X-RAY ABSORPTION IN POLYMERIC CONDUCTORS*

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ABSTRACT: A discussion of the results of x-ray absorption experiments on the polymeric conductors brominated $(SN)_x$ and polyacetylene doped with AsF_5 will be given. The use of x-rays with energies exceeding the K-edge of Br(13.5KeV) and As(11.9KeV) allows studies of the short range order from the backscattering effect of the surrounding atomic arrangement on the ejected 1s electron. Additionally, the shift of the K absorption edge can provide a measure of charge transfer from the SN or acetylene units of the polymer to the incorporated bromine or AsF_5 molecular units. Structural information is derived from the oscillatory part of the extended x-ray absorption (EXAFS). The experiments make use of the highly polarized x-ray beam from the Stanford Linear Accelerator to study the orientational dependence of the x-ray absorption, which allows the determination of the alignment of the incorporated bromine in $(SN)_x$. The short range order in the vicinity of the Br and As atoms is obtained by Fourier transformation of the experimental data, from which average coordination numbers and neighbor distances are then determined. The temperature dependence of these parameters will be given in the regions $300 \leq T \leq 5°K$ and tentative conclusions regarding the relative concentration of Br_2 and Br_3^- molecules at different temperatures are discussed.

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I. INTRODUCTION

In attempts to enhance the transport properties of the conducting polymer polysulfurnitride, $(SN)_x$, halogenated polymers of the type $(SN(Br)_y)_x$ have been synthesized in several laboratories.[1,2] These halogen derivatives of $(SN)_x$ show an order of magnitude increase in conductivity, a 10% increase in superconducting transition temperature T_c and a redshift of the plasma edge[3] relative to pristine $(SN)_x$.

Although Raman experiments have been performed to determine the molecular nature of bromine present in the $(SN)_x$ matrix and to attempt to deduce information about the location of the bromine relative to the $(SN)_x$ fibers,[4,5] ambiguity exists in the assignment of the observed modes to Br_2 and Br_3^- species. IR studies are consistent with bromine being present as a mixture of Br_3^- and Br_2 species.[5a] Electron diffraction experiments[3] and diffuse x-ray measurements[6] indicate the presence of supperlattice lines with a period twice the chain axis repeat unit of $(SN)_x$ in the conducting b direction. The diffuse streaks arising from this one dimensional superlattice disappear below 140°K.

Another very interesting organic polymer, polyacetylene,[7] can be transformed into a highly conducting material by both oxidation and reduction. Currently the most extensively investigated derivative is obtained by treatment of $(CH)_x$ with AsF_5.[8] We report on several sets of experiments on $(SN)_x$ and AsF_5 treated $(CH)_x$[9,10] using synchrotron radiation from the electron-positron storage ring of the Stanford Linear Accelerator Center.

The central idea is to use excitation of Br and As K electrons to the continuum to study the variation of the x-ray absorption at and above the K edge to gain information about charge transfer from the $(SN)_x$ and $(CH)_x$ chains to the Br_n and AsF_5 molecules, as well as to deduce changes in the final state of the Br_2 and AsF_5 molecules after reaction with the respective polymer. For a description of the use of x-ray absorption to determine the short range order of a given atom, whose K or L shell is excited, we refer to several recent review articles.[11] The techniques required to reduce the oscillatory structure in the absorption cross-section above the edge have been extensively discussed and refined in the past years.[12-16] Potentially novel EXAFS effects arise in our studies from the linear form of the Br_3^- molecules and for larger units of Br aligned with the $(SN)_x$ b axis. Multiple scattering of the outgoing photoelectron on its path from the source atom to the backscattering shell and back are important for all but the nearest neighbor shell. Some of these complications make the extraction of coordination numbers in $(SN(Br)_y)_x$ ambiguous.

In addition to the data reduction of the EXAFS oscillations, we have begun to study the edge region itself as a means for determining charge transfer and for identifying molecular species. Self-consistent field Hartree-Fock calculations on AsF_5 molecules have been performed to calculate the excitonic levels, consisting of a 1s hole and one electron in a previously unoccupied electronic state of negative energy relative to the ionization energy. Specifically we have found one exciton state of valence character (inner well) with a binding energy of -5.0eV and two

Rydberg states (outer well) with binding energies of -2.0 and -1.8eV.[17] The distinction into inner and outer well states[18] is made on the basis of whether $\langle r^2 \rangle \lessgtr R^2_{As-F}$ i.e., whether the bound electron resides predominantly outside or inside the fluorine cage surrounding the arsenic atom. We expect that the Rydberg levels will be most sensitive to incorporation into a matrix such as $(CH)_x$.

