OFFICE OF NAVAL RESEARCH

Contract N00014-83-K-0470-P00003

R&T Code NR 33359-718

Technical Report No. 109

Infrared Spectroelectrochemistry of Surface Species: An in situ Surface Fourier Transform Infrared Study of Adsorption of Isoquinoline at a Mercury Electrode

by

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Prepared for publication in Mol. Phen. at Electrode Surf., ACS Symp. Series

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July 15, 1988

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION
Unclassified

1b. RESTRICTIVE MARKINGS

2a. SECURITY CLASSIFICATION AUTHORITY

2b. DECLASSIFICATION / DOWNGRADING SCHEDULE

3. DISTRIBUTION / AVAILABILITY OF REPORT
Approved for public release and sale.
Distribution unlimited.

4. PERFORMING ORGANIZATION REPORT NUMBER(S)
ONR Technical Report No. 109

5. MONITORING ORGANIZATION REPORT NUMBER(S)

6a. NAME OF PERFORMING ORGANIZATION
University of Utah

6b. OFFICE SYMBOL *(If applicable)*

7a. NAME OF MONITORING ORGANIZATION

6c. ADDRESS *(City, State, and ZIP Code)*
Department of Chemistry
Henry Eyring Building
Salt Lake City, UT 84112

7b. ADDRESS *(City, State, and ZIP Code)*

8a. NAME OF FUNDING / SPONSORING ORGANIZATION
Office of Naval Research

8b. OFFICE SYMBOL *(If applicable)*

9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
N00014-83-K-0470-P00003

8c. ADDRESS *(City, State, and ZIP Code)*
Chemistry Program, Code 1113
800 N. Quincy Street
Arlington, VA 22217

10. SOURCE OF FUNDING NUMBERS

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO

11. TITLE *(Include Security Classification)*
Infrared Spectroelectrochemistry of Surface Species: An in situ Surface Fourier Transform Infrared Study of Adsorption of Isoquinoline at a Mercury Electrode

12. PERSONAL AUTHOR(S)
D. Blackwood, C. Korzeniewski, W. McKenna, J. Li and S. Pons

13a. TYPE OF REPORT
Technical

13b. TIME COVERED
FROM 9/87 TO 7/88

14. DATE OF REPORT *(Year, Month, Day)*
July 15, 1988

15. PAGE COUNT
24

16. SUPPLEMENTARY NOTATION

17. COSATI CODES

FIELD	GROUP	SUB-GROUP

18. SUBJECT TERMS *(Continue on reverse if necessary and identify by block number)*
infrared spectroelectrochemistry , mercury

19. ABSTRACT *(Continue on reverse if necessary and identify by block number)*

Attached.

20. DISTRIBUTION / AVAILABILITY OF ABSTRACT
☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT. ☐ DTIC USERS

21. ABSTRACT SECURITY CLASSIFICATION
Unclassified

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22c. OFFICE SYMBOL

DD FORM 1473, 84 MAR

83 APR edition may be used until exhausted.
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ABSTRACT

Infrared spectroscopy, and in particular the method of subtractively normalized interfacial Fourier transform infrared spectroscopy (SNIFTIRS), has been used extensively to examine interactions of species at the electrode/electrolyte interface. In most experiments to date, interactions at solid electrodes have been studied. A method to probe interactions at the mercury solution has been developed and is presented in this presentation. The potential dependent frequency shifts of species adsorbed at mercury electrodes are compared with shifts observed for similar species adsorbed on d-band metals.

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INFRARED SPECTROELECTROCHEMISTRY OF SURFACE SPECIES: AN IN SITU SURFACE FOURIER TRANSFORM INFRARED STUDY OF ADSORPTION OF ISOQUINOLINE AT A STATIONARY MERCURY ELECTRODE.

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INTRODUCTION

Even though the basic technique was developed over two decades ago, infrared spectroscopy of the electrode solution interface has only yielded really useful data in the last few years (1-5). During this time, three basic techniques have been developed. The first successful method was electrochemically modulated infrared spectroscopy, EMIRS (4), which is a dispersive technique in which the electrode potential is modulated at a set frequency (10 Hz) and the resulting attenuation of the reflecting infrared radiation is analyzed by phase sensitive detection. The second common infrared technique used in electrochemistry is infrared reflection absorption spectroscopy, IRRAS (6), in which the polarization of the incident infrared radiation is rapidly modulated (74 kHz) between the s- and the p-states. As s-polarized light undergoes a 180° phase shift upon reflection from a metal surface, the electromagnetically induced standing wave develops a node at the surface so that there is effectively no field intensity. As a result, only p-polarized light can interact with species which are either adsorbed onto, or within a percentage of a wavelength of, the electrode surface. The perturbed infrared signal is again monitored with the aid of phase sensitive detection techniques. The third technique is subtractively normalized interfacial Fourier transform spectroscopy, SNIFTIRS (7). Here, pseudo steady state infrared spectra are collected at two different electrochemical states or potentials, so that the electrochemical difference spectra can be obtained simply by subtracting one from the other and normalizing.

