

Bipolar Transport in Molecularly Doped Polymers*

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Hole and electron mobilities have been measured in ternary solid solutions containing an electron acceptor, 4H-1,1-dioxo-4-dicyanomethylidene-2-*p*-tolyl-6-phenylthiopyran, and an electron donor, tri-*p*-tolylamine, in a polyester host. The results are discussed by a model based on disorder, due to BäSSLer and coworkers. The key result of this study is that the presence of the donor has no effect on electron transport, nor does the presence of the acceptor have any effect on hole transport. The interpretation of the experimental results leads to the conclusion that the electron and hole manifolds are independent and are not influenced by the presence of the transport states of the oppositely charged carriers.

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Introduction

Molecularly doped polymers contain a strong electron donor or acceptor in a host polymer. In these materials, hole or electron transport occurs by charge transfer between adjacent donor or acceptor molecules, respectively. This can be described as a one-electron donor–acceptor or oxidation–reduction process between chemically identical moieties.^{1–3} Due to the widespread use of doped polymers as xerographic photoreceptors,^{4–11} there has been considerable interest in transport phenomena in these materials during the past two decades.

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Mobilities of doped polymers are very low, strongly field- and temperature-dependent, and also dependent on the donor or acceptor molecule and the host polymer. Whereas there have been many studies of hole or electron transport in doped polymers (for reviews, see Refs. 12–17), there have been few studies of bipolar transport. With the exceptions of the early work of Gill^{18–20} and the more recent studies of Yamaguchi²¹ and Yamaguchi et al.,^{22–24} we are not aware of any published work on bipolar transport in doped polymers.²⁵ Because of the relevance of bipolar transport to single-layer xerographic photoreceptors, however, this subject is of considerable technological importance.

In this study, we have investigated a polyester (PE) doped with an acceptor, 4H-1,1-dioxo-4-dicyanomethylidene-2-*p*-tolyl-6-phenylthiopyran (PTS), and a donor, tri-*p*-tolylamine (TTA). The purposes of this study are to compare the general features of bipolar transport with unipolar transport and to ascertain the physical origin of the differences. The results are described within the framework of a formalism based on disorder, due to BäSSLer and coworkers.^{26–29}

The Disorder Formalism

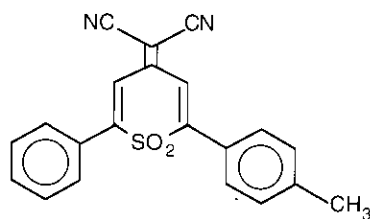
In the disorder formalism, charge propagation occurs by hopping through a manifold of localized states with superimposed energetic and positional disorder. The principal assumptions of the formalism are: (1) the distributions of hopping site energies and distances are Gaussian; (2) the hopping rates can be described by an expression due to Miller and Abrahams,³⁰ originally proposed to describe low-temperature impurity hopping in semiconductors; (3) electron–phonon coupling is sufficiently weak that polaronic effects can be neglected, yet strong enough to guarantee coupling to a heat bath; and (4) the process is incoherent, characterized by a loss of phase memory after each hop. The Miller–Abrahams treatment is based on a single phonon approximation and assumes that hops down

in energy are not impeded by an energy-matching condition for dissipating the excess electronic energy. The analysis also implies that downward jumps are not accelerated by an electric field. The validity of using the Miller-Abrahams approach for calculating hopping rates in disordered solids has been reviewed by Emin³¹ and by Kenkre and Dunlap.³² The key predictions of the formalism are the field and temperature dependencies of the mobility and the temperature dependence of the field dependencies of the mobility.

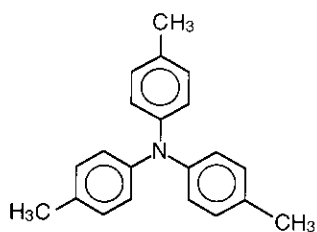
At high fields, the formalism predicts³³

$$\mu(\hat{\sigma}, \Sigma, E) = \mu_0 \exp\left[-\left(\frac{2\hat{\sigma}}{3}\right)^2\right] \exp[C(\hat{\sigma}^2 - \Sigma^2)E^{1/2}], \quad (1)$$

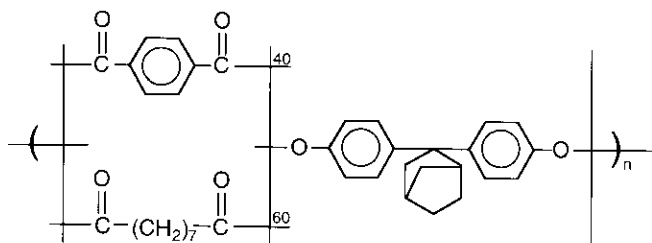
where μ is the mobility, σ the variance of hopping site energies (DOS), $\hat{\sigma} = \sigma/kT$, Σ the degree of positional disorder, μ_0 a prefactor mobility, C an empirical constant, and the other symbols have their usual meanings. Equation 1 applies only for fields in excess of a few multiples of 10^5 V/cm and for $T_g > T > T_c$, where T_g is the glass transition and T_c the low temperature nondispersive-to-dispersive transition temperature.³⁴ At low fields, simulations predict³⁵ that the mobility becomes field-independent or, in the case of large Σ , increases as the field is further reduced. Predictions of Eq. 1



PTS



TTA



PE

Figure 1. Molecular structures of PTS, TTA, and PE.

agree with results reported for a wide range of donor- or acceptor-doped polymers,³⁶⁻⁴⁸ pendant group and main chain polymers,⁴⁹⁻⁵³ and vapor-deposited molecular glasses.^{36,54-62}

Experimental

The molecular structures of PTS, TTA, and PE are shown in Fig. 1. The synthetic methods used for the preparation of these compounds have been described elsewhere.⁶³⁻⁶⁵ Samples were prepared by dissolving the appropriate ratios in dichloromethane, then coating the solutions on semi-transparent Ni-coated polyethylene terephthalate substrates that had previously been coated with a thin dispersion layer of oxotitanium tetrafluorophthalocyanine. To facilitate a comparison between PTS and TTA, the concentrations are expressed in molecular density units (the molecular weights of PTS and TTA are 358.1 and 287.1 g/mol, respectively). To prevent charge injection from the substrate electrode, a 0.02- μ m Nylon layer was interposed between the Ni and phthalocyanine layers. To reduce the residual solvent concentration, the samples were heated to 40°C for 2 h. From cross section photomicrographs and capacitance measurements, the doped polymer thicknesses were between 9.0 and 11.3 μ m. Finally, a 0.02- μ m SiO layer, followed by a 0.03- μ m Au layer, were vapor-deposited on the doped polymer free surface. During the vapor deposition processes, the samples were attached to a liquid N₂-cooled Cu block. Samples prepared in the above manner were amorphous, with no indications of crystallization over extended periods of time.

