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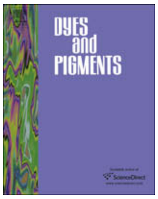

Phenothiazinyl- and 4-diethylaminophenyl-substituted diethylenes as fluorescent and hole-transporting molecular materials

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ABSTRACT

A series of phenothiazinyl- and 4-diethylaminophenyl-substituted diethylenes were synthesized and characterised; their thermal, optical and photoelectrical properties were investigated. TGA showed that the compounds display high thermal stability, achieving 5% mass loss at temperatures up to 385 °C; DSC revealed that many of the compounds exhibited glass transition temperatures ranging from 20 to 124 °C. Dilute solutions of the diethylenes exhibited fluorescence emission in the green-blue region with an efficiency reaching 99%. Electron photoemission spectrometry and time of flight revealed ionization potentials of 5.34–5.52 eV; the room temperature hole drift mobility of one of the compounds molecularly doped in bisphenol Z polycarbonate exceeded 10^{-5} cm^2/Vs under a high electric field.

1. Introduction

Amorphous organic materials exhibiting both charge transport and emission properties are of interest for the application in optoelectronic devices such as organic light emitting diodes [1,2]. The compounds containing aromatic amino groups are known to be capable of both transporting holes and emitting light [3,4]. Among electroactive aromatic amines triphenylamine and carbazole-based materials prevail [5,6]. Phenothiazine having electron-rich sulphur and nitrogen heteroatoms is much less studied and exploited as electroactive moiety [7–9]. Even less works are reported on phenothiazinyl-substituted ethylenes [8,10]. In this presentation we report on the synthesis of new glass-forming electroactive derivatives of phenothiazine i.e. diethylenes containing two phenothiazinyl groups. For the comparison we have synthesized the corresponding diethylenes containing diethylaminophenyl groups. Electron rich phenothiazinyl- and 4-diethylaminophenyl moieties used in this study facilitate the transporting of the positive charge carriers. Increase of the size of molecules usually reinforces their inclination to glass formation. In addition, if the increase of the size of molecules is associated with the increase of the systems of conjugated π-electrons, it leads to the improvement of emission and charge transport properties. The diethylenes were synthesized by Wittig-Horner or Wittig reactions from the corresponding formyl derivatives and ylides or triphenylphosphonium salts. We also report on the thermal, optical, photophysical and photoelectrical properties of the synthesized materials.

2. Experimental

2.1. Instrumentation

^{1}H NMR spectra were obtained on Varian Unity Inova (300 MHz (^{1}H)) or JOEL JNM-FX 100 (100 MHz (^{1}H)) equipment. IR spectra were recordered using Perkin Elmer FT-IR System. The spectra of solid compounds were performed in a form of KBr pellets. Mass spectra were obtained on Waters ZQ 2000. The UV spectra were recorded on a Spectronic Genesys™ 8 spectrometer. Fluorescence (FL) spectra were recorded with Hitachi MPF-4 spectrometer.

Differential scanning calorimetry (DSC) measurements were carried out with Perkin–Elmer DSC-7 at 10 °C min^{-1} heating rate under nitrogen atmosphere. The glass transition temperatures (T_gs) were determined from the second heating scans. Thermogravimetric analysis (TGA) was performed on Mettler Toledo TGA/SDTA 851^e at 10 °C min^{-1} heating rate under nitrogen atmosphere.

