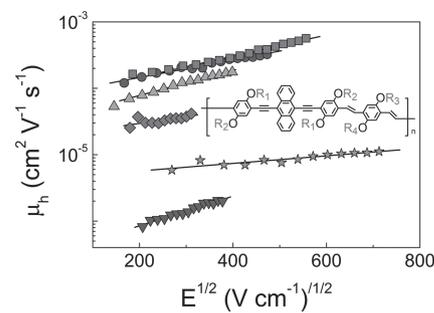


Effect of Side Chains on Charge Transport of Anthracene-Based PPE–PPV Copolymers

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The variation of the drift mobility of positive and negative charge carriers in films of anthracene-containing poly(*p*-phenylene-ethynylene)-*alt*-poly(*p*-phenylene-vinylene)s (AnE-PVs), differently substituted, is investigated as a function of the applied electric field. Branched 2-ethylhexyl and linear alkoxy side chains of different lengths are considered, as well as well-defined and random distributions of lateral substituents. The same conditions are used both for the deposition of the polymer films and for their characterization, which allows for the establishment of a clear relationship between the chemical structure and the charge carrier mobility.



1. Introduction

Soluble π -conjugated polymers are attracting increasing interest in recent years, because of the unique combination of solution-processability, robust mechanical properties, and tunability of their electronic properties, making these innovative materials promising candidates for cost-effective and large-area flexible electronics.^[1,2] Field-effect transistors (OFETs),^[3] photovoltaic cells,^[4] and light-emitting

diodes (OLEDs)^[5] are the key applications of conjugated polymers, and in general of π -conjugated organic materials. Charge transport properties constitute a major determining factor for the operation of these devices. High and balanced hole and electron mobilities are required for high performance in solar cells and the current flowing between source and drain in an OFET is strictly related to charge carrier mobility (μ). Balanced charge transport is also required for high efficiency of charge recombination in OLEDs. Given the strong impact of μ on the properties of any electronic device, the knowledge of how to tune the molecular structure of organic conjugated materials to achieve high mobility values is of prime importance, in addition to the other factors affecting the charge transport properties of organic films (molecular packing, disorder, impurities, etc.).

In this study, the drift mobility of positive and negative charge carriers in a series of six anthracene-containing poly(*p*-phenylene-ethynylene)-*alt*-poly(*p*-phenylene-vinylene)s (AnE-PVs) is investigated and compared. AnE-PVs, with the anthracene unit between two triple bonds, are a relevant class of conjugated polymers,^[6] exhibiting outstanding optoelectronic properties. Light-emitting diodes showing a turn-on voltages below 2 V^[7] and solar cells exhibiting a state-of-art efficiency of around 5%,

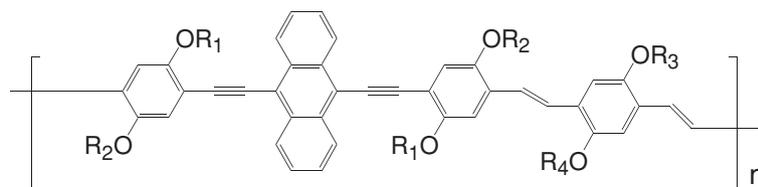
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Polymer	Random	Side-chains
AnE-PV-ab		R1, R2: octyl; R3, R4: 2-ethylhexyl
AnE-PV-ae		R1, R2: octyl; R3, R4: dodecyl
AnE-PV-bb		R1 – R4: 2-ethylhexyl
AnE-PV-stat	X	R1 – R4: octyl(1) or 2-ethylhexyl(1)
AnE-PV-stat4	X	R1, R3: octyl(1) or methyl(1); R2, R4: octyl(1) or 2-ethylhexyl(1)
AnE-PV-stat5	X	R1, R3: octyl(1) or 2-ethylhexyl(1) or methyl(1); R2, R4: octyl(1) or 2-ethylhexyl(2)

Figure 1. Molecular structure of polymers investigated in this study. For the random polymers, the ratio of each side chain type is indicated in parenthesis.

