

Studies of Polymer Electrets. II. Factors Governing the Stabilities of Homoelectrets Obtained from Polystyrene and Its Derivatives

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Synopsis

Polystyrene and its various alkyl and halo derivatives have been studied as homoelectrets and their stabilities under different environmental conditions determined. The stabilities of the homoelectrets prepared from these polymers depended on their volume resistivities, glass transition temperatures, and interactions with electron-acceptor impurities. Excellent stabilities were exhibited by the homoelectrets prepared from polystyrene, poly(vinyltoluene), poly(chlorostyrene), and poly(*t*-butylstyrene). It is postulated that the charged particles are trapped by the π -orbitals of the phenyl rings present in these polymers and that they are adversely affected by the presence of electron-acceptor compounds that generally form charge-transfer complexes with the phenyl groups.

INTRODUCTION

Polystyrene is a common polymeric material possessing a high value of volume resistivity (10^{18} ohm-cm) and a reasonably high glass transition temperature (T_g) of 100° . For this reason, it is extensively used as an insulator.¹ Studies of polystyrene electrets by thermally stimulated discharge (TSD) methods² have indicated that the phenyl groups in this polymer may be responsible for charge trapping, and these electrets are not totally discharged until heated to 125° .

The nonpolar character of polystyrene is reflected in its ability to absorb only a small amount of water (about 0.05–0.20%). Since the homoelectrets of other nonpolar polymers such as polyolefins and polyfluoroolefins have been found to be stable in humid atmospheres,³ it was expected that the polystyrene homoelectrets would behave in a similar manner.

As we have noticed before,³ thermally stimulated techniques provide information on thermal processes affecting an electret. Therefore, it is difficult to understand from these studies how an electret would behave under humid conditions in which charge decay occurs in a manner not related to thermal processes. In most cases, the effect of humidity on electrets has to be determined separately before a polymer can be considered as a promising electret material.

Initially, we studied electrets prepared from commercial polystyrene. Although the electrets were quite stable under dry conditions, they rapidly discharged under 98% relative humidity at room temperature. This was quite unexpected, and we attributed this to the presence of impurities in the commercial polystyrene. To check this, we prepared polystyrene in the laboratory and purified it by standard procedures. As expected, the electrets prepared from this material were stable even under humid conditions.

The above observation indicated that a systematic study of charge stability versus structure in polystyrene would be useful in determining the important variables that govern electret stabilities in this important class of polymers.

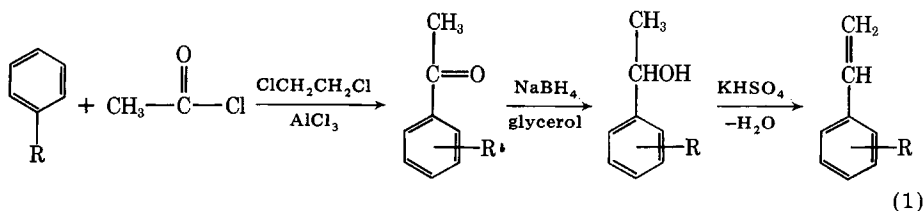
EXPERIMENTAL

Materials

The polymers listed in Table I were prepared and the electrets prepared from them were studied.

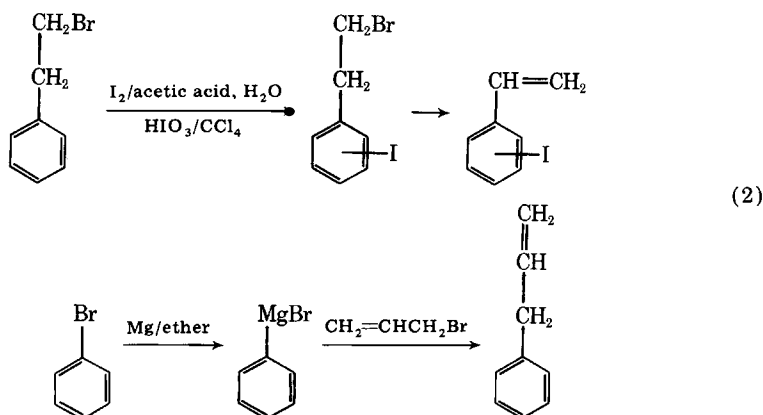
Monomers

Styrene, chlorostyrene, vinyltoluene and *t*-butylstyrene were commercial materials. They were freshly distilled just before polymerization. Ethylstyrene, *n*-butylstyrene, chlorostyrene, and bromostyrene were prepared according to the following reaction:



R being the appropriate substituent.

