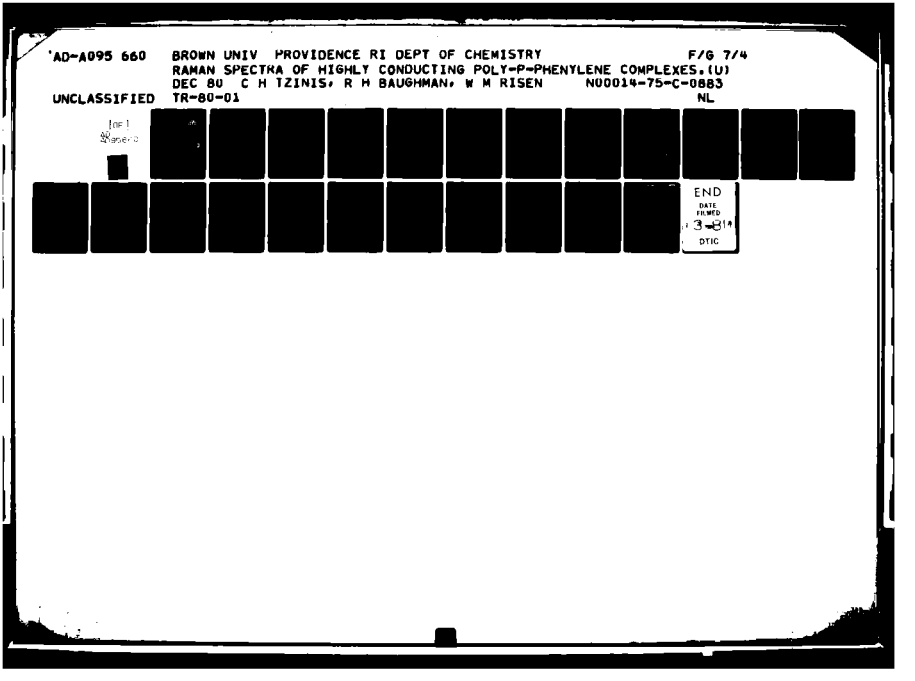
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TECHNICAL REPORT NO. TR-80-01

LEVEL II

# Raman Spectra of Highly Conducting Poly-p-phenylene Complexes

by

Costas H. Tzinis, Ray H. Baughman, and William M. Risen, Jr.

Prepared for Publication

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December 15, 1980



Department of Chemistry
Brown University
Providence, R.I. 02912

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20. (Continued)

the relationship between changes in molecular configuration caused by optical transitions in the donor- or acceptor-doped PPP materials and the vibrational modes of the polymer.

# Raman Spectra of Highly Conducting Poly-p-phenylene Complexes

by

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## Abstract

The resonance Raman spectra of poly(p-phenylene) (PPP) and the highly conducting materials formed by doping it with the electron-donor Na (Na-PPP) or the electron acceptor $AsF_5$ ($AsF_5$-PPP) have been obtained. Doping with either type of dopant causes the C-C bond joining the phenyl-rings to become stronger and at least one of the in-ring bonds to become weaker, so all C-C bonds along the backbone of the π-electron system become more alike than they are in the undoped PPP. The resonance enhancements show the relationship between changes in molecular configuration caused by optical transitions in the donor- or acceptor-doped PPP materials and the vibrational modes of the polymer.

1. Allied Chemical Corporation
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## Introduction

The discovery of several highly conducting polymer systems, including charge-transfer complexes of poly(p-phenylene) (PPP) (1,2) and of polyacetylene (PA) (3,4), has attracted a great deal of attention. Through chemical doping with electron-acceptors, such as $AsF_5$, or donors, such as Na, the electrical conductivity of these conjugated $\pi$-electron systems can be increased from their intrinsic insulating values of less than $1x10^{-10}$ S $m^{-1}$ to metallic values of greater than $1x10^{3}$ S $m^{-1}$. Despite numerous studies of the properties of these materials, relatively little is known about the nature of the polymer chain after doping (5).

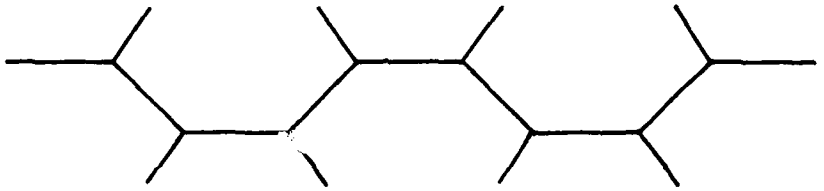
We report that the resonance Raman spectra of poly(p-phenylene) and its highly conductive $AsF_5$- and Na-doped charge transfer complexes reveal the effects on the polymer caused by doping.