In situ infrared reflection techniques can distinguish between absorption bands arising from solution species from those due to species adsorbed on the metal electrode surface by noting the rules listed in Table 1. These rules arise due to the difference in the amount of phase shift undergone upon reflection by s- and p-polarized light as discussed above, and to the fact that the strength of the chemical bond between an adsorbed species and the electrode is expected to be a function of the electrode potential, as this will govern the amount of overlap that can occur between the bonding and antibonding orbitals of the adsorbate.

To illustrate the potential dependence of adsorbed species, the SNIFTIRS spectra of the b_{3u} ring bending mode of p-difluorobenzene at a platinum electrode is shown in Figure 1 (8). The upward pointing band is clearly independent of potential and was thus assigned to a solution species whereas the position of the downward pointing band shifts monotonically (at constant ionic strength) with potential and is therefore clearly due to some adsorbed species.

The particular system investigated for this presentation was the adsorption of isoquinoline on mercury surfaces, which has been previously studied by electrocapillary methods (9), ellipsometry (10), double layer capacity measurements (11), and a range of potential step techniques (12-15). The interest in this system is due in part to the fact that one observes well defined transitions in its physical properties as the adsorbed molecules undergo transitions in surface orientation and packing density under certain experimental conditions. Isoquinoline molecules has been shown to be adsorbed on mercury in four different orientations (Figure 2). The previous investigations indicate the following behavior for the isoquinoline orientation as a function of potential and concentration: at low negative potentials and low bulk concentrations, the molecules are believed to lie flat on the mercury's surface (molecular plane parallel to the surface). However, on increasing either the potential (in the negative direction) or the bulk concentration, the isoquinoline molecules are forced up into either the 4,5 position (10) or the 5,6 position (9). This reorientation occurs gradually with the changing coordinates, and proceeds through a series of phases containing mixtures of these three orientations of isoquinoline molecules.

Increases in the potential to more negative values, and at sufficiently high concentrations, results in an abrupt reorientation to the 6,7 position. The reason that this second transition is much sharper than the first lies in the fact that mixed phases which would contain the 6,7 orientation are energetically less favorable than a complete monolayer of any of the pure standing orientational phases. Gierst et al (9) have produced a graph showing the dependence of the superficial excess on both potential and bulk concentration from their electrocapillary data; we reproduce some of their data in Figure 3.

For an isoquinoline molecule adsorbed onto the surface of mercury, the component of its total dipole moment that is perpendicular to the surface will increase as its orientation changes from:

Flat - 4,5 - 5,6 - 6,7

The surface selection rules for infrared radiation reflection from metal surfaces and its interactions with adsorbed species are such that infrared radiation should be absorbed most strongly by vibrational modes that have a component of the dipole derivative (with respect to the normal coordinate) normal to the metal surface. Therefore as the isoquinoline molecule reorients in the order listed above, the absorption of infrared radiation by the in-plane vibrational modes would be expected to increase, while that of the out-of-plane modes would be predicted to decrease. In the flat orientation there is no component of the dipole moment perpendicular to the surface for the in-plane modes, thus it maybe be thought that these modes will not be able to absorb any of the incident radiation. However, infrared active modes (and in some cases infrared forbidden transitions) can still be observed due to field-induced infrared absorption (16-20).

EXPERIMENTAL

Isoquinoline (Aldrich 97%) was further purified by refluxing with BaO for 30 minutes and distilling under vacuum. The resulting white crystalline solid had a melting point of 26°C. The purified isoquinoline was stored in the dark, at 0°C and under an argon atmosphere. Mercury was triple distilled (American Scientific) and all other chemicals were of AnalaR grade quality. All solutions were prepared with triply distilled water. All glassware was cleaned in a 50:50 (v:v) mixture of HNO_3 and H_2SO_4, rinsed with triply distilled water and steamed (triply distilled water) for half an hour. Because water strongly absorbs infrared radiation a thin layer cell was designed (Figure 4) which could be mounted in a vertical position on the spectrometer. The mercury was held in position by a simple glass tube, and electrical contact was achieved with a piece of platinum wire, dipped directly into the mercury. All potentials reported are with reference to a saturated calomel electrode.

The technique used to acquire all the data shown in this paper was SNIFTIRS, a schematic diagram of the required apparatus is shown in Figure (5). The FTIR spectrometer used was a vacuum bench Bruker IBM Model IR/98, modified so that the optical beam was brought upwards through the sample compartment and made to reflect from the bottom of the horizontal mercury surface. The infrared study of mercury electrode surfaces has been impeded by experimental difficulties in cell design and optical considerations; the methods used herein are adapted from a configuration that has been used by Bewick and co-workers (21).

RESULTS AND DISCUSSION

The abrupt transition to the 6,7 orientation manifests itself in cyclic voltammetry as a sharp current spike (Figure 6). The cathodic spike was found to contain a charge of 2.9 $\mu C\ cm^{-2}$, while its anodic counterpart contained 3.3 $\mu C\ cm^{-2}$. The peak separation was 100 mV, although this relatively large value is due in part to the high iR drop present in thin layer cells.

