Supplementary Information for
Synthesis, Solvent-dependent Emission and Two-photon Absorption of a Triangular -[D-π-A]_3- Macrocyle

Tian Li, Di Zhang, Ranran Wang, Yuanpeng Fan, Xinyan Guo, Shuai Liu, Yuguo Ma* and Dahui Zhao*

Beijing National Laboratory for Molecular Sciences, Center for the Soft Matter Science and Engineering and the Key Laboratory of Polymer Chemistry and Physics of the Ministry of Education, College of Chemistry, Peking University, Beijing 100871, China

Email: dhzhao@pku.edu.cn; ygma@pku.edu.cn

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I. Methods and Materials

All chemicals were used as received unless otherwise indicated. Oxygen or moisture sensitive reactions were performed under nitrogen atmosphere using the standard Schlenk technique. Reagent-grade tetrahydrofuran and toluene were distilled over sodium, while triethylamine and chloroform were distilled over CaH₂ prior to use. NMR spectra were recorded on a Mercury plus 300 (300 MHz), Bruker ARX 400 (400 MHz), Bruker ARX 500 (500 MHz) and Bruker AVANCE III 500 (500 MHz) instrument, using CDCl₃ or d-toluene as the solvent. Chemical shifts were reported in parts per million (ppm) relative to TMS (0 ppm) or toluene-d₈ (7.09 ppm) and CDCl₃ (77.0 ppm) or toluene-d₈ (137.0 ppm) for ¹H and ¹³C NMR spectra. MALDI-TOF was recorded on a Bruker Daltonics Inc. BIFLEX III mass spectrometer. Elemental analyses were performed using a German Vario EL III elemental analyzer. UV-Vis absorption spectra were recorded on a Hitachi U-4100 spectrophotometer, and steady-state linear-excitation fluorescence spectra were recorded on a Horiba Jobin Yvon FluoroMax-4P spectrofluorometer. Fluorescence quantum yields were determined with 9,10-dibenzanthracene in ethanol (φₑ = 0.95; λₑₓ = 372 nm) as the standard.

II. Synthetic Procedures and Characterization Data

General procedures of Sonogashira reaction A Schlenk tube containing aryl iodide, aryl acetylene, Pd(PPh₃)₄, and CuI was evacuated and back-filled with N₂ for three times. Degassed solvents of mixed Et₃N and THF were added via a syringe under N₂ atmosphere. The tube was then sealed and the reaction mixture was heated with stirring for 8–48 hours. After cooling to room temperature, the reaction mixture was concentrated under reduced pressure and diluted with dichloromethane, washed with saturated aq. NH₄Cl and brine sequentially, and then dried over anhydrous Na₂SO₄. The solvent was evaporated under reduced pressure and the residue was purified with silica gel column chromatography to afford the products.
Compound 1a was synthesized according to the procedures previously reported in the literature, with some modifications, and toluene was used as the solvent.

**1b**. A tube containing 1a (15.0 g, 33 mmol), Pd(PPh$_3$)$_2$Cl$_2$ (232 mg, 0.33 mmol), and CuI (64 mg, 0.33 mmol) was evacuated and back-filled with nitrogen for three times. Degassed 2-methylbut-3-yn-2-ol (10.5 mL, 99 mmol), Et$_3$N (70 mL) and THF (80 mL) were added to the tube under nitrogen atmosphere. The reaction mixture was stirred at 70 °C for 12 h and then worked up according to the general procedure. The crude product was purified with silica gel.
column chromatography eluted with dichloromethane/ethyl acetate (DCM/EA=5/1, v/v) to afford the product (12.6 g, 88%); TLC (DCM/EA, 5/1) \( R_f = 0.50 \). \(^1\)H NMR (300 MHz, CDCl\(_3\)): \( \delta 7.94 \) (2H, d, \( J = 1.8 \) Hz), 7.69 (2H, d, \( J = 8.1 \) Hz), 7.46 (2H, dd, \( J_1 = 1.8 \) Hz, \( J_2 = 8.1 \) Hz), 4.19 (4H, t), 3.62 (4H, t), 1.65 (12H, s).

\(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \( \delta 132.8, 132.5, 131.9, 127.2, 126.3, 124.5, 94.8, 92.3, 81.6, 65.6, 61.4, 31.4 \).

