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In Situ Infrared Spectroelectrochemistry

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In situ infrared spectroelectrochemistry

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The vibrational spectrum of molecules at or near an electrode surface can be obtained using subtractively normalized interfacial Fourier transform infrared spectroscopy (SNIFTIRS) and electromodulated infrared spectroscopy (EMIRS). The large electric field existing at the electrode/solution interface is sufficient to induce dipole moments in highly polarizable molecules. The magnitude of the induced dipole moment is proportional to the electric field strength. As a result of this interaction some totally symmetric normal vibrations are made infrared active, and other bands appear in violation of the surface selection rule. This effect is known as the electrochemical Stark effect. Calculation of the expected absorption coefficients for the totally symmetric modes of adsorbed species gives values which are close to those observed experimentally. Experimental evidence of A_g mode activation of adsorbed pyrene is presented. The electric field dependence of the integrated absorption coefficient is demonstrated for acrylonitrile adsorbed on a gold electrode.

I. INTRODUCTION

In recent years several techniques have been developed for obtaining *in situ* the infrared spectra of species at the electrode-solution interface.[1,2] The vibrational spectra is strongly dependent upon the molecular orientation with respect to the electrode surface. Electromagnetic radiation polarized perpendicular to the metal surface (s polarized) undergoes a phase shift close to 180° for all angles of incidence resulting in a standing wave which has little amplitude at the metal surface. The electric vectors of light polarized parallel to the plane of incidence (p polarized) add constructively upon reflection at glancing angles giving rise to a sizable component of electric vector normal to the surface. Therefore, only molecules which have a component of their dipole moment normal to the surface are able to interact with electromagnetic radiation.

Since intense electric fields exist near electrode surfaces, it is possible for the electric field to interact with highly polarizable molecules. This interaction may distort the diffuse electron cloud inducing a dipole in the direction of the electric field. If the polarization of the infrared radiation is in the same direction as the induced dipole, the integrated absorption coefficient for the transition is proportional to the square of the electric field strength. Therefore, the electric field at the electrode surface may allow infrared activity for vibrations normally forbidden by symmetry or the surface selection rule. This effect is known as the electrochemical Stark effect.[6] Perturbation of vibrational spectra by strong electric fields was first predicted by Condon[3] and has been demonstrated in several systems.[4-6]

We report in this work a brief review and some new results that are related to the electrochemical Stark effect and vibronic activation of symmetric vibrational modes. The techniques used are modulated potential specular reflectance types that have recently been introduced to electrochemical science.
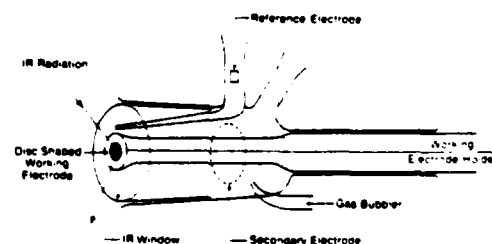
II. EXPERIMENTAL

Experiments were performed in a three-electrode thin-layer cell (Fig. 1). The working electrode consisted of an 8-10-mm-diam platinum or gold disk mounted on the end of a glass syringe barrel. The electrodes were polished to a mirror finish using alumina of decreasing sizes down to 0.05 μm, then positioned in the cell so as to trap a thin layer of solution between the metal surface and the single crystal silicon infrared transmission window. The cell was then mounted in the path of a focused infrared light beam such that the angle of incidence from the normal was slightly less than 70 °C.

Spectra were obtained using both subtractively normalized Fourier transform infrared spectroscopy (SNIFTIRS)[1] and electromodulated infrared spectroscopy (EMIRS).[7]

In the standard SNIFTIRS technique, interferograms are collected and coadded at each of two electrode potentials. After transformation of the interferograms to the frequency domain spectra, the two results are ratioed. The final spectrum represents the difference in the infrared vibrational structure of the interfacial region at the two potentials. If transmission notation is chosen for the display of the spectrum, then the result is in units of $1 + \Delta R/R$, where $\Delta R/R$, the normalized reflectivity, is composed of the difference in reflectivity of the surface region at the two potentials, and R is the reflectance of the electrode at the potential corresponding to the state used in the denominator of the original

FIG. 1. Design of the thin layer spectroelectrochemical cell used for EMIRS and SNIFTIRS experiments.

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ratio. At small values of ΔR compared to R, $\Delta R / R$ corresponds to the absorbance A. ~~throughput~~

In EMIRS, a high throughput dispersive monochromator is used. The potential at the electrode is rapidly modulated between the potentials of interest, thereby modulating the relative amounts of absorbing species in the interfacial region. This results in a small modulation of the reflectivity which is easily extracted by synchronous demodulation techniques. Details of both experimental techniques have been presented earlier.[1,7]

For cleaning cells, triply distilled water was used for steaming previously acid-cleaned cells. Aqueous solutions were prepared from recrystallized salts and triply distilled water. Acetonitrile (Burdick and Jackson, Muskegon MI) was used as received. The water content was nominally < 0.01%. Organic substrates were obtained from Aldrich and used as received. Electrochemistry was controlled by a potentiostat and waveform generator (JAS Instrument Systems, Inc.). Cells and electrodes were also obtained from JAS.

