Honeycomb Spectroelectrochemical Cell User Guide

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1 Preface

1.1 Scope
This User Guide describes the basic functions of the Honeycomb Spectroelectrochemical Cell Kit, hereafter referred to as the “Honeycomb Cell,” including product specifications and limitations, product warranty, and other technical information.

This guide is written for the professional scientist or engineer (or student of science and engineering) and assumes a basic knowledge of scientific measurement and data presentation. Portions of this manual devoted to electrochemical and spectroscopic concepts assume some familiarity with these subjects.

1.2 Copyright
This publication may not be reproduced or transmitted in any form, electronic or mechanical, including photocopying, recording, storing in an information retrieval system, or translating, in whole or in part, without the prior written consent of Pine Research Instrumentation.

1.3 Trademarks
All trademarks are the property of their respective owners.

1.4 Limited Warranty

LIMITED WARRANTY

The Honeycomb Spectroelectrochemical Cell Kit (hereafter referred to as the “INSTRUMENT”) offered by Pine Research Instrumentation (hereafter referred to as “PINE”) is warranted to be free from defects in material at the time of shipment to CUSTOMER. No part of the INSTRUMENT is specifically warranted beyond the date received by the CUSTOMER.

This warranty being expressly in lieu of all other warranties, expressed or implied and all other liabilities.

All specifications are subject to change without notice. The CUSTOMER is responsible for charges associated with non-warranted repairs. This obligation includes but is not limited to travel expenses, labor, parts and freight charges.

1.5 Use Limitation
The Honeycomb Cell is not designed for use in experiments involving human subjects and/or the use of electrodes inside or on the surface of the human body.
1.6 Notes and Hints
Throughout this manual there are highlighted notes and information which are indicated with special icons as shown below (see: Table 1-1).

<table>
<thead>
<tr>
<th>Icon</th>
<th>Note:</th>
<th>Important or supplemental information.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Tip:</td>
<td>Useful hint or advice.</td>
</tr>
</tbody>
</table>

Table 1-1. Special Information Icons Used in this Document.

1.7 Safety Notices
Throughout this user guide there may be safety notices, which are indicated with special icons as shown below (see: Table 1-2). When working with the Honeycomb Cell and related accessories take heed and abide by all safety warnings. Failure to do so may result in damage to property, personal injury, or both.

<table>
<thead>
<tr>
<th>Icon</th>
<th>Caution:</th>
<th>Indicates information needed to prevent injury or death to a person or to prevent damage to equipment.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Stop:</td>
<td>For a procedure involving user action or activity, this icon indicates a point in the procedure where the user must stop the procedure.</td>
</tr>
</tbody>
</table>

Table 1-2. Special Safety Icons Used in this Document.

1.8 Technical Service Contact
For questions about proper operation of the Honeycomb Spectroelectrochemical Cell Kit or other technical issues, please contact Pine Research Instrumentation directly using the contact information below:

Pine Research Instrumentation
http://www.pineinst.com/echem
Phone: +1 (919) 782-8320
FAX: +1 (919) 782-8323
1.9 Factory Return Service Address

In the event that components of this kit must be returned to the factory for service, please contact Pine Research Instrumentation (see contact information above) to obtain a Return Material Authorization (RMA) form. Include a copy of this RMA form in any and all shipping cartons and ship the cartons to the factory address below:

Pine Instrument Company
Attn: RMA #: <RMA number>
104 Industrial Drive
Grove City PA 16127
USA
Phone: +1 (724) 458-6391

Return Material Authorization Required!

Do not ship equipment to the factory address above without first obtaining a Return Material Authorization (RMA) form from Pine Research Instrumentation. Call +1 (919) 782-8320 for RMA.
2 Product Description

The Honeycomb Cell is a tool used to perform spectroelectrochemical experiments. It consists of specially designed components to interrogate the intersection of spectroscopic and electrochemical properties of chemical species.

