Supporting Information

Pd-Indenyl-diphosphine: An Effective Catalyst for the Preparation of Triarylamines

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Experimental Section

General Information

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. (2-mesityl-1H-inden-3-yl) dicyclohexyl phosphine (**6**) was prepared according to the reported procedures.[1] All reactions were performed in a resealable screw cap Schlenk flask (approx. 10 mL volume) in the presence of a Teflon coated magnetic stirrer bar (3 mm × 50 mm). Silica gel (70-230 and 230-400 mesh) was used for column chromatography. ^{1}H NMR, ^{31}P NMR and ^{13}C NMR spectra were recorded on a Mercury-Plus (400 MHz or 600 MHz) spectrometer. HRMS were obtained on an IonSpec FT-ICR mass spectrometer with ESI resource. Elemental analyses were performed on a Perkin-Elmer 240C analyzer. GC analyses were performed on an Agilent 6890N chromatograph. Powder X-ray diffraction patterns were recorded on a Bruker SMART CCD area-detector diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). Compounds described in the literature were characterized by comparison of their ^{1}H and/or ^{13}C NMR spectra to the previously reported.

Preparation of Phosphine Ligands 1 and 2

2-(1H-inden-2-yl) aniline (**4**): A white solid of compound **3** (4.4 g, 18.30 mmol), bromoaniline (3.4 g, 20.00 mmol), anhydrous K_3PO_4 (11.7 g, 55.00 mmol), $Pd(OAc)_2$ (205.4 mg, 0.92 mmol), PPh_3 (480.0 mg, 1.83 mmol) were added to a 100 mL pressure pipe. The tube was evacuated and back-filled with argon (this was repeated two additional times). 50 mL THF was added and the reaction mixture was allowed to stir at 90 ℃ for 24 h. After cooling to room temperature, the system was treated with water (10 mL) and then extraction with ethyl acetate (3 × 20 mL). The organic layer was dried over $MgSO_4$ and filtered. The solvent was removed under reduced pressure, and the residue was purified by flash chromatography (hexane/ethyl acetate = 50/1) to give the product **4** (2.9 g, 77 % yield) as a gray solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 (d, *J* = 6.9 Hz, 1H), 7.40 (d, *J* = 7.1 Hz, 1H), 7.28 (d, *J* = 6.8 Hz, 2H), 7.26-7.18

(m, 1H), 7.16 (s, 1H), 7.10 (t, *J* = 7.1 Hz, 1H), 6.80 (d, *J* = 7.6 Hz, 2H), 4.05 (s, 2H), 3.80 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.30, 144.85, 144.24, 142.34, 128.92, 128.63, 128.10, 126.46, 124.53, 123.37, 122.30, 120.72, 118.50, 116.23, 41.50. HRMS (ESI/ $[M+H]^+$) Calcd for $C_{15}H_{13}N$: 208.1126. Found: 208.1122.

2-(1H-inden-2-yl)-N, N-dimethylaniline (**5)**: 2-(1H-inden-2-yl)aniline **4 (**1.80 g, 8.70 mmol) and K_2CO_3 (2.40 g, 17.40 mmol) was added to a 100 mL round-bottom flask. The tube was evacuated and back-filled with argon (this was repeated two additional times). 20 mL DMF and CH_3I (3.70 g, 26.10 mmol) was added to the flask. The reaction mixture was allowed to stir at room temperature for 24 h. Water (20 mL) was added to quench the reaction, the aqueous phase was extracted with ethyl acetate (3 × 20 mL). The organic layer was dried over $MgSO_4$ and filtered. After evaporation of solvent in vacuo, the residue was subject to column chromatography (CH_2Cl_2/hexane = 1:50) to give a white solid **5** (1.60 g, 80 % yield). ^{1}H NMR (400 MHz, $CDCl_3$)： δ 7.46 (d, *J* = 6.7 Hz, 1H), 7.40 (d, *J* = 7.1 Hz, 2H), 7.25 (d, *J* = 11.4 Hz, 3H), 7.17 (t, *J* = 7.1 Hz, 1H), 7.07 (d, *J* = 7.9 Hz, 1H), 7.00 (t, *J* = 7.1 Hz, 1H), 3.90 (s, 2H), 2.69 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$)： δ 151.74，147.34, 145.48，143.48, 130.36, 129.98, 129.03, 128.06，126.18，124.31，123.36，121.97，120.63，118.11，43.63，39.95. HRMS (ESI/ $[M+H]^+$) Calcd for $C_{17}H_{17}N$: 236.1439. Found: 236.1436.

[2-(2'-N, N-dimethylamine)phenyl-1H-inden-3-yl] dicyclohexyl phosphine **1**·BH_3: In a 250 mL flask, 2-(1H-inden-2-yl)-N, N-dimethylaniline **5** (2.0 g, 8.50 mmol) was dissolved in THF (40 mL) under an argon atmosphere. The mixture was cooled to -78 ℃, and *n*BuLi (6.25 mL, 1.6 M solution in hexane, 10.0 mmol) was added. The solution was stirred for 30 min at -78 ℃ and then for 4 h at room temperature. Then the mixture was cooled to -78 ℃ and Cy_2PCl (2.20 mL, 10.0 mmol) was added. The mixture was warmed to room temperature and stirred for overnight. BH_3·THF (26.0 mL, 1.0 M, 26.0 mmol) was added to the mixture. The resulting solution was stirred at room temperature for 24 h. The LiCl solid was removed by filtration over a pad of Celite. The resulting filtrate was treated with water (5.0 mL). The organic layer was then separated from the aqueous layer, dried over $MgSO_4$ and filtered. After evaporation of solvent in vacuo, the residue was subject to column chromatography

(CH_2Cl_2/hexane = 1:10) to give a white solid **1**·BH_3 (2.6 g, 70 % yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, *J* = 7.5 Hz, 1H), 7.45-7.29 (m, 4H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.11-7.00 (m, 2H), 6.90 (s, 1H), 5.27 (d, *J* = 15.2 Hz, 1H), 2.59-2.49 (m, 6H), 1.91-0.71 (m, 22H), 0.50 (d, *J* = 12.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.22, 149.26, 143.74, 142.72, 132.26, 130.71, 129.84, 128.74, 126.99, 125.99, 124.39, 121.95, 120.83, 117.96, 43.47, 43.29, 42.81, 32.97, 32.70, 30.77, 30.48, 27.47-27.21, 26.77 - 26.55, 25.78, 25.60. ^{31}P NMR (162 MHz, $CDCl_3$): δ 34.98 ppm. Anal. Calcd. for $C_{29}H_{41}BNP$: C, 78.20; H, 9.28; N, 3.14. Found: C, 77.96; H, 9.50; N, 3.17.

1, 3-di(dicyclohexyl) phosphinyl-2-(2'-N, N-dimethylamine) phenyl indene **2**·BH_3: In a 250 mL flask, compound **5** (2.0 g, 8.50 mmol) was dissolved in THF (80 mL) under an argon atmosphere. The mixture was cooled to -78 ℃, and *n*BuLi (6.25 mL, 1.6 M solution in hexane, 10.0 mmol) was added. The solution was stirred for 30 min at -78 ℃ and then for 4 h at ambient temperature. Then the mixture was cooled to -78 ℃ and Cy_2PCl (2.2 mL, 10.0 mmol) was added. The mixture was warmed to room temperature and stirred for overnight. The mixture was cooled to -78 ℃ again and *n*BuLi (6.25 mL, 1.6 M solution in hexane, 10.0 mmol) was added. The solution was stirred for 30 min at -78 ℃ and then for 4 h at ambient temperature. Then the mixture was cooled to -78 ℃ and Cy_2PCl (2.2 mL, 10.0 mmol) was added. The mixture was warmed to room temperature and stirred for overnight. BH_3·THF (50.0 mL, 1.0 M, 50.0 mmol) was added to the mixture. The resulting solution was stirred for 24 h. The LiCl that formed was removed by filtration over a pad of Celite. The resulting filtrate was treated with water (5.0 mL). The organic layer was then separated from the aqueous layer, dried over $MgSO_4$, and filtered. After evaporation of solvent in vacuo, the residue was subject to column chromatography (CH_2Cl_2/ethyl acetate = 50:1) to give a white solid **2**·BH_3 (1.6 g, 30 % yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (br, 1H), 7.82 (br, 1H), 7.49-7.33 (m, 2H), 7.28 (d, *J* = 6.6 Hz, 2H), 7.02 (d, *J* = 7.7 Hz, 2H), 5.51 (d, *J* = 18.3 Hz, 1H), 2.64 (d, *J* = 9.7 Hz, 6H), 2.43-0.84 (m, 44H), 0.73 (d, *J* = 8.5 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.21, 151.01, 143.90, 141.73, 133.36, 130.25, 128.31, 127.95, 126.47, 126.21, 124.77, 122.29, 120.17, 117.95, 53.37, 45.52, 43.06, 35.07, 34.75, 34.27, 33.94, 32.69, 32.43, 30.30, 30.02, 29.00,

28.44, 27.81, 27.53, 27.26, 26.92, 26.81, 26.65, 26.57, 26.49, 25.55. ^{31}P NMR (162 MHz, $CDCl_3$): δ 19.88, 38.80 ppm. Anal. Calcd. for $C_{41}H_{65}B_2NP_2$: C, 75.12; H, 9.99; N, 2.14. Found: C, 75.20; H, 10.10; N, 2.21.

^{31}P NMR Study of Deprotonated Phosphine Ligands

A yellow solution of **1**·BH_3 (100.0 mg, 0.22 mmol) in 1.0 mL of morpholine was heated at 100 ℃ for 2 h. The solvent was removed in vacuo and 2.0 mL of hexane was added. The yellow solution was filtered through a short plug of Celite, and the solvent was evaporated in vacuo. Recrystallization of the residue from degassed EtOH (3.0 mL) gives 90 mg (92 %) of ligand **1** as pale yellow solid. ^{31}P NMR (162 MHz, $CDCl_3$): δ -15.65 ppm.

Ligand **1** (8.6 mg, 0.02 mmol) and *t*BuONa (4.0 mg, 0.04 mmol) were added to a NMR tube. The tube was evacuated and back-filled with argon. Dry DME (0.5 mL) was added to the tube. The reaction mixture was stirred at room temperature for 0.5 h. A red solvent was formed. ^{31}P NMR (162 MHz, DME): δ -19.65 ppm.

A yellow solution of **2**·BH_3 (100 mg, 0.15 mmol) in 1.0 mL of morpholine was heated at 100 ℃ for 4 h. The solvent was removed and 2.0 mL of hexane was added. The yellow solution was filtered through a short plug of Celite, and the solvent was evaporated under reduced pressure. Recrystallization of the residue from degassed EtOH (3.0 mL) gives 84 mg (90%) of ligand **2** as pale yellow solid. ^{31}P NMR (162 MHz, $CDCl_3$): δ -20.18, 7.19 ppm.

Ligand **2** (12.5 mg, 0.02 mmol) and *t*BuONa (4.0 mg, 0.04 mmol) were added to a NMR tube. The tube was evacuated and back-filled with argon. Dry DME (0.5 mL) was added to the tube. The reaction mixture was stirred at room temperature for 0.5 h. A red solvent was formed. ^{31}P NMR (162 MHz, DME): δ -19.65 ppm.

General Procedures for Reaction Condition Screenings.

Pd scource, phosphine ligand, chlorobenzene, diphenylamine, base and solvent were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar. The tube was evacuated and flushed with nitrogen for three times, and then placed into a

preheated oil bath and stirred for 24 h. After completion of reaction, the reaction tube was allowed to cool to room temperature. Ethyl acetate (10 mL) and dodecane (22.6 mg, 0.1 mmol, internal standard) were added. The organic layer was subjected to GC analysis. The GC yield obtained was previously calibrated by authentic sample/dodecane calibration curve. The results are listed in **Table S2**.

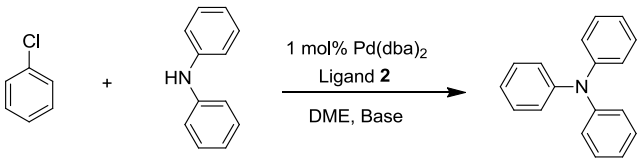
General Procedure for Pd-catalyzed Buchwald-Hartwig Amination Reaction

A disposable tube with a screw cap, Teflon septum and stir bar was charged with $Pd(dba)_2$ (6.0 mg, 0.01 mmol, 1.0 mol %), diphosphine ligand (12.6 mg, 0.02 mmol, 2.0 mol %), aryl halide (1.20 mmol), diphenylamine (169.2 mg, 1.00 mmol) and *t*BuONa (116.0 mg, 1.20 mmol). The tube was evacuated and back-filled with argon (this was repeated two additional times). Aryl halide and degassed DME (2.0 mL) was added to the tube in a glovebox filled with nitrogen. The reaction mixture was allowed to stir at the noted temperature for 24 h. After cooling to room temperature, the crude mixture was filtered through a Celite pad. The filtrate was concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (eluting with hexanes/ethyl acetate = 50:1).

DFT Studies.

Computational method. All calculations were conducted on a home-built Linux cluster consisting of 16 processors. Ground-state geometry optimizations, using all-atom DFT without any approximations, were conducted using Gaussion 03 with B3LYP hybrid functional. For C, H, N, O, Cl and P, the 6-31G (d) basis set was used, and for the Pd center, LANL2DZ+ECP was employed. Single-point energy calculations were conducted using geometries of structures from the 6-31G(d)/LAN2DZ+ECP calculations. All calculated structures were verified to be local minima (all positive eigenvalues) for ground-state structures. The results are listed in **Figure S3**.

Table S1. Optimization of reaction conditions[a]



Entry	Base	Temperature (℃)	Chlorobenzene (mmol)	Diphenyl amine (mmol)	Yield[b] (%)
1	*t*BuOK	120	1.0	1.2	19
2	*t*BuONa	120	1.0	1.2	70
3	Cs_2CO_3	120	1.0	1.2	22
4	K_3PO_4	120	1.0	1.2	5
5	*t*BuONa	110	1.0	1.2	61
6	*t*BuONa	100	1.0	1.2	66
7	*t*BuONa	90	1.0	1.2	75
8	*t*BuONa	80	1.0	1.0	29
9	*t*BuONa	90	1.0	1.0	63
10	*t*BuONa	90	1.2	1.0	81

[a]Reaction conditions: Base (1.4 mmol), $Pd(dba)_2$ (1.0 mol %), ligand **2** (2.0 mol %), DME (2.0 mL), 24 h. [b]Determined by GC using dodecane as the internal standard.

