Electronic Supplementary Material (ESI) for ChemComm.

Electronic Supplementary Information

Cobalt-Catalyzed Cross-Coupling Reactions of Aryl- and Alkylaluminum Derivatives with (Hetero)Aryl and Alkyl Bromides

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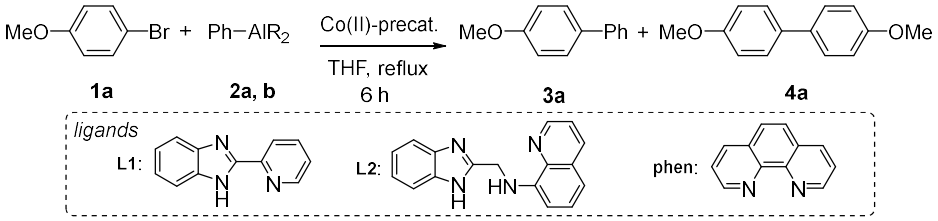
1. General Information

^{1}H-NMR spectra were obtained using a Bruker spectrometer (^{1}H: 400.12 MHz; ^{13}C: 100.62 MHz), $CDCl_3$ was used as the solvent; chemical shifts are reported in parts per million (δ). Dimethyl sulfone has been used as the internal standard for yield determination by ^{1}H NMR analysis on the crude reaction mixtures. IR spectra were recorded with a Jasco FT-IR spectrophotometer. GC/MS analyses, were performed with a gas chromatograph equipped with a 5% phenylpolymethylsiloxane capillary column, 30 m, 0.25 mm i.d., and a mass-selective detector operating at 70 eV. High-resolution mass spectrometry (HRMS) analyses were performed using a Bruker microTOF QII mass spectrometer equipped with an electrospray ion source (ESI). Analytical thin-layer chromatography (TLC) was carried out on pre-coated 0.25 mm thick plates of Kieselgel 60 F_{254}; visualization was accomplished by UV light (254 nm) or by spraying a solution of 5 % (w/v) ammonium molybdate and 0.2 % (w/v) cerium(III) sulfate in 100 mL 17.6 % (w/v) aq. sulphuric acid and heating to 200 °C until blue spots appeared. Chromatography was run by using silica gel 60 with a particle size distribution 40–63 µm and 230–400 ASTM, using petroleum ether/ethyl acetate (AcOEt) mixture as the eluent. Reagents, catalysts and solvents, unless otherwise specified, were purchased from commercial sources at the highest commercial quality and used without further purifications. All reactions were performed in oven-dried glassware using dry tetrahydrofuran (THF), freshly distilled under a nitrogen atmosphere, over sodium/benzophenone ketyl. All operations were performed with Schlenk techniques under a dry nitrogen atmosphere. The following solutions of Grignard reagents were furnished by TCI (Europe) and SigmaAldrich (Sigma-Aldrich, St. Louis, MO, USA), and were used with the following concentration: Me_2AlCl 1.0 M in hexane, PhMgCl 2.0 M in THF, *o*-TolylMgCl 1.0 M in THF, *n*-BuMgCl 2.0 M in THF, *i*PrMgCl·LiCl 1.3 M in THF, 3-ChlorophenylMgBr 0.5 M in THF, 3,5-DimethoxyphenylMgCl 1.0 M in THF. Co(1,10-phenantroline)Cl_2 complex has been prepared according to literature procedure.[1] Full characterization data, including copies of ^{1}H NMR and ^{13}C NMR spectra, have been reported for both the newly synthesized compounds and the known compounds. The following abbreviations have been used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, quin = quintuplet, sext = sextet, sep = septet, br = broad.

[1] B. Brewer, N. R. Brooks, S. J. Abdul-Halim, A. G. Sykes, *Chem. Crystallogr.* 2003, **33**, 651–662

2. Optimization Details

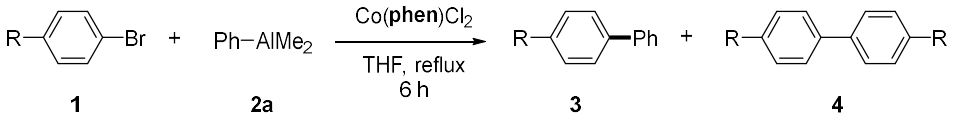
Table S1 Optimization of the Co-catalyzed model reaction between 4-bromoanisole **1a** and organoaluminum reagents **2a,b** in THF.[a]



Entry	Co(II) precatalyst	**2**	R	**1a** conversion (%)	**3a** yield (%)	**4a** yield (%)
1	$CoCl_2$	**2a**	Me	82	21	54
2	$Co(acac)_2$[b]	**2a**	Me	67	5	58
3	Co(**L1**)Cl_2[b]	**2a**	Me	53	<5	43
4	Co(**L2**)Cl_2[b]	**2a**	Me	55	<5	43
5	Co(**phen**)Cl_2[b]	**2a**	Me	95	60	34
6	Co(**phen**)Cl_2[b, c]	**2a**	Me	76	48	20
7	Co(**phen**)Cl_2[b,d]	**2b**	Ph	82	63	17
8	Co(**phen**)Cl_2[b,e]	**2b**	Ph	15	13	N.D.
9	Co(**phen**)Cl_2[b,f]	**2b**	Ph	71	55	11
10	$CoCl_2$[c]	**2b**	Ph	100	69	21
11	$CoCl_2$[c,g]	**2b**	Ph	100	73	18
12	$Co(OAc)_2$[c]	**2b**	Ph	100	70	18
13	$Co(NO_3)_2$[c]	**2b**	Ph	60	45	6

*[a]Reaction conditions: **1a** (0.5 mmol), **2a** (or **2b**) (0.75 mmol, 5 mL of a 0.15 M solution in THF, Co(II)-precatalyst (0.05 mmol, 10 mol%), in THF at reflux temperature. The yields and conversions are calculated via ^{1}H-NMR analysis of the crude reaction mixture using the dimethyl sulfone as internal standard. [b]**acac** = acetylacetonate; **L1** = 2-(2-pyridyl)benzimidazole; **L2** = N-((1H-benzo[d]imidazol-2-yl)methyl)quinolin-8-amine; **phen** = 1,10-phenanthroline. [c]Triphenylaluminum **2b** concentration 0.3 M. [d]5 mol% loading of catalyst. [e]2.5 mol% loading of catalyst. [f]Reaction time 3 hours. [g]Reaction temperature = 50 °C.*

Table S2. Co-catalyzed couplings of dimethyl(phenyl)aluminum **2a** with variously substituted arylbromides **1**.[a]



1 **2a** **3** **4**

Entry	**1**	R	**1a** Conversion (%)	**3** yield (%)	**4** yield (%)
1	**2c**	CF_3	100	13	87
2	**2f**	NMe_2	100	50	50
3	**2g**	OH	50	35	12
4	**2h**	COOH	88	78	4

[a]*Reaction conditions:* ***1*** *(0.5 mmol),* ***2a*** *(0.75 mmol, 5 mL of a 0.15 M solution in THF), Co(****phen****)Cl_2 (0.05 mmol, 10 mol%).*

Table S3. Ligands screening for Co-catalyzed coupling between **1a** and **2b.**[a,b]

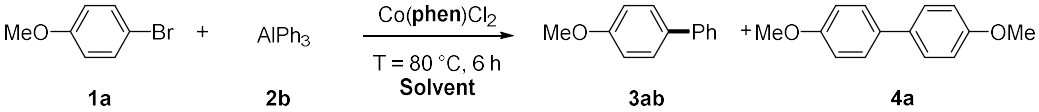
1a **2b** **3ab** **4a**

Entry	**Ligand**	**1a** conversion (%)	**3** yield (%)	**4a** yield (%)
1	dppf	59	42	10
2	TMEDA	52	38	9
3	BINAP	40	27	4
4	N-Methyl-1,2-phenylenediamine	32	24	2
5	(*o*-tolyl)$_3$P	30	18	2
6	1,3-diaminopropane	22	14	<2
7	**L2**	17	13	2
8	**L1**	12	8	<2
9	Ph_3P	0	N.D.	N.D.

[a]*Reaction conditions:* ***1a*** *(0.5 mmol),* ***2b*** *(0.75 mmol, 2.5 mL of a 0.3 M solution in THF), $CoCl_2$ (0.05 mmol, 10 mol%),* ***ligand*** *(0.05 mmol, 10 mol%). dppf: 1,1'-Bis(diphenylphosphino)ferrocene; TMEDA: N,N,N',N'-tetramethylethylenediamine; BINAP: 2,2'-bis(diphenylphosphino)-1,1'-binaphthyl; ;* ***L1*** *= 2-(2-pyridyl)benzimidazole;* ***L2*** *= N-((1H-benzo[d]imidazol-2-yl)methyl)quinolin-8-amine.*

[b] *The preparation of Ar_3Al from ArMgCl and $AlCl_3$ produces three equivs of $MgCl_2$. However, $MgCl_2$ appears as an innocent component of the reaction mixture since when commercially available Ph_3Al was used in the optimal experimental conditions (main text Table 1, entry 10), the product* ***3a*** *formed in very similar yield (75% yield), as observed in the presence of $MgCl_2$.*

Table S4. Solvents screening for Co-catalyzed coupling between **1a** and **2b**.[a]

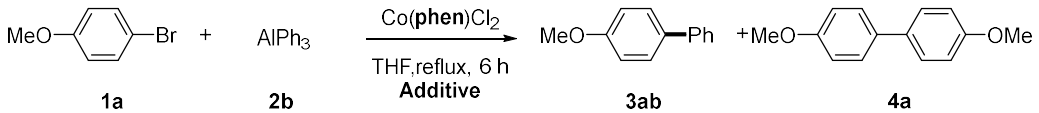


1a **2b** **3ab** **4a**

Entry	**Solvent**	**1a** Conversion (%)	**3** yield (%)	**4a** yield (%)
1	THF[b]	82	61	14
2	2-MeTHF	10	8	N.D
3	Toluene	7	5	N.D
4	CPME	0	N.D.	N.D.

[a]*Reaction conditions:* ***1*** *(0.5 mmol),* ***2a*** *(0.75 mmol, 5 mL of a 0.15 M solution in THF), Co(****phen****)Cl₂ (0.05 mmol, 10 mol%).* 2-MeTHF: 2-methyl tetrahydrofuran; CPME: cyclopentyl methyl ether. [b] T = 66 °C.

Table S5. Additives screening for Co-catalyzed coupling between **1a** and **2b.**[a]



1a **2b** **3ab** **4a**

Entry	**Additive**	**1a** Conversion (%)	**3a** yield (%)	**4a** yield (%)
1	Pyridine	100	77	23
2	NEt_3	100	76	16
3	DMF	100	76	24
4	Butylamine	20	14	N.D.
	Piperidine	13	8	<2
	Pirrolidine	9	5	N.D.

[a]*Reaction conditions:* ***1*** *(0.5 mmol),* ***2a*** *(0.75 mmol, 5 mL of a 0.3 M solution in THF), Co(****phen****)Cl₂ (0.05 mmol, 10 mol%),* ***additive*** *(0.5 mmol).*

3. Supplementary Figures and Schemes

Figure S1. Bromides **1b-u** and organoaluminum reagents **2b-g** employed in Table 2.

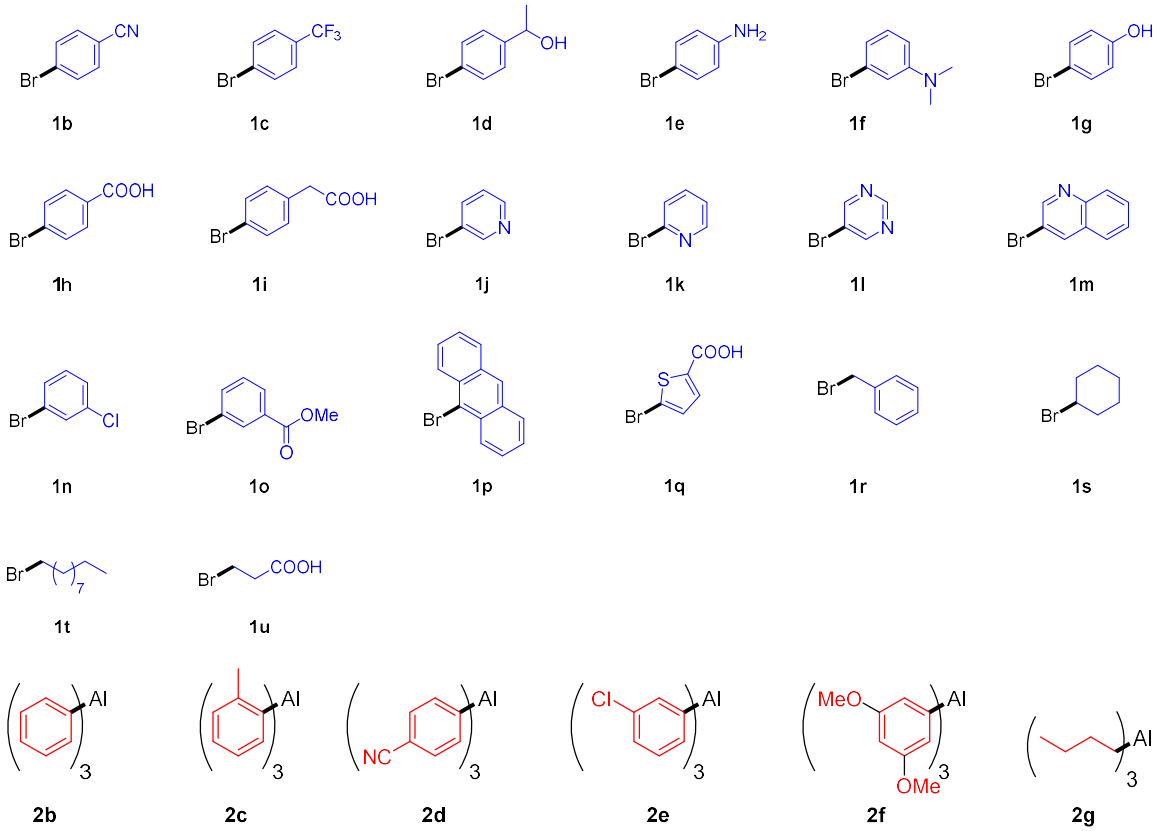
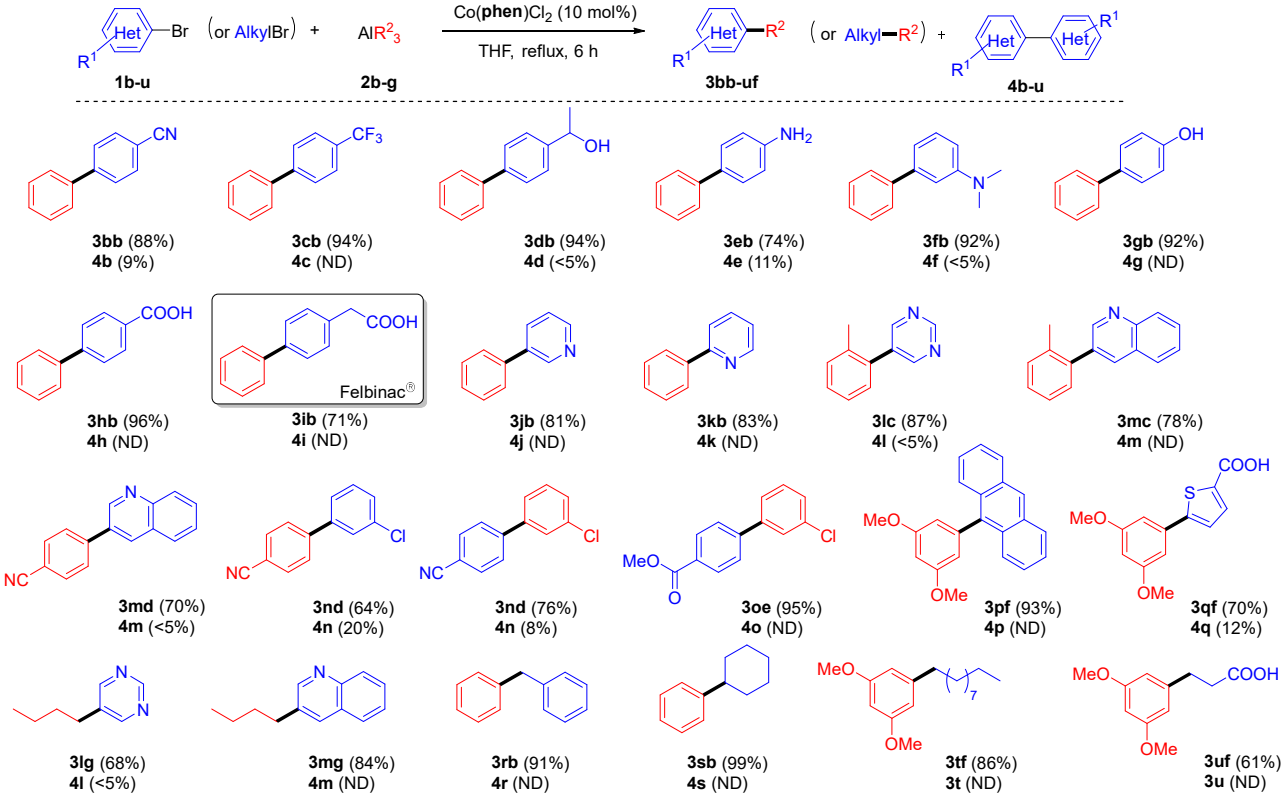
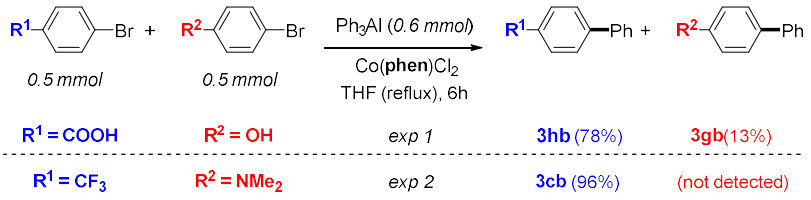


Figure S2. Yields of bromides homocoupling by-products **4b-u** formed during the preparation of corresponding cross-coupling products **3bb-uf**[2]



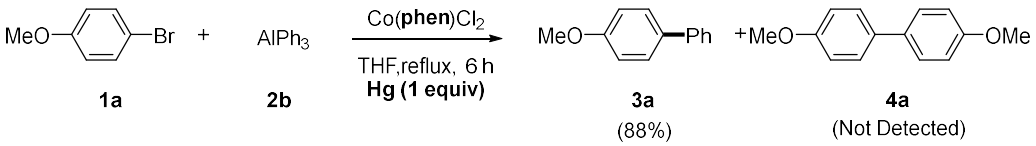
Scheme S1. Competition experiments between differently substituted aryl bromides[a]



Competition experiments procedure: To a solution of $AlCl_3$ (0.6 mmol, 80.0 mg) in 1 mL of freshly distilled THF, phenylmagnesium chloride (1.8 mmol, 0.9 mL, 2.0 M in THF) was added dropwise at 0°C and stirred for 45 min under a nitrogen atmosphere. The solution was warmed up to room temperature, afterwards R^1-bromide (0.5 mmol), R^2-bromide (0.5 mmol) and $Co(Phen)Cl_2$ (10 mol%, 0.05 mmol, 15.5 mg) were sequentially added, under nitrogen stream. The mixture was refluxed for 6 h under magnetic stirring. After this time, the reaction mixture was cooled to room temperature, quenched with brine (2.0 mL), and extracted with ethyl acetate (EtOAc, 3 x 5 mL), the collected organic layers were dried over Na_2SO_4, filtered through a Celite pad and the solvent was removed under reduced pressure. The crude was purified by flash column chromatography on silica gel (hexane/EtOAc 99/1 - 90/10) to provide the products.

[2] Yields of homocoupling products **4b-u** have been determined by ^1H NMR analysis on the crude reaction mixture by means of internal standard (dimethyl sulfone) technique.

Scheme S2. Mercury drop test



1a **2b** **3a** (88%) **4a** (Not Detected)

Mercury drop test procedure: To a solution of $AlCl_3$ (0.75 mmol, 99.8 mg) in 1 mL of freshly distilled THF, PhMgBr (2.25 mmol, 1.13 mL, 2.0 M in THF) was added dropwise at 0°C and the reaction mixture stirred for 45 min under a nitrogen atmosphere. The solution was warmed up to room temperature, afterwards 4-bromoanisole (0.5 mmol, 93.52 mg), $Co(Phen)Cl_2$ (10 mol%, 0.05 mmol, 15.50 mg) and Hg(0) (0.5 mmol, 100.29 mg) were sequentially added, under nitrogen stream. The mixture was refluxed for 6 h under vigorous magnetic stirring. After this time, the reaction mixture was cooled to room temperature, quenched with brine (2.0 mL), and extracted with ethyl acetate (EtOAc, 3 x 5 mL), the collected organic layers were dried over Na_2SO_4, filtered through a Celite pad and the solvent was removed under reduced pressure. The crude was purified by flash column chromatography on silica gel (hexane/EtOAc 99/1 - 90/10) to provide the product **3a**.

4. Experimental Procedures

4.1. Preparation of (4-cyanophenyl)magnesium chloride

To a solution of 4-bromobenzonitrile (2.25 mmol, 409.5 mg) in 1 mL of freshly distilled THF, *i*-PrMgCl·LiCl (2.48 mmol, 1.9 mL, 1.3 M in THF) was added dropwise at 0°C. The resulting mixture was stirred at the above temperature for 3 h under a nitrogen atmosphere and used as such.

4.2. Cross-coupling between dimethyl(phenyl)aluminum reagents and (hetero)aryl bromides in dry THF. General procedure

To a solution of Me_2AlCl (0.75 mmol, 750 µL, 1.0 M in hexane) in 3 mL of freshly distilled THF, PhMgCl (0.75 mmol, 375 µL 2.0 M in THF) was added dropwise at 0 °C and stirred for 45 min under nitrogen atmosphere. The solution was warmed up to room temperature, afterwards (hetero)aryl bromide (0.5 mmol) and Co(II) precatalyst (10 mol%, 0.05 mmol) were sequentially added, under nitrogen stream. The mixture was refluxed for 6 h under magnetic stirring. At the end, the reaction mixture was cooled to room temperature, quenched with Brine (2.0 mL), and extracted with ethyl acetate (EtOAc, 3 x 5 mL), the collected organic layers were dried over Na_2SO_4, filtered through a Celite pad and the solvent was removed under reduced pressure. The crude was purified by column chromatography on silica gel (hexane/EtOAc 99/1 ÷ 90/10) to provide the product **3**.