The structure of PPP is shown in Fig 1 (1). We assume, as in studies of PA- and graphite-complexes, that the basic PPP structure remains in the doped PPP. This result has been confirmed by investigations on $AsF_5$- and Na-doped PPP (1,2). However, since the dopants transfer electrons onto the polymer chain (electron donor complexes, electron carriers) or away from the chain (electron acceptor complexes, hole carriers), it is expected that the strengths and lengths of certain of the in-chain C-C bonds are changed by doping. Such changes in carbon-carbon bond strength should cause changes in the Raman-observable bond-stretching frequencies. And, of course, the directions and magnitudes of the frequency shifts should reflect the changes in electronic structure of the chains.

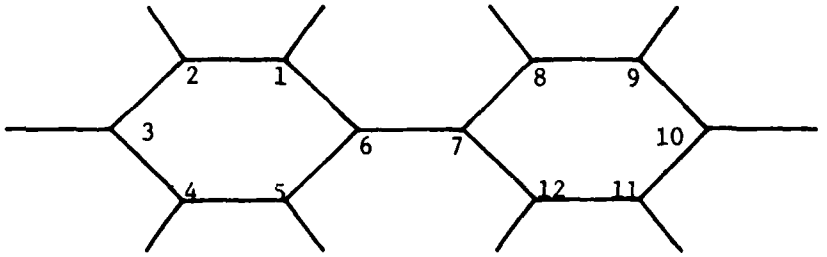
## Experimental

The poly(p-phenylene) used in the present study was synthesized using

COMPARISON OF PA AND PPP LOCAL STRUCTURES



CIS – POLYACETYLENE (CIS-PA)




POLY-P-PHENYLENE (PPP)

Figure 1

the Kovacic synthetic method, as previously reported (1), and consists primarily of uncrosslinked para-linkages. It was prepared into cylindrical pellets. Elemental analyses of the samples showed their C to H atomic ratios to be 1.48-1.50, which are in good agreement with the value of 1.5 expected for $(C_6H_4)_x$. Acceptor doping was accomplished by exposing the PPP pellets to 450 Torr $AsF_5$, under reaction conditions described previously, which resulted in green-black materials with 0.32±0.08 moles $AsF_5$ per mole of monomer units $[C_6H_4(AsF_5)_{0.32}]_x$ and F to As atomic ratios of 4.75-5.10±0.30 and C to H ratios of 1.5. Neutron activation analysis of similar pellets indicated a dopant concentration near the surface of $[C_6H_4(AsF_5)_{0.42}]_x$, and that in the interior of $[C_6H_4(AsF_5)_{0.24}]_x$. Electron donor doping was accomplished by treating PPP with sodium naphthalide in tetrahydrofuran solution, resulting in gold-appearing materials. The doped materials were handled in vacuo or in an argon-filled glove box and were sealed in a glass vial for spectroscopic study. The conductivity of identically $AsF_5$-treated PPP was $\sigma \approx 1.7 \times 10^4$ S $m^{-1}$; those of Na-PPP approach $1 \times 10^3$ S $m^{-1}$.

The Raman spectra of PPP (spinning pellet) and doped PPP (Na-PPP, and $AsF_5$-PPP) (samples sealed in glass tubes) were investigated using 632.8 nm (Spectrophysics 125) and 488.0 nm (Spectrophysics 165) laser radiation and a Jarrell-Ash 25-300 Raman spectrometer operating at 2 $cm^{-1}$ resolution.

## Results

The Raman spectra of PPP, Na-PPP, and $AsF_5$-PPP obtained with the 632.8- and 488.0-nm laser sources are shown in Figs 2 and 3. The positions of three principal bands are tabulated in Table 1, and the forms of the vibrations to which they are assigned are illustrated by analogy to those of biphenyl (6) in Fig 4. Note that the arrows give direction and relative amplitude information only.