II. EXPERIMENTAL DETAILS

The samples were sheets of fibers of $(SN)_x$ approximately 100μm thick, which were cleaved from crystals with dimensions 2x3x3mm^3. They were brominated and then mounted on a cold finger in a sample holder equipped with kapton windows. The fiber axis (crystal b axis) lay in the plane of the samples. These were usually mounted with the crystal b axis parallel to the x-ray polarization vector. The sample could be rotated about an axis perpendicular to the x-ray polarization vector and the sample b axis allowing the angle between these directions to be varied by ±30° to study the anisotropy of the absorption. We also performed measurements on samples with the b axis normal to the x-ray polarization vector to study Br-S and Br-N distances. Most of the data were collected between 86°K and room temperature, however, some data were obtained at 5°K with a Helitran system. For comparison of the edge structure and the EXAFS region as a function of the chemical forms of the bromine, we measured samples of Br_2 in gaseous form and several other samples, such as KBr and $S_4N_3^+ Br_3^-$,[20] as standards for Br^- and Br_3^- ion and to determine from the EXAFS analysis of $S_4N_3^+ Br_3^-$ the strength of the Br-S shells in $(SN)_x$ from their backscattering effect. The standard high-resolution EXAFS I beamline at the Stanford Synchrotron Radiation Laboratory was used, with a typical run at a fixed orientation and temperature requiring 20-25 minutes of running time.

$(CH)_x$ films were prepared by the methods described by Shirakawa.[18] The films were exposed to AsF_5 and the composition determined from the weight uptake of AsF_5. X-ray measurements of pristine $(CH)_x$ indicate a highly disordered polymeric material in which even individual fibers are poorly crystalline.[19] Only a few preliminary experiments have been performed on $(CH(AsF_5)_y)$ ($y \sim 0.1$) at room temperature and at 86°K.[10] In order to have reference data, several spectra on an AsF_5 gaseous sample were taken at pressures of 30 micron.

III. $(SN)_x$ AND BROMINATED $(SN)_x$ STRUCTURE AND EXAFS RESULTS

The unit cell of pristine $(SN)_x$ contains two, almost flat, translationally inequivalent, centrosymmetrically related S_2N_2 units. The inequivalent chains lie in the $10\bar{2}$ plane and alternate along the c axis, while chains of the same type are adjacent to each other along the a axis.

One of the key questions to be resolved in the treatment of $(SN)_x$ with bromine concerns the way in which the bromine is incorporated into the $(SN)_x$ crystals. Although a considerable amount of disorder is introduced by bromination, making a standard x-ray structure determination impossible, the unit cell parameters are changed only slightly in the a and c direction, while the b axis spacing remains the same.[3] In addition, a one-dimensional superlattice of 2b periodicity is observed, suggesting

FIGURE 1.

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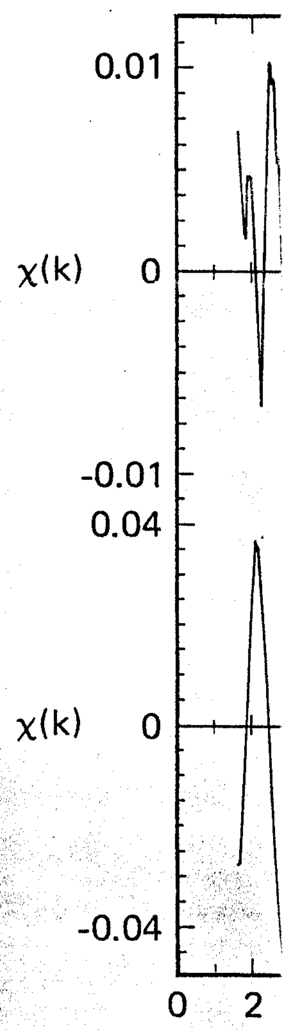

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EXAFS for Brominated $(SN)_x$ with $E \perp b$ and for $S_4N_3Br_3$

