Supporting Information

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General:

$^1$H NMR (400 MHz) and $^{13}$C NMR (100 MHz) were registered on Varian 400 M spectrometers with CDCl$_3$ as solvent and tetramethylsilane (TMS) as internal standard. Chemical shifts were reported in units (ppm) by assigning TMS resonance in the $^1$H spectrum as 0.00 ppm and CDCl$_3$ resonance in the $^{13}$C spectrum as 77.0 ppm. All coupling constants (J values) were reported in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, dd = doublet of doublet, m = multiplet. IR spectra were recorded on a Perkin-Elmer 16 PC FTIR spectrophotometer (KBr disc). High-resolution mass spectra were obtained with a Bruker BioTOF-Q mass spectrometer. Column chromatography was performed on silica gel 200-300 mesh.

All reactions involving Grubbs’ second generation catalyst were carried out under an atmosphere of ethylene (1 atm) using standard Schlenk techniques, and the solution of the catalytic reaction was degassed through freeze-pump-thaw cycle. Toluene was freshly distilled over sodium with the use of diphenyl ketone as an indicator under nitrogen. Dichloromethane was dried by anhydrous calcium chloride and distilled over phosphorus pentoxide. Grubbs’ second generation catalyst was synthesized according to literature procedure$^{1,2}$ and CuI was purchased from Aladdin Reagent Co., Ltd. Tetraisopropyl titanate was provided by Shanghai Darui Fine Chemicals Co., Ltd. Unsymmetrical diarylacetylenes were prepared by the palladium catalyzed coupling of the corresponding aryl iodides and arylacetylenes. Symmetrical diarylacetylenes were synthesized according to the literature procedures$^3$. All other chemicals were purchased commercially and used as received unless indicated otherwise.
Experimental procedures and characterization of products

Typical procedure for the synthesis of 4a

A solution of diarylacetylene 1a (72.3 mg, 0.125 mmol), Grubbs’ second generation catalyst (10 mg, 0.0125 mmol, 10 mol %) and CuI (1.2 mg, 0.00625 mmol, 5 mol %) in dry toluene (5 mL) was stirred at 80 °C for 24 h under 1 atm ethylene pressure (using balloon). After cooling to room temperature, DMAD (89 mg, 0.625 mmol) was added under air conditions and the resulting mixture was stirred at 100 °C for another 24 h in sealed vessel. After cooling to room temperature, the reaction mixture was filtered through a short pad of silica gel eluting with CH₂Cl₂. The volatiles were removed in vacuo. The residue was dissolved in 10 mL dichloromethane and a solution of FeCl₃ (4 equiv., 81 mg) in 1 mL nitromethane is added dropwise at 0 °C under nitrogen atmosphere. After the starting material had disappeared by TLC monitoring, the reaction was quenched by the addition of a few drops of methanol and extracted with dichloromethane (3×15 mL). The organic layer was washed with brine, dried over MgSO₄, and concentrated. The crude product was purified by silica gel column chromatography (elution with 7:1 petroleum ether-ethyl acetate) to give the final product in 71 % yield.

The procedure for the synthesis of 5

4,4'-diiodo-1,1'-biphenyl (406 mg, 1.0mmol), Pd(PPh₃)₂Cl₂ (28 mg, 0.04 mmol, 4 mol %), CuI (15 mg, 0.08 mmol, 8 mol%), PPh₃ (21 mg, 0.08 mmol, 8 mol %), and t-Pr₂NH (15 mL) were placed in a round-bottomed flask under nitrogen atmosphere. The mixture was stirred at 25 °C for 30 min, and 4-ethynyl-1,2-bis(hexyloxy)benzene (664 mg, 2.2 mmol) was then added. After this mixture had been stirred at 25 °C for additional 16 h, a saturated NH₄Cl solution was added. The mixture was extracted with CH₂Cl₂ (3×20 mL). The combined organic layers were dried with MgSO₄ and concentrated under vacuum. The residue was purified by silica gel column chromatography (elution with 2:1 petroleum-dichloromethane) to give the desire product in 76% yield. ¹H NMR (400 MHz, CDCl₃ ) δ 7.59 (s, 4H), 7.12 (dd, J₁=8.4
Hz, \( J_2 = 2.0 \) Hz, 1H), 7.06 (d, \( J = 2.0 \) Hz, 1H), 6.84 (d, \( J = 8.4 \) Hz, 1H), 4.04-4.01 (m, 4H), 1.86-1.82 (m, 4H), 1.51-1.46 (m, 4H), 1.36-1.33 (m, 8H), 0.91 (t, \( J = 6.0 \) Hz, 6H); \(^{13}\text{C}\) NMR (100 MHz, CDCl\(_3\)) \( \delta \) 149.7, 148.7, 139.7, 131.9, 126.8, 124.9, 122.8, 116.5, 115.2, 113.2, 90.7, 87.6, 69.2, 69.1, 31.6, 29.2, 29.1, 25.7, 25.7, 22.6, 14.0.