Iodostyrene⁵ and 3-phenyl-1-propene were prepared as shown below:



The physical properties of all the monomers agreed well with those reported in the literature.

Polymerization Reaction

Polystyrene, polystyrenes with alkyl and halogen substituents, and their copolymers were prepared by the emulsion polymerization technique described by Sorenson and Campbell.⁶ Poly(styrene-co- α -methylstyrene) (2:3) was prepared according to West.⁷ Poly(3-phenyl-1-propene), poly(3-cyclohexyl-1-propene), and poly(4-methyl-1-pentene) were prepared following the procedure described by us before.³ All these polymers were purified by dissolving in

benzene or chloroform followed by precipitation in methanol. They were found to have high molecular weights determined by measurements of their intrinsic viscosity values.⁹

Electret Preparation and Evaluation

Preparation, measurement of apparent surface charge density, and determination of the charge stabilities of the electrets obtained from these polymers were performed following the technique already described by us.³

Temperatures reported are in degrees Celsius.

RESULTS AND DISCUSSION

In our previous work,³ it has been established that in polyolefins, semicrystalline structures are important for imparting stability to their electrets, provided volume resistivity and crystalline dispersion temperatures ($T\alpha$) are reasonably high. In that context, we studied poly(4-methyl-1-pentene) and poly(3-cyclohexyl-1-propene). The difference in stabilities between the electrets of these two polymers was explained on the basis of semicrystalline character of poly(4-methyl-1-pentene) and amorphous character of poly(3-cyclohexyl-1-propene). It was considered that in semicrystalline polymers, the charged particles are trapped at the crystalline/amorphous interface where the vinyl endgroups in these polymers are predominantly present. It was speculated that the π -electrons of a vinyl group are involved in charge trapping. Since in general a fluoroolefin polymer has greater electret stabilities than a polyolefin with comparable physical properties, such as volume resistivity, $T\alpha$ temperature, and degree of crystallinity, it was further postulated that the n -orbitals associated with the fluorine atoms also participate in stabilizing electret charges.

Behavior of aromatic compounds is explained in general on the basis of π -molecular orbitals belonging to the phenyl rings. Most chemical substitution reactions in these compounds have been shown to proceed via initial complex formation of reactive intermediates with the π -molecular orbitals.¹⁰ Spectroscopic measurements such as ultraviolet spectra and nuclear magnetic resonance have amply shown the importance of the π -electrons present in this class of materials.¹¹ Therefore, we expected that these π -orbitals would also be involved in charge trapping by the polymers that contain phenyl and other aromatic groups.

Poly(3-phenyl-1-propene) has been shown to be generally amorphous like poly(3-cyclohexyl-1-propene).¹² Because of structural similarities between the two polymers, one would expect that poly(3-phenyl-1-propene) should give rise to unstable electrets, unless the phenyl group has stabilizing effects:

