)]	R_1 = 0.0454, wR_2 = 0.1116
Final R indexes [all data]	R_1 = 0.0734, wR_2 = 0.1253
Largest diff. peak/hole / e Å^{-3}	0.19/-0.17

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for mo_C19H22O4_1_0m (1). U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O001	41.4(15)	6822.6(12)	1330.4(10)	54.2(3)

O002	8813.6(14)	6225.8(13)	8386.9(12)	57.6(3)
O003	2585.9(15)	7892.9(13)	1110.4(10)	56.7(3)
O004	-2093.5(16)	6401.5(14)	3517.8(12)	64.1(4)
C005	1635(2)	7679.7(16)	2293.3(14)	43.2(4)
C006	1928(2)	7942.3(16)	3356.2(14)	43.2(4)
C007	873.5(19)	7674.0(16)	4520.1(14)	43.1(4)
C008	283(2)	7141.1(16)	2383.9(14)	44.2(4)
C009	4452(2)	6630.3(16)	8018.9(14)	43.5(4)
C00A	2069(2)	8648.5(17)	5813.4(15)	48.9(4)
C00B	6674(2)	8300.3(17)	7535.0(15)	50.1(4)
C00C	3916(2)	8140.7(16)	7451.3(13)	44.0(4)
C00D	7198(2)	6815.2(16)	8089.6(14)	43.6(4)
C00E	6086(2)	5996.6(16)	8328.9(14)	43.8(4)
C00F	1111(2)	7931.3(18)	5685.3(15)	49.1(4)
C00G	5055(2)	8942.4(17)	7226.3(15)	50.2(4)
C00H	-776(2)	6883.6(16)	3538.9(15)	45.8(4)
C00I	-465(2)	7128.9(17)	4602.1(14)	47.0(4)
C00J	2171(2)	8879.2(18)	7063.3(16)	53.6(4)
C00K	3291(2)	5688.8(19)	8308.8(18)	64.3(5)
C00L	3988(2)	8428(2)	944.0(18)	65.1(5)
C00M	-1066(3)	8020(2)	569.6(19)	76.6(6)
C00N	-3274(2)	6202(2)	4657(2)	70.9(5)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for mo_C19H22O4_1_0m (1). The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O001	65.4(8)	54.7(6)	50.0(7)	-21.2(5)	-14.4(5)	-15.7(6)
O002	49.4(7)	55.4(7)	72.0(8)	-17.3(6)	-14.0(6)	-15.6(6)
O003	65.7(8)	69.1(8)	41.7(6)	-18.7(5)	4.3(5)	-31.5(6)
O004	62.4(8)	81.8(9)	63.3(8)	-23.4(6)	-3.0(6)	-39.0(7)
C005	49.0(9)	40.3(7)	37.8(8)	-10.2(6)	-3.2(6)	-11.7(7)
C006	45.3(9)	42.5(8)	43.6(8)	-10.8(6)	-7.7(7)	-14.2(7)
C007	45.0(9)	42.4(8)	39.5(8)	-9.5(6)	-9.9(6)	-7.9(7)
C008	51.0(9)	41.5(8)	42.9(8)	-14.8(6)	-11.3(7)	-10.4(7)
C009	50.8(9)	46.6(8)	37.5(8)	-11.9(6)	-9.4(7)	-16.3(7)
C00A	54.4(10)	47.1(8)	42.7(9)	-8.6(7)	-13.7(7)	-9.7(8)
C00B	56.4(10)	46.6(8)	51.5(9)	-16.7(7)	-1.1(8)	-21.4(8)
C00C	53.9(10)	44.6(8)	33.9(8)	-14.2(6)	-10.4(7)	-8.5(7)
C00D	45.9(9)	48.8(8)	38.8(8)	-15.8(7)	-5.4(6)	-13.3(7)
C00E	53.3(10)	39.7(7)	39.1(8)	-8.8(6)	-9.8(7)	-13.3(7)

C00F	47.5(9)	57.6(9)	38.8(8)	-10.5(7)	-6.8(7)	-11.9(8)
C00G	63.9(11)	39.1(8)	46.1(9)	-10.5(7)	-8.6(8)	-13.0(8)
C00H	45.5(9)	43.2(8)	50.4(9)	-12.5(7)	-6.8(7)	-14.2(7)
C00I	47.7(9)	51.4(9)	38.9(8)	-9.4(7)	-2.6(7)	-15.0(7)
C00J	59.5(11)	51.0(9)	50.3(9)	-19.3(7)	-18.1(8)	-3.8(8)
C00K	68.8(12)	56.0(10)	72.7(12)	-1.7(9)	-26.5(10)	-25.3(9)
C00L	70.2(12)	74.1(12)	56.3(11)	-20.7(9)	11.9(9)	-38.0(10)
C00M	86.4(15)	82.1(13)	65.4(12)	-22.4(10)	-32.9(11)	-11.4(12)
C00N	57.2(12)	78.5(13)	79.6(14)	-16.7(11)	3.3(10)	-34.3(10)

Table 4 Bond Lengths for mo_C19H22O4_1_0m (1).