The mobility measurements were made by conventional time-of-flight techniques.⁶⁶ In the method used here, the displacement of a sheet of either electrons or holes, induced in the phthalocyanine layer, is time resolved. The photoexcitation was by 3-ns exposures of 440-nm radiation derived from a N₂-pumped dye laser (Laser Sciences, Inc., Model 337). The exposures were filtered such that the total charge injected into the sample was less than 5×10^{-2} CV, where C is the capacitance (typically 100 pF) and V the voltage. The field in the sample is thus assumed not to be perturbed by the injected charge and is given as V/L , where L is the sample thickness. The mobilities were determined from the conventional expression, $\mu = L^2/t_0V$, where t_0 is the transit time. The transit times were determined from the intersection of asymptotes to the plateau and trailing edge of the photocurrent transients, in double linear current versus time representation. The transients were measured with a transient digitizer (Tektronix, Model 2301). All measurements were made in air. Figure 2 is a schematic of the experimental arrangement.

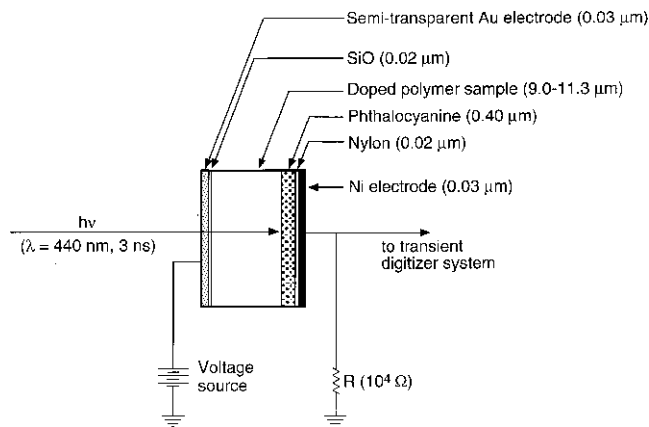


Figure 2. Schematic of the experimental arrangement. In the polarity shown, the photocurrents are due to electron displacement.

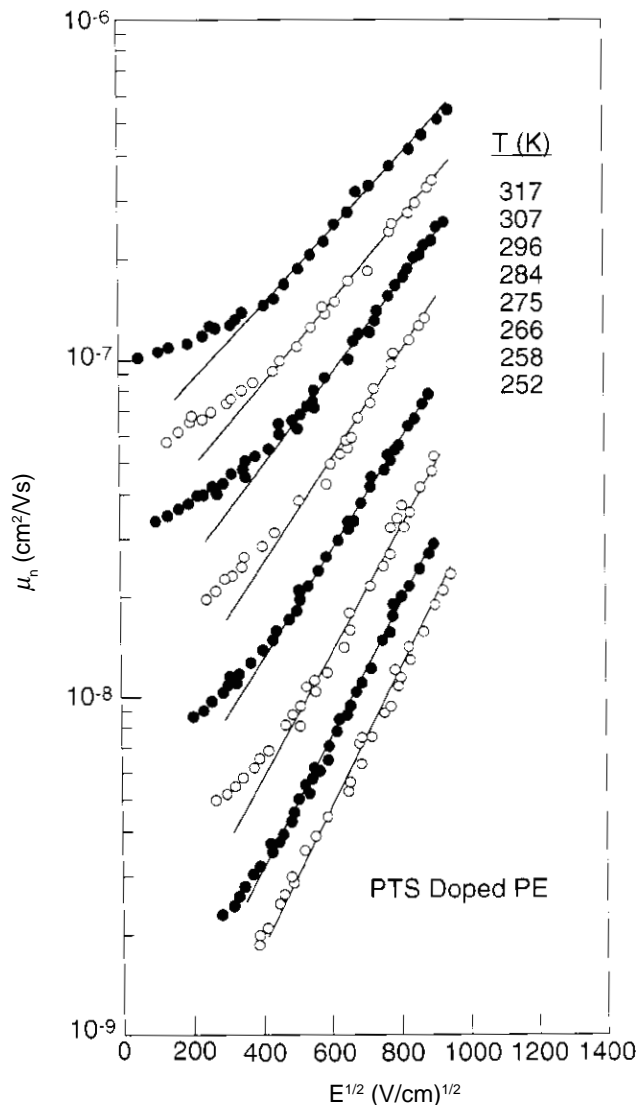


Figure 3. Log μ_n versus $E^{1/2}$, parametric in temperature, for a binary mixture of PTS and PE. The PTS concentration was 4.4×10^{20} mol/cm³ (26.4%).

Results

General Observations. Photocurrent transients in PTS:TTA-doped PE show features normally observed in donor- or acceptor-doped polymers: (1) an initial spike of a few tens of μs , (2) a plateau of variable length, and (3) a long tail. The temporal features of the photocurrent transients were the same for exposures incident on the Au or Ni electrodes. The width of the tail W can be described by an expression due to Schein,⁶⁷ $W = (t_{1/2} - t_0)t_{1/2}$, where t_0 is the time defined by the intersection of asymptotes and $t_{1/2}$ the time required for the photocurrent to decay to 1/2 of its value at t_0 . The width is weakly field- and temperature-dependent, increasing with increasing field and decreasing temperature. At 296 K and 10^5 V/cm, W is in the range of 0.45 to 0.50. The variable W is essentially the same for electron transients in PTS-doped PE and hole transients in TTA-doped PE. The incorporation of TTA has no noticeable effect on the tail broadening for electron transport, nor does the presence of PTS influence the broadening for hole transport. The range of temperatures for which photocurrent transient measurements could be made was determined by the glass transition at high temperatures and signal-to-noise problems at low temperatures. The field

