Monatsh Chem 139, 1011–1017 (2008)
DOI 10.1007/s00706-008-0897-1
Printed in The Netherlands

Monatshefte für Chemie
Chemical Monthly

Thiophene-based glass-forming hole-transporting hydrazones

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Received 9 January 2008; Accepted 16 January 2008; Published online 3 March 2008

Abstract The synthesis, optical, thermal, and photoelectrical properties of new thiophene-based hydrazones are reported. The ionization potentials of the films of thiophene-based hydrazones, measured by the electron photoemission technique, range from 4.99 to 5.58 eV. Hole-drift mobilities in the solid solutions in bisphenol-Z polycarbonate (*PC-Z*) of the synthesized hydrazones were studied by time of flight technique. Room temperature charge mobilities in the solid solution of 5,2″-diformyl-2,2′:5′,5″-terthiophene di(*N*,*N*-diphenylhydrazone) in *PC-Z* exceeded 10^{-5} cm^2/Vs at high applied electric fields.

Keywords Heterocycles; Thiophene; Hydrazones; Ionization potential; Charge transport.

Introduction

Organic charge-transporting materials are used in optoelectronic and electronic devices like electrophotographic photoreceptors, organic light-emitting diodes, photovoltaic cells, and field effect transistors [1, 2]. Hydrazones, due to their simple synthesis and high charge carrier mobilities, play an important role among organic hole-transporting materials, especially those used in electrophotography [3]. Thiophene-based hydrazones until now are poorly studied and represent a relatively new family of organic hole-transporting materials [4]. In this presentation we report on the design, synthesis, and characterization of the new glass-forming thiophene-based hydrazones.

Results and discussion

New 3,4-ethylendioxythiophene-based dihydrazones **1a** and **1b** were obtained by the reaction of 3,4-ethylenedioxythiophene-2,5-dicarbaldehyde with *N*-methyl-*N*-phenylhydrazine and *N*,*N*-diphenylhydrazine hydrochloride. 3,4-Ethylenedioxythiophene-based hydrazone bearing a carbazole moiety (**2**) was obtained in the reaction of 2-(*N*-phenylhydrazonemethyl)-3,4-ethylenedioxythiophene with 9-(6-bromo-*n*-hexyl)carbazole (Scheme 1). A long alkylene bridge was introduced in order to increase the solubility of the resulting hydrazone.

α-Terthiophene-based hydrazones **5a**, **5b**, and **8** were synthesized by the route shown in Scheme 2 involving the preparation of the corresponding formyl derivatives followed by their condensation with hydrazines. The synthesis of carbazolyl-containing

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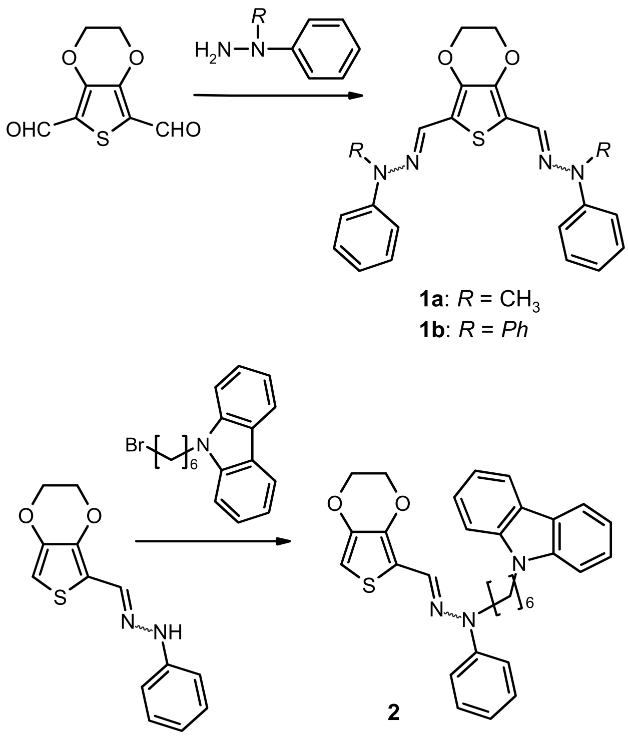


Scheme 1

monohydrazone involved the additional step of alkylation of 2-(*N*-phenylhydrazonemethyl)-3,4-ethylenedioxythiophene with 9-(6-bromo-*n*-hexyl)carbazole.

All the obtained hydrazone derivatives **1a**, **1b**, **2**, **5a**, **5b**, and **8** were purified by column chromatography and characterized by IR, ^{1}H NMR, and mass spectrometry as described in Experimental section. The hydrazones were most likely mixtures of *E*- and *Z*-isomers which was irrelevant with regard to the properties investigated.

