Synthesis of 1,2-Disubstituted Acenaphthylenes via Palladium-Catalyzed Annulation Reactions of Dibromoarenes with Internal Alkynes

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1. General Comments

All reactions were carried out under an atmosphere of nitrogen with freshly distilled solvents, unless otherwise noted. Column chromatography was performed on silica gel (particle size 10-40 μm, Ocean Chemical Factory of Qingdao, China). $^1$H NMR and $^{13}$C NMR spectra were recorded on Bruker 300M and Bruker 400M at ambient temperature with CDCl$_3$ as the solvent. High resolution mass analyses were performed on a Bruker-APEX II mass spectrometer. DMF was distilled from CaH$_2$ under argon before use.
2. Experimental Section

2.1 Preparation for Starting Materials

1) Preparation for dibromoarenes

1,8-dibromonaphthalene (1a)\(^{[1]}\): Solid NaNO\(_2\) (3.5 g, 0.05 mol) was dissolved in 20 mL H\(_2\)O and cooled to 0 °C. The sodium nitrite solution was added dropwise into a solution of 1H-naphtho[1,8-de][1,2,3]triazine (6.8 g, 0.04 mol) in 6.9M H\(_2\)SO\(_4\) (100 mL) at -5 °C over a period of 2 h with vigorous stirring with a mechanical stirrer. The mixture was stirred at -5 °C for an additional 2 h. The solution of CuBr (14.6 g, 0.1 mol) in aqueous HBr (47%, 50 mL) was dropwised into the resulting mixture and stirred at -5°C for 2 h. The reaction mixture was allowed to warm up to 85°C and then stirred for 1 h. The reaction mixture was extracted with dichloromethane and the combined extracts were dried with Na\(_2\)SO\(_4\) and evaporated in vacuum to produce crude product. Flash chromatography on silica gel with petroleum ether gave pure 1a (2.61 g, 23%) as a pale yellow crystal. Melting point: 104-106 °C. \(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) 7.92 (d, \(J = 7.5\) Hz, 2H), 7.79 (d, \(J = 9.0\) Hz, 2H), 7.24 (m, 2H).

5,6-dibromo-1,2-dihydroacenaphthylene (1b)\(^{[2]}\): Acenaphthene (4.6 g, 0.03 mol) and \(N\)-bromosuccinimide (13.4 g, 0.07 mol) were combined in DMF (70 mL) and stirred overnight at 30-32 °C. The reaction was then cooled to room temperature, filtered to obtain the crude product, and purified via recrystallization from ethanol to give 2.15 g of 1b as a beige crystal in 23% yield. Melting point: 169-170 °C (lit. 168-171 °C). \(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) 7.79 (m, 2H), 7.08 (m, 2H), 3.29 (m, 4H).
2) Preparation for alkynes

**Diphenylacetylene (2a):** To a double-necked flask were added phenylacetylene (1.02 g, 10 mmol), iodobenzene (2.45 g, 12 mmol), Pd(PPh₃)₄ (0.57 g, 0.5 mmol), CuI (0.09 g, 0.5 mmol), diisopropylamine (15 mL) and toluene (15 mL). The reaction mixture was stirred overnight at room temperature under nitrogen and then extracted with CH₂Cl₂. The combined extracts were washed with saturated NH₄Cl solution, brine, and dried over Na₂SO₄. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography (SiO₂, eluent: petroleum ether) to give 1.63 g of 2a as a colorless crystal in 92% yield. Melting point: 53-54 °C. ¹H NMR (300 MHz, CDCl₃): δ 7.87 (d, J = 6.0 Hz, 2H), 7.75 (d, J = 6.0 Hz, 2H), 7.58–7.62 (m, 2H), 7.44-7.46 (m, 4H), 7.29 – 7.39 (m, 6H).

