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2,3,7,8,12,13-Hexaaryltruxenes: An *ortho*-Substituted Multiarm Design and Microwave-Accelerated Synthesis toward Starburst Macromolecular Materials with Well-Defined π Delocalization

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Abstract: We present herein a novel design and the efficient synthesis towards a “homogeneous” starburst fluorene system based on the novel 2,3,7,8,12,13-hexaaryltruxene scaffold. Controlled microwave heating provides a facile and powerful approach for each step in the synthesis of these bulky materials with large steric hindrance, suggesting an avenue to access structurally well-defined complex organic semiconductors (OSCs) rapidly and conveniently with high yield and purity. The resulting materials exhibited good thermal stability and an excellent glassy structure as revealed by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) as well as wide-angle X-ray diffraction (WAXD) studies. Moreover, compared with their corresponding three-arm-substituted counterparts **T1**–**T4**, the introduction of the *ortho* substituents around the truxene core in **Tr1**–**Tr4** results in significant blueshifts (of 7–24 nm) of the absorption maxima λ_{max} and higher energy optical gaps (E_g). Comparative studies with corresponding linear, rod-shaped oligofluorene counterparts (OFX) have revealed that the longest *para*-conjugated segment in the **TrX** (X = 1–4) structures plays the dominant role in determining their electronic properties. UV/Vis data and cyclic voltammetry (CV) investigations have indicated that there is little electronic interaction between the arms, even for the shortest armed oligomer **Tr1**. A clear linear relationship of both $1/\lambda_{max}$ and E_g with the inverse of $(n+1)$ for these branched systems was found. Our findings highlight a novel molecular design comprising an *ortho*-substituted, multiarmed architecture that would allow the introduction of isotropic physical and/or mechanical properties, while at the same time maintaining most of the important electronic properties of the rod-shaped constituents of a fully conjugated system.

Keywords: luminescence · microwave chemistry · oligofluorenes · organic semiconductors · starburst macromolecules

Introduction

Organic semiconductors (OSCs) based on π-conjugated systems have been extensively explored and investigated as active materials in the emerging area of organic electronics.[1] The success in this area relies, to a large extent, on the novel design and efficient synthesis of new materials with particular structures and properties. Low-dimensional, linear oligo- and polyfluorenes are among the most prominent OSCs attracting intense research effort due to their interesting properties and potential applications in plastic electronic devices, such as organic light-emitting diodes (OLEDs), optical switches, and lasers.[2–4]

The desire to combine the well-defined electronic properties and high purity of monodisperse oligomers with the mechanical properties of polymers for the design and synthesis of unique OSCs with optimum functional properties motivates the effort to increase the molecular complexity and dimensionality. This poses a major challenge, but also presents an exciting opportunity. Various types of branched OSCs, mainly based on three- or four-substituted core scaffolds, have been realized.[5–10]Among them, star-shaped compounds with a central core and three radial conjugated arms, which bring about interesting physical and chemical properties, are receiving much attention.[6–10] Nevertheless, it

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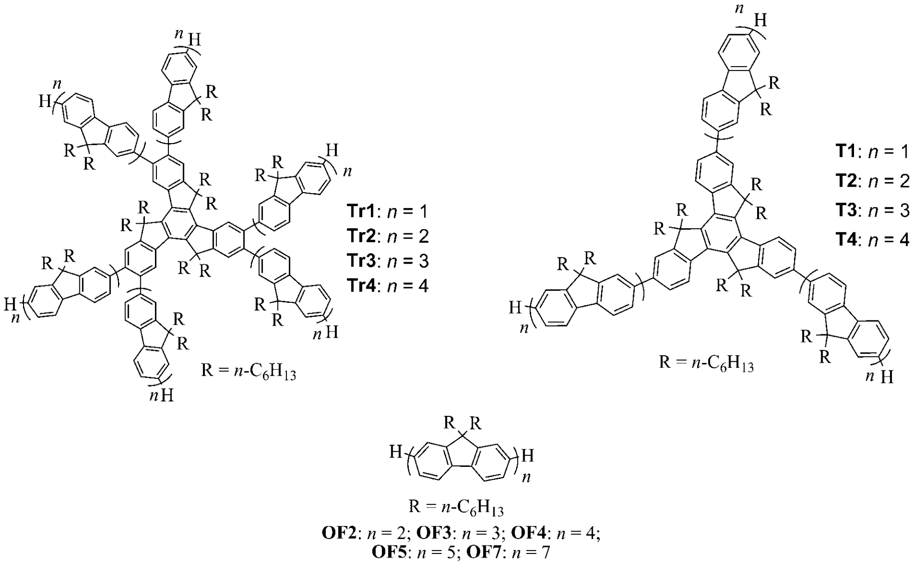
is also of great interest to explore more bulky and complex branched/dendritic materials to mimic the high-mass polymer structures for optimum functional properties.[11–13]

In our quest for efficient materials for luminescence applications, we have recently designed and prepared a new family of multisubstituted starburst macromolecular materials.[11,12] Such highly substituted molecular structures appear to be rather promising for electroluminescence and lasing applications.[11,12] As a further step, we wish to unravel and understand how the effects of increasing molecular complexity and dimensionality impact on associated optoelectronic properties. We thus set out to develop a set of analogues based on a novel 2,3,7,8,12,13-hexaaryltruxene scaffold, as depicted in Scheme 1. The overlapping three-fluorene-fragment structure of the truxene core and the six *ortho*-substituted oligofluorene branches constitute a "homogeneous" starburst fluorene system. This could provide an ideal platform for investigating the impact of such a multisubstituted molecular architecture on material properties.



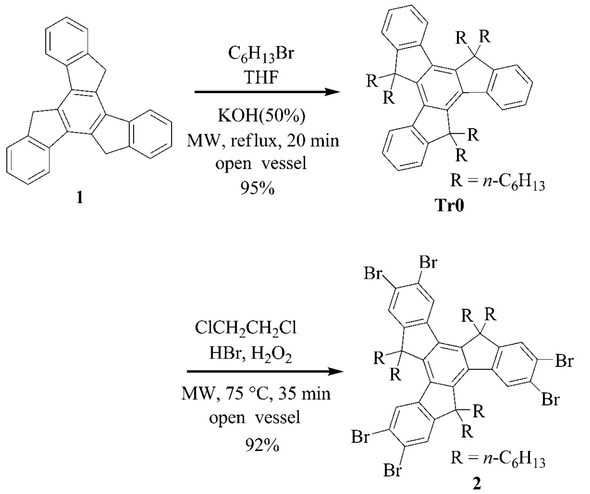
Scheme 1. General structures of **Tr1**–**Tr4** (**TrX**), **TX**,[8] and **OFX**.