The unique Honeycomb Cell features a patterned "honeycomb" electrode card that mounts easily inside a thin layer quartz cuvette. A special cuvette cap securely holds the honeycomb electrode card and a separate reference electrode in the proper position within the cuvette. The working electrode is perforated with a honeycomb pattern of holes, which allow light to pass through the electrode. The active surface of the working electrode includes a metal coating along the inner walls of the holes. As the light beam from the spectrometer passes through the holes, the beam grazes the walls of each hole. Thus, the beam samples a relatively long pathlength. The figure below illustrates the way in which light passes through the Honeycomb electrode card (see: Figure 2-1).

![Figure 2-1. Light Passage through the Honeycomb Electrode.](image)

Electrical connections to the working and counter electrode (both located on the card) are easily made via a convenient mini-B USB connector. Connection to the separate miniature reference electrode is via a simple pin connection, which can accept either an alligator clip or a pin-and-socket connection.
The quartz cuvette is designed to fit any UV-vis spectrometer. The standard 1 cm x 1 cm cuvette fits nearly any sample holder. The Honeycomb Cell has been placed into an Ocean Optics cuvette holder, for illustration purposes.
2.1 Included Components

The Honeycomb Cell is sold as a kit. The components of the Honeycomb Cell Kit are shown and described below (see: Figure 2-3). Each component can be purchased individually to supplement or replace parts of the original kit. Detailed descriptions are provided in Section 2.2.

<table>
<thead>
<tr>
<th></th>
<th>Description</th>
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</thead>
<tbody>
<tr>
<td>A</td>
<td>Quartz Slotted Cell</td>
</tr>
<tr>
<td>B</td>
<td>Cell Cap</td>
</tr>
<tr>
<td>C</td>
<td>Honeycomb Electrodes</td>
</tr>
<tr>
<td>D</td>
<td>Miniature Ag/AgCl Reference Electrode</td>
</tr>
<tr>
<td>E</td>
<td>Plastic Fill Pipette</td>
</tr>
</tbody>
</table>

Figure 2-3. Components Included in Honeycomb Spectroelectrochemical Cell Kit.
2.2 Honeycomb Component Details

2.2.1 Quartz Cuvette Details

Use Caution: Quartz Cuvet is Fragile

The quartz cell is fragile and has thin walls. Use extreme caution when handling the cell.

The Honeycomb quartz cuvette is designed for use with the Honeycomb electrode. The quartz cuvette has a standard 1 cm x 1 cm footprint for use in most UV-vis spectrometers. The lower half of the cell features two notches, which create a solution reservoir and electrode chamber (see: Figure 2-4). Note that the reservoir is offset and not centered in the cuvette. This is intentional and matches with the location of the Honeycomb alignment groove in the cap.

Figure 2-4. Different Angles of the Quartz Cuvette.

If the Quartz Cell Breaks

The quartz cell is not covered by any warranty. The quartz cell can be purchased separately should it need to be replaced.
2.2.2 Honeycomb Electrode Specifications

The Honeycomb Cell Kit includes two gold electrode cards and one platinum card. The cards were designed to fit into a specially designed quartz cuvette, whose footprint is a standard 1 cm × 1 cm. Figure 2-3 shows the dimensions.

![Diagram of Honeycomb Electrode Card]

<table>
<thead>
<tr>
<th>A</th>
<th>14.7 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>17.0 mm</td>
</tr>
<tr>
<td>C</td>
<td>30.0 mm</td>
</tr>
<tr>
<td>D</td>
<td>12.0 mm</td>
</tr>
<tr>
<td>E</td>
<td>9.8 mm</td>
</tr>
<tr>
<td>F</td>
<td>USB connector area (see inset)</td>
</tr>
</tbody>
</table>

The thickness of the electrode card is 1.7 mm. The working electrode consists of channels, drilled in a 19 element array, through the ceramic substrate. The interior walls of the channels are coated with Au or Pt to form the working electrode. Each channel is 0.50 mm in diameter and their center to center distance is 0.75 mm. A counter trace
is placed on the surface of the ceramic, away from the channel area. In Figure 2-3 the areas shaded in light gray indicate the sections coated with an insulating clear ceramic layer.

2.3 Honeycomb Accessories

Accessories for the Honeycomb Cell include cables for making connection from the potentiostat to the honeycomb working electrode and to the externally supplied reference electrode. Gas purge kits for introducing inert gas into the spectroelectrochemical cell (removing dissolved oxygen) are also available (see: Figure 2-4).