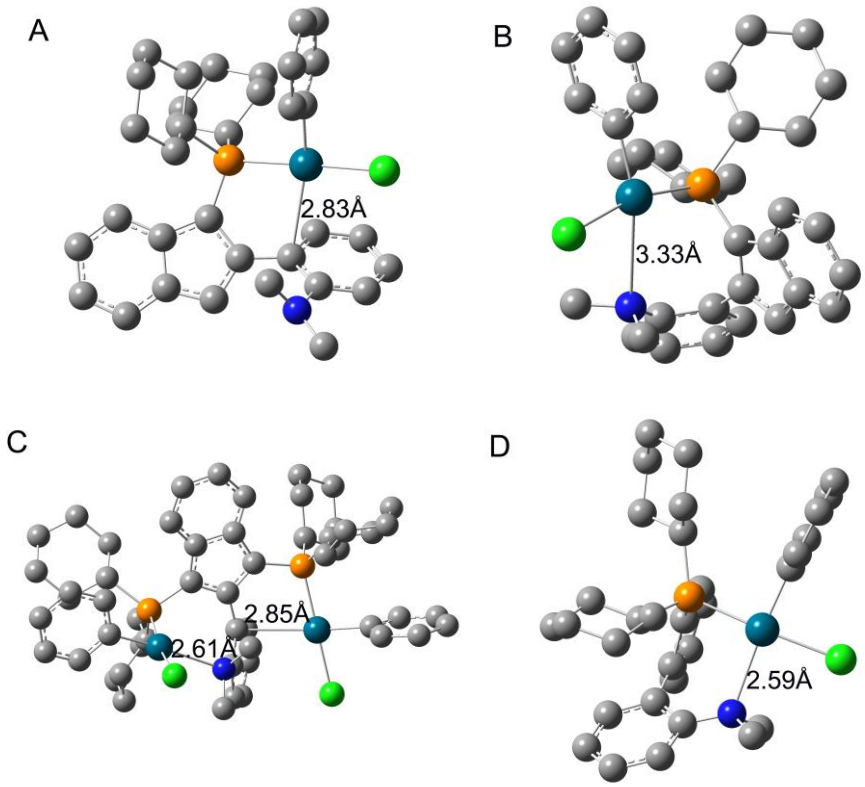
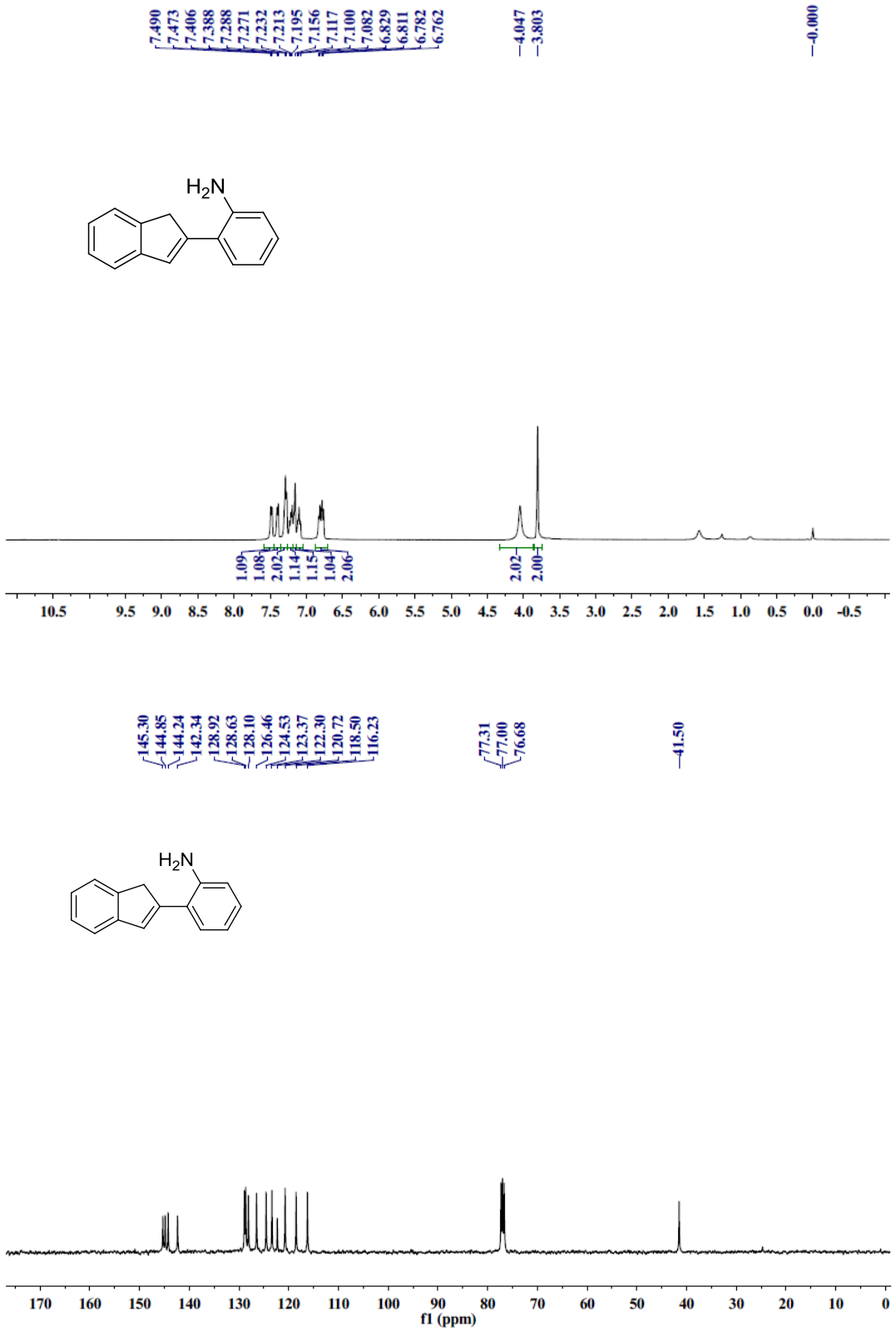
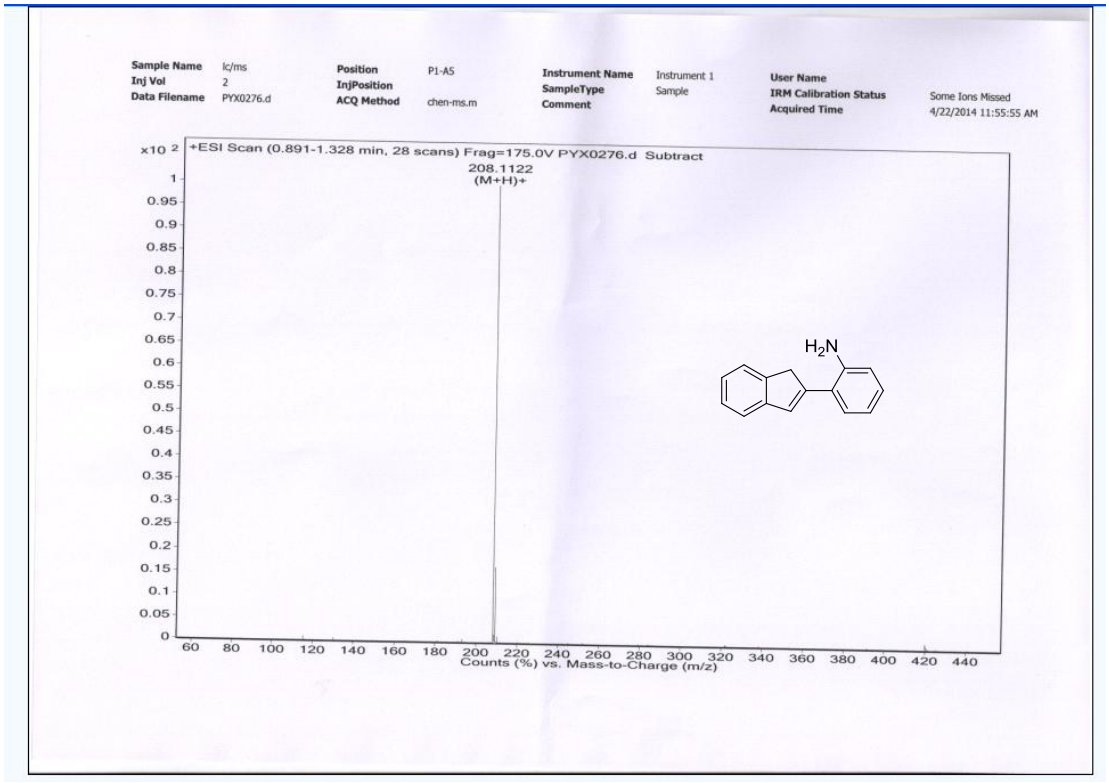


Figure S1. The optimized oxidation addition complexes composed of **1**-Pd(Ph)Cl (**A**, **B**), **2**-Pd(Ph)Cl (**C**), and **8**-Pd(Ph)Cl (**D**).

^{1}H NMR, ^{31}P NMR, ^{13}C NMR spectra and HRMS of compound **4**, **5**, **1·BH$_3$** and **2·BH$_3$**