Typically, spectra were obtained at an electrode potential where the species of interest was either not adsorbed or weakly adsorbed; and at a potential where the species was present on the electrode surface or interfacial region to a large extent. The values of these potentials can be obtained usually by conventional double layer capacitance measurements which can give quantitative values of the surface coverage and information on the thickness of the electrical double layer near the interface.

III. RESULTS AND DISCUSSION

A. Calculation of B

In an experiment where the electric field ($\vec{E}_z$) is applied in a direction z, the induced dipole moment becomes

$$\mu_z = \alpha_{zz}\vec{E}_z,$$

where α_{zz} is the polarizability tensor. The integrated absorption coefficient is given by

$$B = \frac{2\pi^2 vT}{\epsilon_0 hc}|\mu_A|^2,$$

where μ_A is the transition dipole matrix element, T is the number of absorbing molecules per unit area in the beam path, and all other constants have their usual meaning. In the presence of an external electric field the transition dipole matrix element can be expressed as the sum of the permanent
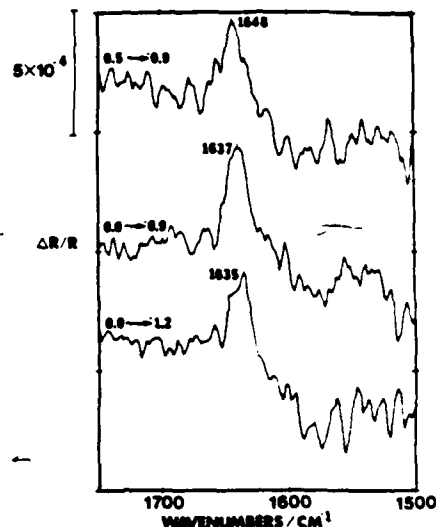

FIG. 2. SNIFTIRS difference spectrum of 0.5 mM pyrene at a platinum electrode in acetonitrile with 0.1 M tetra-n-butylammonium tetrafluoroborate (TBAF) as supporting electrolyte as a function of potential vs Ag/Ag⁻.

dipole moment (μ_0) and the induced dipole moment (μ_z) where

$$\mu_0 = \Sigma_q \langle r \rangle.$$

Thus,

$$|\mu_A| = \langle \Psi_{vf}|\mu_0 + \mu_z|\Psi_{vi}\rangle.$$

If the expression for μ_0 and μ_z are expanded in a Taylor series and the high order terms are neglected the integrated absorption coefficient becomes:

$$B = \frac{2\pi^2 vT}{\epsilon_0 hc}|\langle\Psi_{vf}|\mu_0'|\Psi_{vi}\rangle + E\langle\Psi_{vf}|\alpha'|\Psi_{vi}\rangle|$$

where μ_0' and α' correspond to the change in the permanent dipole moment and polarizability with respect to a normal coordinate, respectively. We have calculated the absorption coefficient for the $C = C$ symmetric stretch of molecules adsorbed flat at an electrode surface. For these molecules μ_0 approaches zero and we are left with evaluating the remaining matrix element. Since the matrix elements $\langle\Psi_{vf}|\alpha'|\Psi_{vi}\rangle$ are not tabulated for molecules larger than diatomic, we have estimated this change to be of the same order of magnitude as the polarizability normal to the molecular axis, providing an upper bound value to the matrix element. The polarizabilities necessary for the calculation were taken from Ref. 11. The value of the absorption coefficient is calculated as a function of electric field strength and shown in Table I.

A_g mode activation

We have observed electric field activation of the totally symmetric A_g mode of pyrene at or near the surface of a platinum electrode. Figure 2 is the SNIFTIRS spectra of 0.5

TABLE I. Predicted values for B.

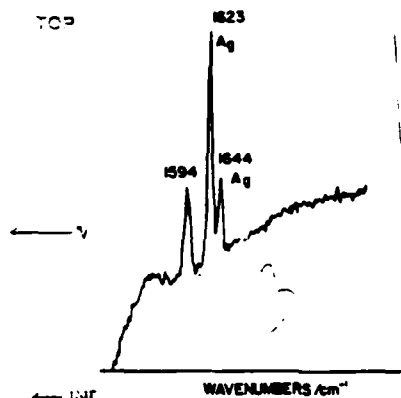| $\frac{E}{V/m}$	C_2H_4	Napthalene	Anthracene	TCNQ⁻	Pyrene
				$B/10^{-1}$ cm⁻¹	
10⁸		0.056	0.1	1.5	0.4
10⁹	0.6	5.6	11	150	36
2 × 10⁹	2.4	22	44	580	140
5 × 10⁹	15	140	290	3 600	900
8 × 10⁹	40	350	710	9 300	2 300
10¹⁰	62	560	1 100	14 500	3 600

