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He(I) Photoelectron Spectrum of Methylcyclopropene Derivatives

by

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Abstract

The He(I) photoelectron spectra of benzo and naptho derivatives of 1,1-diphenylmethylenecyclopropene (DPMC) are presented. A correlation of electrochemical oxidation potentials ($E_{sub\frac{1}{2}}(+)$ values) and gas phase ionization potentials of a series of aromatic hydrocarbons is discussed. This correlation suggests that the difference in $E_{sub\frac{1}{2}}(+)$ values for the two DPMC derivatives is due to structure specific solvation of the radical cations. HAM/3 and HAM/3/CI calculations are compared with a simple structure derived analysis of the observed pe spectra. It is suggested that the effects of the fused cyclopropene grouping are to be found in small (~.2 eV) reductions of the second or higher ionization energies.

THE HE(I) PHOTOELECTRON SPECTRA OF METHYLENECYCLOPROPENE DERIVATIVES

CORRELATION WITH ELECTROCHEMICAL OXIDATION

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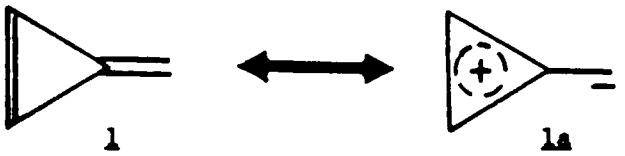
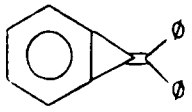
Introduction

Methylenecyclopropene (1), the smallest non-alternant hydrocarbon, has recently been prepared[1] and spectral observations[2] have confirmed the existence of a significant dipolar (1a) contribution to its ground state electronic structure. Characterization of 1 <—> 1a is complicated by its moderately



1 1a

high chemical reactivity but relatively complete spectral and theoretical determinations for this system are possible and forthcoming.[3] The present work concerns results on the kinetically stable cyclopropabenes 2 and 3, the diarylated benzo and naptho derivatives of 1.[4]



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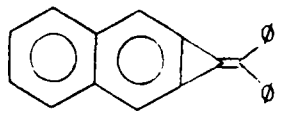


3

The stability, imparted by the aryl substitution, allowed some of us to study[5] the electrochemical behavior of 2 and 3. A significant result of that work was the fact that the half wave oxidation potential ($E_{\frac{1}{2}}^{ox}$) for 3 (0.81 eV) was higher than that for 2 (0.68 eV). This ordering goes against the usual idea that the more delocalized system (3) should be more easily oxidized. It thus seemed of interest to investigate the photoelectron spectra (pe) of 2 and 3 to see if the $E_{\frac{1}{2}}^{ox}$ difference corresponded to an ionization potential (IP) difference in the gas phase. We now report on our determination of the pe spectra of 2 and 3 and on an analysis of the first several ionic states for these systems. A general correlation between $E_{\frac{1}{2}}^{ox}$ values and

pes IP's for aromatic hydrocarbons is presented. This correlation suggests that solvation effects, in the electrochemical oxidation of arenes, increase with increasing IP, due to the smaller molecular size that attends the increased IP with in the series. The difference in $E_{\frac{1}{2}}^{+}$ values for 2 and 3 is rationalized as residual differential solvation effects arising from the difference in hole density distribution.

Experimental

The syntheses of 2 and 3 have been published.[4] The half wave oxidation potentials for 2 and 3 were also as previously described.[5] The photoelectron spectra were obtained from our [6] $\pi/2$ sector instrument at temperatures between 50 and 100°C. The spectra were calibrated in duplicate versus Ar, Xe, methyl iodide mixtures. The beginning resolution in each pe determination was less than 30 mev.

Results and Discussion

The pe spectra of 2 and 3 are shown in Figure 1. They indicate that the observed difference in $E_{1/2}^{+}$ values has no clear counterpart in the gas phase. The first ionization potential of 2 is essentially the same as 3. This applies to both the first peak maximum (~IP (vertical), 7.145 ± .03 eV) and onset (~IP adiabatic, 6.84 ± .03 eV). Figure 2 shows a plot of $E_{1/2}^{+}$ values[5,7a] against first IP values[7,8] (vertical) for several aromatic systems. The correlation predicts an $E_{1/2}^{+}$ value of 0.74 eV for both 2 and 3. The observed value for 2 is slightly lower (0.68 eV) while that for 3 is slightly higher (0.81 eV) than the value (0.74 eV) that the linear correlation predicts.