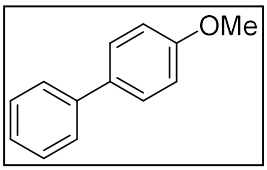
4.3. Synthesis of products 3 by cross-coupling reactions between triaryl- and alkylaluminum reagents and (hetero)aryl bromides in dry THF. General procedure

To a solution of $AlCl_3$ (0.75 mmol, 99.8 mg) in 1 mL of freshly distilled THF, the alkyl or aryl Grignard reagent (2.25 mmol) was added dropwise at 0°C and stirred for 45 min under a nitrogen atmosphere. The solution was warmed up to room temperature, afterwards aryl or alkyl bromide (0.5 mmol) and $Co(Phen)Cl_2$ (10 mol%, 0.05 mmol, 15.5 mg) were sequentially added, under nitrogen stream. The mixture was refluxed for 6 h under magnetic stirring. At the end, the reaction mixture was cooled to room temperature, quenched with brine (2.0 mL), and extracted with ethyl acetate (EtOAc, 3 x 5 mL), the collected organic layers were dried over Na_2SO_4, filtered through a Celite pad and the solvent was removed under reduced pressure. The crude was purified by flash column chromatography on silica gel (hexane/EtOAc 99/1 ÷ 90/10) to provide the product **3**. For carboxylic acids **3hb**, **3ib**, **3qf**, **3uf** hexane/acetone/acetic acid 80/20/2.5 was used as the eluent.

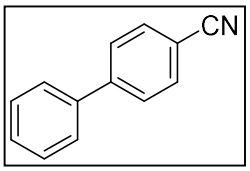
4.4. Synthesis of 4-methoxy-1,1'-biphenyl (3ab) by cross-coupling reaction between 4-bromoanisole (1a) and triphenylaluminium (2b) on a gram-scale

In 250 mL Schlenk flask, $AlCl_3$ (16.05 mmol, 2.140 g) was added in 21.4 mL of freshly distilled THF at 0 °C and dissolved, under magnetic stirring, at room temperature. PhMgCl (48.15 mmol, 24.08 mL, 2.0 M in THF) was added dropwise at 0 °C and solution was stirred for 45 min under a nitrogen atmosphere. The solution was warmed up to room temperature, afterwards 4-bromoanisole (10.7 mmol, 2.0 g) and Co(Phen)Cl_2 (10 mol%, 1.07 mmol, 331.7 mg) were sequentially added, under nitrogen stream. The mixture was refluxed for 6 h under magnetic stirring. Then, the reaction mixture was cooled to room temperature, quenched with Brine (10.0 mL), and extracted with ethyl acetate (EtOAc, 3 x 15 mL). The collected organic layers were dried over Na_2SO_4, filtered through a Celite pad and the solvent was removed under reduced pressure. The crude was purified by column chromatography on silica gel (hexane/EtOAc 99/1) to provide the product **3ab** 78% yield (1.54 g).

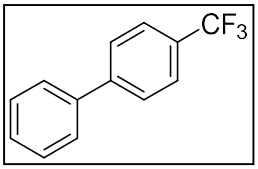
5. Spectroscopic data



4-methoxy-1,1'-biphenyl (3a)[3]: colourless solid, m.p. 89–90 °C, 82%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.58–7.53 (m, 4H), 7.45–7.41 (m, 2H), 7.34–7.29 (m, 1H), 7.00–6.98 (m, 2H), 3.86 (s, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): 159.1, 140.8, 133.7, 128.7, 128.1, 126.7, 126.6, 114.1, 55.3; FT-IR (KBr, cm^{-1}): 3064, 3032, 2979, 1606, 1251, 1035; GC/MS (70 eV) *m/z* (%): 184 [M^+, 100], 169 (77), 141 (56), 115 (35), 43 (34), 40 (56).HRMS (ESI) *m/z* calcd. for $[C_{13}H_{12}O + H]^+$ 185.0961; found: 185.0968.

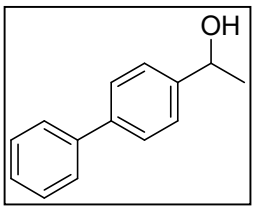


[1,1'-Biphenyl]-4-carbonitrile (3bb)[4]: colourless solid, m.p. 84–85 °C, 88%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.75–7.68 (m, 4H). 7.61–7.58 (m, 2H), 7.51–7.41 (m, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 145.6, 139.2, 132.6, 129.1, 128.6, 127.7, 127.2, 118.9, 110.9; FT-IR (KBr, cm^{-1}): 3401, 3059, 2223, 1603, 1482, 764; GC/MS (70 eV) m/z (%): 179 (M^+ , 100), 178 (23), 151 (12), 102 (15). HRMS (ESI) m/z calcd. for $[C_{13}H_9N + H]^+$: 180.0808; found: 180.0812.

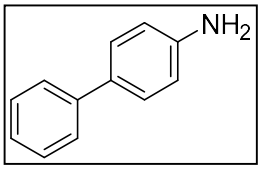


4-(trifluoromethyl)-1,1'-biphenyl (3cb)[5]: white solid, m.p. 70–72 °C, 94%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.75–7.70 (m, 4H), 7.64–7.62 (m, 2H), 7.53–7.48 (m, 2H), 7.47–7.42 (m, 1H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 144.7, 139.7, 129.4 (q, ^{2}J = 32.4 Hz), 128.9, 128.1, 127.4, 127.2, 125.5 (q, ^{3}J = 3.8 Hz), 124.1 (q, ^{1}J = 272.4 Hz); FT-IR (KBr, cm^{-1}): 2923, 1613, 1570, 1489, 1404, 1325, 1274, 1207, 1161, 1111, 1071, 1005, 953, 914, 890, 878, 842, 766, 726, 689; GC/MS (70 eV) *m/z* (%): 223 (15), 222 (100), 201 (7), 153 (24), 152 (29), 151 (10), 86 (7). HRMS (ESI) m/z calcd. for $[C_{13}H_9F_3 + H]^+$: 223.0729; found: 223.0740.

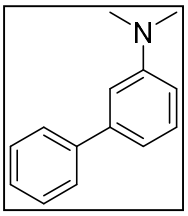
[3] S. Bhadra, W.I. Dzik, L.J. Gooßen, *Angew. Chem. Int. Ed.*, 2013, **52** ,2959.
[4] G. Dilauro, C. S. Azzollini, P. Vitale, A. Salomone, F. M. Perna, V. Capriati, *Angew. Chem. Int. Ed.,* 2021, **60**, 10632.
[5] A. D. Benischke, L. Anthore-Dalion, F. Kohl, P. Knochel, *Chem. Eur. J.,* 2018, **24**, 11103.



1-([1,1'-biphenyl]-4-yl)ethan-1-ol (3db)[6]: white solid, m.p. 96–98 °C, 94%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.61–7.57 (m, 4H), 7.47–7.42 (m, 4H), 7.37–7.32 (m, 1H), 4.96 (q, *J* = 6.4 Hz, 1H), 1.81 (br s, 1H), 1.55 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 144.7, 140.8, 140.4, 128.7, 127.28, 127.27 127.0, 125.8, 70.1, 25.1; FT-IR (KBr, cm^{-1}): 3300, 2970, 1483, 1193, 1001, 1068, 833, 802,759, 686, 505; GC/MS (70 eV) *m/z* (%): 198 (M^+, 5), 181 (55), 166 (100), 153 (17). HRMS (ESI) m/z calcd. for $[C_{14}H_{14}O + Na]^+$: 221.0937; found: 221.0946.

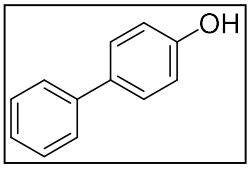


(3eb)[7]: pale brown solid, m.p. 54–55 °C, 74%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.54–7.53 (m, 2H), 7.44–7.38 (m, 4H), 7.28–7.25 (m, 1H), 6.79–6.75 (m, 2H), 3.86 (br s, 2H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 145.7, 141.1, 131.7, 128.6, 128.0, 126.4, 126.2, 115.5; FT-IR (KBr, cm^{-1}): 3422, 3391, 3297, 3199, 3029, 1971, 1631, 1600, 1519, 1482, 1455, 1269, 1257, 1176, 1158, 1131, 1079, 1038, 1003, 967, 945, 853, 831; GC/MS (70 eV) *m/z* (%): 169 (M^+, 100), 115 (17), 83 (11). HRMS (ESI) m/z calcd. for $[C_{12}H_{11}N + H]^+$: 170.0964; found: 170.0971.

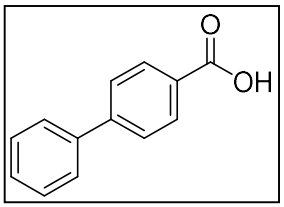


N,N-dimethyl-[1,1'-biphenyl]-3-amine (3fb)[8]: pale yellow oil, 92%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.62–7.60 (m, 2H), 7.46–7.43 (m, 2H), 7.37–7.32 (m, 2H), 6.99–6.97 (m, 2H), 6.80–6.78 (m, 1H), 3.02 (s, 6H) ppm; ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 150.8, 142.23, 142.16, 129.4, 128.5, 127.3, 127.1, 115.9, 111.7, 111.6, 40.7; FT-IR (Film, cm^{-1}): 3008, 2885, 2314, 1847, 1601, 1568, 1487, 1214, 865, 742; GC/MS (70 eV) *m/z* (%): 197(M^+, 100), 196 (99), 153 (15), 152 (23), 98 (17), 76(10), 42(9). HRMS (ESI) m/z calcd. for $[C_{14}H_{15}N + H]^+$: 198.1277; found: 198.1290.

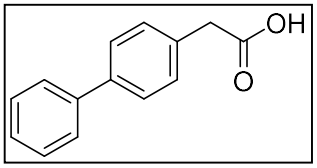
[6] I. Khan, B. G. Reed-Berendt, R. L. Melen, L. C. Morrill, *Angew. Chem. Int. Ed.,* 2018, **57**, 12356.
[7] K. Maeda, R. Matsubara, M. Hayashi, *Org. Lett.,* 2021, **23**,1530.
[8] J. Becht, A. Gissot, . Wagner, C. Mioskowski, *Chem. Eur. J.,* 2003, **9**, 3209.



[1,1'-Biphenyl]-4-ol (3gb)[9]: colourless solid, m.p. 165–166 °C, 92%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.55–7.53 (m, 2H), 7.49–7.47 (m, 2H), 7.43–7.40 (m, 2H), 7.34–7.29 (m, 1H), 6.92–6.90 (m, 2H), 4.90 (br s, 1H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 155.0, 140.7, 134.0, 132.4, 128.6, 128.3, 126.7, 115.6; FT-IR (KBr, cm^{-1}): 3607, 3085, 3036, 1586, 1479, 1438, 1336, 1234, 1183; GC/MS (70 eV) *m/z* (%): 170 (M^+ , 100), 169 (91), 141 (36), 115 (24). HRMS (ESI) *m/z* calcd. for $[C_{12}H_{10}O + H]^+$: 171.0804; found: 171.0807.

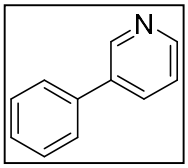


[1,1'-Biphenyl]-4-carboxylic acid (3hb)[10]: white solid, m.p. 222–224 °C, 96%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 8.20–8.18 (m, 2H), 7.72–7.70 (m, 2H), 7.66–7.64 (m, 2H), 7.51–7.41 (m, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 171.0, 146.5, 139.8, 130.7, 130.1, 128.9, 128.4, 127.3, 127.1; FT-IR (KBr, cm^{-1}): 3058, 3018, 2977, 1700, 1695, 1276, 1148, 839, 743, 698; GC/MS (70 eV) *m/z* (%): 198 (M^+, 19), 197 (100), 180 (13), 153 (41), 121 (36). HRMS (ESI) m/z calcd. for $[C_{13}H_{10}O_2 + H]^+$: 199.0754; found: 199.0767.

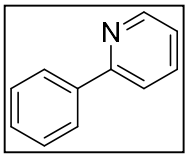


2-([1,1'-biphenyl]-4-yl)acetic acid (3ib) (Felbinac)[11]: white solid, m.p. 161–162 °C, 71%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.59–7.56 (m, 4H), 7.47–7.42 (m, 2H), 7.38–7.34 (m, 3H) 3.71 (s, 2H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 177.4, 140.8, 140.5, 132.4, 130.0, 128.9, 127.9, 127.6, 127.2, 40.8; FT-IR (KBr, cm^{-1}): 3061, 2929, 1683, 1487, 1452, 1412, 1348, 1254, 821, 763, 741, 697; GC/MS (70 eV) *m/z* (%): 212 (M^+ , 53), 167 (100), 152 (16), 115 (9). HRMS (ESI) *m/z* calcd. for $[C_{14}H_{12}O_2 + H]^+$: 213.0910; found: 213.0938.

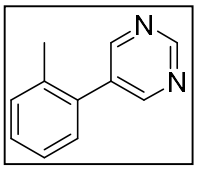
[9] G. Dilauro, S. M. García, D. Tagarelli, P. Vitale, F. M. Perna, V. Capriati, *ChemSusChem* 2018, **11**, 3495.
[10] X. Zhu, Y. Liu, C. Liu, H. Yanga, Hua Fu, *Green Chem.*, 2020, **22**, 4357.
[11] W. Zhang, J. M. Ready, *Angew. Chem. Int. Ed.*, 2014, **53**, 8980.



3-Phenylpyridine (3jb)[12]: white solid, m.p. 128–129 °C, 81%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 8.85–8.84 (m, 1H), 8.59–8.57 (m, 1H), 7.88–7.85 (m, 1H), 7.59–7.56 (m, 2H), 7.49–7.45 (m, 2H), 7.42–7.34 (m, 2H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 148.3, 148.2, 137.7, 136.6, 134.3, 129.0, 128.0, 127.1, 123.5; FT-IR (KBr, cm^{-1}): 3058, 3033, 1580, 1472, 1187, 1076, 1024, 1006, 915, 813, 753, 698; GC/MS (70 eV) m/z (%): 155.1 (M^+, 100), 127.0 (16), 102.0 (14). HRMS (ESI) m/z calcd. For $[C_{11}H_9N + Na]^+$: 178.0627; found: 178.0634.

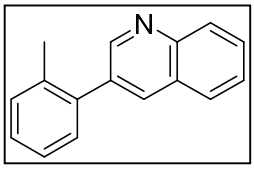


2-Phenylpyridine (3kb)[13]: white solid, m.p. 158–150 °C, 83%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 8.71–8.70 (m, 1H), 8.00–7.98 (m, 2H), 7.78-7.72 (m, 2H), 7.51-7.40 (m, 3H), 7.25-7.22 (m, 1H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 157.4, 149.6, 139.3, 136.7, 128.9, 128.7, 126.9, 122.1, 120.6; FT-IR (KBr, cm^{-1}): 3066, 3010, 1589, 1579, 1468, 1451, 1424, 1020, 1002, 758, 736; GC/MS (70 eV) m/z (%): 155 (M^+, 100), 154 (64), 128 (12), 127 (10), 77 (9), 59 (15), 43 (5). HRMS (ESI) m/z calcd. For $[C_{11}H_9N + Na]^+$: 178.0627; found: 178.0641.

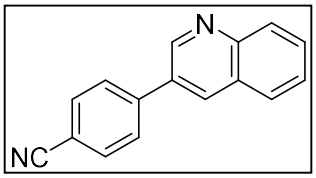


5-(*o*-tolyl)pyrimidine (3lc)[14]: white solid, m.p. 119–122 °C, 87%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 9.20 (s, 1H), 8.74 (s, 2H), 7.38–7.28 (m, 3H), 7.21–7.20 (m, 1H), 2.29 (s, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 157.1, 156.6, 135.6, 135.3, 134.2, 130.8, 129.8, 129.0, 126.4, 20.2; FT-IR (KBr, cm^{-1}): 2919, 2803, 2854, 1548, 1411, 820, 781, 743; GC/MS (70 eV) *m/z* (%): 170 (M^+, 100), 169 (28), 116 (25), 115 (53), 89 (15), 63 (10). HRMS (ESI) *m/z* calcd. For $[C_{11}H_{10}N_2 + Na]^+$: 193.0736; found: 193.0747.

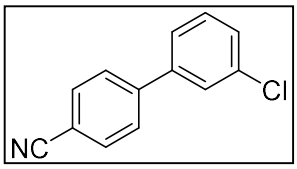
[12] V. Ramakrishna, N. D. Reddy, *Dalton Trans.,* 2017, **46**, 8598.
[13] K. Nozawa-Kumada, Y. Iwakawa, S. Onuma, M. Shigeno Y. Kondo, *Chem. Commun.*, 2020, **56**, 7773.
[14] M. Feuerstein, H. Doucet, M. Santelli, *J. Organomet. Chem.*, 2003, *687*, 327.



3-(*o*-tolyl)quinoline (3mc)[15]: colourless waxy solid, 78%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 8.94 (d, *J* = 2.2 Hz, 1H), 8.25 (d, *J* = 8.5 Hz, 1H), 8.18 (d, *J* = 2.2 Hz, 1H), 7.91–7.88 (m, 1H), 7.81–7.76 (m, 1H), 7.65–7.61 (m, 1H), 7.36-7.32 (m, 4H), 2.34 (s, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 150.5, 145.8, 137.6, 136.4, 135.8, 134.9, 130.7, 130.2, 130.0, 128.4, 127.9, 127.8, 127.3, 126.3, 20.4; FT-IR (KBr, cm^{-1}): 3050, 3033, 2921, 2852, 1616, 1489, 1460, 1358, 1340, 962, 900, 788, 751, 619, 560; GC/MS (70 eV) m/z (%): 219 (M^+, 100), 189 (9), 176 (5), 109 (9). HRMS (ESI) *m/z* calcd. For $[C_{16}H_{13}N + H]^+$: 220.1121; found: 220.1130.

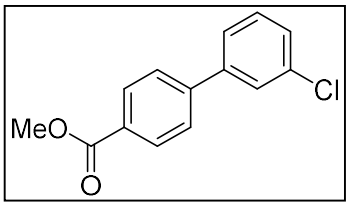


4-(quinolin-3-yl)benzonitrile (3md)[16]: pale yellow solid, m.p. 165–167 °C, 70%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 9.16 (d, *J* = 2.4 Hz, 1H), 8.38 (d, *J* = 2.4 Hz, 1H), 8.19 (d, *J* = 8.5 Hz, 1H), 7.93–7.91 (m, 1H), 7.82- 7.76 (m, 5H), 7.65-7.61 (m, 1H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 148.7, 147.2, 142.1, 134.1, 133.0, 131.9, 130.6, 128.8, 128.2, 128.0, 127.8, 127.7, 118.5, 111.9; FT-IR (KBr, cm^{-1}): 3033, 2922, 2857, 2225, 1739, 1600, 1571, 1493, 1428, 1366, 1339, 1312, 1217, 1192, 1042, 956, 864, 840, 745; GC/MS (70 eV) m/z (%): 230 (M^+ , 100), 201 (9), 175 (12), 88 (11). HRMS (ESI) *m/z* calcd. For $[C_{16}H_{10}N_2 + H]^+$: 231.0917; found: 231.0929.

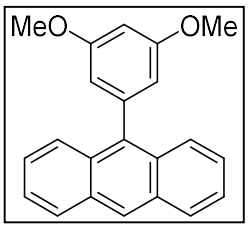


3'-chloro-[1,1'-biphenyl]-4-carbonitrile (3nd)[17]: white solid, m.p. 58–60 °C, 64% from (4-cyanophenyl)magnesium chloride, and 76% from (3-chlorophenyl)magnesium bromide. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.75–7.73 (m, 2H), 7.67–7.65 (m, 2H), 7.57 (s, 1H), 7.48-7.40 (m, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 144.1, 140.9, 135.0, 132.6, 130.3, 128.6, 127.7, 127.3, 125.3, 118.6, 111.6; FT-IR (KBr, cm^{-1}): 2228, 1646, 1573, 1485, 1146, 1132, 1093, 952, 857, 812, 761; GC/MS (70 eV) m/z (%): 213 (M^+, 100), 177 (20), 151 (15), 75 (5). HRMS (ESI) *m/z* calcd. For $[C_{13}H_8CN + H]^+$: 214.0418; found: 214.0429.

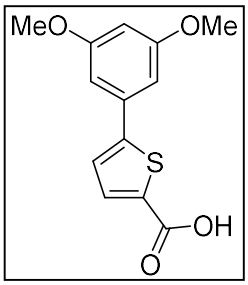
[15]. V.K. Tiwari, G.G. Pawar, R. Das, A. Adhikary, M. Kapur, *Org. Lett.,* 2013, **15**, 3310.
[16] S. Bernhardt, G. Manolikakes, T. Kunz, P. Knochel, *Angew. Chem. Int. Ed.,* **50**, 9205.
[17] N. Sinha, P. A. Champagne, M. J. Rodriguez, Y. Lu, M. E. Kopach, D. Mitchell, M. G. Organ, *Chem. Eur. J.*, 2019, **25**, 6508.



Methyl 3'-chloro-[1,1'-biphenyl]-4-carboxylate (3oe)[18]: colourless oil, 95%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 8.13–8.10 (m, 2H), 7.65–7.60 (m, 3H), 7.51–7.49 (m, 1H), 7.42–7.36 (m, 2H), 3.95 (s, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 166.7, 144.1, 141.8, 134.8, 130.1, 129.4, 128.8, 128.1, 127.4, 127.0, 125.4, 52.1; FT-IR (KBr, cm^{-1}): 2962, 1727, 1433, 1291, 1115, 947, 828, 807, 770, 746; GC/MS (70 eV) m/z (%): 246 (M^+ , 58), 217 (36), 216 (15), 215 (100), 153 (15), 152 (94), 151 (23), 150 (9). HRMS (ESI) *m/z* calcd. For $[C_{14}H_{11}ClO_2 + H]^+$: 247.0520; found: 247.0531.

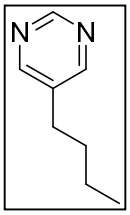


9-(3,5-Dimethoxyphenyl)anthracene (3pf)[19]: white solid, m.p. 144–146 °C, 93%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 8.49 (s, 1H), 8.05–8.03 (m, 2H), 7.77–7.75 (m, 2H), 7.48–7.44 (m, 2H), 7.39–7.35 (m, 2H), 6.66–6.65 (m, 1H), 6.62–6.61 (m, 2H), 3.83 (s, 6H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 160.7, 140.8, 136.8, 131.2, 129.9, 128.2, 126.8, 126.5, 125.3, 125.1, 109.2, 99.7, 55.4; FT-IR (KBr, cm^{-1}): 3047, 2916, 2815, 2160, 1440, 889, 845, 803, 786, 738; GC/MS (70 eV) m/z (%): 314(M^+, 100), 283 (20), 239 (15), 215 (11), 119 (9). HRMS (ESI) *m/z* calcd. For $[C_{22}H_{18}O_2 + H]^+$: 315.1380; found: 315.1391.

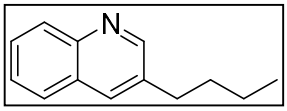


5-(2,6-dimethoxyphenyl)thiophene-2-carboxylic acid (3qf): white solid, m.p. 151–153, 70%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.83 (d, *J* = 3.7 Hz, 1H), 7.30 (d, *J* = 3.7 Hz, 1H), 6.78 (d, *J* = 2.2 Hz, 2H), 6.49–6.48 (m, 1H), 3.84 (s, 6H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 161.6, 157.3, 152.6, 135.6, 135.0, 124.1, 107.3, 104.6, 101.0, 55.4; FT-IR (KBr, cm^{-1}): 3483, 2963, 2848, 2648, 1673, 1590, 1533, 1428, 1348, 1077, 919, 861, 804, 747, 673; GC/MS (70 eV) m/z (%): 220 (M^+, 100), 191 (36), 147 (11), 134 (13), 115 (9), 89 (7). HRMS (ESI) *m/z* calcd. For $[C_{13}H_{12}O_4S + Na]^+$: 287.0349; found: 287.0362.