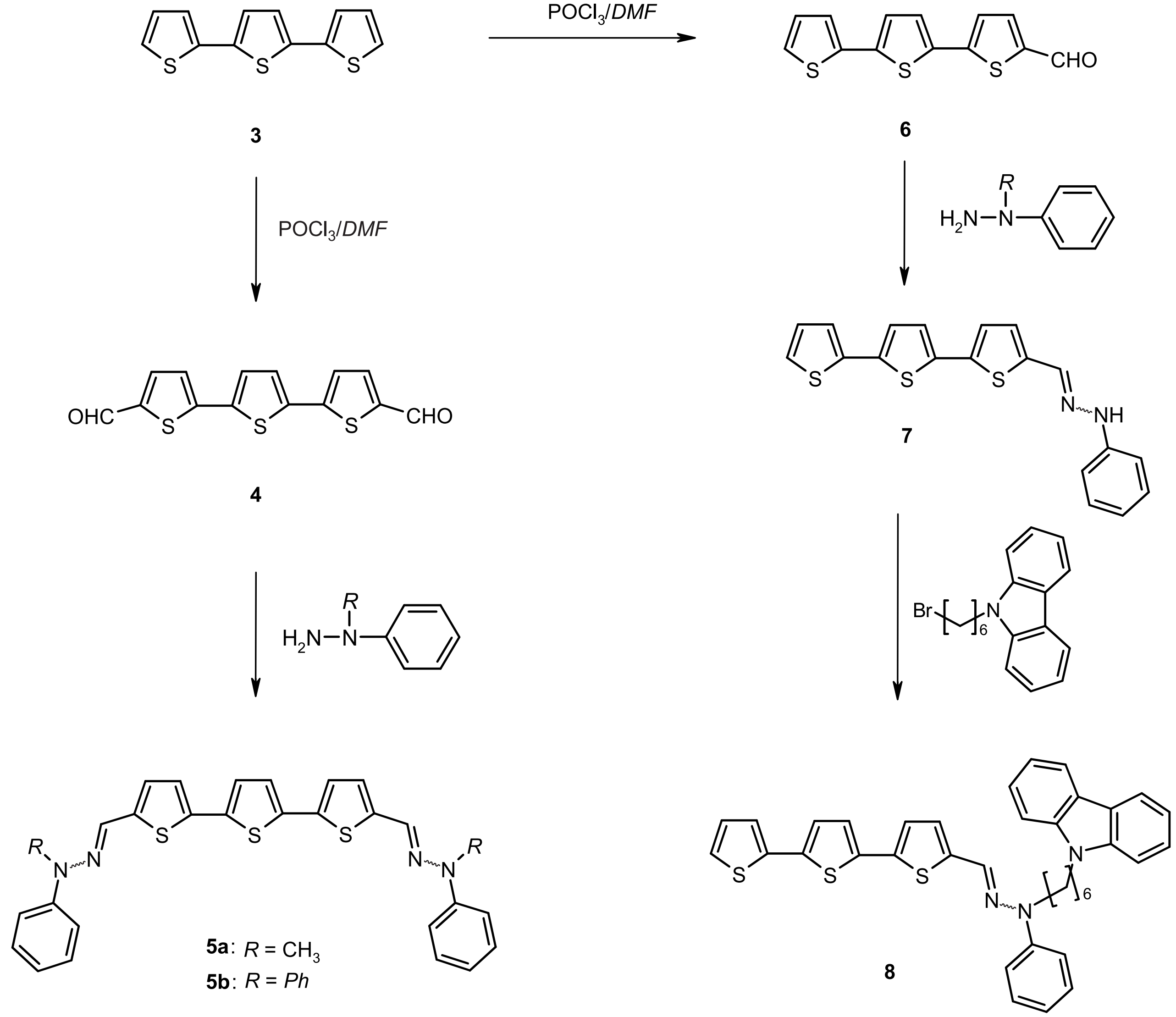
All the synthesized compounds, except **1a** and **1b**, are well soluble in common organic solvents like chloroform, dichloromethane, and tetrahydrofurane. The solubility of compounds **1a** and **1b** in chloroform and tetrahydrofurane is less than $30\,\mathrm{mg/cm^3}$.

Figure 1 shows UV/VIS absorption spectra of dilute solutions of the synthesized hydrazones. α-Terthiophene-based hydrazones **5a** and **5b** absorb radiation in the range of 225–540 nm. 3,4-Ethylendioxythiophene-based hydrazones **1a** and **1b** absorb radiation in the range of 260–490 nm. All the synthesized dihydrazones (**1a** and **1b**, and **5a** and **5b**) exhibit very strong K bands, attributed to $\pi \rightarrow \pi^*$ transitions. The lowest energy absorption bands of α-terthiophene-based dihydrazones **5a** and **5b** are bathochromically shifted by *ca.* 50 nm with respect to the corresponding bands of 3,4-ethylendioxythiophene-based dihydrazones **1a** and **1b**. This observation can be explained by the extended conjugation of electrons in the terthiophene moiety. Indeed, the spectrum of α-terthiophene exhibits a considerable bathochromic shift with respect of the spectrum of 3,4-ethylenedioxythiophene. The UV spectra of monohydrazones show strong hypsochromic effect relative to the spectra of the corresponding dihydrazones. It indicates that the conjugation is extended along the two hydrazone groups in the difunctional structure. The spectra of hydrazones bearing different substituents at hydrazine moiety exhibit almost identical spectra, only the spectra of compounds **1b** and **5b** show a small hyperchromic shift due to the phenyl substituents in comparison with the spectra of **1a** and **5a** bearing methyl substituents. Compounds **2** and **8** bearing carbazole moieties exhibit absorption bands in the shorter range of wavelengths, which is typical absorption for carbazole chromophore.