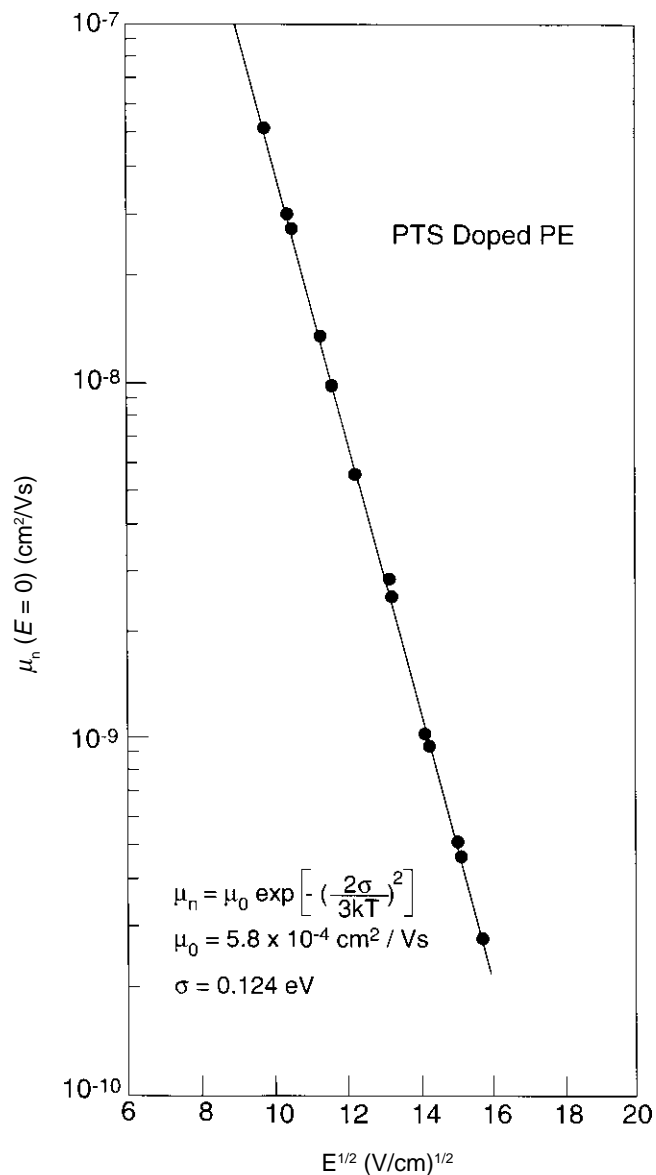


Figure 4. Log $\mu_n(E=0)$ versus T^{-2} for a binary mixture of PTS and PE. The PTS concentration was 4.4×10^{20} mol/cm³ (26.4%). The zero-field mobilities were obtained by the extrapolation of the data in Fig. 3 to $E=0$.

range was determined by dielectric breakdown at high fields and difficulties in resolving transit times at low fields. Over the range of temperatures and fields investigated, the photocurrent transients were reversible, with no indications of hysteresis.

Unipolar Transport in PTS- or TTA-Doped PE. Figure 3 shows the field dependencies of the mobility, parametric in temperature, for PTS-doped PE. At high fields, the results can be described as $\mu \propto \exp(\beta E^{1/2})$, where β is a constant that decreases with increasing temperature. At low fields and high temperatures, the mobility tends to approach a field-independent behavior. Figure 4 shows the temperature dependence of the zero-field mobility for PTS-doped PE, plotted semilogarithmically versus T^{-2} . Describing the data by Eq. 1 yields $\sigma = 0.124$ eV and $\mu_0 = 5.8 \times 10^{-4}$ cm²/Vs. The positional disorder parameter Σ can be determined from the temperature dependence of the field dependencies of the mobility. From Eq. 1, a plot of β versus $\hat{\sigma}^2$ should be linear with a slope of 2.9×10^{-4} (cm/V)^{1/2}.

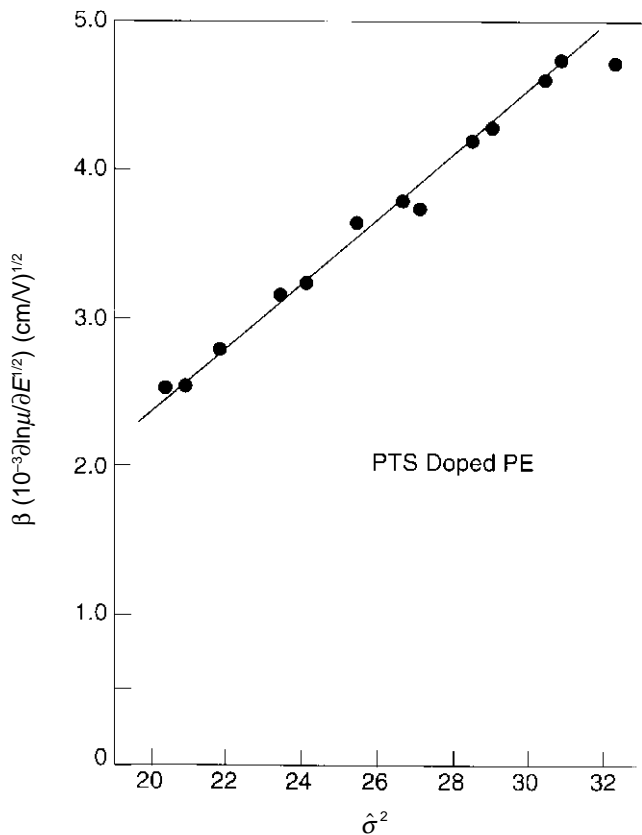


Figure 5. The variable β versus $\hat{\sigma}^2$ for a binary mixture of PTS and PE. The PTS concentration was 4.4×10^{20} mol/cm³ (26.4%). A value of 0.124 eV was used for σ , as derived from the data in Fig 4.

