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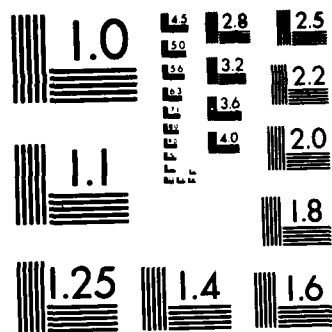
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Surface Infrared Spectroelectrochemistry. The Interaction of the Electric Field in the Electrical Double Layer with Pyrene Adsorbed on a Platinum Electrode: Effects on the Infrared Surface Difference Spectrum

By

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Prepared for Publication in
Langmuir

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Salt Lake City, Utah 84112

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The surface infrared spectrum of pyrene adsorbed at a platinum electrode is obtained by using SNIFTIRS. (Keywords: —————)		

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Surface Infrared Spectroelectrochemistry. The Interaction of the Electric Field in the Electrical Double Layer with Pyrene Adsorbed on a Platinum Electrode: Effects on the Infrared Surface Difference Spectrum

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Received January 13, 1986. In Final Form: March 27, 1986

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The surface infrared spectrum of pyrene adsorbed at a platinum electrode is obtained by using the SNIPTIRS technique. Activation of Raman modes by coupling of the polarizable electrons in the molecule to the strong electric field which exists across the electrical double layer is reported. SNIPTIRS spectra are compared to both the solution IR and Raman spectra.

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PAR03

Introduction

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The perturbation of infrared spectra by strong electric fields was suggested by Condon in 1932.¹ It was predicted that a strong static external electric field could distort polarizable electrons in a molecule in the same way that the electric vector of a light wave induced a dipole moment in symmetric molecules, effects that could be observed by Raman spectroscopy. The selection rules for absorption of infrared radiation require that the vibrational mode give rise to a dipole moment oscillating at the frequency of the exciting radiation. Application of a static electric field can distort polarizable electrons in a molecule resulting in an induced dipole moment. Oscillation of this dipole in response to motion of the nuclei symmetrically about the molecular center of mass can be detected by using infrared radiation. Thus, for highly polarizable molecules, in a strong static electric field, it is possible to detect totally symmetric (A_g) vibrational modes by infrared spectroscopy. Field-induced infrared absorption of this type has been shown convincingly for molecular hydrogen² and crystals of diamond type.³

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Other types of perturbations caused by static electric fields have been observed in vibrational spectra. Changes in the intensity of infrared-active bands have been observed both in gas phase as well as electrochemical systems.⁴ The potential dependence of vibrational frequency has also been investigated.⁵⁻⁷ Attempts have been made to give a more quantitative explanation of the role of the electric field in such systems.⁸⁻¹²

FNT 2
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Since electric fields on the order of 10^7 V/m exist across the polarized electrode/solution interface,¹³ an electrochemical system with absorbers in the interfacial region is ideally suited to study the effects of strong electric fields on infrared spectra. Recently, several techniques have been developed to obtain spectra of species in the electrical double layer.^{14,15} In this report the SNIPTIRS (subtractively normalized interfacial Fourier transform infrared spectroscopy) technique has been used to observe infrared absorption of pyrene at frequencies where, under external field free conditions, only Raman modes are active.

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Experimental Section

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Electrochemical cells used in the experiments were constructed from glass syringes (JAS Instrument Systems, Inc.). An infrared-transparent window (50 mm diameter \times 0.3 mm thickness) made of single-crystal n-type silicon was fitted on the front of the cell. The working electrode was constructed from a platinum disk (9 mm diameter \times 1 mm thick) sealed in glass and polished to a mirror finish with alumina of decreasing sizes down to 0.05 μ m. Final polishing was made from a balsa wood base to minimize the embedding of alumina particles in the platinum surface. Cells were cleaned by acid treatment (50/50 vol % sulfuric/nitric acid) followed by rinsing in triply distilled water.

Acetonitrile (Burdick and Jackson, Muskegon, MI) containing less than 0.009% (nominal) water was handled under a dry argon atmosphere. Pyrene was obtained from Aldrich and purified by vacuum sublimation. Tetra-n-butylammonium tetrafluoroborate

TXT04
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4 (TBAF) was prepared by the metathesis of sodium tetrafluoro-
12 borate (Aldrich) and tetra-*n*-butylammonium hydrogen sulfate
18 (Aldrich). It was dried in vacuo at 80 °C for a minimum of 24
15 h before use.