**Methyl 4-(phenylethynyl)benzoate (2b):** To a double-necked flask were added phenylacetylene (0.49 g, 4.8 mmol), methyl 4-iodobenzoate (1.00 g, 4 mmol), Pd(PPh₃)₄ (0.2311 g, 0.2 mmol), CuI (0.04 g, 0.2 mmol), diisopropylamine (7 mL) and toluene (7 mL). The mixture was stirred overnight under nitrogen at room temperature and then extracted with CH₂Cl₂. The combined extracts were washed with aqueous NH₄Cl, brine, and dried over Na₂SO₄. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography (SiO₂, eluent: petroleum ether) to give 0.53 g of 2b as a pale powder in 56% yield. Melting point: 115-116 °C. ¹H NMR (300 MHz, CDCl₃): δ 7.95 (t, J = 3 Hz, 2H), 7.47 – 7.52 (m, 4H), 7.17-7.29 (m, 3H), 3.85 (m, 3H).
1-Chloro-4-(phenylethynyl)benzene (2c): To a double-necked flask were added phenylacetylene (0.61 g, 6 mmol), 1-chloro-4-iodobenzene (1.57 g, 6.6 mmol), Pd(PPh3)4 (0.34 g, 0.3 mmol), CuI (0.06 g, 0.3 mmol), diisopropylamine (5 mL) and toluene (15 mL). The reaction mixture was stirred overnight under nitrogen at room temperature and then extracted with CH2Cl2. The combined extracts were washed with aqueous NH4Cl, brine, and dried over Na2SO4. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography (SiO2, eluent: petroleum ether) to give 1.29 g of 2c as a pale powder in 98.0% yield. Melting point: 75-76 °C. 1H NMR (300 MHz, CDCl3): δ 7.51-7.54 (m, 2H), 7.44-7.47 (m, 2H), 7.31-7.36 (m, 4H).

1,3,5-Trimethyl-2-(phenylethynyl)benzene (2f): The cuprous iodide (24 mg, 0.12 mmol) and tetrakis(triphenylphosphine)palladium (43 mg, 0.06 mmol) were loaded into a Teflon-sealed flask. 2-iodo-1,3,5-trimethylbenzene (0.51 g, 2.08 mmol) and phenylacetylene (0.26 g, 2.5 mmol) were loaded into a separate flask and dissolved in triethylamine (40 mL). This solution was then cannula-transferred into the flask containing the catalysts, the solution was briefly degassed under vacuum, and the flask was sealed. The mixture was stirred under nitrogen at 80 °C for 6 h. The crude product was purified by column chromatography (SiO2, eluent: petroleum ether) to give 2g as a colorless liquid in 92.0% yield. (0.42 g, 1.91 mmol). 1H NMR (300 MHz, CDCl3): δ 7.51-7.54 (m, 2H), 7.33 (d, J = 7.1 Hz, 3H), 6.88 (s, 2H), 2.47 (s, 6H), 2.28 (s, 3H).
1-Ethyl-4-(phenylethynyl)benzene (2g): To a double-necked flask were added 1-ethyl-4-ethynylbenzene (0.26 g, 2 mmol), iodobenzene (0.49 g, 2.4 mmol), Pd(PPh₃)₄ (0.11 g, 0.1 mmol), CuI (0.02 g, 0.1 mmol), diisopropylamine (5 mL) and toluene (5 mL). The reaction mixture was stirred overnight under nitrogen at room temperature and extracted with CH₂Cl₂. The combined extracts were washed with aqueous NH₄Cl, brine, and dried over Na₂SO₄. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography (SiO₂, eluent: petroleum ether) to give 0.35 g of 2d as a colorless oil in 86.0% yield. ^1H NMR (400 MHz, CDCl₃): δ 7.50-7.52 (m, 2H), 7.43-7.45 (m, 2H), 7.30-7.33 (m, 3H), 7.15-7.20 (m, 2H), 2.64 (q, J = 6.0 Hz, 2H), 1.22 (t, J = 6.0 Hz, 3H).