1c & 1d. A mixture of 1b (6.0 g, 13 mmol) and KOH (730 mg, 13 mmol) in THF (60 ml) was heated at 70 °C with stirring under N\(_2\) atmosphere. TLC was performed every 5 min to monitor the reaction until the diethynyl product appeared. After cooling to r.t., the reaction mixture was washed with water, aq. NH\(_4\)Cl and brine, and then dried over anhydrous Na\(_2\)SO\(_4\). The solvent was evaporated under reduced pressure, and the residue was purified with silica gel column chromatography using PE/DCM (2/1, v/v) to elute 1c (1.2 g, 26%) and then DCM/EA (10/1, v/v) to elute 1d (2.2 g, 42%). \(^1\)H NMR (300 MHz, CDCl\(_3\)) of 1c: \( \delta 8.02 \) (2H, d, \( J = 1.5 \) Hz), 7.72 (2H, d, \( J = 8.1 \) Hz), 7.55 (2H, dd, \( J_1 = 1.5 \) Hz, \( J_2 = 8.1 \) Hz), 4.21 (4H, m), 3.64 (4H, m), 3.15 (2H, s).

1d: \( \delta 8.02 \) (1H, d, \( J = 1.5 \) Hz), 7.94 (1H, d, \( J = 1.5 \) Hz), 7.72 (1H, d, \( J = 8.4 \) Hz), 7.69 (1H, d, \( J = 7.8 \) Hz), 7.54 (1H, dd, \( J_1 = 1.5 \) Hz, \( J_2 = 7.8 \) Hz), 7.47 (1H, dd, \( J_1 = 1.5 \) Hz, \( J_2 = 8.4 \) Hz), 4.19 (4H, m), 3.63 (4H, m), 3.15 (1H, s), 2.08 (1H, s), 1.65 (6H, s). \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) of 1c: \( \delta 133.5, 132.5, 132.4, 127.7, 126.4, 123.9, 92.2, 83.0, 78.3, 61.3 \).

1d: \( \delta 133.4, 132.8, 132.5, 132.34, 132.26, 132.0, 127.6, 127.2, 126.35, 126.30, 124.5, 123.8, 95.0, 92.19, 92.17, 83.0, 81.5, 78.27, 78.24, 65.5, 61.3, 31.4 \).

1e. A mixture of 2,7-diodophenanthrene-9,10-dione (8.0 g, 17 mmol), \(^2\) tetrabutylammonium bromide (1.7 g, 5.2 mmol), sodium hydrosulfite (18.0 g, 102 mmol), H\(_2\)O (80 ml) and THF (80 ml)
was allowed to react at room temperature for 15 min. After a quickly extraction, solvent of the organic phase was evaporated under reduced pressure and the residue was added with 1-bromo-2-octyldodecane (9.2 g, 37 mmol), K₂CO₃ (12.0 g, 85 mmol) and DMF (100 ml). After heating at 100 °C for 6 h under N₂ atmosphere the reaction mixture was dissolved in PE and washed with 2M HCl and brine sequentially, and then dried over anhydrous Na₂SO₄. The solvent was evaporated under reduced pressure and the residue was purified with silica gel column chromatography using PE as the eluent to afford 1e (13.3 g, 75%).

1H NMR (300 MHz, CDCl₃) of 1e: δ 8.59 (2H, d, J = 1.5 Hz), 8.26 (2H, d, J = 8.7 Hz), 7.84 (2H, d, J₁ = 1.5 Hz, J₂ = 8.7 Hz), 4.04 (4H, d), 2.10 (2H, m), 1.60~1.26 (64H, m), 0.88(12H, m)

13C NMR (75 MHz, CDCl₃): δ 142.8, 134.5, 131.5, 131.4, 127.2, 124.0, 108.4, 93.2, 48.8, 40.0, 39.3, 32.02, 31.99, 31.6, 30.3, 29.9, 29.84, 29.79, 29.73, 29.43, 29.40, 27.0, 22.7, 14.2.