TABLE I. Electron and Hole Transport Parameters in PTS-, TTA-, and PTS:TTA-Doped PE

Electron Transport in PTS-Doped PE			
C_{PTS} (10 ²⁰ mol/cm ³)	σ (eV)	μ_0 (cm ² /Vs)	Σ
4.4	0.126	5.8×10^{-4}	2.6
3.3	0.122	1.9×10^{-4}	2.9
2.2	0.119	1.0×10^{-4}	3.4
1.8	0.121	6.0×10^{-5}	4.2
Hole Transport in TTA-Doped PE			
C_{TTA} (10 ²⁰ mol/cm ³)	σ (eV)	μ_0 (cm ² /Vs)	Σ
4.4	0.128	2.0×10^{-3}	3.1
3.3	0.122	9.0×10^{-4}	3.2
2.2	0.127	6.0×10^{-4}	3.9
1.8	0.119	1.8×10^{-4}	4.4
Electron Transport in PTS:TTA-Doped PE with Different Concentrations of TTA ($C_{PTS} = 4.4 \times 10^{20}$ mol/cm ³)			
C_{TTA} (10 ²⁰ mol/cm ³)	σ (eV)	μ_0 (cm ² /Vs)	Σ
4.4	0.118	6.0×10^{-4}	2.1
3.3	0.127	3.0×10^{-4}	2.3
2.2	0.121	5.0×10^{-4}	2.0
1.8	0.130	4.7×10^{-4}	2.0
Hole Transport in TTA:PTS-Doped PE with Different Concentrations of PTS ($C_{TTA} = 4.4 \times 10^{20}$ mol/cm ³)			
C_{PTS} (10 ²⁰ mol/cm ³)	σ (eV)	μ_0 (cm ² /Vs)	Σ
4.4	0.121	2.6×10^{-3}	2.3
3.3	0.122	2.9×10^{-3}	1.9
2.2	0.129	3.0×10^{-3}	2.4
1.8	0.120	1.9×10^{-3}	2.0

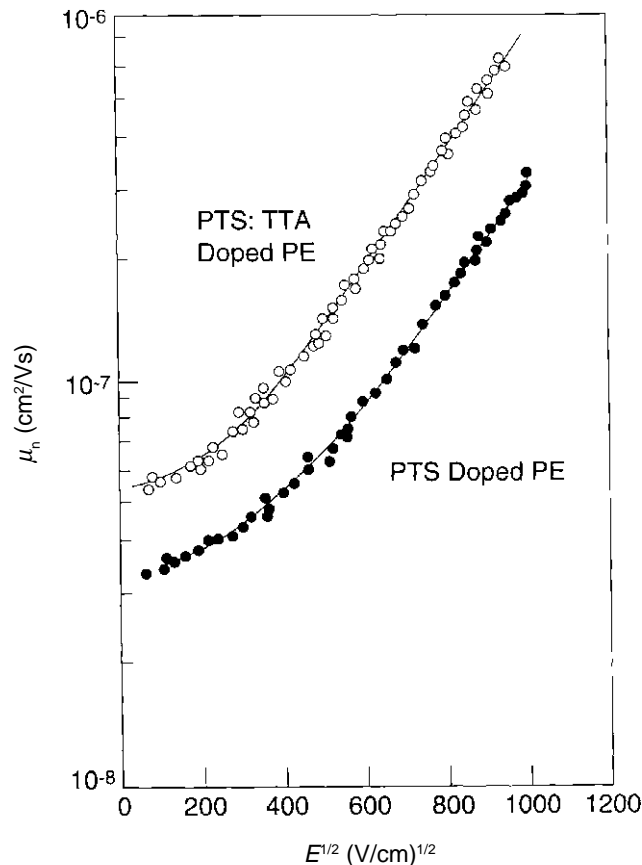


Figure 6. Log μ_n versus $E^{1/2}$ for a binary mixture of PTS and PE (solid circles) and a ternary mixture of PTS, TTA, and PE (open circles). The PTS and TTA concentrations were 4.4×10^{20} mol/cm³ ($C_{PTS} = 26.4\%$, $C_{TTA} = 21.0\%$). The temperature was 296 K.

Here $\beta = \partial \ln \mu / \partial E^{1/2}$. The variable Σ can then be determined from the $\beta = 0$ intercept from the condition $\hat{\sigma}^2 = \Sigma^2$. Figure 5 shows results for PTS-doped PE. Values of β were determined from the high field region of the data illustrated in Fig. 3. A value of 0.124 eV was used for σ , as derived from the data in Fig. 4. As predicted, a linear relationship between β and $\hat{\sigma}^2$ is observed. The slope in Fig. 5 is 2.2×10^{-4} (cm/V)^{1/2}, in acceptable agreement with the simulated value of 2.9×10^{-4} (cm/V)^{1/2}. From the $\beta = 0$ intercept, Σ is 3.0. In a similar manner, values of σ , μ_0 , and Σ were determined for hole transport in TTA-doped PE. The results are summarized in Table I.

Bipolar Transport in PTS- and TTA-Doped PE. Figure 6 compares the electron mobilities of PE doped with PTS and with a mixture of PTS and TPA. Figure 7 shows similar data for hole mobilities. Figure 8 shows electron and hole mobilities for PE doped with equal molecular concentrations of PTS and TTA. The effects of the PTS concentration on electron mobilities of PTS-doped PE and PTS:TTA-doped PE are illustrated in Figs. 9 and 10. Figures 11 and 12 show similar data for hole mobilities in TTA-doped PE and TTA:PTS-doped PE. In the manner described previously, values of σ , μ_0 , and Σ were determined for the ternary mixtures described in Figs. 9 to 12. Table I summarizes the results.

