Supporting Information

Light-Induced Arylation (Alkylation) of *N*-Sulfonylhydrazones with Boronic Acids

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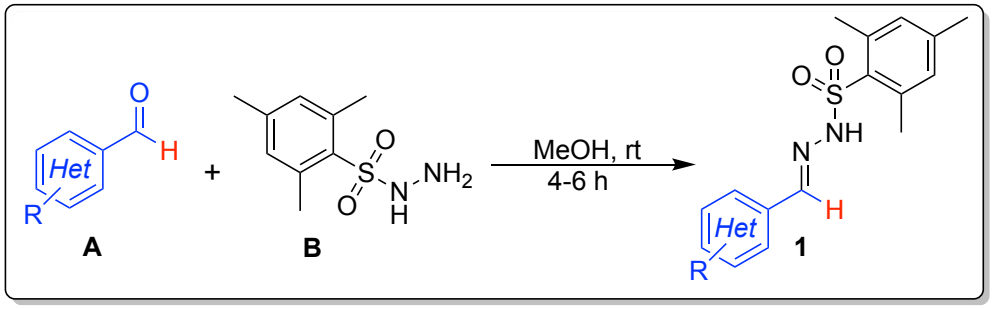
1. General Information

All chemicals were purchased from Aldrich, TCI, GLR Innovations and Avra chemicals in analytical grade and were used as supplied. All reaction were carried out under nitrogen atmosphere using oven dried reaction vials. Dry solvents were prepared by distilling over sodium metal with benzophenone, Calcium hydride and stored over molecular sieves 4 Å under N_2 atmosphere. All compounds were purified by column chromatography using silica gel (60-120 mesh). Thin layer chromatography was performed on 0.25 mm thick aluminium-baked silica gel plates purchased from Merck and visualized with ultraviolet (λ = 254 nm). ^{1}H, $^{13}C\{^{1}H\}$, ^{11}B and ^{19}F NMR spectra were recorded on JEOL ECZ500R/S1 (500, 126, 160 and 471 MHz respectively) instrument. ^{1}H signals are referenced to residual $CDCl_3$ at 7.26 ppm. ^{13}C signals are referenced to $CDCl_3$ at 77.16 ppm. IR spectra were recorded on Bruker Alpha II compact FT-IR spectrophotometer. High resolution mass spectra quadrupole time-of-flight (HRMS-QTOF) was obtained in ESI mode. The UV-Vis spectrum of compounds was recorded using a UV-Vis spectrophotometer (Shimadzu-2600). The reaction vials were purchased from Aldrich. The small Air Circulator fan Vornado 133 were purchased from amazon.com. The 40W Purple LED lamps were used Kessil LED photoreaction lighting PR160L.

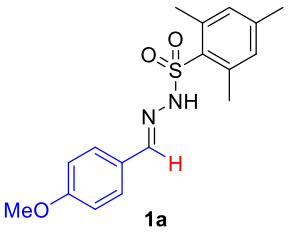
2. Synthesis of Starting Materials

General procedure 2.1: Synthesis of *N*-sulfonylhydrazones **1** derived from aldehydes



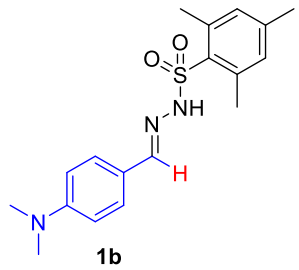
To a stirred solution of 2-mesitylenesulfonyl hydrazide **B** (1.5 mmol, 1.0 equiv) in MeOH (0.5 M), corresponding aldehydes **A** (1.5 mmol, 1.0 equiv) in MeOH (0.25 M) solution were added dropwise. The reaction mixture was stirred at room temperature until complete conversion was observed by TLC. Solvent was removed under reduced pressure by rotary evaporator and crude compound was recrystallized with hot methanol to get the desired product. Starting material is prepared from the reported literature.[1]

(E)-N'-(4-Methoxybenzylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(1a)**



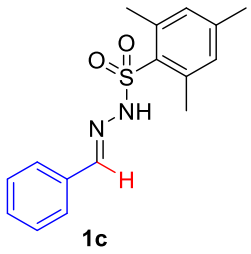
1a was prepared according to the general procedure **2.1** in 75% (359.0 mg) white solid compound. Purification done by recrystallization with hot MeOH. **TLC** (30% ethyl acetate in hexanes): R_f **= 0.30**; **MP:** 167-168 °C; **IR neat (cm^{-1}):** 3245, 2915, 1645**,** 1150**;** ^{1}**H NMR** (500 MHz, $CDCl_3$)**:** δ 8.05 (br s, 1H), 7.69 (s, 1H), 7.45 (d, *J* = 9.0 Hz, 2H), 6.98 (s, 2H), 6.85 (d, *J* = 9.1 Hz, 2H), 3.80 (s, 3H), 2.73 (s, 6H), 2.29 (s, 3H); ^{13}**C NMR** (126 MHz, $CDCl_3$)**:** δ 161.4, 146.9, 143.1, 140.3, 132.4, 132.0, 128.9, 126.1, 114.2, 55.4, 23.4, 21.1; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{17}H_{21}N_2O_3S$ 333.1267, Found 333.1267.

(E)-N'-(4-(Dimethylamino)-benzylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(1b)**



1b was prepared according to the general procedure **2.1** in 69% (357.0 mg) yellow solid compound. Purification done by recrystallization with hot MeOH. **TLC** (30% ethyl acetate in hexanes): R_f **= 0.33; MP:** 157– 158°C; **IR neat (cm^{-1}):** 3215, 2945, 1595**,** 1270; ^{1}**H NMR** (500 MHz, DMSO-d_6)**:** δ 7.60 (s, 1H), 7.30 (d, *J* = 6.81 Hz, 2H), 6.96 (s, 2H), 6.60 (d, *J* = 9.30 Hz, 2H), 2.97 (s, 6H), 2.73 (s, 6H), 2.28 (s, 3H) (The -NH proton is not visible); ^{13}**C NMR** (126 MHz, DMSO-d_6)**:** δ 190.5, 142.8, 140.2, 132.0, 128.8, 125.2, 112.1, 111.1, 40.5, 40.2, 23.4, 21.10; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{18}H_{24}N_3O_2S$ 346.1584, Found 346.1589.

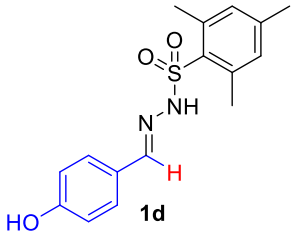
(E)-*N*′-Benzylidene-2,4,6-trimethylbenzenesulfonohydrazide **(1c)**



1c

1c was prepared according to the general procedure **2.1** in 60% (271.0 mg) white solid compound. Purification done by recrystallization with hot MeOH. **TLC** (20% ethyl acetate in hexanes): R_f **= 0.35**; **MP:** 155-156 °C; **IR neat (cm^{-1}):** 3208, 2989, 1603**,** 1364; **^{1}H NMR** (500 MHz, $CDCl_3$)**:** δ 7.76 (br s, 1H), 7.52-7.50 (m, 2H), 7.34-7.32 (m, 3H), 6.97 (s, 2H), 2.74 (s, 6H), 2.29 (s, 3H) (The -NH proton is not visible); **^{13}C NMR** (126 MHz, $CDCl_3$)**:** δ 146.7, 143.2, 140.3, 133.4, 132.3, 132.1, 130.3, 128.7, 127.3, 23.4, 21.1; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{19}N_2O_2S$ 303.1162, Found 303.1157.

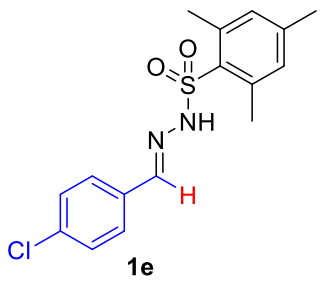
(E)-*N*′-(4-Hydroxybenzylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(1d)**



1d

1d was prepared according to the general procedure **2.1** in 72% (343.0 mg) yellow solid compound. Purification done by recrystallization with hot MeOH. **TLC** (10% methanol in DCM): R_f **= 0.30**; **MP:** 182-184°C; **IR neat (cm^{-1}):** 3224, 2934, 2319, **1**604**,** 1326; **^{1}H NMR** (500 MHz, DMSO-d_6)**:** δ 11.22 (s, 1H), 9.84 (s, 1H), 7.76 (s, 1H), 7.28 (d, J = 9 Hz, 2H), 6.97 (s, 2H), 6.70 (d, J = 8.8 Hz, 2H), 2.59 (s, 6H), 2.19 (s, 3H); **^{13}C NMR** (126 MHz, DMSO-d_6)**:** δ 159.7, 146.3, 142.6, 139.7, 133.9, 132.1, 128.7, 125.5, 116.1, 23.3, 20.9; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{19}N_2O_3S$ 319.1111, Found 303.1112.

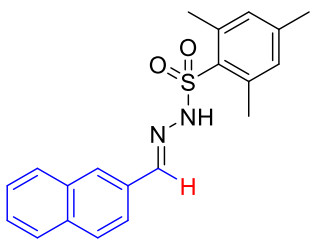
(E)-*N*′-(4-Chlorobenzylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(1e)**



1e

1e was prepared according to the general procedure **2.1** in 55% (277.0 mg) white solid compound. Purification done by recrystallization with hot MeOH. **TLC** (30% ethyl acetate in hexanes): R_f **= 0.28**; **MP:** 144-145 °C; **IR neat (cm^{-1}):** 3309, 2942, 1601**,** 1372; **^{1}H NMR** (500 MHz, $CDCl_3$)**:** δ 7.70 (s, 1H), 7.43 (d, J = 8.4 Hz, 2H), 7.29 (d, J = 8.8 Hz, 2H), 6.98 (s, 2H), 2.73 (s, 6H), 2.30 (s, 3H) (The -NH proton is not visible); **^{13}C NMR** (126 MHz, $CDCl_3$)**:** δ 191.2, 145.4, 143.42, 140.4, 136.3, 132.16, 131.8, 131.4, 131.1, 129.6, 129.0, 128.5, 23.4, 21.1; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{18}ClN_2O_2S$ 337.0772, Found 337.0777.

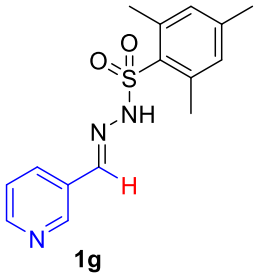
(E)-2,4,6-Trimethyl-*N*′-(naphthalen-2-ylmethylene)benzenesulfonohydrazide **(1f)**



1f

1f was prepared according to the general procedure **2.1** in 68% (359.0 mg) white solid compound. Purification done by recrystallization with hot MeOH. **TLC** (20% ethyl acetate in hexanes): R_f **= 0.35**; **MP:** 190-191 °C; **IR neat (cm^{-1}):** 3216, 2949, 1596, 1319; **^{1}H NMR** (500 MHz, $CDCl_3$)**:** (500 MHz $CDCl_3$): δ 8.0 (s, 1H), 7.82-7.79 (m, 2H), 7.76 (d, *J* = 1.75 Hz, 2H), 7.52-7.47 (m, 2H), 6.99 (s, 2H), 2.77 (s, 6H), 2.29 (s, 3H)**; ^{13}C NMR** (126 MHz $CDCl_3$): δ 146.9, 143.3, 140.4, 134.4, 132.46, 132.44, 133.0, 132.1, 131.0, 129.2, 128.7, 128.5, 128.0, 127.3, 126.8, 122.8, 23.4, 21.1; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{20}H_{21}N_2O_2S$ 353.1318, Found 353.1321.

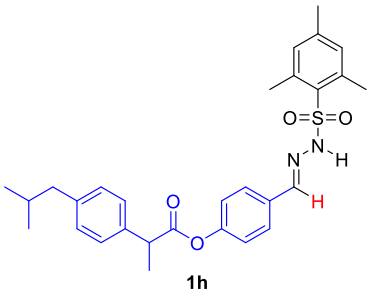
(E)-2,4,6-Trimethyl-*N'*-(pyridin-3-ylmethylene) benzenesulfonohydrazide **(1g)**



1g

1g was prepared according to the general procedure **2.1** in 68% (309.0 mg) white solid compound. Purification done by recrystallization with hot MeOH. **TLC** (10% Methanol in DCM): R_f **= 0.28**; **MP:** 188 – 189 °C; **IR neat (cm^{-1}):** 3309, 2388, 1606, 1326; **^{1}H NMR** (500 MHz, DMSO-d_6)**:** δ 11.80 (br s, 1H), 8.61 (d, *J* = 3 Hz, 1H), 8.49 (dd, *J* = 1.8, 4.9 Hz, 1H), 7.91 (s, 1H), 7.83-7.81 (m, 1H), 7.38-7.35 (m, 1H), 7.00 (s, 2H), 2.60 (s, 6H), 2.19 (s, 3H); **^{13}C NMR (**126 MHz, DMSO-d_6)**:** δ 151.0, 148.6, 142.95, 142.93, 139.7, 133.7, 133.4, 132.1, 130.2, 124.4, 23.2, 20.9; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{15}H_{18}N_3O_2S$ 304.1114, Found 304.1113.

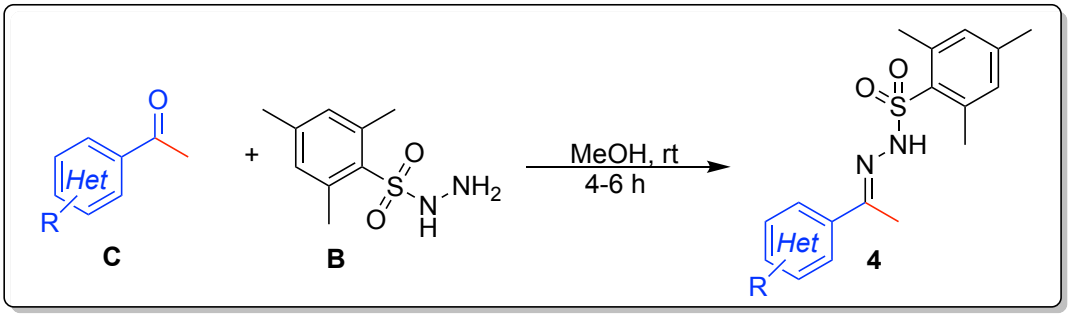
(E)-4-((2-(Mesitylsulfonyl)hydrazineylidene)methyl)phenyl 2-(4-isobutylphenyl)propanoate **(1h)**



1h

1h was prepared according to the general procedure **2.1** in 68% (516.0 mg) white solid compound. Purification done by recrystallization with hot MeOH. **TLC** (30% ethyl acetate in hexanes): R_f **= 0.32; MP:** 162-163 °C; **IR neat (cm^{-1}):** 3309, 2388, 1606, 1326; **^{1}H NMR (**500 MHz, $CDCl_3$)**:** δ 7.4 (d, *J* = 9 Hz, 2H), 7.20 (d, *J* = 8.3 Hz, 2H), 7.11 (d, *J* = 8.3 Hz, 2H), 6.96 (d, *J* = 7.3 Hz, 2H), 6.94 (s, 2H), 3.9 (q, 1H), 2.71 (s, 6H), 2.4 (d, *J* = 7.1 Hz, 2H), 2.2 (s, 3H), 1.90-1.82 (m, 1H), 1.60 (d, *J* = 6.5 Hz, 3H), 0.9 (d, *J* = 6.2 Hz, 6H) (The -NH proton is not visible); **^{13}C NMR (**126 MHz, $CDCl_3$)**:** δ 173.2, 152.3, 145.5, 143.2, 141.0, 140.3, 137.0, 132.3, 132.1, 131.0, 129.7, 128.3, 127.3, 121.9, 45.3, 45.1, 30.3, 23.4, 22.5, 21.1, 18.5; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{29}H_{35}N_2O_4S$ 507.2312, Found 507.2315.

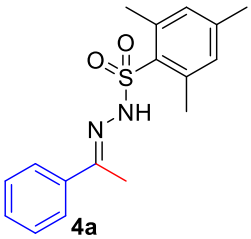
General procedure 2.2: Synthesis of *N*-sulfonylhydrazones **4** derived from ketones



To a stirred solution of 2-mesitylenesulfonyl hydrazide **B** (1.5 mmol, 1.0 equiv) in MeOH (0.5 M), corresponding ketones **C** (1.5 mmol, 1.0 equiv) in MeOH (0.25 M) solution were added dropwise. The reaction mixture was stirred at room temperature until complete conversion was observed by TLC. Solvent was removed under reduced pressure by rotary evaporator and crude compound was recrystallised with hot methanol to get the desired product. Starting material is prepared from the reported literature.[1]

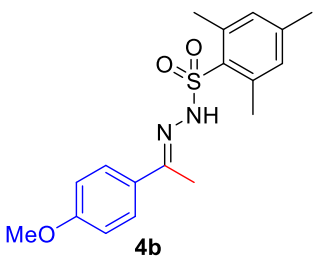
If crude product is sticky then washed two to three times with minimum amount of hexane and diethyl ether. Solvent was removed under reduced pressure by rotary evaporator and then recrystallized with hot MeOH.

(E)-2,4,6-trimethyl-*N'*-(1-phenyl-ethylidene)-benzenesulfonohydrazide **(4a)**



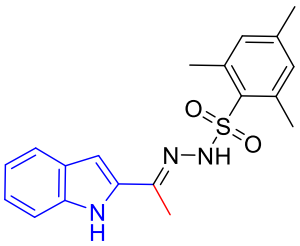
4a was prepared according to the general procedure **2.2** in 68% (321.0mg) white solid compound. Purification done by recrystallisation with hot MeOH. **TLC** (20% ethylacetate in hexanes): R_f **= 0.33**; **MP:** 110-112 °C; **IR neat (cm^{-1}):** 3325, 3130, 2940, 1603, 1358, 1140; **^{1}H NMR:** δ 7.04 (br s, 1H), 7.58-7.55 (m, 2H), 7.32-7.29 (m, 3H), 6.90 (s, 2H), 2.75 (s, 6H), 2.28(s, 3H), 2.16 (s, 3H); **^{13}C NMR** (126 MHz $CDCl_3$): δ 151.0, 143.0, 140.4, 137.4, 132.4, 131.9, 129.5, 128.3, 126.2, 23.4, 21.1, 13.0; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{13}H_{21}N_2O_2S$ 317.1323, Found 317.1323.

(*E*)-N'-(1-(4-methoxyphenyl)ethylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(4b)**



4b was prepared according to the general procedure **2.2** in 68% (352.0 mg) white solid compound. Purification done by recrystallization with hot MeOH. **TLC** (30% ethylacetate in hexanes): R_f **= 0.30; MP:** 125-127 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.50 (d, J = 8.5 Hz, 2H), 6.92 (s, 2H), 6.80 (d, J = 9.0 Hz, 2H), 3.81 (s, 3H), 2.70 (s, 6H), 2.22 (s, 3H), 2.10 (s, 3H) (The -NH proton is not visible); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 160.8, 151.1, 143.0, 140.4, 132.0, 130.7, 130.0, 127.7, 113.7, 55.4, 23.4, 21.1, 13.01; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{18}H_{22}N_2O_3S$ 347.1427, Found 347.1427.

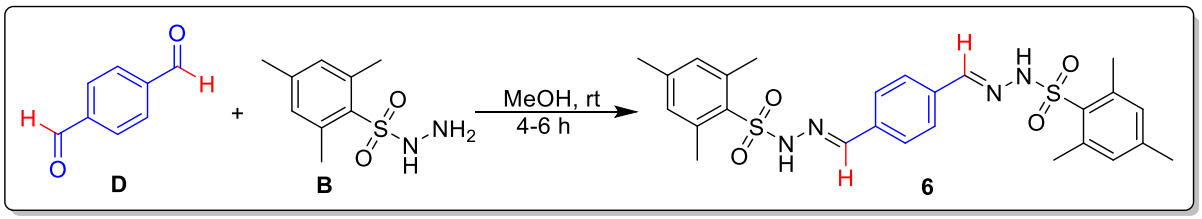
(E)-N'-(1-(1H-indol-2-yl)ethylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(4c)**



4c

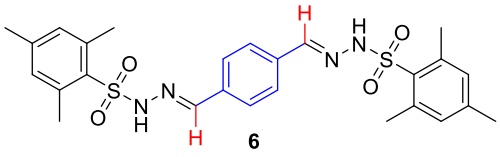
4c was prepared according to the general procedure **2.2** in 70% (533.0 mg) yellow solid compound. Purification done by recrystallization with methanol. **TLC** (10% Methanol in DCM): R_f **= 0.30; MP:** 220-221 °C; **IR neat (cm^{-1}):** 3362, 3226, 2944, 1603, 1358; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 11.36 (s, 1H), 10.14 (s, 1H), 7.74 (d, *J* = 3.0 Hz, 1H), 7.66 (d, *J* = 8.7 Hz, 1H), 7.31 (d, *J* = 8.4 Hz, 1H), 7.09-7.05 (m, 3H), 6.87 (t, *J* = 8.25 Hz, 1H), 2.66 (s, 6H), 2.26 (s, 3H), 2.22 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 153.5, 143.12, 140.2, 136.4, 132.5, 132.1, 132.0, 130.2, 129.9, 129.8, 128.4, 128.2, 127.5, 23.27, 21.15; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{19}H_{22}N_3O_2S$ 356.1427, Found 356.1429.

General procedure 2.3: Synthesis of *N*-sulfonylhydrazones **6** derived from terephthaldehdye.



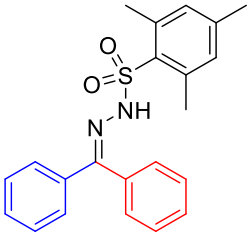
To a stirred solution of 2-mesitylenesulfonyl hydrazide **B** (1.0 mmol, 1.0 equiv) in MeOH (0.5 M), solution of corresponding terephthaldehdye **D** (0.5 mmol, 0.5 equiv) in MeOH (0.25 M) solution were added dropwise. The reaction mixture was stirred at room temperature until complete conversion was observed by TLC. Solvent was removed in under reduced pressure by rotary evaporator and crude compound was washed two times with MeOH to obtained the desired product. Starting material is prepared from the reported literature.[1]

N',N''-((*1E,1'E*)-1,4-phenylenebis(methaneylylidene))bis(2,4,6-trimethylbenzenesulfonohydrazide) **(6)**



6 was prepared according to the general procedure **2.3** in 70% (368.0 mg) white solid compound. Purification done by washing with methanol. **TLC** (10% Methanol in DCM): R_f **= 0.28; MP:** 203-204 °C; **IR neat (cm^{-1}):** 3219, 2945, 1695, 1101; **^{1}H NMR** (500 MHz DMSO-d_6): δ 11.66 (s, 2H), 7.83 (s, 2H), 7.45 (s, 4H), 7.00 (s, 4H), 2.59 (s, 12H), 2.19 (s, 6H) (The -NH proton is visible,) ; **^{13}C NMR** (126 MHz, DMSO-d_6): δ 149.9, 142.8, 139.7, 135.5, 133.8, 132.1, 127.3, 23.26, 20.9; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{26}H_{31}N_4O_4S_2$ 527.1781, Found 527.1784.

N'-(Diphenylmethylene)-2,4,6-trimethylbenzenesulfonohydrazide (**8**)



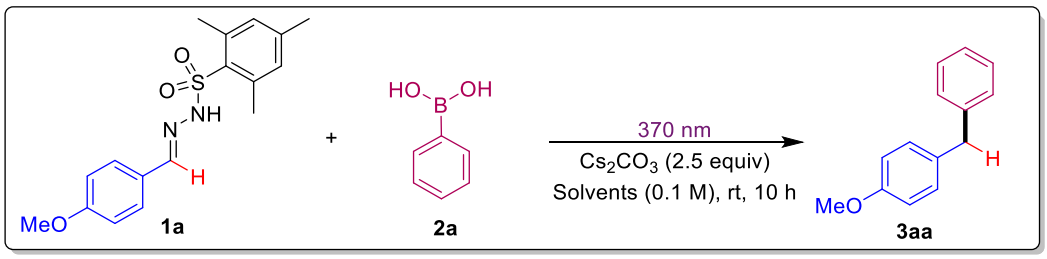
8

8 was prepared according to the general procedure **2.2** in 64% (362.8 mg) white solid compound. Purification done by recrystallization with methanol. **TLC** (20% ethyl acetate in hexanes): R_f **= 0.35; MP:** 130-131 °C; **IR neat (cm^{-1}):** 3335, 2926, 2900, 1706, 1457; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.68 (br s, 1H), 7.54-7.51 (m, 3H), 7.38-7.36 (m, 2H), 7.30-7.24 (m, 3H), 7.16-7.14 (m, 2H), 6.97 (s, 2H), 2.65 (s, 6H), 2.29 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 153.5, 143.1, 140.1, 136.5, 132.5, 132.0, 131.1, 130.2, 129.9, 129.8, 128.3, 128.2, 127.5, 23.2, 21.1; **HRMS** (ESI/Q-TOF) m/z: $[M+H]^+$ Calcd for $C_{22}H_{23}N_3O_2S$ 379.1475, Found 379.1470.

3. Reaction optimization

3.1 Optimization of diarylmethanes (3)

3.1.1 Solvent optimization.



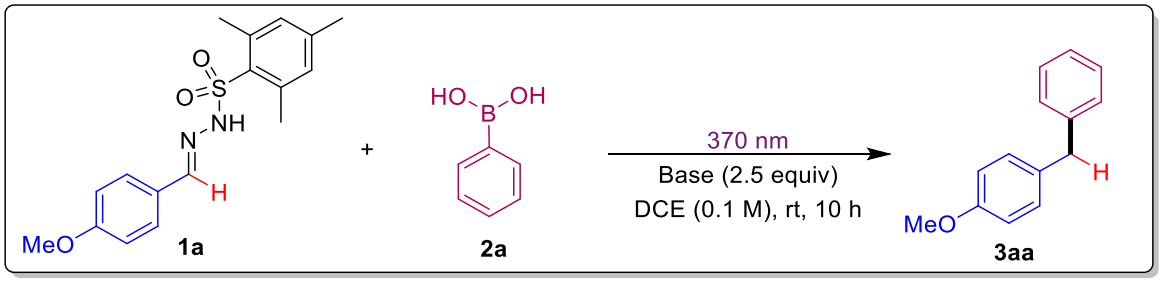
To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **1a** (33.2 mg, 0.1 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (81.5 mg, 0.25 mmol, 2.5 equiv) and arylboronic acid **2a** (18.3 mg, 0.15 mmol, 1.5 equiv) were added followed by dry solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan (we control the temperature by using the fan, temperature of the reaction reaching ~35˚C) for 10 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **3aa** was determined by using CH_2Br_2 as an internal standard.

Entry	Dry Solvents (0.1 M)	NMR Yield (**3aa**) [a]
1	DCM	63%
2	DCE	93% (90%) [b]
3	1,4-dioxane	69%

4	CH_3CN	4.0%
5	Toluene	60%
6	Mesitylene	62%
7	Trifluoro- toluene	60%
8	DMF	ND
9	THF	62%
10	Chlorobenzene	56%
11	Benzene	56%

[a]Crude yield and [b]isolated yield of **3aa**

3.1.2: Base optimization.



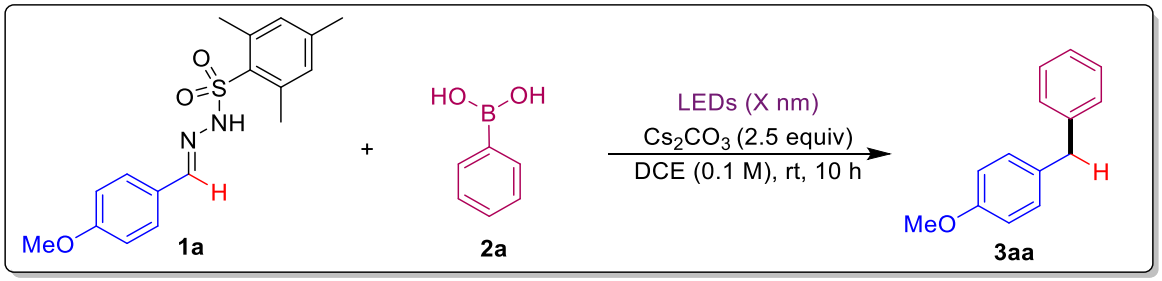
To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **1a** (33.2 mg, 0.1 mmol, 1.0 equiv) derived from aldehyde was added, then base (xx mg, 0.25 mmol, 2.5 equiv) and arylboronic acid **2a** (18.3 mg, 0.15 mmol, 1.5 equiv) were added followed by DCE solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 10 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **3aa** was determined by using CH_2Br_2 as an internal standard.

Entry	Base	NMR Yield **(3aa)** [a]
1	No base	ND
2	Na_2CO_3	ND
3	K_2CO_3	26%
4	Cs_2CO_3	93% (90%) [b]

5	K_3PO_4	80%
6	$KHCO_3$	2.0%
7	NaOH	32%
8	KOH	24%
9	NaOMe	19%
10	$NaNH_2$	45%
11	CH_3CO_2K	12%
12	NaH	15%
13	CsF	28%
14	Li^{t}BuO	6%
15	DIPEA	8%
16	DMAP	16%
17	Et_3N	24%
18	DBU	10%

[a]Crude yield and [b]isolated yield of **3aa**

3.1.3: Optimization of light source (LEDs nm)



To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **1a** (33.2 mg, 0.1 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (81.5 mg, 0.25 mmol, 2.5 equiv) and arylboronic acid **2a** (18.3 mg, 0.15 mmol, 1.5 equiv) were added followed by DCE solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of various light sources LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 10 h and we also tried the reaction under dark condition in presence of 370 nm LEDs by covering of two layers of aluminium foil upon glass vial. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through

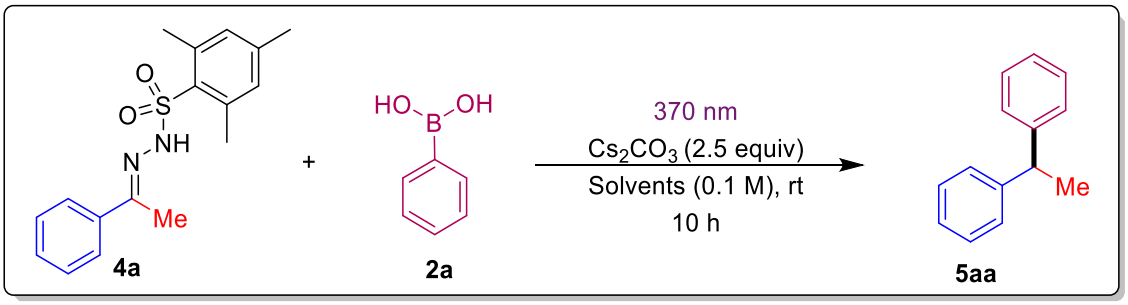
the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **3aa** was determined by using CH_2Br_2 as an internal standard.

Entry	LEDs (nm)	NMR Yield (**3aa**) [a]
1	370 nm	93% (90%) [b]
2	390 nm	72%
3	427 nm	5%
4	440 nm	5%
5	456 nm	ND
6	Dark and 40 °C	20%

[a]Crude yield and [b]isolated yield of **3aa**

3.2. Optimization of diarylalkylmethanes **(5)**

3.2.1. Optimization of solvents.



To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **4a** (31.6 mg, 0.1 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (81.5 mg, 0.25 mmol, 2.5 equiv) and arylboronic acid **2a** (18.3 mg, 0.15 mmol, 1.5 equiv) were added followed by dry solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 10 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **5aa** was determined by using CH_2Br_2 as an internal standard.

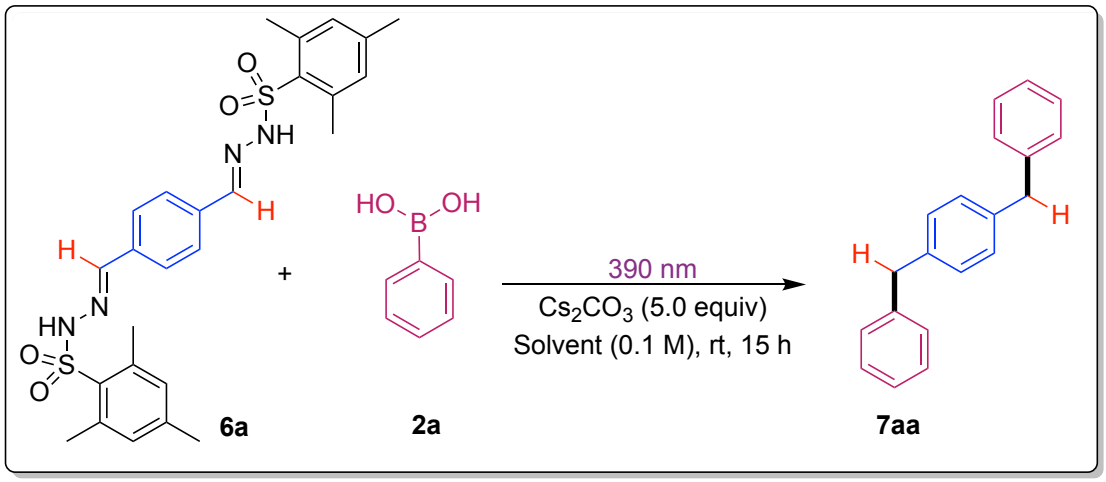
Entry	Dry Solvents (0.1 M)	NMR Yield (**5aa**) [a]
1	DCM	64% (63%) [b]
2	DCE	35%

3	1,4-dioxane	10%
4	Toluene	50%
5	Mesitylene	32%
6	THF	12%

[a]Crude yield and [b]isolated yield of **3aa**

3.3 Optimization of bis(1,4-diaryl/napthylmethyl)benzenes **(7)**.

3.3.1 Optimization of solvents.



To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **6a** (52.7 mg, 0.1 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (163.0 mg, 0.5 mmol, 5 equiv) and arylboronic acid **2a** (36.6 mg, 0.3 mmol, 3.0 equiv) were added followed by dry solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 15 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **7aa** was determined by using CH_2Br_2 as an internal standard.

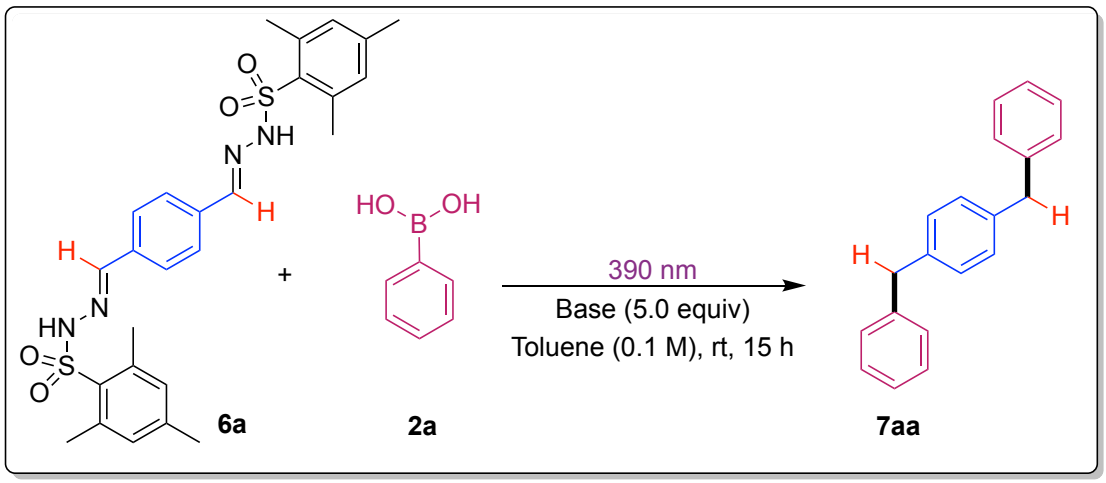
Entry	Solvents	NMR Yield **(7aa)** [a]
1	Mesitylene	ND
2	Toluene	22%
3	DCM	3.0 %

4	1,4-dioxane	ND
5	DMF	ND
6	THF	12%
7	$CHCl_3$	17%
8	Toluene: H_2O	18%

[a]Crude yield

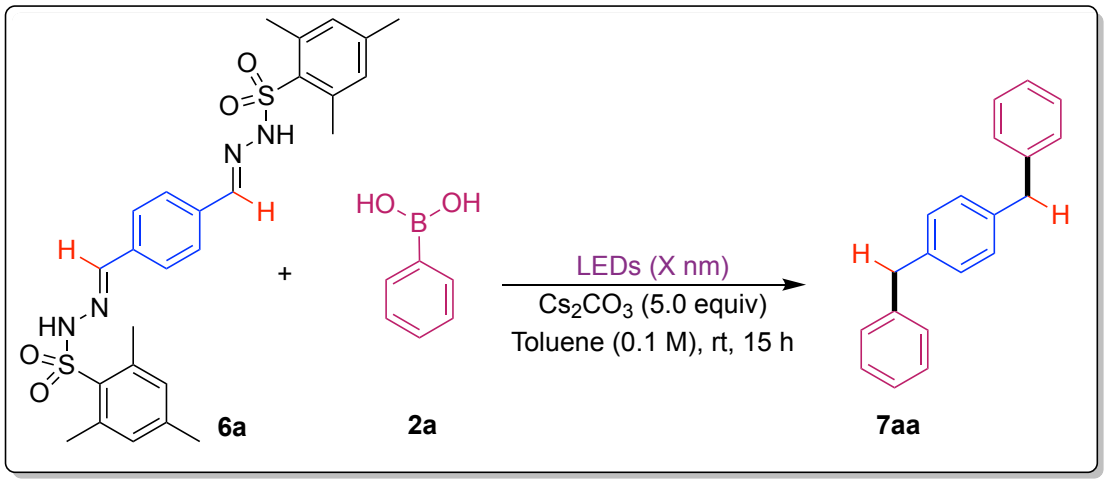
Scheme 3.3.2: Base optimization.

To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **6a** (52.7 mg, 0.1 mmol, 1.0 equiv) derived from aldehyde was added, then base (163.0 mg, 0.25 mmol, 2.5 equiv) and arylboronic acid **2a** (36.6 mg, 0.3 mmol, 3.0 equiv) were added followed by toluene solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 15 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **7aa** was determined by using CH_2Br_2 as an internal standard.



Entry	Mixture of base (1:1)	NMR Yield **(7aa)** [a]
1	Cs_2CO_3	25%
2	Cs_2CO_3: DBU	ND
3	Cs_2CO_3: H_2O	33%

Scheme 3.3.3: Optimization of light source (LEDs nm).



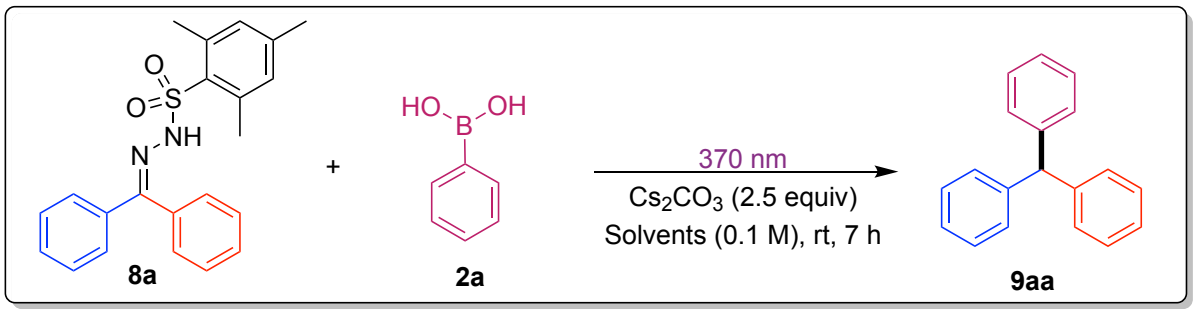
To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **6a** (52.7 mg, 0.1 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (163.0 mg, 0.5 mmol, 5 equiv) and arylboronic acid **2a** (36.6 mg, 0.3 mmol, 3.0 equiv) were added followed by toluene solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of various light sources LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 15 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **7aa** was determined by using CH_2Br_2 as an internal standard.

Entry	LEDs (nm)	NMR Yield **(7aa)** [a]
1	370 nm	30%
2	390 nm	70%
3	427 nm	ND
4	440 nm	ND
5	456 nm	ND

[a]Crude yield

3.4 Synthesis of triarylmethanes (**9**).

3.4.1: Optimization of solvents.



To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **8a** (37.8 mg, 0.1 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (81.5 mg, 0.25 mmol, 2.5 equiv) and aryl boronic acid **2a** (18.3 mg, 0.15 mmol, 1.5 equiv) were added followed by dry solvent (0.1 M, 1 mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 7 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **9aa** was determined by using CH_2Br_2 as internal standard.

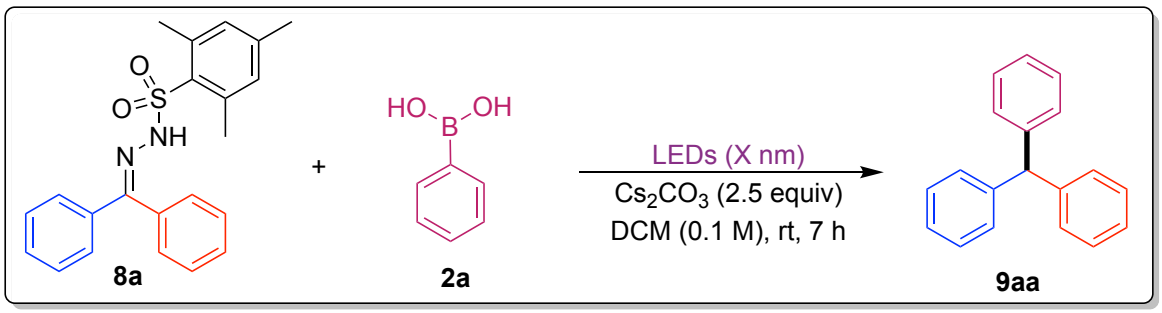
Entry	Solvents	NMR Yield **(9aa)** [a]
1	Mesitylene	15%
2	Toluene	22%
3	DCM	28%
4	DCE	20%

[a]Crude yield

3.4.2: Optimization of light source (LEDs nm).

To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **8a** (37.8 mg, 0.1 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (0.25 mmol, 2.5 equiv) and aryl boronic acid **2a** (18.3 mg, 0.15 mmol, 1.5 equiv) were added followed by dry solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of various LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 7 h.

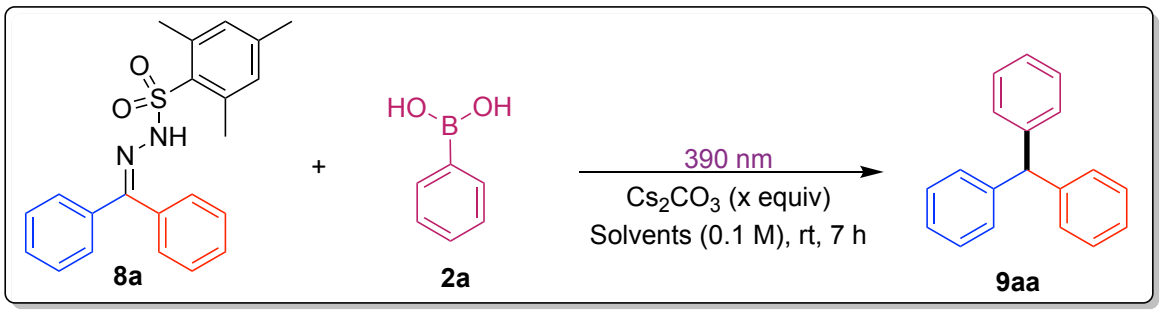
Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **9aa** was determined by using CH_2Br_2 as internal standard.



Entry	LEDs (nm)	NMR Yield (**9aa**) [a]
1	370 nm	28%
2	390 nm	35%
3	427 nm	20%
4	440 nm	ND
5	456 nm	ND

[a]Crude yield

3.4.3: Optimization of equivalent of Cs_2CO_3



To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylenesulphonylhydrazones **8a** (37.8 mg, 0.1 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (xx equiv) and arylboronic acid **2a** (18.3 mg, 0.15 mmol, 1.5 equiv) were added followed by dry solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35 °C) for 7 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl

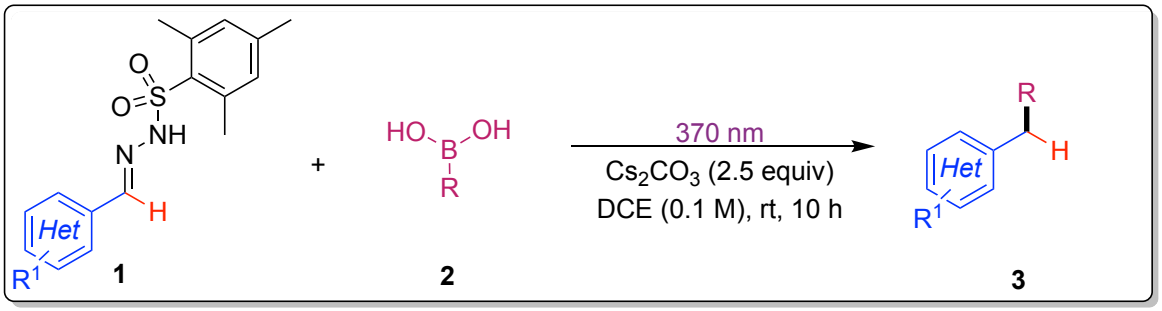
acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **9aa** was determined by using CH_2Br_2 as internal standard.

