Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Modular synthesis of unsymmetrical 2,4-diaryl substituted pyridines through four-component cyclization strategy under metal-free conditions

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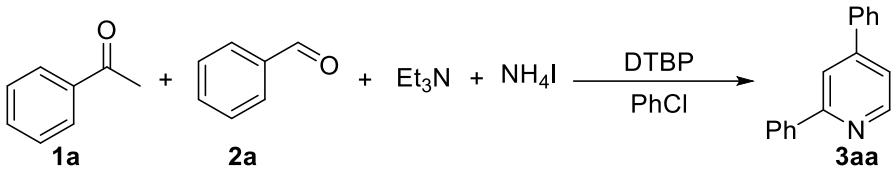
1. General information

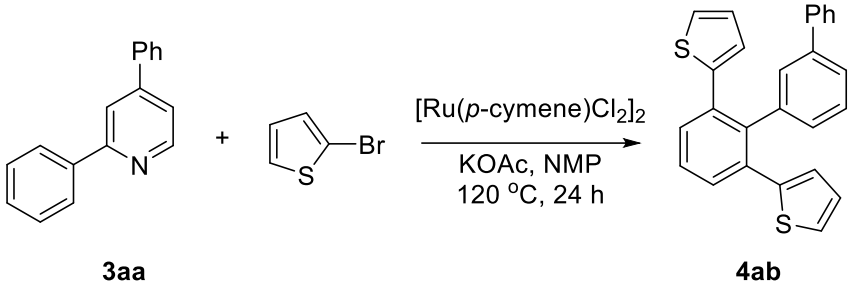
All reactions were carried out under an atmosphere of oxygen unless otherwise noted. Column chromatography was performed using silica gel (200-300 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker-AV (400 and 100 MHz, respectively) instrument using $CDCl_3$ as solvent and TMS as an internal standard. Mass spectra were measured on Agilent 5975 GC-MS instrument (EI). High-resolution mass spectra (HRMS) was performed on Agilent 6230 TOF LC/MS. The structures of known compounds were further corroborated by comparing their ^{1}H NMR, ^{13}C NMR data and MS data with those of literature. Most reagents were obtained from commercial suppliers and used without further purification.

2. General procedure



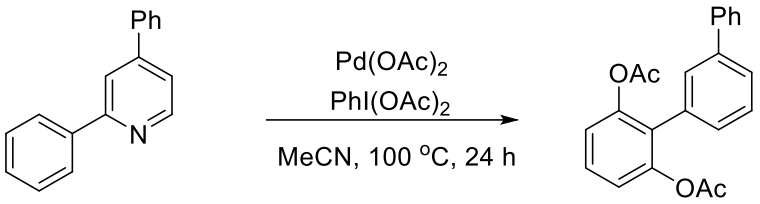
A 10 mL reaction vessel was charged with acetophenone (**1a**, 0.2 mmol), benzaldehyde (**2a**, 0.4 mmol), Et_3N (0.6 mmol), NH_4I (0.6 mmol), DTBP (0.4 mmol), PhCl (2.0 mL) under air. The reaction vessel was stirred at 150 ^{o}C for 24 h. After cooling to room temperature, the combined organic layer was dried over magnesium sulfate and the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 40:1) to give the desired product **3aa** as yellow oil, 30.0 mg, yield 65%.

3. Synthetic Transformation of Product 3aa



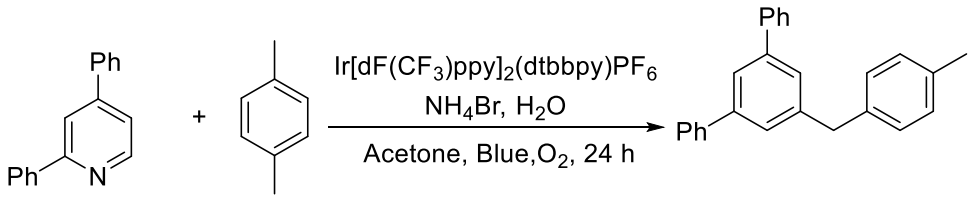
A 10 mL reaction vessel was charged with acetophenone 2,4-diphenylpyridine (0.2 mmol), 2-bromothiophene (2.5 equiv), [Ru(*p*-cymene)Cl_2]$_2$ (2.5 mol %), KOAc (3 equiv), NMP (0.6 mL). The reaction vessel was stirred at 120 ^{o}C for 24 h. After cooling to room temperature, the mixture was diluted with water (30 mL) and then extracted by EtOAc (10 mL×3). The combined organic phases were dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo and the crude product was obtained. Purified directly by flash column chromatography

(on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (59 mg, 75% yield).



3aa **4ac**

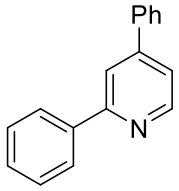
A 10 mL reaction vessel was charged with acetophenone 2,4-diphenylpyridine (0.2 mmol), *p*-xylene (2 mmol, 10 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (2 mol%), NH_4Br (0.5 equiv.), H_2O (10 equiv.), acetone (3 mL), at 35 °C under O_2 atmosphere (sealed tube) and 35 W blue LED irradiation for 20 h. After the reaction was complete, the mixture was diluted with water (30 mL) and then extracted by EtOAc (10 mL×3). The combined organic phases were dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo and the crude product was obtained. Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a colorless oil (46 mg, 67% yield).



3aa **4ad**

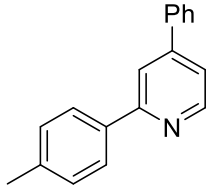
A 10 mL reaction vessel was charged with acetophenone 2,4-diphenylpyridine (0.2 mmol), *p*-xylene (2 mmol, 10 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (2 mol%), NH_4Br (0.5 equiv.), H_2O (10 equiv.), acetone (3 mL), at 35 °C under O_2 atmosphere (sealed tube) and 35 W blue LED irradiation for 20 h. After the reaction was complete, the mixture was diluted with water (30 mL) and then extracted by EtOAc (10 mL×3). The combined organic phases were dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo and the crude product was obtained. Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a colorless oil (41 mg, 61% yield).

4. Analytical data for the compounds prepared



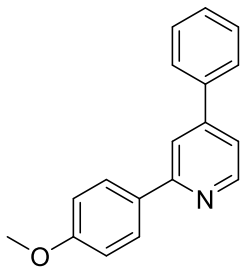
2,4-Diphenylpyridine (3aa, CAS: 26274-35-1)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (30.0 mg, 65% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, *J* = 5.1 Hz, 1H), 8.10 – 8.02 (m, 2H), 7.93 (d, *J* = 1.6 Hz, 1H), 7.70 (d, *J* = 7.3 Hz, 2H), 7.54 – 7.43 (m, 7H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.1, 150.1, 149.3, 139.5, 138.5, 129.1, 129.0, 129.0, 128.7, 127.1, 127.0, 120.2, 118.8.



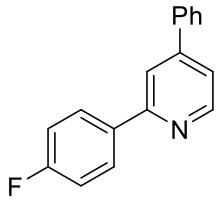
4-Phenyl-2-(*p*-tolyl)pyridine (3ba)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (32.8 mg, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71 (d, *J* = 5.1 Hz, 1H), 7.97 – 7.89 (m, 3H), 7.72 – 7.66 (m, 2H), 7.53 – 7.45 (m, 3H), 7.43 – 7.40 (m, 1H), 7.30 (d, *J* = 7.9 Hz, 2H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 150.0, 149.2, 139.0, 138.6, 136.6, 129.5, 129.1, 129.0, 127.0, 126.8, 119.9, 118.4, 21.3.



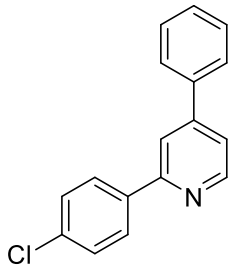
2-(4-Methoxyphenyl)-4-phenylpyridine (3ca)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 10:1) to give a yellow solid (33.4 mg, 64% yield). Mp: 60-62 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (d, *J* = 5.2 Hz, 1H), 8.05 – 7.98 (m, 2H), 7.87 (d, *J* = 1.7 Hz, 1H), 7.72 – 7.67 (m, 2H), 7.53 – 7.45 (m, 3H), 7.41 – 7.38 (m, 1H), 7.06 – 7.00 (m, 2H), 3.87 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.5, 157.7, 149.9, 149.2, 138.7, 132.1, 129.1, 128.9, 128.3, 127.0, 119.6, 118.0, 114.1, 55.3.



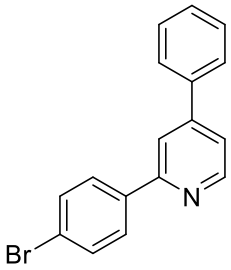
2-(4-Fluorophenyl)-4-phenylpyridine (3da)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (28.4 mg, 57% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71 (d, *J* = 5.1 Hz, 1H), 8.09 – 8.00 (m, 2H), 7.87 (s, 1H), 7.68 (d, *J* = 7.1 Hz, 2H), 7.53 – 7.41 (m, 4H), 7.17 (t, *J* = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5 (d, *J* = 246.9 Hz), 156.9, 150.0, 149.3, 138.3, 135.5 (d, *J* = 3.1 Hz), 129.1, 129.0, 128.8 (d, *J* = 8.3 Hz), 127.0, 120.1, 118.3, 115.6 (d, *J* = 21.5 Hz).



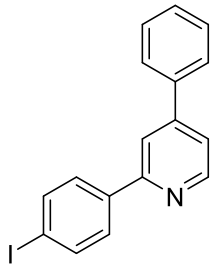
2-(4-Chlorophenyl)-4-phenylpyridine (3ea)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (27.3 mg, 51% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (d, *J* = 5.2 Hz, 1H), 8.00 (d, *J* = 8.1 Hz, 2H), 7.89 (s, 1H), 7.68 (d, *J* = 7.4 Hz, 2H), 7.54 – 7.44 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.8, 150.1, 149.4, 138.3, 137.8, 135.1, 129.1, 128.9, 128.3, 127.0, 120.5, 118.5.



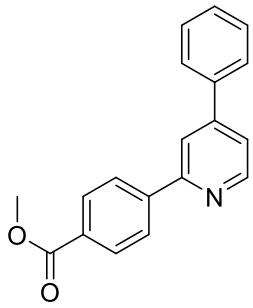
2-(4-Bromophenyl)-4-phenylpyridine (4fa)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow solid (26.6 mg, 43% yield). Mp: 80-82 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, *J* = 5.0 Hz, 1H), 7.97 – 7.89 (m, 3H), 7.74 – 7.60 (m, 4H), 7.56 – 7.43 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.8, 150.1, 149.6, 138.3, 138.3, 131.9, 129.2, 129.2, 128.6, 127.1, 123.6, 120.6, 118.5.



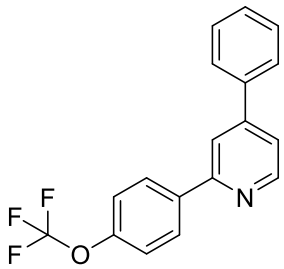
2-(4-Iodophenyl)-4-phenylpyridine (3ga)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow solid (25.7 mg, 36% yield). Mp: 88-90 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, *J* = 4.7 Hz, 1H), 7.94 – 7.78 (m, 5H), 7.69 (d, *J* = 7.2 Hz, 2H), 7.59 – 7.40 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.9, 150.2, 149.5, 138.9, 138.3, 137.9, 129.1, 128.7, 127.1, 120.6, 118.4, 95.4.



Methyl 4-(4-phenylpyridin-2-yl)benzoate (3ha)

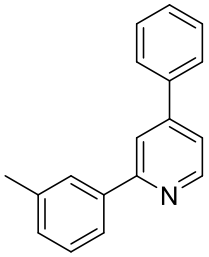
Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 10:1) to give a yellow solid (30.6 mg, 53% yield). Mp: 118-120 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.77 (d, *J* = 5.1 Hz, 1H), 8.19 – 8.10 (m, 4H), 7.98 (d, *J* = 1.7 Hz, 1H), 7.74 – 7.68 (m, 2H), 7.56 – 7.47 (m, 4H), 3.96 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9, 156.7, 150.2, 149.6, 143.5, 138.2, 130.4, 130.1, 129.2, 129.2, 127.1, 127.0, 121.0, 119.2, 52.2. HRMS (ESI) m/z calcd. for $C_{19}H_{16}NO_2^+$ $(M+H)^+$ 290.1176, found 290.1185.



4-Phenyl-2-(4-(trifluoromethoxy)phenyl)pyridine (3ia)

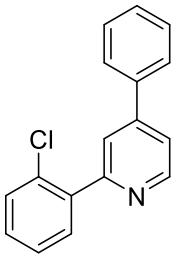
Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (29.6 mg, 47% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, *J* = 5.1 Hz, 1H), 8.11 – 8.04 (m, 2H), 7.90 (d, *J*

= 1.7 Hz, 1H), 7.72 – 7.66 (m, 2H), 7.54 – 7.46 (m, 4H), 7.34 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 150.2, 149.9, 149.5, 138.3, 138.1, 129.2, 128.7, 128.5, 127.0, 121.1, 120.6, 118.6. HRMS (ESI) m/z calcd. for $C_{18}H_{13}F_3NO^+$ $(M+H)^+$ 316.0944, found 316.0948.



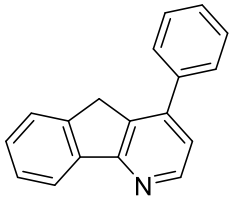
4-Phenyl-2-(*m*-tolyl)pyridine (3ja)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (26.4 mg, 54% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, *J* = 5.1 Hz, 1H), 7.95 – 7.87 (m, 2H), 7.82 (d, *J* = 7.8 Hz, 1H), 7.72 – 7.65 (m, 2H), 7.53 – 7.43 (m, 4H), 7.39 (t, *J* = 7.6 Hz, 1H), 7.27 – 7.24 (m, 1H), 2.46 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.2, 150.0, 149.2, 139.4, 138.5, 138.4, 129.8, 129.1, 129.0, 128.6, 127.7, 127.1, 124.1, 120.2, 118.8, 21.5. HRMS (ESI) m/z calcd. for $C_{18}H_{16}N^+$ $(M+H)^+$ 246.1277, found 246.1280.



2-(2-Chlorophenyl)-4-phenylpyridine (3ka)[1]

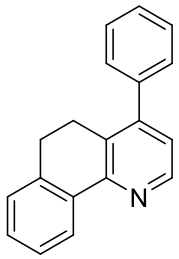
Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow solid (15.4 mg, 29% yield). Mp: 58-60 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.77 (d, *J* = 5.1 Hz, 1H), 7.88 (s, 1H), 7.73 – 7.63 (m, 3H), 7.55 – 7.42 (m, 5H), 7.41 – 7.33 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3, 149.9, 148.3, 139.2, 138.1, 132.2, 131.5, 130.1, 129.6, 129.1, 129.1, 127.1, 127.0, 122.9, 120.4.



4-Phenyl-5*H*-indeno[1,2-*b*]pyridine (3ma)

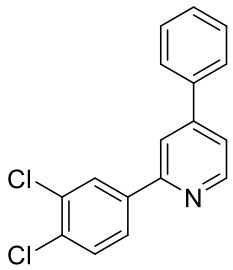
Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow solid (30.6 mg, 63% yield). Mp: 93-95 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 (d, *J* = 5.2 Hz, 1H), 8.16 (d, *J* =

7.5 Hz, 1H), 7.65 – 7.59 (m, 2H), 7.57 – 7.50 (m, 3H), 7.48 – 7.40 (m, 3H), 7.27 – 7.23 (m, 1H), 3.99 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.8, 148.7, 146.0, 143.7, 140.7, 138.4, 134.3, 128.8, 128.8, 128.6, 128.2, 127.3, 125.0, 121.1, 120.9, 34.5. HRMS (ESI) m/z calcd. for $C_{18}H_{14}NO^+$ $(M+H)^+$ 244.1121, found 244.1125.



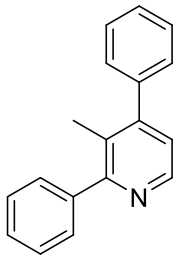
4-Phenyl-5,6-dihydrobenzo[*h*]quinoline (3na)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a white solid (32.3 mg, 62% yield). Mp: 100-102 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (d, *J* = 4.9 Hz, 1H), 8.37 (d, *J* = 7.5 Hz, 1H), 7.51 – 7.30 (m, 7H), 7.26 – 7.20 (m, 1H), 7.14 (d, *J* = 4.9 Hz, 1H), 2.94 – 2.89 (m, 2H), 2.85 – 2.81 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.0, 148.6, 147.1, 138.9, 138.1, 134.8, 129.5, 129.1, 128.7, 128.4, 128.0, 127.5, 127.1, 125.5, 123.3, 28.0, 25.4.



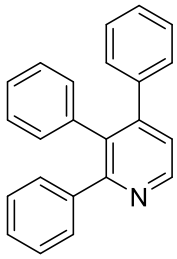
2-(3,4-Dichlorophenyl)-4-phenylpyridine (3oa)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow solid (22.8 mg, 38% yield). Mp: 96-98 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, *J* = 5.1 Hz, 1H), 8.18 (d, *J* = 2.1 Hz, 1H), 7.92 – 7.85 (m, 2H), 7.72 – 7.66 (m, 2H), 7.57 – 7.47 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.5, 150.2, 149.7, 139.3, 138.1, 133.2, 133.1, 130.7, 129.3, 129.2, 128.9, 127.1, 126.1, 120.9, 118.5.



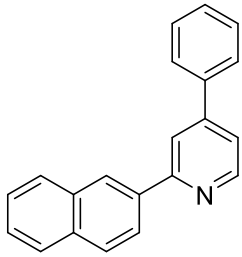
3-Methyl-2,4-diphenylpyridine (3pa)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (28.9 mg, 58% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (d, *J* = 4.8 Hz, 1H), 7.55 (d, *J* = 7.4 Hz, 2H), 7.49 – 7.35 (m, 8H), 7.14 (d, *J* = 4.8 Hz, 1H), 2.21 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 150.8, 146.3, 141.0, 139.8, 129.0, 128.6, 128.4, 128.3, 128.1, 127.8, 123.1, 17.93.



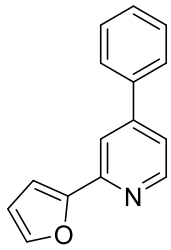
2,3,4-Triphenylpyridine (3qa)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a white solid (31.9 mg, 52% yield). Mp: 174-176 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, *J* = 4.7 Hz, 1H), 7.35 (d, *J* = 4.8 Hz, 1H), 7.30 – 7.24 (m, 3H), 7.23 – 7.17 (m, 5H), 7.14 – 6.97 (m, 5H), 6.88 (d, *J* = 6.1 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.4, 149.9, 148.2, 140.7, 139.4, 137.7, 134.4, 131.3, 129.8, 129.3, 127.9, 127.7, 127.6, 127.3, 127.3, 126.6, 123.6.



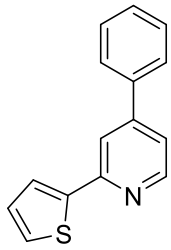
2-(Naphthalen-2-yl)-4-phenylpyridine (3ra)[2]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (31.4 mg, 56% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.79 (d, *J* = 5.1 Hz, 1H), 8.54 (d, *J* = 1.8 Hz, 1H), 8.24 – 8.18 (m, 1H), 8.07 (d, *J* = 1.6 Hz, 1H), 7.97 (d, *J* = 8.8 Hz, 2H), 7.90 – 7.86 (m, 1H), 7.76 – 7.71 (m, 2H), 7.55 – 7.46 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 150.2, 149.4, 138.5, 136.7, 133.6, 133.5, 129.1, 129.1, 128.7, 128.5, 127.6, 127.1, 126.5, 126.4, 126.3, 124.6, 120.3, 119.0.



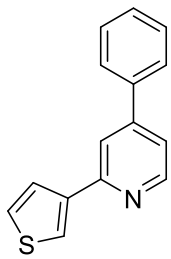
2-(Furan-2-yl)-4-phenylpyridine (3sa)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (24.3mg, 55% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, *J* = 5.1 Hz, 1H), 7.93 (s, 1H), 7.70 (d, *J* = 7.2 Hz, 2H), 7.58 – 7.43 (m, 4H), 7.39 (d, *J* = 4.1 Hz, 1H), 7.14 (d, *J* = 3.0 Hz, 1H), 6.57 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.5, 149.9, 149.7, 149.3, 143.4, 138.1, 129.2, 129.1, 127.0, 119.9, 116.6, 112.1, 109.0.



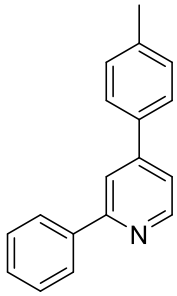
4-Phenyl-2-(thiophen-2-yl)pyridine (3ta)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (27.5 mg, 58% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (d, *J* = 5.0 Hz, 1H), 7.85 (s, 1H), 7.66 (d, *J* = 4.7 Hz, 3H), 7.54 – 7.39 (m, 4H), 7.35 (d, *J* = 4.8 Hz, 1H), 7.17 – 7.08 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.9, 149.8, 149.1, 144.7, 138.0, 129.0, 129.0, 127.9, 127.5, 126.8, 124.5, 119.9, 116.7.



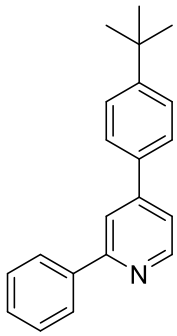
4-Phenyl-2-(thiophen-3-yl)pyridine (3ua)[2]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a black oil (26.5 mg, 56% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 (d, *J* = 5.1 Hz, 1H), 8.00 – 7.95 (m, 1H), 7.82 (d, *J* = 1.7 Hz, 1H), 7.74 – 7.66 (m, 3H), 7.53 – 7.46 (m, 3H), 7.43 – 7.38 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.9, 149.9, 149.3, 142.1, 138.4, 129.1, 129.1, 127.0, 126.4, 126.2, 123.7, 120.0, 118.4.



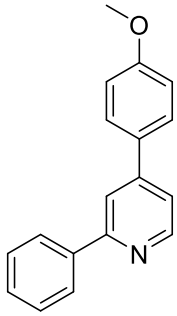
2-Phenyl-4-(*p*-tolyl)pyridine (3ab)[3]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (31.4 mg, 64% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (d, *J* = 5.2 Hz, 1H), 8.04 (d, *J* = 7.6 Hz, 2H), 7.92 (d, *J* = 1.7 Hz, 1H), 7.60 (d, *J* = 7.9 Hz, 2H), 7.53 – 7.42 (m, 4H), 7.32 (d, *J* = 7.8 Hz, 2H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 150.0, 149.1, 139.5, 139.2, 135.5, 129.8, 129.0, 128.7, 127.0, 126.9, 120.0, 118.5, 21.2.



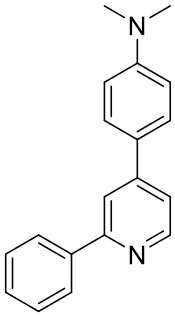
4-(4-(*tert*-Butyl)phenyl)-2-phenylpyridine (3ac)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a white solid (39.0 mg, 68% yield). Mp: 130-132 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (d, *J* = 5.1 Hz, 1H), 8.07 – 8.00 (m, 2H), 7.94 – 7.90 (m, 1H), 7.65 (d, *J* = 8.2 Hz, 2H), 7.55 – 7.43 (m, 6H), 1.38 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 152.3, 150.0, 149.1, 139.5, 135.5, 128.9, 128.7, 127.0, 126.7, 126.1, 120.1, 118.6, 34.7, 31.2. HRMS (ESI) m/z calcd. for $C_{21}H_{22}N^+$ $(M+H)^+$ 288.1747, found 288.1755.



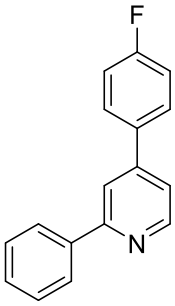
4-(4-Methoxyphenyl)-2-phenylpyridine (3ad)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 10:1) to give a yellow solid (28.7 mg, 55% yield). Mp: 73-75 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71 (d, *J* = 4.8 Hz, 1H), 8.04 (d, *J* = 7.1 Hz, 2H), 7.90 (s, 1H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.58 – 7.37 (m, 4H), 7.04 (d, *J* = 8.3 Hz, 2H), 3.88 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.5, 157.9, 149.9, 148.7, 139.5, 130.6, 128.9, 128.7, 128.2, 127.0, 119.6, 118.2, 114.5, 55.3.



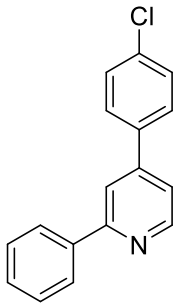
N,*N*-Dimethyl-4-(2-phenylpyridin-4-yl)aniline (3ae)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 10:1) to give a yellow solid (18.6 mg, 34% yield). Mp: 112-114 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 (d, *J* = 5.3 Hz, 1H), 8.07 – 8.01 (m, 2H), 7.90 (d, *J* = 1.8 Hz, 1H), 7.67 – 7.62 (m, 2H), 7.52 – 7.48 (m, 2H), 7.45 – 7.41 (m, 2H), 6.84 – 6.80 (m, 2H), 3.04 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 151.1, 149.6, 149.2, 139.6, 128.9, 128.7, 127.7, 127.0, 125.2, 119.0, 117.5, 112.4, 40.3. HRMS (ESI) m/z calcd. for $C_{19}H_{19}N_2^+$ $(M+H)^+$ 275.1543, found 275.1548.



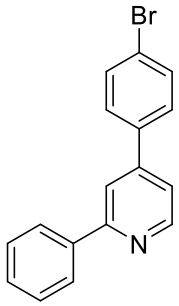
4-(4-Fluorophenyl)-2-phenylpyridine (3af)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (28.3 mg, 57% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, *J* = 5.0 Hz, 1H), 8.04 (d, *J* = 7.2 Hz, 2H), 7.88 (s, 1H), 7.71 – 7.63 (m, 2H), 7.53 – 7.38 (m, 4H), 7.28 – 7.12 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.3 (d, *J* = 247.6 Hz), 158.0, 150.0, 148.0, 139.2, 134.4 (d, *J* = 2.8 Hz), 129.0, 128.7 (d, *J* = 7.9 Hz), 128.6, 126.9, 119.9, 118.3, 116.0 (d, *J* = 21.5 Hz).



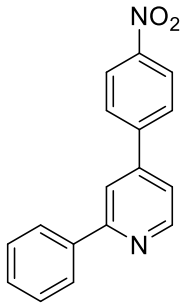
4-(4-Chlorophenyl)-2-phenylpyridine (3ag)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (28.0 mg, 53% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, *J* = 5.1 Hz, 1H), 8.03 (d, *J* = 7.6 Hz, 2H), 7.88 (s, 1H), 7.62 (d, *J* = 8.2 Hz, 2H), 7.52 – 7.44 (m, 5H), 7.42 – 7.39 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.2, 150.2, 148.0, 139.2, 136.9, 135.2, 129.3, 129.1, 128.8, 128.3, 127.0, 120.0, 118.5. HRMS (ESI) m/z calcd. for $C_{17}H_{13}ClNO^+$ $(M+H)^+$ 266.0731, found 266.0739.



4-(4-Bromophenyl)-2-phenylpyridine (3ah)

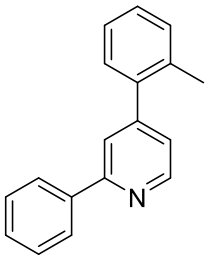
Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (27.2 mg, 44% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, *J* = 5.2 Hz, 1H), 8.03 (d, *J* = 7.6 Hz, 2H), 7.88 (s, 1H), 7.64 (d, *J* = 8.2 Hz, 2H), 7.57 – 7.44 (m, 5H), 7.42 – 7.39 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.2, 150.2, 148.1, 139.2, 137.4, 132.3, 129.1, 128.8, 128.6, 127.0, 123.5, 119.9, 118.4. HRMS (ESI) m/z calcd. for $C_{17}H_{13}BrN^+$ $(M+H)^+$ 310.0226, found 310.0236.



4-(4-Nitrophenyl)-2-phenylpyridine (3ai)[1]

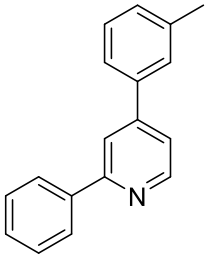
Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 10:1) to give a yellow

solid (13.8 mg, 25% yield). Mp: 132-134 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.83 (d, *J* = 5.2 Hz, 1H), 8.38 (d, *J* = 8.5 Hz, 2H), 8.06 (d, *J* = 7.4 Hz, 2H), 7.94 (s, 1H), 7.85 (d, *J* = 8.6 Hz, 2H), 7.57 – 7.45 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.5, 150.4, 148.2, 146.9, 144.8, 138.8, 129.4, 128.8, 128.0, 127.0, 124.3, 120.1, 118.7.



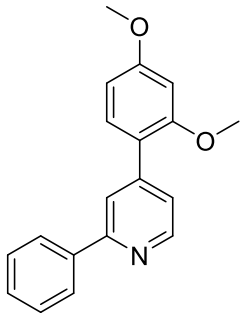
2-Phenyl-4-(*o*-tolyl)pyridine (4aj)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (26.4 mg, 54% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, *J* = 5.0 Hz, 1H), 8.04 – 7.99 (m, 2H), 7.70 (d, *J* = 1.6 Hz, 1H), 7.51 – 7.41 (m, 3H), 7.33 – 7.20 (m, 5H), 2.33 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3, 150.6, 149.4, 139.3, 139.3, 135.0, 130.6, 129.2, 129.0, 128.7, 128.4, 127.0, 126.1, 122.7, 121.2, 20.3. HRMS (ESI) m/z calcd. for $C_{18}H_{16}N^+$ $(M+H)^+$ 246.1277, found 246.1280.



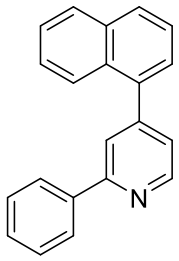
2-Phenyl-4-(*m*-tolyl)pyridine (4ak)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (27.9 mg, 57% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, *J* = 5.1 Hz, 1H), 8.08 – 8.02 (m, 2H), 7.92 (d, *J* = 1.7 Hz, 1H), 7.49 (d, *J* = 7.4 Hz, 4H), 7.45 – 7.38 (m, 3H), 7.30 – 7.24 (m, 1H), 2.46 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 149.9, 149.5, 139.4, 138.8, 138.4, 129.8, 129.0, 128.7, 127.8, 127.0, 124.2, 120.3, 118.8, 21.51. HRMS (ESI) m/z calcd. for $C_{18}H_{16}N^+$ $(M+H)^+$ 246.1277, found 246.1279



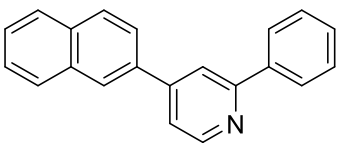
4-(2,4-Dimethoxyphenyl)-2-phenylpyridine (3al)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 10:1) to give a yellow oil (20.9 mg, 36% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 (d, *J* = 5.2 Hz, 1H), 8.04 – 7.98 (m, 2H), 7.88 (s, 1H), 7.51 – 7.46 (m, 2H), 7.44 – 7.39 (m, 2H), 7.35 (d, *J* = 8.3 Hz, 1H), 6.63 – 6.58 (m, 2H), 3.87 (s, 3H), 3.84 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.4, 157.7, 157.2, 149.1, 147.0, 139.7, 131.1, 128.7, 128.7, 127.0, 122.7, 121.3, 120.6, 105.0, 99.0, 55.6, 55.5. HRMS (ESI) m/z calcd. for $C_{19}H_{18}NO_2^+$ $(M+H)^+$ 292.1332, found 292.1333.



4-(Naphthalen-1-yl)-2-phenylpyridine (3am)[4]

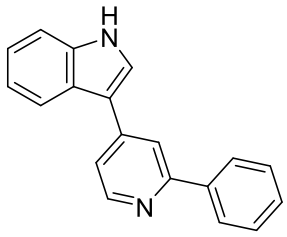
Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (27.0 mg, 48% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81 (d, *J* = 5.0 Hz, 1H), 8.10 – 8.03 (m, 2H), 7.95 – 7.87 (m, 4H), 7.59 – 7.52 (m, 2H), 7.51 – 7.42 (m, 5H), 7.39 – 7.37 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.5, 149.6, 149.5, 139.2, 137.6, 133.7, 130.8, 129.1, 128.8, 128.8, 128.5, 127.0, 126.8, 126.6, 126.2, 125.3, 125.2, 123.5, 122.0.



4-(Naphthalen-2-yl)-2-phenylpyridine (3an)[4]

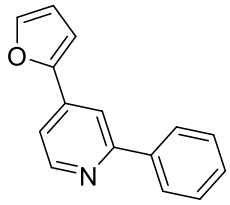
Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (28.7mg, 51% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.78 (d, *J* = 5.2 Hz, 1H), 8.17 (d, *J* = 1.8 Hz, 1H), 8.11 – 8.03 (m, 3H), 7.99 – 7.93 (m, 2H), 7.92 – 7.88 (m, 1H), 7.82 – 7.78 (m, 1H), 7.58 – 7.50 (m, 5H), 7.48 – 7.45 (m,

1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.1, 150.0, 149.3, 139.4, 135.7, 133.4, 133.4, 129.1, 128.9, 128.8, 128.4, 127.7, 127.1, 126.8, 126.7, 126.5, 124.6, 120.4, 119.0.



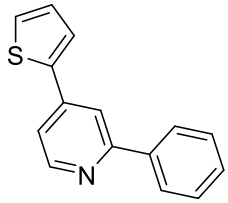
3-(2-Phenylpyridin-4-yl)-1*H*-indole (3ao)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a black solid (23.8 mg, 44% yield). Mp: 93-95 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.76 (s, 1H), 8.62 (d, J = 5.2 Hz, 1H), 8.26 – 8.09 (m, 4H), 8.07 – 7.99 (m, 1H), 7.74 – 7.68 (m, 1H), 7.54 – 7.41 (m, 4H), 7.23 – 7.15 (m, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 156.8, 150.2, 145.0, 139.5, 137.7, 129.4, 129.2, 127.2, 127.0, 125.1, 122.4, 121.0, 119.8, 117.3, 113.4, 112.8. HRMS (ESI) m/z calcd. for $C_{19}H_{14}N_2^+$ $(M+H)^+$ 271.1230, found 271.1228.



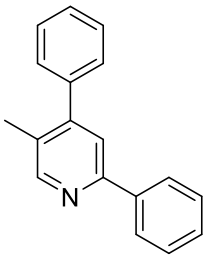
4-(Furan-2-yl)-2-phenylpyridine (3ap)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil. (20.8 mg, 47% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (d, J = 5.0 Hz, 1H), 8.04 (d, J = 7.3 Hz, 2H), 7.98 (s, 1H), 7.58 (s, 1H), 7.53 – 7.41 (m, 4H), 6.95 (d, J = 3.0 Hz, 1H), 6.56 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 151.5, 150.0, 143.7, 139.3, 138.3, 129.1, 128.7, 126.9, 116.3, 114.7, 112.1, 108.7.



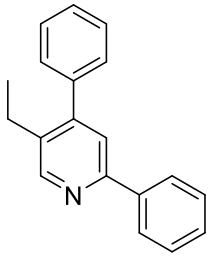
2-Phenyl-4-(thiophen-2-yl)pyridine (3aq)[5]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow solid (22.4 mg, 47% yield). Mp: 62-65 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 (d, J = 4.5 Hz, 1H), 8.02 (d, J = 7.4 Hz, 2H), 7.90 (s, 1H), 7.57 – 7.42 (m, 6H), 7.16 (d, J = 4.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.2, 150.2, 142.2, 141.4, 139.2, 129.1, 128.7, 128.4, 127.1, 127.0, 125.3, 118.4, 116.9.



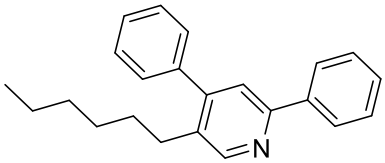
5-Methyl-2,4-diphenylpyridine (3ar)[1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (30.4 mg, 62% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (s, 1H), 8.02 – 7.98 (m, 2H), 7.60 (s, 1H), 7.48 – 7.44 (m, 4H), 7.43 – 7.36 (m, 4H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.2, 151.1, 149.9, 139.4, 139.3, 129.2, 128.7, 128.6, 128.5, 128.4, 127.9, 126.7, 121.0, 17.0.