Hex-1-ynylbenzene (2h): To an anhydrous THF solution (35 mL) of phenylacetylene (0.51 g, 5 mmol) was added 3.9 mL n-BuLi (10.5 mmol, 2.7 M solution in hexane) dropwise within 30 min under N₂ atmosphere at -20°C. 30 min later, n-BuI (1.93 g, 10.5 mmol) was added and the mixture was stirred for an additional 30 min at -20°C. The reaction temperature was raised to 25°C and remained for 30 minutes. The reaction mixture was extracted with CH₂Cl₂, washed with aqueous NH₄Cl, brine, and dried over Na₂SO₄. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography (SiO₂, eluent: petroleum ether) to give 0.38 g of 2f as a colorless oil in 48.0% yield. ^1H NMR (400 MHz, CDCl₃): δ 7.38-7.40 (m, 2H), 7.23 – 7.26 (m, 3H), 2.38-2.42 (m, 2H), 1.57 – 1.61 (m, 2H), 1.47 – 1.51 (m, 2H), 0.93-0.97 (m, 3H).
1-Phenyl-2-propylacetylene (2i): To a double-necked flask were added 1-pentyne (0.14 g, 2 mmol), iodobenzene (0.49 g, 2.4 mmol), Pd(PPh₃)₄ (0.11g, 0.1 mmol), CuI (0.02 g, 0.1 mmol), diisopropylamine (5 mL) and toluene (5 mL). The reaction mixture was stirred overnight under nitrogen at room temperature and extracted with CH₂Cl₂. The combined extracts were washed with aqueous NH₄Cl, brine, and dried over Na₂SO₄. After filtration, the solvents were evaporated. The crude product was subjected to column chromatography (SiO₂, eluent: petroleum ether) to give 0.16 g of 2e as a yellow oil in 56.0% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.38 – 7.40 (m, 2H), 7.24 – 7.27 (m, 3H), 2.38 (t, J = 6.0 Hz, 2H), 1.63 (q, J = 7.2 Hz, 2H), 1.05 (t, J = 7.4 Hz, 3H).

3) General procedure for synthesis of 1,2-disubstituted acenaphthylenes

Dibromoarene 1 (0.5 mmol), alkyne 2 (0.6 mmol), Pd(OAc)₂ (5 mol%, 5.6 mg), Xantphos (5.5 mol%, 16 mg), K₂CO₃ (1.5 mmol, 207 mg) were added into a Schlenk flask. The flask was evacuated and backfilled with nitrogen (3 cycles). Dry DMF (5 mL) was added by syringe. The mixture was heated to 120 °C and stirred until the reaction finished (TLC analysis). After cooling to room temperature, the mixture was extracted three times with ethyl acetate. The organic layers were combined and dried over Na₂SO₄. After filtration, the solvents were evaporated. The crude product was further purified by silica gel chromatography, using petroleum ether and ethyl acetate as eluents to provide the pure product.

The spectra of compounds 3a¹⁰, 3j¹³, 3k¹³ are identical to those reported in the literatures.

1,2-Diphenylacenaphthylene (3a):⁴ Orange solid, m.p. 161–163 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.67 (d, J = 8.0 Hz, 2H), 7.75 (d, J = 6.8 Hz, 2H), 7.60 (dd, J = 8, 3.6 Hz, 2H), 7.44–7.46 (m, 4H), 7.29–7.39 (m, 6H).

Methyl 4-(2-phenylacenaphthylene-1-yl)benzoate(3b): Orange solid, m.p. 166–167
$^\circ$C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 8.03 (d, $J = 8.3$ Hz, 2H), 7.88 (dd, $J = 8.0$, 2.2 Hz, 2H), 7.73–7.78 (m, 2H), 7.59–7.64 (m, 2H), 7.51 (d, $J = 8.3$ Hz, 2H), 7.35–7.43 (m, 5H), 3.93 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 167.1, 140.3, 139.7, 139.6, 139.4, 136.9, 134.8, 130.0, 129.7, 128.6, 128.6, 128.3, 127.9, 127.9, 127.8, 127.6, 127.5, 124.5, 124.0, 52.1. HRMS (EI): m/z calcd for C$_{26}$H$_{18}$O$_2$: 362.1307; Found: 362.1305.