The procedure for the synthesis of 6

A solution of diarylacetylene 5 (94.3 mg, 0.125 mmol), Grubbs’ second generation catalyst (21 mg, 0.025 mmol, 20 mol \%) and CuI (2.4 mg, 0.0125 mmol, 10 mol \%) in dry toluene (5 mL) was stirred at 80 °C for 24 h under 1 atm ethylene pressure (using balloon). After cooling to room temperature, DMAD (178 mg, 1.25 mmol) was added under air conditions and the resulting mixture was stirred at 100 °C for another 24 h in sealed vessel. After cooling to room temperature, the reaction mixture was filtered through a short pad of silica gel eluting with CH\(_2\)Cl\(_2\). The volatiles were removed in vacuo. The residue is dissolved in 20 mL dichloromethane and a solution of FeCl\(_3\) (8 equiv., 162 mg) in 2 mL nitromethane is added dropwise at 0 °C under nitrogen atmosphere. After the starting material had disappeared by TLC monitoring, the reaction was quenched by the addition of a few drops of methanol and extracted with dichloromethane (3×15 mL). The organic layer was washed with brine, dried over MgSO\(_4\), and concentrated. The crude product was purified by silica gel column chromatography (elution with 3:1 petroleum-acetone) to give the final product in 38% yield.
Dimethyl 6,7,10,11-tetrakis(hexyloxy)triphenylene-2,3-dicarboxylate 4a

4a: 71% Yield. A white solid. $^1$H NMR (400 MHz, CDCl$_3$ ) δ 8.82 (s, 1H), 7.99 (s, 1H), 7.82 (s, 1H), 4.23-4.27 (m, 4H), 4.01 (s, 3H), 1.92-1.99 (m, 4H), 1.55-1.63 (m, 4H), 1.40-1.42 (m, 8H), 0.92-0.96 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$ ) δ 168.7, 150.5, 149.3, 130.2, 128.1, 125.1, 124.5, 122.4, 106.9, 106.3, 69.4, 69.4, 52.8, 31.6, 29.3, 29.3, 25.8, 25.8, 22.6, 14.1; IR:(KBr) $\nu_{max}$ 2951, 1723, 1615, 1515, 1465, 1387, 1267, 727 cm$^{-1}$; HRMS (ESI): Calcd for C$_{48}$H$_{64}$O$_8$ (M+H$^+$) 745.4674, Found: 745.4651; (M+Na$^+$) 767.4493, Found: 767.4480; Elemental anal. calcd. for C$_{46}$H$_{64}$O$_8$: C, 74.36; H, 8.62; O, 17.18; Found: C, 74.25; H, 8.56.

Dimethyl 6,11-bis(hexyloxy)-7,10-dimethoxytriphenylene-2,3-dicarboxylate 4b

4b: 62% Yield. A pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$ ) δ 8.83 (s, 1H), 7.98 (s, 1H), 7.81 (s, 1H), 4.28 (t, $J$=6.8 Hz, 2H), 4.13 (s, 3H), 4.02 (s, 3H), 1.96-2.03 (m, 2H), 1.57-1.59 (m, 2H), 1.37-1.45 (m, 4H), 0.94 (t, $J$=7.2 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$ ) δ 168.7, 150.7, 148.8, 130.2, 128.2, 124.9, 124.4, 122.3, 106.1, 104.3, 69.2, 56.2, 52.8, 31.7, 29.2, 25.7, 22.6, 14.1; IR:(KBr) $\nu_{max}$ 2998, 1733, 1618, 1518, 1435, 1288, 1264, 727 cm$^{-1}$; HRMS (ESI): Calcd for C$_{36}$H$_{44}$O$_8$ (M+H$^+$) 605.3109, Found: 605.3133; (M+Na$^+$) 627.2928, Found: 627.2944.
Dimethyl 6,7-bis(hexyloxy)-10-methoxytriphenylene-2,3-dicarboxylate 4c