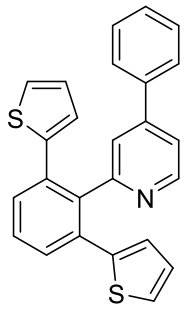
5-Ethyl-2,4-diphenylpyridine (3as)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (30.5 mg, 59% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (s, 1H), 8.03 – 7.97 (m, 2H), 7.57 (s, 1H), 7.48 – 7.35 (m, 8H), 2.68 (q, *J* = 7.6 Hz, 2H), 1.13 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.9, 150.3, 149.7, 139.4, 139.2, 135.3, 128.7, 128.6, 128.5, 128.4, 127.9, 126.7, 121.3, 23.3, 15.5. HRMS (ESI) m/z calcd. for $C_{19}H_{18}N^+$ $(M+H)^+$ 260.1434, found 260.1434.



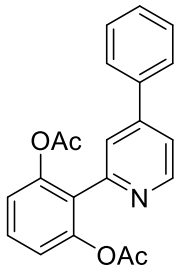
5-Hexyl-2,4-diphenylpyridine (3at)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (34.6 mg, 55% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (s, 1H), 8.00 (d, *J* = 7.7 Hz, 2H), 7.57 (s, 1H), 7.47 – 7.38 (m, 6H), 7.37 – 7.33 (m, 2H), 2.67 – 2.60 (m, 2H), 1.49 – 1.42 (m, 2H), 1.24 – 1.15 (m, 6H), 0.84 – 0.79 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.8, 150.7, 149.9, 139.5, 139.2, 134.0, 128.7, 128.6, 128.5, 128.4, 127.8, 126.7, 121.2, 31.3, 30.9, 29.9, 28.9, 22.4, 14.0. HRMS (ESI) m/z calcd. for $C_{23}H_{26}N^+$ $(M+H)^+$ 316.2060, found 316.2056.



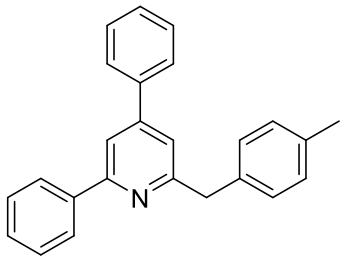
2-(2,6-di(thiophen-2-yl)phenyl)-4-phenylpyridine (4ab) [1]

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a yellow oil (59 mg, 75% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (d, *J* = 5.1 Hz, 1H), 7.59 (d, *J* = 7.7 Hz, 2H), 7.52 – 7.30 (m, 8H), 7.14 (d, *J* = 5.1 Hz, 2H), 6.84 – 6.80 (m, 2H), 6.72 (d, *J* = 3.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.9, 149.2, 148.0, 142.6, 138.5, 138.1, 134.7, 130.0, 129.0, 128.9, 128.40, 127.2, 126.9, 126.8, 125.8, 124.4, 120.1.



2-(4-phenylpyridin-2-yl)-1,3-phenylene diacetate (4ac)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 10:1) to give a yellow oil (46 mg, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, *J* = 5.1 Hz, 1H), 7.65 (d, *J* = 6.7 Hz, 2H), 7.58 (s, 1H), 7.55 – 7.39 (m, 5H), 7.13 (d, *J* = 8.2 Hz, 2H), 2.02 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 152.7, 149.9, 149.1, 148.2, 137.5, 129.3, 129.3, 129.21, 129.16, 127.6, 126.8, 122.8, 120.7, 120.3, 20.57. HRMS (ESI) m/z calcd. for $C_{21}H_{18}NO_4^+$ $(M+H)^+$ 348.1230, found 348.1236.



2-(4-methylbenzyl)-4,6-diphenylpyridine (4ad)

Purified directly by flash column chromatography (on silica gel, petroleum ether/EtOAc = 20:1) to give a colorless oil (41 mg, 61% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 7.3 Hz, 2H), 7.73 (s, 1H), 7.60 (d, *J* = 7.0 Hz, 2H), 7.53 – 7.39 (m, 6H), 7.28 (d, *J* = 7.7 Hz, 2H), 7.23 (s, 1H), 7.13 (d, *J* = 7.4 Hz, 2H). 4.25 (s, 2H), 2.32 (s,

3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.7, 157.5, 149.7, 139.7, 138.8, 136.6, 135.8, 129.2, 129.1, 128.9, 128.8, 128.8, 128.7, 127.12, 127.08, 119.5, 116.4, 44.5, 21.0. HRMS (ESI) m/z calcd. for $C_{25}H_{22}N^+$ $(M+H)^+$ 336.1747, found 336.1746.

5. References

[1] H. Huang, J. Cai, L. Tang, Z. Wang, F. Li, G.-J. Deng, *J. Org. Chem.*, 2016, **81**, 1499−1505.

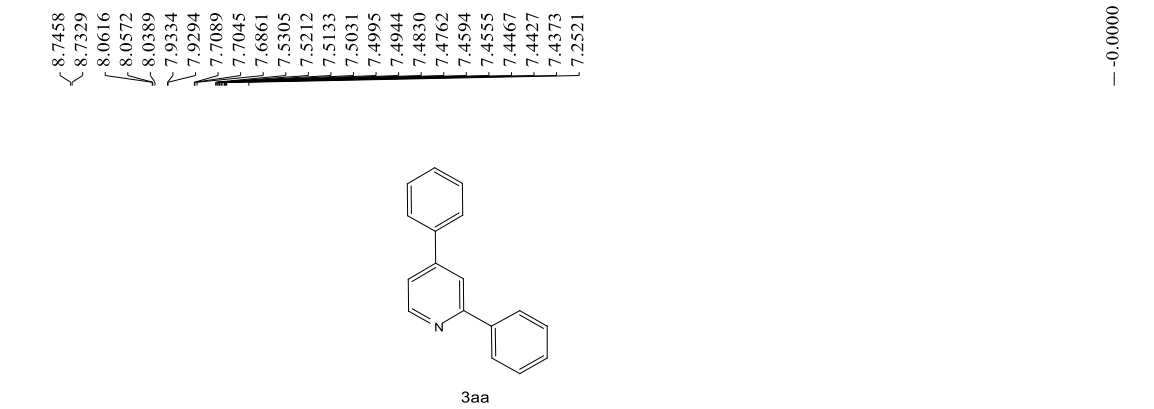
[2] Y. Wei, N. Yoshikai, *J. Am. Chem. Soc.*, 2013, **135**, 3756−3759.

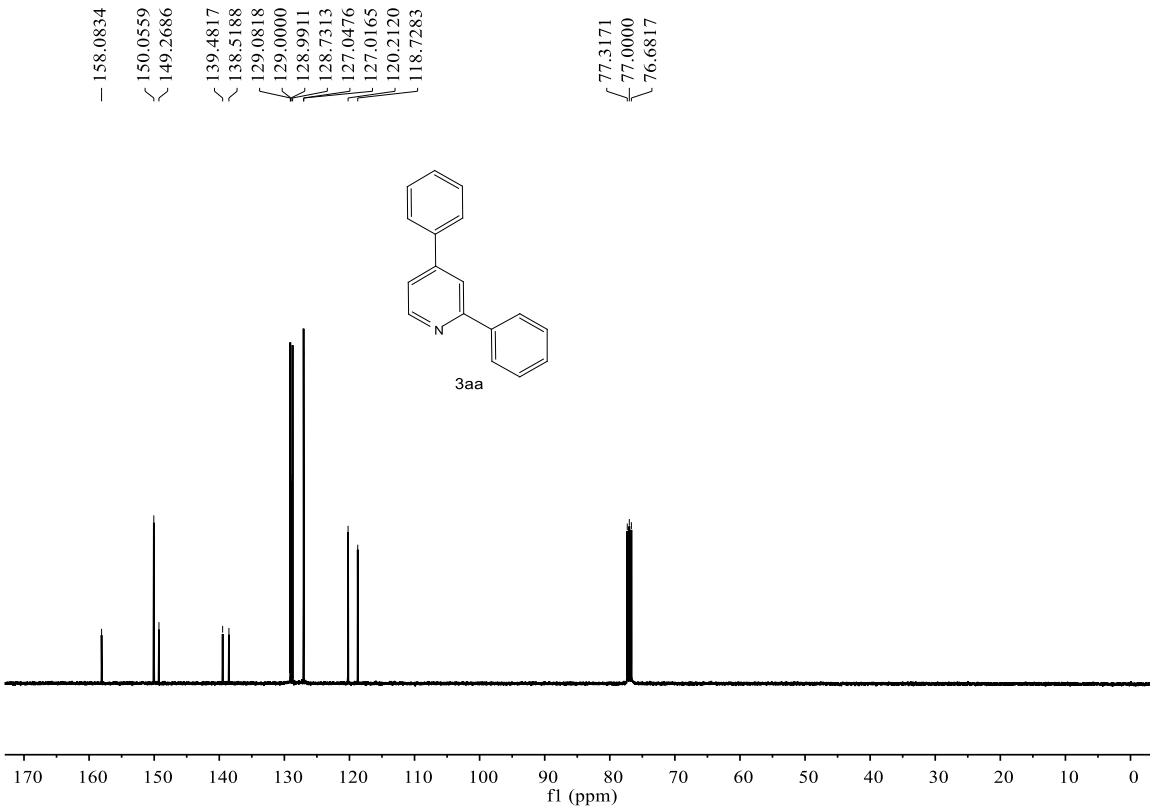
[3] Y.-Y. Chuaa, H. A. Duong, *Chem. Commun.*, 2014, **50**, 8424-8427.

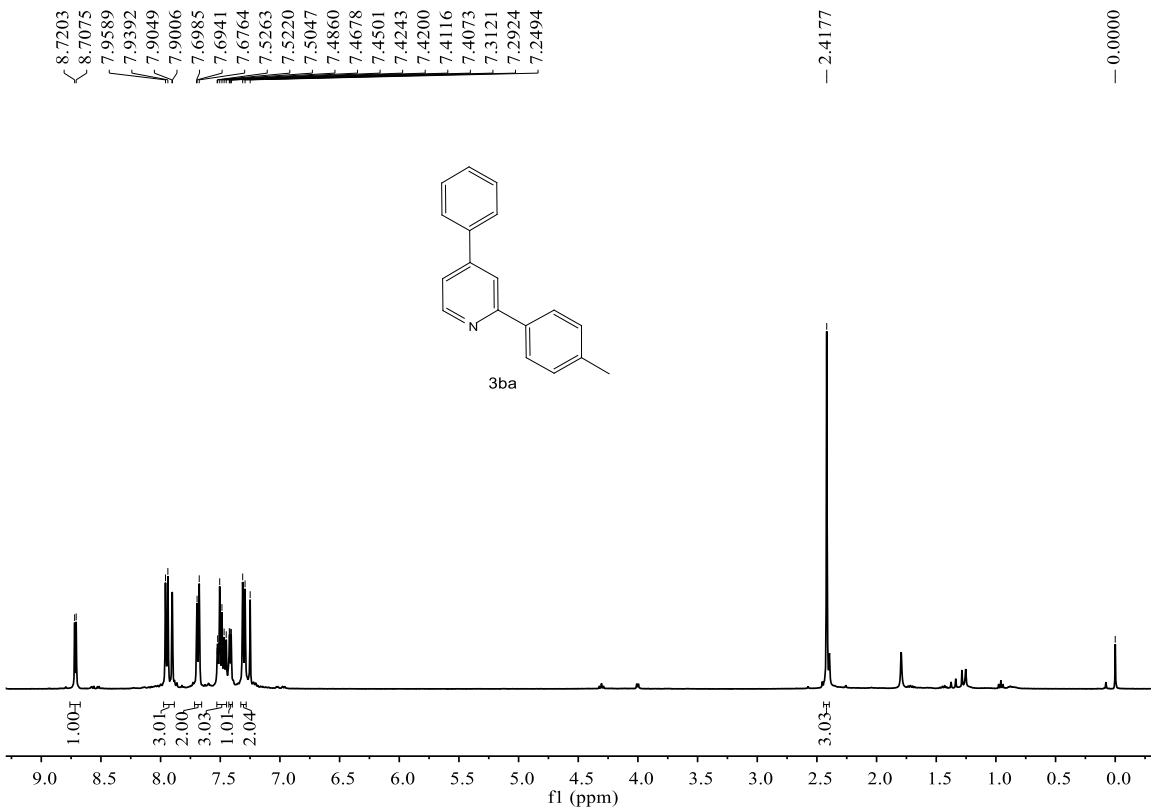
[4] R. Singha, S. Dhara, J. K. Ray, T*etrahedron Letters.*, 2013, **54**, 4841 – 4843.

[5] M. Guzel, J. Watts, M. McGilvary, M. Wright, S. Kiren, *Tetrahedron Letters* 2015, **56**, 5275 – 5277.

6. NMR Spectra for the compounds prepared



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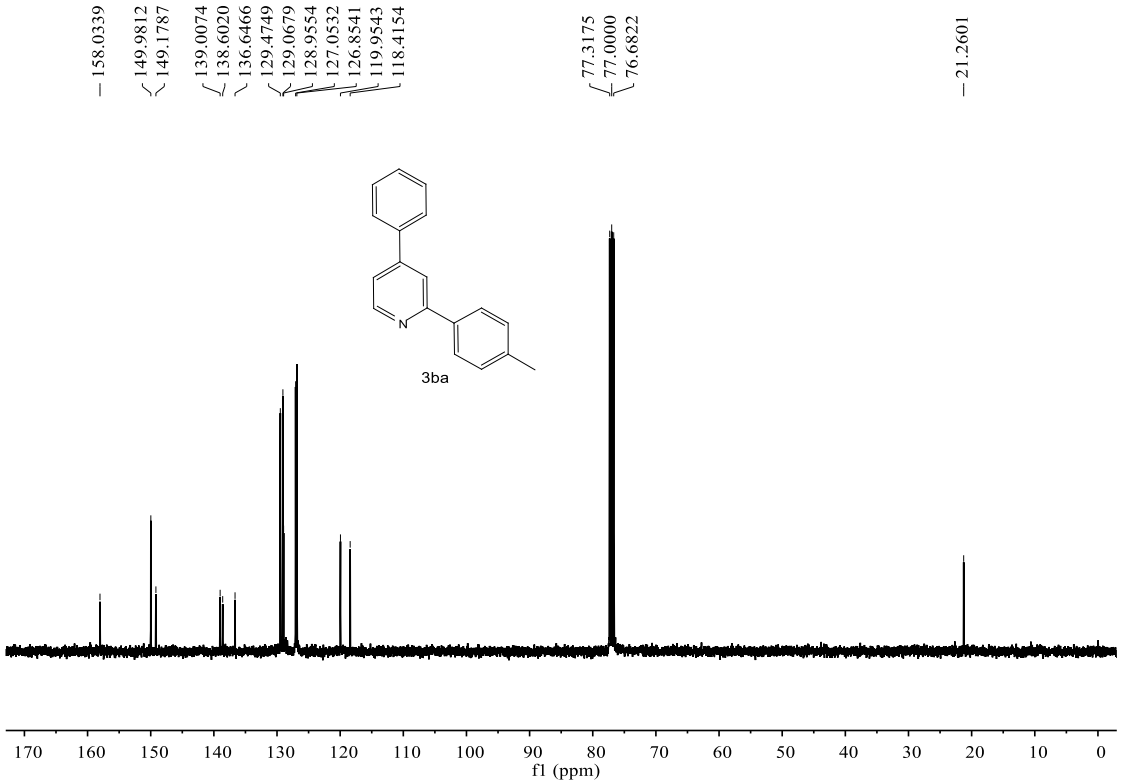
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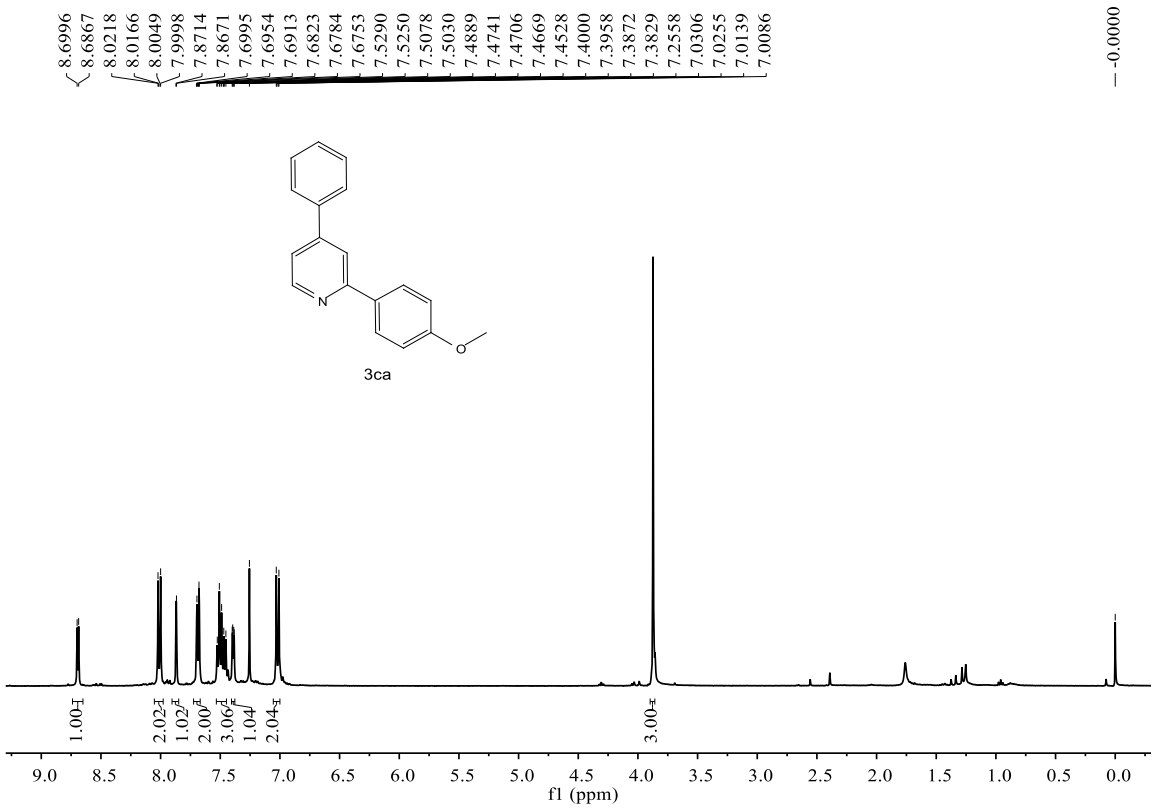
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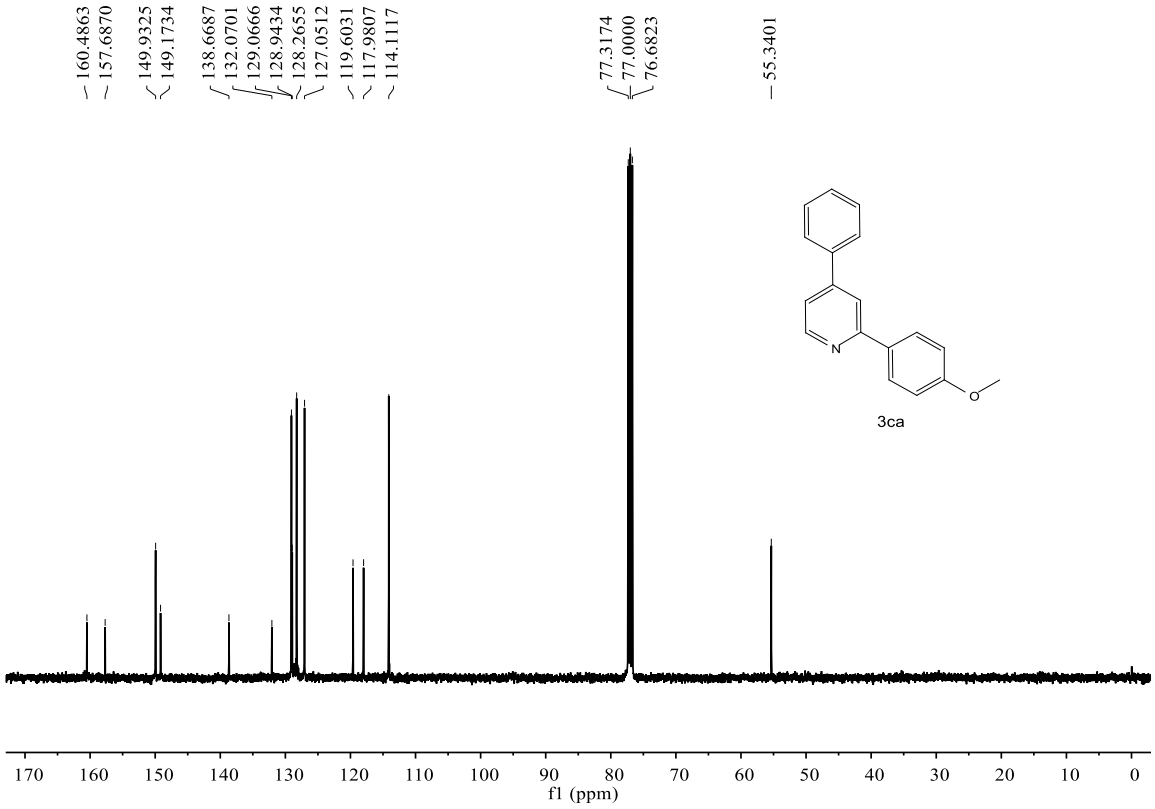
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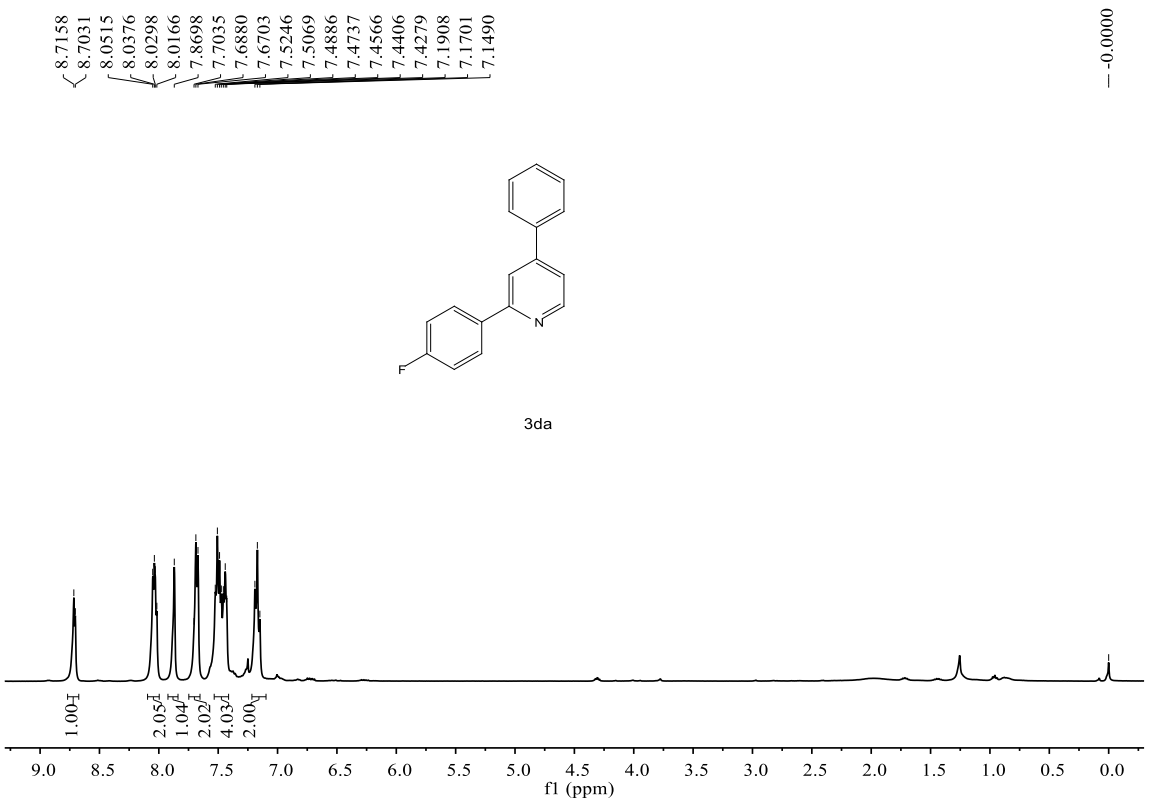
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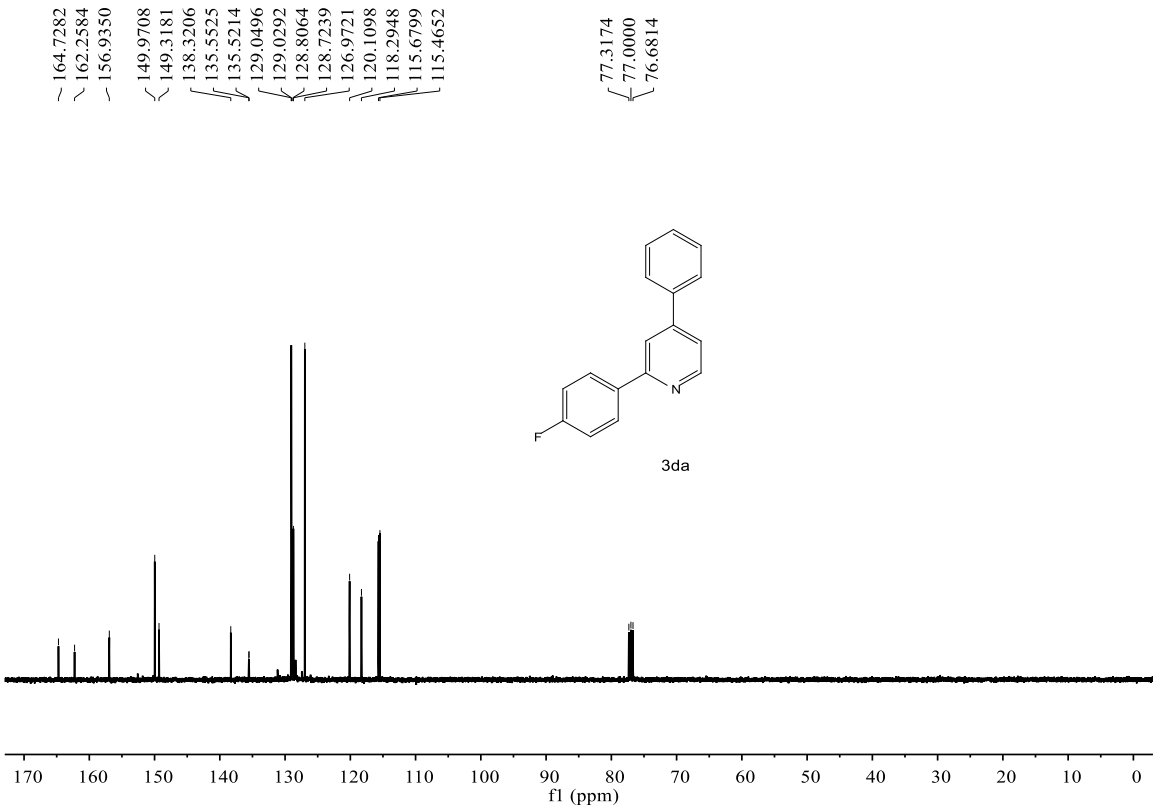
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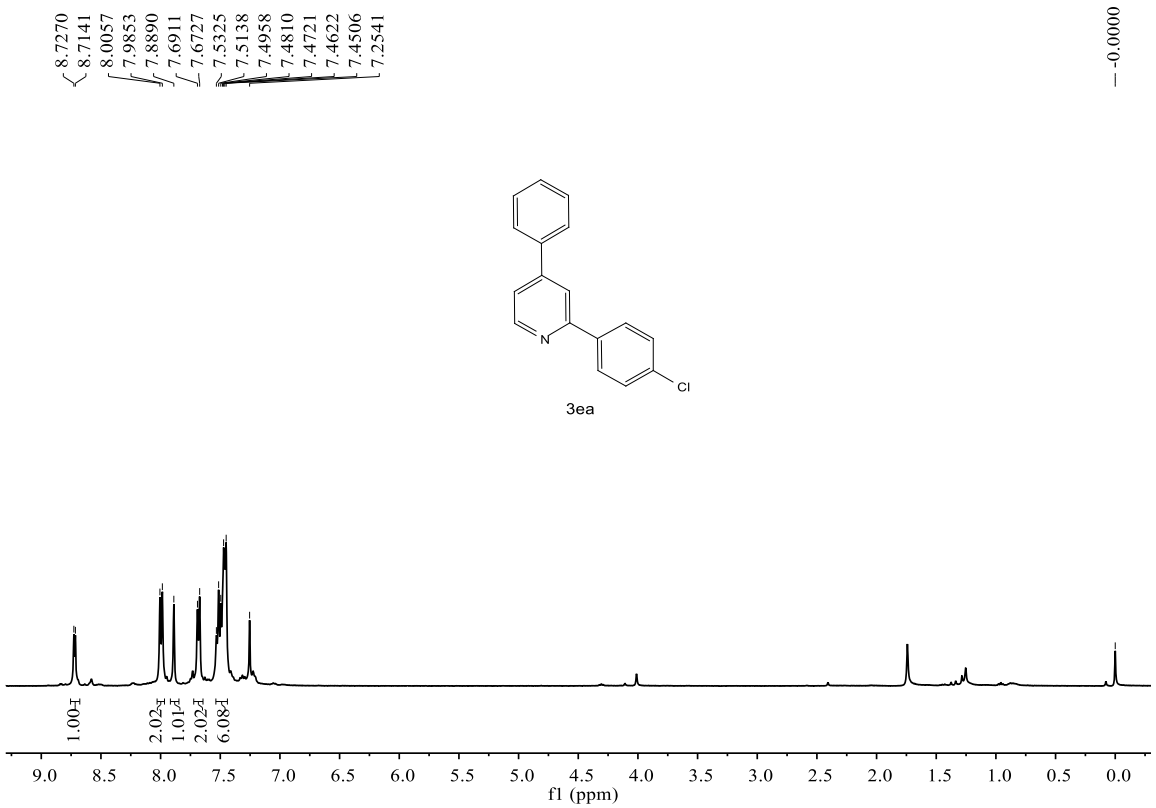
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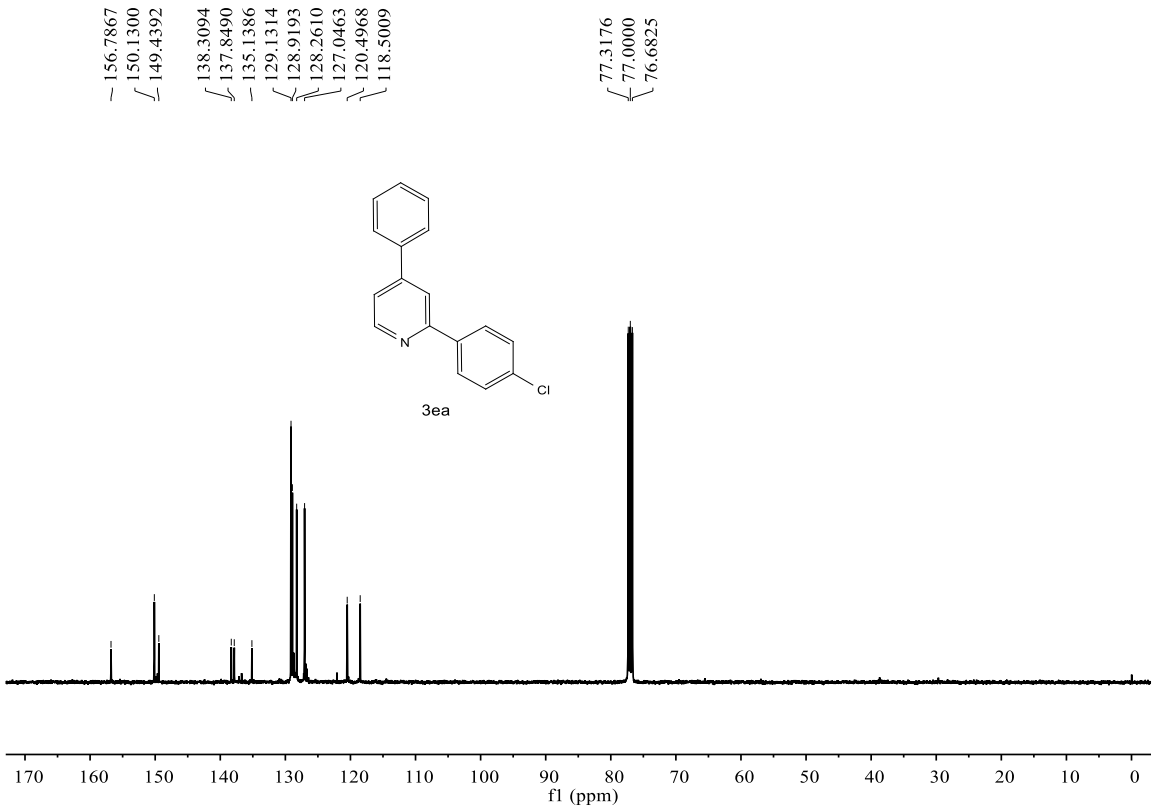
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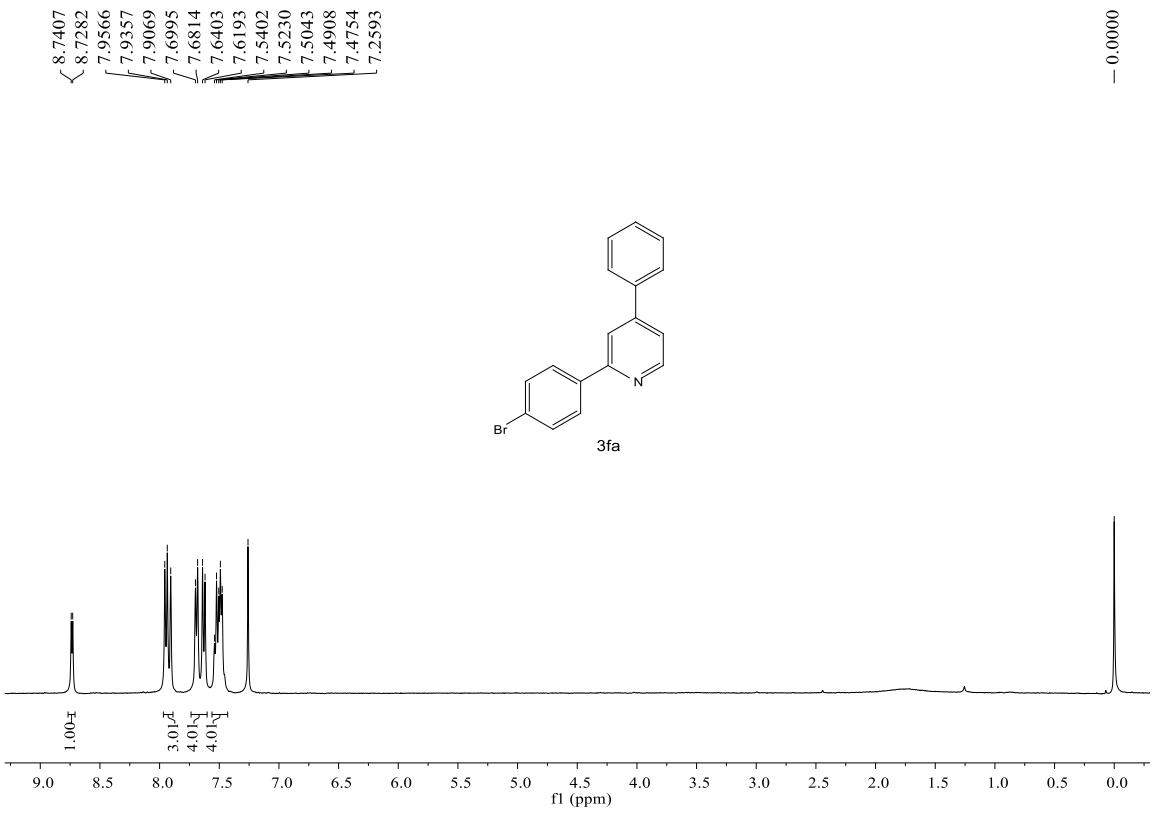
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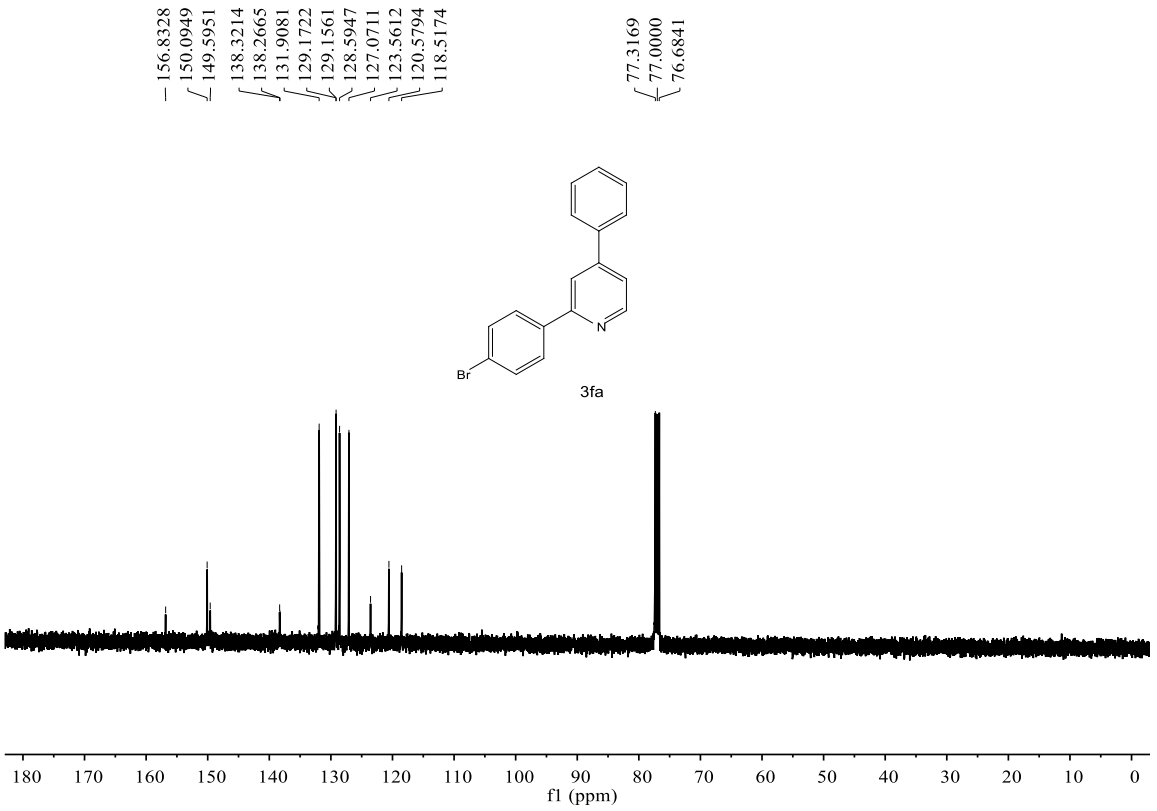
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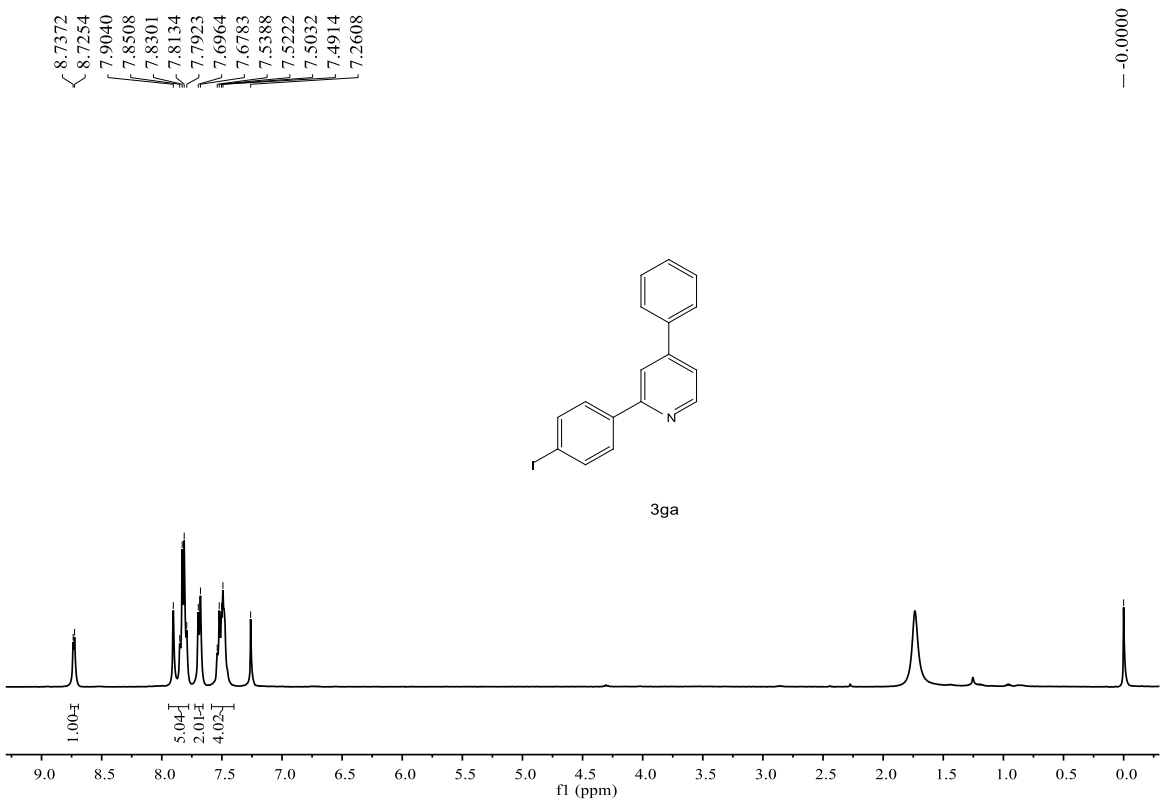
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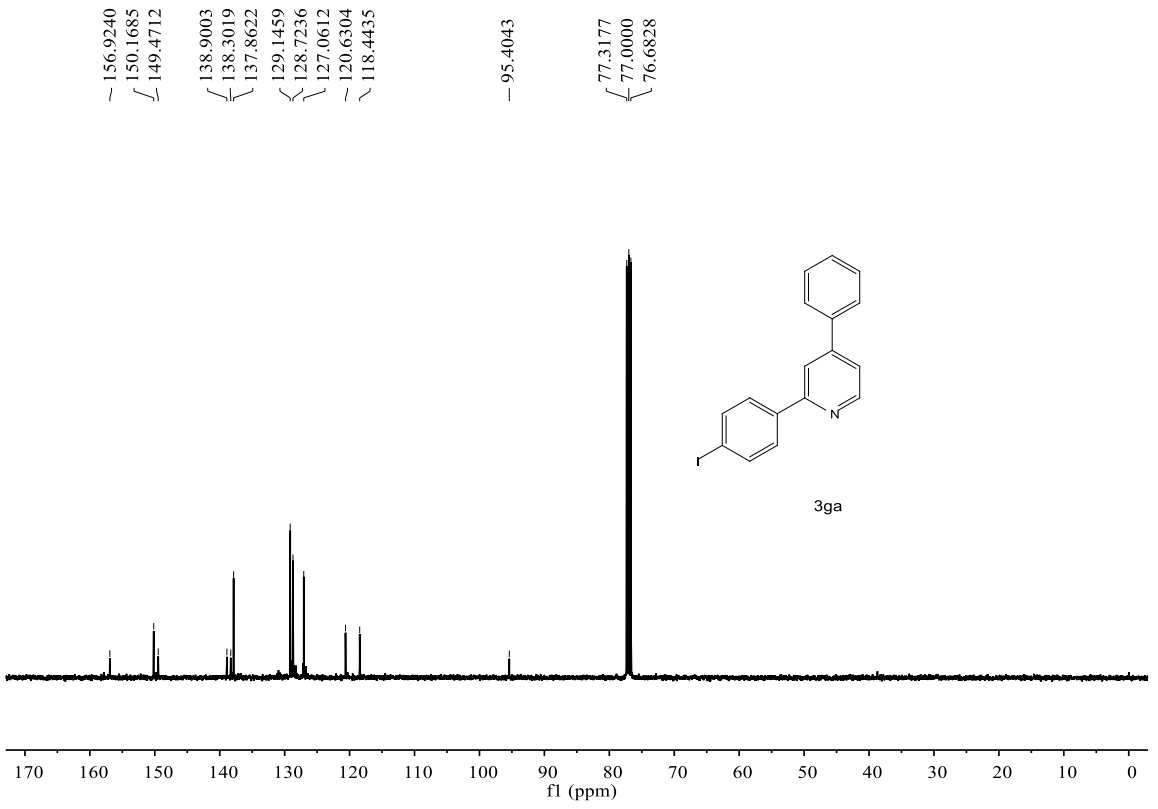
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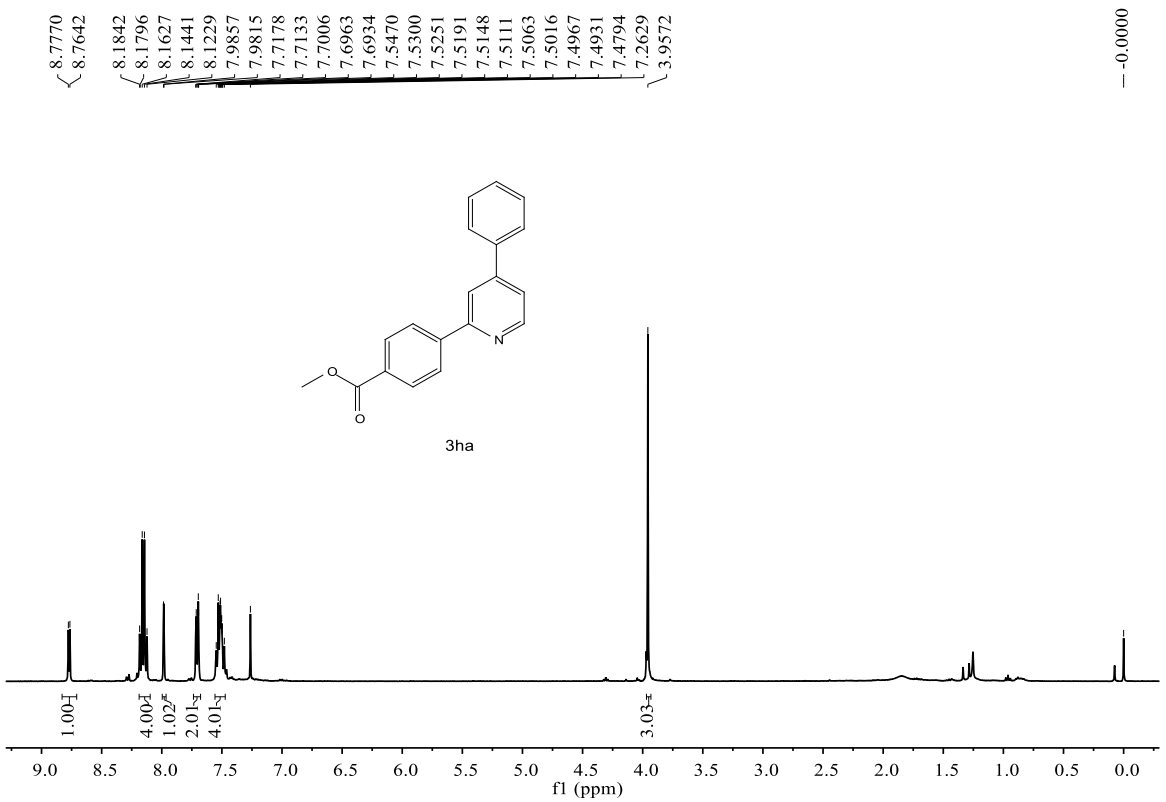
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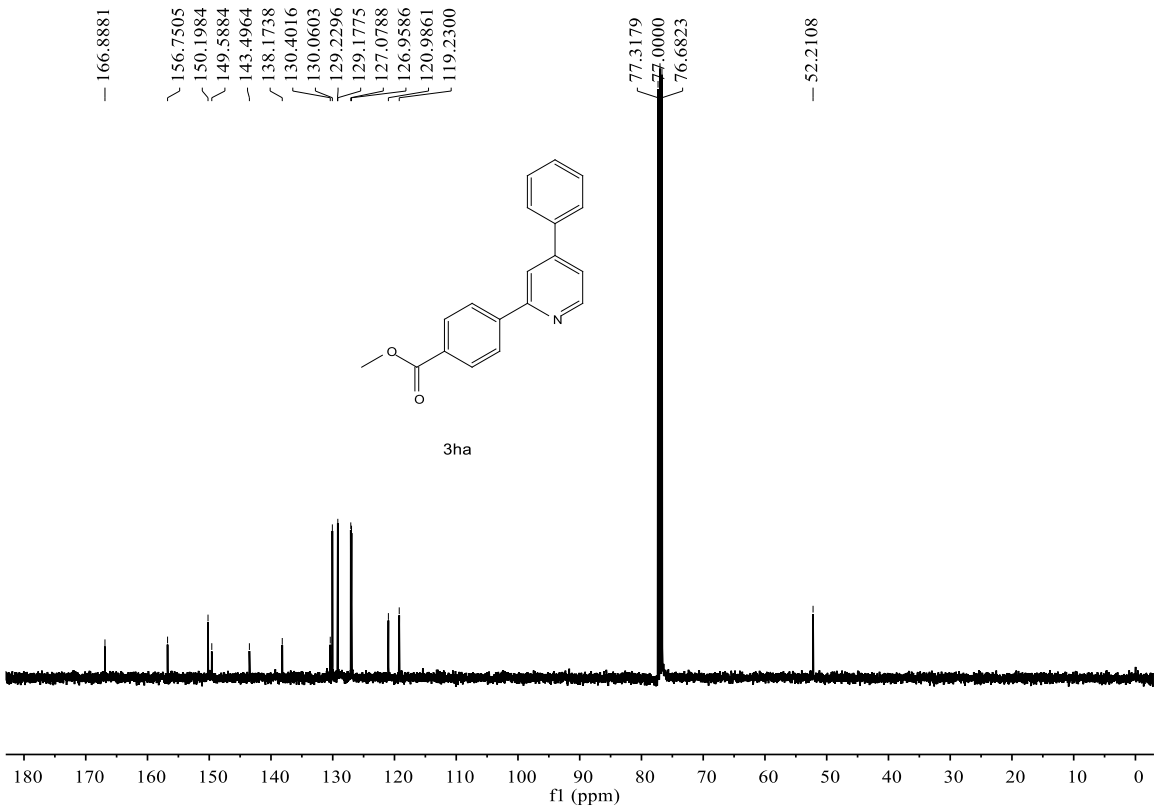
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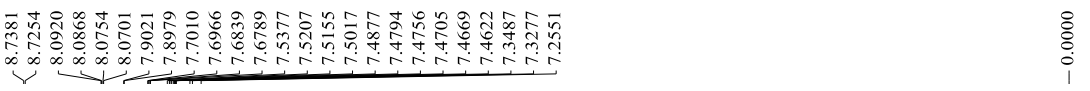
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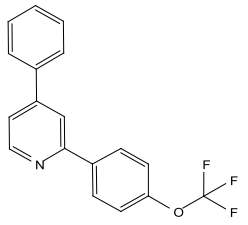
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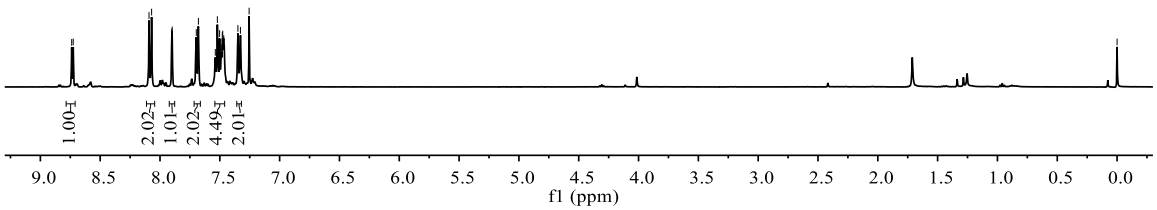
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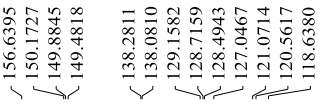
f1 (ppm)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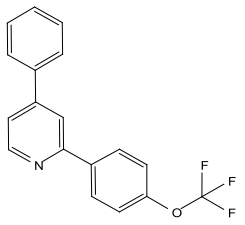
149.8845
149.4818
138.2811
138.0810
129.1582
128.7159
128.4943
127.0467
121.0714
120.5617
118.6380
77.3172
77.0000
76.6829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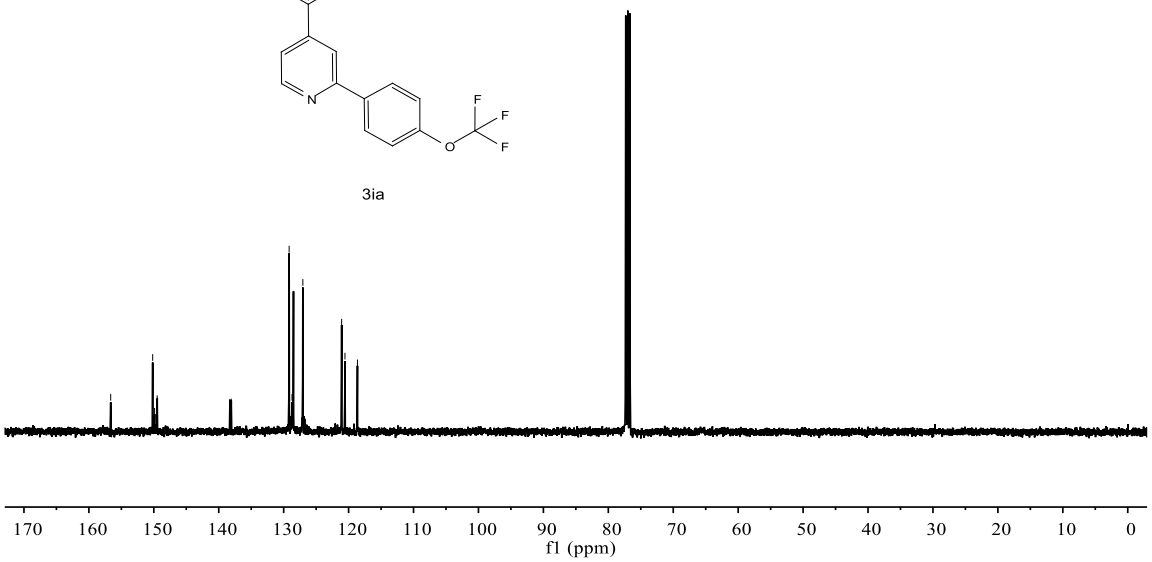
F
F
F
O