1-(4-Chlorophenyl)-2-phenylacenaphthylene(3c): Orange solid, m.p. 122–123$^\circ$C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.87 (d, $J = 8.1$ Hz, 2H), 7.72 (dd, $J = 10.9$, 6.9 Hz, 2H), 7.58–7.63 (m, 2H), 7.31–7.44 (m, 9H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 157.2, 139.8, 139.6, 138.7, 136.8, 135.0, 133.8, 133.1, 131.4, 130.1, 128.7, 128.6, 128.2, 127.9, 127.6, 127.5, 127.4, 124.3, 123.8. HRMS (EI): m/z calcd for C$_{24}$H$_{15}$Cl: 338.0862; Found: 338.0862.

1,2-Bis(2-chlorophenyl)acenaphthylene(3d): Yellow solid, m.p. 152–154$^\circ$C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.85–7.88 (m, 2H), 7.54–7.62 (m, 4H), 7.41–7.45 (m, 2H), 7.28 (ddd, $J = 10.1$, 4.8, 2.7 Hz, 3H), 7.25–7.15 (m, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 139.3, 138.7, 138.1, 134.5, 134.1, 134.1, 133.8, 132.1, 132.1, 130.2, 129.5, 129.0, 128.8, 128.5, 128.0, 127.7, 127.7, 127.6, 126.7, 126.3, 124.7, 124.5. HRMS (EI): m/z calcd for C$_{24}$H$_{14}$Cl$_2$: 372.0473; Found: 362.0477.

1,2-Bis(4-tert-butylphenyl)acenaphthylene(3e): Orange solid, m.p. 164–165$^\circ$C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.83(d, $J = 8.1$ Hz, 2H), 7.75 (d, $J = 6.8$ Hz, 2H), 7.54–7.59 (m, 2H), 7.35–7.42 (m, 8H), 1.36 (s, 18H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 149.9, 140.4, 137.6, 132.4, 129.7, 128.3, 128.3, 127.7, 127.0, 125.2, 123.9, 34.6, 31.4. HRMS (EI): m/z calcd for C$_{32}$H$_{32}$: 416.2504; Found: 416.2507.

1-Mesityl-2-phenylacenaphthylene(3f): Yellow oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.80 –7.85 (m, 3H), 7.60 (dd, $J = 8.0$, 7.1 Hz, 1H), 7.50 (dd, $J = 8.1$, 6.9 Hz, 1H), 7.37–7.39 (m, 2H), 7.27 –7.32 (m, 3H), 7.24 (d, $J = 2.6$ Hz, 1H), 6.91 (s, 2H), 2.33 (s,
1-(4-Ethylphenyl)-2-phenylacenaphthylene (3g): Orange oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.83 (dd, $J = 8.1$, 2.3 Hz, 2H), 7.73 (dd, $J = 14.9$, 6.8 Hz, 2H), 7.55-7.59 (m, 2H), 7.44–7.46 (m, 2H), 7.34–7.38 (m, 4H), 7.29–7.31 (m, 1H), 7.18 (d, $J = 8.2$ Hz, 2H), 2.68 (q, $J = 7.6$ Hz, 2H), 1.27 (t, $J = 7.6$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 143.2, 140.2, 140.1, 138.2, 137.7, 135.5, 132.5, 130.1, 130.0, 128.4, 128.4, 128.3, 127.9, 127.8, 127.2, 127.1, 127.0, 124.1, 123.8, 28.7, 15.3. HRMS (EI): m/z calcd for C$_{26}$H$_{20}$: 332.1565; Found: 332.1567.

