

Base-Catalysed [3+2] Cycloaddition of Propargylamines and Aldehydes to Substituted Furans

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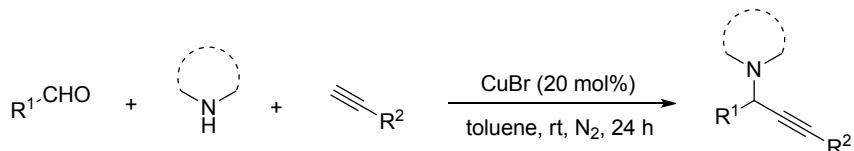
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1. General information:

Unless otherwise stated, all commercial materials and solvents were used directly without further purification. ^1H NMR spectra were recorded on 400 MHz spectrometers, and ^{13}C NMR spectra were recorded on a 100 MHz spectrometer. Chemical shifts (in ppm) were referenced to tetramethylsilane ($\delta = 0$ ppm) in CDCl_3 as an internal standard at room temperature. ^{13}C NMR spectra were obtained by using the same NMR spectrometers and were calibrated with CDCl_3 ($\delta = 77.00$ ppm). High-resolution mass spectra (HRMS) were equipped with an ESI source and a TOF detector. Column chromatography was performed on silica gel (70-230 mesh ASTM) using the reported eluents. Thin-layer chromatography (TLC) was carried out on 4×15 cm plates with a layer thickness of 0.2 mm (silica gel 60 F254).

The A3-propargylamines were prepared according to the literatures.¹



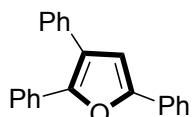
To a 25 mL round-bottom flask equipped with a magnetic stir bar were added amine (1.3 mmol), aldehyde (1.0 mmol), acetylene (1.3 mmol), copper (I) bromide (20 mol%) and toluene (5 mL). The mixture was degassed and backfilled with argon, and then stirred in room temperature for 24 h (monitored by TLC). The resulting reaction mixture was diluted with CH_2Cl_2 (10 mL) and filtered through a thin pad of silica gel. The filter cake was washed with CH_2Cl_2 , and the combined filtrate was concentrated in vacuum. The crude product was purified by flash column chromatography on silica gel to afford the corresponding A3-propargylamines **2**.

2. General procedure for synthesis of furans 3:

A mixture of aldehydes **1** (0.25 mmol), propargylamines **2** (0.25 mmol, 1 equiv), and TBAOH (0.05 mmol, 0.2 equiv) in DMSO (1.25 mL) was stirred in room temperature (25 °C) under air atmosphere for 2 h (monitored by TLC). Then H_2O was added and the resultant was extracted with EtOAc (3 x 10 mL). The combined EtOAc extracts were dried over Na_2SO_4 and concentrated. Then solvent was evaporated and the residue was purified by silica gel flash chromatography (EtOAc/PE=1/100 to EtOAc/PE=1/10) to give furans **3**.

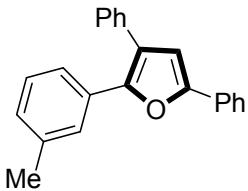
For **3aa** (gram-scale): A mixture of benzaldehyde **1a** (0.53g, 5 mmol), 2-(1,3-diphenylprop-2-yn-1-yl)-1,2,3,4-tetrahydroisoquinoline **2a** (1.62g, 5 mmol, 1 equiv), and TBAOH (1 mmol, 0.2 equiv) in DMSO (12.5 mL) was stirred in room temperature (25 °C) under air atmosphere for 2 h (monitored by TLC). Then H_2O was added and the resultant was extracted with EtOAc (3 x 20 mL). The combined EtOAc extracts were dried over Na_2SO_4 and concentrated. Then solvent was evaporated and the residue was purified by silicagel flash chromatography (EtOAc/PE=1/100) to give 2,3,5-triphenylfuran **3aa** in a 69% yield (1.02 g).

3. Characterization data for products:



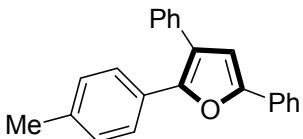
2,3,5-triphenylfuran **3aa**²

56.7 mg, 78% yield. White solid. Mp 91-93 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.74 (d, *J* = 8.0 Hz, 2H), 7.60 (d, *J* = 8.1 Hz, 2H), 7.44 (d, *J* = 7.9 Hz, 2H), 7.36 (dd, *J* = 17.8, 7.7 Hz, 4H), 7.32 – 7.19 (m, 5H), 6.77 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 152.5, 147.9, 134.3, 131.1, 130.5, 128.7, 128.6, 128.4, 127.5, 127.3, 126.1, 124.5, 123.8, 109.4.



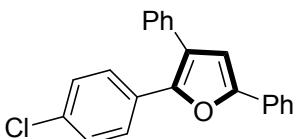
3,5-Diphenyl-2-(m-tolyl)furan **3ba**

57.2 mg, 74% yield. White solid. Mp 60-62 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, *J* = 7.5 Hz, 2H), 7.46 (d, *J* = 6.8 Hz, 3H), 7.42 – 7.34 (m, 5H), 7.33 – 7.24 (m, 2H), 7.16 (t, *J* = 7.7 Hz, 1H), 7.06 (d, *J* = 7.5 Hz, 1H), 6.80 (s, 1H), 2.31 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 152.4, 148.1, 138.0, 134.3, 131.0, 130.6, 128.7, 128.6, 128.3, 128.2, 127.5, 127.2, 126.8, 124.4, 123.8, 123.4, 109.4, 21.5; HRMS (ESI) m/z calcd for C₂₃H₁₉O (MH⁺) 311.1430, found 311.1426.



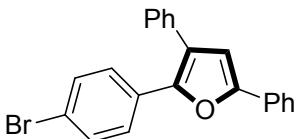
3,5-Diphenyl-2-(p-tolyl)furan **3ca**³

48.4 mg, 62% yield. White solid. Mp 108-110 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.76 (d, *J* = 8.3 Hz, 2H), 7.52 – 7.45 (m, 4H), 7.40 (dd, *J* = 13.6, 7.1 Hz, 4H), 7.31 (dd, *J* = 14.9, 7.6 Hz, 2H), 7.12 (d, *J* = 7.9 Hz, 2H), 6.81 (s, 1H), 2.35 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 152.2, 148.2, 137.4, 134.4, 130.6, 129.1, 128.7, 128.6, 128.3, 127.4, 127.2, 126.1, 123.8, 123.7, 109.3, 21.3.



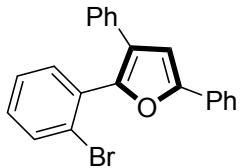
2-(4-Chlorophenyl)-3,5-diphenylfuran **3da**³

60.3 mg, 73% yield. White solid. Mp 100-102 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.78 (d, *J* = 7.3 Hz, 2H), 7.56 (d, *J* = 8.7 Hz, 2H), 7.49 – 7.30 (m, 10H), 6.84 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 152.8, 146.7, 134.0, 133.1, 130.3, 129.5, 128.8, 128.6, 128.6, 127.7, 127.5, 127.2, 125.0, 123.8, 109.6.



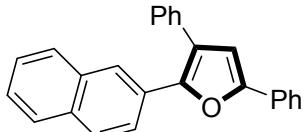
2-(4-Bromophenyl)-3,5-diphenylfuran **3ea**³

65.3 mg, 70% yield. White solid. Mp 103-105 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.74 (d, *J* = 7.4 Hz, 2H), 7.49 – 7.32 (m, 11H), 7.29 (t, *J* = 7.4 Hz, 1H), 6.79 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 152.9, 146.8, 134.0, 131.6, 130.3, 130.0, 128.8, 128.8, 128.6, 127.7, 127.5, 127.5, 125.2, 123.9, 121.3, 109.6.



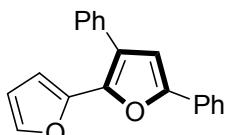
2-(2-Bromophenyl)-3,5-diphenylfuran **3fa**

60.1 mg, 64% yield. White solid. Mp 51-53 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.80 (d, *J* = 7.8 Hz, 2H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.44 (t, *J* = 7.0 Hz, 3H), 7.36 – 7.24 (m, 8H), 7.01 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 153.5, 147.2, 133.5, 133.3, 132.8, 132.6, 130.5, 130.2, 128.7, 128.5, 127.7, 127.4, 127.3, 127.0, 125.7, 124.1, 123.9, 106.7; HRMS (ESI) m/z calcd for C₂₂H₁₆BrO (MH⁺) 375.0379, found 375.0376.



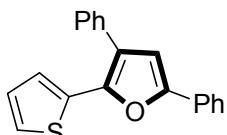
2-(Naphthalen-2-yl)-3,5-diphenylfuran **3ga**

65.5 mg, 76% yield. White solid. Mp 110-112 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 8.12 (s, 1H), 7.77 (ddd, *J* = 19.6, 10.0, 5.5 Hz, 5H), 7.68 – 7.63 (m, 1H), 7.53 – 7.48 (m, 2H), 7.48 – 7.33 (m, 7H), 7.30 (td, *J* = 7.1, 1.1 Hz, 1H), 6.86 (d, *J* = 1.3 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 152.8, 148.0, 134.3, 133.4, 132.6, 130.5, 128.8, 128.8, 128.7, 128.5, 128.2, 127.9, 127.6, 127.6, 127.4, 126.3, 126.1, 125.0, 124.9, 124.2, 123.9, 109.6; HRMS (ESI) m/z calcd for C₂₆H₁₉O (MNa⁺) 369.1250, found 369.1252.



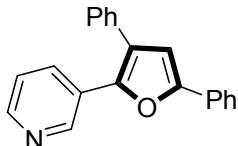
3,5-Diphenyl-2,2'-bifuran **3ha**

60.2 mg, 84% yield. Brown oil. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.76 – 7.71 (m, 2H), 7.55 (dd, *J* = 5.2, 3.3 Hz, 2H), 7.43 – 7.36 (m, 5H), 7.35 – 7.30 (m, 1H), 7.27 (dd, *J* = 10.5, 4.3 Hz, 1H), 6.81 (s, 1H), 6.63 – 6.50 (m, 1H), 6.42 (dd, *J* = 3.4, 1.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 152.8, 146.2, 142.0, 140.4, 133.2, 130.2, 128.7, 128.6, 128.4, 127.7, 127.4, 124.6, 123.9, 111.3, 108.6, 107.3; HRMS (ESI) m/z calcd for C₂₀H₁₅O₂ (MH⁺) 287.1067, found 287.1071.



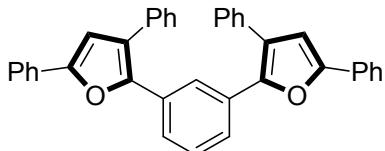
3,5-Diphenyl-2-(thiophen-2-yl)furan **3ia**

54.8 mg, 73% yield. Brown oil. $R_f = 0.3$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.73 (d, $J = 7.4$ Hz, 2H), 7.55 – 7.49 (m, 2H), 7.44 – 7.32 (m, 5H), 7.27 (t, $J = 7.4$ Hz, 1H), 7.23 – 7.21 (m, 1H), 7.21 – 7.14 (m, 1H), 6.96 (dd, $J = 4.9, 3.8$ Hz, 1H), 6.77 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.3, 143.9, 133.5, 133.1, 130.2, 128.9, 128.7, 128.6, 127.6, 127.6, 127.3, 124.7, 124.2, 124.0, 123.8, 109.2; HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{15}\text{OS}$ (MH^+) 303.0838, found 303.0834.



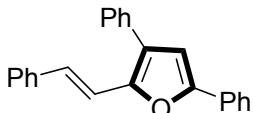
3-(3,5-Diphenylfuran-2-yl)pyridine **3ja**

58.0 mg, 78% yield. White solid. Mp 105–107 °C. $R_f = 0.4$ (EtOAc/PE = 1:10). ^1H NMR (400 MHz, CDCl_3) δ 8.86 (s, 1H), 8.50 – 8.41 (m, 1H), 7.88 – 7.82 (m, 1H), 7.76 (dd, $J = 8.3, 1.0$ Hz, 2H), 7.42 (ddt, $J = 12.6, 9.6, 4.8$ Hz, 6H), 7.37 – 7.29 (m, 2H), 7.21 (dd, $J = 8.0, 4.8$ Hz, 1H), 6.82 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.6, 148.1, 147.1, 144.9, 133.5, 132.7, 130.1, 128.9, 128.8, 128.5, 127.9, 127.8, 127.3, 126.3, 123.9, 123.2, 109.5; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{16}\text{NO}$ (MH^+) 298.1226, found 298.1228.



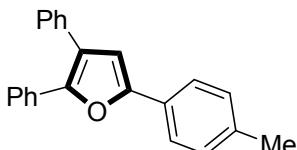
1,3-Bis(3,5-diphenylfuran-2-yl)benzene **3kb**

45.2 mg, 35% yield. Pale yellow solid. Mp 117–119 °C. $R_f = 0.3$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.98 (d, $J = 1.2$ Hz, 1H), 7.66 (d, $J = 7.8$ Hz, 4H), 7.49 – 7.43 (m, 6H), 7.43 – 7.32 (m, 10H), 7.28 (t, $J = 7.3$ Hz, 2H), 7.20 (dd, $J = 12.0, 4.5$ Hz, 1H), 6.78 (d, $J = 1.5$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.5, 147.4, 134.2, 131.3, 130.4, 128.8, 128.7, 128.7, 128.4, 127.5, 127.3, 124.9, 124.9, 123.8, 123.4, 109.6; HRMS (ESI) m/z calcd for $\text{C}_{38}\text{H}_{27}\text{O}_2$ (MH^+) 515.2006, found 515.2008.



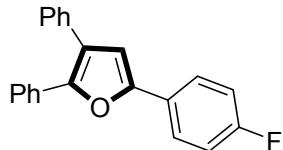
3,5-Diphenyl-2-styrylfuran **3la**

27.8 mg, 35% yield. Colorless oil. $R_f = 0.4$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.84 – 7.75 (m, 2H), 7.48 (ddt, $J = 15.4, 13.6, 4.8$ Hz, 8H), 7.38 – 7.28 (m, 4H), 7.27 – 7.22 (m, 2H), 7.10 (d, $J = 16.1$ Hz, 1H), 6.87 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.9, 148.1, 137.3, 133.5, 130.4, 128.8, 128.8, 128.7, 128.3, 127.9, 127.7, 127.5, 127.2, 126.9, 126.4, 124.0, 115.2, 108.1; HRMS (ESI) m/z calcd for $\text{C}_{24}\text{H}_{19}\text{O}$ (MH^+) 323.1430, found 323.1426.



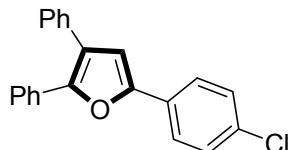
2,3-Diphenyl-5-(p-tolyl)furan **3af**³

51.8 mg, 67% yield. White solid. Mp 97-99 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.64 (d, *J* = 8.0 Hz, 2H), 7.59 (d, *J* = 7.8 Hz, 2H), 7.45 (d, *J* = 7.5 Hz, 2H), 7.36 (t, *J* = 7.4 Hz, 2H), 7.29 (dd, *J* = 15.2, 7.7 Hz, 3H), 7.21 (t, *J* = 8.9 Hz, 3H), 6.74 (s, 1H), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 152.8, 147.5, 137.4, 134.4, 131.2, 129.4, 128.7, 128.6, 128.4, 127.9, 127.3, 127.2, 126.1, 124.5, 123.8, 108.8, 21.3.