3ia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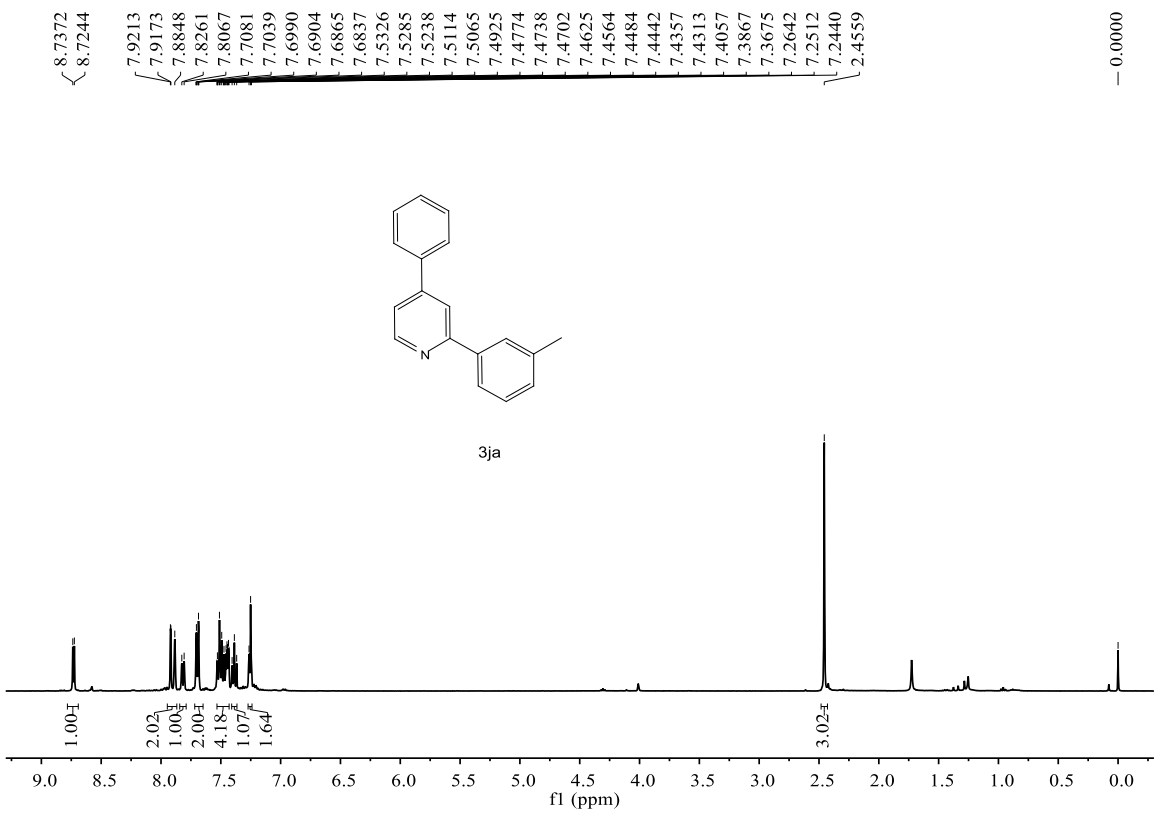
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.6990
7.6904
7.6865
7.6837
7.5326
7.5285
7.5238
7.5114
7.5065
7.4925
7.4774
7.4738
7.4702
7.4625
7.4564
7.4484
7.4442
7.4357
7.4313
7.4057
7.3867
7.3675
7.2642
7.2512
7.2440
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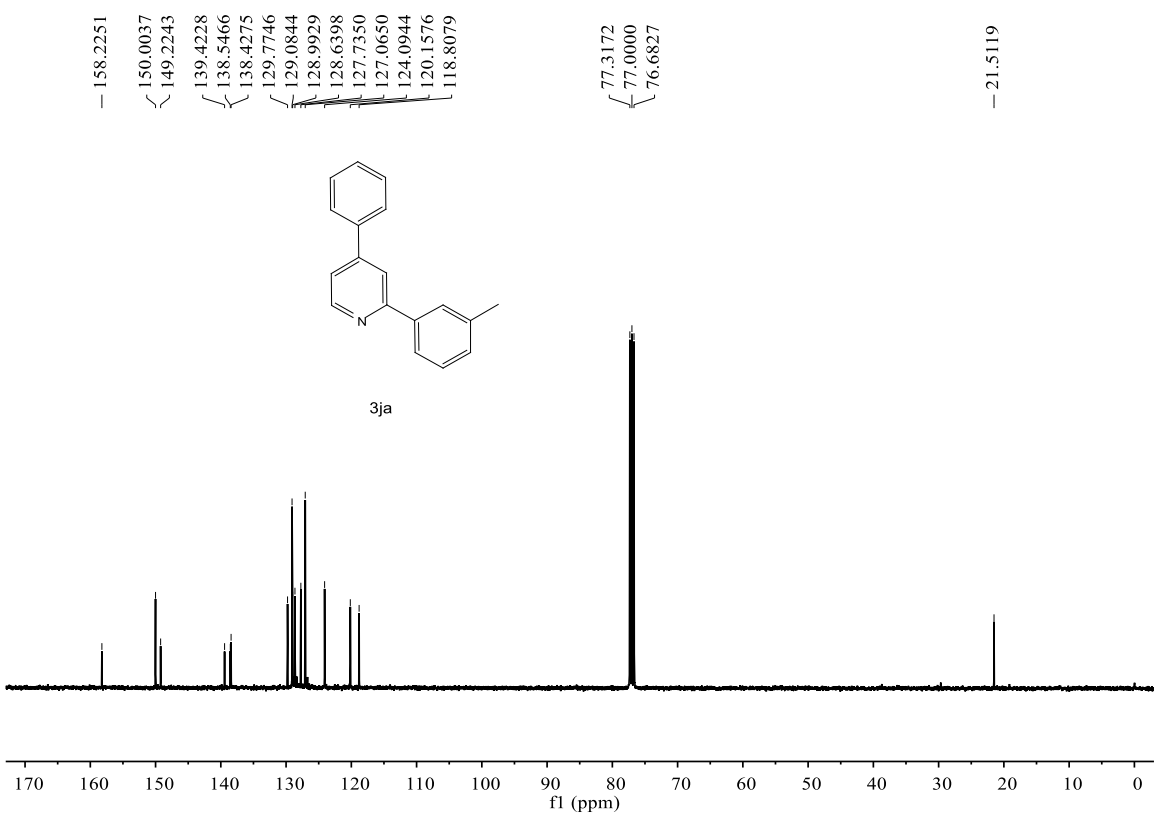
0.0000
3ja
1.00
2.02
1.00
2.00
4.18
1.07
1.64
3.02
f1 (ppm)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127.7350
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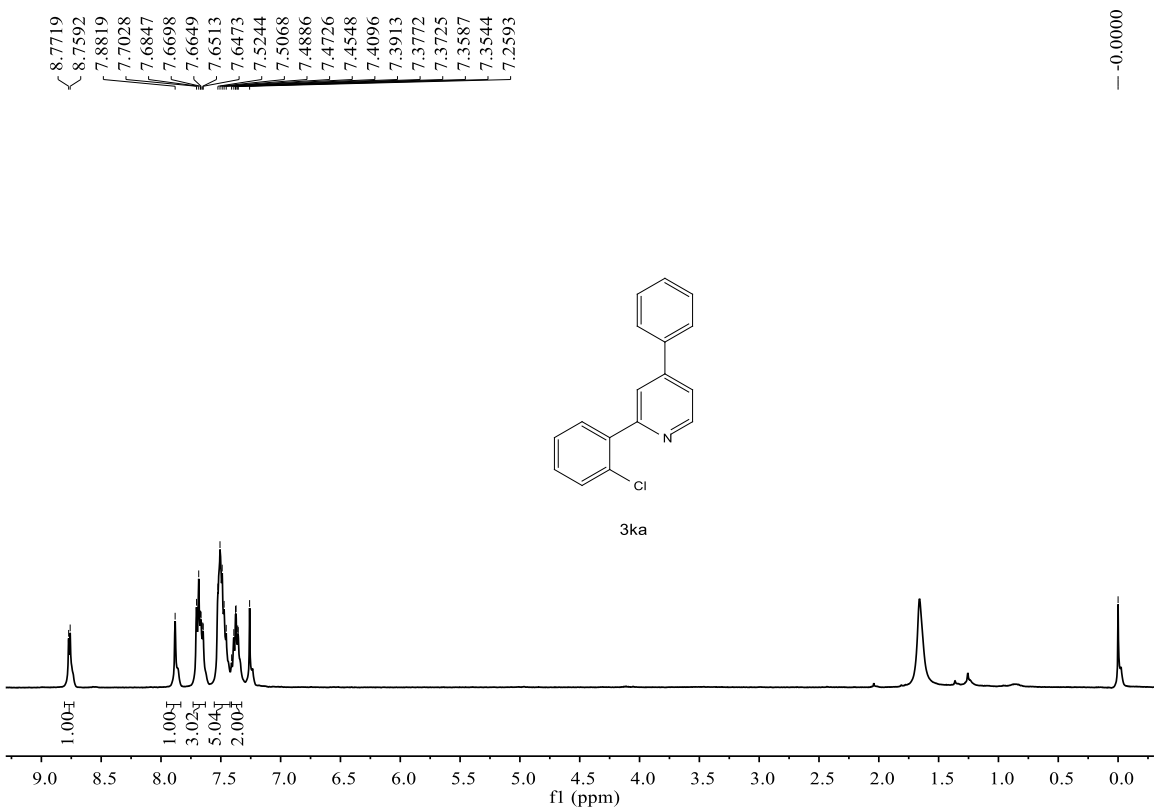
77.0000
76.6827
21.5119
3ja
f1 (ppm)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7.8819
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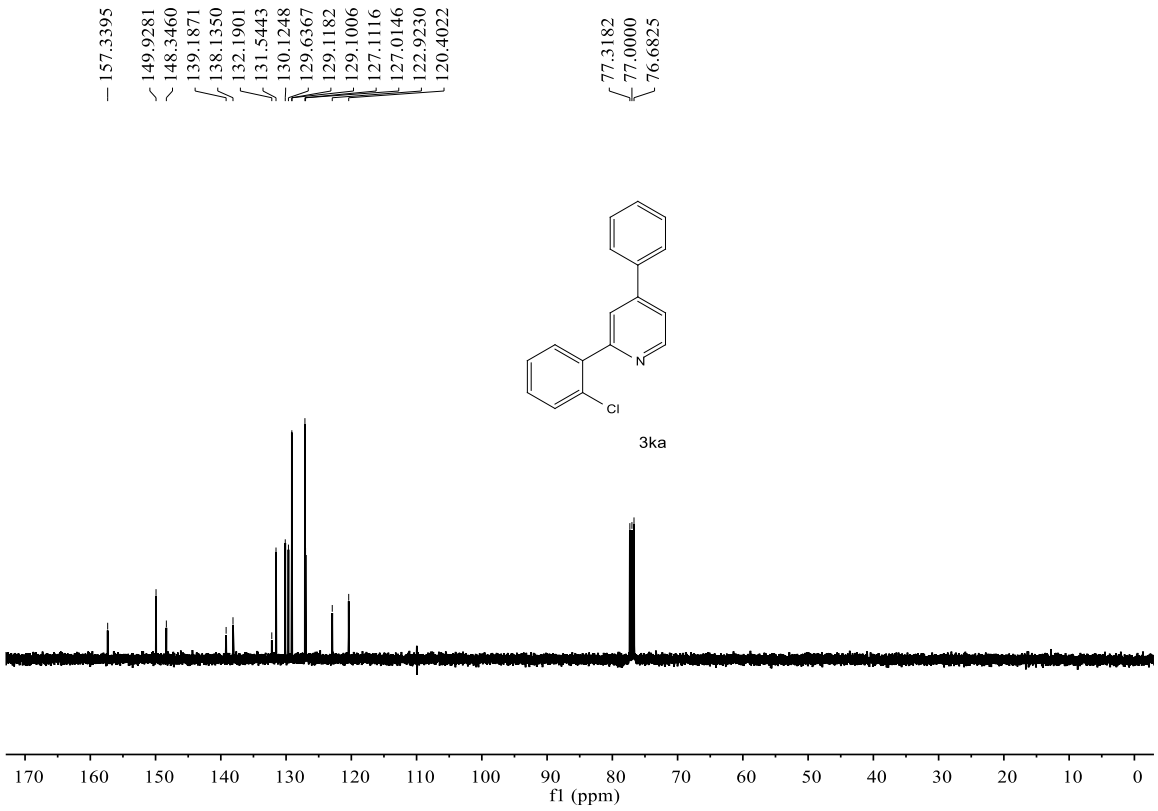
7.6473
7.5244
7.5068
7.4886
7.4726
7.4548
7.4096
7.3913
7.3772
7.3725
7.3587
7.3544
7.2593
-0.0000
N
Cl
3ka
1.00
1.00
3.02
5.04
2.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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132.1901
131.5443
130.1248
129.6367
129.1182
129.1006
127.1116
127.0146
122.9230
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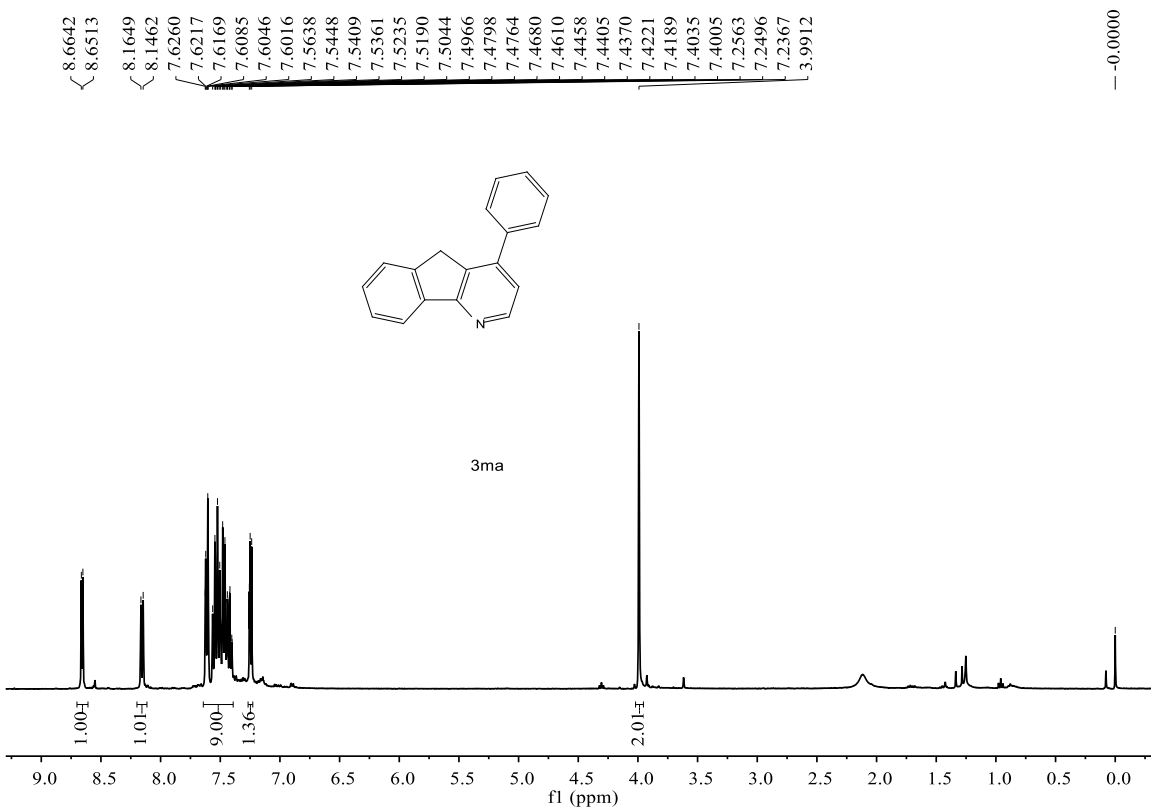
77.0000
76.6825
N
Cl
3ka
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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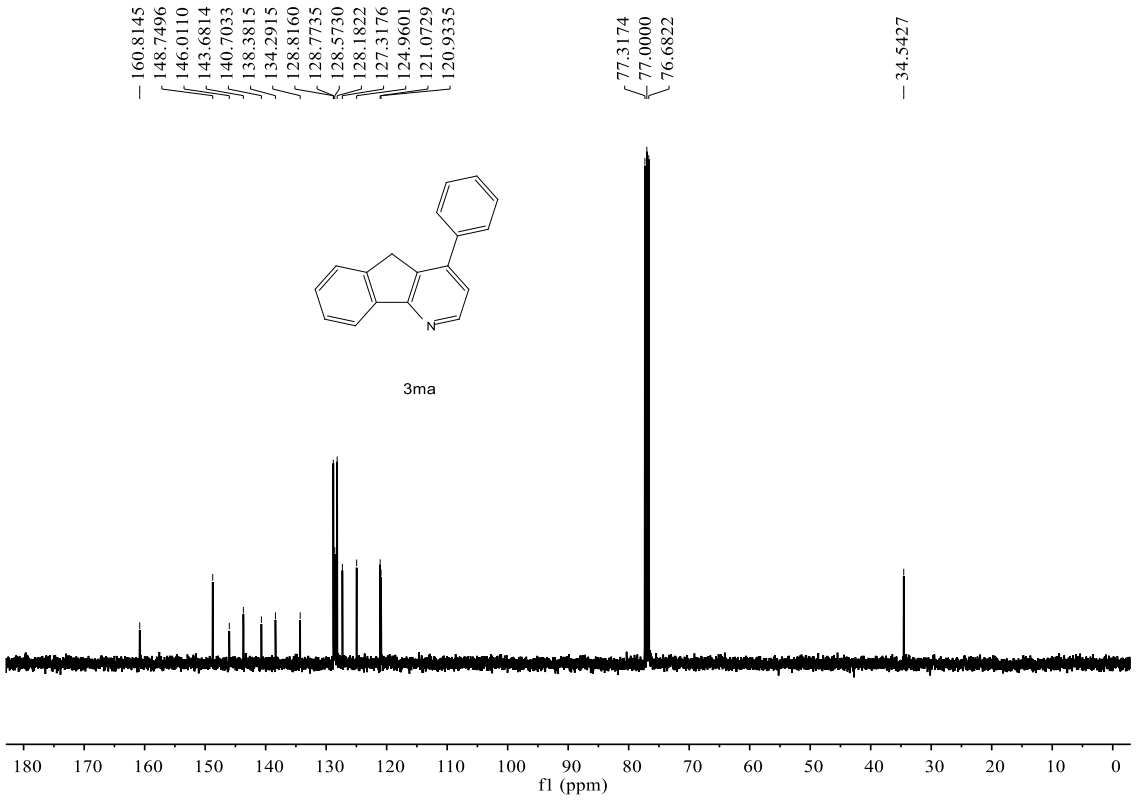
8.6513
8.1649
8.1462
7.6260
7.6217
7.6169
7.6085
7.6046
7.6016
7.5638
7.5448
7.5409
7.5361
7.5235
7.5190
7.5044
7.4966
7.4798
7.4764
7.4680
7.4610
7.4458
7.4405
7.4370
7.4221
7.4189
7.4035
7.4005
7.2563
7.2496
7.2367
3.9912
-0.0000
3ma
1.00
1.01
9.00
1.36
2.01
f1 (ppm)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146.0110
143.6814
140.7033
138.3815
134.2915
128.8160
128.7735
128.5730
128.1822
127.3176
124.9601
121.0729
120.9335
77.3174
77.0000
76.6822
34.5427
3ma
f1 (ppm)