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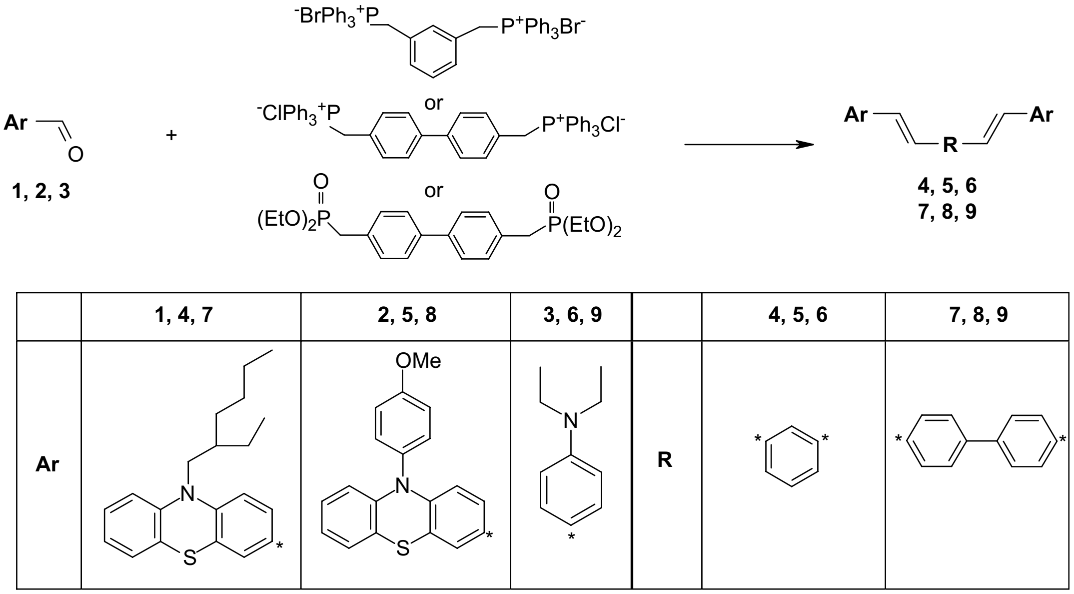

Scheme 1.

The samples for ionization potential measurements were prepared as described previously [11]. The ionization potentials were measured by the electron photoemission in air method [12]. The standard error in the mean to 95% confidence for the values of ionization potential was 0.04 eV. Hole-drift mobilities (μ) were measured by xerographic time of flight (XTOF) technique [13,14]. The samples for the measurements were prepared by casting the solutions of synthesized materials and bisphenol Z polycarbonate (PC-Z) on glass plates with conductive SnO_2 layer or polyester films with Al layer. The thickness of the transporting layer varied in the range of 3–10 μm.

2.2. Materials

4,4′-Bis(chloromethyl)1,1′-biphenyl, α,α′-dibromo-*m*-xylene, potassium *tert*-butoxide, sodium hydride, 4-diethylaminobenzaldehyde (**3**) were received from Aldrich and used as-received.

3-Formyl-10-(2-ethylhexyl)phenothiazine (**1**) and 3-formyl-10-(*p*-methoxyphenyl)phenothiazine (**2**) were synthesized by the known procedures including alkylation or Ullmann coupling followed by Vilsmeier reactions [15]. 1,3-Xylene-bis(triphenylphosphonium bromide), biphenyl-4,4′-diyl-dimethyldi(triphenylphosphonium chloride) and 4,4′-di[(diethoxyphosphonyl)methyl]biphenyl were synthesized by the known procedures [16,17].

2.2.1. 1,3-Di{2-[10-(2-ethylhexyl)phenothiazin-3-yl]vinyl}benzene (**4**)

3-Formyl-10-(2-ethylhexyl)phenothiazine (**1**) (0.85 g, 2.5 mmol) and 1,3-xylene-bis(triphenylphosphonium bromide) (0.94 g, 1.19 mmol) were dissolved in 5 ml of chloroform under an atmosphere of nitrogen. The solution obtained was then added dropwise to the solution of potassium *tert*-butoxide 0.4 g (3.57 mmol) in 7 ml of anhydrous ethanol and the reaction mixture was stirred at room temperature for 24 h. Pouring of the reaction mixture into a large amount of methanol gave rise to yellow precipitates. Separated by filtration the crude product was further purified by silica gel column chromatography using an eluent mixture of chloroform and hexane in a vol. ratio of 1:4. The yield of **4** as amorphous resin was 0.15 g (48%). (FW = 748 g/mol). ^1H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.87–1.06 (m, 12H, CH_3); 1.28–1.60 (m, 16H, CH_2); 1.92–2.12 (m, 2H, CH); 3.71–3.85 (m, 4H, CH_2–N); 6.55–7.85 (m, 22H, ar, CH=CH–). IR (in KBr) ν/cm^{-1}: 3019 (CH_{ar}); 2958, 2927, 2857 ($CH_{aliphatic}$); 1593, 1573 ($C=C_{ar}$); 1250 (C–N); 958 (CH=CH *trans*); 688 (CH=CH *cis*). MS (APCI$^+$, 20 V), *m*/*z* (%): 749 ($[M+H]^+$, 100).

