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VIBRATIONAL SPECTROSCOPY OF THE ELECTRODE-SOLUTION INTERPHASE. --ETC(11)

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OFFICE OF NAVAL RESEARCH
Contract N00014-82-G-0017
Task No. NR 359-718
TECHNICAL REPORT NO. 15

Vibrational Spectroscopy of the Electrode-Solution Interphase. II.
Use of FTIR Spectroscopy for Recording IR Spectra of Radical Ion
Intermediates.

by
Stanley Pons, Timothy Davidson, and Alan Bewick*

Prepared for Publication in

Journal of the American Chemical Society

University of Alberta
Department of Chemistry
Edmonton, Alberta, Canada
T6G 2G2

*Department of Chemistry
The University
Southampton, U.K.

August 18, 1982

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REPORT DOCUMENTATION PAGE		
1. REPORT NUMBER 15	2. GOVT ACCESSION NO. AD-A111870	3. REPORT TYPE AND PERIOD COVERED 705
4. TITLE (and Subtitle) Vibrational Spectroscopy of the Electrode-Solution Interphase. II. Use of FTIR Spectroscopy for the Recording of IR Spectra of Radical Ion Intermediates.		5. TYPE OF REPORT & PERIOD COVERED Technical Report # 15
6. AUTHOR(s) Stanley Pons, Timothy Davidson, and Alan Bewick		7. PERFORMING ORG. REPORT NUMBER N00014-82-G-0017
8. PERFORMING ORGANIZATION NAME AND ADDRESS Department of Chemistry University of Alberta Edmonton, Alberta, Canada T6G 2G2		9. CONTRACT OR GRANT NUMBER(s) Task No. NR 359-718
10. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research Chemistry Program - Chemistry Code 472 Arlington, Virginia 22217		11. REPORT DATE August 18, 1982
12. DISTRIBUTION STATEMENT (of the abstract entered in Block 26. If different than Report) This document has been approved for public release and sale; its distribution is unlimited.		13. NUMBER OF PAGES 24
14. SUPPLEMENTARY NOTES		15. SECURITY CLASS (of this report) Unclassified
16. KEY WORDS (Continue on reverse side if necessary and identify by block number) Electrochemistry, FTIR, Spectroelectrochemistry, Radical Ions, IR Spectra		17. DISTRIBUTION STATEMENT (of the abstract entered in Block 26. If different than Report)
18. ABSTRACT (Continue on reverse side if necessary and identify by block number) Results for the recording of the radical ions formed in the reduction of benzophenone, tetracyanoethylene and anthracene are presented.		19. DISTRIBUTION STATEMENT (of the abstract entered in Block 26. If different than Report)

DD FORM 1, 1473 EDITION OF 1 NOV 65 IS OBSOLETE
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ABSTRACT

High quality infrared spectra of the radical ions of benzophenone, anthracene and tetracyanoethylene were recorded during electrolysis of the substrates at a platinum electrode using reflectance techniques in a Fourier transform infrared spectrometer. Evidence of adsorption of benzophenone was indicated, whereas simple ion spectra were observed for anthracene and tetracyanoethylene. Versatility of the technique is discussed.

VIBRATIONAL SPECTROSCOPY OF THE ELECTRODE-SOLUTION INTERFACE.

II. USE OF FOURIER TRANSFORM SPECTROSCOPY FOR RECORDING
IR SPECTRA OF RADICAL ION INTERMEDIATES

Stanley Pons* and T. Davidson

Department of Chemistry
University of Alberta
Edmonton, Alberta, Canada
T6G 2G2

and

A. Bewick

Department of Chemistry
The University
Southampton, Hampshire
SO9 5NH
U.K.

* To whom correspondence should be sent.

The investigation of heterogeneous and homogeneous electrode processes by infrared coupled spectroelectrochemical techniques has been reported. Thus work on the electrosorption of methanol on platinum electrodes (1), adsorbed hydrogen on platinum (2,3), studies of the platinum-acetonitrile interface, (4) adsorption of acrylonitrile (5), and changes in the water structure at metal electrodes (3,6) has been completed.

The use of Fourier transform infrared spectrometers has proven to be a convenient rapid method for obtaining infrared spectral differences between two polarization potentials at an electrode interface (4). We report here the ease with which the vibrational spectra of free radical ions may be observed using this technique. Traditionally, it has been rather difficult to obtain free radical ion spectra in the infrared region for obvious reasons of sample handling and the large IR absorptions of the common solvents used when generating ion radicals. Conventional techniques used to observe such spectra include co-condensation of the parent molecule and alkali metal atoms onto infrared transparent windows (7), examination of salts of

prepared salts of stable radical ions (8), and crossed molecular beam co-condensation techniques (9-11). If the radicals of interest can be generated electrochemically, then it is likely, using the technique described herein, that their infrared spectra may be recorded with minimal experimental effort. The traditional problem of strong solvent absorption is minimized by using reasonably thin layers of solution and efficient software subtraction routines available with most commercial FTIR instruments.