RAMAN SPECTRA OF PPP AND ITS DERIVATIVES (LASER WAVELENGTH : 6328 Å)

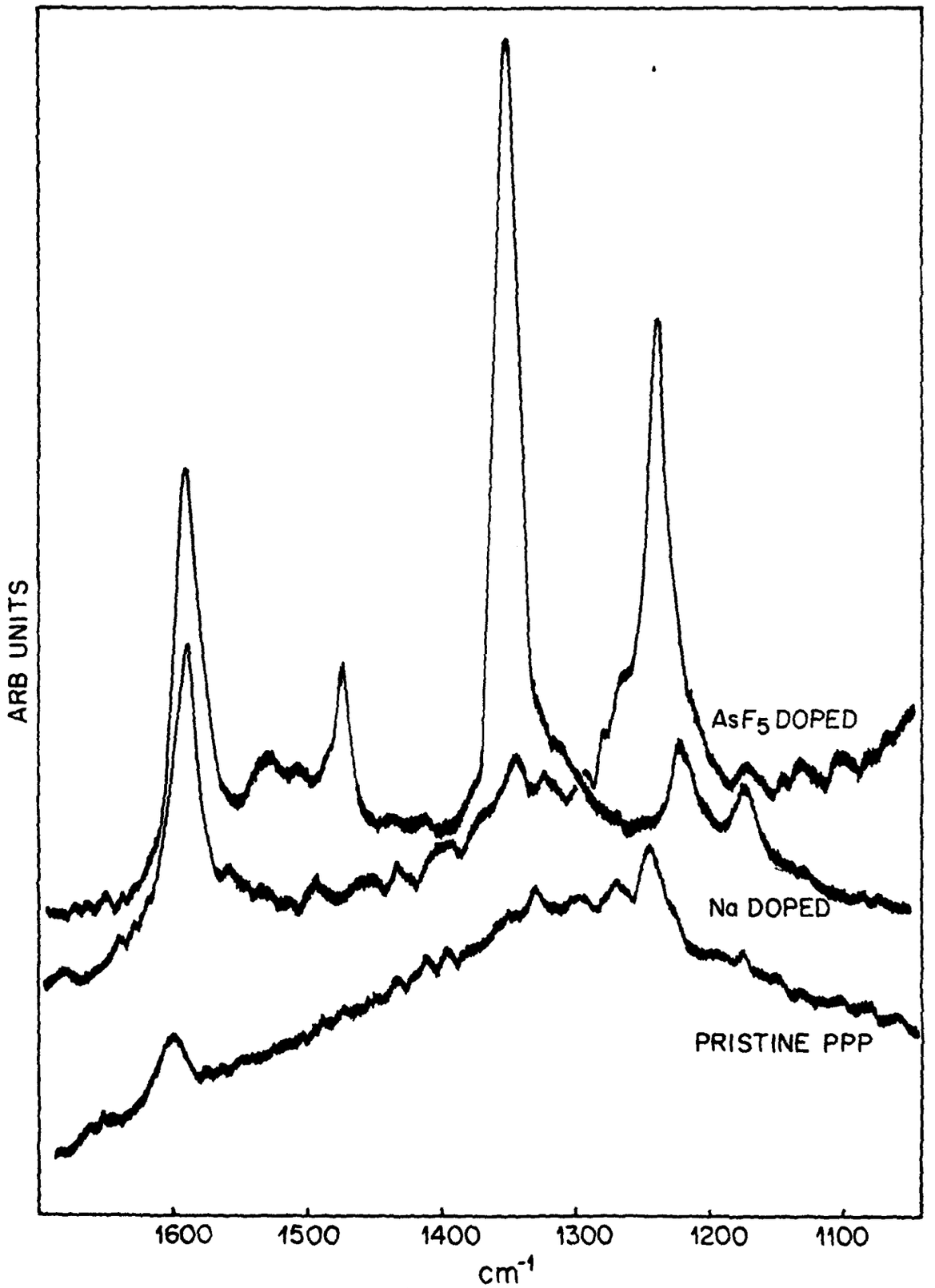



Figure 2

RAMAN SPECTRA OF PPP AND ITS DERIVATIVES (LASER WAVELENGTH: 4880 Å)