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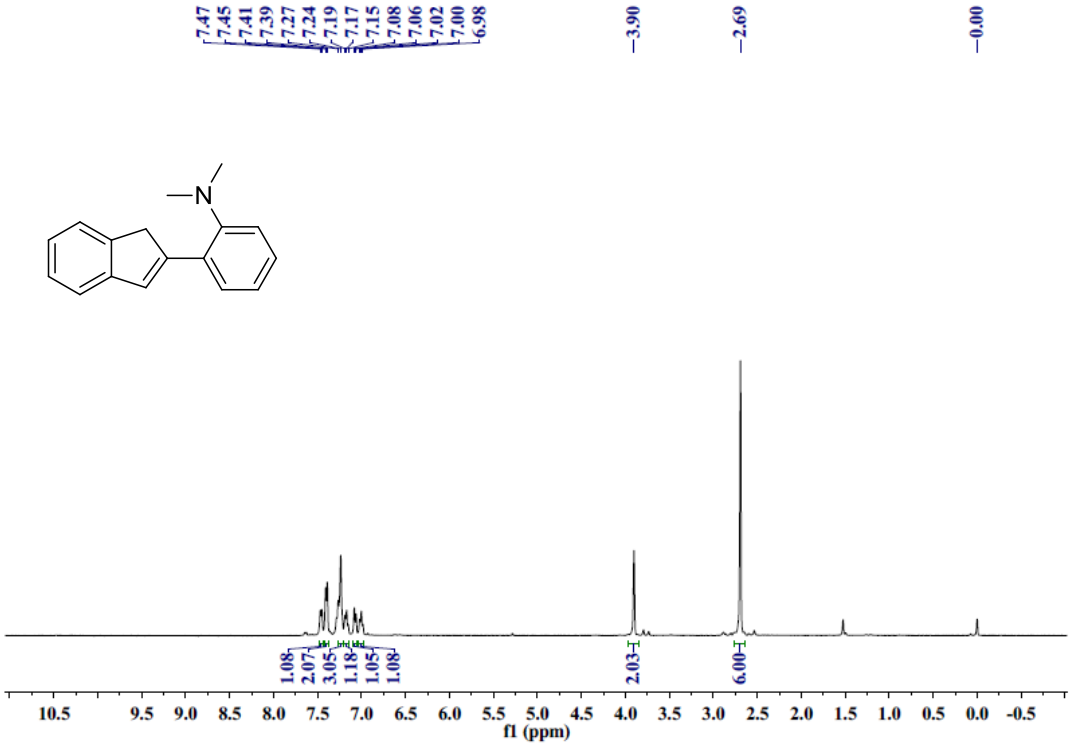
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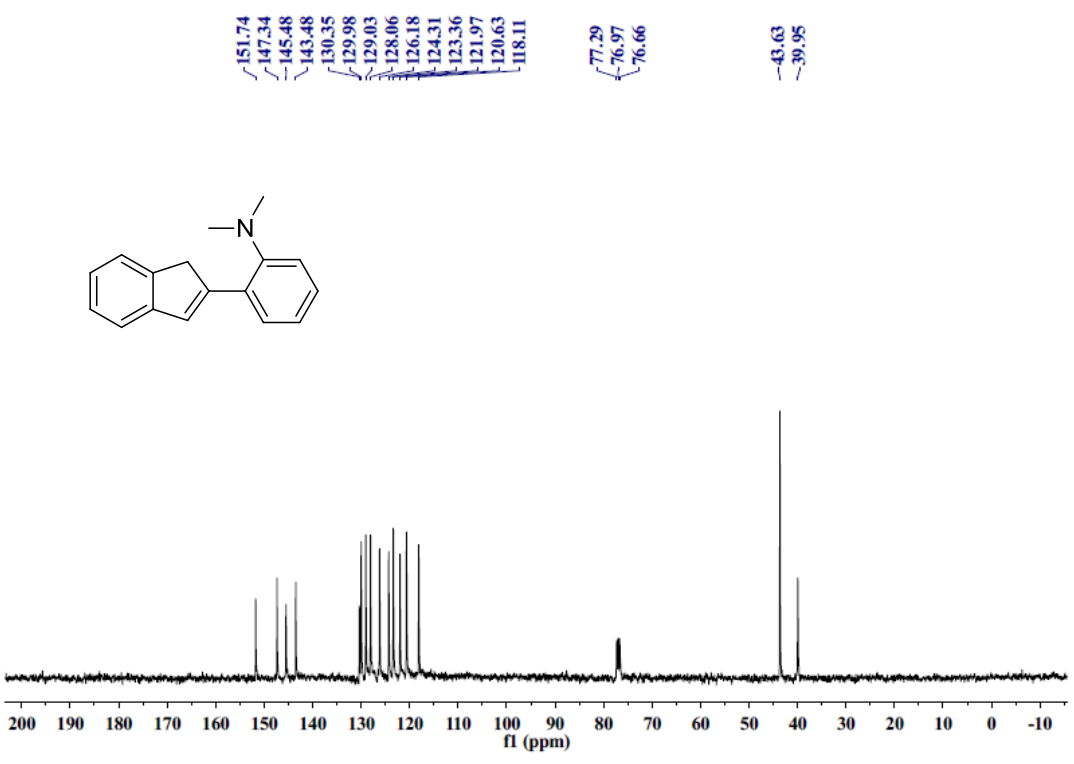
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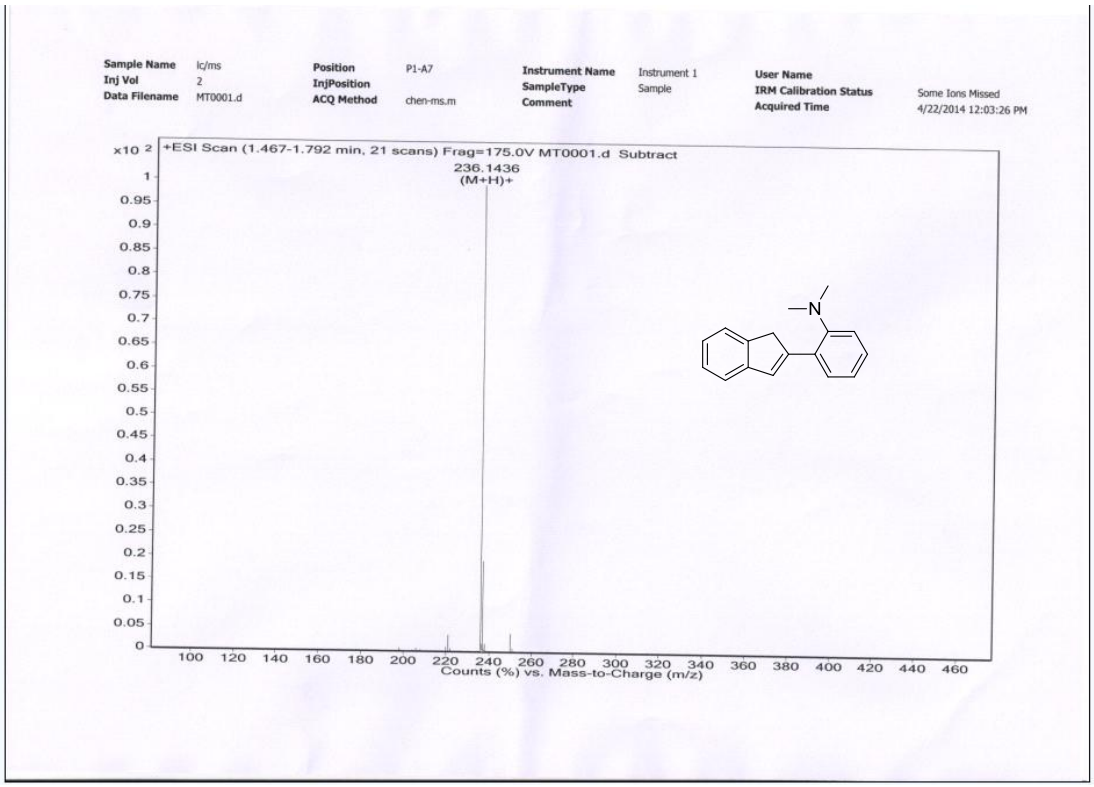
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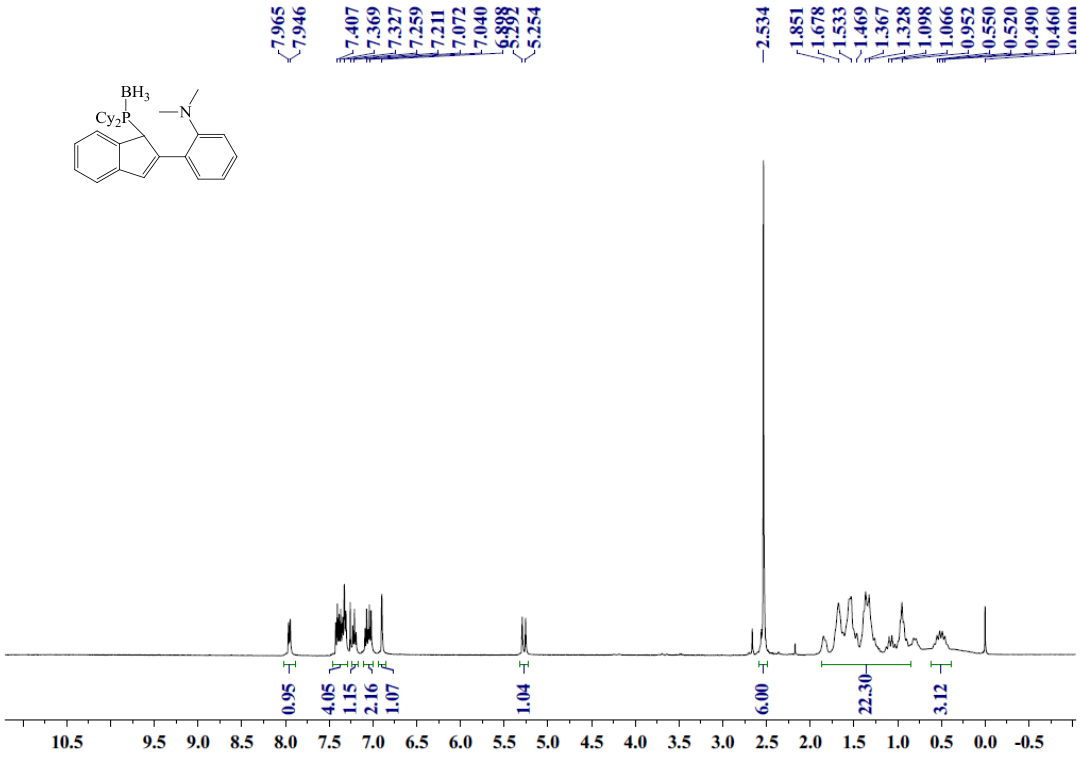
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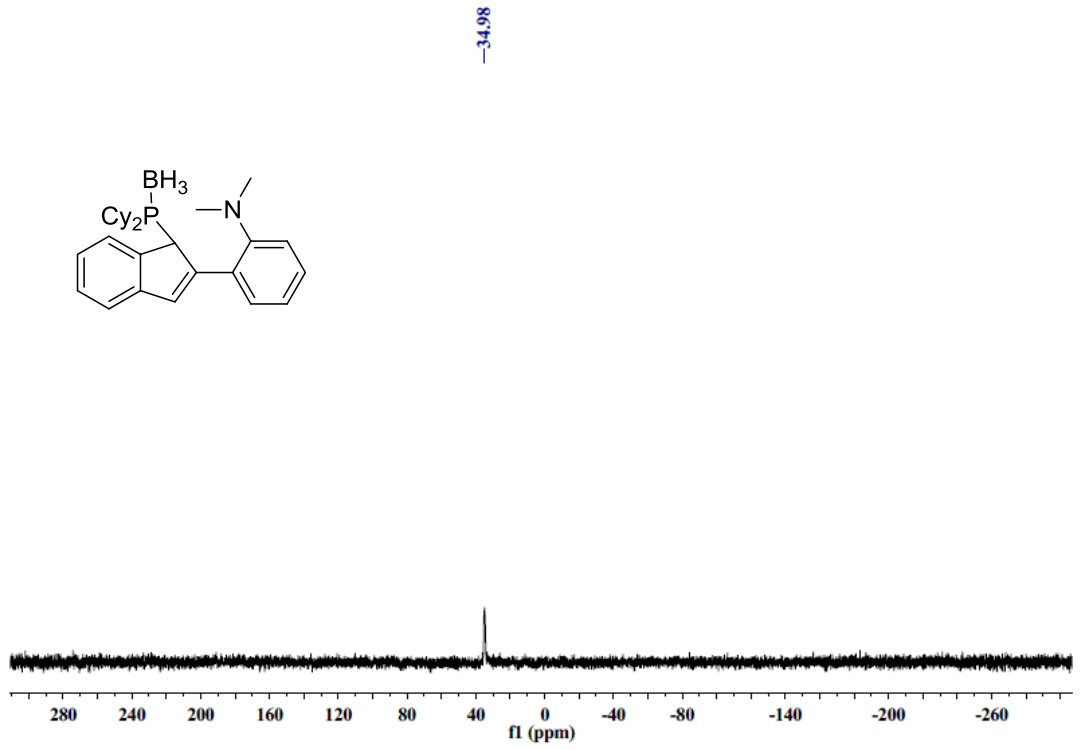
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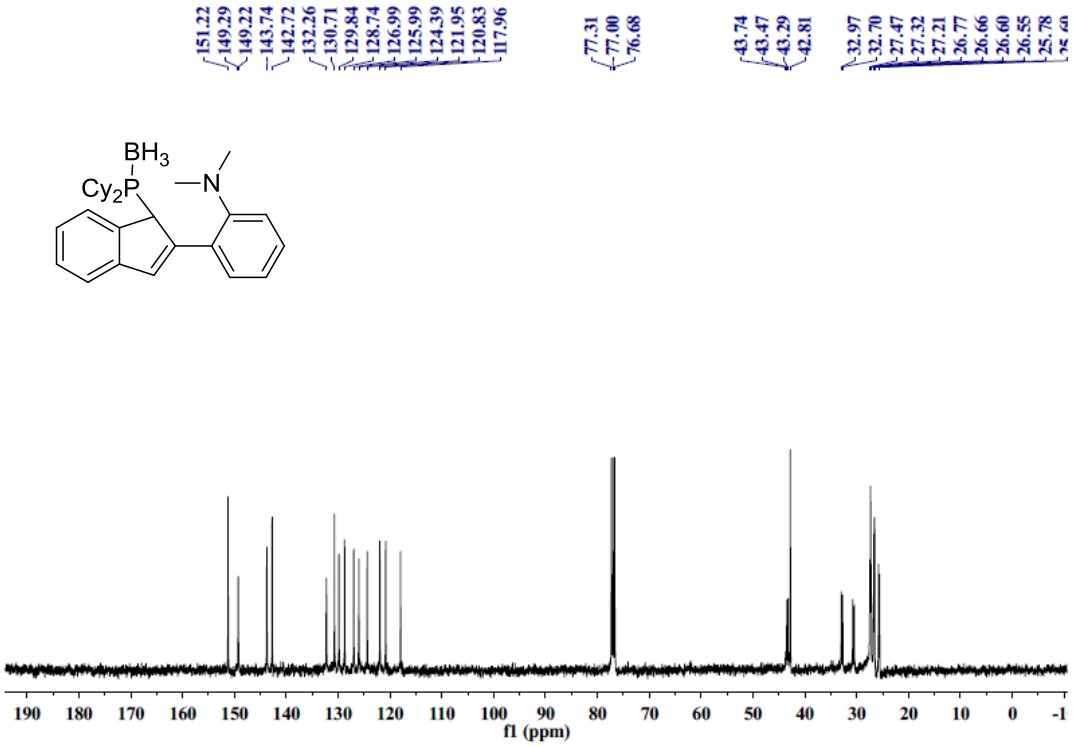
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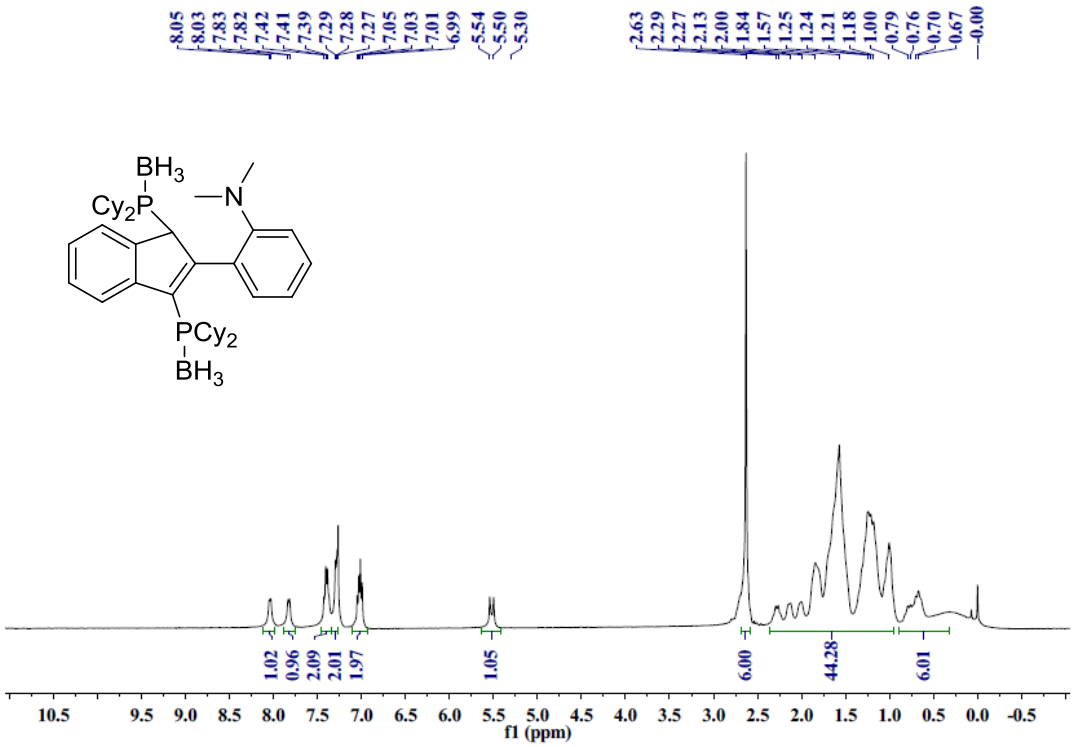
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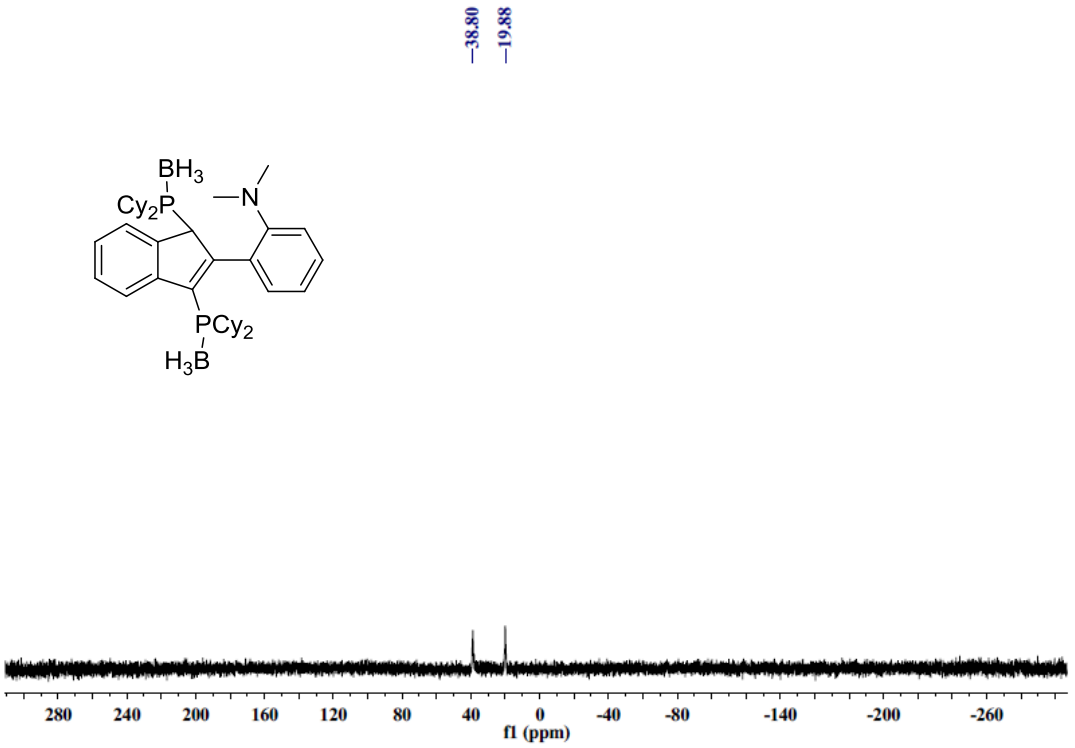
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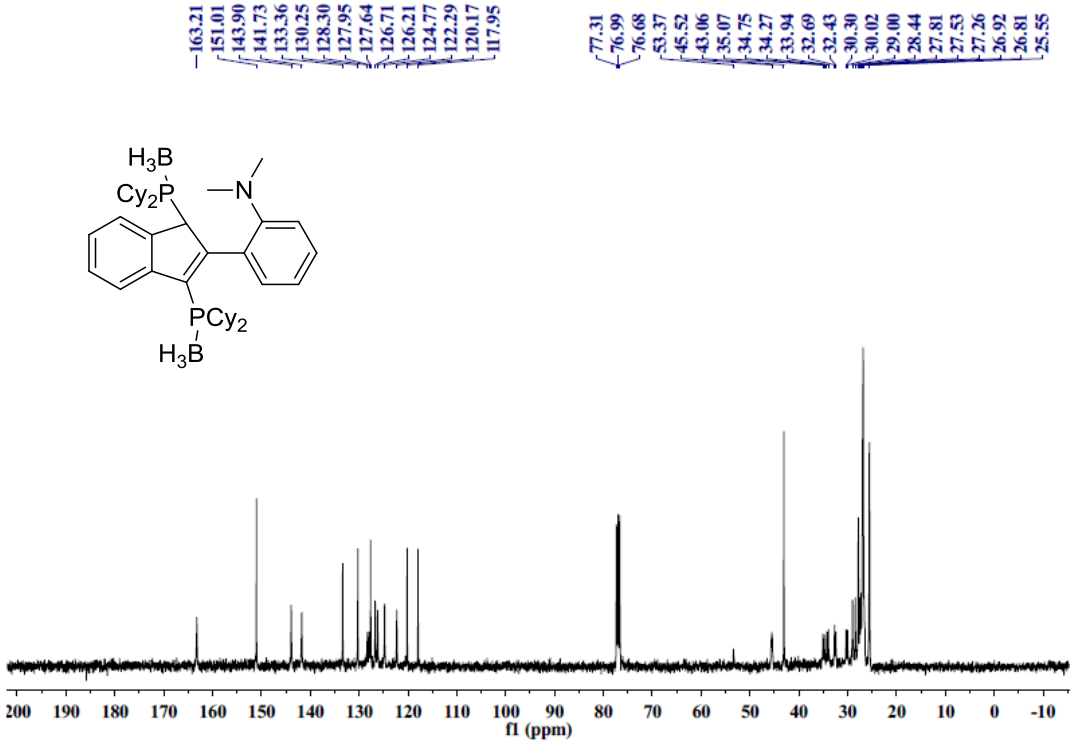
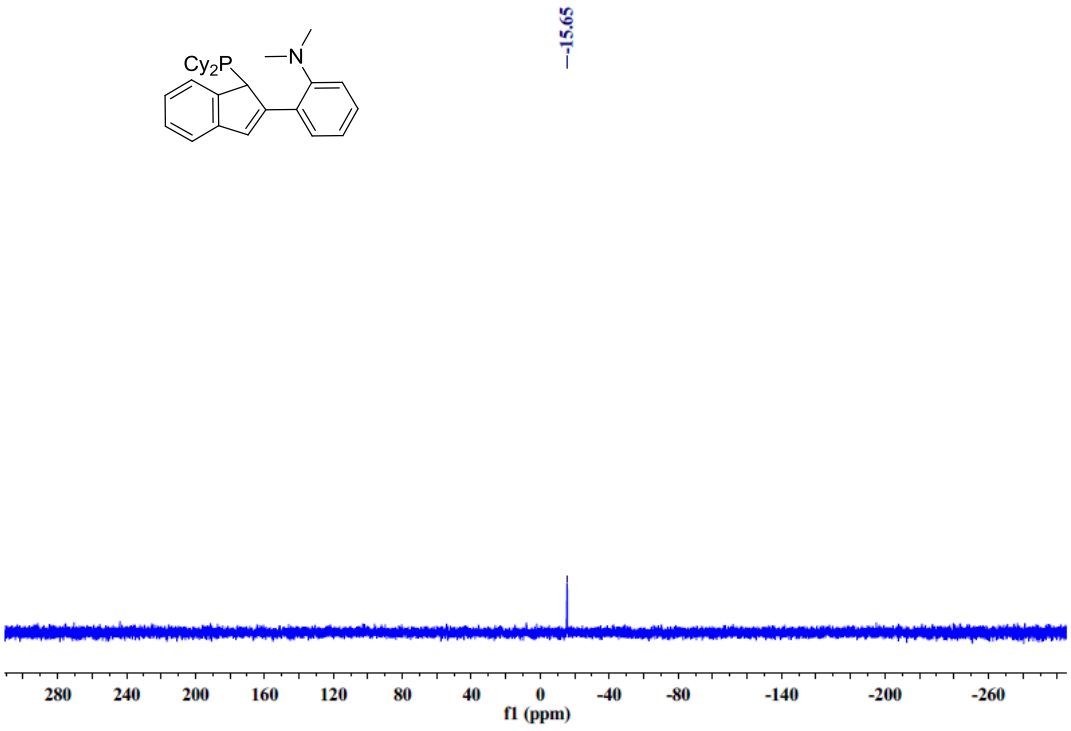
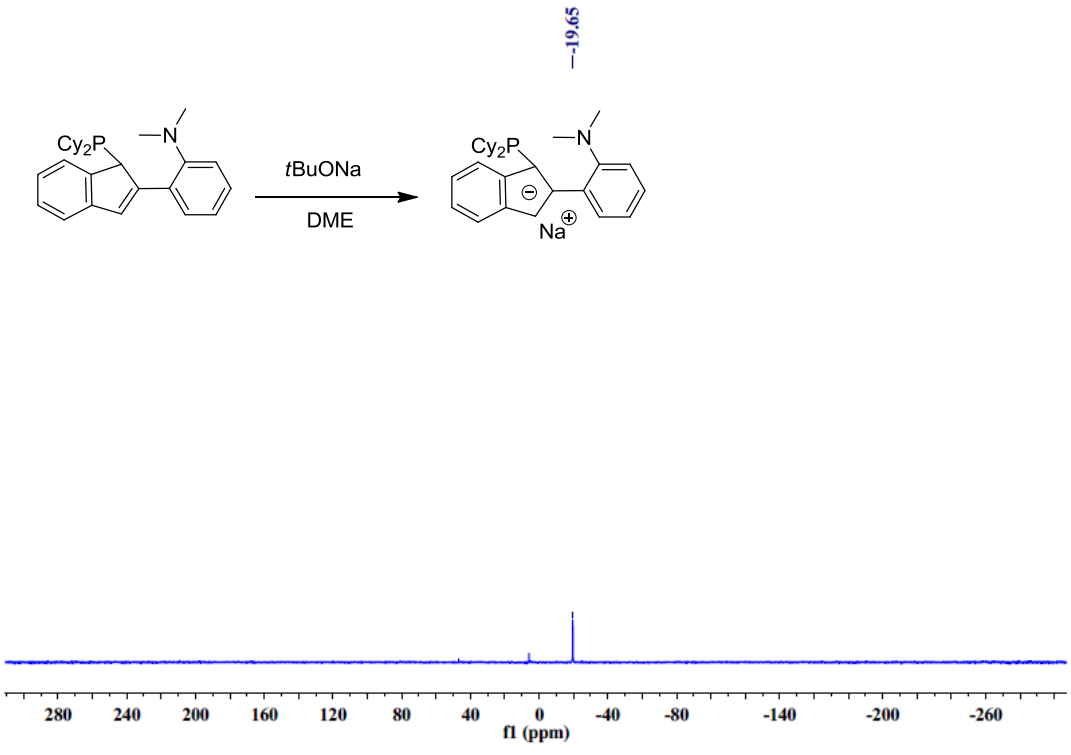
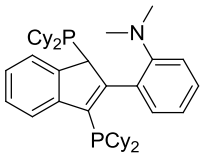
f1 (ppm)


