Supporting Information
for DOI: 10.1055/s-0029-1219957
© Georg Thieme Verlag KG Stuttgart · New York 2010
Supporting Information

Synthesis and Structures of 1,10-Phenanthroline-Based Extended Triptycene Derivatives

Yi Jiang, a,b Chuan-Feng Chen*, a

aBeijing National Laboratory for Molecular Sciences, CAS Key Laboratory of Molecular Recognition and Function, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, China. bGraduate School, Chinese Academy of Sciences, Beijing 100049, China

E-mail: cchen@iccas.ac.cn

1. Synthesis and Characterizations for Compounds 2b-d and 1d

2. Copies of ¹H NMR and ¹³C NMR Spectra of New Compounds
1. Synthesis and Characterizations for Compounds 2b-d and 1d

Typical Preparation Procedure and Characterizations for of 2b-d

To a solution of compound 4 (1 mmol) in MeOH (50 mL) was added 3b-d (1.3 mmol). The solution was stirred under N2 overnight to give a yellow solution. The solvent was removed by rotary evaporation and the red-orange residue was chromatographed on silica gel to give the product.

2b: Yellow solid; yield 70%. Mp > 300 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta =$ 9.45 (d, J = 8.3 Hz, 2H), 8.20 (s, 2H), 7.60 (d, J = 8.3 Hz, 2H), 7.53-7.49 (m, 4H), 7.12-7.09 (m, 4H), 5.72 (s, 2H), 2.99 (s, 6H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta =$ 161.7, 147.0, 146.8, 143.6, 141.6, 140.2, 133.9, 126.1, 125.4, 124.3, 124.1, 122.7, 53.8, 25.7. MALDI-TOF MS: m/z 487.2 [M+H]$^+$, 509.2 [M+Na]$^+$. Anal. Calcd. for C$_{34}$H$_{22}$N$_4$: C, 83.93; H, 4.56; N, 11.51. Found: C, 83.79; H, 4.48; N, 11.72.

2c: Yellow solid; yield 61%. Mp > 300 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta =$ 9.49 (d, J = 8.3 Hz, 2H), 8.21 (s, 2H), 7.65 (d, J = 8.3 Hz, 2H), 7.53-7.50 (m, 4H), 7.12-7.09 (m, 4H), 5.72 (s, 2H), 3.29-3.24 (m, 4H), 1.99-1.90 (m, 4H), 1.58-1.48 (m, 4H), 1.01 (t, J = 7.3 Hz, 6H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta =$ 165.6, 147.4, 146.5, 143.7, 141.4, 140.4, 133.6, 126.1, 125.5, 124.1, 123.1, 122.7, 53.7, 39.0, 31.9, 22.9, 14.1. MALDI-TOF MS: m/z 572.5 [M+2H]$^+$, 593.5 [M+Na]$^+$. Anal. Calcd. for C$_{40}$H$_{34}$N$_4$: C, 84.18; H, 6.00; N, 9.82. Found: C, 84.11; H, 6.08; N, 9.74.

2d: Pale yellow solid; yield 68%. Mp > 300 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta =$ 9.35 (d, J = 8.5 Hz, 2H), 8.02 (s, 2H), 7.75 (d, J = 8.5 Hz, 2H), 7.50-7.47 (m, 4H),
7.09-7.07 (m, 4H), 5.64 (s, 2H), 3.61 (s, 18H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ = 171.7, 147.0, 146.4, 143.7, 141.3, 140.7, 133.5, 126.1, 125.2, 124.1, 122.6, 120.1, 53.8, 38.8, 30.3. MALDI-TOF MS: $m/z$ 572.2 [M+2H]$^{+}$. Anal. Calcd. for C$_{40}$H$_{34}$N$_{4}$: C, 84.18; H, 6.00; N, 9.82. Found: C, 84.27; H, 6.11; N, 9.64.

**Synthesis and Characterizations for 1d**

To a solution of compound 5 (344 mg, 1 mmol) in MeOH (50 mL) was added 3d (1.38 g, 4.3 mmol). The solution was stirred under N$_2$ overnight to give a yellow solution. The solvent was removed by rotary evaporation and the red-orange residue was purified by silica gel column chromatography (eluant: 1:50 methanol/CH$_2$Cl$_2$) to give the product 1d in 32% yield as pale yellow solid. Mp > 300 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ = 9.45 (d, $J$ = 8.5 Hz, 6H), 8.42 (s, 6H), 7.83 (d, $J$ = 8.5 Hz, 6H), 6.24 (s, 2H), 1.61 (s, 54H). $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ = 172.2, 147.2, 143.5, 141.6, 141.4, 133.7, 125.0, 124.2, 120.3, 53.3, 38.8, 30.3. MALDI-TOF MS: $m/z$ 1203.7 [M+H]$^{+}$. Anal. Calcd. for C$_{78}$H$_{74}$N$_{12}$: C, 79.43; H, 6.32; N, 14.25. Found: C, 79.55; H, 6.45; N, 14.12.
2. Copies of $^1$H NMR and $^{13}$C NMR Spectra of New Compounds

Figure S1. $^1$H NMR spectrum (300 MHz, CDCl$_3$) of 3d.

Figure S2. $^{13}$C NMR spectrum (75 MHz, CDCl$_3$) of 3d.
**Figure S3.** $^1$H NMR spectrum (300 MHz, CDCl$_3$) of 2a.

**Figure S4.** $^{13}$C NMR spectrum (75 MHz, CDCl$_3$) of 2a.
Figure S5. $^1$H NMR spectrum (300 MHz, CDCl$_3$) of 2b.

Figure S6. $^{13}$C NMR spectrum (75 MHz, CDCl$_3$) of 2b.
Figure S7. $^1$H NMR spectrum (300 MHz, CDCl$_3$) of 2c.

Figure S8. $^{13}$C NMR spectrum (75 MHz, CDCl$_3$) of 2c.
Figure S9. $^1$H NMR spectrum (300 MHz, CDCl$_3$) of 2d.

Figure S10. $^{13}$C NMR spectrum (75 MHz, CDCl$_3$) of 2d.
Figure S11. $^1$H NMR spectrum (300 MHz, CDCl$_3$) of 1c.

Figure S12. $^{13}$C NMR spectrum (75 MHz, CDCl$_3$) of 1c.
Figure S13. $^1$H NMR spectrum (300 MHz, CDCl$_3$) of 1d.

Figure S14. $^{13}$C NMR spectrum (75 MHz, CDCl$_3$) of 1d.