Fluorescence spectra of dilute *THF* solutions of hydrazones synthesized are shown in Fig. 2. The emission maxima of α-terthiophene-based hydrazones **5a** and **5b** are observed at 538 and 542 nm. They are shifted bathochromically by 24 and 57 nm with respect to the emission maxima of hydrazones **1a** and **1b**. Fluorescence maxima of phenyl substituted dihydrazones **1b** and **5b** are slightly red shifted relative to maxima of the corresponding methyl substituted dihydrazones **1a** and **5a**. The emission maxima of monohydrazones **2** and **8** are observed at 398 and 504 nm. They exhibit considerable hypsochromic effects with respect of the spectra of dihydrazones.

The thermal properties of the newly synthesized compounds were investigated by TG and DSC. It was earlier established that the hydrazone moiety is the critical unit with respect of their thermal stability [5]. Therefore the initial weight loss temperatures of different hydrazones are rather close. Usually it is below 300°C. 3,4-Ethylenedioxythiophene- and α-terthiophene-based hydrazones do not make any exception. Their initial weight loss temperature is around 270°C.

All the newly synthesized hydrazones, except **5a**, can be transformed into the amorphous state. Hydrazones **1a**, **5b**, and **8** were isolated as crystalline compounds after their synthesis. The DSC measurements revealed melting peaks in the first DSC scans of **1a**, **5b** (as an example, see Fig. 3), and **8**, but neither melting nor crystallization was observed in the second and the following DSC scans. How-



Scheme 2

ever, the T_g of the hydrazone **8** was not detected in the DSC curves, perhaps due to the low sensitivity of the apparatus.

Compounds **1b** and **2** were isolated as amorphous compounds. The glass transition temperatures (T_g) of 3,4-ethylenedioxythiophene- and α-terthiophene-based hydrazones detected by DSC are summarized in Table 2. The highest T_gs were showed by 3,4-ethylenedioxythiophene-based dihydrazones **1a** and **1b**. The very low T_g of compound **2** is determined by the long alkylene bridge. Crystalline compound **5a** showed a polymorphic melting in the second DSC heating run. Such behavior of large molecules inclined to form different crystalline forms was described in literature earlier [6].

Figure 4 shows electron photoemission spectra of the films of the newly synthesized hydrazones. The ionization potentials (I_p) established from these spectra (Fig. 4) are summarized in Table 2. 3,4-Ethylenedioxythiophene-based dihydrazones **1a** and **1b** exhibit by *ca.* 0.2 eV lower ionization potentials than the corresponding α-terthiophene-based dihydrazones **5a** and **5b**. Carbazolyl-containing monohydrazones **2** and **8** show higher ionization potentials than dihydrazones. The ionization potential of 3,4-ethylenedioxythiophene-based monohydrazone **2** is very close to that earlier observed for the similar monohydrazone structure having a methyl substituent at the hydrazone N atom [4]. This observation shows that electronically isolated carbazolyl group has