for poly(*p*-phenylene-vinylene)-based materials, have been already demonstrated for this class of conjugated polymers.^[8]

The AnE-PV polymers here investigated (Figure 1), with the same conjugated backbone, differ in the nature of the grafted solubilizing alkoxy side chains (linear or branched), and possess either well-defined or randomly distributed side chains (random polymers).^[7] Substituents can affect the electronic properties of conjugated polymers,^[9,10] other than modifying their processability in organic solvents. Depending on their nature, size, and position, substituents can influence the molecular packing of polymer chains, thus greatly affecting charge transport properties of polymer films. In order to clearly establish the relationship between chemical structure and charge carrier mobility for the investigated polymers, the polymer films were prepared in the same way and the same technique, time-of-flight (TOF), was used for their investigation. Indeed, TOF^[11] considered as the “direct” method for the investigation of bulk transport properties in disordered materials, has two great advantages over other common techniques for the investigation of charge carrier mobility in organic film: i) it is an injection-free method, thus not limited by the charge-injection capability of the device contacts; ii) it allows a facile selection of the sign of charge carriers, thus enabling the measurement of both hole and electron mobility on the same sample.

2. Experimental Section

The synthesis of the polymers here investigated has been already reported in the literature.^[6,7,12] The number-average molec-

ular weights of the investigated polymer samples were 40 000, 13 300, 16 000, 41 200, 9100 and 7500 g mol⁻¹, while the polydispersities were 3.54, 1.97, 2.98, 2.01, 3.30, and 4.00 for AnE-PV-ab, AnE-PV-ae, AnE-PV-bb, AnE-PV-stat, AnE-PV-stat4, and AnE-PV-stat5, respectively. Polymer films for TOF experiments were drop-casted onto ITO-coated (ITO is indium-tin-oxide) glass substrates from chlorobenzene solutions (ranging between 15 and 35 g L⁻¹ for the six polymers), stirred for 4 d at 45–50 °C. After the deposition, the films were solvent-vapor annealed (chlorobenzene) overnight. The device structure was completed with a vacuum-evaporated semitransparent aluminum electrode (18 nm thick). The device area was 0.25 cm² and the film thickness 9.4, 1.4, 2.9, 3.6, 7.0, and 15.5 μm for AnE-PV-ab, AnE-PV-ae, AnE-PV-bb, AnE-PV-stat, AnE-PV-stat4, and AnE-PV-stat5, respectively.

A nitrogen laser ($\lambda = 337$ nm) with a pulse duration of 6–7 ns was used to photogenerate charge carriers in TOF experiments. A variable DC potential was applied to the samples and, in order to ensure a uniform electric field inside the device, the total photogenerated charge was kept less than 0.1 CV (where C is the sample capacitance and V is the applied potential) by attenuating the laser beam intensity with quartz neutral filters. The photocurrent was monitored across a variable load resistance by using a Tektronix TDS620A digital oscilloscope. TOF measurements were performed at room temperature and under dynamic vacuum (10⁻⁵ mbar).

Polymer films for X-ray diffraction (XRD) investigation were drop-casted from the same chlorobenzene solutions used for the preparation of TOF samples. Mineral quartz “zero background” was used as substrate (The Gem Dugout, State College, PA, USA), in order to strongly minimize its contribution to the total scattering. After the deposition, the films were solvent-vapor annealed (chlorobenzene) overnight. XRD analysis was carried out by means of a PANalytical X’Pert diffractometer equipped with a copper anode ($\lambda_{\text{mean}} = 0.15418$ nm) and a fast X’Celerator detector, with a step of 0.05° (2 θ) and counting time of 120 s/step. The films were directly investigated in reflection geometry.