FIG. 3. Raman spectrum of pyrene in the region 1485–1800 cm^{-1}. Ag mode assignments taken from Ref. 8.

mM pyrene in acetonitrile. The limits of the modulation potential were set between $+0.5$ and -1.2 V vs Ag/Ag$^+$ where no electrochemical reaction was occurring. Therefore, the bands appearing in the difference spectra are due to the changing electric field rather than a chemical process. The band which occurs at ~ 1640 cm^{-1} corresponds to the totally symmetric (A_g) $C–C$ stretching mode of pyrene. The Raman spectrum of pyrene was obtained in our labs and three bands were found in this frequency region (Fig. 3). The
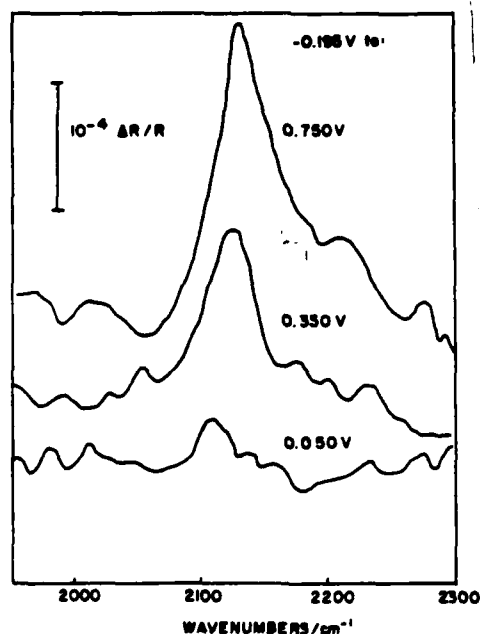

FIG. 4 EMIRS spectrum of 0.05 M acrylonitrile adsorbed at a gold electrode in 1 M H$_2$SO$_4$ as a function of potential vs SCE.
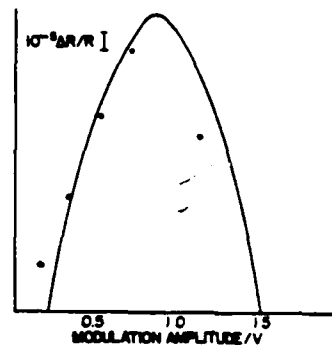

FIG. 5 Potential dependence of the intensity of the C≡N stretch band from adsorbed acrylonitrile. The dashed curve is calculated from the squared electric field dependence.

two bands at 1628 and 1644 cm^{-1} are assigned to the A_g $C–C$ stretching mode of pyrene.[8] The positions of the activated band is very dependent on the potential, and is the subject of a forthcoming report.[10]

C. Electric field dependence of B

An example of the field dependence of band intensity of an adsorbed oscillator is demonstrated in the acrylonitrile system. Differential capacity measurements show that acrylonitrile is adsorbed on gold over a wide range of potentials $(+0.5-−1.0$ V vs SCE). Spectroscopic evidence indicates that the molecule is adsorbed flat and interacts with the surface via the conjugated π system.[9]

Figure 4 is the EMIRS spectra of acrylonitrile adsorbed on gold. The band at about 2130 cm^{-1} corresponds to the C≡N stretching mode of adsorbed acrylonitrile. The intensity of the band is about an order of magnitude weaker than expected for perpendicular adsorption, and is strongly potential dependent.[7] As the potential is made more positive the band intensity increases. Figure 5 shows the band intensity as a function of the amplitude of the potential modulation. If only desorption of acrylonitrile at higher potentials was occuring, the band intensity should increase over the entire potential range and a band of opposite sign corresponding to solution free acrylonitrile should appear. However, as the modulation amplitude increases the band intensity increases, reaches a maximum, and begins to decrease. The observed spectra can be explained in terms of an electric field effect.

Since the potential dependence of the electric field at the electrode surface is unknown, it is difficult to test the exact field dependence of the intensity. However, there should be a maximum in band intensity when one of the modulation limits is at the potential point of zero charge at the interface. The dashed curve sketched in the figure is the theoretical curve predicted by the squared electric field dependence of intensity. The maximum in the curve appears at about -0.7 V vs SCE which is not an unreasonable position for the potential of zero field.

Also, the position of the band is not a linear function of

potential. In systems were perpendicular adsorption is certain, such as CO adsorbed on platinum and CN⁻ adsorbed on silver, band position is a linear function of potential.

Although the fit is only approximate it is sufficiently good to demonstrate the effect of strong electric fields on infrared spectra. Intense electric fields present near the electrode surface may interact with polarizable molecules and induce a dipole moment in the direction of the field which give rise to very weak absorptions. With refinements in the calculation it may be possible to use the electrochemical Stark effect to study the electric field intensity in the double layer region of the electrode.

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