Generally, the synthesis towards these well-defined, highly substituted, and branched structures suffers from intractable multiple couplings and arduous and time-consuming, repetitive synthetic steps, as well as low overall yields. Microwave (MW) irradiation is known for its dielectric heating pattern that allows energy to be directly applied onto the reactants, so-called "molecular heating", rather than conductively through the vessel.[14] Being energy efficient, this type of novel heating pattern not only provides the opportunity to complete reactions in minutes, but can also promote transformations that are currently not readily accessible using conventional heating methods. MW chemistry is therefore creating faster reaction times, improving yields, producing cleaner reaction sequences, and thus paving the way to rapid and efficient chemical syntheses. In contrast to its widespread application in reaction investigations,[15] the use of MW in material exploitation is still in its infancy.[10,11,16] In particular, research focused on the development of complex organic semiconducting materials, which adopts this flash heating technology throughout the synthetic procedures, attracts less attention.

Herein, we wish to present the novel molecular design and the efficient synthesis toward increasing molecular complexity and dimensionality in the accessing of structurally well-defined, highly pure luminescent macromolecular materials. This is achieved by constructing multiple *ortho*-substituted starburst architectures. A straightforward microwave-accelerated synthetic procedure was proposed to afford all of the intermediates and the final multisubstituted targets quite efficiently, and with high yields and chemical purities. The demonstrated high efficiency overcomes one of the key challenges in synthetic organic chemistry—the preparation and isolation of complex molecules with functionalities in specific spatial orientations. In contrast to their three-substituted, 2D counterparts (**TX**),[8] the special sixfold *ortho*-substituted arrangement surrounding the truxene core in the **TrX** structures makes the adjacent conjugated rigid branches twist out of the plane, adopting a nonplanar 3D spatial orientation because of steric hindrance. This imparts the materials with exceptional electronic properties.

Results and Discussion

The general chemical structures of **TrX**, **TX**,[8] and **OFX** investigated in this study are depicted in Scheme 1. Although truxene derivatives mainly based on three-substituted core scaffold have been widely studied,[7,8,17] 2,3,7,8,12,13-hexaaryltruxenes remain largely unexplored. We have recently explored the synthesis of a hexabromotruxene derivative **2**,[18] a key building block for the six-armed starburst systems. This was accomplished by the alkylation of truxene **1** followed by a bromination procedure. Traditionally, the alkylation of truxene involves lithiation with nBuLi, followed by quenching with alkyl bromide.[8,17a] To accelerate the process, we describe here an alternative route. The open-vessel irradiation in a focused monomode MW device (CEM Discover) of compound **1** with 1-bromohexene in THF at reflux doped with KOH afforded **Tr0** in 95 % yield in 20 min (Scheme 2). The subsequent bromination was carried out

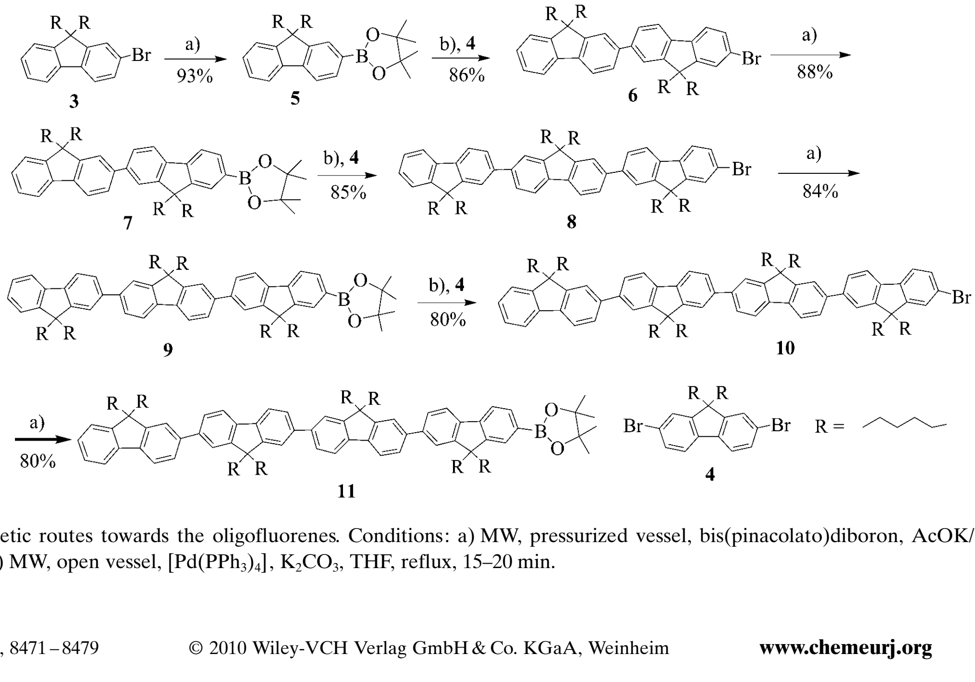


Scheme 2. The synthetic routes toward the hexabromotruxene core structure.