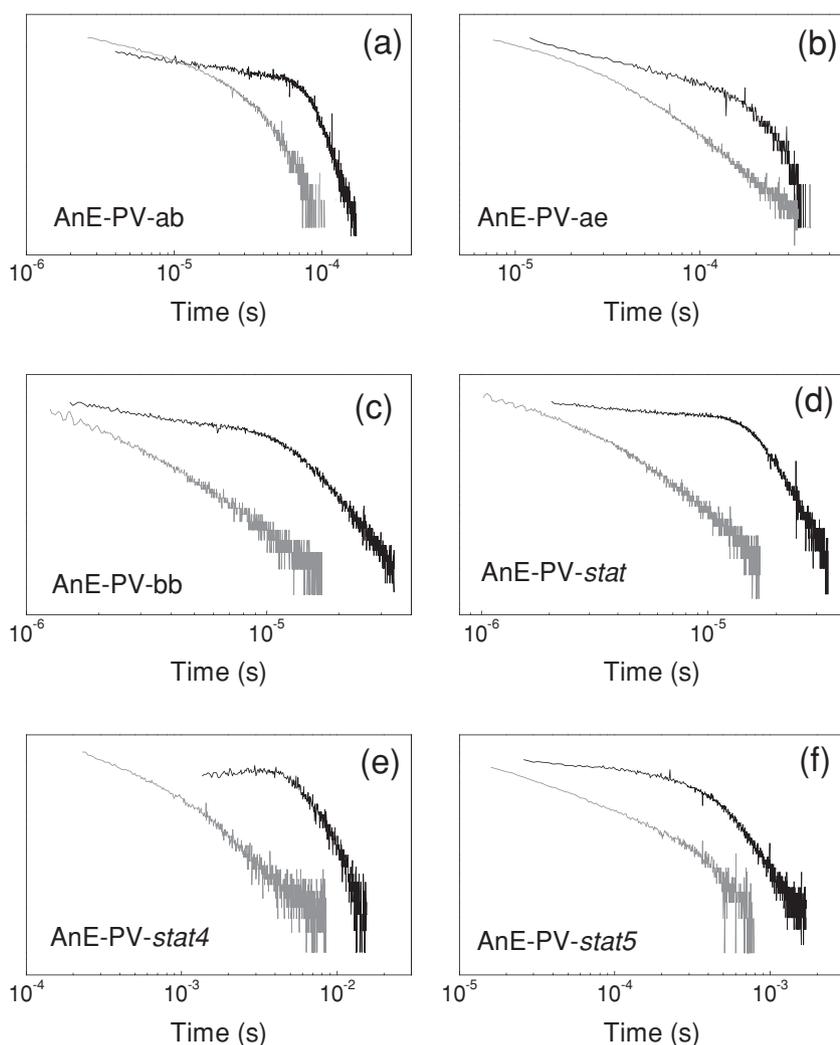


Figure 2. Typical TOF signals in log–log scales for an applied electric field of about $1 \times 10^5 \text{ V cm}^{-1}$. Positive carriers (black lines) and negative carriers (gray lines).

3. Results and Discussion

The drift mobility of charge carriers was studied as a function of the applied electric field (E) by using differential small-signal-mode TOF.^[11] The TOF experiments were performed on sandwich-type devices with the structure glass/ITO/polymer/Al, with the top aluminum layer being semitransparent. For a better comparison of the charge transport properties of the investigated AnE-PVs, the same conditions were used for the deposition of all polymer films. Transport of charge carriers is indeed highly affected by the organization of polymers chains in the solid state, depending on the used solvent, on the deposition technique and deposition parameters, as well as on post-deposition treatments.^[13,14] For this reason, the polymer layers were drop-casted from solutions prepared from the same solvent and treated in the same way onto the glass/ITO substrates and underwent solvent-vapor annealing

overnight, in order to allow an optimum organization of polymer chains through a very slow-drying process of the films. The sign of charge carriers generating the TOF photocurrent signals was selected by appropriately biasing the illuminated electrode (Al for holes, positively biased, and ITO for electrons, negatively biased).