2.3.1 Cell Cables

The cell cable for the Honeycomb Cell is sold separately to accommodate the appropriate potentiostat connection. Two USB-terminated cables are available: one to connect to a Pine WaveNow or WaveNano potentiostat and another to connect to any potentiostat (see: Figure 2-6).

A Generic Cable

This cable is needed to access individual electrode traces on a screen-printed honeycomb electrode. A mini-USB connector is on one end of the cable, and banana plugs terminate on the other end.

B WaveNow Cable

This cable connects a honeycomb electrode directly to a Pine portable potentiostat (WaveNow or WaveNano). A break-out (white) socket connector on the mini-USB end allows access to the externally supplied reference electrode.

Figure 2-6. The Cables to Connect the Honeycomb Electrode to Potentiostat.
2.3.2 Gas Purge Kits

There are purge kits available to assist with purging, sparging, and blanketing of solutions within the cell. For the Honeycomb Cell, two different kits should be used and include the three-way valve purge kit and micro-connection purge kit (see: Figure 2-7).

The three-way valve purge kit contains 3/8" OD / 1/4" ID PVC tubing and three-way PTFE valve to switch an inert purge gas to either bubbling under an electrolyte solution or blanketing over the solution.

The micro-connection purge kits contains 1/8" OD / 1/16" ID PVC tubing, 1/16" OD / 1/32" ID PTFE tubing, 1/32" OD / 1/64" ID PEEK tubing, and male/female luer lock connectors.

A  Three-Way Valve Purge Kit
   Connects purge gas (e.g. Nitrogen or Argon) to either cell connection or micro-connection purge kit.

B  Micro-Connection Purge Kit
   Kit connects three-way valve purge system to the Honeycomb cell. The Kit contains:

Figure 2-7. Purge Kits for use with the Honeycomb Cell.
The three-way valve and micro-connection purge kits are designed to be used together. The three-way valve purge kit links the purge gas source (e.g. Nitrogen or Argon) with the three-way valve. From the three way valve, the micro-connection purge kit connects to the Honeycomb cell for purging, sparging, and blanketing solutions within the Honeycomb quartz cuvette.

### 2.3.3 Reference Electrodes

The Honeycomb Cell Kit contains a miniature Ag/AgCl gel reference electrode (see: Figure 2-8). The Ag/AgCl electrode is ideal for aqueous sampling or systems where chloride ion contamination is not of concern. The reference electrode has a ceramic fritted tip, a 3.5 mm diameter glass body, and an overall length of 60 mm. The gel inside is a saturated KCl solution.

Alternatively, a miniature silver wire pseudo reference electrode is also separately available to fit the cell. The miniature reference electrodes can be purchased separately from Pine (see: Figure 2-8).

![Figure 2-8. Miniature External Reference Electrodes for the Honeycomb Cell.](image)

**Use Proper Orientation**

Always ensure the Honeycomb electrode is inserted properly into the quartz cuvette. The externally supplied reference electrode should always be inserted behind the Honeycomb electrode, the side on which there is no printing (with gold or platinum).
3 Background and Theory

3.1 Introduction to Absorption Spectroscopy

The irradiance of light ($I$) is defined as the energy per second per unit area of the light beam ($W/m^2$). When a light beam (of initial irradiance, $I_0$) passes through a colored solution sample of length $b$, the molecules in the solution can absorb some or all of the light. Therefore, the irradiance of the exiting light beam ($I$) depends on the extent of light-molecule interactions and $I \leq I_0$. The ratio of initial light beam irradiance to exiting light beam irradiance is called Transmittance, $T$ (see: Equation 1).

$$ T = \frac{I}{I_0} $$ (1)

$T$ ranges from zero (the light passing through solution is 100% absorbed) to one (0% of the light beam is absorbed). Another way to consider the extent to which molecules in solution absorb light energy is by considering the Absorbance ($A$), the logarithm of Transmittance (see Equation 2).

$$ A = \log\left(\frac{I_0}{I}\right) = -\log T $$ (2)

Some refer to Absorbance as optical density. The extent of Absorbance is described by the Beer-Lambert relationship (see: Equation 3).

$$ A = \varepsilon b C $$ (3)

where $\varepsilon$ is the molar absorption (extinction) coefficient ($L/mol \cdot cm$) of a light-absorbing molecule, $b$ is the optical path length ($cm$); and $C$ is the concentration ($mol/L$).