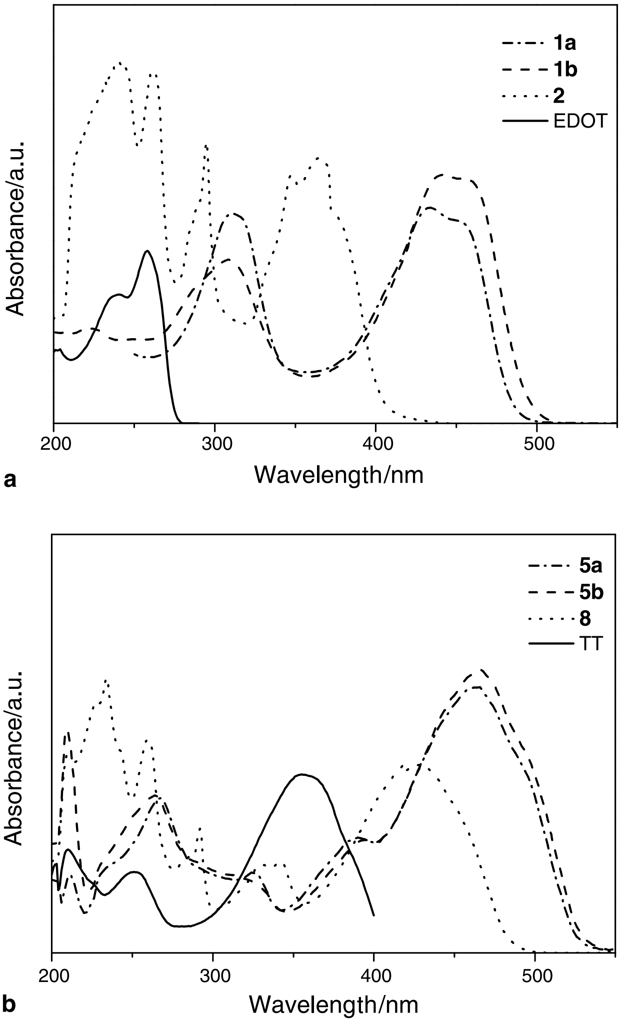


Fig. 1 UV/VIS absorption spectra of dilute *THF* solutions (10^{-5} mol/dm^3) of hydrazones **1a**, **1b**, **2**, 3,4-ethylenedioxythiophene (*EDOT*) (a); and hydrazones **5a**, **5b**, **8**, and α-terthiophene (*TT*) (b)

practically no influence on the value of the ionization potential of compound **2**.

The I_p values of thiophene-based hydrazones show that these compounds are suitable for the application in electrographic photoreceptors. Holes are easily injected into the charge transport layer from the charge generation layer with I_p close to I_p of charge transport layer. The $I_p$values for charge generation materials widely used in electrographic photoreceptors such as titanyl phthalocyanines, perylene pigments, and bisazo pigments are in the range of 5.1–5.6 eV [1].

The xerographic time-of-flight technique was used to characterize charge transport properties of the selected hydrazones. Hydrazones **5b** and **8** which showed the best solubility were chosen for these experiments. Hole-drift mobilities in the amorphous films of thiophene-based hydrazones **5b** and **8** molecularly doped in polymer bisphenol Z polycarbon-

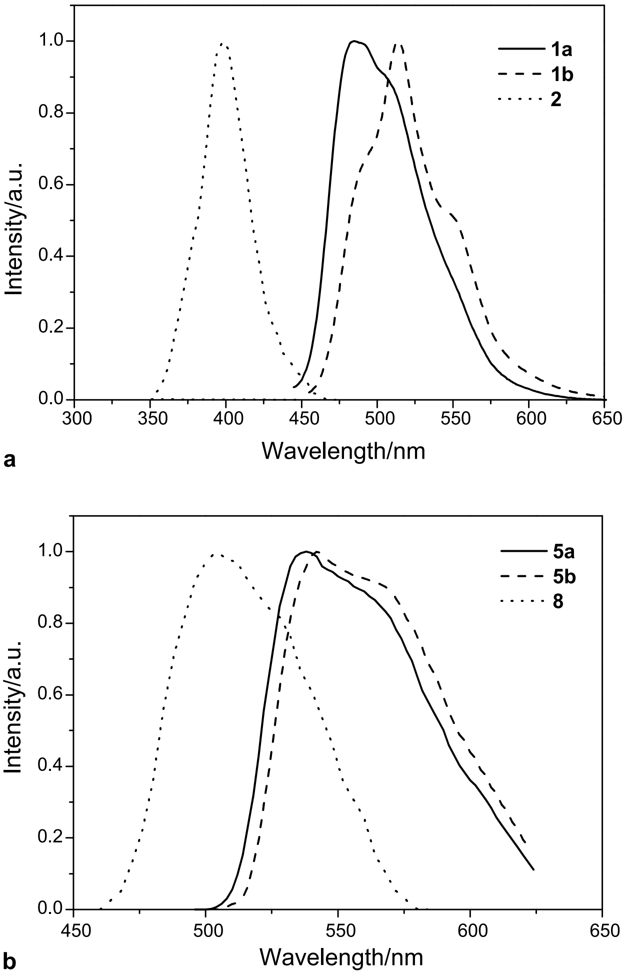


Fig. 2 Fluorescence spectra of dilute *THF* solutions (10^{-5} mol/dm^3) of hydrazones **1a**, **1b** ($\lambda_{ex} = 380$ nm), and **2** ($\lambda_{ex} = 290$ nm) (a); and hydrazones **5a**, **5b** ($\lambda_{ex} = 380$ nm), 380 nm), and **8** ($\lambda_{ex} = 290$ nm) (b)

Table 1 Absorption and fluorescence maxima of 3,4-ethylenedioxythiophene and α-terthiophene-based hydrazones

Compound	Absorption maxima of the lowest energy bands/nm	Fluorescence maxima/nm
1a	432	485
1b	439	513
2	365	398
5a	462	538
5b	465	542
8	428	504

