Luminescence of New 2,6- and 1,5-Naphthalene-Based PPV-Type Trimers and Polymers

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A series of new soluble 2,6- and 1,5-conjugated naphthalene-based PPV-analogous trimers (**1a**,**b**–**6a**,**b**) and polymers (**24**, **25**, **26**) have been synthesized by means of Knoevenagel and Wittig reactions, combining appropriate 1,5- and 2,6-functionalized naphthylene or phenylene building blocks. The influence of cyano substituents at the vinylic double bonds of the aromatic chromophores, and of the way in which they are linked, on the absorption and photoluminescence spectra has been investigated. The electroluminescent behaviour of the new compounds has been studied in ITO/copper phthalocyanine (PcCu)/emitter (trimer or polymer)/Al devices, demonstrating the aptitude of trimeric and polymeric 1,5-naphthylenevinylenes for use in organic light-emitting diodes for the first time.

Introduction

Since the first demonstration of the light-emitting property of poly(*p*-phenylenevinylene) (PPV),[1] numerous modified oligomers and polymers of this type have been developed and applied as emissive layers in organic light-emitting diodes (OLEDs).[2,3] One of the most notable advantages that stems from the use of organic substances in LEDs is the possibility of tuning the properties of the emitted light as well as the efficiencies of the devices through chemical modification of the material. Besides the introduction of substituents, such as cyano groups to increase the electron affinity (EA),[4] partial or complete replacement of the phenylene rings in the PPV backbone by different moieties (for example naphthalene, anthracene, heterocycles) has been realized.[5] In this context, we have previously described the photoluminescence (PL) and electroluminescence (EL) of some three-ring PPV-analogous model compounds (in the following referred to as trimers), in which one or more phenylene units were replaced by naphthylene moieties linked through their 2,6-positions.[6] Incorporation of the naphthalene ring leads to a bathochromic shift in the emissions of these compounds if it is present as a side unit, whereas the opposite effect is observed when it forms the central unit. We also found that trimers made up exclusively of naphthalene units[6c] exhibit properties akin to those of the homopolymer poly(cyano-2,6-naphthylenevinylene), which shows a marked hypsochromic shift in the PL and EL maxima when compared with poly(cyano-1,4-phenylenevinylene).[4a,6a]

These results demonstrate that the electronic properties of the extended π-system, and consequently the potential applications of conjugated organic compounds in LEDs, are highly dependent on both the nature of the active building blocks and the way in which they are linked.[6,7] Figure 1 depicts the different ways in which naphthylenevinylene-type polymers can be linked, i.e. through their 1,4-, 1,5-, or 2,6-positions, respectively.

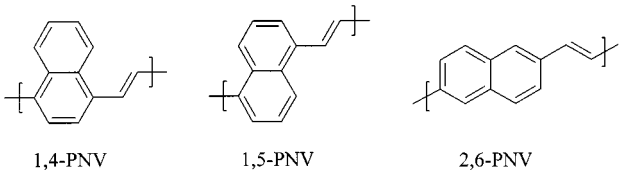

Figure 1. Chemical structures of poly(naphthylenevinylenes) with different linkages

To date, only 1,4-[6a] and 2,6-conjugated naphthalene derivatives[8] have been employed in the synthesis of conjugated polymers. The synthesis of a copoly(1,4-phenylenevinylene-1,5-naphthylenevinylene) has also been reported,[9] but its photo- and electroluminescent properties were not investigated in a systematic way.

We report herein on the synthesis of a new series of PPV-analogous trimers and polymers incorporating naphthalene moieties conjugated through their 2,6- and, for the first time, 1,5-positions. Furthermore, we have investigated the photo- and electroluminescent properties of the new compounds in a systematic way, using a standard device set-up.

Results and Discussion

1. Synthesis and Optical Properties

Figure 2 depicts the new trimers **2a**,**b**, **4a**,**b**, **5a**,**b**, and **6a**,**b**, along with the cyano-2,6-conjugated naphthalene trimers **1a**,**b** and **3a**,**b**, on which we have recently reported.[6c]

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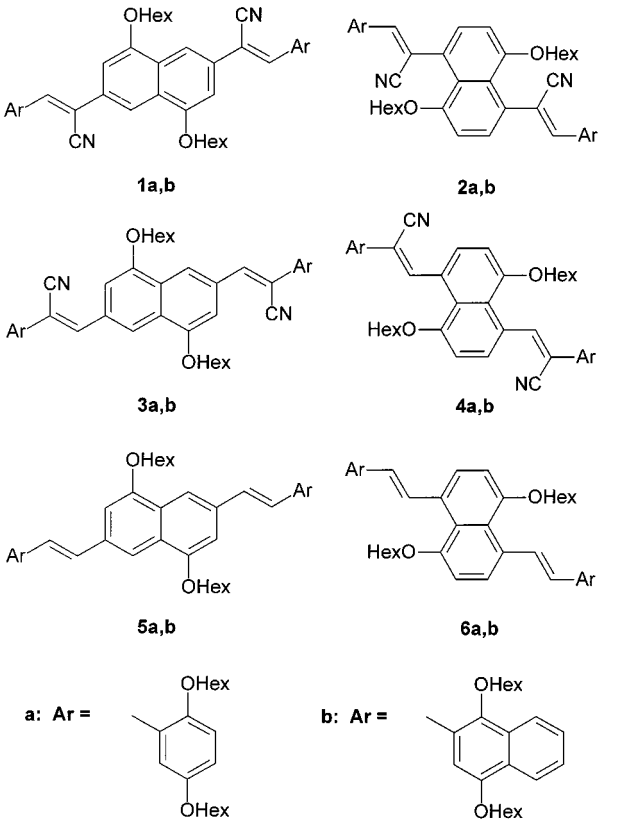
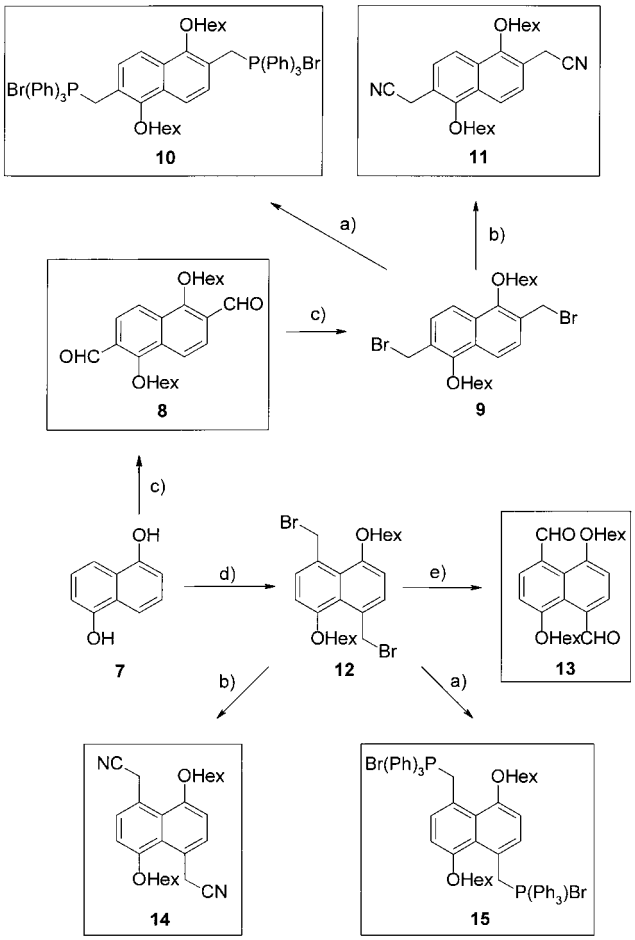


