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The synthesis of π-electron molecular rods with a thiophene or thieno[3,2-*b*]thiophene core unit and sulfur alligator clips

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ABSTRACT

A series of short oligo(*p*-phenylene-ethynylene)- and oligo(*p*-phenylenevinylene)-type molecular rods with an electronically rich thiophene or thieno[3,2-*b*]thiophene core unit and sulfur anchoring groups (AcS–, *t*-BuS–) at the termini have been synthesised using Sonogashira coupling or Horner–Wadsworth–Emmons (*E*)-olefination methodology. The collection of linear/bent, conjugated/cross-conjugated systems has been characterised by UV–vis and fluorescence spectroscopy, optical/calculated HOMO–LUMO gaps and calculated excitation energies.

π-Electron molecules, oligomers and polymers are the subject of intense research since they can mediate charge transport along the π-conjugated pathway.[1] This key attribute is characterised by the conductance of a bulk material or a single molecule.[2] In particular, the latter approach is notably challenging and rewarding, because it provides ‘neat’ conductance of a single molecular wire or, better, a triad given by the metallic electrode–molecule–metallic electrode system[3] (provided that the intermolecular interaction can be neglected).[4] Nowadays, there are two experimental techniques most frequently used, which allow for quantifying single-molecule conductance: mechanically controllable break junction (MCBJ)[5] and scanning tunnelling microscopy-based break junction (STMBJ).[6] In both cases, functional rod-like molecules with anchoring groups at both ends are needed to provide a strong covalent attachment to the metallic contacts (usually a gold source and drain electrodes). Accordingly, the thiol moiety and its analogues are routinely used,[7] although they suffer from some drawbacks.[8] Various molecular wires have been investigated ranging from alkane to carotenoid dithiols,[2c] but short oligo(*p*-phenylene-ethynylene) (OPE) and oligo(*p*-phenylenevinylidene) (OPV) rod molecules and their congeners equipped with acetylsulfanyl anchoring groups at the termini are popular in single-molecule conductivity measurements owing to, inter alia, their variable structure, reasonable synthesis and stability. Although the incorporation of a heterocyclic unit such as thiophene into the OPE- and OPV-type molecular wires might result in interestingly altered electronic and geometrical properties, their single-molecule conductance has not been studied systematically. There are only a few examples of molecular wires comprising a single thiophene unit,[9] in contrast to the systems represented by thiophene-based switchable photochromic molecules,[10] oligo(2,5-thienylene-ethynylene)[11] and oligothiophenes,[12] whose derivatives exhibit low band gaps and high charge mobility.[13]

Herein, we report on the synthesis of the short OPE- and OPV-type rods **1a**–**d** and **2a**–**d**, in which the central 1,4-phenylene unit is displaced by electronically rich 2,5-, 2,4- and 3,4-thienylenes as well as thieno[3,2-*b*]thiophene-2,5-diyl counterparts (Fig. 1). For the sake of comparison of the single-molecule conductance of these compounds with that of the parent OPE and OPV structures, the molecular rods **1e** and **2e**, have also been synthesised based on published procedures.[14,15] All compounds **1a**–**e** and **2a**–**e** are equipped with sulfur moieties to serve as alligator clips during the measurement of the single-molecule conductance by the MCBJ technique.[16] The results of these physical experiments will be published elsewhere.

The preparation of thiophene- and thieno[3,2-*b*]thiophene-derived OPE molecular rods **1a**–**c** started from commercially available 2,5-, 2,4- and 3,4-dibromothiophenes **3a**–**c** (Scheme 1). The known 2,5-diiodothieno[3,2-*b*]thiophene (**3d**),[17] which was em-