Figure 7 shows typical SNIFTIRS spectra for isoquinoline molecules adsorbed on mercury. The reference spectrum in each case was obtained at 0.0V vs. SCE reference electrode; at this potential the molecules are believed to be oriented flat on the metal surface. The vibrational frequencies of the band structure (positive values of absorbance) are easily assigned since they are essentially the same as those reported by Wait et al.(22) for pure isoquinoline. The differences in the spectra are that the bands for the adsorbed species exhibit blue shifting of 3-4 cm^{-1} relative to the neat material, and the relative intensities of the bands in each case are markedly changed.

The major vibrational modes observed for isoquinoline are listed in Table 2. The assignments made by Wait et al.(22) are also included. These authors made their assignments from considerations of the higher $D_{\infty h}$ symmetry parent species, instead of the C_s symmetry group; they demonstrated that the assignments arising from this representation are reasonable.

The difference spectra show a complete absence of bands with negative absorbances (Figure 7), this can be explained if the vibrational frequencies of the bands do not shift with changes in the electrode potential, and that they are adsorbed over the entire potential region investigated. This is consistent with results of electrochemical double layer experiments. The spectra can now be interpreted as relatively simple changes in the absorption of infrared radiation.

The intensities of the bands are markedly potential dependent; an especially large change in the intensities is observed at potentials where the orientation changes to the vertical 6,7 configuration.

In order to explain the lack of potential dependence of the vibrational frequencies of the bands it is necessary to examine how this characteristic of the vibrational modes of adsorbed species is believed to arise. Two types of mechanisms have been proposed to explain the potential dependent shift of vibrational frequencies. The first involves molecular orbital arguments; the second is based on arguments for interactions between the electric field across the double layer and the highly polarizable electrons of the adsorbed molecule (an electrochemical Stark effect). In the molecular orbital mechanism, electrons can be donated to empty metal orbitals through σ-type overlap with filled ligand orbitals of the appropriate symmetry. The metal can "back" donate electrons from filled d-orbitals to empty π^* antibonding orbitals on the adsorbate. When a molecule is adsorbed on a clean uncharged metal surface, its vibrational frequency may either increase or decrease from the frequency of the unadsorbed molecule depending upon the relative contributions of the σ- and π-bonding interactions. If the π-bonding interaction is dominant the frequency will decrease; conversely, the frequency will increase if the σ-bonding interaction is dominant. When the charge on the electrode is made negative, the bond is weakened due to donation of charge from the metal into adsorbate π^* orbitals and the band frequency shifts to lower wavenumber. When the charge on the metal is made positive a shift to higher frequency occurs. At a mercury electrode, however, there are no p- or d-electrons available to participate in a back-bonding interaction. The observation of potential dependent frequency shifts are therefore not expected according to this model.

The electric field mechanism involves coupling of the electric field across the double layer with highly polarizable electrons of the adsorbate. According to the Gouy-Chapman-Stern model, for high concentrations of supporting electrolytes, most of the potential drop will occur within the first 5-10Å of the electrode surface, and the drop will be approximately linear with distance. When a layer of adsorbed species is present, it can act as a dielectric across which the greatest portion of the potential drop will occur. Electric fields on the order of 10^9 V m^{-1} can exist in this region. Interaction of this electric field with the dipole moment of the molecule leads to changes in the vibrational frequency of the molecule.

The absorbances in Figure 7 have positive values. This indicates that the absorption of infrared radiation is strongest when the isoquinoline molecules are lying flat on the electrode surface (in these difference spectra, positive values of absorbance denote stronger absorption at the positive potential, i.e. potentials where the isoquinoline is adsorbed in the flat configuration). This is an opposite result than that suggested from the surface selection rule, and suggests that there is a strong field-induced absorption for the in-plane modes in this configuration, similar to that observed in previous work for pyrene adsorbed on platinum (18).

A closer examination of the SNIFTIRS difference spectra shows that there are marked differences in the changes in intensity of the in-plane and out-of-plane vibrational modes of the adsorbed isoquinoline with potential (Figure 8). The normalized intensities (against their intensity at -0.1V vs. SCE) of the vibrational bands shown in Figure 7 were plotted against potential (Figure 9). Since the bands in Figure 7 have positive values of absorbance, the positive vertical axis in Figure 9 represents a decrease in the amount of infrared radiation absorbed. The figure clearly shows that the amount of radiation absorbed by the out-of-plane vibrational modes decreases up to a factor of 10 as the potential is changed from -0.1 to -1.0V, whereas the in-plane vibrational modes only change by a factor of 3-4 over the same potential range. (We point out at this point that solution soluble isoquinoline would not exhibit this effect).