Figure 2. Comparison of the trimeric Knoevenagel and Wittig structures

With this array of naphthalene trimers at hand, it has now been possible to systematically study the influence of several parameters on their absorption and luminescent behaviour, specifically: (i) the introduction of a cyano group at both vinylic double bonds, (ii) the type of building block incorporated as a side unit (phenyl or naphthyl), and (iii) the position of linkage at the central naphthalene moiety (2,6- or 1,5-conjugated). For the sake of simplicity, the central naphthalene ring is referred to as the central unit and the peripheral phenylene or naphthylene rings as side units. The position of the cyano group has been varied from an α- (adjacent to the central unit, compounds **1a**,**b** and **2a**,**b**) to a β-position (adjacent to the side-units, **3a**,**b** and **4a**,**b**). The cyano derivatives have been compared with the corresponding compounds lacking a cyano group (**5a**,**b** and **6a**,**b**). Similarly, a 2,6-linked trimer has been compared with the corresponding 1,5-linked derivative. In all these trimers, the side units are either phenylene (**a**) or naphthylene (**b**) moieties, both substituted with solubilizing hexyloxy chains.

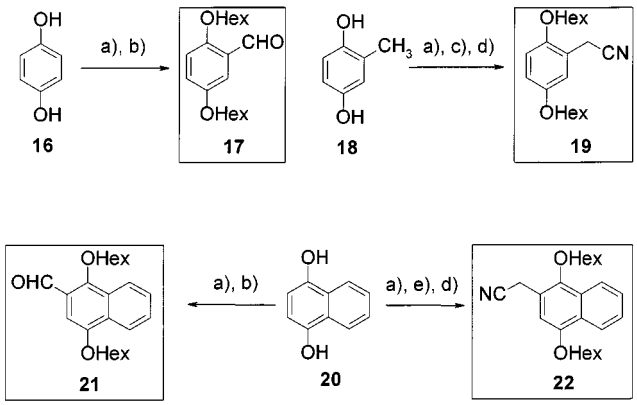
Scheme 1 shows the synthetic pathways and reaction conditions by which the precursors of the central units of all the new compounds were obtained. The commercially available 1,5-dihydroxynaphthalene (**7**) could be used as starting material for all the required 2,6- and 1,5-functionalized naphthalene derivatives (**8**, **10**, **11**, **13**, **14**, and **15**). The 2,6-derivatives **8**, **10**, and **11** have been described in our previous reports.[6a,6b] Dialdehyde **13** could be obtained by means of a Sommelet reaction analogous to a procedure described in the literature,[10a] but with a slight modification of the purification step (see Experimental Section). Bromo−cyano exchange in the dibromomethyl compound **12**,[10b] using tetraethylammonium cyanide according to a literature procedure,[10c] furnished the dicyano derivative **14**. The diphosphonium salt **15** was prepared by treating **12** with triphenylphosphane in dry toluene.[10b] Scheme 2 shows the synthetic protocol used to obtain the side unit precursors required for preparing the trimers. These procedures have been described in detail in an earlier report.[6c]



Scheme 1. Synthesis of the naphthylene central unit precursors **8**, **10**, **11**, **13**, **14**, and **15**: (a) $P(Ph)_3$, DMF, Δ*T*; (b) $[Et_4N]^+CN^-$, room temp.; (c) ref.[6b]; (d) (i) HexBr, EtOH, Δ*T*; (ii) $(CH_2O)_n$, HBr, AcOH, room temp.; (e) (i) HMTA; (ii) 50% AcOH

The functionalized precursor molecules obtained according to Schemes 1 and 2 could then be combined as building blocks to furnish the new naphthalene-based trimers and polymers presented herein. The syntheses of the trimers **1a**,**b** to **4a**,**b** and of the polymers **23**, **24**, **25**, and **26** were based on the Knoevenagel reaction, whereas compounds **5a**,**b** and **6a**,**b** were obtained by means of Wittig reactions. The reaction schemes are given in the appropriate sections (Schemes 3, 4, and 5, respectively).

All the compounds have been fully characterized by ^{1}H-NMR, ^{13}C-NMR, FT-IR, and UV/Vis spectroscopy, HRMS, elemental analysis, and PL and EL spectroscopy. The hexyloxy substituents on both the naphthalene and



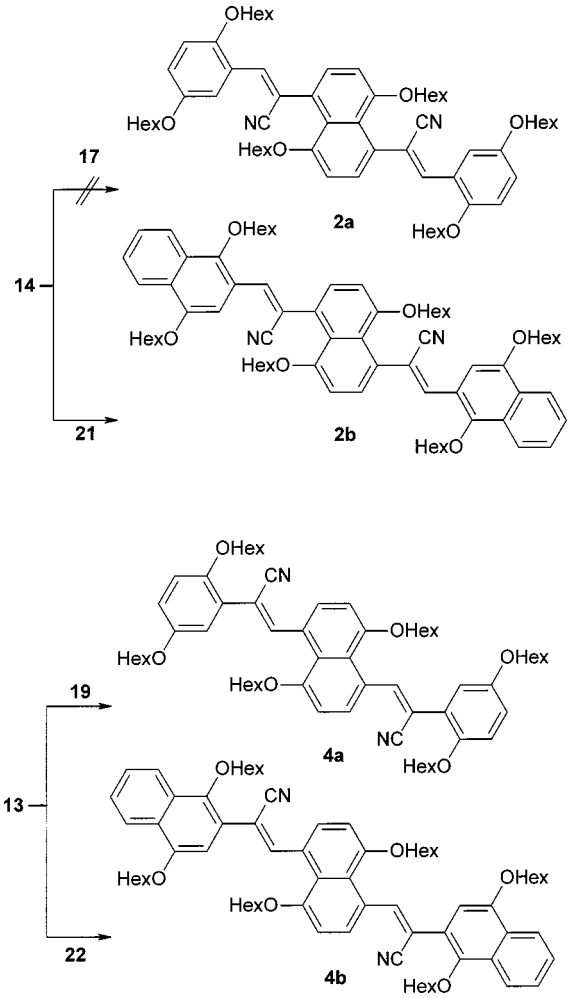
Scheme 2. Synthesis of the side-unit precursors **17**, **19**, **21**, and **22**: (a) HexBr, EtOH, ΔT; (b) $Cl_2HCOCH_3/TiCl_4$, CH_2Cl_2; (c) NBS, CCl_4, ΔT; (d) $(n\text{Bu})_4NCN$, CH_2Cl_2; (e) HBr/AcOH, AcOH; for further details, see ref.[6c]

phenyl units render these materials highly soluble in common organic solvents such as dichloromethane, chloroform, tetrahydrofuran, etc. Moreover, the processability required for the preparation of thin films for PL and EL devices is enhanced.