163.21
151.01
143.90
141.73
133.36
130.25
128.30
127.95
127.64
126.71
126.21
124.77
122.29
120.17
117.95
77.31
76.99
76.68
53.37
45.52
43.06
35.07
34.75
34.27
33.94
32.69
32.43
30.30
30.02
29.00
28.44
27.81
27.53
27.26
26.92
26.81
25.55
H3B
Cy2P
N
PCy2
H3B
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

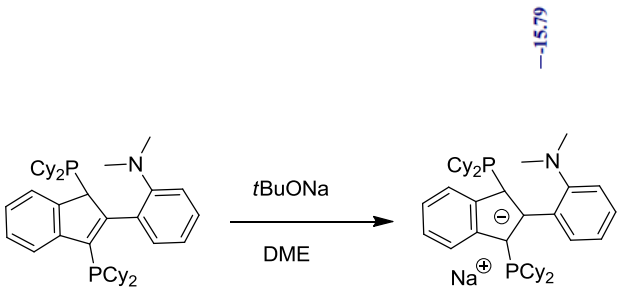
^{31}P NMR spectra of compound **1**, **2**, **1**$^{-}$ **and 2**$^{-}$





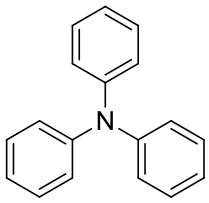

Cy2P
N
PCy2

-7.19
-20.18
280
240
200
160
120
80
40
0
-40
-80
-140
-200
-260
f1 (ppm)

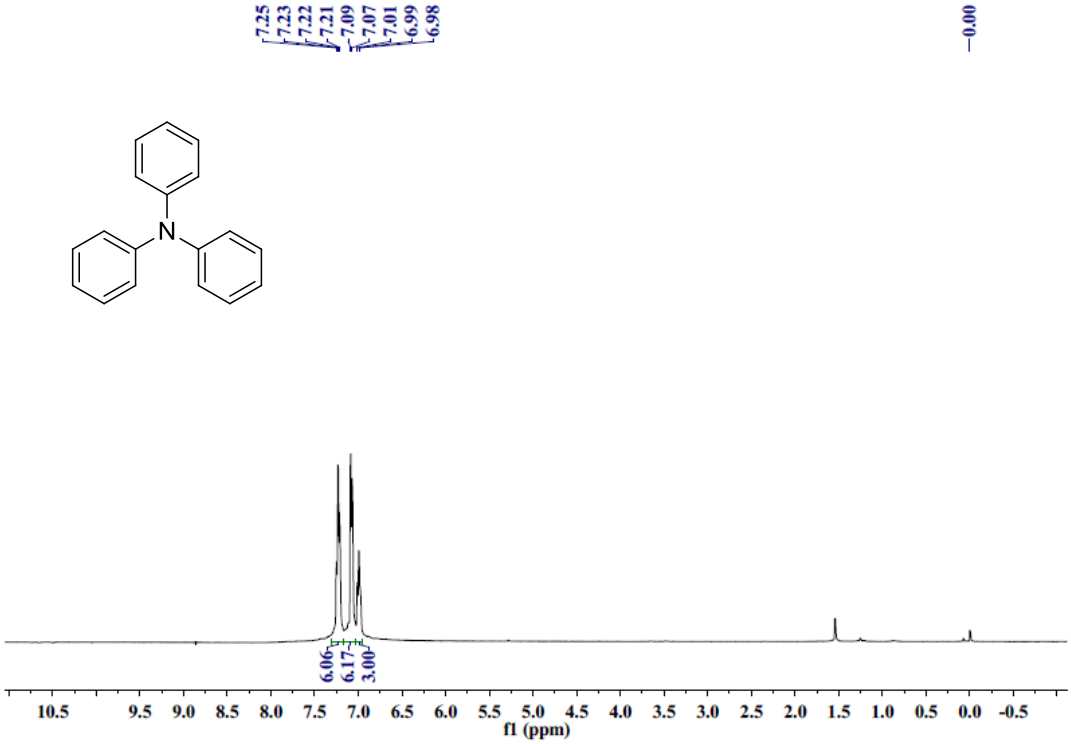

Cy2P
N
PCy2
tBuONa
DME
Cy2P
N
Na
PCy2

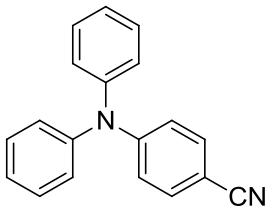
-15.79
280
240
200
160
120
80
40
0
-40
-80
-140
-200
-260
f1 (ppm)

^{1}H NMR, ^{13}C NMR and HRMS Spectra of Triaryl Amines Products

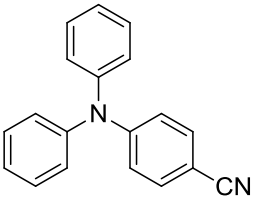


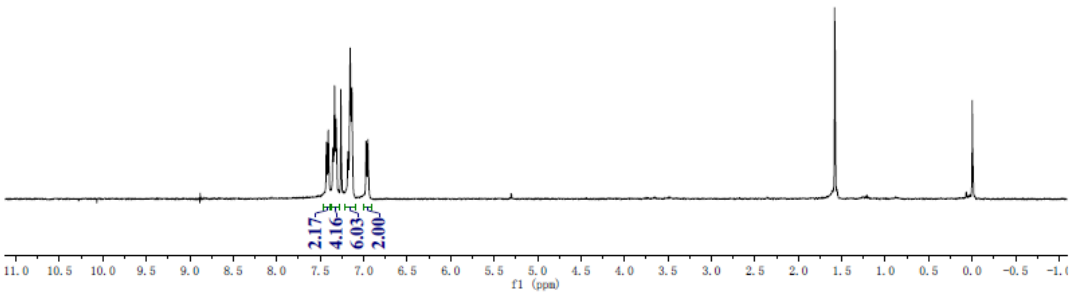
Triphenylamine. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, *J* = 4.3 Hz, 6H), 7.08 (d, *J* = 6.5 Hz, 6H), 6.99 (t, *J* = 6.2 Hz, 3H). Data is consistent with that reported in the literature.[2]

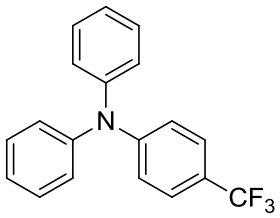




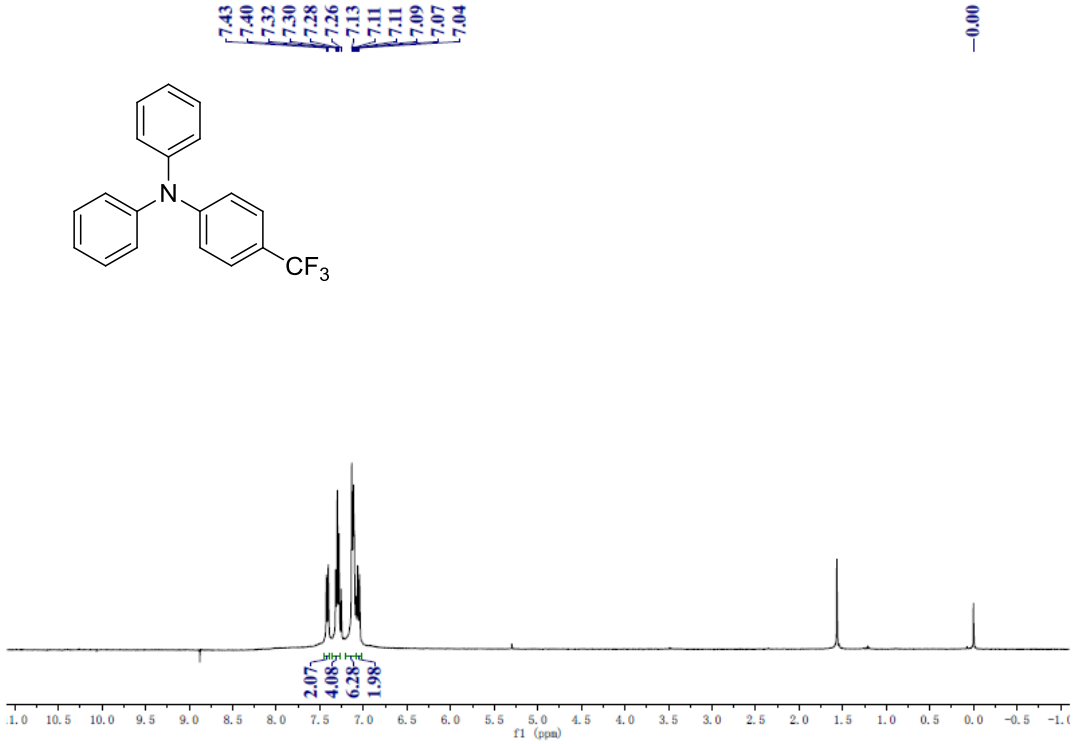
4-(diphenylamino)benzonitrile. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.7 Hz, 2H), 7.33 (t, *J* = 7.1 Hz, 4H), 7.16 (t, *J* = 9.1 Hz, 6H), 6.96 (d, *J* = 8.8 Hz, 2H). Data is consistent with that reported in the literature.[3]