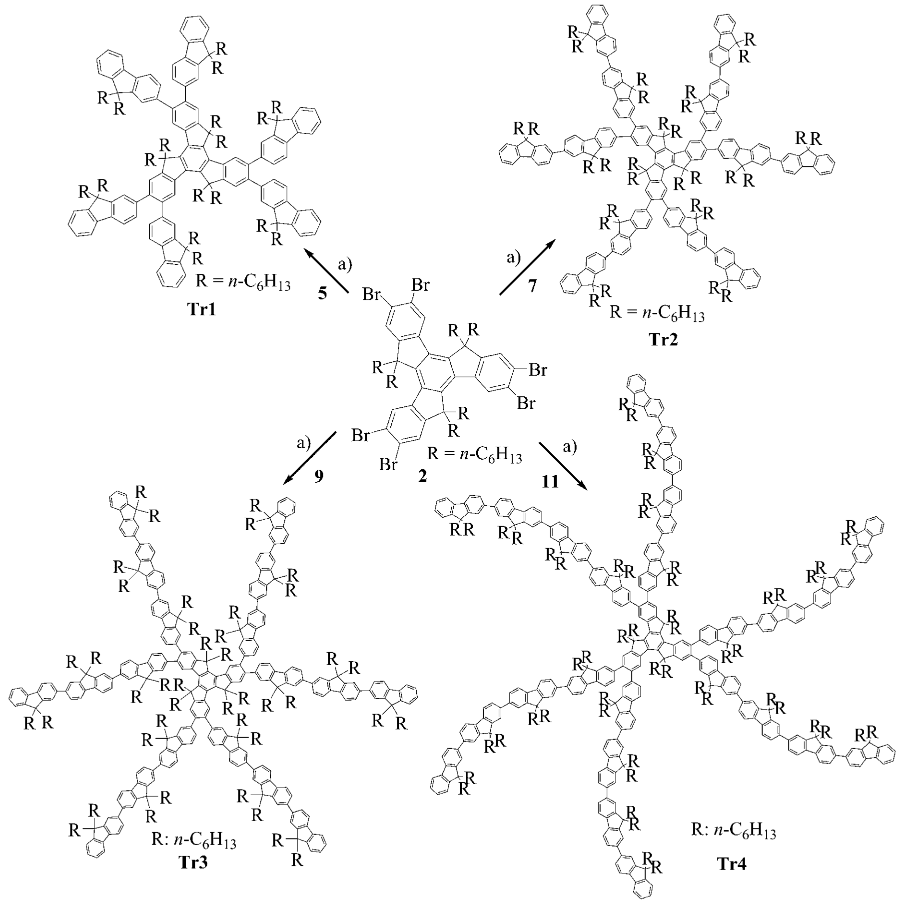
with hydrobromic acid (40 wt %) and aqueous hydrogen peroxide (30 wt %) under refluxing conditions. The efficient MW irradiation led to completion of the reaction within 35 min, in 92 % yield. Unlike traditional bromination reactions, the potentially hazardous elemental halogens and the corrosive side products are avoided during this process, providing a rapid and green pathway toward efficient bromination.

The synthetic routes toward the oligofluorene arms are shown in Scheme 3. In general, lengthy multistep sequences and intensive labor are required to obtain these compounds.[2] Here, we describe the rapid and facile assembly of these compounds using MW heating. With an optimized MW-assisted Miyaura reaction,[19] using [$PdCl_2$(dppf)] (dppf = 1,1′-bis(diphenylphosphino)ferrocene) as the catalyst and a temperature of 110 °C in pressurized vessels, the oligofluorene bromides **3**, **6**, **8**, and **10** were successfully transformed into the corresponding boronic esters **5**, **7**, **9**, and **11** in high yields (>80 %) within 10–25 min. Significantly, this transformation avoids utilizing strongly basic organometallic reagents, allowing a facile entry into a wide variety of functionalized aryl boronic esters. The oligofluorene bromides **6**, **8**, and **10** could also be obtained within minutes by the reaction of **5**, **7**, and **9**, respectively, with excessive equivalents of compound **4**, using MW-assisted Suzuki cross-couplings in open vessels under refluxing conditions. Fast column chromatography gave the desired compounds in high purities and quantities.

The Suzuki reaction[20] is recognized as one of the most versatile and, at the same time, one of the most widely used cross-coupling reactions in modern organic synthesis. The facile access to the simple biaryls[15] under MW conditions prompted us to explore the possibility of complex multiple couplings,[11b] which were generally limited by their poor efficiency under conventional conditions (more than five days are needed for a simple threefold transformation) and unfortunately often resulted in low yields and troublesome, partially substituted by-products.[8] Concomitant with the high temperatures and pressures accessible in sealed vessels under MW irradiation, we surprisingly found that the complex multiple Suzuki couplings proceeded smoothly with [$Pd(PPh_3)_4$] as a catalyst in a K_2CO_3/THF solvent system. We were therefore able to access macromolecules **Tr1**–**Tr4** using MW-enhanced sixfold Suzuki transformations efficiently and rapidly within 33 min, simply by optimizing the irradiation times (Scheme 4). The desired monodisperse star targets were easily isolated by flash column chromatography on silica gel in excellent yields (>82 %). Not only is the procedure simplified, but most importantly the efficiency of preparation, the product yields, and the purities were greatly enhanced by MW heating. This powerful and facile methodology could open the possibility to the development of a



Scheme 3. The synthetic routes towards the oligofluorenes. Conditions: a) MW, pressurized vessel, bis(pinacolato)diboron, AcOK/DMF, [$PdCl_2$(dppf)], 110 °C, 10–25 min; b) MW, open vessel, [$Pd(PPh_3)_4$], K_2CO_3, THF, reflux, 15–20 min.



Scheme 4. The synthesis towards the macromolecular stars **Tr1**–**Tr4**. Conditions: a) MW, pressurized vessel, $[Pd(PPh_3)_4]$, K_2CO_3, THF, 150 °C, 15–33 min, 82–89 %.

great variety of complex materials through similar multiple couplings using controlled MW heating.

The well-defined structures and chemical purities of all intermediates and the final star targets **Tr1**–**Tr4** have been adequately confirmed by ^{1}H and ^{13}C NMR spectroscopy, elemental analysis, and MALDI-TOF mass spectrometry. The results agree very well with the expected structures. The ^{1}H and ^{13}C NMR spectra of the key building block **2** and the final targets **Tr1**–**Tr4** are given in Figures S1–S10 of the Supporting Information. The eluting curves from the gel permeation chromatography (GPC) (Figure 1) of the resulting targets **Tr1**–**Tr4** exhibit symmetrical narrow peaks with polydispersities of 1.01–1.05, suggesting a high monodispersity and purity. No distinct precursors or partially substituted byproducts are observed. Considering the steric hindrance for the sixfold transformations and the rigid nature of the **TrX** structures, the overall yields and the purity obtained were quite notable.

The thermal stabilities of the resulting **TrX** star targets were investigated by thermogravimetric analysis (TGA) in an inert N_2 atmosphere, as shown in Figure 2. All of these compounds exhibited good thermal stability. The decomposition temperatures based on 5 % weight loss increased slightly with an increase in the oligofluorene arm length and the molecular weights from 395 °C for **Tr1** to 420 °C for **Tr4** (Table 1). Differential scanning calorimetry (DSC) measurements showed that these stars exhibited glassy morphologies with no obvious crystallization or melting processes detectable during the repeated scan cycles. The glass transition temperatures (T_g) increased stepwise from 49 °C for **Tr1** to 85 °C for **Tr3** with the increasing number of fluorene units, whereas the T_g for **Tr4** was weak and hard to identify. The DSC results are shown in Figure 3 and summarized in Table 1. Their wide-angle X-ray diffraction (WAXD) patterns (Figure 4) showed weak and broad halos at approximately $2\theta = 20°$, further confirming the amorphous glassy morphology of these stars.