By the Beer-Lambert relationship (see: Equation 3), absorbance ($A$) is determined by cell geometry ($b$) and the chemical properties of solution ($\varepsilon C$). Concentration ($C$) is linearly proportional to absorbance ($A$).

Maximization of the optical signal ($A$) is often an important aspect of spectroscopic experiments. The standard path length for routine measurements is 10 mm. There are few ways in which to maximize absorption, one of which is to extend the measured path length ($b$) (see Equation 3). The schematic drawing shows the basic principle of absorption spectroscopy (see Figure 3-1).
3.2 Introduction to Electrochemistry

Electrochemistry focuses on chemical reactions that involve electrical potential and current. Some chemical reactions can proceed spontaneously can generate electrical current, which can be used to do useful work; while other chemical reaction can be forced to proceed by using electrical current. Most frequently, electrochemical reactions are oxidation and reduction reactions (redox reactions). Current or voltage, which arise from electrons that flow through an electric circuit during a redox reaction, are characteristic measurements for a specific chemical system. Electrical current is proportional to the rate of reaction and the cell voltage is proportional to the free energy change for the electrochemical reaction.

One of the most basic relationships is that of electric charge, \( q \), measured in coulombs (C). The magnitude of charge of a single electron is \( 1.602 \times 10^{-19} \) and a mole of electrons must therefore have charge equal to \( (1.602 \times 10^{-19} \text{ C})(6.022 \times 10^{23} \text{ mol}^{-1}) = 9.649 \times 10^4 \text{ C/mol} \). This value is known as Faraday’s constant, \( (F) \). By the relationship shown, charge is related to the number of electrons transferred, where \( n \) = number of electrons transferred (see: Equation 4).

\[
q = nF
\]  

(4)

Energy, measured in Joules (J), drives all chemical reactions. In a redox reaction, the movement of charged particles releases energy. Charged particle movement gives

![Figure 3-1. Absorption Spectroscopy in a Cuvette.](image)
rise to an electrical potential difference \((E)\), measured in volts \((V)\). When a charged particle moves through a potential difference, thermodynamic work \((W)\) is done (see: Equation 5). Since potential difference is not a point source, it is sometimes denoted as a difference, \(\Delta E\).

\[
W = qE
\]

(5)

Gibb’s free energy \((\Delta G)\) is the negative value of the maximum electrical work possible (see: Equation 6).

\[
W = -\Delta G
\]

(6)

Therefore, the relationship between Gibb’s free energy and electrical potential difference is established (see: Equation 7).

\[
\Delta G = -nFE
\]

(7)

At standard conditions \((T = 273.15 \text{ K} \text{ and } P = 100 \text{ kPa})\), free energy and electrical potential are denoted as \(\Delta G^*\) and \(E^*\) and are often tabulated values useful for finding free energy and potential differences at non-standard conditions.

For the following general chemical reaction (balanced, overall)

\[
aA + bB \rightleftharpoons cC + dD
\]

the reaction quotient \((Q)\) is the ratio of the activity \((a)\) of each component, raised to its stoichiometric coefficient. At low concentrations, activity closely follows concentration (see: Equation 8).

\[
Q = \frac{a_c^a \cdot a_D^d}{a_A^a \cdot a_B^b} = \frac{[C]^c \cdot [D]^d}{[A]^a \cdot [B]^b}
\]

(8)

Free energy relates to the reaction quotient,

\[
\Delta G = \Delta G^* + RT \ln Q
\]

(9)

where \(R\) is the universal gas constant \((8.314 \text{ J/mol} \cdot \text{K})\) and \(T\) is temperature \((K)\) (see: Equation 9). The relationships for free energy, electrical potential difference, and reaction quotient combine to give the Nernst equation (see: Equation 10), a fundamental relationship used in electrochemistry. If a system follows the Nernst equation then the reaction is often said to be thermodynamically (or electrochemically) reversible (i.e. Nernstian)

\[
E = E^* + \frac{RT}{nF} \ln \frac{[C]^c \cdot [D]^d}{[A]^a \cdot [B]^b}
\]