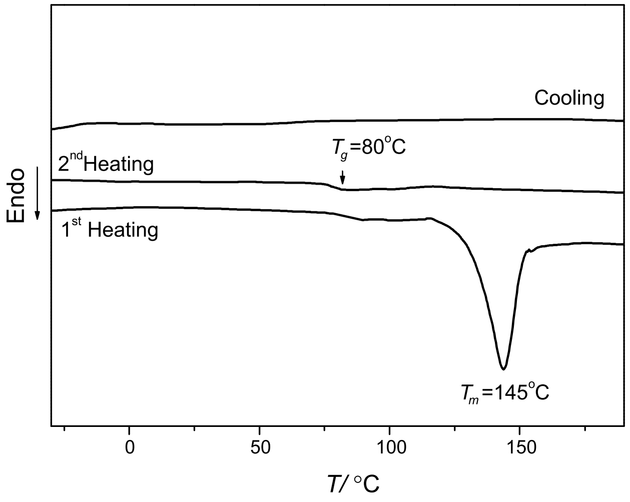


Fig. 3 DSC curves of **5b** at the heating/cooling rate of 10 K/min, N_2 atmosphere

Table 2 DSC measurements of 3,4-ethylenedioxythiophene- and α-terthiophene-based hydrazones

Compound	T_m/°C	T_{cr}/°C	T_g/°C
1a	235[a]	–	120
1b	–	–	160
2	–	–	5
5a	185[b], 194[b]	124	–
5b	145[a]	–	80
8	110[a]	–	–

[a] Only in the first heating run of DSC
[b] Second heating run of DSC

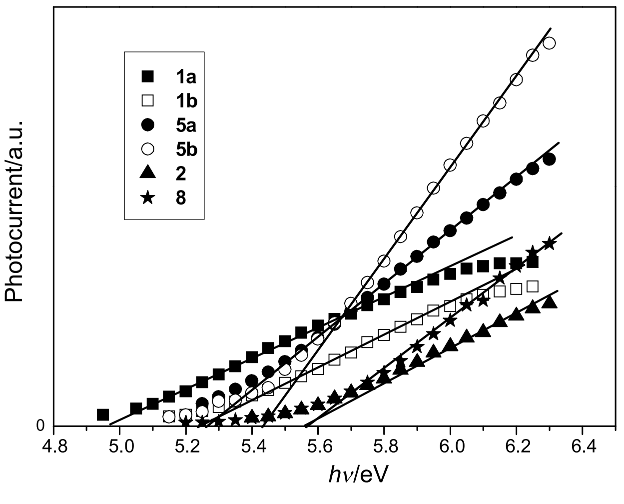


Fig. 4 Electron photoemission spectra of thin films of hydrazones

Table 3 Ionization potentials of thin films of 3,4-ethylene-dioxythiophene and α-terthiophene-based hydrazones

Compound	**1a**	**1b**	**2**	**5a**	**5b**	**8**
I_p/eV	4.99	5.24	5.58	5.25	5.43	5.58

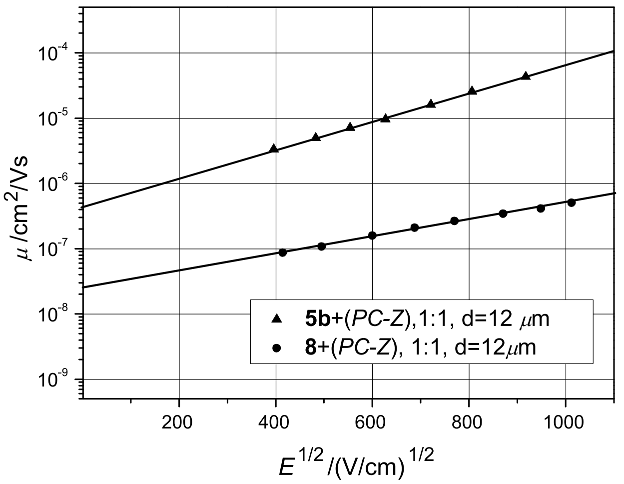


Fig. 5 Electric field dependencies of hole-drift mobilities in the amorphous films of thiophene-based hydrazones **5b** and **8** molecularly doped in *PC-Z* (50 *wt%*)

ate host *PC-Z* (50 *wt%*) were measured at different electric fields at room temperature. The results are shown in Fig. 5. The room temperature hole-drift mobilities in these materials showed linear dependencies on the square root of the electric field. This observation is characteristic of the majority of non-crystalline organic semiconductors and is attributed to the effects of disorder on charge transport [1]. Hole-drift mobilities observed in the layers containing dihydrazone **5b** were by *ca.* two orders of magnitude higher than those observed in the layers containing monohydrazone **8**. They exceed 10^{-5} cm^2/Vs at high electric fields.