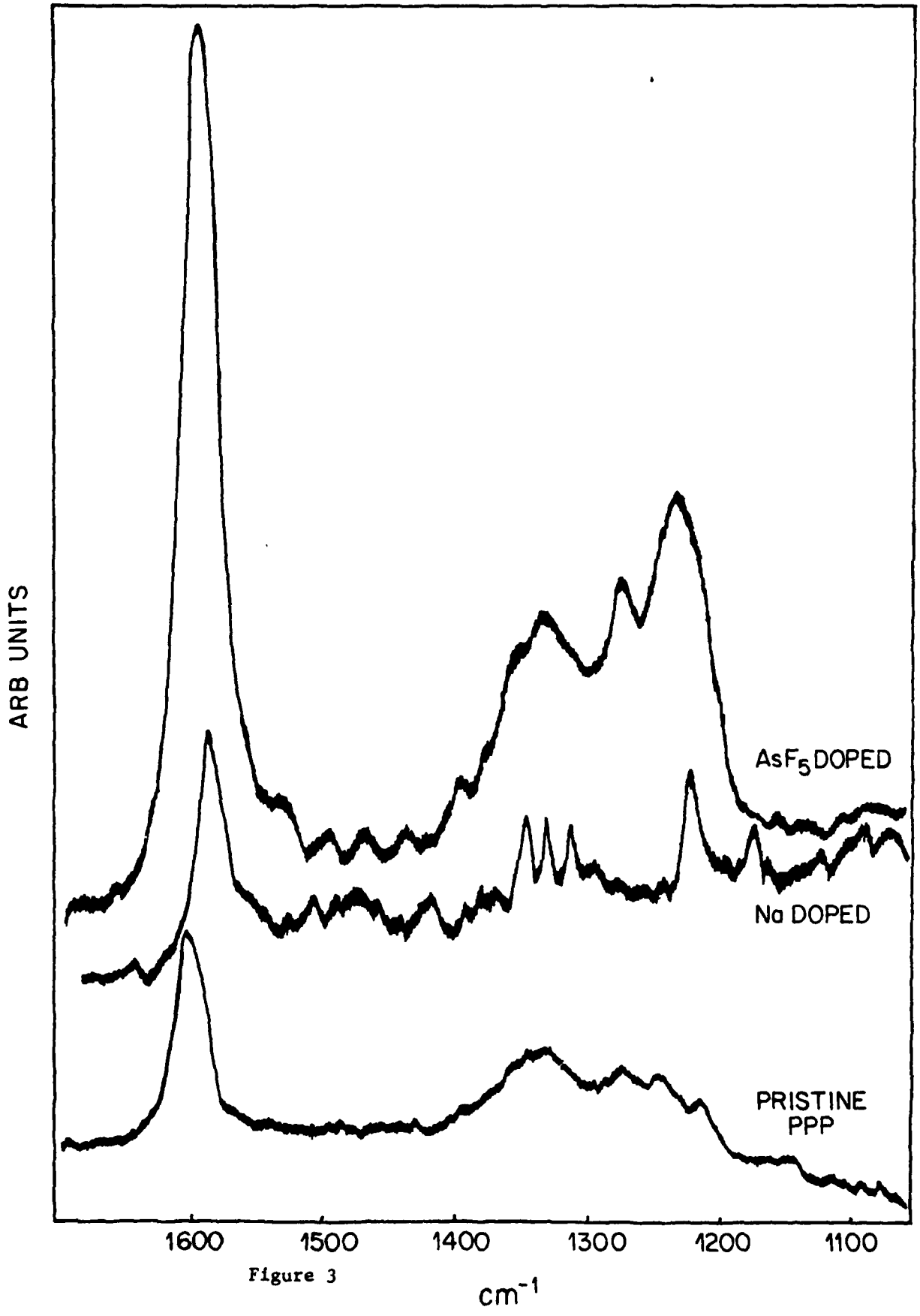


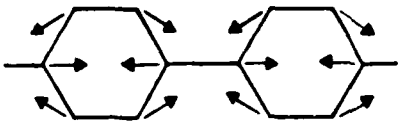

Figure 3

Table 1

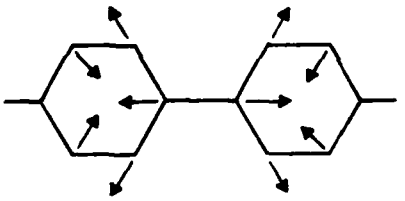
Observed Raman bands assigned to $\nu_1$, $\nu_2$, and $\nu_3$ of PPP and doped-PPP

| Raman source | Material | $\nu_1(cm^{-1})$ | $\nu_2(cm^{-1})$ | $\nu_3(cm^{-1})$ |
|---|---|---|---|---|
| 488.0 (nm) | PPP | 1605 | 1337 | 1247 |
| 488.0 | Na-PPP | 1591 | 1350 | 1232 |
| 488.0 | $AsF_5$-PPP | 1588 | 1352 | 1223 |
| 632.8 | PPP | 1601 | 1330 | 1245 |
| 632.8 | Na-PPP | 1590 | 1342 | 1239 |
| 632.8 | $AsF_5$-PPP | 1588 | 1346 | 1222 |