Entry	Cs_2CO_3 (equiv.)	NMR Yield (**9aa**) [a]
1	1.5	30%
2	2.0	65% (58%) [b]
3	2.5	35%

[a]Crude yield

4.0 General procedure for Visible Light-Induced Arylation (Alkylation)

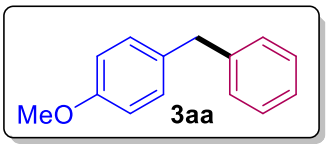
4.1 General procedure for light-induced synthesis of diarylmethanes (**3**)



General procedure A: To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **1** (0.1 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (0.25 mmol, 2.5 equiv) and aryl/alkylboronic acid **2** (0.15 mmol, 1.5 equiv) were added followed by DCE solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 10 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **3aa** was determined by using CH_2Br_2 as an internal standard.

Spectral data of Isolated compounds:

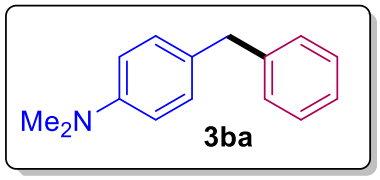
1-Benzyl-4-methoxybenzene (**3aa**)



3aa was prepared according to the general procedure **A** in 90% (35.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 20:1) as an eluent, **TLC** (1% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.45**; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.35 - 7.33 (m, 2H), 7.26 - 7.24 (m, 3H), 7.19 (d, *J* = 7.1 Hz, 2H), 6.92 (d, *J* = 7.1 Hz, 2H), 4.00 (s, 2H), 3.86 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.1, 141.7, 133.4, 130.0, 128.9, 128.6, 126.1, 114.0, 55.4, 41.0.

Spectral data matched with the reported literature.[2]

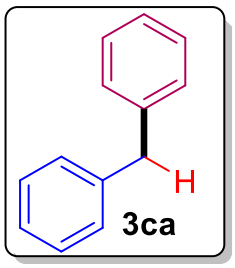
4-Benzyl-*N*,*N*-dimethylaniline (**3ba**)



3ba was prepared according to the general procedure **A** in 76% (32.0 mg) yellow oil. Purification done by column chromatography on silica gel using (hexane: EtOAc =98:2) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.4; ^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.28 (t, *J* = 7.4 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 3H), 7.08 (d, *J* = 8.5 Hz, 2H), 6.71 (d, *J* = 8.2 Hz, 2H), 3.90 (s, 2H), 2.91(s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 149.2, 142.1, 129.6, 128.9, 128.4, 126.9, 125.9, 113.1, 41.05, 41.01.

Spectral data matched with the reported literature.[2]

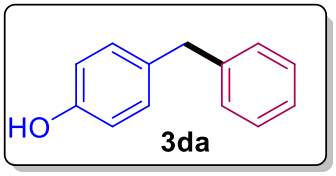
Diphenylmethane (**3ca**)



3ca was prepared according to the general procedure **A** in 73% (31.0 mg) Colorless oil. Purification done by column chromatography on silica gel using (hexanes) as the eluent, **TLC** (hexanes): $\boldsymbol{R_f}$ **= 0.6**; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.40-7.29 (m, 5H), 7.25-7.19 (m, 5H), 4.01 (s, 2H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 141.2, 129.0, 128.5, 126.1, 42.0

Spectral data matched with the reported literature.[4]

4-Benzylphenol (**3da**)

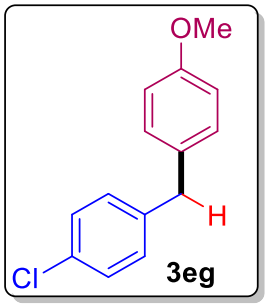


3da was prepared according to the general procedure **A** in 42% (15.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 95:5) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.35. ^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.30-

7.27(m, 2H), 7.21-7.17 (m, 3H), 7.07 (d, *J* = 8.7 Hz, 2H), 6.77 (d, *J* = 8.7 Hz, 2H), 3.92 (s, 2H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 153.8, 141.5, 133.5, 130.1, 128.8, 128.5, 126.0, 115.3, 41.1.

Spectral data matched with the reported literature.[5]

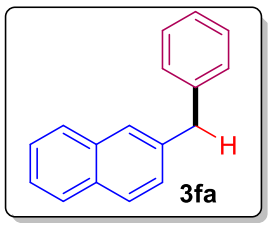
1-Chloro-4-(4-methoxybenzyl) benzene (**3eg**)



3eg was prepared according to the general procedure **A** in 75% (35.0 mg) Colorless oil. Purification done by column chromatography on silica gel using (hexanes: EtOAc = 98:2) as the eluent, **TLC** (5% ethyl acetate in hexanes): ***R*$_f$ = 0.28; ^{1}H NMR** (500 MHz, $CDCl_3$): δ7.25-7.22(m, 2H), 7.13-7.10 (m, 4H), 6.86- 6.80 (m, 2H), 3.88 (s, 2H), 3.77 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.2, 140.1, 132.7, 131.8, 130.2, 129.9, 128.6, 114.0, 55.3, 40.6.

Spectral data matched with the reported literature.[3]

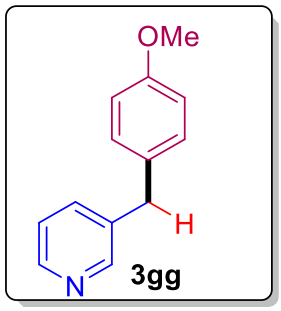
2-Benzylnaphthalene (**3fa**)



3fa was prepared according to the general procedure **A** in 78% (72.0 mg) white solid. Purification done by column chromatography on silica gel using (hexanes: EtOAc = 99:1) as the eluent, **TLC** (5% ethyl acetate in hexanes): ***R*$_f$ = 0.45**; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.83-7.77 (m, 3H), 7.66 (s, 1H), 7.50-7.42 (m, 2H), 7.36-7.30 (m, 3H), 7.27-7.20 (m, 3H), 4.17 (s, 2H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 141.0, 138.7, 133.7, 132.1, 129.1, 128.6, 128.1, 127.75, 127.73, 127.6, 127.2, 126.2, 126.0, 125.4, 42.2.1.

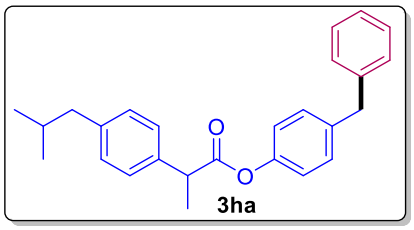
Spectral data matched with the reported literature.[6]

3-(4-Methoxybenzyl)pyridine (**3gg**)



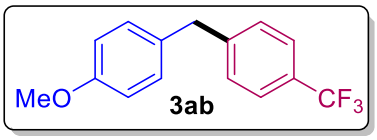
3gg was prepared according to the general procedure **A** in 42% (17.0 mg) Colorless oil. Purification done by column chromatography on silica gel using (hexanes: EtOAc = 97:3) as the eluent, **TLC** (10% ethyl acetate in hexanes): ***R*$_f$ = 0.25**; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.25-7.23 (m, 2H), 7.12-7.06 (m, 4H), 6.85-6.81 (m, 2H), 3.89 (s, 2H), 3.79 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.1, 140.1, 132.7, 131.8, 130.2, 129.9, 128.6, 114.0, 55.3, 40.4.Spectral data matched with the reported literature.[7]

4-Benzylphenyl 2-(4-isobutylphenyl)propanoate (**3ha**)



3ha was prepared according to the general procedure **A** in 90% (33.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexanes: EtOAc= 99:1) as the eluent, **TLC** (10% ethyl acetate in hexanes): $\boldsymbol{R_f}$ **= 0.55;** ^{1}**H NMR** (500 MHz, $CDCl_3$): 7.32-7.26 (m, 4H), 7.23-7.15 (m, 7H), 6.93 (d, *J* = 8.8 Hz, 2H), 3.97 (s, 2H), 3.96-3.92 (m, 1H), 2.40 (d, *J* = 7.1 Hz, 2H), 1.93-1.84 (m, 1H), 1.60 (d, *J* = 8.0 Hz, 3H), 0.9 (d, *J* = 7.5 Hz, 6H); ^{13}**C NMR** (126 MHz, $CDCl_3$): δ 173.4, 149.2, 140.9, 140.8, 138.6, 137.4, 129.8, 129.6, 129.0, 128.6, 127.3, 126.2, 45.3, 45.1, 41.3, 30.2, 22.5, 18.6. **HRMS** (ESI/Q-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{26}H_{29}O_2$ 373.2162., Found 373.2164.

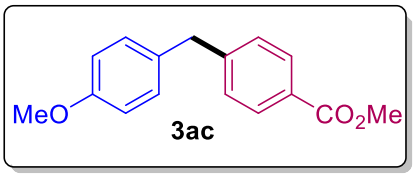
1-Methoxy-4-(4-(trifluoromethyl)benzyl) benzene (**3ab**)



3ab was prepared according to the general procedure **A** in 72% (38.0 mg) white solid. Purification done by column chromatography on silica gel using (hexane: EtOAc = 98:2) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.52.** ^{1}**H NMR** (500 MHz, $CDCl_3$): δ 7.55 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 7.10 (d, *J* = 8.5 Hz, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 3.98 (s, 2H), 3.80 (s, 3H); ^{13}**C NMR** (126 MHz, $CDCl_3$): δ 158.3, 145.8, 132.2, 130.4, 129.1, 125.5(q, 15.5 Hz), 123.3, 114.1, 55.3, 40.9; ^{19}**F NMR** (471 MHz, $CDCl_3$): δ -62.2.

Spectral data matched with the reported literature.[6]

Methyl 4-(4-methoxybenzyl) benzoate (**3ac**)

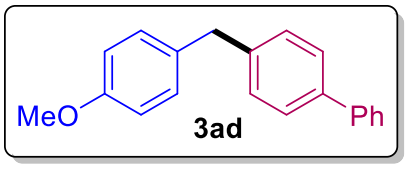


3ac was prepared according to the general procedure **A** in 71% (36.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 97:3) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.40**.

^{1}**H NMR** (500 MHz, $CDCl_3$): δ 7.96 (d, *J* = 8.5 Hz, 2H), 7.25 (d, *J* = 8.5 Hz, 2H), 7.10 (d, *J* = 8.7 Hz, 2H), 6.85 (d, *J* = 8.7 Hz, 2H), 3.97(s, 2H), 3.90 (s, 3H), 3.79 (s, 3H); ^{13}**C NMR** (126 MHz, $CDCl_3$): δ 167.7, 158.2, 147.1, 132.3, 130.4, 129.9, 128.9, 128.1, 114.1, 58.3, 52.1, 41.1 **HRMS** (ESI/Q-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{16}H_{17}O_3$ 257.1172, Found 257.1175.

Spectral data matched with the reported literature.[7]

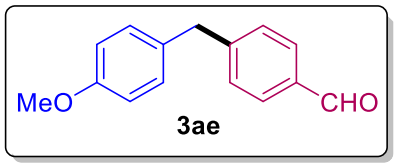
4-(4-Methoxybenzyl)-1,1'-biphenyl (**3ad**)



3ad was prepared according to the general procedure **A** in 72% (37.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexanes: EtOAc = 99:1) as an eluent, **TLC** (10% ethyl acetate in hexanes): $\boldsymbol{R_f}$ **= 0.60**. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.59-7.57 (m, 2H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.44 (td, *J* = 8.5 Hz, 1Hz, 2H), 7.34 (tt, *J* = 7.5 Hz, 1Hz, 1H), 7.27 (dt, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 9.0 Hz, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 3.98 (s, 2H), 3.80(s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.1, 141.4, 140.8, 139.0, 133.2, 130.2, 129.3, 128.8, 127.3, 127.2, 127.1, 114.0, 55.3, 40.7.

Spectral data matched with the reported literature.[7]

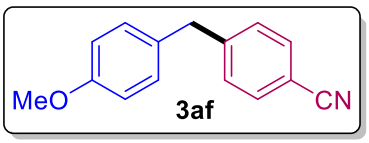
(4-Methoxybenzyl) benzaldehyde (**3ae**)



3ae was prepared according to the general procedure **A** in 45% (20.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 98:2) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.30**. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.97 (s, 1H), 7.79 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 6.84 (d, *J* = 8.3 Hz, 2H), 4.00 (s, 2H), 3.79 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 192.1, 158.3, 149.1, 134.7, 131.9, 130.1, 130.0, 129.5, 114.2, 55.4, 41.3; **HRMS** (ESI/Q-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{15}H_{15}O_2$ 227.1056, Found 227.1056.

Spectral data matched with the reported literature.[7]

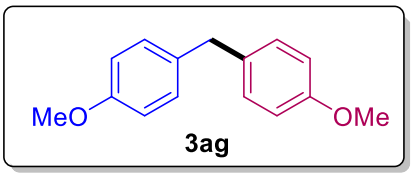
4-(4-Methoxybenzyl) benzonitrile (**3af**)



3af was prepared according to the general procedure **A** in 58% (25.8 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 97:3) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.42**; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.57 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 7.8 Hz, 2H), 7.06 (d, *J* = 7.8 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H), 3.97 (s, 2H), 3.79 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.4, 147.4, 132.4, 131.6, 130.1, 129.7, 119.2, 114.3, 110.1, 55.4, 41.2.

Spectral data matched with the reported literature.[7]

Bis(4-Methoxyphenyl) methane **(3ag)**



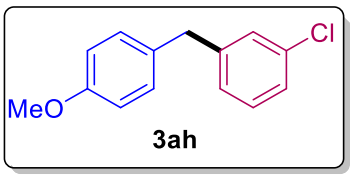
3ag

3ag was prepared according to the general procedure **A** in 79% (36.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 98:2) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.53.**

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.11 (d, *J* = 8.5 Hz, 4H), 6.84 (d, *J* = 8.5 Hz, 4H), 3.88 (s, 2H), 3.79 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.0, 133.8, 129.8, 113.9, 55.3, 40.2; **HRMS** (ESI/Q-TOF) *m/z*: $[M+NH_4]^+$ Calcd for $C_{14}H_{19}NO_2$ 245.1410, Found 246.1489.

Spectral data matched with the reported literature.[9]

1-Chloro-3-(4-methoxybenzyl) benzene **(3ah)**



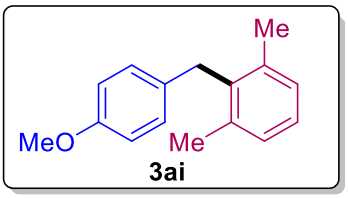
3ah

3ah was prepared according to the general procedure **A** in 92% (42.8 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 97:3) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.45.**

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.21 (dd, *J* = 7.0 Hz, 1.50 Hz, 3H), 7.11 (d, *J* = 9.0 Hz, 2H), 7.09-7.07 (m, 1H), 6.86 (d, *J* = 8.1 Hz, 2H), 3.90 (s, 2H), 3.80 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.6, 139.2, 134.6, 132.7, 130.3, 130.1, 129.3, 127.4, 126.6, 114.4, 55.7, 41.1.

Spectral data matched with the reported literature.[10]

2-(4-Methoxybenzyl)-1,3-dimethylbenzene(**3ai**)

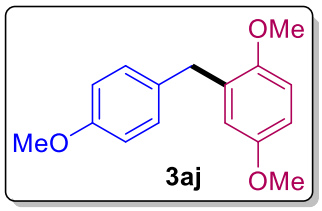


3ai

3ai was prepared according to the general procedure **A** in 55% (25.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 99:1) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.45**; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.09-7.05 (m, 3H), 6.93 (d, *J* = 8.8 Hz, 2H), 6.79 (d, *J* = 8.8 Hz, 2H), 3.99 (s, 2H), 3.76 (s, 3H), 2.24 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 157.7, 137.3, 137.1, 131.7, 128.8, 128.1, 126.3, 113.8, 55.31, 34.18, 20.27; **HRMS** (ESI/Q-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{16}H_{19}O$ 227.1430, Found 227.1421.

Spectral data matched with the reported literature.[6]

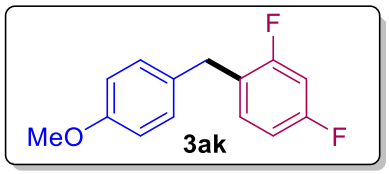
1,4-Dimethoxy-2-(4-methoxybenzyl) benzene **(3aj)**



3aj was prepared according to the general procedure **A** in 52% (26.0 mg) white solid. Purification done by column chromatography on silica gel using (hexane: EtOAc = 96:4) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.33**; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 8.02-8.00 (m, 1H), 7.87-7.85 (m, 1H), 7.77 (d, *J* = 8.1 Hz, 1H), 7.47-7.40 (m, 3H), 7.29 (dd, , *J* = 7 Hz, 1Hz, 1H), 7.13 (d, *J* = 8.75 Hz, 2H), 6.83 (d, *J* = 8.70 Hz, 2H) 4.40 (s, 2H), 3.77 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.4, 137.1, 134.0, 132.8, 132.2, 129.8, 128.8, 127.3, 127.2, 126.0, 125.8, 125.6, 124.4, 114.0, 55.4, 38.3.

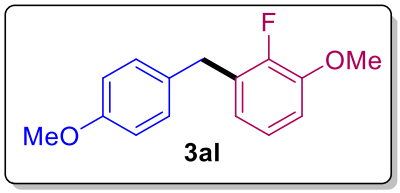
Spectral data matched with the reported literature.[11]

2,4-Difluoro-1-(4-methoxybenzyl) benzene (**3ak**)



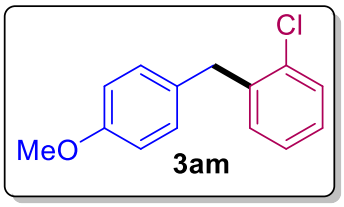
3ak was prepared according to the general procedure **A** in 47% (22.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 97:3) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.32; ^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.12 (d, *J* = 8.3 Hz, 2H), 7.08-7.06 (m, 1H), 6.84 (d, *J* = 8.8 Hz, 2H), 6.80-6.77 (m, 2H), 3.89 (s, 2H), 3.78 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.2, 131.7, 131.4, 131.5, 129.9, 129.8, 114.1, 111.2, 111.1, 104, 55.4, 33.5; **^{19}F NMR** (471 MHz, $CDCl_3$): δ -113.3, -113.7; **HRMS** (ESI/Q-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{14}H_{13}F_2O$ 235.0929, Found 235.0987.

2-Fluoro-1-methoxy-3-(4-methoxybenzyl) benzene **(3al)**



3al was prepared according to the general procedure **A** in 49% (24.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 95:5) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.31; ^{1}H NMR** (500 MHz, $CDCl_3$): δ 8.01 (d, *J* = 2.5 Hz, 1H), 7.36 (dd, *J* = 8.5 Hz, 2.5 Hz, 1H), 7.08 (d, *J* = 8.7 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.67 (d, *J* = 8.4 Hz, 1H), 3.91 (s, 2H), 3.83 (s, 3H), 3.78 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 162.8, 158.2, 146.2, 139.5, 132.7, 129.8, 129.7 114.1, 114.0, 110.8, 55.4, 53.5, 37.3; **^{19}F NMR** (471 MHz, $CDCl_3$): δ -132.6; **HRMS** (ESI/Q-TOF) *m/z*: $[M]^+$ Calcd for $C_{15}H_{15}FO_2$ 246.1056, Found 246.1030.

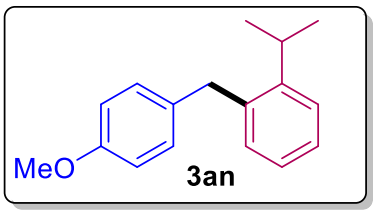
1-Chloro-2-(4-methoxybenzyl) benzene (**3am**)



3am was prepared according to the general procedure **A** in 90% (40.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 97:3) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f = 0.45;}$ ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40-7.35 (m, 1H), 7.18-7.10 (m, 5H), 6.85 (dt, J = 9.1 Hz, 2.3 Hz, 2H), 4.04 (s, 2H), 3.79 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 139.6, 138.8, 134.5, 131.1, 129.1. 128.8, 127.5, 127.3, 126.9, 126.3, 39.2.

Spectral data matched with the reported literature.[13]

1-Isopropyl-2-(4-methoxybenzyl) benzene (**3an**)

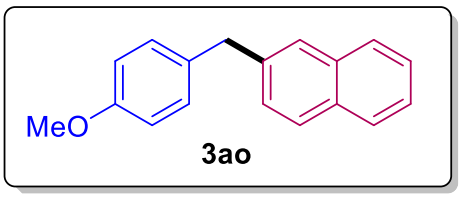


3an was prepared according to the general procedure **A** in 83% (40.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 99:1) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f = 0.35.}$

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32 (d, J = 7.8 Hz, 1H), 7.27 (t, J = 5.0 Hz, 1H), 7.16 (t, J = 7.2 Hz, 1H), 7.12 (d, J = 7.5 Hz, 1H), 7.05 (d, J = 8.2 Hz, 2H), 6.84 (d, J = 8.7 Hz, 2H), 4.02 (s, 2H), 3.79 (s, 3H), 3.18 (sept, J = 7.9 Hz, 1H), 1.17 (d, J=7.9 Hz, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.2, 147.6, 138.1, 133.7, 130.9, 130.8, 129.9, 127.2, 125.9,125.1,114.2, 55.6, 38.3, 29.3, 24.2; **HRMS** (ESI/Q-TOF) *m/z*: $[M+K]^+$ Calcd for $C_{17}H_{20}KO$ 279.1146, Found 279.1179.

Spectral data matched with the reported literature.[9]

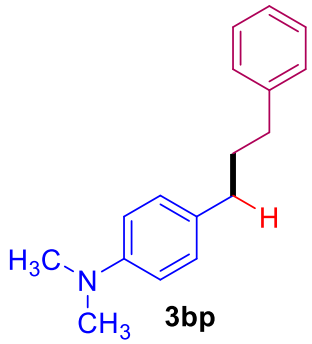
2-(4-Methoxy-benzyl) naphthalene **(3ao)**



3ao was prepared according to the general procedure **A** in 82% (40.6 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 99:1) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f = 0.52;}$ **^{1}H NMR** (500 MHz, $CDCl_3$): δ 8.02-8.00 (m, 1H), 7.87-7.85 (m, 1H), 7.77 (d, J = 8.1 Hz, 1H), 7.47-7.40 (m, 3H), 7.29 (dd, J = 7.0 Hz, 1H), 7.13 (d, J = 8.75 Hz, 2H), 6.83 (d, J = 8.7 Hz, 2H) 4.40 (s, 2H), 3.77 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.4, 137.1, 134.0, 132.8, 132.2, 129.8, 128.8, 127.3, 127.2, 126.0, 125.8, 125.6, 124.4, 114.0, 55.4, 38.3; **HRMS** (ESI/Q-TOF) *m/z*: $[M+NH_4]^+$ Calcd for $C_{18}H_{20}NO$ 266.1539, Found 266.1537.

Spectral data matched with the reported literature.[12]

1-Isopropyl-2-(4-methoxybenzyl) benzene (**3bp**)



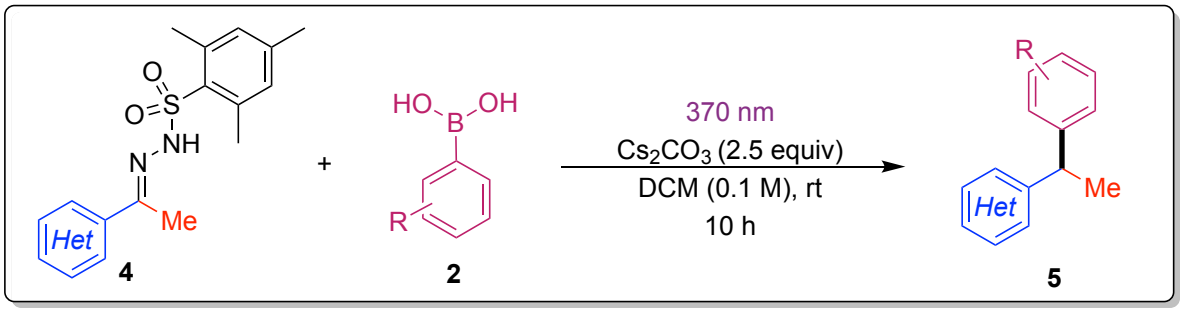
3bp

3bp was prepared according to the general procedure **A** in 72% (40.0 mg) colorless oil. Purification done by column chromatography on silica gel using (hexane: EtOAc= 97:3) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f = 0.35}$.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30-7.18 (m, *J* = 7.8 Hz, 5H), 7.08 (d, *J* = 8.5 Hz, 2H), 6.70 (d, *J* = 8.5 Hz, 2H), 2.92 (s, 6H), 2.63 (t, *J* = 7.7 Hz, 2H), 2.57 (t, *J* = 7.7 Hz, 2H), 1.96-1.90 (m, 2H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 142.6, 129.10, 128.5, 128.3, 127.0, 125.7, 113.1, 41.0, 35.5, 34.4, 33.3.

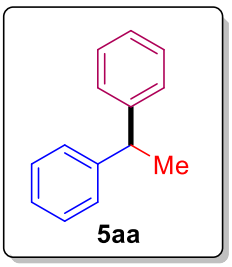
Spectral data matched with the reported literature.[14]

4.2 Synthesis of diarylalkylmethanes **(5)**



General procedure B: To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **4a** (0.1 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (0.2 mmol, 2.5 equiv) and arylboronic acid **2a** (0.15 mmol, 1.5 equiv) were added followed by DCM solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 10 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **5aa** was determined by using CH_2Br_2 as internal standard.

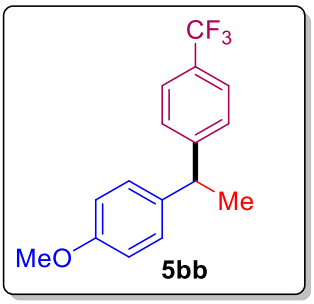
Ethane-1,1-diyldibenzene (**5aa**)



5aa was prepared according to the general procedure **B** in 63% (23.0 mg) Colorless oil. Purification done by column chromatography on silica gel using (hexanes: EtOAc = 99:1) as the eluent, **TLC** (hexanes): R_f **= 0.45;** ^{1}**H NMR** (500 MHz, $CDCl_3$): δ 7.31-7.26 (m, 6H), 7.25-7.17 (m, 4H), 4.19 (q, *J* = 7.2 Hz, 1H), 1.66 (d, *J* = 7.2 Hz, 3H); ^{13}**C NMR** (126 MHz, $CDCl_3$): δ 146.5, 128.4, 127.7, 126.1, 44.9, 22.0.

Spectral data matched with the reported literature.[15]

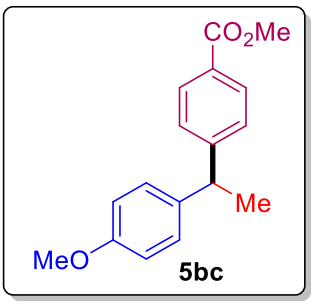
(R)-1-Methoxy-4-(1-(4-(trifluoromethyl)phenyl)ethyl)benzene (**5bb**)



5bb was prepared according to the general procedure **B** in 48% (27.0 mg) Colorless oil. Purification done by column chromatography on silica gel using (hexanes: EtOAc = 98:2) as the eluent, **TLC** (10% ethyl acetate in hexanes): R_f **= 0.45;** ^{1}**H NMR** (500 MHz, $CDCl_3$): δ 7.53 (d, *J*=8 Hz, 2H), 7.32 (d, *J* = 8 Hz, 2H), 7.13 (d, *J* = 8.2 Hz, 2H), 6.85 (d, *J* = 8.2 Hz, 2H), 4.17 (q, *J* = 7.1 Hz 1H), 3.78 (s, 3H), 1.63 (d, *J* = 7.1 Hz, 3H); ^{13}**C NMR** (126 MHz, $CDCl_3$): δ 158.2, 141.7, 133.4, 130.0, 128.9, 128.6, 126.1, 114.0, 55.4, 41.1; ^{19}**F NMR** (471 MHz, $CDCl_3$): δ -62.09.

Spectral data matched with the reported literature.[8]

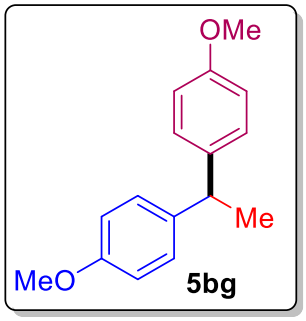
Methyl (R)-4-(1-(4-methoxyphenyl) ethyl) benzoate **(5bc)**



5bc was prepared according to the general procedure **B** in 71% (38.0 mg) Colorless oil. Purification done by column chromatography on silica gel using (hexanes: EtOAc = 98:2) as the eluent, **TLC** (10% ethyl acetate in hexanes): R_f **= 0.35;** ^{1}**H NMR** (500 MHz, $CDCl_3$): δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.16 (q, *J* = 7.2 Hz 1H), 3.78 (s, 3H), 1.63 (d, *J* = 7.2 Hz, 3H); ^{13}**C NMR** (126 MHz, $CDCl_3$): δ 167.2, 158.1, 152.3, 137.7, 129.8, 128.6, 127.7, 113.9, 55.3, 52.1, 44.1, 21.9.

Spectral data matched with the reported literature.[8]

4,4'-(Ethane-1,1-diyl) bis(methoxybenzene) (**5bg**)

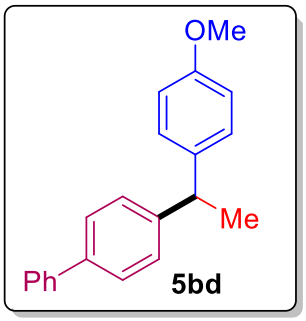


5bg was prepared according to the general procedure **B** in 72% (35.0 mg) white solid. Purification done by column chromatography on silica gel using (hexanes: EtOAc = 70:30) as the eluent, **TLC** (10% ethyl acetate in hexanes): $\boldsymbol{R_f}$ **= 0.32.**

^{1}H NMR (500 MHz, $CDCl_3$): 7.61 (d, *J* = 7.6 Hz, 2H), 7.55 (d, *J* = 7.6 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.37-7.31 (m, 3H), 7.22 (d, *J* = 7.2 Hz, 2H), 6.89 (d, *J* = 6.89 Hz, 2H), 4.19 (q, *J* = 7.2 Hz, 1H), 3.81 (s, 3H), 1.68 (d, *J*=7.2 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.0, 146.0, 141.0, 138.9, 138.5, 128.8, 128.6, 128.0, 127.7, 127.2, 127.1, 113.9, 43.3, 22.2.

Spectral data matched with the reported literature.[8]

(S)-4-(1-(4-Methoxyphenyl) ethyl)-1,1'-biphenyl) (**5bd**)

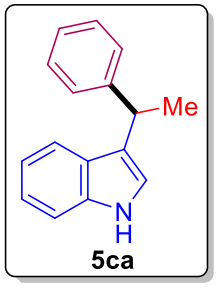


5bd was prepared according to the general procedure **B** in 55% (30.0 mg) white solid. Purification done by column chromatography on silica gel using (hexanes: EtOAc = 70:30) as the eluent, **TLC** (10% ethyl acetate in hexanes): $\boldsymbol{R_f}$ **= 0.32**.

^{1}H NMR (500 MHz, $CDCl_3$): 7.60 (d, *J* = 7.60Hz, 2H), 7.56 (d, *J* = 7.6 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.37-7.31(m, 3H), 7.22 (d, *J* = 7.2 Hz, 2H), 6.89 (d, *J* = 6.89Hz, 2H), 4.19 (q, *J* = 7.2 Hz, 1H), 3.81(s, 3H), 1.68 (d, *J* = 7.2 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 158.0, 146.0, 141.0, 138.9, 138.5, 128.8, 128.6, 128.0, 127.7, 127.2, 127.1, 113.9, 43.3, 22.2; **HRMS** (ESI/Q-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{21}O$ 289.1611, Found 289.1611.

Spectral data matched with the reported literature.[8]

(R)-3-(1-Phenylethyl)-1H-indole (**5ca)**

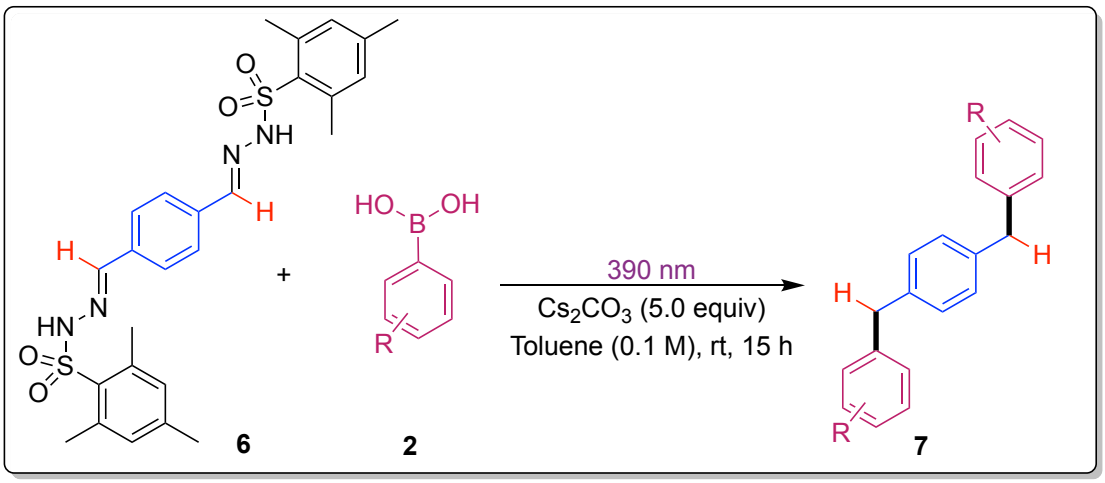


5ca was prepared according to the general procedure **B** in 82% (36.0 mg) yellow oil. Purification done by column chromatography on silica gel using (hexanes: EtOAc = 93:7) as the eluent, **TLC** (10% ethyl acetate in hexanes): $\boldsymbol{R_f}$ **= 0.3; ^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.96 (s,1H), 7.37-7.32 (m, 2H), 7.30-7.26 (m, 2H), 7.20-7.13 (m, 2H), 7.02-6.98 (m, 2H), 4.38 (q, *J* = 7.38 Hz, 1H), 1.71 (d, *J* = 7.1 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 146.8, 136.7, 128.3, 127.5, 126.9, 125.9,

122.0, 121.5, 121.1, 119.7, 119.2, 37.0, 22.4; **HRMS** (ESI/Q-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{16}H_{16}N$ 222.1277., Found 222.1272.

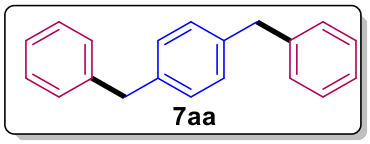
Spectral data matched with the reported literature.[16]

4.3 Synthesis of bis(1,4-diaryl/naphthylmethyl)benzenes **(7)**



General procedure C: To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **6** (0.1 mmol, 1.0 equiv) was added, then Cs_2CO_3 (0.2 mmol, 2.0 equiv) and arylboronic acid **2** (0.3 mmol, 3 equiv) were added followed by toluene solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 390 nm LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching ~35˚C) for 15 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^1H NMR, yield of **7aa** was determined by using CH_2Br_2 as internal standard.

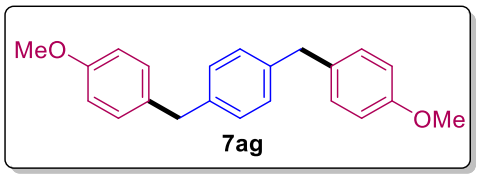
1,4-Dibenzylbenzene (**7aa**)



7aa was prepared according to the general procedure **C** in 70% (18 mg) yellow oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 99.5:0.5) as an eluent, **TLC** (5% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.50;** ^{1}**H NMR** (500 MHz, $CDCl_3$): 7.36-7.29 (m, 5H), 7.23-7.20 (m, 5H), 7.13 (s, 4H), 3.97 (s, 4H); ^{13}**C NMR** (126 MHz, $CDCl_3$): δ 141.3, 138.9, 129.12, 129.04, 128.5, 126.1, 41.6**.**

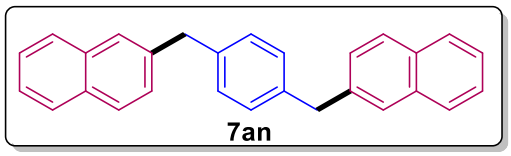
Spectral data matched with the reported literature.[17]

1,4-Bis(4-methoxybenzyl) benzene (**7ag**)



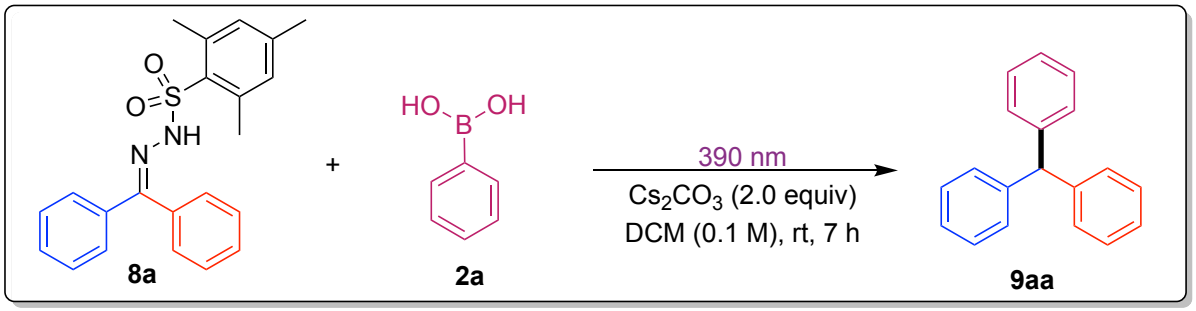
7ag was prepared according to the general procedure **C** in 68% (21.5 mg) yellow oil. Purification done by column chromatography on silica gel using (hexane: EtOAc = 98:2) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.35;** $^{\mathbf{1}}$**H NMR** (500 MHz, $CDCl_3$): 7.11-7.09 (m, 8H), 6.82 (d, *J* = 7.1 Hz, 4H), 3.89 (s, 4H), 3.79 (s, 6H); $^{\mathbf{13}}$**C NMR** (126 MHz, $CDCl_3$): δ 158.0, 139.3, 133.5, 129.9, 120.0, 114.0, 55.4, 40.8; **HRMS** (ESI/Q-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{23}O_2$ 319.1693, Found 319.1665.

1,4-Bis(naphthalen-2-ylmethyl) benzene (**7an**)



7an was prepared according to the general procedure **C** in 65% (17.0 mg) white solid. Purification done by column chromatography on silica gel using (hexane: EtOAc = 99.5:0.5) as an eluent, **TLC** (10% ethyl acetate in hexane); $\boldsymbol{R_f}$ **= 0.52;** $^{\mathbf{1}}$**H NMR** (500 MHz, $CDCl_3$): 8.00-7.98(m, 2H), 7.86-7.84 ((m, 2H), 7.76 (d, *J* = 7.75 Hz, 2H), 7.46-7.39 (m, 6H), 7.29 (d, *J* = 7.3 Hz, 2H),7.10 (s, 4H), 4.41 (s, 4H); $^{\mathbf{13}}$**C NMR** (126 MHz, $CDCl_3$): δ 138.4, 136.8, 134.0, 132.0, 128.9, 128.7, 127.4, 127.2, 127.1, 125.6, 124.4, 38.7; **HRMS** (ESI/Q-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{28}H_{26}N$ 376.2060, Found 376.2029.

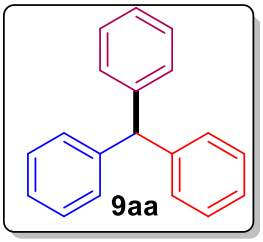
4.4 Synthesis of triarylmethanes **(9aa)**



General procedure D: To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylenesulphonylhydrazones **8a** (75.6 mg, 0.2 mmol, 1.0 equiv) derived from aldehyde was added, then Cs_2CO_3 (130.3 mg, 0.4 mmol, 2.0 equiv) and arylboronic acid **2a** (36.5 mg, 0.3 mmol, 1.5 equiv) were added followed by DCM solvent (0.1 M, 2mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 390 nm LEDs at room temperature using fan (we control the temperature by using the fan the temperature of the reaction reaching

~35˚C) for 7 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad the solvents were removed under reduced pressure and residue submitted for ^{1}H NMR, yield of **9aa** was determined by using CH_2Br_2 as internal standard.

Triphenylmethane **(9aa)**

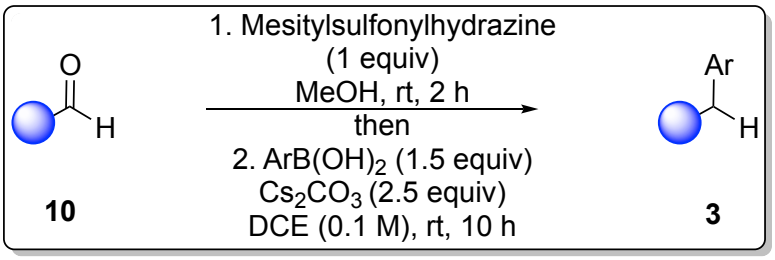


9aa was prepared according to the general procedure **D** in 58% (28 mg) white solid. Purification done by column chromatography on silica gel using (hexanes: DCM = 98:2) as the eluent, **TLC** (10% DCM in hexanes): $\boldsymbol{R_f}$ **= 0.55;** **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.31-7.27(m, 6H), 7.24-7.20 (m, 3H), 7.14-7.11(m, 6H), 5.56 (s, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 144.10, 129.6, 128.4, 126.4, 56.9; **HRMS** (ESI/Q-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{19}H_{17}$ 245.1341., Found 245.1341.

Spectral data matched with the reported literature.[18]

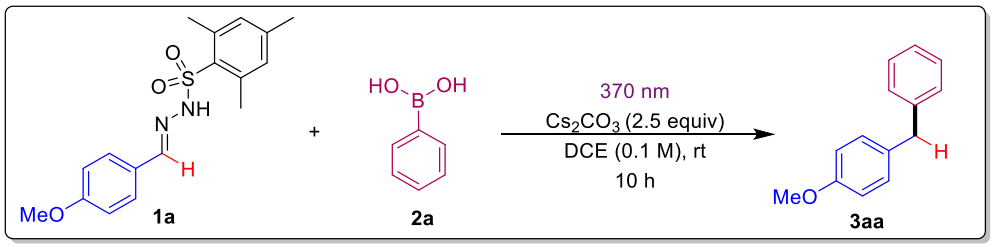
4.5 One-pot synthesis of diarylmethanes **(3)**

Procedure for the one-pot synthesis of diarylmethanes **3** from aldehydes and 2-mesitylenesulfonyl hydrazide.



To a stirred solution of 2-mesitylenesulfonyl hydrazide (43.0 mg, 0.2 mmol, 1.0 equiv) in MeOH (0.5 M), corresponding aldehydes (0.2 mmol, 1.0 equiv) in MeOH (0.25 M) solution were added dropwise. The reaction mixture was stirred at room temperature for 2 h followed by removal of MeOH under pressure. In the crude reaction mixture, Cs_2CO_3 (163.0 mg, 0.5 mmol, 2.5 equiv) and arylboronic acid (0.3 mmol, 1.5 equiv) were added followed by DCE solvent (0.1 M, 2 mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at rt for 10 h. The reaction mixture was diluted with ethyl acetate and then passed through the celite pad. The organic solvent was removed under reduced pressure, and the crude obtained was separated by column chromatography on silica gel pad (1% EtOAc/hexanes) to afford the title product **3**.