Typical photocurrent transients, both for holes and electrons, are displayed in Figure 2 in a double-logarithmic representation and for a comparable applied electric field of about $1 \times 10^5 \text{ V cm}^{-1}$. The comparison of the two signals of Figure 2a–f confirms the usual finding that transport of electrons is more dispersed than that of positive carriers in conjugated polymers.^[15] This is commonly attributed to trapping effects by typical impurities, acting as trapping states for negative carriers, because close in energy to the polymer lowest unoccupied molecular orbital (LUMO) level.^[16,17] Though the dispersion of photocurrent signals, an inflection point was visible also for electrons in the double-logarithmic plots, from which the transit time (τ) of charge carriers can be evaluated.^[18] The inspection of Figure 2a–f also suggests that this class of conjugated polymers shows an ambipolar behavior, as already reported for AnE-PV-stat,^[19] with the photocurrent due to negative carriers decreasing even more rapidly than that of holes

and indicating that the time required for electrons to travel through the same sample is not longer than that for holes.

The values of charge carrier mobility were calculated as a function of the applied field through the well-known expression $\mu = d/\tau E$ where d is the thickness of the polymer layer, and with the transit times extracted from the TOF signals by using the same method in all cases, that is from the inflection point observed in the double-logarithmic representation. By plotting the logarithm of mobility as a function of the square root of E , a good linear trend was obtained in most cases, as shown in Figure 3, indicating a Poole–Frenkel behavior:^[20,21]

$$\mu(E) = \mu_0 \exp(\beta\sqrt{E}) \quad (1)$$

where μ_0 denotes the mobility at zero-field and β is the parameter describing how strong is the dependence of μ_0

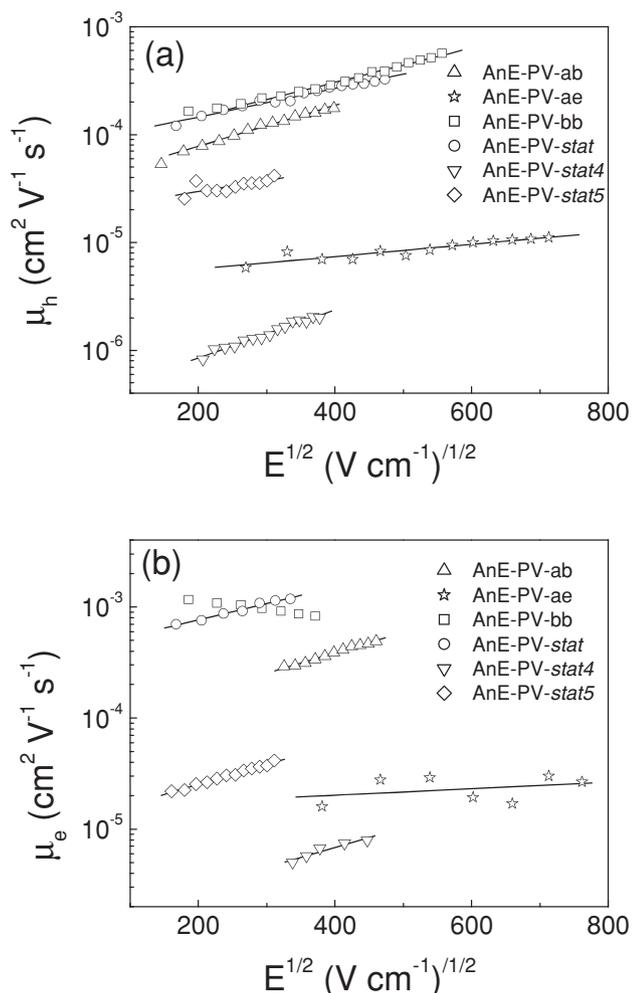


Figure 3. a) Hole mobility and b) electron mobility as a function of the square root of the electric field for: AnE-PV-ab (up triangles); AnE-PV-ae (stars); AnE-PV-bb (squares); AnE-PV-stat (circles); AnE-PV-stat4 (down triangles); AnE-PV-stat5 (diamonds). The lines are the linear fit to the experimental data.

on the electric field. The parameters obtained from the Poole–Frenkel fit to mobility data of Figure 3 are collected in Table 1. The only decreasing trend with E was observed for electron mobility, μ_e , in AnE-PV**bb** films (Figure 3b). Both the Poole–Frenkel-like trend of mobility, often observed for disordered organic materials, and a decreasing μ with E can be explained within the same model of an hopping conduction in a Gaussian distribution of localized states, as demonstrated by Monte Carlo simulations.^[22,23] Indeed, depending on the relative contribution of energetic (diagonal) and positional (off-diagonal) disorder to the random walk of charge carriers, a Poole–Frenkel-like behavior or a change of sign of the field-dependence of mobility can be observed.