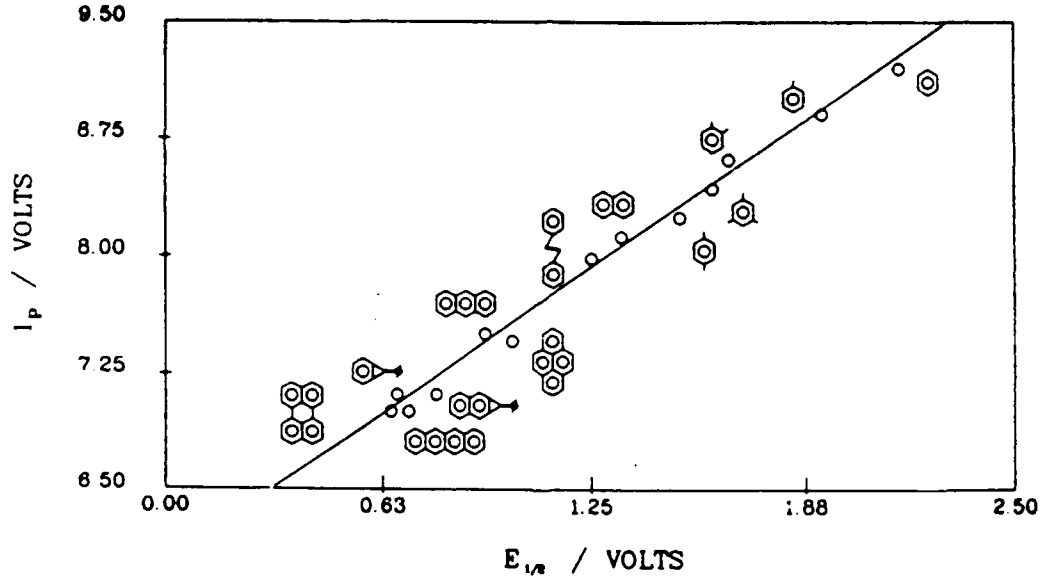


Figure 2. Correlation of $E_{1/2}^{+}$ with IP.

The regression line (correlation coefficient 0.989) shown in Figure 2 is expressed by equation (1). This can be compared with the simplistic theor-

$$E_{1/2}^{+} = 0.667 \cdot IP - 4.023 \qquad (1)$$

etical expression given in equation (2) where $\Delta\Delta G^{\circ}_{sol}$ is the difference in

$$E_{1/2}^{+} = IP + \Delta\Delta G^{\circ}_{sol} + \text{constant} \qquad (2)$$

solvation of the neutral (reduced) and ionic (oxidized) forms of the hydrocarbon and the constant corrects for the reference electrode potential. The linearity and less than unit slope in equation (1) suggest that $\Delta\Delta G^{\circ}_{sol}$ contains a term that is proportional to IP. Equation 3 incorporates this observation and defines the residuel differential solvation, $\delta\Delta\Delta G^{\circ}_{sol}$.

$$\Delta\Delta G^{\circ}_{sol} = -S \cdot IP + \delta\Delta\Delta G^{\circ}_{sol} \qquad (3)$$

Equation (4) results from these definitions.

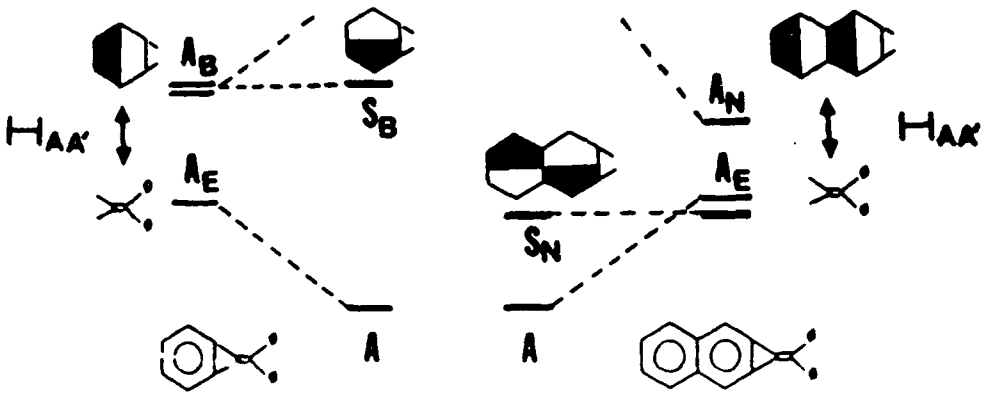
$$E_{1/2}^{+} = (1-S) \cdot IP + \delta\Delta\Delta G^{\circ}_{sol} + \text{constant} \qquad (4)$$