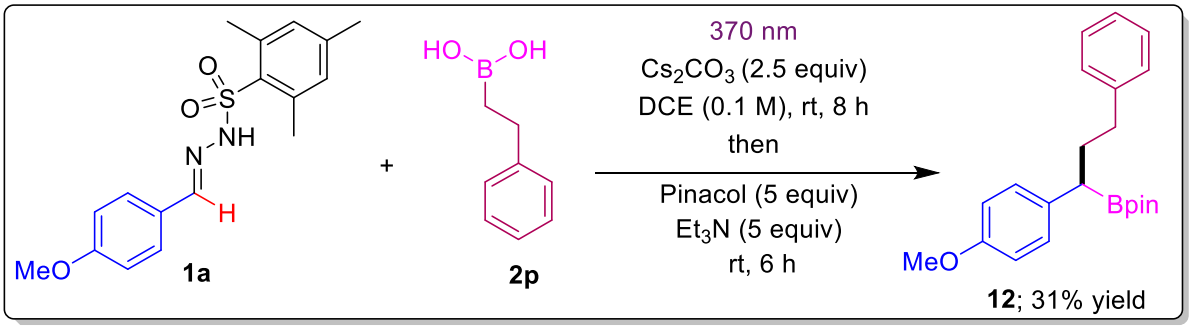
Procedure for the gram scale synthesis of diarylmethanes **3aa** from *N*-sulphonyl-hydrazones **1a**



To an oven dried 25 mL round bottom flask charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **1a** (332.4 mg, 1 mmol, 1.0 equiv) derived from aldehyde were added, then Cs_2CO_3 (814.5 mg, 2.5 mmol, 2.5 equiv) and arylboronic acid **2a** (182.8 mg, 1.5 mmol, 1.5 equiv) was added in DCE solvent (0.1 M) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at rt for 10 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad. The organic solvent was removed under reduced pressure, and the crude obtained was separated by column chromatography on silica gel pad (1% EtOAc/hexanes) to afford the title product **3aa** (168.0 mg, 85%).

5. Synthetic Application

5.1. Synthesis of (R)-2-(1-(4-Methoxyphenyl)-3-phenylpropyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **(12)**

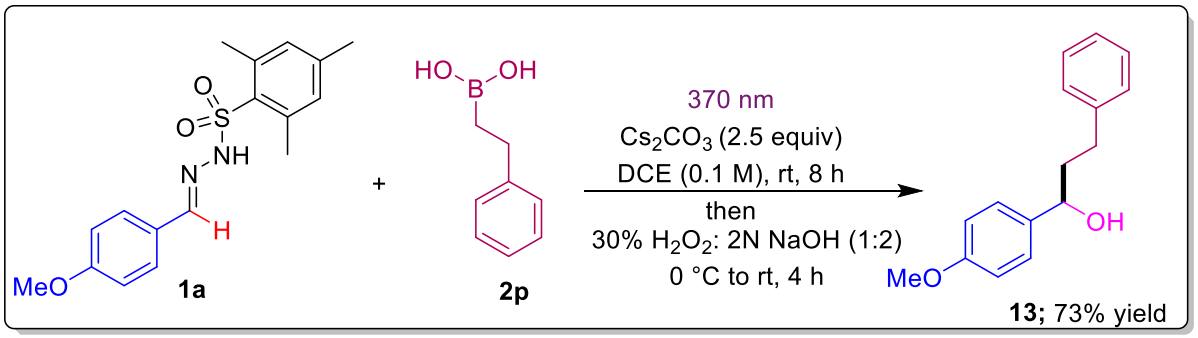


To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **1a** (66.4 mg, 0.2 mmol, 1.0 equiv) was added, then Cs_2CO_3 (197.7 mg, 0.6 mmol, 3 equiv) and phenylethyl boronic acid **2p** (45.0 mg, 0.3 mmol, 1.5 equiv) were added followed by DCE solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan for 8 h. Upon the complete consumption of the starting materials, pinacol (118.0 mg, 1 mmol, 5 equiv) along with Et_3N

(101.1 mg, 1 mmol, 5 equiv) and stirred at rt for 6 h. Upon the complete consumption of the starting materials, the reaction mixture was diluted with ethyl acetate and then passed through the celite pad. The organic solvent was removed under reduced pressure, and the crude obtained was separated by column chromatography on silica gel pad (2.5% EtOAc/hexanes) to afford the title product **12** (14 mg, 31%); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.28-7.24 (m, 2H), 7.18-7.13 (m, 5H), 6.83 (d, *J* = 7.7, 2H), 3.79 (s, 3H), 2.59-2.54 (m, 2H), 2.33-2.28 (m, 1H) 2.18-2.09 (m, 1H), 1.99-1.91 (m, 1H), 1.22 (s, 6H), 1.20 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 157.4, 142.7, 134.9, 129.3, 128.6, 128.3, 125.7, 113.8, 83.4, 55.2, 35.4, 34.7, 24.8, 24.7; δ **^{11}B NMR** (160 MHz, $CDCl_3$): -32.9 (s).

Spectral data matched with the reported literature.[19]

5.2 Synthesis of (*R*)-1-(4-Methoxyphenyl)-3-phenylpropan-1-ol **(13)**



To an oven dried 5 mL glass vial was charged with magnetic stir bar, *N*-mesitylene-sulfonylhydrazone **1a** (66.4 mg, 0.2 mmol, 1.0 equiv) was added, then Cs_2CO_3 (197.7 mg, 0.6 mmol, 3 equiv) and phenethyl boronic acid **2p** (45.0 mg, 0.3 mmol, 1.5 equiv) were added followed by DCE solvent (0.1 M, 1mL) under nitrogen atmosphere. The reaction mixture was stirred in presence of 370 nm LEDs at room temperature using fan for 8 h. Upon the complete consumption of the starting materials, the reaction mixture was set in 0 °C, followed by addition of 30% H_2O_2:2N NaOH (1:2). The reaction mixture was warmed to rt and stirred for 4 h and extracted with CH_2Cl_2 and water, the combined organic layer was dried over anhydrous sodium sulphate and filtered the crude product was by chromatography on a column of silica gel eluted with (14% EtOAc/hexanes) to give **13** (35 mg ,73%). **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.29-7.25(m, 4H), 7.20-7.16 (m, 3H), 6.88 (d, *J* = 7.88, 2H), 4.62 (q, *J* =7.38, 1H), 3.80 (s, 3H), 2.75-2.60 (m, 2H), 2.17-2.09 (m, 1H), 2.02-1.96 (m, 1H), 1.91 (br s, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ 159.2, 140.9, 136.8, 128.58, 128.52, 127.3, 125.97, 114.02, 73.6, 55.4, 40.8, 32.2, 29.8.

Spectral data matched with the reported literature.[19]

6. UV-Vis spectra

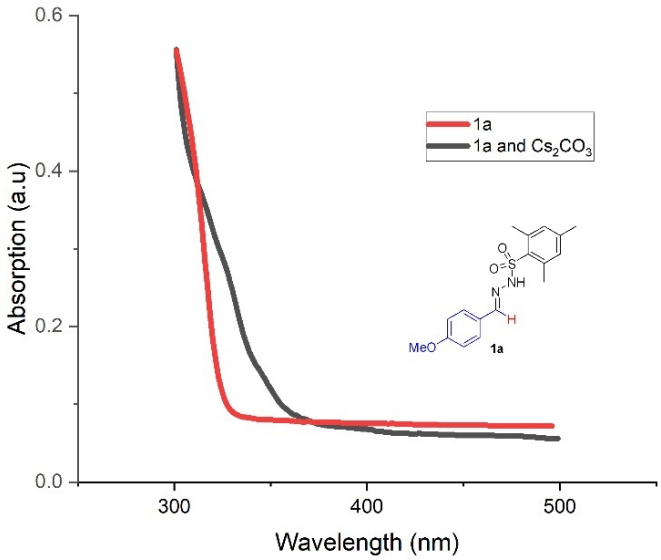


Figure S1: UV-vis spectrum of *N-mesitylene-sulfonylhydrazone* **1a** (0.1 M, red line) and **1a** + Cs_2CO_3 (0.1M, black) in MeCN.

7. References:

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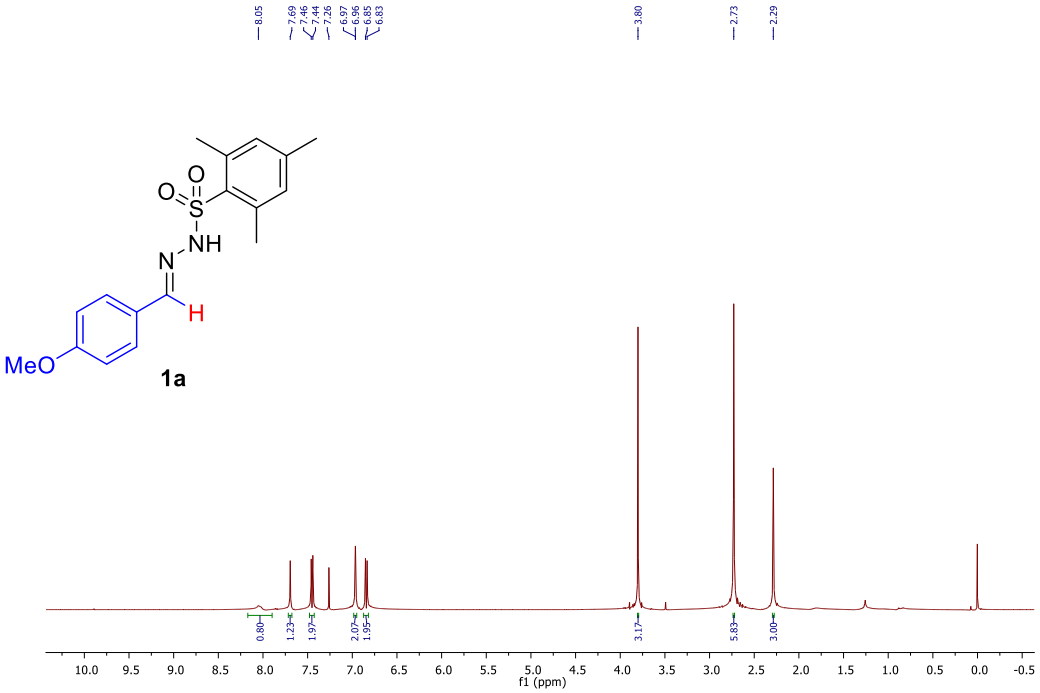
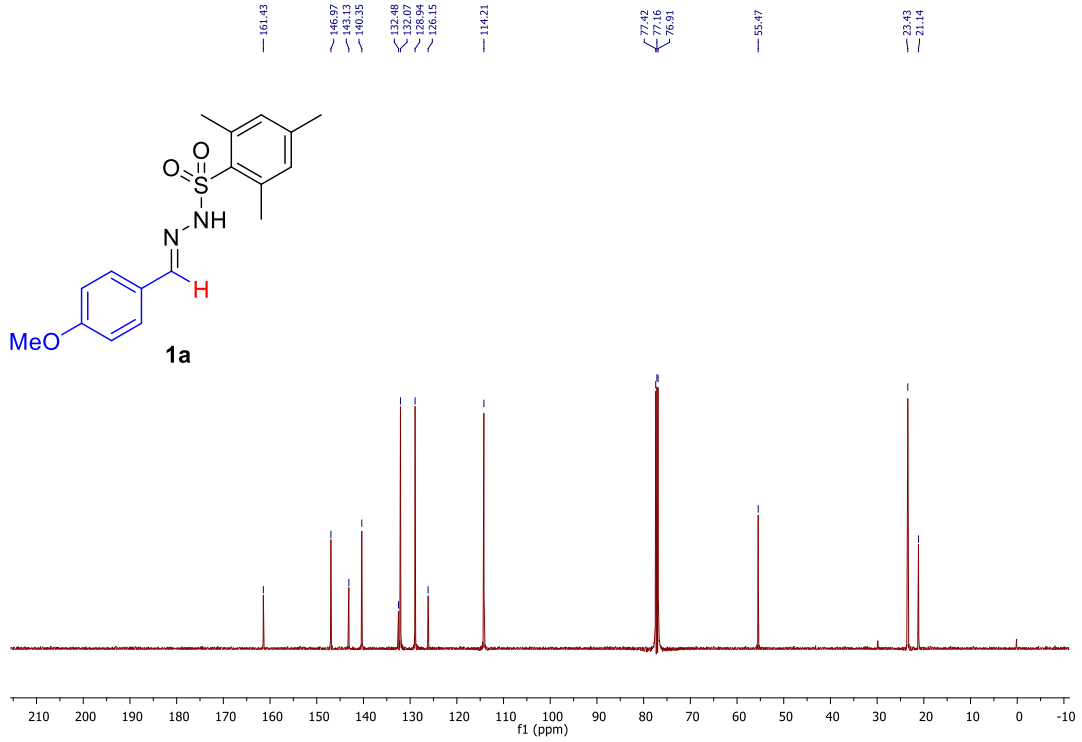
8. Spectral Data:

(E)-N'-(4-Methoxybenzylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(1a)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

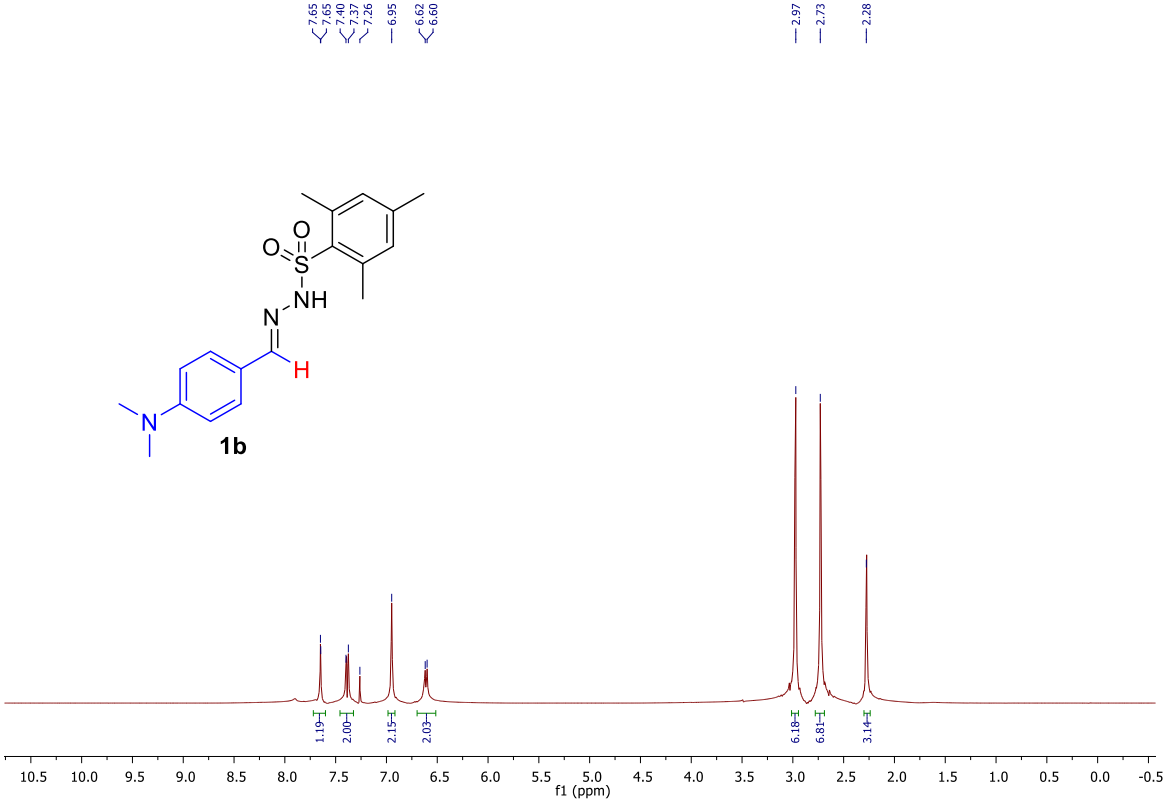


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

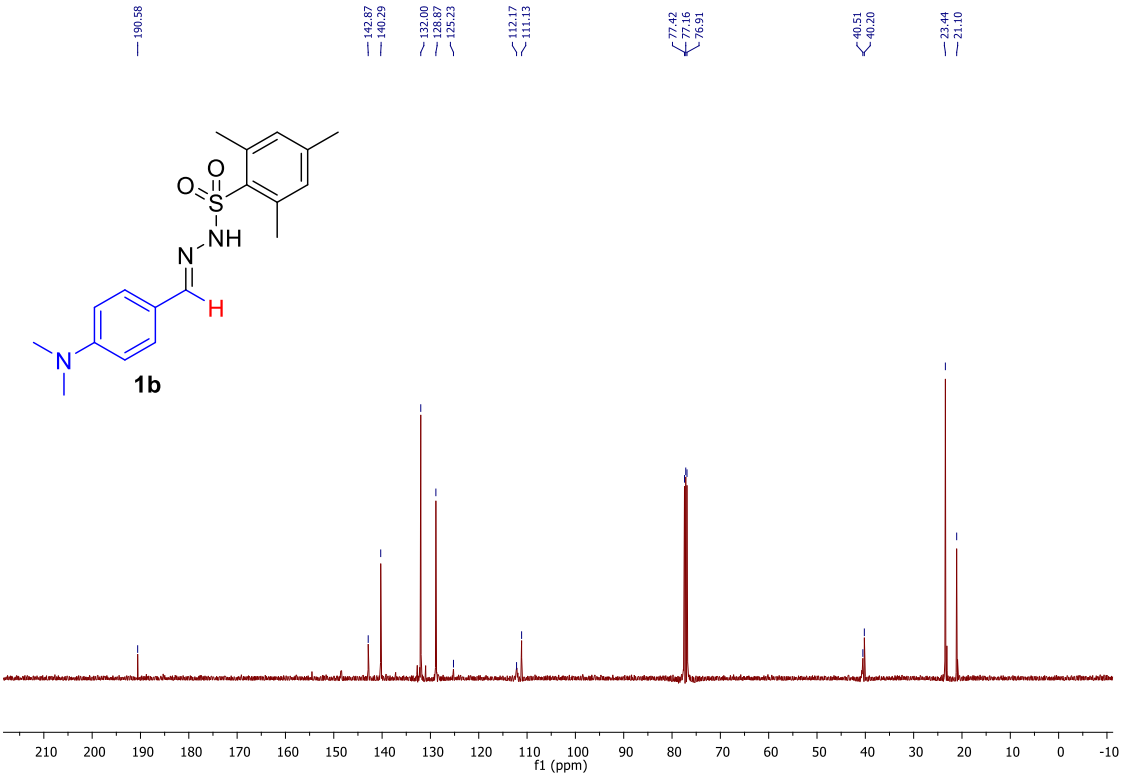


(E)-N'-(4-(Dimethylamino)benzylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(1b)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

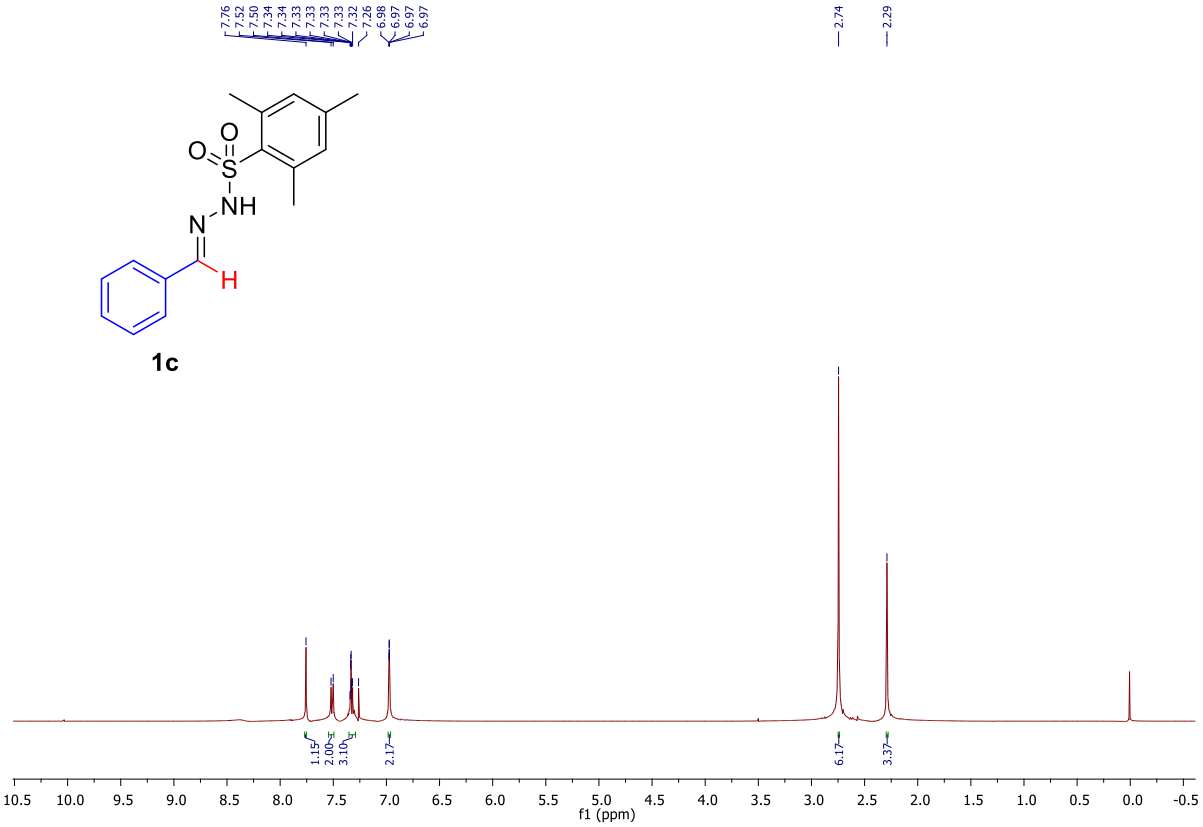


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)



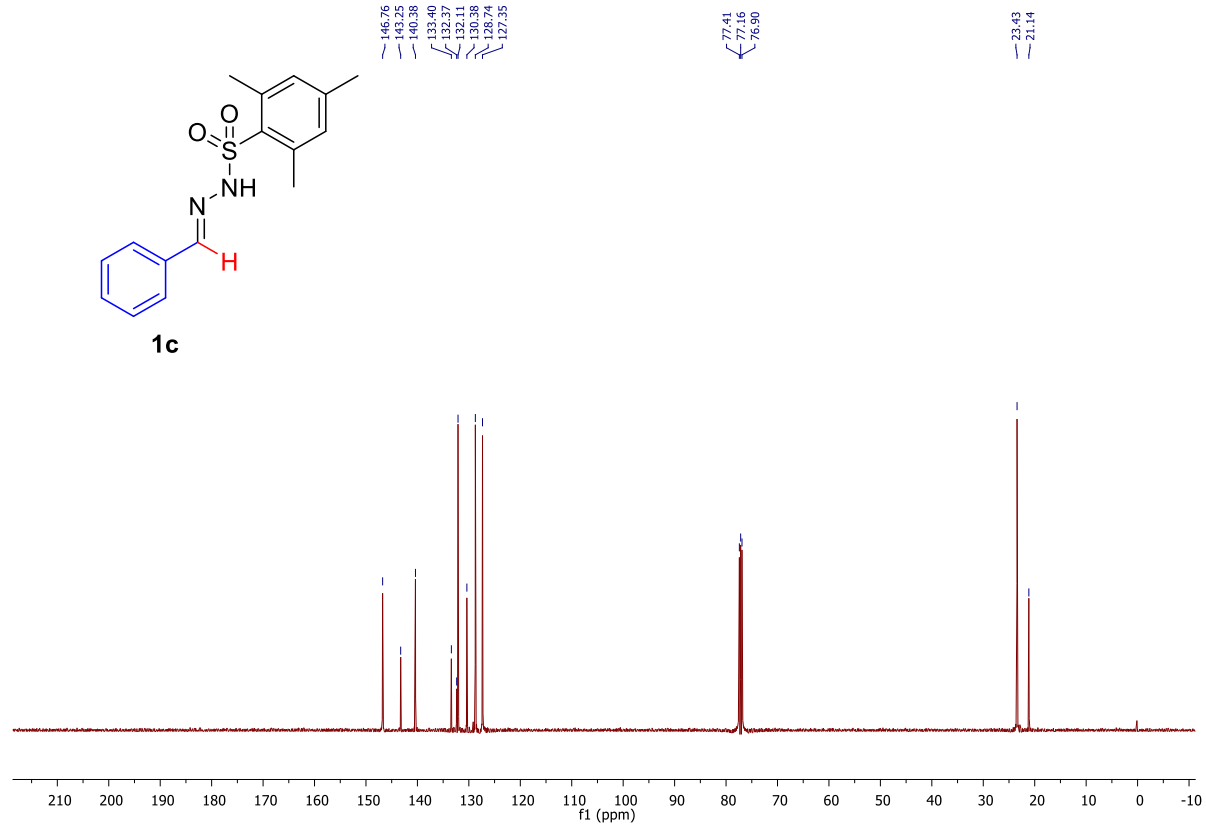
(E)-*N'*-Benzylidene-2,4,6-trimethylbenzenesulfonohydrazide **(1c)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)



1c

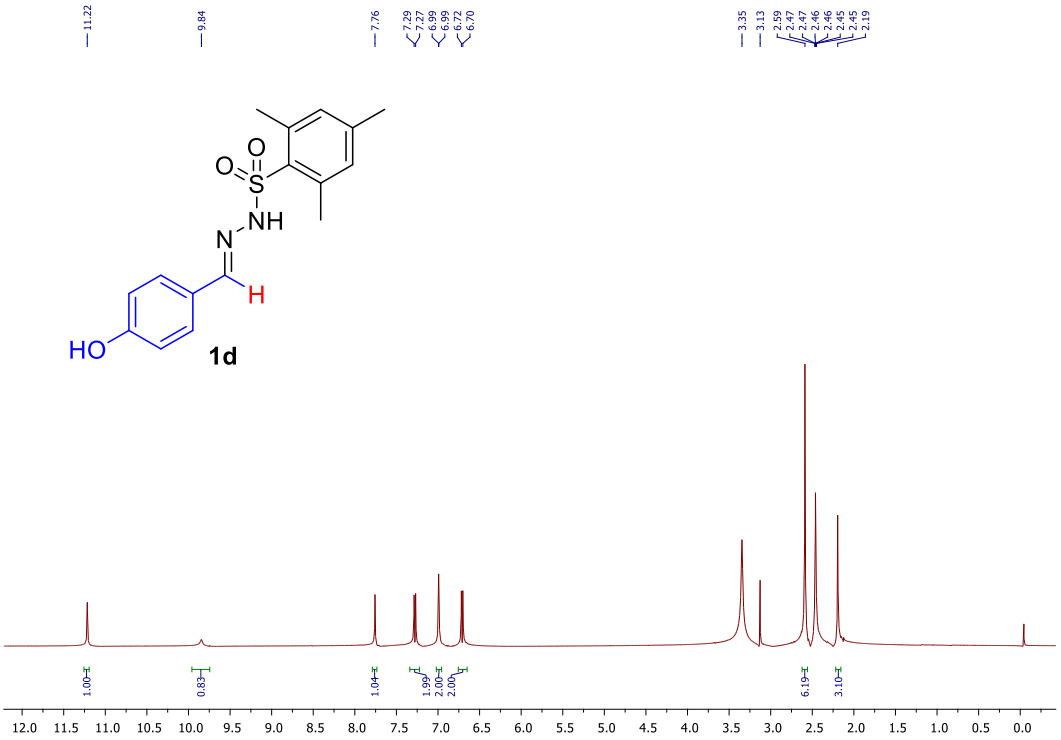
^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)



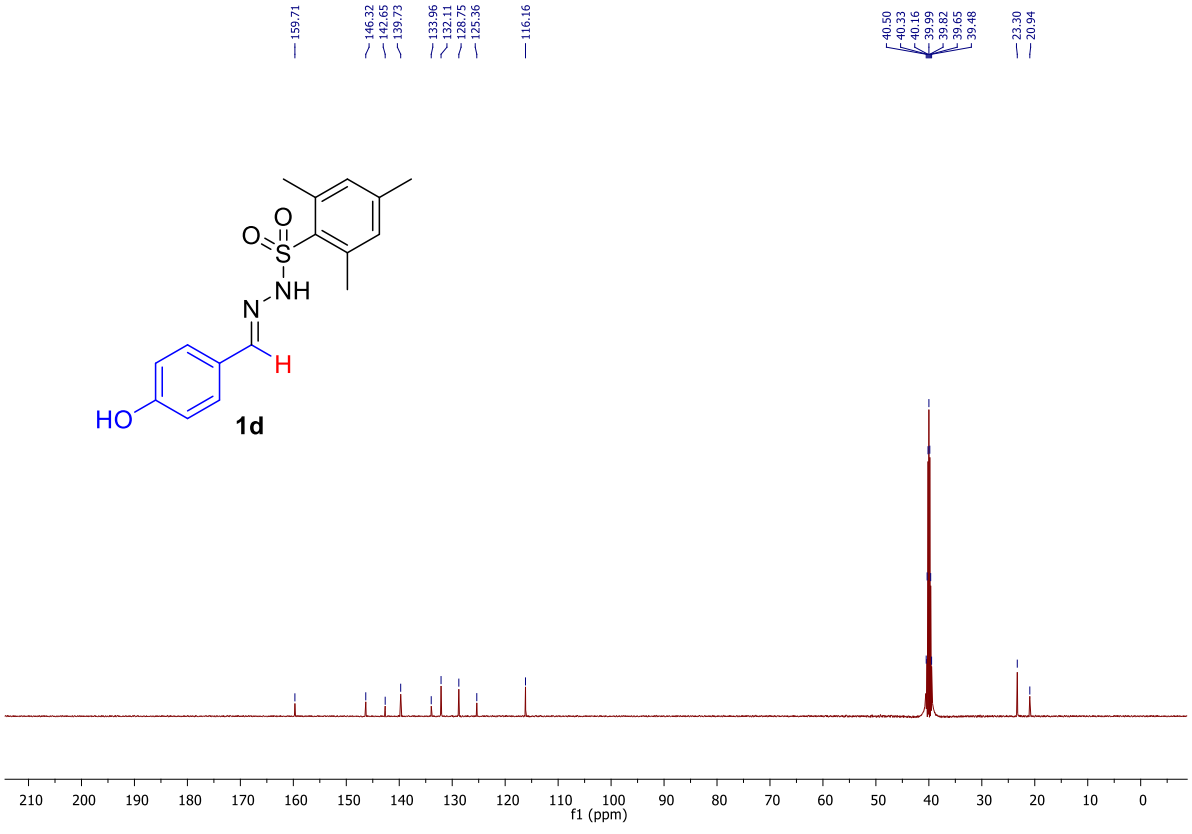
1c

(E)-N'-(4-Hydroxybenzylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(1d)**

^{1}H NMR (500 MHz, DMSO-d$_6$, 24 °C)

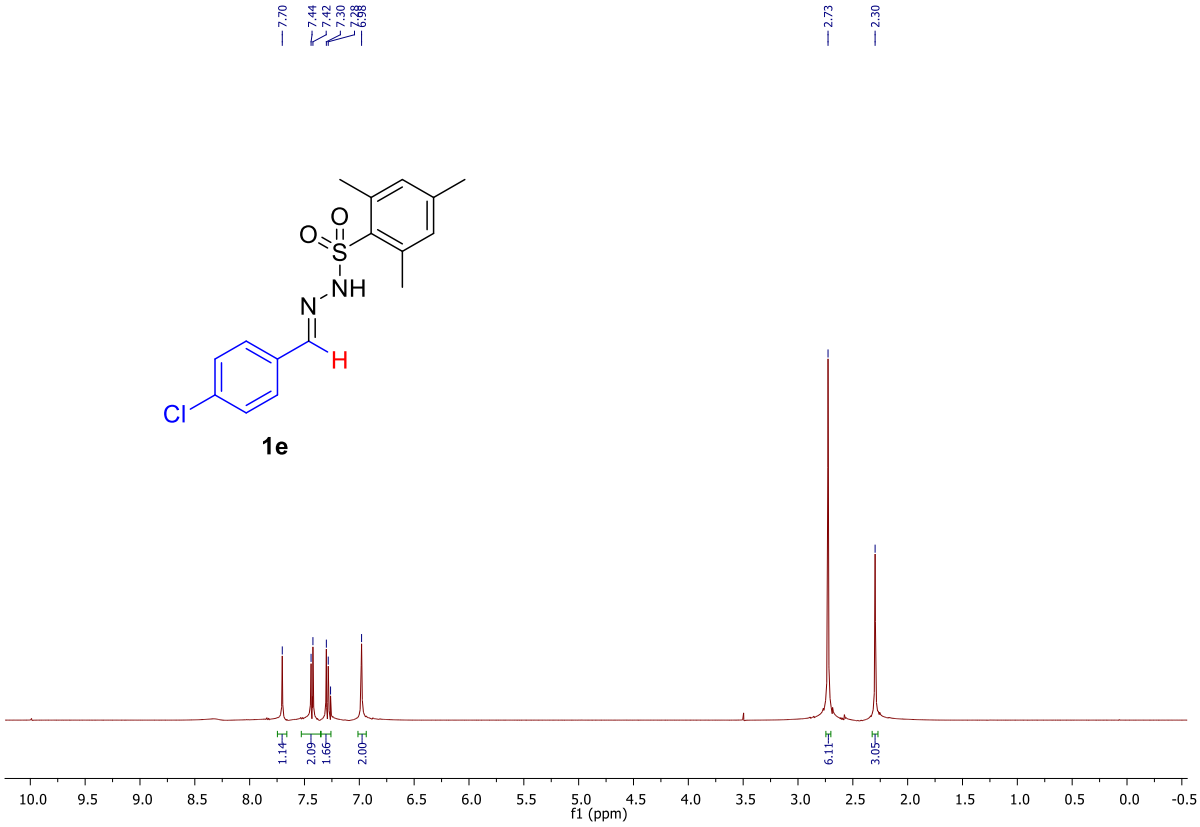


^{13}C{^{1}H} NMR (126 MHz, DMSO-d$_6$, 24 °C)

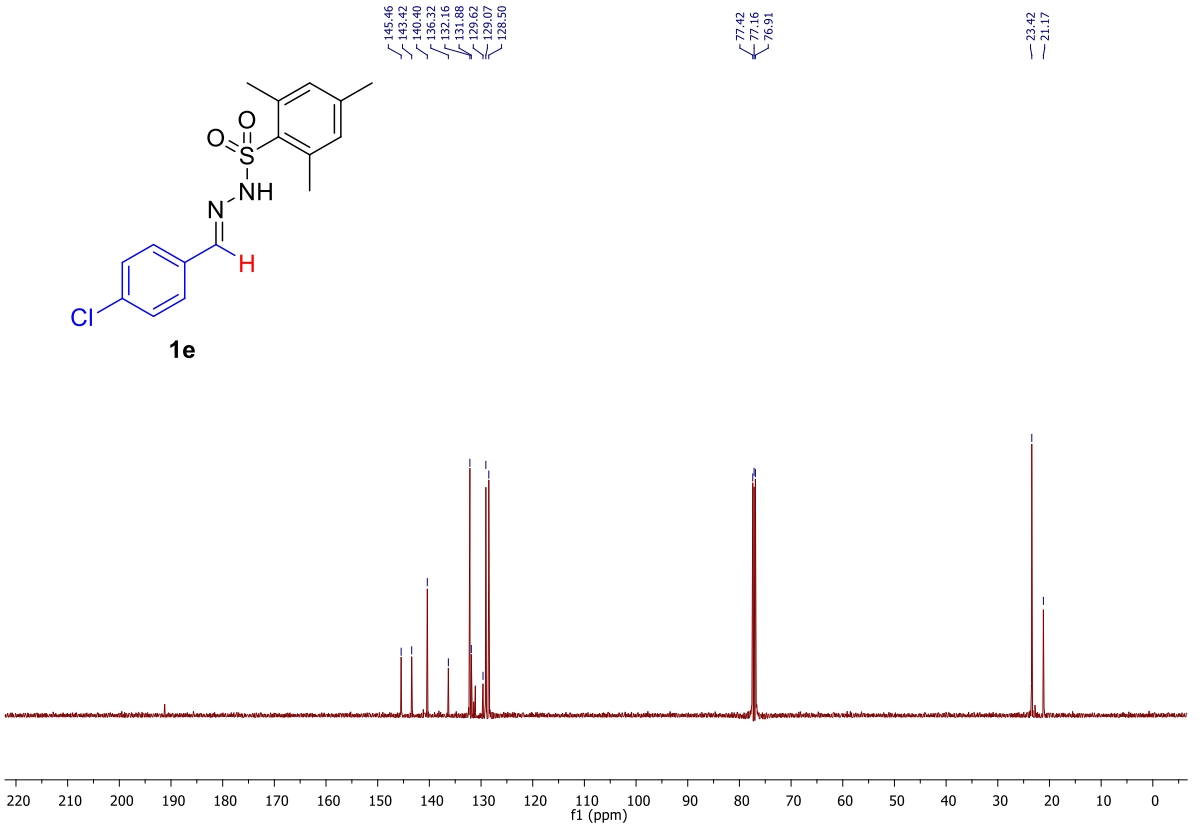


(E)-*N'*-(4-Chlorobenzylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(1e)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

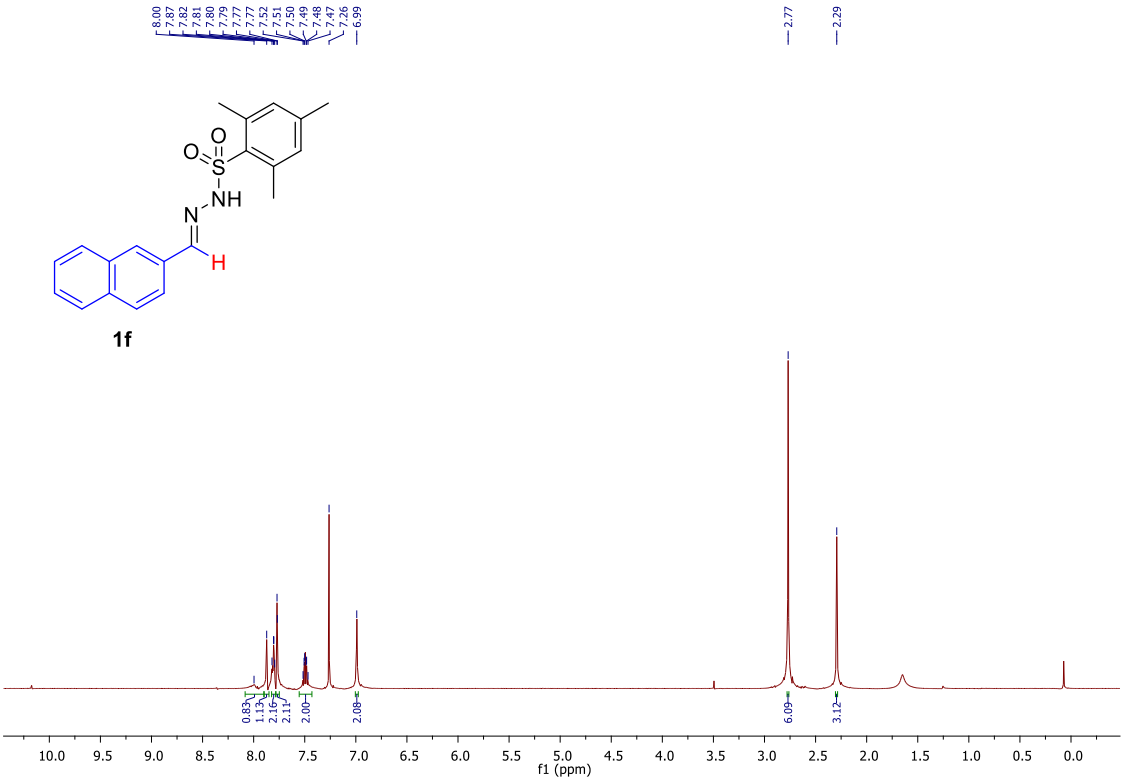


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

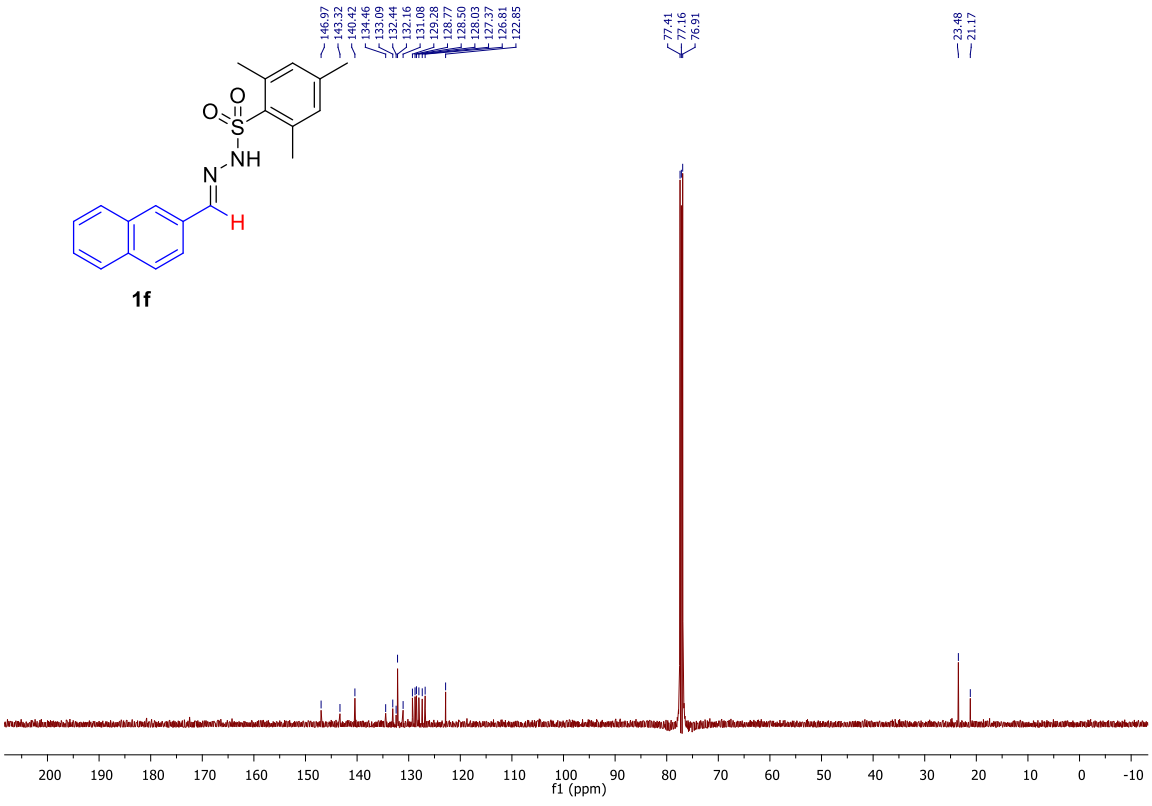


(E)-2,4,6-Trimethyl-*N′*-(naphthalen-2-ylmethylene)benzenesulfonohydrazide **(1f)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