PAR18

SEN03 1 The electrochemistry was controlled by a potentiostat and
SEN06 10 waveform generator (Hi-Tek Instruments). The instrumentation
4 for phase-sensitive detection used in the differential capacity
SEN06 12 measurements was obtained from Bentham Instruments. Infrared
3 spectra were obtained with the use of an IBM IR/98 Series FTIR
SEN12 5 spectrometer. Light from the source was focused onto the electro-
SEN15 10 de and then reflected out of the cell onto the detector. The
2 spectrum was obtained by collecting interferograms at two
SEN18 11 electrode potentials, E_1 and E_2 . After transformation of the
6 interferograms to the frequency domain, the two spectra were
18 ratioed to give R_2/R_1 , where the R_i correspond to the reflectances
SEN21 26 at each potential E_i . The spectra are normally displayed as R_2/R_1
9 on the spectrometer, and may be converted to the more common
SEN24 20 units of $\delta R/R$ through the relation $\delta R/R = R_2/R_1 - 1$. This final
4 result represents the difference spectrum of the species being
13 observed between the two potentials.

TXT09

Results

SEN03 1

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SEN03 1 Figure 1 is a plot of the differential capacity of the
13 platinum electrode used in the SNIFTIRS experiments in
21 acetonitrile solutions (a) without and (b) with pyrene.
SEN06 1 Addition of pyrene to the system significantly lowers the
11 differential capacitance of the platinum electrode indi-
17 cating adsorption of pyrene across the entire potential
SEN09 25 range investigated. Reduction at large negative potentials
7 results in the desorption of the species and increase in the
SEN12 18 capacitance as expected. Since we are working at pyrene
8 concentrations below 0.5 mM, it is likely that the pyrene
18 is adsorbed in a flat orientation.

FIG 1 (003, 3- 4)

PAR24

SEN03 1 Figure 2 shows the electrochemical behavior of pyrene
SEN06 10 in acetonitrile. It is noted that the current is constant and
11 essentially capacitive over the region of +0.5 to -1.5 V (vs.
SEN09 22 Ag/Ag⁺). Reduction of pyrene occurs at potentials more
SEN12 9 negative than -2.4 V. To study the effect that the electric
10 field has on the spectra independent of chemical effects
SEN15 26 such as reduction of pyrene, we have collected interfero-
15 grams at potentials between +0.5 and -1.5 V. Thus, any
4 changes in the spectra are due to changes induced by the
15 electric field rather than a chemical change.

FIG 2 (003, 3- 4)

PAR27

SEN03 1 Figure 3 shows SNIFTIRS spectra in the region of the
12 ring stretching modes of pyrene as a function of the in-
SEN06 22 tensity of potential modulation. A band, 1640 cm⁻¹ is ob-
7 served, which is weakly dependent on the magnitude of
SEN09 18 the electrode potential. The position of the band appears
8 to shift to higher energy as the electrode potential is made
SEN12 19 more positive. This result was reproducible in several
SEN15 9 independent experiments. The SNIFTIRS spectra can be
7 compared to the Raman (Figure 4) and infrared (Figure
SEN18 16 5) solution spectra. The bands in the Raman spectrum
8 are assigned to symmetric C-C stretching ring modes
SEN21 16 (A_g).¹⁶ The infrared spectrum has a strong band at 1598
11 cm⁻¹ assigned to the B_{2g} ring mode and a strong band at
23 3049 cm⁻¹ assigned to the aromatic C-H stretch.¹⁶

FIG 3 (003, 3- 4)

FIG 4 (015,12-13)

FIG 5 (015,16-17)

FNT 16

PAR30

SEN03 1 Figure 6 is the SNIFTIRS difference spectra of the same
12 pyrene platinum electrode system in the region of the
SEN06 21 pyrene aromatic C-H stretch. We note the absence of any
8 SNIFTIRS absorption bands in the 3049-cm⁻¹ region.

FIG 6 (003, 3- 4)

TXT12

Discussion

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PAR33

SEN03 1 Pyrene was chosen to study field-induced infrared absorp-
9 tion because of its large molecular polarizability¹⁷ and
17 the likelihood that the molecule would undergo flat adsorption
25 on the platinum electrode under low bulk solution
SEN06 34 conditions (vide infra). Measurements have been made
SEN09 6 to ensure adsorption and flat orientation of pyrene. The
3 differential capacity of the electrode indicates that pyrene
SEN12 11 is adsorbed over the potential range studied. Further, we
4 are working at concentrations where pyrene should adsorb
SEN15 12 flat on the surface rather than edgewise. Soriaga and
4 Hubbard have studied the concentration dependence of

FNT 17-20

TXT12
 PAR33

11 the orientation of several π -electron-rich molecules ad-
 12 sorbed at platinum electrodes.²¹ They have shown that
 13 in general, flat adsorption is the primary surface configura-
 14 tion at bulk concentrations less than about 0.5 mM.