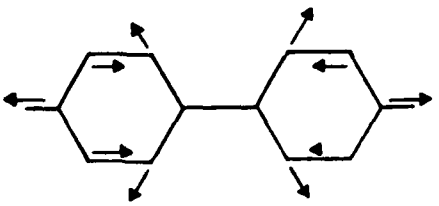
THREE TOTALLY SYMMETRIC (Ag) VIBRATIONS
OF THE $-C_{12}H_8-$ REPEAT UNIT IN PPP



1605 $cm^{-1}$



1271 $cm^{-1}$



1245 $cm^{-1}$

Figure 4

Interpretation of the resonance Raman observations requires knowledge of the optical absorption spectra of PPP and doped PPP in the region of the Raman source frequencies. The most useful available information of this type (2) is given in Fig 5, which shows the spectrum of doped polymerized p-terphenyl, which is an acceptor-doped highly conducting poly(p-phenyl) polymer, and of p-terphenyl itself. In reporting these spectra, Shacklette et al. noted that the high frequency absorption (near 3.6 eV) is that for a $\pi$-$\pi^*$ transition of the chain, and is essentially the same for the $AsF_5$-doped or $(CH_3)_2NH$-compensated material, while the lower frequency band, peaking below 1 eV (specifically 3600 $cm^{-1}$), vanishes when the $AsF_5$-doped material is compensated. The two vertical arrows mark the frequencies of the Raman sources.

It is also useful in interpreting the Raman spectra to note the results of molecular orbital (Hückel SCF-MO) calculations on biphenyl and related poly(p-phenyls) (7). Much of the information required is contained in the symmetry properties of the orbitals and for the undoped PPP such properties are represented well (and simply) by the orbitals on biphenyl. Of the twelve $\pi$-electron SCF-MO's computed using the C 2p atomic orbitals as the basis set, the key ones for the present purpose are $\psi_3$ and $\psi_{10}$, whose energy levels are represented in Fig 6, and, particularly, $\psi_3$, $\psi_6$, $\psi_7$, and $\psi_{10}$, whose symmetry properties are represented by the Hückel coefficients in Table 2. In Table 2 the coefficients $C_{ij}$ of the 2p orbitals, $\psi_j$, normal to the plane on each of the twelve carbons as numbered on Fig 1, in the molecular orbitals, $\psi_i$ are listed. Thus, $C_{ij}$ for $\psi_i = \sum_j C_{ij}\phi_j$, are listed. Coefficients of the same sign on adjacent atoms designate a net bonding effect, and when they are of opposite sign a node appears between the atoms and the net effect of occupancy of the orbital in an antibonding contribution to the interaction of these two atoms. Identical sets of absolute values of coefficients

Table 2

Hückel coefficients, $C_{ij}$, for biphenyl

| j \ i* | 3 | 6 | 7 | 10 |
|---|---|---|---|---|
| 1 | .06 | .30 | .30 | .06 |
| 2 | -.29 | -.14 | .14 | .29 |
| 3 | -.44 | -.40 | -.40 | -.44 |
| 4 | -.29 | -.14 | .14 | .29 |
| 5 | .06 | .30 | .30 | .06 |
| 6 | .37 | .37 | -.35 | -.37 |
| 7 | .37 | -.35 | -.35 | .37 |
| 8 | .06 | -.30 | .30 | -.06 |
| 9 | -.29 | .14 | .14 | -.29 |
| 10 | -.44 | .40 | -.40 | .44 |
| 11 | -.29 | .14 | .14 | -.29 |
| 12 | .06 | -.30 | .30 | -.06 |

*The coefficients $C_{ij}$ in the equation, $\psi_i = \sum_j C_{ij}\phi_j$, are listed. Here the $\psi_j$ are the C 2p orbitals normal to the biphenyl plane, numbered as shown in Figure 1, and the $\psi_i$ are numbered as shown in Figure 6 and in reference (7), from which the results are abstracted.

on certain sets of orbitals, e.g. those of $\psi_6$ and $\psi_7$ and those of $\psi_3$ and $\psi_{10}$, are symmetry required.