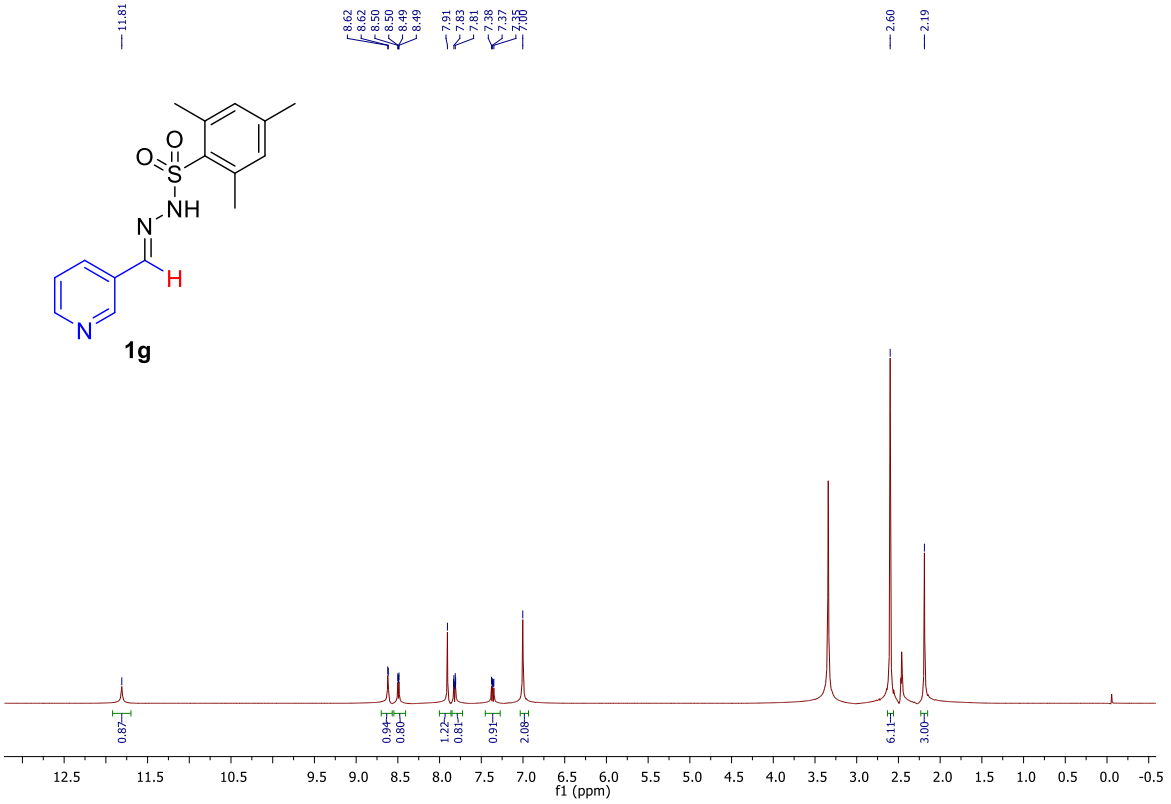


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

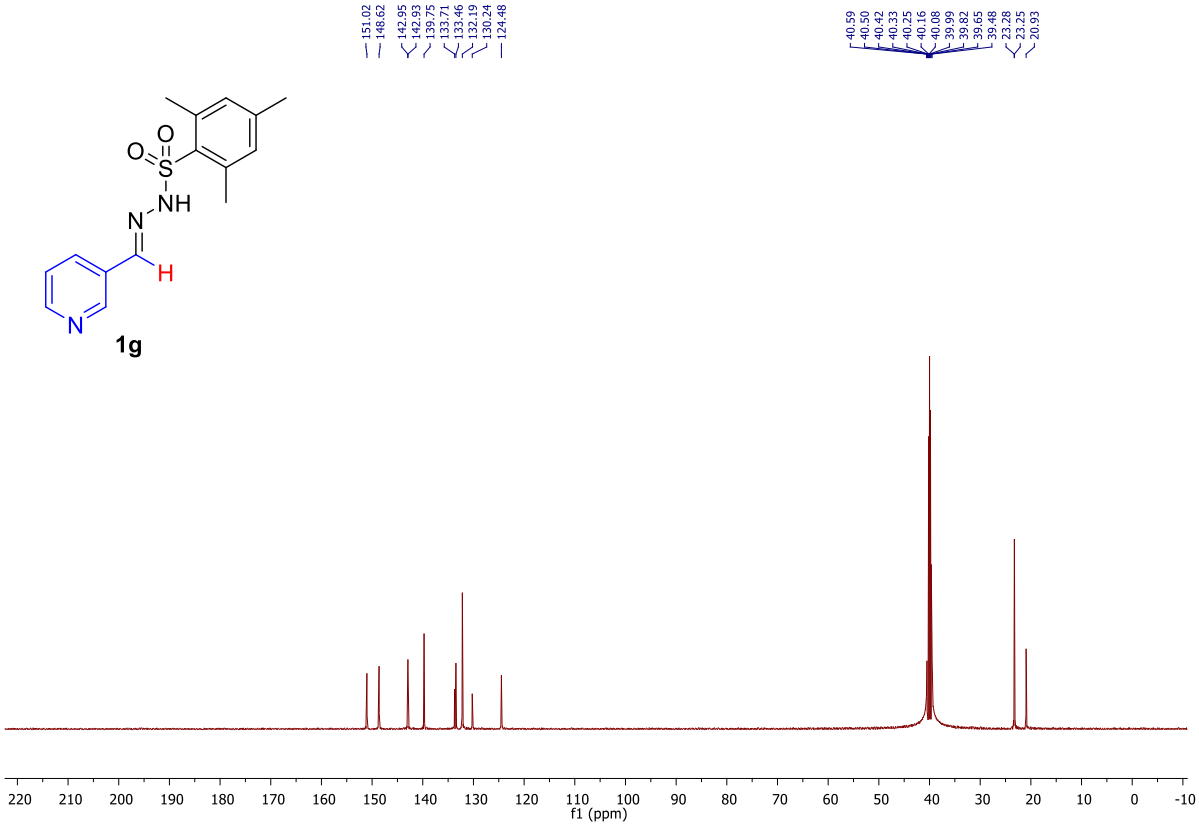


(E)-2,4,6-Trimethyl-*N*'-(pyridin-3-ylmethylene)benzenesulfonohydrazide **(1g)**

^{1}H NMR (500 MHz, DMSO-d$_6$, 24 °C)

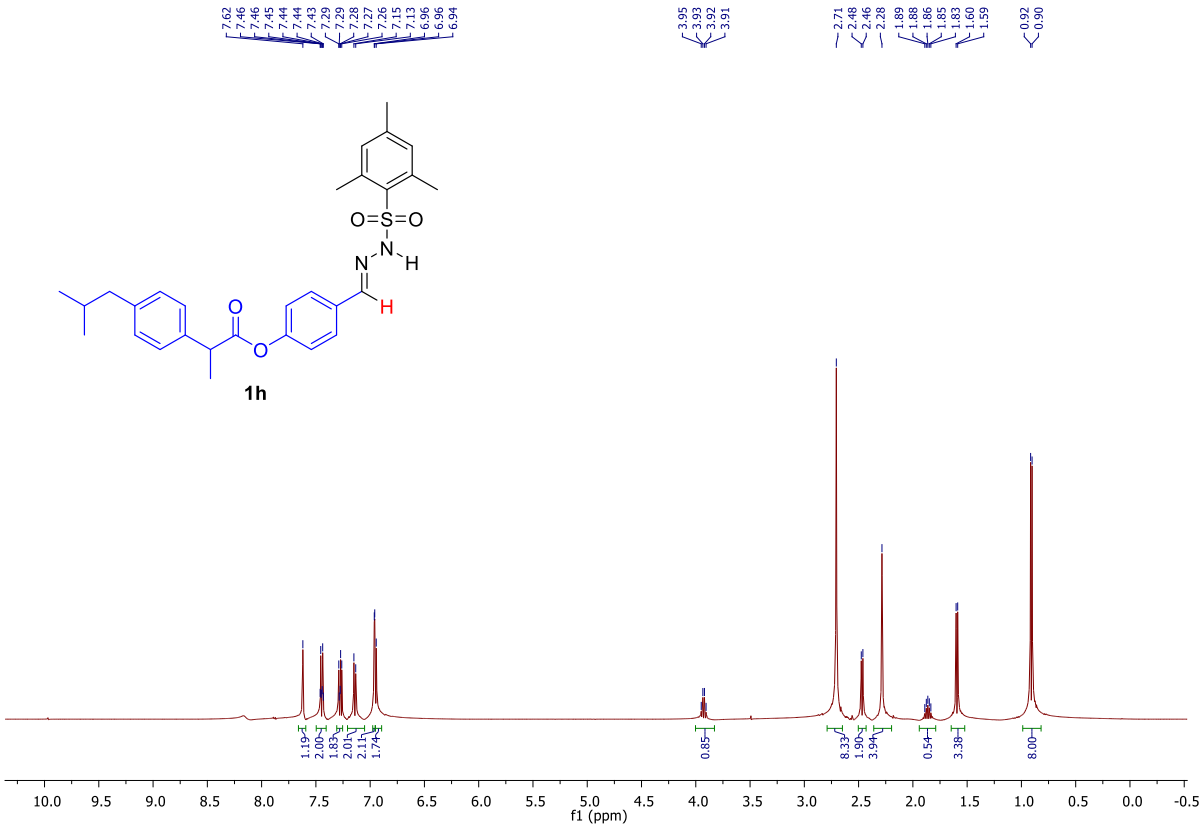


^{13}C{^{1}H} NMR (126 MHz, DMSO-d$_6$, 24 °C)

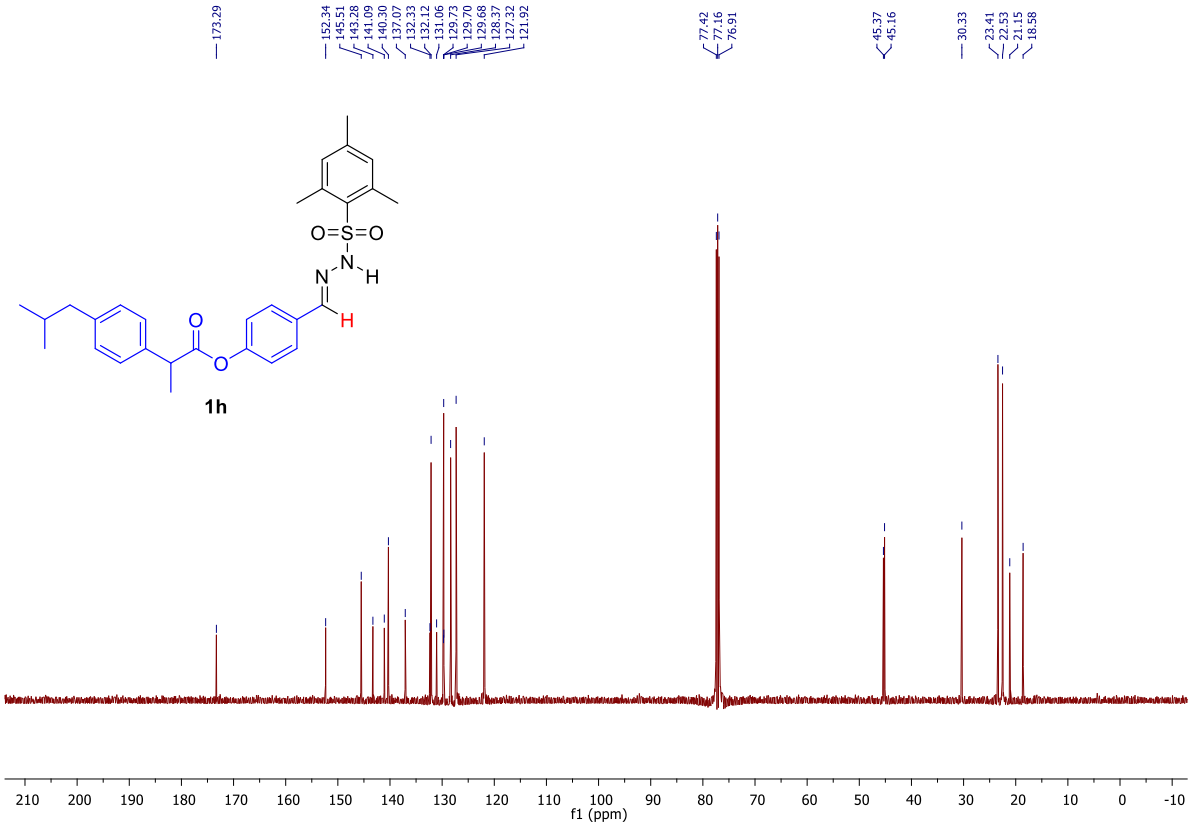


(E)-4-((2-(Mesitylsulfonyl)hydrazineylidene)methyl)phenyl 2-(4-isobutylphenyl)propanoate **(1h)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

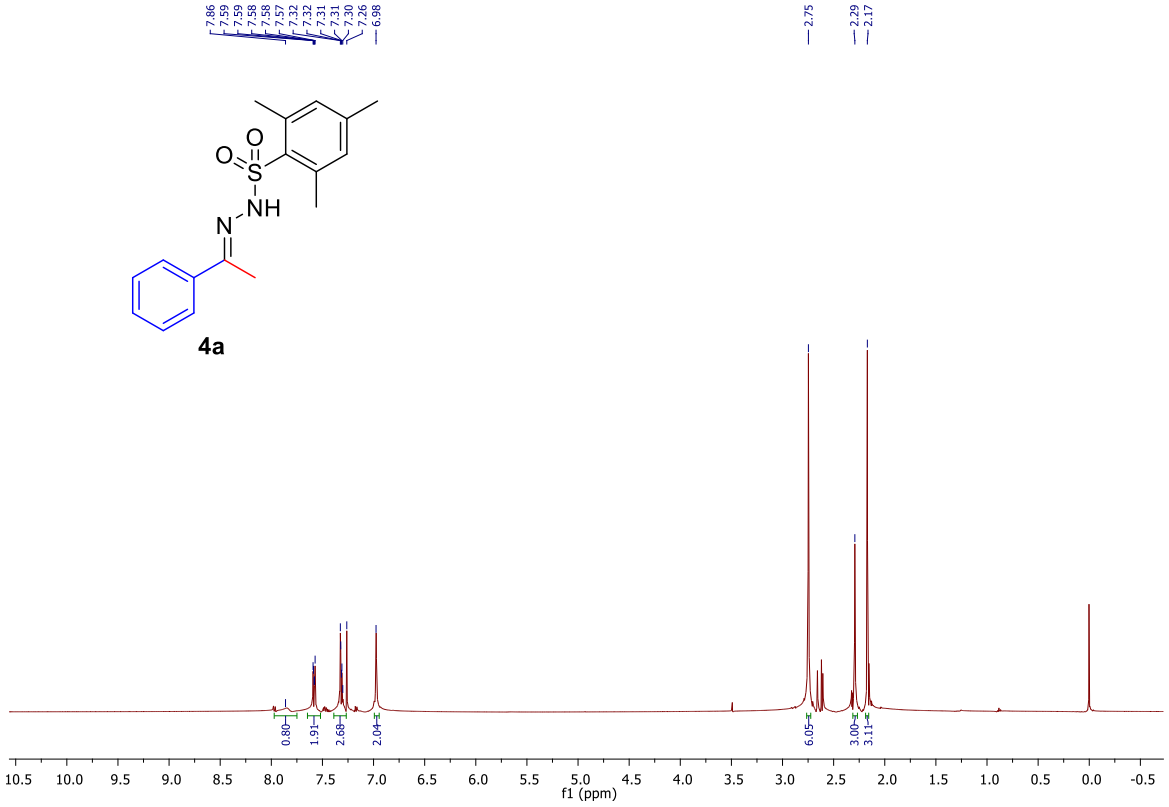


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

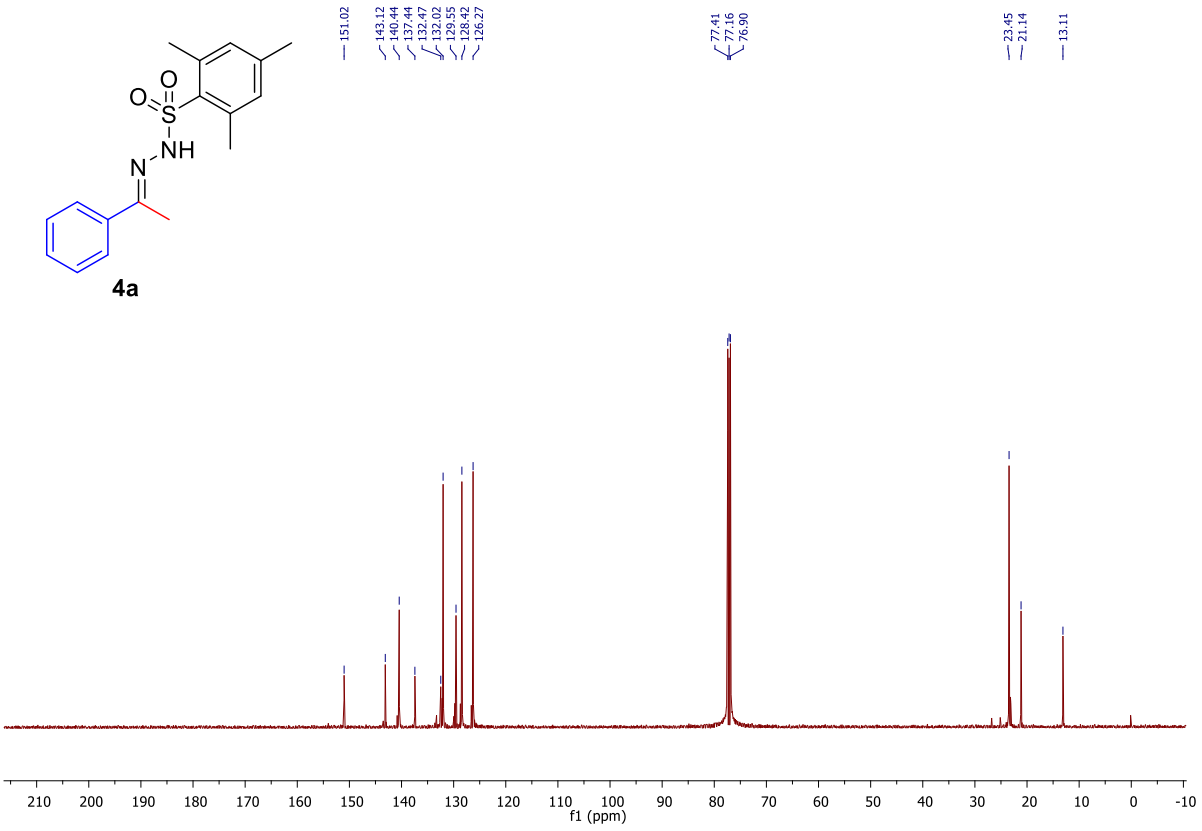


(E)-2,4,6-Trimethyl-*N'*-(1-phenylethylidene)benzenesulfonohydrazide **(4a)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

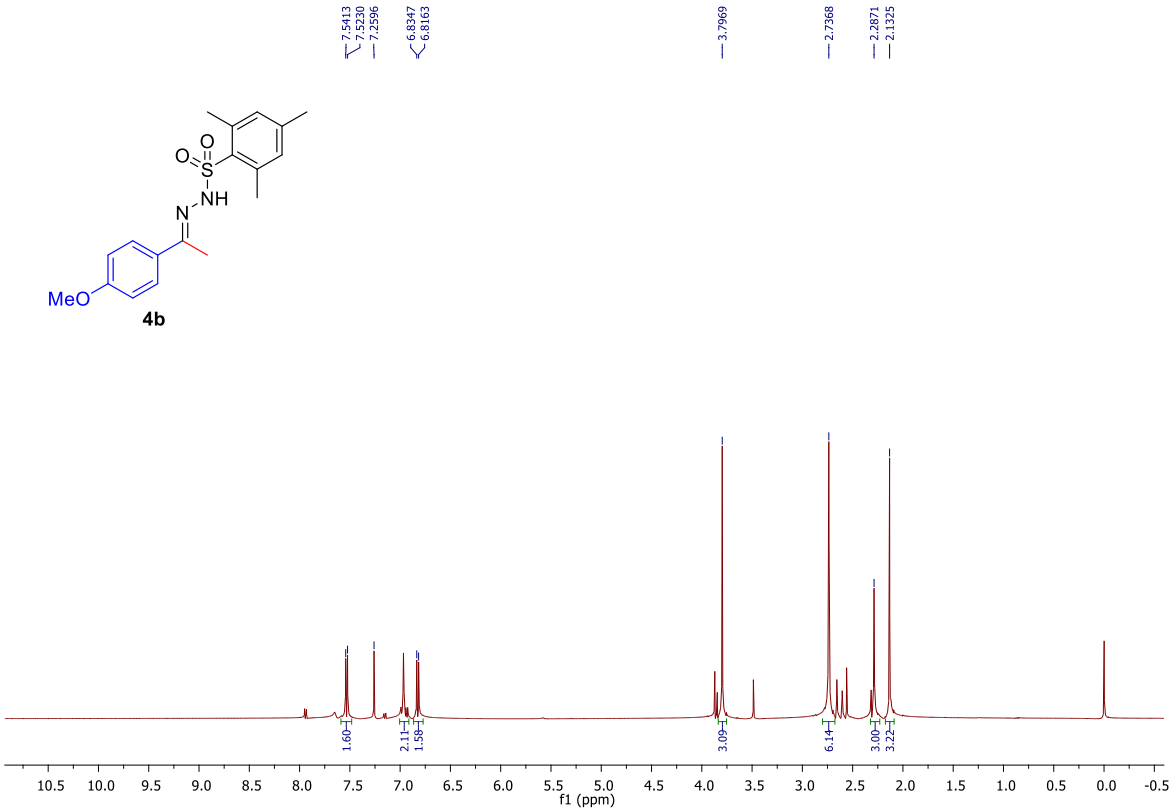


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

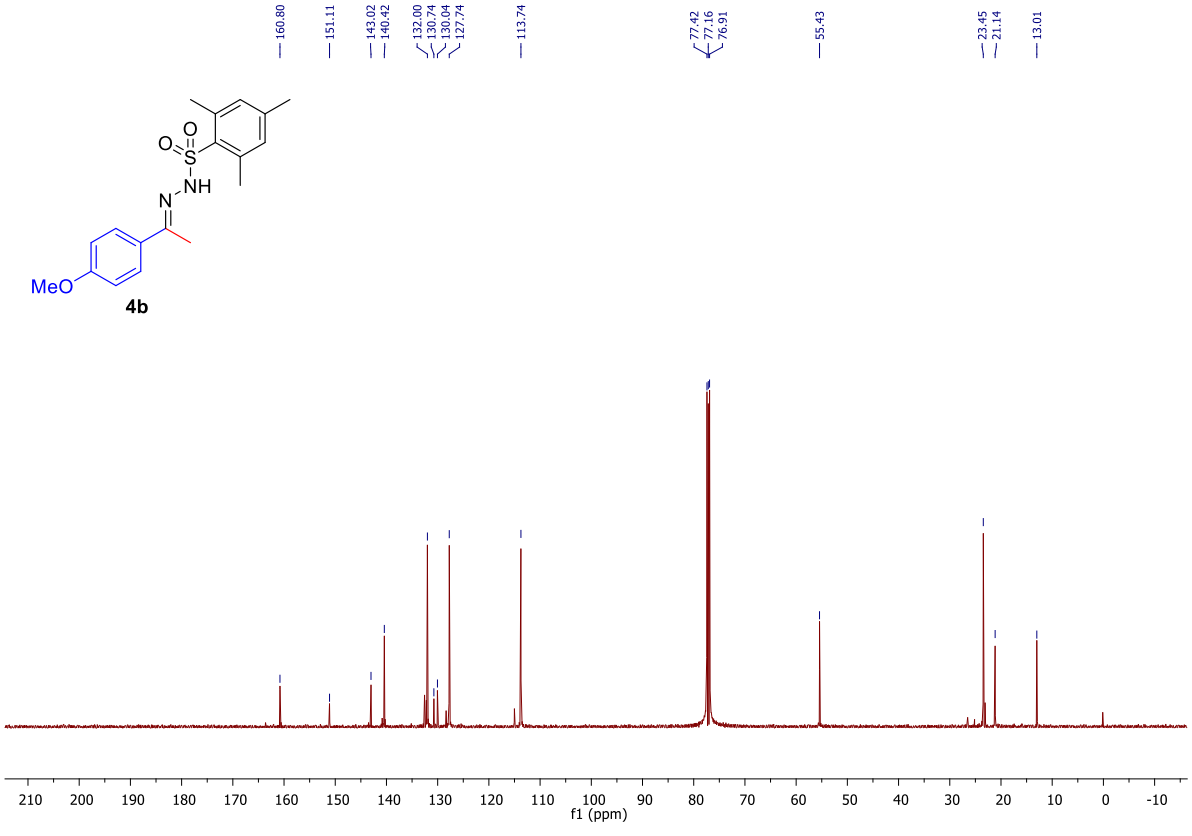


(E)-N'-(1-(4-Methoxyphenyl) ethylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(4b)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

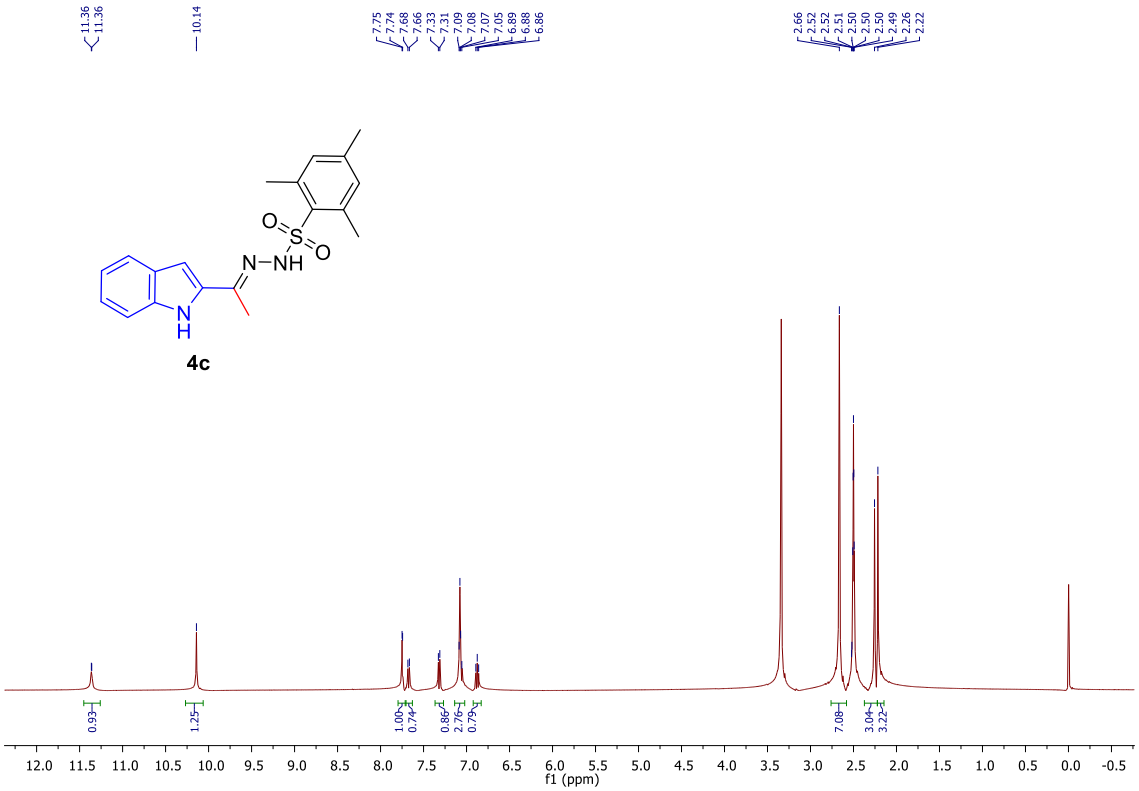


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

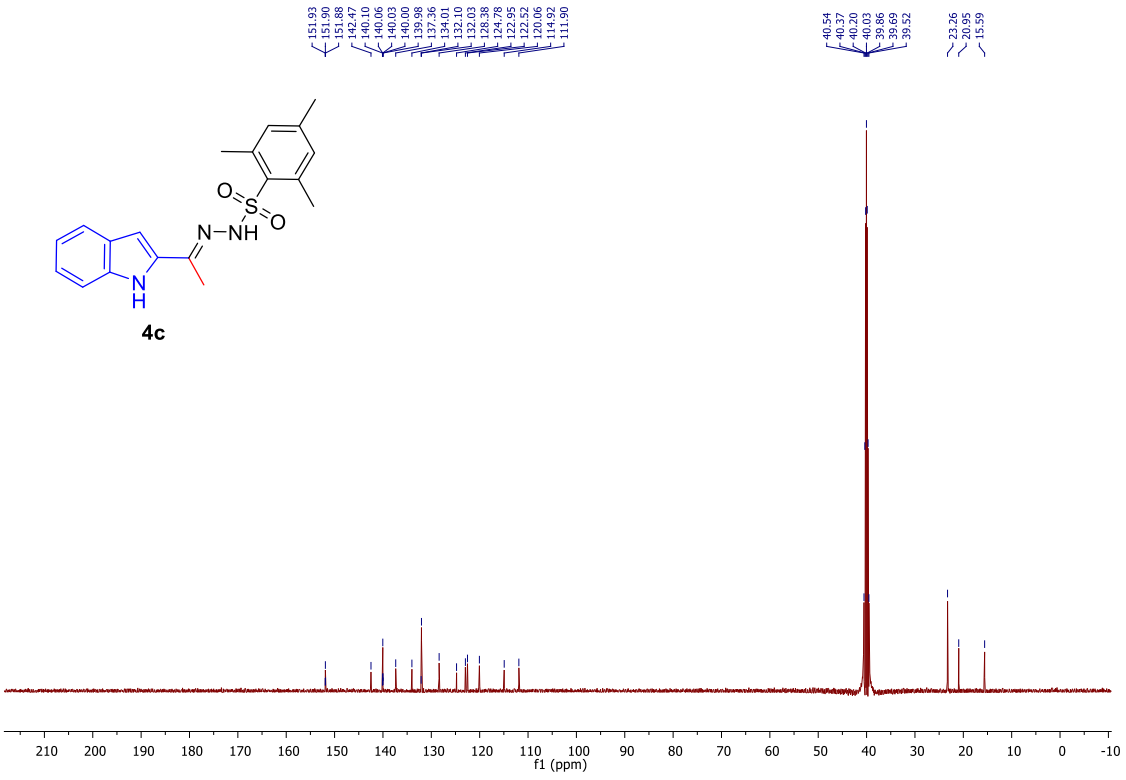


(E)-N'-(1-(1H-Indol-2-yl)ethylidene)-2,4,6-trimethylbenzenesulfonohydrazide **(4c)**

^{1} H NMR (500 MHz, DMSO-d_6, 24 °C)

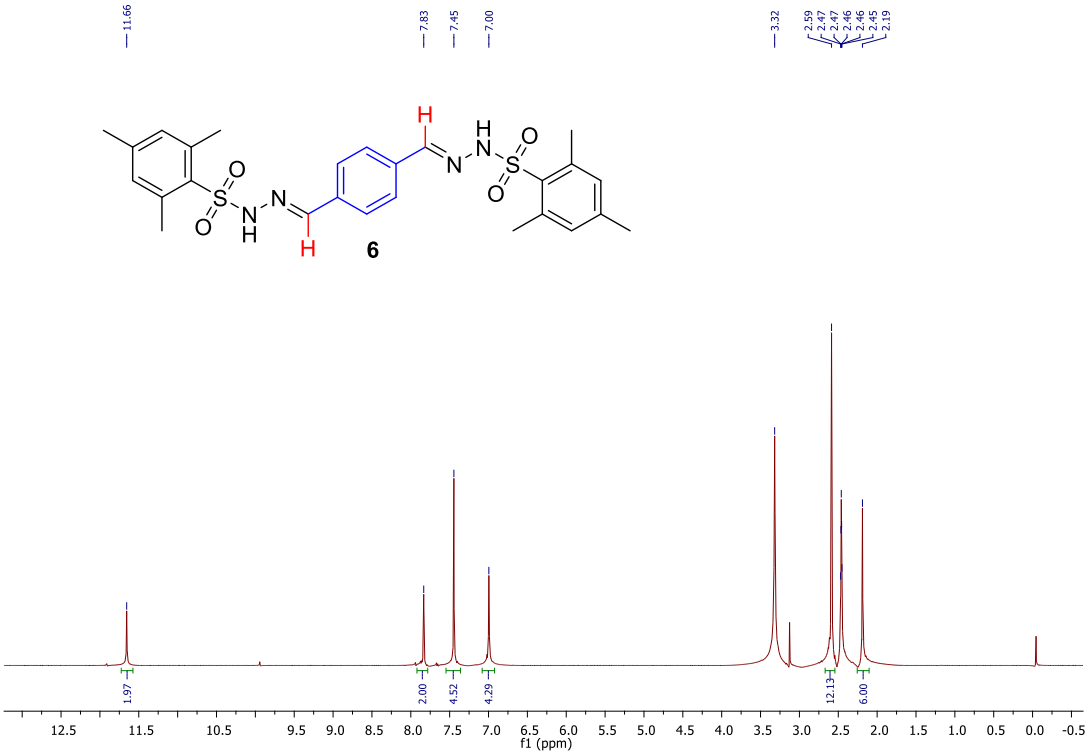


^{13} C{^{1}H} NMR (126 MHz, DMSO-d_6, 24 °C)

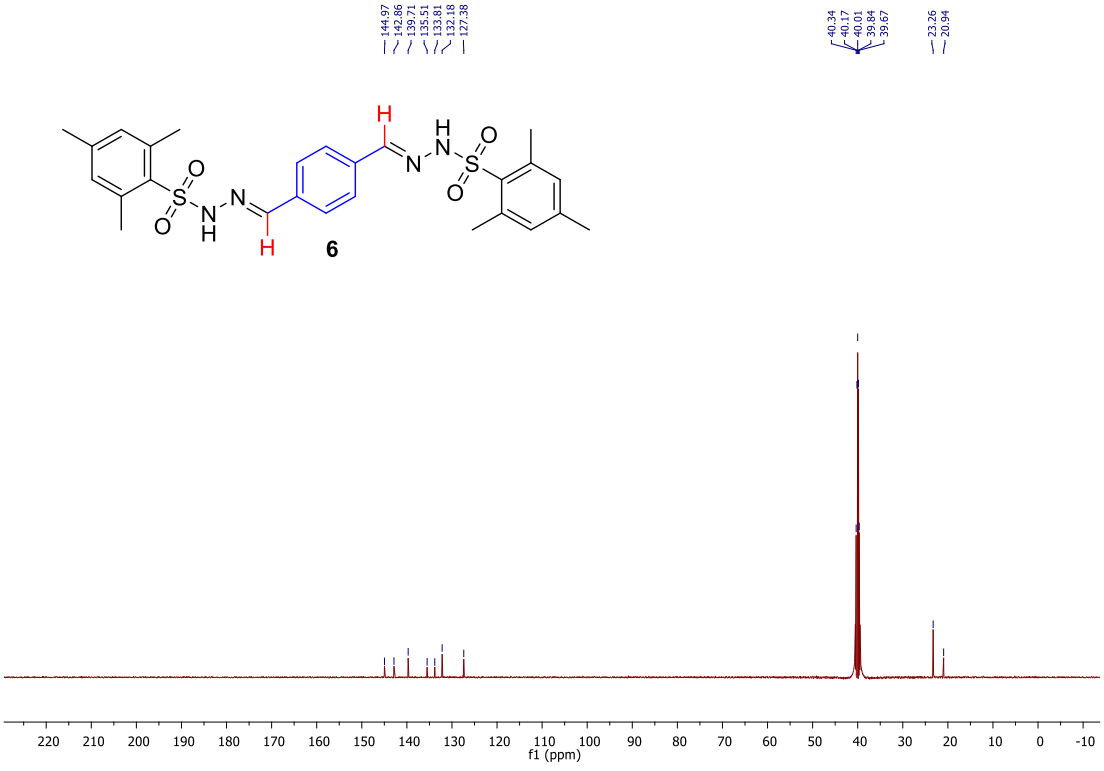


N',N'''-((*1E,1'E*)-1,4-Phenylenebis(methaneylylidene))bis(2,4,6-trimethylbenzenesulfonohydrazide) **(6)**

^{1}H NMR (500 MHz, DMSO-d$_6$, 24 °C)

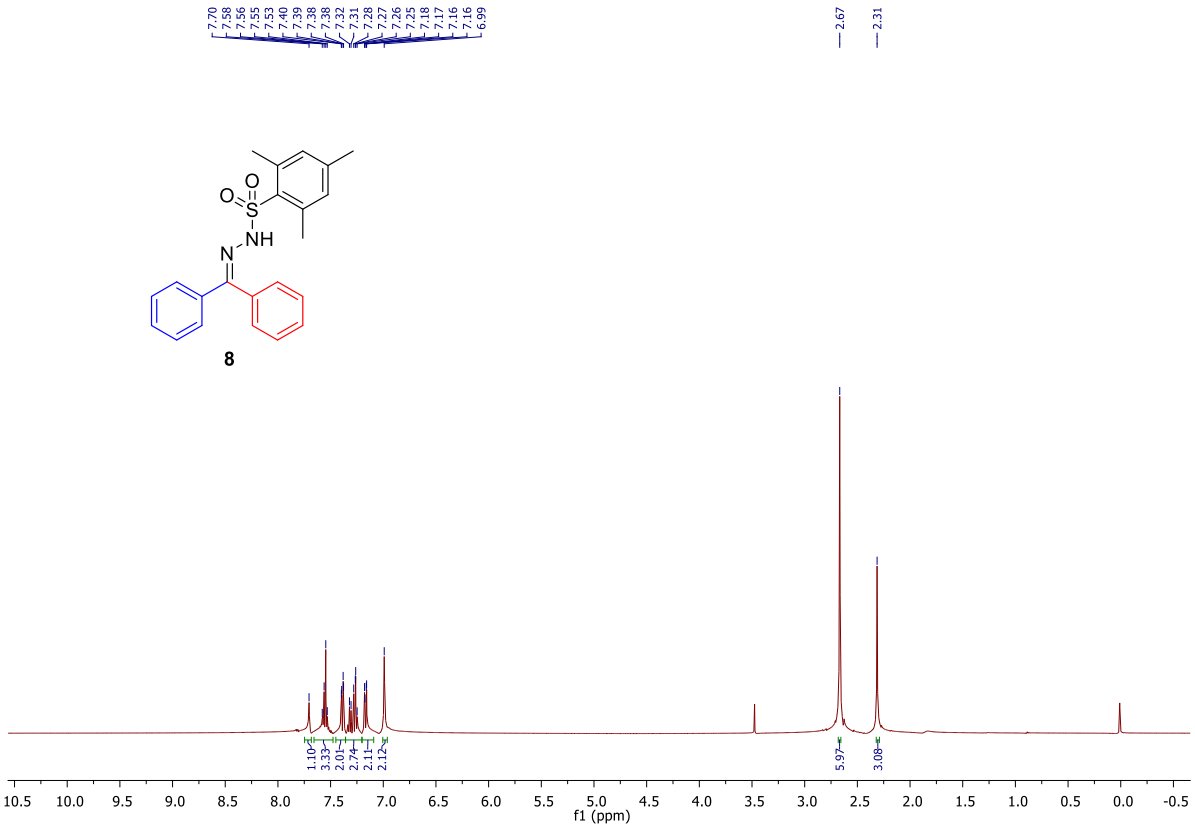


^{13}C{^{1}H} NMR (126 MHz, DMSO-d$_6$, 24 °C)

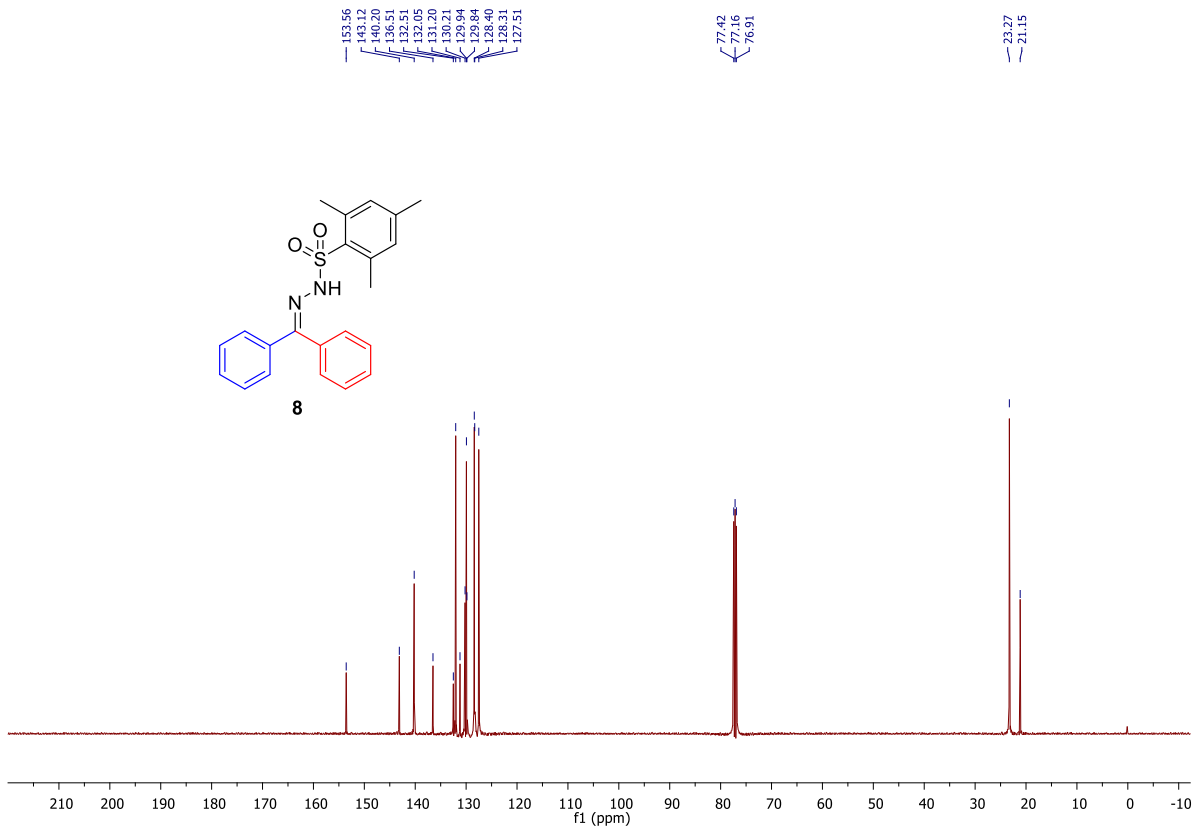


*N'-(*diphenylmethylene)-2,4,6-trimethylbenzenesulfonohydrazide **(8)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

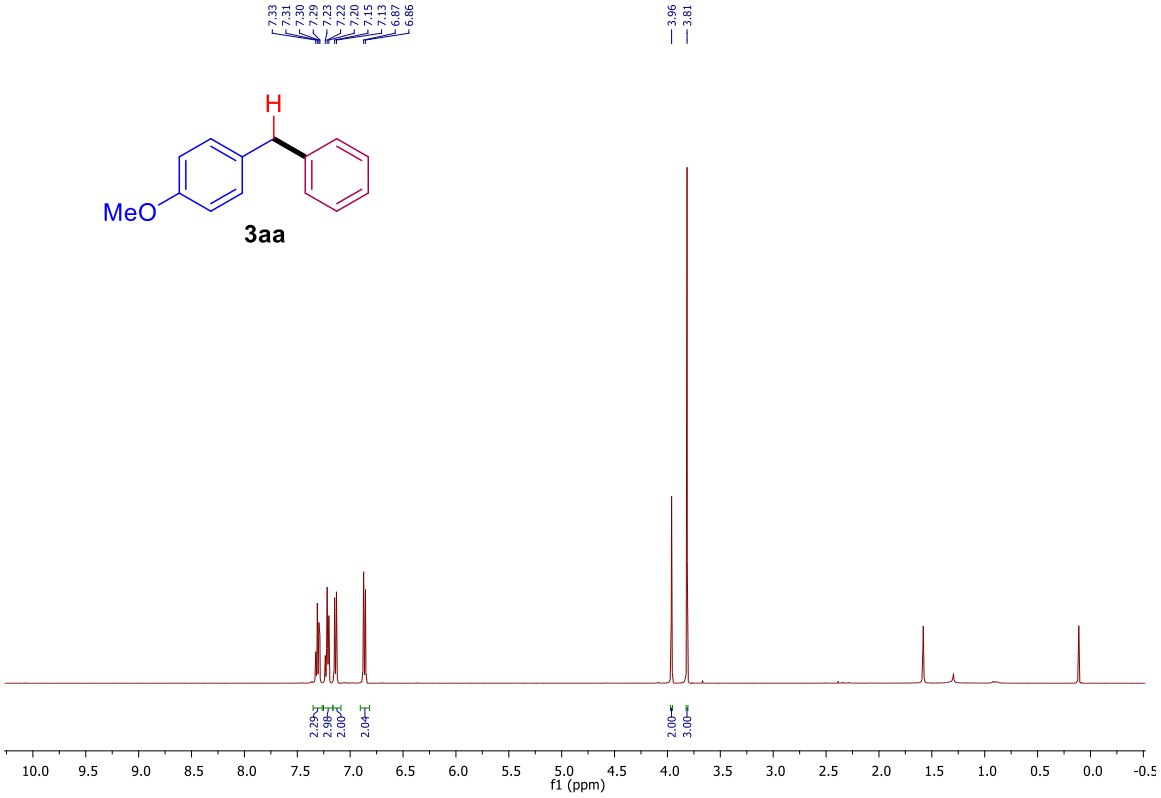


^{13}C {^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

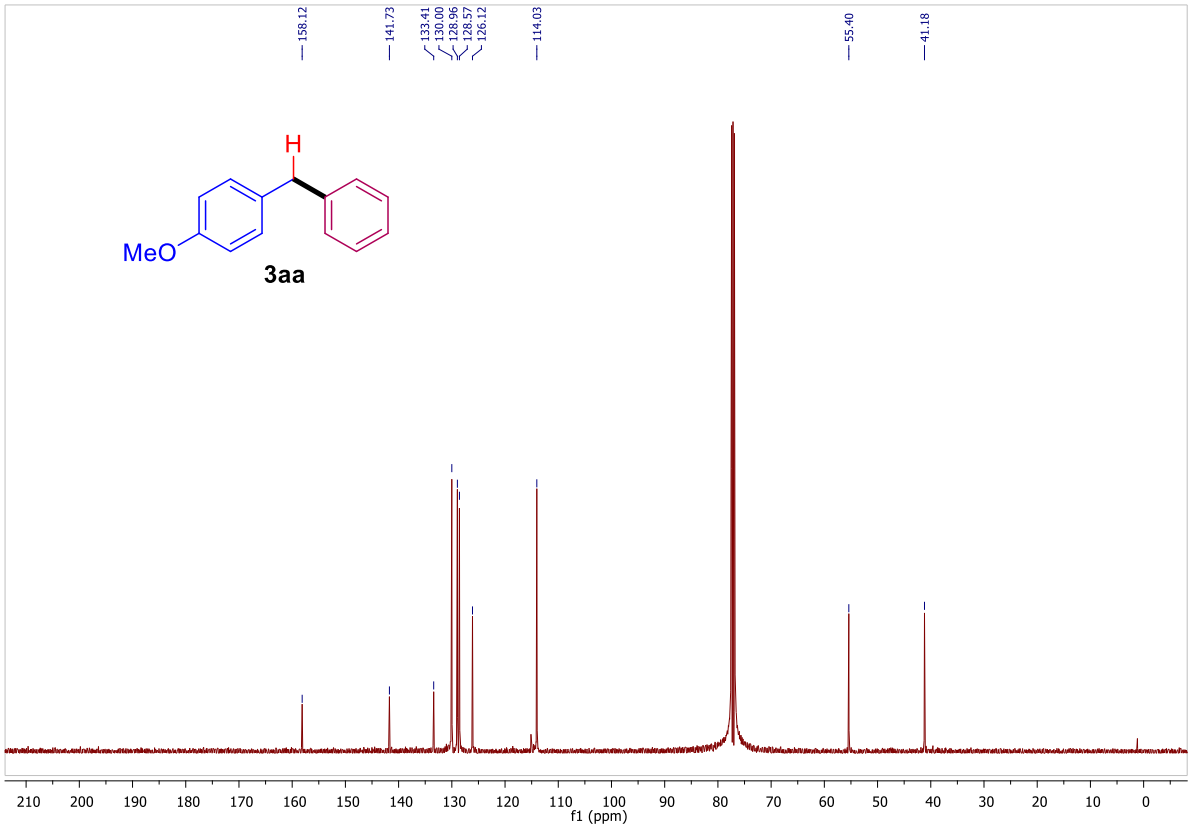


1-Benzyl-4-methoxybenzene **(3aa)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