Discussion

From the results in Figs. 3 to 12, it may be concluded that the basic predictions of the disorder formalism are recovered. These predictions include the field and temperature dependencies of the mobility and the temperature

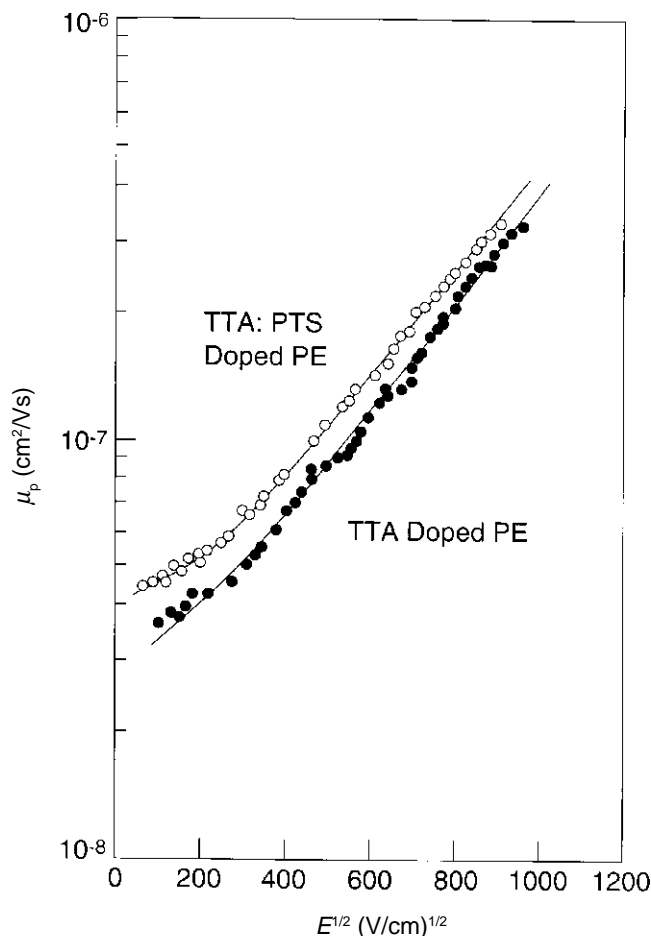


Figure 7. Log μ_p versus $E^{1/2}$ for a binary mixture of TTA and PE (solid circles) and a ternary mixture of TTA, PTS, and PE (open circles). The TTA and PTS concentrations were 4.4×10^{20} mol/cm³ ($C_{TTA} = 21.0\%$, $C_{PTS} = 26.4\%$). The temperature was 296 K.

dependence of the field dependencies of the mobility. In addition, the field and temperature dependencies of the tail-broadening parameter W are in qualitative agreement with simulations by Borsenberger, Pautmeier, and Bässler⁶⁸ and by Borsenberger and Bässler.⁶⁹ We thus conclude that the formalism provides a meaningful framework to describe transport phenomena in this class of materials. Values of σ , μ_0 , and Σ are in qualitative agreement with results reported previously for electron transport in PTS-doped polymers^{40,46} and hole transport in TTA-doped polymers^{41,43,44}. The principal result of this work is that the presence of the TTA donor has no significant effect on electron transport in PTS-doped PE, and, similarly, the incorporation of the PTS acceptor has no effect on hole transport in TTA-doped PE.

The key parameter of the disorder formalism is the width of the DOS. Figure 13 shows the width for electron transport in PTS- and PTS:TTA-doped PE. Figure 14 shows similar data for hole transport in TTA- and TTA:PTS-doped PE. These results show that the PTS and TTA manifolds are independent and not influenced by the presence of the transport states of the oppositely charged carriers. In the disorder formalism, the total width σ is determined by a dipolar component, σ_d , and a van der Waals component, σ_{vdw} . Assuming that the dipolar and van der Waals components are independent, the total width is

$$\sigma = (\sigma_d^2 + \sigma_{vdw}^2)^{1/2}. \quad (2)$$

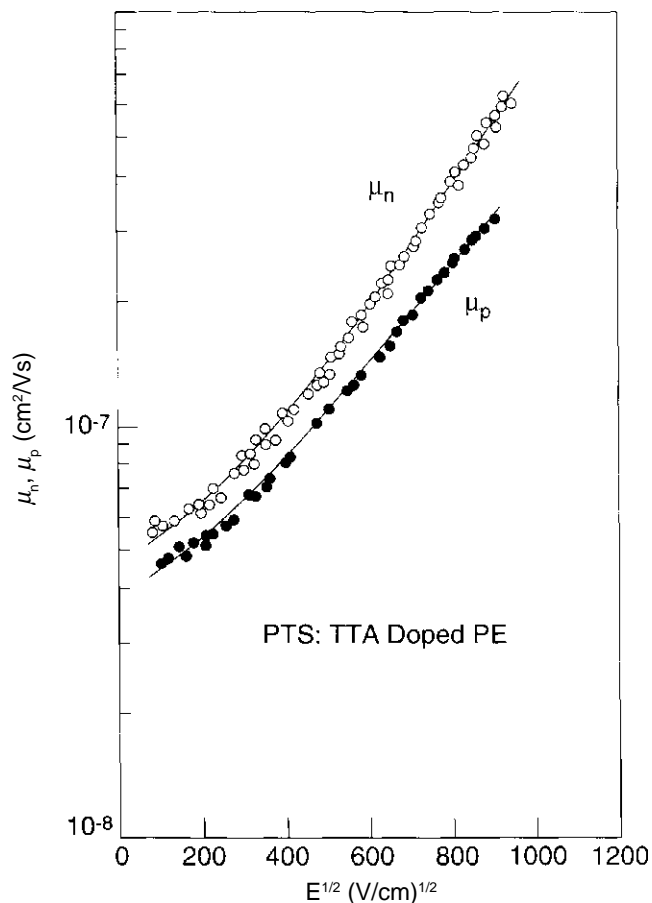


Figure 8. Log μ_n (open circles) and log μ_p (solid circles) versus $E^{1/2}$ for a ternary mixture of PTS, TTA, and PE. The PTS and TTA concentrations were 4.4×10^{20} mol/cm³ ($C_{PTS} = 26.4\%$, $C_{TTA} = 21.0\%$). The temperature was 296 K.

For a dopant concentration of 40%, previous work^{41–46} has indicated $\sigma_{vdw} = 0.070$ eV for a wide range of donor-doped polymers. Substituting $\sigma_{vdw} = 0.070$ eV into Eq. 2 requires $\sigma_d \sim 0.10$ eV for both electron and hole transport. The dipolar component is determined by dipole moments associated with the PTS and TTA molecules, as well as the carbonyl functionalities of the PE repeat unit. In principal, the dipolar components can be determined from expressions due to Dieckmann, Bässler, and Borsenberger,⁷⁰ and Young,⁷¹ or by the methods described by Novikov and Vannikov^{72,73} and Richert and Loring.⁷⁴ The limitation of these approaches is that they are premised on the assumption that the dipole distributions are random. In materials such as these, where there is considerable freedom of motion, it is likely that the dopant and polymer dipoles will align in such a way that the dipolar components will be reduced. The observation that the dipolar components for electron and hole transport are essentially the same, whereas the dipole moments of PTS and TTA are substantially different, suggests that the dipolar components are mainly due to the highly polar carbonyl groups of the PE repeat unit, rather than the PTS or TTA dopant molecules.