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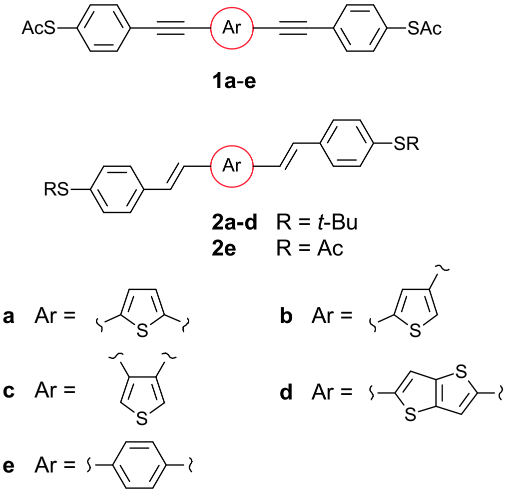
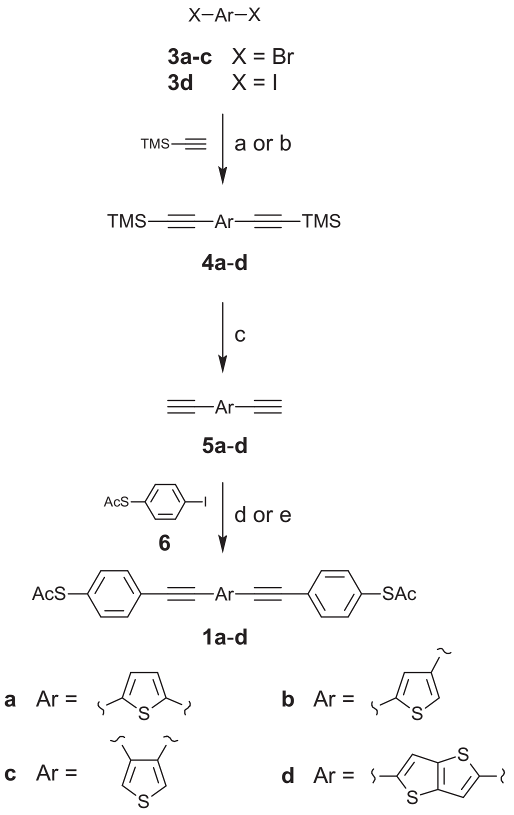


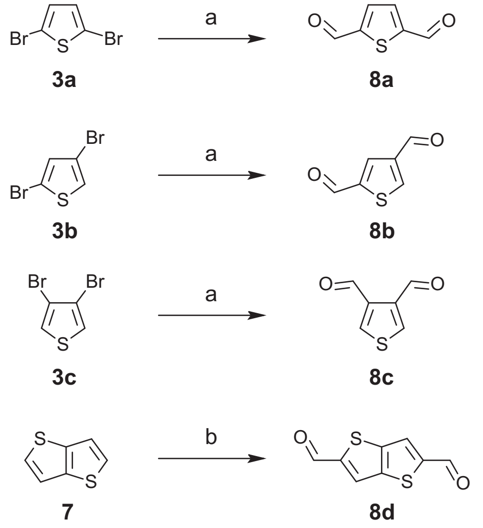
Figure 1. π-Electron molecular rods **1a**–**e** and **2a**–**e** with a thiophene or thieno[3,2-*b*]thiophene core unit and sulfur anchoring groups.



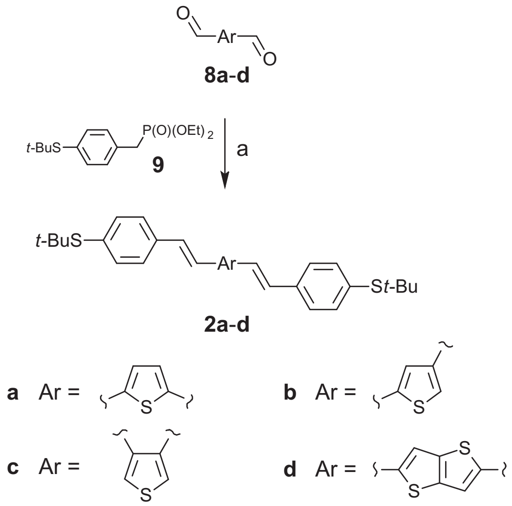
Scheme 1. The synthesis of the OPE-type molecular rods **1a**–**d**. Reagents and conditions: (a) TMS–C≡CH (3.0 equiv), $Pd(PPh_3)_2Cl_2$ (10 mol %), CuI (10 mol %), *i*-Pr_2NH (3.2 equiv), THF, room temperature, 2 d, 90% for **4a**; (b) TMS–C≡CH (10.0 equiv), $Pd(PPh_3)_4$ (10 mol %), CuI (10 mol %), Et_3N, 75 °C, 3 d, 87% for **4b**, 78% for **4c**, 51% for **4d**; (c) *n*-Bu_4NF (16 equiv), THF, room temperature, 1–3 h, 98% for **5a**, 98% for **5b**, 99% for **5c**, 81% for **5d**; (d) **6** (2.0 equiv), $Pd(PPh_3)_2Cl_2$ (10 mol %), CuI (10 mol %), *i*-Pr_2NH (1.5 equiv), THF, room temperature, 3 d, 40% for **1a**, 27% for **1b**, 21% for **1c**; (e) **6** (2.0 equiv), $Pd(PPh_3)_4$ (10 mol %), CuI (10 mol %), Et_3N, 75 °C, 40 h, 32% for **1d**.

ployed in the synthesis of **1d**, was obtained by iodination of commercially available thieno[3,2-*b*]thiophene (**7**) using I_2 in the presence of an Ag(I) salt.