8.5872
8.5750
8.3794
8.3607
7.4970
7.4802
7.4618
7.4472
7.4298
7.4121
7.3942
7.3716
7.3681
7.3511
7.3329
7.3146
7.2589
7.2334
7.2154
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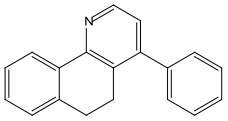
7.1373
2.9385
2.9240
2.9057
2.8447
2.8270
2.8120
0.0000



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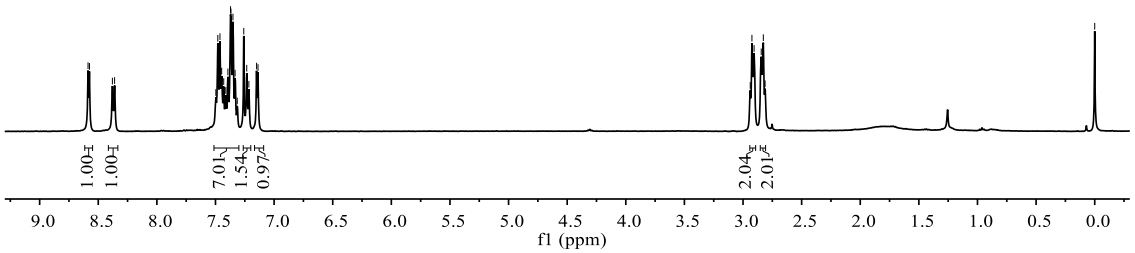
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1.00
7.01
1.54
0.97
2.04
2.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


152.9661
148.6338
147.1020
138.8895
138.1068
134.8195
129.4738
129.1054
128.7380
128.4087
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127.1485
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123.2997
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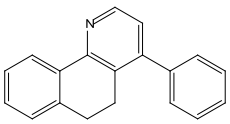
77.0000
76.6818
28.0447
25.4541



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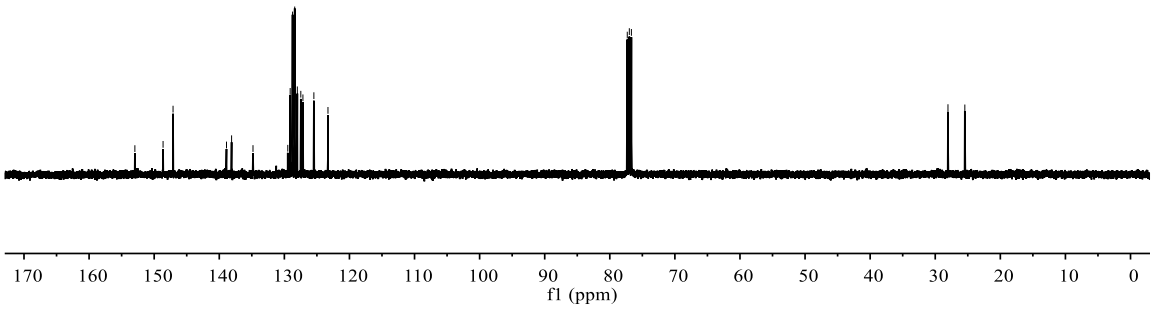
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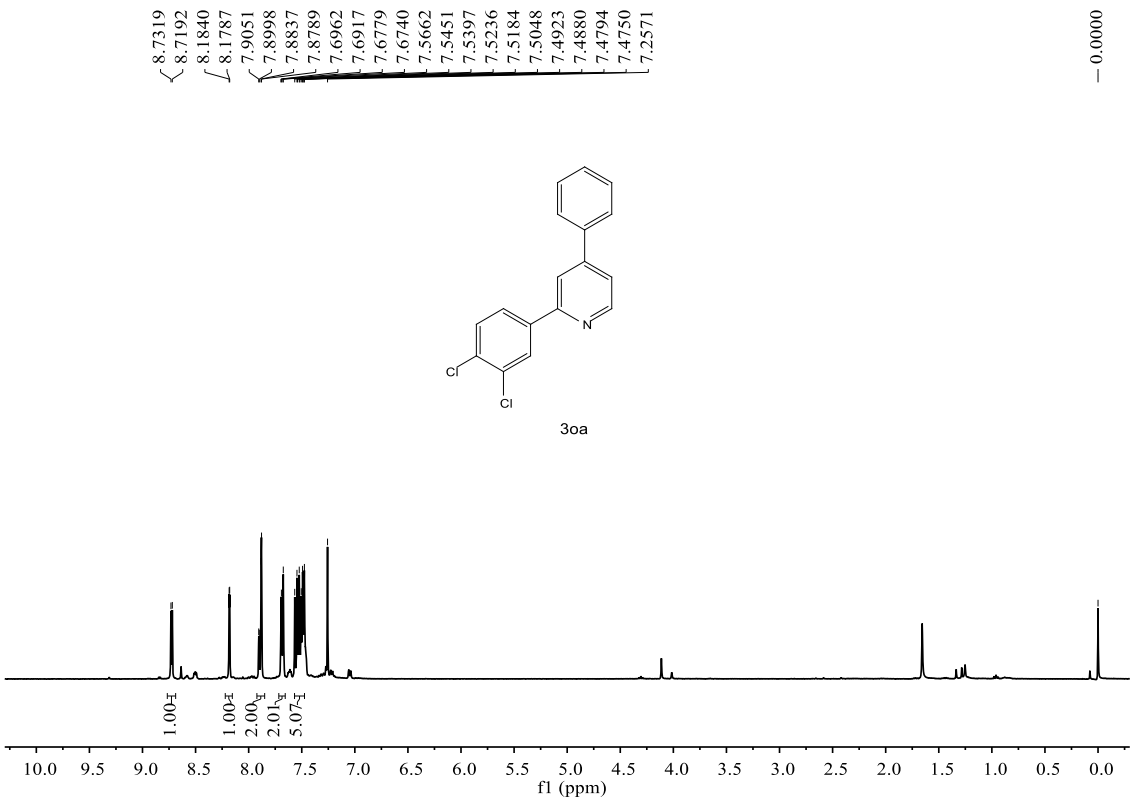
f1 (ppm)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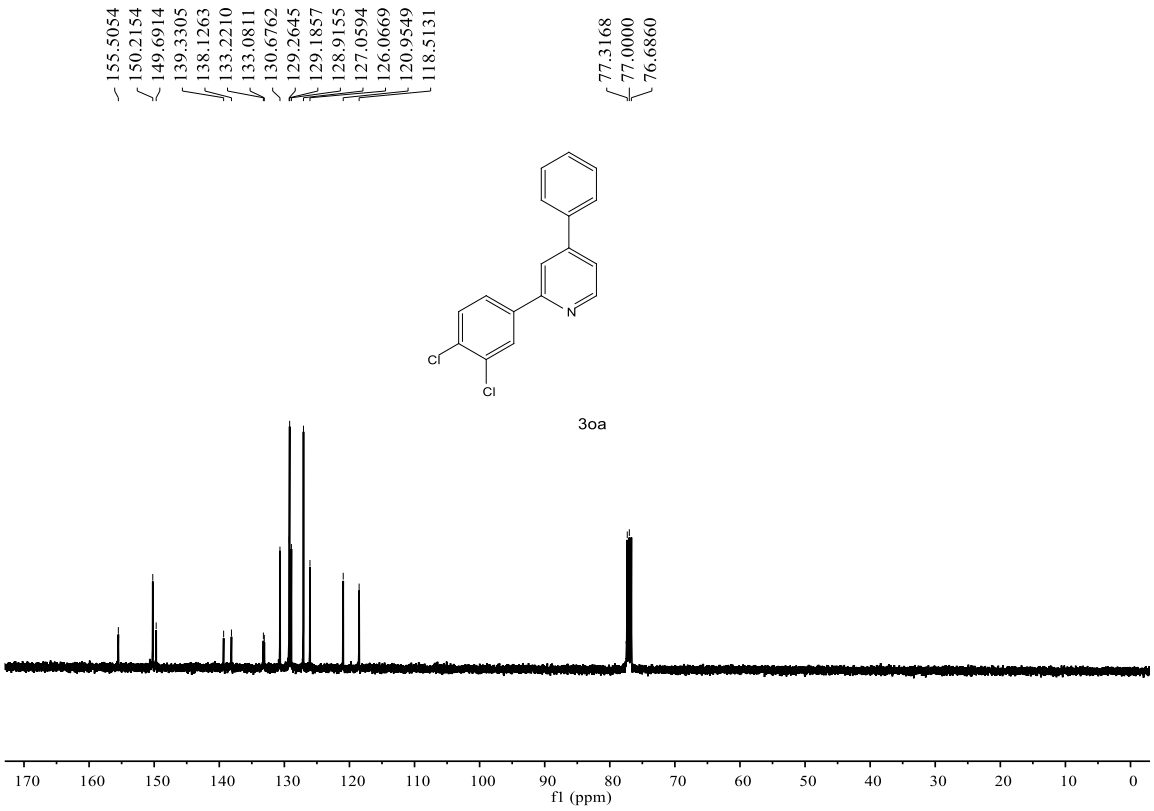
8.1840
8.1787
7.9051
7.8998
7.8837
7.8789
7.6962
7.6917
7.6779
7.6740
7.5662
7.5451
7.5397
7.5236
7.5184
7.5048
7.4923
7.4880
7.4794
7.4750
7.2571
0.0000
N
Cl
Cl
3oa
1.00
1.00
2.00
2.01
5.07
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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150.2154
149.6914
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138.1263
133.2210
133.0811
130.6762
129.2645
129.1857
128.9155
127.0594
126.0669
120.9549
118.5131
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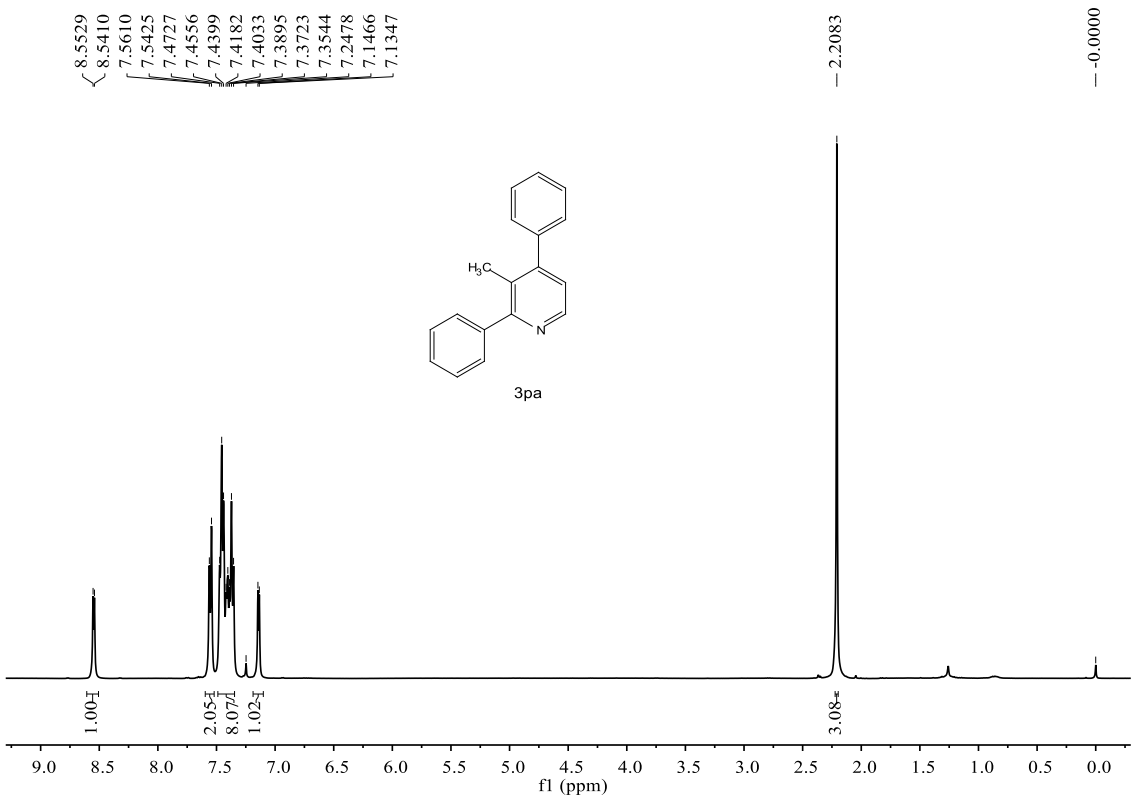
76.6860
N
Cl
Cl
3oa
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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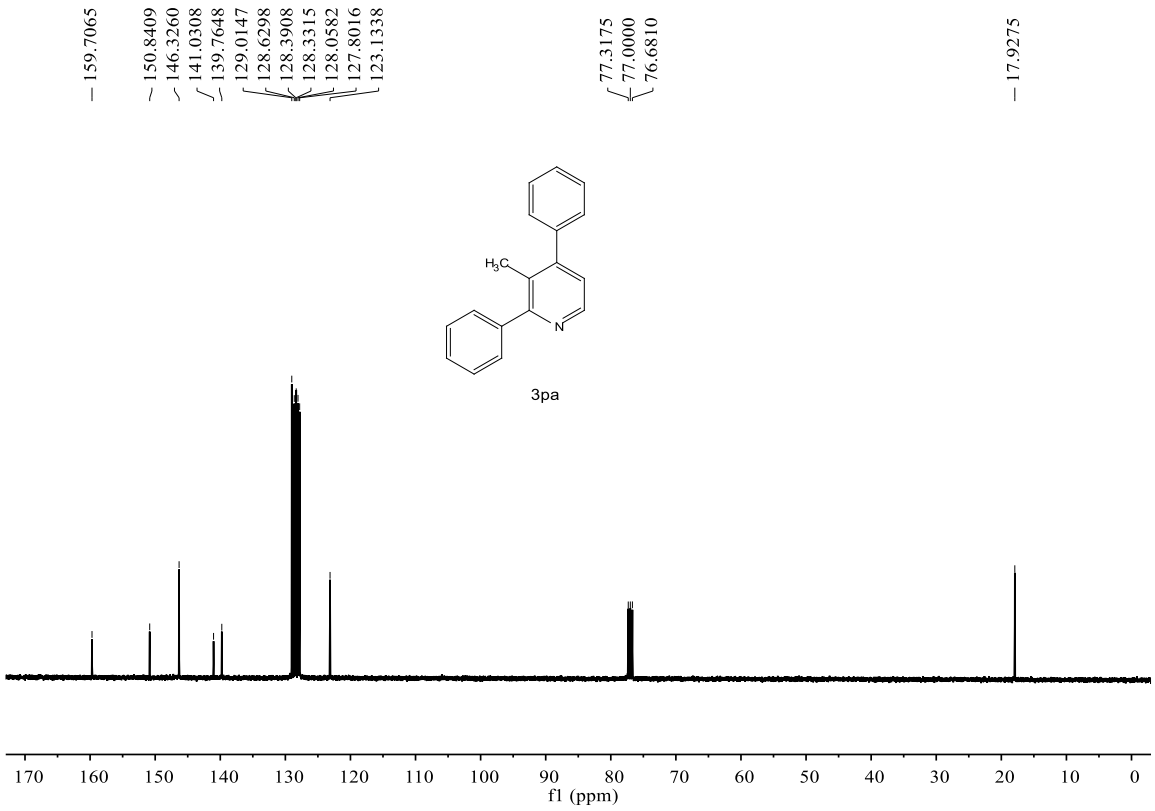
7.5610
7.5425
7.4727
7.4556
7.4399
7.4182
7.4033
7.3895
7.3723
7.3544
7.2478
7.1466
7.1347
2.2083
-0.0000
3pa
1.00
2.05
8.07
1.02
3.08
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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139.7648
129.0147
128.6298
128.3908
128.3315
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127.8016
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77.0000
76.6810
17.9275
3pa
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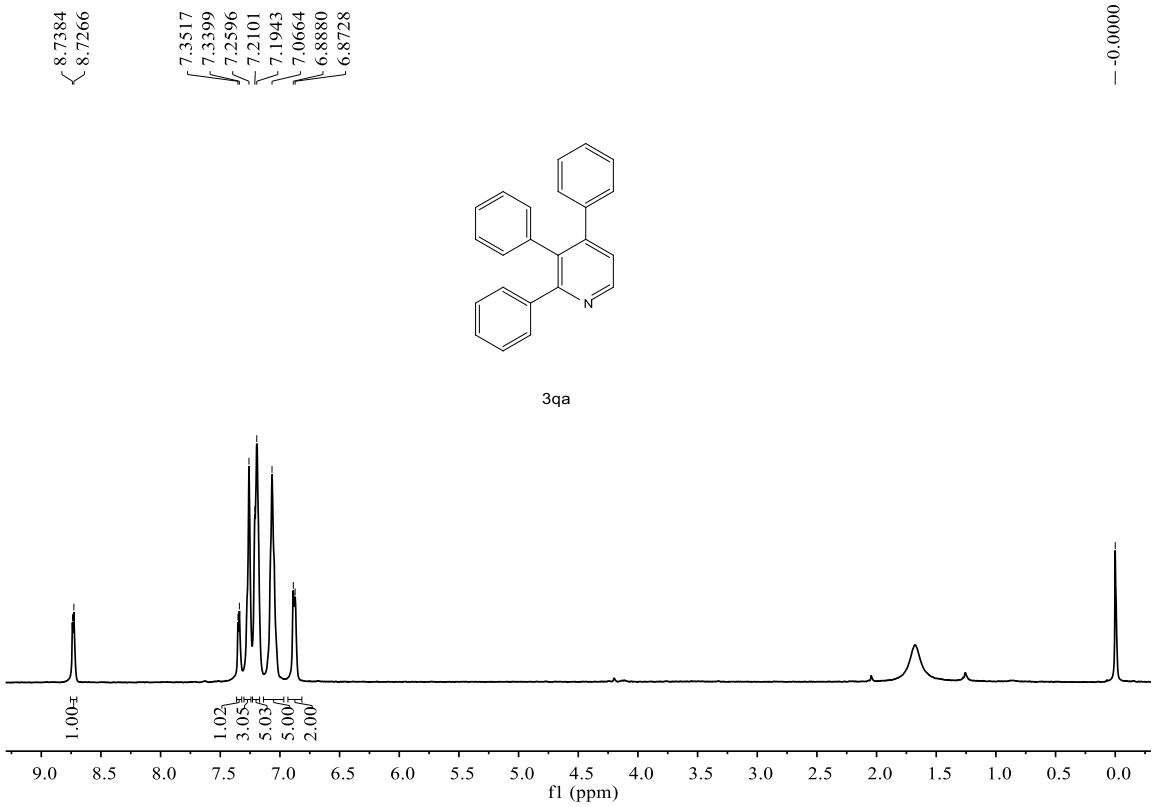
f1 (ppm)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7.2596
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6.8728
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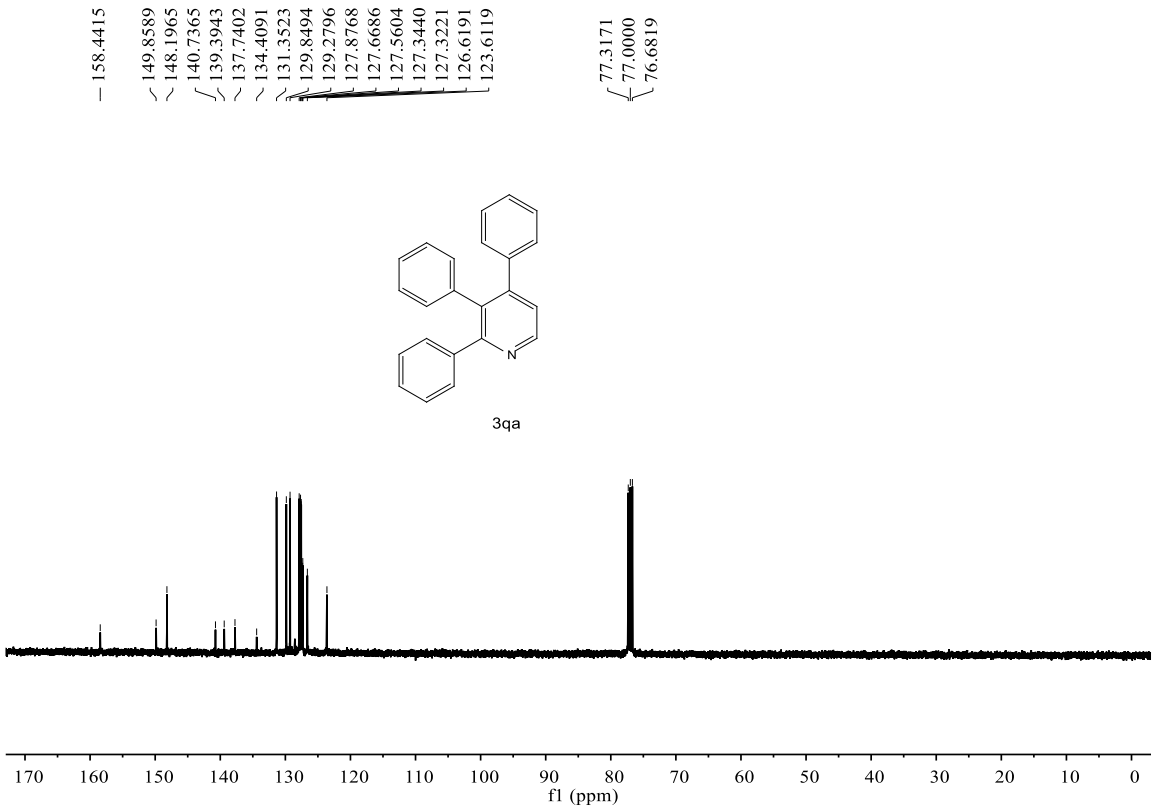
N
3qa
1.00
1.02
3.05
5.03
5.00
2.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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134.4091
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127.6686
127.5604
127.3440
127.3221
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77.0000
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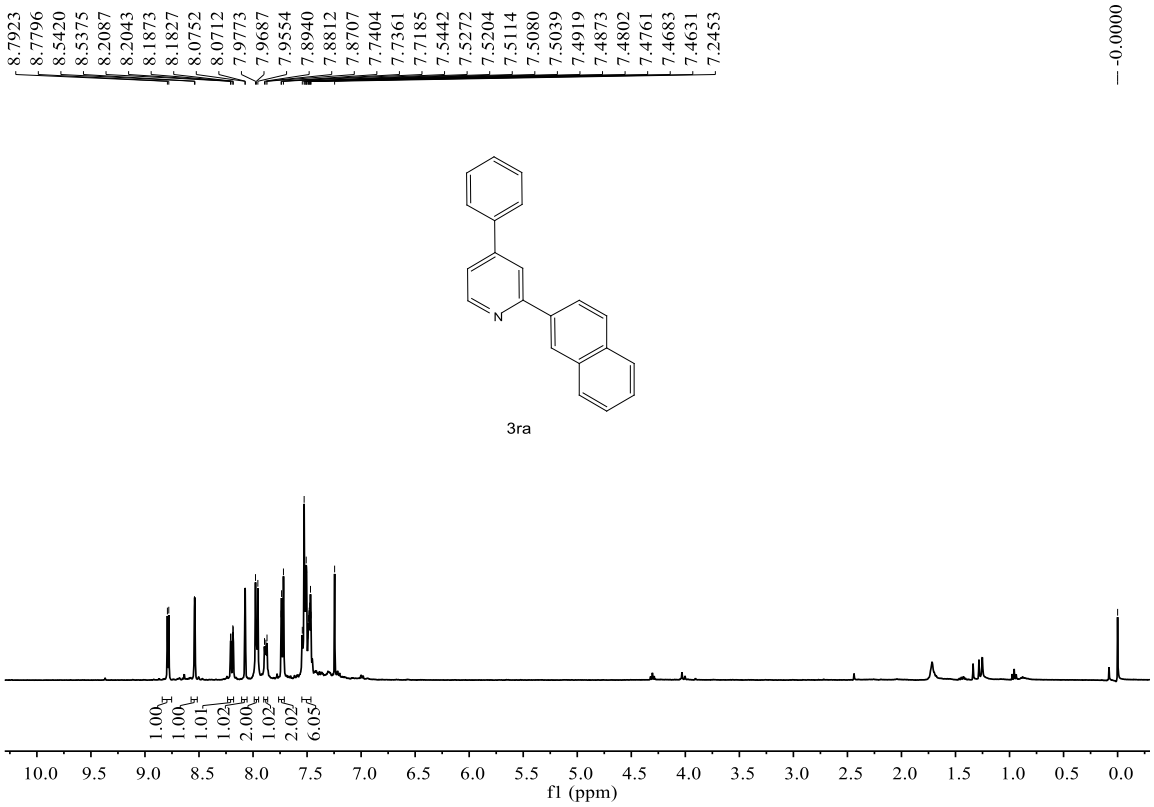
f1 (ppm)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8.0752
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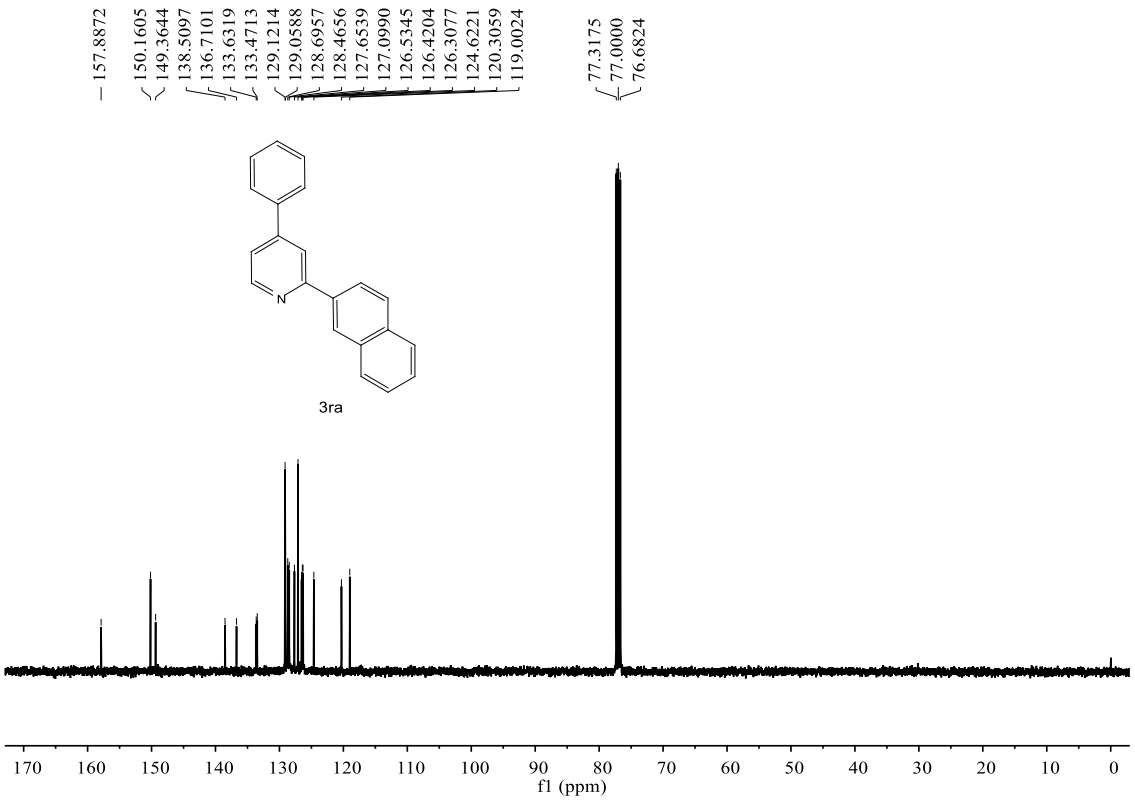
7.9687
7.9554
7.8940
7.8812
7.8707
7.7404
7.7361
7.7185
7.5442
7.5272
7.5204
7.5114
7.5080
7.5039
7.4919
7.4873
7.4802
7.4761
7.4683
7.4631
7.2453
-0.0000
N
3ra
1.00
1.00
1.01
1.02
2.00
1.02
2.02
6.05
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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133.4713
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128.6957
128.4656
127.6539
127.0990
126.5345
126.4204
126.3077
124.6221
120.3059
119.0024
77.3175
77.0000
76.6824
N
3ra
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.6441
8.6315
7.9342
7.7096
7.6917
7.5614
7.5283
7.5112
7.4925
7.4778
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7.3914
7.3813
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7.1455
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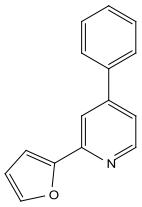
6.5656
-0.0000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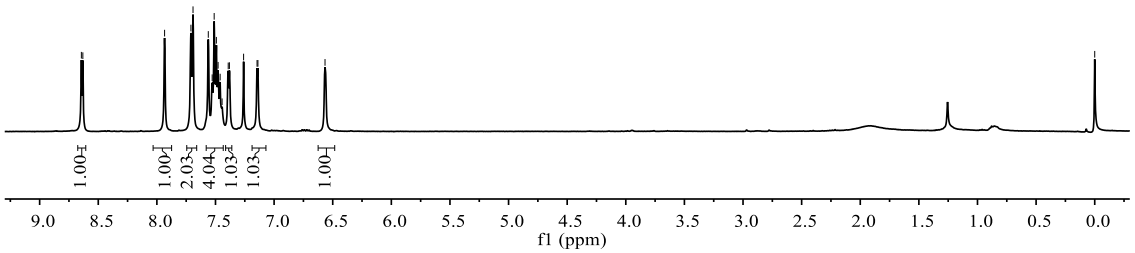
O

3sa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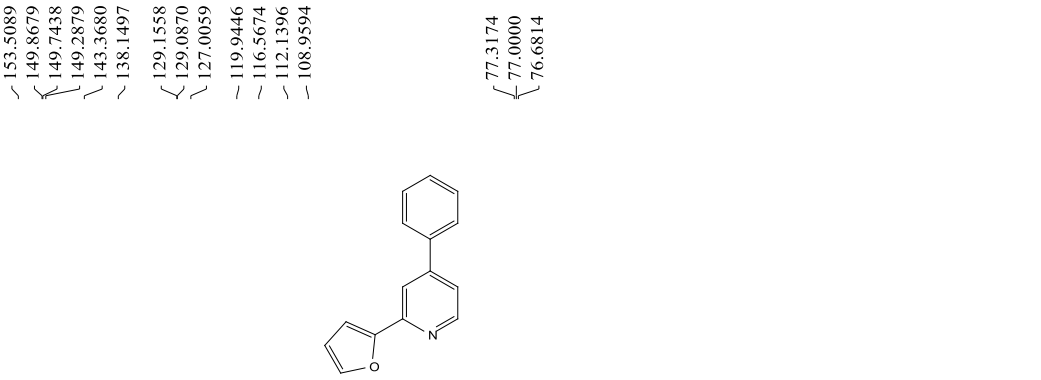
1.00
2.03
4.04
1.03
1.03
1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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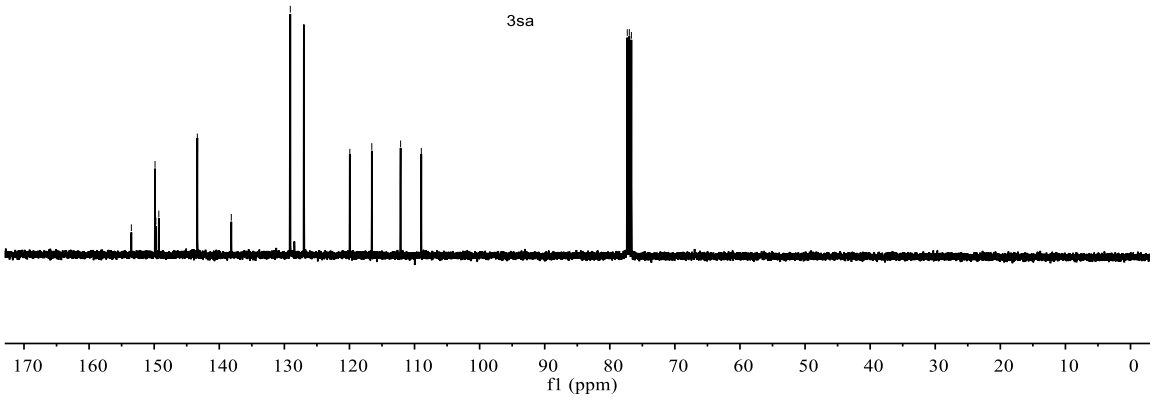
N
O