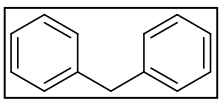
[18] L. Niu, H. Zhang, H. Yang, H. Fu, *Synlett.*, 2014, **25**, 995.
[19] E. Wanga, M. Chen, *Chem. Sci.*, 2019, **10**, 8331.



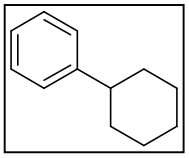
5-Butylpyrimidine (3lg)[2]: colourless oil, 68%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 9.06 (s, 1H), 8.58 (s, 2H), 2.60 (t, *J* = 7.0 Hz, 2H), 1.64–1.57 (m, 2H), 1.41–1.32 (m, 2H), 0.93 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 157.8, 154.8, 128.4, 31.9, 29.3, 22.7, 13.9; FT-IR (film, cm^{-1}): 3014, 2974, 2859, 1661, 1381, 986, 837, 742; GC/MS (70 eV) *m/z* (%): 136 (M^+, 82), 107 (11), 94 (100), 80 (15). HRMS (ESI) *m/z* calcd. for $[C_8H_{12}N_2 + H]^+$: 137.1073; found: 137.1079.



3-Butylquinoline (3mg)[2], light yellow oil, 84%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 8.80–8.79 (m, 1H), 8.11–8.09 (m, 1H), 7.92–7.91 (m, 1H), 7.77–7.75 (m, 1H), 7.66–7.62 (m, 1H), 7.53–7.49 (m, 1H), 2.79 (t, *J* = 7.6 Hz, 2H), 1.73–1.66 (m, 2H), 1.45–1.36 (m, 2H), 0.95 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 152.0, 146.4, 135.4, 134.4, 128.8, 128.6, 128.2, 127.3, 126.5, 33.2, 32.8, 22.2, 13.8; FT-IR (film, cm^{-1}): 2958, 2931, 2870, 2856, 1570, 1498, 1466, 1376, 1319, 1001, 945, 785, 750; GC/MS (70 eV) *m/z* (%): 185 (M^+, 35), 142 (100), 128 (5), 115 (22), 89 (5), 77 (5). HRMS (ESI) *m/z* calcd. for $[C_{13}H_{15}N + H]^+$: 186.1277; found: 186.1290.

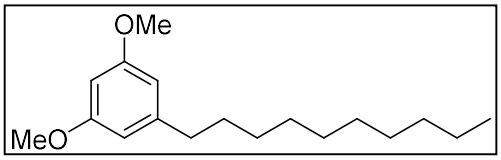


Diphenylmethane (3rb)[20]: colourless oil, 91%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.33– 7.29 (m, 4H), 7.24–7.21 (m, 6H), 4.01 (s, 2H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 141.1, 128.9, 128.4, 126.0, 41.9; FT-IR (KBr, cm^{-1}): 2904, 1606, 1497, 1481, 1452, 1398, 1370, 1276, 1178, 1094, 1082, 1020, 1012, 907, 887, 853, 801, 740, 698; GC/MS (70 eV) m/z (%): 168 (M^+, 94), 167 (100). HRMS (ESI) *m/z* calcd. for $[C_{13}H_{12} + H]^+$: 169.1012; found: 169.1021.

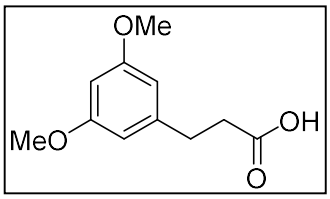


Cyclohexylbenzene (3sb)[21]: colourless oil, 99%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 7.32– 7.24 (m, 2H), 7.23–7.16 (m, 3H), 2.53–2.46 (m, 1H), 1.90–1.82 (m, 4H), 1.78–1.73 (m, 1H), 1.48–1.34 (m, 4H), 1.28–1.25 (m, 1H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 148.2, 128.0, 126.5, 125.8, 44.4, 34.5, 26.9, 26.0; FT-IR (Film, cm^{-1}): 2978, 2041, 1603, 945, 871, 841, 793, 700; GC/MS (70 eV) m/z (%): 160 (M^+, 60), 131 (25), 117 (75), 104 (100), 91 (42), 78 (11). HRMS (ESI) *m/z* calcd. for $[C_{12}H_{16} + H]^+$: 161.1325; found: 161.1342.

[20] L. L. Anka-Lufford, K. M. M. Huihui, N. J. Gower, L. K. G. Ackerman, D. J. Weix, *Chem. Eur. J.,* 2016, **22**, 11564.
[21] J. Zeng, K.M. Liu, X.F. Duan, *Org. Lett.,* 2013, **15**, 5342.



1-decyl-3,5-dimethoxybenzene (3tf): colourless oil, 86%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 6.36–6.35 (m, 2H), 6.31–6.30 (m, 1H), 3.78 (s, 6H), 2.57–2.53 (m, 2H), 1.64–1.56 (m, 2H), 1.31–1.27 (m, 14H), 0.89 (t, *J* = 7.9 Hz, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 160.7, 145.4, 106.5, 97.5, 55.2, 36.3, 31.9, 31.3, 29.62, 29.58, 29.5, 29.34, 29.32, 22.7, 14.1; FT-IR (Film, cm^{-1}): 2997, 1596, 1461, 1453, 1428, 1349, 1292, 1154, 1060, 927, 828, 695; GC/MS (70 eV) m/z (%): 278 (M^+, 20), 194 (9), 165 (21), 152 (100), 121 (19), 91 (17). HRMS (ESI) *m/z* calcd. for $[C_{18}H_{30}O_2 + H]^+$: 279.2319; found: 279.2328.

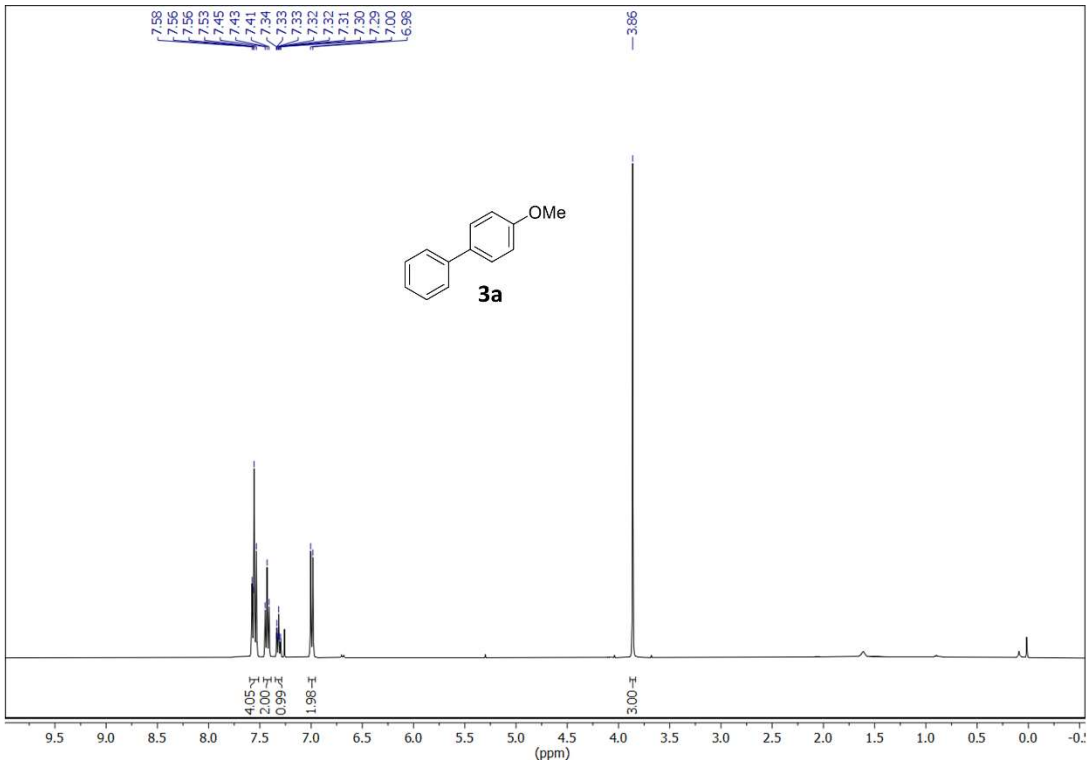


3-(3,5-Dimethoxyphenyl)propanoic Acid (3uf)[22]: white solid, m.p. 59- 61 °C, 61%. ^{1}H NMR (400.12 MHz, $CDCl_3$): δ 6.37–6.36 (m, 2H), 6.33 (s, 1H), 3.78 (s, 6H), 2.90 (dd, ^{2}J = 8.5 Hz, ^{3}J = 7.1 Hz, 2H), 2.67 (dd, ^{2}J = 8.4 Hz, ^{3}J = 7.0 Hz, 2H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ 178.6, 160.9, 142.5, 106.3, 98.3, 55.2, 35.4, 30.8; FT-IR (Film, cm^{-1}): 3400, 3250, 2501, 2851, 2998, 2940, 2836, 1708, 1593, 1157, 820, 735; GC/MS (70 eV) m/z (%): 210 (M^+, 48), 165 (100), 57 (26), 55 (19). HRMS (ESI) *m/z* calcd. for $[C_{11}H_{14}O_4 + Na]^+$: 233.0784; found: 233.0801.

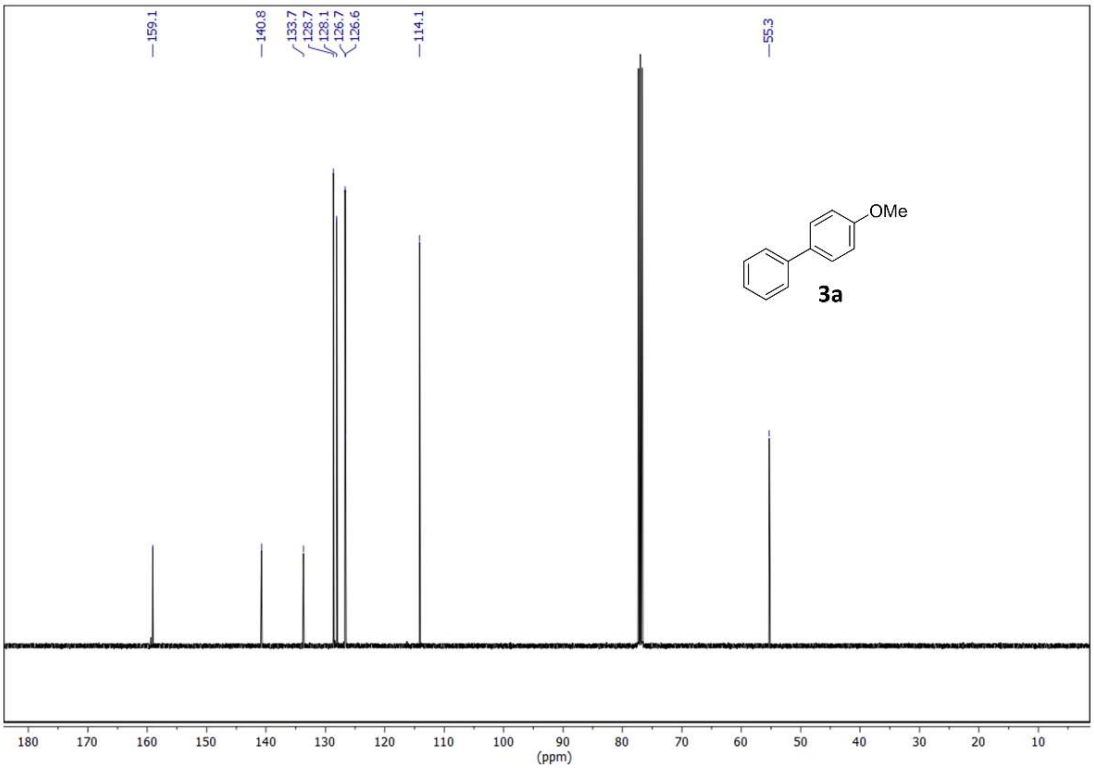
[22] K. Tangdenpaisal, K. Chuayboonsong, P. Sukjarean, V. Katesampao, N. Noiphrom, S. Ruchirawat, P. Ploypradith, *Chem. Asian J.*, 2015, **10**, 1050.