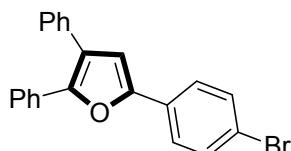
5-(4-Fluorophenyl)-2,3-diphenylfuran **3ag**³

65.2 mg, 83% yield. White solid. Mp 85-87 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.76 – 7.69 (m, 2H), 7.59 (dd, *J* = 8.3, 1.1 Hz, 2H), 7.45 (dd, *J* = 8.1, 1.3 Hz, 2H), 7.40 – 7.24 (m, 6H), 7.10 (dd, *J* = 12.1, 5.4 Hz, 2H), 6.74 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 162.3 (d, *J* = 247.4 Hz), 151.7, 147.9, 134.2, 131.0, 128.7, 128.7, 128.4, 127.5 (d, *J* = 19.7 Hz), 126.9 (d, *J* = 3.2 Hz), 126.1, 125.6, 125.5, 124.5, 115.8 (d, *J* = 21.9 Hz), 109.1.



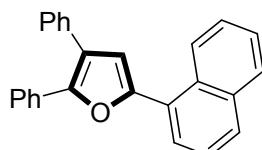
5-(4-Chlorophenyl)-2,3-diphenylfuran **3ah**³

75.1 mg, 91% yield. White solid. Mp 98-100 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.69 – 7.64 (m, 2H), 7.61 – 7.56 (m, 2H), 7.44 (dd, *J* = 8.1, 1.3 Hz, 2H), 7.40 – 7.23 (m, 8H), 6.79 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 151.5, 148.2, 134.1, 133.1, 130.9, 129.0, 129.0, 128.7, 128.7, 128.4, 127.7, 127.4, 126.2, 125.0, 124.6, 109.9.



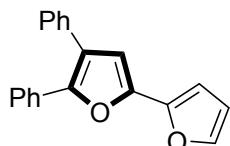
5-(4-Bromophenyl)-2,3-diphenylfuran **3ai**³

61.8 mg, 66% yield. White solid. Mp 103-104 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ¹H NMR (400 MHz, CDCl₃) δ 7.61 – 7.55 (m, 4H), 7.50 (d, *J* = 8.6 Hz, 2H), 7.45 – 7.40 (m, 2H), 7.39 – 7.23 (m, 6H), 6.78 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 151.4, 148.2, 134.0, 131.9, 130.9, 129.4, 128.7, 128.6, 128.4, 127.7, 127.4, 126.1, 125.2, 124.6, 121.2, 110.0.



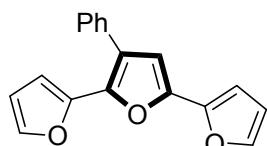
5-(Naphthalen-1-yl)-2,3-diphenylfuran **3aj**

51.9 mg, 60% yield. Colorless oil. $R_f = 0.4$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 8.57 (d, $J = 8.4$ Hz, 1H), 7.94 – 7.83 (m, 3H), 7.66 (d, $J = 7.3$ Hz, 2H), 7.60 – 7.49 (m, 5H), 7.41 (t, $J = 7.5$ Hz, 2H), 7.37 – 7.24 (m, 4H), 6.91 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.2, 148.3, 134.4, 134.0, 131.1, 130.3, 128.8, 128.7, 128.6, 128.5, 128.2, 127.5, 127.3, 126.7, 126.2, 126.1, 126.0, 125.5, 125.4, 124.2, 113.7; HRMS (ESI) m/z calcd for (MNa^+) 369.1250, found 369.1252.



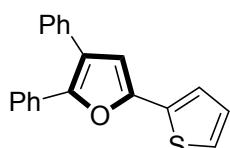
4,5-Diphenyl-2,2'-bifuran **3ak**

45.1 mg, 63% yield. Brown oil. $R_f = 0.3$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.59 – 7.54 (m, 2H), 7.44 (d, $J = 7.2$ Hz, 3H), 7.29 (dd, $J = 14.5, 10.6, 7.5, 4.4$ Hz, 6H), 6.71 (s, 1H), 6.65 (d, $J = 3.4$ Hz, 1H), 6.50 – 6.44 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.6, 146.3, 145.3, 142.0, 134.0, 130.8, 128.7, 128.7, 128.4, 127.6, 127.4, 126.2, 124.1, 111.5, 109.3, 105.5; HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{15}\text{O}_2$ (MH^+) 287.1067, found 287.1063.



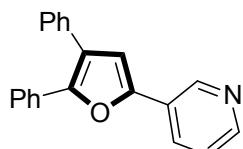
3'-Phenyl-2,2':5',2''-terfuran **3hk**

50.1 mg, 72% yield. Brown oil. $R_f = 0.3$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.53 (d, $J = 7.1$ Hz, 2H), 7.45 – 7.42 (m, 1H), 7.42 – 7.36 (m, 3H), 7.35 – 7.30 (m, 1H), 6.72 (s, 1H), 6.67 (d, $J = 3.4$ Hz, 1H), 6.56 (d, $J = 3.4$ Hz, 1H), 6.50 – 6.46 (m, 1H), 6.45 – 6.38 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 146.0, 145.9, 145.5, 142.2, 142.1, 140.1, 132.9, 128.6, 128.4, 127.5, 124.3, 111.5, 111.3, 108.6, 107.6, 105.9; HRMS (ESI) m/z calcd for $\text{C}_{18}\text{H}_{13}\text{O}_3$ (MH^+) 277.0859, found 277.0855.



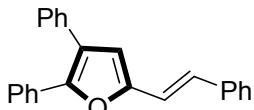
2,3-Diphenyl-5-(thiophen-2-yl)furan **3al**

47.8 mg, 63% yield. Pale yellow solid. Mp 72–74 °C. $R_f = 0.3$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.83 – 7.74 (m, 4H), 7.49 – 7.39 (m, 4H), 7.38 – 7.30 (m, 3H), 7.20 (dd, $J = 3.5, 1.1$ Hz, 1H), 7.11 (dd, $J = 5.1, 3.5$ Hz, 1H), 6.87 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.5, 148.5, 135.5, 130.7, 130.2, 128.7, 128.4, 127.9, 127.7, 127.4, 126.4, 126.0, 125.1, 123.9, 117.5, 109.6; HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{15}\text{OS}$ (MH^+) 303.0838, found 303.0840.



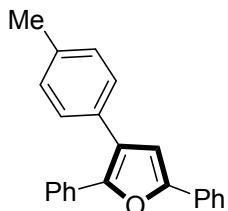
3-(4,5-Diphenylfuran-2-yl)pyridine **3am**

59.6 mg, 80% yield. White solid. Mp 103-104 °C. R_f = 0.4 (EtOAc/PE = 1:10). ^1H NMR (400 MHz, CDCl_3) δ 9.01 (s, 1H), 8.51 (s, 1H), 7.99 (d, J = 7.9 Hz, 1H), 7.59 (d, J = 7.5 Hz, 2H), 7.44 (d, J = 7.2 Hz, 2H), 7.40 – 7.23 (m, 7H), 6.88 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.5, 148.9, 148.2, 145.3, 133.7, 130.6, 130.6, 128.7, 128.6, 128.4, 127.8, 127.4, 126.5, 126.2, 124.5, 123.5, 110.7; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{16}\text{NO}$ (MH^+) 298.1226, found 298.1227.



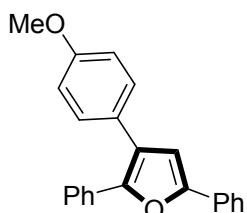
2,3-diphenyl-5-styrylfuran 3an

18.0 mg, 22% yield. White solid. Mp 93-95 °C. R_f = 0.4 (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.62 – 7.57 (m, 2H), 7.51 (d, J = 7.4 Hz, 2H), 7.45 – 7.41 (m, 2H), 7.39 – 7.24 (m, 9H), 7.16 (d, J = 16.2 Hz, 1H), 6.93 (d, J = 16.2 Hz, 1H), 6.52 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.0, 148.1, 137.0, 134.2, 131.0, 128.7, 128.7, 128.4, 127.7, 127.6, 127.5, 127.3, 126.4, 126.2, 124.6, 116.1, 113.1; HRMS (ESI) m/z calcd for $\text{C}_{24}\text{H}_{19}\text{O}$ (MH^+) 323.1430, found 323.1432.



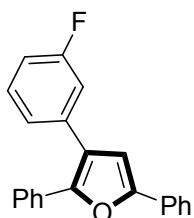
2,5-Diphenyl-3-(p-tolyl)furan 3ao⁴

46.0 mg, 59% yield. White solid. Mp 126-127 °C. R_f = 0.4 (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.77 – 7.72 (m, 2H), 7.65 – 7.59 (m, 2H), 7.41 – 7.33 (m, 4H), 7.31 – 7.16 (m, 6H), 6.77 (d, J = 1.4 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.4, 147.7, 137.0, 131.3, 131.2, 130.6, 129.4, 128.7, 128.5, 128.3, 127.4, 127.4, 126.0, 124.5, 123.8, 109.5, 21.2.



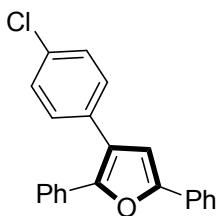
3-(4-Methoxyphenyl)-2,5-diphenylfuran 3ap⁴

53.2 mg, 65% yield. Colorless oil. R_f = 0.4 (EtOAc/PE = 1:20). ^1H NMR (400 MHz, CDCl_3) δ 7.78 – 7.71 (m, 2H), 7.61 (dd, J = 5.3, 3.3 Hz, 2H), 7.42 – 7.35 (m, 4H), 7.32 – 7.20 (m, 4H), 6.95 – 6.88 (m, 2H), 6.76 (s, 1H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.9, 152.4, 147.5, 131.2, 130.6, 129.8, 128.7, 128.4, 127.4, 127.3, 126.6, 126.0, 124.2, 123.8, 114.1, 109.6, 55.2.



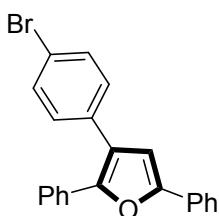
3-(3-Fluorophenyl)-2,5-diphenylfuran **3aq**

65.2 mg, 83% yield. White solid. Mp 79-80 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.81 (d, $J = 7.7$ Hz, 2H), 7.66 (d, $J = 7.4$ Hz, 2H), 7.47 (t, $J = 7.5$ Hz, 2H), 7.35 (ddd, $J = 25.7, 12.5, 4.6$ Hz, 6H), 7.23 (d, $J = 9.9$ Hz, 1H), 7.07 (t, $J = 8.3$ Hz, 1H), 6.85 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 163.0 (d, $J = 245.9$ Hz), 152.8, 148.3, 136.5 (d, $J = 8.2$ Hz), 130.7, 130.3, 130.1 (d, $J = 8.5$ Hz), 128.8, 128.5, 127.8, 127.7, 126.3, 124.4 (d, $J = 2.8$ Hz), 123.8, 123.3 (d, $J = 2.2$ Hz), 115.5 (d, $J = 21.8$ Hz), 114.2 (d, $J = 21.0$ Hz), 109.0; HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{16}\text{FO}$ (MH^+) 315.1180, found 315.1176.



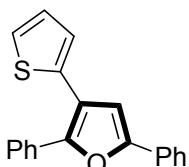
3-(4-Chlorophenyl)-2,5-diphenylfuran **3ar**

50.4 mg, 61% yield. White solid. Mp 117-119 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.72 (d, $J = 7.9$ Hz, 2H), 7.56 (d, $J = 7.8$ Hz, 2H), 7.32 (dtd, $J = 23.3, 15.4, 7.8$ Hz, 10H), 6.73 (d, $J = 0.7$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.7, 148.1, 133.1, 132.7, 130.8, 130.3, 129.9, 128.9, 128.7, 128.5, 127.7, 127.6, 126.2, 123.8, 123.2, 109.0; HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{16}\text{ClO}$ (MH^+) 331.0884, found 331.0882.



3-(4-Bromophenyl)-2,5-diphenylfuran **3as⁴**

56.1 mg, 60% yield. White solid. Mp 119-121 °C. $R_f = 0.4$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.82 (d, $J = 8.0$ Hz, 2H), 7.67 (d, $J = 8.1$ Hz, 2H), 7.57 (d, $J = 8.5$ Hz, 2H), 7.49 (t, $J = 7.6$ Hz, 2H), 7.44 – 7.31 (m, 6H), 6.83 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.8, 148.0, 133.2, 131.8, 130.7, 130.3, 130.2, 128.7, 128.5, 127.7, 127.6, 126.2, 123.8, 123.2, 121.2, 108.9.