1,3-Di{2-[10-(4-methoxyphenyl)phenothiazin-3-yl]vinyl}benzene (**5**) was prepared from 2.1 g (6.3 mmol) of 3-formyl-10-(*p*-methoxyphenyl)phenothiazine (**2**) and 2.37 g (3 mmol) of 1,3-xylene-bis(triphenylphosphonium bromide) in the same way as compound **4**. The compound was purified by silica gel column chromatography using an eluent mixture of ethylacetate and hexane in a vol. ratio of 1:4. The yield of amorphous powder of **5** was 1.7 g (77%). (FW = 736 g/mol). ^1H NMR (300 MHz, $CDCl_3$, δ, ppm): 3.89; 3.94 (s; s, 6H, CH_3, *cis*; *trans*). 6.0–7.6 (m, 30H, ar, CH=CH–). IR (in KBr) ν/cm^{-1}: 3013 (CH_{ar}); 2952, 2930, 2833 ($CH_{aliphatic}$); 1591, 1573 ($C=C_{ar}$); 1245 (C–N); 956 (CH=CH *trans*); 688 (CH=CH *cis*). MS (APCI$^+$, 20 V), *m*/*z* (%): 737 ($[M+H]^+$, 10).

2.2.2. 1,3-Di[4-(diethylamino)phenyl-2-vinyl]benzene (**6**)

4-(Diethylamino)benzaldehyde (1.11 g, 6.27 mmol), 1,3-xylene-bis(triphenylphosphonium bromide) (1 g, 2.98 mmol) and sodium hydride (1 g, 41.75 mmol) were stirred in 30 ml dry THF at the room temperature for 24 h under N_2 blanket. Then the reaction mixture was poured into a large amount of methanol and yellow precipitate was obtained. Separated by filtration the crude product was further purified by silica gel column chromatography using an eluent mixture of ethylacetate and hexane in a vol. ratio of 1:4. It was crystallized from the eluent mixture of solvents. The yield of **6** was 0.66 g (52%). M.p. 134 °C (DSC). (FW = 424 g/mol). ^1H NMR

Table 1
Melting points, glass transition temperatures and 5% mass loss temperatures of compounds **4**–**9**.

Compound	T_g (°C)	T_m (°C)	T_{ID} (°C)
4	20[a,b]	–	351
5	105[a,b]	–	352
6	23[b]	134[a,b]	308
7	56[a,b]	–	370
8	124[b]	266[a,b]	345
9	–	304[a]	385

[a] From the first DSC scan.
[b] From the second DSC scan.