The data of Figure 3 and Table 1 clearly show the great effect of lateral chains on charge carrier mobility

of conjugated polymers, with μ varying by two orders of magnitude for the AnE-PVs here considered, both for holes (ranging between 1.6×10^{-6} and 2.3×10^{-4} $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ for E of about 10^5V cm^{-1}) and electrons (between 5.0×10^{-6} and 1.2×10^{-3} $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ for the same field). Apart from the case of AnE-PV-*stat5*, for which comparable hole and electron mobilities were obtained, the mobility of negative charge carriers was found to be higher than that of holes from two- to sixfold, as shown in Table 1, confirming the good ambipolar behavior of this class of conjugated polymers. As expected, the polymer with the longest substituents (AnE-PV-*ae*, with octyl and dodecyl chains) shows low mobility compared with the other ones, consistent with the reduction of the electronic interaction between conjugated backbones^[9,10] as the extension of later chains increases. This is also confirmed by the comparison between AnE-PV-*ab* and AnE-PV-*bb*, only differing for the octyl side chains. The latter, with only 2-ethylhexyl substituents, shows higher mobility values (2.3×10^{-4} vs. 1.3×10^{-4} $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ for holes, 9.1×10^{-4} vs. 2.9×10^{-4} $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ for electrons, for the field of about 10^5V cm^{-1}). However, the most striking difference of AnE-PV-*ae*, compared with the other polymers, is represented by the low dependence of mobility on electric field, as demonstrated by the lowest values of β (Table 1). This could be due to a lower energetic disorder^[24] in the fluctuation of the energy of the hopping sites for charge transport, which could be attributed to a more ordered arrangement of polymer chains in the film. Indeed, a layered structure consisting of π - π -stacked backbones has been already reported for films made of AnE-PVs with all-linear side chains attached close to the anthracenylene-ethynylene unit, in contrast to the more amorphous structure of polymers with branched lateral chains attached to the same backbone.^[25] The other five polymers, all bearing branched 2-ethylhexyl chains, show a β value ranging between 3.1×10^{-3} and 5.1×10^{-3} $\text{cm}^{1/2} \text{V}^{-1/2}$, without a clear trend with the molecular structure.

The comparison between statistical and non-statistical polymers can be done by considering AnE-PV-*ab* and AnE-PV-*stat*, bearing the same octyl and 2-ethylhexyl side chains. Better values both for mobility and β were obtained for the polymer with lateral chains statistically distributed, confirming the superior features of the random polymer compared to the counterpart based on well-defined side chain.^[12] Finally, looking at the data of Table 1, it is surprising the difference of roughly one order of magnitude between the mobility values of AnE-PV-*stat4* and AnE-PV-*stat5*, two random polymers with the same side chains and just differing for the different amount of short methyl chains. The former, with more methyl groups in the molecular structure, shows lower values compared with AnE-PV-*stat5*, as well as the lowest ones for the six considered polymers, indicating that the

Table 1. Summary of mobility data: hole mobility (μ_h) and electron mobility (μ_e) for an applied electric field of about $1 \times 10^5 \text{ V cm}^{-1}$, zero-field mobility (μ_{0h} and μ_{0e} , for holes and electrons, respectively) and Poole–Frenkel factor (β_h and β_e , for holes and electrons, respectively).