3sa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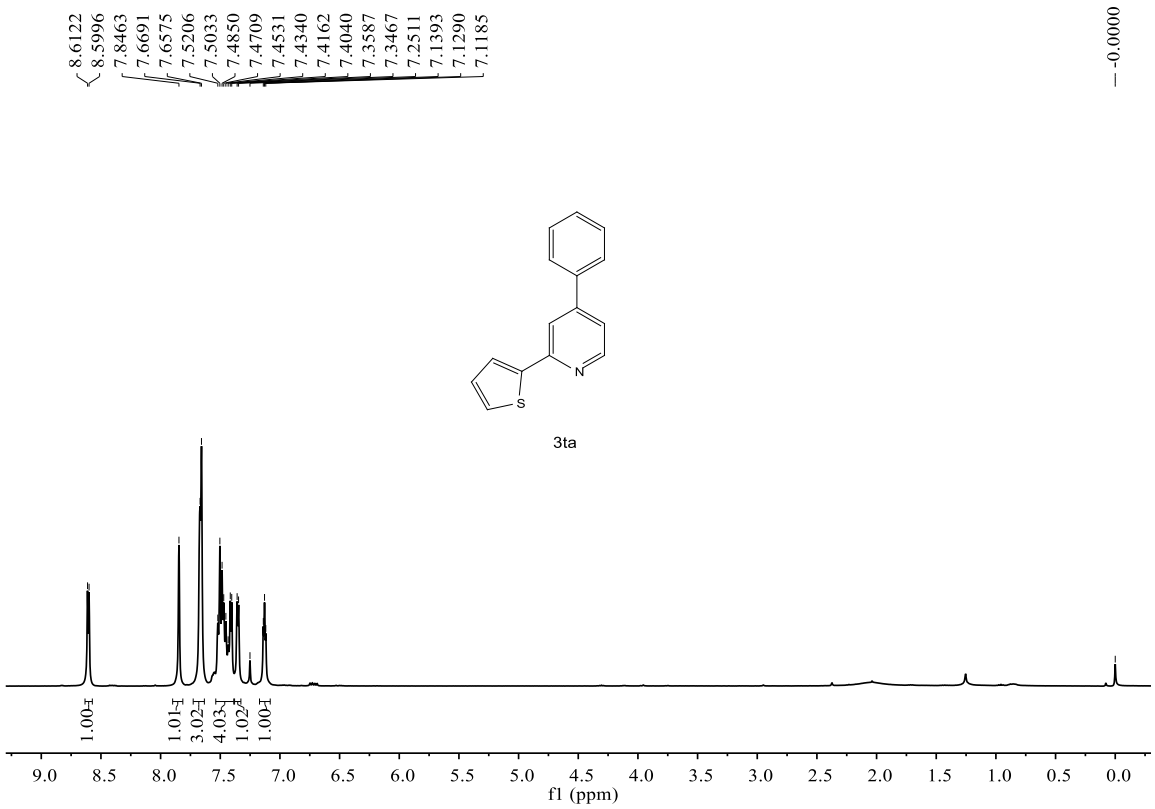
f1 (ppm)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7.5033
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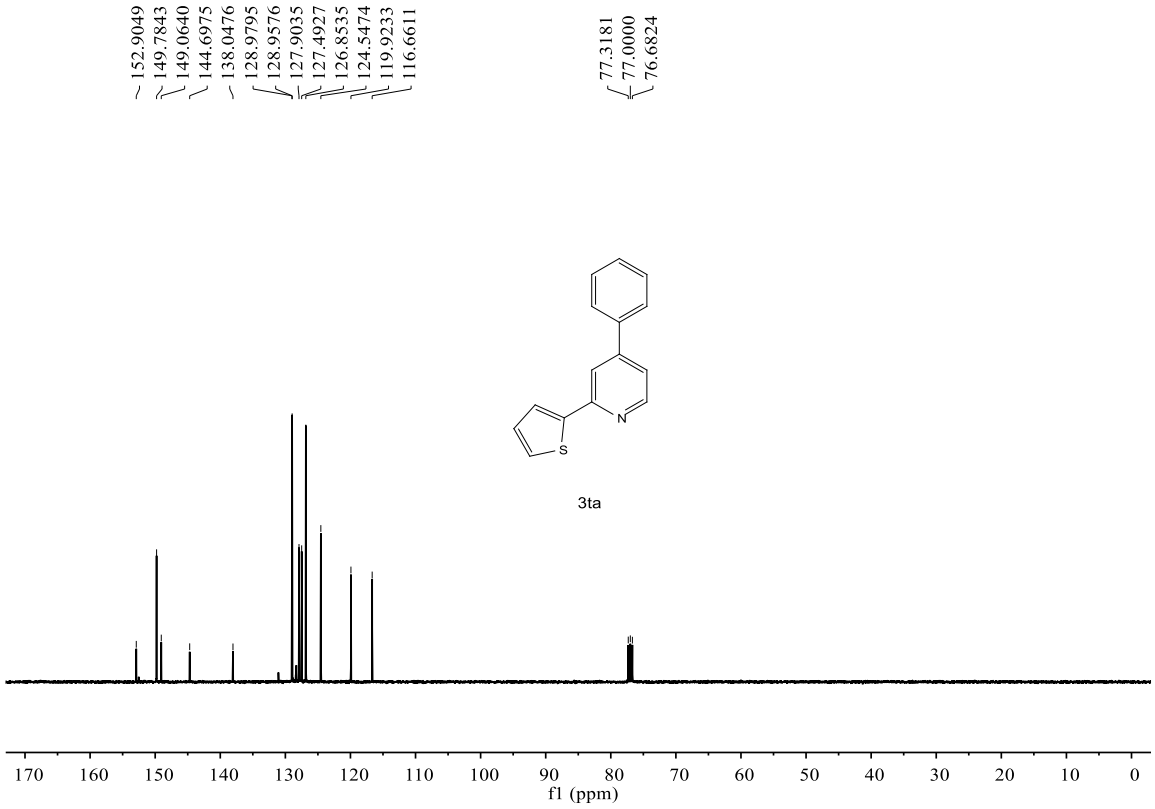
7.4531
7.4340
7.4162
7.4040
7.3587
7.3467
7.2511
7.1393
7.1290
7.1185
-0.0000
N
S
3ta
1.00
1.01
3.02
4.03
1.02
1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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124.5474
119.9233
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77.0000
76.6824
N
S
3ta
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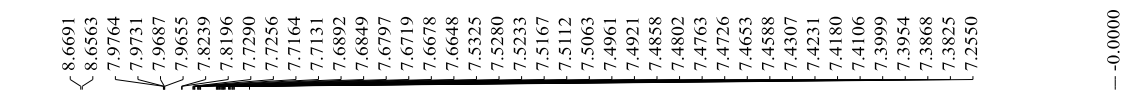
f1 (ppm)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7.4588
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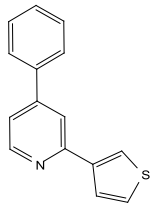
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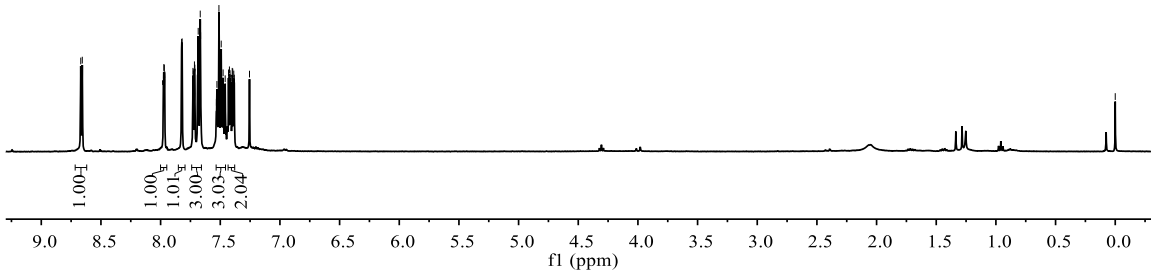
S

3ua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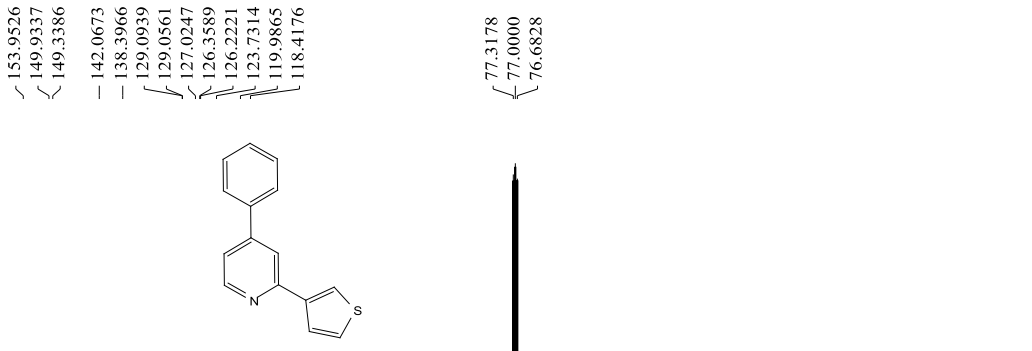
1.00
1.01
3.00
3.03
2.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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149.3386
142.0673
138.3966
129.0939
129.0561
127.0247
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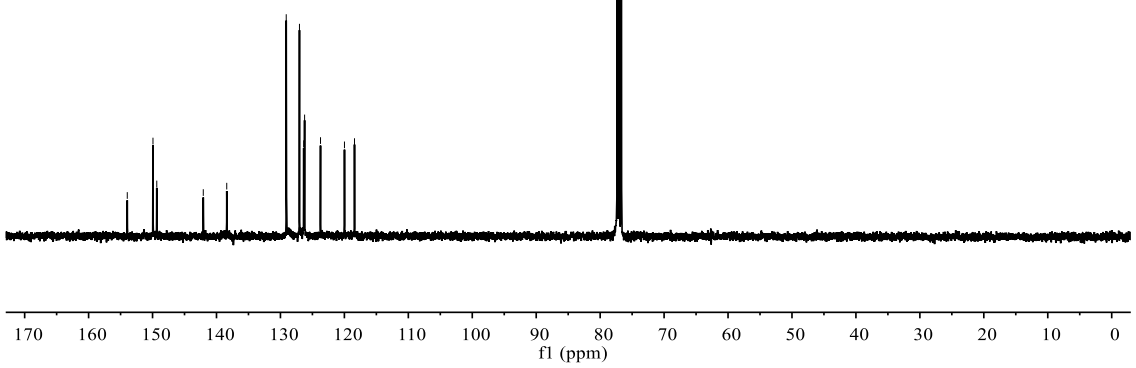
126.2221
123.7314
119.9865
118.4176
77.3178
77.0000
76.6828