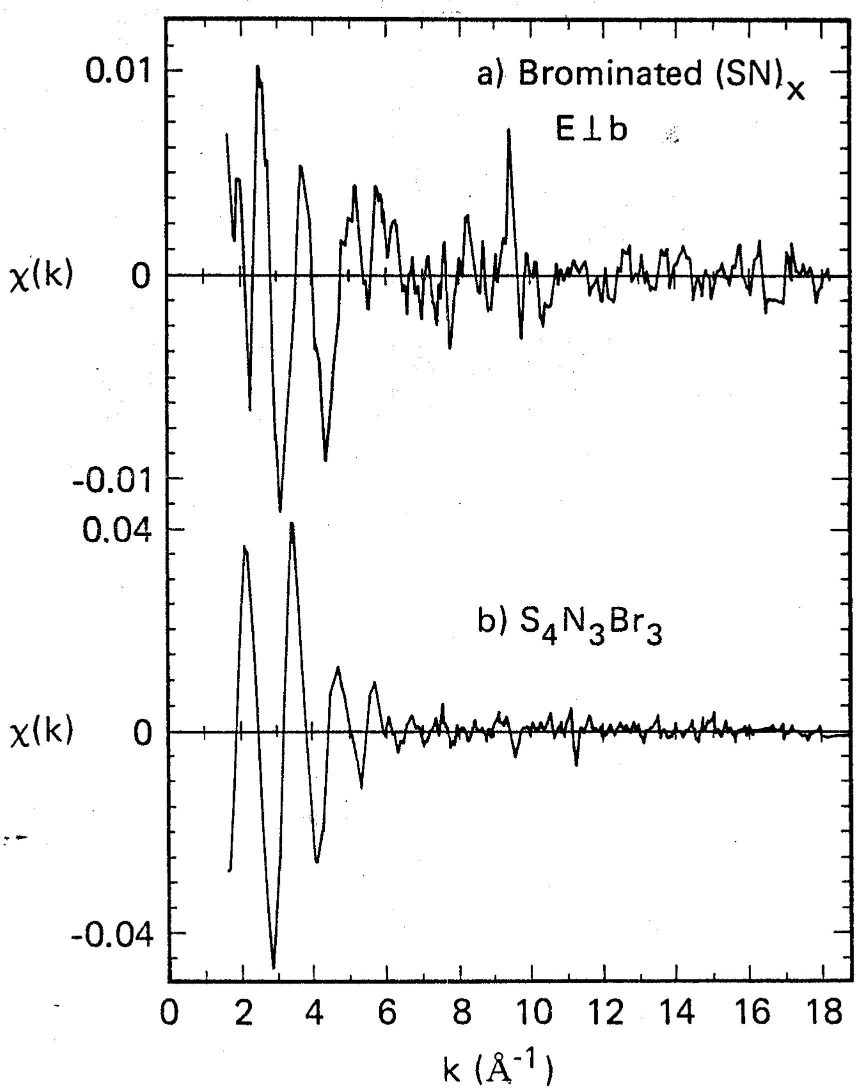

TABLE 1. SUMMARY OF TRANSFORM RESULTS FOR BROMINATED $(SN)_x$

First Shell				Second Shell			
Sample	Position		Amplitude	Position		Amplitude	A_2/A_1
	R_1'		A_1	R_2'		A_2	
Br_2 Gas	1.93		203	-		-	
Br on $(SN)_x$ (86 K)	2.15	(2.50)	363	4.60	(4.95)	103	0.28
Br on $(SN)_x$ (170 K)	2.15	(2.50)	275	4.62	(4.97)	68	0.25
Br on $(SN)_x$ (300 K)	2.13	(2.48)	162	4.58	(4.93)	42	0.26

Br_2 Gas Measured Br-Br = 2.28 A Therefore $\delta = 2.28 - 1.93 = 0.35$ A

Table II	300° K	150° K	86° K
First Neighbor Coordination Number N_1 ( )	0.7 ± 0.1	0.7 ± 0.1	0.7 ± 0.1
Ratio of second Neighbor $\frac{N_2}{N_1}$ = Coordination Number N_2 to N_1	0.34 ± 0.2	0.56 ± 0.2	0.41 ± 0.2

( ) Footnote: Apart from the multiple scattering corrections discussed in the text, a very recent reexamination of EXAFS amplitudes for bromine gas standards has been made, which may affect the values given. (S. Heald and E. Stern, reprint 1979)