The absorption and emission spectra of the *ortho*-substituted, six-armed stars **Tr1**–**Tr4** measured in dilute toluene (1×10^{-7} M) are shown in Figure 5. The electronic properties of **Tr1**–**Tr4** have been investigated and compared with those of their corresponding linear (**OFX**) and 2D three-armed (**T1**–**T4**) counterparts.[8] These results are summarized in Table 2. As a general trend, an increase in the oligofluorene arm length results in a distinct red-shift of the absorption maximum λ_{max}, together with a strong increase in the molar extinction coefficient ($\log\varepsilon$). It is interesting to note, however, that in comparison with their three-armed counterparts **T1**–

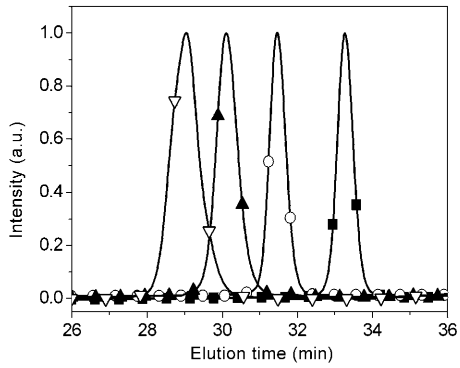


Figure 1. The GPC elution curves for **Tr1** (■), **Tr2** (○), **Tr3** (▲), and **Tr4** (▽).

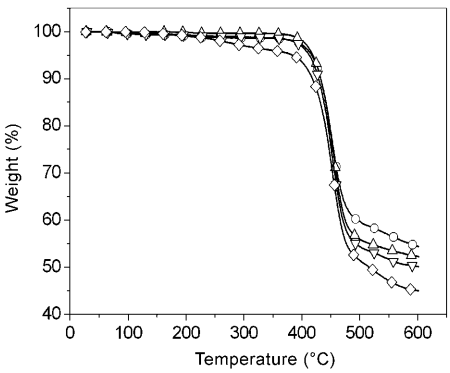


Figure 2. The TGA curves of **Tr1** (◇), **Tr2** (▽), **Tr3** (△), and **Tr4** (○). Heating rate: 10 °C min^{-1}.

Table 1. The glass transition temperatures and thermal stabilities of **Tr1**–**Tr4**.

Compound	M_w [g mol^{-1}]	T_g[a] [°C]	T_d[b] [°C]
Tr1	2842.5	49	395
Tr2	4837.7	75	416
Tr3	6832.8	85	419
Tr4	8827.9	NA	420

[a] Measured at 10 °C min^{-1}. [b] Measured at 10 °C min^{-1}. Values determined by TGA for 5 % weight loss.

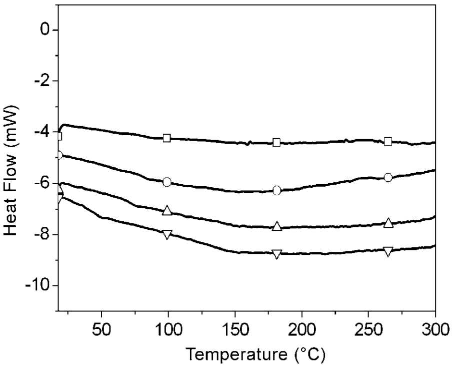


Figure 3. The DSC curves of **Tr1** (▽), **Tr2** (△), **Tr3** (○), and **Tr4** (□). Heating rate: 10 °C min^{-1}.

T4,[8] the doubling of the number of arms around the truxene core in **Tr1**–**Tr4** (by *ortho* substitution) leads to significant blueshifts (of 7–24 nm) of λ_{max} and, correspondingly, higher energy optical gaps (E_g) (Table 2). This suggests a nonplanar 3D geometry induced by the *ortho* substituents that limits π delocalization through the core or by extension between the arms. The emission spectra of **Tr1**–**Tr4** show little vibronic structure relative to **T1**–**T4** (especially for **Tr1** and **Tr2**) again suggesting a less planar, less rigid geometry.

The normalized absorption spectra of the **TrX** compounds in dilute toluene, in comparison with the **OFX** counterparts, are shown in Figure 6. On the basis of the spectroscopic investigation, we deduce that the effective conjugation length of **TrX** is constrained to be not much longer than $n+1$ fluorene units, which corresponds to the longest *para*-conjugated segment. Moreover, no strong tailing at lower energies and intensive additional bands at higher energies are notable, in-

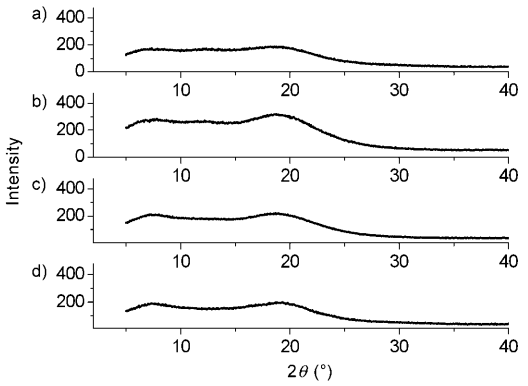


Figure 4. WAXD patterns (5–40°) of powdered **Tr1** (a), **Tr2** (b), **Tr3** (c), and **Tr4** (d). All samples were tested under the same conditions and each pattern is at its original intensity.

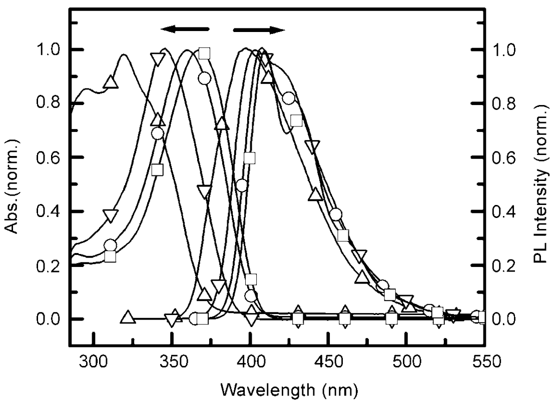


Figure 5. The normalized absorption and photoluminescence (PL) emission spectra of **Tr1** (△), **Tr2** (▽), **Tr3** (○), and **Tr4** (□) in toluene.