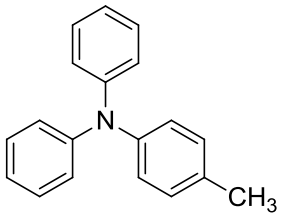




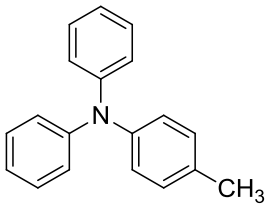


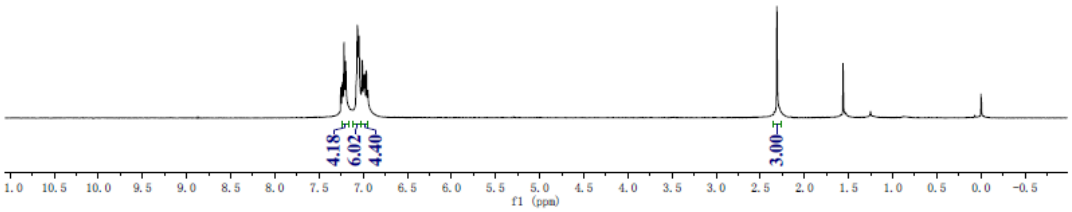
N,N-diphenyl-4-(trifluoromethyl)aniline. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 (d, *J* = 8.6 Hz, 2H), 7.30 (t, *J* = 7.8 Hz, 4H), 7.11 (d, *J* = 7.8 Hz, 6H), 7.06 (d, *J* = 8.6 Hz, 2H). Data is consistent with that reported in the literature.[2]

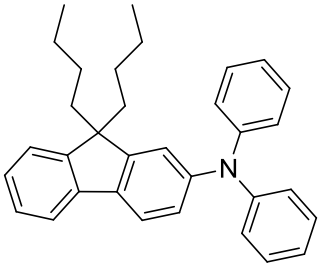




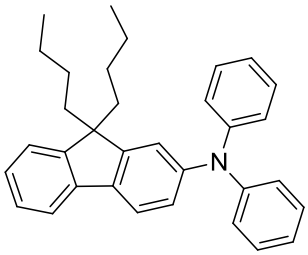
4-methyl-N,N-diphenylaniline. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22 (t, *J* = 7.1 Hz, 4H), 7.11-7.03 (m, 6H), 6.98 (d, *J* = 8.8 Hz, 4H), 2.31 (s, 3H). Data is consistent with that reported in the literature.[2]

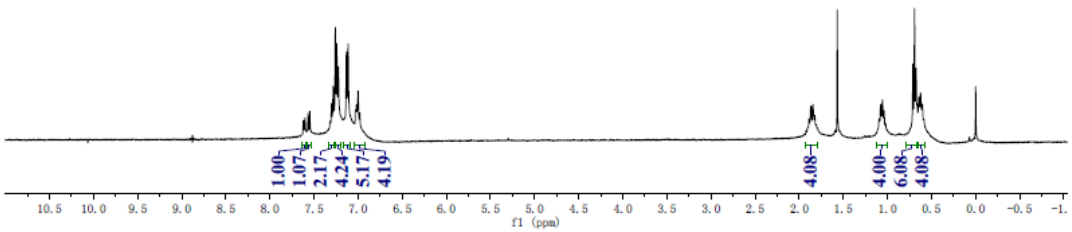


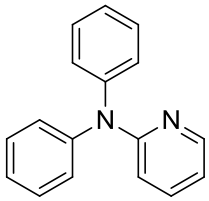




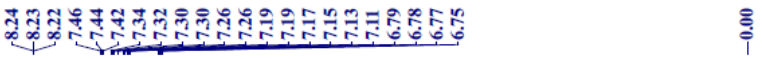
9,9-dibutyl-N,N-diphenyl-9H-fluoren-2-amine. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61 (d, *J* = 7.5 Hz, 1H), 7.56 (d, *J* = 8.1 Hz, 1H), 7.29 (d, *J* = 6.7 Hz, 2H), 7.23 (d, *J* = 7.6 Hz, 4H), 7.12 (d, *J* = 7.8 Hz, 5H), 7.00 (t, *J* = 7.2 Hz, 4H), 1.86 (d, *J* = 9.0 Hz, 4H), 1.12 - 1.01 (m, 4H), 0.69 (t, *J* = 7.3 Hz, 6H), 0.66-0.58 (m, 4H). Data is consistent with that reported in the literature.[4]

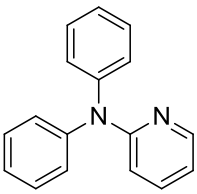


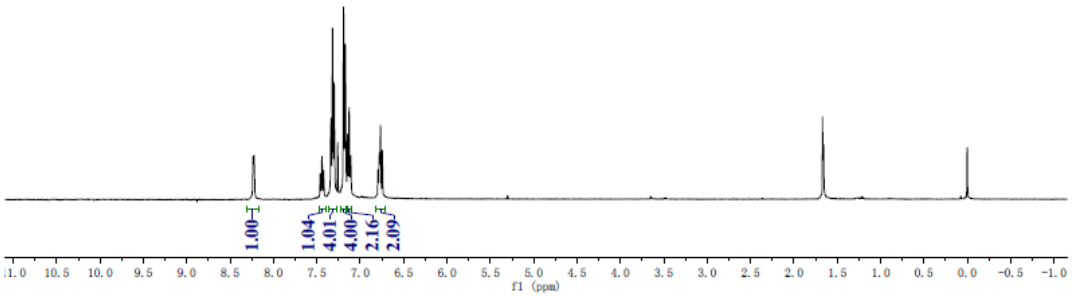


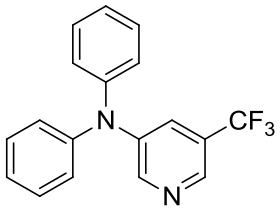


N,N-diphenylpyridin-2-amine. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.31-8.17 (m, 1H), 7.44 (t, *J* = 7.8 Hz, 1H), 7.31 (d, *J* = 4.4 Hz, 4H), 7.23-7.15 (m, 4H), 7.13 (t, *J* = 7.3 Hz, 2H), 6.77 (d, *J* = 7.0 Hz, 2H). Data is consistent with that reported in the literature.[2]

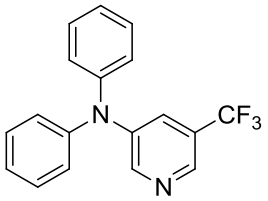


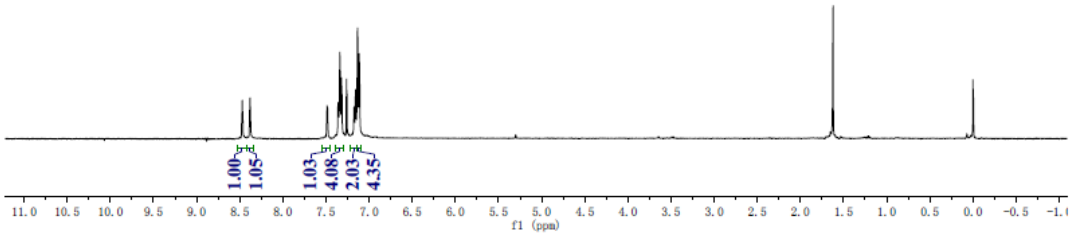


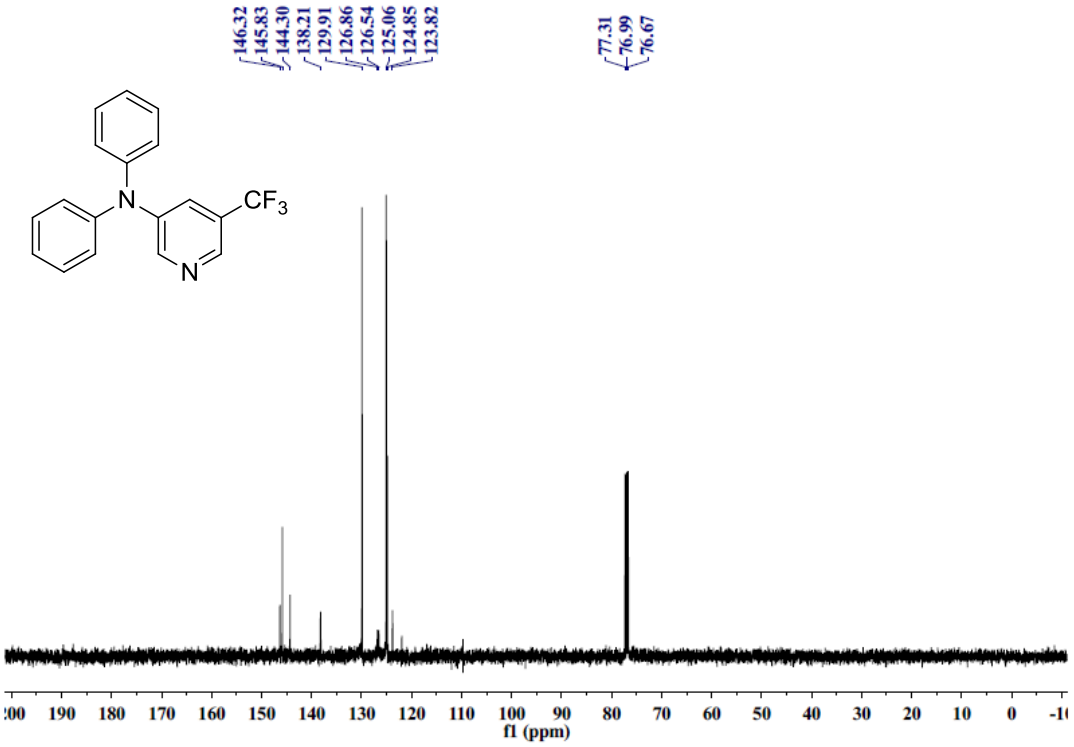


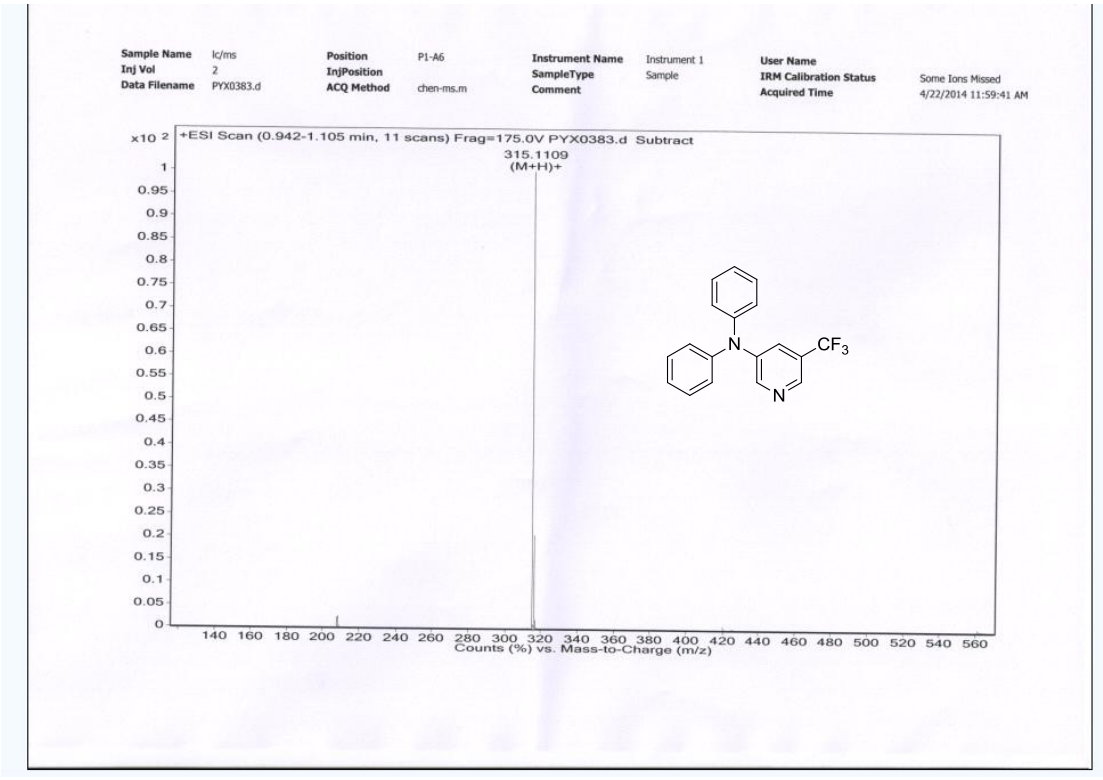


N,N-diphenyl-5-(trifluoromethyl)pyridin-3-amine. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.47 (s, 1H), 8.38 (s, 1H), 7.49 (s, 1H), 7.34 (t, *J* = 6.7 Hz, 4H), 7.16 (d, *J* = 7.4 Hz, 2H), 7.12 (d, *J* = 8.3 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.32, 145.83, 144.30, 138.21, 129.91, 126.86, 126.54, 125.06, 124.85, 123.82. HRMS (ESI/ $[M+H]^+$): Calcd for $C_{18}H_{13}F_3N_2$: 315.1109; Found: 315.1109.






146.32
145.83
144.30
138.21
129.91
126.86
126.54
125.06
124.85
123.82
77.31
76.99
76.67
N
CF3
N
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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Inj Vol 2
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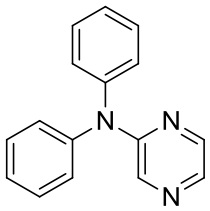
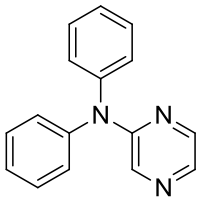
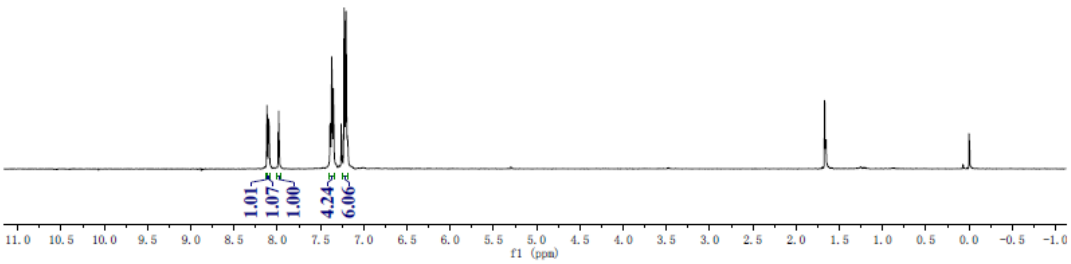
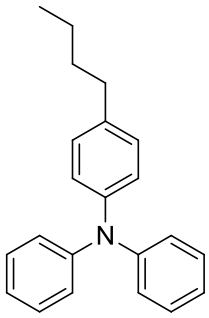
InjPosition
ACQ Method chen-ms.m
Instrument Name Instrument 1
SampleType Sample
Comment
User Name
IRM Calibration Status Some Ions Missed
Acquired Time 4/22/2014 11:59:41 AM
x10 2
+ESI Scan (0.942-1.105 min, 11 scans) Frag=175.0V PYX0383.d Subtract
315.1109
(M+H)+
1
0.95
0.9
0.85
0.8
0.75
0.7
0.65
0.6
0.55
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500 520 540 560
Counts (%) vs. Mass-to-Charge (m/z)
N
CF3
N