While it is not strictly valid to assume that addition or subtraction of an electron from the π-electron system of either PPP or biphenyl leaves its one-electron eigenstates unchanged, both spectral (8,9) and theoretical (10,11) studies of the biphenyl radical anion indicate that the effects on vibrational and electronic transitions are as would be expected if an electron were added to $\psi_7$ of the parent biphenyl. This result clearly is only an approximation, but using it in the present case of doped PPP provides a useful framework in which to describe an interpretation of the observed Raman spectra.

## Discussion

The Raman spectra yield two main types of information. The first is the effect of doping on the vibrational frequencies of the polymer. As shown in Table 1, $\nu_1$ and $\nu_3$ decrease and $\nu_2$ increases on doping. As illustrated by the forms of the vibrations in biphenyl, this means that doping causes the bridge C-C bond ($C_6$-$C_7$ in biphenyl) to become stronger and the in-ring C-C bonds (e.g. $C_2$-$C_1$ and $C_1$-$C_6$ in biphenyl) to become weaker. Thus, doping with an electron-donor or with an electron-acceptor causes essentially the same effect. While the magnitudes of the shifts differ slightly (note that the dopant levels are not identical), the directions are the same for both types of dopants.

These results are qualitatively consistent with the conclusions that would be drawn from the assumption that the one-electron eigenstates of the π-system with an electron added or subtracted are quite similar to those of the undoped material. That is, that the effect on bond strengths of adding an electron to the π-system (in a $\psi_7$-like orbital) would be substantially the same as removing one (from a $\psi_6$-like orbital). For example, as can be seen from the coefficients in Table 2, The $C_6$-$C_7$ interaction in $\psi_6$ is antibonding, so removal of an electron should make it stronger, while the $C_6$-$C_7$ interaction in $\psi_7$ is bonding so addition of

an electron to it should also make the $C_6$-$C_7$ bond stronger. Similarly, both types of in-ring C-C bonds should become weaker as they do.

The resonance Raman effects in the spectra also aid in understanding the bonding changes in the π-electron system that accompany doping. As shown in Fig 2, the $\nu_1$ band (ca 1600 $cm^{-1}$) is of appreciable intensity in all three materials when the Raman source wavelength is 488.0 nm. This Ag mode is expected to be strong. Since it is relatively stronger in the 488.0 nm spectrum than in the 632.8 nm spectrum, it is assigned as being preresonantly enhanced through coupling with the optical transition at ca 3.6 eV.

The striking intensity enhancements are of $\nu_2$ in Na-PPP and $\nu_3$ in $AsF_5$-PPP with 632.8 nm escitation. These are resonantly enhanced by their coupling with electronic transitions giving rise to a lower frequency optical absorption in either of the doped materials. In the case of $AsF_5$-PPP this transition leads to the strong 0.6-2.0 eV band in Fig 5. In the Na-PPP case a similar low frequency band will be due to similar transitions, but in the $\pi^*$ band.

The electronic transition of Na-PPP excited at 632.8 nm gives rise to electron density changes and a consequent molecular distortion that causes the $\nu_2$ vibration to be resonantly enhanced. A similar observation in the biphenyl and biphenyl-anion ($BP^-$) system was shown by Yamaguchi, et al.(10) to be due to the fact that the changes in the equilibrium bond lengths of $BP^-$ associated with the $\psi_7$ to $\psi_{10}$ transition is of the same form as $\nu_2$. Assuming the analogy between BP and PPP and between $BP^-$ and Na-PPP to hold at least insofar as their symmetry properties are concerned, the resonance enhancement of $\nu_2$ in Na-PPP can be understood. Using it, the 632.8 nm Raman source would excite a transition from $\psi_7$-like to a $\psi_{10}$-like orbital. The electron density changes accompanying that transition affects most the C-C bond that joins the rings, affects $C_1$-$C_6$-like bonds significantly and $C_2$-$C_1$ bonds very little.