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O001	C008	1.3823(17)	C008	C00H	1.388(2)
O001	C00M	1.425(2)	C009	C00C	1.404(2)
O002	C00D	1.3697(19)	C009	C00E	1.389(2)
O003	C005	1.3656(18)	C009	C00K	1.502(2)
O003	C00L	1.415(2)	C00A	C00F	1.308(2)
O004	C00H	1.3669(19)	C00A	C00J	1.506(2)
O004	C00N	1.421(2)	C00B	C00D	1.380(2)
C005	C006	1.383(2)	C00B	C00G	1.378(2)
C005	C008	1.396(2)	C00C	C00G	1.389(2)
C006	C007	1.395(2)	C00C	C00J	1.510(2)
C007	C00F	1.470(2)	C00D	C00E	1.380(2)
C007	C00I	1.391(2)	C00H	C00I	1.381(2)

Table 5 Bond Angles for mo_C19H22O4_1_0m (1).

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C008	O001	C00M	113.89(12)	C00F	C00A	C00J	124.27(16)
C005	O003	C00L	118.04(12)	C00G	C00B	C00D	119.65(15)
C00H	O004	C00N	117.69(13)	C009	C00C	C00J	121.52(14)
O003	C005	C006	125.31(14)	C00G	C00C	C009	118.05(14)
O003	C005	C008	114.67(13)	C00G	C00C	C00J	120.42(14)
C006	C005	C008	120.01(14)	O002	C00D	C00B	117.64(14)
C005	C006	C007	120.18(14)	O002	C00D	C00E	123.05(14)
C006	C007	C00F	122.98(14)	C00B	C00D	C00E	119.31(15)
C00I	C007	C006	119.36(13)	C00D	C00E	C009	121.59(14)
C00I	C007	C00F	117.66(13)	C00A	C00F	C007	129.03(15)
O001	C008	C005	119.66(13)	C00B	C00G	C00C	122.13(14)

O001	C008	C00H	120.39(14)	O004	C00H	C008	115.37(13)
C00H	C008	C005	119.92(13)	O004	C00H	C00I	124.78(14)
C00C	C009	C00K	121.38(14)	C00I	C00H	C008	119.85(14)
C00E	C009	C00C	119.28(14)	C00H	C00I	C007	120.66(14)
C00E	C009	C00K	119.34(14)	C00A	C00J	C00C	113.03(13)

Table 6 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for mo_C19H22O4_1_0m (1).

Atom	*x*	*y*	*z*	U(eq)
H002	9061.78	5326.08	8569.36	86
H006	2831.42	8298.76	3294.79	52
H00A	2729.43	9041.62	5085.33	59
H00B	7409.43	8864.92	7370.49	60
H00E	6438.96	4996.7	8706.74	53
H00F	495.33	7528.01	6436.26	59
H00G	4713.58	9944.85	6855.29	60
H00I	-1155.96	6927.68	5380.37	56
H00C	1342.42	8498.03	7721.33	64
H00D	1871.92	9925.53	6998.8	64
H00H	2219.25	6140.37	8771.28	96
H00J	3803.17	4735.14	8813.24	96
H00K	3109.85	5589.48	7527.09	96
H00L	4583.23	8468.92	88.84	98
H00M	4745	7783.62	1532.2	98
H00N	3585.28	9395.75	1097.95	98
H00O	-1228.69	7706.87	-113.74	115
H00P	-569.86	8814.01	225.09	115
H00Q	-2145.47	8344.73	1084.66	115
H00R	-4158.27	5904.99	4506.97	106
H00S	-3766.75	7109.5	4922.55	106
H00T	-2695.83	5456.87	5311.69	106

mo_C19H22O4_1_0m (1)

Table 1 Crystal data and structure refinement for mo_C19H22O4_1_0m (1).