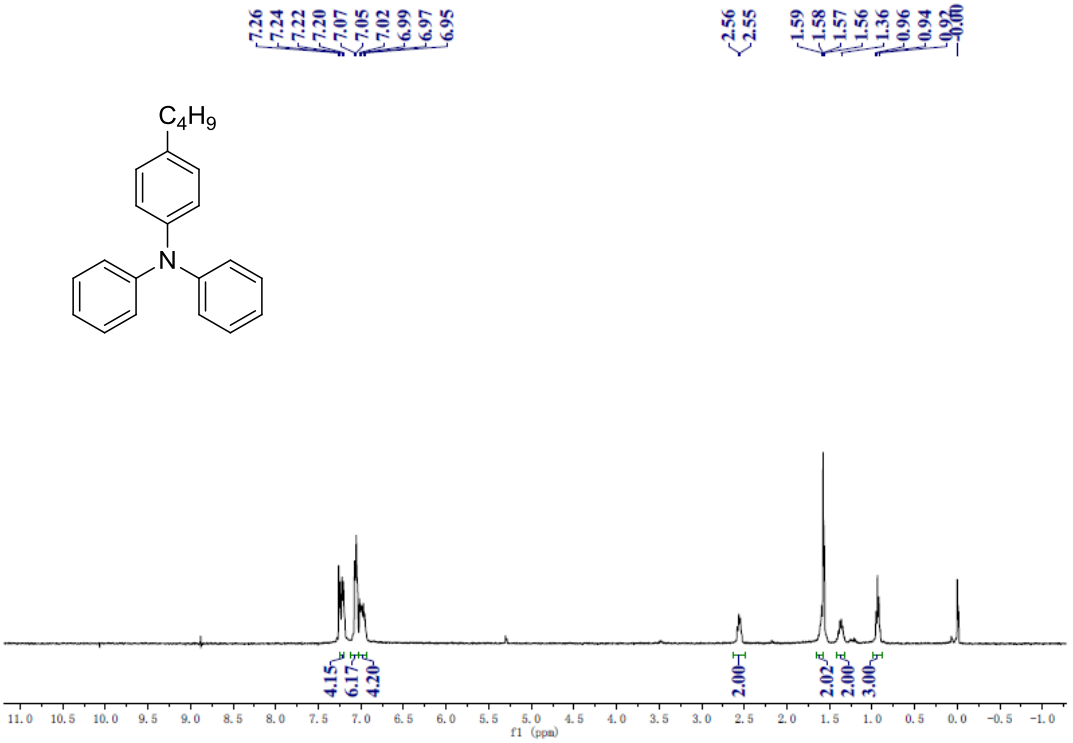
N,N-diphenylpyrazin-2-amine. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (s, 1H), 8.11-8.08 (m, 1H), 7.99 (d, *J* = 2.5 Hz, 1H), 7.40-7.34 (m, 4H), 7.21 (d, *J* = 3.9 Hz, 6H). Data is consistent with that reported in the literature.[5]

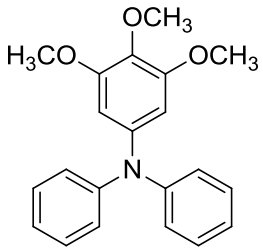




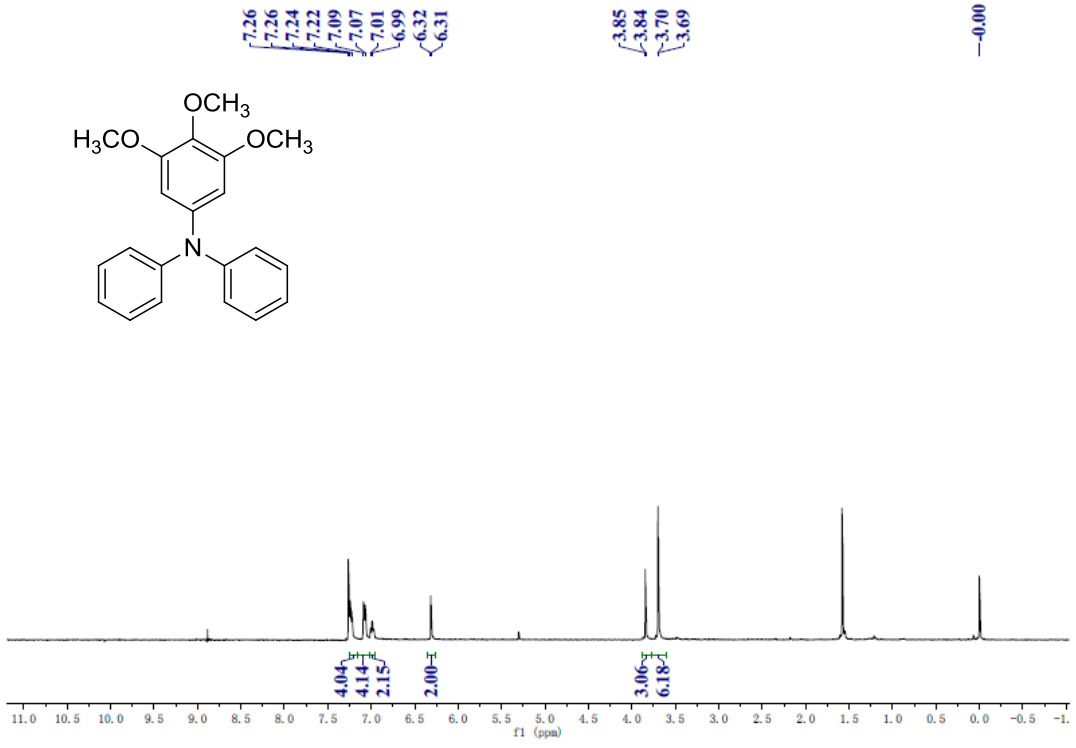


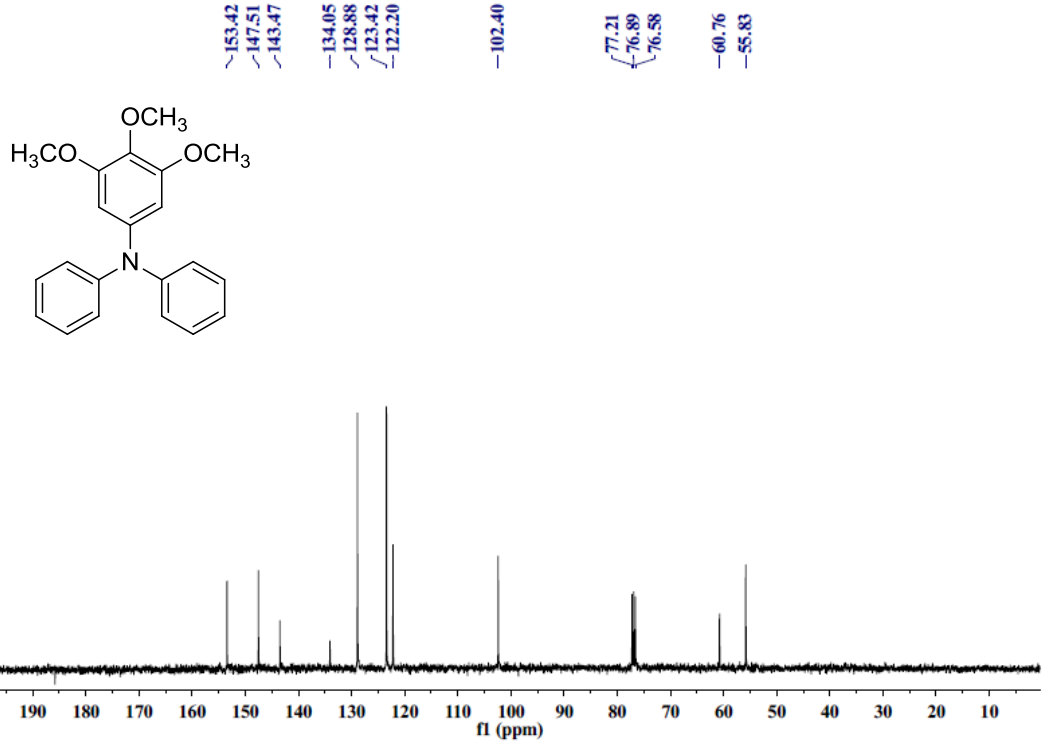
4-butyl-N,N-diphenylaniline. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25-7.19 (m, 4H), 7.06 (d, *J* = 7.9 Hz, 6H), 6.98 (d, *J* = 8.9 Hz, 4H), 2.55 (d, *J* = 7.4 Hz, 2H), 1.59 (d, *J* = 5.2 Hz, 2H), 1.42-1.31 (m, 2H), 0.94 (t, *J* = 7.2 Hz, 3H). Data is consistent with that reported in the literature.[6]

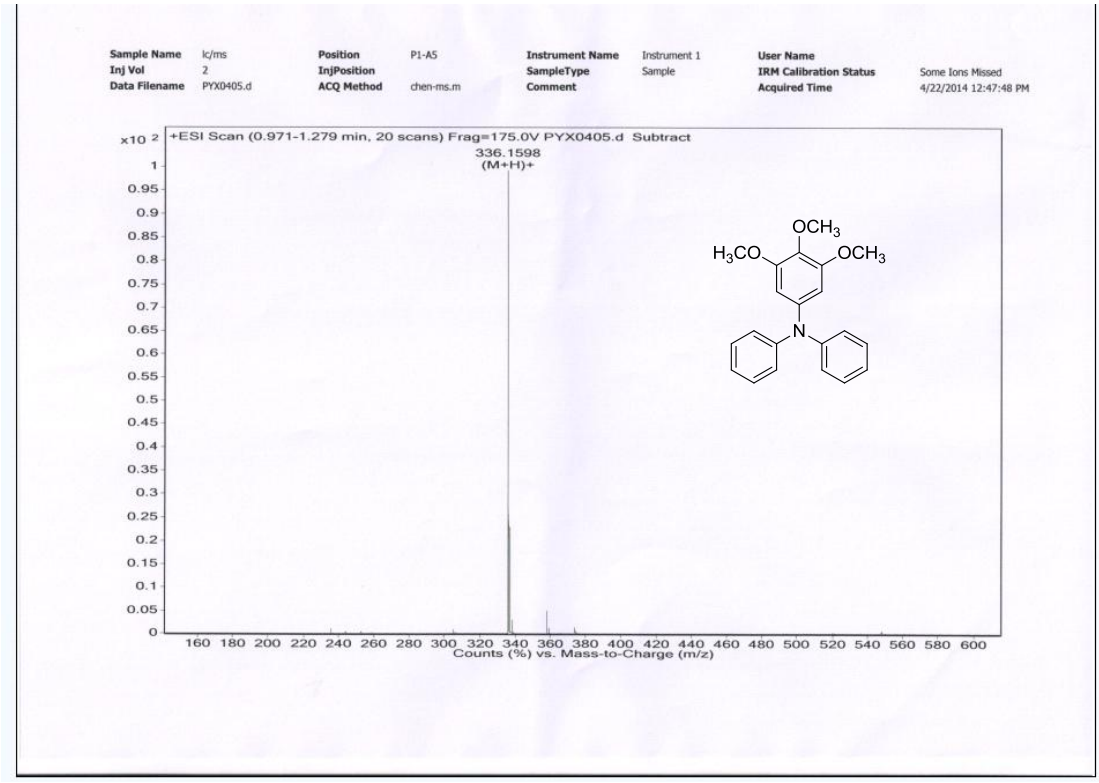




3,4,5-trimethoxy-N,N-diphenylaniline. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, *J* = 7.3 Hz, 4H), 7.08 (d, *J* = 8.3 Hz, 4H), 7.00 (d, *J* = 7.5 Hz, 2H), 6.31 (d, *J* = 2.6 Hz, 2H), 3.84 (d, *J* = 2.6 Hz, 3H), 3.70 (d, *J* = 2.6 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.42, 147.51, 143.47, 134.05, 128.88, 123.42, 122.20, 102.40, 60.76, 55.83. HRMS (ESI/ $[M+H]^+$): Calcd for $C_{21}H_{21}NO_3$: 336.1600. Found 336.1598.




153.42
147.51
143.47
134.05
128.88
123.42
122.20
102.40
77.21
76.89
76.58
60.76
55.83
OCH3
H3CO
OCH3
N
f1 (ppm)


Sample Name lc/ms
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Instrument Name Instrument 1
User Name
Inj Vol 2
InjPosition
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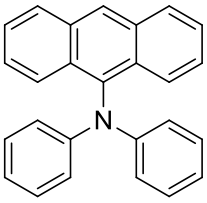
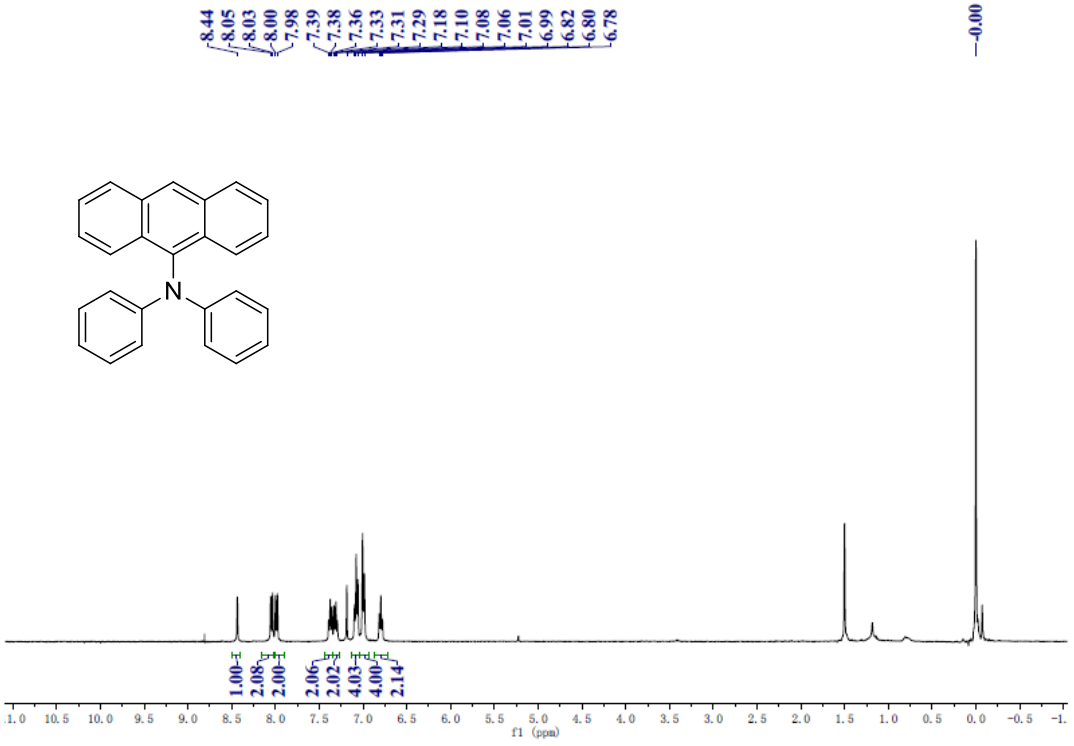
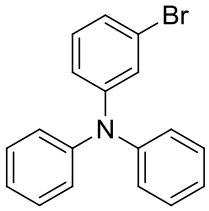
IRM Calibration Status Some Ions Missed
Data Filename PYX0405.d
ACQ Method chen-ms.m
Comment
Acquired Time 4/22/2014 12:47:48 PM
+ESI Scan (0.971-1.279 min, 20 scans) Frag=175.0V PYX0405.d Subtract
336.1598
(M+H)+
x10 2
Counts (%) vs. Mass-to-Charge (m/z)
OCH3
H3CO
OCH3
N