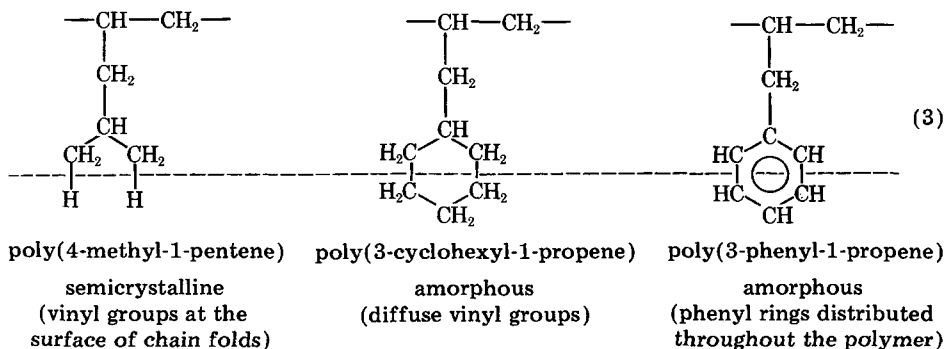


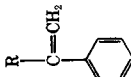




TABLE I
Polymers Studied in This Work

| Substituent R | Polymer | T_g , °C (Ref. 4) |
|---|--|---------------------|
| <p>A. Polystyrene with Alkyl Substitution in the Benzene Ring:</p> <p style="text-align: center;"> $\text{---CH---CH}_2\text{---}$  </p> | | |
| H | polystyrene | 100 |
| CH ₃ | poly(vinyltoluene) | 110 |
| C(CH ₃) ₃ | poly(<i>t</i> -butylstyrene) | 130 |
| CH ₂ CH ₃ | poly(ethylstyrene) | 27 |
| CH ₂ CH ₂ CH ₂ CH ₃ | poly(<i>n</i> -butylstyrene) | 6 |
| <p>B. Copolymers of styrene</p> <p style="text-align: center;"> CH=CH_2  </p> <p style="text-align: center;">with substituted styrene</p> <p style="text-align: center;">  </p> | | |
| Substituent R | Polymer | |
| CH ₃ | poly(styrene-co- α -methylstyrene) (-3) | |
| <p>C. Copolymers of Substituted Styrenes</p> <p style="text-align: center;"> CH=CH_2  </p> <p style="text-align: center;">and</p> <p style="text-align: center;"> CH=CH_2  </p> | | |

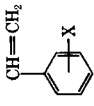
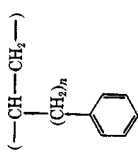
| Substituent R ₁ | Substituent R ₂ | Polymer |
|---|---|---|
| H | | |
| H | CH ₃ | poly(styrene-co-vinyltoluene) (1:1) |
| CH ₃ | C(CH ₃) ₃ | poly(styrene-co- <i>t</i> -butylstyrene) (1:1) |
| | C(CH ₃) ₃ | poly(vinyltoluene-co- <i>t</i> -butylstyrene) (1:1) |
| D. Polystyrenes with Halogen Substituents in the Benzene Ring | | |
| |  | |
| | Polymer | <i>T_g</i> , °C |
| Cl | | 110 |
| Br | | 137 |
| I | | 156 |
| E. Polyolefins with Phenyl Substitution at the Side Chain | | |
| <i>n</i> |  | |
| | Polymer | <i>T_g</i> , °C |
| I | | 60 |
| | poly(3-phenyl-1-propene) | |

TABLE II
Charge Decay Characteristics of Poly(4-methyl-1-pentene), Poly(3-cyclohexyl-1-propene), and Poly(3-phenyl-1-propene) Electrets^a

| Time, days | Poly(4-methyl-1-pentene), $T_g = 40^\circ$ | | Poly(3-cyclohexyl-1-propene), $T_g = 65^\circ$ | | Poly(3-phenyl-1-propene), $T_g = 60^\circ$ | |
|------------|--|---------|--|---------|--|---------|
| | Voltage, V | V/V_0 | Voltage, V | V/V_0 | Voltage, V | V/V_0 |
| 0 | 1600 | 1.00 | 1400 | 1.00 | 2000 | 1.00 |
| 1 | 1500 | 0.94 | 370 | 0.26 | 1600 | 0.80 |
| 4 | 1480 | 0.93 | 270 | 0.19 | — | — |
| 8 | — | — | 210 | 0.15 | 1300 | 0.65 |
| 20 | 1340 | 0.84 | — | — | — | — |
| 30 | 1280 | 0.80 | — | — | 900 | 0.45 |
| 40 | 1230 | 0.77 | — | — | — | — |

^a Environment: 50° , 100% relative humidity.