The explanation of the trends seen in figure 9 is that the out-of-plane vibrational modes have dipole derivative changes perpendicular to the metal surface when the molecules are lying flat on the surface. Absorption of radiation by the surface selection rule is thus allowed. When the orientation changes to the 6,7 configuration, absorption by these modes, which are now parallel to the surface is forbidden, as is any field induced interaction since most of the molecule lies outside of most of the field gradient. A large decrease in absorption (large increase in positive absorbance in the difference spectra) is thus expected and observed. In the case of the in-plane modes, the high electric field in the double layer leads to strong field induced absorption for the flatly adsorbed isoquinoline as expected. When reorienting to the 6,7 configuration, the modes become allowed by the surface selection rule. A smaller decrease in absorption is then observed.

CONCLUSIONS

Subtractively normalize interfacial Fourier transform infrared spectroscopy has been successfully used to follow the reorientations of isoquinoline molecules adsorbed onto a mercury electrode. It has been shown that field-induced infrared absorption makes a major contribution to the intensities of the vibrational band structure of aromatic organic molecules adsorbed on mercury.

The isoquinoline was observed to go through an abrupt reorientation at potentials more negative than about -0.73 V vs SCE (the actual transition potential being dependent on the bulk solution concentration) to the erect 6,7 standing position.

There was a lack of any potential dependence in the frequencies of the vibrational modes of the isoquinoline adsorbed to the mercury surface. The explanation of this may be the fact that mercury has no available vacant π- *op* δ-orbitals into which back-bonding (which is observed on all d metals) can occur. In this case, therefore, adsorption occurs only through σ-bonding. This point is under continued investigation.

ACKNOWLEDGEMENTS

We thank the Office of Naval Research for support of this work.

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Table 1. A comparison of the characteristics of infrared vibrational bands arising from bulk solution species and adsorbed species.

BULK SOLUTION SPECIES	ADSORBED SPECIES
i) Bands appear with both s & p polarized light.	i) Bands appear with only p polarized light.
ii) Band positions independent of potential.	ii) Band positions may shift with potential.
iii) Relative intensity between bands is independent of potential.	iii) Relative intensity between bands may change with potential.
	iv) Some normally ir active bands may not be observed.
	iv) Some normally ir inactive bands may be observed.

Table 2. Assignments of the major infrared bands for isoquinoline at the mercury /water interface.

Band	Assignment	C_s	D_{2h}	In- or out-of-plane
1628	ν_8	A'	B_{3g}	In
1589	ν_9	A'	A_g	In
1575	$\nu_{26} + \nu_{38}$	A' + A''	$A_g + B_{3u}$	In + Out
1500	ν_{11}	A'	B_{2u}	In
1462	ν_{12}	A'	A_g	In
1435	ν_{13}	A'	B_{3g}	In
1380	ν_{14}	A'	A_g	In
1376	ν_{15}	A'	B_{1u}	In
1273	ν_{17}	A'	B_{1u}	In
1257	ν_{18}	A'	B_{2u}	In
1215	$\nu_{36} + \nu_{42}$	A''+ A''	$A_u + B_{1g}$	Out + Out
1180	ν_{19}	A'	B_{3g}	In

FIGURE LEGENDS

Figure 1. SNIFTIRS difference spectra of the b_{3u} ring bending mode of p-difluorobenzene at a platinum electrode as a function of modulation potential.

Figure 2. Possible orientations for the adsorption of isoquinoline on mercury.

Figure 3. Superficial excess as a function of potential for a mercury electrode in contact with 0.5 mol dm^{-3} Na_2SO_4 and the following isoquinoline concentrations: (a) 2.1 x 10^{-2} (saturated), (b) 6.3 x 10^{-3}, and (c) 2.1 x 10^{-4} mol-dm^{-3}. (Data from Gierst et al.,(1))

Figure 4. Construction of the thin layer mercury reflectance cell used for all experiments.

Figure 5. Schematic repesentation of the instrumentation used for SNIFTIRS experiments.

Figure 6. Cyclic voltammogram for a mercury electrode in contact with a solution 2.1 x 10^{-2} mol-dm^{-3} in isoquinoline / 0.5 mol-dm^{-3} Na_2SO_4 at 10 mV-s^{-1}.

Figure 7. SNIFTIRS difference spectra for a mercury electrode in contact with a solution that is 1.3 x 10^{-2} mol-dm^{-3} in isoquinoline. Reference potential at 0.0 V vs SCE, sample potential (a) -0.60V, (b) -0.75V, (c) -0.80V and (d) -0.9V vs SCE.

Figure 8. Expanded section of Figure 5 showing examples of differences in the magnitude of integrated area for two absorption bands with the same changes in electrode potential. The potentials for the difference spectra are -0.6 and -0.9V. The change in area for the 1380cm^{-1} in-plane mode has increased by a factor of ≈4 , whereas that of the 1215cm^{-1} out-of-plane mode has increased by a factor of ≈10.

Figure 9. Plot of the normalized intensities of the bands observed in the SNIFTIRS difference spectra at a mercury electrode in contact with a 1.3×10^{-2} mol·dm^{-3} solution of isoquinoline vs the sample potential. Reference potential = 0.0V vs SCE.