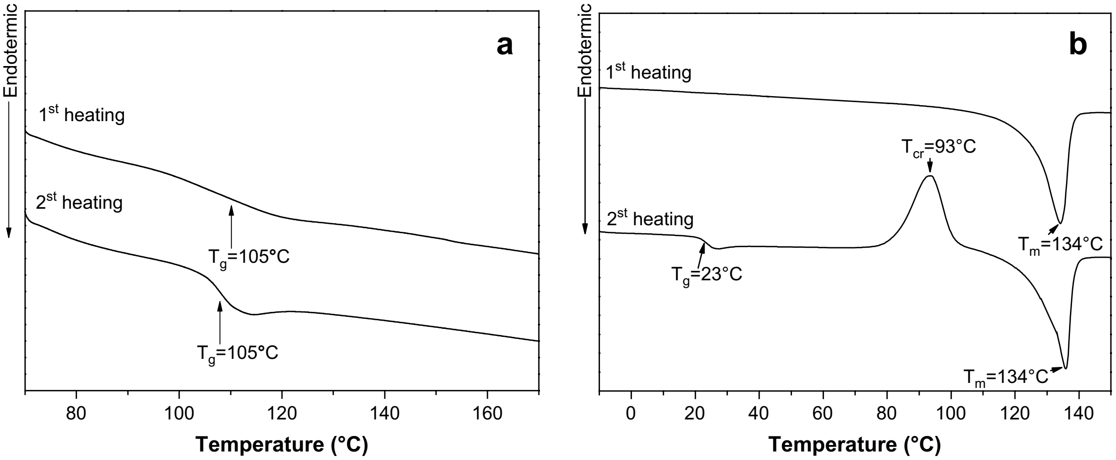

Fig. 1. DSC curves of **5** (a) and **6** (b). Heating rate: 10 °C/min, nitrogen atmosphere.

(300 MHz, $CDCl_3$, δ, ppm): 1.15–1.35 (m, 12H, CH_3); 3.32–3.35 (m, 18H, CH_2); 6.69–7.51 (m, 22H, ar, CH=CH–). IR (in KBr) ν/cm^{-1}: 3014 (CH_{ar}); 2966, 2927, 2887 ($CH_{aliphatic}$); 1606, 1587 ($C{=}C_{ar}$); 1265 (C–N); 957 (CH=CH *trans*); 687 (CH=CH *cis*). MS (APCI$^+$, 20 V), *m*/*z* (%): 425 ($[M+H]^+$, 100).

4,4′-Di{2-[10-(2-ethylhexyl)phenothiazin-3-yl]vinyl}biphenyl (**7**) was prepared from 1 g (2.95 mmol) of 3-formyl-10-(2-ethylhexyl)phenothiazine (**1**), 1.08 g (1.4 mmol) of biphenyl-4,4′-diyldimethyldi(triphenylphosphonium chloride) and 0.46 g (19.09 mmol) of sodium hydride in the same way as compound **6**. The product was purified by silica gel column chromatography using an eluent mixture of chloroform and hexane in a vol. ratio of 1:8. The yield of amorphous powder of **7** was 2.3 g (96%). (FW = 825 g/mol). ^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.86 (t, 12H, CH_3); 1.05–2.10 (m, 16H, CH_2); 2.10–2.20 (m, 2H, CH); 3.71 (d, 4H, CH_2–N); 6.42–7.25 (m, 26H, ar, CH=CH–). IR (in KBr) ν/cm^{-1}: 3022 (CH_{ar}); 2956, 2925, 2856 ($CH_{aliphatic}$); 1595, 1574 ($C{=}C_{ar}$); 1286 (C–N); 962 (CH=CH *trans*); 699 (CH=CH *cis*). MS (APCI$^+$, 20 V), *m*/*z* (%): 826 ($[M+H]^+$, 70).

4,4′-Di{2-[10-(4-methoxyphenyl)phenothiazin-3-yl]vinyl}biphenyl (**8**) was prepared from 0.5 g (1.5 mmol) of 3-formyl-10-(*p*-methoxyphenyl)phenothiazine (**2**), 0.33 g (0.72 mmol) of 4,4′-di[(diethoxyphosphonyl)methyl]biphenyl and 0.23 g (9.72 mmol) of sodium hydride in the same way as compound **6**. The product was purified by silica gel column chromatography using an eluent mixture of ethylacetate and hexane in a vol. ratio of 1:6. It was crystallized from the eluent mixture of solvents. The yield of **8** was 1.9 g (97%). M.p. 266 °C (DSC). (FW = 813 g/mol). ^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 3.95 (s, 6H, CH_3); 6.13–7.7 (m, 34H, ar, CH=CH–). IR (in KBr) ν/cm^{-1}: 3022 (CH_{ar}); 2948, 2928, 2828 ($CH_{aliphatic}$); 1606, 1593, 1574 ($C{=}C_{ar}$); 1307 (C–N); 965 (CH=CH *trans*); 668 (CH=CH *cis*). MS (APCI$^+$, 20 V), *m*/*z* (%): 814 ($[M+H]^+$, 60).