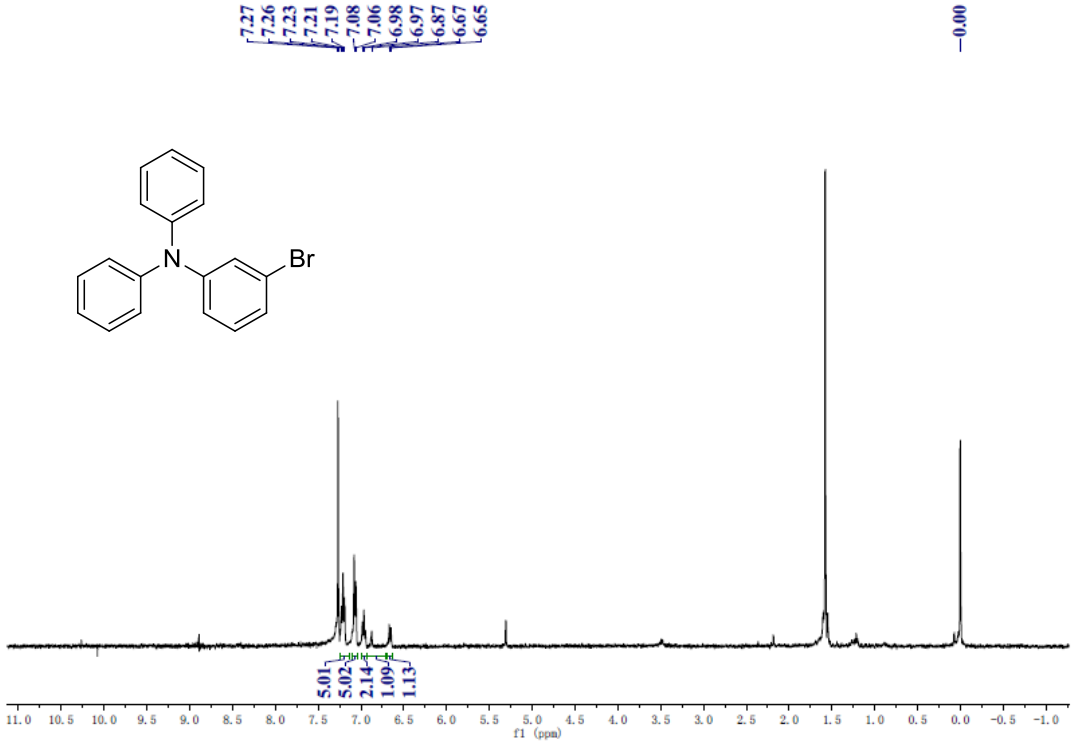


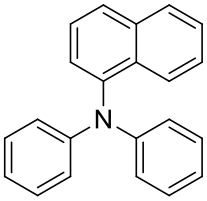
N,N-diphenylanthracen-9-amine. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.44 (s, 1H), 8.04 (d, *J* = 8.6 Hz, 2H), 7.99 (d, *J* = 8.4 Hz, 2H), 7.43-7.34 (m, 2H), 7.35-7.27 (m, 2H), 7.08 (t, *J* = 7.2 Hz, 4H), 7.00 (d, *J* = 8.2 Hz, 4H), 6.80 (t, *J* = 7.1 Hz, 2H). Data is consistent with that reported in the literature.[7]



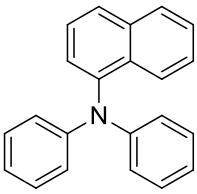


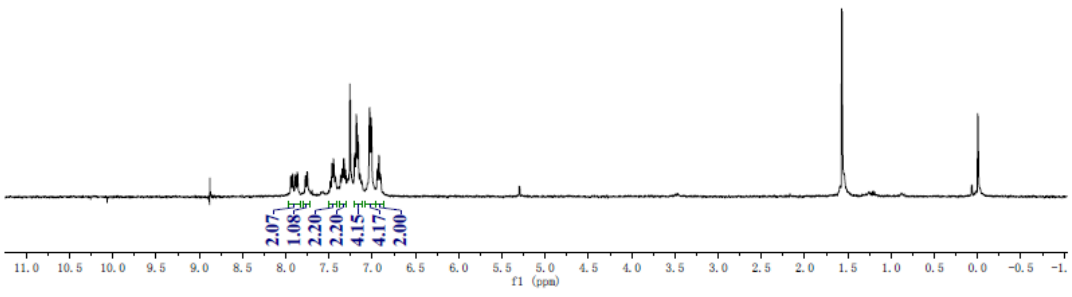
3-bromo-N,N-diphenylaniline. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21 (t, *J* = 6.7 Hz, 5H), 7.07 (d, *J* = 7.7 Hz, 5H), 6.97 (d, *J* = 7.1 Hz, 2H), 6.87 (s, 1H), 6.66 (d, *J* = 8.0 Hz, 1H). Data is consistent with that reported in the literature.[8]

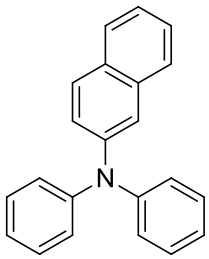




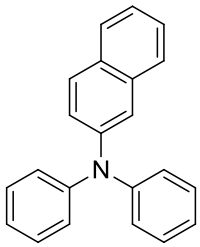
N,N-diphenylnaphthalen-1-amine. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.5 Hz, 2H), 7.76 (d, *J* = 7.7 Hz, 1H), 7.46 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 6.9 Hz, 2H), 7.17 (d, *J* = 7.9 Hz, 4H), 7.02 (d, *J* = 6.7 Hz, 4H), 6.93 (d, *J* = 7.2 Hz, 2H). Data is consistent with that reported in the literature.[2]

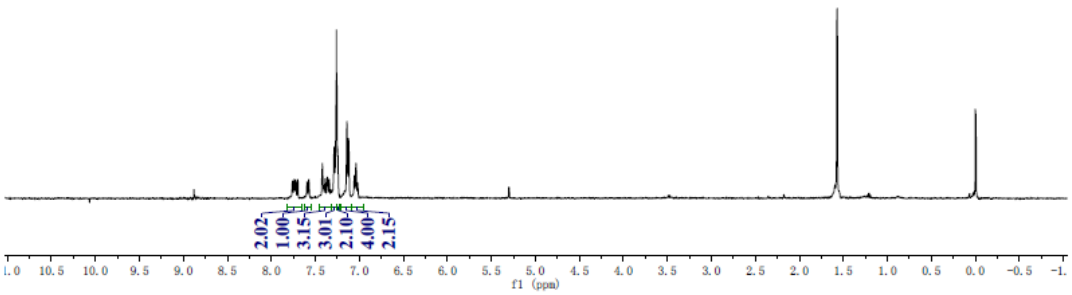


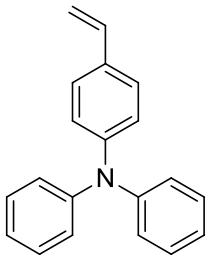




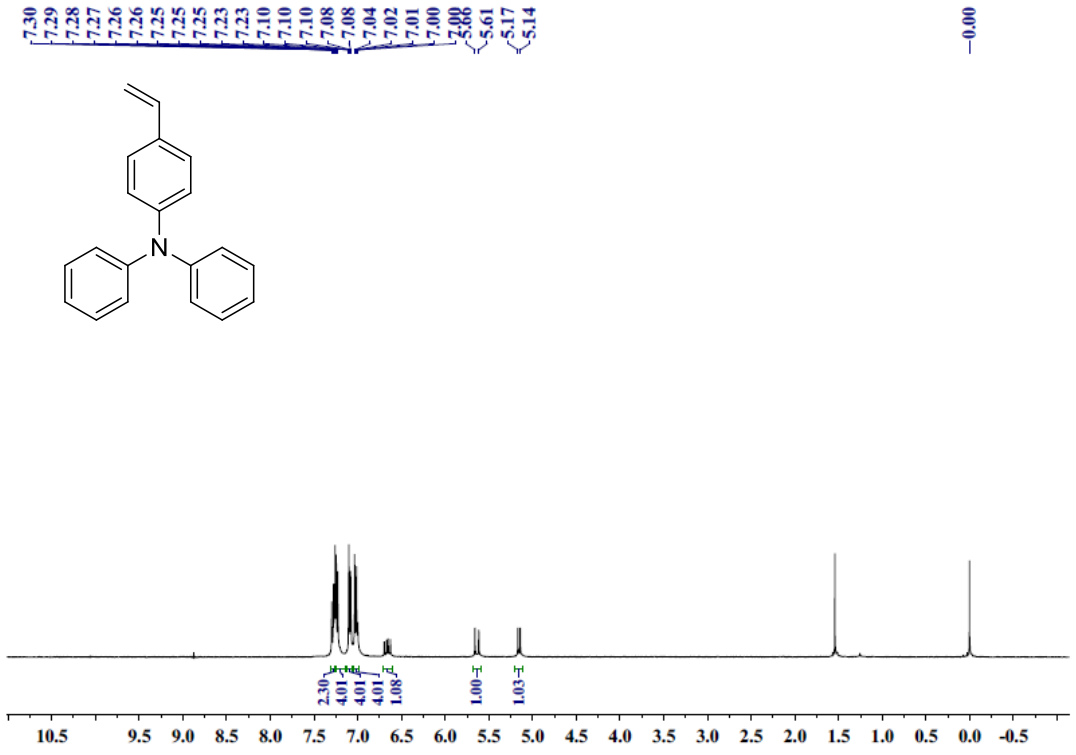
N,N-diphenylnaphthalen-2-amine. White solid,[1]H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, *J* = 8.3 Hz, 2H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.46 - 7.32 (m, 3H), 7.28 (d, *J* = 9.3 Hz, 3H), 7.25 (d, *J* = 2.8 Hz, 2H), 7.13 (d, *J* = 8.1 Hz, 4H), 7.04 (t, *J* = 7.3 Hz, 2H). Data is consistent with that reported in the literature.[2]

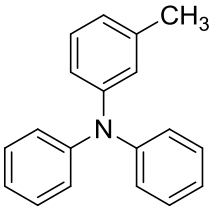




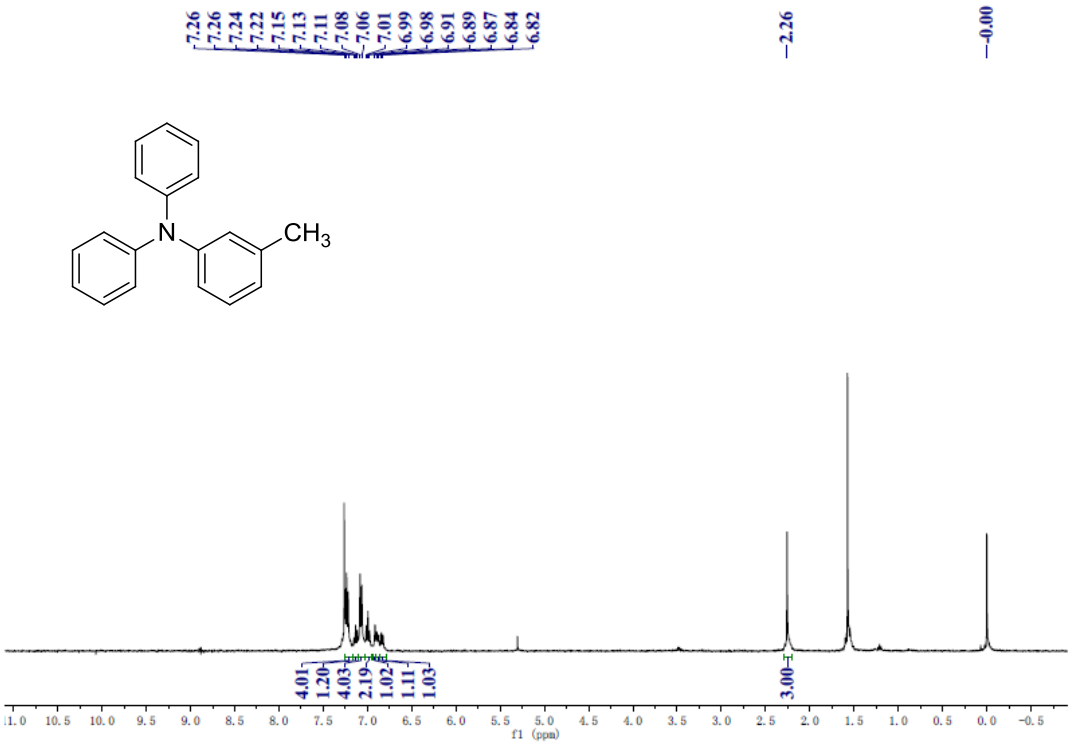


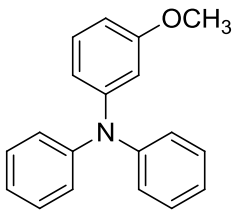
N,N-diphenyl-4-vinylaniline. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28 (d, *J* = 2.7 Hz, 2H), 7.26-7.15 (m, 4H), 7.09 (d, *J* = 4.4 Hz, 4H), 7.05-6.99 (m, 4H), 6.66 (d, *J* = 10.9 Hz, 1H), 5.64 (d, *J* = 17.5 Hz, 1H), 5.15 (d, *J* = 10.9 Hz, 1H). Data is consistent with that reported in the literature.[9]



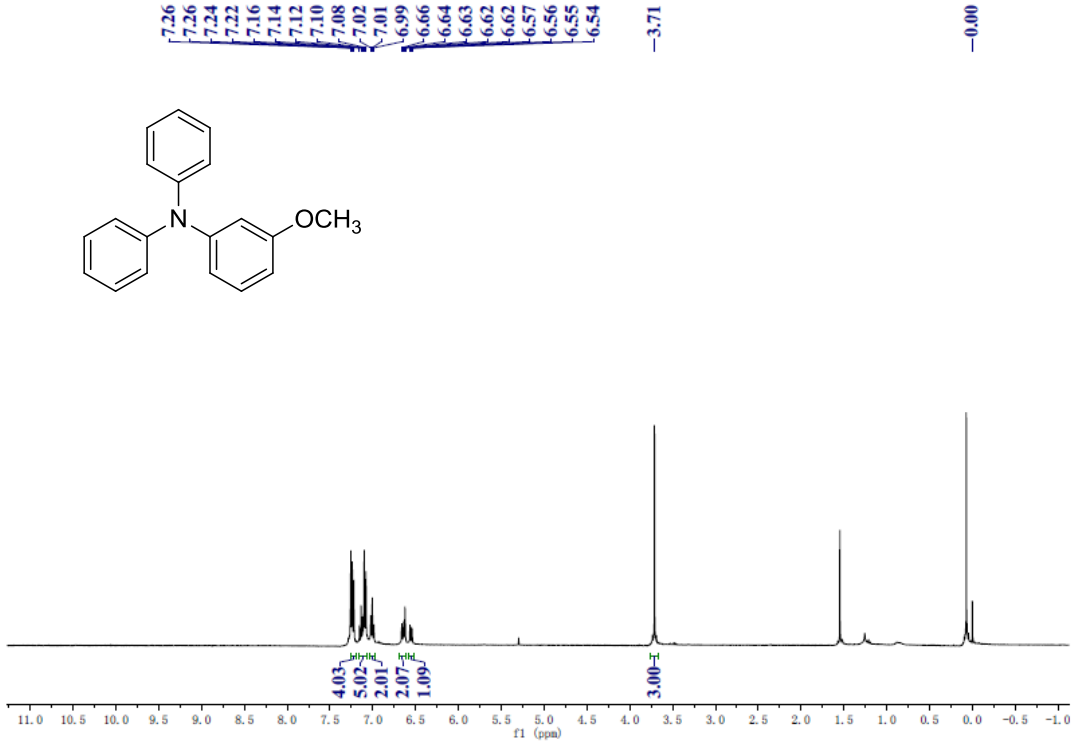


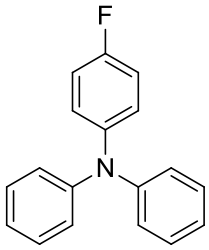
3-methyl-N,N-diphenylaniline. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, *J* = 7.5 Hz, 4H), 7.13 (t, *J* = 7.7 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 4H), 6.99 (t, *J* = 7.2 Hz, 2H), 6.91 (s, 1H), 6.88 (d, *J* = 8.5 Hz, 1H), 6.83 (d, *J* = 7.5 Hz, 1H), 2.26 (s, 3H). Data is consistent with that reported in the literature.[2]