The prefactor mobilities are between 10^{-3} to 10^{-5} cm²/Vs, increasing with increasing dopant concentration. The prefactors for electron transport in PTS- and PTS:TTA-doped PE are in agreement with PTS-doped polycarbonate (PC),⁴⁰ 2-*t*-butyl-9,10-N,N'-dicyanoanthraquinone diimine (DCAQ)-doped PC and poly(styrene),³⁹ and 1-phenyl-3-((diethylamino)styryl-5-(*p*-(diethylamino)phenyl)pyrazoline (DEASP)-doped PS and PC.⁷⁵ DCAQ and DEASP are highly

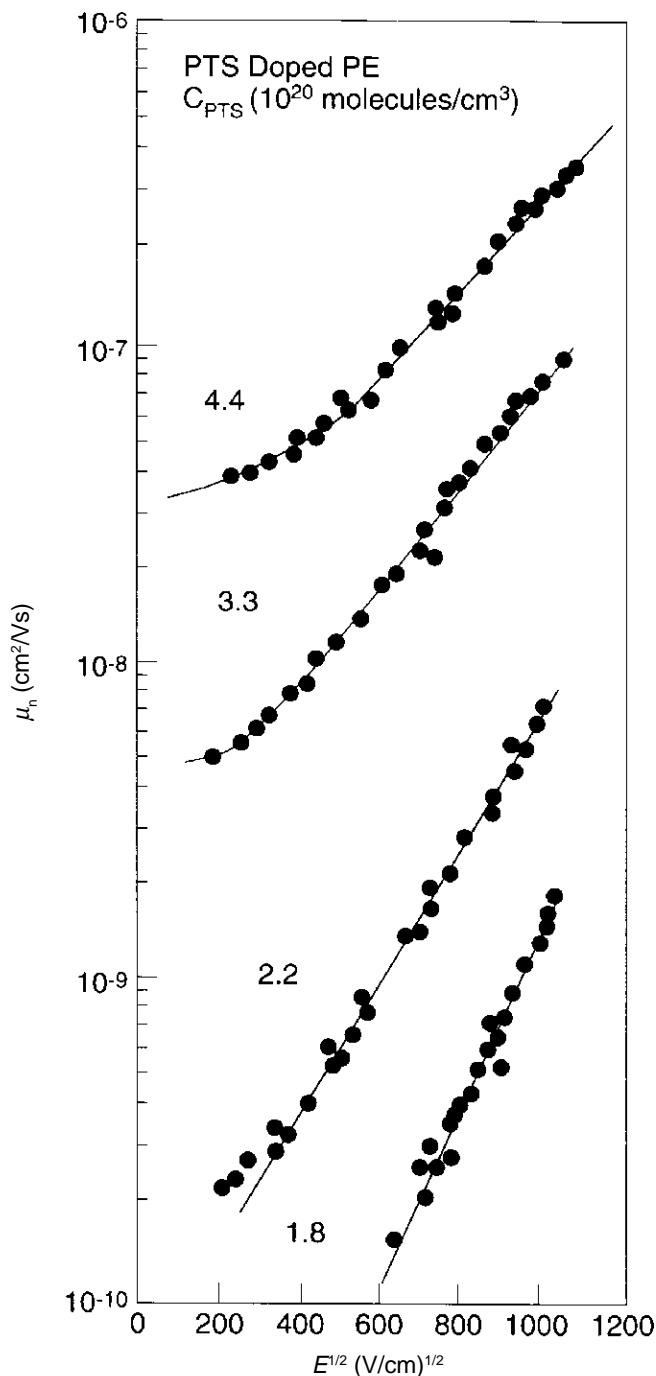


Figure 9. Log μ_n versus $E^{1/2}$ for PTS-doped PE containing different concentrations of PTS (10.8 to 26.4%). The temperature was 296 K.

polar compounds with dipole moments comparable to PTS. The prefactors for TTA- and TTA:PTS-doped PE, however, are approximately an order of magnitude less than values reported for TTA-doped PC⁷⁶ and 1,1-bis(di-4-tolylamino-phenyl)cyclohexane (TAPC)-doped PC.⁷⁷ TAPC is a weakly polar compound with a dipole moment comparable to that of TPA. These results suggest that the prefactors may be influenced by the polymer host. Finally, previous work has shown that the degree of positional disorder is strongly dependent on the donor or acceptor concentration, increasing with decreasing concentration. The values determined from this study agree with results reported for a wide range of donor- or acceptor-doped polymers. These observations suggest that positional disorder is largely of geometrical