The empirical relationship (equation 1) is obtained by neglecting the $\delta\Delta\Delta G^{\circ}_{sol}$ term with s equal to 0.333 and the constant equal to -4.023 (eV). The deviations from the value (0.74 eV) predicted for 2 and 3 can be interpreted as residual differential solvation effects ($\delta\Delta\Delta G^{\circ}_{sol}$). The values of these deviations (-0.06 eV for 2 and +0.07 eV for 3) are not atypical of the other members of the series shown in Figure 2. Small structure specific solvation effects are certainly present and the proposed $\delta\Delta\Delta G^{\circ}_{sol}$ values are one way to recognize them at least phenomenologically.

The -S·IP term of equation (4) may be related to recent observations[9] of the $E_{1/2}^{+}$ values of a series of molecules of high IP. In those studies it was found that the correlation between $E_{1/2}^{+}$ and IP changed from one which was linear and of unit slope (saturated hydrocarbons) to one of less than unit slope at the high IP -- small molecular size end. The assistance to oxidation by the solvent is of greater magnitude in the high IP - small molecular size

range. Reduced molecular size accompanies increased IP in the aromatic hydrocarbon series of Figure 2 and the -S·IP term of equation (4) may be explained by the higher vacancy density per unit volume in the ions of high IP and smaller π orbital size. The difference in $E_{\frac{1}{2}}^{+}$ for 2 and 3 are well within expectations for structure specific solvation energy differences which we describe by the $\delta\Delta\Delta G^{\circ}_{sol}$ term.

SCHEME I



The observed (Figure 1) near equality in the first IP's of 2 and 3 can be understood by considering their π electron systems as perturbed[10] 1,1-diphenylethenes.[11] The energetic positions and symmetries of the perturbing benzynic (2) and napthynic (3) π electron groups, with respect to that of the lowest ionic state of 1,1-diphenylethene[11] (4), are shown in Scheme I. The conrotation of the two phenyl groups with respect to coplanarity, shown by the X-ray structure for 2, leaves an approximate[12] C_2 axis along the exo methylene group. The labelling in Scheme I designates the basis structures as symmetric (S_B, 2; S_N, 3) or antisymmetric (A_B, 2, A_N, 3; A_E, 4) with respect to this symmetry element. The first IP's of 2 and 3 are then describable as combinations from the appropriate A_1 manifold.

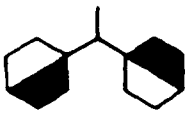
The first naptho (A_N, Scheme I) hole structure in 3, that will perturb the first one derived from 4 (A_E, 8.25 eV,[11] Scheme I), corresponds to the second ionic state of napthalene ($^2B_{1u}$, 8.88 eV[8]). The corresponding hole structure for 2 (A_B, Scheme I) is one of the e_g pair of benzene (9.3 eV). The difference in energy between A_N and A_E is 0.6 eV (3) while that for A_B and A_E is 1.02 eV (2). The smaller difference for 3 compared to 2 leads one to expect a lower first IP for 3 as is the usual case. The interaction constant (H_{AA}, Scheme I) for mixing A_N and A_E (3) will be smaller than that for A_B and A_E (2). The lowered interaction constant for 3 would lead to a high first IP in 3 compared to 2. The net result of the smaller energy gap and smaller interaction constant for 3 is the fortuitous equality in the first IP of 3 with that of 2.