FNT 21,22

PAR34

1 The absence of an absorption band in the SNIFTIRS
 2 spectra of Figure 6 provides further evidence that pyrene
 3 is orientated flat on the surface. If the molecule were
 4 orientated edgewise on the electrode a strong band at
 5 about 3049 cm^{-1} corresponding to the aromatic C-H
 6 stretch would be expected to appear. However, if the
 7 molecule were orientated flat on the surface absorption
 8 would be forbidden. This differential absorption is known
 9 as the surface selection rule. The surface selection rule
 10 arises due to the properties of radiation reflection from a
 11 metal surface. These properties may be derived through
 12 analysis of the Fresnel relations and have been discussed
 13 in detail elsewhere.¹⁸ When infrared radiation is reflected
 14 from a metal surface, only the component polarized parallel
 15 to the plane of incidence (p polarized) has any amplitude
 16 at the surface after reflection. Light polarized perpen-
 17 dicular to the plane of incidence (s polarized) undergoes
 18 a phase shift of close to 180° for all angles of incidence
 19 resulting in a standing wave that has little amplitude near
 20 the surface. Infrared radiation will interact with an os-
 21 cillating dipole of a species when both the electric field of
 22 the radiation and the oscillator have spatial components
 23 in the same direction. Thus, only molecules that have a
 24 component of the dipole derivative (the change in the
 25 dipole moment with respect to the normal coordinate)
 26 oriented in a direction perpendicular to the surface can
 27 interact with the p-polarized radiation. The s-polarized
 28 radiation is blind to species adsorbed near the surface. For
 29 a molecule adsorbed flat on the surface, absorption of
 30 infrared radiation is forbidden by the surface selection rule.
 However, if a dipole moment is induced in the species
 perpendicular to the surface, for example, by external fields
 or bonding effects, a vibrational transition can be observed
 by using infrared radiation.

PAR39

1 The appearance of symmetry-forbidden bands in the
 2 spectra of molecules adsorbed on metal surfaces has been
 3 observed. The interpretation of such bands includes
 4 mechanisms involving chemical bonding of the molecule
 5 to the surface and interaction of the molecule with electric
 6 fields near the metal surface. The chemical mechanism
 7 suggests that bonding to the surface decreases the sym-
 8 metry of the molecule causing disallowed modes to become
 9 active.¹⁹ In addition, distortion of the molecule by dona-
 10 tion of electrons from the metal to orbitals on the molecule
 11 has also been suggested. More quantitative explanations
 12 based on electric fields present near the metal surface have
 13 been discussed. Sass et al.²⁰ have shown that electric field
 14 gradients arising from interaction of radiation with the
 15 metal surface are strong enough to couple with quadrupole
 16 moments in the molecule, giving rise to activation of in-
 17 frared-forbidden modes. In electrochemical systems, it has
 18 been shown quantitatively that large electric fields which
 19 exist across the electrical double layer are strong enough
 20 to interact with electrons of highly polarizable molecules.
 21 This interaction results in a dipole moment which can
 22 oscillate normal to the metal surface.⁹ Applying this
 23 calculation⁹ to pyrene predicts a $\Delta R/R$ on the order of 10^{-4}
 24 for an electric field strength of 10^6 V/cm. Therefore, we
 25 believe that the mechanism for appearance of bands in the
 26 SNIFTIRS difference spectrum of Figure 3 is through
 27 interaction of polarizable electrons in the molecule with
 28 the large static electric field across the double layer. This
 29 interaction can induce a dipole moment normal to the
 30 surface which can oscillate at the vibrational frequency of
 the A_1 ring mode. The band appears at potentials very
 close in energy to those observed for ring stretching modes
 in the Raman spectrum (which are infrared-forbidden).

PAR42

1 Thus, for adsorbed pyrene a dipole moment can be in-
 2 duced normal to the surface by coupling the highly po-
 3 larizable electrons in the aromatic ring of the molecule to



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SEN06 30 the electric field across the double layer. The aromatic
 4 C-H stretching modes would not be expected to be en-
 13 hanced by the electric field because of the small polariz-
 22 ability of the C-H bond.

TXT15

Conclusion

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PAR45

SEN03 1

11 This report demonstrates that the electric field in the
 18 double layer is sufficiently strong to induce infrared
 26 selection rules. Field induced absorption can be used to
 9 study the electric field in the double layer.