Table 2. The electronic properties of **TrX**,[a] **TX**,[b] and **OFX**.[c]

	λ_{max} [nm] (log ε)	λ_{onset} [nm]	E_g[d] [eV]	λ_{PL} [nm]
Tr1	295,319 (5.63)	371	3.34	398
Tr2	346 (5.92)	389	3.18	403, 421 (sh)
Tr3	360 (6.22)	400	3.10	408, 428 (sh)
Tr4	367 (6.17)	404	3.07	410, 433, 464 (sh)
T1	343 (4.97)	376.5[e]	3.29	375 (sh), 396, 416 (sh)
T2	360 (5.50)	394.5[e]	3.14	399, 422, 443 (sh)
T3	370 (5.61)	402[e]	3.08	408, 431, 460 (sh)
T4	374 (5.67)	406[e]	3.05	411, 436, 460 (sh)
OF2	328 (5.19)	364	3.41	364, 384, 402 (sh)
OF3	351 (5.27)	387.5	3.20	393, 415, 439 (sh)
OF4	362 (5.42)	399	3.11	404, 427, 454 (sh)
OF5	369 (5.54)	404	3.07	409, 432, 462 (sh)
OF7	375 (5.70)	410	3.02	412, 436, 469 (sh)

[a] Measured in 1×10^{-7} M toluene solution at 295 K. [b] Data from ref. [8]. [c] Measured in 5×10^{-7} M toluene solution at 295 K. [d] Band gap estimated from the onset absorption (in toluene). [e] Deduced from its optical band gap data.

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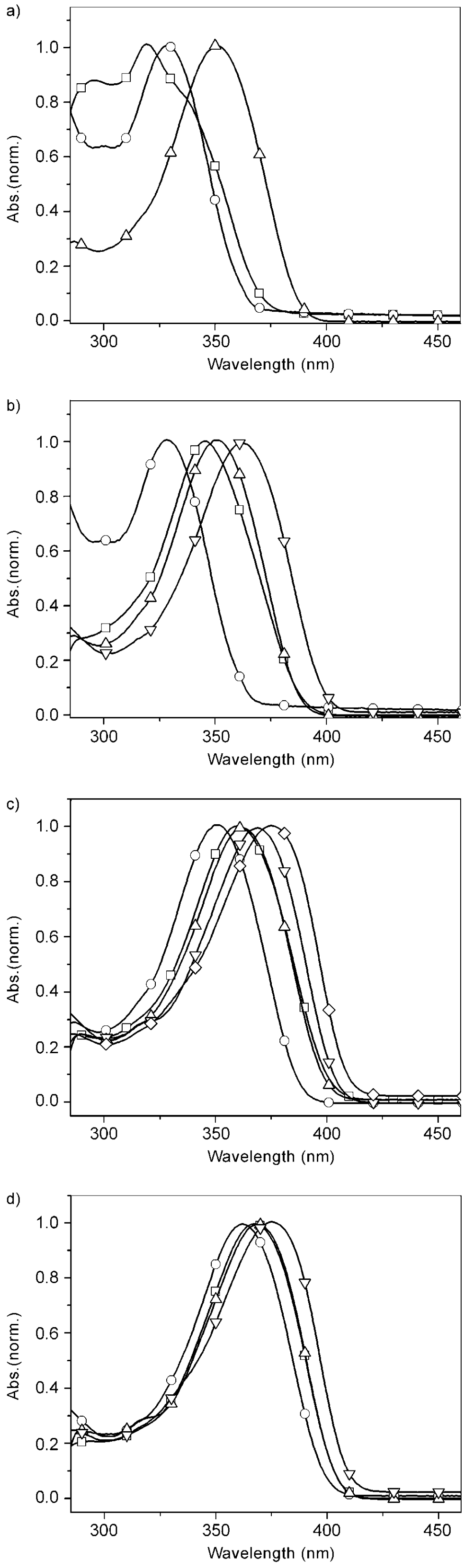

Figure 6. The normalized absorption spectra of a) **Tr1** (□) compared with those of **OF2** (○) and **OF3** (△), b) **Tr2** (□) compared with those of **OF2** (○), **OF3** (△), and **OF4** (▽), c) **Tr3** (□) compared with those of **OF3** (○), **OF4** (△), **OF5** (▽), and **OF7** (◇), and d) **Tr4** (□) compared with those of **OF4** (○), **OF5** (△) and **OF7** (▽) in dilute toluene solution.

dicating no significant electron delocalization occurred between the arms.

We have examined the relationship of λ_{max} and E_g with the number of the fluorene units in the **TrX** macromolecules. Consequently, as seen in Figure 7, we found an excellent linear correlation of $1/\lambda_{max}$ and E_g with $1/(n+1)$ ($R = 0.99877$, slope $= 0.00138$ and $R = 0.99827$, slope $= 0.91807$, respectively). This is a common feature in linear rod-shaped systems[2f] and the fact that a similar correlation is observed for our branched systems clearly demonstrates that the longest *para*-conjugated segment in the multiple *ortho*-substituted architecture plays the dominant role in determining their electronic properties.

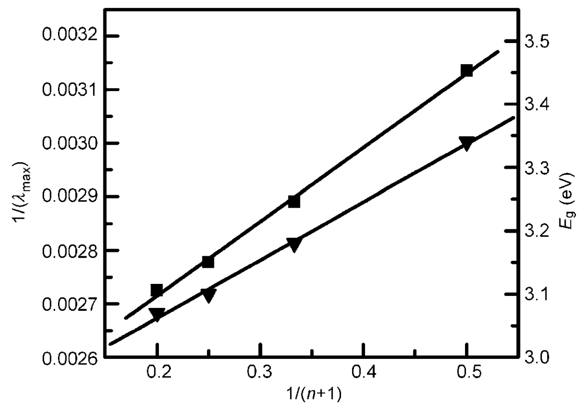

Figure 7. The relationship between the absorption maximum (λ_{max}) and the optical gap (E_g) with the number of fluorene units in the longest *para*-conjugated segment ($n+1$) in **TrX**. ■: $1/(\lambda_{max})$; ▼: E_g. $1/(\lambda_{max}) = 0.00244 + 0.00138/(n+1)$, $R = 0.99877$. $E_g = 2.87795 + 0.91807/(n+1)$, $R = 0.99827$.