The preparation of **1a**–**d** followed a general synthetic scheme, which relied on Sonogashira coupling methodology (Scheme 1). Performing the double cross-couplings of trimethylsilyl acetylene with **3a**–**d** under $Pd(PPh_3)_2Cl_2$/CuI catalysis in the presence of diisopropylamine in THF at room temperature, or $Pd(PPh_3)_4$/CuI catalysis in triethylamine at 75 °C, the silylated bis(ethynyl) derivatives **4a**,[18] **4b**,[19] **4c**[20] and **4d**[21] were obtained. We adjusted the reaction conditions (such as relatively long reaction times at ambient temperature) to obtain high yields and clean products. On reaction with tetrabutylammonium fluoride, the double desilylation proceeded smoothly to afford bis(ethynyl) heteroaromatics **5a**,[18] **5b**,[22] **5c**[22] and **5d**[21] in good yields. Thus, we performed the final double Sonogashira coupling with *S*-(4-iodophenyl)ethanethioate (**6**)[23] under $Pd(PPh_3)_2Cl_2$/CuI catalysis in the presence of diisopropylamine in THF at room temperature, or $Pd(PPh_3)_4$/CuI catalysis in triethylamine at 75 °C to afford the thiophene- and thieno[3,2-*b*]thiophene-derived OPE molecular rods **1a**–**d** in moderate yields but in good purities, which were further increased by chromatographic separation. Using the same methodology, we prepared the parent compound **1e**.[14] It is worth noting that the alternative



Scheme 2. Formylation of dibromothiophenes **3a**–**c** and thieno[3,2-*b*]thiophene **7**. Reagents and conditions: (a) *n*-BuLi (1.1 equiv), THF, −78 °C to 0 °C, 30 min, then DMF (1.2 equiv), room temperature, 2 h, then *t*-BuLi (1.3 equiv), THF, −78 °C, 5 min, then DMF (excess), −78 °C to room temperature, overnight, 42% for **8a**, 22% for **8b**, 29% for **8c**; (b) *n*-BuLi (2.2 equiv), THF, −78 °C to 0 °C, 2 h then DMF (excess), −78 °C to room temperature, overnight, 17%.



Scheme 3. The synthesis of OPV-type molecular rods **2a**–**d**. Reagents and conditions: (a) diethyl [4-(*tert*-butylsulfanyl)-benzyl]phosphonate (**9**) (2.0 equiv), *t*-BuOK (3.2–3.8 equiv), THF, room temperature, 12 h, 48% for **4a**, 57% for **4b**, 38% for **4c**, 78% for **4d**.

Table 1
The optical properties of the molecular rods **1a**–**e** and **2a**–**e**

Compound	$\lambda_{max,abs}$[a] (nm)	Calculated $\lambda_{max,abs}$[b] (nm)	$\lambda_{max,em}$[c] (nm) (excitation λ, nm)
1a	356	370	416 (390)
1b	296	311	weak fluorescence
1c	286	300	weak fluorescence
1d	375	387	430 (405)
1e	329	339	386 (360)
2a	400	396	475 (435)
2b	318	330	weak fluorescence
2c	314	305	weak fluorescence
2d	409	413	483 (430)
2e	365	360	429 (400)

[a] The maximum absorption wavelength $\lambda_{max,abs}$ corresponds to the absorption maximum of the longest wavelength band in the experimental UV–vis spectrum (at concentrations of 0.10–26.9 × 10^{-5} mol/L in acetonitrile).

[b] The maximum absorption wavelength $\lambda_{max,abs}$ was calculated by TD-DFT (B2PLYP/cc-pVDZ) on the structures optimised by DFT (B3LYP/ccpVDZ).