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PAR51

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SYF03

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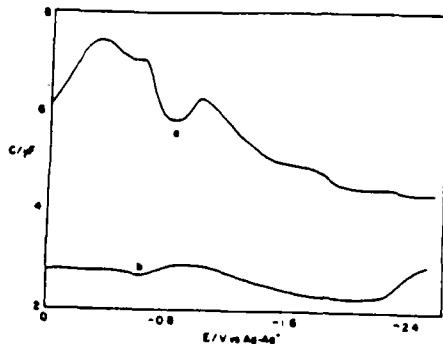
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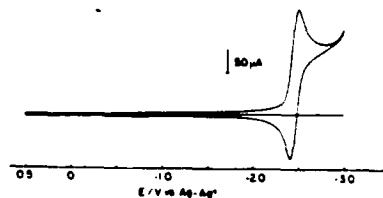
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12 1965, 89, 5246.

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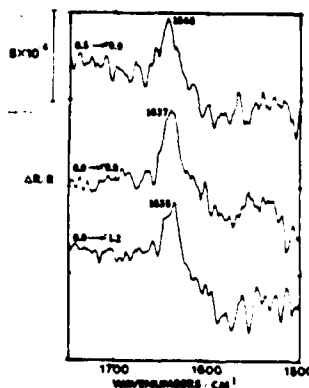


CAP00 1 Figure 1. Differential capacity curves for a platinum electrode
 CAP03 9 in a solution of (a) 0.1 M TBAF in acetonitrile and (b) same as
 CAP06 23 (a) with 0.5 mM pyrene. Results were obtained by using a 5 mV
 10 (pp amplitude) 400 Hz sine wave superimposed on a 10 mV/s
 CAP09 31 voltage ramp applied to the working electrode and measuring the
 4 in and out of phase components of the ac current. All potentials
 16 are with reference to the Ag/Ag⁰ (0.01 M Ag⁰ in acetonitrile with
 0.1 M TBAF).



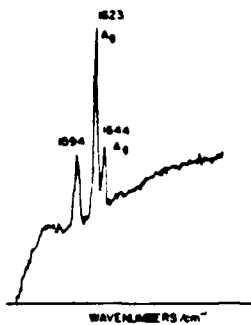
CAP00 1 Figure 2. Cyclic voltammetry of the solution in Figure 1b. Sweep
 CAP06 3 rate is 50 mV/s.

EXCEPT WITH 5 mM PYRENE

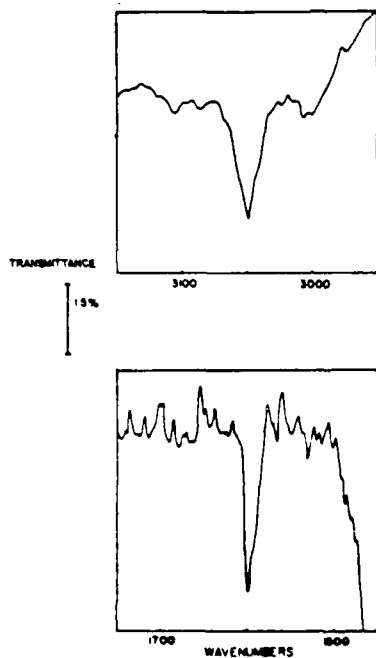


CAP00 1 Figure 3. SNIPTIRS difference spectrum of the solution in
 CAP06 9 Figure 1b between the indicated potential regions. Bands ex-
 3 tending down are present increased absorbance at the more
 12 negative electrode potential.

FNN23
FNF66

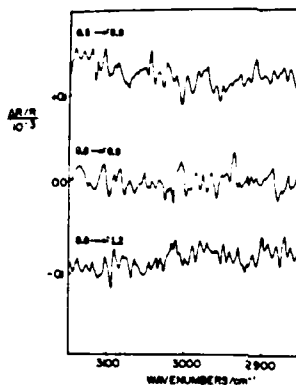


CAPO0 1 Figure 4. Solution Raman spectrum of pyrene in carbon tetra-
CAPO6 9 chloride. Band assignments are taken from ref 16



CAPO0 1 Figure 5. Solution infrared spectrum of pyrene in acetonitrile.
CAPO6 10 (a) The ring stretching mode region. (b) The aromatic C-H stretch
CAPO9 7 region (see ref 16).

FIG. 3
FN. 56



CAP00 1 Figure 6. SNIPTIRS difference spectrum in the aromatic C-H
CAP03 9 stretch region for the solution described in Figure 1b.

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Running Heads

Surface Infrared Spectroelectrochemistry

Korzeniewski and Pons

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