In this very simple analysis, the ionic ground states of 2 and 3 would both be completely (π) delocalized. However, the fortuitous equality in first IP's of 2 and 3 implies that essentially the same (~70%) fraction of the vacancy should reside on the 1,1-diphenylethene grouping in the two ionic ground states. The fraction of the hole distributed on the benzo and naptho grouping would also be the same (30%). The vacancy density, on a per atom basis, would be lower in 3 compared to 2 because of the large size of the naptho substructure. This would be expected to lead to a lowered differential solvation energy ($\delta\Delta\Delta G^{\circ}_{sol}$) and hence an increased $E_{\gamma_2}^{+}$ for 3 compared to 2. This explanation of the difference in the $E_{\gamma_2}^{+}$ values of 2 compared to 3 is at least internally consistent since it is derived from the same physical source as suggested for the larger -S·IP term in equation (4).

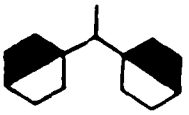
Scheme I also predicts that the second pe band for 3 should correspond to the first ionic state of napthalene (8.15 eV, ^2Au[8]). The symmetry of this basis representation (S_N) is such that it will not be overlap-sensitive to the

attached 1,1-diphenylethene unit. The second ionic state of 3 should thus be describable as one localized on the naptho unit with bond distance changes that correspond to those for the lowest ionic state of napthalene itself. Indeed, the position (7.95 eV) and vibrational shape of the second pe band of 3 (Figure 1) strongly support this assignment.

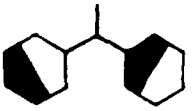
Analysis of the pe spectra of 2 and 3 in terms of perturbed diphenylethene (4) ionic states can also be extended to the 9 eV region. The experimental spectrum of 4[11] shows that two ionic states are located (both) at 9.05 eV with a third state at 9.2 eV. This set is describable in terms of combinations of hole structures localized on the two phenyl groups with little or no ethene (π) cationic character. Two of the three are of S symmetry ($S_{\phi_2}(1)$, $S_{\phi_2}(2)$ below). The one of A symmetry (A_{ϕ_2}) has low coefficients at the phenyl carbons that are joined to the ethene group giving negligible ethene-phenyl mixing.



$S_{\phi_2}(1)$



A_{ϕ_2}



$S_{\phi_2}(2)$

The S_β structure (9.3 eV) for 2, shown in Scheme I, is likewise isolated by symmetry from the ethene unit. Thus, for 2, at least 4 ionic states should reside in the 9 eV region. The observed intensity pattern in the spectrum of 2 supports this analysis since the relative area of the 8.7-9.9 eV section is 4.5 times that of the resolved 7.14 eV band. These arguments also apply to 3 (except for the S_β structure) and the gross appearance of the 9 eV region of the pe spectrum of 3 is expected to be similar to that for 2.

While the major features of the low energy region of the pe spectra of 2 and 3 are simply understandable, the above analysis has ignored possible

effects due to dipolar contributions analogous to 1a in the neutral hydrocarbons and effects due to distortions of the fused benzo and naptho groups. The X-ray data for 2[4] show a significant reduction in the bond distance between the two carbons in the fused cyclopropene ring. Table 1 shows results of HAM/3[13] and HAM/3/CI[14] calculations for 2 and 3. The X-ray structure was used for 2 and the additional C-H groups of 3 were simply added at napthalene positions. These calculations predict essentially equal first ionization potentials for 2 and 3 although the calculated values are appreciably higher than observed. The high values are not too surprising since the HAM/3 transition state method overestimates the first IP of benzene by a similar (~0.4 eV) amount. The HAM/3 method has not been previously tested on such large systems and the results of Table 1 suggest that the existing parameterization may not yet be optimal for such systems.

The position of the S_N state of 3 (7.95 eV) is shifted downwards compared to the corresponding band in the spectrum of napthalene (8.15 eV).[8] This decrease is in the direction expected for the shortened (1.355Å) C-C distance between the two carbon atoms common to the cyclopropene and napthalene rings. The HAM/3 calculations give a calculated shift of -0.19 eV in the S_N band for 3 compared to napthalene at its experimental geometry. A similar shift is calculated for the S_B position in 2 compared to benzene but the S_B band is not resolved from the other 4 states seen in the 8.75-9.7 eV group for 2. The position of the S_N band in the spectrum of 3 may be taken as a measure of the net shift attributable to the shortened bond length in the ring fusing carbon atoms in 3.