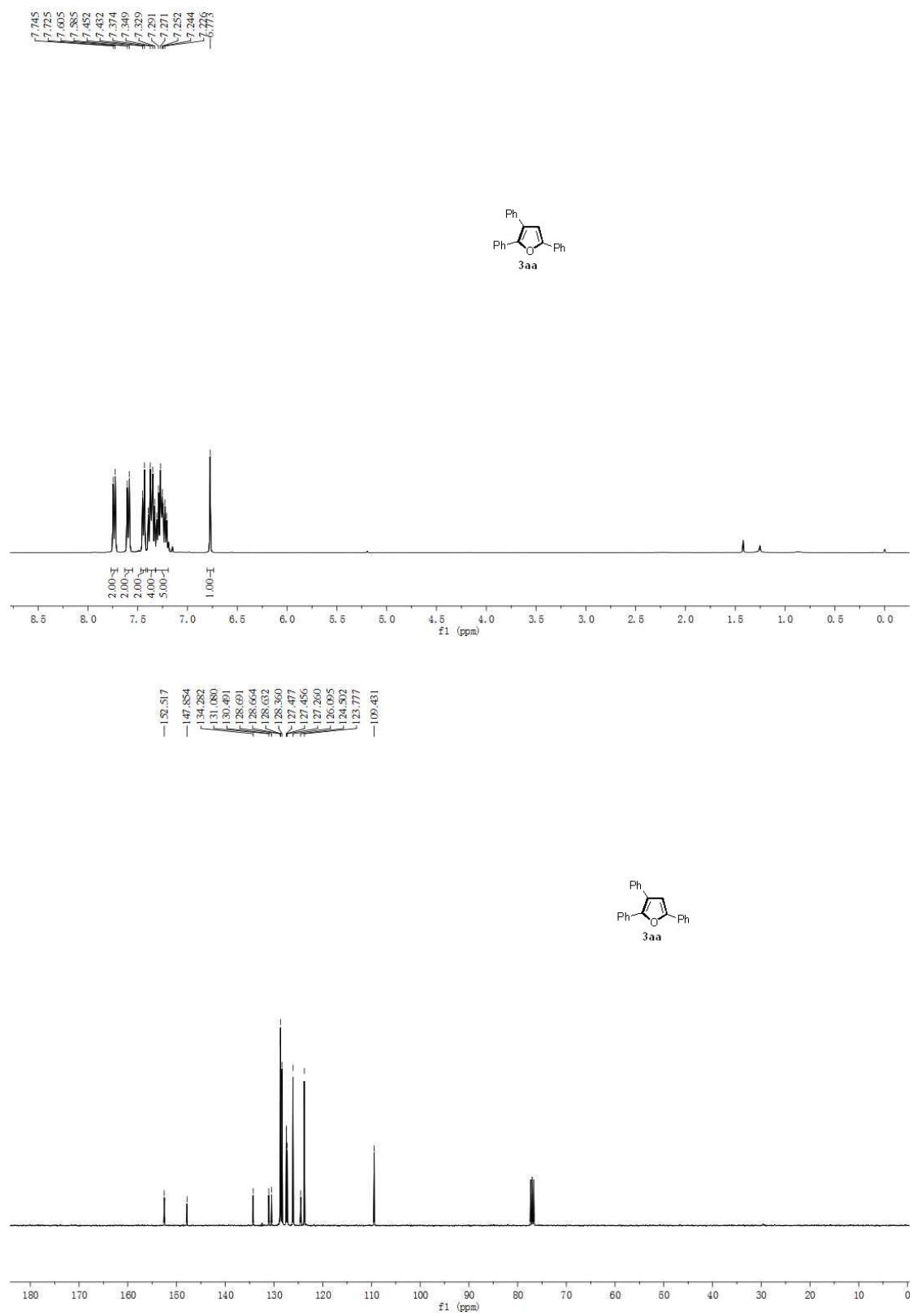
2,5-Diphenyl-3-(thiophen-2-yl)furan **3at**⁵

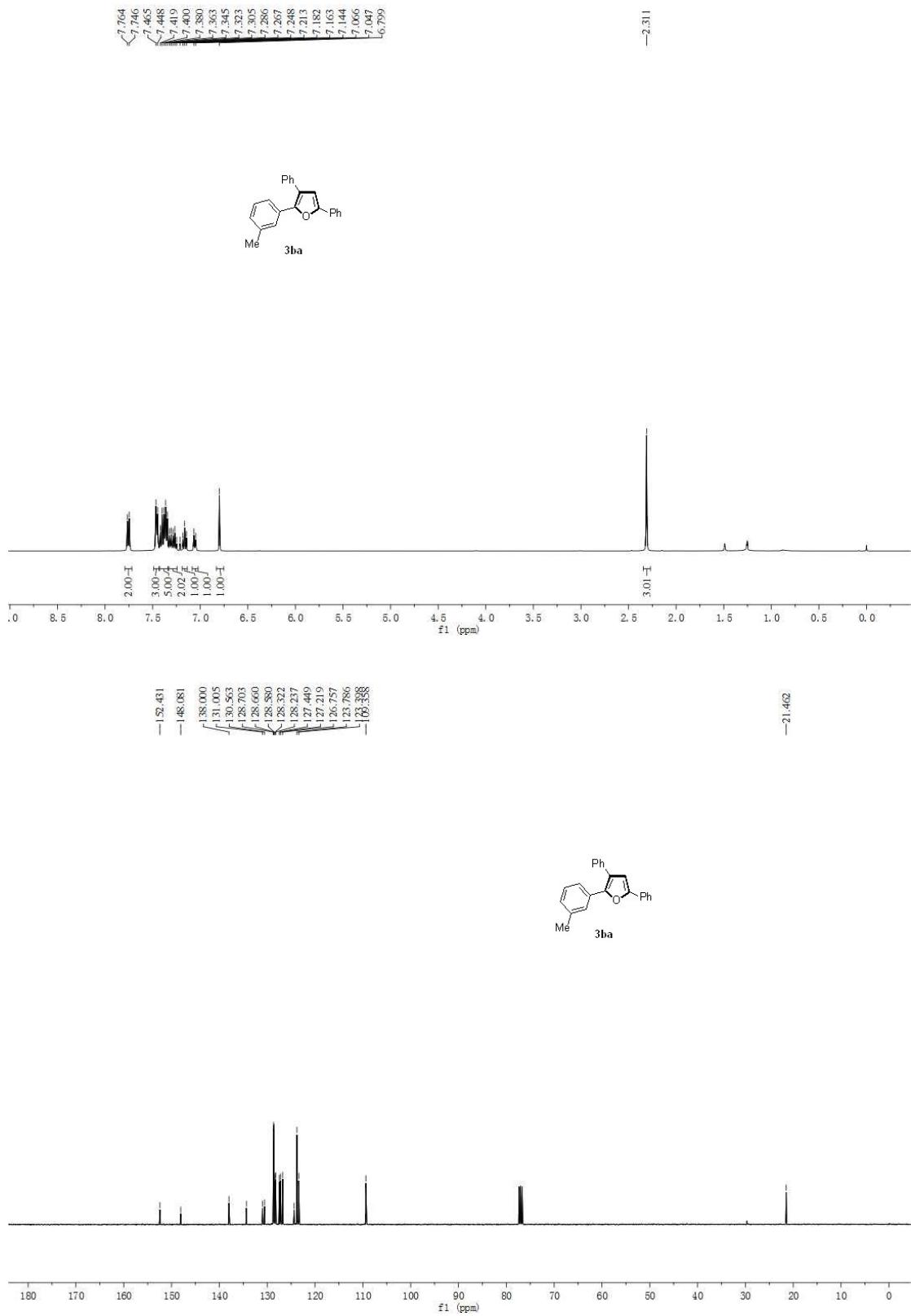
54.5 mg, 72% yield. Colorless oil. $R_f = 0.4$ (EtOAc/PE = 1:100). ^1H NMR (400 MHz, CDCl_3) δ 7.83 – 7.74 (m, 4H), 7.49 – 7.39 (m, 4H), 7.38 – 7.30 (m, 3H), 7.20 (dd, $J = 3.5, 1.1$ Hz, 1H), 7.11 (dd, $J = 5.1, 3.5$ Hz, 1H), 6.87 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.5, 148.5, 135.5, 130.7, 130.2, 128.7, 128.4, 127.9, 127.7, 127.4, 126.4, 126.0, 125.1, 123.9, 117.5, 109.6.

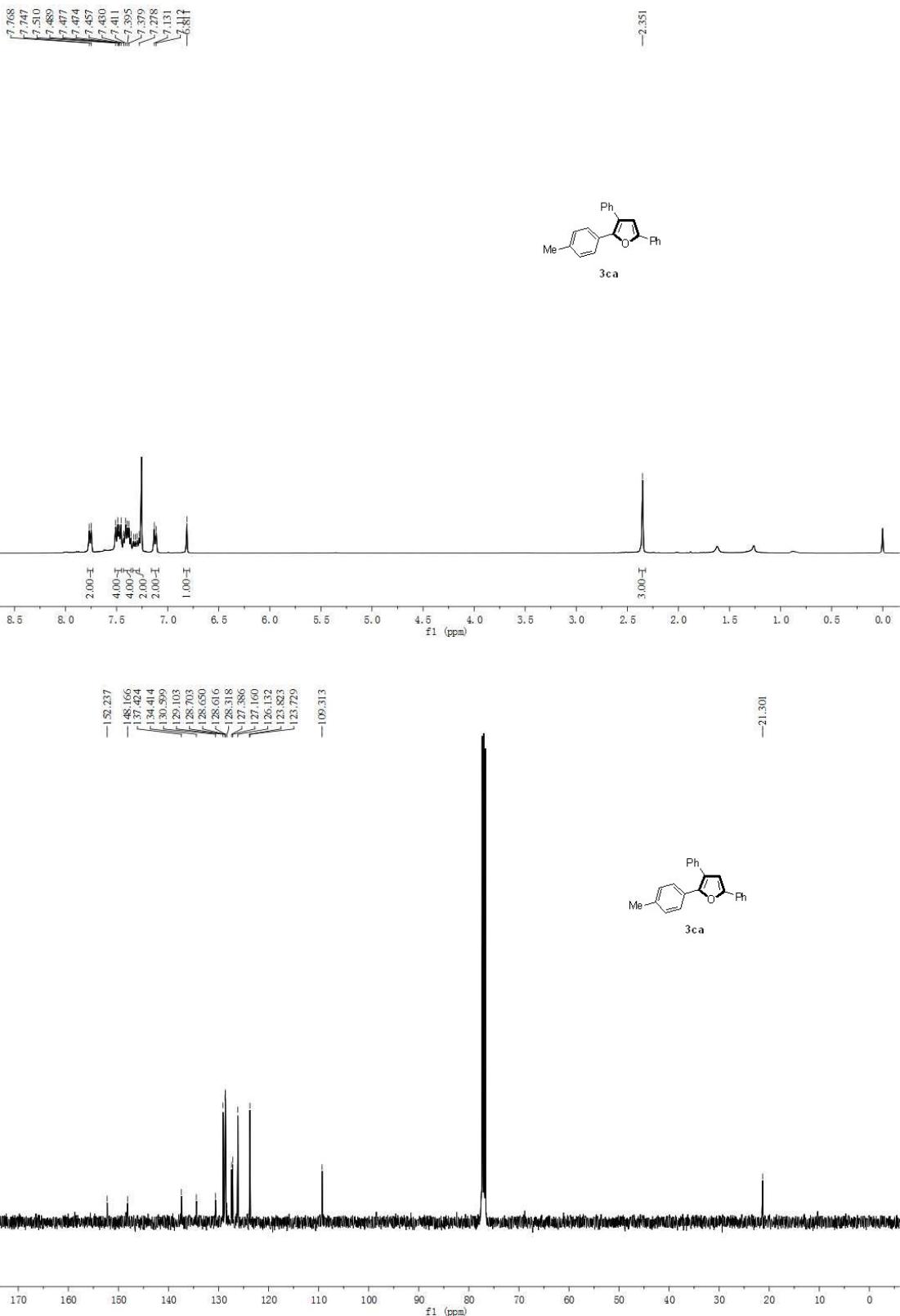
References:

1. (a) Q.-H. Zheng, W. Meng, G.-J. Jiang and Z.-X. Yu, *Org. Lett.*, 2013, **15**, 5928; (b) Y. Odabachian, S. Tong, Q. Wang, M.-X. Wang and J. Zhu, *Angew. Chem. Int. Ed.*, 2013, **52**, 10878.
2. Dudnik, A. S.; Gevorgyan, V. *Angew. Chem. Int. Ed.*, 2007, **46**, 5195.
3. Y. Wu, Z. Huang, Y. Luo, D. Liu, Y. Deng, H. Yi, J. Lee, C. Pao, J. Chen and A. Lei, *Org. Lett.*, 2017, **19**, 2330.
4. Y. Xia, Y. M. Xia, R. Ge, Z. Liu, Q. Xiao, Y. Zhang and J. B. Wang, *Angew. Chem. Int. Ed.*, 2014, **53**, 3917.
5. K.-W. Chen, S. Syu, Y.-J. Jang and W. Lin, *Org. Biomol. Chem.*, 2011, **9**, 2098.

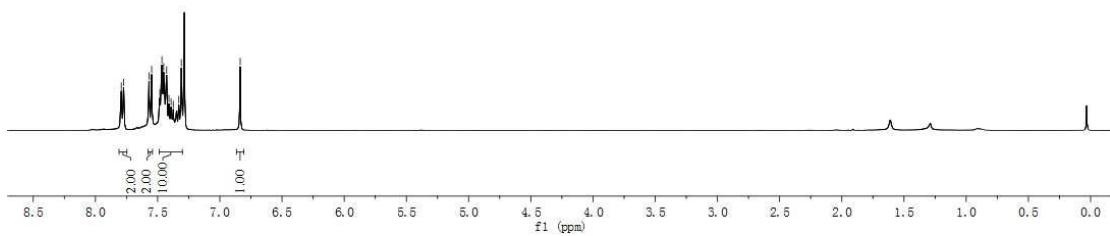
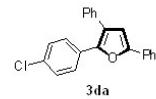
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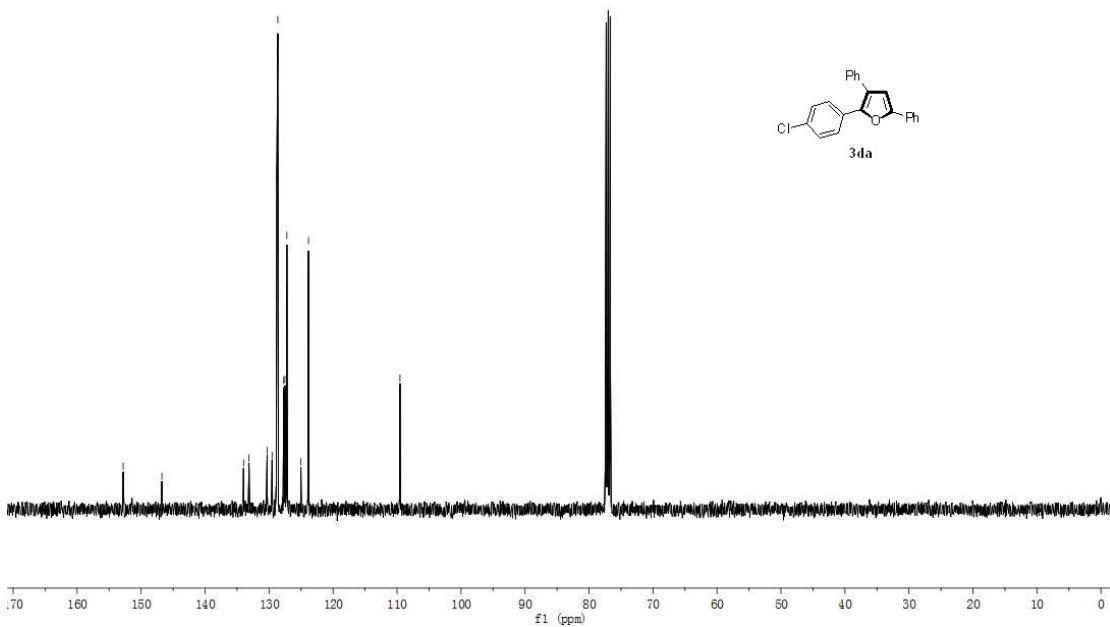
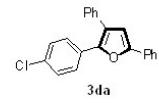