AnE-PV	ab	ae	bb	stat	stat4	stat5
Side chains	octyl 2-ethylhexyl	octyl dodecyl	2-ethyl- hexyl	octyl 2-ethylhexyl	octyl 2-ethylhexyl methyl	octyl 2-ethylhexyl methyl
μ_h ($@ \approx 10^5 \text{ V cm}^{-1}$) [$\text{cm}^2 \text{ V}^{-1} \text{ s}^{-1}$]	1.3×10^{-4}	8.2×10^{-6}	2.3×10^{-4}	2.0×10^{-4}	1.6×10^{-6}	4.0×10^{-5}
μ_{0h} [$\text{cm}^2 \text{ V}^{-1} \text{ s}^{-1}$]	3.3×10^{-5}	3.8×10^{-6}	7.9×10^{-5}	7.9×10^{-5}	3.0×10^{-7}	1.3×10^{-5}
β_h [$(\text{V cm}^{-1})^{-1/2}$]	4.3×10^{-3}	1.6×10^{-3}	3.4×10^{-3}	3.1×10^{-3}	5.1×10^{-3}	3.6×10^{-3}
μ_e ($@ \approx 10^5 \text{ V cm}^{-1}$) [$\text{cm}^2 \text{ V}^{-1} \text{ s}^{-1}$]	2.9×10^{-4}	1.6×10^{-5}	9.1×10^{-4}	1.2×10^{-3}	5.0×10^{-6}	4.1×10^{-5}
μ_{0e} [$\text{cm}^2 \text{ V}^{-1} \text{ s}^{-1}$]	6.7×10^{-5}	1.5×10^{-5}	n.a.	3.9×10^{-4}	1.3×10^{-6}	1.1×10^{-5}
β_e [$(\text{V cm}^{-1})^{-1/2}$]	4.4×10^{-3}	6.7×10^{-4}	n.a.	3.4×10^{-3}	4.1×10^{-3}	4.2×10^{-3}

short methyl chains have a detrimental effect on the transport properties of charge carriers. It is worth noting that the two polymers with methyl side chains are among the ones exhibiting the lowest mobilities (Figure 3 and Table 1) of the six considered, confirming that the shortest lateral chains prevent a favorable organization of polymer films for the transport of charge carriers. To support this hypothesis, the XRD pattern of AnE-PV-*stat4* film is compared to that of AnE-PV-*stat*, without methyl chains and showing very good mobility values. The polymer films, prepared in the same conditions used for mobility investigation, showed very different XRD patterns (Figure 4). Differently from AnE-PV-*stat*, showing a semicrystalline character, AnE-PV-*stat4* reveals its complete amorphous feature. Indeed, AnE-PV-*stat* sample shows a crystalline peak at 5.53° (2θ), corresponding to a staking interlayer distance of 1.53 nm, while only a bell-shaped profile is

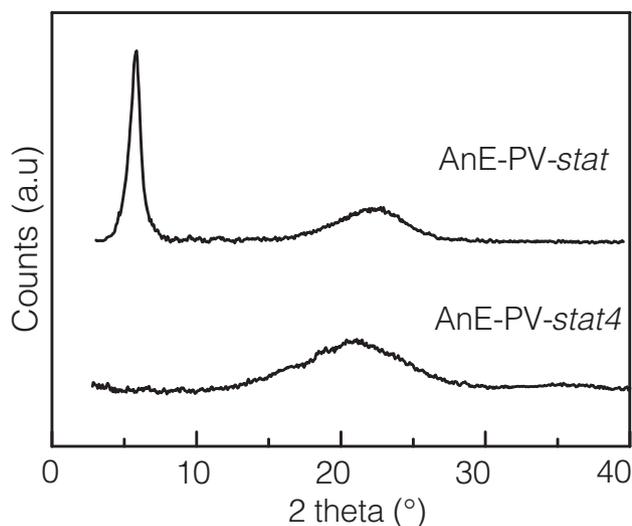


Figure 4. XRD patterns of AnE-PV-*stat* and AnE-PV-*stat4* films drop-cast onto quartz substrate.