Furthermore, the cyclic voltammetry (CV) of **Tr1**–**Tr4** showed simple redox patterns and large electrochemical band gaps (3.27–3.62 eV) even in film states (Table 3). The results also suggest there is little electronic interaction between the arms, even in **Tr1**, consistent with the electronic spectral properties. The HOMO and LUMO values of the six-armed nanostars **Tr1**–**Tr4** were estimated to be in the ranges of −5.66 to −5.75 and −2.39 to −2.12 eV, respectively-

Table 3. Cyclic voltammetric data of **Tr1**–**Tr4**.[a]

Compound	E_{ox}[b] [V]	E_{red}[b] [V]	E_{HOMO}[c] [eV]	E_{LUMO}[c] [eV]	E_g^{CV}[d] [eV]
Tr1	0.95	−2.32	−5.66	−2.39	3.27
Tr2	0.99	−2.52	−5.70	−2.19	3.51
Tr3	1.03	−2.59	−5.74	−2.12	3.62
Tr4	1.04	−2.52	−5.75	−2.19	3.56

[a] Cyclic voltammogram curves of drop-coated films measured in 0.1 M Bu_4NPF_6 acetonitrile solution at a scan rate of 50 mV s^{-1} at room temperature (vs. Ag/Ag$^+$). [b] Onset oxidation (p-doping) and reduction (n-doping) potentials (vs. Ag/Ag$^+$). [c] Estimated from the onset oxidation and reduction potential by using $E_{HOMO} = -E_{ox} - 4.71$ eV and $E_{LUMO} = -E_{red} - 4.71$ eV.[10a] [d] Electrochemical band gaps determined using $E_g = -(E_{HOMO} - E_{LUMO})$.

ly, which are similar to those of other homogeneous fluorene systems.[2d,3e,8]

Our findings highlight a novel molecular design comprising an *ortho*-substituted multiarm structure that could allow the introduction of isotropic physical and/or mechanical properties,[5a] while at the same time maintaining most of the important electronic properties of the rod-shaped constituents of a fully conjugated system. It can be further noted that short oligofluorenes, for example, terfluorenes, are of sufficient length for many luminescence requirements,[2c] therefore, we can envisage practical applications of our compounds without the requirement of longer arms than those reported herein.

Conclusion

We have successfully designed and synthesized a novel series of structurally well-defined, highly pure macromolecular materials **Tr1**–**Tr4** based on the novel 2,3,7,8,12,13-hexaaryltruxene scaffold. Controlled microwave heating proved to be a facile and powerful approach throughout the whole synthetic procedure, which opens an avenue for accessing well-defined, highly substituted complex structures rapidly and conveniently with high yields and purities. The novel *ortho*-substituted multiarm designs provide the materials with a nonplanar, 3D molecular geometry that helps to define π delocalization. This might provide a useful design guideline for exploring unique branched OSCs, with the aim of fine-tailoring electronic properties. We have recently demonstrated that these kind of materials with appropriate arm lengths are rather attractive for OLED and laser applications with enhanced solid-state luminescent characteristics. These results will be reported elsewhere.[21]

Experimental Section

5,5,10,10,15,15-Hexakishexyl-2,3,7,8,12,13-hexabromo-10,15-dihydro-5*H*-diindeno[1,2-a;1′,2′-c]fluorene (2): Compound **1** (5.0 g, 5.9 mmol), $FeCl_3$ (55 mg), $ClCH_2CH_2Cl$ (50 mL), and aqueous HBr (40 %, 5.7 mL, 43 mmol), with a catalytic amount of I_2, were mixed in a 100 mL round-bottomed flask with a reflux condenser. The setup was placed into the microwave cavity. The mixture was irradiated at a power efficiency of 80 W to ramp to 75 °C and held at that temperature for 35 min. Simultaneously, aqueous H_2O_2 (30 %, 4.3 mL, 43 mmol) was added dropwise to the mixture by means of a syringe. The reaction mixture was cooled to room temperature; the organic phase was separated and washed with water (2×), dried, and concentrated. The resulting residue was purified with column chromatography using hexane as an eluant to give **2** as a light yellow solid (7.2 g, 94 %). ^1H NMR (400 MHz, $CDCl_3$): $\delta = 8.52$ (s, 3H), 7.67 (s, 3H), 2.75–2.68 (m, 6H), 2.07–2.00 (m, 6H), 0.98–0.86 (m, 36H), 0.66–0.63 (t, 18H), 0.52–0.38 ppm (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 154.6$, 146.2, 140.6, 137.1, 129.4, 127.5, 123.5, 122.6, 56.2, 36.9, 31.6, 29.5, 24.2, 22.5, 14.1 ppm; (MALDI-TOF): *m*/*z* (%): 1235.6 (88) $[M-C_6H_{13}]^+$, 1155.4 (100) $[M-C_6H_{13}-Br]^+$, 1073.4 (60) $[M-C_6H_{13}-2\,Br]^+$, 993.4 (20) $[M-C_6H_{13}-3\,Br]^+$.

General procedure for the synthesis of oligofluorene boronates 5, 7, 9, and 11 through a microwave-assisted Miyaura reaction: A 10 mL pressurized vessel containing a magnetic stirrer bar was charged with the corresponding oligofluorene bromides (**3**, **6**, **8**, or **10**) (1.0 equiv), bis(pinacolato)diboron (1.6 equiv), KOAc (3.0 equiv), and DMF (2–5 mL). The mixture was carefully degassed before and after the addition of $[PdCl_2(dppf)]$ (0.06 equiv). The vessel was then sealed and heated in the CEM Discover system with a preset power of 50 W. Once the set temperature of 110 °C was reached, the microwave power regulated itself to keep that temperature for an additional 10 (for **3**), 18 (for **6**), and 25 min (for **8**). After the reaction mixture was cooled to RT, it was poured onto crushed ice and extracted several times with diethyl ether. The extracts were washed with brine (3×) and dried over anhydrous Na_2SO_4. After filtration and evaporation of the solvents, the resulting product was purified by flash chromatography on a silica gel column with hexane/ethyl acetate (95:5) to afford oligofluorene boronates (**5**, **7**, **9**, or **11**).

*Compound **5***: Pale-yellow oil; yield 93 %; ^1H NMR (400 MHz, $CDCl_3$): $\delta = 7.81$–7.80 (d, 1H), 7.74–7.69 (m, 3H), 7.34–7.31 (m, 2H), 1.99–1.95 (m, 4H), 1.39 (s, 12H), 1.11–1.01 (m, 12H), 0.77–0.73 (t, 6H), 0.60–0.57 ppm (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 151.5$, 150.1, 144.3, 141.1, 133.9, 129.1, 127.7, 126.9, 123.1, 120.3, 119.6, 119.2, 83.9, 55.3, 40.5, 31.7, 29.9, 25.2, 25.0, 23.9, 22.8, 14.2 ppm; MS (MALDI-TOF): *m*/*z*: 460.3 $[M]^+$.