1.a) Knoevenagel Trimers

By combining the appropriate building blocks depicted in Schemes 1 and 2, the cyano-1,5-naphthalene trimers should be accessible. Indeed, as mentioned above, by Knoevenagel reactions of dialdehyde **13** with the cyanomethyl compounds **19** and **22**, and of dicyano compound **14** with aldehyde **21** (Scheme 3), **2b**, **4a**, and **4b** were obtained in moderate yields (26−45%).

It is noteworthy that the yields of the cyano-2,6-naphthalene trimers **1a**,**b** and **3a**,**b**[6c] were considerably higher than those of the corresponding cyano-1,5-naphthalene derivatives **2a**,**b** and **4a**,**b**. Furthermore, the synthesis of **2b** required a higher reaction temperature compared with those of **4a** and **4b** (see Experimental Section). The synthesis of **2a** could not be achieved under a variety of reaction conditions, despite varying different parameters (temperature, time, solvent). We ascribe the lower yields or even failure of the Knoevenagel reaction to greater steric hindrance of the cyano groups when the reaction takes place at the 1,5-positions, as these will lie in very close proximity to the hexyloxy substituents at the central ring. This steric hindrance will also influence the luminescent properties of the compounds (vide infra). The optical properties of the new Knoevenagel trimers **2b** and **4a**,**b** were investigated by recording UV/Vis spectra of their CH_2Cl_2 solutions and of thin films. The UV/Vis and PL data of compounds **2b**, **4a**,



Scheme 3. Synthesis of the Knoevenagel trimers **2a**,**b** and **4a**,**b**

and **4b** are listed in Table 1, along with those of **1a**,**b** and **3a**,**b**.

In the following, the positions of the UV/Vis maxima refer to the bands at lowest energy in CH_2Cl_2 solution, which are assigned to the $S_0 \rightarrow S_1$ transitions. The UV/Vis absorbances of the thin films are only slightly shifted (see Table 1, $\Delta\lambda \leq 16$ nm for the transition at lowest energy) compared with the solution spectra, except in the case of **3b**, for which a blue-shift of 24 nm is observed. The PL maxima relate to spectra recorded from thin films on glass carriers, which were prepared by evaporation of the solvent from solutions of the material (trimers) or by spin-coating (polymers).

Although we have no data for **2a**, comparison of the UV/Vis and PL data for the other compounds allows an insight

Table 1. UV/Vis (in CH_2Cl_2 solution and as thin films) and photoluminescence (PL) data of the Knoevenagel trimers **1a**,**b**, **2b**, **3a**,**b** and **4a**,**b**

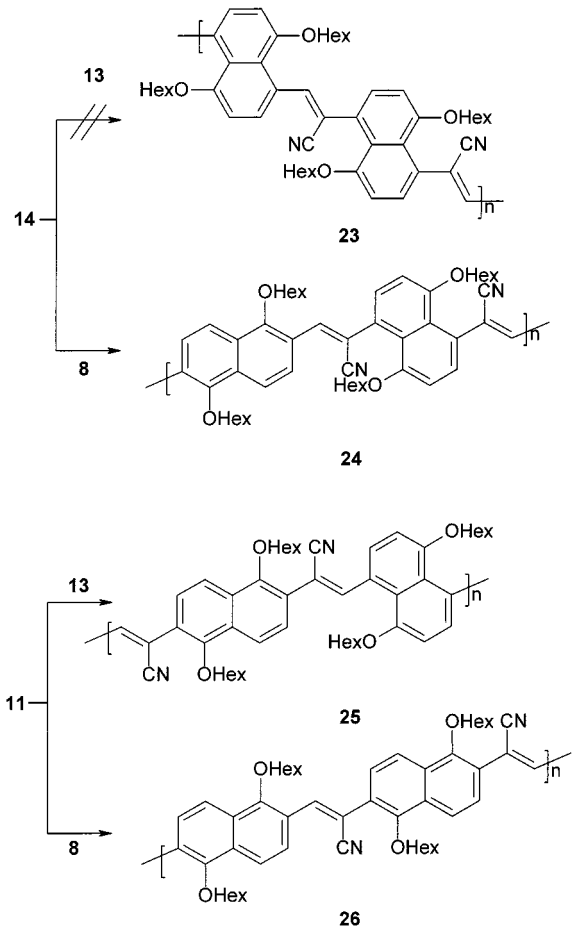
Compound	**1a**	**1b**	**3a**	**3b**	**2b**	**4a**	**4b**
UV/Vis (CH_2Cl_2) [nm]	383, 294	402, 295	395, 342, 291	412, 349, 297	378, 318, 286	371, 250 (sh)	380, 250
UV/Vis (solid state) [nm]	399, 306	399, 295	401, 349, 301	388, 346, 297	387, 370, 319	366	374
PL (solid state) [nm]	477	482	510	506	452	485	516

into the effects of the nature of side units, the presence of cyano groups at the vinylic system, and the position of linkage at the central naphthalene ring on the absorption and luminescent behaviour. Thus, on changing the position of the cyano group from an α- (**2b**) to a β-position (**4b**), a bathochromic shift in the UV/Vis and PL maxima is observed, a phenomenon that we have encountered previously with the cyano-2,6-trimers (i.e. on going from **1a** to **3a** or from **1b** to **3b**).[6c,6d] Replacing the phenyl side unit (**4a**) by a naphthyl moiety (**4b**) also shifts the maxima to longer wavelengths, which again corroborates the results for the cyano-2,6-derivatives (i.e. going from **1a** to **1b** or from **3a** to **3b**, considering the UV/Vis maxima). However, the PL maximum of **3b** is slightly blue-shifted compared with that of **3a**. This is believed to be due to suppression of the wide-amplitude torsional modes about the phenyl−C(CN) bond in the solid state, as has also been found for the phenyl analogues of **3a** and **3b**.[6c] This suppression is more marked for **3b**, in which the cyano group is present in the β-position, thus leading to a hypsochromic shift. At this point, it must be stressed that in the condensed state the fluorescence of molecules with weak intermolecular coupling stems from a rather complex interplay of several effects.[6e] These *inter*molecular effects are superimposed on the *intra*molecular effects that originate from the electronic influence of the side unit. Nevertheless, in most cases the PL shifts of the new compounds are in agreement with the UV/Vis shifts. By measuring PL in thin solid films, the spectra can be compared with the EL data (see below).

Changing from a 2,6- to a 1,5-linkage influences the absorption and luminescence in the following way: the UV/Vis and PL spectra clearly indicate a hypsochromic shift, for example when comparing **2b** with **1b** or **4a** with **3a**. Comparison of **4b** with **3b** reveals a blue shift in the UV/Vis absorbance, but a red shift in the PL. This gives a further indication that in the solid state the influence exerted by the molecular interaction can compensate or even outweigh the intramolecular effects. The hypsochromic shift can be attributed to the steric interactions between the hexyloxy groups at the central unit and the vinylene double bonds, which are greater in the case of 1,5-linkage (cf. the angles between C−O and C−H/C−N). The resulting torsion of the molecule decreases the conjugation, leading to a hypsochromic shift for 1,5-conjugated compounds (Table 3). In the case of the Knoevenagel trimers, the steric demand of the vinylic cyano group is greater and, therefore, the shift is more pronounced.[6d]

1.b) Knoevenagel Polymers

The precursor molecules shown in Scheme 1 represent useful building blocks not only for the synthesis of 1,5-linked naphthalene trimers but also for the preparation of the corresponding polymeric materials. Therefore, we have combined the dicyanomethyl compounds **11** and **14** with the dialdehydes **8** and **13** in multiple Knoevenagel reactions. In this way, we obtained the polymers **24** and **25**, as illustrated in Scheme 4.