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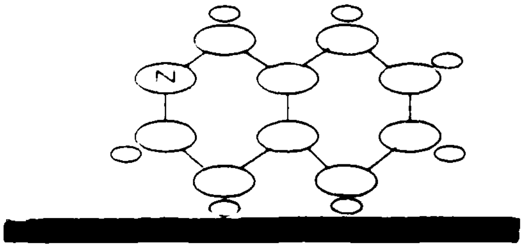

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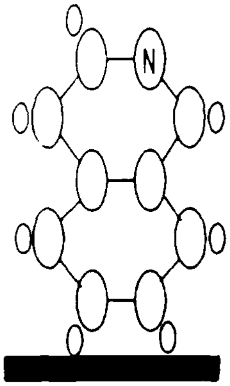

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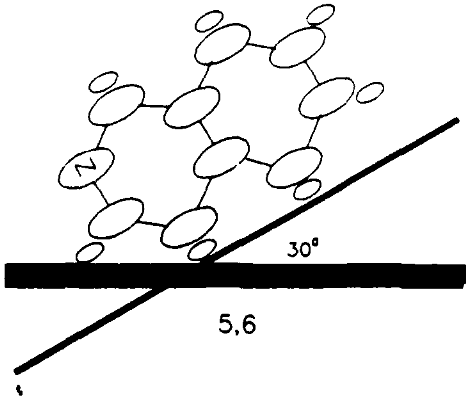

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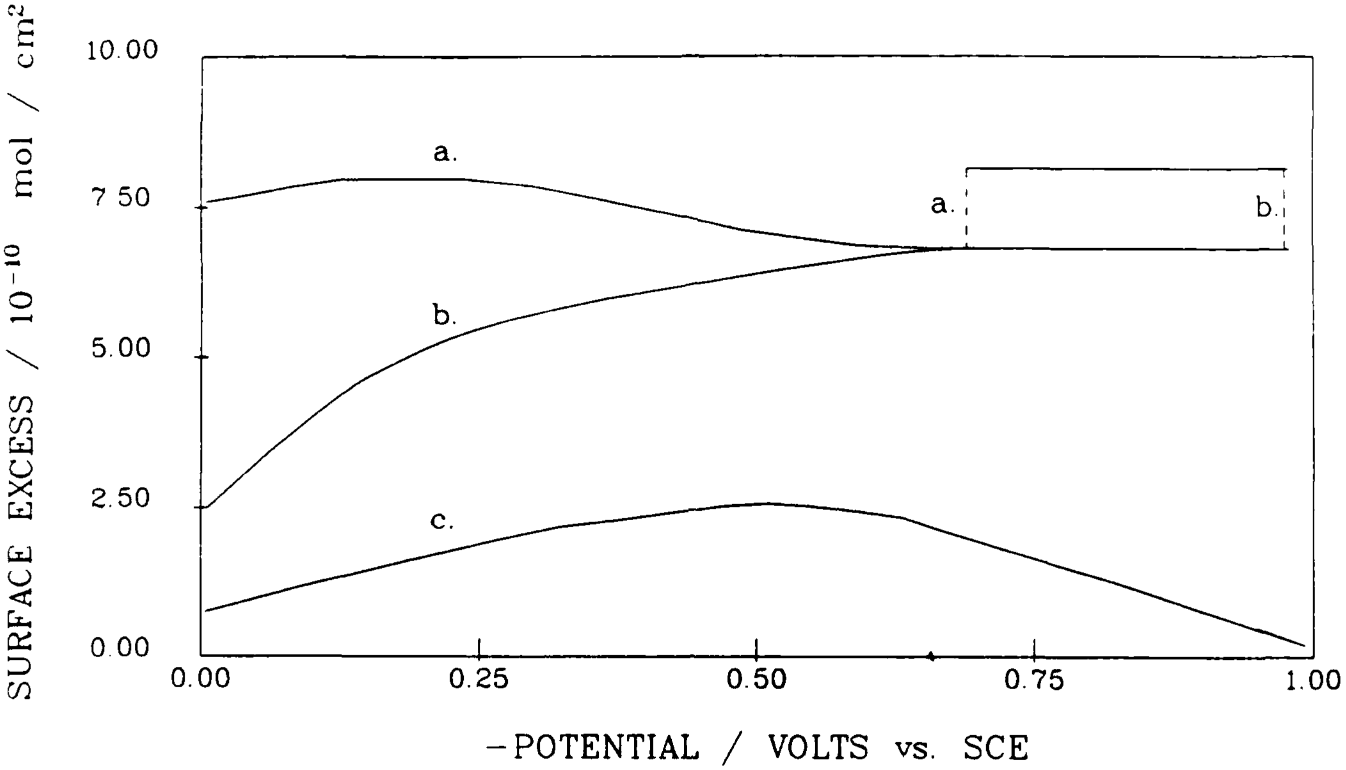