6. ^{1}H and ^{13}C NMR spectra

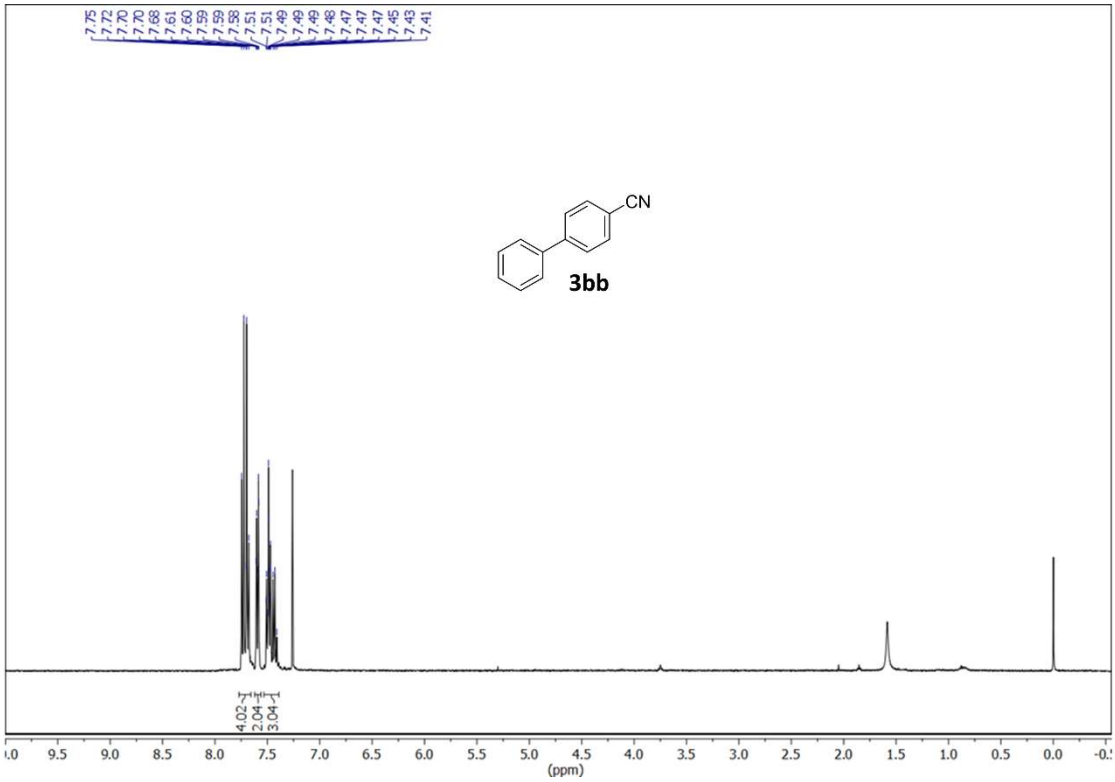
^{1}H NMR 400.12 MHz, $CDCl_3$



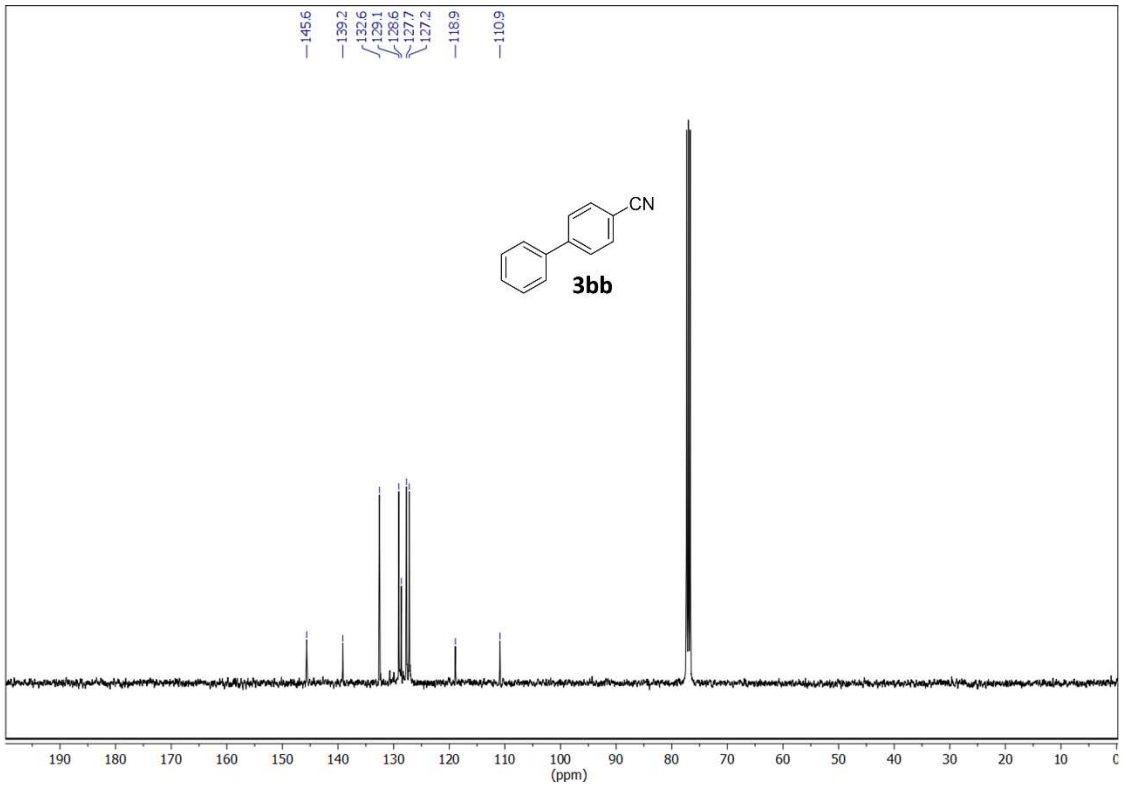
^{13}C NMR 100.62 MHz, $CDCl_3$



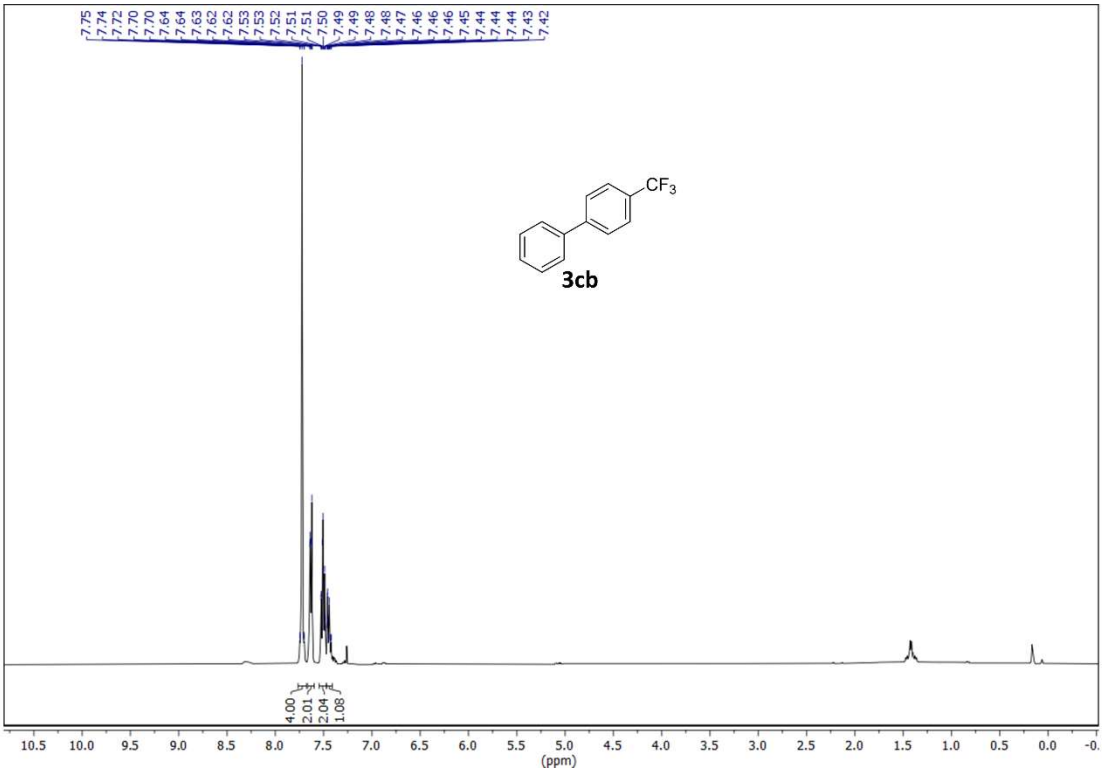
^{1}H NMR 400.12 MHz, $CDCl_3$



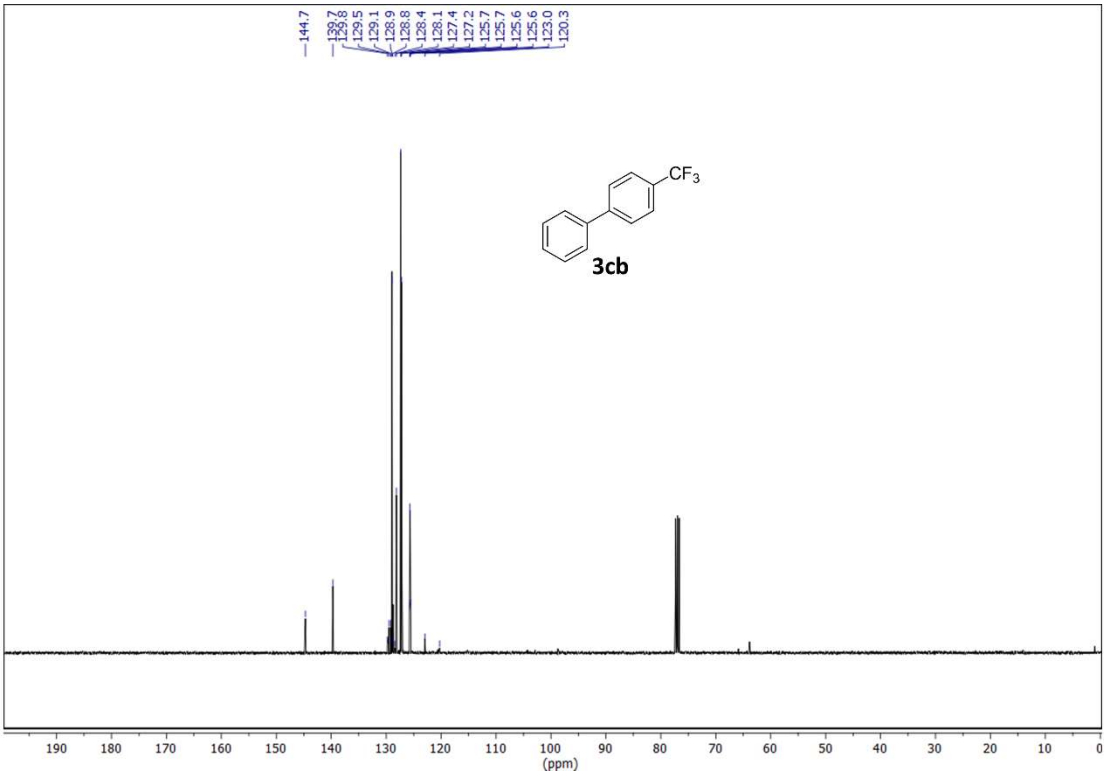
^{13}C NMR 100.62 MHz, $CDCl_3$



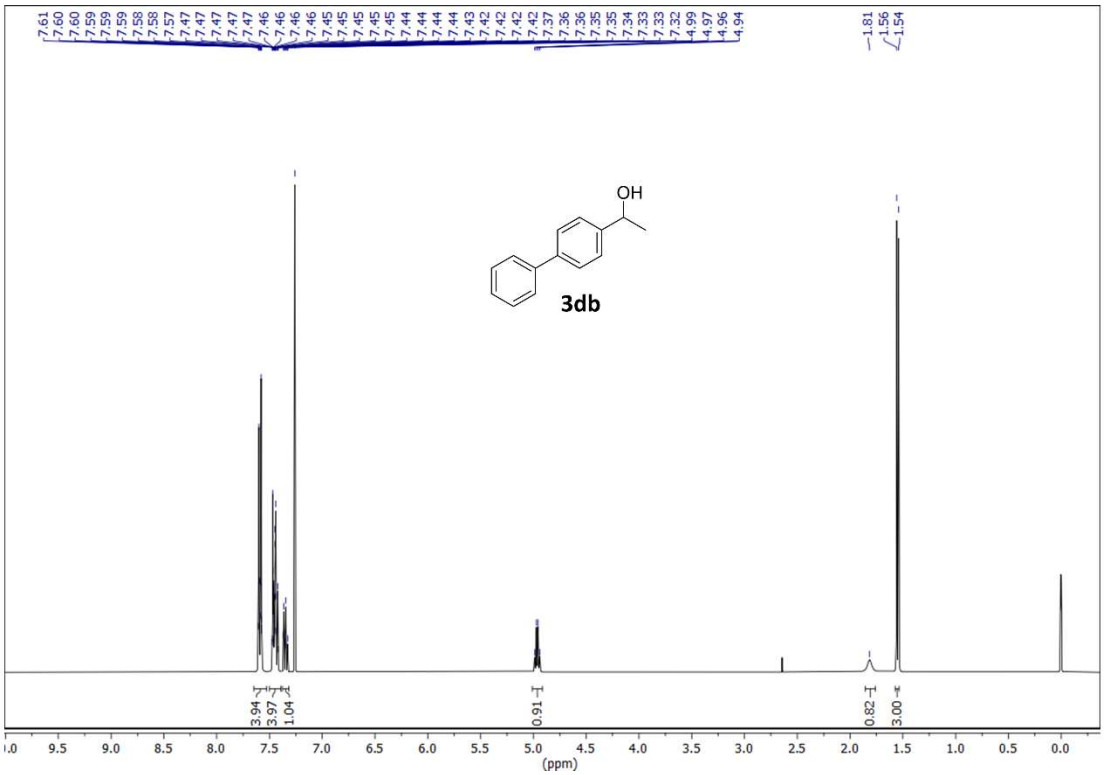
^{1}H NMR 400.12 MHz, $CDCl_3$



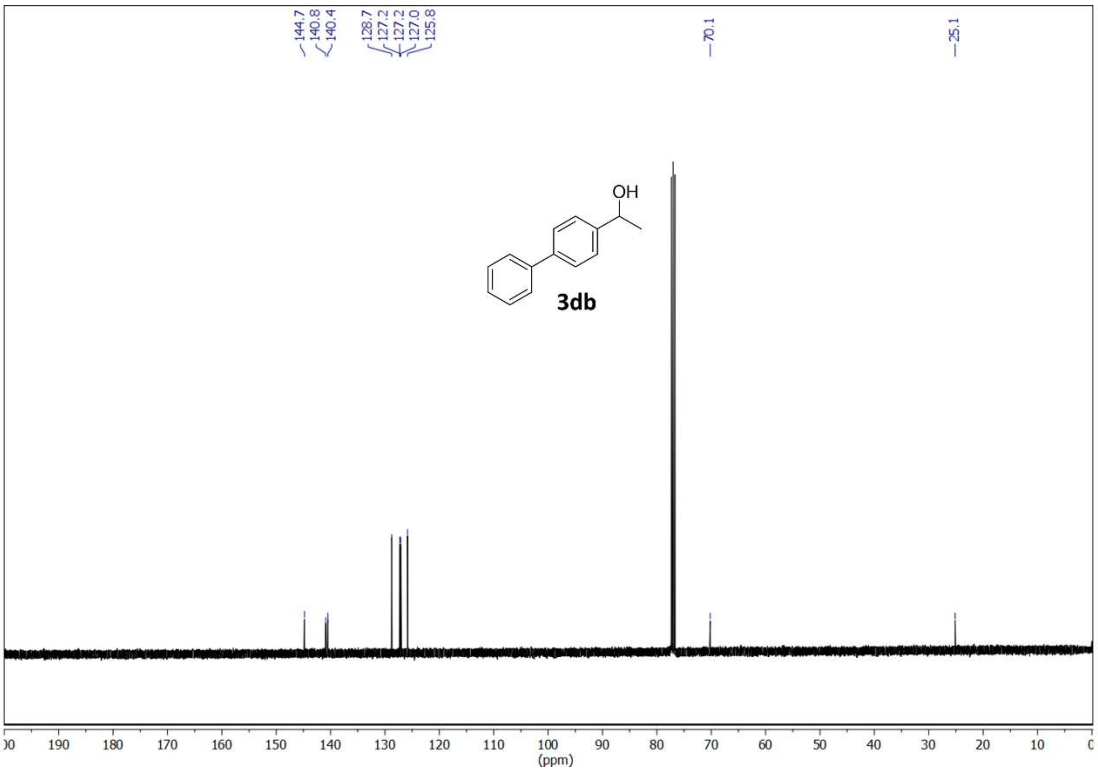
^{13}C NMR 100.62 MHz, $CDCl_3$



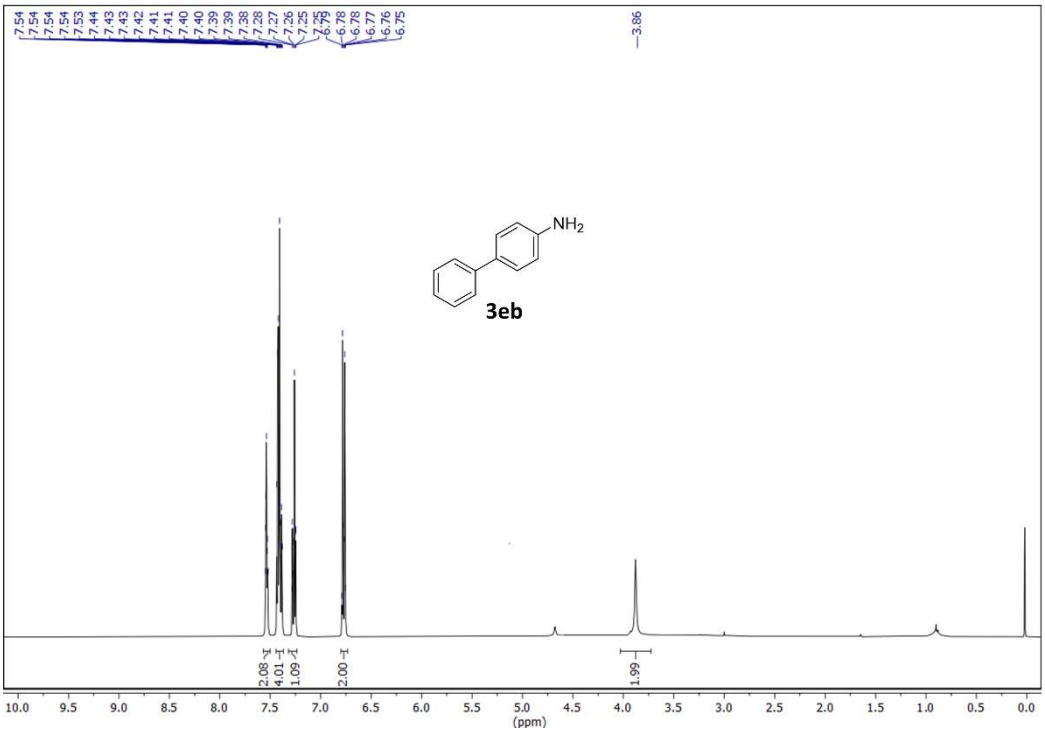
^{1}H NMR 400.12 MHz, $CDCl_3$



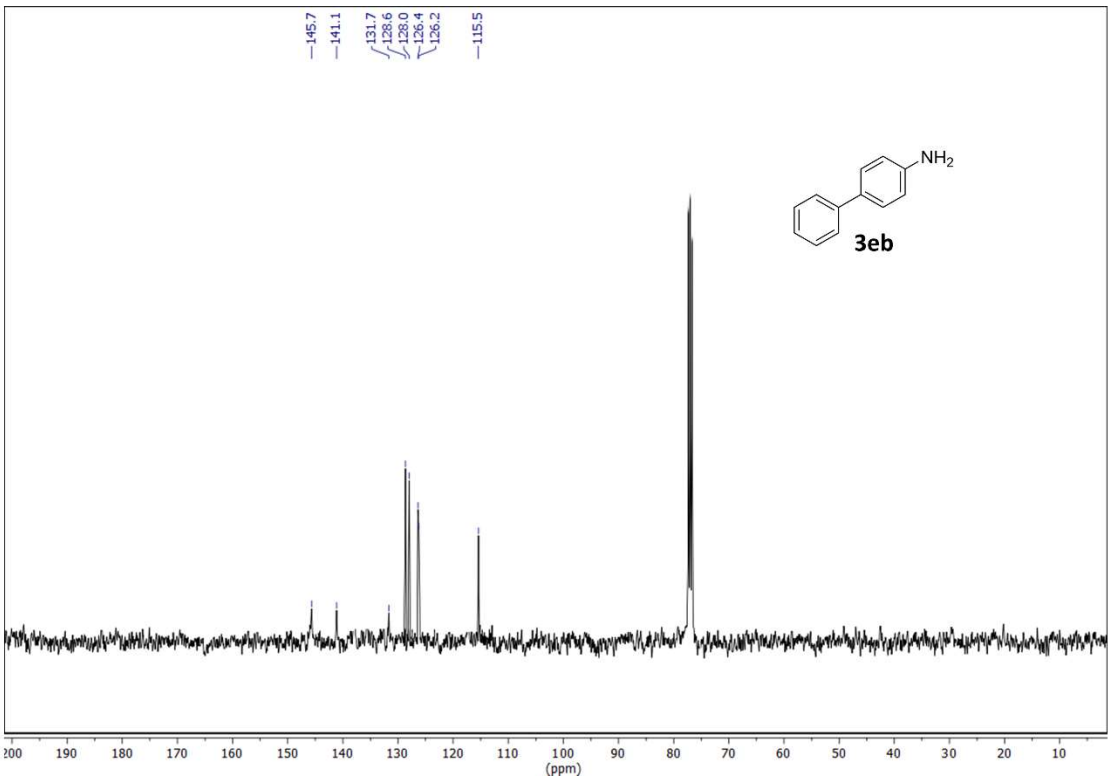
^{13}C NMR 100.62 MHz, $CDCl_3$



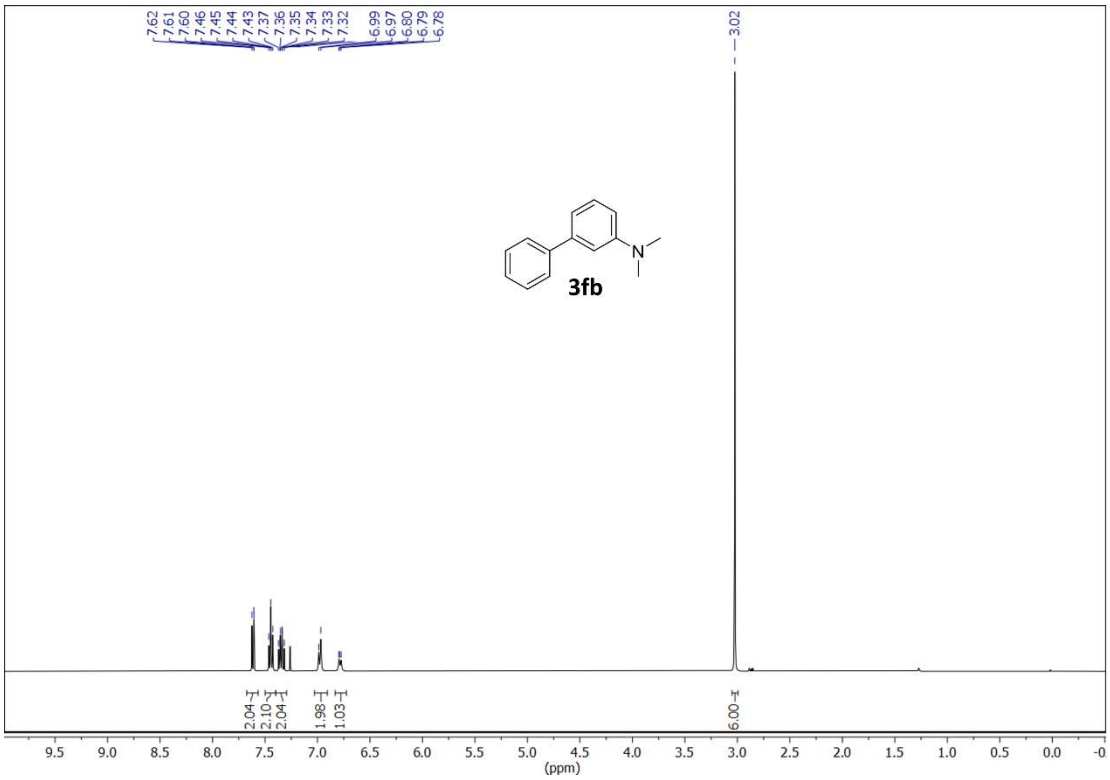
^{1}H NMR 400.12 MHz, $CDCl_3$