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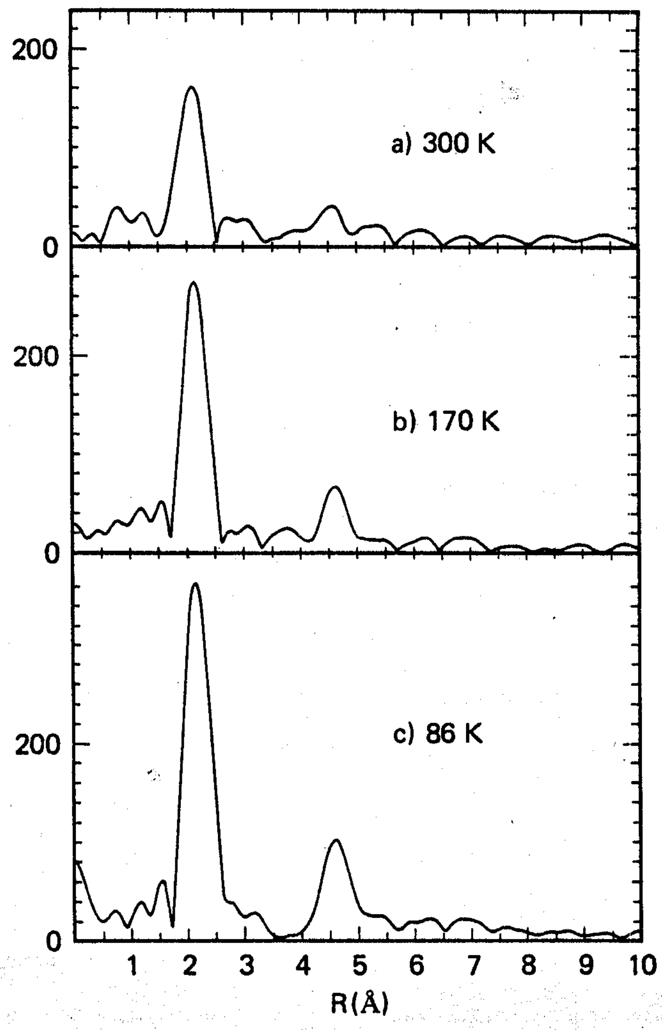
one dimensional ordering along the b direction. As the dimensions of the Br_3^- molecular ion are close to 2b (8.86Å, and 8.9Å, respectively), it is suggestive that the superlattice arises from Br_3^- ions aligned parallel to the chains. As for the presence of expanded Br_2 molecules in $(SN)_x$, we note that x-ray absorption studies of graphite exposed to bromine,[21] a system, which is analogous to brominated $(SN)_x$--albeit in a 2 dimensional rather than 1 dimensional fashion--have shown that the Br_2 units expand to optimize the molecule-graphite interaction from 2.28Å to 2.43Å.

The physical situation, a molecule intercalated between graphite planes or as in our case, a Br_3^- or Br_2 molecule between $(SN)_x$ chains or outside $(SN)_x$ fibers, resembles the molecule-surface system, extensively investigated in chemisorption.[22] We expect some mixing of orbitals from the substrate with the orbitals of the molecule leading to charge transfer and local bonding. (The other extreme, the disruption of the molecular structure of the intercalated molecule, to a uniform stack of bromine, is theoretically also conceivable, stabilized in the solid state by charge-transfer, Coulomb and dispersion forces.) The amount of charge transferred from the $(SN)_x$ chains controls the band filling of the conduction electron π band, which is thought to be responsible for the change in transport properties in the rigid band model.[23]

The oscillatory part of the x-ray absorption spectra obtained at 300°K, 150°K and 86°K are shown in Figure 1 after subtracting out the monotonically decreasing single atom absorption cross section. The Fourier transforms of this oscillatory function, which contain information about near neighbor

FIGURE 2.

Transforms of Brominated $(SN)_x$
K^3 Transforms from 3.7 to 13.7 A^{-1}



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shells and coordination numbers are shown in Figure 2. The first nearest neighbor shell at 2.15Å, to which a phaseshift correction of .34Å has to be added, clearly indicates Br_3^- ions. The entire set of results on first and second nearest neighbor shells are collected in Table I. We have assumed the standard expansion for the oscillatory part of the absorption cross-section $\chi(h)$[12-16]

$$\chi(k) = \frac{1}{k} \sum_i \frac{N_i}{R_i^2} |f(k,\pi)| (\sin (2kR_i+2\delta(k))e^{-2\sigma^2k^2} e^{-2R_i/\bar{l}} \qquad (1)$$