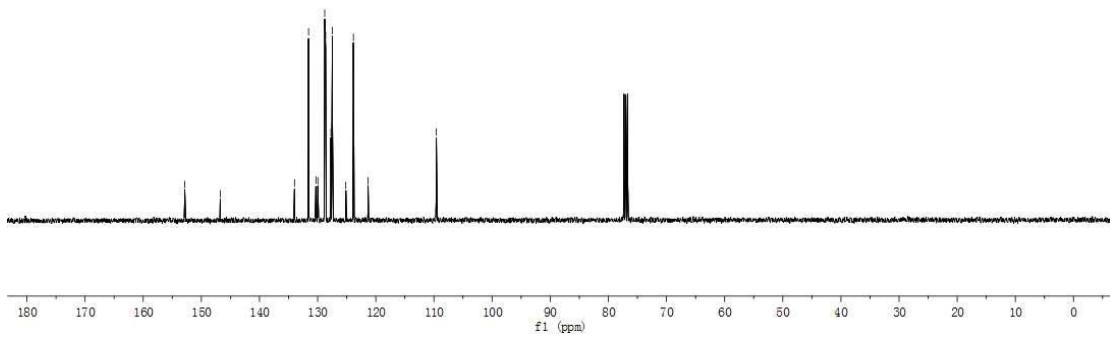
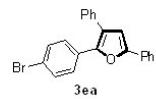
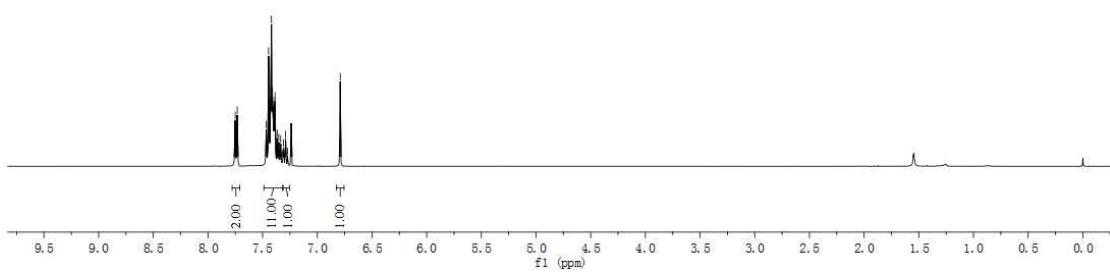
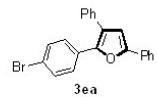
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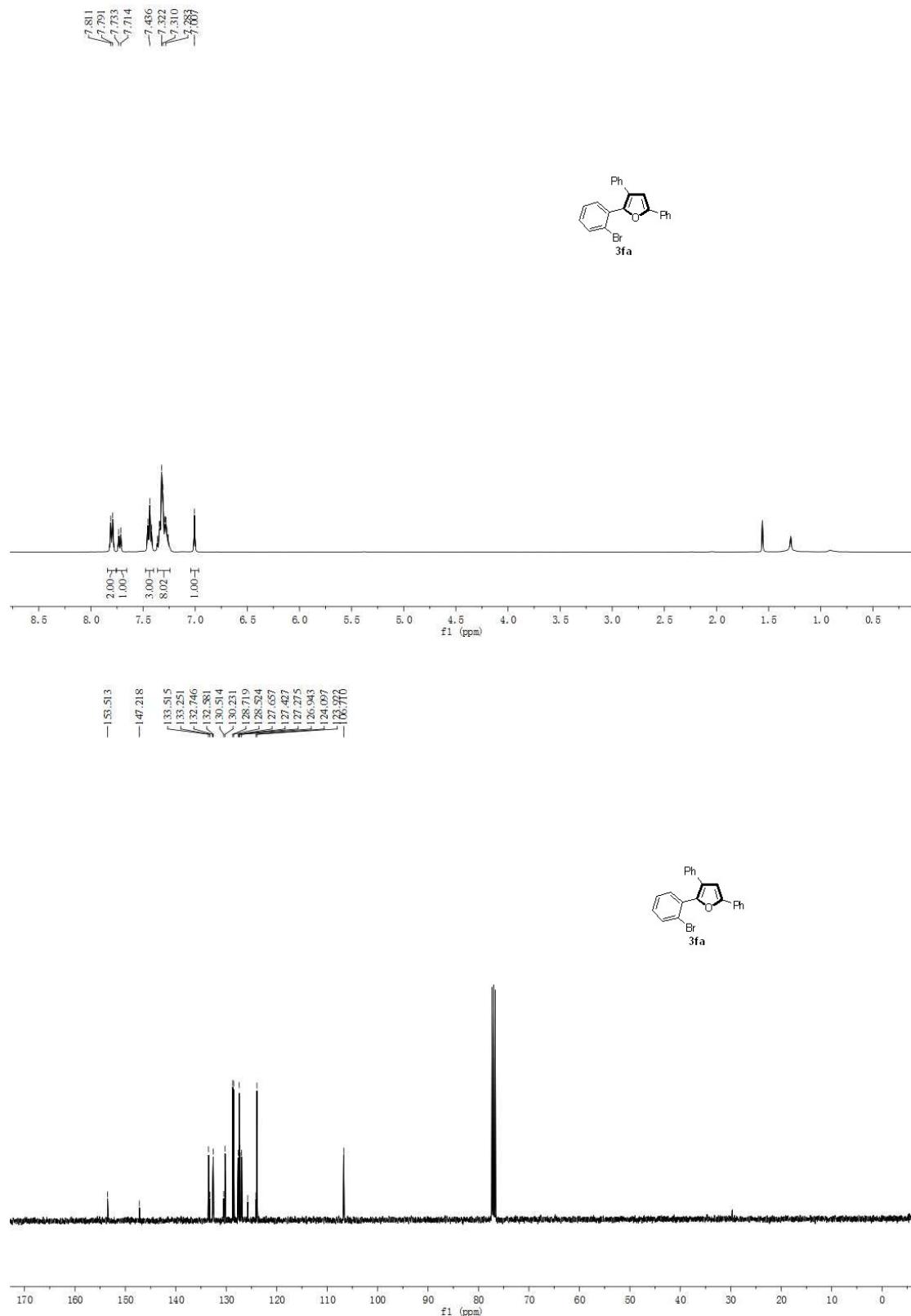


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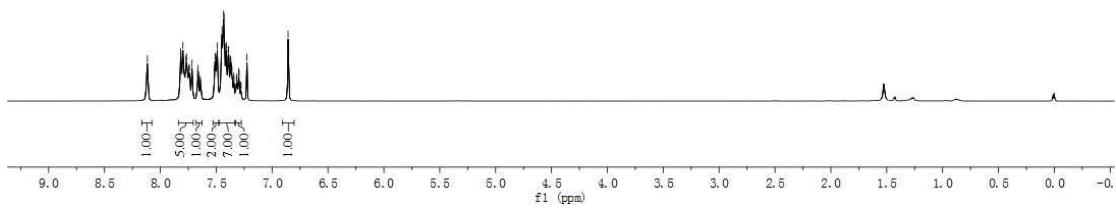
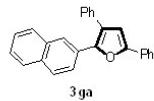


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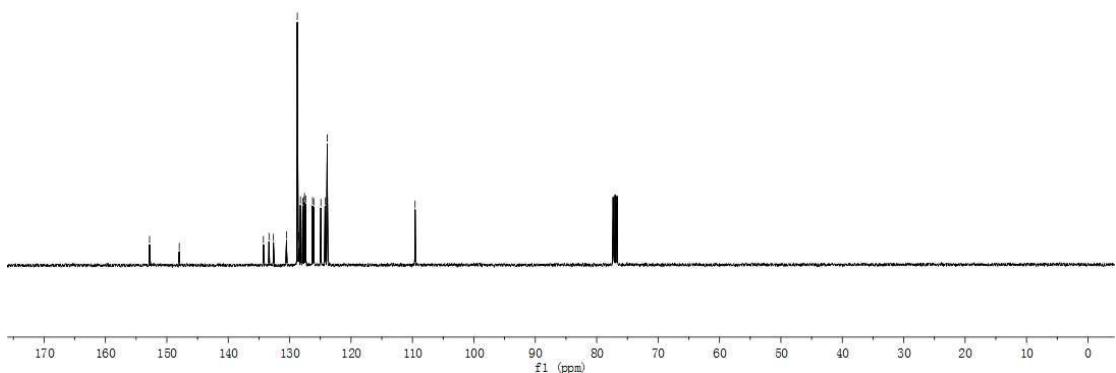
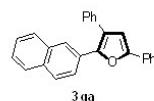


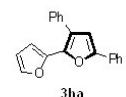
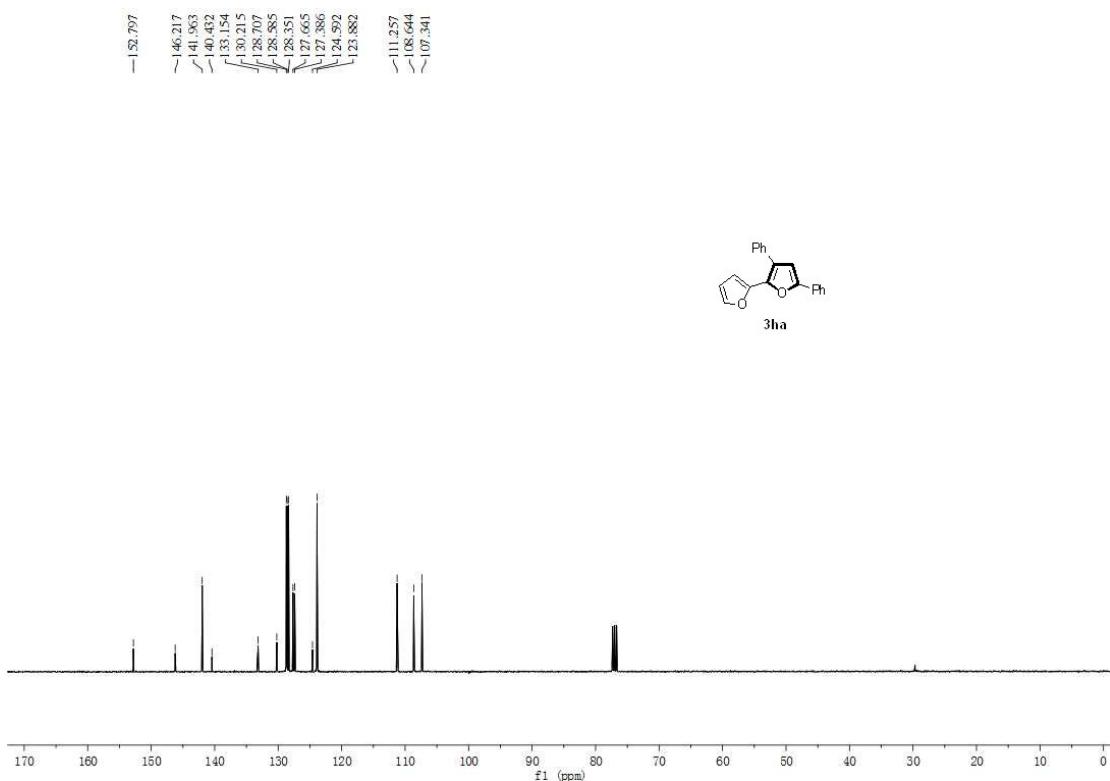
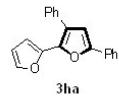
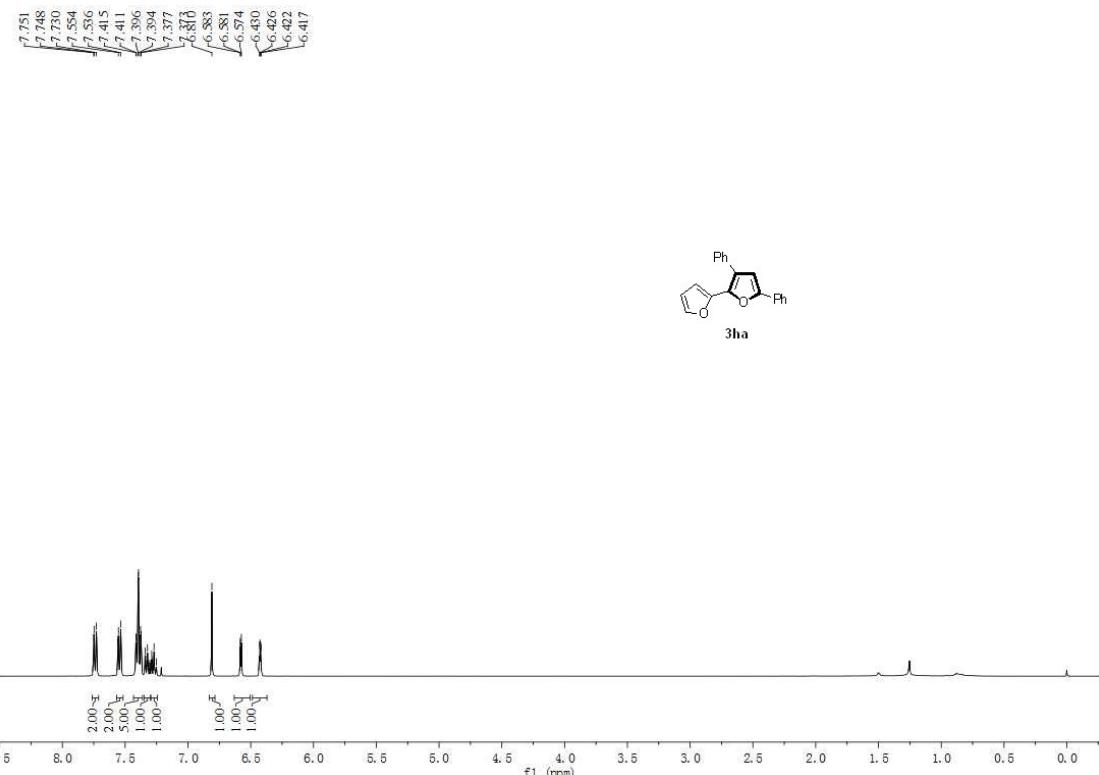