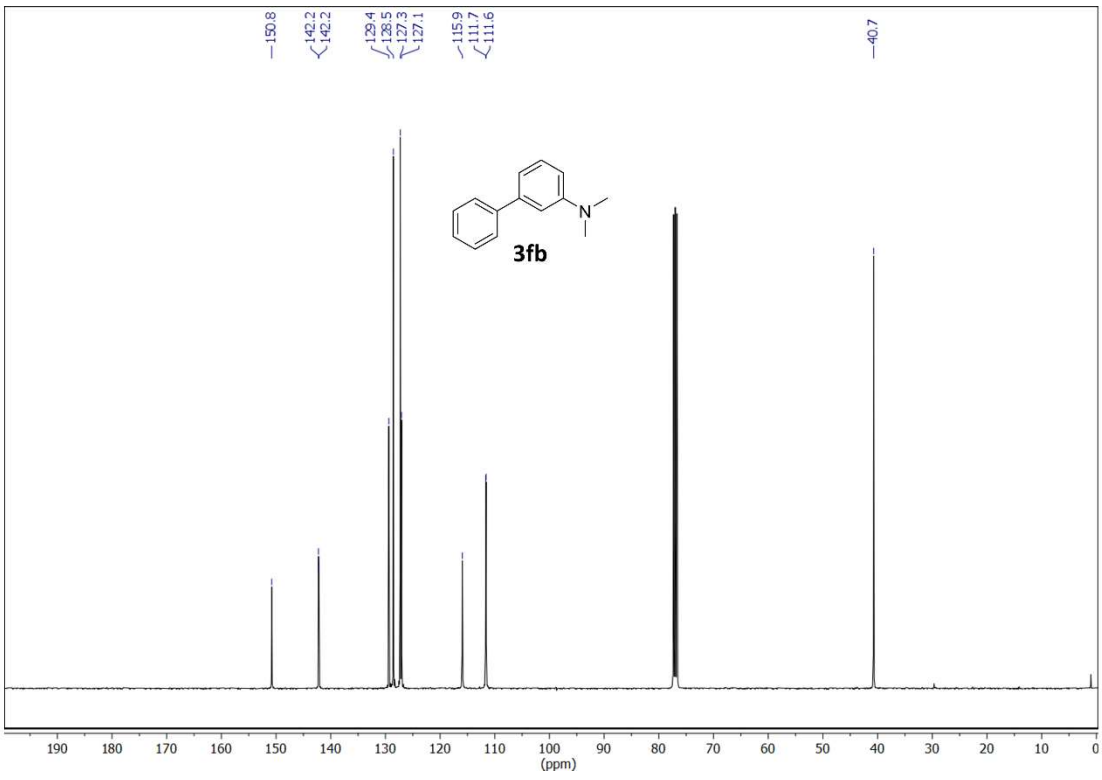
^{13}C NMR 100.62 MHz, $CDCl_3$



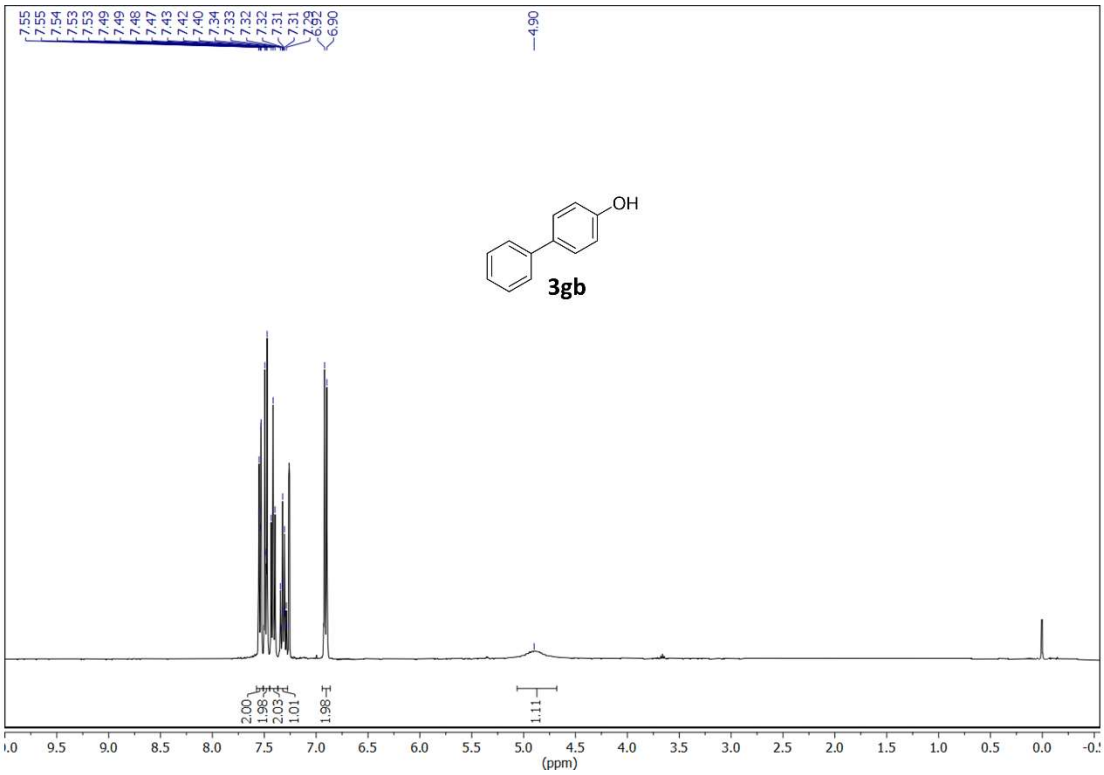
^{1}H NMR 400.12 MHz, $CDCl_3$



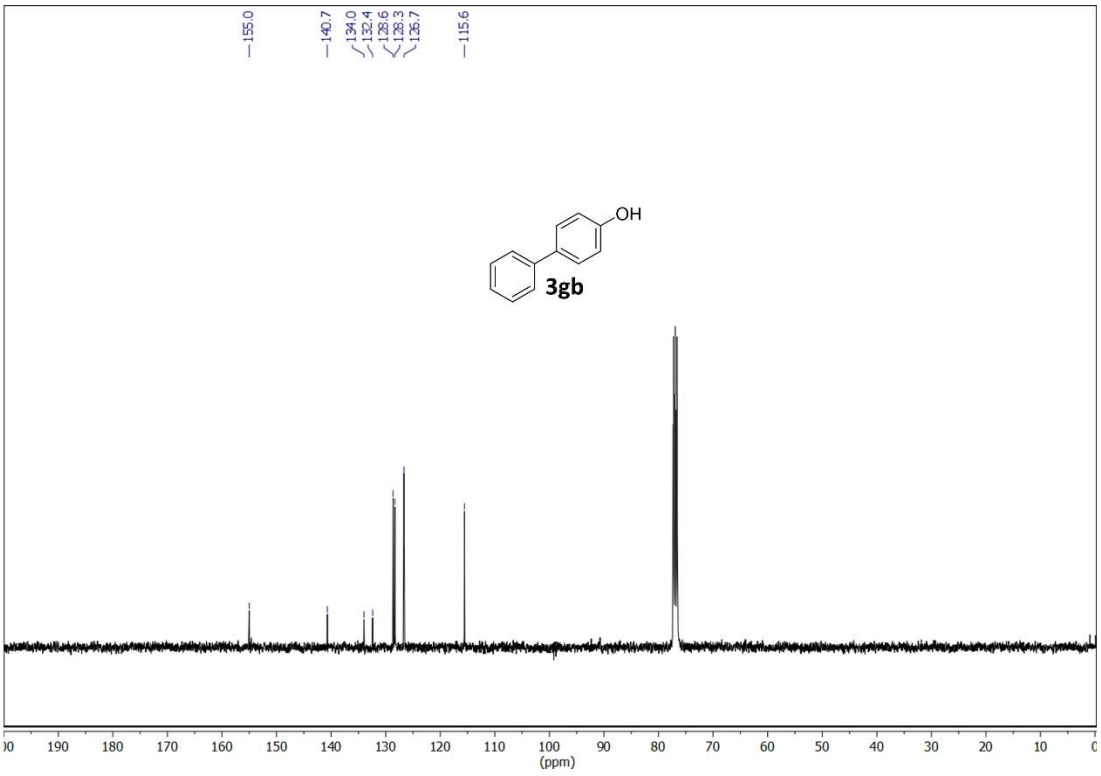
^{13}C NMR 100.62 MHz, $CDCl_3$



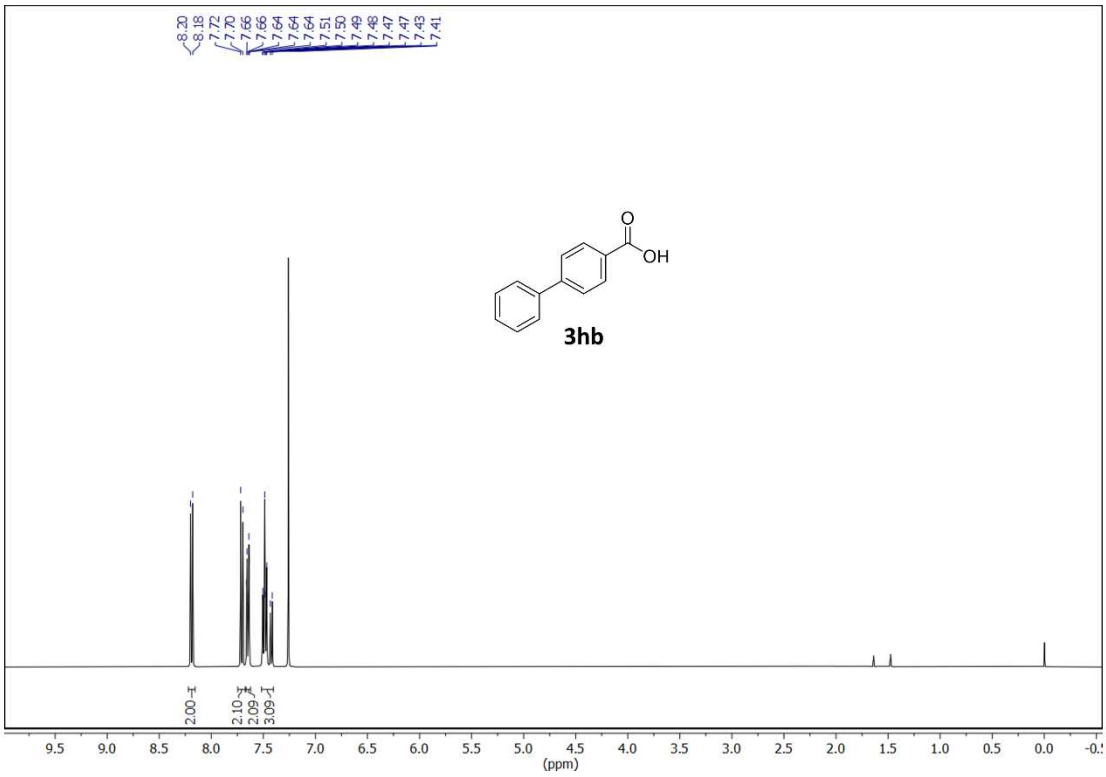
^{1}H NMR 400.12 MHz, $CDCl_3$



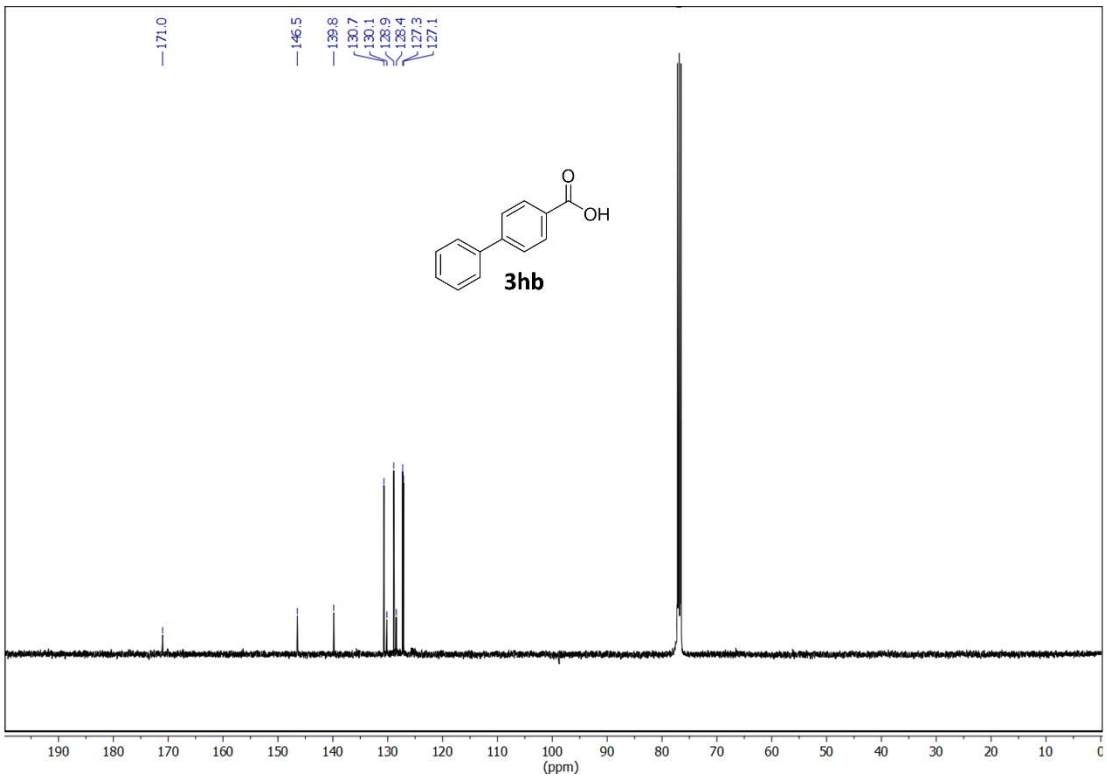
^{13}C NMR 100.62 MHz, $CDCl_3$



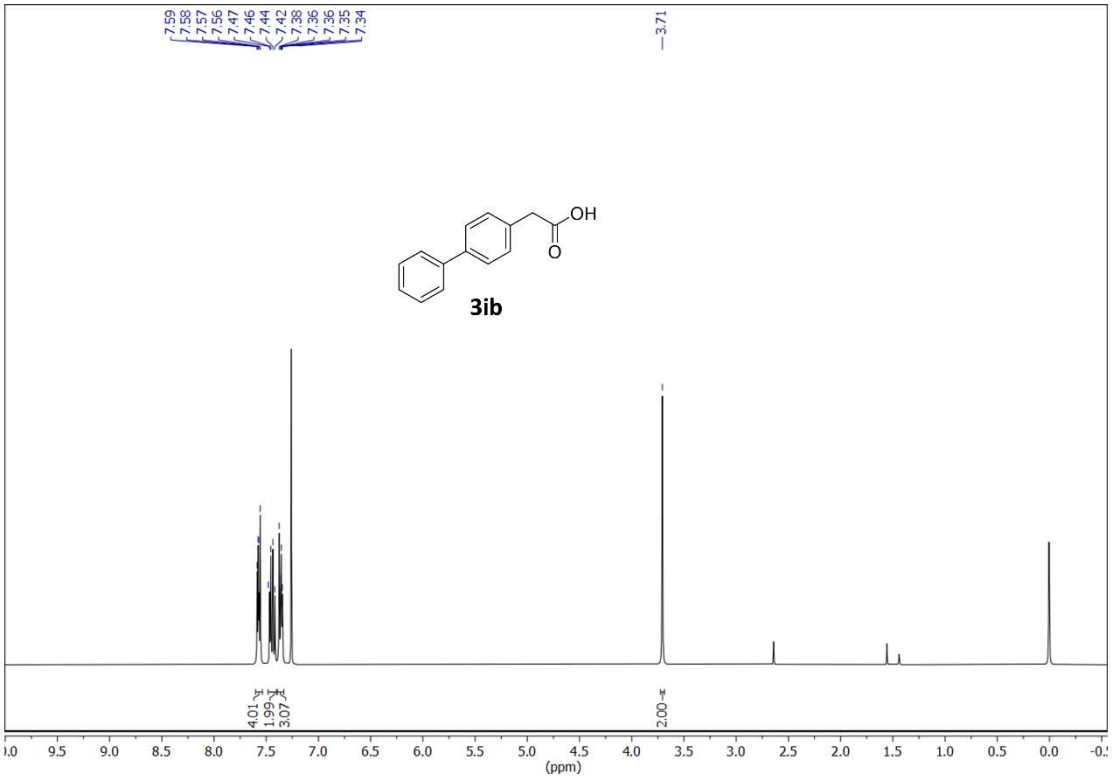
^{1}H NMR 400.12 MHz, $CDCl_3$



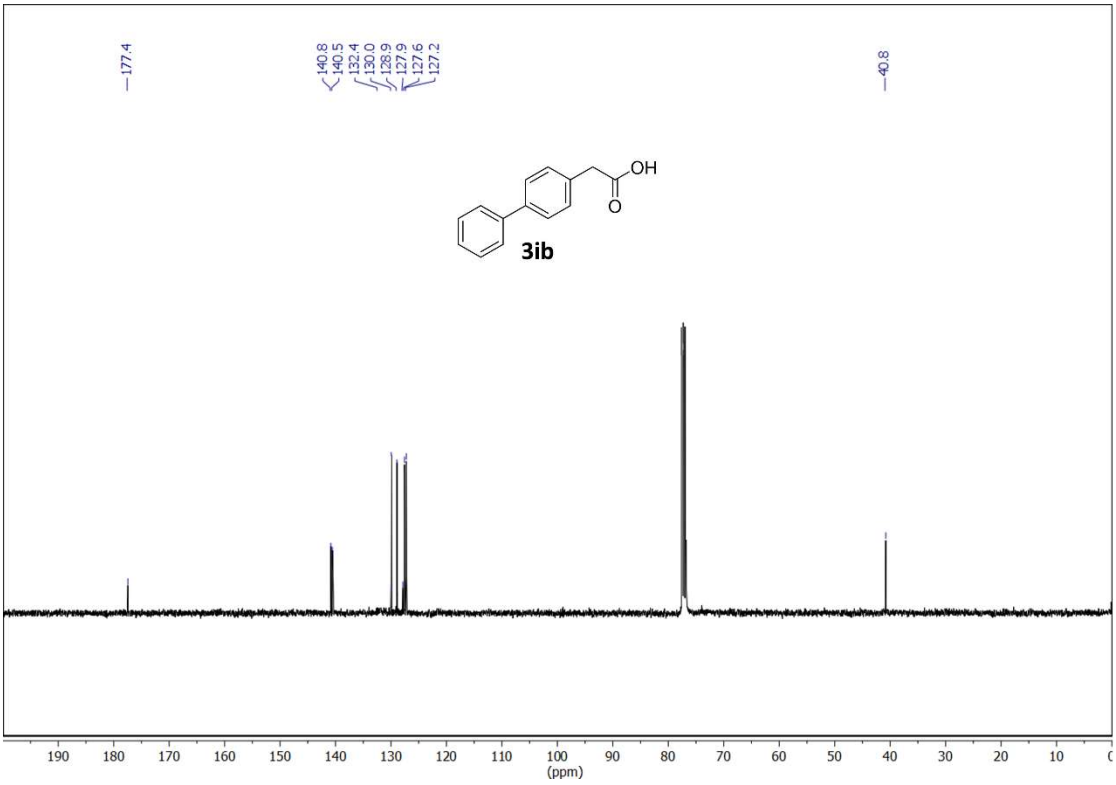
^{13}C NMR 100.62 MHz, $CDCl_3$



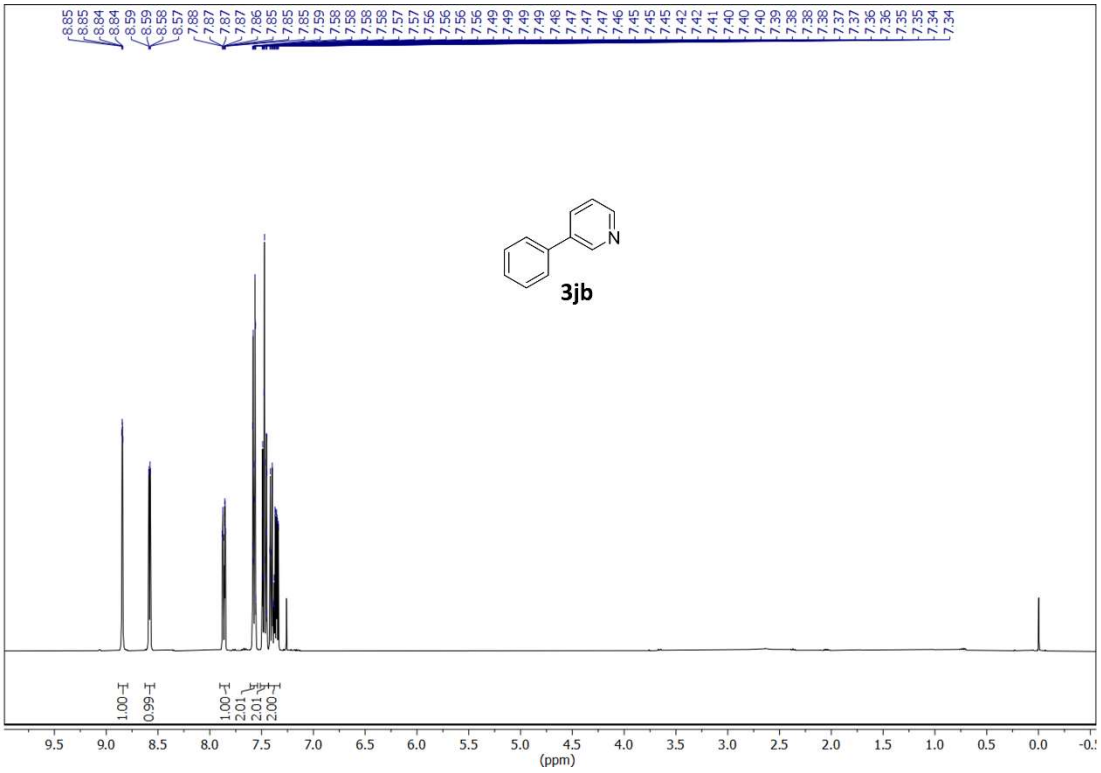
^{1}H NMR 400.12 MHz, $CDCl_3$



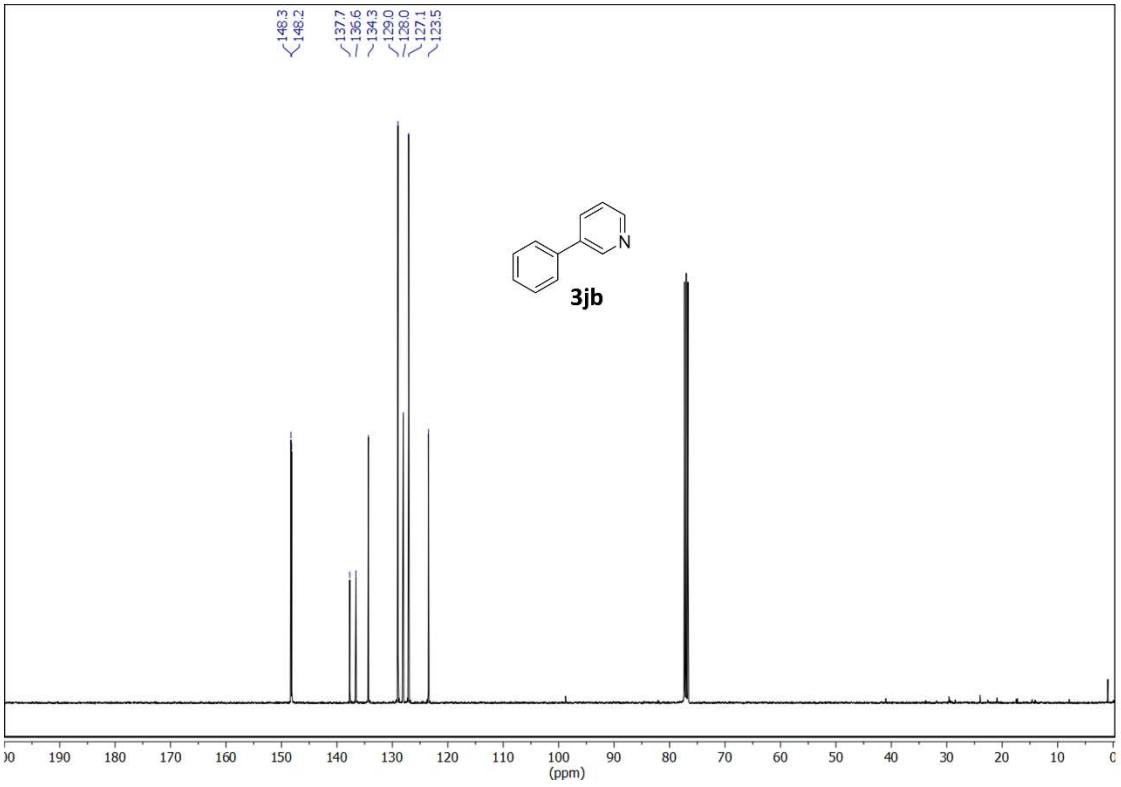
^{13}C NMR 100.62 MHz, $CDCl_3$