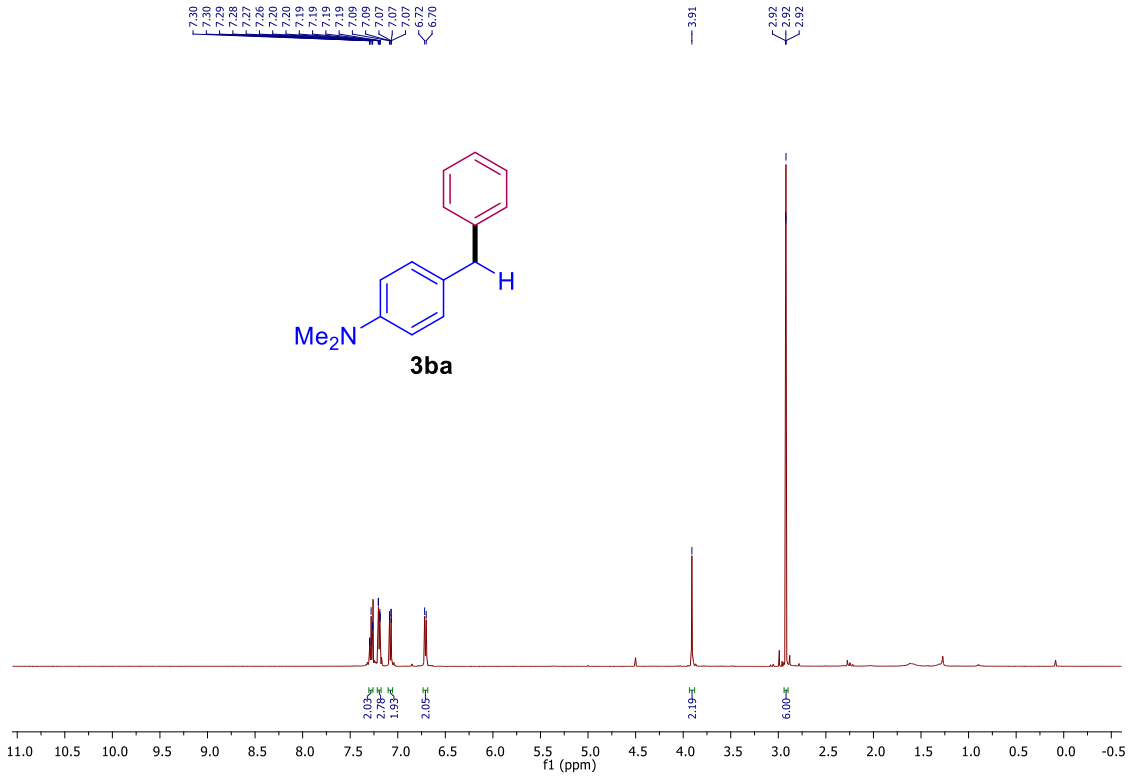


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

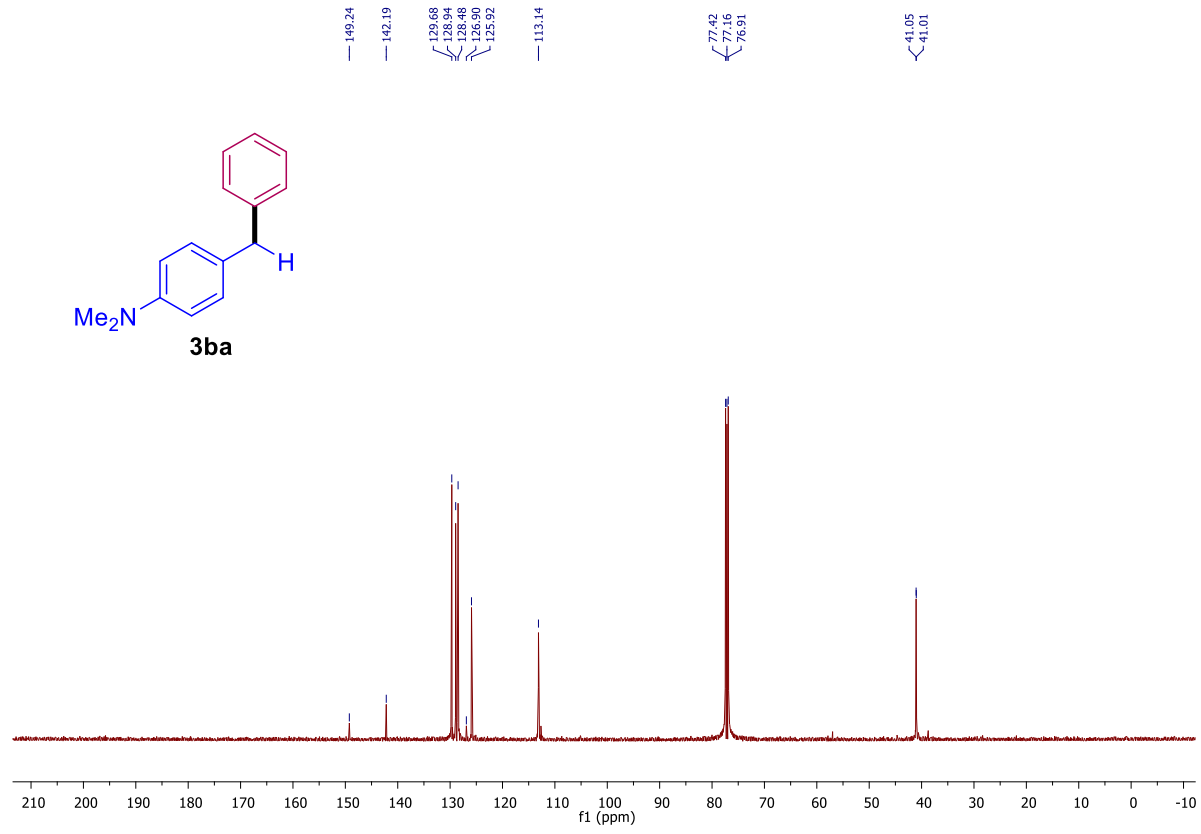


4-Benzyl-N,N-dimethylaniline **(3ba)**

^{1} H NMR (500 MHz, $CDCl_3$, 24 °C)

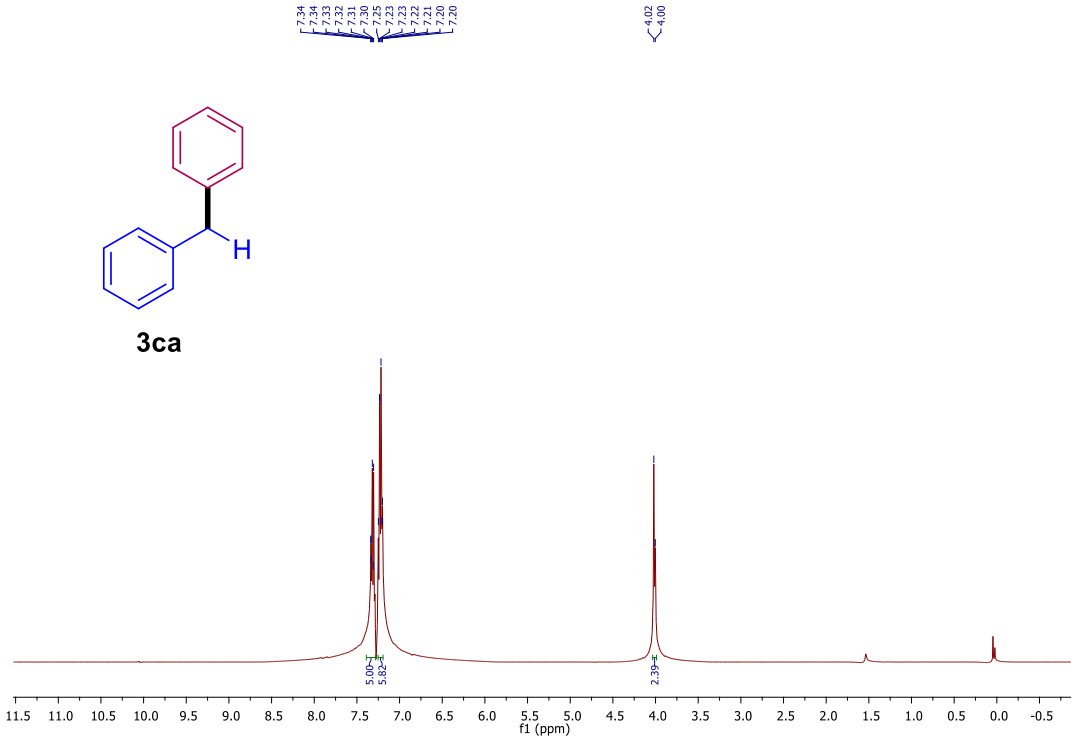


^{13} C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

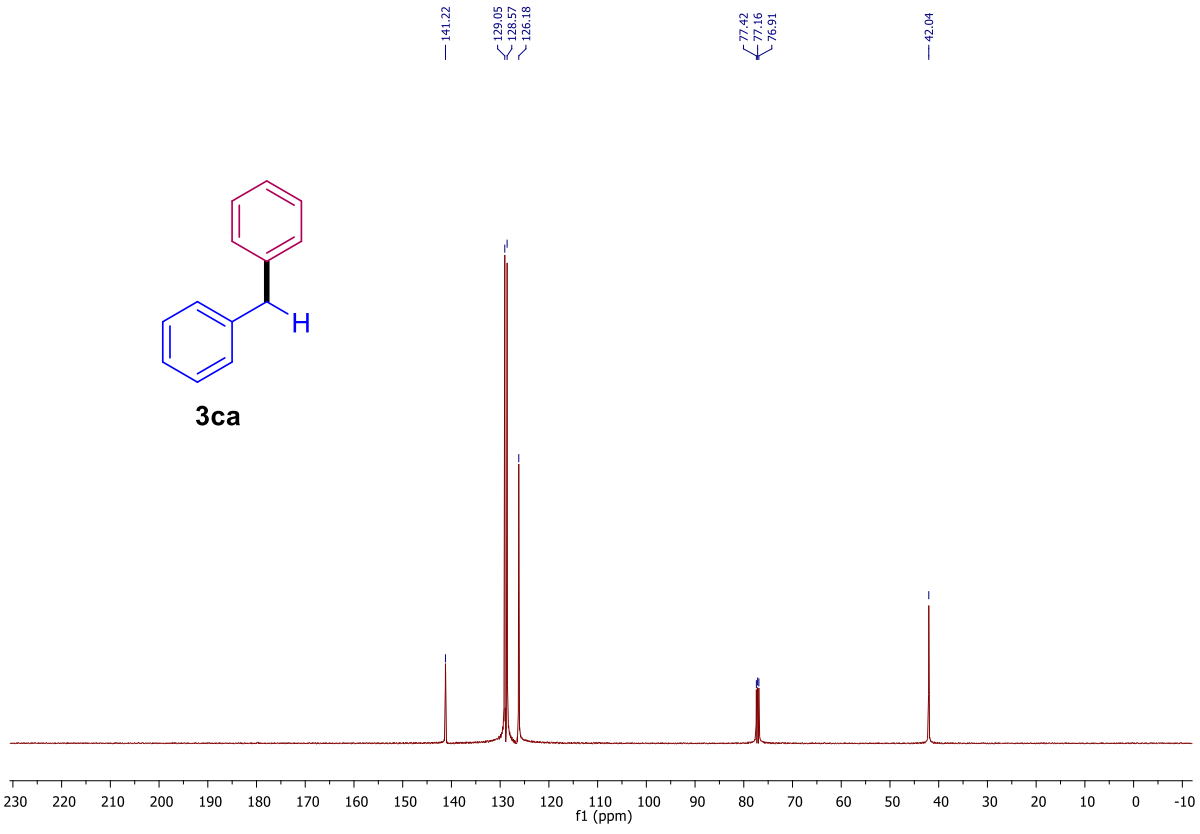


Diphenylmethane **(3ca)**

^{1} H NMR (500 MHz, $CDCl_3$, 24 °C)

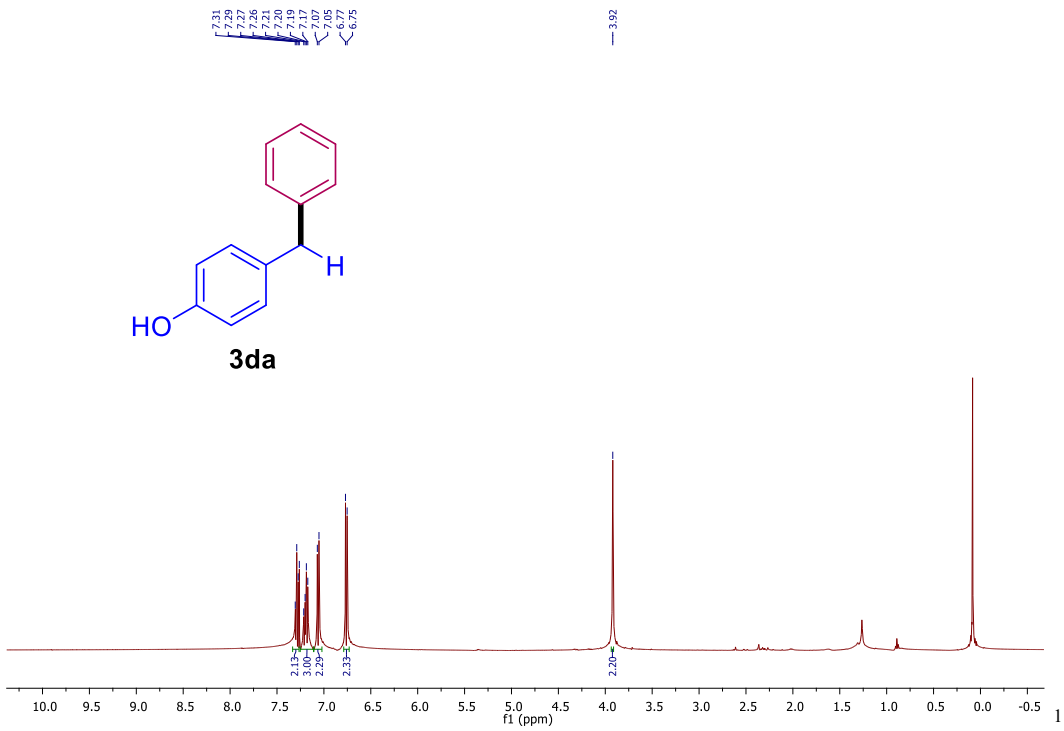


^{13} C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

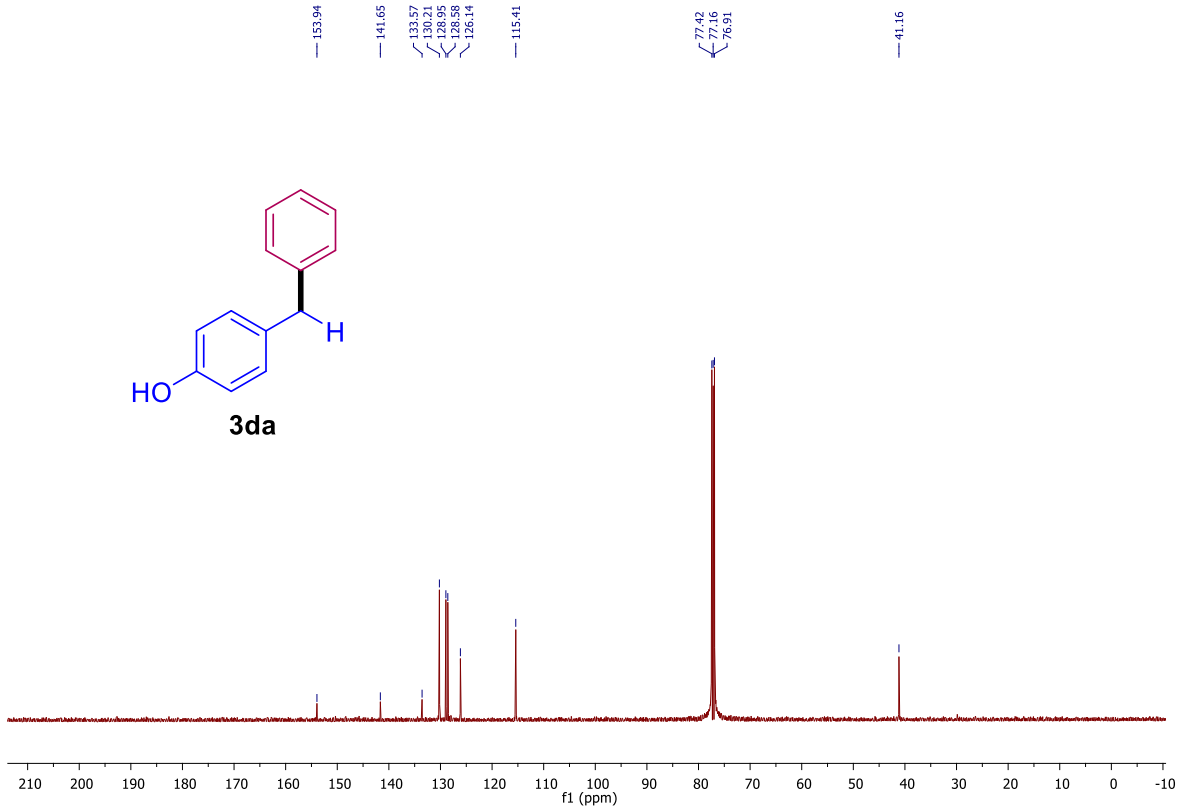


Benzylphenol **(3da)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

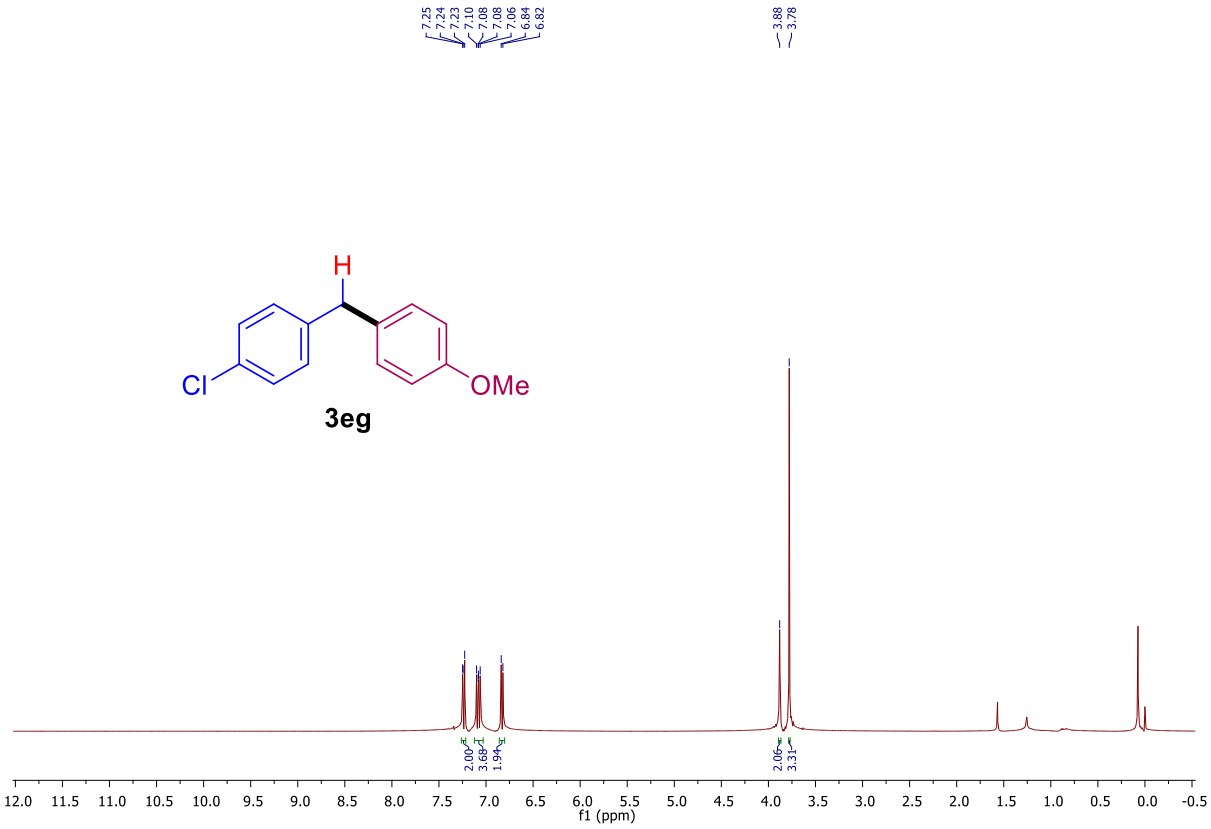


^{3}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

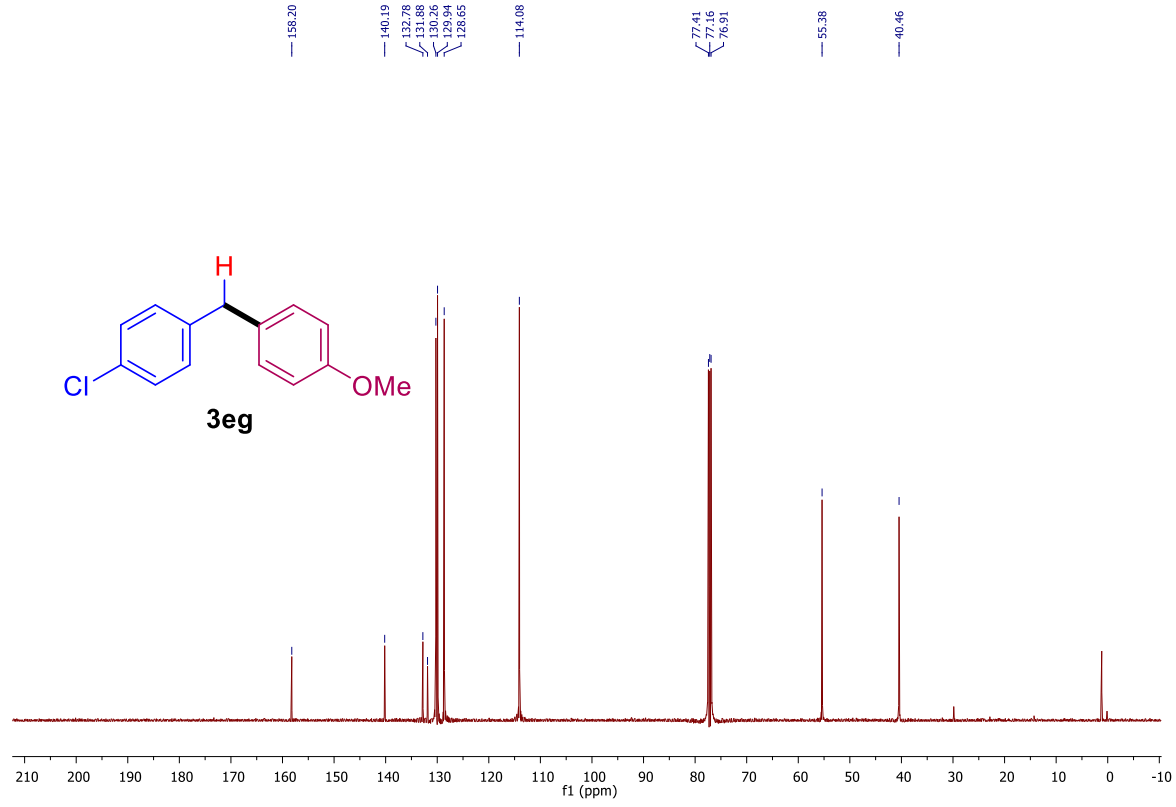


1-Chloro-4-(4-methoxybenzyl)benzene **(3eg)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

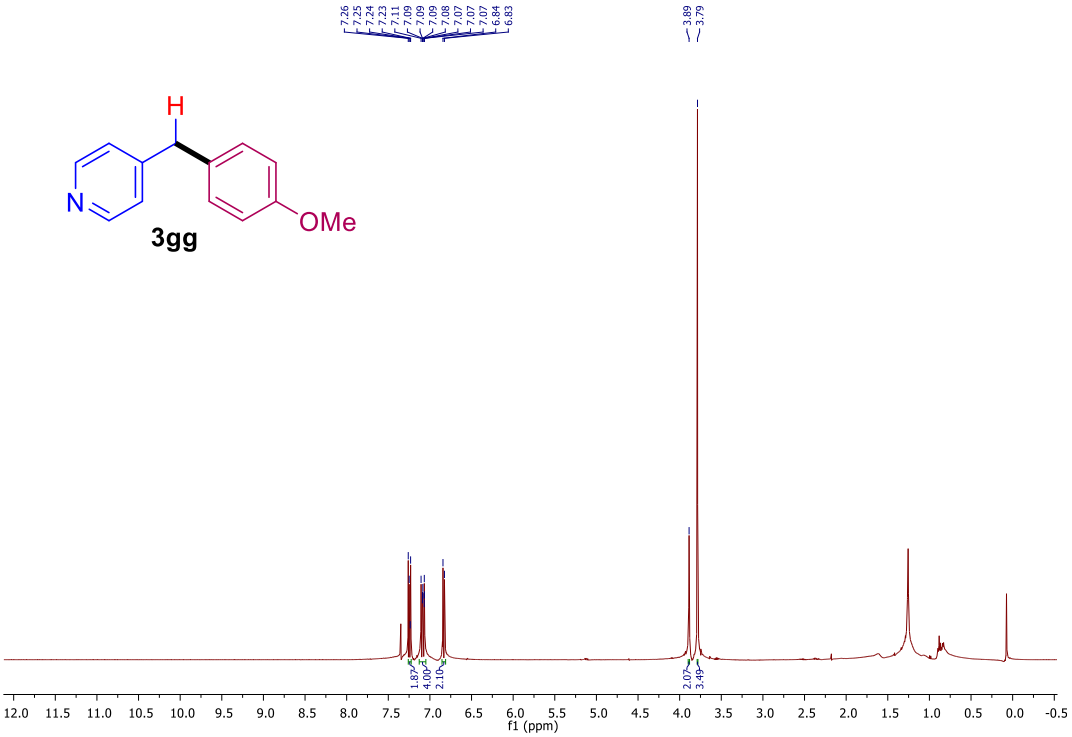


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

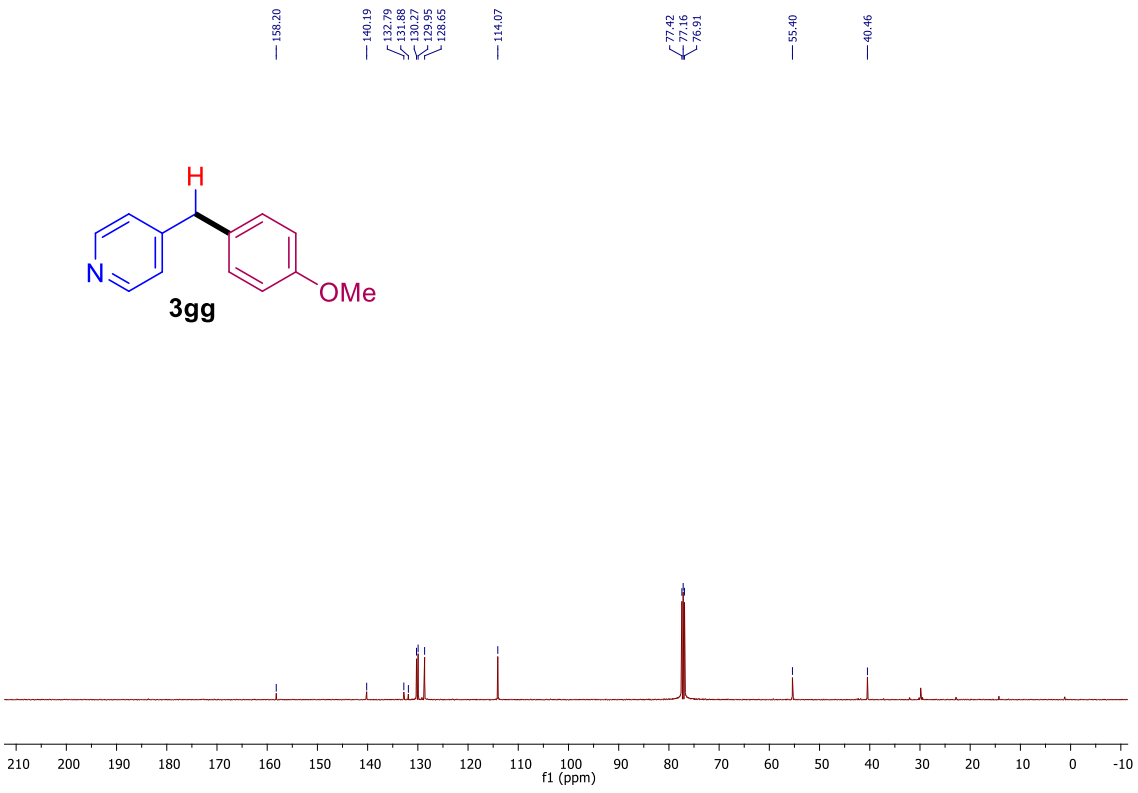


4-(4-Methoxybenzyl)pyridine **(3gg)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

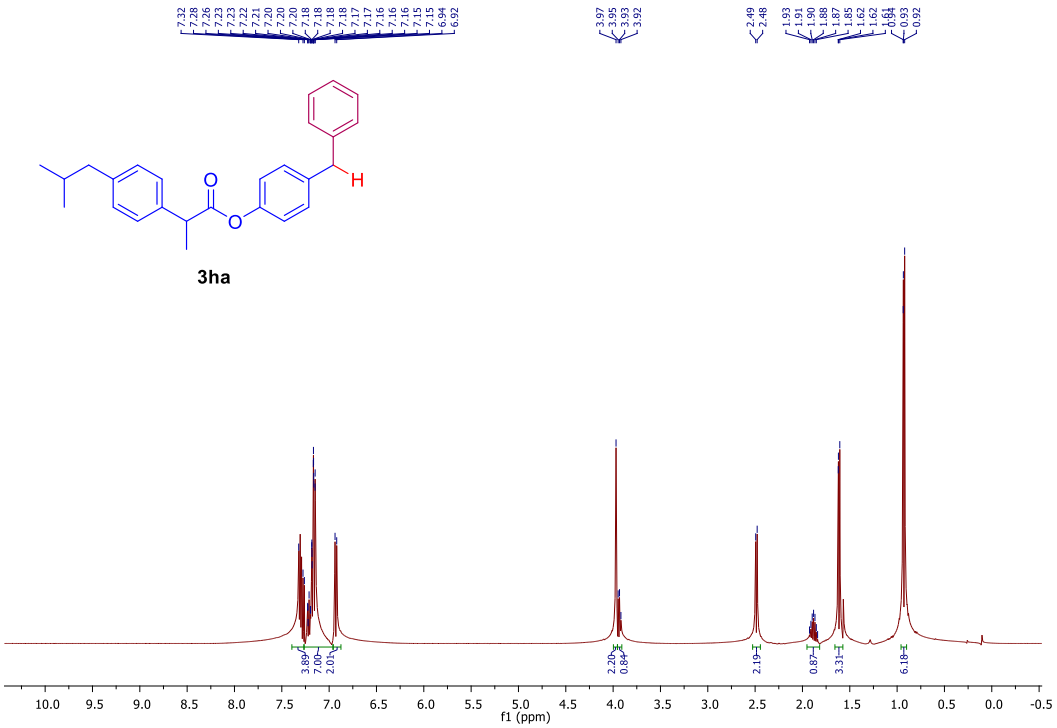


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

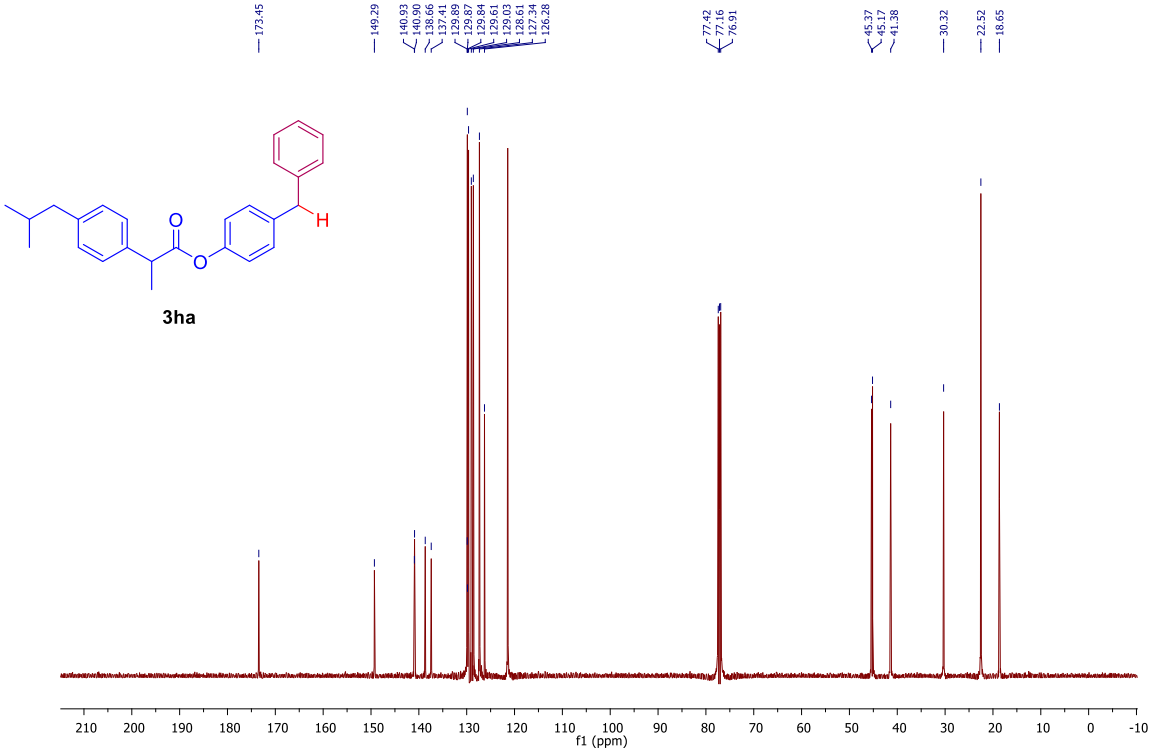


4-Benzylphenyl 2-(4-isobutylphenyl)propanoate **(3ha)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

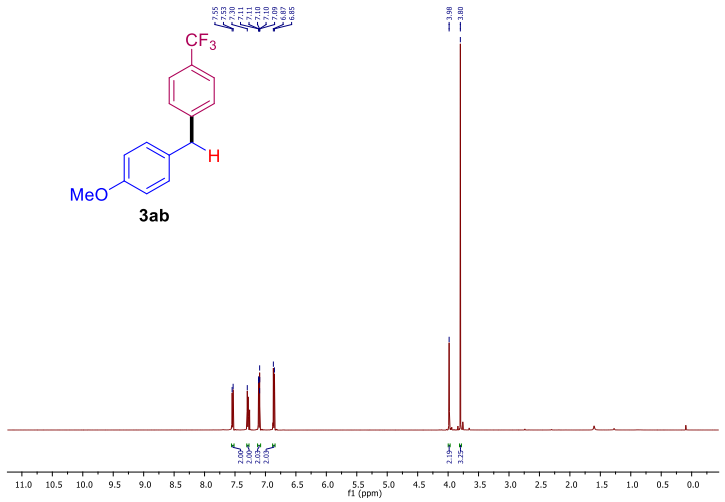


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

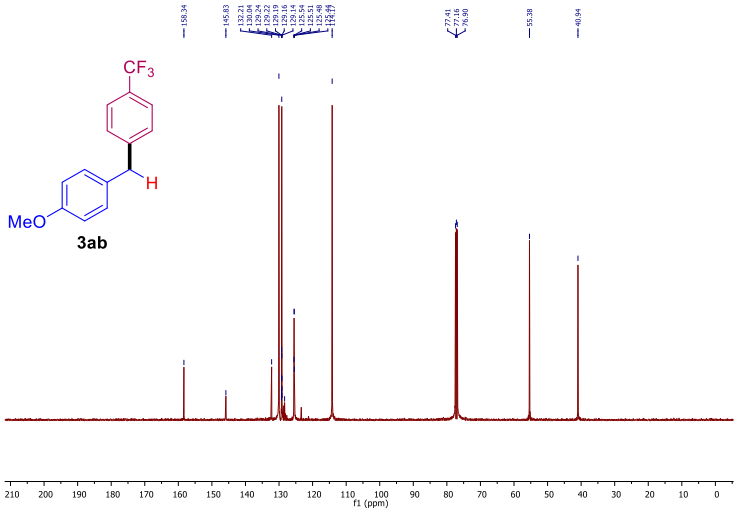


1-Methoxy-4-(4-(trifluoromethyl)benzyl)benzene **(3ab)**

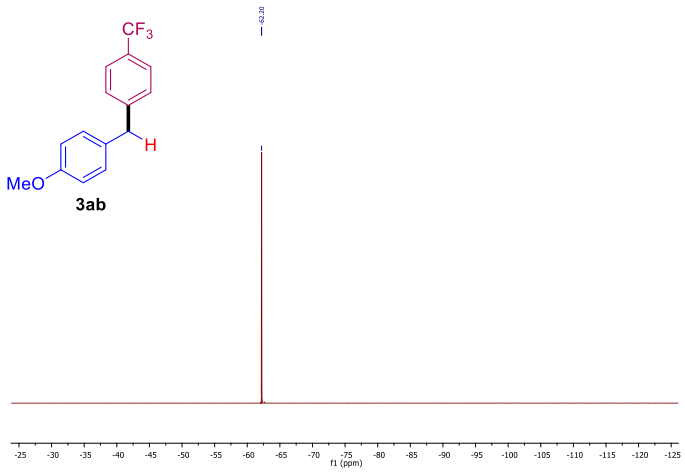
^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)



^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

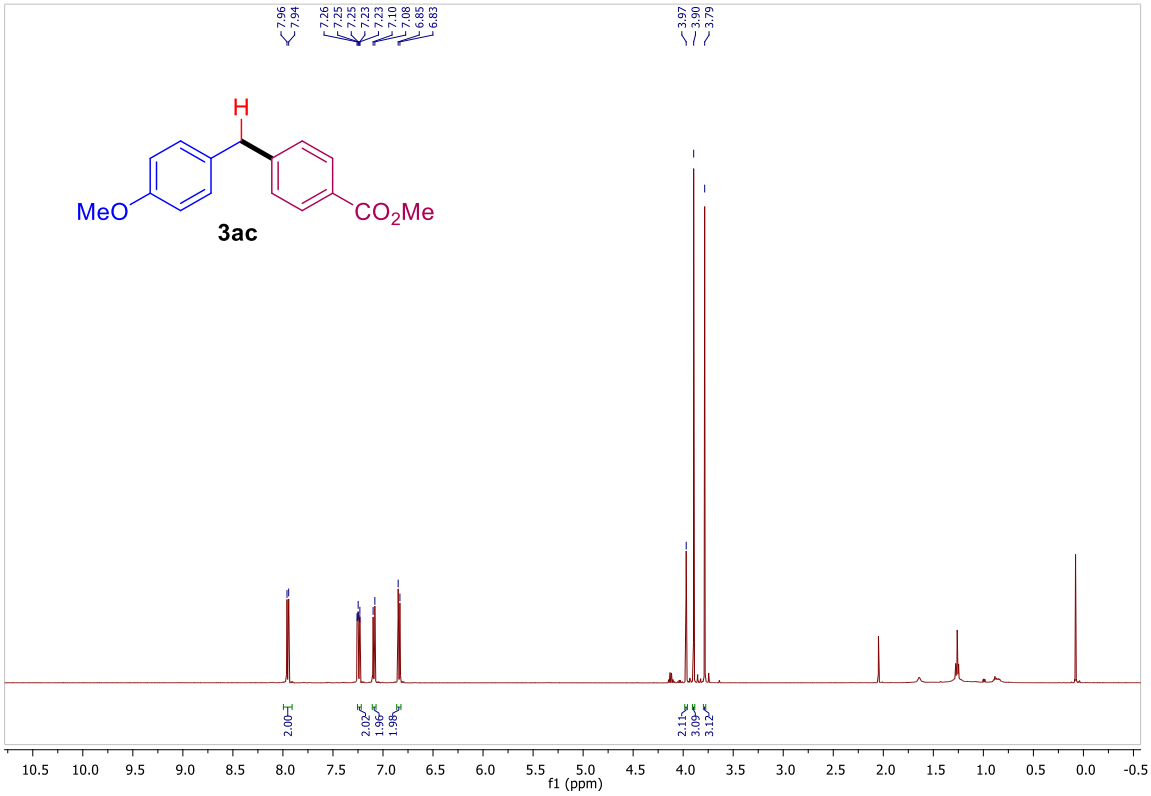


^{19}F NMR (471 MHz, $CDCl_3$, 24 °C)