(10)

There are always two reactions occurring in the electrochemical cell; a reaction occurs at the negative terminal (cathode) and at the positive terminal (anode). The Nernst equation is most commonly used to determine the half cell potential, or the standard
reduction potential of one of the electrochemical reactions in the electrochemical cell. The form of the Nernst equation given (see: Equation 10) is often simplified to only consider the oxidized \([O]\) and reduced \([R]\) species of a half reaction, raised to their stoichiometric coefficients (see Equation 11).

\[
E = E^\circ + \frac{RT}{nF} \ln \frac{[O]}{[R]}
\]  

(11)

The \([O]/[R]\) ratio from the Nernst equation can be interrogated during a spectroelectrochemical experiment. Due to the confined reaction space within the Honeycomb holes, the electrochemical reaction approaches exhaustive electrolysis within the holes. The time to reach exhaustive electrolysis depends highly on the kinetics of mass transport and electron transfer for the chemical species of interest.

In electrochemistry, it is common to write half cell reactions as reductions (see: Equation 12),

\[
O + ne \rightarrow R
\]  

(12)

where \(n\) is the number of electrons transferred in the half reaction, \(e\) represents an electron, and \(O\) and \(R\) are the oxidized and reduced forms of the molecule, respectively. Standard reduction potentials \((E^\circ)\) for electron transfer reactions are useful in determining reduction potentials as non-standard or non-equilibrium conditions \((E)\). \(E^\circ\) are thermodynamically calculated and do not consider kinetic limitations in the heterogeneous or homogenous electron transfer rates.

Electrolysis is a general term for the process by which a chemical reaction is forced to occur at an electrode due to an applied voltage. For a general reduction reaction (see: Equation 12), applying a voltage which is \(\geq E^\circ\) (called an overpotential) will drive the redox reaction at a faster rate; therefore, causing more charge to flow per unit time. The quantity of charge that flows per unit time through a circuit is called current (amperes, \(A\)) and 1 A of current is the charge of 1 C per second \((1C/s = 1A)\). The general equation for relating current \((i)\) to amount of chemical electrolyzed \((N)\) depends on time \((t)\) and number of electrons transferred in the half reaction \((n)\) (see: Equation 13).

\[
N = \frac{it}{nF}
\]  

(13)

The term exhaustive electrolysis indicates that all moles of redox active species \((O\) in Equation 12) within the diffusional limit of the working electrode have been reduced \((R\) in Equation 12).

### 3.3 Introduction to Spectroelectrochemistry

Spectroelectrochemistry includes a range of experimental techniques that provide integrated information from both spectroscopy and electrochemistry. Among these techniques, integration of UV-vis absorption spectroscopy and electrochemistry represents a classical example that was studied during the pioneering days of this field.
In fact, the term “spectroelectrochemistry” often specifically refers to methods that integrate UV-vis absorption and electrochemistry. The Honeycomb Cell was designed for use with UV-vis absorption and electrochemistry.

In a typical spectroelectrochemical setup, the working electrode must permit transmission of a collimated beam of light. Transparent and conductive electrodes, such as indium tin oxide (ITO) electrodes, satisfy this requirement; however, ITO does not permit passage of ultraviolet light. By comparison, mini-grid electrodes, electrodes composed of thin strands of woven noble metals like platinum and gold, allow partial transmission of UV light and are commonly used in spectroelectrochemical experiments (see Figure 3-2).

A mini-grid electrode is made of thin metal wires of diameter \( w \), woven into a mesh of a pitch distance \( p \). The mini-grid is inserted into a thin-layer cell of width \( b \), which is also the optical path length in an absorption measurement.

![Figure 3-2. Spectroelectrochemical Analysis with a Mini-Grid Electrode.](image)

Electron transfer reactions (see: Equation 12) at the working electrode surface can lead to a change in the optical absorption measured if the oxidized and reduced species involved have significantly different molar extinction coefficients. According to the Beer-Lambert relationship (see: Equation 3), the magnitude of absorbance change is directly proportional to the optical path length \( b \).