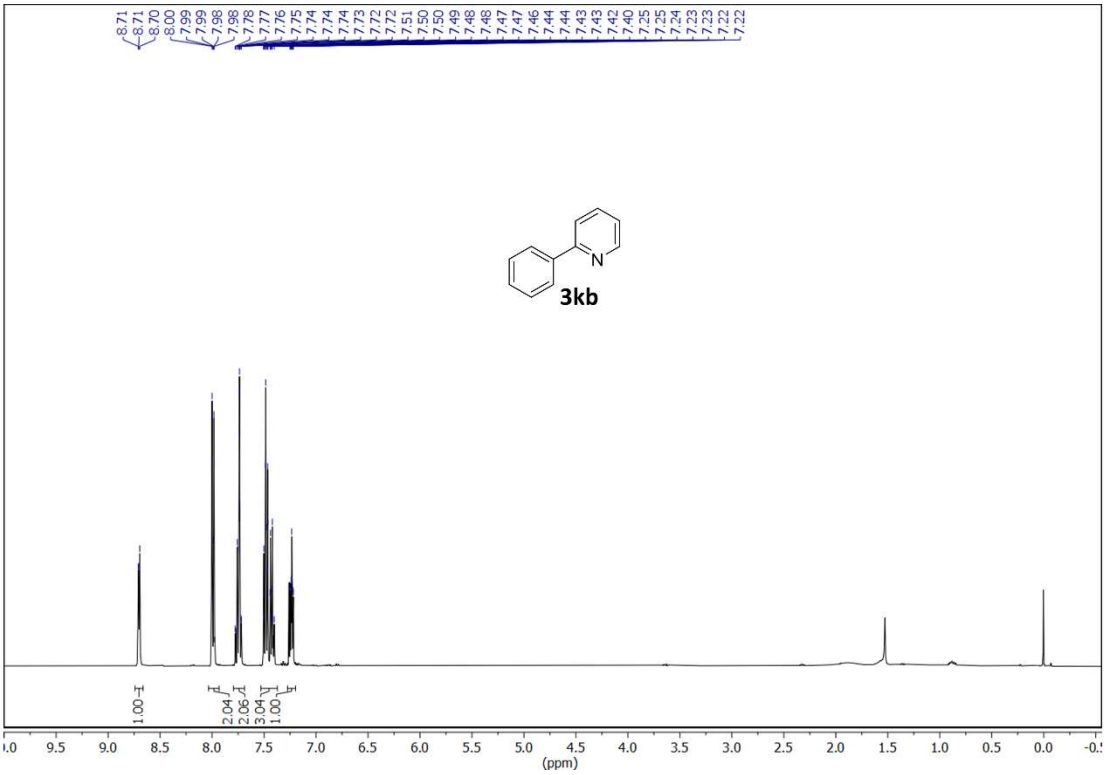
^{1}H NMR 400.12 MHz, $CDCl_3$



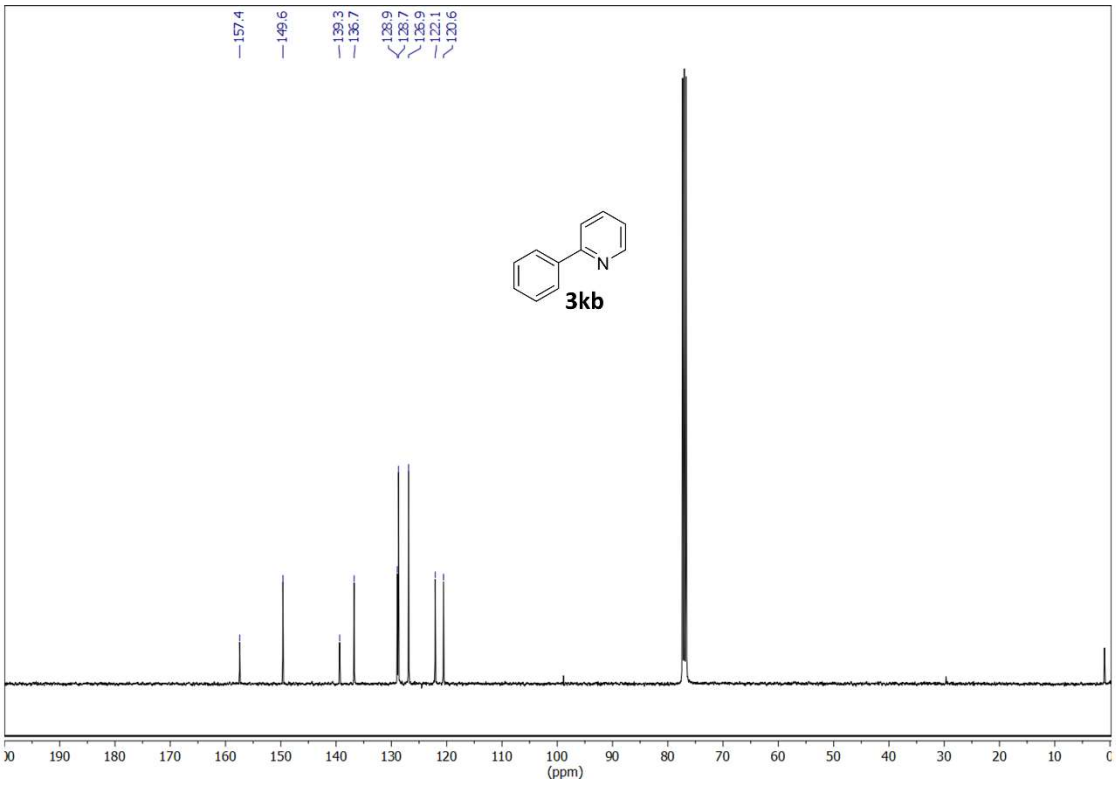
^{13}C NMR 100.62 MHz, $CDCl_3$



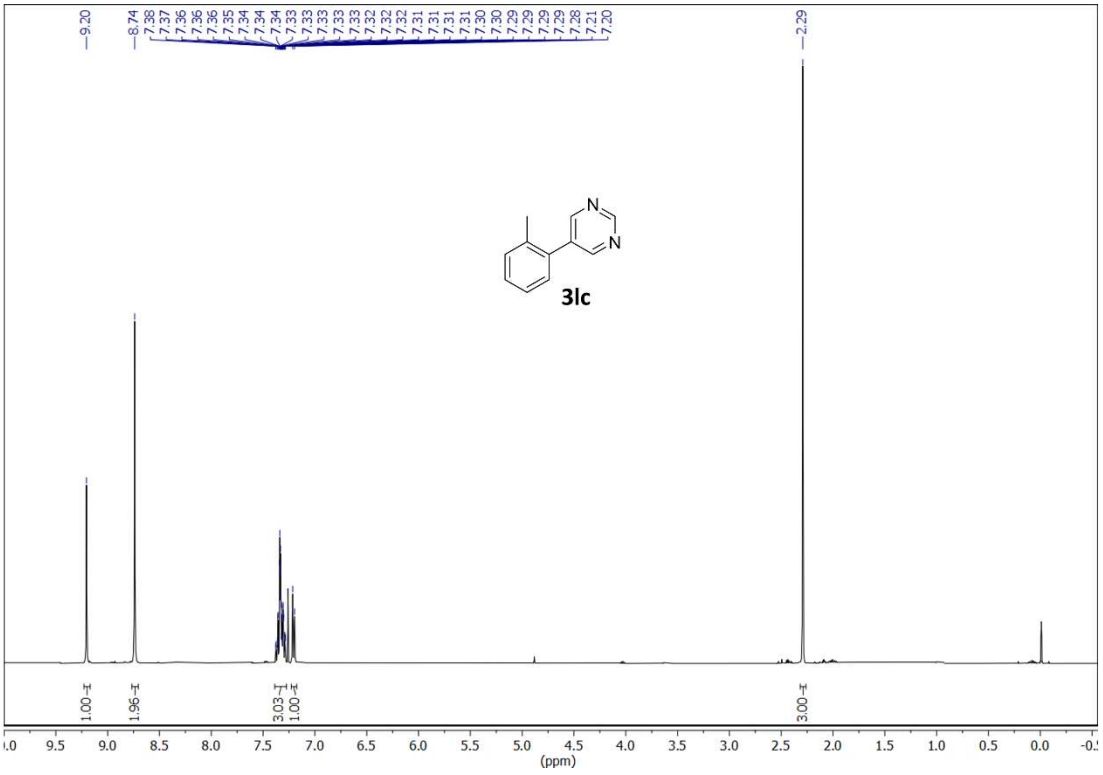
^{1}H NMR 400.12 MHz, $CDCl_3$



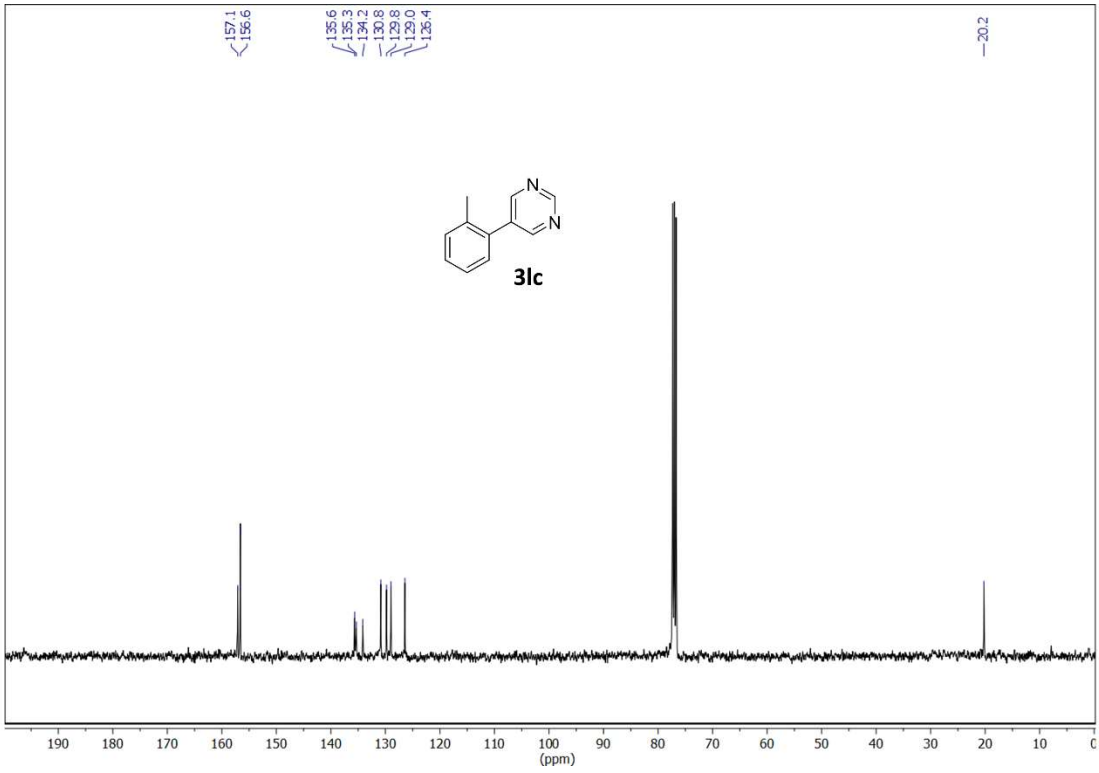
^{13}C NMR 100.62 MHz, $CDCl_3$



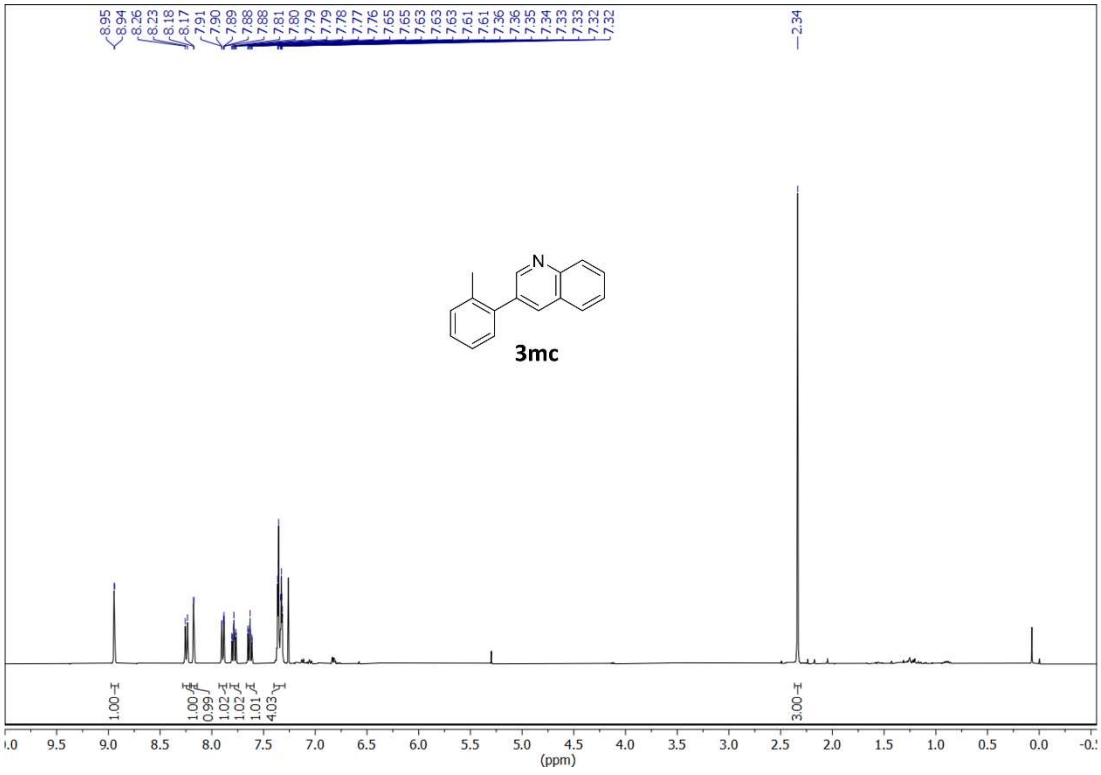
^{1}H NMR 400.12 MHz, $CDCl_3$



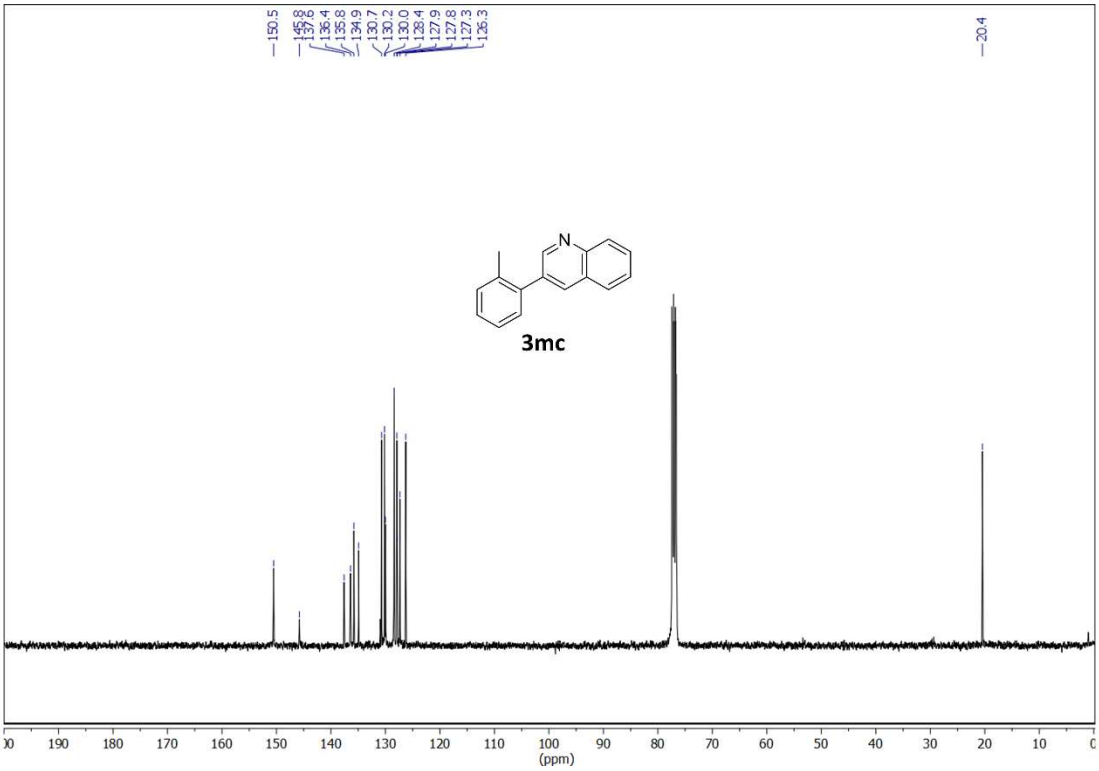
^{13}C NMR 100.62 MHz, $CDCl_3$



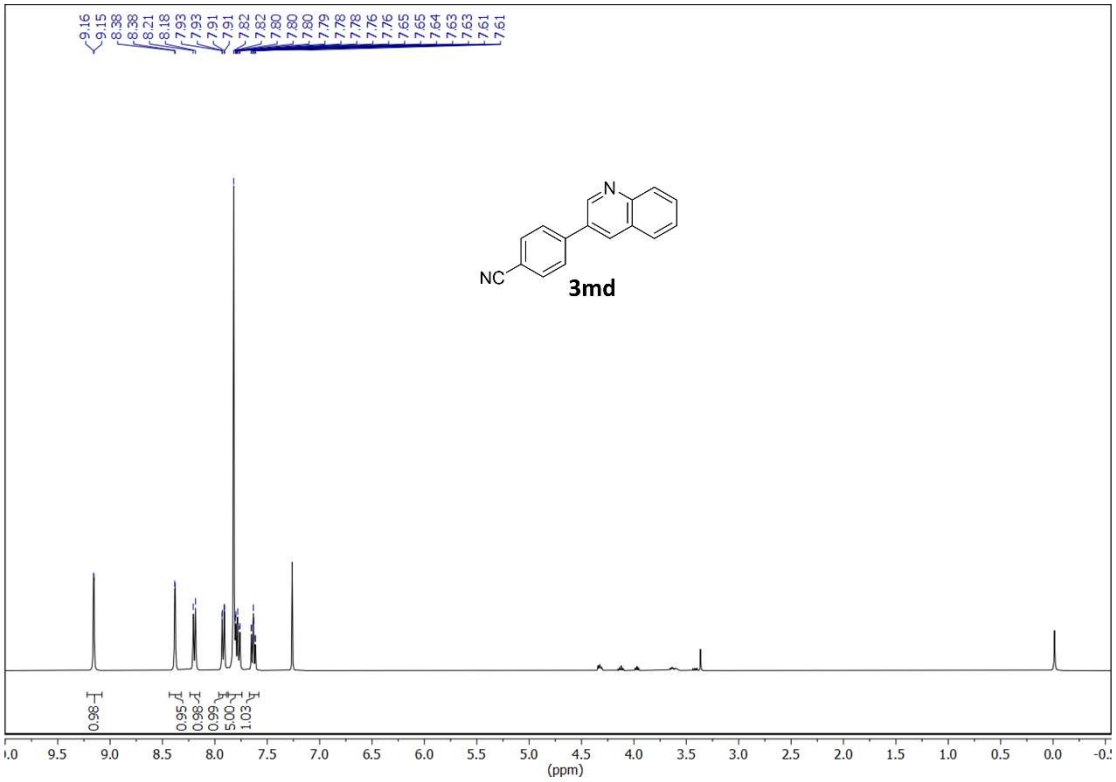
^{1}H NMR 400.12 MHz, $CDCl_3$



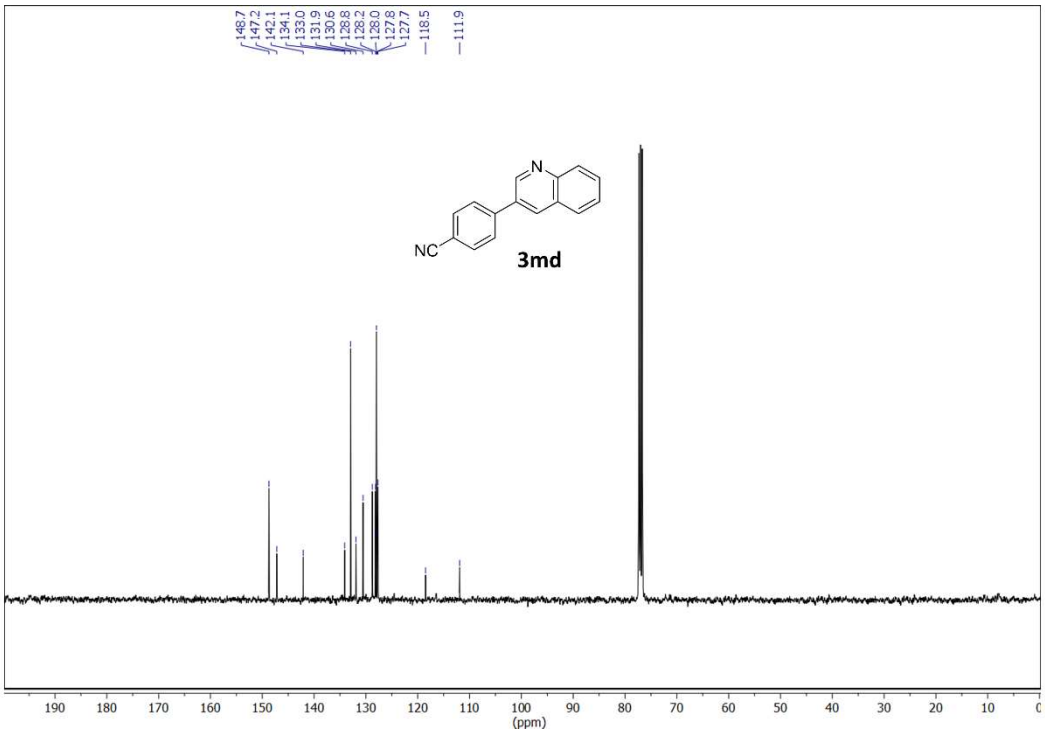
^{13}C NMR 100.62 MHz, $CDCl_3$



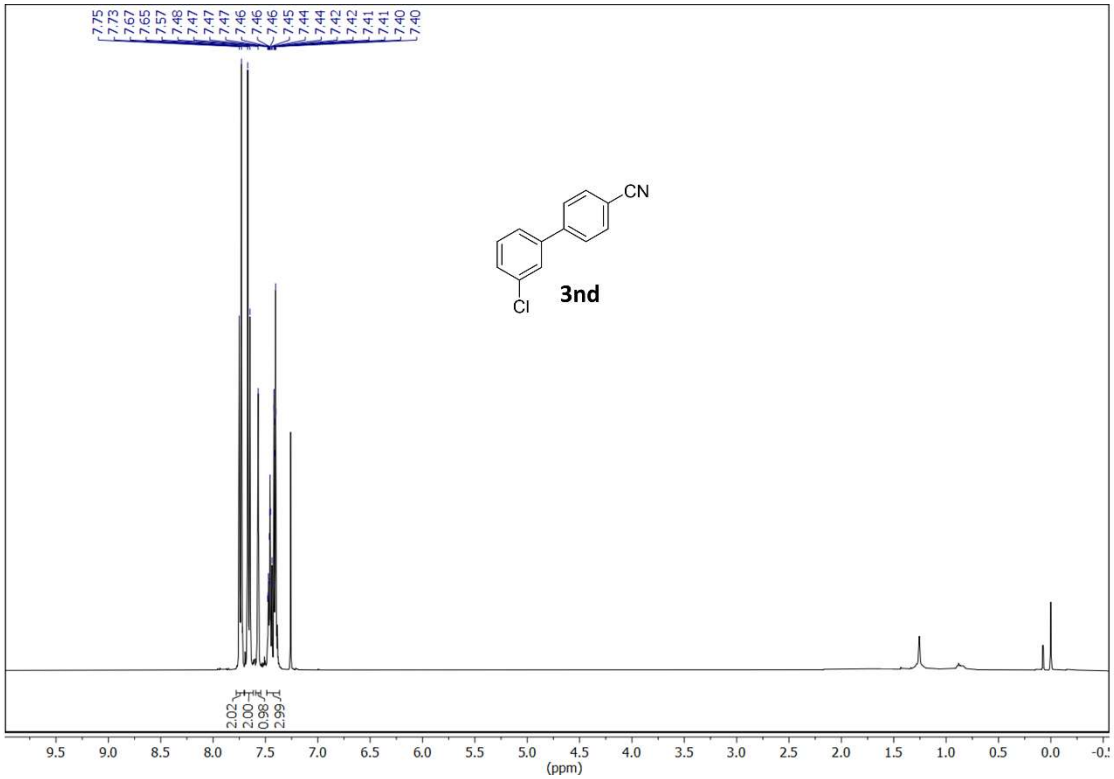
^{1}H NMR 400.12 MHz, $CDCl_3$



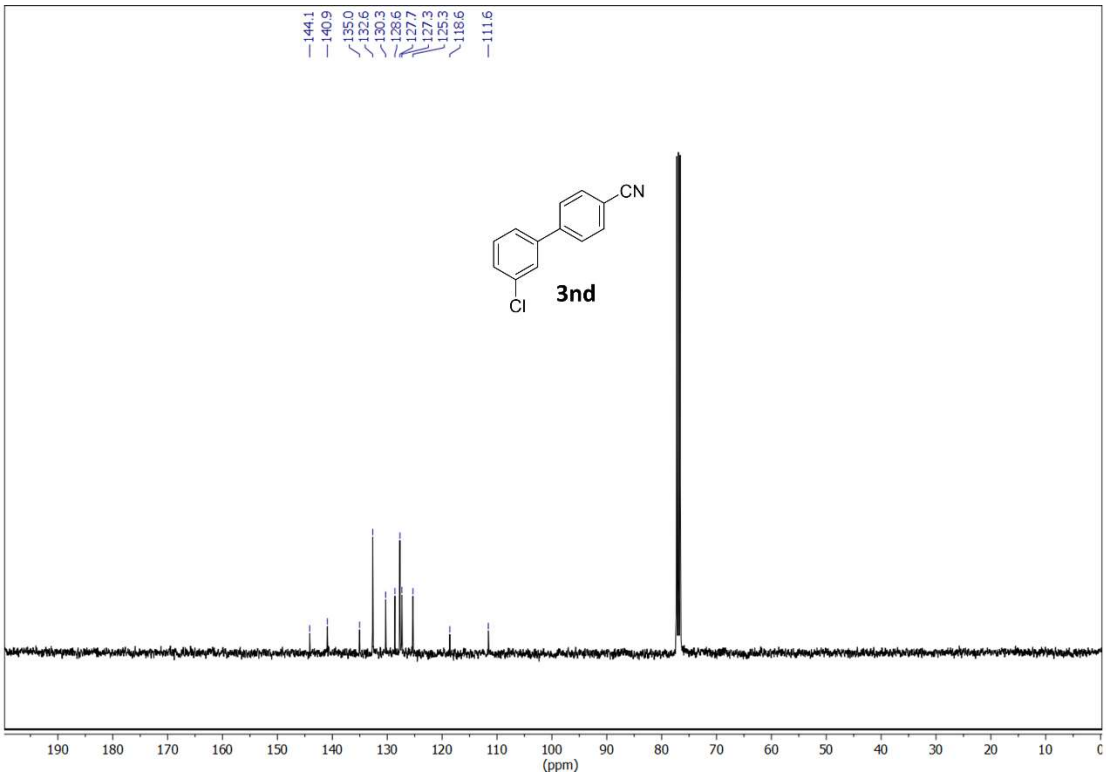