4c: 57% Yield. A pale yellow solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.91 (s, 1H), 8.75 (s, 1H), 8.58 (d, \(J=9.2\) Hz, 1H), 7.954 (s, 1H), 7.89 (s, 1H), 7.86 (d, \(J=2.4\) Hz, 1H), 7.25-7.28 (m, 1H), 4.23-4.27 (m, 4H), 4.04 (s, 3H), 4.01 (s, 3H), 4.01 (s, 3H), 1.93-2.00 (m, 4H), 1.55-1.60 (m, 4H), 1.40-1.43 (m, 8H), 0.92-0.95 (m, 6H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 168.9, 168.3, 159.5, 150.3, 149.9, 131.9, 130.4, 130.0, 128.6, 129.0, 125.6, 124.7, 124.4, 124.1, 123.3, 121.8, 114.5, 106.6, 106.3, 105.8, 69.3, 69.2, 55.5, 52.8, 52.7, 31.6, 29.3, 29.2, 25.8, 22.6, 14.0; IR:(KBr) \(\nu_{\text{max}}\) 2950, 1732, 1613, 1516, 1516, 1427, 1376, 1288, 724 cm\(^{-1}\); HRMS (ESI): Calcd for C\(_{35}\)H\(_{42}\)O\(_7\) (M+H\(^+\)) 575.3003, Found: 575.3021; (M+Na\(^+\)) 597.2823, Found: 597.2852.

Dimethyl 6,7,10-trimethoxytriphenylene-2,3-dicarboxylate 4d

4d: 65% Yield. A pale yellow solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.79 (s, 1H), 8.61 (s, 1H), 8.46 (d, \(J=8.8\) Hz, 1H), 7.77-7.74 (m, 3H), 7.22 (dd, \(J_1=8.8\) Hz, \(J_2=2.4\) Hz, 1H), 4.11 (s, 3H), 4.10 (s, 3H), 4.03 (s, 3H), 4.02 (s, 3H), 4.01 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 168.8, 168.2, 159.6, 150.2, 149.7, 131.7, 130.5, 129.8, 128.7, 128.1, 125.6, 124.7, 124.4, 123.9, 123.2, 121.9, 114.6, 105.8, 104.6, 104.3, 56.1, 56.0, 55.5, 52.7, 52.7; IR:(KBr) \(\nu_{\text{max}}\) 3422, 2999, 1724, 1615, 1514, 1471, 1377, 1262, 674 cm\(^{-1}\); HRMS (ESI): Calcd for C\(_{25}\)H\(_{22}\)O\(_7\) (M+H\(^+\)) 435.1438, Found:435.1430; (M+Na\(^+\))
457.1258, Found: 457.1248.

Dimethyl 6,7-bis(hexyloxy)-10-phenyltriphenylene-2,3-dicarboxylate 4e

4e: 50% Yield. A pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.98 (s, 1H), 8.70 (s, 1H), 8.64 (d, $J$=8.4 Hz, 1H), 8.57 (s, 1H), 7.98 (s, 1H), 7.90 (s, 1H), 7.83 (dd, $J_1$=8.4 Hz, $J_2$=1.6 Hz, 1H), 7.79 (d, $J$=7.6 Hz, 2H), 7.56 (t, $J$=7.6 Hz, 2H), 7.46 (t, $J$=7.2 Hz, 1H), 4.22-4.26 (m, 4H), 4.02 (s, 3H), 4.01 (s, 3H), 1.93-2.00 (m, 4H), 1.58-1.62 (m, 4H), 1.40-1.42 (m, 8H), 0.92-0.97 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 168.9, 168.0, 150.6, 149.9, 141.0, 140.9, 131.1, 130.5, 130.1, 129.8, 128.9, 127.7, 127.5, 126.9, 125.8, 125.2, 125.1, 124.4, 124.0, 121.2, 106.6, 106.2, 69.3, 69.3, 52.8, 52.7, 30.6, 29.3, 29.2, 25.8, 22.6, 14.0; IR (KBr) $\nu_{\text{max}}$ 2931, 1725, 1613, 1518, 1465, 1372, 1265, 758 cm$^{-1}$; HRMS (ESI): Calcd for C$_{40}$H$_{44}$O$_6$ (M+H$^+$) 621.3211, Found: 621.3220; (M+Na$^+$) 643.3030, Found: 643.3055.