Identification code	mo_C19H22O4_1_0m (1)
Empirical formula	$C_{19}H_{22}O_4$
Formula weight	314.36
Temperature/K	298

Crystal system	triclinic
Space group	P-1
a/Å	8.4709(3)
b/Å	9.9830(3)
c/Å	11.1522(4)
α/°	72.4800(10)
β/°	75.8640(10)
γ/°	69.7340(10)
Volume/Å^3	833.28(5)
Z	2
ρ_{calc}g/cm^3	1.253
μ/mm^{-1}	0.087
F(000)	336.0
Crystal size/mm^3	0.09 × 0.08 × 0.06
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.878 to 54.278
Index ranges	-10 ≤ h ≤ 10, -12 ≤ k ≤ 12, -14 ≤ l ≤ 14
Reflections collected	19020
Independent reflections	3672 [R_{int} = 0.0474, R_{sigma} = 0.0344]
Data/restraints/parameters	3672/0/213
Goodness-of-fit on F^2	1.039
Final R indexes [I>=2σ (I)]	R_1 = 0.0454, wR_2 = 0.1116
Final R indexes [all data]	R_1 = 0.0734, wR_2 = 0.1253
Largest diff. peak/hole / e Å^{-3}	0.19/-0.17

exp_9978

Table 1 Crystal data and structure refinement for exp_9978.

Identification code	exp_9978
Empirical formula	$C_{17}H_{17}BrO$
Formula weight	317.21
Temperature/K	293(2)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	8.2775(7)
b/Å	5.2464(5)
c/Å	17.4065(11)
α/°	90
β/°	92.906(7)
γ/°	90
Volume/Å^3	754.94(11)
Z	2
ρ_{calc}g/cm^3	1.395
μ/mm^{-1}	2.712
F(000)	324.0
Crystal size/mm^3	0.3 × 0.2 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.686 to 57.918
Index ranges	$-11 \leq h \leq 7$, $-6 \leq k \leq 3$, $-18 \leq l \leq 23$
Reflections collected	3212
Independent reflections	2481 [R_{int} = 0.0217, R_{sigma} = 0.0619]
Data/restraints/parameters	2481/1/174
Goodness-of-fit on F^2	1.055
Final R indexes [I>=2σ (I)]	R_1 = 0.0543, wR_2 = 0.1280
Final R indexes [all data]	R_1 = 0.1214, wR_2 = 0.1726
Largest diff. peak/hole / e Å^{-3}	0.29/-0.23
Flack parameter	0.14(2)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_9978. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Br1	3094.8(17)	10319(4)	6271.3(7)	163.0(8)
O1	436(6)	2671(12)	324(3)	81.4(16)
C6	2413(9)	3738(14)	1314(4)	61.0(19)
C1	1961(9)	2312(17)	664(4)	64(2)
C2	3010(9)	580(20)	377(4)	80(2)
C9	1261(9)	5600(20)	1635(4)	79(2)
C12	2136(11)	8360(20)	3699(5)	87(3)
C10	1453(11)	6135(19)	2486(6)	89(3)
C15	2682(11)	9480(20)	5227(5)	84(3)
C16	1752(12)	7450(20)	5018(6)	96(3)
C17	1481(12)	6910(20)	4238(7)	102(3)
C13	3075(12)	10390(20)	3923(6)	102(3)
C14	3332(12)	10950(20)	4694(6)	101(3)
C11	1938(12)	7980(20)	2850(7)	107(3)
C3	4525(10)	220(20)	729(5)	93(3)
C5	3942(9)	3361(18)	1656(5)	80(2)
C7	6759(16)	1590(40)	1743(9)	171(7)
C4	5009(10)	1630(20)	1366(6)	106(4)
C8	7220(30)	-440(50)	1966(15)	271(13)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for exp_9978. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br1	158.5(12)	232.7(18)	93.5(8)	-54.5(10)	-36.4(7)	46.1(12)
O1	67(3)	105(5)	69(3)	-2(3)	-23(3)	6(3)
C6	68(5)	60(5)	55(4)	11(4)	4(4)	4(4)
C1	58(4)	80(5)	54(4)	15(4)	-4(3)	-6(4)
C2	65(5)	101(6)	72(5)	-9(6)	-8(4)	-3(5)
C9	79(5)	87(6)	69(4)	1(5)	-12(4)	4(5)
C12	77(6)	110(8)	74(6)	-12(6)	-1(5)	20(6)
C10	96(6)	70(6)	99(6)	-8(6)	-20(5)	16(5)
C15	79(6)	88(8)	81(6)	-21(6)	-16(5)	14(5)
C16	109(7)	93(7)	87(7)	7(6)	12(6)	-1(7)
C17	95(7)	94(8)	117(9)	-23(7)	-4(7)	-11(6)
C13	106(7)	98(7)	102(7)	2(8)	1(6)	-3(7)
C14	99(7)	93(8)	111(8)	-23(7)	-9(6)	1(6)