EXPERIMENTAL

FTIR Spectroscopy and Electrochemical Generation of Ions

Spectra were recorded on a Nicolet 7199 Fourier transform infrared spectrometer with a type A mercury-cadmium-telluride solid state 77°K cooled detector. Light was polarized by a Cambridge optical polarizer mounted before the cell. Potential programs were generated at the electrode by a MI-TEK UT2101 potentiostat and PPK1 waveform generator. The spectra were taken after near steady state conditions had been reached at the appropriate potential. Synchronization between the application of the potential step and the beginning of the spectral data collection was made by tapping the TTL signal accompanying the beginning of data collection at the Nicolet A/D converter and applying the signal to a 74190 down counter. Thus the waveform

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generator could be triggered at chosen sweep increments. Further details are given elsewhere (12).

Solvents and Chemicals

Acetonitrile (Caledon HPLC grade, 0.003% water) was used without further purification. Benzophenone (Aldrich) was vacuum sublimed twice before use. Anthracene (Aldrich) was used as received, and tetracyanoethylene was recrystallized twice from chlorobenzene. All solutions were degassed with argon before use.

Tetrabutylammonium tetrafluoroborate (TBAF) (13) was the supporting electrolyte used in all solutions.

Cells, Mounts and Electrodes

The details of the cells and associated mountings have been described elsewhere (12). In these experiments a polished platinum mirror electrode was used, and the reference electrode was Ag/Ag⁺ (0.01 M in 0.10 M tetrabutylammonium tetrafluoroborate).

RESULTS

The differential reflectance FTIR spectrum of a 10 mM solution of benzophenone in acetonitrile (0.1 M TBAF) at a platinum mirror electrode is shown in Figure 1b. The potential limits were -1.75 V and -2.50 V vs the Ag/Ag⁺ reference. The

light was polarized such that the plane of the electric field vector was perpendicular to the surface of the electrode (p-polarized with respect to the plane of incidence). The thickness of the solution layer was 1.0 microns. The corresponding transmission spectrum of benzophenone in the same cell arrangement is shown in Figure 1a. As will be discussed later, bands extending downward (Figure 1b) correspond to increased absorbance at the higher potential (-2.75 V), while peaks extending upward correspond to decreased absorbance at the higher potential. Thus the downward extending bands correspond to absorbance due to the anion radical, while the upward extending bands correspond to disappearance of the substrate benzophenone as it is reduced to form the anion radical. Table 1 lists absorbance bands observed and compares them with literature values. Slight differences in wavenumber is attributed to solvent effects (the literature values were determined in carbon tetrachloride (benzophenone) and tetrahydrofuran (benzophenone ketyl anion radical)). There is a very good correlation between this work and reported values, except for the weakest bands. No attempt was made to enhance the signal to noise by further signal averaging. The reported spectra were the average of 40 scans (about 5 minutes).

The band at 1464 cm⁻¹ in Figure 1b has not been previously reported for the benzophenone ketyl anion radical. If the solution thickness is increased to 200 microns, and the benzophenone in the thin layer of solution is 90% reduced to the ketyl structure, the difference spectrum has a greatly enhanced intensity and it is not possible to detect the band at 1464

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cm^{-1} . The 1664 cm^{-1} band is also absent from the spectrum if the light polarization is changed by 90° to the s-polarized form. A comparison of the p-polarized spectrum with an s-polarized spectrum is shown in Figure 2. We note changes in bandwidths and intensities of the bands as a function of polarization, and wavelength.

Figures 3a and 3b show the spectrum of anthracene and the difference spectrum of anthracene between -1.5 V and -2.5 V respectively. The light was p-polarized and the solution thickness was 14 microns. The solvent and other parameters were the same as noted above for benzophenone.

Figure 4a and 4b show the results for the tetracyanoethylene (TCNE) system. The modulation region was $+0.25 \text{ V}$ to -0.25 V . The solution thickness was 17 microns.

DISCUSSION

Care must be taken when interpreting difference spectra. In Figures 3 and 4, where the extinction coefficients of the products (the anion radicals of anthracene and tetracyanoethylene) are apparently much greater than the reactants (11) then the results are quite straightforward. The resulting difference spectra closely resemble pure absorption spectra of the species formed at the more negative potential. In spectra where the extinction coefficient of reactant and product are about the same (Figure 1), the spectra may be more complex. A discussion of these effects has been made previously (12).