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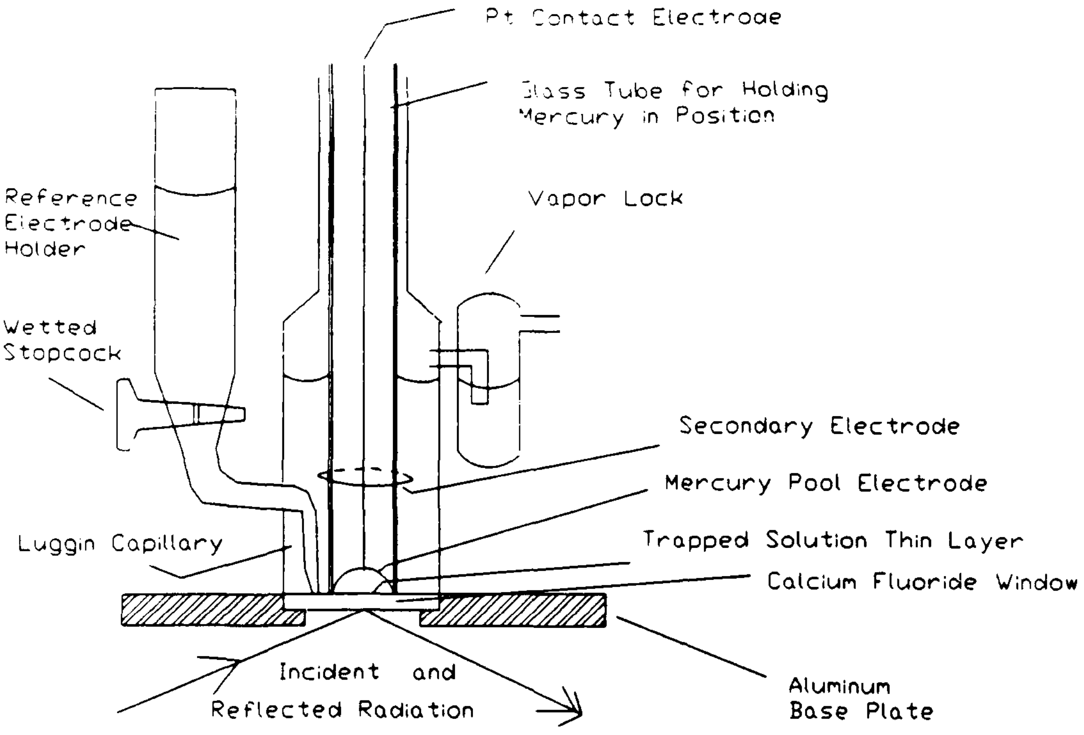
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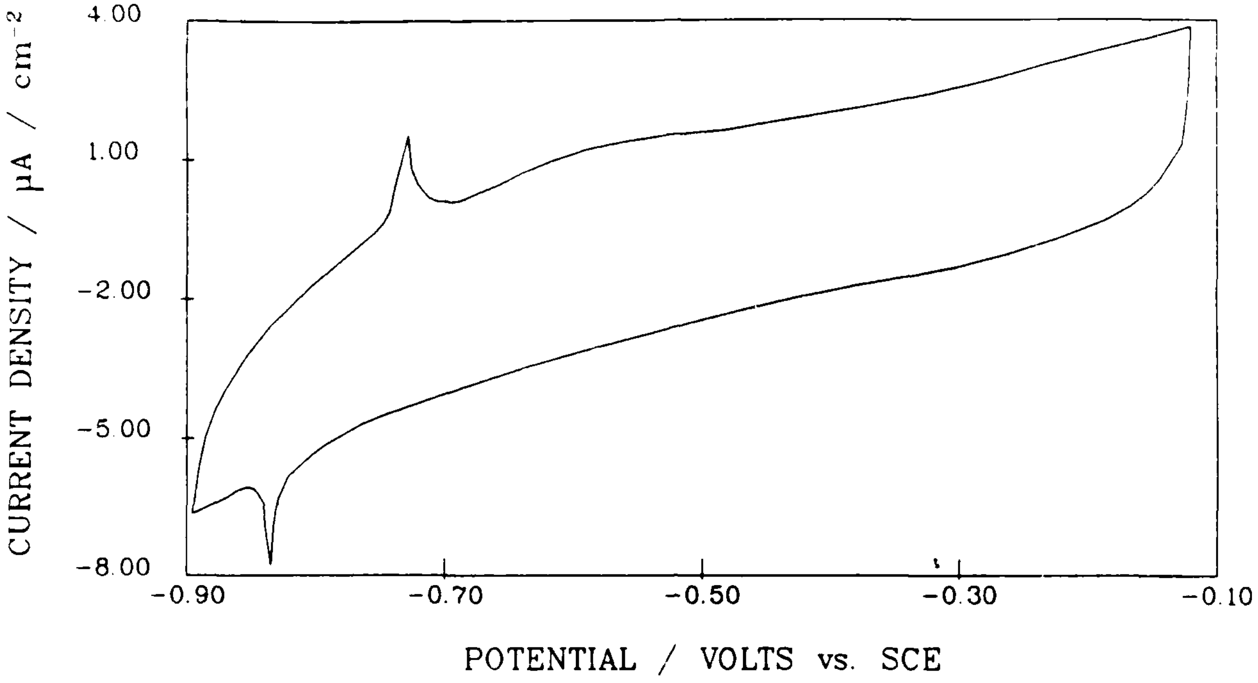
Incident and Reflected Radiation
Aluminum Base Plate