There are various ways of determining the phaseshift $2\delta(k)$, namely by comparison with a reference compound or by some atomic calculation. As forward scattering of the outgoing and returning electron wave-front is very strong, caution is required in using a phaseshift obtained for the first neighbor shell for the second neighbor shell as well. Multiple scattering by the central Br atom on the way from an end bromine in a linear molecular ion such as Br_3^- to the other end gives additional scattering contributions beyond the single path from the one end bromine to the other and back. In Table II we have collected the coordination numbers estimated by using only the Br_2 gas phaseshift of -.34Å. In principle, multiple scattering could change the estimate of Table II by up to 50%. More detailed estimates of the multiple scattering effects based on quantitative calculations are being pursued in analogy to model calculations on purely 1D systems.[24]

It is clear from the data given in Tables I and II that the predominant form of bromine in $(SN)_x$ is Br_3^- or a more extended chain with a nearest neighbor spacing of 2.5Å. This does not exclude the presence of some Br_2

molecules with some charge transfer allowing expansion of the Br-Br diatomic gas phase spacing of 2.28Å to 2.5Å in analogy to the bromine-graphite system;[21] however, the existence of the second nearest neighbor bromine shell at 5Å is incontrovertible evidence for the dominant presence of Br_3^- or larger Br_n units.

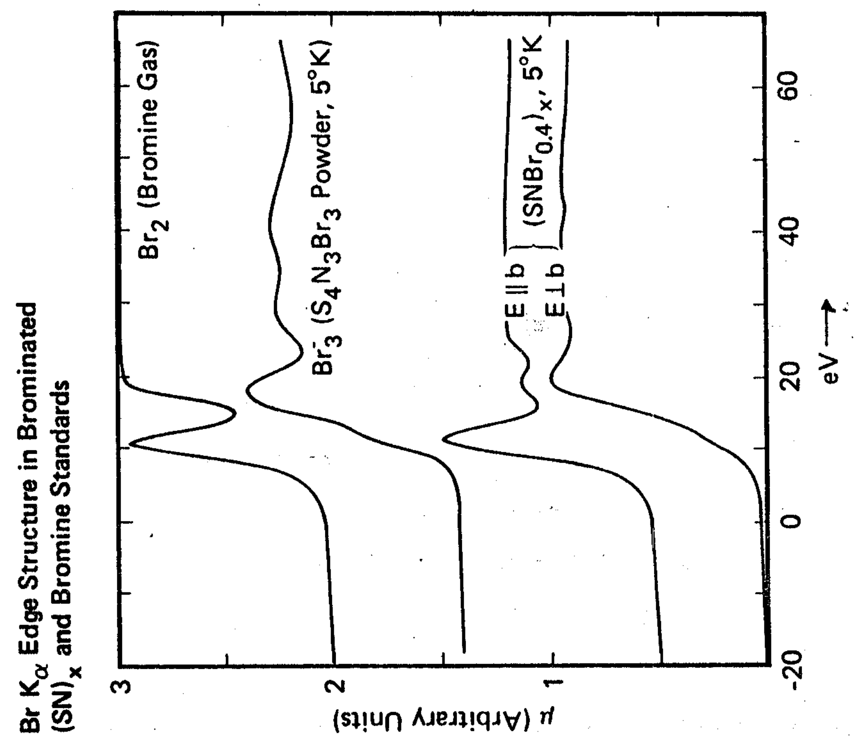
We have also performed measurements with the x-ray polarization perpendicular to the b axis of our samples to study the arrangement of the bromine relative to the $(SN)_x$ chains. We expect the sulphur backscattering to dominate transverse excitation of the Br K-shell electron into continuum states and some chemical arguments favor preferred interaction between S and Br neighbors. It was also thought very useful to perform x-ray absorption experiments on the compound $S_4N_3^+\ Br_3^-$,[20] to obtain information about the resolution of the EXAFS measurements of the 2 unequal Br-Br bond lengths of 2.43 and 2.68Å deduced from the x-ray structure. Despite cooling the powdered $S_4N_3^+\ Br_3^-$ samples to 5°K to minimize the thermal smearing of the peak (Debye-Waller factor) we resolve only a single peak centered at 2.5Å. In this compound, the region from 3.1-3.6Å contains too many bromine-sulphur shells of small coordination numbers to show any sharp structure.