1. A mixture of 1c (447 mg, 1.3 mmol), 1e (8.0 g, 7.8 mmol), Pd(PPh₃)₄ (35 mg, 0.03 mmol), CuI (6 mg, 0.03 mmol), Et₃N (10 ml), and THF (10 ml) was applied to the general Sonogashira reaction procedure and reacted at 45 °C for 10 h. The crude product was purified with silica gel column chromatography and eluted with PE/CH₂Cl₂ (4/1, v/v) to afford the product 1f (1.4 g, 52%). TLC (PE/CH₂Cl₂, 3/1) Rₚ = 0.40. 1H NMR (300 MHz, CDCl₃): δ 8.61 (2H, d, J = 2.1 Hz), 8.54 (2H, d, J = 8.7 Hz), 8.44 (2H, d, J = 1.8 Hz), 8.30 (2H, d, J = 8.7 Hz), 8.18 (2H, d, J = 0.9 Hz), 7.85 (2H, dd, J₁ = 2.1Hz, J₂ = 8.7 Hz), 7.79 (2H, d, J = 8.1 Hz), 7.76 (2H, dd, J₁ = 1.8 Hz, J₂ = 8.7 Hz), 7.65 (2H, dd, J₁ = 0.9Hz, J₂ = 8.1 Hz), 4.24 (4H, m), 4.08 (8H, m), 3.69 (4H, m), 3.69 (4H, m), 1.92 (4H, m), 1.20~1.62 (128H, m), 0.87 (12H, t), 0.81 (12H, t). 13C NMR (100 MHz, CDCl₃): δ 143.5, 142.6, 134.3, 133.1, 132.7, 132.0, 131.6, 131.4, 129.6, 128.7, 127.8, 127.20, 127.16, 126.5, 126.0, 125.1, 124.4, 122.6, 121.6, 93.2, 92.4, 90.9, 89.7, 61.4, 39.2, 31.95, 31.91, 31.45, 31.44, 30.2, 29.77, 29.71, 29.42, 29.40, 29.39, 29.36, 27.00, 26.97, 22.71, 22.67, 14.13, 14.10.
A mixture of 1d (2.5 g, 6.2 mmol), 1e (3.2 g, 3.1 mmol), Pd(PPh₃)₄ (116 mg, 0.1 mmol), CuI (19 mg, 0.1 mmol), Et₃N (10 ml), and THF (10 ml) was applied to the general procedure and reacted at 45 °C for 14 h. The crude product was purified with silica gel column chromatography and eluted with CH₂Cl₂/EA (3/1, v/v) to afford the product 1g (3.8 g, 83%).

**1H NMR (300 MHz, CDCl₃):** δ 8.59 (2H, d, J = 8.7 Hz), 8.46 (2H, d, J = 1.5 Hz), 8.13 (2H, d, J = 1.2 Hz), 8.02 (2H, d, J = 1.5 Hz), 7.78 (2H, d, J = 8.1 Hz), 7.77 (2H, dd, J₁ = 1.8 Hz, J₂ = 8.7 Hz), 7.72 (2H, d, J = 8.1 Hz), 7.64 (2H, d, J₁ = 1.2 Hz, J₂ = 8.1 Hz), 7.49 (2H, d, J₁ = 1.5 Hz, J₂ = 8.1 Hz), 4.22 (8H, m), 4.12 (4H, d), 3.68 (8H, m), 2.09 (2H, s), 1.97 (2H, m), 1.67 (12H, s), 1.22-1.60 (64H, m), 0.81 (12H, t).

**13C NMR (100 MHz, CDCl₃):** δ 143.4, 132.93, 132.90, 132.6, 132.5, 131.98, 131.93, 129.9, 128.6, 127.8, 127.2, 127.1, 126.4, 126.3, 125.9, 125.1, 124.5, 123.0, 121.5, 94.8, 92.31, 92.29, 91.0, 89.7, 81.6, 65.6, 61.4, 39.2, 31.9, 31.4, 30.2, 29.69, 29.68, 29.65, 29.64, 29.36, 29.31, 26.9, 22.6, 14.1.

A mixture of 1g (3.1 g, 2.0 mmol) and KOH (1.1 g, 20 mmol) in toluene (30 ml) was heated at 110 °C under N₂ atmosphere with stirring for 2 h. After cooling to r.t., the reaction mixture was washed with 2M HCl and brine sequentially, and then dried over anhydrous Na₂SO₄. The solvent was evaporated under reduce pressure and the residue was purified with silica gel column chromatography and eluted with PE/DCM (2/1, v/v) to afford 1h (2.5 g, 86%).