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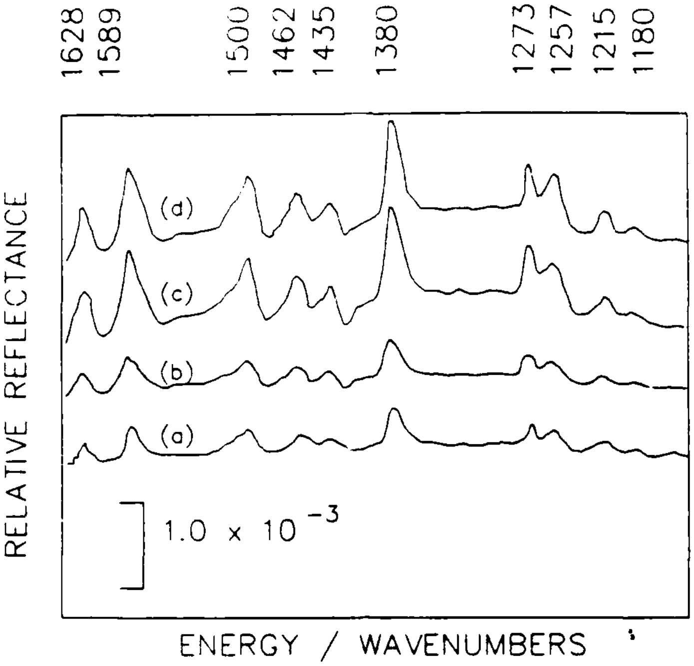