1-Butyl-2-phenylacenaphthylene (3h): Yellow oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.69–7.79 (m, 3H), 7.51–7.58 (m, 7H), 7.36–7.49 (m, 1H), 2.87 (t, $J = 7.6$ Hz, 2H), 1.75 (t, $J = 7.6$ Hz, 2H), 1.42 (dd, $J = 14.8$, 7.4 Hz, 2H), 0.91 (t, $J = 7.3$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 140.7, 139.9, 137.9, 135.8, 129.6, 128.5, 128.5, 128.2, 127.7, 127.7, 127.1, 126.9, 126.4, 122.6, 122.4, 33.6, 26.1, 23.1, 14.0. HRMS (EI): m/z calcd for C$_{22}$H$_{20}$: 284.1565; Found: 284.1566.

1-Phenyl-2-propylacenaphthylene (3i): Yellow oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.69–7.79 (m, 3H), 7.47–7.58 (m, 7H), 7.38 (t, $J = 7.2$ Hz, 1H), 2.84 (t, $J = 7.2$ Hz, 2H), 1.76-1.82 (m, 2H), 1.00 (t, $J = 7.3$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 140.6, 140.6, 139.6, 138.0, 135.7, 129.5, 128.4, 128.3, 128.1, 127.7, 127.6, 127.0, 126.9, 126.4, 122.6, 122.3, 28.4, 24.6, 14.5. HRMS (EI): m/z calcd for C$_{21}$H$_{18}$: 270.1409; Found: 270.1410.

1,2-Dipropylacenaphthylene (3j): Pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.66–7.69 (m, 1H), 7.52 (d, $J = 6.5$ Hz, 1H), 7.46 (dd, $J = 7.9$, 6.9 Hz, 1H), 2.70 (t, $J = 6.5$ Hz, 2H), 1.69–1.75 (m, 2H), 1.02 (t, $J = 7.4$ Hz, 3H).
**Dimethyl acenaphthylene-1,2-dicarboxylate (3k):** Dark red solid. m.p. 99–101°C. 

$^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 8.07 (d, $J = 6.8$ Hz, 2H), 7.92 (d, $J = 8.0$ Hz, 2H), 7.56–7.61 (m, 2H), 3.94 (s, 6H).

**7,8-Bis(4-tert-butylphenyl)-3,4-dihydrocyclopenta[f,g]acenaphthylene (3l):**

Orange solid, m.p. 170–171°C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.81 (d, $J = 7.0$ Hz, 2H), 7.46–7.48 (m, 4H), 7.42 (d, $J = 7.0$ Hz, 2H), 7.36–7.38 (m, 4H), 3.52 (s, 4H), 1.36 (s, 18H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 149.6, 146.5, 137.1, 136.2, 135.3, 133.5, 129.6, 127.0, 126.0, 125.2, 120.7, 34.6, 32.6, 31.4. HRMS (EI): m/z calcd for C$_{34}$H$_{34}$: 442.2661; Found: 442.2658.

**7,8-Diphenyl-3,4-dihydrocyclopenta[f,g]acenaphthylene (3m):** Orange solid, m.p. 219–221°C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.82 (d, $J = 6.9$ Hz, 2H), 7.51–7.53 (m, 4H), 7.46 (d, $J = 7.0$ Hz, 2H), 7.34–7.38 (m, 4H), 7.30 (m, 2H), 3.55 (s, 4H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 146.9, 137.6, 136.3, 135.7, 135.4, 130.0, 128.4, 126.8, 126.0, 120.8, 32.6. HRMS (EI): m/z calcd for C$_{26}$H$_{18}$: 330.1409; Found: 330.1399.

**7,8-Bis(2-chlorophenyl)-3,4-dihydrocyclopenta[f,g]acenaphthylene (3n):** Yellow solid, m.p. 207–210°C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.69 (dd, $J = 15.4$, 6.9 Hz, 2H), 7.41–7.45 (m, 4H), 7.30 (d, $J = 6.0$ Hz, 1H), 7.11–7.29 (m, 5H), 3.52 (s, 4H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 147.3, 147.3, 138.2, 137.6, 135.4, 135.1, 134.0, 133.9, 132.3, 132.2, 130.2, 129.5, 128.7, 128.5, 126.8, 126.6, 126.2, 120.7, 120.6, 32.7. HRMS (EI): m/z calcd for C$_{26}$H$_{16}$Cl$_2$: 398.0629; Found: 398.0616.