Table II gives the charge decay behavior of the electrets obtained from the above three polymers; the data are shown graphically in Figure 1. From the data given in Table II and Figure 1, it seems that the electrets derived from poly(3-phenyl-1-propene) are decidedly more stable than those from poly(3-cyclohexyl-1-propene).

Next, we prepared polystyrene by the emulsion technique⁶ and purified this material by dissolving it in reagent-grade benzene followed by precipitation in reagent-grade methanol. The electrets prepared from this material were very stable, as can be seen from Table III. This proves that the phenyl groups can stabilize charged species in polymer electrets.

Although the polystyrene electrets as shown in Table III are very stable, we soon discovered that the stability depends on how the polymer is purified. Thus, in Table IV, it is shown that when we used commercial benzene for reprecipitation, the stabilities of the electrets were significantly reduced, and the decrease in stability was directly dependent on the number of times purification of the

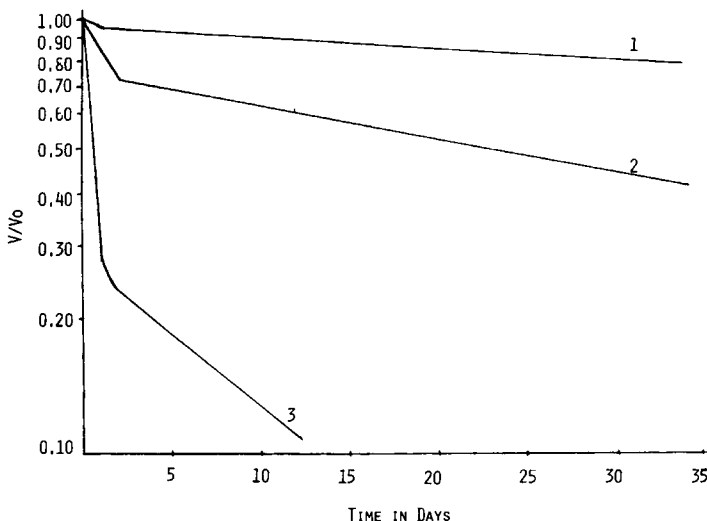


Fig. 1. Charge decay characteristics of electrets prepared from polyolefins with similar structures, at 50° , 100% relative humidity: 1, poly(4-methyl-1-pentene); 2, poly(3-phenyl-1-propene); 3, poly(3-cyclohexyl-1-propene).

TABLE III
Charge Decay Characteristics of Polystyrene Electrets

| Time, days | Room temperature, dry | | Room temperature, 100% relative humidity | | 50°, 100% Relative humidity | |
|------------|-----------------------|------------------|--|------------------|-----------------------------|------------------|
| | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ |
| 0 | 1380 | 1.00 | 1400 | 1.00 | 1360 | 1.00 |
| 3 | 1350 | 0.98 | 1360 | 0.97 | 1290 | 0.95 |
| 10 | 1300 | 0.94 | 1330 | 0.95 | 1260 | 0.93 |
| 17 | 1350 | 0.98 | 1290 | 0.92 | 1240 | 0.91 |
| 24 | 1370 | 0.99 | 1350 | 0.96 | 1230 | 0.90 |
| 38 | 1330 | 0.96 | 1220 | 0.87 | 1220 | 0.90 |
| 48 | 1370 | 0.99 | 1260 | 0.90 | 1110 | 0.82 |
| 123 | 1310 | 0.95 | 1130 | 0.81 | 1080 | 0.79 |

polymer was attempted through use of this commercial benzene solvent. This indicated that something present in the commercial benzene was being absorbed by the polystyrene during attempted purification, and this may be responsible for charge decay. Therefore, we tried to isolate this chemical from the commercial benzene. The material was found to be a mixture of organic phosphites and phosphates, presumably extracted from polyethylene liner used in the storage containers.