Scheme 4. Synthesis of the Knoevenagel polymers **23**, **24**, **25**, and **26**

Polymer **26**, which we have described previously,[6a] was synthesized once more for comparison purposes. The synthesis of **23** failed, presumably due to the high steric demand of the 1,5-functionalized building blocks in the Knoevenagel reaction (see above). The UV/Vis and PL data of **24**, **25**, and **26** are presented in Table 2.

Table 2. UV/Vis and PL data of the Knoevenagel polymers **24**, **25**, and **26**

Compound	**24**	**25**	**26**
UV/Vis (CH_2Cl_2) [nm]	396, 330, 280	386, 251	417, 360, 295
UV/Vis (solid state) [nm]	402	396	427, 351
PL (solid state) [nm]	499	506	499, 526 (sh)

As in the case of the trimers **2b** and **4a**,**b**, substitution of a 2,6-linked naphthalene moiety by a 1,5-linked unit leads to a hypsochromic shift in the UV/Vis bands attributable to the $S_0 \rightarrow S_1$ transition, e.g. on going from **26** to **24** or **25**. The PL spectra do not show this effect, the maxima re-

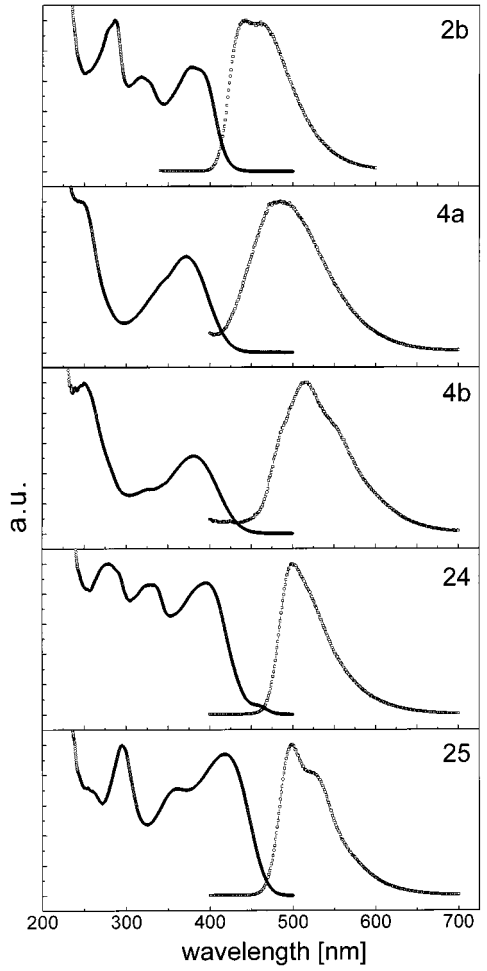
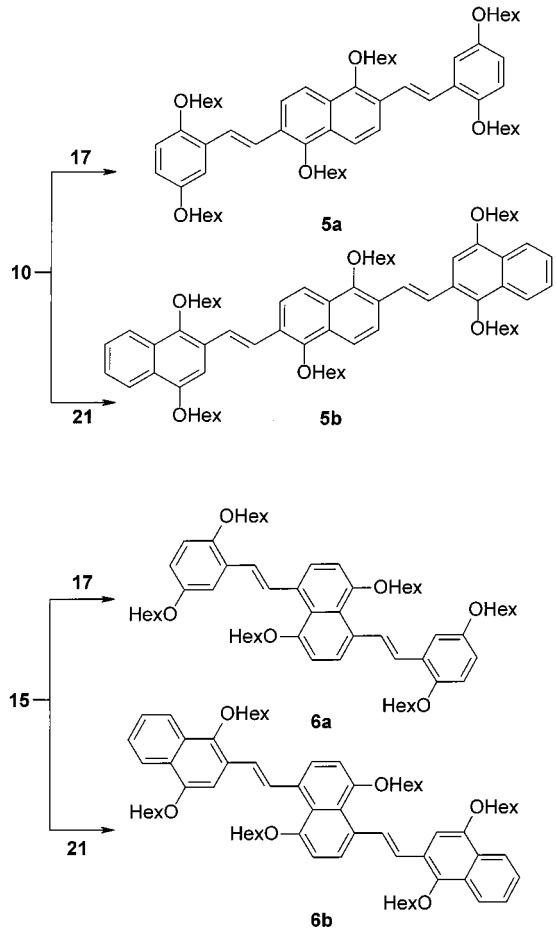


Figure 3. UV/Vis spectra in CH_2Cl_2 solution (solid line) and PL (squares) of the Knoevenagel trimers **2b**, **4a**,**b** and of polymers **24** and **25**

maining at approximately 500 nm. Figure 3 shows the UV/Vis and PL spectra of both the new Knoevenagel trimers (**2b**, **4a**,**b**) and the new polymers (**24**, **25**).

1.c) Wittig Trimers

The results described thus far would seem to suggest that in the case of 1,5-linkage the cyano groups have a high steric requirement and thus impede the Knoevenagel reaction, leading to low yields or even to a failure of the synthesis. To circumvent these difficulties, we decided to prepare the corresponding compounds *without* cyano groups, bearing only hydrogen atoms at the vinylic double bonds. As mentioned above, Wittig reactions of the bis(triphenylphosphonium) salts **10** and **15** with the aldehydes **17** and **21**, respectively, using potassium *tert*-butoxide as the base, furnished the trimers **5a**,**b** and **6a**,**b** in good yields (56−80%) (Scheme 5).



Scheme 5. Synthesis of the Wittig trimers **5a**,**b** and **6a**,**b**

Trimers **5a**,**b** and **6a**,**b** were obtained as mixtures of three isomers, (*E*/*E*), (*Z*/*Z*), and (*E*/*Z*), as evidenced by the NMR spectra. By recrystallization from ethyl acetate, the pure (*E*/*E*) isomers could be separated for all compounds, identifiable on the basis of a coupling constant $J > 16$ Hz for the vinylic protons and three triplet signals at $\delta = 3.86-4.22$ for the OCH_2 protons of the hexyloxy substituents in their ^{1}H-NMR spectra. The UV/Vis and PL maxima are listed in Table 3 and may be compared with those of the corresponding compounds with cyano groups attached to the vinylic systems **1a**,**b**, **3a**,**b**, **2b**, and **4a**,**b** (Table 1).