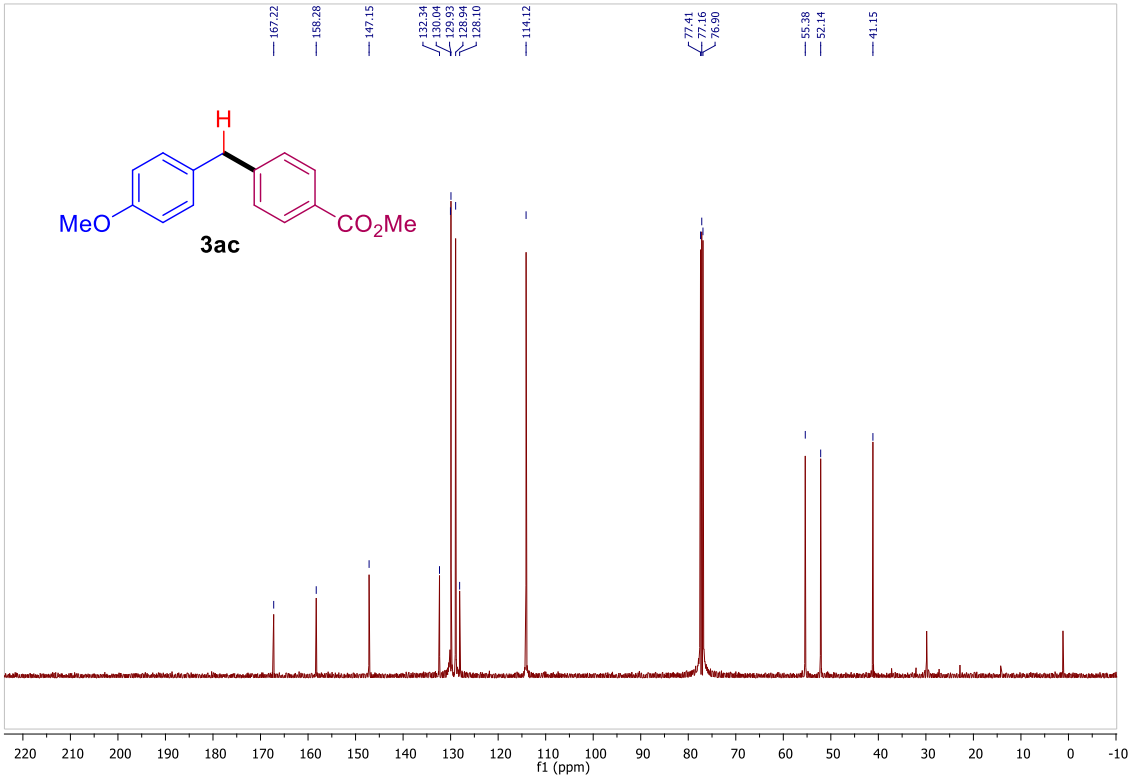


Methyl 4-(4-methoxybenzyl)benzoate **(3ac)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

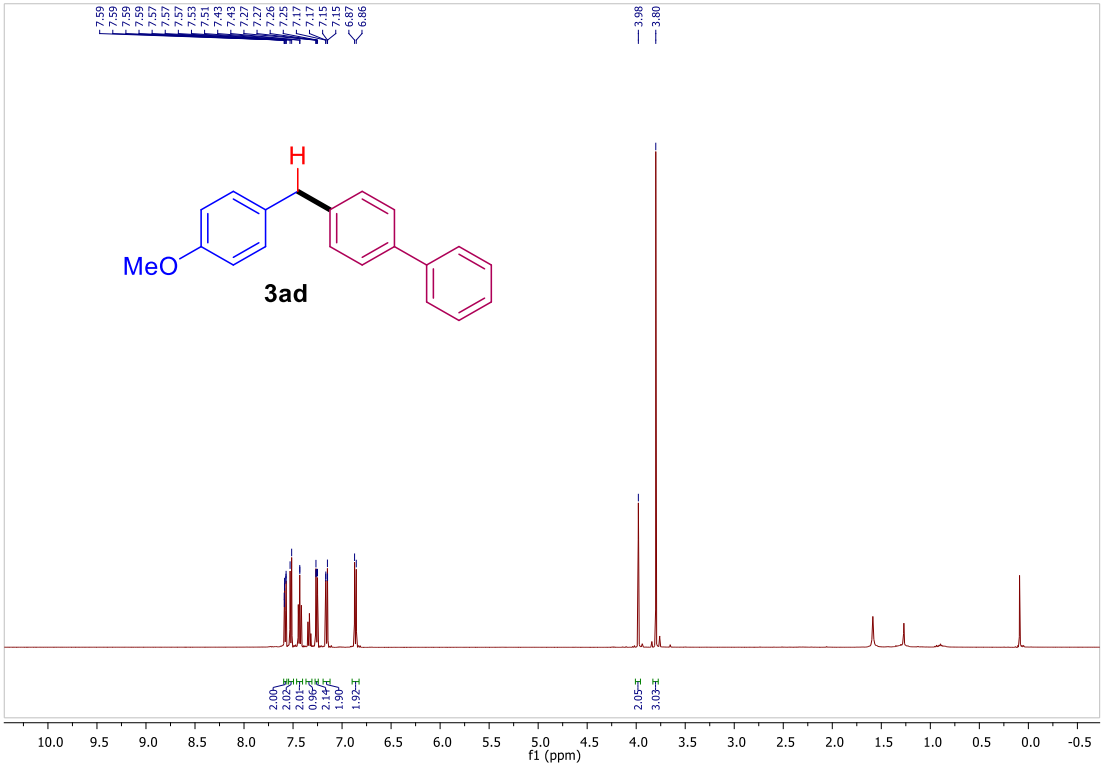


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C

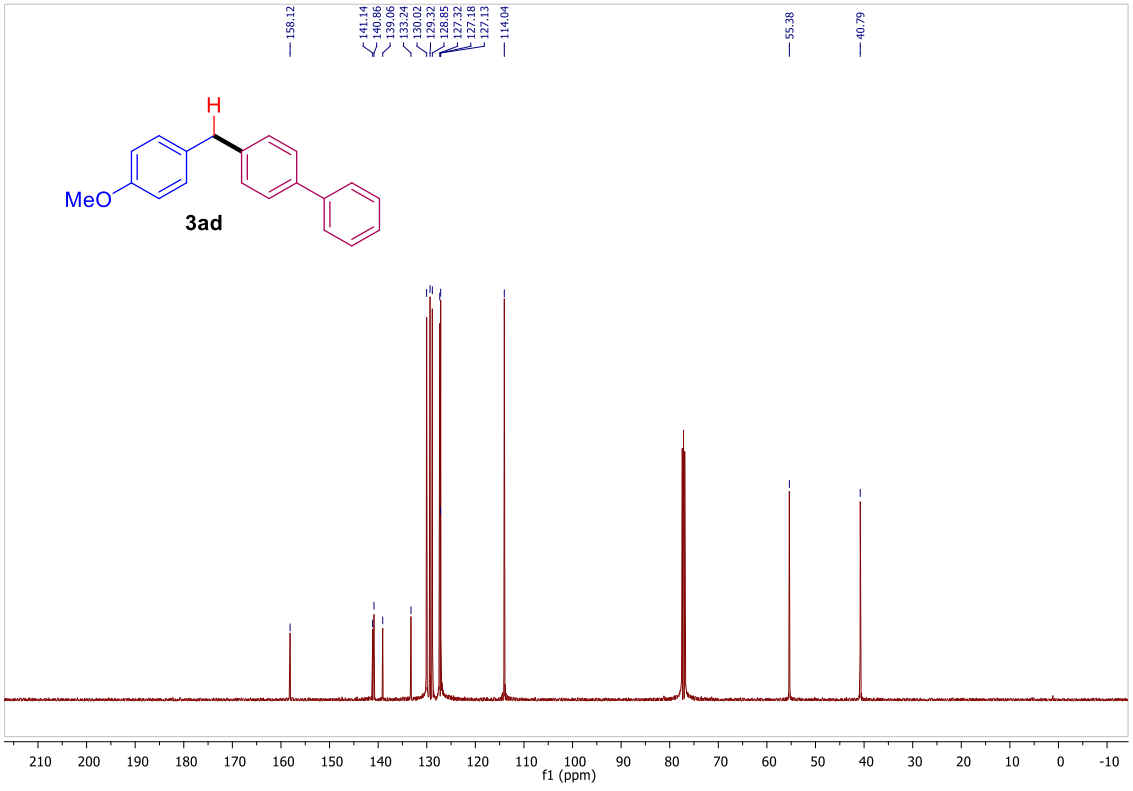


4-(4-Methoxybenzyl)-1,1'-biphenyl **(3ad)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

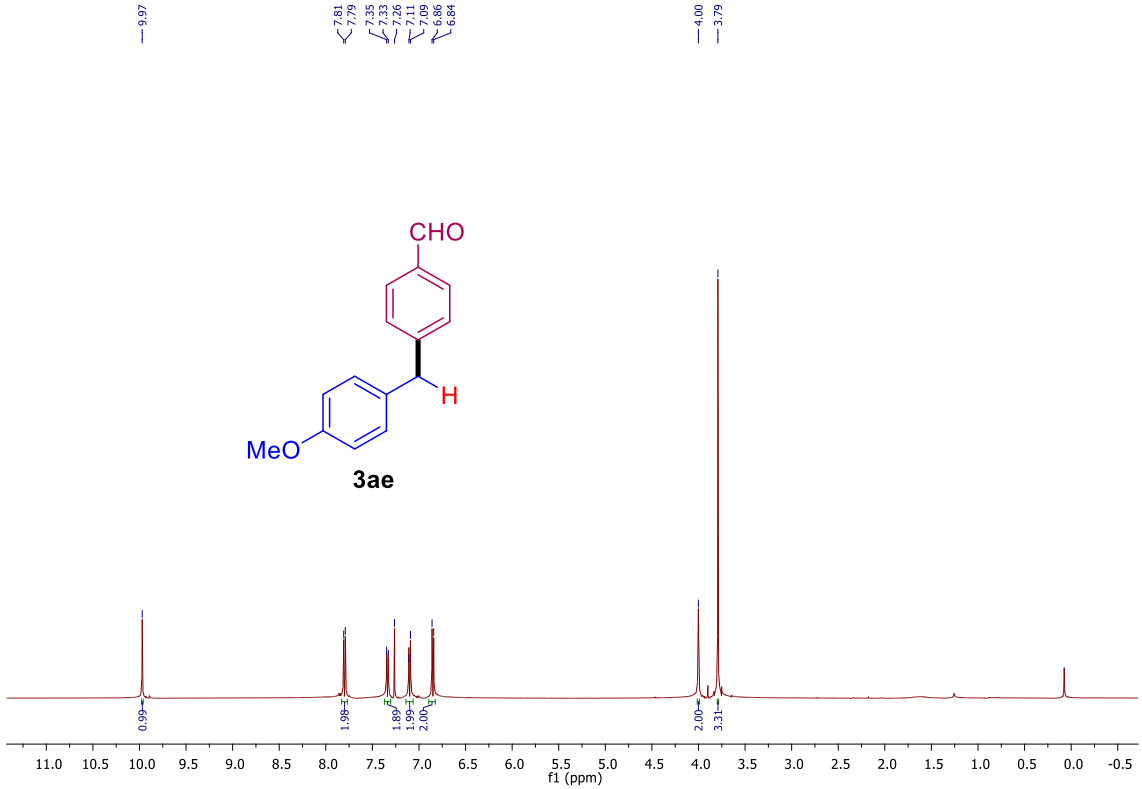


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

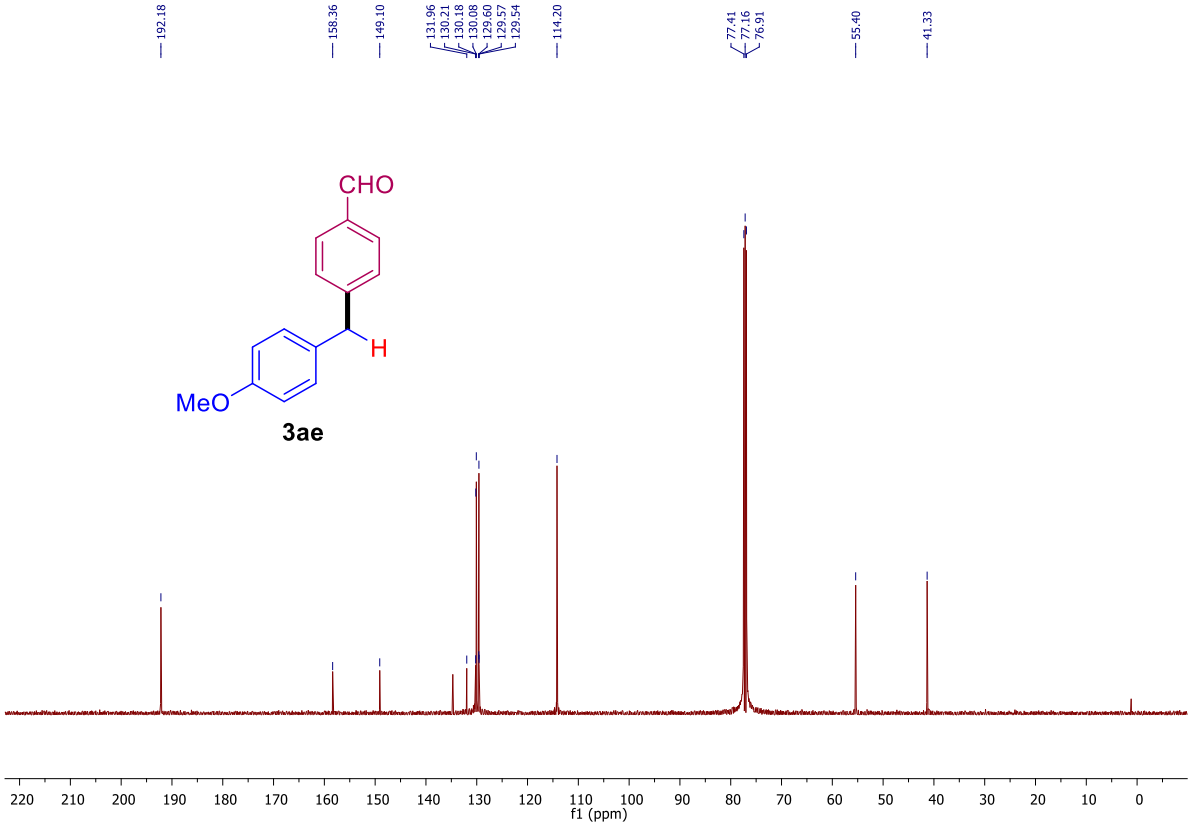


4-(4-methoxybenzyl)benzaldehyde **(3ae)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

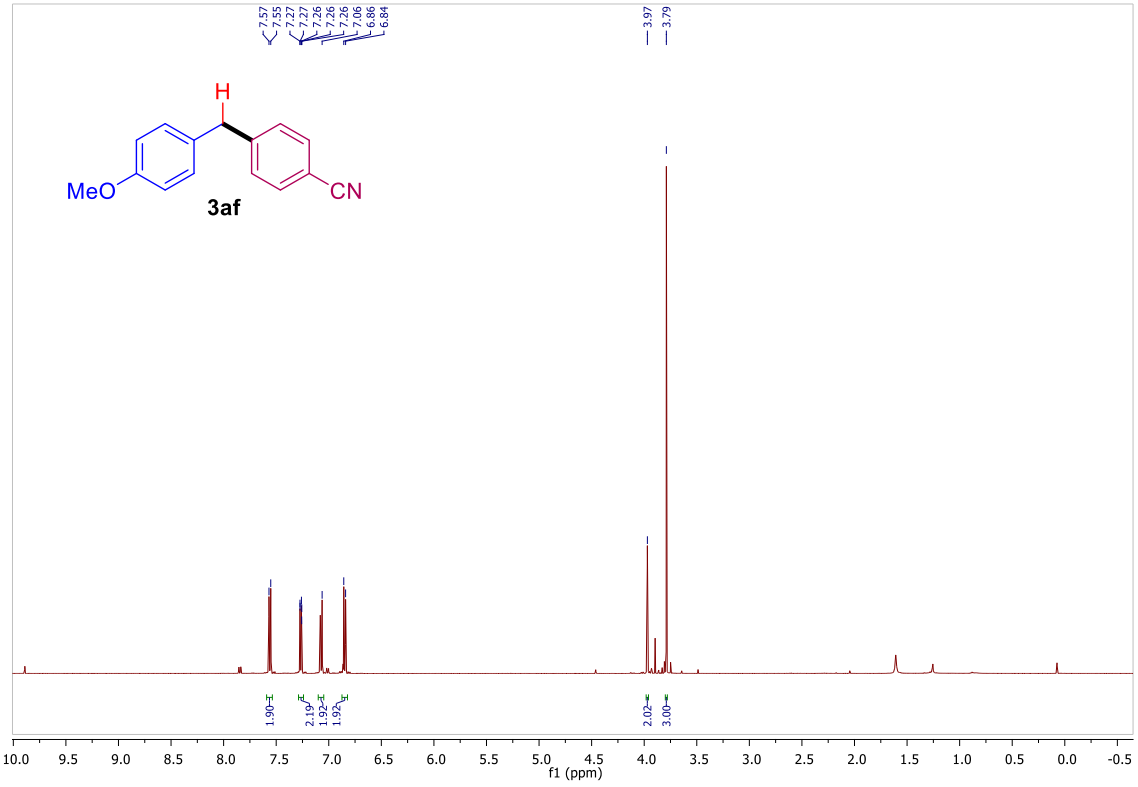


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

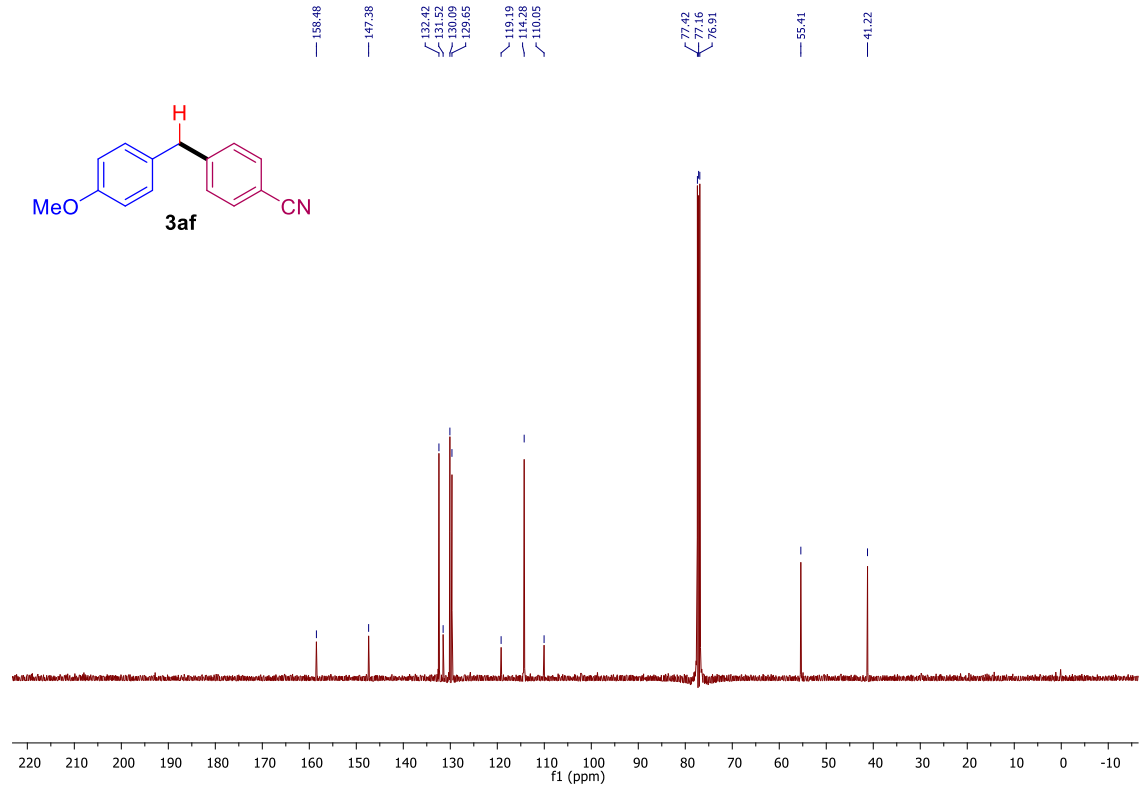


4-(4-Methoxybenzyl) benzonitrile **(3af)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

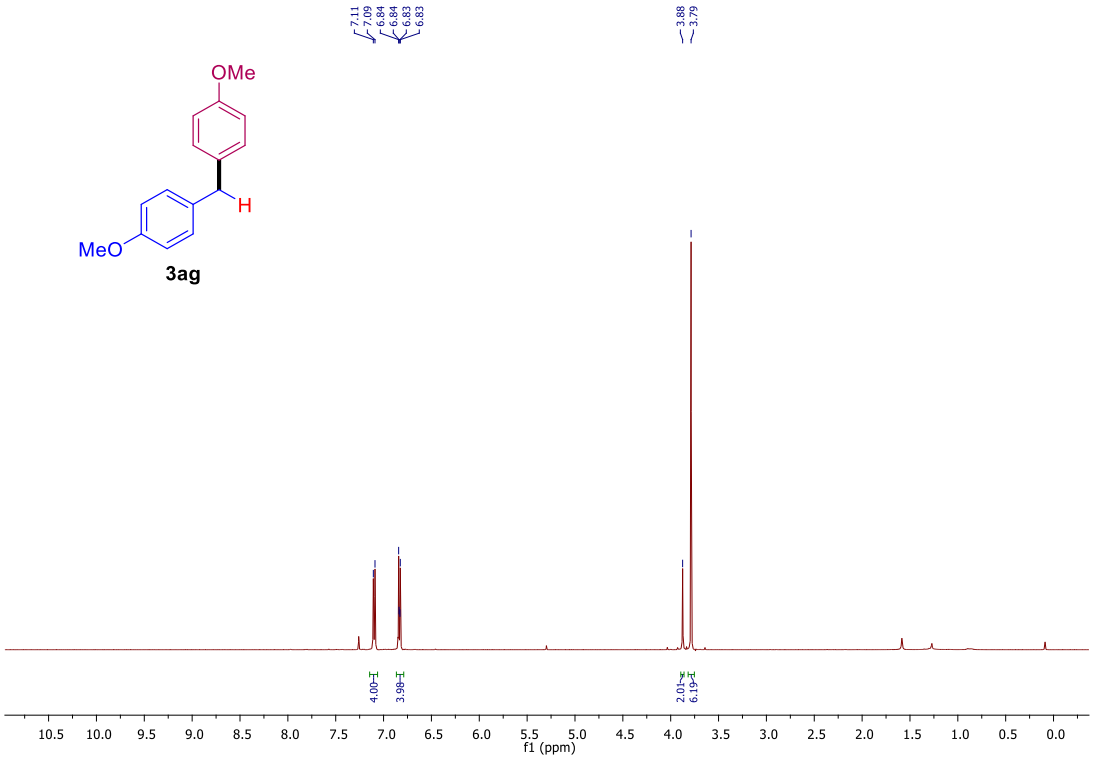


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

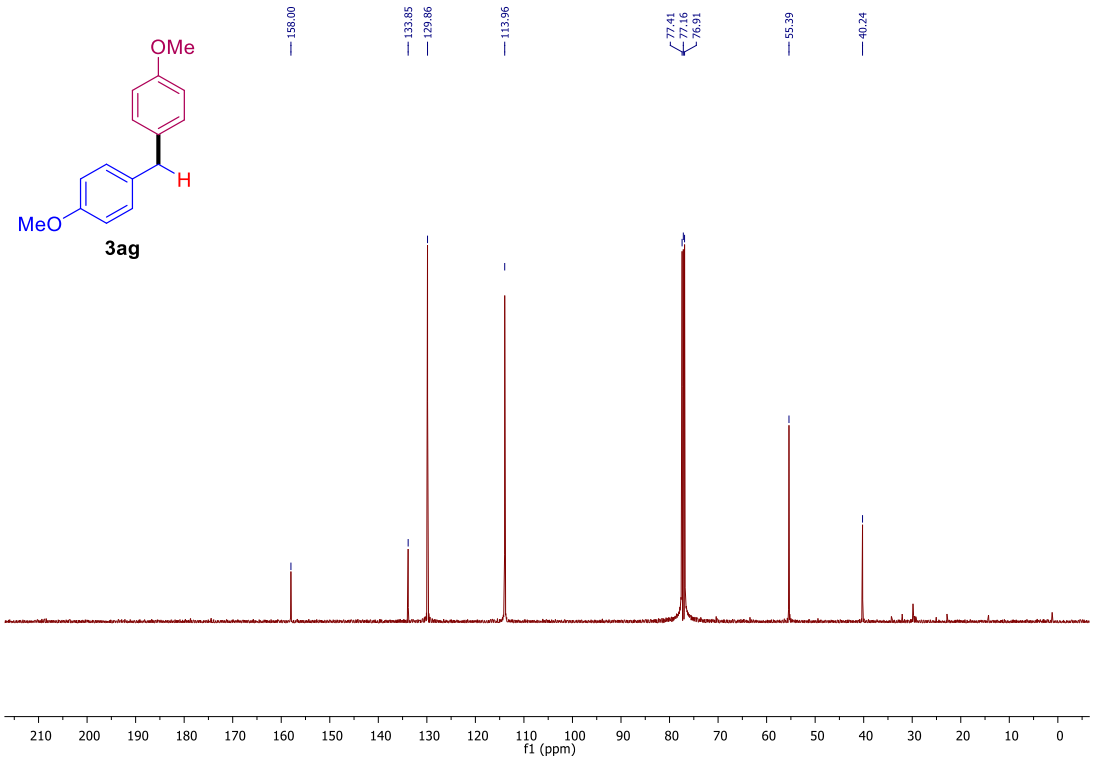


Bis(4-methoxyphenyl) methane **(3ag)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

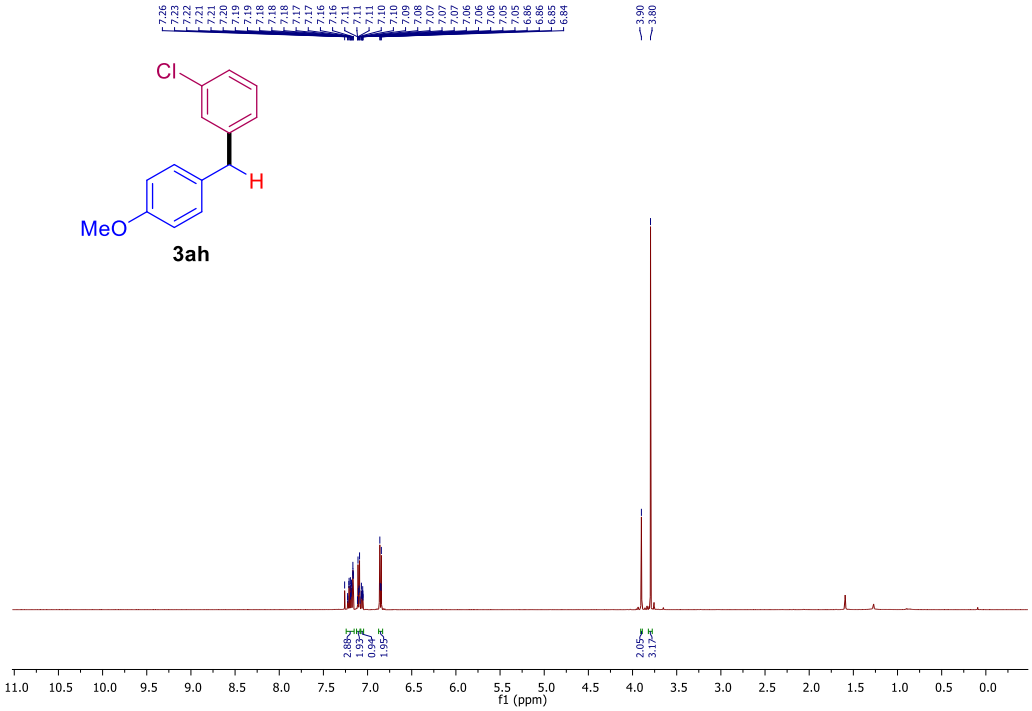


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

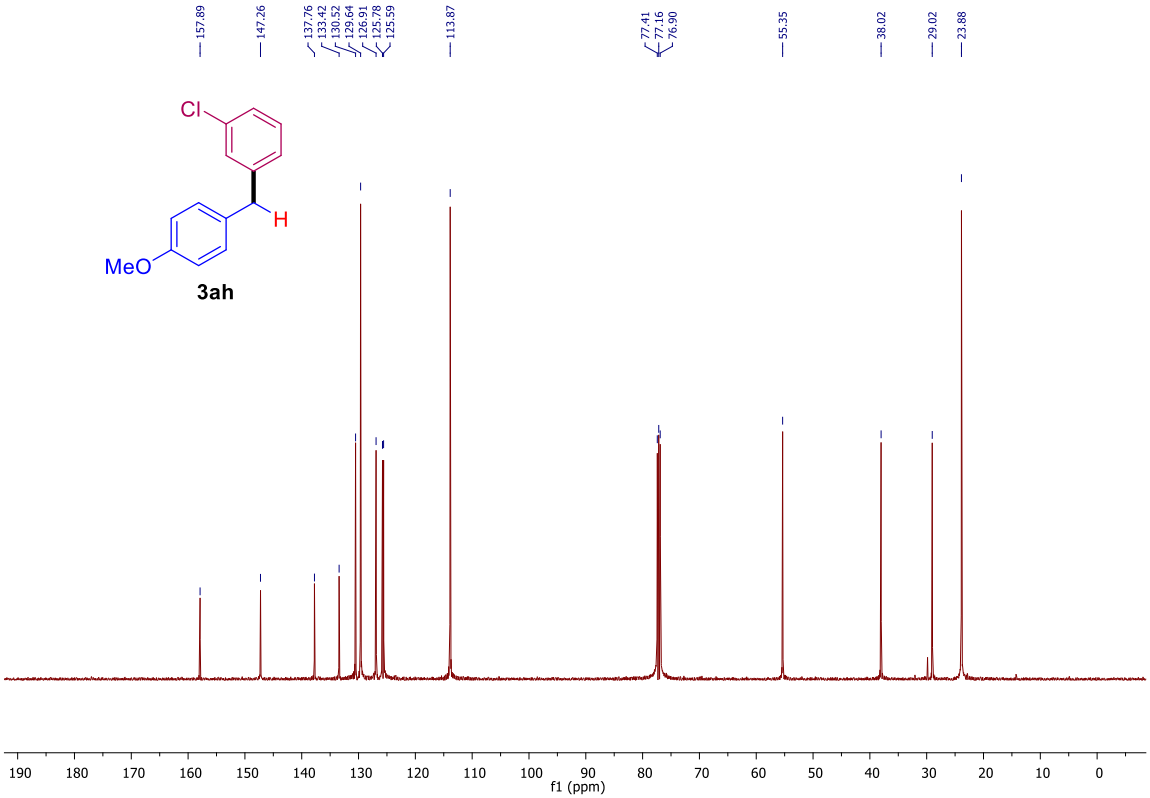


1-Chloro-3-(4-methoxybenzyl) benzene **(3ah)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

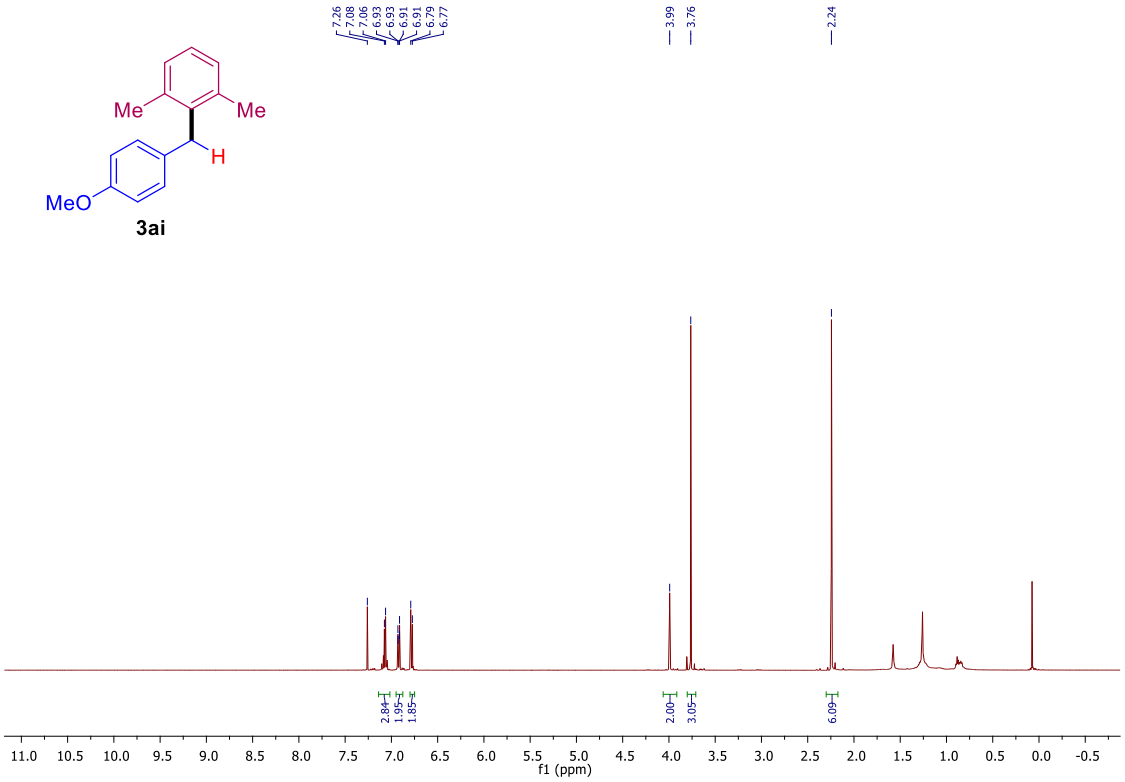


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

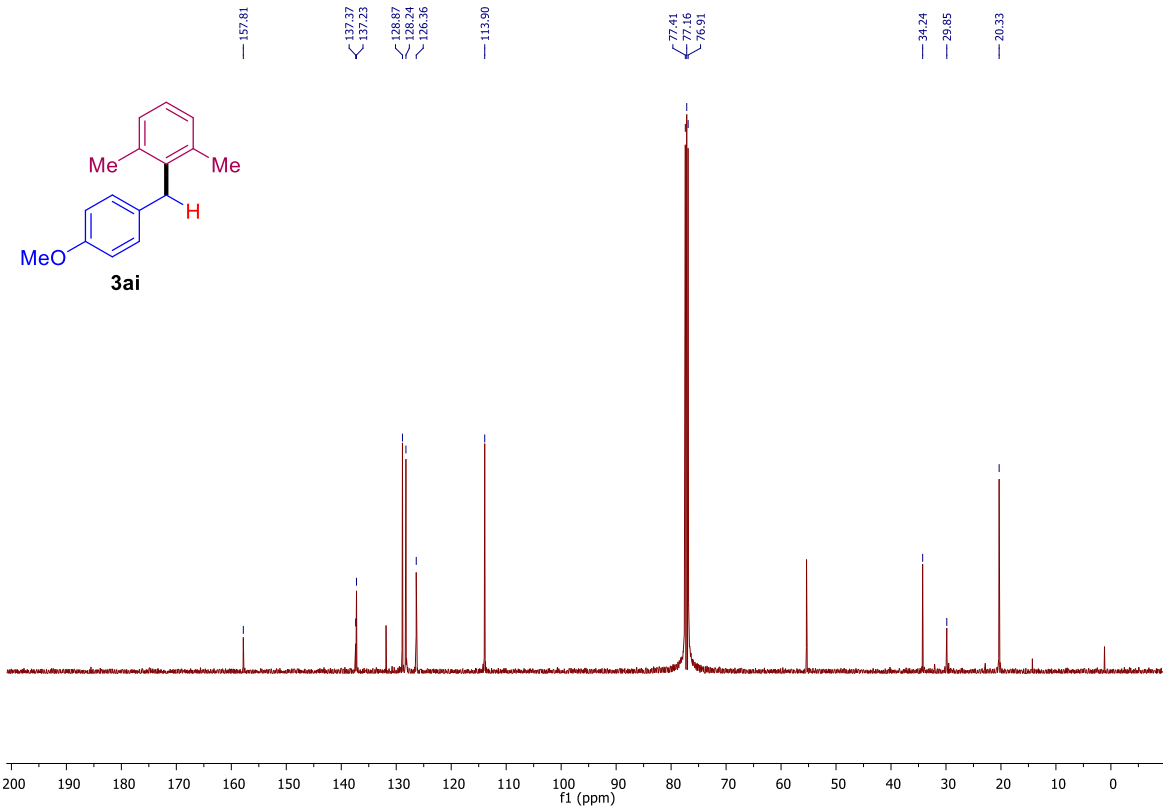


2-(4-Methoxybenzyl)-1,3-dimethylbenzene **(3ai)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

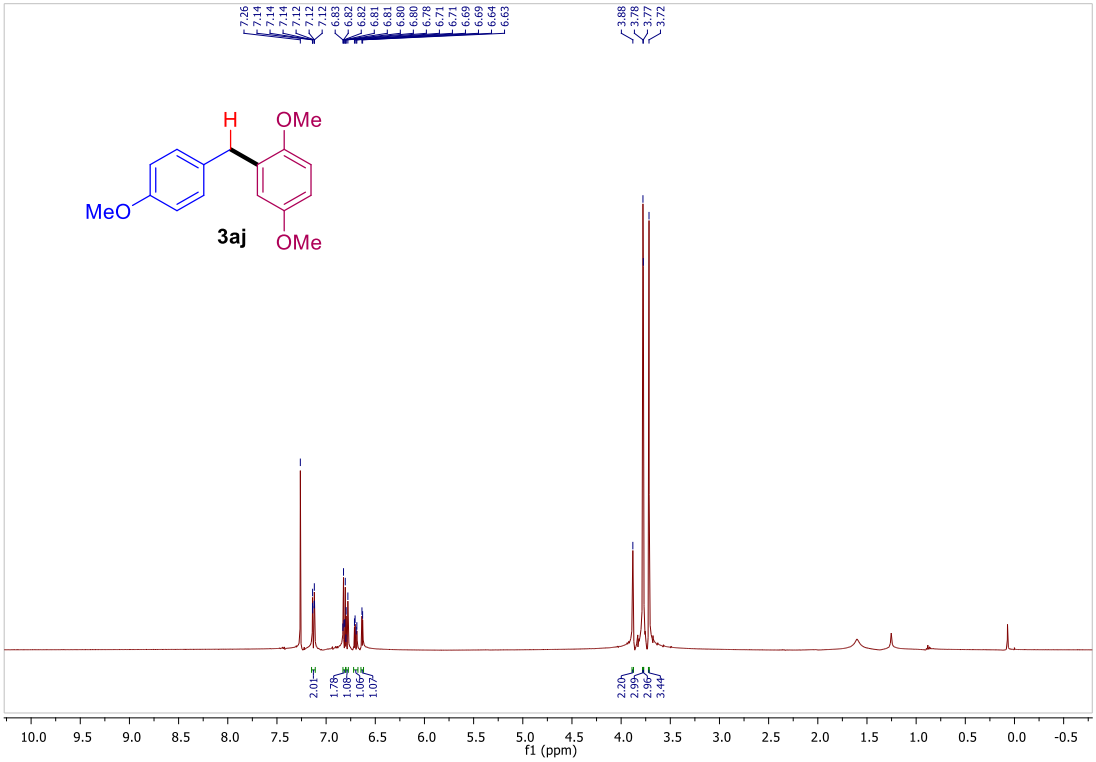


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

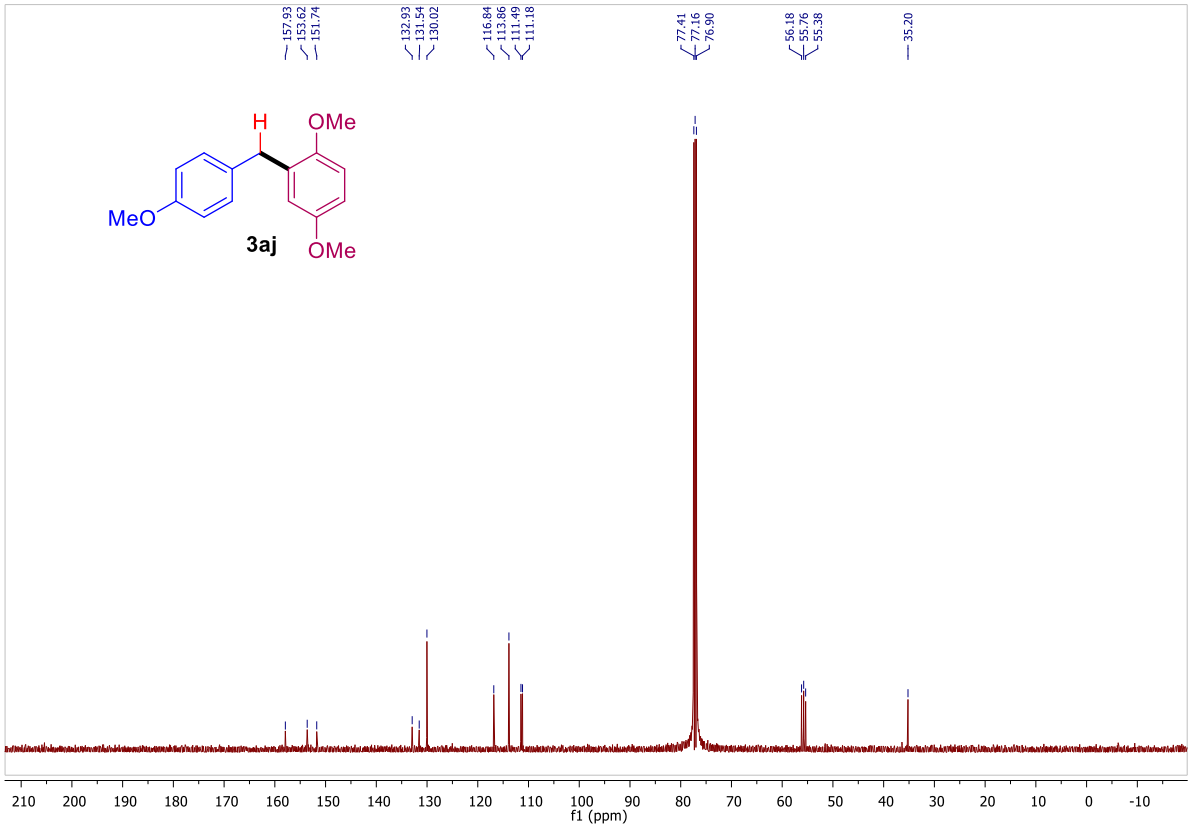


1,4-Dimethoxy-2-(4-methoxybenzyl) benzene **(3aj)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

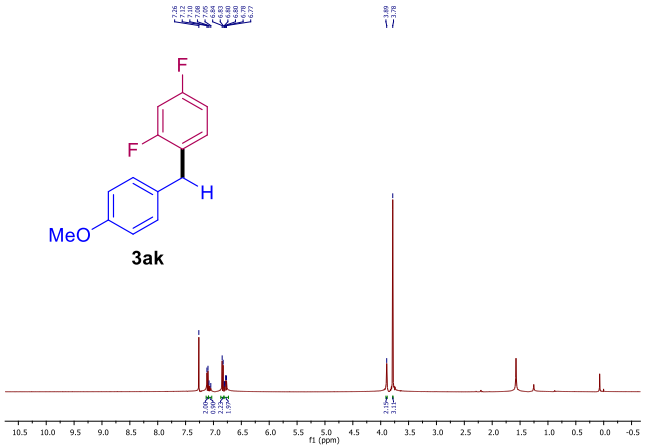


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

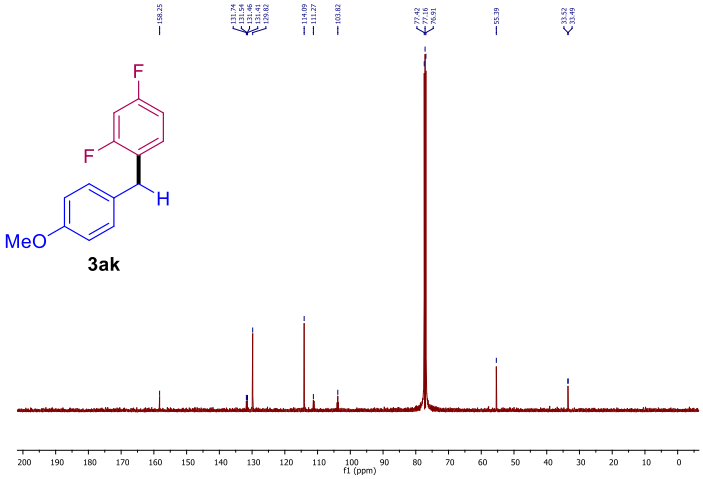


2,4-Difluoro-1-(4-methoxybenzyl) benzene **(3ak)**

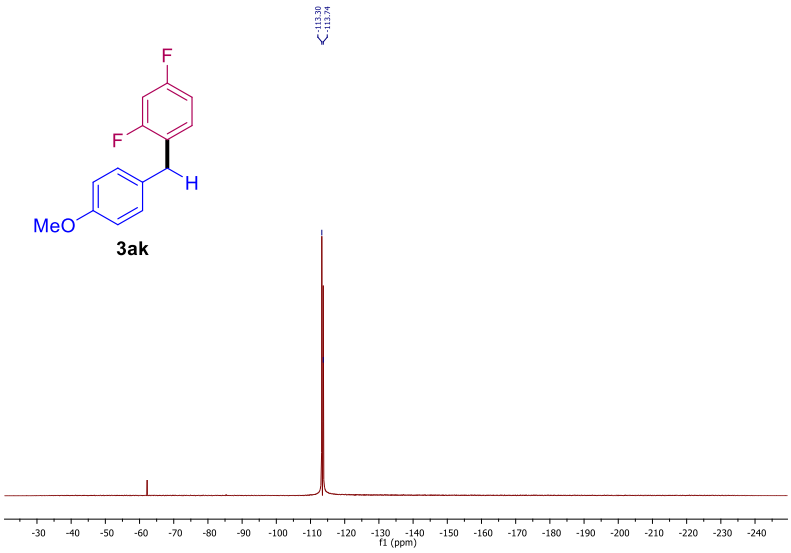
^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)