The effects of impurities are more drastic under humid conditions than under dry conditions. This suggests that the changes brought about by the impurities are related to the development in these polymers of polar sites that can interact with moisture. Of all the commercial solvents employed for the purification of polystyrene materials, chloroform was found to be the most suitable, presumably because it did not contain any deleterious impurities.

Since the polystyrene electret stabilities were found to be so susceptible to the presence of impurities, we decided to study the effects of addition of various electron-donor and electron-acceptor compounds at 1-2% concentrations. These

TABLE IV
Charge Decay Characteristics of Polystyrene Electrets^a

| Time, days | 50° Dry | | 50°, 100% Relative humidity | |
|------------|--------------------------|------------------|-----------------------------|------------------|
| | Voltage V | V/V ₀ | Voltage, V | V/V ₀ |
| | Precipitated Once | | | |
| 0 | 1550 | 1.00 | 1660 | 1.00 |
| 2 | 1550 | 1.00 | 1140 | 0.69 |
| 8 | 1510 | 0.97 | 890 | 0.54 |
| 29 | 1410 | 0.91 | 740 | 0.45 |
| 38 | 1390 | 0.90 | 730 | 0.44 |
| 77 | 1310 | 0.85 | 680 | 0.41 |
| | Precipitated Three Times | | | |
| 0 | 1640 | 1.00 | 1650 | 1.00 |
| 1 | 1700 | 1.04 | 330 | 0.20 |
| 8 | 1350 | 0.82 | 250 | 0.15 |
| 19 | 1280 | 0.78 | 190 | 0.12 |
| 33 | 1240 | 0.76 | — | — |
| 47 | 1190 | 0.72 | — | — |

^a Attempted purification by dissolving in commercial benzene, followed by precipitation in methanol.

TABLE V
 Charge Decay Characteristics of Electrets of Polystyrene Doped with Various Electron-Donor Additives.^a

| Time, days | No additive | | 1% Anthracene | | 2% α -Methylstyrene | | 1% Trioctylphosphite | |
|------------|-------------|---------|---------------|---------|----------------------------|---------|----------------------|---------|
| | Voltage, V | V/V_0 | Voltage, V | V/V_0 | Voltage, V | V/V_0 | Voltage, V | V/V_0 |
| 0 | 1400 | 1.00 | 1500 | 1.00 | 1600 | 1.00 | 1400 | 1.00 |
| 1 | 1300 | 0.93 | 1400 | 0.93 | 1500 | 0.94 | 1200 | 0.86 |
| 3 | 1260 | 0.90 | 1210 | 0.81 | 1360 | 0.85 | 1200 | 0.86 |
| 9 | 1230 | 0.88 | 1170 | 0.78 | 1220 | 0.76 | 1150 | 0.82 |
| 21 | 1120 | 0.80 | 980 | 0.65 | 1080 | 0.68 | 1110 | 0.79 |

^a Environment: 50°, 100% relative humidity.

TABLE VI
Charge Decay Characteristics of Electrets of Polystyrene Doped with Various Electron-Acceptor Additives^a

| Time, days | No additive | | 1% Iodine | | Tetracyanoethylene | | 1% Tributylphosphate | |
|---------------|-------------|------------------|------------|------------------|--------------------|------------------|----------------------|------------------|
| | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ |
| 0 | 1400 | 1.00 | 1400 | 1.00 | 1400 | 1.00 | 1500 | 1.00 |
| 1 | 1300 | 0.93 | 380 | 0.27 | 1030 | 0.74 | 700 | 0.47 |
| 3 | 1260 | 0.90 | — | — | — | — | — | — |
| 4 | — | — | 100 | 0.07 | 650 | 0.46 | 560 | 0.37 |
| 9 | 1230 | 0.88 | — | — | — | — | — | — |
| 11 | — | — | 0 | 0.00 | 430 | 0.31 | 480 | 0.32 |

^a Environment: 50°, 100% relative humidity.