Table 3. UV/Vis and PL data of the Wittig trimers **5a**,**b** and **6a**,**b**

Compound	**5a**	**5b**	**6a**	**6b**
UV/Vis (CH_2Cl_2) [nm]	388, 298, 290	419, 399, 296, 261	372, 270 (sh)	381, 272
UV/Vis (solid state) [nm]	388	435, 407	373	374
PL (solid state) [nm]	545	536 (sh), 500, 469	468	475

Considering the electronic effect, cyano substitution leads to an overall stabilization of the frontier orbital levels, in accord with its acceptor nature, with the LUMO level being more affected.[11] This asymmetry in stabilization

would be expected to result in a shift towards longer wavelengths of the UV/Vis absorptions and PL emissions of the cyano-substituted molecules. However, the spectra show only slight bathochromic or even hypsochromic shifts when the Knoevenagel trimers **2b** and **4a**,**b** are compared with their Wittig counterparts **6a**,**b** (or **1a**,**b** and **3a**,**b** with **5a**,**b**, respectively). This lowering or even compensation of the bathochromic shift can be rationalized in terms of steric hindrance,[6b] since this effect is particularly distinct in the case of **1b**, **2b**, and **3b**, in which the cyano group is located in an α-position, in close proximity to the bulky central dihexyloxynaphthalene ring. Steric interaction between the dihexyloxy-substituted naphthalene and the cyano moiety leads to a torsion of the molecular backbone and, thus, to a loss of conjugation. These two opposite effects, i.e. the unsymmetrical stabilization of the frontier orbitals, causing a bathochromic shift, and the steric hindrance, leading to a hypsochromic shift, make it difficult to predict the exact influence of the cyano moiety on the maxima.[6c]

Nevertheless, the results obtained concerning the influence of the linkage position and of the nature of the side unit are more clear-cut: Replacement of a phenyl side unit by a naphthyl moiety (i.e. going from **5a** to **5b** and from **6a** to **6b**) again leads to a bathochromic shift, while the 1,5-linked trimers (**6a**,**b**) exhibit a marked hypsochromic shift in their absorbances and emissions compared with their 2,6-linked counterparts (**5a**,**b**).

2. Device Preparation and Electroluminescence

In addition to UV/Vis and PL measurements, the naphthalene trimers **2b**, **4a**,**b**, **5a**,**b**, and **6a**,**b** and the polymers **24**, **25**, and **26** were tested with regard to their electroluminescent (EL) properties. For this purpose, OLEDs were prepared with an ITO/copper phthalocyanine (PcCu)/emitter (trimer or polymer)/Al configuration, which is illustrated in Figure 4.

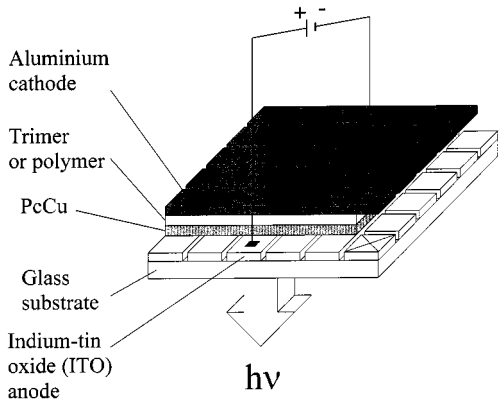


Figure 4. Schematic set-up of the devices used for the electroluminescence screening tests

The ITO glass substrate acting as the anode was structured in five columns according to a method that we have described previously.[12] The aluminium cathode on the top was deposited in five stripes in an orthogonal arrangement, thus yielding a total of 25 electroluminescent devices, which could be driven separately. PcCu was condensed onto the ITO as a hole transport layer[13] with a layer thickness of approximately 30 nm. The emitting layer was generated by vacuum deposition of the trimer or by spin-coating of the polymer from chloroform solution, yielding films with thicknesses of 100−200 nm. The EL measurements were carried out in air at room temperature.

The Wittig trimers **5a**,**b** and **6a**,**b** showed bright electroluminescence that was visible in daylight. Their EL spectra are shown in Figure 5, together with the corresponding UV/Vis and PL spectra. In contrast, only for the Knoevenagel trimer **4a** and for polymers **24** and **26** was EL detected, and the brightness was much reduced compared with that of the Wittig trimers. No EL was detected at all for compounds **2b**, **4b** and the polymer **25**. The observed maxima are listed in Table 4.

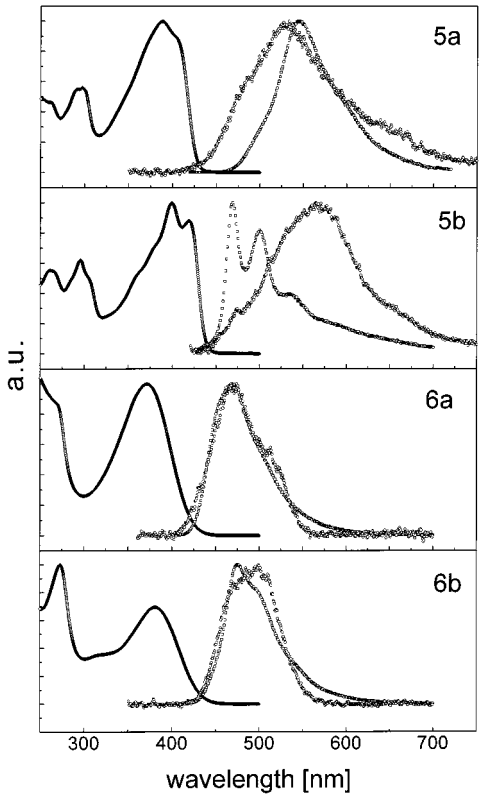


Figure 5. UV/Vis spectra in CH_2Cl_2 solution (solid line), PL (squares) and EL (circles) of the Wittig trimers **5a**,**b** and **6a**,**b**

Table 4. Electroluminescence (EL) maxima obtained for the trimers **4a**, **5a**,**b**, and **6a**,**b**, and for the polymers **24** and **26**

Compound	**4a**	**5a**	**5b**	**6a**	**6b**	**24**	**26**
EL [nm]	511	527	565	467	499	513	527

For **5a**,**b** and **6a**,**b**, the colours of the emitted light range from blue to green-yellow. The EL spectra are in good agreement with the corresponding thin-film photoluminescence spectra, except in the case of **5b**, where the EL maximum is red-shifted by 88 nm compared with the PL maximum. While slight differences (blue-shifts) can be ex-

plained in terms of re-absorption of the red emission part by the PcCu layer,[6c] which shifts the maximum to a shorter wavelength (see the spectra of **5a**), the strong bathochromic shift for **5b** originates from a different energy transition. The fine structure that is particularly distinct in the UV/Vis and PL spectra of this compound gives a further indication of the occurrence of interesting optoelectronic processes in this case. Investigations concerning these phenomena are currently in progress. In the case of compound **6b**, the EL maximum (λ = 499 nm) is slightly bathochromically shifted compared to the PL maximum (λ = 475 nm), but corresponds to a shoulder ($\lambda \approx$ 500 nm) in the corresponding PL spectrum. We therefore conclude that the EL maximum and the PL shoulder originate from the same energy transition. In general, we found that the Wittig trimers showed better electroluminescent properties, although we did not quantify these findings. At this point, it should be stressed that there are quite a number of parameters that influence the EL intensity and that have to be taken into consideration, such as layer thickness, driving voltage, degradation mechanisms, and film characteristics. Studies on the promising Wittig trimers with regard to these parameters are currently in progress.