The direction of the dipole (1a) of 1 suggests that the ionic states that are localized on the two phenyl substituents in 2 and 3 might be stabilized so that they would appear at lower IP in 2 and 3 than in 4. The observed spectra

could be viewed as being in agreement with this expectation in that the maxima in the second pe bands of 2 (8.7 eV) and the third band in 3 (8.8 eV) are lower than that corresponding in 4 (9.05 eV) by 0.2-0.3 eV. The HAM/3 calculated shifts for these (S_{ϕ_2}, A_{ϕ_2}, Table 1) states, in 2 and 3 compared to 4, are between -0.24 eV and -0.34 eV. The small reduction in the positions of the second and third bands in the spectra of 2 and 3 could thus be a reflection of the increased polarization or polarizability of the ethene group in 2 and 3 compared to 4 and thus reflect the nonalternacy of the system.

The calculations at detailed geometries confirm the simple analysis given above in terms of band assignments up to the ~9.5 eV point of the observed spectra. However, the simple analysis ignored some of the basis functions in the A_i manifold which are included in the HAM/3 calculations. These include the basis function associated with the 10.26 eV band in the spectrum of 4[11] which we label A_ξ' in Table 2. Also, the naptho compound (3) should show a second symmetry factored (S) state which corresponds to the third ($^2B_{2g}$) ionic state in napthalene (10.08 eV). This state for 3 is labeled S_N' in Table 1. The calculated position of the first member of the σ manifold is also included in Table 1. The HAM/3 calculations and assignments seem to be in acceptable agreement with the experimental spectra.

The HAM/3/CI[14] results (Table 1) suggest that non-Koopmans' effects are appreciable in the ionic states of 2 and 3 beginning in the 10-11 eV region. Such shake-up phenomena give an increased number of bands which are broader than single excitation counterparts. The observed spectra of 2 and 3, between 10 and 11 eV, mildly suggest that these effects may be present but the number of states is too high to show shake up effects very clearly. As has been discussed[15] in the case of stilbene, the rotational angle of the phenyl groups with respect to the ethene group in 2, 3 and 4 may not be the same as given by

crystal structures. Additional calculations on these relative large systems at variable geometries would be required before their pe spectra can be analyzed to the non-Koopmans (configuration interaction) level. Other derivatives such as the fluorenylidene analogs of 2 and 3, would be better candidates for revealing the non-Koopmans effects.

Conclusions

The present work shows that there is little or no difference in the first ionization potentials of 2 and 3. An apparently linear correlation between $E_{\frac{1}{2}}^{+}$ values and gas phase IP's of a series of aromatic hydrocarbons indicates that the difference in $E_{\frac{1}{2}}^{+}$ values of 2 and 3 is due to structure specific solvation effects. The HAM/3 method gives acceptable agreement with the experimental spectra in terms of order of states but appears to overestimate all of the IP's to a fairly significant extent. The HAM/3/CI method indicates that shakeup phenomena are expected in this series above the 9.5 eV level. Studies of other members of this series could serve to test the $E_{\frac{1}{2}}^{+}$-IP correlation and might show the non-Koopmans effects more clearly.

Acknowledgement

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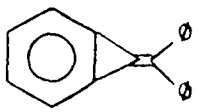
TABLE 1. Calculated and Observed Outer Valence PE Spectra of 2 and 3.



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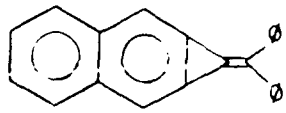

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Γ^a	obs (eV)	HAM/3	HAM3/CI[b]	obs	HAM/3	HAM3/CI[b]
A	7.15	7.67	7.53	7.14	7.66	7.59
S_N				7.95	8.21	8.14
$S_{\phi_2(1)}$[c]	8.75	8.97	9.02	8.91	8.88	9.09
A_{ϕ_2}[c]	d	9.00	9.10	d	8.92	9.17
$S_{\phi_2(2)}$[c]	9.12	9.08	9.11	9.08	9.00	9.19
S_B	d	9.52	9.83			
A_B or A_N	d	9.67	9.79	d	9.40	9.33
A_E'	10.2	10.64	10.80* 11.06*	9.95	10.02	9.99* 10.55* 10.67*
S_N'				d	10.05	10.37
σ	10.8	10.79		(10.7)	10.67	

[a]Approximate symmetry. [b]A * designates a state with appreciable shakeup character (Ref. 14). [c]State with vacancy localized on the diphenyl substituent. [d]Peak unresolved but present.

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