In our measurement of $(SNBr_{0.4})_x$ with the x-ray polarization vector perpendicular to the b axis, oscillations of the absorption cross-section were only detectable to a $k=5\text{Å}^{-1}$, indicating backscattering from a low Z element (i.e., sulphur). As the phase-shift has nonlinear functional behavior for small k,[25] we estimated a correction of -1.3Å to the transform peak centered at 2.08Å. This distance of 3.3Å is consistent with a weakly bonding Br-S contact. As this peak is quite broad, it seems likely that several different Br-S distances occur in the $(SNBr_{0.4})_x$ material, and indicates that the environment of the Br_3^- ions is not unique.

IV. EDGE STRUCTURE IN $(SNBr_{.4})_x$ AND $[CH(AsF_5)_y]x$

In addition to extracting bond distances and coordination numbers from the analysis of EXAFS data, the near edge region with its sharp excitonic structure and the contribution from resonant states in the continuum provides additional information on charge transfer and molecular structure. For K shell excitation only final states of angular momentum $\ell=1$, contribute to the absorption cross-section, due to the dipole character of the x-ray absorption process. For the bromine--$(SN)_x$ system, the molecular excitonic states are made up of appropriately phased 4p (antibonding) orbitals. In Figure III we show, referenced to a common zero of energy, the edge structure for $(SNBr_{.4})_x$ for the x-ray polarization vector parallel, 30° and 90° to the b axis.

The origin corresponds to an energy of 13.452keV above the K shell binding energy. Besides the very prominent white line peak, which is familiar from gas phase studies,[26] we see a second peak, which may arise from the Rydberg series np(n=5,6,...). Interestingly enough, the second peak dominates the excitonic features in the $\vec{\epsilon}\perp b$ case, which is consistent with this interpretation as the $4p_x$, $4p_y$ orbitals are occupied in the molecular Br_3^- ion, so the lowest empty states are $5p_x$, $5p_y$ combinations.



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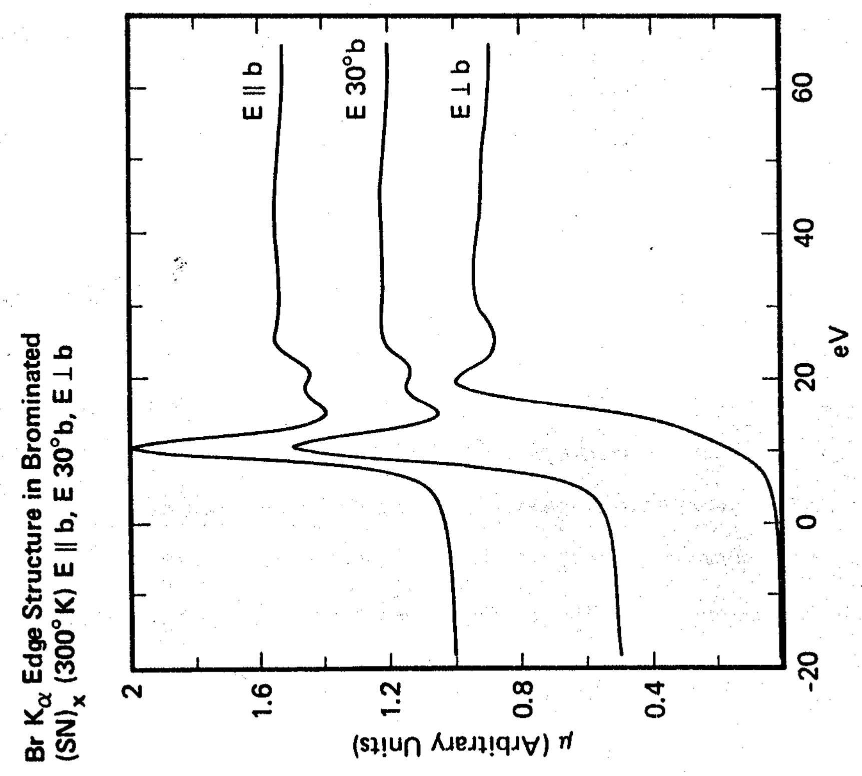

FIGURE 3.