**7-Butyl-8-phenyl-3,4-dihydrocyclopenta[f,g]acenaphthylene (3o):** Yellow solid, m.p. 87–88°C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.76 (d, $J = 6.9$ Hz, 1H), 7.58–7.62 (m, 3H), 7.47–7.50 (m, 2H), 7.34–7.41 (m, 3H), 3.48 (s, 4H), 2.94 (t, $J = 6.5$ Hz, 2H), 1.80 (t, $J = 6.4$ Hz, 2H), 1.41 –1.50 (m, 2H), 0.92 (t, $J = 7.3$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 146.3, 145.7, 139.7, 137.4, 136.7, 136.5, 136.3, 135.1, 129.5, 128.4,
127.0, 126.7, 124.6, 124.6, 120.5, 120.5, 33.6, 32.6, 32.4, 27.0, 23.0, 14.0. HRMS(EI): m/z calcd for C_{24}H_{22}: 310.1722; Found: 310.1718.

7-Phenyl-8-propyl-3,4-dihydrocyclopenta[f,g]acenaphthylene(3p): Yellow solid, m.p. 105–106 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.75 (d, \(J = 6.9\) Hz, 1H), 7.58–7.62 (m, 3H), 7.48 (t, \(J = 7.6\) Hz, 2H), 7.34–7.40 (m, 3H), 3.48 (s, 4H), 2.92 (t, \(J = 6.5\) Hz, 2H), 1.82–1.87 (m, 2H), 1.01 (t, \(J = 7.3\) Hz, 3H). \(^1^3\)C NMR (101 MHz, CDCl\(_3\)): \(\delta\) 146.3, 145.7, 139.5, 137.6, 136.7, 136.5, 136.3, 135.1, 129.5, 128.4, 126.9, 126.7, 124.6, 124.6, 120.6, 120.5, 32.6, 32.4, 29.4, 24.7, 14.5. HRMS (EI): m/z calcd for C\(_{23}\)H\(_{20}\): 296.1565; Found: 296.1568.

4) Procedure for synthesis of dibenzo[j,l]fluoranthene using 3d:

A mixture of Cs\(_2\)CO\(_3\) (391 mg, 1.2 mmol), PdCl\(_2\)(PCy\(_3\))\(_2\) (59 mg, 0.08 mmol), 1,2-bis(2-chlorophenyl)acenaphthylene 3d (149.2 mg, 0.4 mmol) and NMP (4 mL) in a thick-wall pyrex tube was purged with nitrogen for 5 min. The sealed tube was heated at reflux for 24 h. After cooling to room temperature, the suspension was diluted with CH\(_2\)Cl\(_2\) (10 mL) and filtered through a 3 cm thick layer of Celite. The solvents of the filtrate were removed under reduced pressure, and the residue was subjected to chromatography on silica gel. Elution with PE/EA (20:1) gave 4 (58 mg, 48%) as yellow needle crystal. \(^1\)H NMR(400 MHz, CDCl\(_3\)): \(\delta\) 8.93 (d, \(J = 7.5\) Hz, 2H), 8.82 (d, \(J = 7.9\) Hz, 2H), 8.58 (d, \(J = 7.1\) Hz, 2H), 7.91 (d, \(J = 8.2\) Hz, 2H), 7.81–7.67 (m, 6H). \(^1^3\)C NMR (101 MHz, CDCl\(_3\)): \(\delta\) 137.9, 133.8, 132.0, 130.9, 129.9, 129.4, 128.0, 127.6, 127.2, 126.2, 125.0, 123.7.
3. Copies of $^1$H NMR and $^{13}$C NMR Spectra
4. References