[c] The maximum emission wavelength $\lambda_{max,em}$ corresponds to the emission maximum of the longest wavelength band in the experimental fluorescence spectrum (at concentrations of 8.4–10.6 × 10^{-5} mol/L in acetonitrile).

approach to **1a**–**e** through Sonogashira coupling between **3a**–**e** and *S*-(4-ethynylphenyl)ethanethioate[11] under Pd^0/Cu^I catalysis led generally to complex mixtures, from which the model compounds **1a**–**e** were difficult to isolate.

With the aim of synthesising the analogous thiophene- and thieno[3,2-*b*]thiophene-derived OPV molecular rods **2a**–**d** through the Horner–Wadsworth–Emmons (*E*)-olefination methodology, we prepared the corresponding dialdehydes **8a**,[24] **8b**,[25] **8c**[26] and **8d**[27] by modification of the literature procedures (Scheme 2). The double formylation of dibromothiophenes **3a**–**c** was carried out as a two-step process; the first bromine-to-lithium exchange at −78 °C was performed using *n*-butyllithium and, after adding an equivalent amount of *N*,*N*-dimethylformamide, the second lithiation proceeded in the presence of *t*-butyllithium at −78 °C, followed by the addition of an excess of DMF. The dialdehyde **8d** was prepared by the double metallation of **7** with 2 equiv of *n*-butyllithium at 0 °C, after which the dilithium intermediate was reacted with an excess of DMF.

The syntheses of **2a**–**d** were completed by the double Horner–Wadsworth–Emmons reaction of diethyl [4-(*tert*-butylsulfanyl)benzyl]phosphonate (**9**)[15d] with dialdehydes **8a**–**d** to give the corresponding olefins in moderate or good yield (Scheme 3). Using the same methodology, we also prepared the parent compound **2e**,[15] which was obtained from its *t*-butylsulfanyl analogue by treatment with acetyl chloride and boron tribromide.[15d] Although the protecting group transformation proceeded well in this case, the same operation with the thiophene- and thieno[3,2-*b*]thiophene-derived OPV molecular rods **2a**–**d** failed since a complex mixture was generally formed.

The thiophene-based OPE and OPV molecular rods **1a**–**c** and **2a**–**c** are bent at angles ranging from 68° to 150°. The rods **1d**, **e** and **2d**, **e** are linear, as a result of which they exhibit the largest distances, in this series, between the sulfur atoms of the anchoring groups (19.8–22.1 Å).

In the UV–vis spectra of **1a**–**e** and **2a**–**e**, a red shift of the absorption maxima of the longest wavelength bands can be seen uniformly when going from the corresponding OPE to OPV derivatives (22–44 nm, Table 1). Interestingly, the λ_{max} values of 2,4- and 3,4-thiophene derivatives **1b**, **c** (296 vs 286 nm) are comparable, which was also observed with **2b**, **c** (318 vs 314 nm), and the absorption maxima are shifted to shorter wavelengths. This fact can be ascribed to weaker conjugation along the backbone of 3,4-thiophene derivatives **1c** and **2c** and cross-conjugated **1b** and **2b**. Such an effect can be demonstrated by comparison of the electron probability distribution within the frontier orbitals of linear-conjugated **1a** and cross-conjugated **1b**, as calculated by density functional theory (DFT) methods (Fig. 2). The weak fluorescence of **1b**, **c** and **2b**, **c** (Table 1) was also in accord with the reduced conjugation in these rod-shaped compounds. In fact, the fluorescence emission efficiency corresponds to the π-conjugation length in the excited singlet state,[28] which might be correlated with the π-conjugation length in the ground state. The absorption or fluorescence spectra allowed calculation of the optical HOMO–LUMO gap of **1a**–**e** and **2a**–**e** (Table 2). Among both the OPE and OPV series, the thieno[3,2-*b*]thiophene derivatives **1d** and **2d** exhibited the largest red shifts and, accordingly, the smallest optical HOMO–LUMO band gaps of 3.0 or 2.7 eV, respectively. The parent OPE/OPV rods **1e** and **2e** with 1,4-phenylene core units absorb at shorter wavelengths than their electron rich 2,5-thiophene counterparts **1a** and **2a**, which results in a narrowing of the gap by about 0.2 eV in favour of the thiophene derivatives. The magnitudes of the HOMO–LUMO gaps of **1a**–**e** and **2a**–**e** calculated by DFT methods generally over-