**TLC (PE/CH₂Cl₂, 2/1) Rₜ = 0.3.** ¹H NMR (300 MHz, CDCl₃): δ 8.59 (2H, d, J = 8.7 Hz), 8.46 (2H, d, J = 1.5 Hz), 8.13 (2H, s), 8.10 (2H, s), 7.77 (2H, dd, J₁ = 1.5 Hz, J₂ = 8.7 Hz), 7.76 (2H, d, J = 6.9 Hz), 7.73
(2H, d, J = 6.9 Hz), 7.64 (2H, d, J = 6.9 Hz), 7.56 (2H, d, J = 6.9 Hz), 4.22 (8H, m), 4.13 (4H, d), 3.67 (8H, m), 3.17 (2H, s), 1.98 (2H, m), 1.22-1.60 (64H, m), 0.81 (12H, t). $^{13}$C NMR (100 MHz, CDCl$_3$): δ 143.5, 133.7, 133.1, 132.7, 132.6, 132.5, 132.2, 130.0, 128.7, 128.0, 127.8, 127.2, 126.6, 126.1, 125.2, 124.0, 123.1, 121.6, 92.4, 92.3, 91.2, 89.8, 83.2, 78.3, 61.4, 39.3, 32.0, 31.5, 30.3, 29.81, 29.79, 29.76, 29.44, 29.43, 27.0, 22.7, 14.2.

**MC3.** A mixture of 1 (293 mg, 0.14 mmol), 2 (200 mg, 0.14 mmol), Pd(PPh$_3$)$_4$ (80 mg, 0.07 mmol), CuI (13 mg, 0.07 mmol), Et$_3$N (300 ml) and THF (300 ml) was allowed to react according to the general procedure at 45 °C for 48 hrs. The crude product was further purified by column chromatography on silica gel with PE/CH$_2$Cl$_2$ (1/1, v/v) as the eluent to afford the product (250 mg, 55%). TLC (PE/CH$_2$Cl$_2$, 1/1) $R_f = 0.50$. $^1$H NMR (400 MHz, CDCl$_3$): δ 8.63 (6H, d, J = 8.4 Hz), 8.48 (6H, s), 8.24 (6H, s), 7.83 (6H, d, J = 8.4 Hz), 7.80 (6H, d, J = 7.6 Hz), 7.67 (6H, d, J = 7.6 Hz), 4.26 (12H, m), 4.15 (12H, d), 3.72 (12H, m), 2.00 (6H, m), 1.98–1.21 (192H, m), 0.83 (36H, t). $^{13}$C NMR (100 MHz, CDCl$_3$): δ 143.5, 133.1, 132.7, 131.8, 130.0, 128.7, 127.9, 127.5, 126.5, 126.0, 125.2, 123.0, 121.6, 92.5, 91.2, 89.8, 81.5, 39.4, 32.0, 31.9, 31.6, 30.3, 30.0, 29.79, 29.78, 29.76, 29.73, 29.44, 29.39, 27.1, 22.7, 14.1. Calc. for C$_{228}$H$_{306}$O$_{18}$: C, 82.12; H, 9.25. Found: C, 82.05; H, 9.21.
MC2. In a round-bottom flask, MC3 (100 mg, 0.03 mmol) was dissolved in a mixture of CHCl₃ (20 ml), H₂O (4 ml), CH₃CN (4 ml) and CF₃CO₂H (10 ml). The mixture was heated at reflux for 2 days. After cooling to rt, it was washed with water and aq. NaHCO₃. Then the organic layer was separated and dried over anhydrous Na₂SO₄. The solvent was evaporated and the residue was purified with silica gel column chromatography and eluted with CHCl₃/EtOH (50/1, v/v) to afford the product (87 mg, 95%). TLC (CHCl₃/EtOH, 50/1) Rₗ = 0.20. ¹H NMR (300 MHz, CDCl₃): δ 8.25 (6H, s), 8.03 (6H, d, J = 8.4 Hz), 7.99 (6H, s), 7.98 (6H, d, J = 8.1 Hz), 7.52 (6H, d, J = 8.4 Hz), 7.46 (6H, d, J = 8.1 Hz), 4.08 (12H, d), 1.98 (6H, m), 1.69–1.23 (192H, m), 0.83 (36H, t). ¹H NMR (500 MHz, toluene-d₈): δ 8.83 (6H, s), 8.39 (6H, d, J = 8.5 Hz), 8.09 (6H, s), 8.03 (6H, d, J = 8.0 Hz), 7.79 (6H, d, J = 8.5 Hz), 7.47 (6H, d, J = 8.0 Hz), 4.33 (12H, d), 2.18 (6H, m), 1.87–1.28 (192H, m), 0.89 (36H, m). ¹³C NMR (125 MHz, toluene-d₈): δ 178.0, 143.7, 134.9, 131.9, 131.1, 130.6, 130.2, 130.0, 128.7, 126.8, 126.7, 123.1, 121.6, 94.9, 89.9, 77.5, 39.8, 32.0, 31.80, 31.74, 30.25, 30.24, 29.69, 29.63, 29.58, 29.3, 29.2, 27.3, 22.44, 22.41, 13.47, 13.46. MALDI-TOF MS: Calc. for C₂₁₆H₂₈₂O₁₂: 3070.2 (m/z). Found: 3093.2 (M+Na⁺).
MC1. A Schlenk tube containing the MC2 (50 mg, 0.02 mmol) and diaminomaleonitrile (16 mg, 0.15 mmol) was evacuated and back-filled with N₂ for three times. Then, a mixture of CF₃CO₂H (5 ml) and chloroform (5 ml) was added via a syringe under N₂ atmosphere. The tube was then sealed and the reaction mixture was heated at 70 °C with stirring for 3 hours. After cooling to room temperature, the reaction mixture was diluted with dichloromethane and washed with water and NaHCO₃ to pH=7, and then dried over anhydrous Na₂SO₄. The solvent was evaporated and the residue was purified with silica gel column chromatography and eluted with PE/toluene = (1/1, v/v) to afford product MC1 (43 mg, 80%). TLC (PE/ toluene, 1/1) Rf = 0.50. 