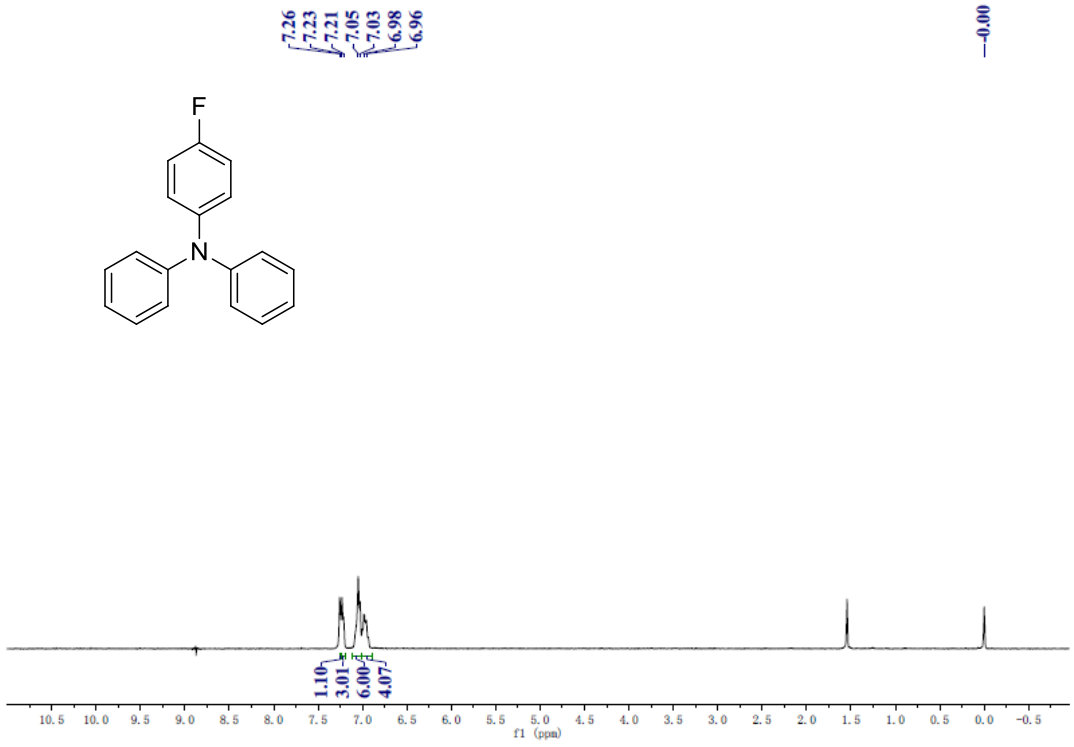


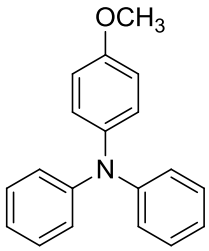
3-methoxy-N,N-diphenylaniline. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26-7.20 (m, 4H), 7.17-7.07 (m, 5H), 7.01 (t, *J* = 7.3 Hz, 2H), 6.70-6.61 (m, 2H), 6.55 (d, *J* = 2.4 Hz, 1H), 3.76-3.66 (m, 3H). Data is consistent with that reported in the literature.[2]



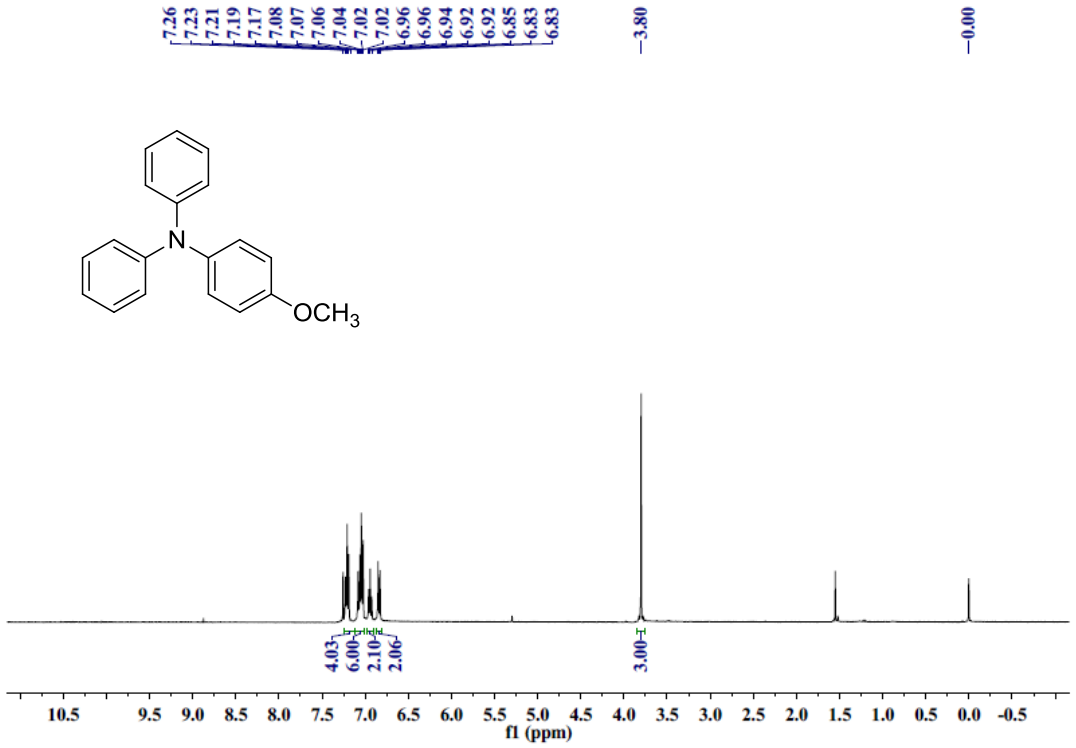


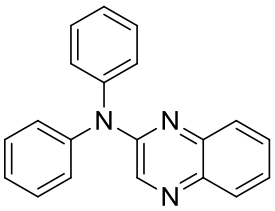
4-fluoro-N,N-diphenylaniline. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26-7.24 (m, 1H), 7.22 (d, *J* = 7.2 Hz, 3H), 7.04 (d, *J* = 8.2 Hz, 6H), 6.97 (d, *J* = 10.8 Hz, 4H). Data is consistent with that reported in the literature.[10]



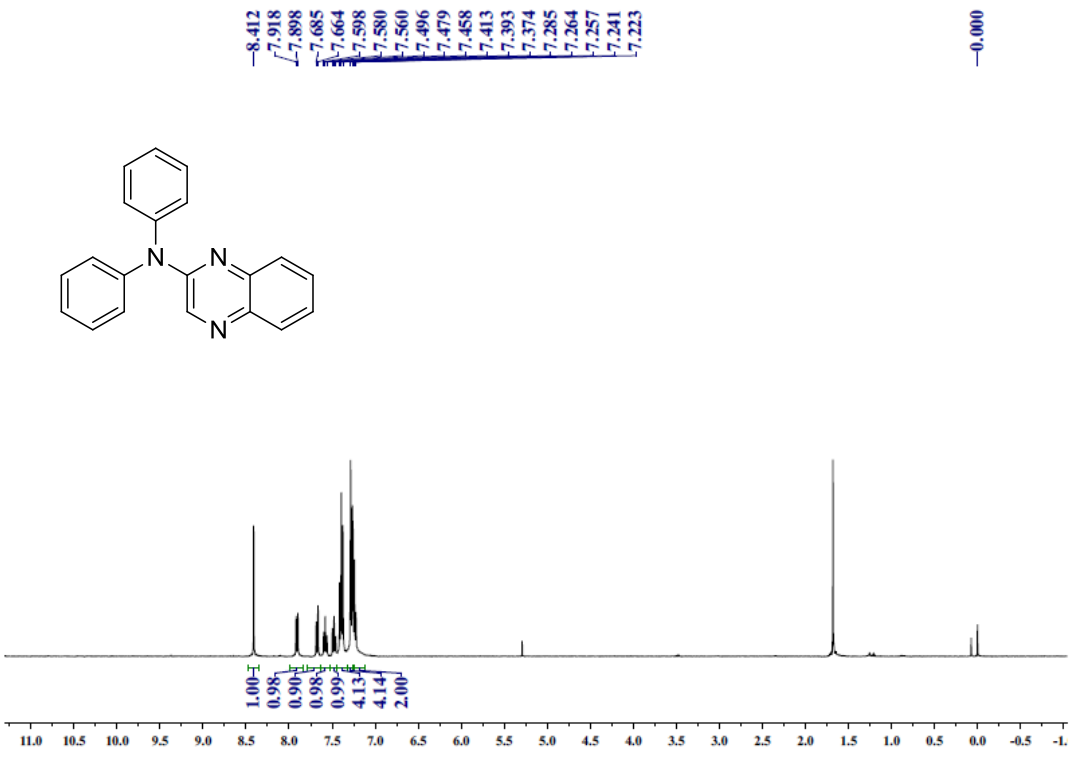


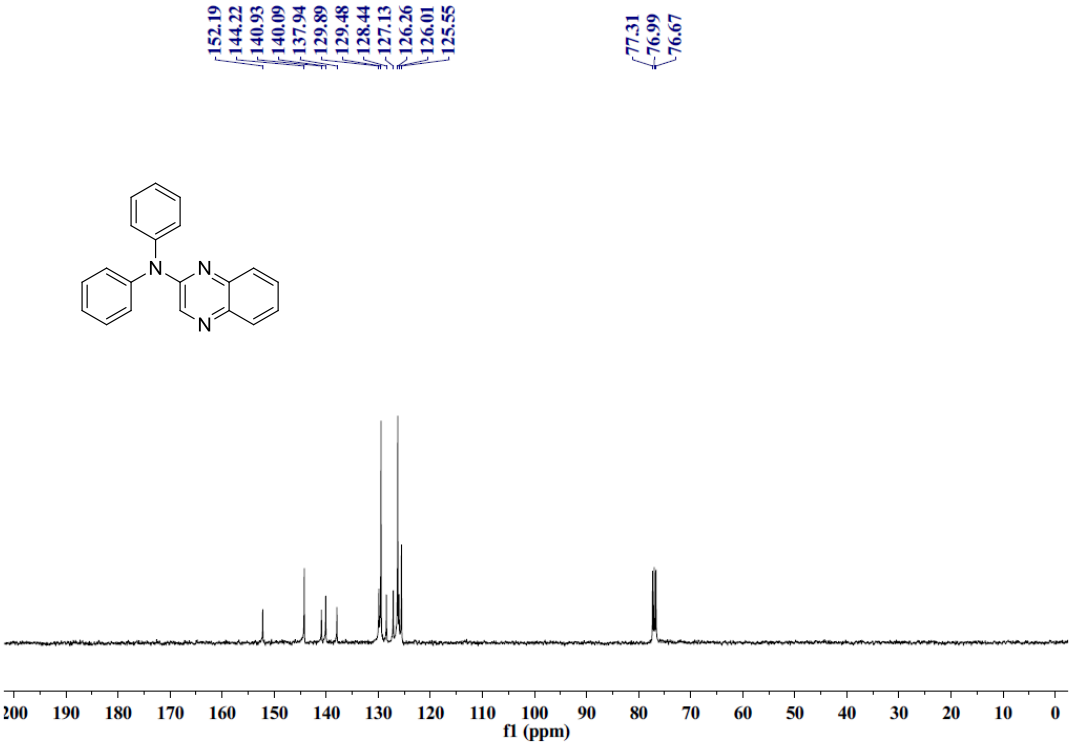
4-methoxy-N,N-diphenylaniline. White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21 (t, *J* = 7.9 Hz, 4H), 7.12-7.00 (m, 6H), 6.98-6.91 (m, 2H), 6.87-6.81 (m, 2H), 3.85-3.75 (m, 3H). Data is consistent with that reported in the literature.[2]





N, N-diphenylquinoxalin-2-amine. **y**ellow solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (s, 1H), 7.91 (d, *J* = 8.2 Hz, 1H), 7.67 (d, *J* = 8.3 Hz, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.39 (t, *J* = 7.8 Hz, 4H), 7.27 (d, *J* = 8.4 Hz, 4H), 7.23 (d, *J* = 7.1 Hz, 2H). Data is consistent with that reported in the literature. ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.19, 144.22, 140.93, 140.09, 137.94, 129.89, 129.48, 128.44, 127.13, 126.26, 126.01, 125.55. HRMS (ESI/ $[M+H]^+$) Calcd for $C_{20}H_{15}N_3$: 298.1344. Found: 298.1346.

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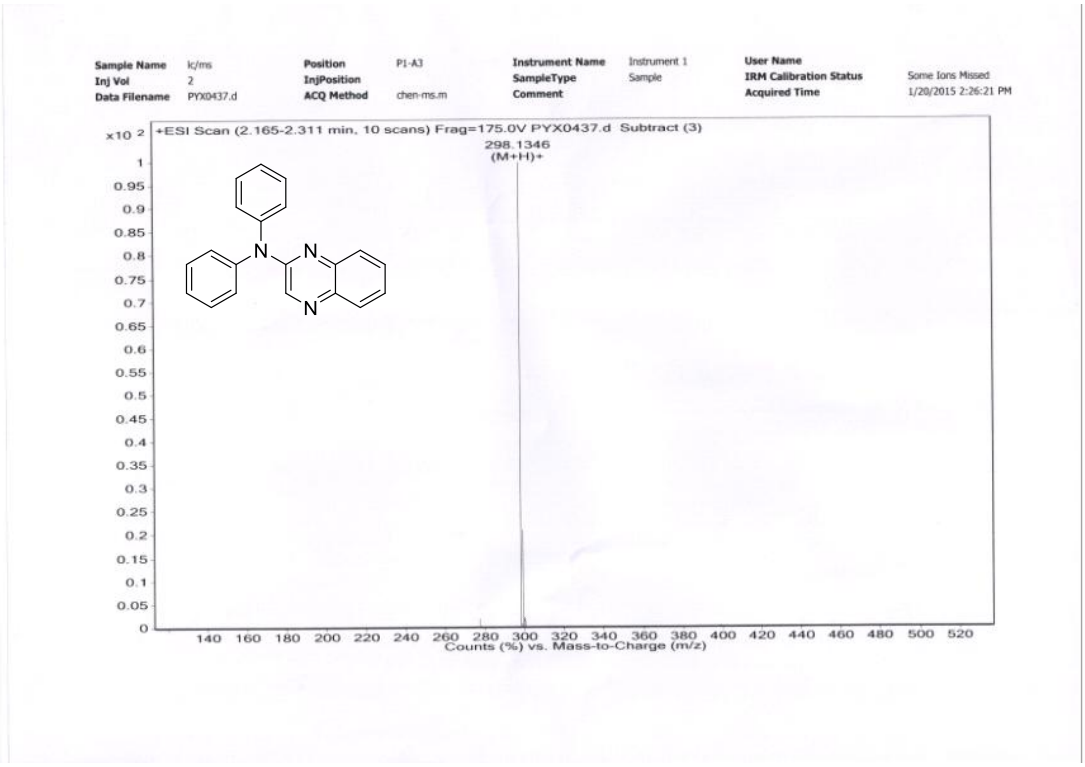
f1 (ppm)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Comment
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Acquired Time 1/20/2015 2:26:21 PM
+ESI Scan (2.165-2.311 min, 10 scans) Frag=175.0V PYX0437.d Subtract (3)
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(M+H)+
x10 2
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0.85
0.8
0.75
0.7
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Counts (%) vs. Mass-to-Charge (m/z)
N
N
N

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