Dimethyl 6,7-bis(hexyloxy)-11-methoxytriphenylene-2,3-dicarboxylate 4f

4f: 57% Yield. A pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.89 (s, 1H), 8.72 (s, 1H), 8.36 (d, $J$=9.2 Hz, 1H), 7.97 (d, $J$=2.4 Hz, 1H), 7.90 (s, 1H), 7.85 (s, 1H), 7.28 (dd, $J_1$=9.2 Hz, $J_2$=2.8 Hz, 1H), 4.23 (t, $J$=6.4 Hz, 4H), 4.03 (s, 3H), 4.02 (s, 3H), 4.01 (s, 3H), 1.93-2.00 (m, 4H), 1.55-1.60 (m, 4H), 1.40-1.44 (m, 8H), 0.93-0.96 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 168.8, 168.2, 158.4, 150.8, 149.1, 131.7, 129.9, 128.9, 127.5, 126.9, 125.8, 125.2, 125.1, 124.4, 124.0, 121.2, 106.6, 106.2, 69.3, 69.3, 52.8, 52.7, 30.6, 29.3, 29.2, 25.8, 22.6, 14.0; IR (KBr) $\nu_{\text{max}}$ 2931, 1725, 1613, 1518, 1465, 1372, 1265, 758 cm$^{-1}$; HRMS (ESI): Calcd for C$_{40}$H$_{44}$O$_6$ (M+H$^+$) 621.3211, Found: 621.3220; (M+Na$^+$) 643.3030, Found: 643.3055.
129.8, 129.3, 127.7, 125.6, 125.1, 124.5, 124.4, 121.7, 117.3, 107.0, 105.8, 69.5, 69.1, 55.6, 52.8, 52.7, 31.6, 31.6, 29.3, 29.2, 25.8, 22.6, 14.0; IR:(KBr) $\nu_{\text{max}}$ 2951, 1731, 1617, 1511, 1467, 1382, 1266, 723 cm$^{-1}$; HRMS (ESI): Calcd for C$_{35}$H$_{42}$O$_7$ (M+Na$^+$) 597.2823, Found: 597.2825.

Dimethyl 6,7-bis(hexyloxy)triphenylene-2,3-dicarboxylate $4g$

$4g$: 55% Yield. A pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.06 (s, 1H), 8.77 (s, 1H), 8.67 (d, $J=8.0$ Hz, 1H), 8.5 (d, $J=8.0$ Hz, 1H), 7.98 (d, $J=11.6$ Hz, 2H), 7.63-7.73 (m, 2H), 4.26 (t, $J=6.8$ Hz, 4H), 4.02 (s, 3H), 4.02 (s, 3H), 1.95-1.99 (m, 4H), 1.55-1.60 (m, 4H), 1.40-1.42 (m, 8H), 0.93-0.96 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 168.9, 168.0, 168.0, 166.8, 150.7, 149.9, 131.3, 130.3, 130.0, 128.2, 127.9, 127.8, 126.6, 125.3, 125.2, 124.0, 123.9, 122.9, 122.9, 69.4, 69.2, 52.8, 52.7, 31.6, 29.3, 29.2, 25.8, 22.6, 14.0; IR:(KBr) $\nu_{\text{max}}$ 2932, 1736, 1611, 1514, 1452, 1373, 1268, 755 cm$^{-1}$; HRMS (ESI): Calcd for C$_{34}$H$_{40}$O$_6$ (M+H$^+$) 545.2898, Found: 545.2922; (M+Na$^+$) 567.2717, Found: 567.2726.

Dimethyl 6,11-bis(hexyloxy)triphenylene-2,3-dicarboxylate $4h$

$4h$: 60% Yield. A pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.87 (s, 1H), 8.43 (d, $J=9.2$ Hz, 1H), 7.97 (d, $J=2.4$ Hz, 1H), 7.29 (dd, $J_1=9.2$ Hz, $J_2=2.4$ Hz, 1H), 4.17 (t, $J=6.8$ Hz, 2H), 4.02 (s, 3H), 1.86-1.93 (m, 2H), 1.54-1.61 (m, 2H), 1.39-1.42 (m,
4H), 0.94 (t, J=6.8 Hz, 3H); 13C NMR (100 MHz, CDCl₃) δ 168.4, 157.9, 131.5, 129.3, 128.8, 124.9, 124.6, 124.4, 117.7, 106.8, 68.3, 52.8, 31.6, 29.3, 25.8, 22.6, 14.1; IR(KBr) νmax 2945, 1736, 1614, 1495, 1437, 1388, 1235, 731 cm⁻¹; HRMS (ESI): Calcd for C₄₄H₄₀O₆ (M+H⁺) 545.2898, Found: 545.2921; (M+Na⁺) 567.2717, Found: 567.2728.