4,4′-Di[4-(diethylamino)phenyl-2-vinyl]biphenyl (**9**) was prepared from 2 g (11.3 mmol) of 4-(diethylamino)benzaldehyde, 2.44 g (5.38 mmol) of 4,4′-di[(diethoxyphosphonyl)methyl]biphenyl and 1.76 g (73.13 mmol) of sodium hydride in the same way as compound **6**. The product was purified by silica gel column chromatography using an eluent mixture of ethylacetate and hexane in a vol. ratio of 1:4. It was crystallized from the eluent mixture of solvents. The yield of **9** was 5.3 g (94%). M.p. 304 °C (DSC). (FW = 500 g/mol). ^{1}H NMR (100 MHz, TFA + acetone-d_6, δ, ppm): 1.06 (t, 12H, CH_3); 3.40–3.82 (m, 8H, CH_2); 7.00–7.72 (m, 20H, ar, CH=CH–). IR (in KBr) ν/cm^{-1}: 3021 (CH_{ar}); 2972, 2931, 2883 ($CH_{aliphatic}$); 1609, 1595 ($C{=}C_{ar}$); 1265 (C–N); 963 (CH=CH *trans*). MS (APCI$^+$, 20 V), *m*/*z* (%): 502 ($[M+H]^+$, 50).

3. Results and discussion

The synthetic route to compounds **4**–**9** is shown in Scheme 1. Compounds **4**–**9** were prepared via Wittig or Wittig-Horner reactions of aldehydes and triphenylphosphonium salts.

All the synthesized compounds were identified by IR, ^{1}H NMR and mass spectrometries.

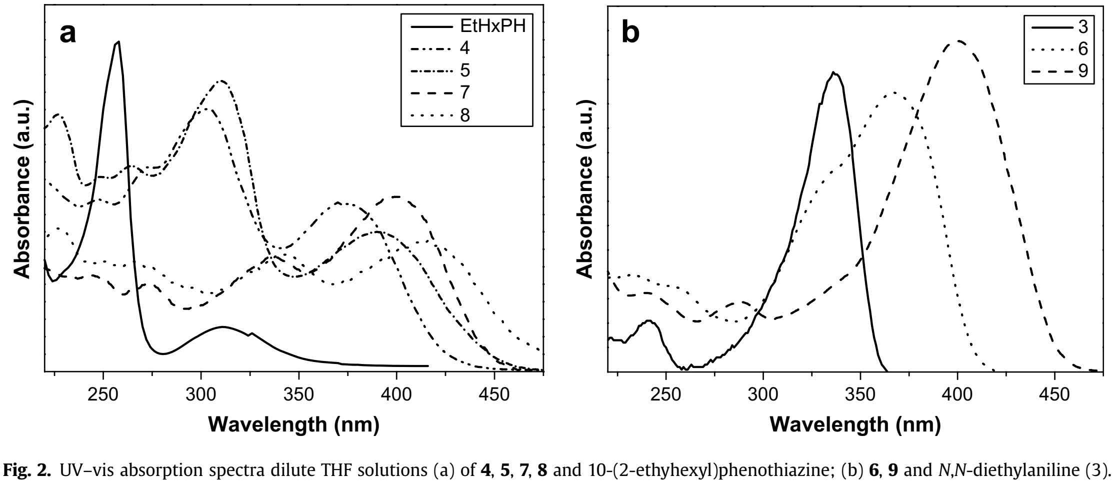

Fig. 2. UV–vis absorption spectra dilute THF solutions (a) of **4**, **5**, **7**, **8** and 10-(2-ethyhexyl)phenothiazine; (b) **6**, **9** and *N*,*N*-diethylaniline (3).