Experimental

^{1}H NMR and ^{13}C NMR spectra were recorded with Bruker AC 250 (250 MHz) and Varian Unity Inova (300 MHz) spectrometers. All the data are given as chemical shifts δ (ppm) downfield from $(CH_3)_4Si$. IR spectroscopy measurements were performed on a Perkin Elmer Spectrum GX spectrophotometer, using KBr pellets. UV/VIS spectra were recorded with Spectronic Unicam Genesys 8 spectrophotometer. Mass spectra were obtained on a Waters Micromass ZQ mass spectrometer. Elemental analyses (C, H, N, S, and O) were conducted using the Elemental Analyzer CE-440, their results were found to be in good agreement (±0.2%) with the calculated values. Thermogravimetry (TG) was performed on a Perkin Elmer Thermal Analysis System 409 apparatus at a heating rate of 10 K/min under nitrogen atmosphere. Differential scanning calorimetry (DSC) measurements were performed on a Perkin Elmer Pyris Diamond DSC apparatus at a 10 K/min heating rate under nitrogen atmosphere. The samples for ionization potential and hole-drift mobility measurements were prepared as described previously [7]. The ionization potentials were measured by the electron photoemission in air method [8]. Hole-drift mobilities were measured by a xerographic time-of-flight method [9].

The starting compounds 3,4-ethylenedioxythiophene, *N*-methyl-*N*-phenylhydrazine, *N*,*N*-diphenylhydrazine hydrochloride, *N*-phenylhydrazone, 2-bromothiophene, 1,6-dibromo-*n*-hexane, 9*H*-carbazole (all purchased from Aldrich), 2,5-dibromothiophene (from Across), and the required chemicals, *i.e.* buthyllithium 2.0 *M* sol. in cyclohexane, potassium hydroxide (both from Aldrich), phosphorus oxychloride (from Riedel-de Haen), magnesium, 1,3-bis(diphenylphosphino)propane-nickel(II) (both from Fluka) were purchased as reagent grade chemicals and used as received. α-Terthiophene (**3**) was synthesized according to Refs. [10, 11]. 5,2″-Diformyl-2,2′:5′,5″-terthiophene (**4**) and 5-formyl-2,2′:5′,5″-terthiophene (**6**) were synthesized according to Ref. [12]. The solvents were purified and dried using standard procedures.

3,4-Ethylenedioxythiophene-2,5-dicarbaldehyde di(N-methyl-N-phenylhydrazone) (**1a**, $C_{22}H_{22}N_4O_2S$)

A solution of *N*-methyl-*N*-phenylhydrazine (1.22 g, 10 mmol) in 20 cm^3 *Et*OH was added dropwise to a solution of 3,4-ethylenedioxythiophene-2,5-dicarbaldehyde [13] (0.5 g, 2.52 mmol) in 30 cm^3 *Et*OH with stirring. The reaction mixture was refluxed until all aldehyde reacted (TLC monitoring). Then the reaction mixture was cooled to room temperature. The precipitated product was filtered off, washed with *Et*OH, and purified by silica gel column chromatography (eluent: CH_2Cl_2/*n*-hexane, 2/1). The yield of yellow crystals of **1a**, crystallized from eluent, was 0.88 g (85%). Mp 215°C; ^1H NMR (250 MHz, $CDCl_3$): $\delta = 3.37$ (s, 6H, CH_3), 4.28 (s, 4H, CH_2), 6.90 (m, 2H, Ar), 7.3 (m, 8H, Ar), 7.59 (s, 2H, CH) ppm; ^{13}C NMR (250 MHz, $CDCl_3$): $\delta = 33.68$ (CH_3), 65.45 (CH_2), 115.57 (Ar), 120.87 (C–S), 124.37 (Ar), 129.43 (CH), 139.58 (C(Ar)-N), 147.91 (=C(Thioph)) ppm; IR (KBr): $\bar{\nu} = 3050$ (C–H in Ht, Ar), 2940 (C–H in Aliph.), 1630 (C=N), 1594, 1496 (C=C in Ar), 1107, 1082 (C–O), 750, 685 (C–H in Ht, Ar) cm^{-1}; MS ($APCI^+$, 20 V): m/z (%) = 407 ($[M+H]^+$, 100).

3,4-Ethylenedioxythiophene-2,5-dicarbaldehyde di(N,N-diphenylhydrazone) (**1b**, $C_{32}H_{26}N_4O_2S$)

Compound **1b** was synthesized by the same procedure as compound **1a** only *N*,*N*-diphenylhydrazine hydrochloride was used instead of *N*-methyl-*N*-phenylhydrazine. The yield of yellow crystals of **1b** was 1.2 g (79%). Mp 270°C; ^1H NMR (300 MHz, $CDCl_3$): $\delta = 4.15$ (s, 4H, CH_2), 7.13–7.18 (m, 12H, Ar), 7.36–7.40 (m, 8H, Ar), 7.43 (s, 2H, CH) ppm; ^{13}C NMR (250 MHz, $CDCl_3$): $\delta = 65.16$ (CH_2), 122.89 (Ar), 124.90 (Ar), 127.76 (C–S), 130.20 (CH), 139.98 (C(Ar)-N), 143.85 (–C = (Thioph)) ppm; IR (KBr): $\bar{\nu} = 3050$ (C–H in Ht, Ar), 2950 (C–H in Aliph.), 1630 (C=N), 1590, 1587 (C=C in Ar), 1082, 1064 (C–O), 747, 700 (C–H in Ht, Ar) cm^{-1}; MS ($APCI^+$, 20 V): m/z (%) = 531 ($[M+H]^+$, 100).