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\begin{align*}
\text{Dimethyl 10,11-bis(hexyloxy)-6,7,8-trimethoxytriphenylene-2,3-dicarboxylate 4i} \\
\text{4i: 41% Yield. A pale yellow solid.} \\
\text{1H NMR (400 MHz, CDCl₃) δ} \\
9.17 (s, 1H), 8.85 (s, 1H), 8.78 (s, 1H), 7.98 (s, 1H), 7.89 (s, 1H), 4.28-4.22 (m, 4H), 4.13 (s, 3H), 4.08 (s, 3H), 4.02 (s, 3H), 4.01 (s, 3H), 3.95 (s, 3H), 1.92-1.99 (m, 4H), 1.54-1.60 (m, 4H), 1.40-1.44 (m, 8H), 0.92-0.96 (m, 6H); 13C NMR (100 MHz, CDCl₃) δ 168.7, 168.5, 152.2, 152.0, 150.1, 148.5, 144.2, 131.4, 130.0, 129.3, 128.0, 125.6, 125.1, 125.0, 124.3, 122.8, 119.5, 110.4, 110.4, 106.8, 101.7, 69.4, 68.9, 61.4, 60.7, 56.1, 52.8, 52.8, 31.6, 31.6, 29.3, 29.2, 25.8, 25.8, 22.6, 14.0. IR(KBr) νmax 2955, 1725, 1611, 1438, 1371, 1287, 728 cm⁻¹; HRMS (ESI): Calcd for C₃₇H₄₆O₉ (M+H⁺) 635.3215, Found: 635.3232; (M+Na⁺) 657.3034, Found: 657.3057.
\end{align*}
\]

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\begin{align*}
\text{Trimethyl 10,11-bis(hexyloxy)triphenylene-2,3,7-tricarboxylate 4j} \\
\text{4j: 26% Yield. A pale yellow solid.} \\
\text{1H NMR (400 MHz, CDCl₃) δ} \\
9.21 (d, J=1.6 Hz, 1H), 9.10 (s, 1H), 8.77 (s, 1H), 8.73 (d, J=8.8 Hz, 1H), 8.25 (dd, J₁=8.4 Hz, J₂=1.2 Hz}
\end{align*}
\]
1H), 8.07 (s, 1H), 7.98 (s, 1H), 4.25-4.32 (m, 4H), 4.06 (s, 3H), 4.03 (s, 3H), 4.02 (s, 3H), 1.94-2.02 (m, 4H), 1.57-1.64 (m, 4H), 1.37-1.45 (m, 8H), 0.95 (t, J=6.8 Hz, 6H);

\[ ^{13} \text{C} \text{ NMR (100 MHz, CDCl}_3 \text{ )} \delta 168.8, 167.7, 167.1, 150.9, 150.2, 132.3, 131.2, 131.1, 130.1, 129.4, 129.3, 127.8, 126.5, 125.9, 125.0, 124.1, 124.0, 123.1, 106.5, 106.2, 69.4, 69.3, 52.9, 52.8, 52.5, 31.6, 29.2, 25.8, 22.6, 14.1; IR:(KBr) \nu_{\text{max}} 2944, 1736, 1614, 1495, 1388, 1256, 731 \text{ cm}^{-1}; \text{HRMS (ESI): Calcd for } \text{C}_{36}\text{H}_{42}\text{O}_8 (\text{M}+\text{H}^+) 625.2772, \text{Found: 625.2789.} \]

![Chemical structure of Dimethyl 10-bromo-6,7-bis(hexyloxy)triphenylene-2,3-dicarboxylate 4k](attachment:image.png)