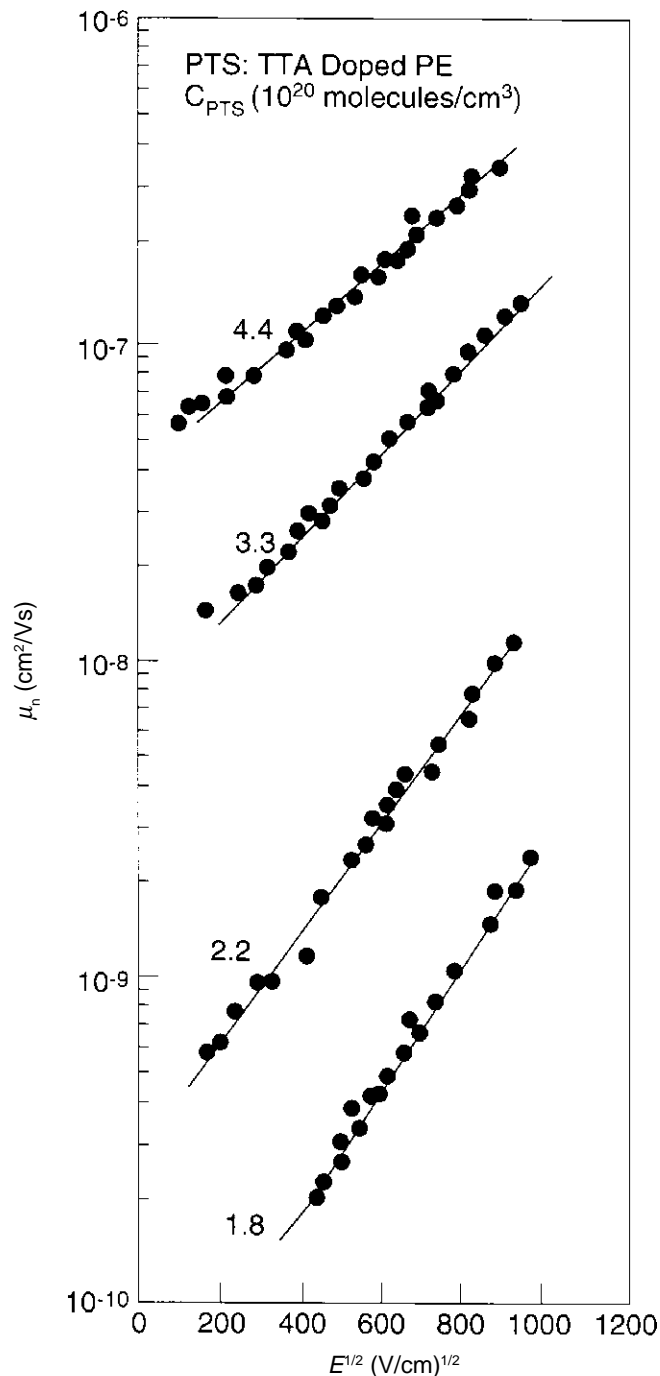


Figure 10. Log μ_n versus $E^{1/2}$ for PTS:TTA-doped PE containing different concentrations of PTS (10.8 to 26.4%). The TTA concentration was 4.4×10^{20} mol/cm³ (21.0%). The temperature was 296 K.

origin and not due to the molecular structure of the dopant molecule or polymer host. Positional disorder is usually attributed to packing constraints. \blacktriangle

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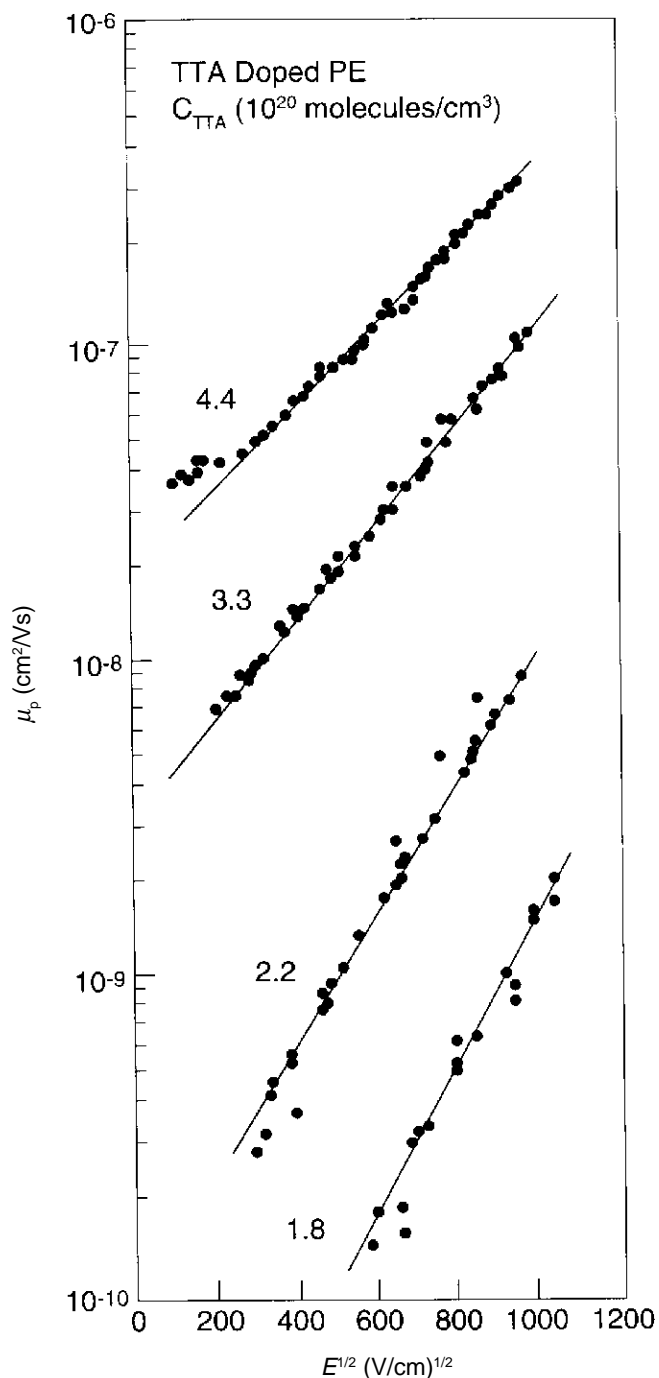


Figure 11. Log μ_p versus $E^{1/2}$ for TTA-doped PE containing different concentrations of TTA (8.5 to 21.0%). The temperature was 296 K.