Table 2
UV–vis absorption, FL emission maxima and FL quantum efficiency (QE) of the dilute THF solutions of compounds **4**–**9**.

Compound	UV, λ_{max} (nm)	FL, λ_{max} (nm)	QE (%)
4	303, 375	504	48
5	309, 392	492	44
6	367	438	17
7	338, 398	510	64
8	344, 416	511	67
9	400	487	99

Compounds **4**–**6** were readily soluble in common organic solvents, such as tetrachloroethane, chloroform, THF, acetone at room temperature. Compounds **8** and **9** were moderately soluble in THF, chloroform, dichloromethane and other chlorinated solvents.

The behavior under heating of compounds **4**–**9** was studied by TGA and DSC under nitrogen atmosphere. The values of the glass transition temperatures (T_gs), melting points (T_ms), and the temperatures at which initial loss of mass (5%) was observed (T_{ID}) are summarized in Table 1. All the newly synthesized diethylenes demonstrate high thermal stability. The mass loss occurs at the temperatures higher than 308 °C, as confirmed by TGA with a heating rate of 10 °C/min.

Compounds **4**, **5** and **7** were obtained as amorphous materials as confirmed by DSC. When the samples of these compounds were heated the glass transitions were observed at 20, 105 and 56 °C, respectively, and no peaks due to crystallization and melting appeared in the DSC thermograms. As an example, DSC curves of compound **5** are given in Fig. 1(a). Compounds **6**, **8** and **9** were isolated by crystallization from solutions. Compounds **6** and **8** showed the similar behaviour in the DSC experiments. They showed endothermic melting signals in the first heating scan of the DSC experiment with the maxima at 134 °C and 266 °C, respectively, and formed glasses when cooled from the melt. The second heating scans revealed glass transitions at 23 °C and 124 °C, followed by crystallization at 93 °C and 232 °C, and melting at 134 °C and 266 °C. Fig. 1(b) shows DSC curves of compound **6**.

Compound **9** showed endothermic melting signal in the first heating scan of the DSC experiment at 304 °C. In the cooling scan it showed crystallization at 292° and in the following second heating scan it showed melting at 304 °C again. Thus compound **9** could not be transformed into the glassy state by cooling from the melt.

Compounds **4**–**9** were also studied by UV–vis and fluorescence spectrometries. UV–vis absorption spectra of dilute solutions of the synthesized compounds are presented in Fig. 2. For the comparison the spectra of the dilute solutions of 10-(2-ethylhexyl)phenothiazine (EtHxPH) and *N*,*N*-diethylaniline are also given. The wavelengths of the absorption maxima are summarized in Table 2. The absorption bands do not possess any vibrational structure. Compounds **4**, **5**, **7**, **8** exhibit broad absorption up to 475 nm with λ_{max} in the range of 303–416 nm. The comparison of the absorption spectra of compounds **4**, **5**, **7**, **8** with that of Et HxPH shows that these compounds have new absorption maxima in the longer wavelength region at 375, 392, 398 and 416 nm, respectively. Compounds **6** and **9** containing diethylaminophenyl groups exhibit broad absorption bands with λ_{max} values of 367 and 400 nm. Their absorption spectra are strongly red shifted with those of *N*,*N*-diethylaniline. This is, apparently, the consequence of the increased size of the conjugated double bonds systems. The absorption bands of 4,4′-biphenylene-centered compounds **7**–**9** are red shifted with respect to the absorption bands of the corresponding 1,3-phenylene-centered compounds **4**–**6**. This observation can apparently be explained by the more planar structure 4,4′-biphenylene-centered compounds, which ensure more effective conjugation of electrons.