Conclusions

By applying Wittig or Knoevenagel reactions, we have synthesized and then characterized a series of new, highly soluble, naphthalene-based PPV-analogous trimers and polymers. The precursor molecules synthesized for this purpose represent useful building blocks for the preparation of a variety of luminescent naphthalene-based materials. The trimers **2b**, **4a**,**b**, and **6a**,**b** represent the first examples of such compounds in which a central naphthalene ring is conjugated through its 1,5-positions. Comparison of the UV/Vis and thin-film PL spectra of these trimers with those of the 2,6-conjugated analogues **1a**,**b**, **3a**,**b**, and **5a**,**b** has revealed a marked hypsochromic shift of the maxima, which can be attributed to the effects of steric hindrance. The influence of cyano groups at the vinylic double bonds and of the type of aromatic ring (phenyl vs. naphthyl) present as a side-unit has also been studied and the results were in accordance with previous findings made for similar molecules.[6c] The electroluminescent capabilities of the new materials have been tested by preparing segmented ITO/PcCu/emitter (trimer or polymer)/Al devices. The Wittig trimers **5a**,**b** and **6a**,**b** showed better electroluminescent properties than the Knoevenagel compounds, thus making them promising candidates for OLED applications.

Experimental Section

Chemicals received from commercial sources (Aldrich and Fluka) were used without further purification. All reactions were performed under dry argon. Solvents were dried according to standard procedures. Melting points are uncorrected. − Infrared spectra were recorded from samples in KBr pellets using a Bruker IFS 48 spectrometer. − UV/Vis spectra were recorded from samples in CH_2Cl_2 solution or as thin films with a Perkin−Elmer Lambda 2 spectrophotometer. − The PL of films deposited by solvent evaporation was measured with a SPEX fluorolog 112 in the 45° configuration. For EL measurements, an HP 6030A voltage source was used together with a Keithley 171 DMM. EL spectra were recorded in air at room temperature from devices with an ITO/(phthalocyanine)copper (PcCu)/trimer or polymer/Al configuration using a waveguide diode-array set-up. − NMR spectra were recorded with a Bruker AC 250 spectrometer at 250 MHz (^{1}H) and 62.9 MHz (^{13}C) in $CDCl_3$ and internally referenced to $CHCl_3$ (^{1}H: δ = 7.24, ^{13}C: δ = 77.00). − Elemental analyses were carried out with a VarioEL V analyzer. − HRMS (EI) spectra were measured with a Finnigan ISQ 70 mass spectrometer.

General Procedure for the Knoevenagel Reaction: As a representative example, 0.30 mmol of 2-(cyanomethyl)-1,4-bis(hexyloxy)naphthalene (**22**) and 0.60 mmol of dialdehyde **13** were dissolved in a mixture of THF (1 mL) and *t*BuOH (2 mL) under argon at room temperature. A drop of tetra-*n*-butylammonium hydroxide solution (25%, 0.8 M) and 0.05 mmol of anhydrous potassium *tert*-butoxide were then added, and the mixture was vigorously stirred while it warmed to 40 °C. After 20 min, the mixture was poured into methanol (50 mL, acidified with a drop of acetic acid), which led to precipitation of the product. The collected crude product was redissolved in dichloromethane and reprecipitated by adding methanol. Trimers **3** and **4** were recrystallized from methanol/dichloromethane. Due to the high steric hindrance, the reaction of dicyanomethyl compound **14** required a slight modification of the aforementioned procedure. Thus, the aldehyde and nitrile were suspended in a mixture of THF (3 mL) and *t*BuOH (1 mL), and the mixture was heated to 40 °C prior to addition of the base.

General Procedure for the Wittig Reaction: As a representative example, 0.60 mmol of 1,4-bis(hexyloxy)benzaldehyde (**17**) and 0.30 mmol of phosphonium salt **15** were suspended in dry ethanol (8 mL) under argon at room temperature. Then, 0.60 mmol of anhydrous potassium *tert*-butoxide was added and the mixture was stirred for 12 h or until the starting aldehyde had been consumed. The reaction was quenched by adding 10% HCl (5 mL), the phases were separated, and the organic phase was washed with water and dried with magnesium sulfate. After evaporation of the solvent, the crude product was purified by column chromatography (silica gel, hexane/dichloromethane) and subsequent crystallization from ethyl acetate to furnish the pure (*E*/*E*) isomer.

Synthesis of 4,8-Bis(hexyloxy)-1,5-naphthalenedicarbaldehyde (13): Hexamethylenetetraamine (0.40 g, 2.80 mmol) was added to a solution of 4,8-bis(bromomethyl)-1,5-bis(hexyloxy)naphthalene (**12**) (0.50 g, 1.00 mmol) in chloroform (10 mL) and the mixture was refluxed for 4 h.[10a] After cooling, the precipitate was collected and dried. A solution of this salt in 50% acetic acid (10 mL) was refluxed for 15 h. Once cooled, the mixture was diluted with water (20 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic extracts were washed with water (2 × 10 mL), dried with magnesium sulfate, and the solvent was evaporated. The crude product was purified by column chromatography (silica gel, hexane/dichloromethane).

(*E*)-1,5-Bis[β-cyano-1′,4′-bis(hexyloxy)naphth-2-ylvinyl]-4,8-bis(hexyloxy)naphthalene (2b): Yield: 37%, colorless crystals, blue fluorescence; m.p. 180−182 °C. − ^{1}H NMR: δ = 8.24 (m, 2 H), 8.01 (m, 2 H), 7.90 (s, 2 H), 7.55 (s, 2 H), 7.47 (m, 4 H), 7.31 (d, J = 7.9 Hz, 2 H), 6.86 (d, J = 8.2 Hz, 2 H), 4.21 (t, J = 6.1 Hz, 4 H), 4.14 (br. s, 2 H), 3.95 (br. s, 2 H), 3.88 (t, J = 6.4 Hz, 4 H),

1.96−1.72 (m, 12 H), 1.60−1.09 (m, 28 H), 1.02−0.80 (m, 8 H), 0.85 (t, J = 7.0 Hz, 6 H), 0.73 (t, J = 7.0 Hz, 6 H), 0.48 (t, J = 7.0 Hz, 6 H). − ^{13}C NMR: δ = 156.4, 151.3, 149.0, 135.3, 130.5, 128.7, 127.8, 126.9, 126.6, 126.0, 125.3, 123.0, 122.7 (2 C), 119.6, 115.4, 106.7, 101.4, 76.7, 69.3, 68.5, 31.6, 30.3, 29.3, 29.1, 26.1, 25.8, 22.7, 22.6, 22.4, 14.1, 14.0, 13.8. − IR (KBr): $\tilde{\nu}$ = 2935 cm^{-1}, 2205, 1589, 1522, 1404, 1232, 1132, 773. − HRMS (EI): calcd. for $C_{72}H_{94}N_2O_6$ 1082.7112; found 1082.7019. − $C_{72}H_{94}N_2O_6$ calcd. C 79.81, H 8.74, N 2.59; found C 79.51, H 9.05, N 2.55. − UV (CH_2Cl_2): λ_{max} = 378 nm, 318, 286. − PL (solid state): λ = 452 nm.