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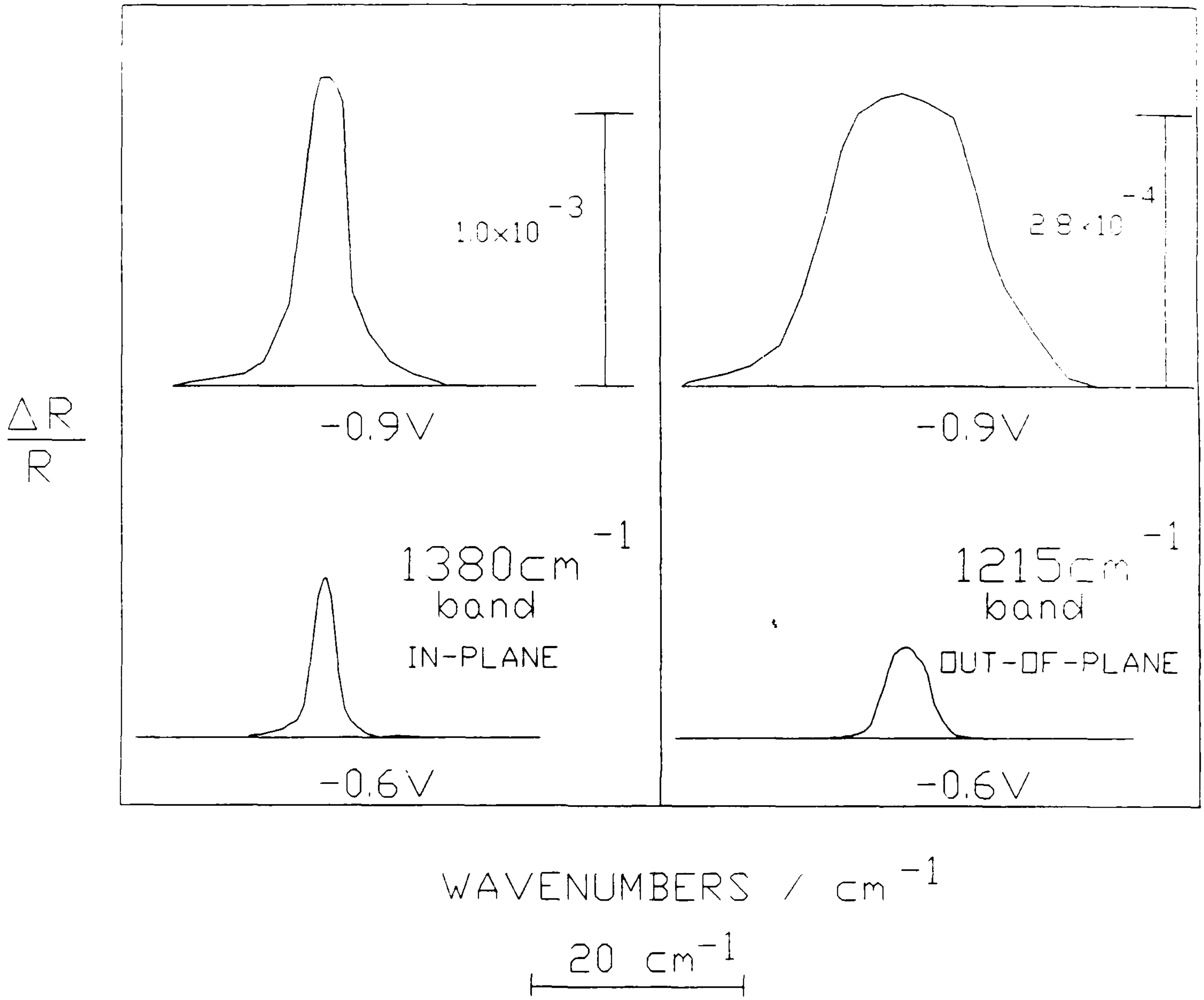
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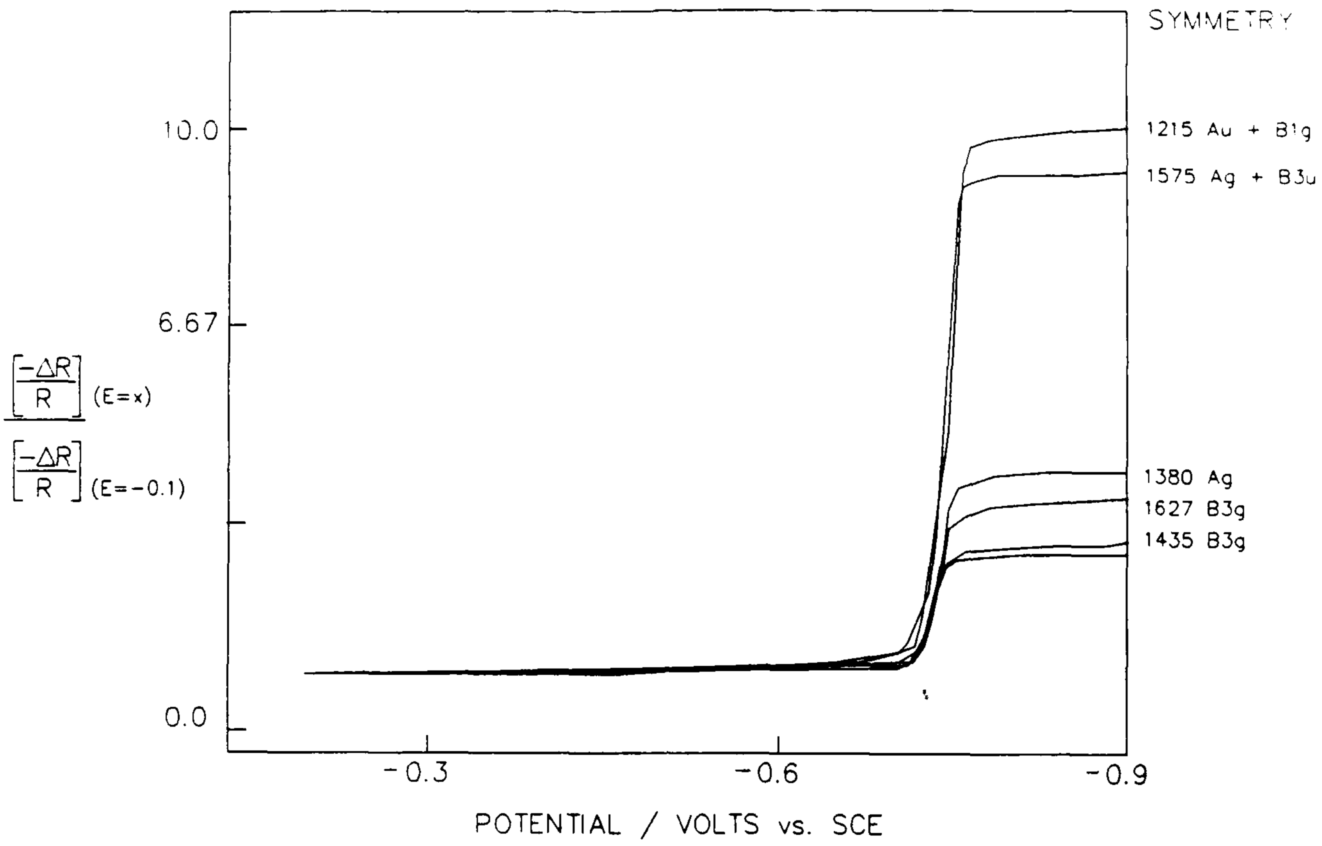
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-0.6
-0.9
POTENTIAL / VOLTS vs. SCE
1215 Au + B1g
1575 Ag + B3u
1380 Ag
1627 B3g
1435 B3g

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