^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

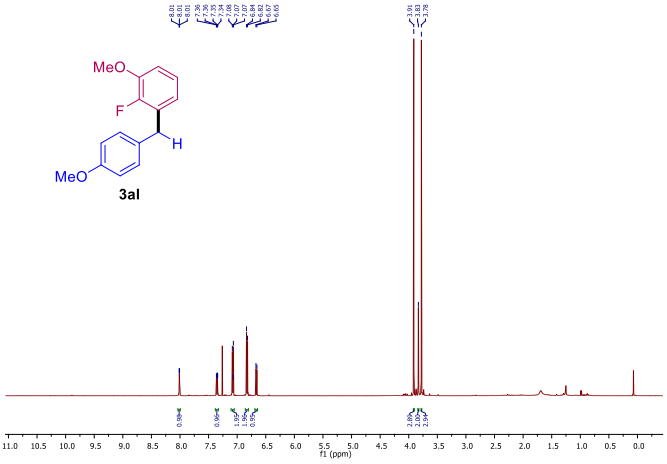


^{19}F NMR (471 MHz, $CDCl_3$, 24 °C)

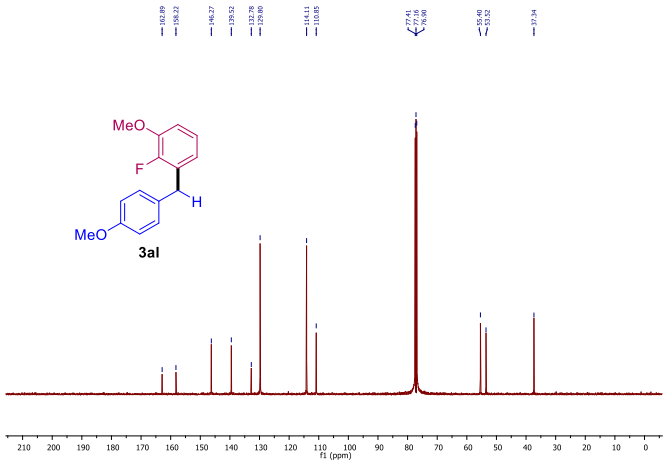


2-Fluoro-1-methoxy-3-(4-methoxybenzyl) benzene **(3al)**

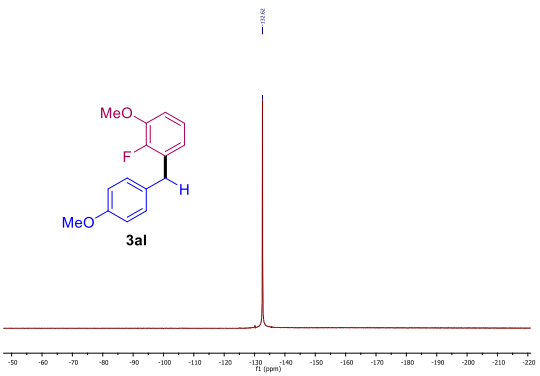
^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)



$^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$, 24°C)

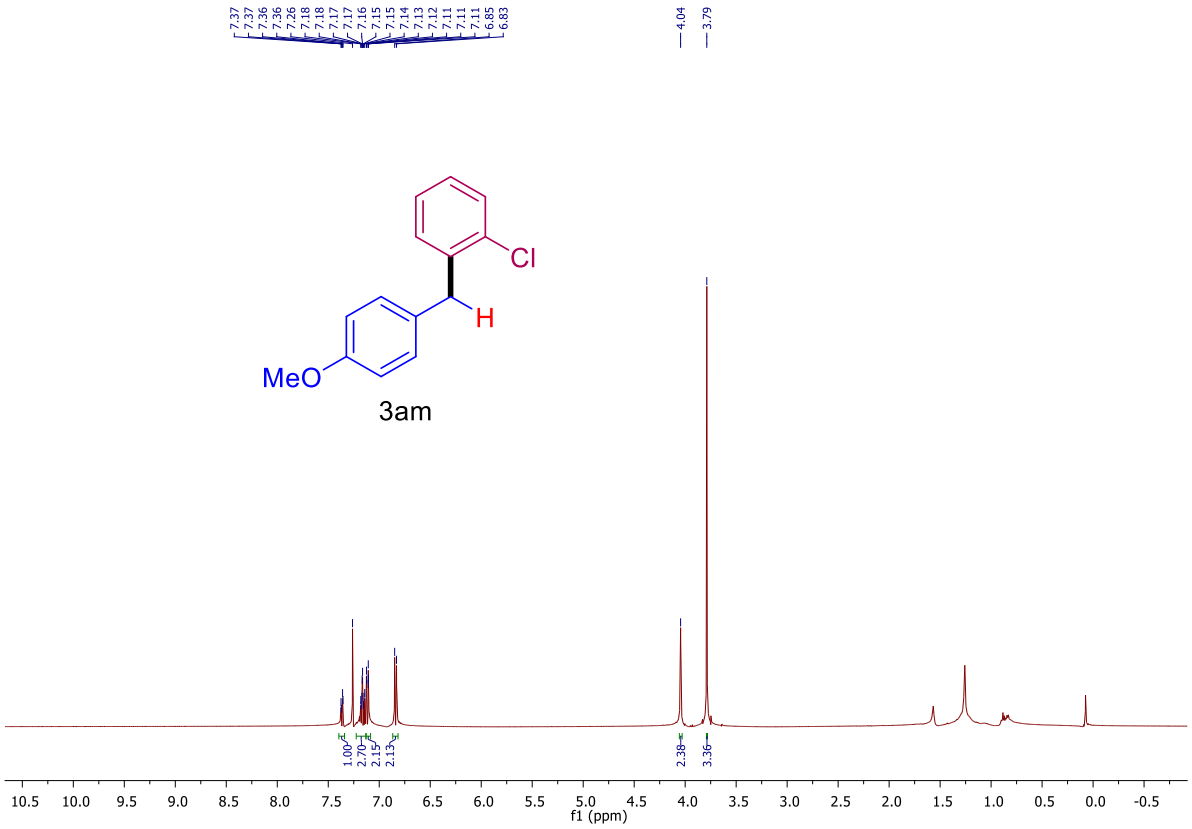


^{19}F NMR (471 MHz, $CDCl_3$, 24 °C)

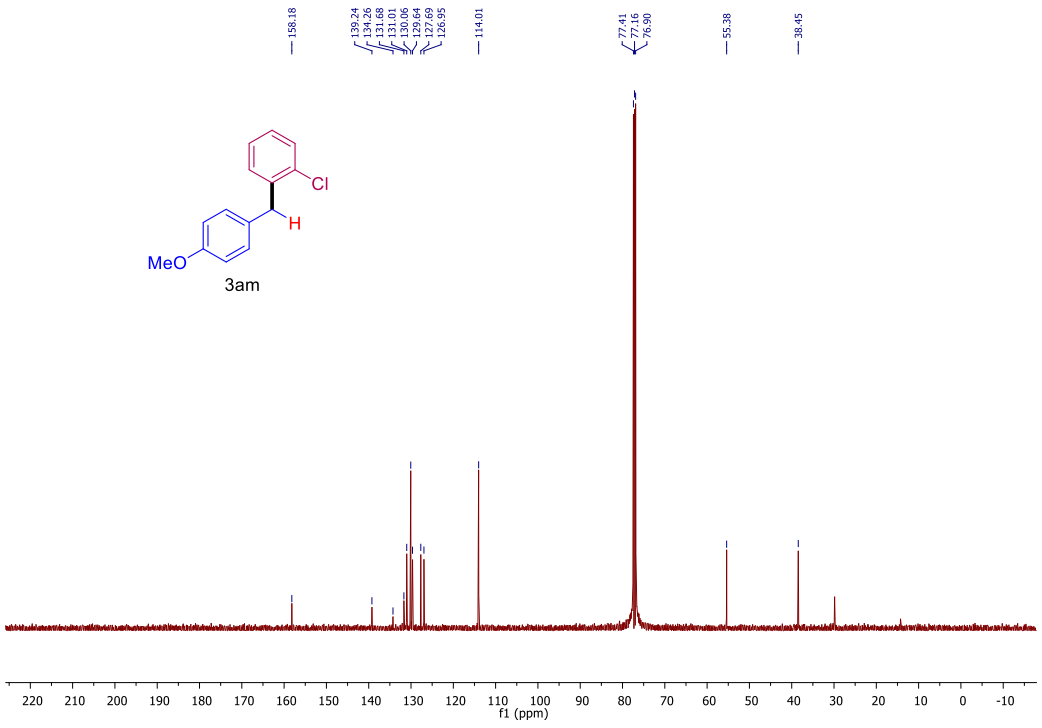


1-Chloro-2-(4-methoxybenzyl)benzene (**3am)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

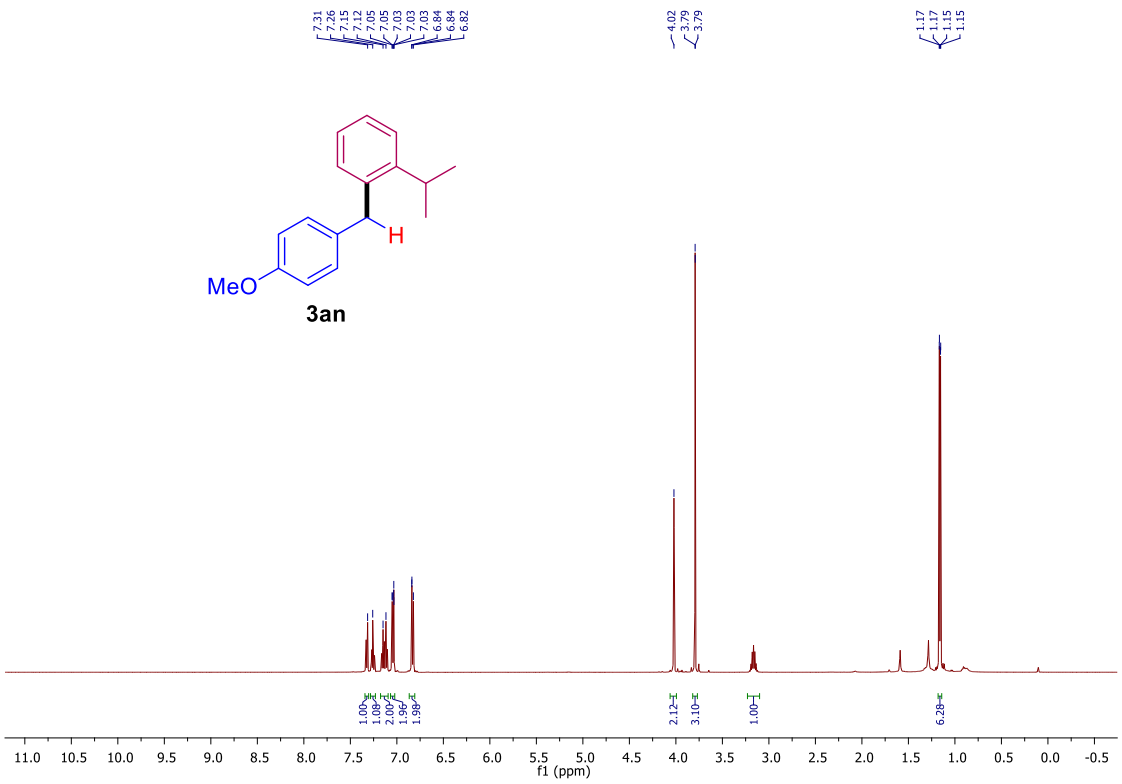


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

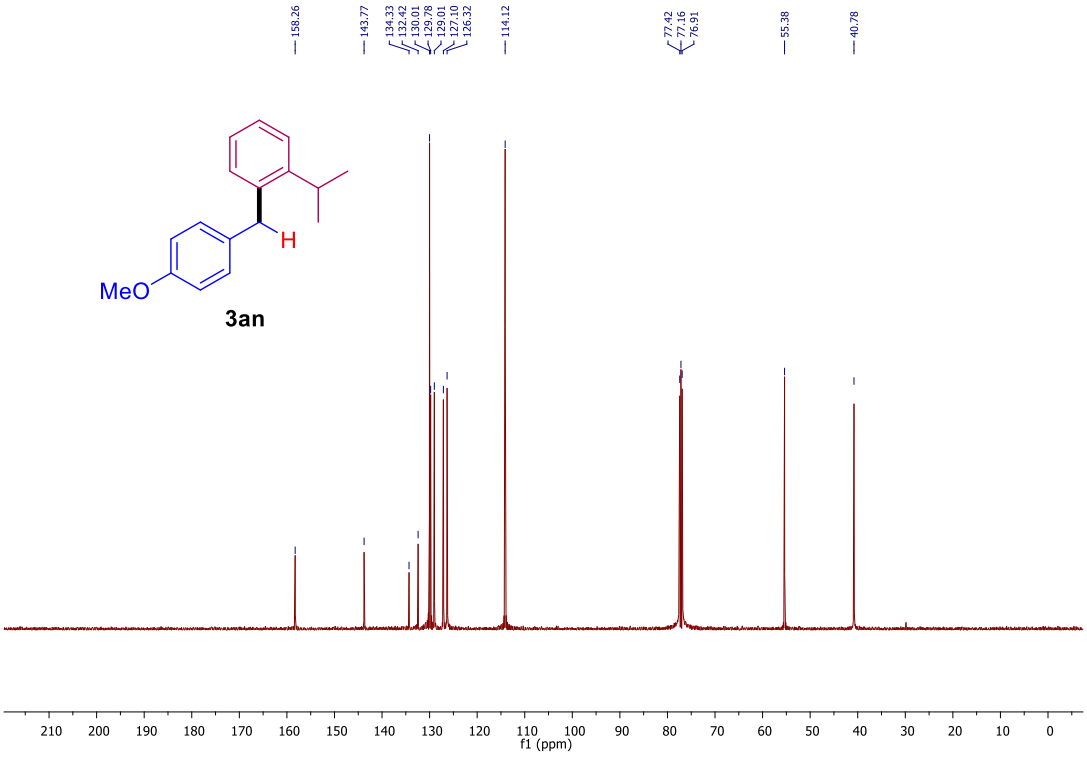


1-Isopropyl-2-(4-methoxybenzyl)benzene (**3an)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

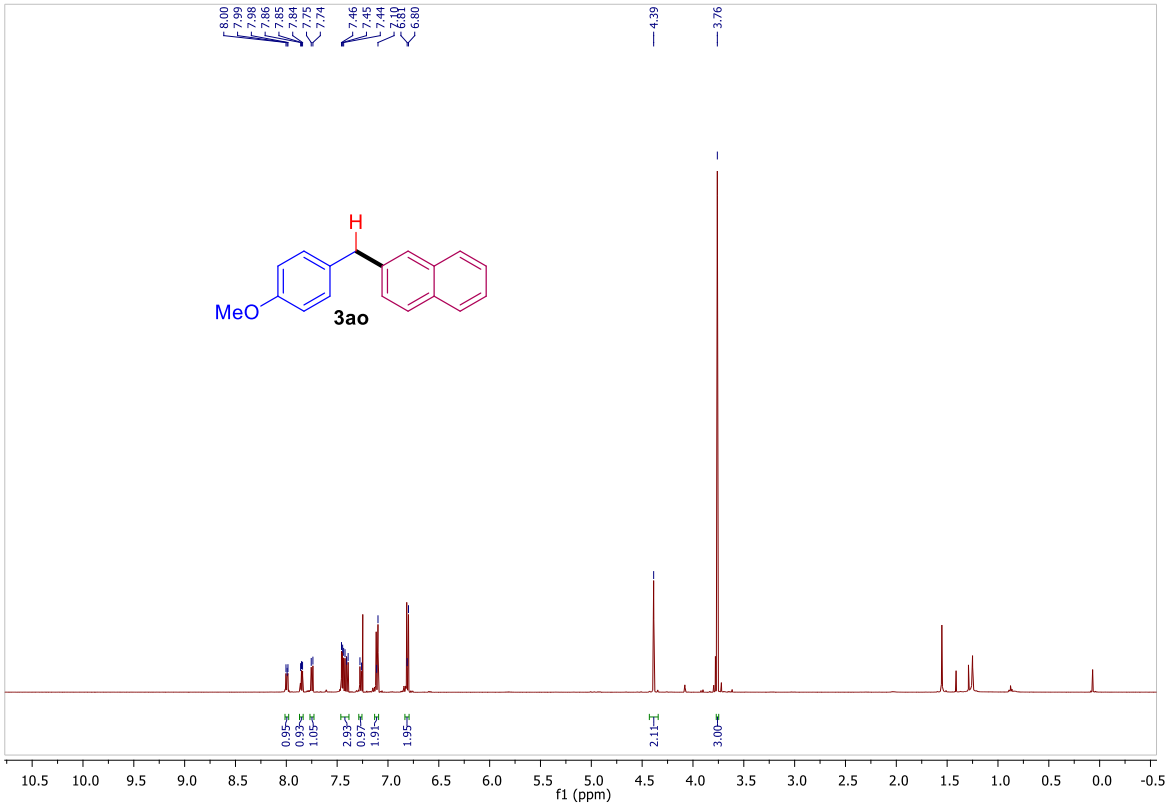


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

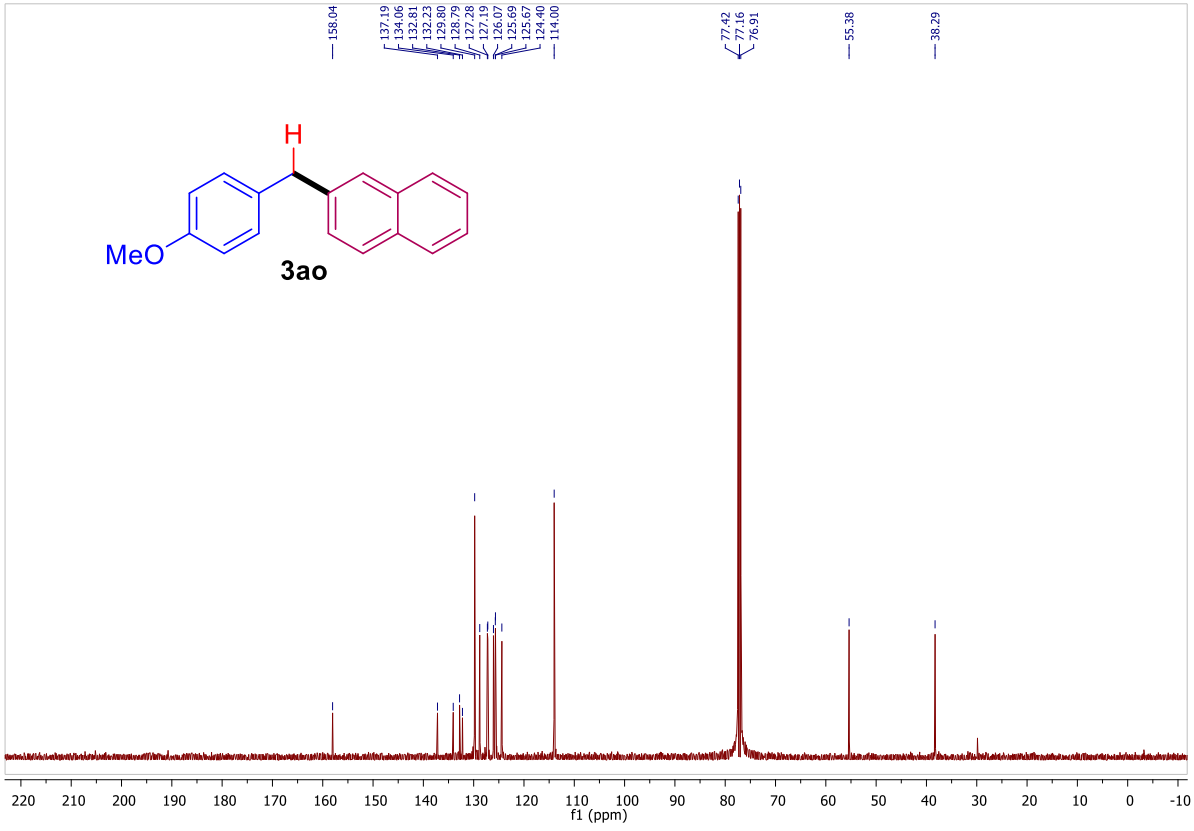


2-(4-Methoxybenzyl) naphthalene **(3ao)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

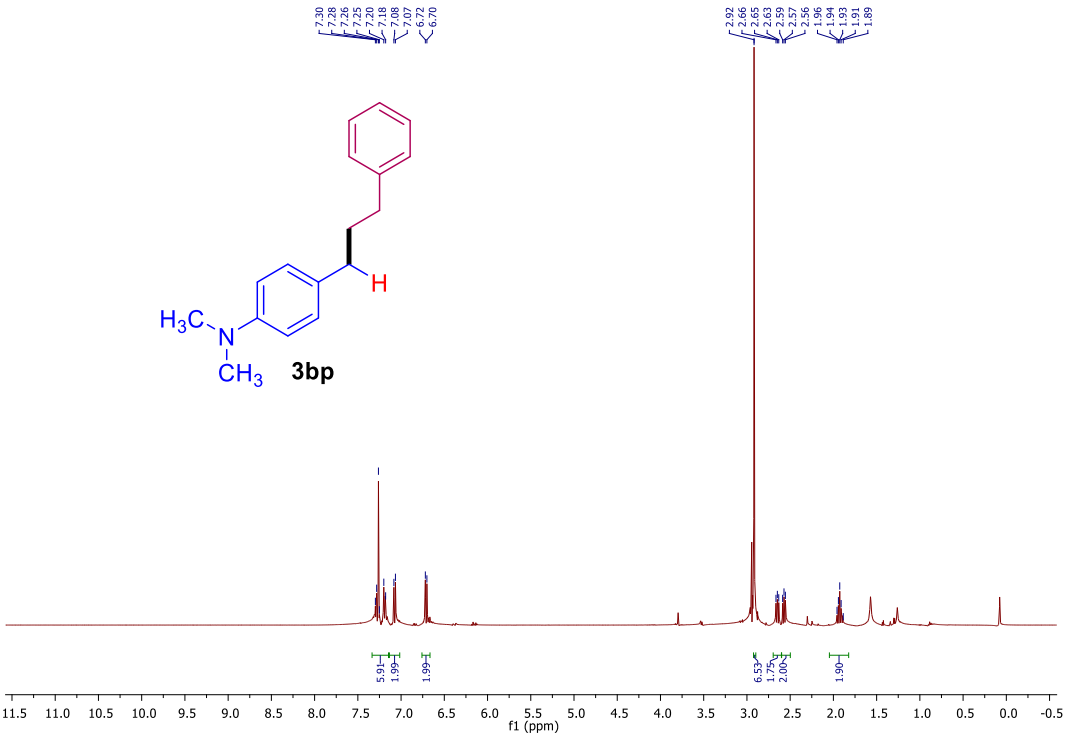


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

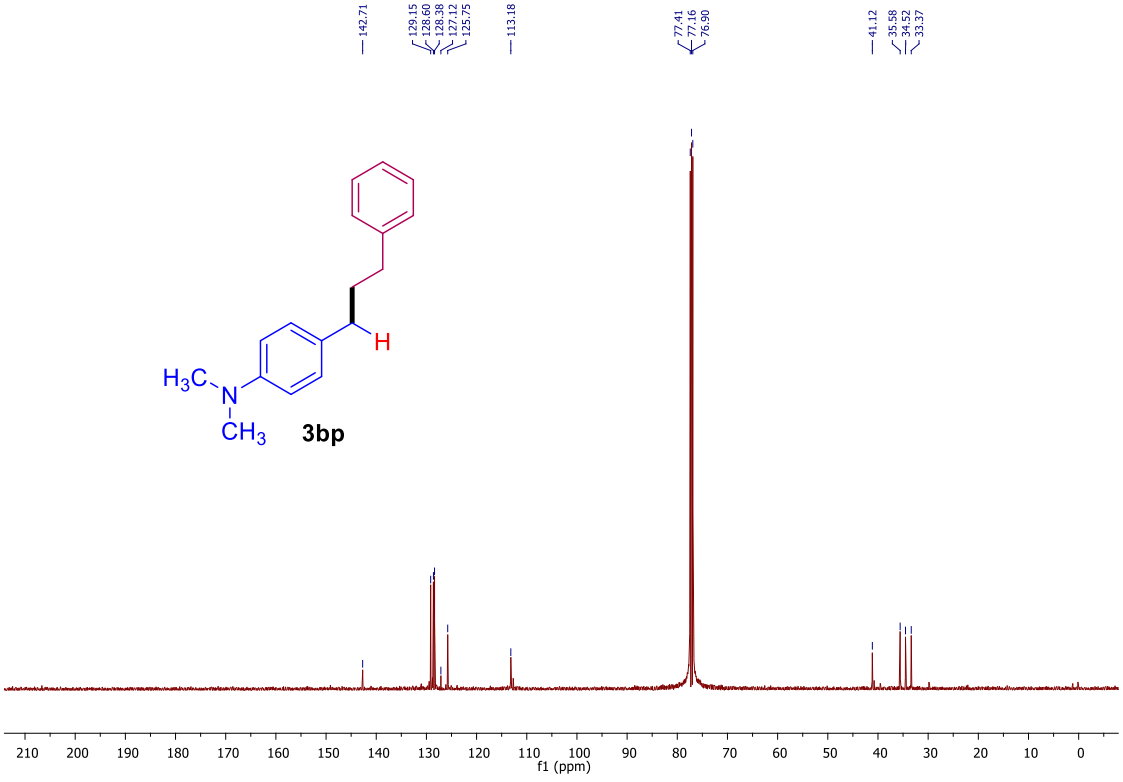


N,N-Dimethyl-4-(3-phenylpropyl)aniline (**3bp)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

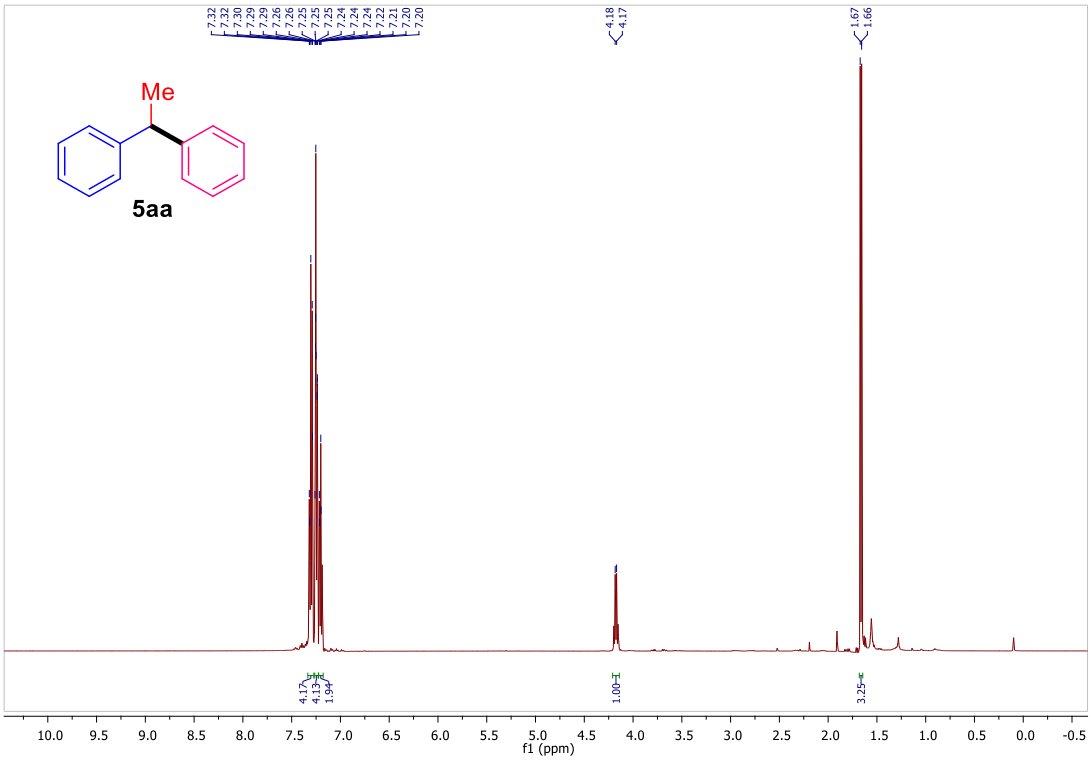


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

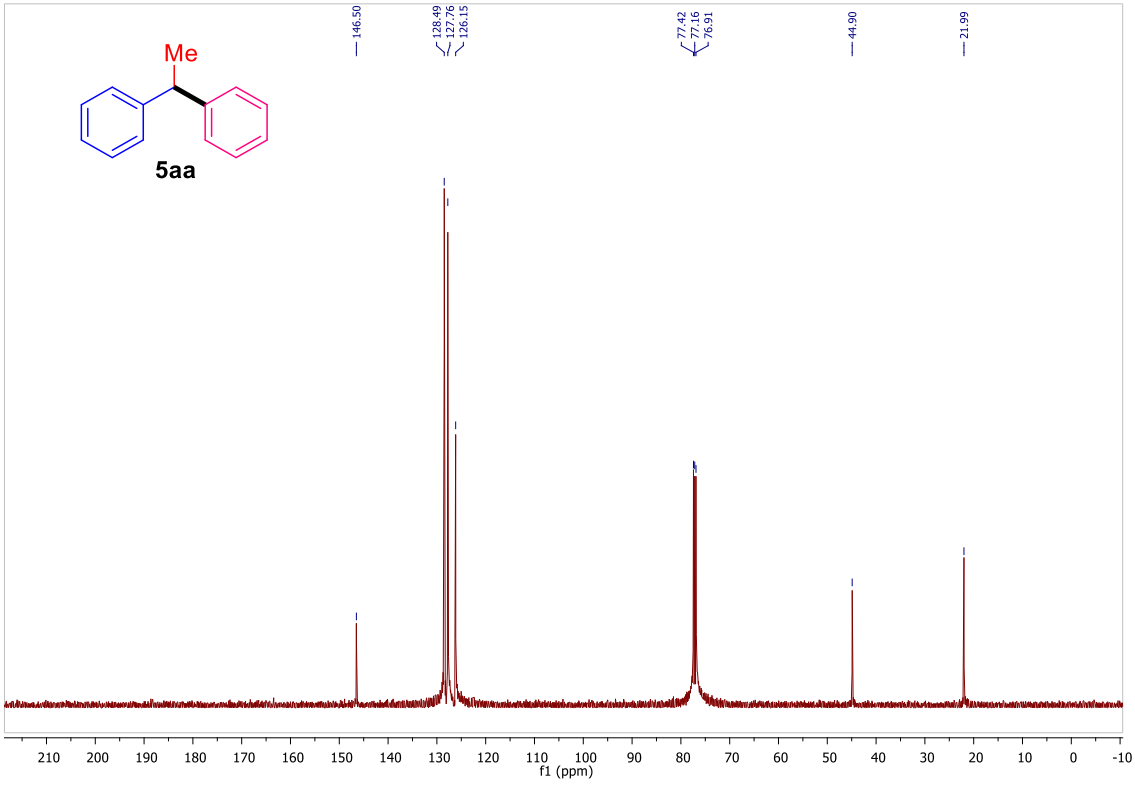


Ethane-1,1-diyldibenzene (**5aa)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

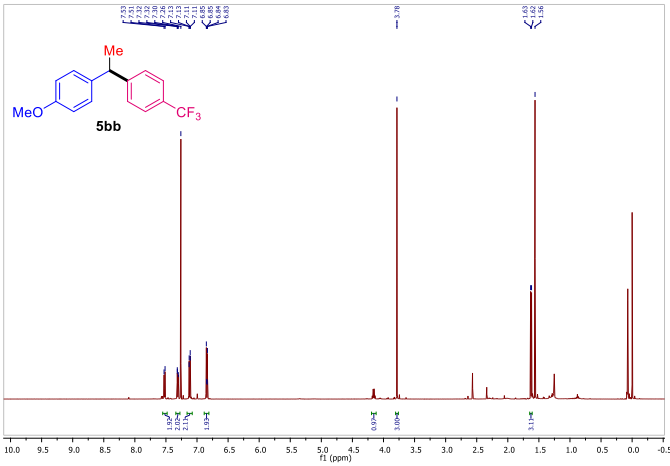


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

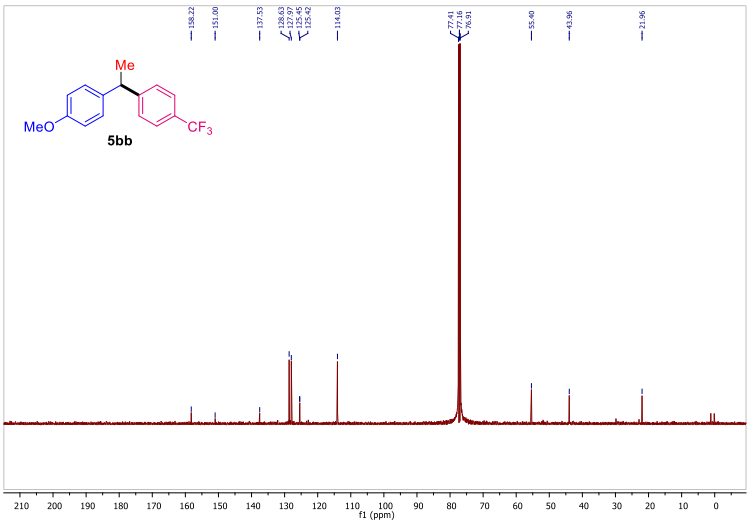


(S)-1-Methoxy-4-(1-(4-(trifluoromethyl) phenyl) ethyl) benzene **(5bb)**

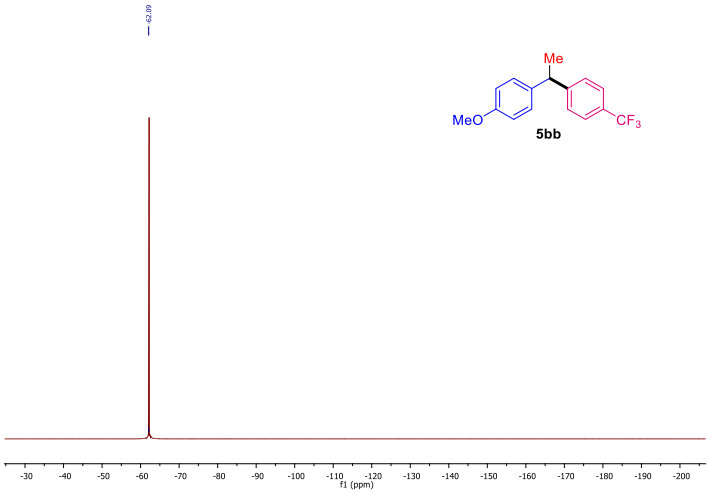
^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)



^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

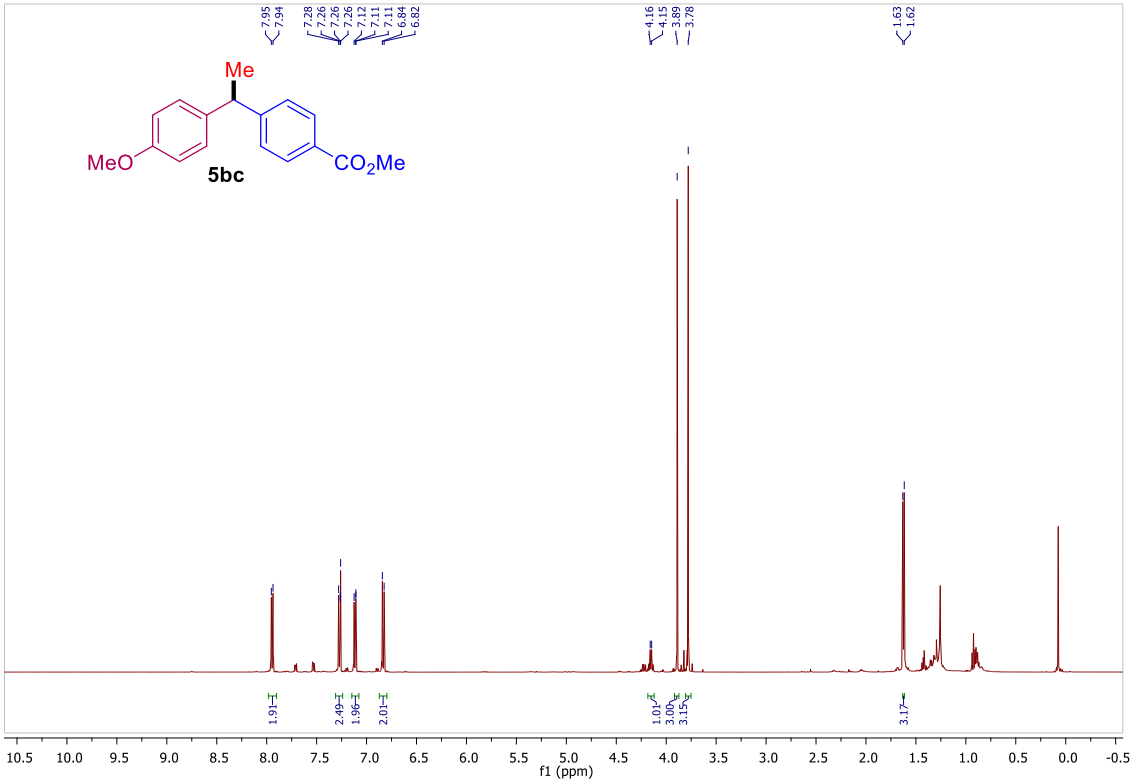


^{19}F NMR (471 MHz, $CDCl_3$, 24 °C)

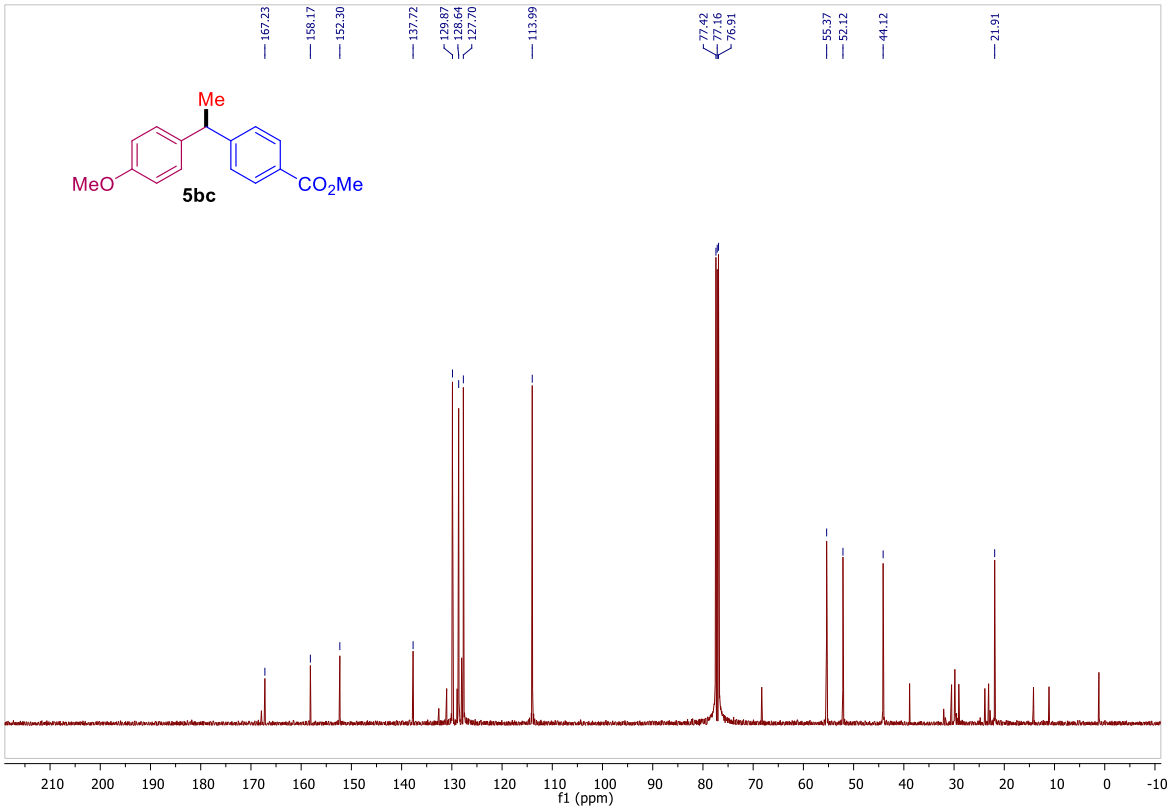


Methyl (S)-4-(1-(4-methoxyphenyl)ethyl)benzoate **(5bc)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

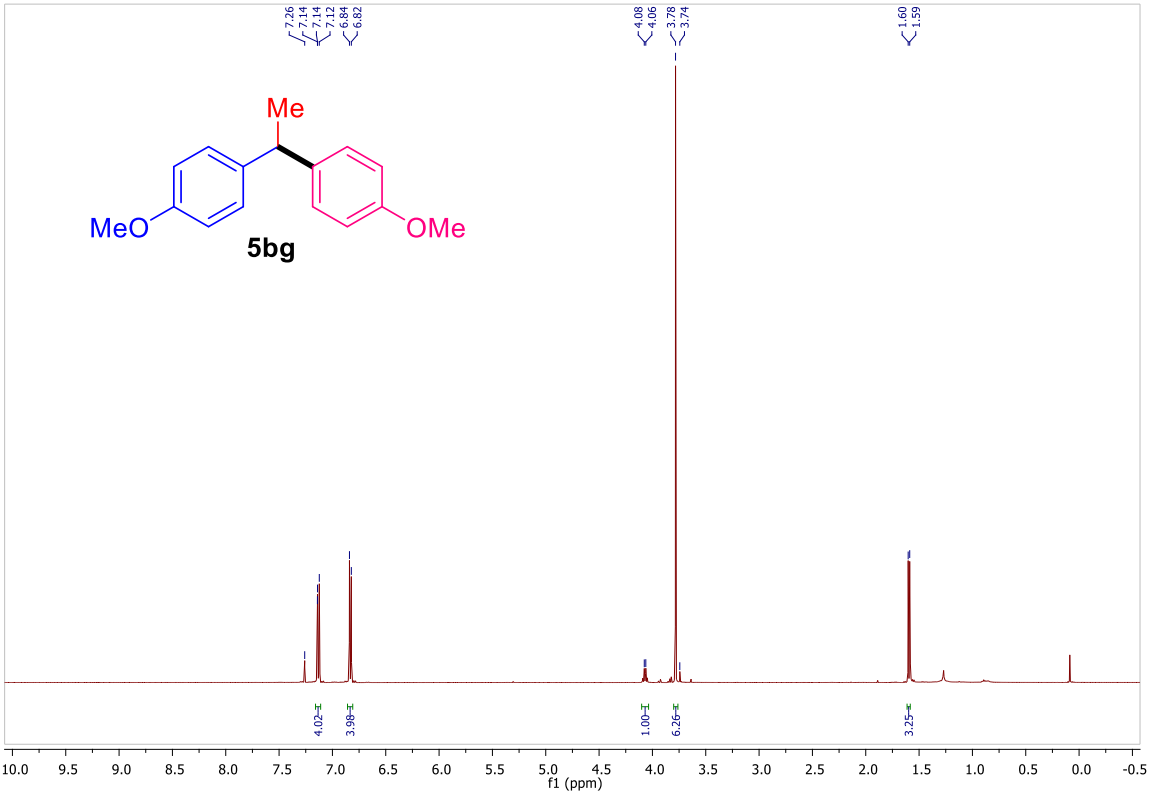


^{13}C {^{1}H} NMR (500 MHz, $CDCl_3$, 24 °C)