(*E*)-1,5-Bis[α-cyano-2′,5′-bis(hexyloxy)styryl]-4,8-bis(hexyloxy)-naphthalene (4a): Yield: 26%, yellow needles, weak greenish-yellow fluorescence; m.p. 84−86 °C. − ^{1}H NMR: δ = 8.51 (s, 2 H), 7.61 (dd, J = 8.2 Hz, 0.9 Hz, 2 H), 7.02 (d, J = 2.1 Hz, 2 H), 6.83 (d, J = 8.2 Hz, 2 H), 6.81−6.77 (m, 4 H), 3.95 (t, J = 6.4 Hz, 4 H), 3.91 (t, J = 6.7 Hz, 4 H), 3.87 (t, J = 6.4 Hz, 4 H), 1.80−0.80 (m, 48 H), 0.83 (t, J = 7.0 Hz, 6 H), 0.74 (t, J = 7.0 Hz, 6 H), 0.72 (t, J = 7.0 Hz, 6 H). − ^{13}C NMR: δ = 157.3, 153.2, 152.8, 150.8, 129.0, 125.6, 124.7, 118.7, 115.9, 115.2, 113.7, 106.9, 106.6, 77.2, 69.5, 68.7, 31.7, 31.5, 29.4, 29.3, 29.1, 26.1, 25.9, 25.8, 22.6, 22.5, 14.1. − IR (KBr): $\tilde{\nu}$ = 2934 cm^{-1}, 2216, 1497, 1285, 1231, 1043, 804. − HRMS (EI): calcd. for $C_{64}H_{90}N_2O_6$ 982.6799; found 982.6764. − $C_{64}H_{90}N_2O_6$ calcd. C 77.62, H 9.45, N 2.92; found C 76.85, H 9.12, N 2.48. − UV (CH_2Cl_2): λ_{max} = 371 nm, 250 (sh). − PL (solid state): λ = 485 nm.

(*E*)-1,5-Bis[α-cyano-1′,4′-bis(hexyloxy)naphth-2-ylvinyl]-4,8-bis-(hexyloxy)naphthalene (4b): Yield: 45%, yellow plates, weak yellow fluorescence; m.p. 148−150 °C. − ^{1}H NMR: δ = 8.21 (dm, J = 7.0 Hz, 2 H), 8.05 (dm, J = 7.0 Hz, 2 H), 7.74 (dd, J = 8.2 Hz, 0.9 Hz, 2 H), 7.48 (dq, J = 7.0 Hz, 1.5 Hz, 2 H), 7.47 (dq, J = 7.0 Hz, 1.5 Hz, 2 H), 6.83 (s, 2 H), 6.82 (d, J = 8.2 Hz, 2 H), 4.08 (t, J = 6.1 Hz, 4 H), 3.94 (t, J = 6.1 Hz, 4 H), 3.89 (t, J = 6.7 Hz, 4 H), 1.87 (q, J = 6.6 Hz, 4 H), 1.76 (q, J = 7.0 Hz, 4 H), 1.60−0.60 (m, 40 H), 0.85 (t, J = 6.7 Hz, 6 H), 0.72 (t, J = 7.0 Hz, 6 H), 0.45 (t, J = 6.7 Hz, 6 H). − ^{13}C NMR: δ = 157.5, 153.7, 151.5, 146.5, 129.4, 129.0, 126.9, 126.8, 126.0, 125.7, 124.7, 123.1, 122.5, 122.4, 119.0, 106.9, 106.1, 104.2, 74.9, 69.4, 68.5, 31.8, 31.7, 31.4, 30.3, 29.3, 29.1, 26.1, 26.0, 25.9, 22.7, 22.6, 22.3, 14.0, 13.8. − IR (KBr): $\tilde{\nu}$ = 2930 cm^{-1}, 2216, 1520, 1458, 1404, 1229, 1097, 798. − HRMS (EI): calcd. for $C_{72}H_{94}N_2O_6$ 1082.7112; found 1082.7040. − $C_{72}H_{94}N_2O_6$ calcd. C 79.81, H 8.74, N 2.59; found C 78.98, H 8.45, N 2.48. − UV (CH_2Cl_2): λ_{max} = 380 nm, 250. − PL (solid state): λ = 516 nm.

(*E*)-2,6-Bis[2′,5′-bis(hexyloxy)styryl]-1,5-bis(hexyloxy)naphthalene (5a): Yield: 66%, yellow powder, bright yellow fluorescence; m.p. 98−99 °C. − ^{1}H NMR: δ = 7.82 (d, J = 8.8 Hz, 2 H), 7.73 (d, J = 8.8 Hz, 2 H), 7.52 (s, 2 H), 7.17 (d, J = 2.7 Hz, 2 H), 6.78 (d, J = 9.1 Hz, 2 H), 6.72 (dd, J = 8.8 Hz, 2.7 Hz, 2 H), 3.94 (t, J = 6.4 Hz, 4 H), 3.92 (t, J = 6.4 Hz, 4 H), 3.90 (t, J = 6.4 Hz, 4 H), 1.95−1.67 (m, 12 H), 1.63−1.22 (m, 36 H), 0.85 (t, J = 7.0 Hz, 18 H). − ^{13}C NMR: δ = 153.4, 153.1, 151.0, 129.5, 128.0, 126.8, 124.0, 123.8, 123.3, 118.6, 114.8, 114.0, 111.9, 77.7, 69.7, 68.7, 31.9, 31.6, 30.5, 29.5, 29.4, 26.0, 25.9, 25.8, 22.7, 14.1. − IR (KBr): $\tilde{\nu}$ = 2928 cm^{-1}, 1501, 1265, 1217, 1018, 800. − HRMS (EI): calcd. for $C_{62}H_{92}O_6$ 932.6894; found 932.6965. − $C_{62}H_{92}O_6$ calcd. C 79.78, H 9.93; found C 79.98, H 7.87. − UV (CH_2Cl_2): λ_{max} = 388 nm, 298, 290. − PL (solid state): λ = 545 nm.

(*E*)-2,6-Bis[1′,4′-bis(hexyloxy)naphth-2-ylvinyl]-1,5-bis(hexyloxy)-naphthalene (5b): Yield: 65%, yellow crystals, green fluorescence; m.p. 142−143 °C. − ^{1}H NMR: δ = 8.19 (dm, J = 7.6 Hz, 2 H), 8.02 (dm, J = 7.3 Hz, 2 H), 7.88 (d, J = 8.9 Hz, 2 H), 7.82 (d, J = 8.9 Hz, 2 H), 7.73 (d, J = 16.6 Hz, 2 H), 7.60 (d, J = 16.6 Hz, 2 H), 7.50−7.35 (m, 4 H), 7.07 (s, 2 H), 4.22 (t, J = 6.3 Hz, 4 H), 4.08 (t, J = 6.4 Hz, 4 H), 4.02 (t, J = 6.5 Hz, 4 H), 2.00−1.84 (m, 12 H), 1.70−1.25 (m, 36 H), 0.89 (t, J = 6.1 Hz, 6 H), 0.88 (t, J = 6.4 Hz, 6 H), 0.86 (t, J = 7.0 Hz, 6 H). − ^{13}C NMR: δ = 153.2, 151.5, 147.0, 129.5, 129.3, 126.9 (2 C), 126.7, 125.9, 125.5, 124.3, 123.6, 123.1, 122.5, 122.3, 118.8, 100.8, 75.9, 75.8, 68.3, 31.9, 31.7, 30.6, 29.4, 26.2, 26.1, 22.8, 22.7, 14.2, 14.1. − IR (KBr): $\tilde{\nu}$ = 2928 cm^{-1}, 1595, 1366, 1281, 1221, 1085, 770. − HRMS (EI): calcd. for $C_{70}H_{96}O_6$ 1032.7207; found 1032.7176. − $C_{70}H_{96}O_6$ calcd. C 81.35, H 9.36; found C 81.20, H 9.70. − UV (CH_2Cl_2): λ_{max} = 419 nm, 399, 296, 261. − PL (solid state): λ = 509 nm, 477, 450.