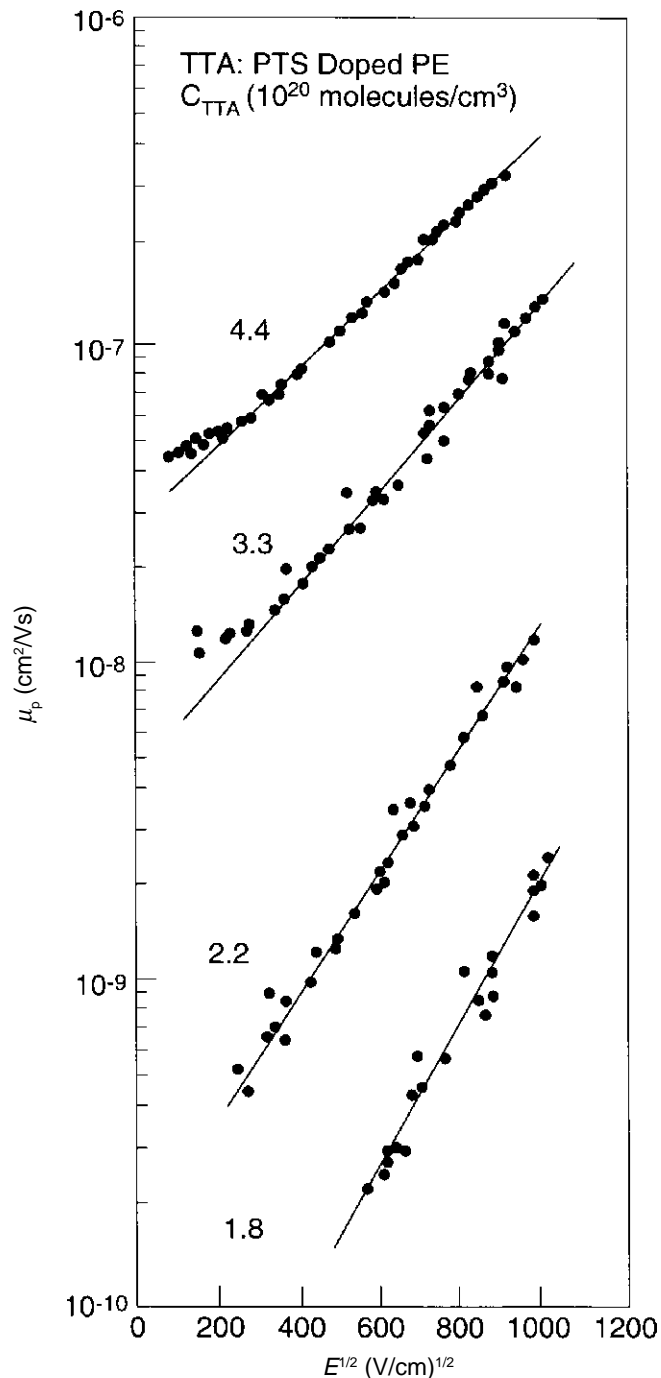


Figure 12. Log μ_p versus $E^{1/2}$ for TTA:PTS-doped PE containing different concentrations of TTA (8.5 to 21.0%). The PTS concentration was 4.4×10^{20} mol/cm³ (26.4%). The temperature was 296 K.

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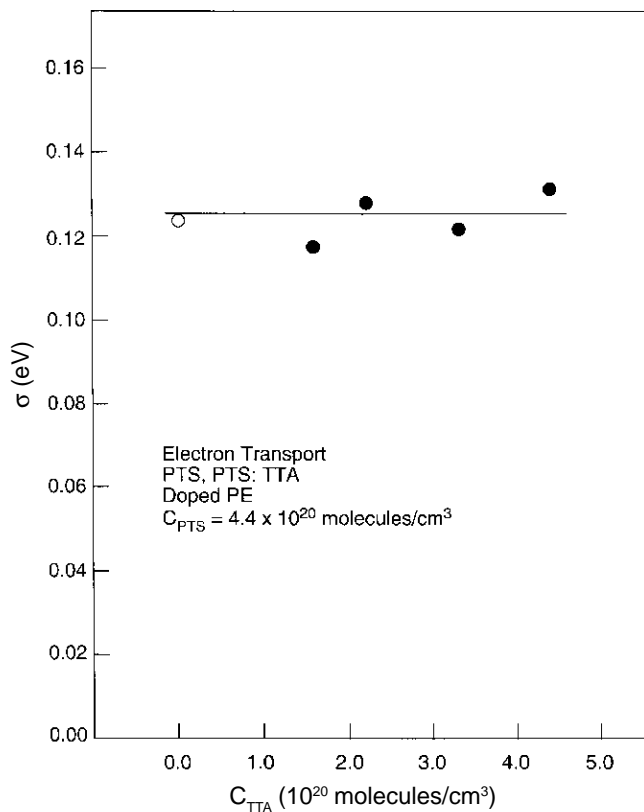


Figure 13. Width of the DOS for PTS-doped PE (open circle) and PTS-doped PE containing different concentrations of TTA (solid circles) (8.5 to 21.0%).

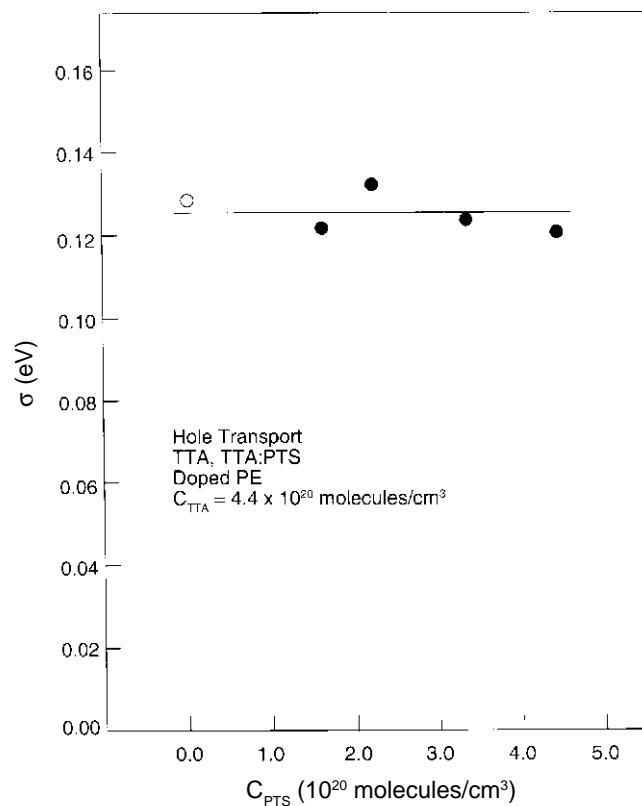


Figure 14. Width of the DOS for TTA-doped PE (open circle) and TTA-doped PE containing different concentrations of PTS (solid circles) (10.8 to 26.4%).

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