visible in the wide angle region of the XRD pattern of AnE-PV-*stat4*, also present in the pattern of AnE-PV-*stat* and related to the amorphous component of the investigated films. Likely, the short methyl side chains, reducing the overall rough symmetry of the repeating unit, could hinder the possibility of a regular stacking of the polymer main chains. This leads to a very different organization in the solid state and highly affects the charge transport properties of the films made with the two different polymers, with a variation of two orders of magnitude in the mobility values. It is worth noting that also the photo-physical properties of the two statistical polymers are greatly influenced, with AnE-PV-*stat* showing improved absorption and emission spectra compared to the amorphous AnE-PV-*stat4*.^[7]

The very different transport properties of AnE-PV-*stat* and AnE-PV-*stat4* could be also attributed to a different extent of charge trapping processes. In this case, a different trend of charge carrier transit times with the applied electric field should be observed. It has been shown,^[26] that in a multiple trapping model, the transit time of charge carriers exhibits the electric field dependence of $\tau(E) \propto E^{-1/\alpha}$, where α is a dispersion parameter ($0 < \alpha < 1$; $\alpha = 1$ for non dispersive transport) introduced by Scher and Montroll^[18] in their model for the description of dispersive transport in amorphous solids. The electric field dependence of transit times derived from the TOF measurements are reported in Figure 5 for AnE-PV-*stat* and AnE-PV-*stat4*. From the slope of the lines representing the linear fit to the experimental data, the values of the dispersion parameter were extracted. 0.57 and 0.55 were obtained for AnE-PV-*stat4*, for holes and electrons, respectively, compared with the expected higher values for α of 0.68 (holes) and 0.71 (electrons) calculated for AnE-PV-*stat* and indicating that charge transport is less affected by charge trapping events in this latter polymer.

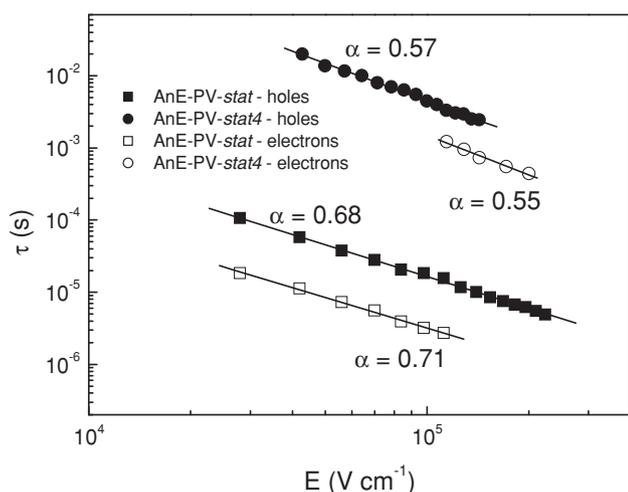


Figure 5. Transit time of holes (filled symbols) and electrons (open symbols) as a function of the electric field for AnE-PV-stat (squares) and AnE-PV-stat4 (circles).

4. Conclusion

A systematic investigation of charge carrier mobility in a series of anthracene-containing poly(*p*-phenylene-ethynylene)-*alt*-poly(*p*-phenylene-vinylene)s, differing for the solubilizing alkoxy lateral chains, has been performed, in order to establish a clear relationship between molecular structure and charge transport properties. The ambipolar behavior of this class of conjugated polymers is confirmed, with transit times of electrons even higher than those of holes, but with a higher dispersion of the photocurrent signals generated by the drift of negative carriers. The nature of the substituents has a great impact on charge carrier mobility of the investigated polymers, with a variation observed of 2–3 orders of magnitude. Long octyl and dodecyl linear chains have a detrimental effect on μ , likely because of a less compact molecular packing resulting in a decreased interaction between adjacent hopping sites. Similarly, short methyl substituents induce low mobility values, but because of a highly disordered arrangement of polymer chains in the solid phase, also leading to an increased charge trapping. A decreasing mobility with the amount of methyl chains in the molecular structure is clearly observed.

Finally, the used experimental conditions, allowing for a straight comparison of the investigated films, definitely confirmed the better properties of random polymers, compared to the counterpart based on a well-defined side chain.

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