(*E*)-1,5-Bis[2′,5′-bis(hexyloxy)styryl]-4,8-bis(hexyloxy)naphthalene (6a): Yield: 56%, pale-yellow powder, blue-green fluorescence; m.p. 69−70 °C. − ^{1}H NMR: δ = 8.13 (d, J = 16.1 Hz, 2 H), 7.38 (d, J = 7.9 Hz, 2 H), 7.13 (d, J = 2.7 Hz, 2 H), 6.91 (d, J = 16.1 Hz, 2 H), 6.79 (d, J = 7.9 Hz, 2 H), 6.74 (d, J = 8.9 Hz, 2 H), 6.64 (dd, J = 8.9 Hz, 2.7 Hz, 2 H), 3.96 (t, J = 6.4 Hz, 4 H), 3.88 (t, J = 6.4 Hz, 4 H), 3.86 (t, J = 6.4 Hz, 4 H), 1.80−1.63 (m, 12 H), 1.45−1.15 (m, 36 H), 0.83 (t, J = 7.0 Hz, 6 H), 0.79 (t, J = 7.0 Hz, 6 H), 0.69 (t, J = 6.7 Hz, 6 H). − ^{13}C NMR: δ = 156.6, 153.5, 150.7, 134.4, 129.6, 128.9, 126.7, 125.8, 120.6, 114.2, 113.5, 111.9, 107.5, 69.8, 69.4, 68.6, 31.7, 31.6, 29.6, 29.5, 26.1, 25.9, 25.8, 22.6, 22.5, 14.0. − IR (KBr): $\tilde{\nu}$ = 2932 cm^{-1}, 1495, 1273, 1209, 1043, 793. − HRMS (EI): calcd. for $C_{62}H_{92}O_6$ 932.6894; found 932.6965. − $C_{62}H_{92}O_6$ calcd. C 79.78, H 9.93; found C 79.71, H 10.35. − UV (CH_2Cl_2): λ_{max} = 372 nm, 270 (sh). − PL (solid state): λ = 468 nm.

(*E*)-1,5-Bis[1′,4′-bis(hexyloxy)naphth-2-ylvinyl]-4,8-bis(hexyloxy)-naphthalene (6b): Yield: 80%, yellow powder, greenish-yellow fluorescence; m.p. 133−134 °C. − ^{1}H NMR: δ = 8.22 (d, J = 16.5 Hz, 2 H), 8.18 (d, J = 8.8 Hz, 2 H), 8.00 (d, J = 7.8 Hz, 2 H), 7.50−7.32 (m, 8 H), 7.10 (d, J = 16.5 Hz, 2 H), 7.03 (s, 2 H), 6.84 (d, J = 8.2 Hz, 2 H), 4.11 (t, J = 6.3 Hz, 4 H), 3.99 (t, J = 6.4 Hz, 4 H), 3.89 (t, J = 6.5 Hz, 4 H), 1.96−1.68 (m, 12 H), 1.64−1.43 (m, 8 H), 1.42−1.18 (m, 20 H), 0.98−0.75 (m, 8 H), 0.86 (t, J = 6.7 Hz, 6 H), 0.79 (t, J = 7.0 Hz, 6 H), 0.47 (t, J = 7.0 Hz, 6 H). − ^{13}C NMR: δ = 156.6, 151.3, 146.1, 134.4, 129.6, 129.4, 126.8, 126.6, 126.4, 126.3, 125.8, 124.9, 122.3, 122.1, 121.1, 107.7, 107.4, 75.3, 69.5, 68.3, 31.8, 31.7, 31.6, 30.4, 29.7, 29.4, 26.1, 26.0, 25.9, 22.7, 22.4, 14.0, 13.8. − IR (KBr): $\tilde{\nu}$ = 2922 cm^{-1}, 1522, 1373, 1280, 1210, 1088, 1040, 768. − HRMS (EI): calcd. for $C_{70}H_{96}O_6$ 1032.7207; found 1032.7157. − $C_{70}H_{96}O_6$ calcd. C 81.35, H 9.36; found C 81.19, H 9.47. − UV (CH_2Cl_2): λ_{max} = 381 nm, 272. − PL (solid state): λ = 475 nm.

Polymer 24: Yield: 45%, yellow powder, bright yellow fluorescence. − ^{1}H NMR: δ = 8.63−7.33 (m, 8 H), 6.98−6.85 (m, 2 H), 4.23−4.02 (m, 8 H), 1.92−1.89 (m, 8 H), 1.53−1.30 (m, 16 H), 1.01−0.93 (m, 8 H), 0.84−0.58 (m, 12 H). − ^{13}C NMR (DEPT-135): δ = 189.4, 189.2, 134.3, 133.9, 130.3, 130.0, 129.4, 124.5, 124.1, 123.0, 119.3, 118.9, 106.5, 106.3, 79.2, 69.1, 31.4, 31.3, 30.1, 29.4, 28.7, 26.4, 25.8, 25.6, 25.5, 22.3, 22.1, 13.7, 13.6. − IR (KBr): $\tilde{\nu}$ = 2953 cm^{-1}, 2930, 2214, 1676, 1520, 1321, 1049, 825. − GPC: M_w = 3114; M_n = 2334. − UV (CH_2Cl_2): λ_{max} = 396 nm, 330, 280. − PL (solid state): λ = 499 nm.

Polymer 25: Yield: 69%, yellow powder, weak yellow fluorescence. − ^{1}H NMR: δ = 8.82−7.53 (m, 8 H), 7.09−6.90 (m, 2 H), 4.24−3.94 (m, 8 H), 1.91 (br. s, 8 H), 1.49−1.16 (m, 16 H), 0.94−0.80 (m, 8 H), 0.65−0.52 (m, 12 H). − ^{13}C NMR (DEPT-135): δ = 193.9, 153.7, 153.2, 129.5, 129.1, 126.9, 119.7, 119.1, 118.5, 107.4, 107.0, 106.7, 75.1, 69.5, 69.4, 31.6, 31.4, 31.3, 30.2,

29.0, 25.9, 25.8, 22.5, 22.3, 14.0. – IR (KBr): $\tilde{\nu}$ = 2953 cm^{-1}, 2928, 2212, 1684, 1520, 1377, 1232, 1049, 825. – GPC: M_w = 3629; M_n = 2522. – UV (CH_2Cl_2): λ_{max} = 386 nm, 251. – PL (solid state): λ = 506 nm.

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