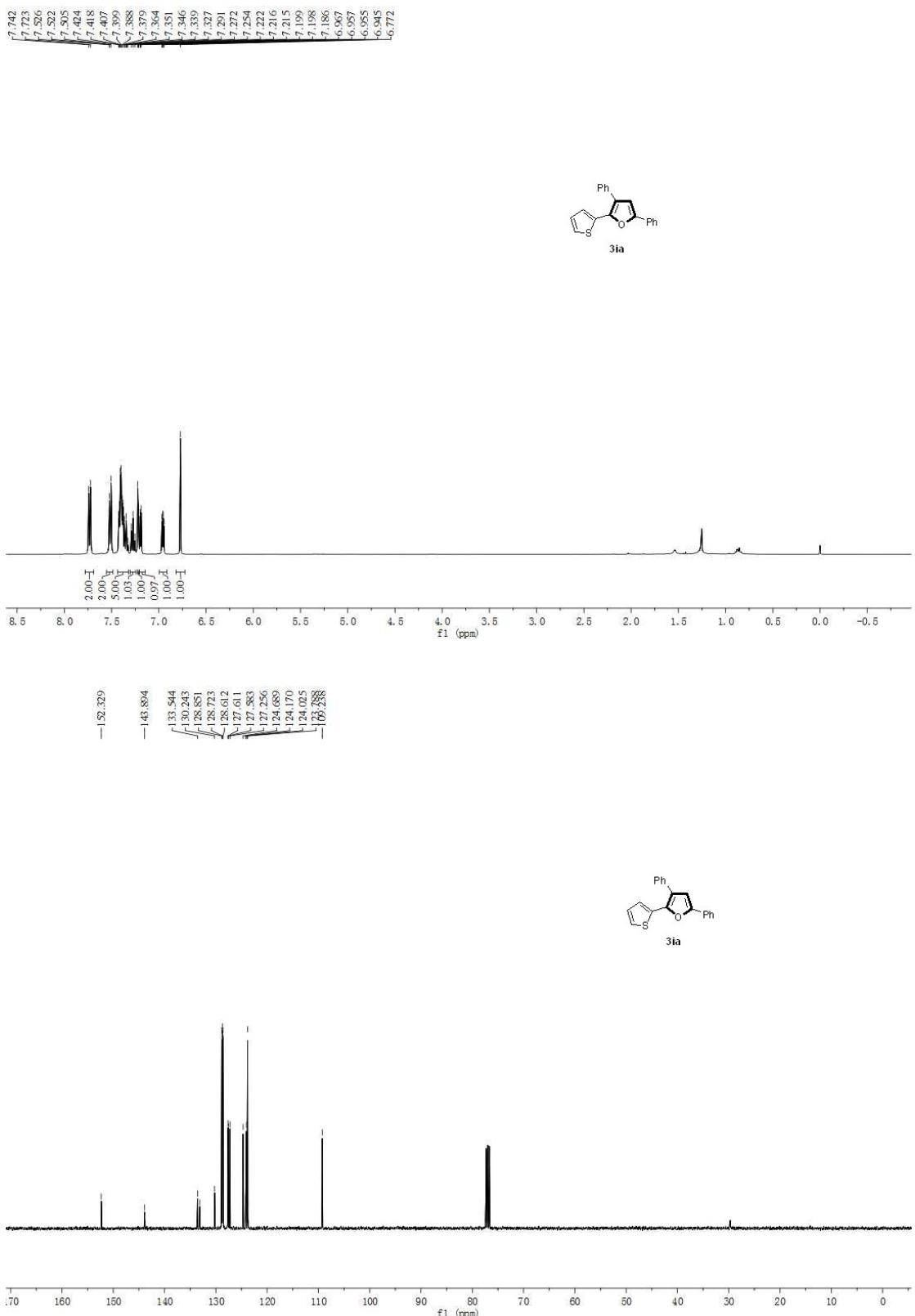
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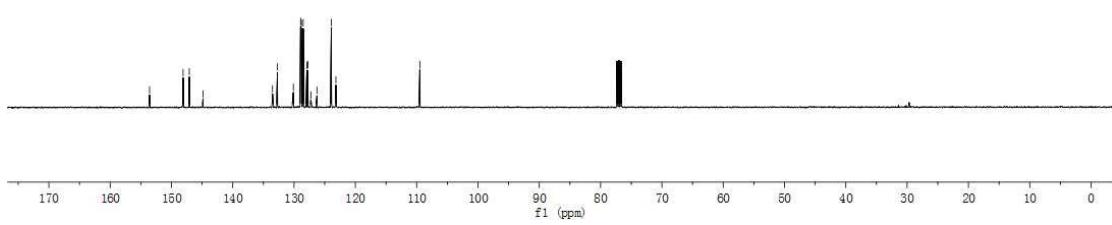
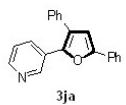
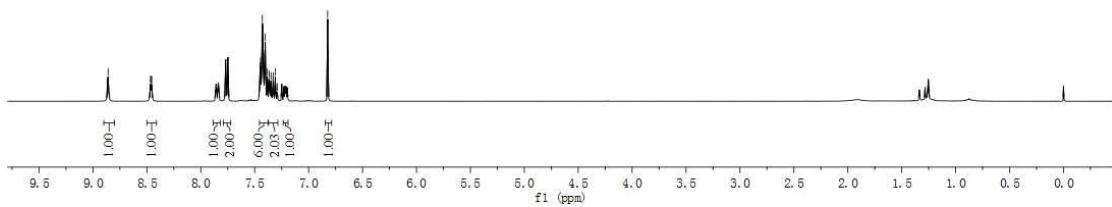
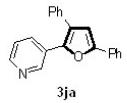
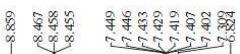


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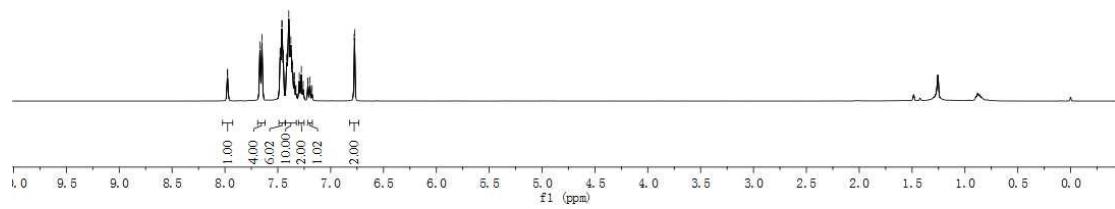




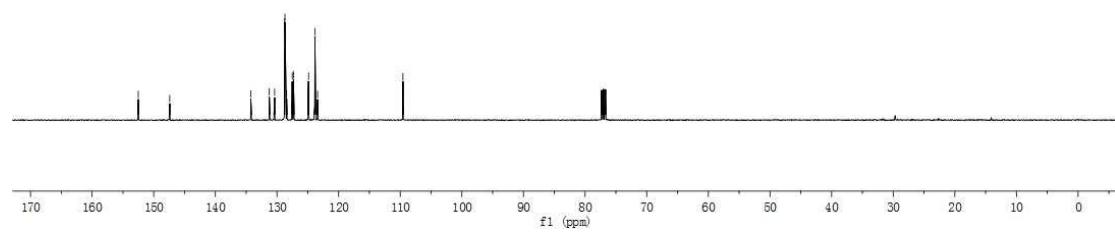
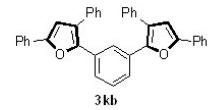




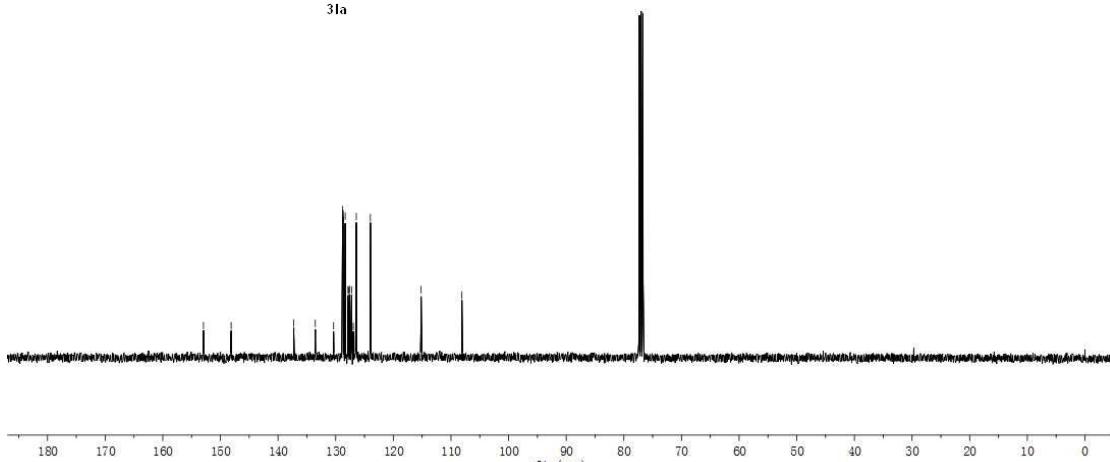
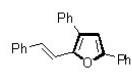
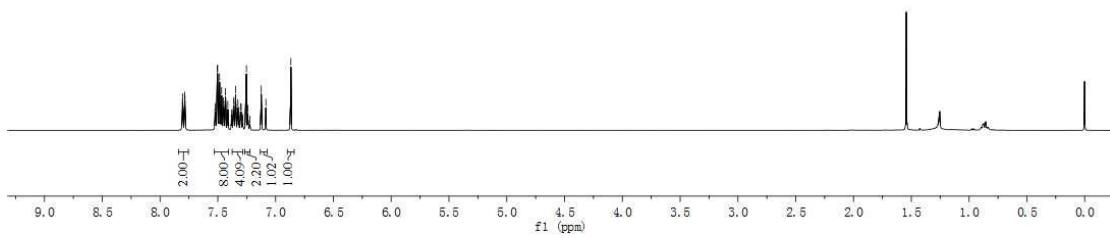
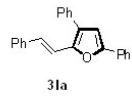
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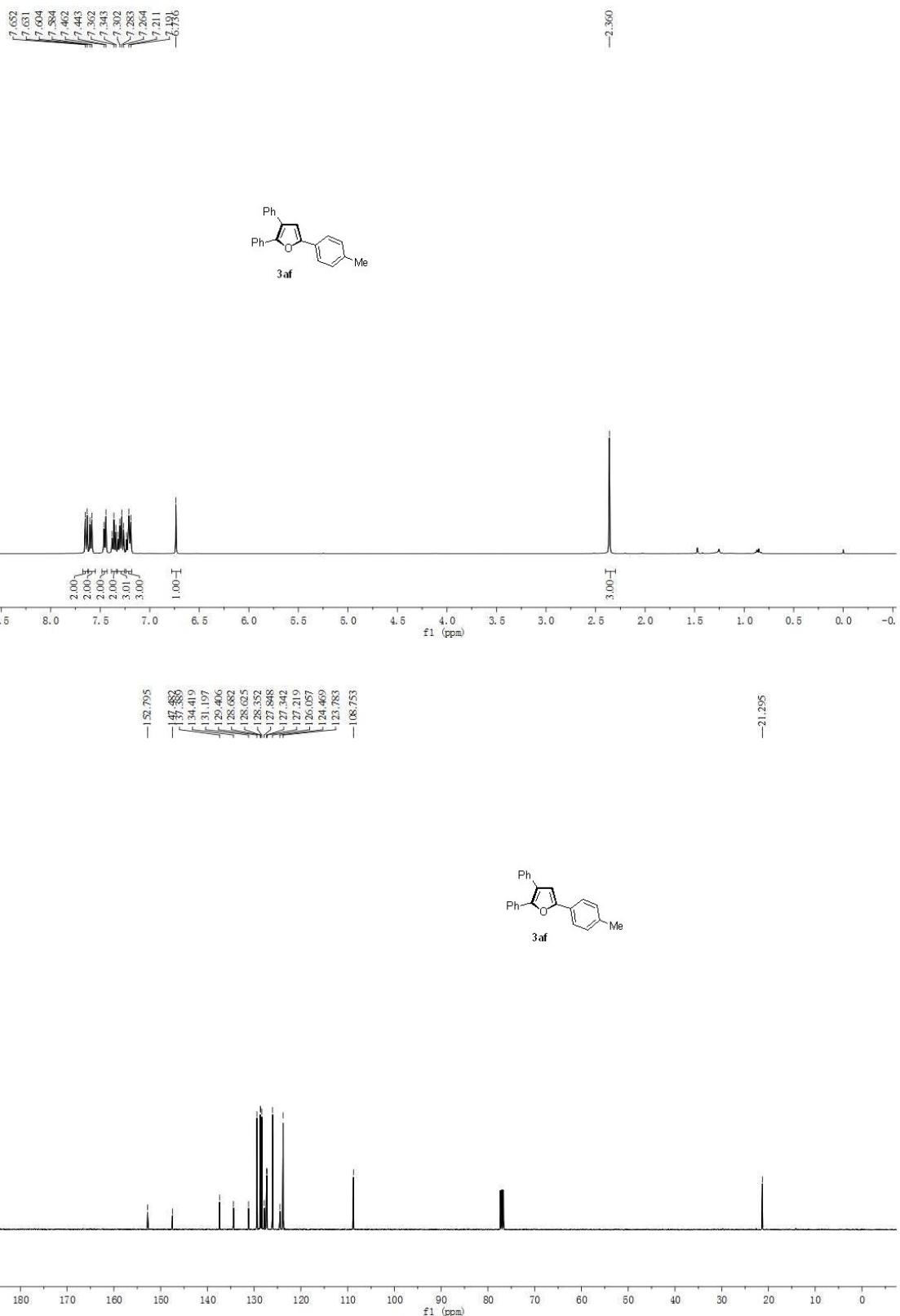


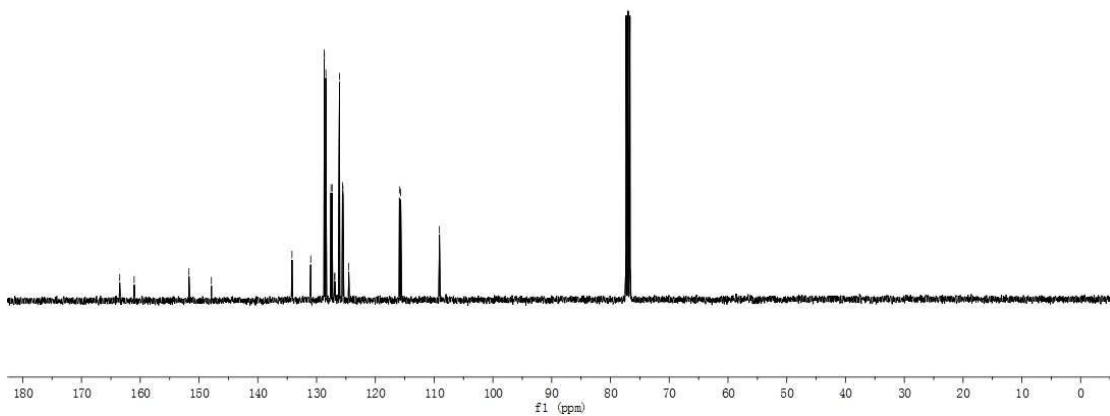
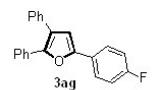
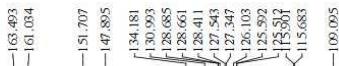
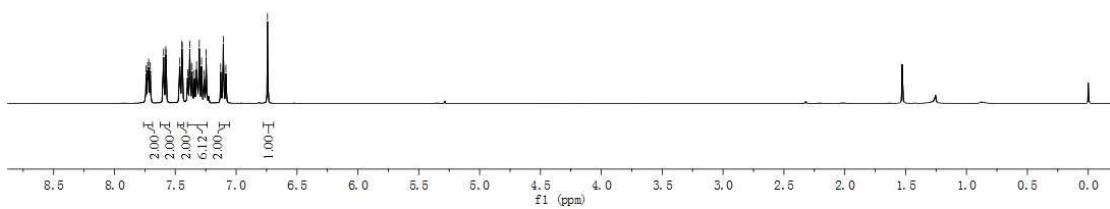
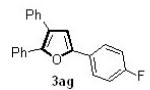
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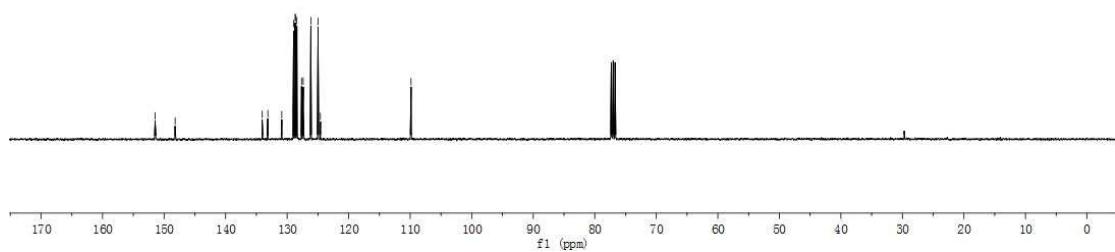
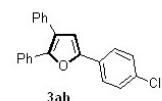
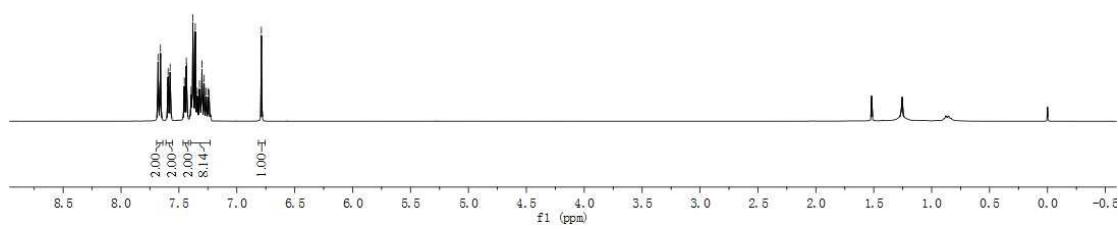
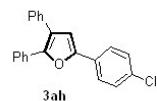
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7.735
7.734
7.732
7.731
7.730
7.729
7.728
7.728
7.727
7.726
7.725
7.723
7.721
7.720
7.719
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7.709
7.708
6.568