TABLE VII
Charge Decay Characteristics of Poly(styrene-co- α -methylstyrene) (2:3) Electrets Under Various Environmental Conditions

| Time, days | Room temperature, 75% relative humidity | | 50°, Dry | | 50°, 100% Relative humidity | |
|------------|---|------------------|------------|------------------|-----------------------------|------------------|
| | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ |
| 0 | 1710 | 1.00 | 1730 | 1.00 | 1700 | 1.00 |
| 1 | 100 | 0.06 | 1500 | 0.87 | 100 | 0.06 |
| 2 | 30 | 0.02 | 920 | 0.53 | 30 | 0.02 |
| 8 | — | — | 500 | 0.29 | — | — |

compounds were dissolved in a large volume of analytical-grade solvent such as methanol or acetone, and the mixture was then added to the purified polystyrene to make a slurry. Most of the solvent was allowed to evaporate at ambient conditions, and the rest was removed at 40–50° under vacuum during 6 h. The electrets were prepared and studied in the usual manner. Tables V and VI summarize the results. It is obvious that electron-acceptor impurities have drastic effects on the stabilities of polystyrene electrets. Since polystyrene has electron-donor characteristics, this effect suggests that there is formation of charge-transfer structures which are responsible for charge decay and dramatic interaction with moisture in these electrets.

We next investigated the effects of alkyl substituents in the main chain and in the phenyl rings of polystyrene on electret charge decay. It is known¹³ that poly(α -methylstyrene) has a higher stabilization constant value for its charge-transfer complexes than polystyrene itself. This means that poly(α -methylstyrene) will form a charge-transfer complex very easily and the effects of impurities on the electret stabilities will be significant. Therefore, it is not surprising to see that the electrets prepared from poly(styrene-co- α -methylstyrene) (2:3) have very poor stabilities, as shown in Table VII.

Even under dry conditions at 50°, the electrets lost charges. Interestingly under humid conditions, the rate of charge decay is relatively independent of temperatures, humidity being the dominant variable.

In comparison, introduction of alkyl groups into the phenyl ring has a different effect. The electret properties, however, were found to depend on whether the alkyl substituent was normal or branched.

If the phenyl ring is the site for charge trapping in these polymers, the important transition temperature at which the charge loss will be maximum should

TABLE VIII
Charge Decay Characteristics of Electrets of Various *n*-Alkyl Substituted Polystyrene Derivatives^a

| Time days | Poly(vinyltoluene), T _g = 100° | | Poly(ethylstyrene), T _g = 27° | | Poly(<i>n</i> -butylstyrene), T _g = 6° | |
|-----------|---|------------------|--|------------------|--|------------------|
| | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ |
| 0 | 1560 | 1.00 | 1400 | 1.00 | 2000 | 1.00 |
| 2 | 1500 | 0.96 | 800 | 0.57 | 100 | 0.05 |
| 8 | 1450 | 0.93 | 190 | 0.14 | — | — |
| 15 | 1420 | 0.91 | 50 | 0.04 | — | — |

^a Environment: 50°, 100% relative humidity.

TABLE IX
Charge Decay Characteristic of Electrets from Polystyrene, Poly(vinyltoluene), and Poly(*t*-butylstyrene), Each Precipitated Three Times from Commercial Benzene^a

| Time, days | Polystyrene | | Poly(vinyltoluene) | | Poly(<i>t</i> -butylstyrene) | |
|------------|-------------|------------------|--------------------|------------------|-------------------------------|------------------|
| | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ |
| 0 | 1650 | 1.00 | 1800 | 1.00 | 1700 | 1.00 |
| 1 | 330 | 0.20 | 1060 | 0.59 | 1540 | 0.91 |
| 8 | 250 | 0.15 | 880 | 0.49 | 1500 | 0.88 |
| 20 | — | — | 790 | 0.44 | 1520 | 0.89 |
| 30 | 190 | 0.12 | 730 | 0.41 | 1510 | 0.89 |
| 50 | — | — | 690 | 0.38 | 1510 | 0.89 |

^a Environment: 50°, 100% relative humidity.

be the glass transition temperature (T_g) of these polymers. Table VIII shows how electret stability and T_g are directly correlated in the *n*-alkyl-substituted polystyrenes.