*Compound **7***: White solid; yield 88 %; ^1H NMR (400 MHz, $CDCl_3$): $\delta = 7.85$–7.73 (m, 6H), 7.66–7.61 (m, 4H), 7.38–7.30 (m, 3H), 1.97–1.93 (m, 8H), 1.33 (s, 12H), 1.06–0.99 (m, 24H), 0.70–0.61 ppm (m, 20H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 152.3$, 151.7, 151.2, 150.4, 144.0, 141.2, 141.0, 140.7, 140.6, 140.3, 134.0, 129.2, 127.3, 127.1, 126.4, 123.2, 121.8, 120.7, 120.2, 120.0, 119.4, 84.1, 55.6, 55.5, 40.7, 40.6, 31.8, 30.0, 25.3, 24.1, 24.0, 22.9, 14.4 ppm; MS (MALDI-TOF): *m*/*z*: 792.9 $[M]^+$.

*Compound **9***: White solid; yield 84 %; ^1H NMR (400 MHz, $CDCl_3$): $\delta = 7.86$–7.79 (m, 6H), 7.77–7.75 (d, 2H), 7.69–7.64 (m, 8H), 7.39–7.30 (m, 3H), 2.13–2.05 (m, 12H), 1.41 (s, 12H), 1.27–1.09 (m, 36H), 0.79–0.72 ppm (m, 30H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 152.4$, 152.1, 151.8, 151.3, 150.5, 144.2, 141.1, 140.8, 140.7, 140.5, 140.4, 140.3, 134.2, 129.2, 127.3, 127.1, 126.5, 126.4, 123.3, 121.9, 121.8, 120.7, 120.3, 120.2, 120.0, 119.4, 84.1, 55.7, 55.5, 40.7, 40.6, 31.8, 30.0, 25.3, 22.9, 14.4 ppm; MS (MALDI-TOF): *m*/*z*: 1124.9 $[M]^+$.

*Compound **11***: White solid; yield 80 %; ^1H NMR (400 MHz, $CDCl_3$): $\delta = 7.88$–7.65 (m, 20H), 7.70–7.60 (m, 2H), 7.40–7.35 (m, 3H), 2.08–1.99 (m, 16H), 1.40 (s, 12H), 1.27–1.10 (m, 48H), 0.85–0.73 ppm (m, 40H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 153.6$, 152.1, 151.7, 151.3, 151.2, 141.2, 141.0, 140.8, 140.7, 140.6, 140.5, 140.4, 140.3, 140.2, 140.1, 139.4, 130.2, 127.2, 127.0, 126.4, 126.3, 123.1, 121.7, 121.3, 121.2, 120.2, 120.1, 119.9, 84.1, 55.7, 55.6, 55.4, 40.6, 40.5, 31.5, 29.7, 24.1, 23.9, 22.8, 14.4 ppm; MS (MALDI-TOF): *m*/*z*: 1457.2 $[M]^+$.

General procedure for the synthesis of oligofluorene bromides 6, 8, and 10 through an open-vessel, microwave-assisted Suzuki reaction: A solution of oligofluorene boronates (1.0 equiv, 4.6 mmol), **4** (3.0 equiv), and K_2CO_3 (2.0 equiv) in THF (40 mL) in a 100 mL round-bottomed flask with a reflux condenser was carefully degassed before and after the addition of $[Pd(PPh_3)_4]$ (0.02 equiv). The mixture was then irradiated at 80 W for 15–20 min in the microwave cavity under nitrogen. After cooling to RT, the reaction mixture was diluted with water and extracted with dichloromethane. The extracts were washed with brine, dried (Na_2SO_4), and evaporated. The resulting residue was purified with column chromatography using hexane as an eluant to afford oligofluorene bromides (**6**, **8**, and **10**).

*Compound **6***: White solid; yield 86 %; ^1H NMR (400 MHz, $CDCl_3$): $\delta = 7.85$–7.75 (3H, m), 7.72–7.60 (5H, m), 7.54–7.48 (2H, m), 7.44–7.34 (3H, m), 2.18–1.92 (8H, m), 1.24–1.02 (24H, m), 0.86–0.66 ppm (20H, m); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 153.17$, 151.42, 151.01, 150.91, 140.96, 140.64, 140.38, 140.18, 139.77, 139.09, 129.91, 126.98, 126.73, 126.20, 126.12, 125.96, 122.85, 121.33, 121.00, 120.89, 119.92, 119.84, 119.68, 55.45, 55.10, 40.30, 40.22, 31.40, 29.63, 29.56, 23.70, 23.66, 22.51, 13.97 ppm; MS (MALDI-TOF): *m*/*z*: 744.4 $[M]^+$, 666.5 $[M-Br]^+$.

*Compound **8***: White solid; yield 85 %; ^1H NMR ($CDCl_3$, 400 MHz): $\delta = 7.84$–7.74 (m, 6H), 7.69–7.60 (m, 8H), 7.50–7.47 (m, 1H), 7.38–7.26 (m, 4H), 2.16–1.93 (m, 12H), 1.27–1.09 (m, 36H), 0.93–0.72 ppm (m, 30H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 152.1$, 151.8, 151.3, 141.1, 140.9, 140.7, 140.3, 130.3, 127.3, 127.1, 126.5, 126.4, 123.3, 121.8, 121.7, 120.3, 120.2, 120.0, 55.5, 40.7, 31.8, 30.0, 24.7, 24.2, 24.1, 22.9, 14.4 ppm; MS (MALDI-TOF): m/z: 1077.4 $[M]^+$, 998.0 $[M-\mathrm{Br}]^+$.

*Compound **10***: White solid; yield 80%; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta=$ 7.86–7.60 (m, 20H), 7.52–7.46 (m, 2H), 7.38–7.31 (m, 3H), 2.14–1.99 (m, 16H), 1.28–1.11 (m, 48H), 0.90–0.74 ppm (m, 40H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta=$153.5, 152.0, 151.7, 151.3, 151.2, 141.2, 141.0, 140.8, 140.7, 140.6, 140.5, 140.4, 140.3, 140.2, 140.1, 139.4, 130.2, 127.2, 127.0, 126.4, 126.3, 123.1, 121.7, 121.3, 121.2, 120.2, 120.1, 119.9, 55.8, 55.6, 55.4, 40.6, 40.5, 31.7, 29.9, 24.1, 24.0, 22.8, 14.3 ppm; MS (MALDI-TOF): m/z: 1410.8 $[M]^+$, 1331.8 $[M-\mathrm{Br}]^+$.