There are several considerations that need to be made when interpreting the results of experiments that use polarized light

for reflection studies at metal interfaces. First, s-polarized light has an almost zero electric field intensity at the surface of the metal especially at IR wavelength, and therefore cannot interact with dipole oscillators of any orientation that might exist at that surface, i.e. s-polarized radiation is "blind" to adsorbed species. Second, p-polarized light will interact with dipole oscillators encountered in solution, and also dipole oscillators at the metal surface oriented such that the dipole derivative with respect to the normal coordinate, $(\partial\mu/\partial y)$, has a non-zero component perpendicular to the electrode surface, i.e. p-polarized radiation sees adsorbed species but the absorption bands observed depend upon the orientation of the molecule (6). Third, for s-polarized light, the spectra observed may possess features from: (a) absorption by species present in the bulk solution as the IR beam is transmitted through it, reflected off the electrode, and transmitted back through the solution again; (b) internal reflectance absorption at the interface between the inner face of the window and the bulk solution. This latter effect is rather complex and can lead to severe distortion of the normal spectra. Many examples of this phenomenon have been reported (14), and it is apparent that for the purposes herein, it is very difficult to obtain quantitative data from spectra obtained with s-polarized light.

Thus there are two simple ways in which to distinguish between the spectra of intermediates that are adsorbed at the

14. See for example N.J. Harrick, "Internal Reflection Spectroscopy", Interscience, New York, 1967.

electrode surface and free in the thin layer of solution. The former will give bands only with p-polarized radiation, whereas the latter will show no partiality towards either polarization state. The other test is the comparison between the spectra obtained for following the generation of smaller quantities of intermediate and much larger amounts. The former will have significant contributions from both adsorbed and non-adsorbed species, while the latter will show bands of greatly increased intensity for the non-adsorbed species with little or no increase in the bands from the adsorbed state. It is also important to note that, in general, only some of the bands from an adsorbed species will be expected at the same frequency as those of the corresponding non-adsorbed species; others will be shifted due to the perturbation in structure and bonding accompanying the interaction with the atoms in the metallic surface. In the case of benzophenone these effects are observed and it is clear that the anion radical is present in both the adsorbed and the non-adsorbed state. Two new bands, a strong absorption at 1464 cm^{-1} and a weaker absorption at 1340 cm^{-1} , are observed for the adsorbed anion radical. The former is almost certainly the carbonyl stretch of the adsorbed species (shifted from 1555 cm^{-1}) and the latter is at the position expected for the combination band from the carbonyl stretch and the symmetrical stretch of the aromatic ring (16). It is to be concluded, therefore, that the anion radical on the electrode surface is adsorbed via the carbonyl group, as would be expected. Voltammetric measurements using a Pt electrode also support the conclusion that adsorbed anion radical is present.

The differential reflectance spectrum recorded during the reduction of tetracyanoethylene (TCNE) in acetonitrile at a platinum electrode is shown in Figure 1. The positive bands due to disappearance of the TCNE substrate at the more negative potential are not visible. This is because the sensitivity of the transmission scale has been decreased to accommodate the full amplitude of the bands at 2187 cm^{-1} and 2148 cm^{-1} . The band at 2148 cm^{-1} is almost 10^4 times larger than is predicted from a simple Beer's law calculation for the total amount of TCNE anion that has been found during the potential step. The weaker band at 2187 cm^{-1} corresponds closely to an assignment made by Devlin *et al* to the $\text{-C}\equiv\text{N}$ stretch which has been charge transfer enhanced by formation of a complex between the anion radical and a surface platinum atom. The stronger band increases markedly with an increase in the cell thin layer gap, and is therefore due to solution free $\text{-C}\equiv\text{N}$ fundamental stretch, enhanced most likely by the formation of an electron donor-acceptor complex between the anion radical and neutral TCNE. This point will be the subject of a forthcoming paper.

CONCLUSIONS

The technique described is a very powerful new method for the rapid recording of the spectra of radical ion and other intermediates. The method is capable of detecting spectra from short-lived species that could not otherwise be obtained. Modern FTIR techniques include time resolved modes which will enable kinetic and mechanistic information to be obtained for unstable intermediates. This will be the subject of a forthcoming report.

ACKNOWLEDGEMENT

Part of this work was supported by the Office of Naval Research.