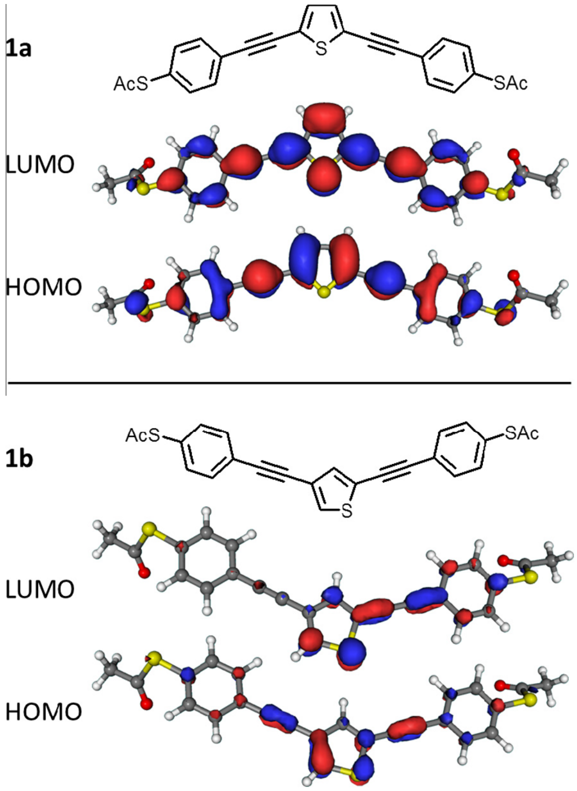


Figure 2. The electron probability distributions within the frontier orbitals of linear-conjugated **1a** and cross-conjugated **1b** were calculated by DFT (B2PLYP/ccpVDZ) on the structures optimised by DFT (B3LYP/ccpVDZ).

Table 2
The optical/calculated HOMO–LUMO gaps and calculated excitation energies of the molecular rods **1a**–**e** and **2a**–**2e**

Compound	Optical HOMO–LUMO gap[a] (eV)	Calculated HOMO–LUMO gap[b] (eV)	Calculated excitation energy[c] (eV)
1a	3.1 (3.0)	5.2	3.4
1b	3.5	5.9	4.0
1c	3.7	6.4	4.1
1d	3.0 (2.9)	5.0	3.2
1e	3.3 (3.2)	5.6	3.7
2a	2.8 (2.6)	4.9	3.1
2b	3.1	5.5	3.8
2c	3.3	6.2	4.1
2d	2.7 (2.6)	4.7	3.0
2e	3.0 (2.9)	5.2	3.4

[a] The optical HOMO–LUMO gap was calculated from the onset of the longest wavelength band in the experimental UV–vis spectra or from the wavelength of the maximum emission band in the experimental fluorescence spectra (in parentheses).

[b] The HOMO–LUMO gap in the ground state was calculated by DFT (B2PLYP/ccpVDZ) on the structures optimised by DFT (B3LYP/ccpVDZ).

[c] The energy needed for the electron excitation to the first singlet state was calculated by TD-DFT (B2PLYP/cc-pVDZ) on the structures optimised by DFT (B3LYP/ccpVDZ).

estimate the actual excitation energies by 67–88% (Table 2) and, therefore, the energies needed for the electron excitation to the first singlet state calculated by the TD-DFT method better approximate the corresponding optical HOMO–LUMO gaps, being overestimated now by only 7–24%.

In conclusion, we have prepared a series of oligo(*p*-phenyleneethynylene)- and oligo(*p*-phenylenevinylene)-type molecular rods with thiophene or thieno[3,2-*b*]thiophene core units and end-capped with sulfur anchoring groups (AcS−, *t*-BuS−). These π-electron systems have been characterised by UV–vis/fluorescence spectroscopy and by DFT/TD-DFT calculations. The possibly reduced conjugation or cross-conjugation in these molecular rods has led to hypsochromic shifts of the corresponding $\lambda_{\max}$ in the UV–vis spectra (about 60−86 nm) as well as to weak or no fluorescence, in contrast to the properties of linearly-conjugated molecular rods. Good correlations between the experimental optical HOMO–LUMO gaps and calculated excitation energies have been found. Experiments to estimate their molecular conductivities by using the mechanically controllable break-junction method are currently underway.

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Supplementary data

Supplementary data (the experimental procedures and NMR spectra of the substrates and products) associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.tetlet.2013.03.084.

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