N
S

3ua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160
150
140
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110
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90
80
70
60
50
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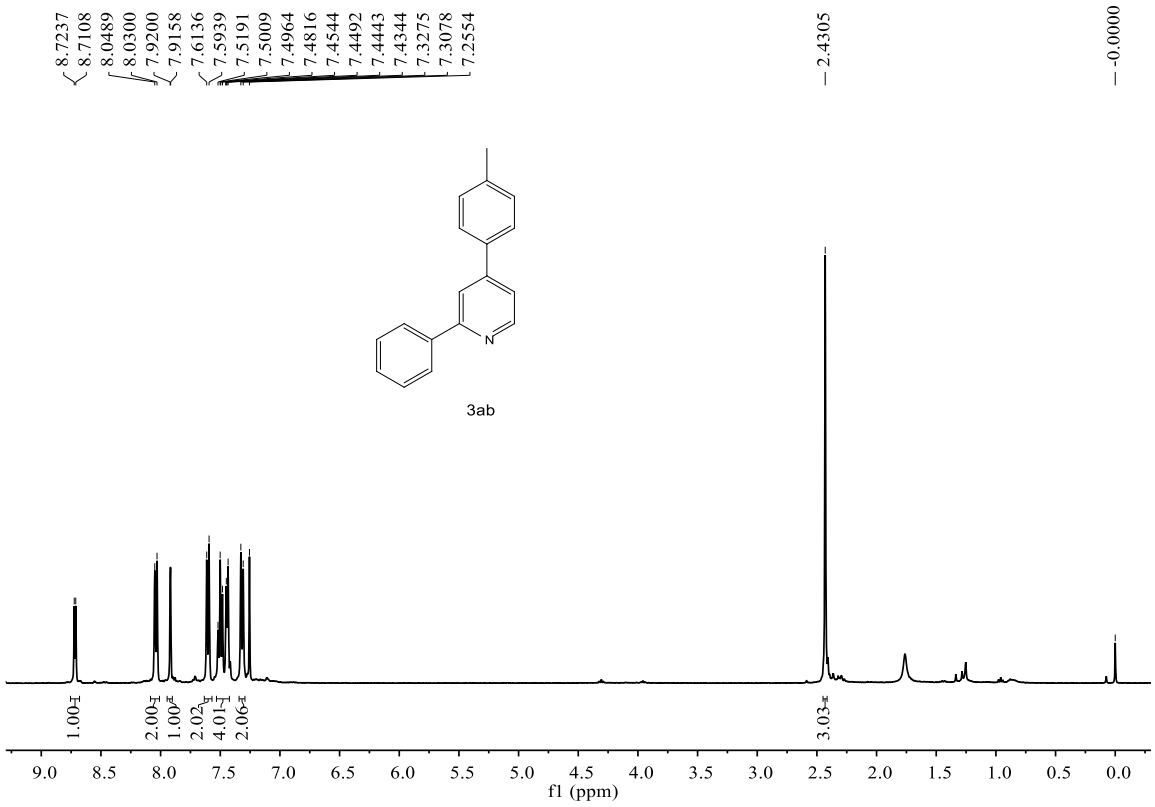
30
20
10
0
f1 (ppm)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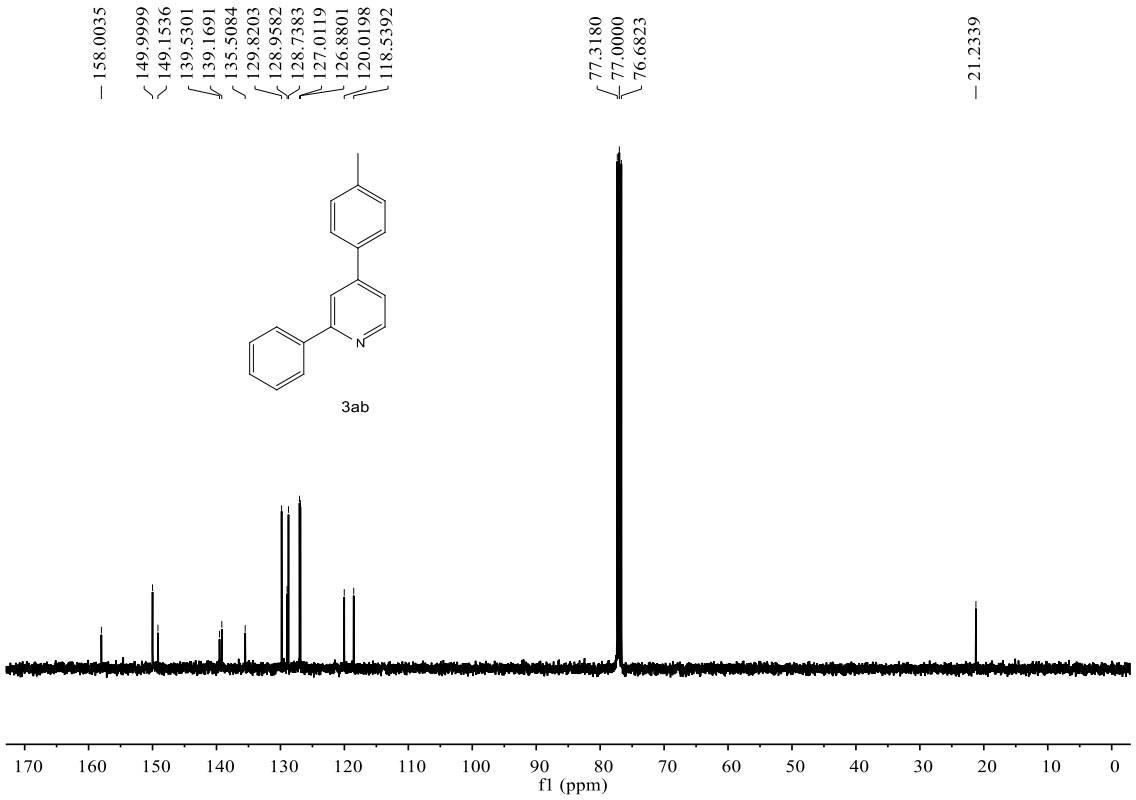
8.7108
8.0489
8.0300
7.9200
7.9158
7.6136
7.5939
7.5191
7.5009
7.4964
7.4816
7.4544
7.4492
7.4443
7.4344
7.3275
7.3078
7.2554
2.4305
-0.0000
3ab
1.00
2.00
1.00
2.02
4.01
2.06
3.03
f1 (ppm)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129.8203
128.9582
128.7383
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120.0198
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77.3180
77.0000
76.6823
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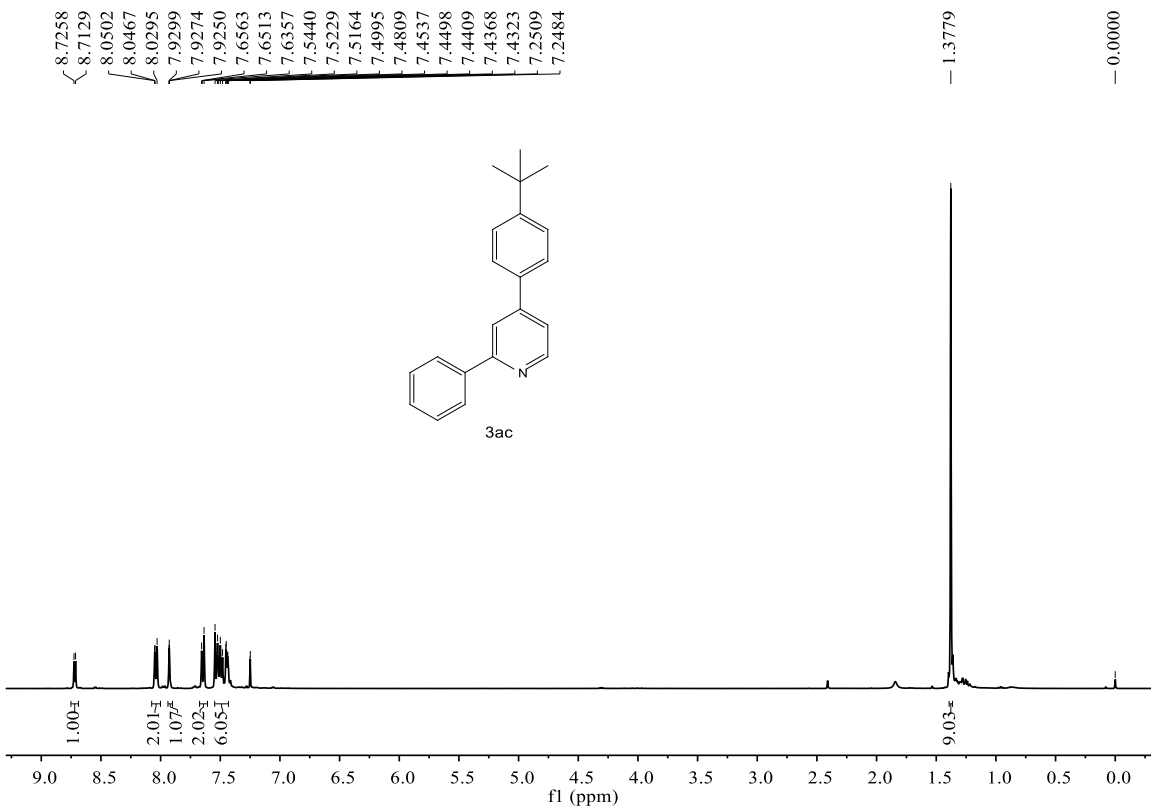
3ab
f1 (ppm)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8.7129
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8.0467
8.0295
7.9299
7.9274
7.9250
7.6563
7.6513
7.6357
7.5440
7.5229
7.5164
7.4995
7.4809
7.4537
7.4498
7.4409
7.4368
7.4323
7.2509
7.2484
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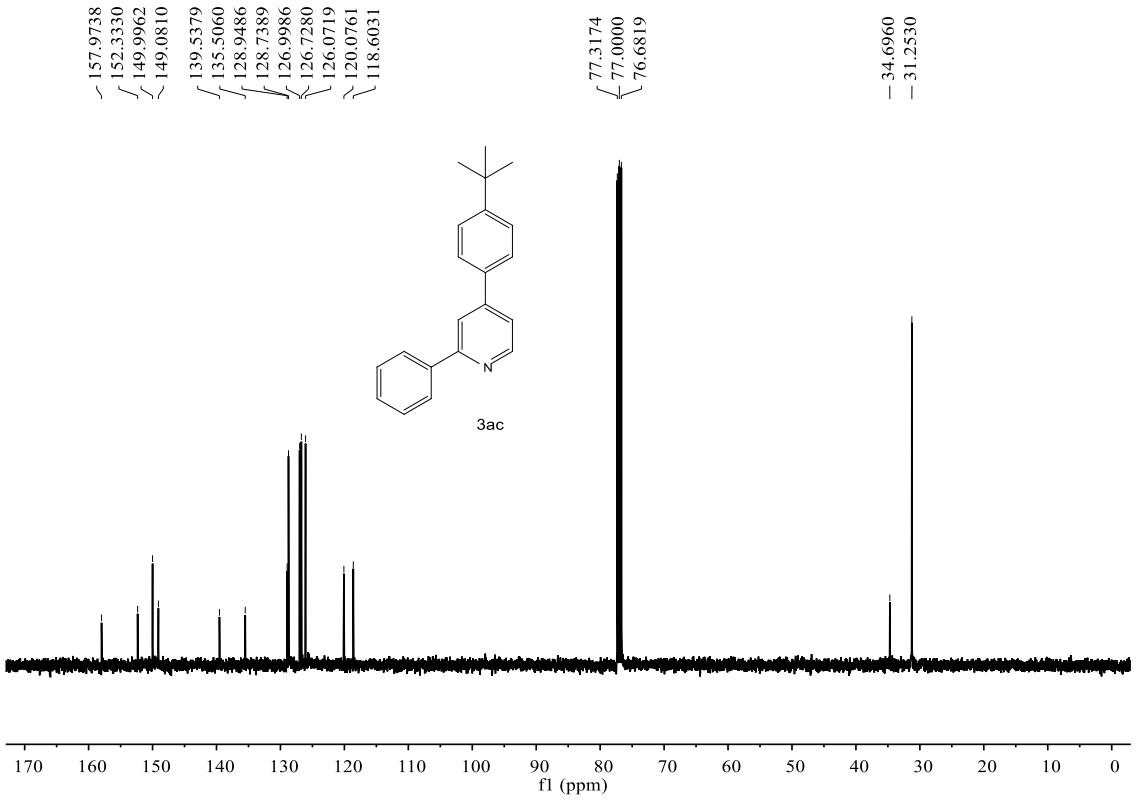
0.0000
3ac
1.00
2.01
1.07
2.02
6.05
9.03
f1 (ppm)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126.9986
126.7280
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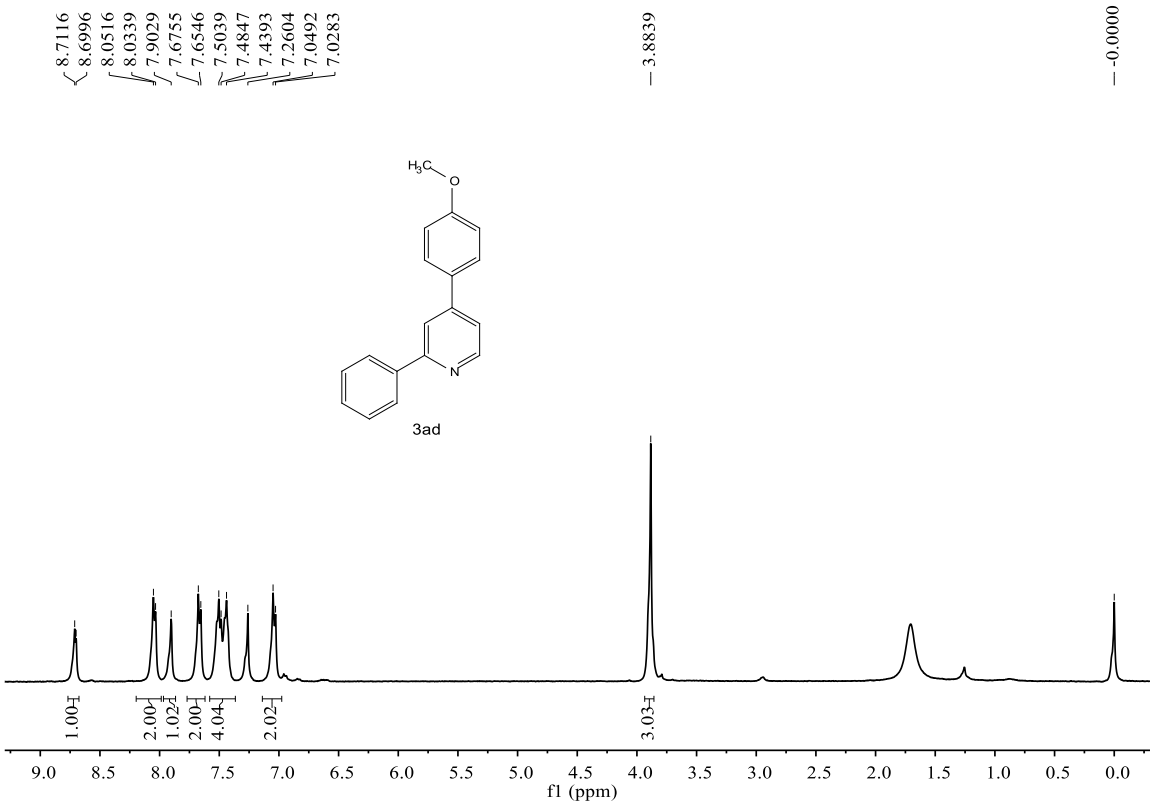
120.0761
118.6031
77.3174
77.0000
76.6819
34.6960
31.2530
3ac
f1 (ppm)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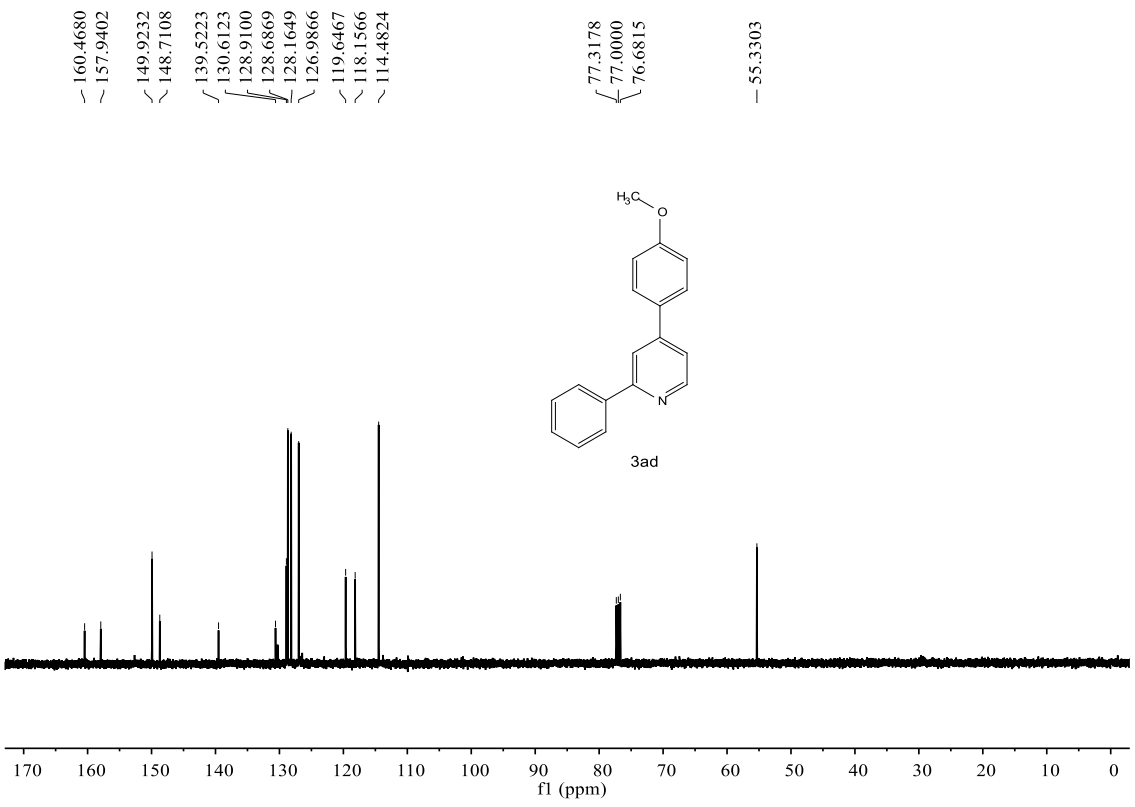
8.0516
8.0339
7.9029
7.6755
7.6546
7.5039
7.4847
7.4393
7.2604
7.0492
7.0283
3.8839
-0.0000
3ad
1.00
2.00
1.02
2.00
4.04
2.02
3.03
f1 (ppm)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139.5223
130.6123
128.9100
128.6869
128.1649
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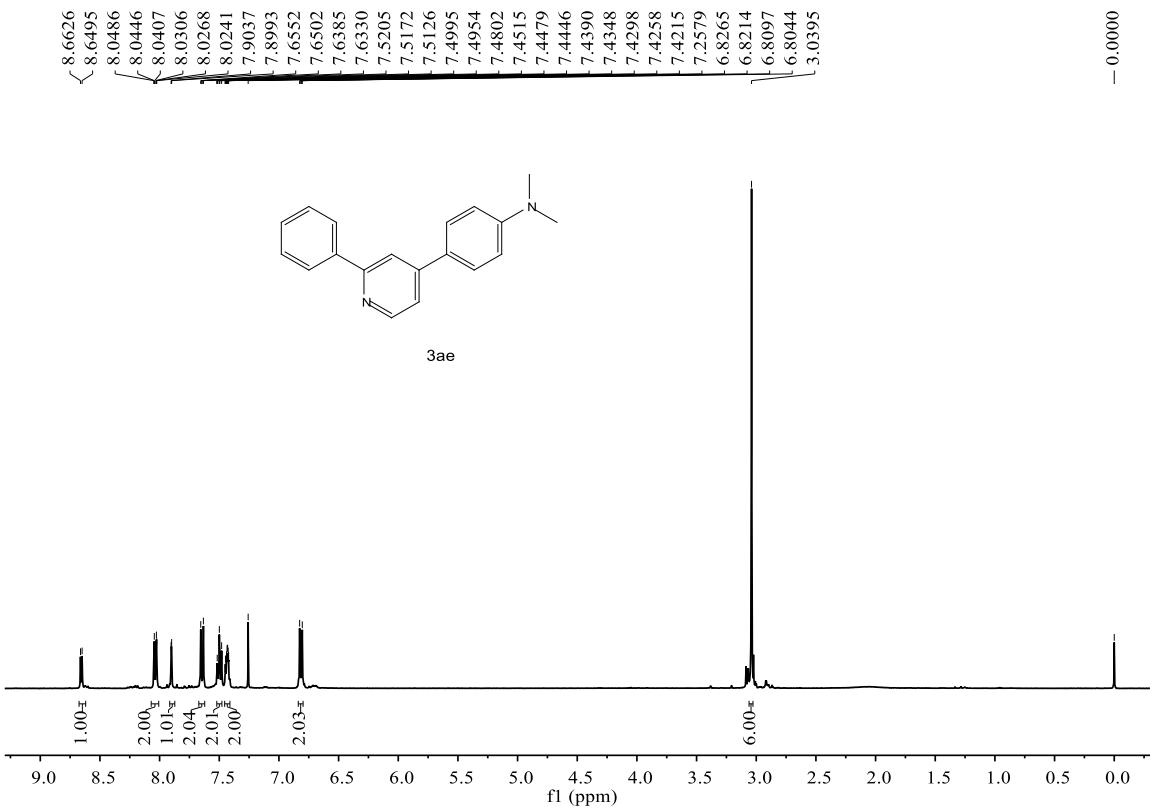
119.6467
118.1566
114.4824
77.3178
77.0000
76.6815
55.3303
3ad
f1 (ppm)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8.0407
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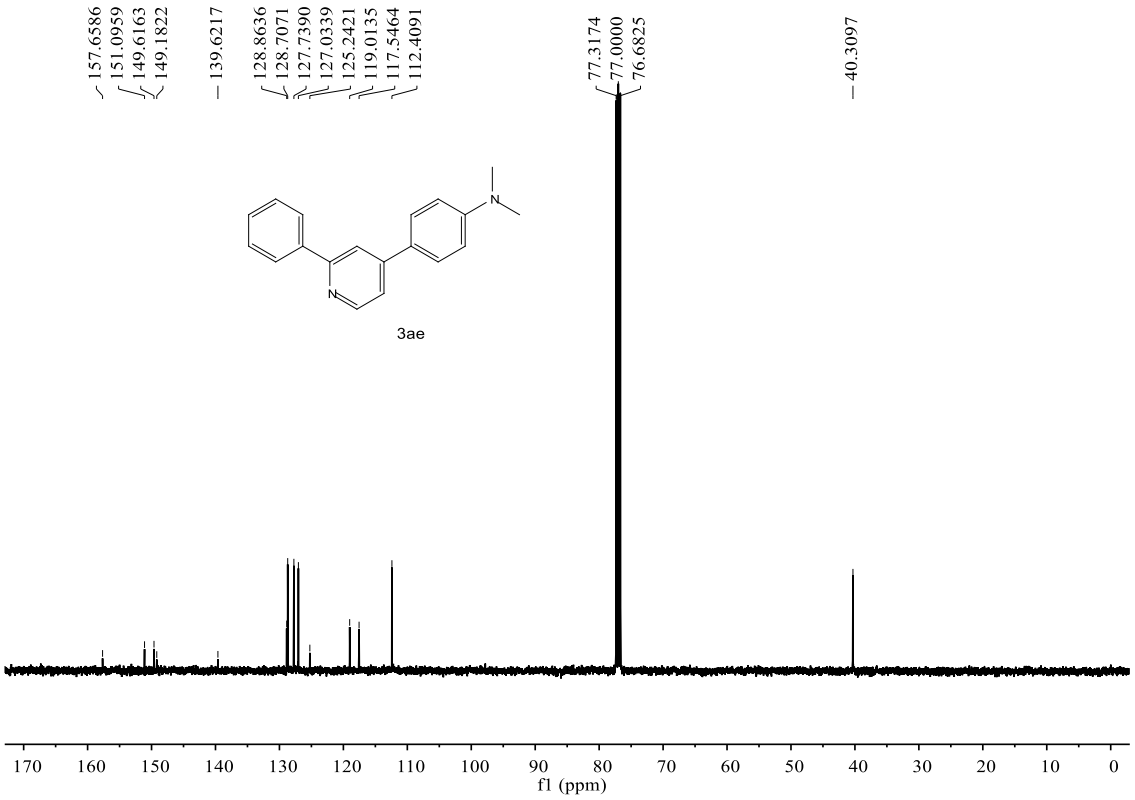
8.0268
8.0241
7.9037
7.8993
7.6552
7.6502
7.6385
7.6330
7.5205
7.5172
7.5126
7.4995
7.4954
7.4802
7.4515
7.4479
7.4446
7.4390
7.4348
7.4298
7.4258
7.4215
7.2579
6.8265
6.8214
6.8097
6.8044
3.0395
0.0000
N
N
3ae
1.00
2.00
1.01
2.04
2.01
2.00
2.03
6.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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127.0339
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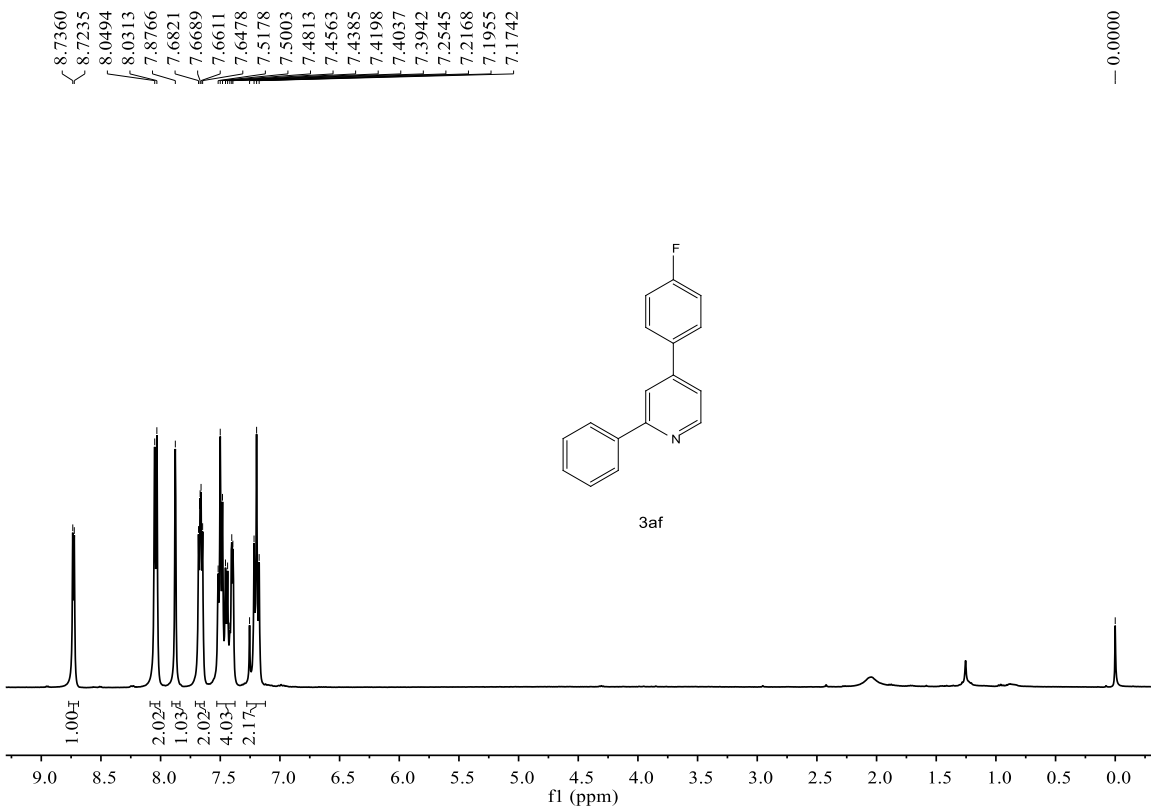
119.0135
117.5464
112.4091
77.3174
77.0000
76.6825
40.3097
N
N
3ae
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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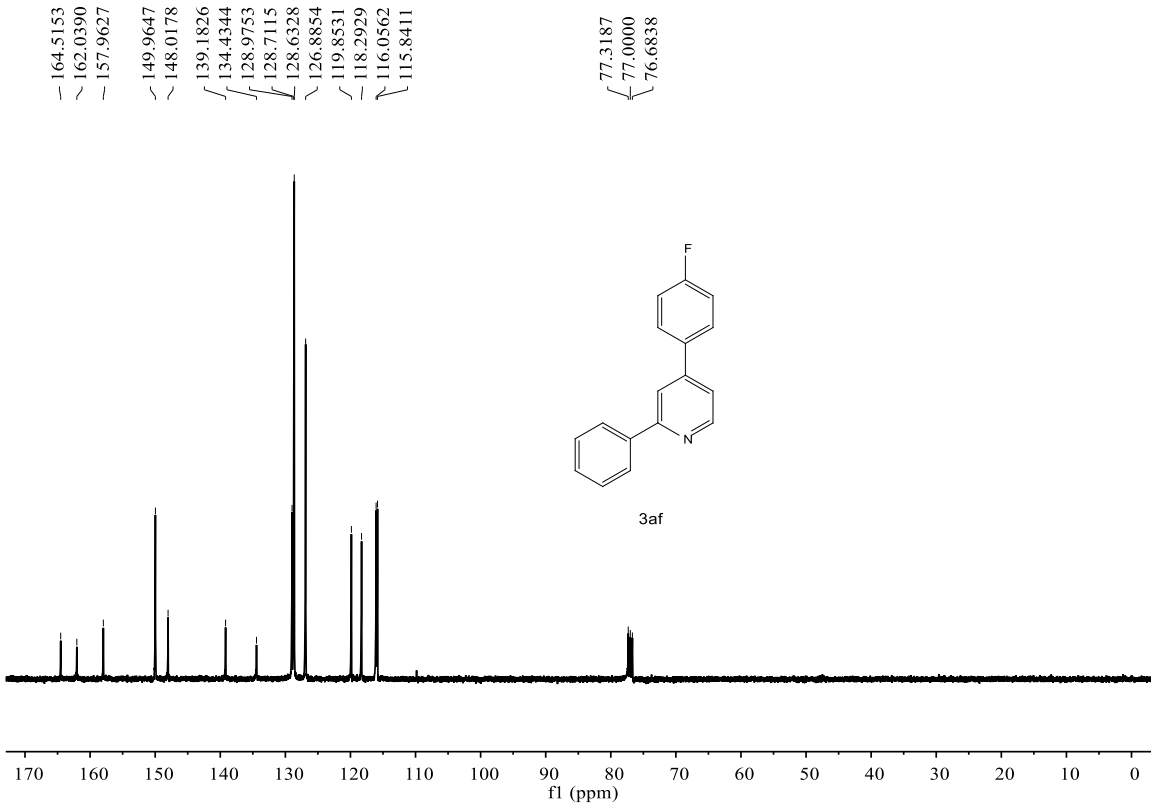
8.7235
8.0494
8.0313
7.8766
7.6821
7.6689
7.6611
7.6478
7.5178
7.5003
7.4813
7.4563
7.4385
7.4198
7.4037
7.3942
7.2545
7.2168
7.1955
7.1742
0.0000
F
N
3af
1.00
2.02
1.03
2.02
4.03
2.17
f1 (ppm)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139.1826
134.4344
128.9753
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128.6328
126.8854
119.8531
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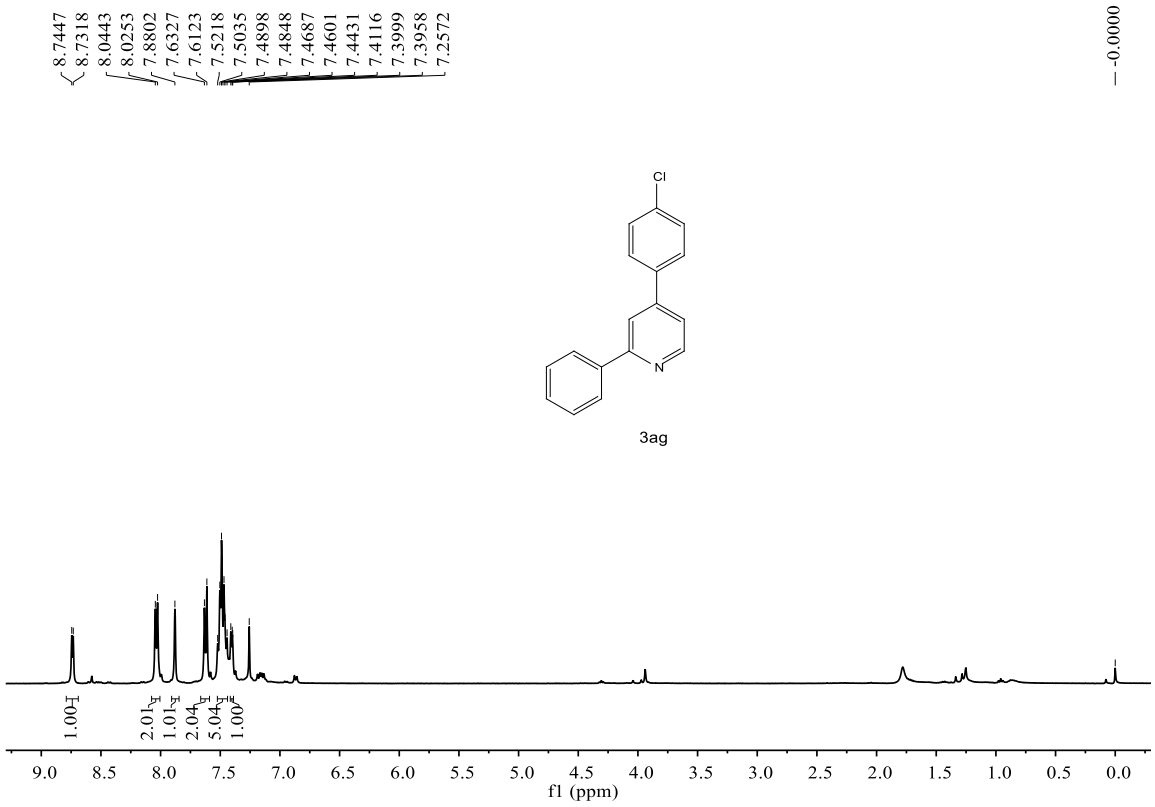
116.0562
115.8411
77.3187
77.0000
76.6838
F
N
3af
f1 (ppm)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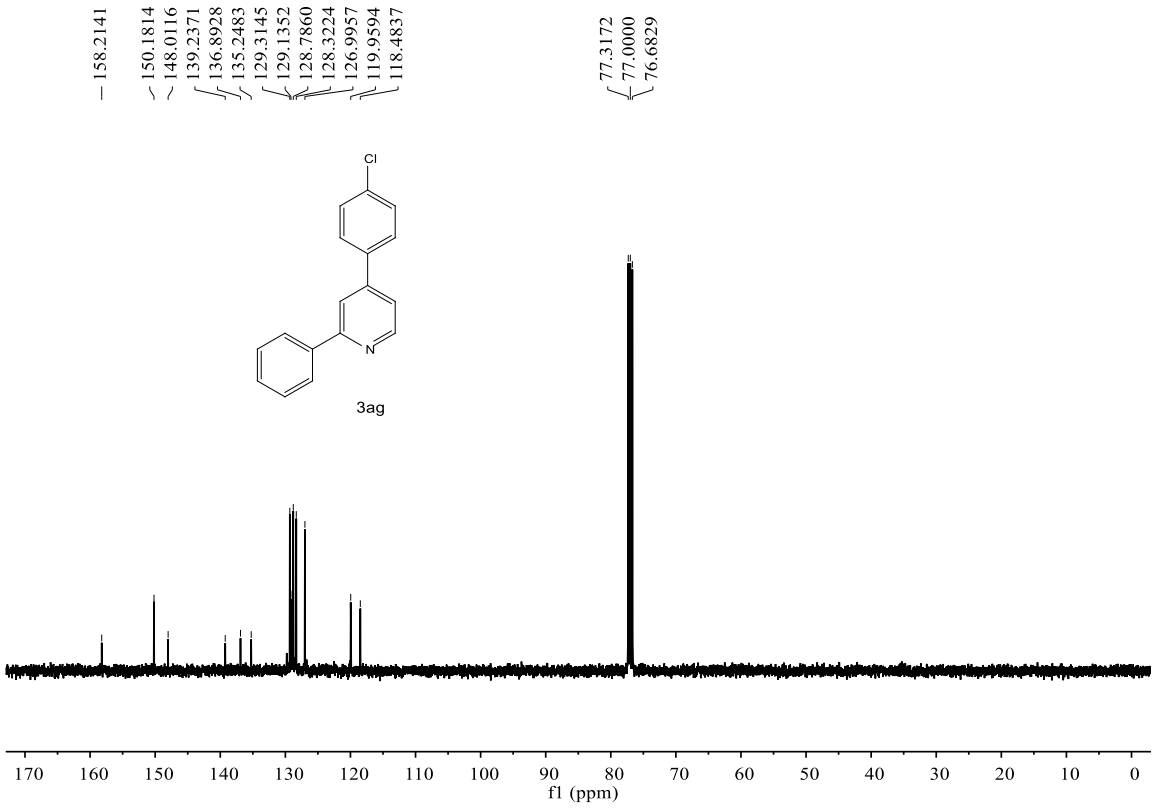
8.0253
7.8802
7.6327
7.6123
7.5218
7.5035
7.4898
7.4848
7.4687
7.4601
7.4431
7.4116
7.3999
7.3958
7.2572
-0.0000
Cl
N
3ag
1.00
2.01
1.01
2.04
5.04
1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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148.0116
139.2371
136.8928
135.2483
129.3145
129.1352
128.7860
128.3224
126.9957
119.9594
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Cl
N
3ag
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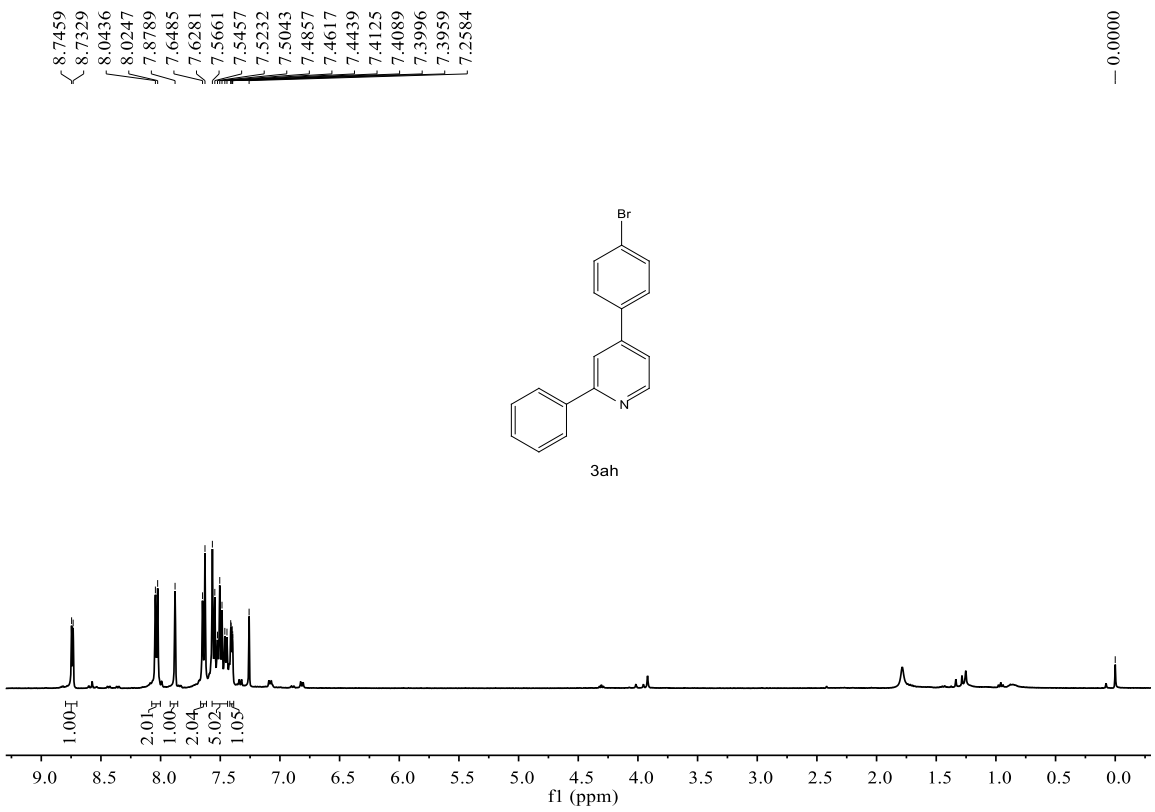
f1 (ppm)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8.0247
7.8789
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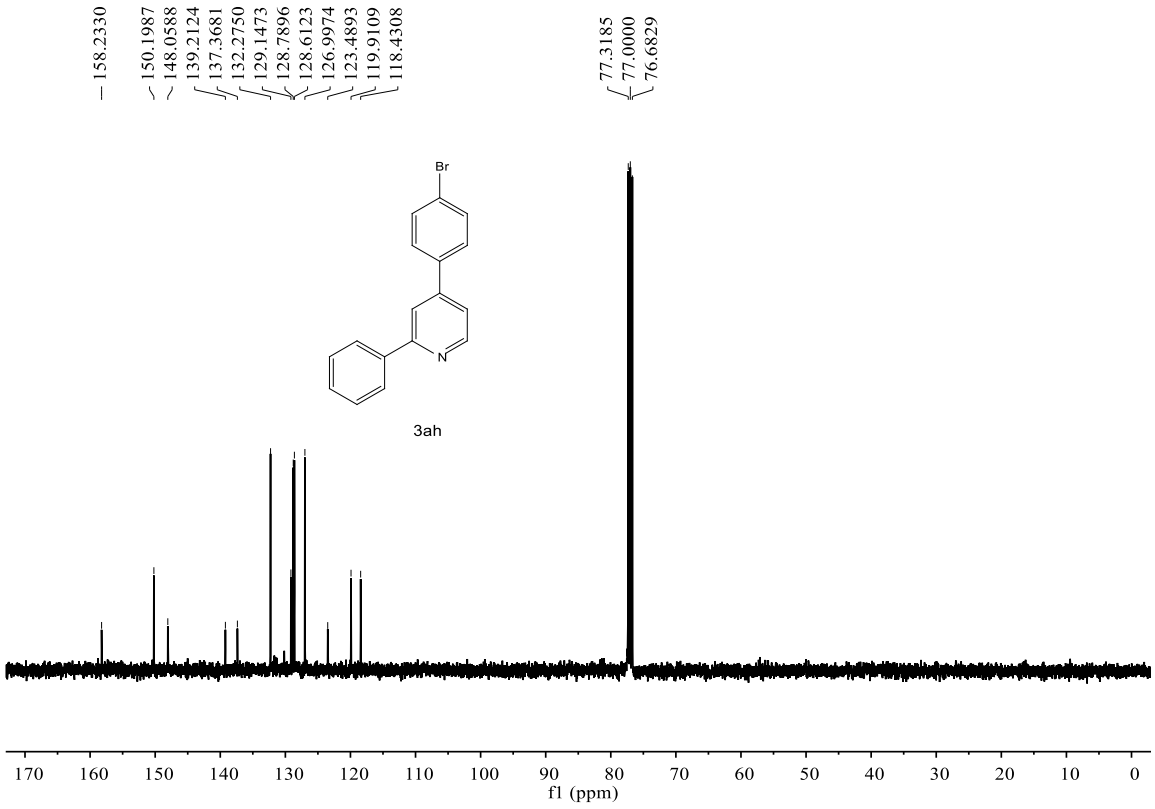
7.5457
7.5232
7.5043
7.4857
7.4617
7.4439
7.4125
7.4089
7.3996
7.3959
7.2584
0.0000
Br
N
3ah
1.00
2.01
1.00
2.04
5.02
1.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.1473
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128.6123
126.9974
123.4893
119.9109
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77.0000
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Br
N
3ah
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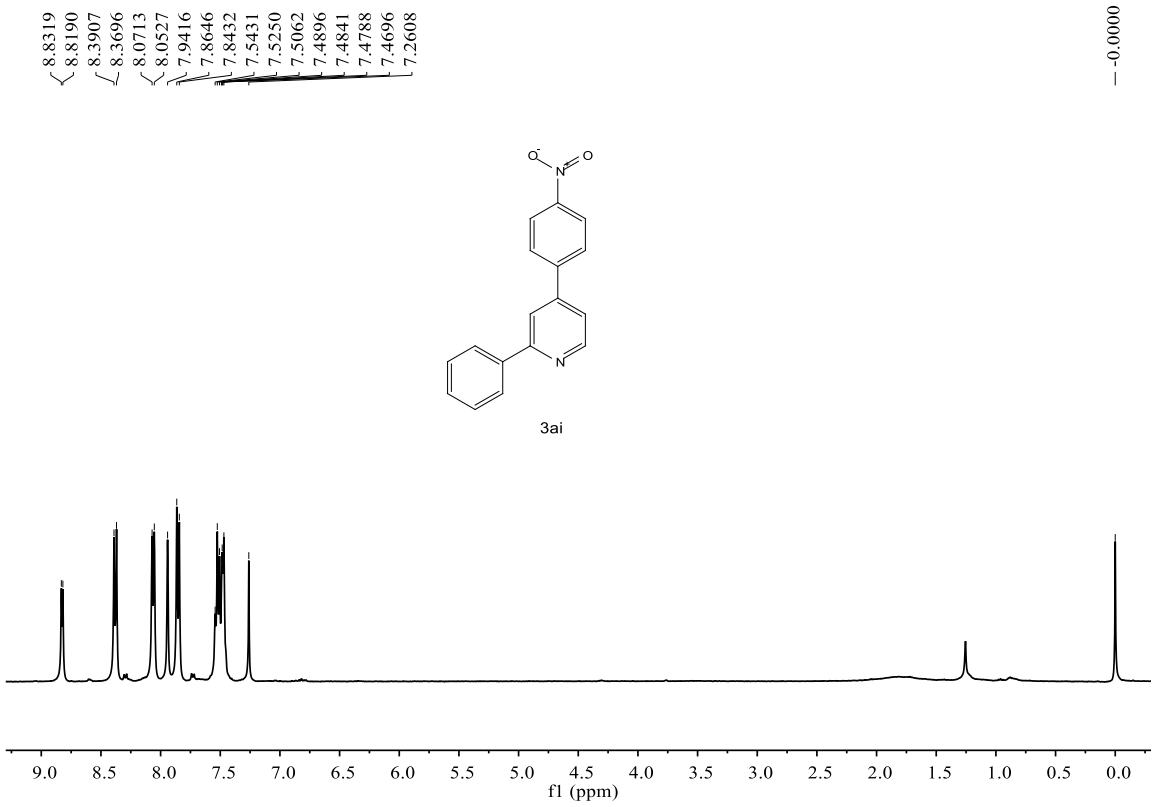
f1 (ppm)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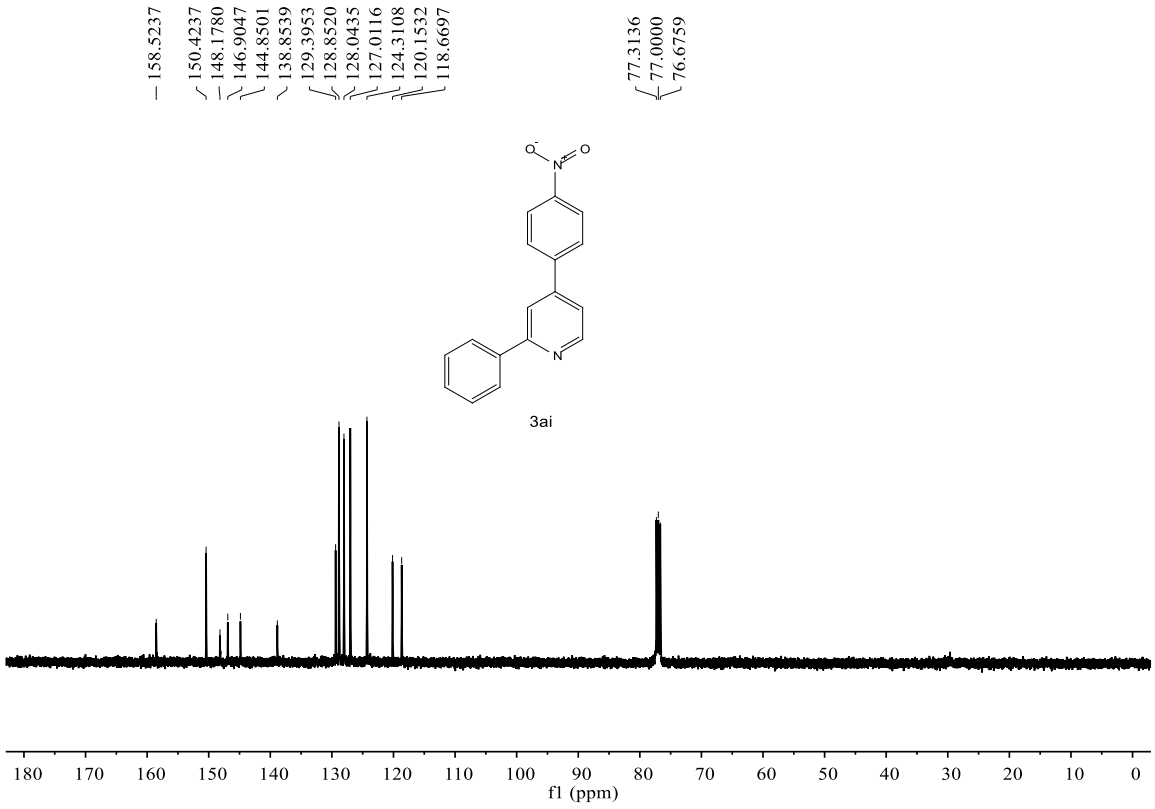
8.0713
8.0527
7.9416
7.8646
7.8432
7.5431
7.5250
7.5062
7.4896
7.4841
7.4788
7.4696
7.2608
-0.0000
3ai
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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127.0116
124.3108
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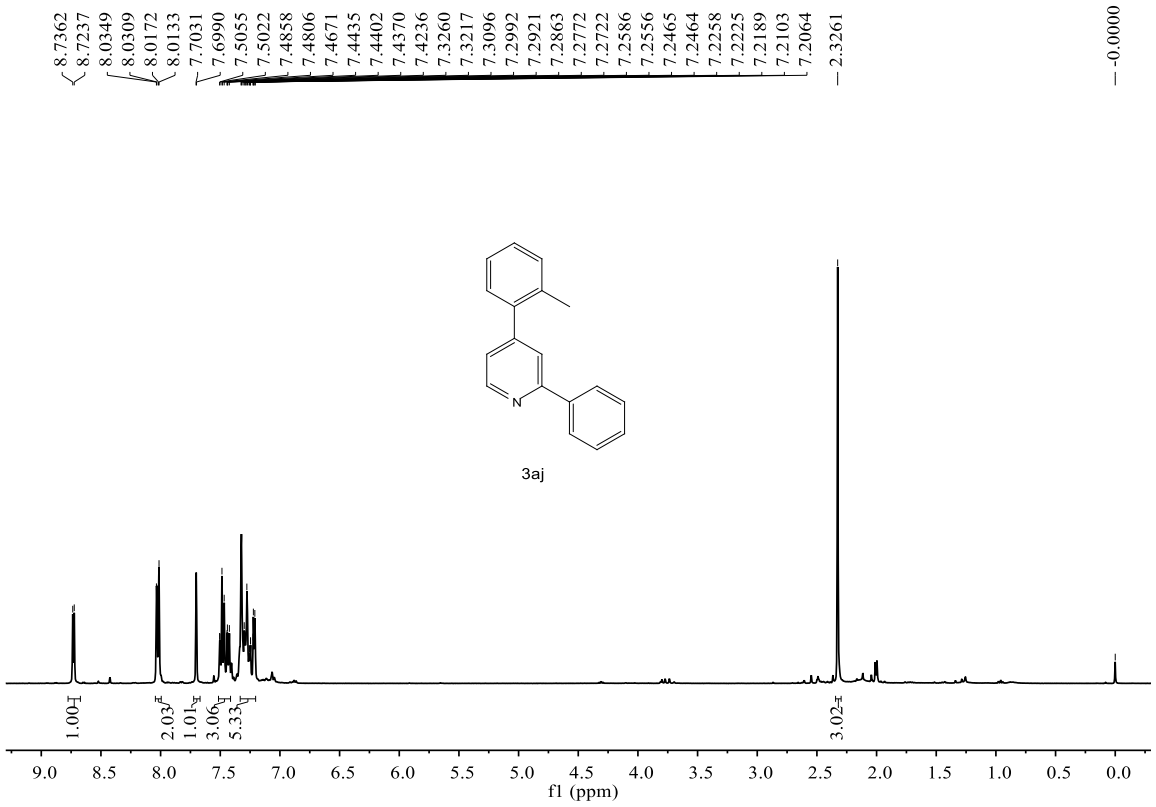
f1 (ppm)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7.5022
7.4858
7.4806
7.4671
7.4435
7.4402
7.4370
7.4236
7.3260
7.3217
7.3096
7.2992
7.2921
7.2863
7.2772
7.2722
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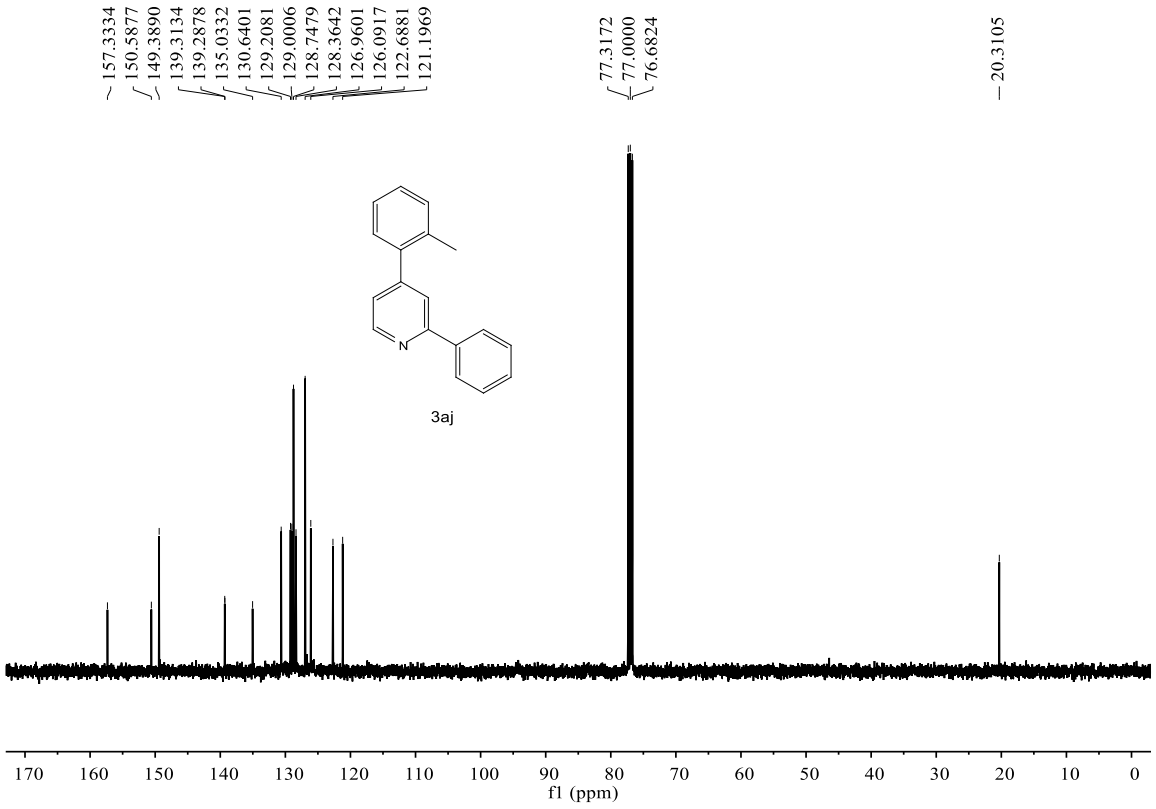
7.2556
7.2465
7.2464
7.2258
7.2225
7.2189
7.2103
7.2064
2.3261
-0.0000
3aj
1.00
2.03
1.01
3.06
5.33
3.02
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.0006
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126.0917
122.6881
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77.0000
76.6824
20.3105
3aj
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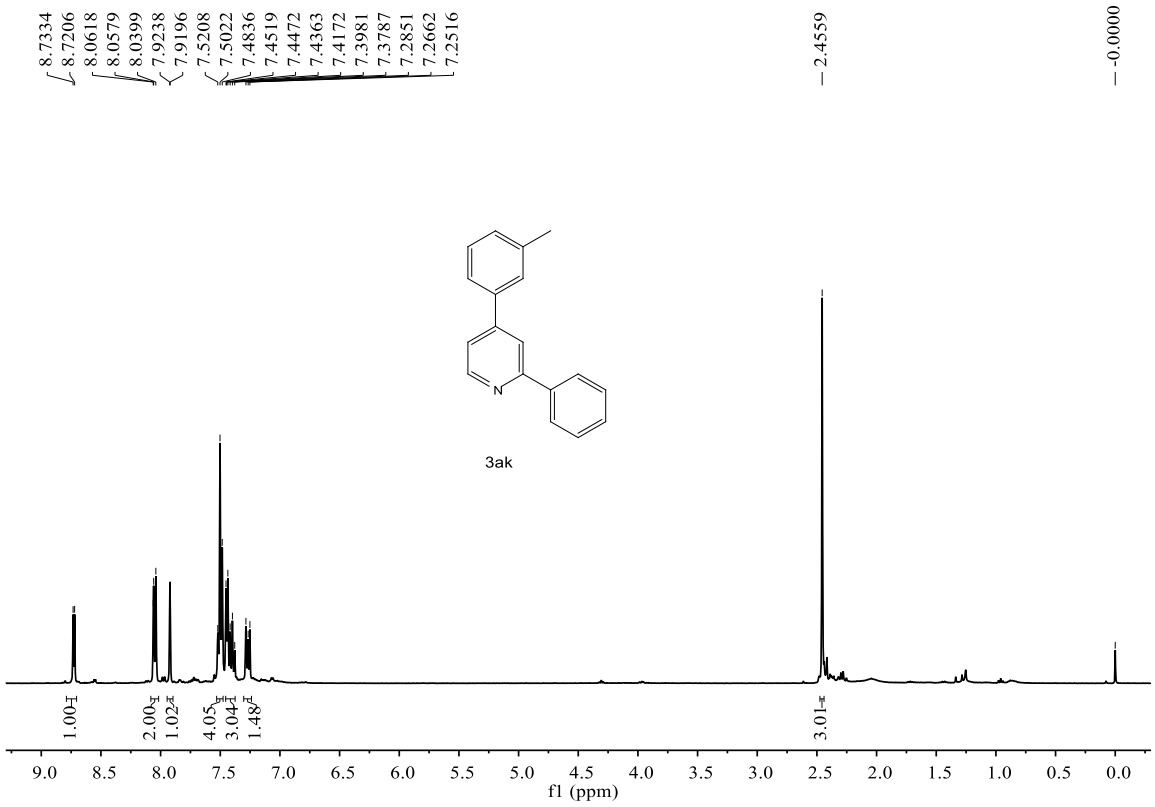
f1 (ppm)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8.0579
8.0399
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7.9196
7.5208
7.5022
7.4836
7.4519
7.4472
7.4363
7.4172
7.3981
7.3787
7.2851
7.2662
7.2516
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2.00
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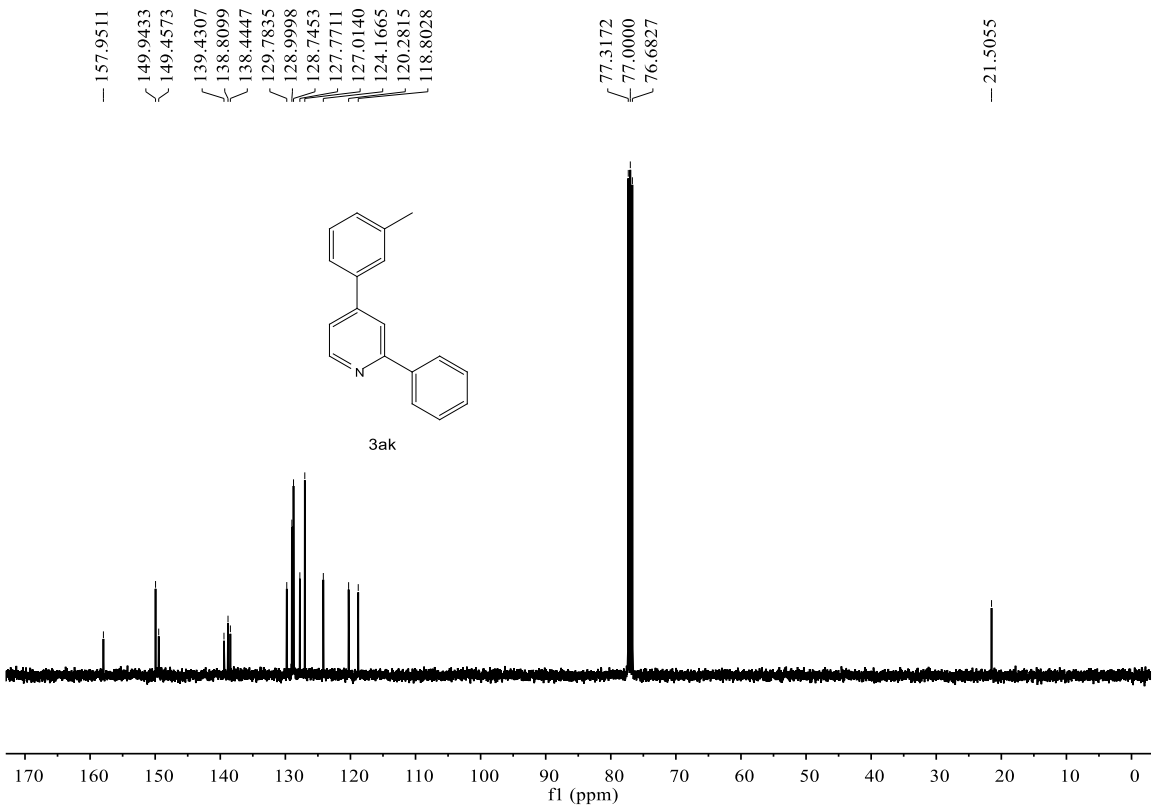
4.05
3.04
1.48
3.01
f1 (ppm)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76.6827
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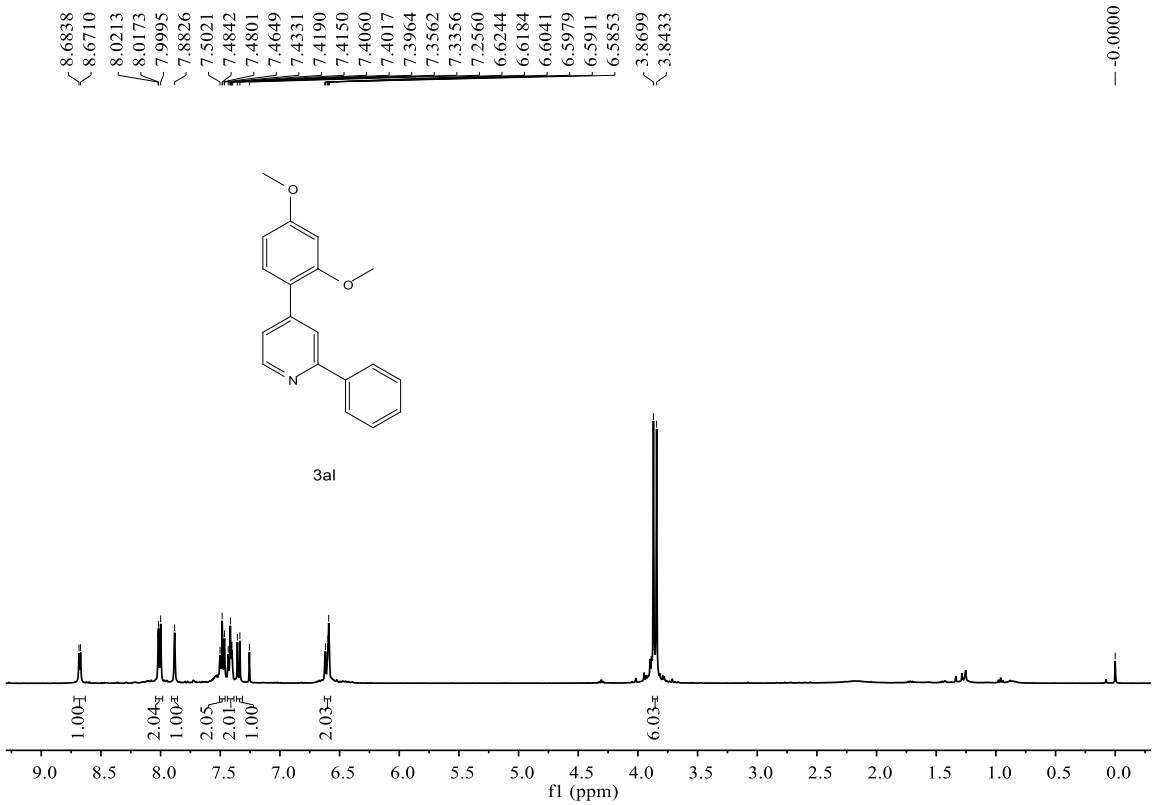
f1 (ppm)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7.4190
7.4150
7.4060
7.4017
7.3964
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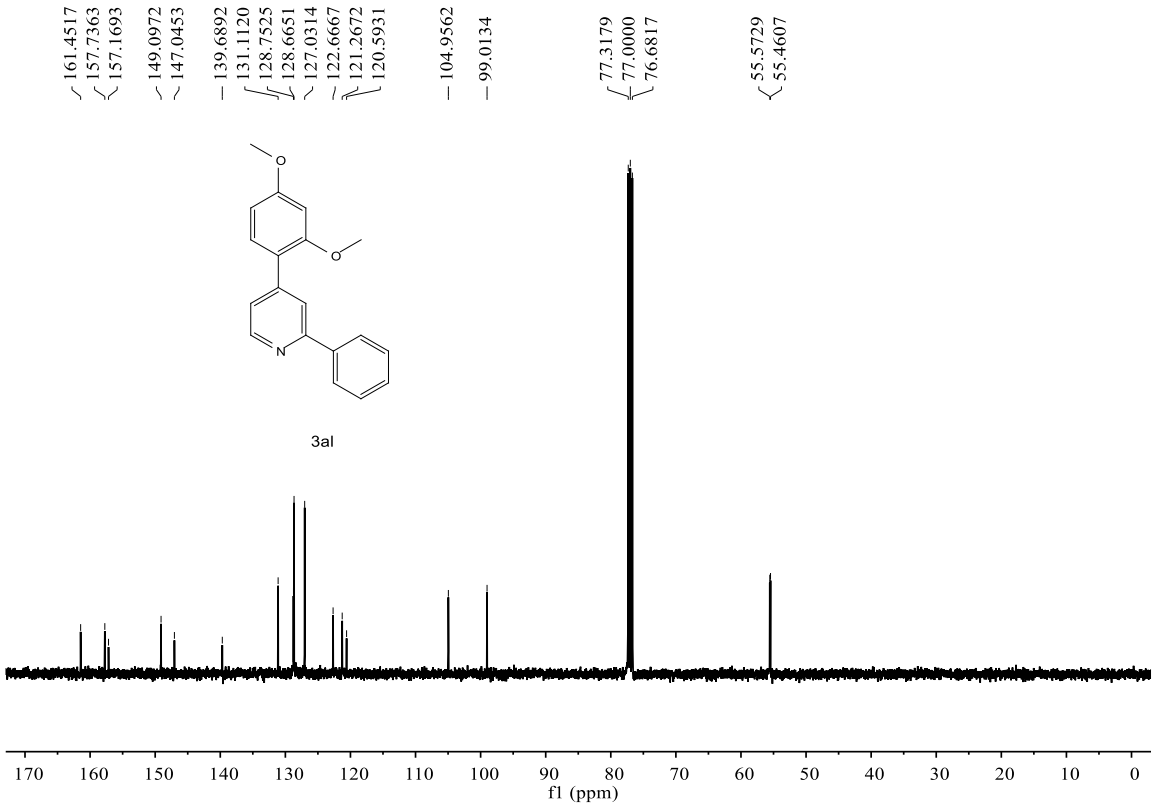
7.3356
7.2560
6.6244
6.6184
6.6041
6.5979
6.5911
6.5853
3.8699
3.8433
-0.0000
O
O
N
3al
1.00
2.04
1.00
2.05
2.01
1.00
2.03
6.03
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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157.1693
149.0972
147.0453
139.6892
131.1120
128.7525
128.6651
127.0314
122.6667
121.2672
120.5931
104.9562
99.0134
77.3179
77.0000
76.6817
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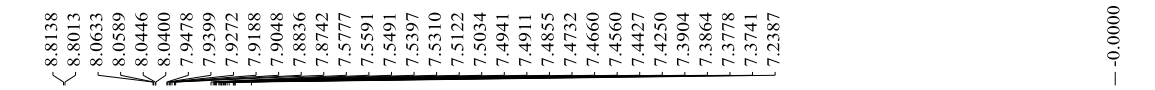
55.4607
O
O
N
3al
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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8.0446
8.0400
7.9478
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7.9272
7.9188
7.9048
7.8836
7.8742
7.5777
7.5591
7.5491
7.5397
7.5310
7.5122
7.5034
7.4941
7.4911
7.4855
7.4732
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7.4250
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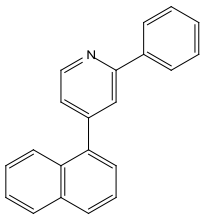
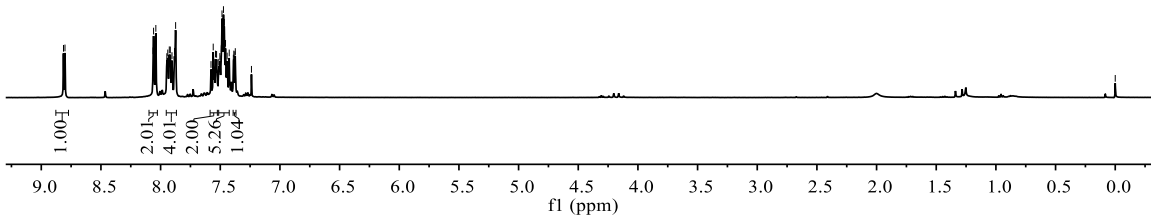
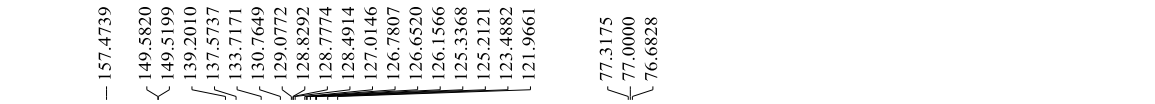
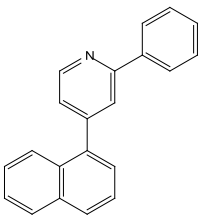