^{13}C NMR 100.62 MHz, $CDCl_3$



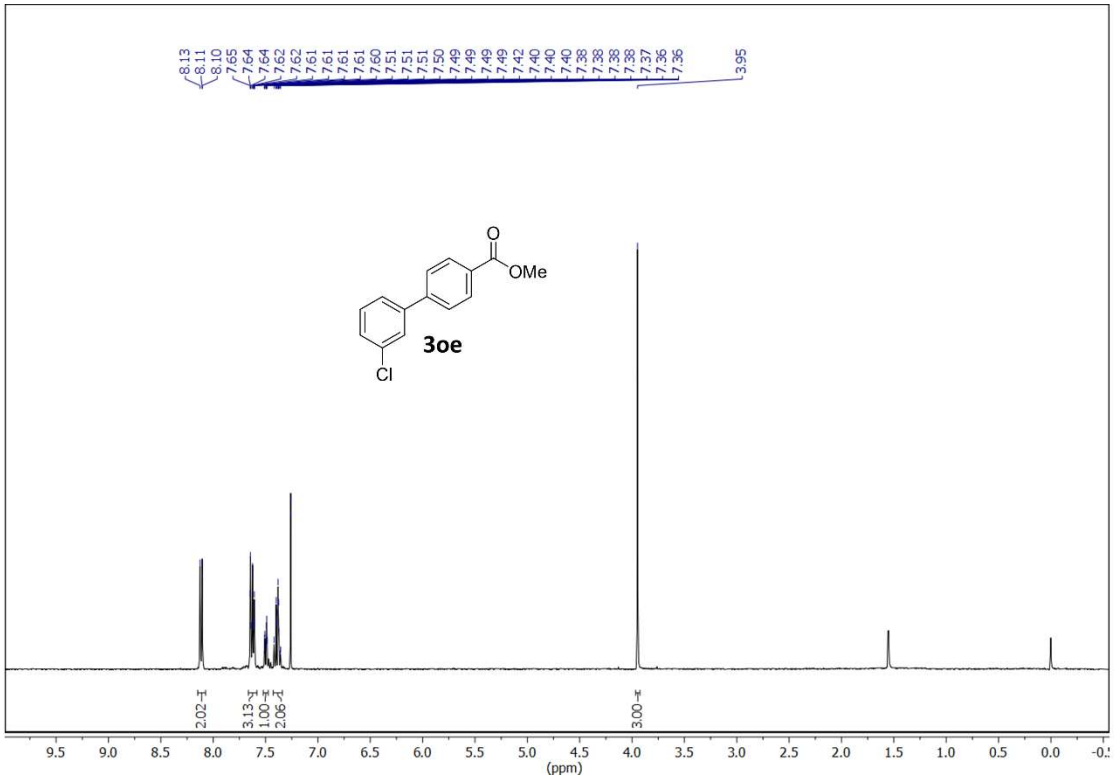
^{1}H NMR 400.12 MHz, $CDCl_3$



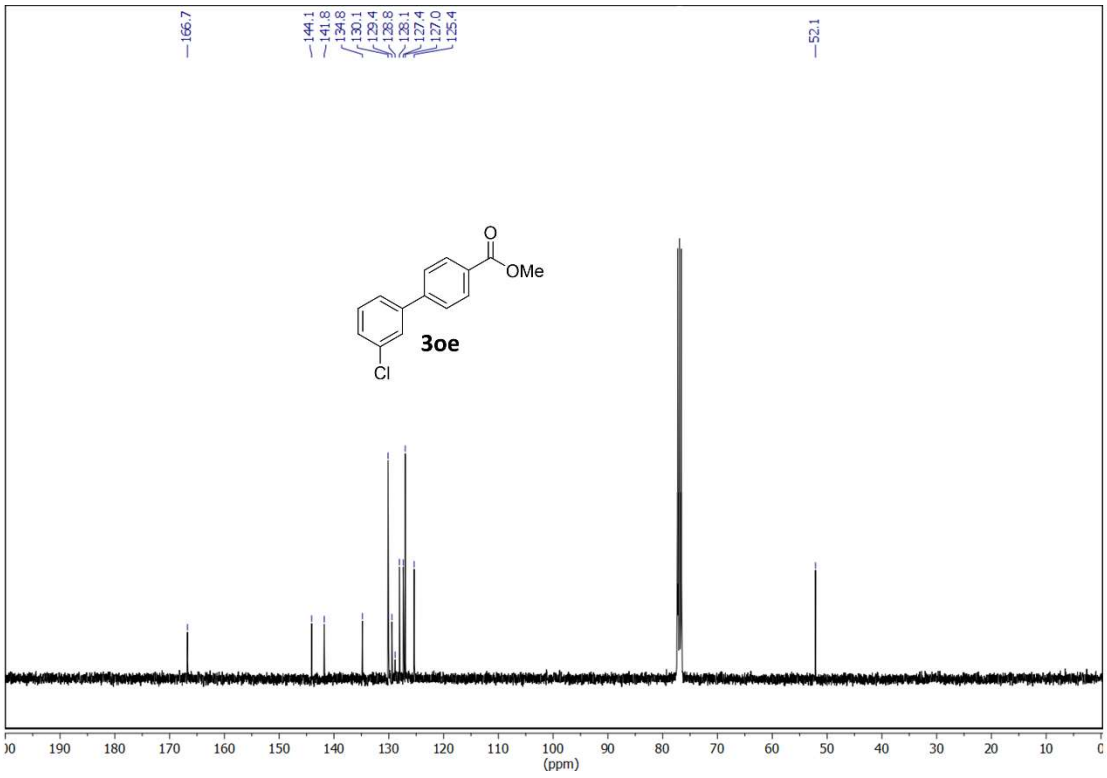
^{13}C NMR 100.62 MHz, $CDCl_3$



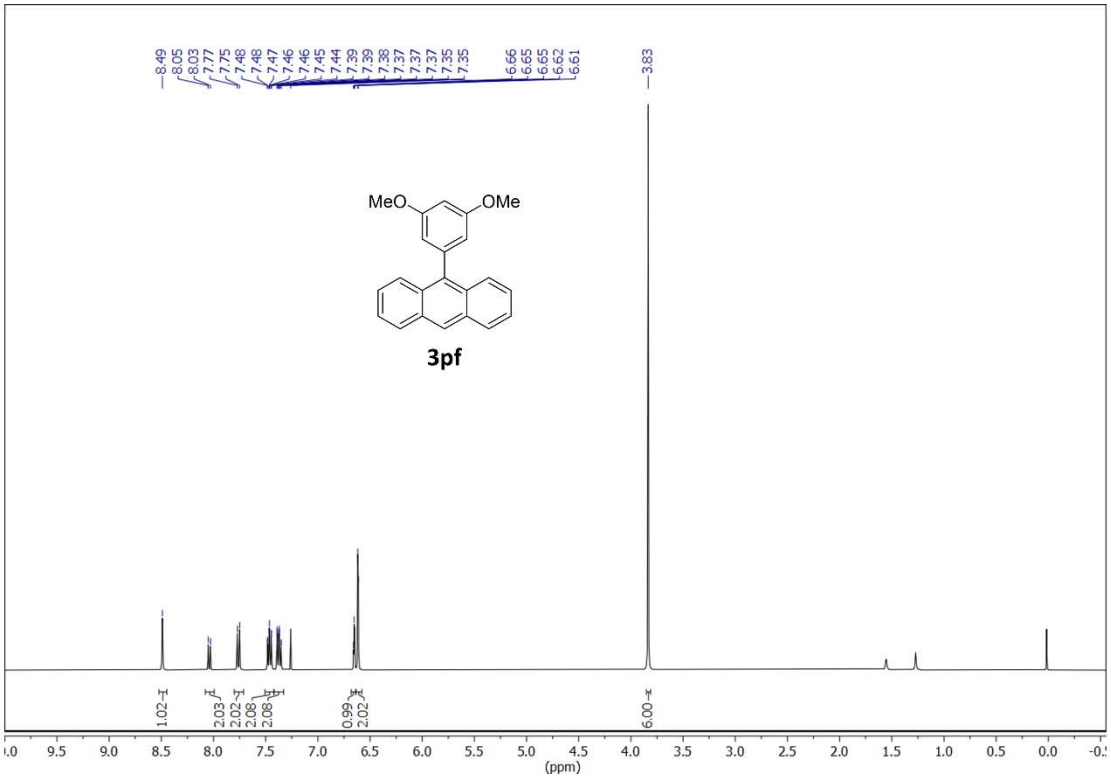
^{1}H NMR 400.12 MHz, $CDCl_3$



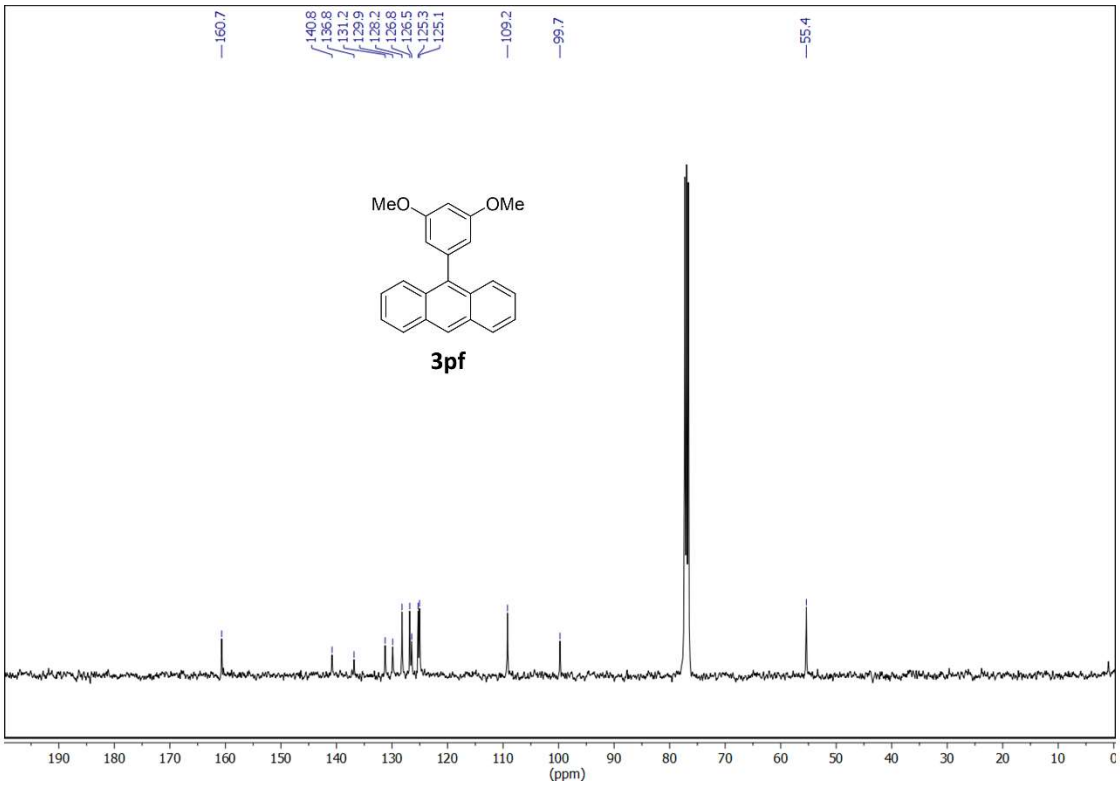
^{13}C NMR 100.62 MHz, $CDCl_3$



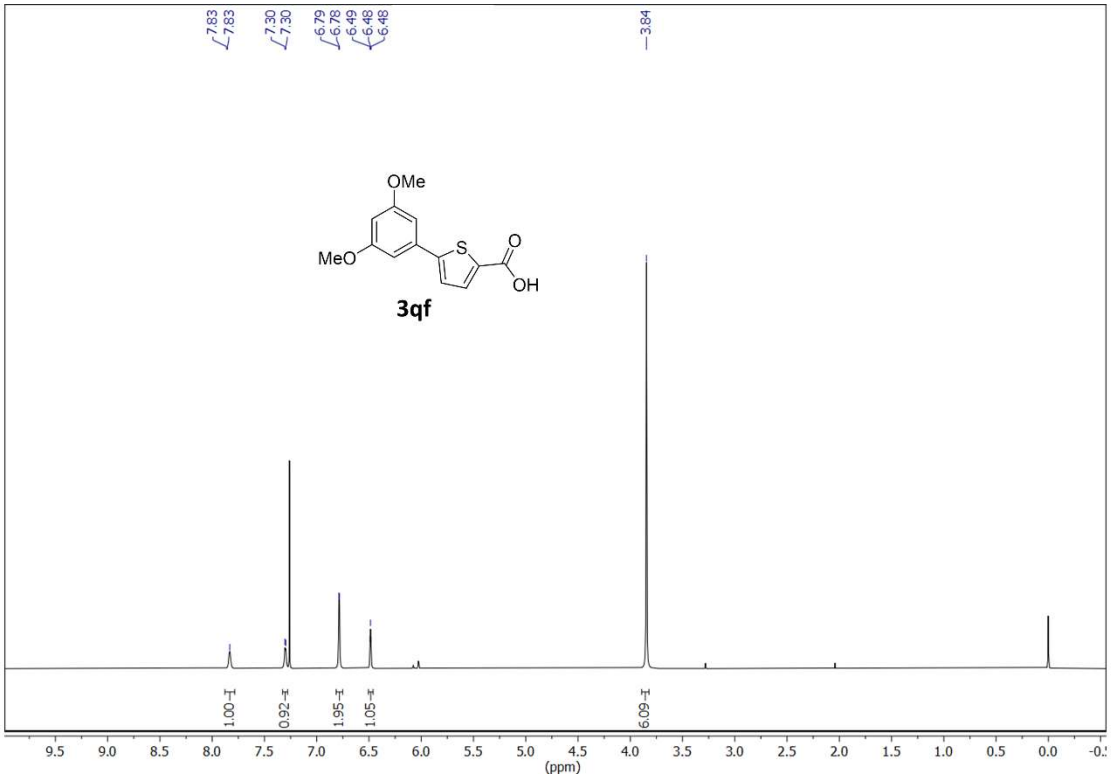
^{1}H NMR 400.12 MHz, $CDCl_3$



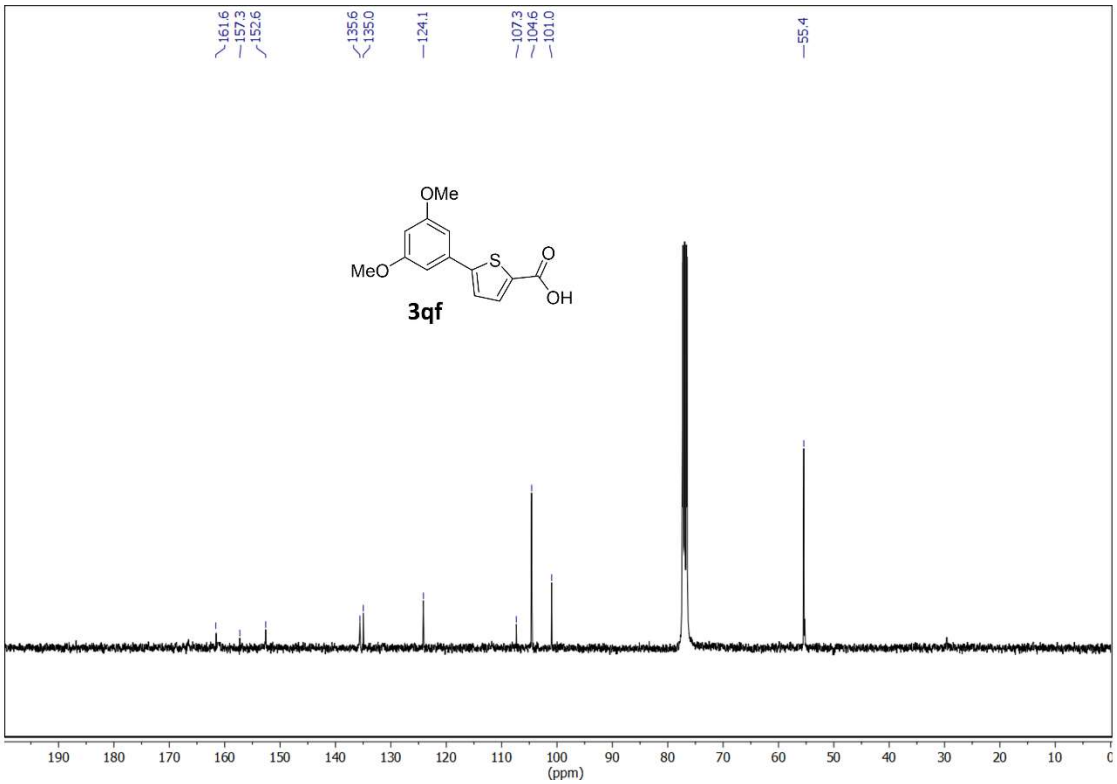
^{13}C NMR 100.62 MHz, $CDCl_3$



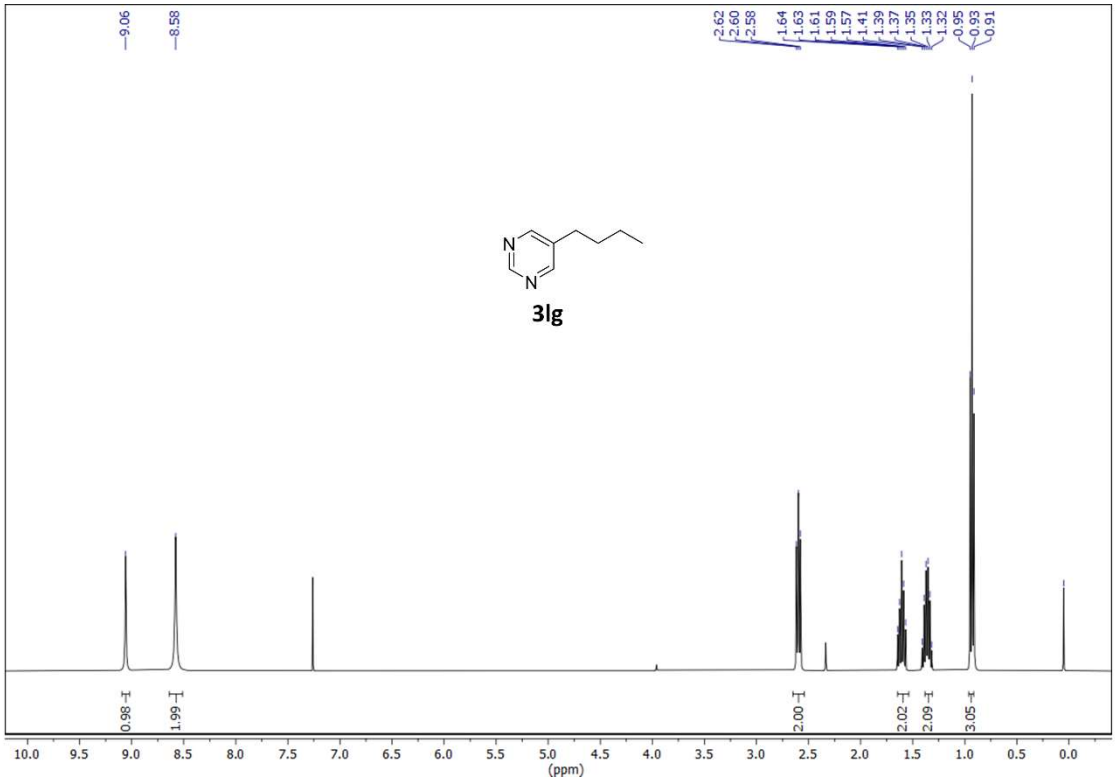
^{1}H NMR 400.12 MHz, $CDCl_3$



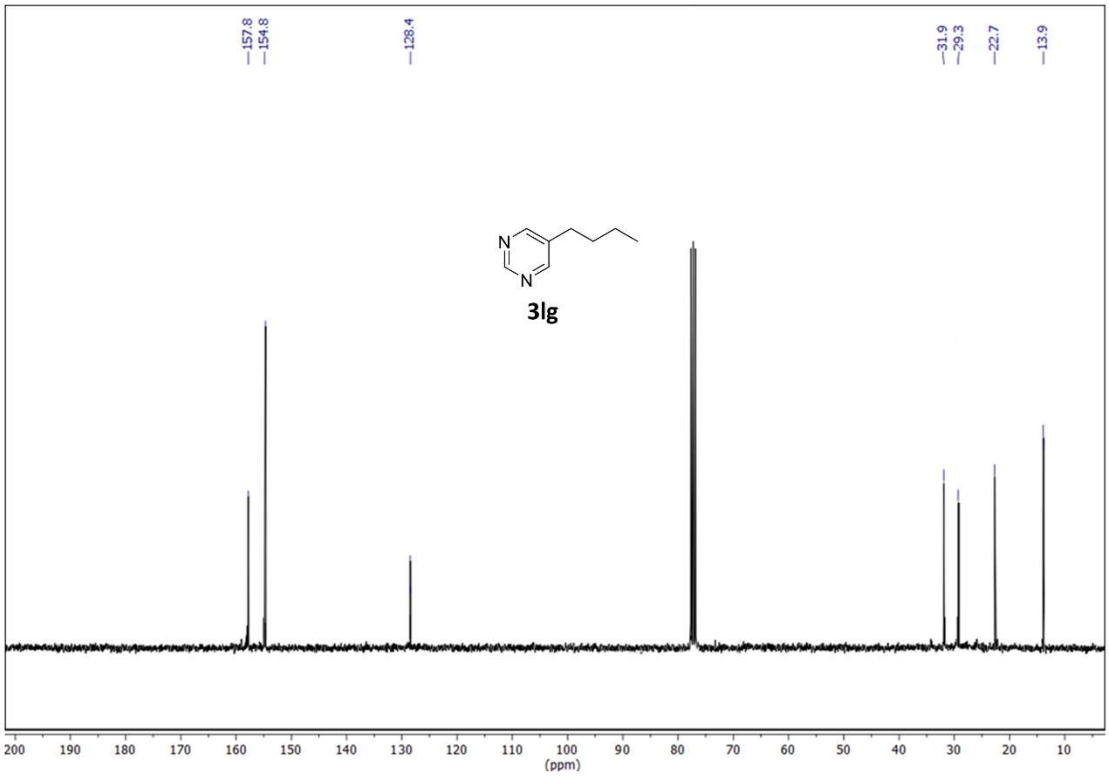
^{13}C NMR 100.62 MHz, $CDCl_3$



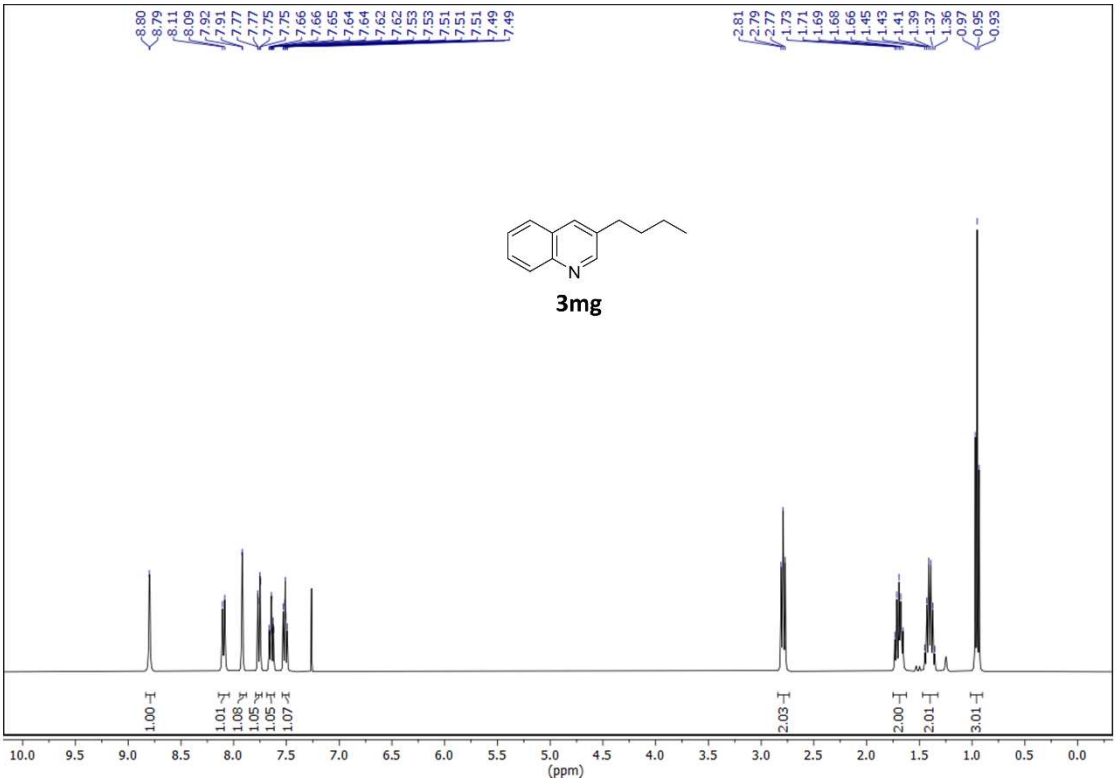
^{1}H NMR 400.12 MHz, $CDCl_3$