General procedure for the synthesis of Tr1–Tr4: A solution of **2** (1.0 equiv, 0.10 mmol), **5**, **7**, or **9** (7.8 equiv) and K_2CO_3 (15 equiv) in THF (3–5 mL) in a 10 mL pressurized vessel was carefully degassed before and after the addition of $[Pd(PPh_3)_4]$ (0.6 equiv). The vessel was then sealed and heated in the CEM Discover system. The initial microwave power was set at 100 W. After the set temperature of 150 °C was reached, the microwave power regulated itself to keep that temperature for 15–33 min before cooling to RT. The mixture was subsequently diluted with CH_2Cl_2 and then washed with aqueous HCl (1 M) and with a saturated solution of brine, dried (Na_2SO_4), and evaporated. The resulting residue was purified with column chromatography using hexane/CH_2Cl_2 as eluant to give the final product. The temperature of the reaction was monitored by using a calibrated infrared temperature control mounted under the reaction vessel. A load cell, connected to the vessel through the septum, controlled the pressure.

*Compound **Tr1***: Colorless solid; yield 88.6%; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta=$8.52 (s, 3H), 7.70–7.55 (m, 18H), 7.48–7.33 (m, 9H), 7.32–7.25 (d, 12H), 7.14–7.08 (m, 6H), 3.15–3.05 (m, 6H), 2.24–2.17 (m, 6H), 2.08–2.04 (m, 12H), 1.79–1.73 (m, 6H), 1.64–1.59 (m, 6H), 1.22–0.96 (m, 108H), 0.79–0.47 ppm (m, 90H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta=$153.0, 151.0, 150.9, 150.4, 145.3, 142.0, 141.3, 141.2, 141.1, 139.9, 139.7, 139.6, 139.3, 138.6, 132.4, 132.3, 129.7, 128.8, 128.7, 127.7, 127.0, 126.9, 126.8, 125.2, 124.5, 124.4, 123.0, 122.8, 119.9, 119.8, 119.6, 119.1, 56.2, 55.3, 55.1, 41.3, 40.6, 37.6, 32.2, 31.8, 31.6, 30.5, 29.9, 29.7, 24.4, 24.1, 23.9, 23.2, 22.8, 22.6, 14.3, 14.2 ppm; MS (MALDI-TOF): m/z (%): 2949.2 (20) $[M+\mathrm{Ag}]^+$, 2756.3 (100) $[M-C_6H_{13}]^+$, 2692.8 (25) $[M-C_6H_{13}-63.3]^+$, 2638.6 (25) $[M-C_6H_{13}-117.7]^+$, 2575.2 (25) $[M-C_6H_{13}-181.1]^+$; elemental analysis calcd (%) for $C_{213}H_{282}$: C 90.00, H 10.00; found: C 89.86, H 10.12.

*Compound **Tr2***: Colorless solid; yield 85.2%; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta=$8.59 (s, 3H), 7.80–7.59 (m, 48H), 7.54–7.49 (m, 9H), 7.38–7.30 (m, 18H), 7.20–7.16 (m, 6H), 3.15–3.13 (m, 6H), 2.26–1.85 (m, 48H), 1.72–1.66 (m, 6H), 1.29–1.05 (m, 180H), 0.90–0.63 ppm (m, 150H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta=$153.1, 151.8, 151.7, 151.6, 151.4, 151.2, 150.8, 145.4, 142.1, 141.1, 141.0, 140.8, 140.5, 140.4, 140.0, 139.4, 139.3, 138.7, 129.8, 128.8, 127.7, 127.3, 127.2, 127.0, 126.4, 126.2, 125.3, 124.5, 123.2, 123.1, 121.7, 121.6, 121.4, 120.2, 120.1, 119.9, 119.7, 119.2, 56.2, 55.5, 55.4, 55.3, 44.9, 41.3, 40.6, 37.7, 32.1, 31.8, 31.7, 31.5, 30.5, 29.9, 29.8, 29.7, 29.6, 24.5, 24.2, 24.1, 24.0, 23.3, 23.2, 22.8, 22.6, 14.4, 14.2 ppm; MS (MALDI-TOF): m/z (%): 4944.5 (8) $[M+\mathrm{Ag}]^+$, 4750.6 (100) $[M-C_6H_{13}]^+$, 4722.4 (15), 4687.6 (10), 4633.2 (12), 4570.2 (10); elemental analysis calcd (%) for $C_{363}H_{474}$: C 90.12, H 9.88; found: C 89.89, H 10.03.

*Compound **Tr3***: Pale yellow solid; yield 84.4%; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta=$8.60 (s, 3H), 7.84–7.51 (m, 93H), 7.36–7.30 (m, 18H), 7.22–7.17 (m, 6H), 3.20–3.13 (m, 6H), 2.29–1.77 (m, 72H), 1.70–1.69 (m, 6H), 1.26–0.96 (m, 252H), 0.83–0.60 ppm (m, 210H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta=$152.0, 151.7, 151.2, 141.0, 140.8, 140.7, 140.5, 140.2, 127.2, 127.0, 126.4, 126.2, 123.1, 121.7, 120.2, 120.1, 119.9, 55.5, 55.4, 40.6, 32.2, 31.8, 31.7, 31.6, 29.9, 29.8, 24.0, 23.2, 22.8, 22.6, 14.4, 14.2 ppm; MS (MALDI-TOF): m/z (%): 6937.9 (20) $[M+\mathrm{Ag}]^+$, 6745.5 (100) $[M-C_6H_{13}]^+$; elemental analysis calcd (%) for $C_{513}H_{666}$: C 90.18, H 9.82; found: C 90.12, H 9.95.

*Compound **Tr4***: Pale yellow solid, yield 82.3%. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta=$8.61 (s, 3H), 7.85–7.52 (m, 129H), 7.36–7.31 (m, 18H), 7.22–7.14 (m, 6H), 3.16 (m, 6H), 2.27–1.66 (m, 102H), 1.26–1.00 (m, 339H), 0.80–0.72 ppm (m, 255H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta=$152.0, 151.2, 141.0, 140.8, 140.7, 140.2, 126.4, 126.2, 123.1, 121.7, 120.2, 120.1, 119.9, 55.5, 55.4, 40.6, 31.7, 29.9, 29.8, 24.1, 24.0, 22.8, 22.6, 14.2 ppm; MS (MALDI-TOF): m/z: 4837.65 $[M]^+$, 2507.6 $[M-C_6H_{13}]^+$, 2423.8 $[M-2C_6H_{13}]^+$; elemental analysis calcd (%) for $C_{663}H_{858}$: C 90.20, H 9.80; found: C 90.16, H 9.88.

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