N

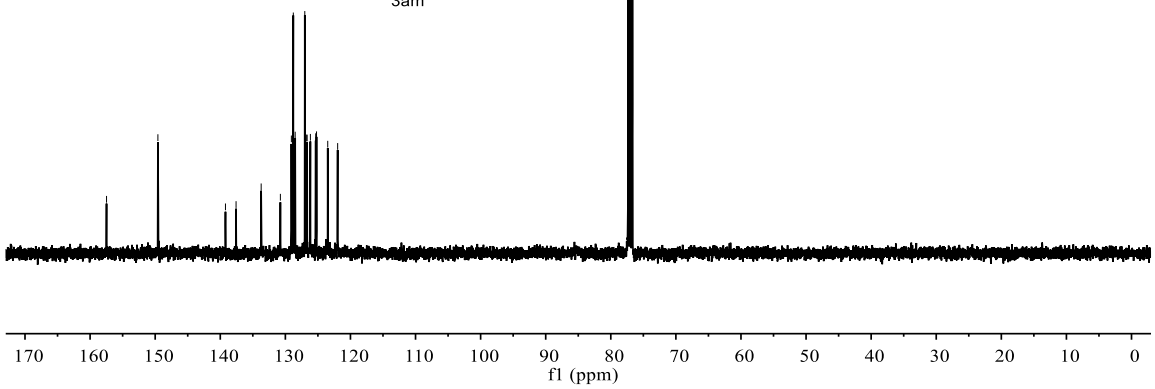
3am

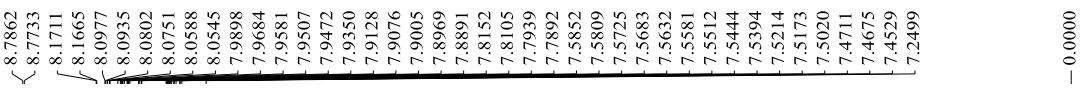

1.00
2.01
4.01
2.00
5.26
1.04
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

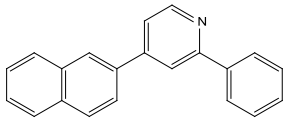

157.4739
149.5820
149.5199
139.2010
137.5737
133.7171
130.7649
129.0772
128.8292
128.7774
128.4914
127.0146
126.7807
126.6520
126.1566
125.3368
125.2121
123.4882
121.9661
77.3175
77.0000
76.6828


N

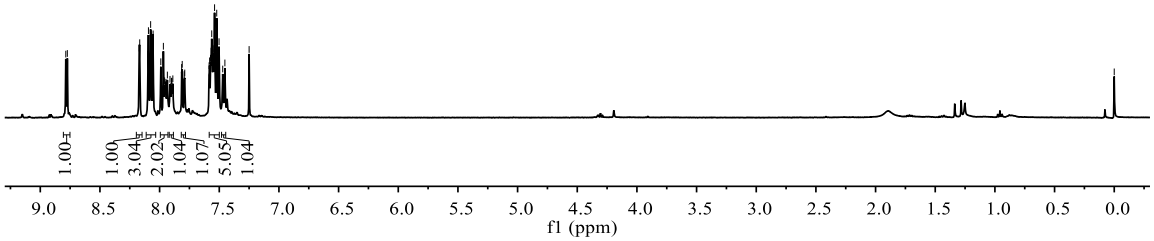
3am

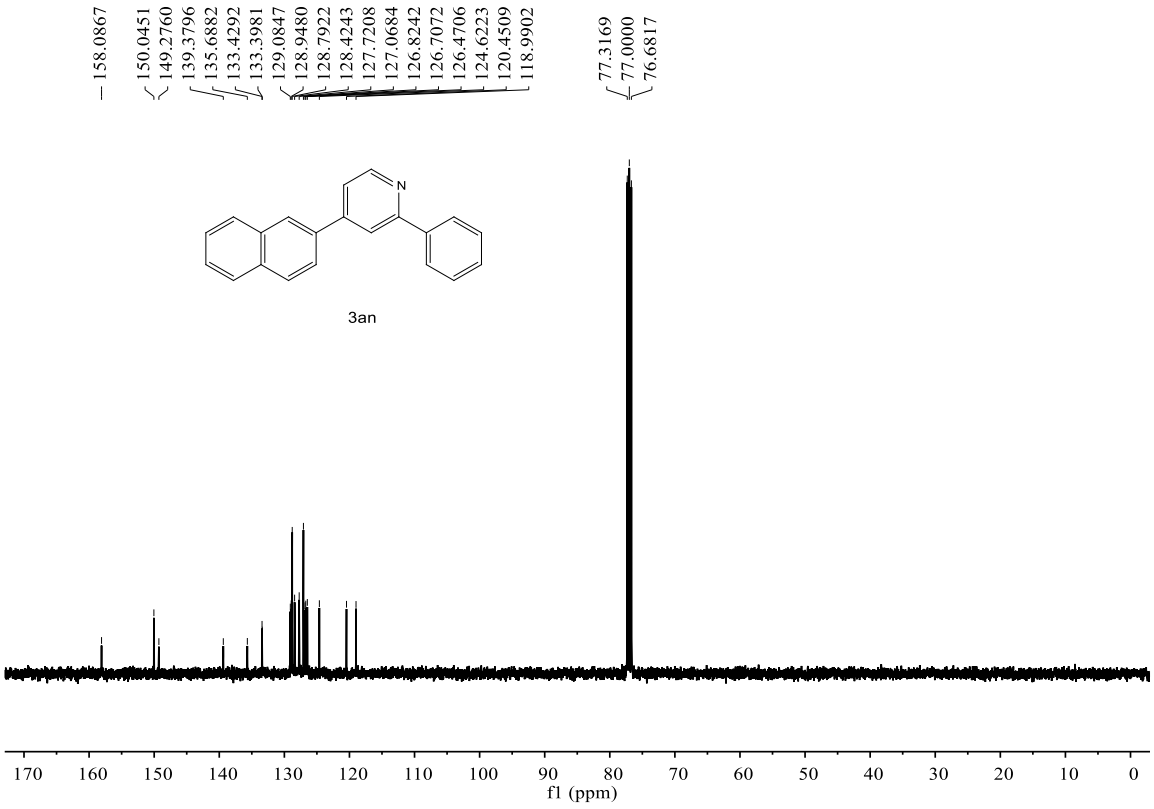

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.7862
8.7733
8.1711
8.1665
8.0977
8.0935
8.0802
8.0751
8.0588
8.0545
7.9898
7.9684
7.9581
7.9507
7.9472
7.9350
7.9128
7.9076
7.9005
7.8969
7.8891
7.8152
7.8105
7.7939
7.7892
7.5852
7.5809
7.5725
7.5683
7.5632
7.5581
7.5512
7.5444
7.5394
7.5214
7.5173
7.5020
7.4711
7.4675
7.4529
7.2499
0.0000


N

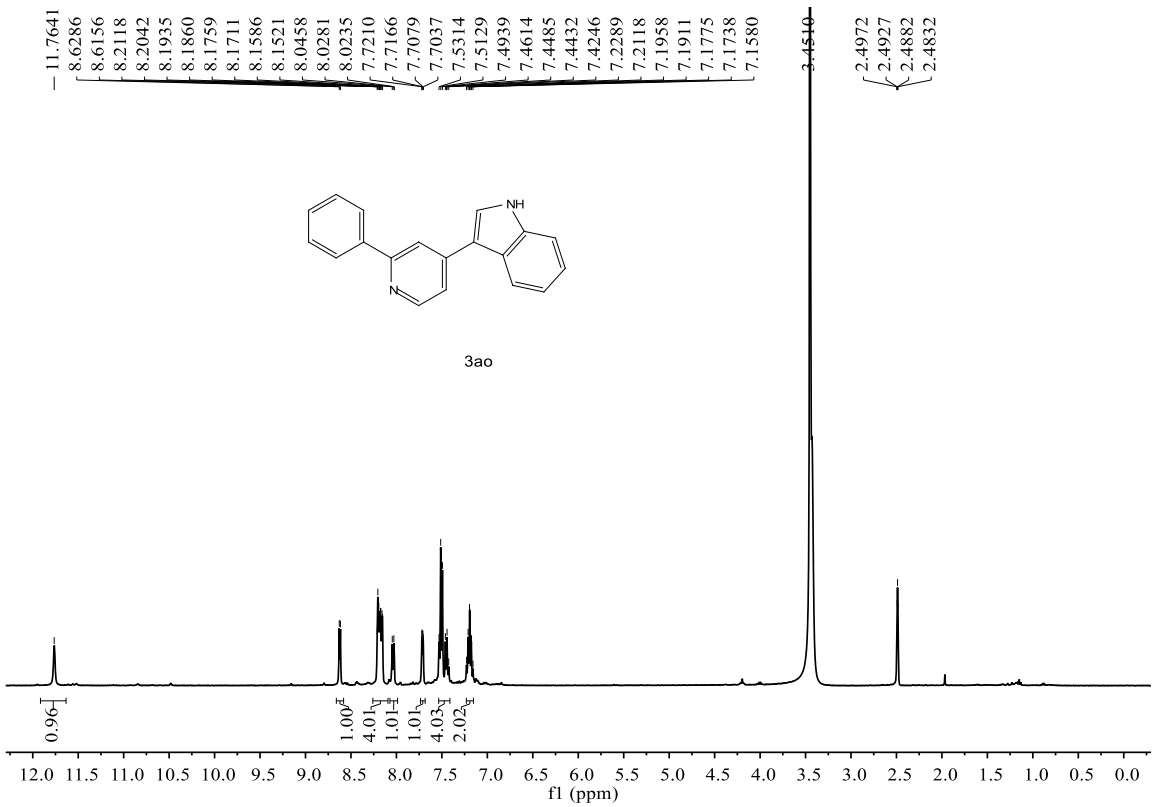
3an

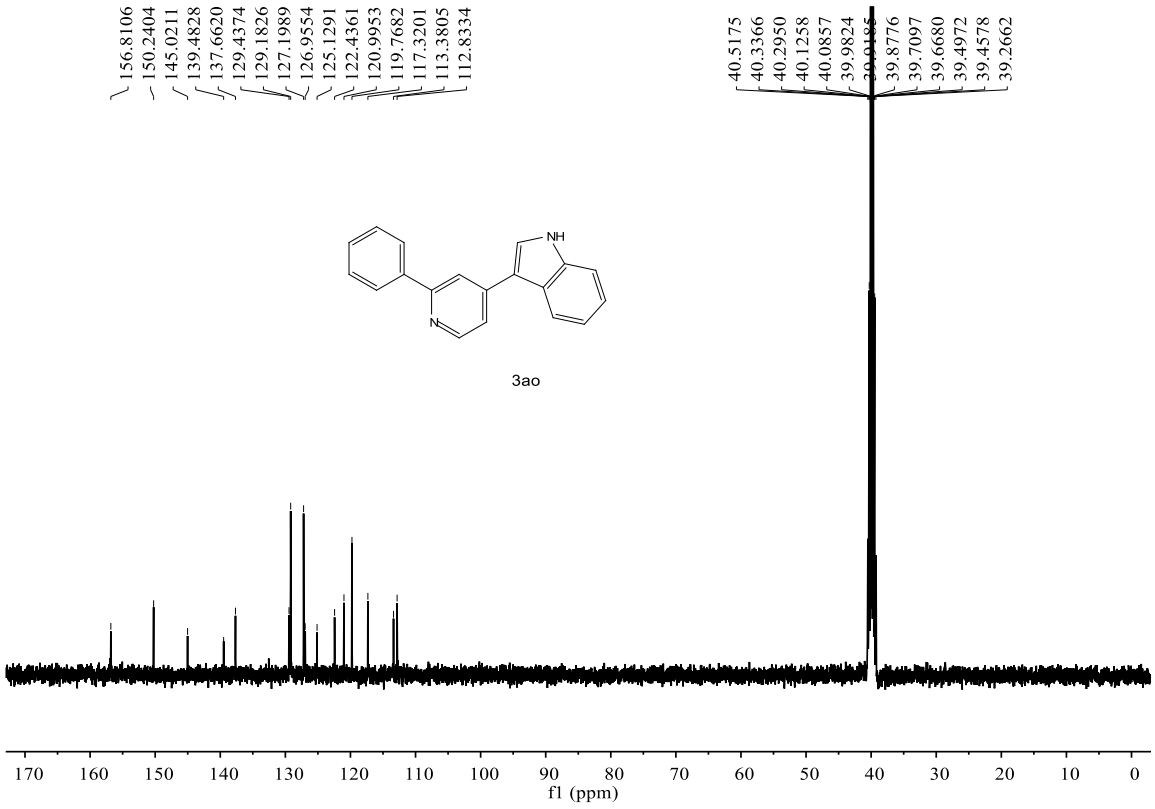

1.00
1.00
3.04
2.02
1.04
1.07
5.05
1.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

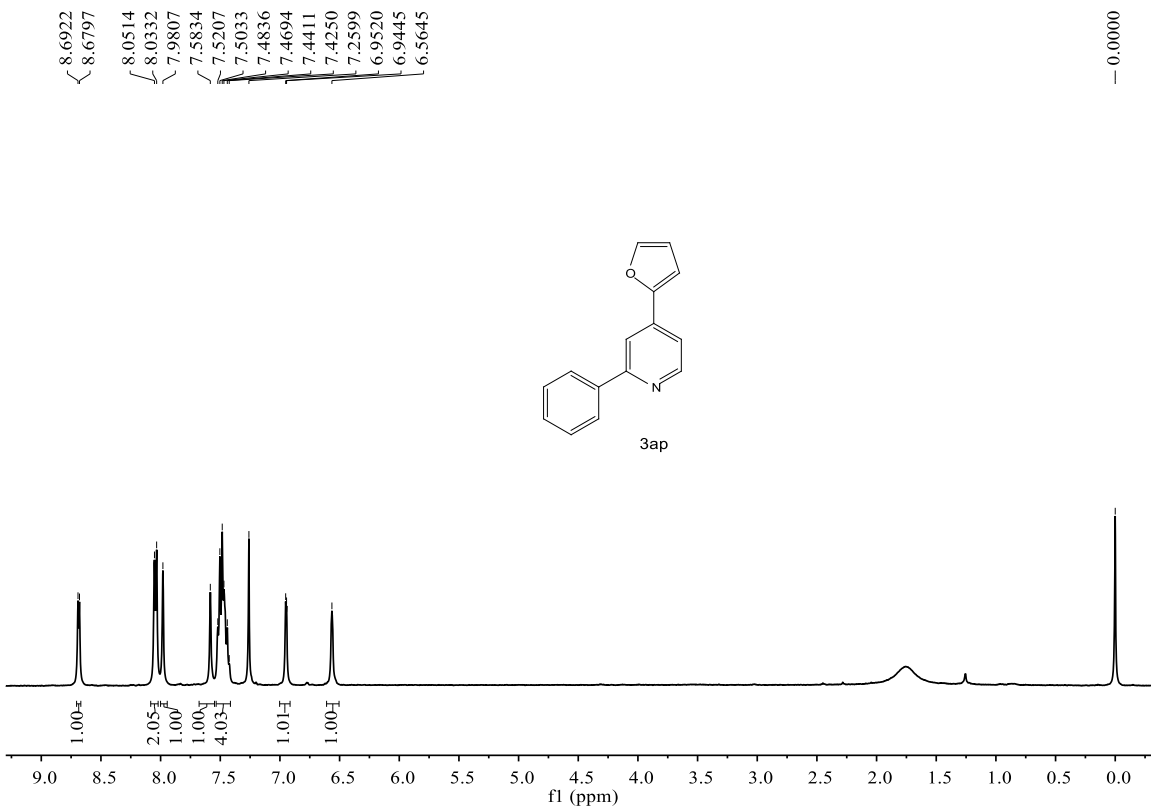

158.0867
150.0451
149.2760
139.3796
135.6882
133.4292
133.3981
129.0847
128.9480
128.7922
128.4243
127.7208
127.0684
126.8242
126.7072
126.4706
124.6223
120.4509
118.9902
77.3169
77.0000
76.6817
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

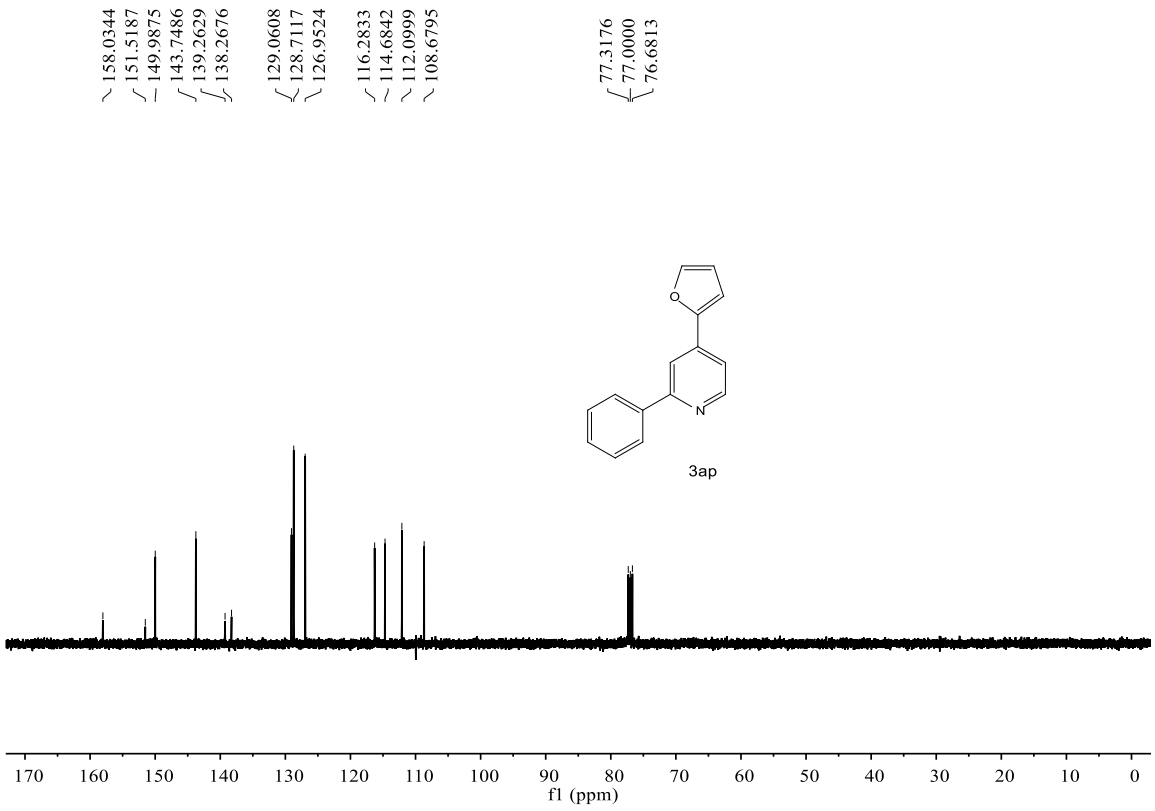
N

3an

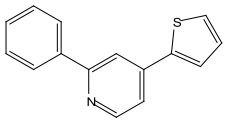

11.7641
8.6286
8.6156
8.2118
8.2042
8.1935
8.1860
8.1759
8.1711
8.1586
8.1521
8.0458
8.0281
8.0235
7.7210
7.7166
7.7079
7.7037
7.5314
7.5129
7.4939
7.4614
7.4485
7.4432
7.4246
7.2289
7.2118
7.1958
7.1911
7.1775
7.1738
7.1580
3.4510
2.4972
2.4927
2.4882
2.4832
NH
N
3ao
0.96
1.00
4.01
1.01
1.01
4.03
2.02
f1 (ppm)


156.8106
150.2404
145.0211
139.4828
137.6620
129.4374
129.1826
127.1989
126.9554
125.1291
122.4361
120.9953
119.7682
117.3201
113.3805
112.8334
40.5175
40.3366
40.2950
40.1258
40.0857
39.9824
39.8776
39.7097
39.6680
39.4972
39.4578
39.2662
NH
N
3ao
f1 (ppm)

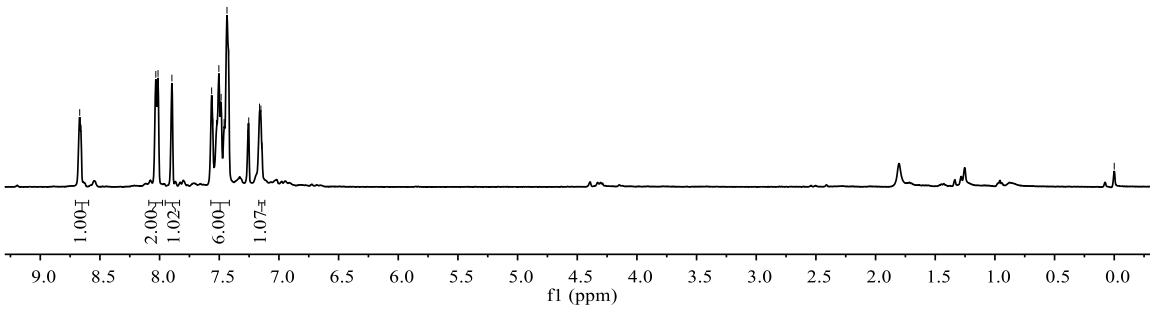

8.6922
8.6797
8.0514
8.0332
7.9807
7.5834
7.5207
7.5033
7.4836
7.4694
7.4411
7.4250
7.2599
6.9520
6.9445
6.5645
0.0000
O
N
3ap
1.00
2.05
1.00
1.00
4.03
1.01
1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

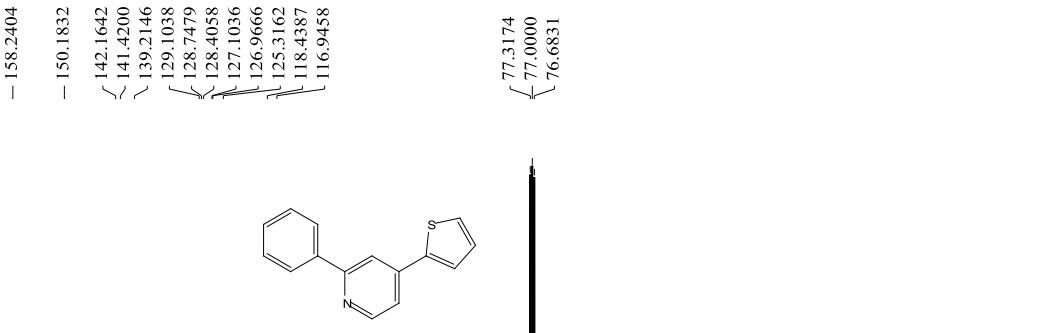

158.0344
151.5187
149.9875
143.7486
139.2629
138.2676
129.0608
128.7117
126.9524
116.2833
114.6842
112.0999
108.6795
77.3176
77.0000
76.6813
O
N
3ap
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.6695
8.6583
8.0318
8.0133
7.8970
7.5640
7.5541
7.5233
7.5034
7.4844
7.4595
7.4353
7.4214
7.2605
7.2531
7.1623
7.1513
7.1412
0.0000


S
N

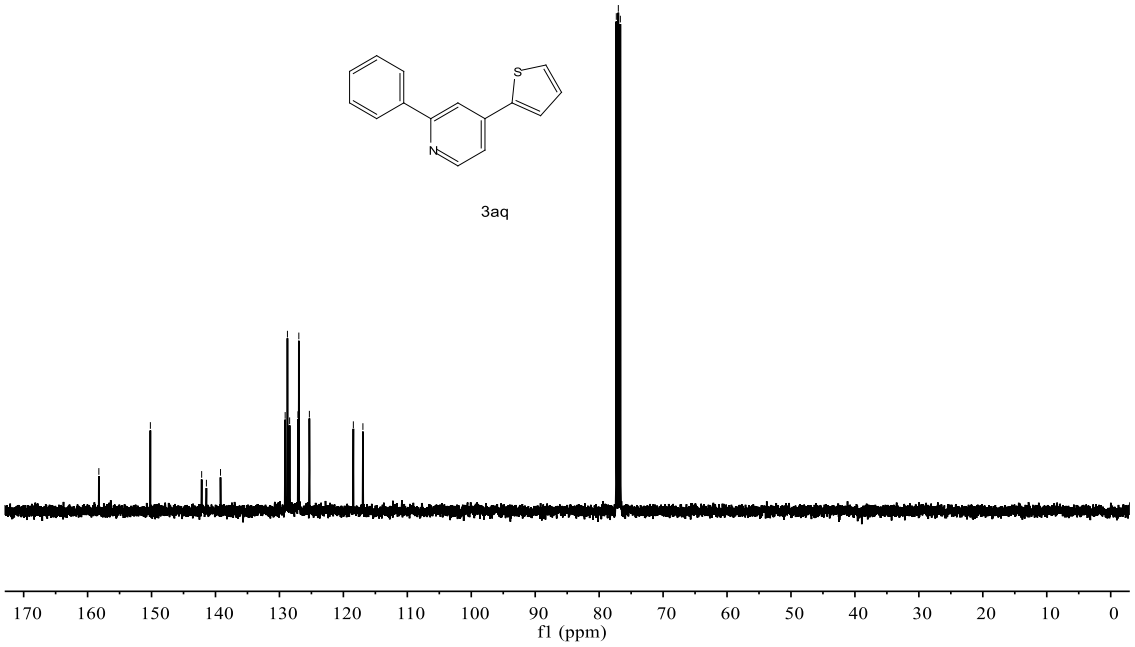
3aq

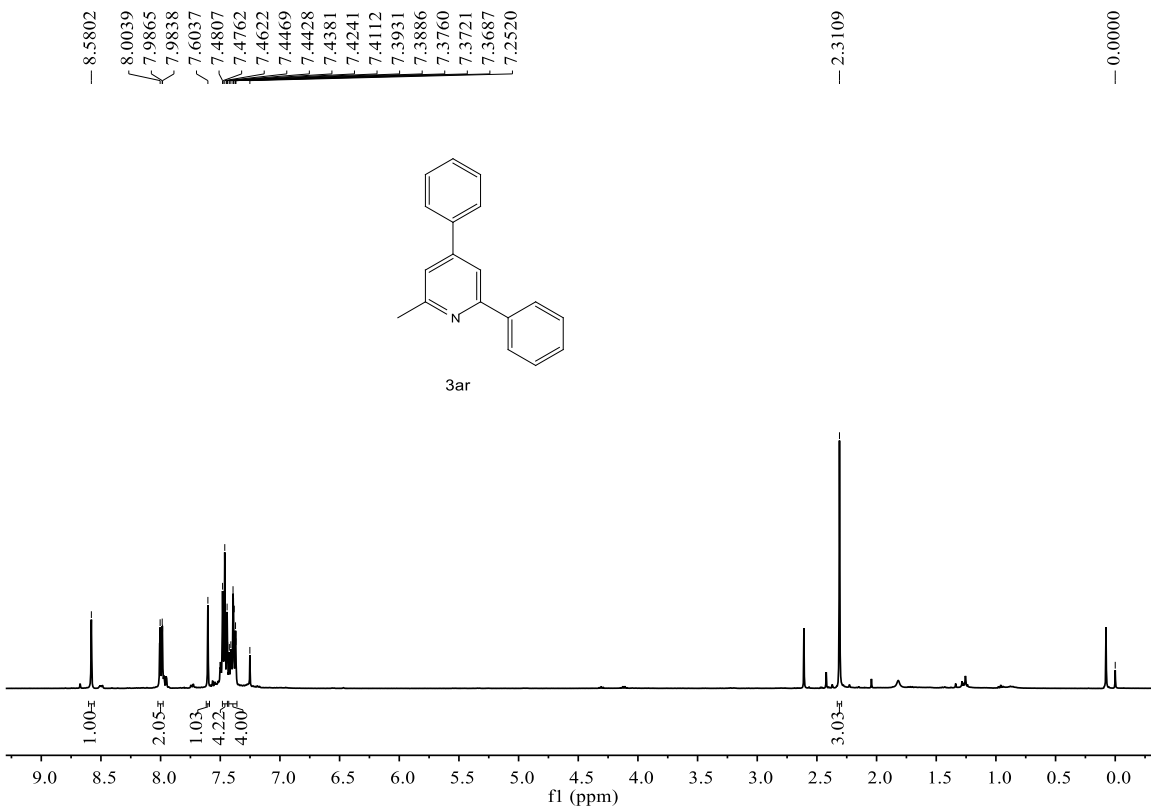

1.00
2.00
1.02
6.00
1.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

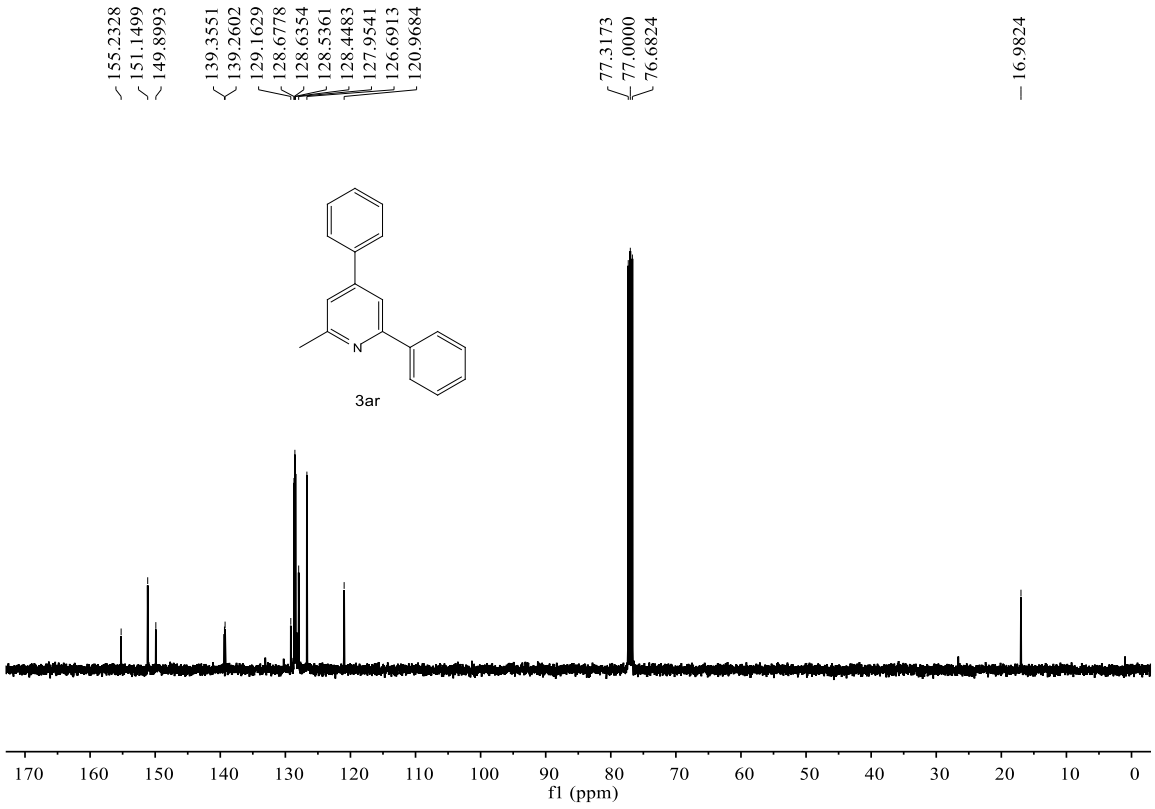

158.2404
150.1832
142.1642
141.4200
139.2146
129.1038
128.7479
128.4058
127.1036
126.9666
125.3162
118.4387
116.9458
77.3174
77.0000
76.6831