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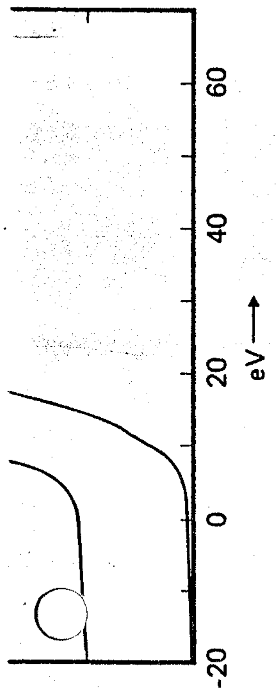

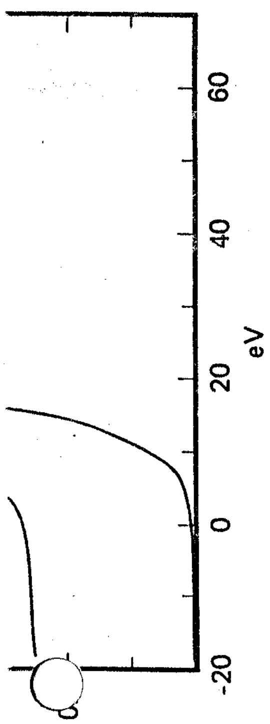

In Figure IV we have compared the edge structure of Br_2, $S_4N_3^+$ Br_3^- and the $(SNBr_{0.4})_x$ with $\vec{\varepsilon}||b$, and $\vec{\varepsilon}\perp b$ ($\vec{\varepsilon}$ polarization vector of the x-ray beam). As the Br_2 excitation clearly involves 4p antibonding states to form the exciton, the parentage of the first feature in all the edges is apparent (apart from shifts of order a few eV due to different inner potentials and charge distribution). The most intriguing aspect of the reference $S_4N_3^+$ Br_3^- compound is the dominance of the higher lying Rydberg states compared to the 4p antibonding state (seen only as a shoulder). Detailed molecular calculations on the excitonic (core-hole-np Rydberg level) states are planned to resolve this point.

H. Morawitz and P. Bagus[17] have performed detailed self-consistent field Hartree-Fock calculations on AsF_5 molecules to interpret the edge structure of the AsF_5 doped polyacetylene polymer. These calculations allow the determination of the ionization energy of AsF_5, corresponding to removal of 1 electron from the 1s shell. In addition, the calculation on the ionized AsF_5^+ (1 core hole) system with full relaxation identifies potential excitonic states corresponding to virtual orbitals of negative energy. Using these states with an additional electron placed into one of these orbitals allows the determination of the wavefunctions, energies and oscillator strengths of the corresponding excitonic states of the AsF_5 molecule.

The AsF_5 molecule is an interesting example of a cage molecule in which Coulomb barrier effects of the five surrounding F^- ions arranged in a bi-pyramidal structure may spatially confine electrons excited from a core level of the central As to the continuum.[18] In addition, even for states, whose wavefunctions extend beyond the fluorine cage, the central core hole acts as spherically symmetric Coulomb potential leading to a Rydberg series. Indeed, we have identified in our calculations a valence type (inner well) excitonic state of E' symmetry with a binding energy of -5.0eV relative to the ionization limit and two Rydberg-like states with binding energy of -2.1eV and -1.8eV of E' and A_2'' symmetry. This calculated feature correlates well with structure in the very large excitonic peak observed in the AsF_5 gas phase edge structure.[10] It should be noted here that we expect large matrix effects on the extended Rydberg states upon incorporation of AsF_5 in a matrix with potentially larger local fields. Preliminary edge data on AsF_5 in $(CH)_x$ show a considerable enhancement of the strength of the edge factors relative to the K-edge step height, which measures the individual atom absorption increase.

V. SUMMARY AND CONCLUSIONS

In this paper we have discussed the first use of x-ray absorption experiments to determine molecular structure, orientation and charge transfer of bromine in $(SN)_x$ and AsF_5 in $(CH)_x$. As the incorporated molecules have a profound influence on the physical properties of these polymeric conductors, a detailed answer to these questions is essential for the development of other new conducting polymers as well as for the clarification of the mechanisms responsible for the striking behavior of these materials.

We have demonstrated that near neighbor distance determination for bromine suggests that the bromine in $(SNBr_{0.4})_x$ is in the form of Br_3^- or longer bromine chains and confirms the orientation of the bromine parallel to the conducting b axis. In addition, we have performed theoretical studies of the excitonic region for the case of AsF_5 in order to use these features in a quantitative manner to determine possible chemical changes in the $(CH)_x$ matrix, to ascertain charge transfer from the shift of edge features and to determine changes in oscillator strengths after incorporation in a matrix such as $(CH)_x$.

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