2-(3,4-Ethylenedioxythiophene)carboxaldehyde-N-phenyl-N-(carbazole-N-yl-hexyl)hydrazone (**2**, $C_{26}H_{21}N_3O_2S$)

9-(6-Bromo-*n*-hexyl)carbazole (1.9 g, 5.77 mmol) and tetrabutylammonium hydrogen sulfate were added to a solution of 2-(*N*-phenylhydrazonemethyl)-3,4-ethylenedioxythiophene [4] (1 g, 3.85 mmol) in 15 cm^3 ethyl methyl ketone. Then 0.65 g (11.54 mmol) KOH and 0.22 g (1.54 mmol) Na_2SO_4 were added in 3 portions. The reaction mixture was heated at 40°C for 20 h. Then the inorganic components were filtered off. The solvent was removed from the filtrate by rotary evaporation. The product was purified by silica gel column chromatography (eluent: ethyl acetate/*n*-hexane, 1/6). The yield of yellow powders of **2** was 0.66 g (34%). ^1H NMR (300 MHz, $CDCl_3$): $\delta = 1.46$–1.47 (m, 4H, CH_2), 1.64–1.69 (m, 2H, CH_2), 1.91–1.96 (m, 2H, CH_2), 3.85 (t, 2H, $J = 7.5$ MHz, CH_2–N), 4.24 (s, 4H, CH_2–O), 4.33 (t, 2H, $J = 6.9$ MHz, CH_2–N), 6.32 (s, 1H, CH–S), 6.97–7.02 (m, 1H, Ar), 7.30–7.58 (m, 10H, Ar), 7.71 (s, 1H, CH–N), 8.20 (d, 2H, $J = 7.8$ MHz, Ar) ppm; MS ($APCI^+$, 20 V): m/z (%) = 510 ($[M+H]^+$, 100). ^{13}C NMR (300 MHz, $CDCl_3$): $\delta = 24.96$ (CH_2), 27.12 (CH_2), 27.36 (CH_2), 29.13 (CH_2), 43.12 (CH_2), 45.41 (CH_2), 65.03 (CH_2), 65.09 (CH_2), 98.89 (CH), 108.86 (CH), 114.70 (CH), 117.05 (CH), 119.06 (CH), 120.41 (CH), 120.64 (CH), 123.07 (CH), 123.47 (CH), 125.89 (CH), 129.34 (CH), 139.53 (C), 140.63 (C), 141.85 (C), 147.03 (C) ppm; IR (KBr): $\bar{\nu} = 3053$, 3023 (C–H in Ht, Ar), 2934, 2869 (C–H in Aliph.), 1626 (C=N), 1597, 1563, 1494 (C=C in Ht, Ar), 1105, 1079 (C–O), 751, 724 (C–H in Ht, Ar) cm^{-1} MS ($APCI^+$, 20 V): m/z (%) = 510 ($[M+H]^+$, 100).

5,2″-Diformyl-2,2′:5′,5″-terthiophene di(N-methyl-N-phenylhydrazone) (**5a**, $C_{28}H_{24}N_4S_3$)

A solution of *N*-methyl-*N*-phenylhydrazine (0.53 g, 4.34 mmol) in 20 cm^3 isopropyl alcohol was added dropwise to a solution of 2-(5-(thiophen-2-yl)thiophen-2-yl)thiophene-2,5-dicarbaldehyde (**4**) (0.3 g, 1.09 mmol) in 250 cm^3 isopropyl alcohol by stirring. The reaction mixture was refluxed until all aldehyde reacted (TLC monitoring). Then the reaction mixture was cooled down to room temperature. A part of isopropyl alcohol was evaporated. The precipitated product was filtered off and washed with methyl alcohol and purified by silica gel column chromatography (eluent: CH_2Cl_2/*n*-hexane, 1/3). The yield of **5a** was 0.195 g (35%). ^1H NMR (300 MHz, $CDCl_3$): $\delta = 3.43$ (s, 6H, CH_3), 6.90–6.95 (m, 2H, Ar), 7.21 (d, 2H, $J = 3.9$ MHz, Ar), 7.21–7.39 (m, 12H, Ar), 7.21 (s, 2H, Ar) ppm; IR (KBr): $\bar{\nu} = 3058$, 3023 (C–H in Ht, Ar), 2961, 2923 (C–H in Aliph.), 1597, 1570, 1497 (C=C in Ht, Ar, C=N), 746, 688 (C–H in Ht, Ar) cm^{-1}; ^{13}C NMR (300 MHz, $CDCl_3$): $\delta = 118.53$ (Ar), 119.91 (Ar), 124.75 (CH), 127.06 (CH), 127.37 (CH), 129.12 (Ar), 137.26 (C), 137.78 (C), 142.22 (C), 143.52 (C) ppm; MS ($APCI^-$, 20 V): m/z (%) = 512 ($[M–H]^-$, 100).