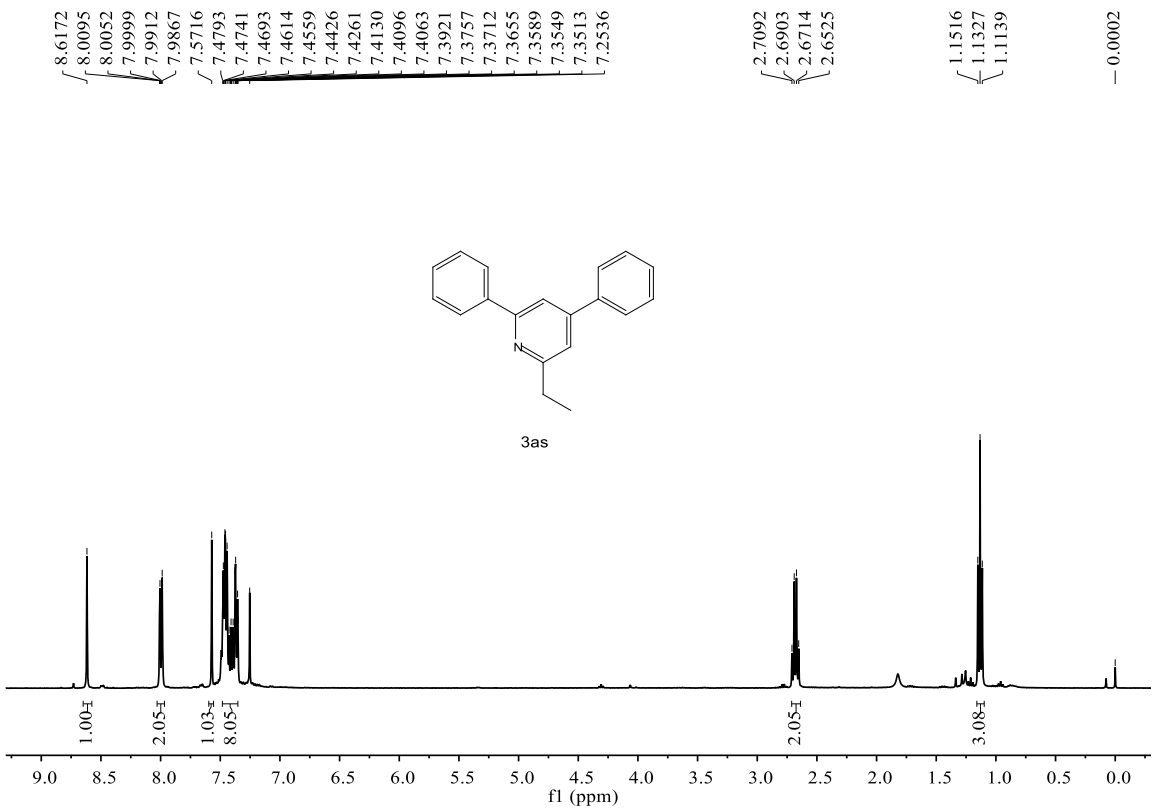
S
N

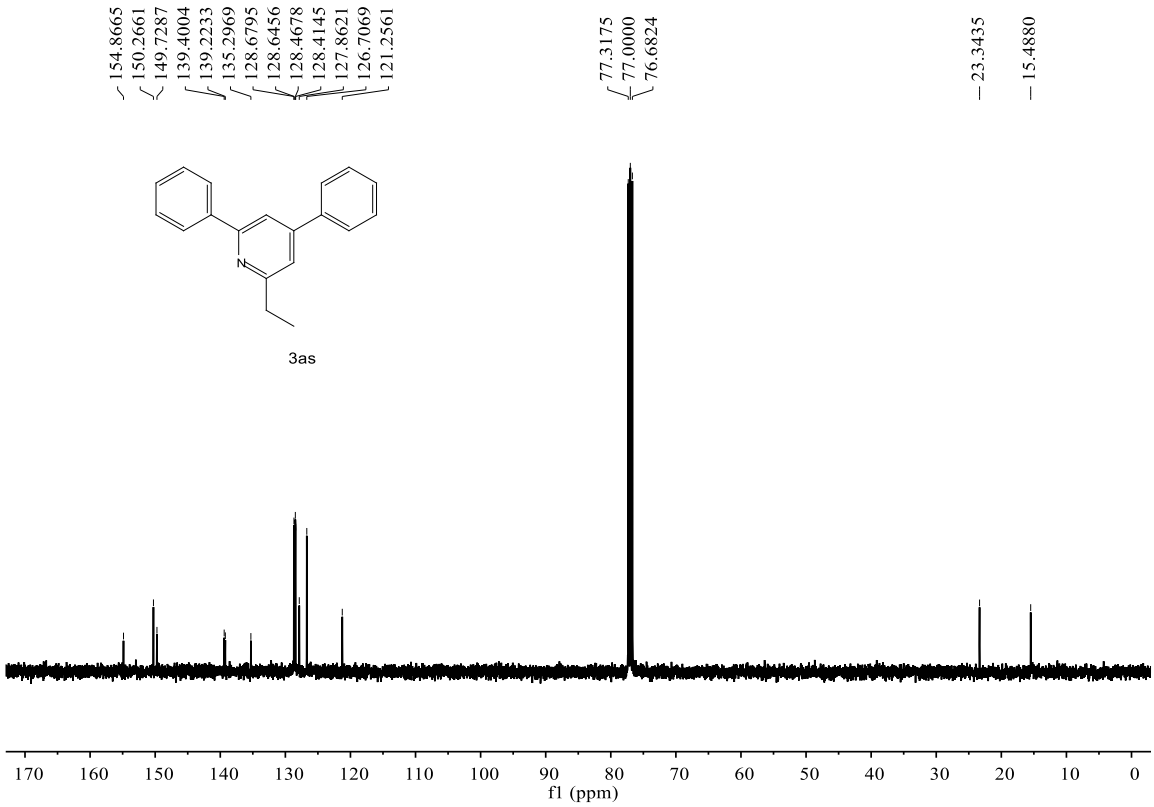
3aq


170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.5802
8.0039
7.9865
7.9838
7.6037
7.4807
7.4762
7.4622
7.4469
7.4428
7.4381
7.4241
7.4112
7.3931
7.3886
7.3760
7.3721
7.3687
7.2520
2.3109
0.0000
3ar
1.00
2.05
1.03
4.22
4.00
3.03
f1 (ppm)


155.2328
151.1499
149.8993
139.3551
139.2602
129.1629
128.6778
128.6354
128.5361
128.4483
127.9541
126.6913
120.9684
77.3173
77.0000
76.6824
16.9824
3ar
f1 (ppm)


8.6172
8.0095
8.0052
7.9999
7.9912
7.9867
7.5716
7.4793
7.4741
7.4693
7.4614
7.4559
7.4426
7.4261
7.4130
7.4096
7.4063
7.3921
7.3757
7.3712
7.3655
7.3589
7.3549
7.3513
7.2536
2.7092
2.6903
2.6714
2.6525
1.1516
1.1327
1.1139
0.0002
3as
1.00
2.05
1.03
8.05
2.05
3.08
f1 (ppm)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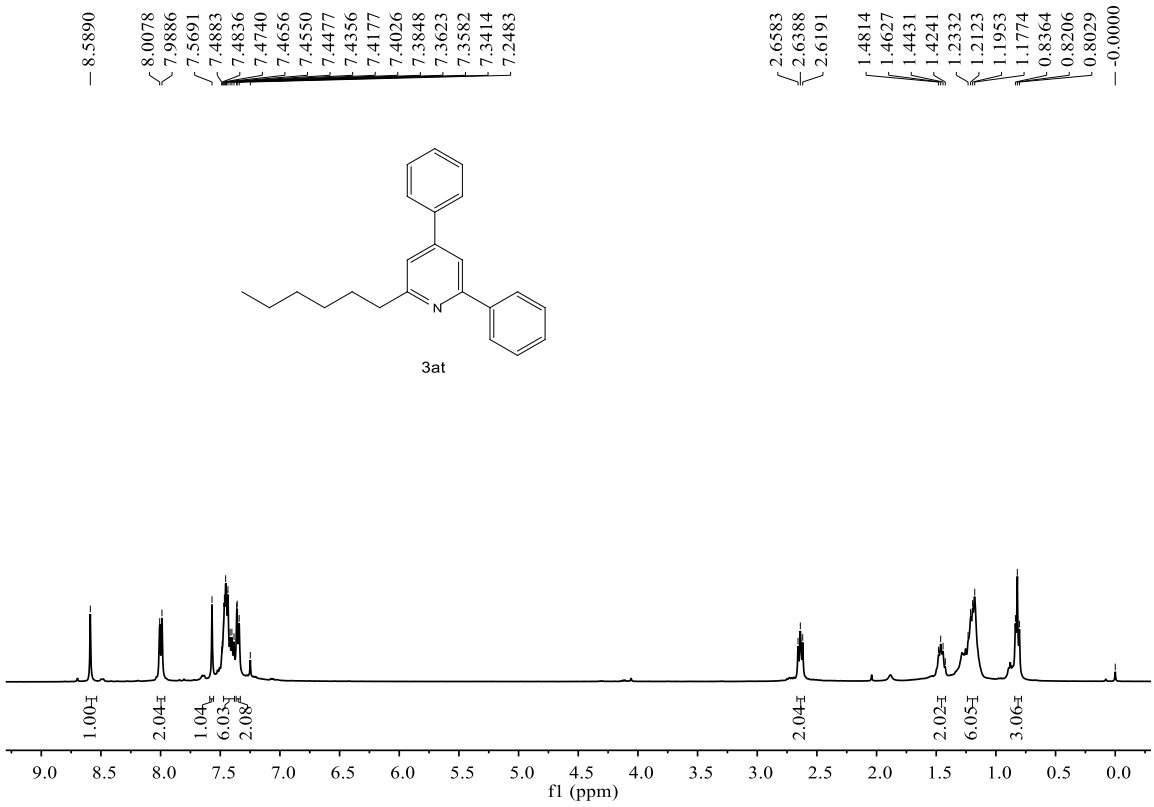
149.7287
139.4004
139.2233
135.2969
128.6795
128.6456
128.4678
128.4145
127.8621
126.7069
121.2561
77.3175
77.0000
76.6824
23.3435
15.4880
3as
f1 (ppm)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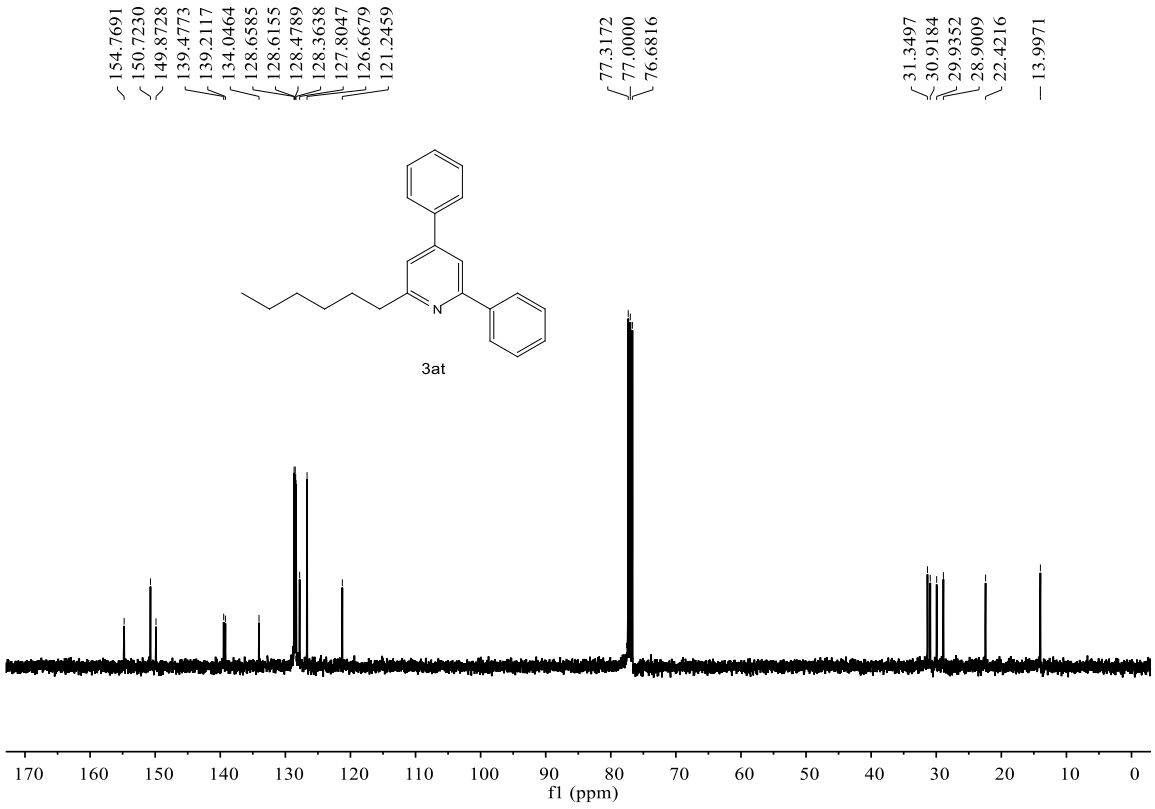
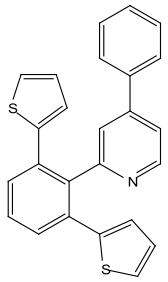
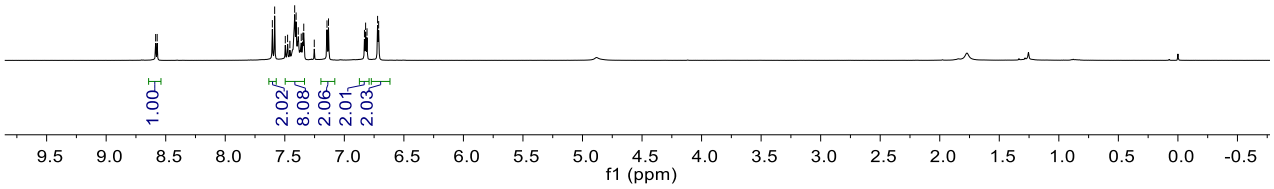
8.0078
7.9886
7.5691
7.4883
7.4836
7.4740
7.4656
7.4550
7.4477
7.4356
7.4177
7.4026
7.3848
7.3623
7.3582
7.3414
7.2483
2.6583
2.6388
2.6191
1.4814
1.4627
1.4431
1.4241
1.2332
1.2123
1.1953
1.1774
0.8364
0.8206
0.8029
-0.0000
N
3at
1.00
2.04
1.04
6.03
2.08
2.04
2.02
6.05
3.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

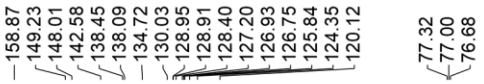

154.7691
150.7230
149.8728
139.4773
139.2117
134.0464
128.6585
128.6155
128.4789
128.3638
127.8047
126.6679
121.2459
77.3172
77.0000
76.6816
31.3497
30.9184
29.9352
28.9009
22.4216
13.9971
N
3at
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.58
8.57
7.60
7.59
7.50
7.48
7.46
7.42
7.41
7.39
7.36
7.35
7.34
7.25
7.15
7.13
6.83
6.82
6.81
6.72
6.71


S
N
S

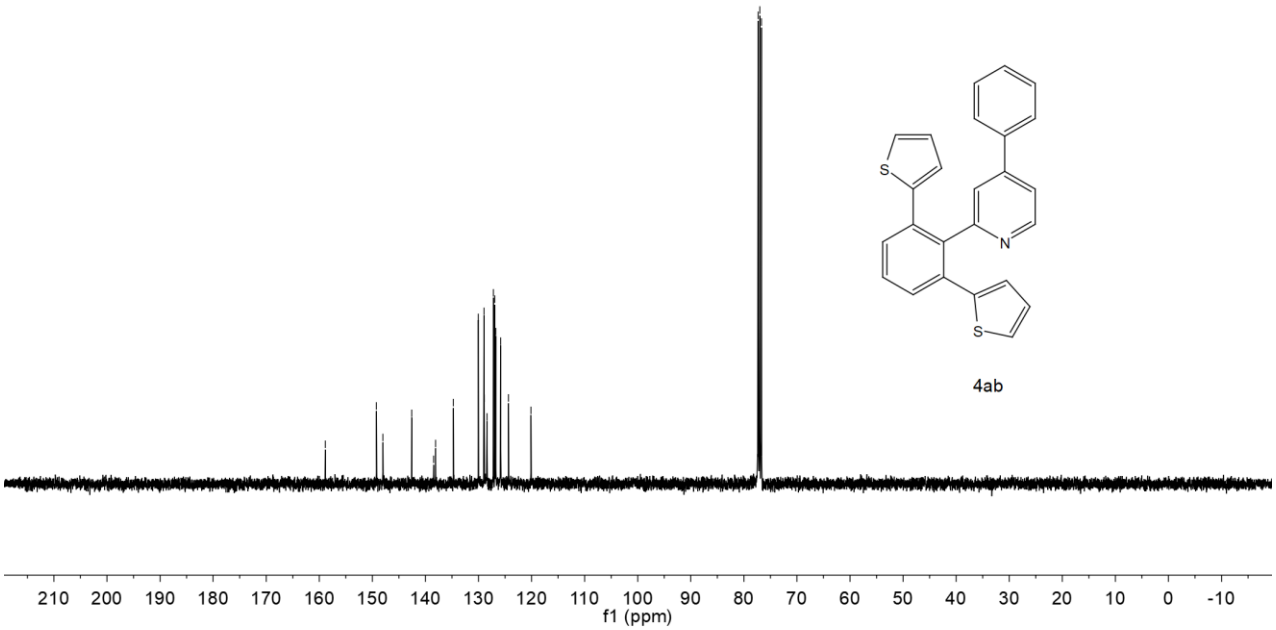
4ab

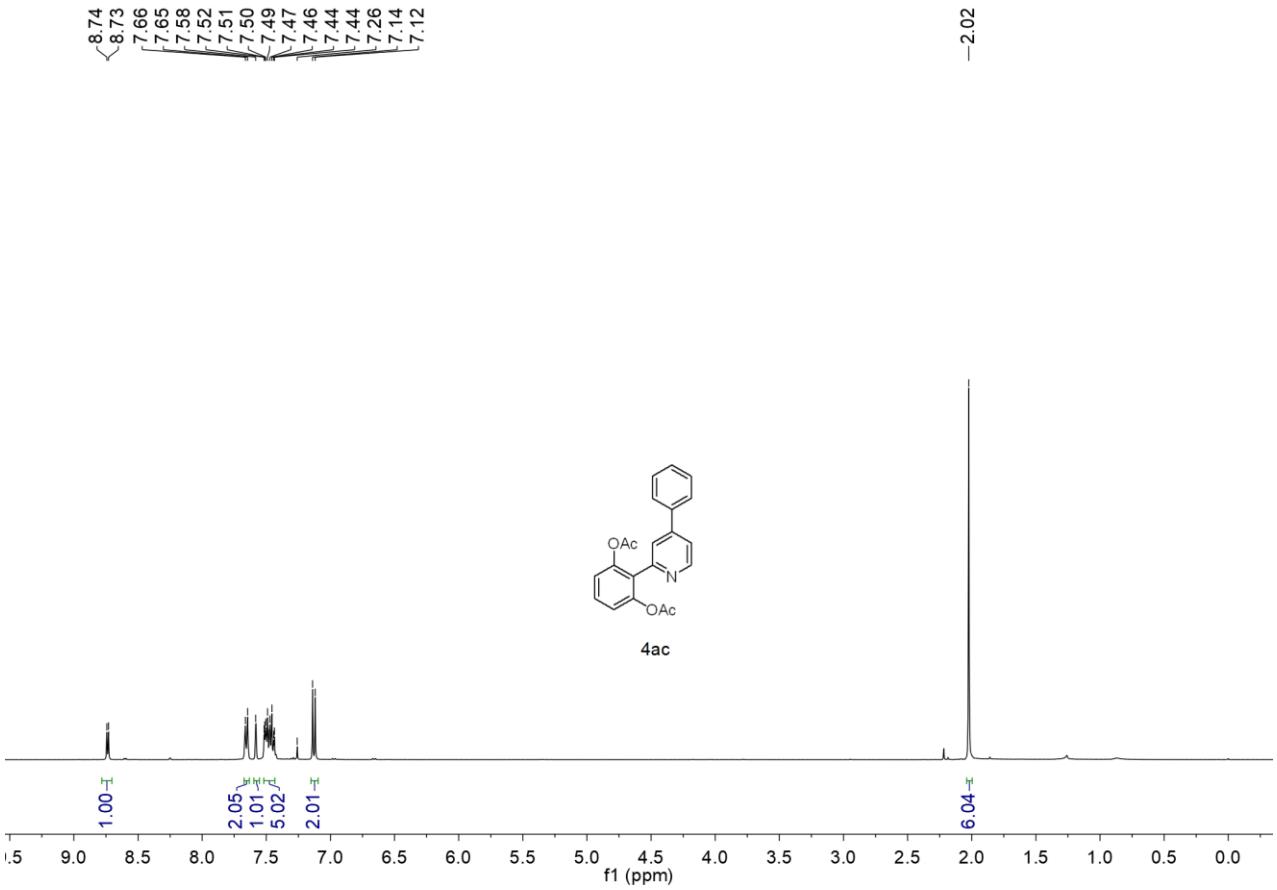

1.00
2.02
8.08
2.06
2.01
2.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

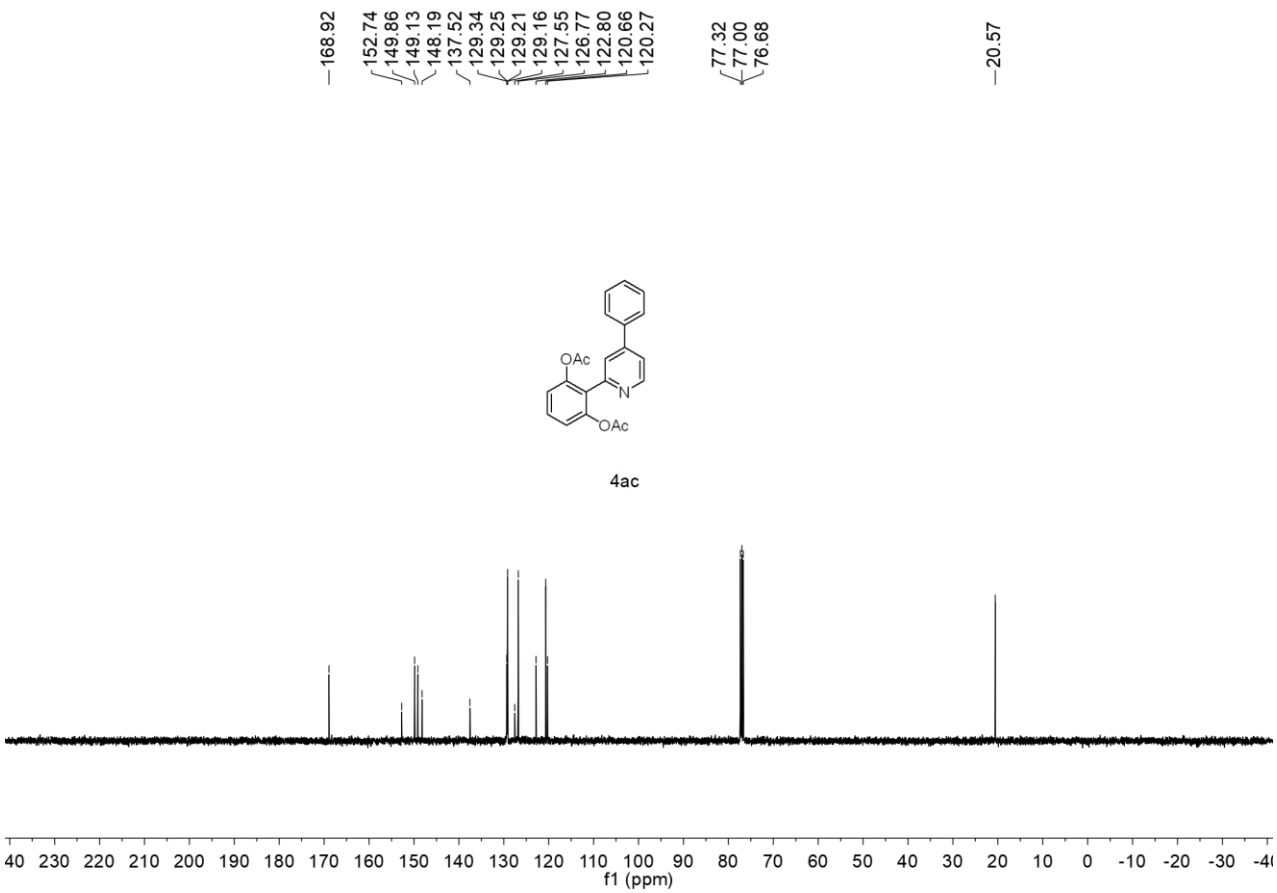

158.87
149.23
148.01
142.58
138.45
138.09
134.72
130.03
128.95
128.91
128.40
127.20
126.93
126.75
125.84
124.35
120.12
77.32
77.00
76.68

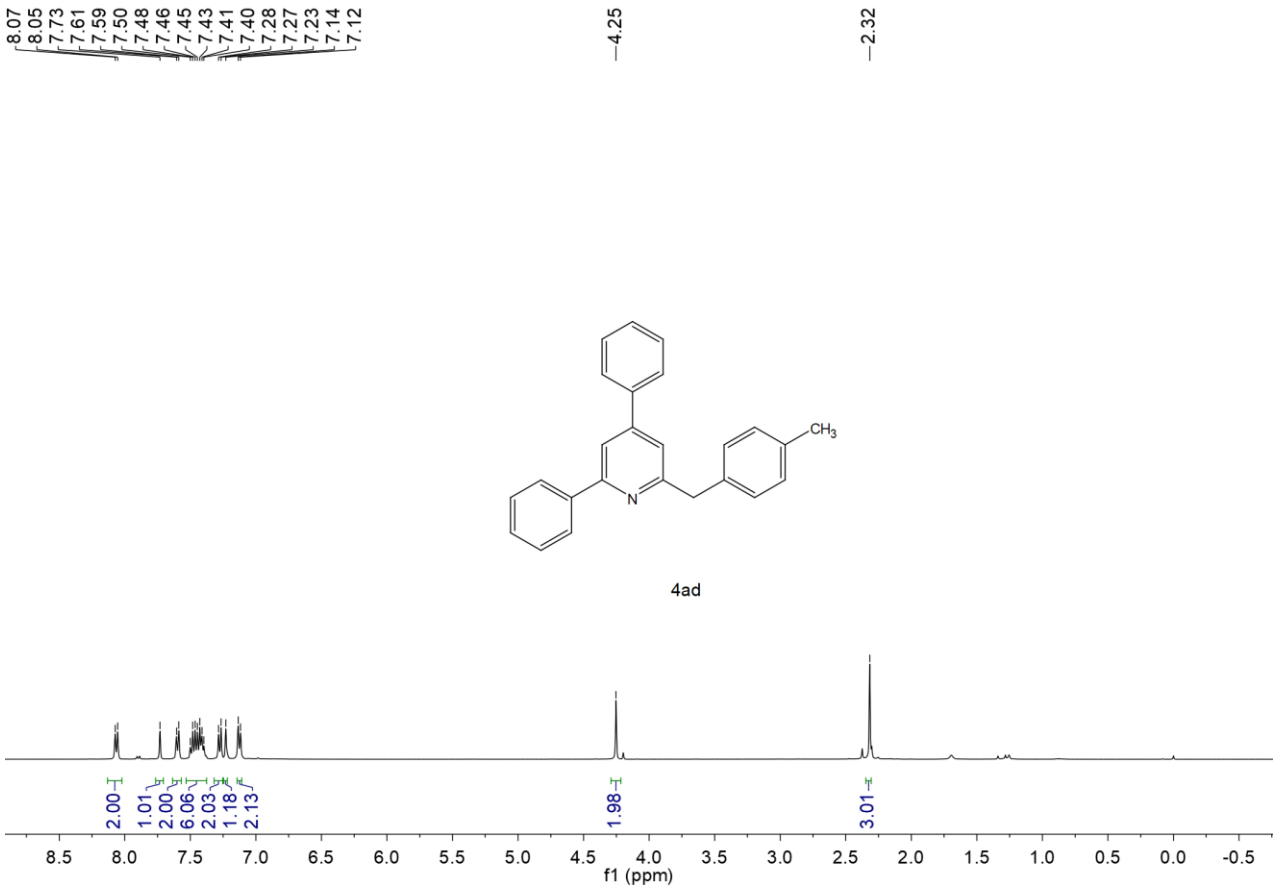
S
N
S

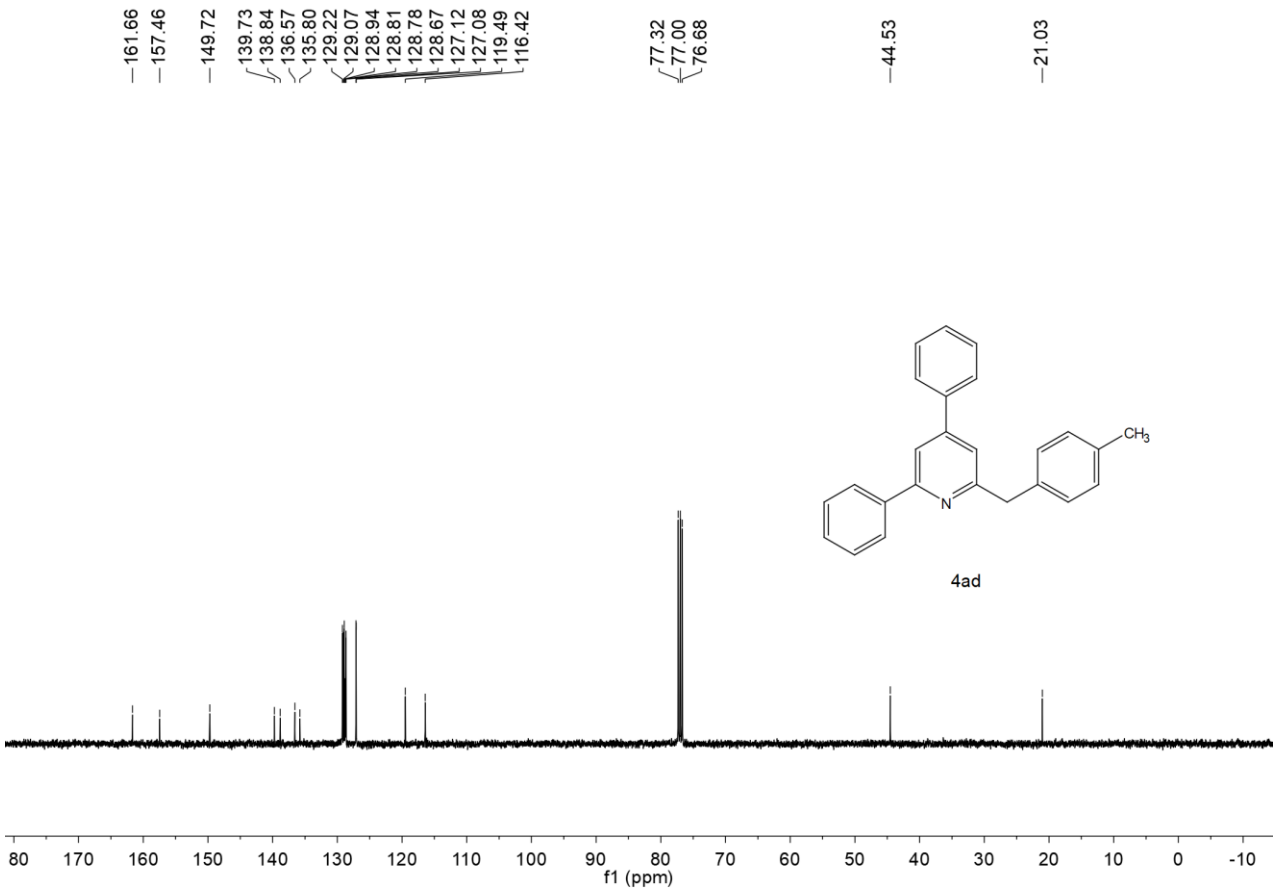
4ab


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.74
8.73
7.66
7.65
7.58
7.52
7.51
7.50
7.49
7.47
7.46
7.44
7.44
7.26
7.14
7.12
2.02
OAc
N
OAc
4ac
1.00
2.05
1.01
5.02
2.01
6.04
f1 (ppm)


168.92
152.74
149.86
149.13
148.19
137.52
129.34
129.25
129.21
129.16
127.55
126.77
122.80
120.66
120.27
77.32
77.00
76.68
20.57
OAc
N
OAc
4ac
f1 (ppm)


8.07
8.05
7.73
7.61
7.59
7.50
7.48
7.46
7.45
7.43
7.41
7.40
7.28
7.27
7.23
7.14
7.12
4.25
2.32
CH3
N
4ad
2.00
1.01
2.00
6.06
2.03
1.18
2.13
1.98
3.01
f1 (ppm)


161.66
157.46
149.72
139.73
138.84
136.57
135.80
129.22
129.07
128.94
128.81
128.78
128.67
127.12
127.08
119.49
116.42
77.32
77.00
76.68
44.53
21.03
CH3
N
4ad
f1 (ppm)