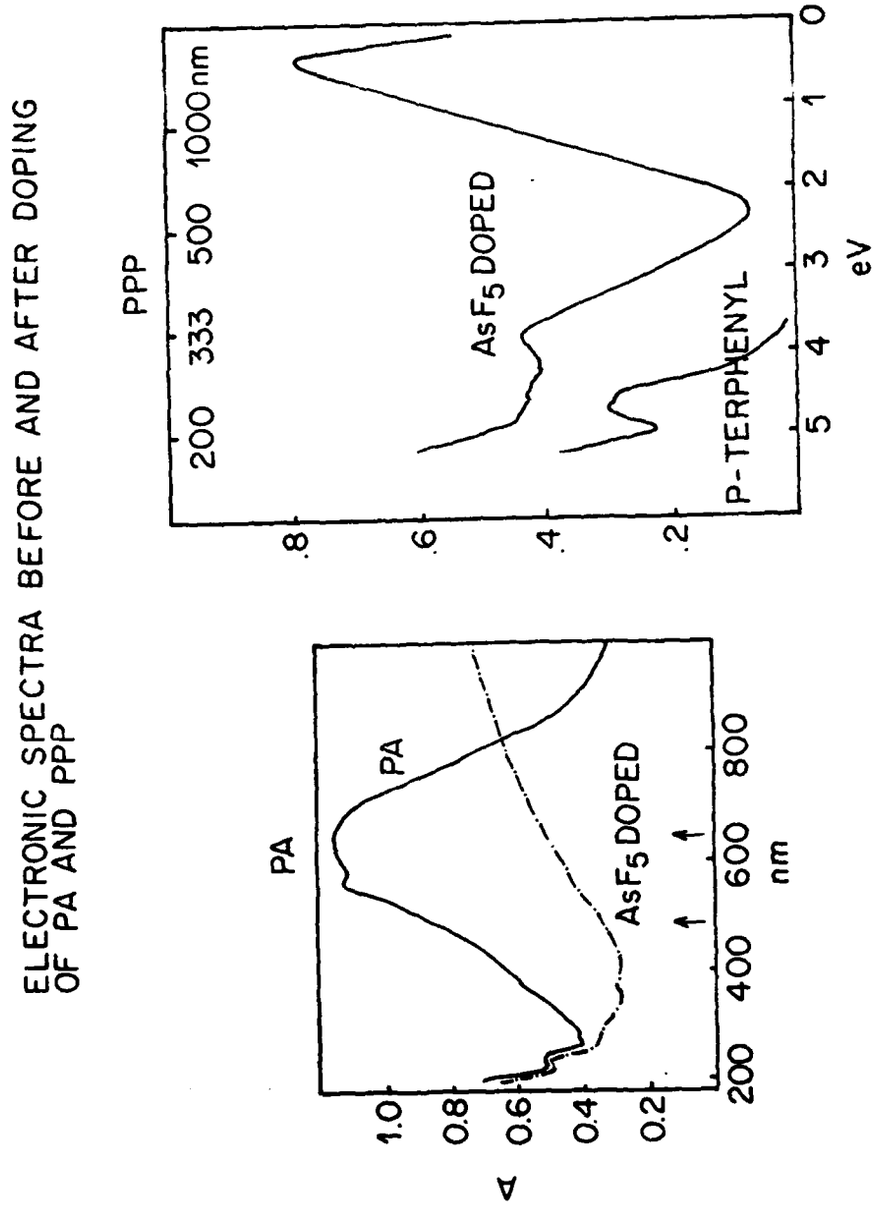

ELECTRONIC SPECTRA BEFORE AND AFTER DOPING
OF PA AND PPP
PPP
200 333 500 1000 nm
AsF5 DOPED
P-TERPHENYL
.8
.6
.4
.2
0
1
2
3
4
5
eV
PA
PA
AsF5 DOPED
1.0
0.8
0.6
0.4
0.2
A
200
400
600
800
nm

Figure 5

ALLOWED ELECTRONIC TRANSITIONS, IN THE VISIBLE, OF A SIMPLE TWO RING SYSTEM (BIPHENYL)