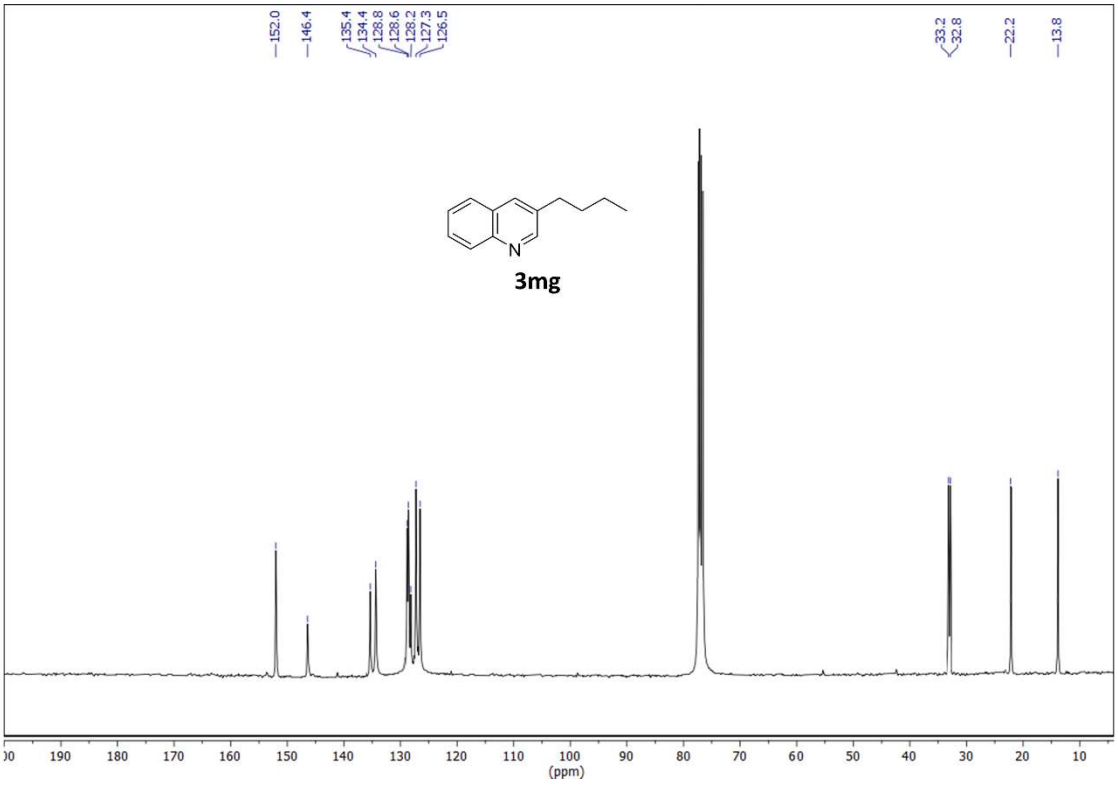
^{13}C NMR 100.62 MHz, $CDCl_3$



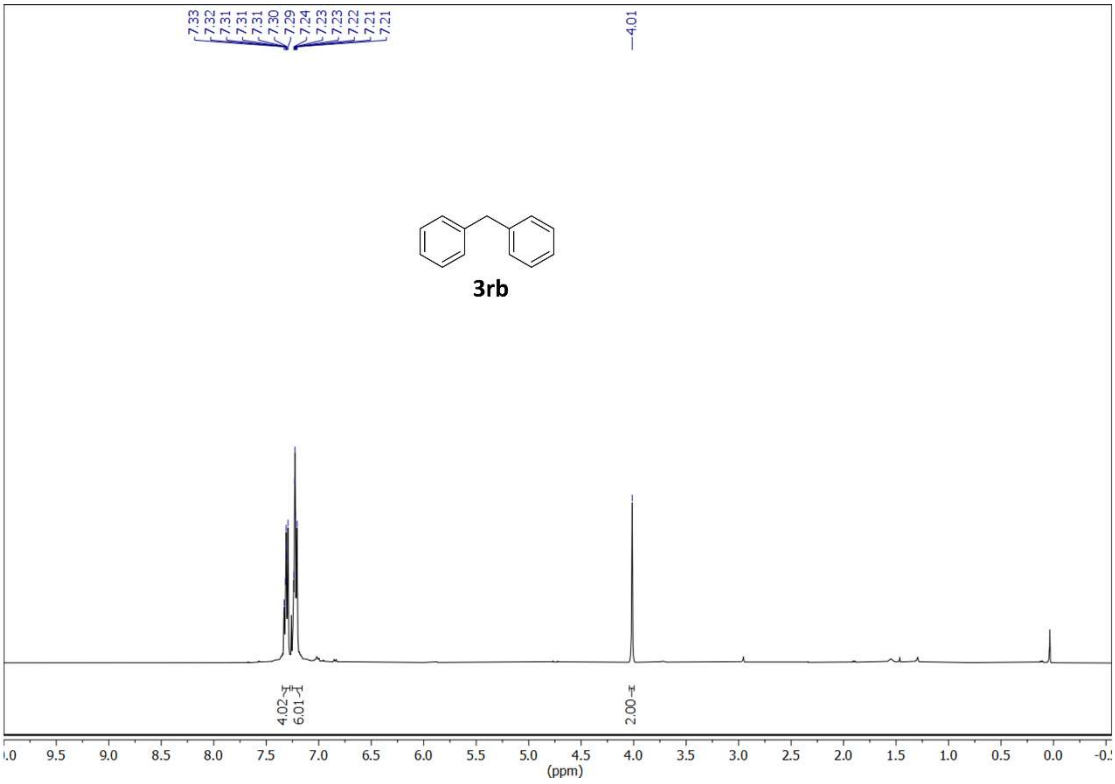
^{1}H NMR 400.12 MHz, $CDCl_3$



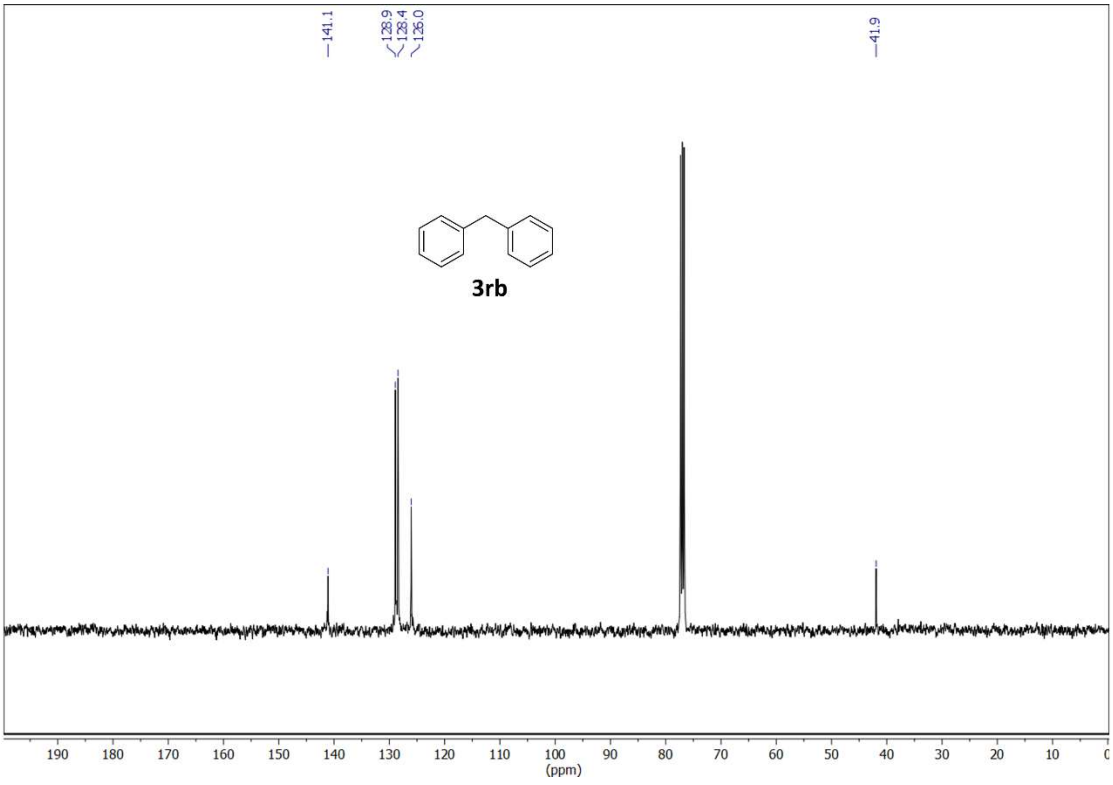
^{13}C NMR 100.62 MHz, $CDCl_3$



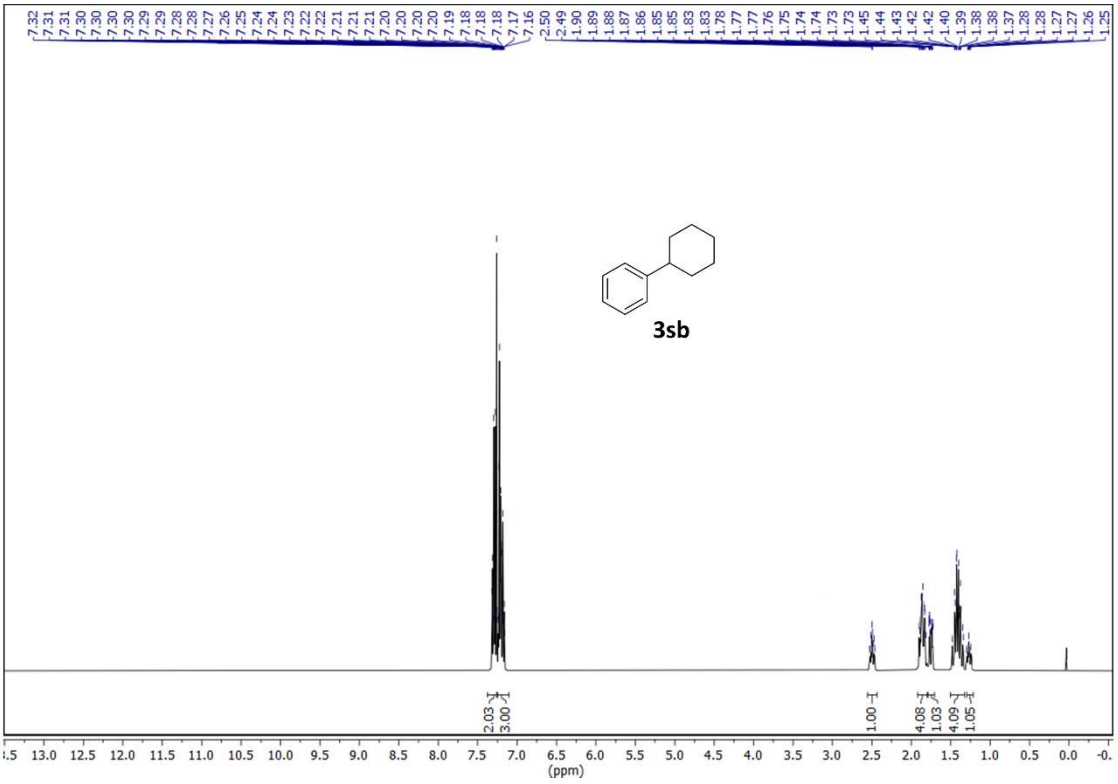
^{1}H NMR 400.12 MHz, $CDCl_3$



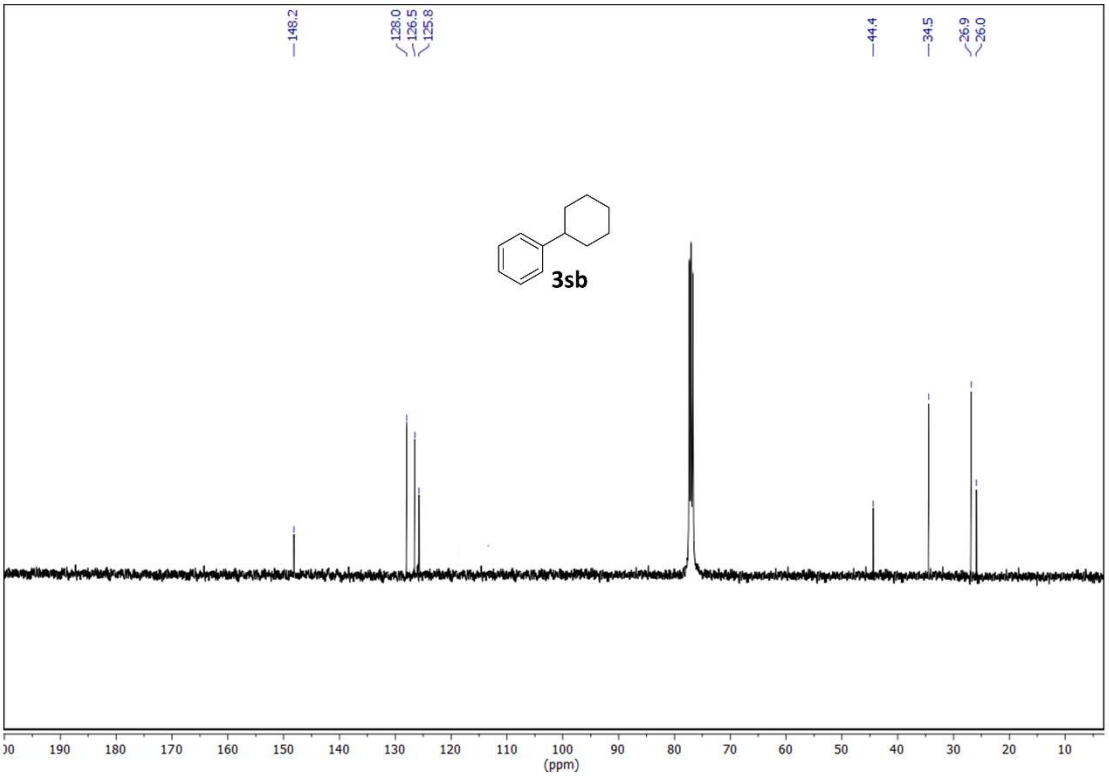
^{13}C NMR 100.62 MHz, $CDCl_3$



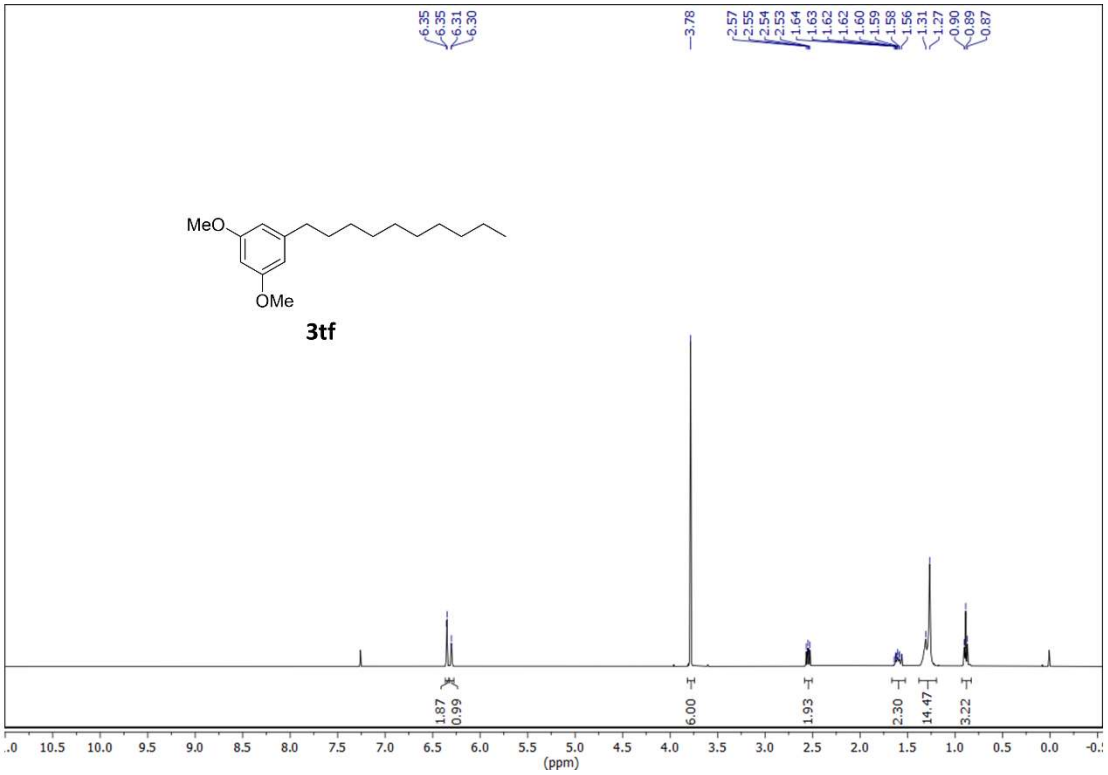
^{1}H NMR 400.12 MHz, $CDCl_3$



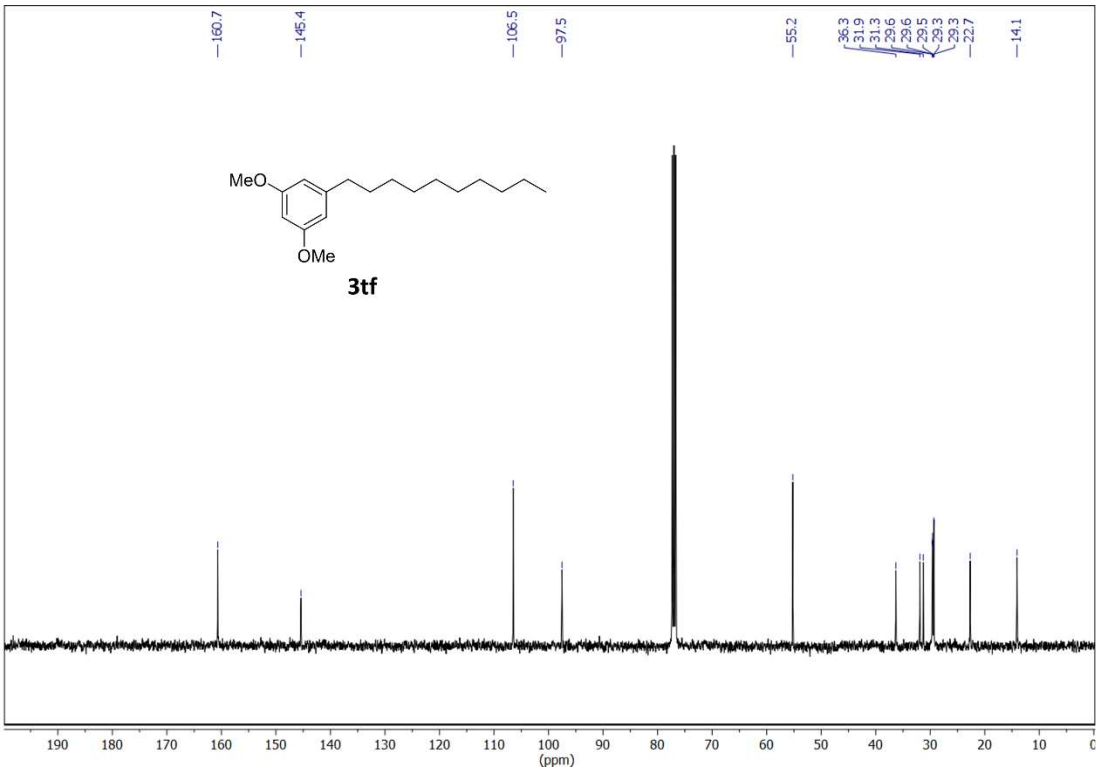
^{13}C NMR 100.62 MHz, $CDCl_3$



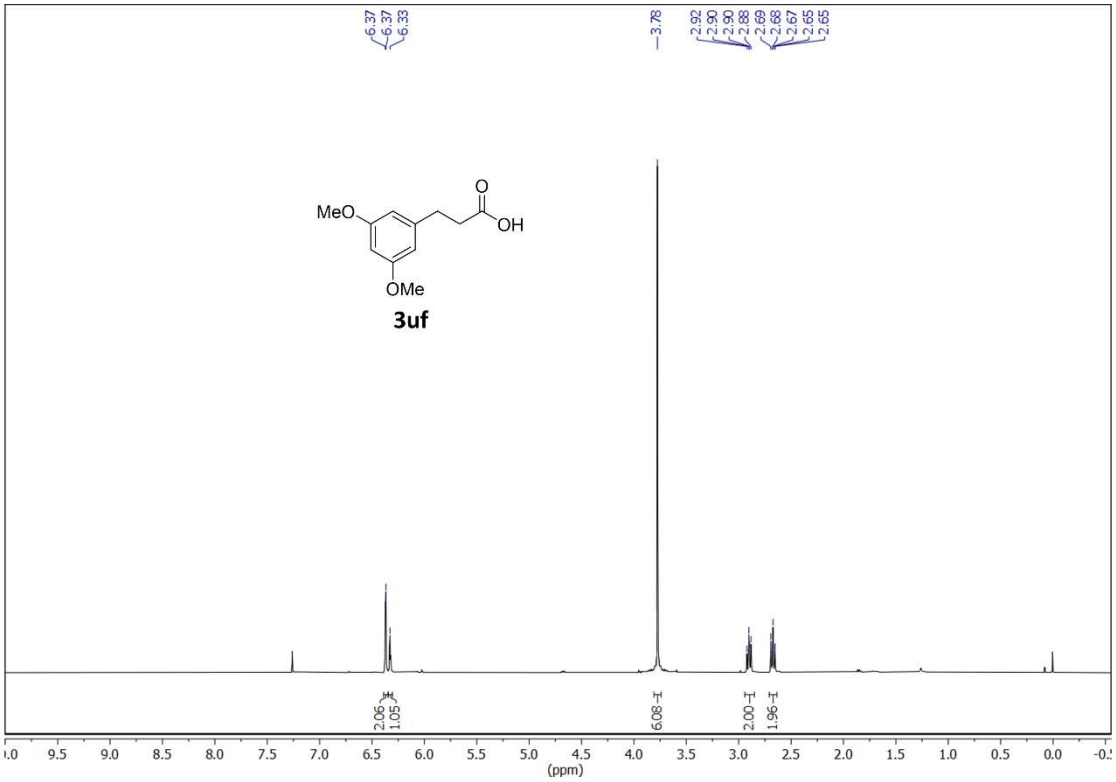
^{1}H NMR 400.12 MHz, $CDCl_3$



^{13}C NMR 100.62 MHz, $CDCl_3$



^{1}H NMR 400.12 MHz, $CDCl_3$



^{13}C NMR 100.62 MHz, $CDCl_3$