Fluorescence spectra of the dilute solutions of compounds **4**–**9** are presented in Fig. 3. The wavelengths of the emission maxima are summarized in Table 2. The dilute solutions of the materials emit light in the green-blue spectral range with the intensity maxima ranging from 438 nm to 511 nm. The fluorescence bands of 4,4′-biphenylene-centered compounds **7**–**9** are red shifted with respect of the emission bands of the corresponding 1,3-phenylene-centered compounds **4**–**6**. This observation is consistent with the UV–vis spectrometry data. Fluorescence quantum efficiencies of the THF solutions of 1,2-diarylethylenes **4**–**9** are presented in Table 2. They are estimated to range from 17% to 99%, as determined by comparison with the standard of known quantum yield. 4,4′-Biphenylene-centered compounds exhibit higher fluorescence quantum yields than the corresponding 1,3-phenylene-centered compounds. The highest fluorescence quantum yield was observed for compound **9**.

Chromaticity coordinates (x, y) compounds **4**–**9** were evaluated on the basis of the trichromatic system of modern colorimetry developed by CIE in 1931. First, the tristimulus values X, Y, and Z representing the relative quantities of the primary colors were obtained by integrating the emission spectra with the standard color matching functions $\bar{x}(\lambda)$, $\bar{y}(\lambda)$, and $\bar{z}(\lambda)$ tabulated in Ref. [18]. Following the normalization procedure of the tristimulus values, the normalized color value components x and y i.e., chromaticity coordinates, were calculated. The detailed description of the evaluation procedure is described [19]. Chromaticity coordinates which refer to the bluish-green part of the chromaticity diagram, were obtained for compounds **4**–**9**. We note that the chromaticity coordinates of **6** [(0.15, 0.08)] are very close to those of the primary blue color [(0.15, 0.06)] used in the modern NTSC, PAL and SECAM systems. The use of **6** as the primary blue when developing new

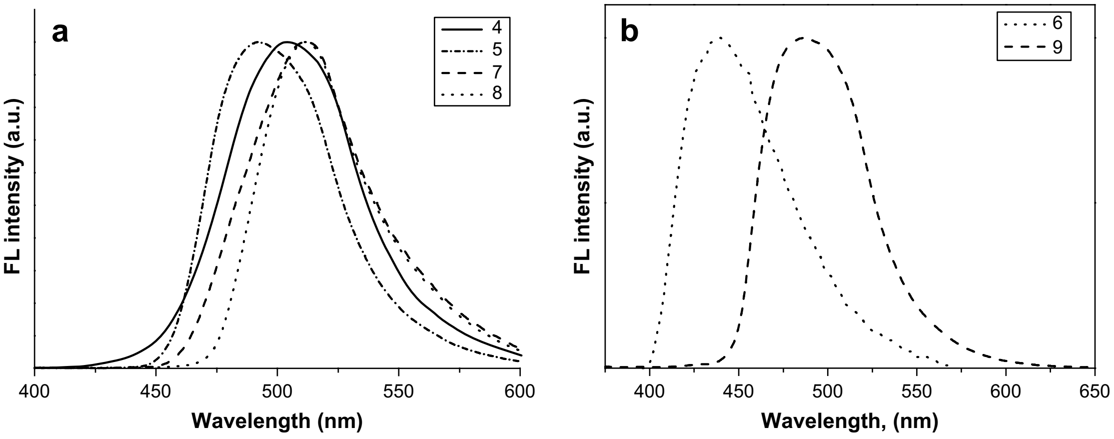

Fig. 3. FL emission ($\lambda_{ex} = 360$ nm) spectra dilute THF solutions (a) of **4**, **5**, **7**, **8**, and (b) of **6** and **9**.

Table 3
The electronic characteristics of 1,2-diarylethylenes compounds.

Compound		4	5	6	7	8	9
Energy, (eV)	HOMO	5.46	5.47	5.34	5.51	5.52	5.30
	LUMO	2.67	2.88	2.41	2.78	3.06	2.36

OLED displays could avoid inconsistencies with modern broadcasting standards caused by color mismatch.

An important characteristic of electronically active compounds used in optoelectronic devices is ionization potential (I_p), which characterizes the electron releasing work under illumination. The ionization potential (I_p) was measured by electron photoemission technique in air. The values of I_p, which correspond the HOMO values, are presented in Table 3. Using the values of optical band gap (δE) which can be estimated by the absorption spectroscopy, the LUMO values were calculated by the method described before [20]. They are also given in Table 3.