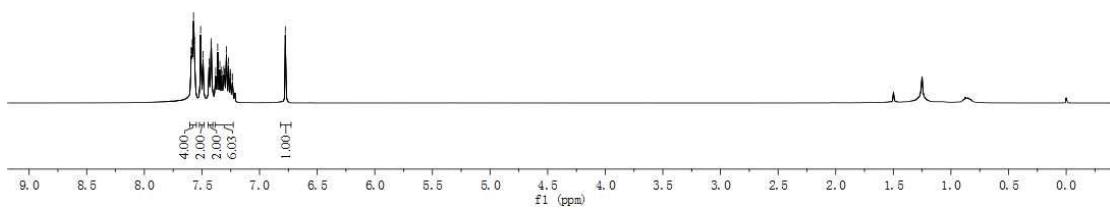
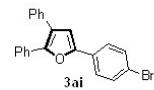




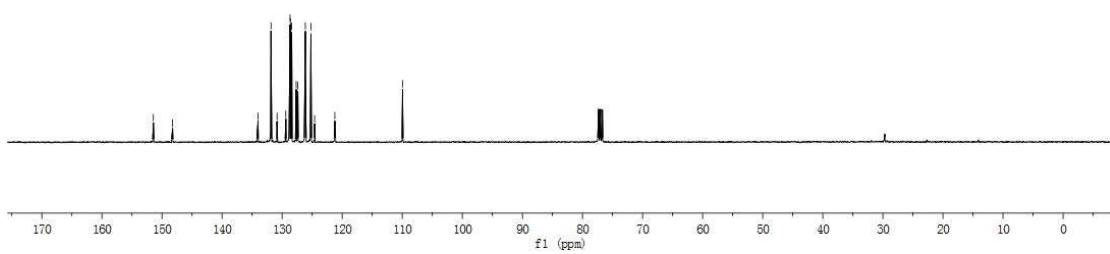
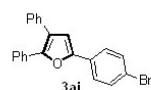
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7.301
7.282
6.788



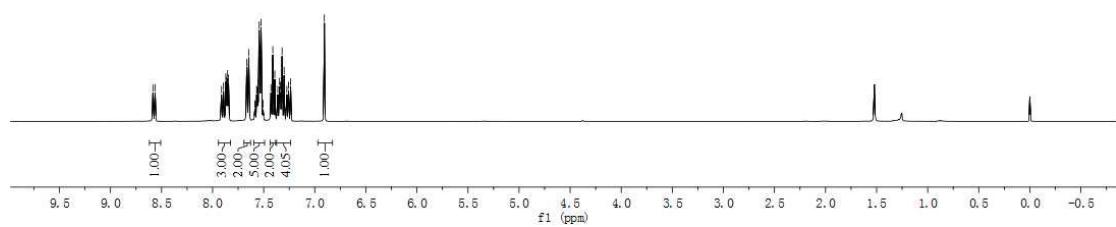
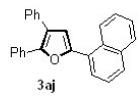
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7.567
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7.345
7.310
7.288
7.292
6.976



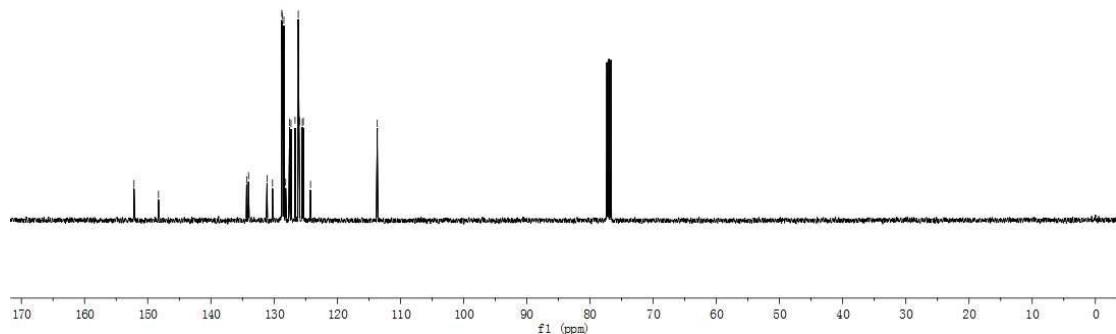
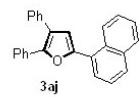
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-1.28,680
-1.28,632
-1.28,499
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-1.09,954



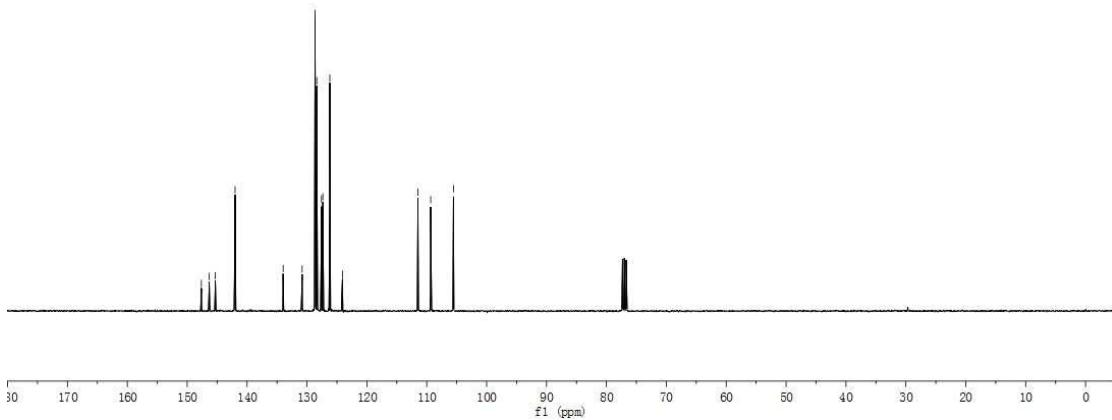
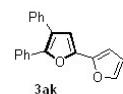
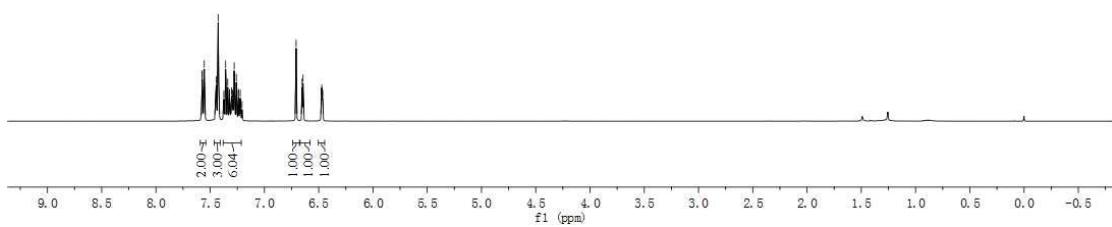
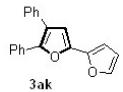
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7.644
7.544
7.540
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7.411
7.320
7.310

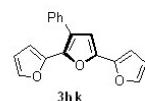
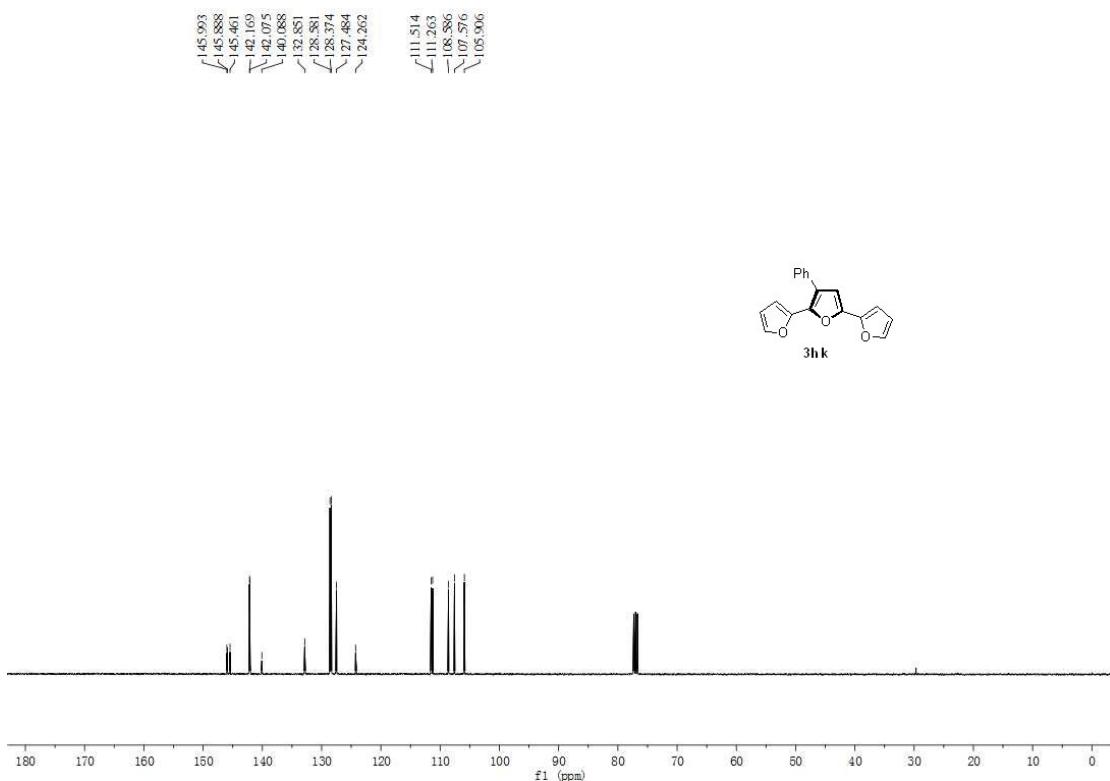
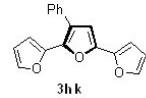
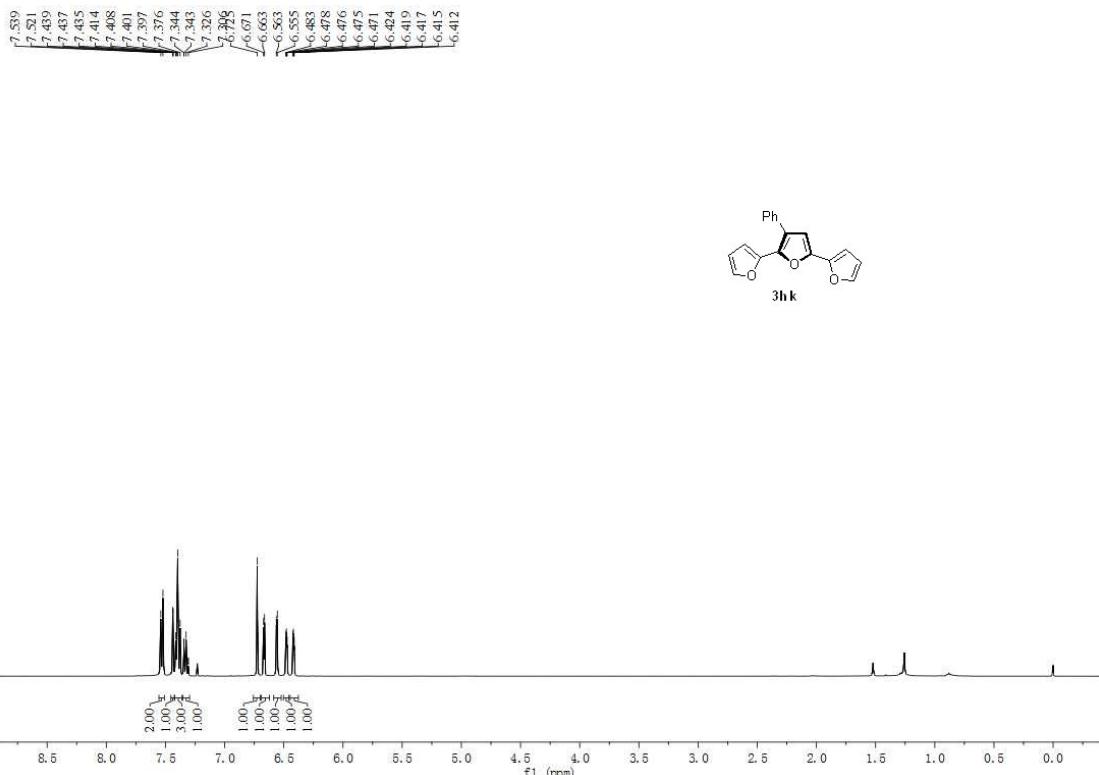


-1.52, 1.80
-1.48, 2.98
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128.671
128.605
128.446
127.543
126.680
126.169
126.060
125.975
125.530
125.368