H NMR (300 MHz, CDCl₃): δ 8.77 (6H, s), 8.45 (6H, s), 8.34 (6H, s), 8.14 (6H, s), 7.76 (6H, s), 7.61 (6H, s), 4.08 (12H, d), 2.01 (6H, m), 1.64 ~ 1.21 (192H, m), 0.83 (36H, t). 

H NMR (500 MHz, toluene-d₈): δ 8.91 (6H, s), 8.87 (6H, d, J = 8.5 Hz), 8.72 (6H, s), 8.44 (6H, d, J = 8.5 Hz), 7.89 (12H, d, J = 8.5 Hz), 4.38 (12H, d), 2.12 (6H, m), 1.93 ~ 1.30 (192H, m), 0.90 (36H, m). 

C NMR (125 MHz, toluene-d₈): δ 143.8, 141.5, 132.1, 131.4, 130.6, 128.8, 127.8, 126.8, 126.7, 126.3, 123.2, 121.6, 113.3, 94.4, 90.1, 77.6, 39.8, 32.0, 31.9, 31.8, 30.3, 29.8, 29.6, 29.4, 29.2, 27.3, 22.5, 22.4, 13.51, 13.47. MALDI-TOF MS: Calc. for C₂₂₈H₂₃₀N₁₂O₆: 3286.2 (m/z). Found: 3286.2 (m/z). Calc. for C₂₂₈H₂₃₂N₁₂O₆; C, 83.32; H, 8.65, N, 5.11. Found: C, 83.26; H, 8.67, N, 5.03.

III. DFT Calculations of MC1

Density functional theory (DFT) calculations were carried out using Gaussian09 program. Molecular geometry was optimized using B3LYP hybrid functional 9 with 6-31G basis set, and visualized by Chem3D program. The polarized continuum model method (PCM) with toluene as
the solvent was used to calculate the electronic structures in solution. In these calculations, side chains are shortened to methoxyl groups.