The HOMO values of the synthesized materials are rather close and range from 5.30 to 5.52 eV. Diethylenes containing 4-diethylaminophenyl groups (**6** and **9**) exhibit a little lower HOMO values than their phenothiazinyl-containing counterparts. The HOMO values of the phenothiazinyl-containing ethylenes are lower than those of derivatives having electronically isolated phenothiazine rings (5.7–5.8 eV) [21]. Holes would be easily injected into the layers of these materials from a charge generation layer or a conductive anode with I_p or work function close to 5.3 eV. It should be mentioned that I_p values for charge generation materials, including those widely used in electrophotographic photoreceptors, e.g. pigments, such as titanyl phthalocyanines [22,23], perylene pigments [24] and bisazo pigments [25] are in the range of 5.1–5.6 eV. The I_p values of compounds **4**–**9** are close to those of indium-tin oxide (ITO) which is widely used as an anode in electroluminescent devices [26]. The injection barrier of holes from the electrode into the layers of the materials synthesized would be 0.4–0.5 eV.

The solubility of compounds **8**, **9** was not sufficient for the preparation of the layers with the thickness required for the estimation of the charge mobilities. Compounds **4**–**7** were used for the charge carrier mobility studies by the xerographic time of flight technique. Hole drift mobilities (μ_h) were estimated for the materials molecularly dispersed in polymer host bisphenol Z polycarbonate (PC-Z). Electric field dependencies of μ_h for the molecular dispersions of **4**–**7** in PC-Z (50%) are shown in Fig. 4.

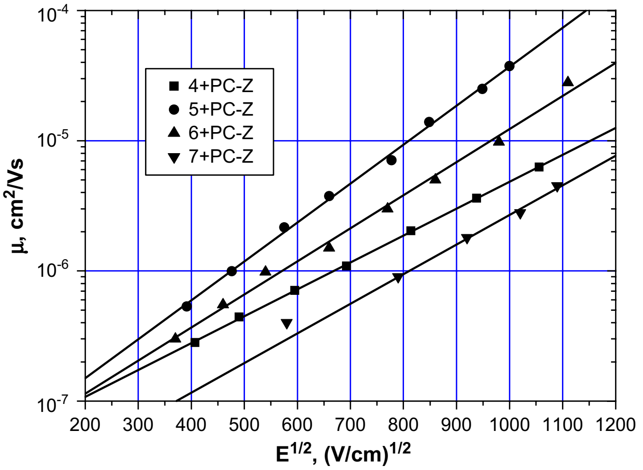

Fig. 4. Electric field dependencies of hole drift mobility in charge transport layers of compounds **4**–**7** doped in PC-Z (50%).

The room temperature μ_h showed linear dependencies on the square root of the electric field. Such dependence is observed for the majority of non-crystalline organic systems and can be attributed to the effects of disorder on charge transport [27]. The solid solutions of **4**–**7** in PC-Z (50%) demonstrated hole drift mobility values in the range from 2.6×10^{-6} cm^2/Vs to 3.7×10^{-5} cm^2/Vs at an electric field of 10^6 V/cm at room temperature.

In conclusion, we have synthesized various phenothiazinyl- or 4-diethylaminophenyl-containing diethylenes which exhibit relatively high thermal stability with 5% mass loss temperatures ranging from 308 to 385 °C. Most of them form glasses with glass transition temperatures in the range of 20–124 °C. Their dilute solutions feature fluorescence emission in the green-blue spectral range with efficiency ranging from 17% to 99%. The synthesized compounds exhibit ionization potentials of 5.34–5.52 eV. Time-of-flight hole mobilities in the films of one synthesized compound molecularly dispersed in bisphenol Z polycarbonate were found to exceed 10^{-5} cm^2/Vs at moderate and high electric fields.

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