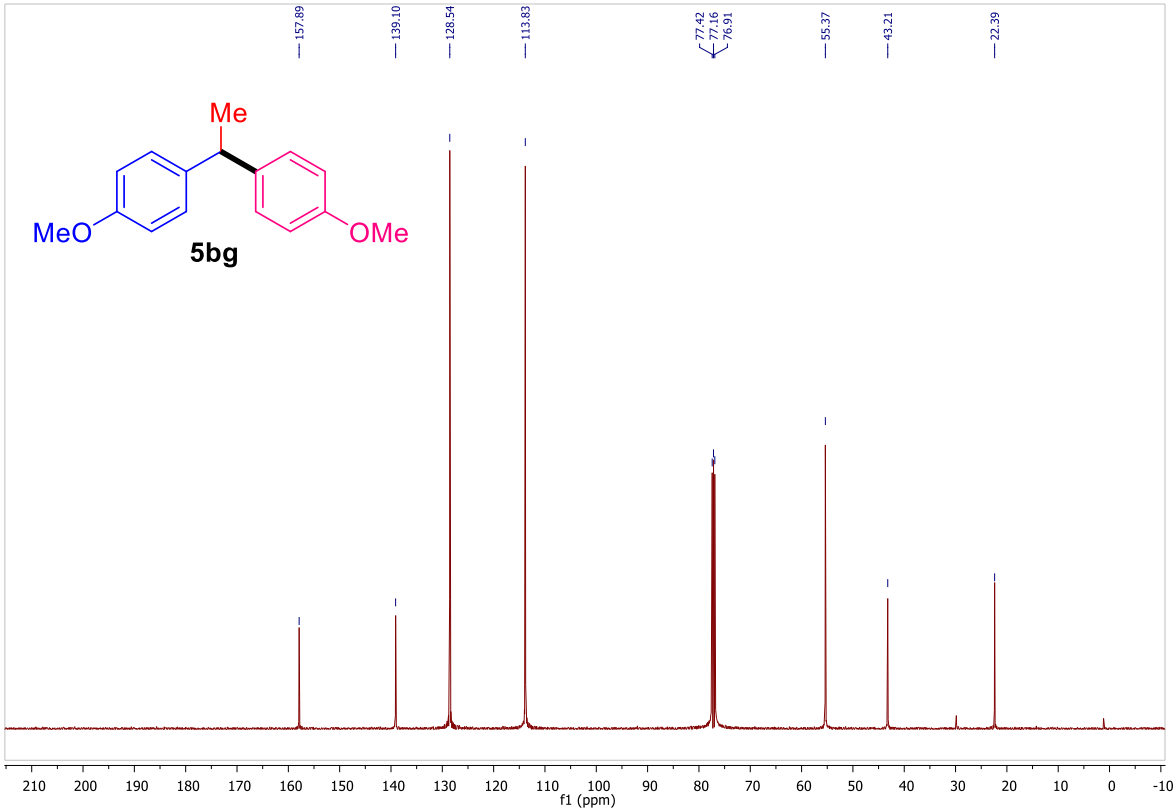


4,4'-(Ethane-1,1-diyl) bis(methoxybenzene) **(5bg)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

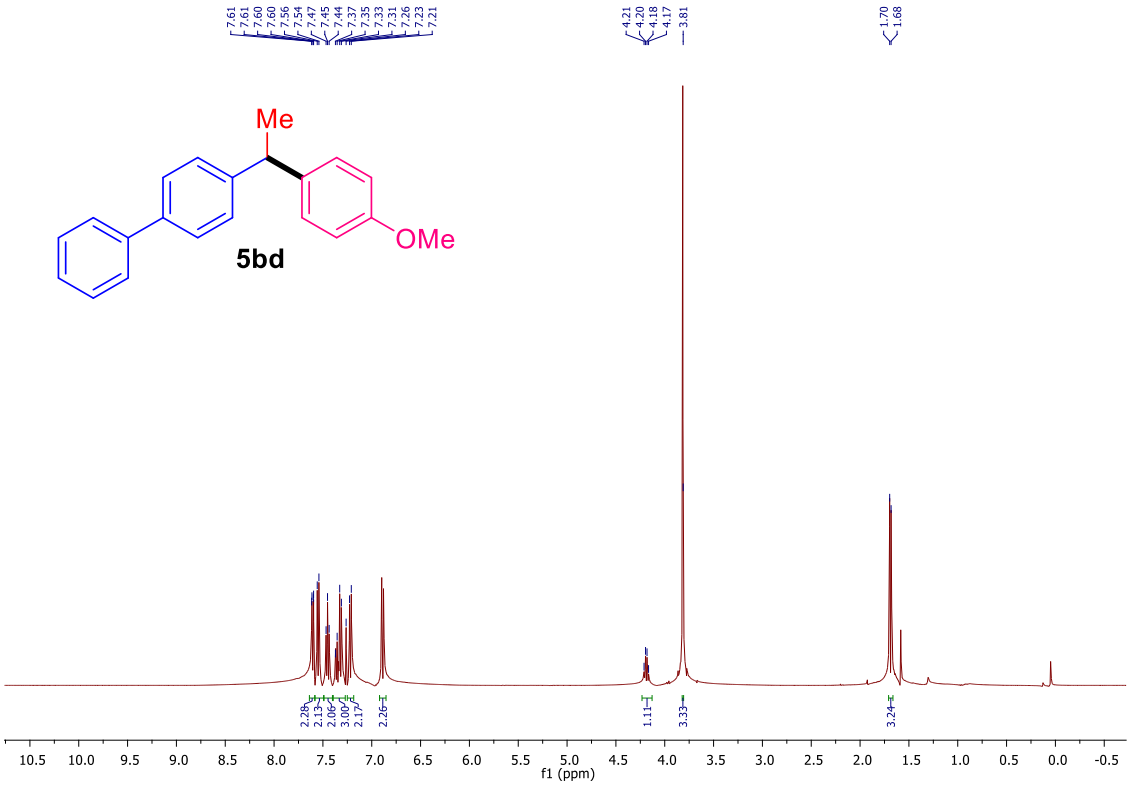


^{13}C {^{1}H} NMR (500 MHz, $CDCl_3$, 24 °C)

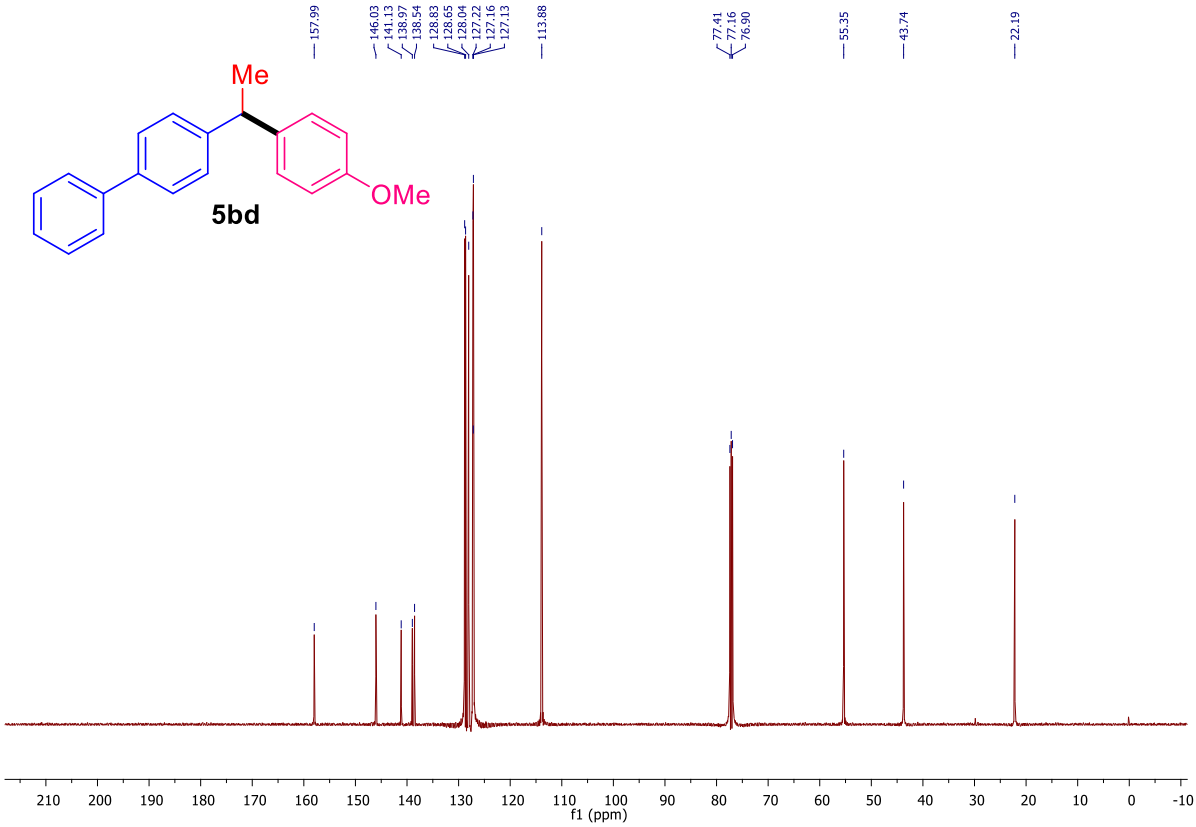


(S)-4-(1-(4-Methoxyphenyl) ethyl)-1,1'-biphenyl **(5bd)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

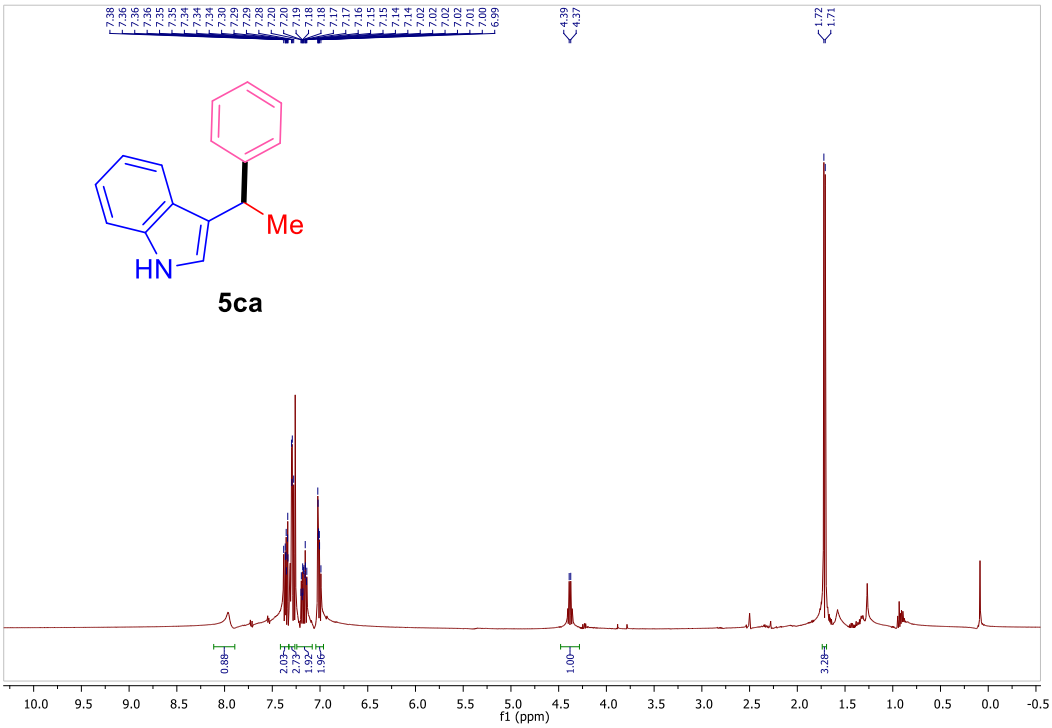


^{13}C {^{1}H} NMR (500 MHz, $CDCl_3$, 24 °C)

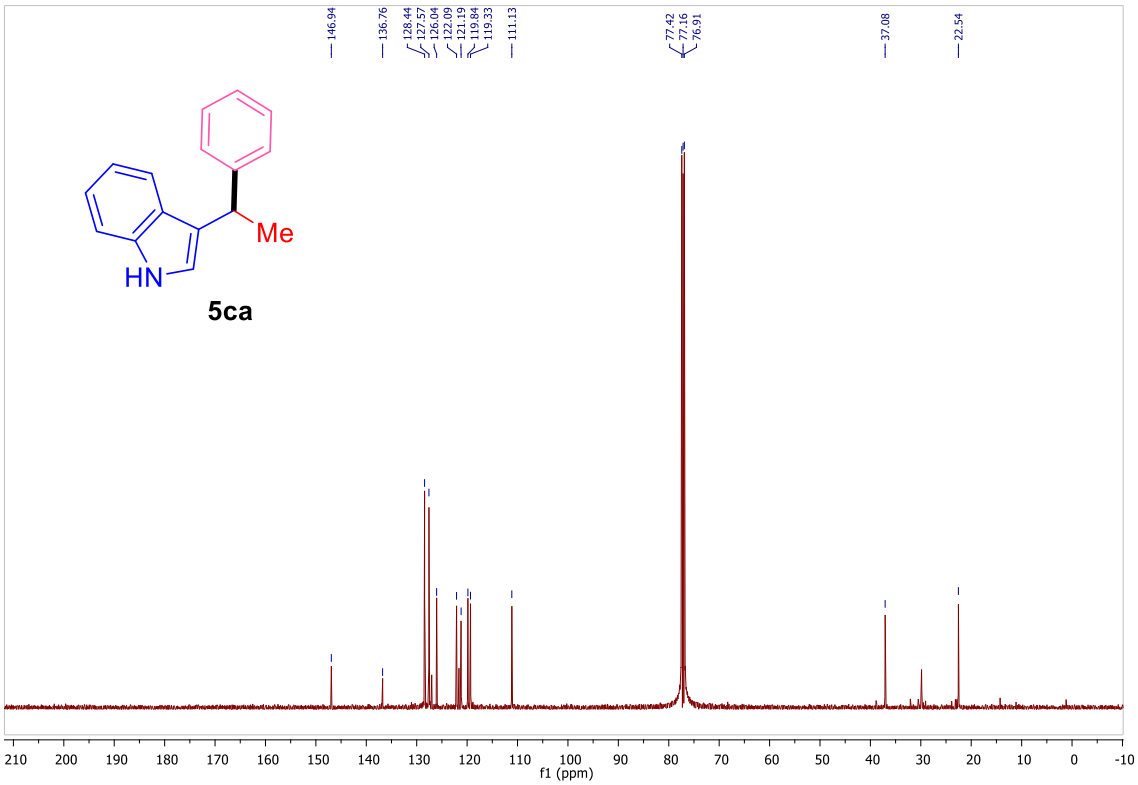


3-(1-Phenylethyl)-1H-indole **(5ca)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

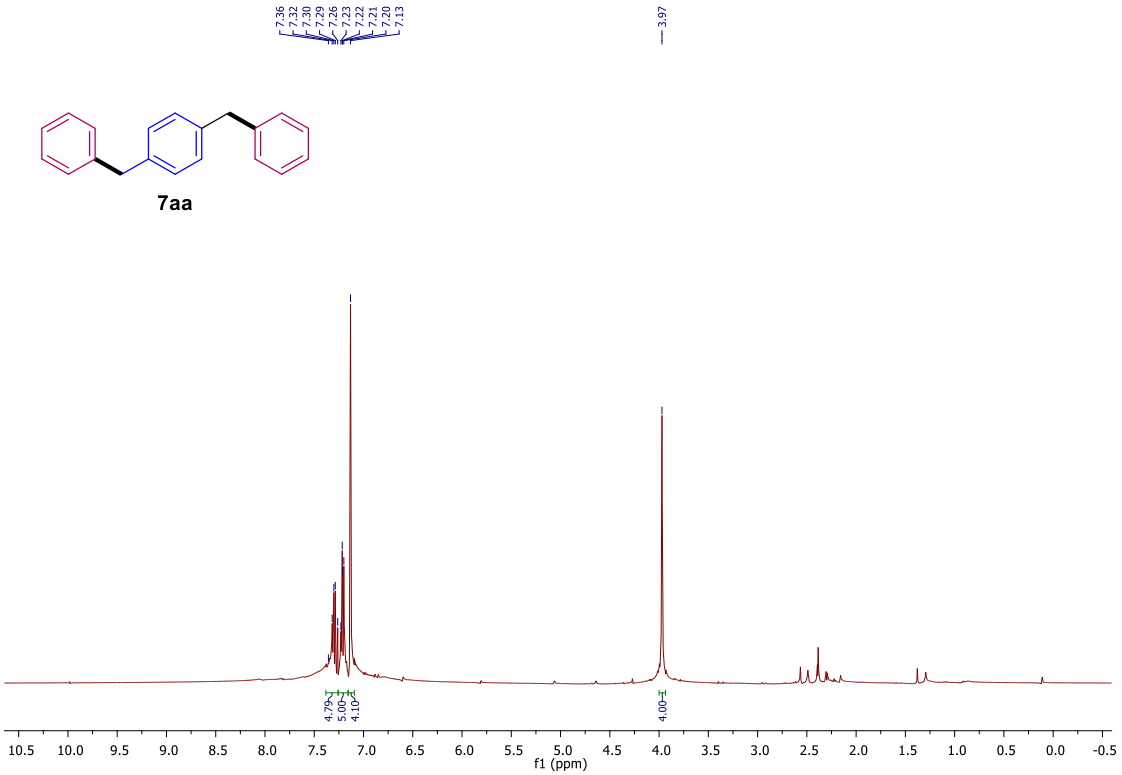


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24 °C)

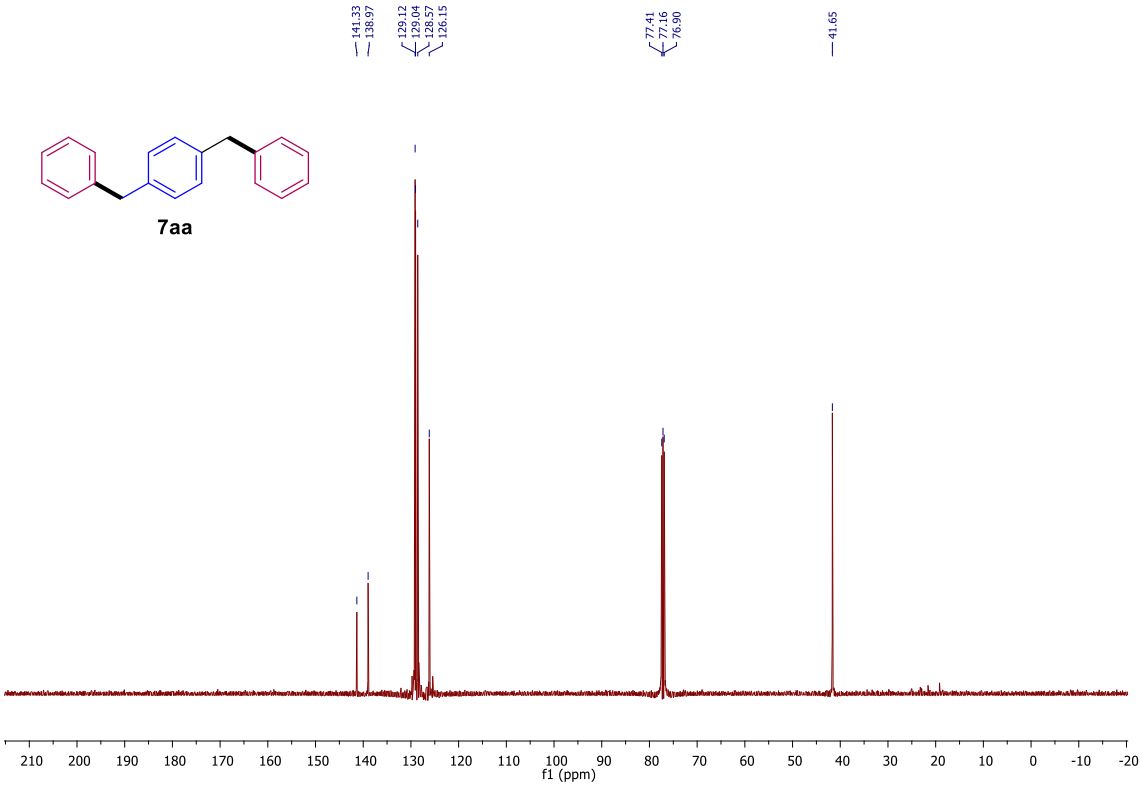


Bis(1,4-diarylmethylbenzene)

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

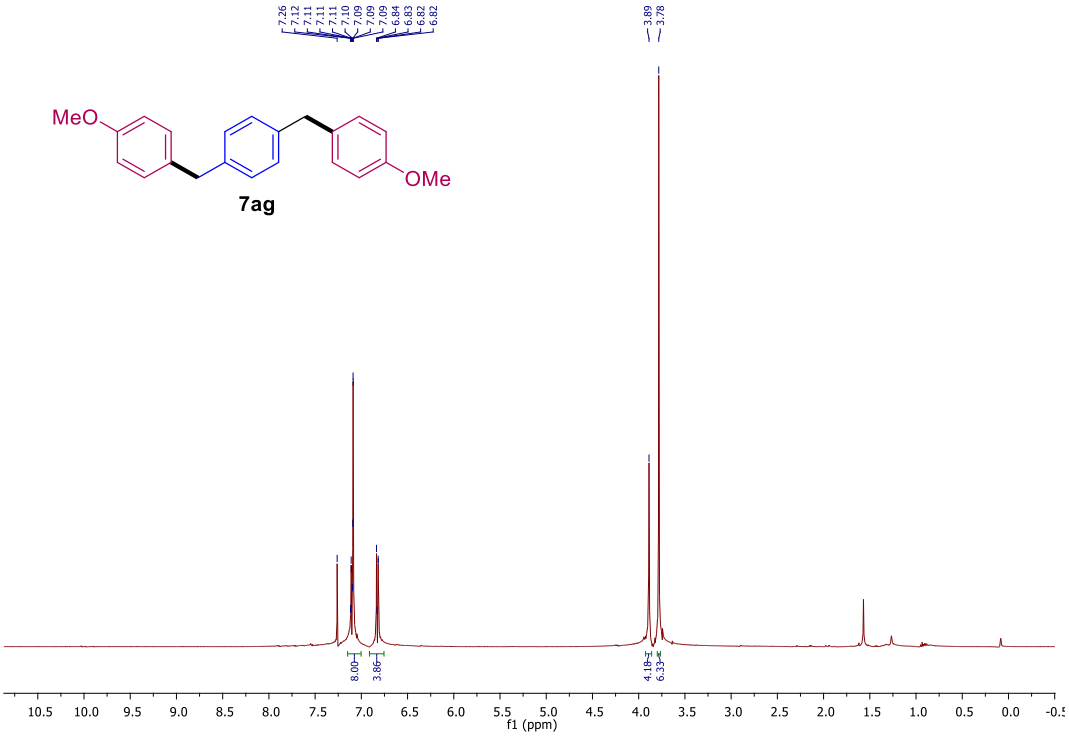


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

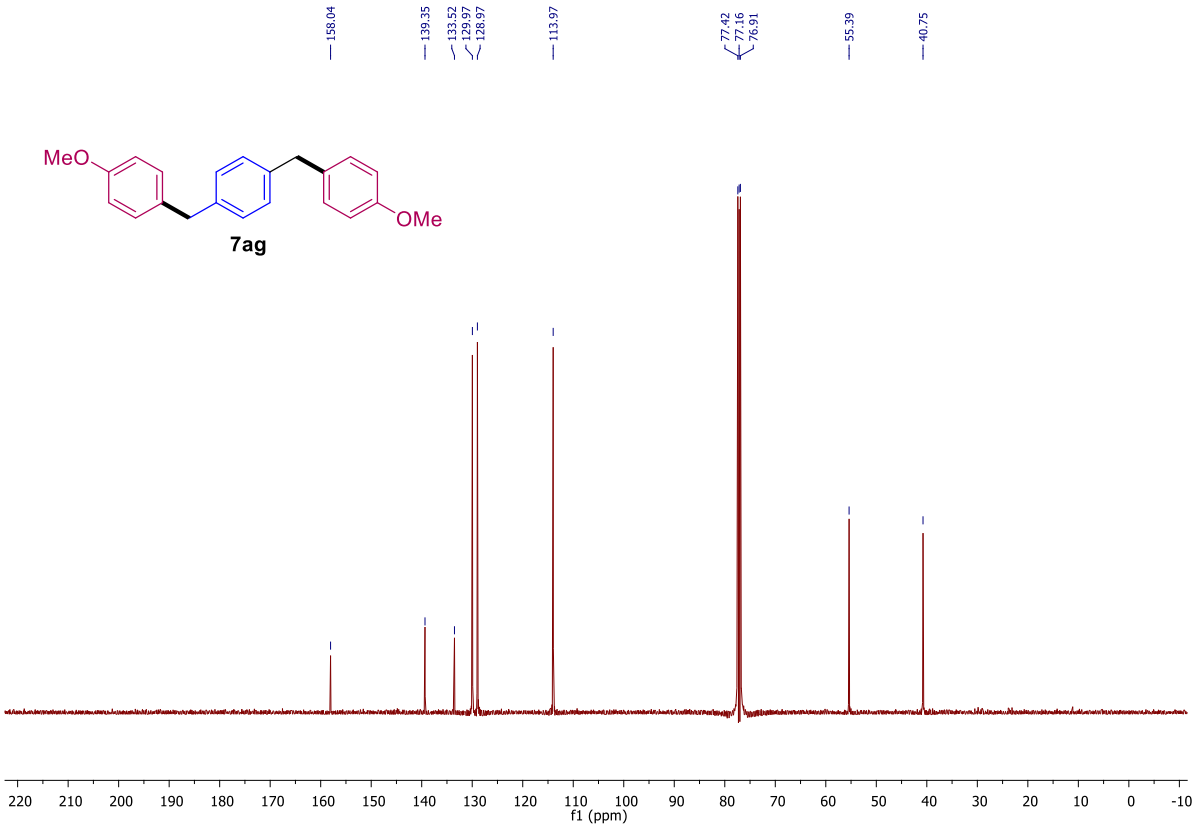


1,4-Bis(4-methoxybenzyl)benzene **(7ag)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

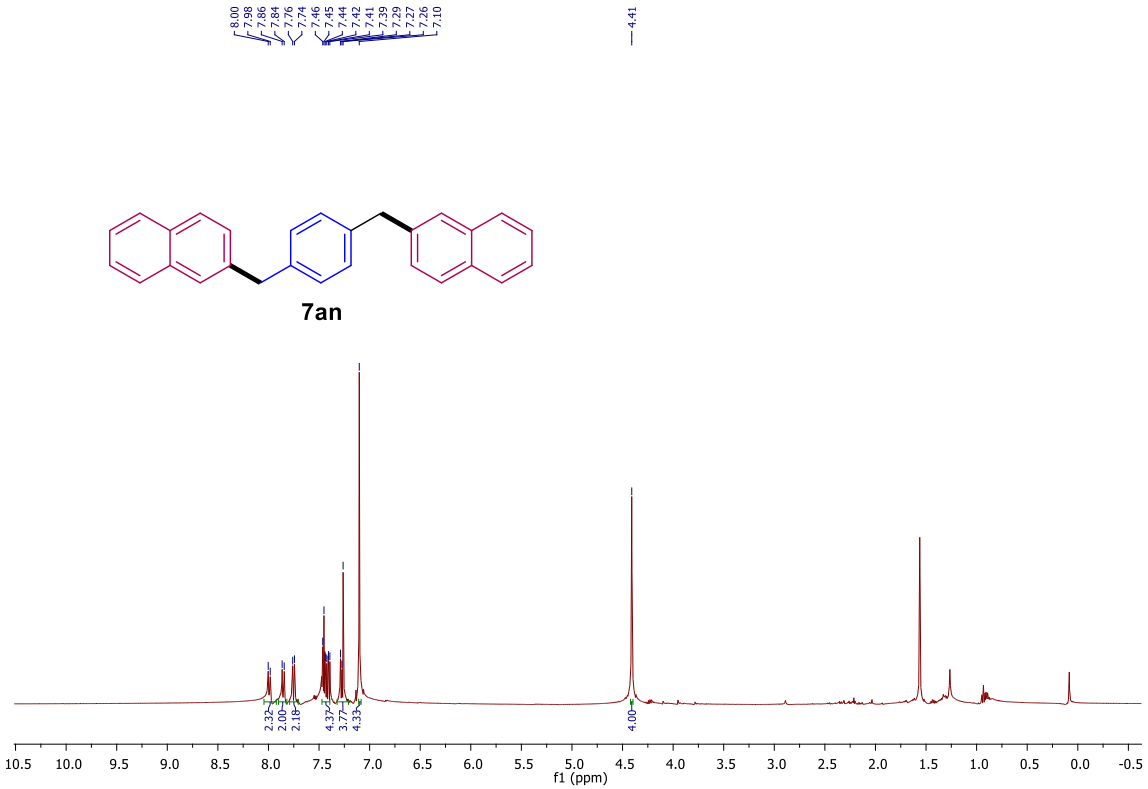


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

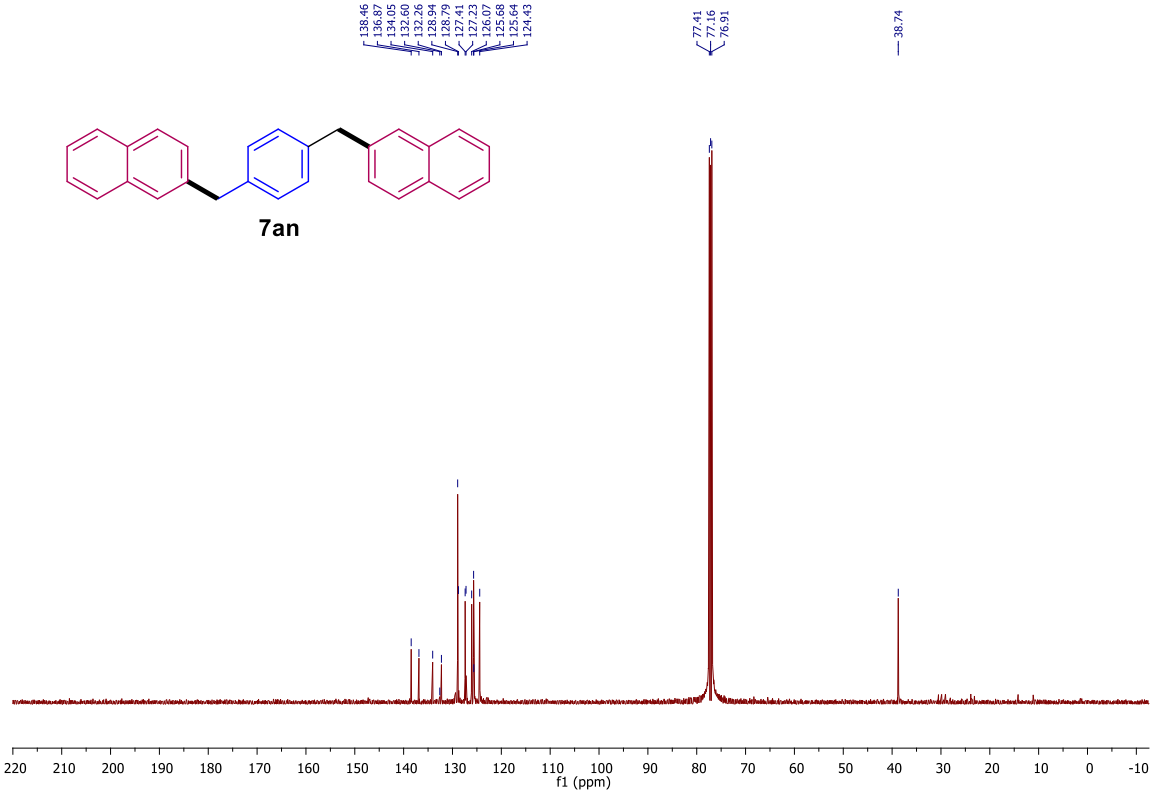


1,4-Bis(naphthalen-2-ylmethyl) benzene **(7an)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

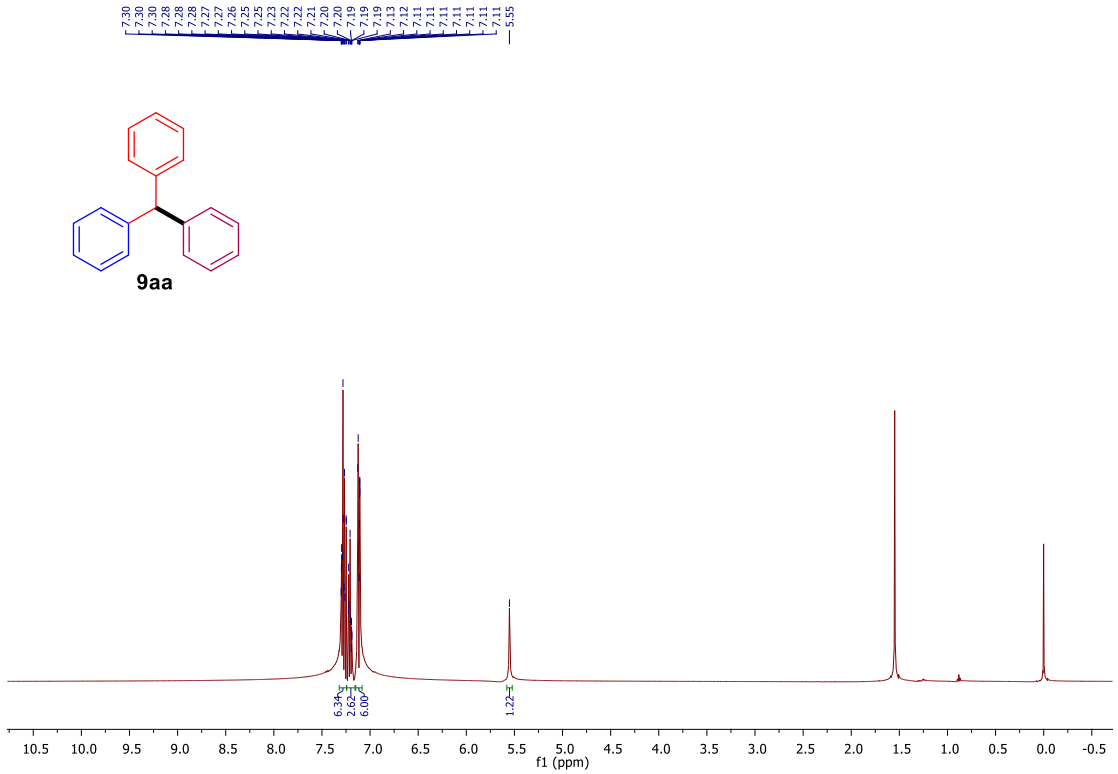


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

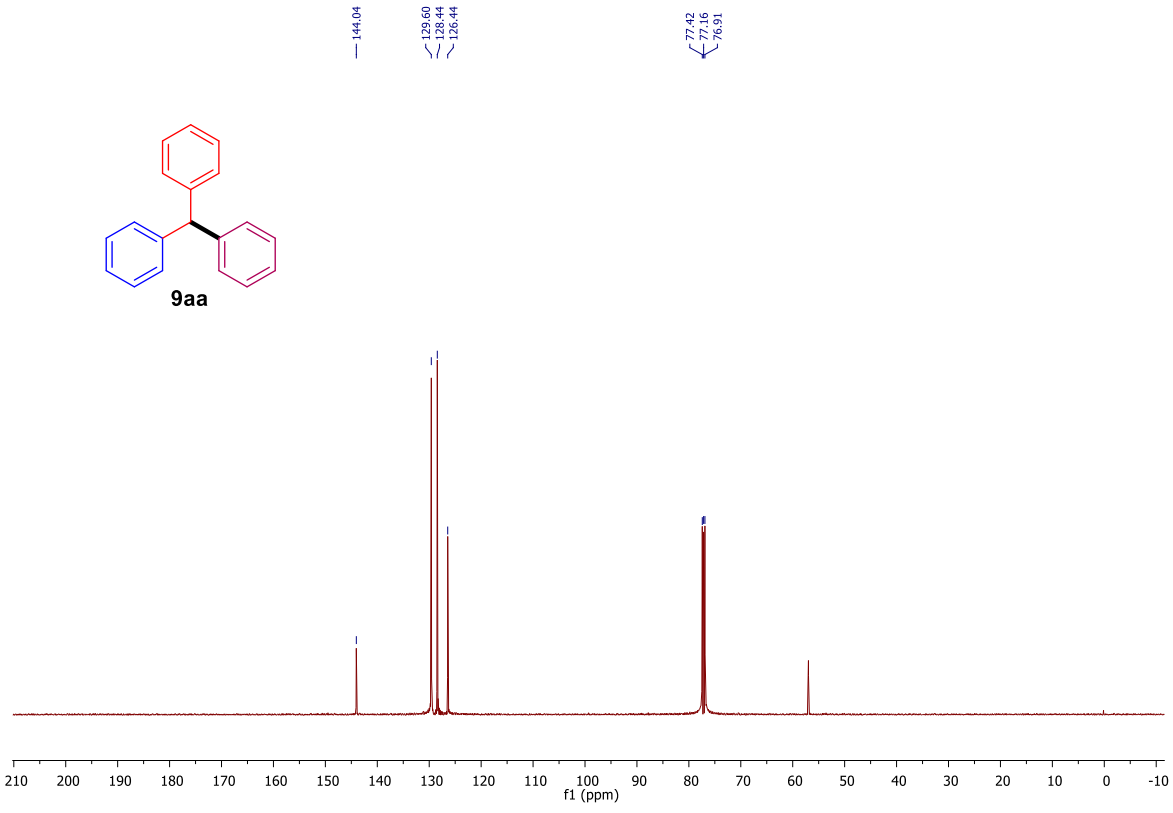


Triphenylmethane **(9aa)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)

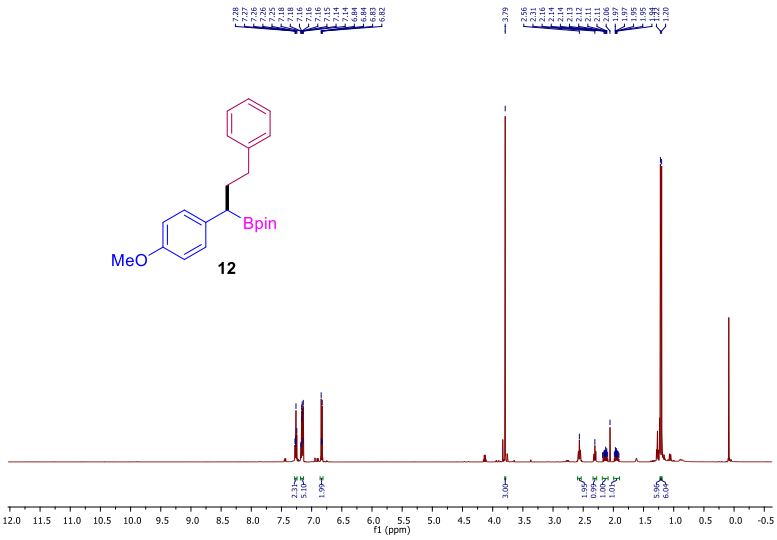


^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)

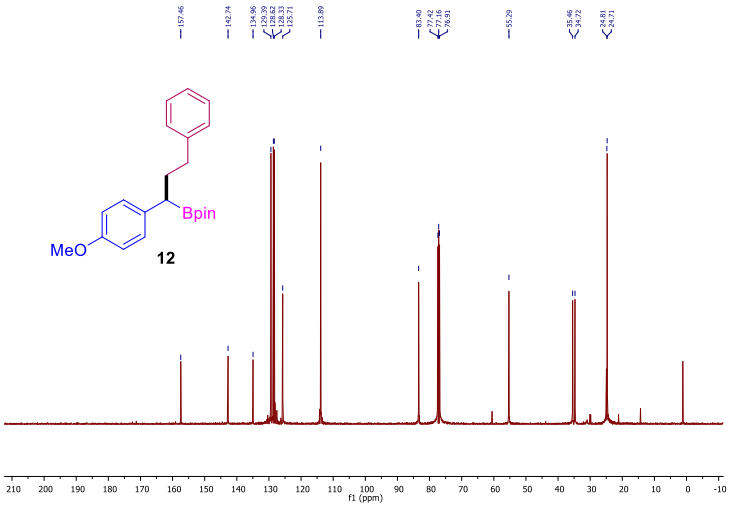


(R)-2-(1-(4-Methoxyphenyl)-3-phenylpropyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **(12)**

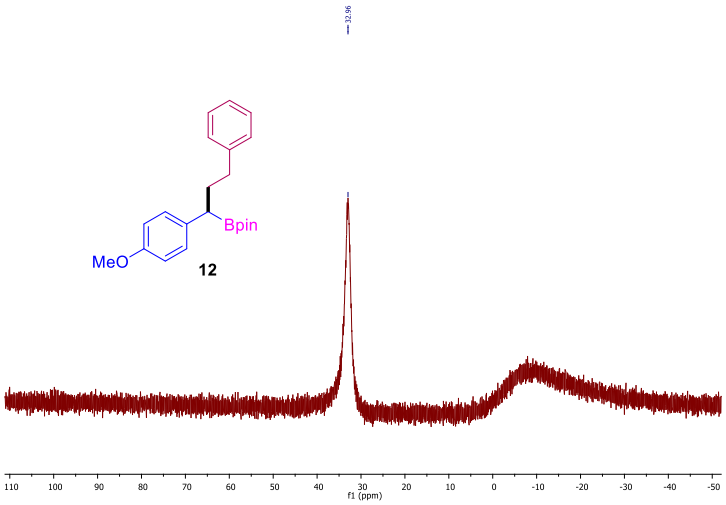
^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)



^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C

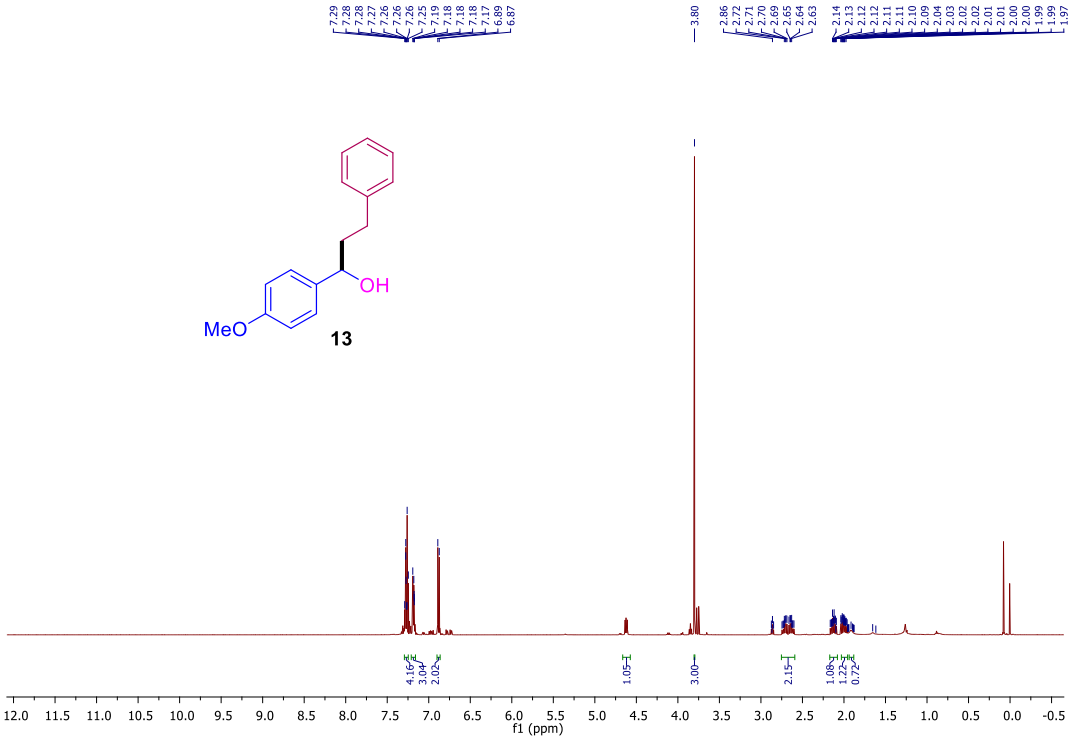


^{11}B NMR (160 MHz, $CDCl_3$, 24°C)



(*R*)-1-(4-Methoxyphenyl)-3-phenylpropan-1-ol **(13)**

^{1}H NMR (500 MHz, $CDCl_3$, 24 °C)



^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$, 24°C)