-0.11921 -0.11753 -0.11753 -0.11238 -0.11238

Lumo  Lumo+1  Lumo+2  Lumo+3  Lumo+4

-0.21024 -0.21027 -0.21027 -0.22422 -0.22422

Homo  Homo-1  Homo-2  Homo-3  Homo-4

**Figure S1.** Selected MOs of ground-state **MC1** with energy levels (eV) calculated in the optimized geometry in toluene solution.

**IV. Concentration-dependent Absorptions and Self-association Constants**

By fitting the extinction coefficient changes as a function of concentration to an isodesmic (i.e., equal $K$) self-association model at the longer wavelength, the aggregation constants ($K_{agg}$) were obtained for **MC1**. The apparent extinction coefficients measured in the concentration-dependent absorption spectra were fitted by the nonlinear regression analyses to the following expression,$^{4,5}$

$$
\varepsilon = \frac{(2K_{agg}c + 1 - (4K_{agg}c + 1)^{0.5}) / 2 \times c^2 \times (\varepsilon_m - \varepsilon_a) + \varepsilon_a}{K_{agg}^2 / \varepsilon_m - \varepsilon_a}
$$

where $\varepsilon$ is the apparent extinction coefficient; $\varepsilon_m$ and $\varepsilon_a$ are the extinction coefficient of the monomer and aggregated species, respectively; $K_{agg}$ is the association constant, and $c$ is the total concentration of the compounds.
Figure S2. Concentration-dependence of absorption spectra of MC1 in (a) chloroform, (b) chlorobenzene and (c) toluene (insets showing the fitting of absorption data).
Figure S3. Concentration-dependent emission spectra of MC1 in chloroform (arrow indicates the direction of intensity change at increased concentration).

V. Nonlinear Optical Characterizations

Open-aperture Z-scan measurements were conducted with a femtosecond laser system composed of a Spectra-Physics TOPAS optical parametric amplifier (OPA) pumped by a Spfire ACE-F-1KXP Ti:sapphire amplifier (~120 fs, 1KHz) as the light source. With the Z-scan technique, the 2PA cross section experiments were carried out at a number of wavelengths in the range of 670-800 nm. To obtain these wavelengths, the second harmonic of the signal wavelength was used. The spatial profile of the laser beam was a nearly Gaussian distribution. The beam was focused by a plano-convex lens with a focal length of 200 mm. by fitting the \( T(z) \) curve using the 2PA theoretical equation:

\[
T(z) = \frac{1}{q_0 \sqrt{\pi}} \int_{-\infty}^{+\infty} t \ln(1 + q_0 e^{-\delta^2}) \, d\delta, \quad q_0 = \frac{I_0 \beta L w_0^2}{w^2(z)} \quad (1)
\]

where \( T(z) \) is the sample transmittance, \( I_0 \) was the laser peak power density at \( z = 0 \), \( L \) was the optical distance, \( w_z \) and \( w_0 \) were the laser beam areas at \( z \) and \( z = 0 \), being \( w^2(z)/w_0^2 = 1 + z^2/z_0^2 \), where \( z_0 \) is the Rayleigh distance of the beam. The two-photon absorption coefficient \( \beta \) was obtained. The absolute 2PA cross sections were calculated according to Eq. (2):

\[
\sigma_{2PA} = \frac{\beta}{N_0} (h\nu) \quad (2)
\]

Where \( h\nu \) is one photon energy; \( N_0 \) is the concentration of absorbing species/cm\(^3\).

Two-photon excited fluorescence (2PEF) spectra were recorded on SD2000 spectrometer (Ocean Optical), excited by a mode-locked Ti:sapphire femtosecond laser (Mai Tai HP, Spectra
Physics) with pulse width and repetition rate of 100 fs and 82 MHz. The fluorescence emission curves were detected at $2.0 \times 10^{-4}$ M from 720 nm to 870 nm.

![Figure S4. Emission spectra of MC1 in toluene upon excitation at indicated wavelengths](image)

**Figure S4.** Emission spectra of MC1 in toluene upon excitation at indicated wavelengths

**References**


Gonzalez, C.; Pople, J. A. *Gaussian 03*, Revision C.02; Gaussian Inc.: Wallingford CT, 2004.


Copies of $^1$H and $^{13}$C NMR Spectra
Copies of $^1$H and $^{13}$C NMR Spectra in toluene-$d_8$ at 80 °C

at 50 °C