Introduction of *n*-alkyl groups in the phenyl rings of polystyrene decreases T_g ; but if a branched chain alkyl such as *t*-butyl group is introduced, the T_g is increased. It is, therefore, not surprising to find that poly(vinyltoluene) and poly(*t*-butylstyrene) with T_g values of 110 and 130°, respectively, give rise to more stable electrets than polystyrene ($T_g = 100^\circ$). What is more interesting is that the effects of impurities also progressively decrease in the same order. Table IX shows the electret charge decay behavior of these polymers at 50° and 100% relative humidity after attempts have been made to purify these polymers by the use of commercial benzene containing deleterious impurities.

Thus, the combined effects of T_g and of interaction with impurities determine the electret stabilities of these polymers. These effects, however, have to be in balance as illustrated by the behavior of the electrets prepared from halogenated polystyrenes. There is a steady increase in T_g when polystyrene is substituted in the benzene ring with chlorine, bromine, and iodine, respectively. But the electret stability decreases in the same order (Table X). Therefore, the effects of substituents on charge-transfer complex formation by the phenyl ring result in decreased electret stabilities, even though the T_g values of these polymers are high. In this respect, these polymers behave similarly as poly(styrene-co- α -methylstyrene) (2:3) (Table VII).

We can conclude from above that in styrene-type polymers, the conditions for obtaining stable electrets are the following. High values of volume resistivities and glass transition temperatures, together with a lower tendency to form charge-transfer complexes with electron-acceptor materials, are required for

TABLE X
Charge Decay Characteristics of Halogenated Polystyrene Electrets^a

| Time, days | Poly(chlorostyrene) | | Poly(bromostyrene) | | Poly(iodostyrene) | |
|------------|---------------------|------------------|--------------------|------------------|-------------------|------------------|
| | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ |
| 0 | 1900 | 1.00 | 1700 | 1.00 | 1450 | 1.00 |
| 7 | 1670 | 0.88 | 1200 | 0.71 | 450 | 0.31 |
| 82 | 1300 | 0.68 | 800 | 0.47 | 230 | 0.16 |
| 250 | 1100 | 0.58 | — | — | — | — |

^a Environment: room temperature, 100% relative humidity.

TABLE XI
Charge Decay Characteristics of Various Copolymer (1:1) Electrets^a

| Time, days | Poly(styrene-co- vinyltoluene) | | Poly(styrene-co- <i>t</i> -butylstyrene) | | Poly(vinyltoluene-co- <i>t</i> -butylstyrene) | |
|---------------|-----------------------------------|------------------|---|------------------|--|------------------|
| | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ | Voltage, V | V/V ₀ |
| 0 | 1160 | 1.00 | 1140 | 1.00 | 1480 | 1.00 |
| 4 | 1100 | 0.95 | 1080 | 0.95 | 1450 | 0.98 |
| 10 | 1120 | 0.97 | 1000 | 0.88 | 1440 | 0.97 |
| 17 | 1090 | 0.94 | 990 | 0.87 | 1370 | 0.93 |
| 24 | 1080 | 0.93 | 960 | 0.84 | 1360 | 0.92 |
| 31 | 1070 | 0.92 | 940 | 0.82 | 1330 | 0.90 |
| 45 | 1080 | 0.93 | 920 | 0.81 | 1350 | 0.91 |
| 57 | 1060 | 0.91 | 920 | 0.81 | 1340 | 0.91 |
| 100 | 950 | 0.82 | 860 | 0.75 | 1200 | 0.81 |

^a Environment: 50°, 100% relative humidity.

stabilizing electret charges. Among the polymers of this class that we have studied, poly(*t*-butylstyrene) seems to possess the best combination of physical properties for stable electret formation.

Lastly, we studied the stabilities of electrets prepared from 1:1 copolymers of styrene, vinyltoluene, and *t*-butylstyrene to determine if these offer any advantage over the homopolymers. The results are shown in Table XI. The results indicate that the copolymers do not offer any real advantage over their homopolymers.

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