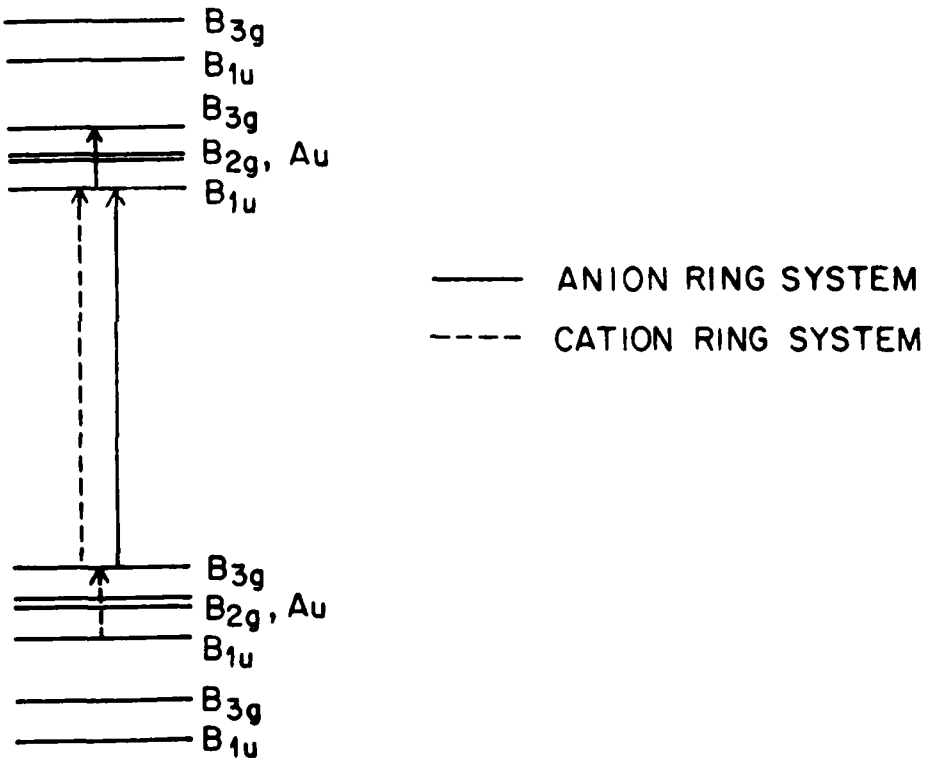



Figure 6

Similar arguments lead to describing the low frequency transition in $AsF_5$-PPP as being that between $\psi_3$-like and $\psi_6$-like orbitals, which is possible because $AsF_5$ has removed an electron from the $\psi_6$-like orbital. The electron-density changes accompanying this transition primarily affect the in-ring C-C bonds, do not affe t the C-C bonds that connect the rings, and enhance $\nu_3$ predominantly.

While it is helpful in interpreting and discussing the Raman spectra to use the terms provided by the Hückel calculations, it is important to note the conclusions that can be drawn on the basis of the Raman results alone. First, the bridge carbon-carbon bond (analogous to $C_6$-$C_7$) becomes stronger (and presumably shorter) while at least one of the in-ring carbon-carbon bonds become weaker (and longer) when PPP is doped with either an electron donor or acceptor. Second, the electron density changes that result from the optical transitions excited at 632.8 nm causes distortions similar in form to $\nu_2$ in the Na-PPP case and to that of $\nu_3$ in $AsF_5$-PPP. These conclusions neither depend on nor demonstrate the validity of the Hückel description, but they are qualitatively consistent with it.

Overall, both the frequency shifts and the resonance enhancement behavior of the Raman bands for both acceptor- and donor-doped poly(p-phenylene) are in accord with the assumption that an electron is added to or taken from the $\pi$-system of the parent polymer. They are qualitatively in accord with the predictions that can be made by assuming molecular orbitals of the extended Hückel SCF-MO type and assuming that the one-electron eigenstates give a reasonable approximation to the states that obtain upon doping. More detailed analyses of the nature of the materials after doping must include the rearrangements affected by changes in electron population and bond length changes, of course, and will be reported later.

## Acknowledgments

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11. See ref (5), in which the $\pi \rightarrow \pi$ and $\pi^* \rightarrow \pi^*$ transitions are calculated.

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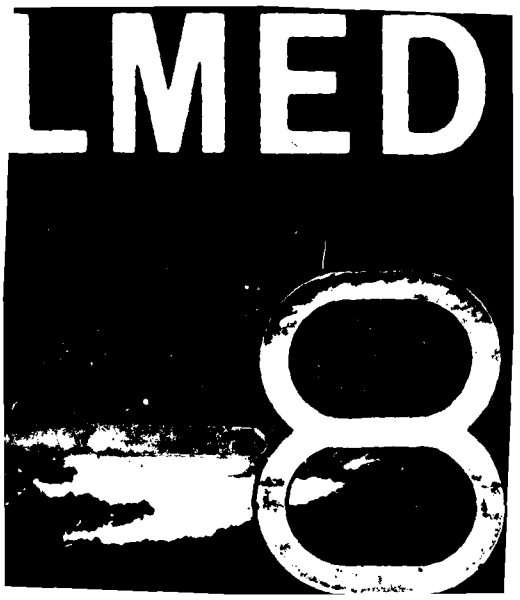
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