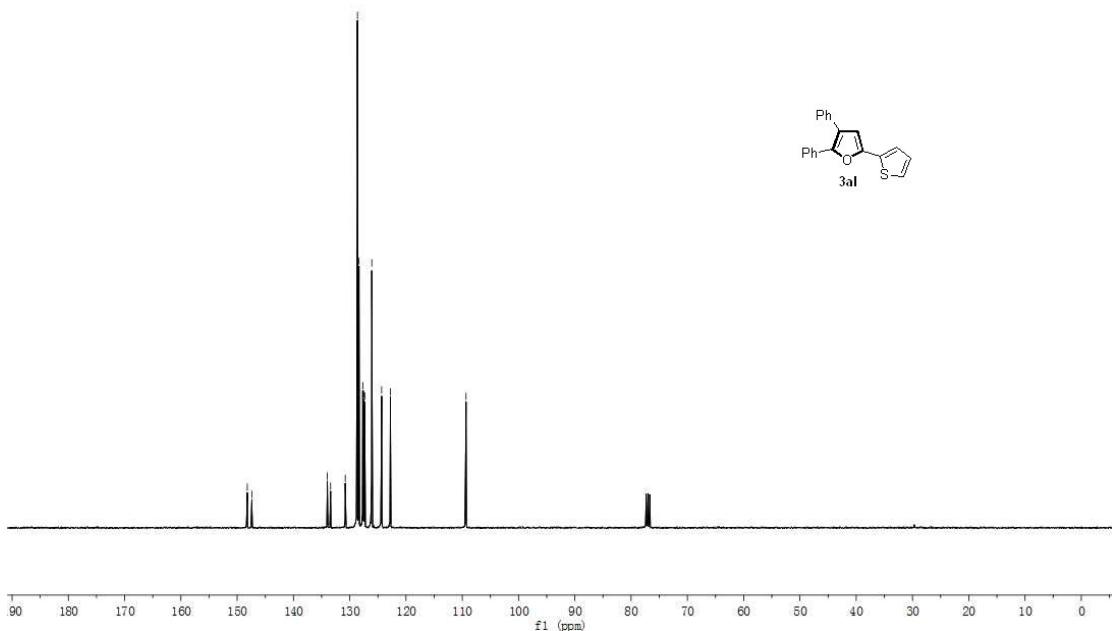
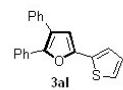
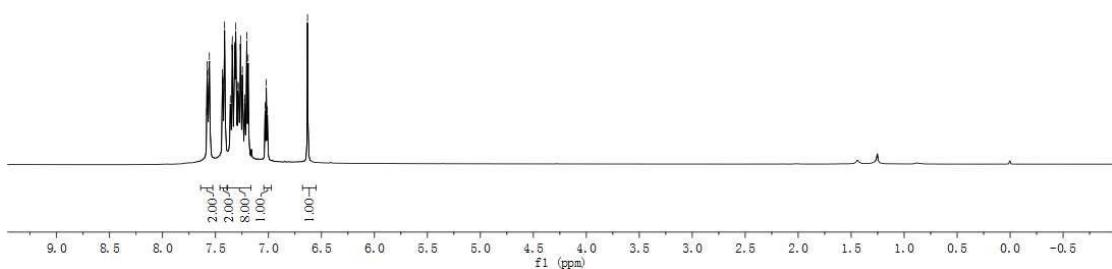
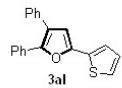


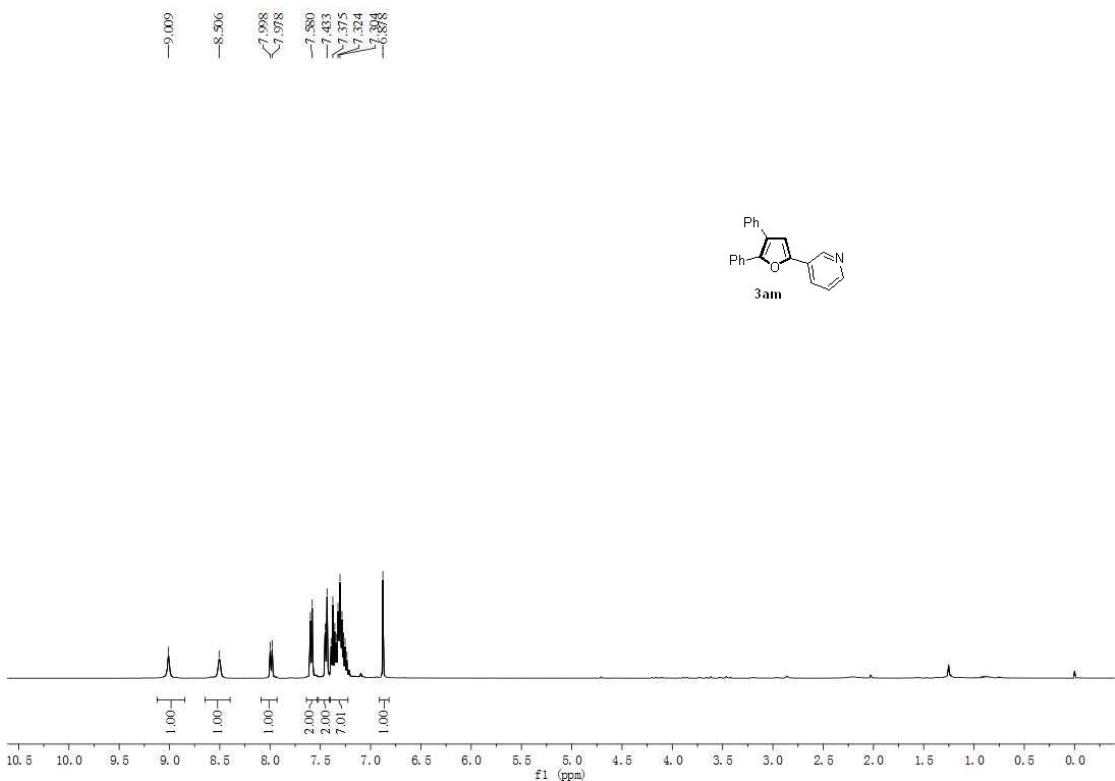
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 7.322
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 7.303
 7.297
 7.295
 7.279
 7.259
 7.240
 7.223
 6.768
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 6.644
 6.476
 6.474
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 6.468
 6.466
 6.464

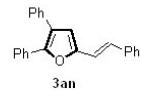
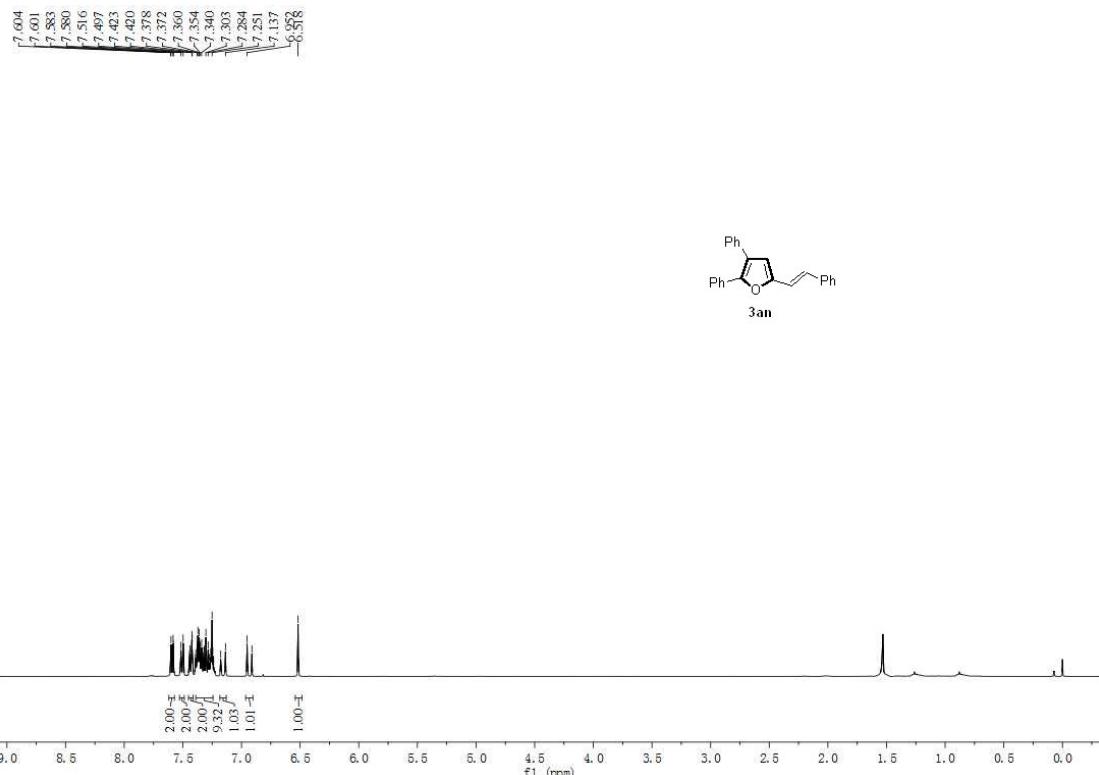




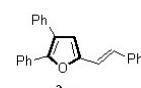
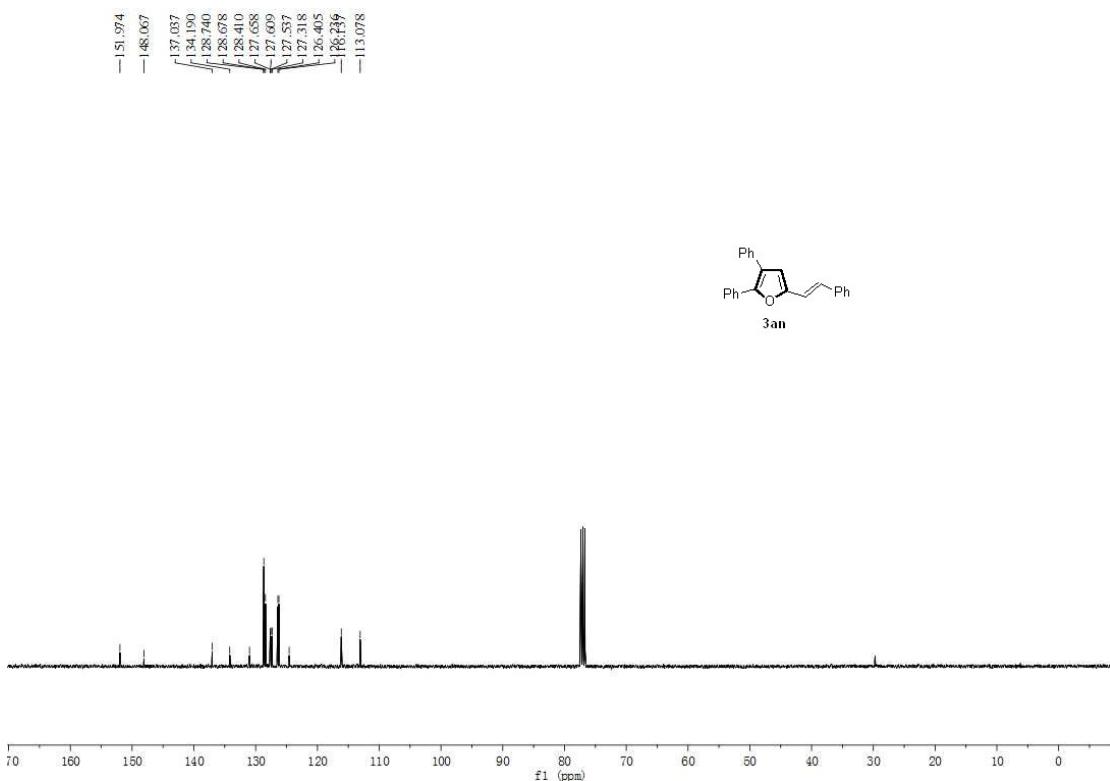
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7.484
7.481
7.450
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7.341
7.321
7.319
7.312
7.308
7.266
7.264
7.246
7.205
7.037