C11	99(7)	92(8)	128(9)	-17(7)	-21(7)	14(6)
C3	77(6)	107(7)	94(6)	-14(7)	8(5)	9(6)
C5	59(5)	102(7)	75(5)	-10(5)	-22(4)	7(5)
C7	143(11)	187(16)	176(14)	-41(12)	-68(10)	89(11)
C4	67(6)	130(9)	118(8)	-14(7)	-32(5)	31(6)
C8	260(20)	163(19)	370(30)	3(19)	-190(20)	42(16)

Table 4 Bond Lengths for exp_9978.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br1	C15	1.884(8)	C12	C11	1.493(14)
O1	C1	1.380(8)	C10	C11	1.214(12)
C6	C1	1.391(10)	C15	C16	1.353(14)
C6	C9	1.495(11)	C15	C14	1.340(13)
C6	C5	1.386(10)	C16	C17	1.395(13)
C1	C2	1.368(12)	C13	C14	1.379(12)
C2	C3	1.380(10)	C3	C4	1.377(13)
C9	C10	1.509(11)	C5	C4	1.379(12)
C12	C17	1.343(14)	C7	C4	1.560(14)
C12	C13	1.365(15)	C7	C8	1.19(2)

Table 5 Bond Angles for exp_9978.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	C6	C9	120.4(7)	C14	C15	Br1	118.4(8)
C5	C6	C1	118.3(7)	C14	C15	C16	120.6(9)
C5	C6	C9	121.3(7)	C15	C16	C17	118.9(9)
O1	C1	C6	118.5(7)	C12	C17	C16	120.9(10)
C2	C1	O1	121.2(7)	C12	C13	C14	120.1(10)
C2	C1	C6	120.3(7)	C15	C14	C13	120.3(10)
C1	C2	C3	120.6(8)	C10	C11	C12	129.8(12)
C6	C9	C10	116.8(7)	C4	C3	C2	120.2(9)
C17	C12	C13	119.1(9)	C4	C5	C6	121.6(8)
C17	C12	C11	126.0(11)	C8	C7	C4	115.4(18)
C13	C12	C11	114.8(10)	C3	C4	C5	118.9(8)
C11	C10	C9	132.5(11)	C3	C4	C7	123.8(10)
C16	C15	Br1	121.0(9)	C5	C4	C7	117.0(10)

Table 6 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for exp_9978.

Atom	*x*	*y*	*z*	U(eq)
H1	347.97	4142.87	169.05	122
H2	2698.95	-360.63	-58.14	95
H9A	169.7	4987.06	1522.15	95
H9B	1366.5	7206.59	1363.44	95
H10	1140.23	4785.66	2791.76	107
H16	1300.9	6437.49	5389.17	115
H17	836.75	5527.42	4089.13	123
H13	3542.02	11402.93	3555.34	122
H14	3959.79	12353.08	4843.65	122
H11	2229.43	9367.01	2554.52	129
H3	5220.84	-988.09	534.52	111
H5	4257.97	4298.64	2091.15	95
H7A	7496.26	2208.44	1369.66	205
H7B	6807.22	2762.01	2173.21	205
H8A	7203.29	-1617.18	1543.55	407
H8B	6529.88	-1049.87	2351.69	407
H8C	8307.62	-284.4	2182.28	407

exp_9978

Table 1 Crystal data and structure refinement for exp_9978.

Identification code	exp_9978
Empirical formula	$C_{17}H_{17}BrO$
Formula weight	317.21
Temperature/K	293(2)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	8.2775(7)
b/Å	5.2464(5)
c/Å	17.4065(11)
α/°	90
β/°	92.906(7)
γ/°	90
Volume/Å^3	754.94(11)
Z	2
ρ_{calc}g/cm^3	1.395
μ/mm^{-1}	2.712
F(000)	324.0

Crystal size/mm^3	0.3 × 0.2 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.686 to 57.918
Index ranges	$-11 \leq h \leq 7$, $-6 \leq k \leq 3$, $-18 \leq l \leq 23$
Reflections collected	3212
Independent reflections	2481 [R_{int} = 0.0217, R_{sigma} = 0.0619]
Data/restraints/parameters	2481/1/174
Goodness-of-fit on F^2	1.055
Final R indexes [I>=2σ (I)]	R_1 = 0.0543, wR_2 = 0.1280
Final R indexes [all data]	R_1 = 0.1214, wR_2 = 0.1726
Largest diff. peak/hole / e $Å^{-3}$	0.29/-0.23
Flack parameter	0.14(2)