5,2″-Diformyl-2,2′:5′,5″-terthiophene di(N,N-diphenylhydrazone) (**5b**, $C_{38}H_{28}N_4S_3$)

Compound **5b** was synthesized by the same procedure as compound **5a** only *N*,*N*-diphenylhydrazine hydrochloride was used instead of *N*-methyl-*N*-phenylhydrazine. The yield of **5b** was 2.03 g (96%). ^{1}H NMR (300 MHz, $CDCl_3$): $\delta =$ 6.83 (d, 2H, $J = 4.2$ Hz, CH), 7.07 (d, 2H, $J = 3.9$ Hz, CH), 7.18 (s, 2H, CH=N), 7.23–7.29 (m, 14H, CH, Ar), 7.48 (t, 8H, $J = 7.95$ Hz, Ar). IR (KBr): $\bar{\nu} = 3064$, 3037 (C–H in Ht, Ar), 1630 (C=N), 1598, 1588, 1494, 1456 (C=C in Ht, Ar), 744, 700 (C–H in H, Ar) cm^{-1}; ^{13}C NMR (300 MHz, $CDCl_3$): $\delta = 122.73$ (Ar), 123.92 (CH), 124.83 (CH), 125.00 (Ar), 127.37 (CH), 130.12 (Ar), 130.26 (CH), 136.78 (C), 136.95 (C), 141.20 (C), 143.53 (C) ppm; MS (APCI$^+$, 20 V): $m/z(\%) = 637$ ($[M + H]^+$, 100).

5-Formyl-2,2′:5′,5″-terthiophene-N-phenylhydrazone (**7**, $C_{19}H_{14}N_2S_3$)

Compound **7** was synthesized by the same procedure as **5a**. The yield of **7** was 88%. ^{1}H NMR (300 MHz, $CDCl_3$): $\delta =$ 6.90–7.62 (m, 8H, Ar), 7.82 (s, 1H, CH=N) ppm; ^{13}C NMR (300 MHz, $CDCl_3$): $\delta = 118.63$ (CH), 121.95 (CH), 123.83 (CH), 124.12 (CH), 127.37 (CH), 129.12 (CH), 137.72 (C), 138.92 (C), 141.20 (C), 142.53 (C) ppm; IR (KBr): $\bar{\nu} = 3317$ (N–H), 3058, 3025 (C–H in Ht, Ar), 1596, 1567, 1495 (C=C in Ht, Ar, C=N), 745, 681 (C–H in Ht, Ar) cm^{-1}; MS (APCI$^+$, 20 V), m/z (%) $= 367$ ($[M + H]^+$, 100).

5-Formyl-2,2′:5′,5″-terthiophene-N-phenyl-N-(carbazole-9-ylhexyl)hydrazone (**8**, $C_{32}H_{23}N_3S_3$)

Compound **8** was synthesized by the same procedure as compound **2**. The yield of **8** was 78%. ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.45$–1.48 (m, 4H, CH_2), 1.61–1.67 (m, 2H, CH_2), 1.89–1.99 (m, 2H, CH_2), 3.81 (t, 2H, $J = 7.65$ Hz, CH_2–N), 4.34 (t, 2H, $J = 7.05$ Hz, CH_2–N), 6.93–7.56 (m, 20H, CH, Ar), 8.15 (d, 2H, $J = 7.8$ MHz, Ar) ppm; ^{13}C NMR (300 MHz, $CDCl_3$): $\delta = 25.08$ (CH_2), 27.17 (CH_2), 27.45 (CH_2), 29.24 (CH_2), 43.13 (CH_2), 45.65 (CH_2), 108.88 (CH), 115.03 (CH), 119.11 (CH), 120.68 (CH), 120.93 (CH), 123.11 (CH), 123.95 (CH), 124.52 (CH), 124.71 (CH), 125.93 (CH), 126.33 (CH), 128.20 (CH), 129.44 (CH), 136.37 (C), 136.79 (C), 137.44 (C), 140.65 (C), 142.13 (C), 146.76 (C) ppm; IR (KBr): $\bar{\nu} = 3063$, 3042 (C–H in Ht, Ar), 2929, 2852 (C–H in Aliph.), 1624 (C=N), 1594, 1563, 1484 (C=C in Ht, Ar), 750, 722 (C–H in Ht, Ar) cm^{-1}; MS (APCI$^+$, 20 V): m/z (%) $= 616$ ($[M + H]^+$, 100).

Acknowledgements

Financial support from the Lithuanian Science and Studies Foundation is gratefully acknowledged. Dr. habil. *Valentas Gaidelis* is thanked for the recording of photoemission spectra and the ionization potential measurements.

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