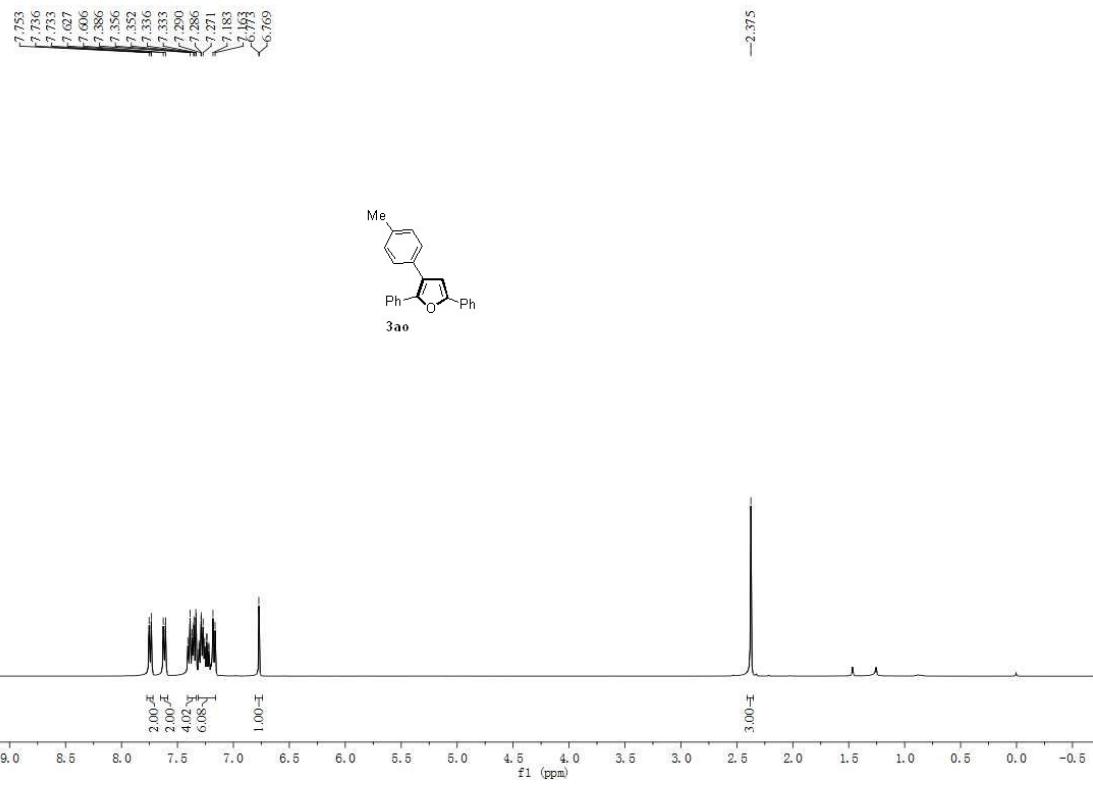




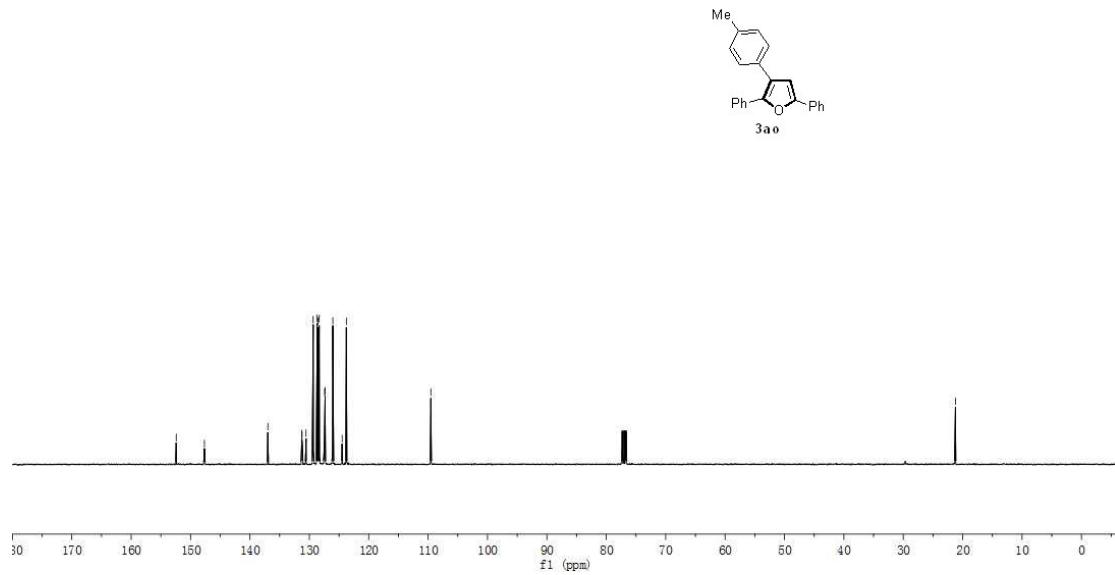
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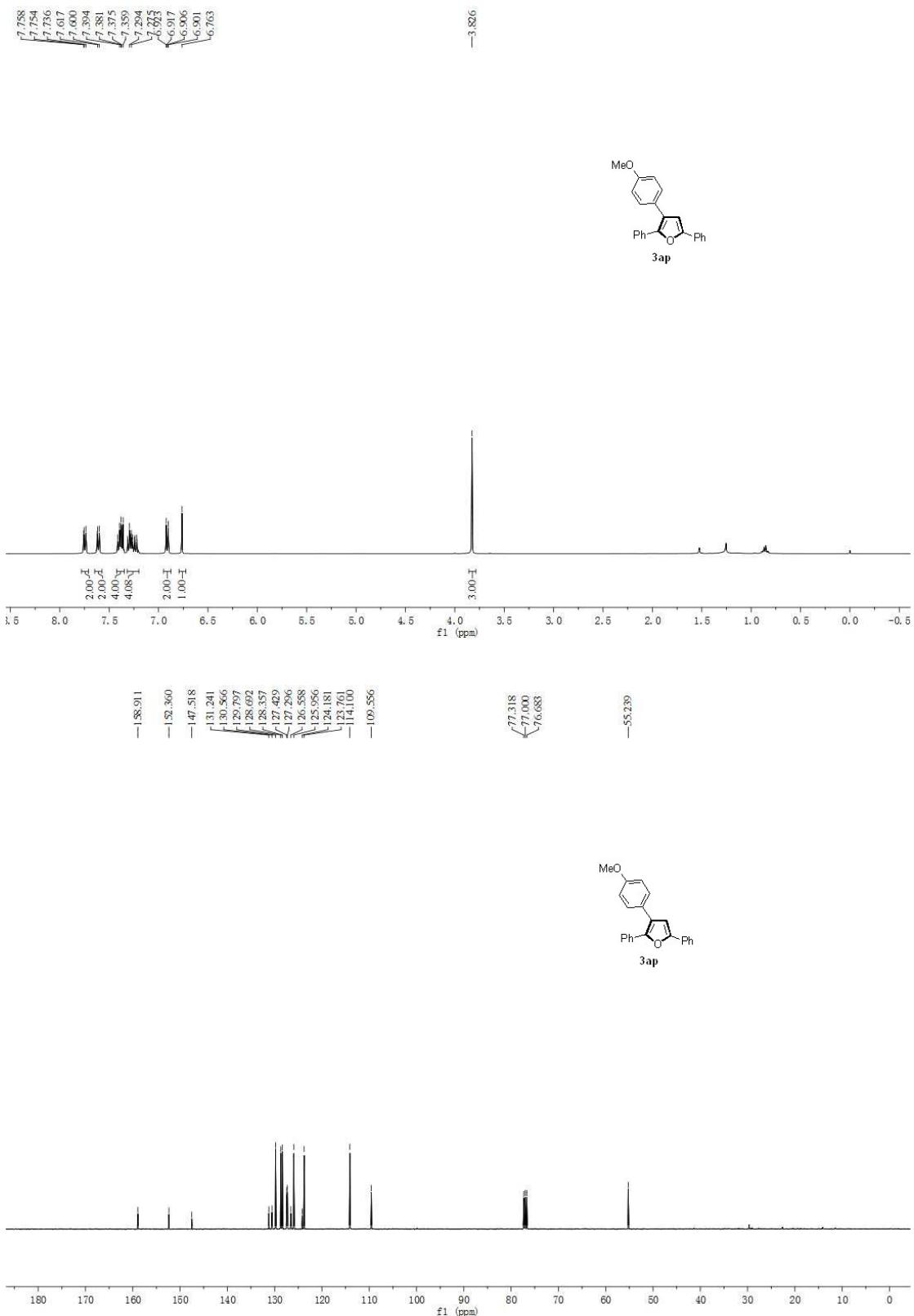


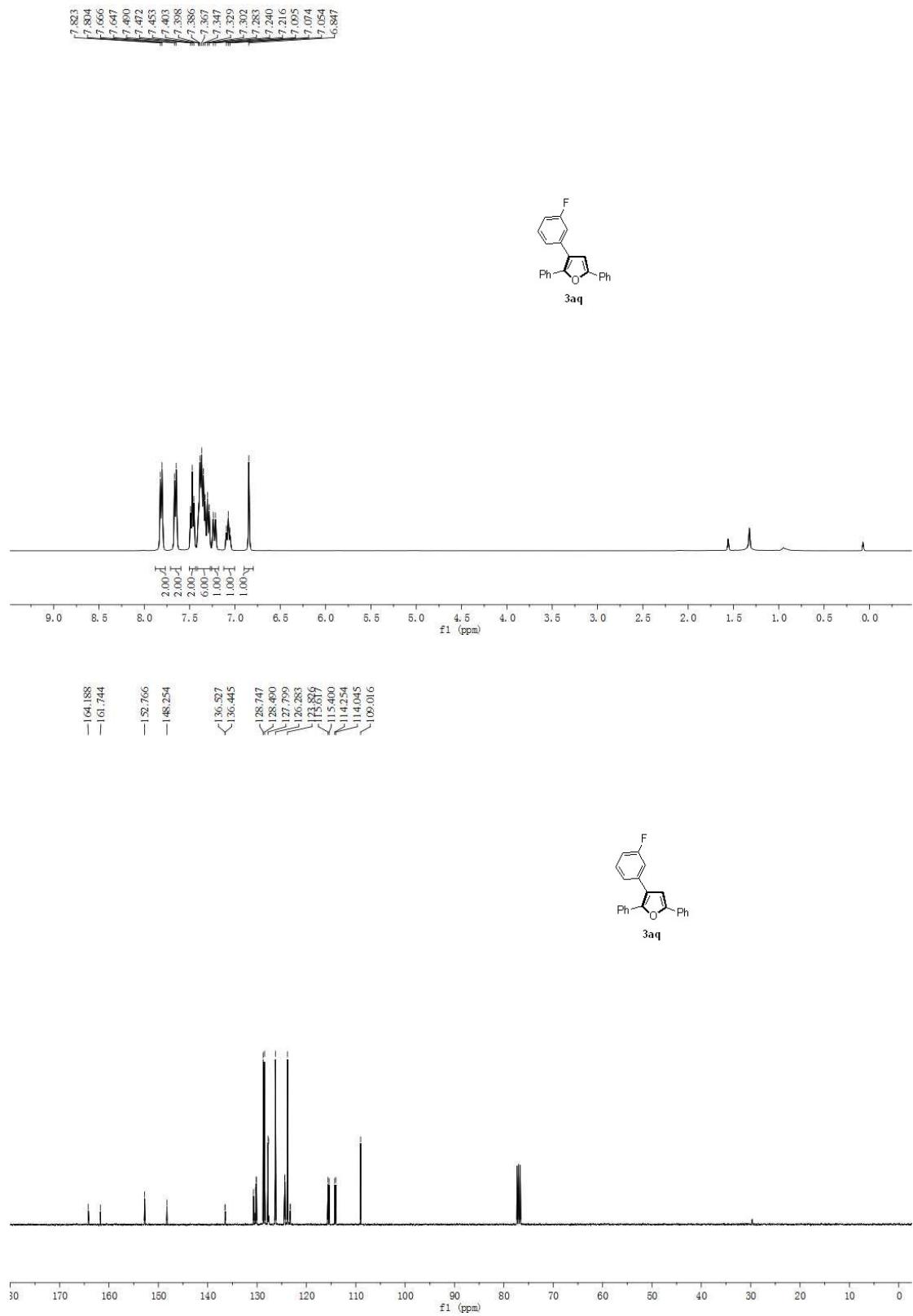
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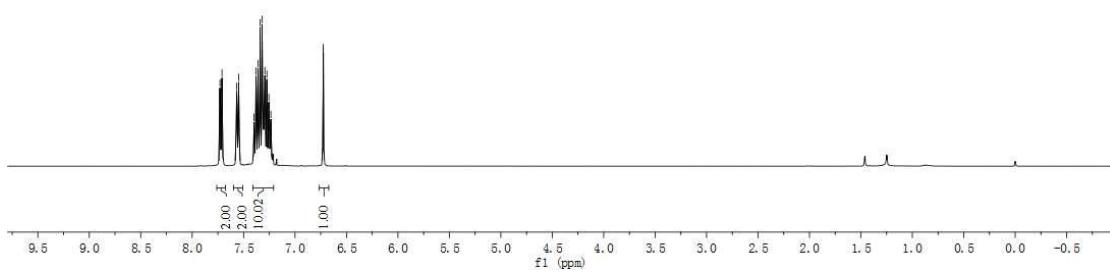
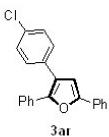
¹H NMR chemical shifts (δ , ppm): 7.753, 7.736, 7.733, 7.627, 7.606, 7.388, 7.356, 7.352, 7.336, 7.290, 7.286, 7.271, 7.183, 7.093, 6.969, 3.00, 2.375.



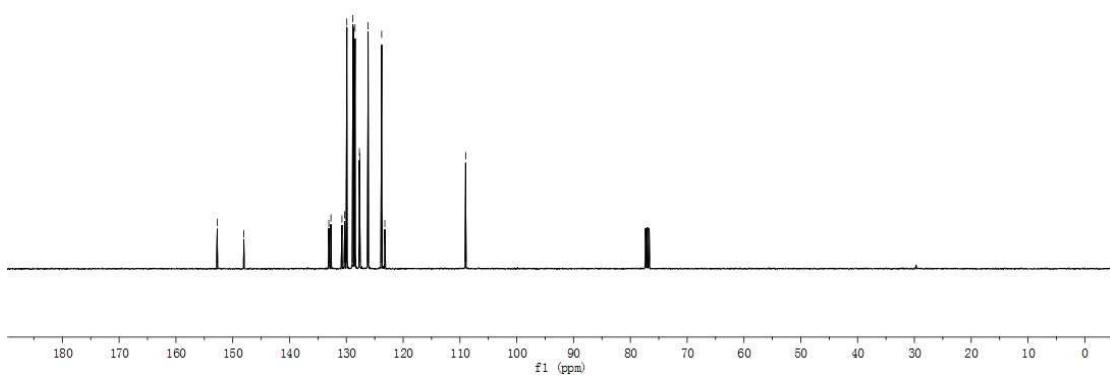
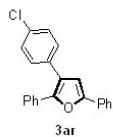




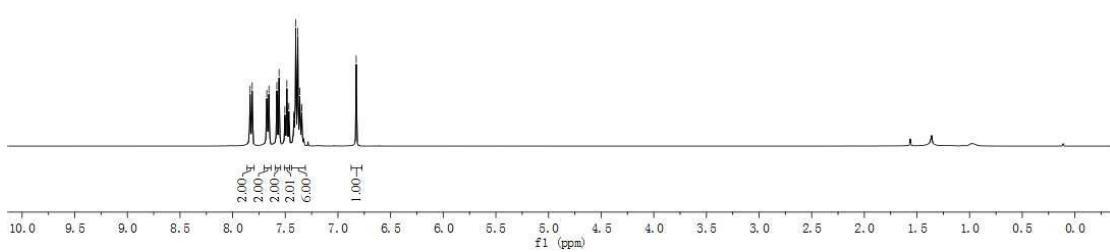
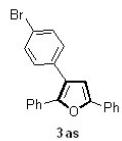
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7.274
7.254
7.235
6.727
6.725



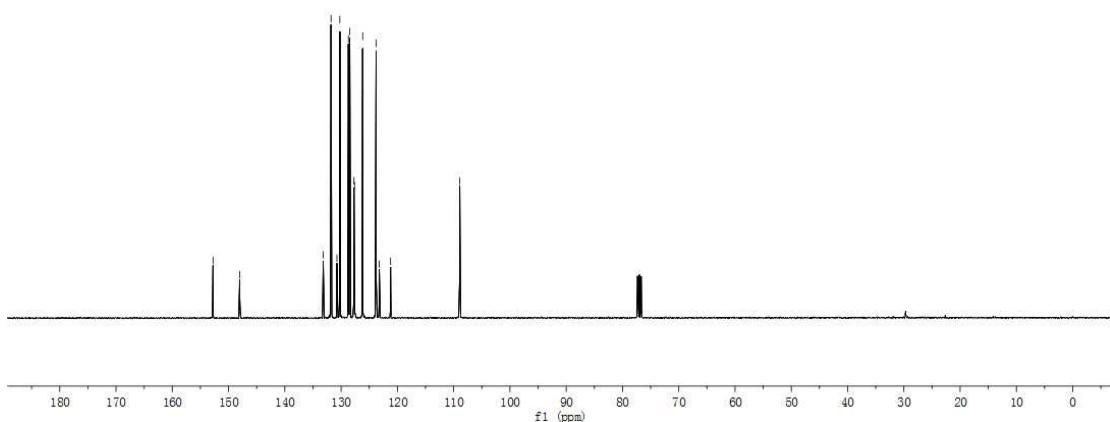
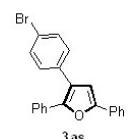
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-133.089
-132.716
-130.777
-129.918
-128.854
-128.715
-128.466
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-126.622
-126.167
-123.788
-123.223
-109.002



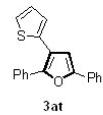
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7.466
7.419
7.416
7.401
7.381
7.362
7.343
6.826



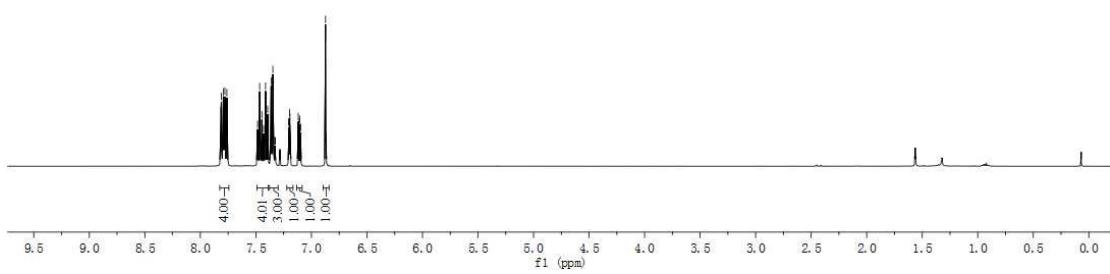
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-1.30, 2.56
-1.30, 2.22
-1.28, 7.04
-1.28, 4.64
-1.27, 7.17
-1.27, 6.20
-1.26, 1.68
-1.23, 7.81
-1.23, 2.09
-1.21, 2.10
-1.08, 9.11



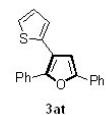
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7.7466
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7.7413
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7.7110
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7.7097
6.6873



3at



-1.52.540
-1.48.513
1.30.238
1.28.720
1.28.413
1.27.908
1.27.674
1.27.427
1.26.435
1.25.960
1.25.124
1.25.853



3at