TABLE I

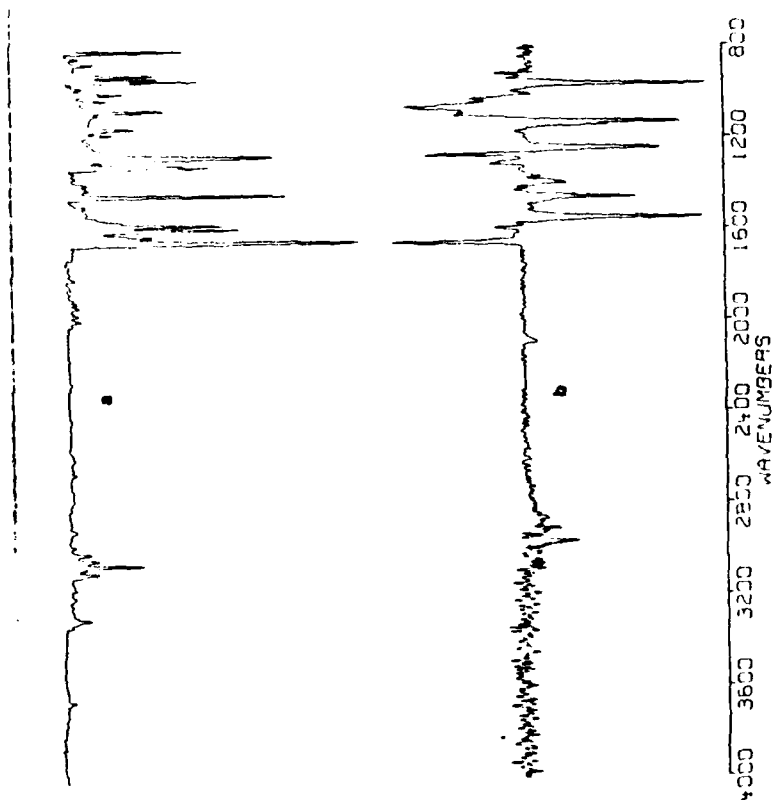
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Literature (15)	This Work	Literature (16)	This Work
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1010 (m)	1000 (m)	1019 (m)	1025 (w)
1317 (s)	1319 (m)	1143 (vs)	1147 (vs)
1277 (vs)	1277 (vs)	1150 (vs)	1155 (s)
917 (m)	922 (m)	1260 (vs)	1249 (vs)
		1288 (m)	1288 (w)
		1396 (s)	1394 (m)
		1560 (s)	1555 (vs)

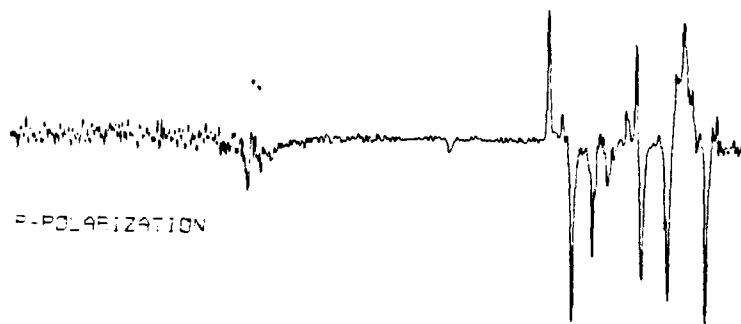
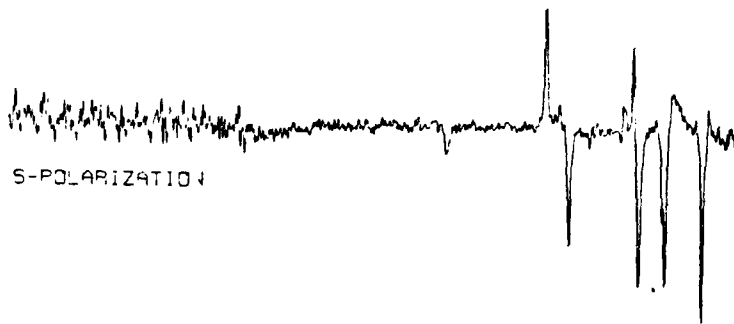
Comparison of absorption bands of benzophenone and benzophenone ketyl radical. (Relative band intensity nomenclature: vs = very strong, s = strong, m = medium, w = weak).

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FIGURE LEGENDS

1. a) Transmission spectrum of benzophenone. b) Differential FTIR spectrum of benzophenone between -1.75 V and -2.50 V.
2. The differential spectrum of benzophenone with s- and p-polarized light.
3. a) Transmission spectrum of anthracene. b) Differential FTIR spectrum of anthracene between -1.5 V and -2.5 V.
4. a) Transmission spectrum of TCNE. b) Differential FTIR spectrum of TCNE between +0.25 V and -0.25 V.





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