**Dimethyl 10-bromo-6,7-bis(hexyloxy)triphenylene-2,3-dicarboxylate 4k**

4k: The reaction of 2k was dissolved in MoCl$_5$ (2.5 equiv., 85 mg) and 10 mL dichloromethane at 0 °C under nitrogen atmosphere. After the reaction was quenched by the addition of a few drops of methanol and extracted with dichloromethane (3×15 mL). The organic layer was washed with brine, dried over MgSO$_4$, and concentrated. The crude product was purified by silica gel column chromatography (elution with 7:1 petroleum ether-ethyl acetate) to give a pale yellow solid (27 % yield). $^1$H NMR (400 MHz, CDCl$_3$ ) δ 8.95 (s, 1H), 8.72 (s, 1H), 8.56 (d, J=1.6 Hz, 1H), 8.48 (d, J=8.8 Hz, 1H), 7.92 (s, 1H), 7.83 (s, 1H), 7.72 (dd, J$_1$=8.8 Hz, J$_2$=2.0 Hz, 1H), 4.24-4.28 (m, 4H), 4.02 (s, 3H), 4.02 (s, 3H), 1.93-2.01 (m, 4H), 1.58-1.62 (m, 4H), 1.38-1.45 (m, 8H), 0.95 (t, J=7.2 Hz, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$ ) δ 168.8, 167.8, 150.8, 150.3, 132.0, 131.2, 130.3, 129.6, 129.5, 128.0, 126.6, 125.7, 125.6, 125.1, 124.1, 124.0, 123.3, 122.9, 106.5, 105.9, 69.4, 69.3, 52.9, 52.8, 31.6, 29.2, 29.2, 25.8, 22.6, 14.1; IR:(KBr) \nu_{\text{max}} 2952, 1727, 1614, 1535, 1465, 1393, 1269, 734, 593 \text{ cm}^{-1}; \text{HRMS (ESI): Calcd for } \text{C}_{36}\text{H}_{39}\text{BrO}_6 (\text{M}+\text{H}^+) 623.0203, \text{Found: 623.2027; (M+Na}^+) 645.1822, \text{Found: 645.1837.}
dimethyl triphenylene-2,3-dicarboxylate 4l

4l: The reaction of 2l was dissolved in MoCl$_5$ (5.0 equiv., 170 mg) and 10 mL dichloromethane at 0 °C under nitrogen atmosphere. After the reaction was quenched by the addition of a few drops of methanol and extracted with dichloromethane (3×15 mL). The organic layer was washed with brine, dried over MgSO$_4$, and concentrated. The crude product was purified by silica gel column chromatography (elution with 7:1 petroleum ether-ethyl acetate) to give a pale yellow solid (18 % yield). $^1$H NMR (400 MHz, CDCl$_3$) δ 8.98 (s, 1H), 8.65 (d, $J$=7.2 Hz, 2H), 7.74-7.68 (m, 2H), 4.02 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 168.3, 131.4, 130.6, 129.7, 128.6, 128.5, 127.7, 124.9, 123.9, 123.4, 52.8; IR:(KBr) $\nu_{\text{max}}$ 2925, 1734, 1607, 1539, 1290, 748 cm$^{-1}$.

Dimethyl 12,13-bis(hexyloxy)benzo[g]chrysene-2,3-dicarboxylate 4n

4n: 51% Yield. A pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 9.06 (s, 1H), 8.96-8.98 (m, 1H), 8.85 (s, 1H), 8.60 (d, $J$= 9.2 Hz, 1H), 8.38 (s, 1H), 8.07 (s, 1H), 8.03-8.05 (m, 1H), 7.99 (d, $J$= 8.8 Hz, 1H), 7.63-7.65 (m, 2H), 4.31 (t, $J$= 6.8 Hz, 2H), 4.19 (t, $J$= 6.8 Hz, 2H), 4.04 (s, 3H), 4.03 (s, 3H), 1.92-2.01 (m, 4H), 1.56-1.63 (m, 4H), 1.37-1.44 (m, 8H), 0.93-0.95 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 168.9, 168.1, 149.5, 149.3, 133.9, 131.0, 130.1, 129.8, 129.7, 128.5, 128.4, 128.0, 127.8, 127.2 126.5, 126.2, 125.9, 125.8, 125.2, 124.5, 124.0, 120.8, 112.4, 106.5, 69.4, 69.2, 52.9, 52.8, 31.6, 29.2, 29.2, 25.8, 25.8, 22.6, 26.6, 14.1; IR:(KBr) $\nu_{\text{max}}$ 2956, 1728, 1612, 1508, 1444, 1380, 1201, 753 cm$^{-1}$; HRMS (ESI): Calcd for C$_{38}$H$_{42}$O$_6$ (M+H$^+$)
Dimethyl 2,3-bis(hexyloxy)benzo[g]chrysene-12,13-dicarboxylate 4o

4o: 26% Yield. A pale yellow solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.34 (s, 1H), 8.89 (s, 1H), 8.84 (d, $J$=8.4 Hz, 1H), 8.48 (d, $J$=8.8 Hz, 1H), 8.05 (s, 1H), 8.04-8.03 (m, 2H), 7.73-7.61 (m, 3H), 4.31-4.27 (m, 4H), 4.04 (s, 3H), 3.98 (s, 3H), 2.00-1.96 (m, 4H), 1.61-1.55 (m, 4H), 1.42-1.41 (m, 8H), 0.96-0.93 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 168.9, 167.9, 150.8, 150.1, 137.8, 133.0, 131.8, 130.9, 130.1, 129.7, 129.2, 129.0, 128.8, 128.3, 128.2, 127.9, 126.8, 126.6, 125.9, 125.4, 125.3, 125.2, 124.2, 123.5, 120.5, 106.5, 106.3, 69.4, 69.3, 52.8, 52.7, 31.6, 29.3, 29.2, 25.8, 22.6, 21.4, 14.0; IR (KBr) $\nu_{\text{max}}$ 2954, 1727, 1611, 1529, 1468, 1378, 1276, 753 cm$^{-1}$; HRMS (ESI): Calcd for C$_{38}$H$_{42}$O$_6$ (M+H$^+$) 595.3060, Found: 595.3044; (M+ Na$^+$) 617.2879, Found: 617.2875.

2,3,6,7-tetrakis(hexyloxy)benzo[f]tetraphene-10,13-dione 4p

4p: 43% Yield. A red solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.88 (s, 1H), 7.86 (s, 1H), 7.69 (s, 1H), 6.99 (s, 1H), 4.26-4.20 (m, 4H), 2.00-1.94 (m, 4H), 1.62-1.58 (m, 4H), 1.44-1.43 (m, 8H), 0.98-0.94 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 185.0, 150.9, 149.5, 139.4, 131.9, 127.9, 125.4, 122.9, 122.2, 106.9, 106.1, 69.4, 69.3, 31.7, 31.7,
29.4, 29.3, 25.9, 22.7, 14.1, 14.0; IR:(KBr) \( \nu_{\text{max}} \) 2955, 1666, 1611, 1515, 1445, 1341, 1269, 673 cm\(^{-1}\); HRMS (ESI): Calcd for C\(_{46}H_{60}O\_6\) (M+H\(^+\)) 709.4469, Found: 709.4462.

Tetramethyl10,10',11,11'-tetrakis(hexyloxy)-[2,2'-bitriphenylene]-6,6',7,7'-tetracarboxylate 6

6: 38% Yield. A pale yellow solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 9.06 (s, 1H), 8.80-8.75 (m, 3H), 8.09 (s, 1H), 8.04 (d, \( J=\)8.8 Hz, 1H), 7.98 (s, 1H), 4.27 (t, \( J=\)6.0 Hz, 4H), 4.04 (s, 3H), 4.03 (s, 3H), 1.97-1.94 (m, 4H), 1.42-1.37 (m, 10H), 0.97-0.88 (m, 8H); \(^1\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 168.9, 167.9, 150.7, 150.1, 140.6, 131.2, 130.6, 130.0, 129.9, 127.8, 127.2, 125.9, 125.1, 125.1, 124.5, 124.0, 123.3, 121.6, 106.7, 106.4, 69.4, 69.3, 52.8, 52.7, 31.7, 29.3, 29.3, 25.9, 25.8, 22.6, 22.6, 14.0, 14.0; IR:(KBr) \( \nu_{\text{max}} \) 2951, 1728, 1611, 1516, 1465, 1372, 1265, 726 cm\(^{-1}\); HRMS (ESI): Calcd for C\(_{68}H_{72}O_{12}\) (M+Na\(^+\)) 1110.5391, Found: 1110.5423.
References:
$^1$H and $^{13}$C NMR
Figure 1 POM image of compound 4a at 150 °C.