Exhaustive electrolysis is often desired in spectroelectrochemical experiments. It is desirable to shorten the total electrolysis time for two reasons: 1) molecules can become unstable (transient) upon oxidation or reduction and require rapid detection
before they are lost to irreversible chemical reactions; and 2) short equilibrium time reduces total experiment time, useful when many spectra must be obtained at equilibrium conditions. For example, the formal potential of a redox protein can be mapped by measuring the ratio of redox species concentrations as a function of applied electrode potential. The ratio of redox species concentration is obtained from absorption spectrum at each potential when the redox pair reaches Nernstian equilibrium at the electrode surface. Such a measurement depends upon exhaustive electrolysis, which depending on the nature of the electron transfer kinetics, may take a long time to achieve.

The equilibrium time of a thin-layer spectroelectrochemical cell can be estimated if diffusion toward the working electrode is the rate limiting step (see: Equation 14),

$$\tau = \frac{\delta^2}{D}$$  \hspace{1cm} (14)

where $D$ (cm$^2$/s) is the diffusion coefficient of a redox active molecule and $\delta$ (cm) is the largest distance molecules have to travel to reach the nearest electrode surface. By assuming typical grid dimensions ($w = 10\mu m; p = 100\mu m$), standard cell dimensions ($b = 100\mu m; H = 15\mu m$), and assuming a typical value for the diffusion coefficient ($D = 5 \times 10^{-6}$ cm$^2$/s), the equilibrium time can be estimated ($\tau = 20s$). If cell path increases ten-fold in length to approximately 1 mm, the diffusional time increases 100 fold to 2000 s. Therefore, there exists a clear tradeoff between measured signal strength (absorbance) and the time required to obtain spectroelectrochemical measurements.

The Honeycomb Spectroelectrochemical electrodes by Pine (see: Figure 3-3) feature geometries that achieve longer path lengths than possible with classic mini grid configurations. Primarily, the increased absorbance results from the substrate thickness (1.7 mm) and through pore coating with active electrode material, which defines path length. Compared to traditional mesh type electrodes, path length is about an order of magnitude higher for Honeycomb electrodes. Radial diffusion within the small pores ($125\mu m$) of the Honeycomb electrode pattern decreases the diffusion time, thereby offering slightly higher absorbance than traditional mesh electrodes.

### 3.4 Common Spectroelectrochemical Experiments

#### 3.4.1 Measurement of Transient Chemical Species or Equilibrium Spectra

A common application of UV-vis spectroelectrochemistry is to obtain absorption spectra of transient species, which form because of electrochemical reactions. In such an experiment, the electrode potential is initially biased at a potential where electrochemical reactions do not occur. Then, potential is stepped to a value where a transient species generates at the electrode surface due to electron transfer reactions. Optical absorption measurement starts immediately after the potential step is applied. The result is a mapping of the absorption spectrum signature as a function of applied potential.
With certain electroactive species, signal strength (absorbance) initially increases as potential increases as a function of time. After sufficient electrolysis time, absorption reaches a limiting value after the electroactive species in solution have been exhaustively electrolyzed.

If the transient species generated electrochemically is not stable (e.g. spontaneous decomposition, homogenous chemical reaction, etc.), then the temporal resolution of the optical absorption measurement must be improved to measure the signal that arises from the short lived species. In this case, longer optical pathlength becomes particularly desirable because it can compensate signal loss resulting from short measurement time.

3.4.2 Determination Electroactive Species Reduction Potential

By UV-vis analysis of electrochemically active chemicals subjected to changes in applied potential, certain absorbance peaks will grow and/or decay as a function of applied potential. Specific wavelengths at which the absorbance change due to applied potential are most notable can be followed to determine the reduction potential of the chemical.

If an electrochemical reaction generates stable, measureable products, and if the products quickly reach equilibrium concentrations (by exhaustive electrolysis), then equilibrium absorbance spectra obtained at a series of electrochemical potentials can
be measured. In this case, the absorbance value is expected to follow the Nernst equation (see: Equation 11).

Consider the general electrochemical reaction (see: Equation 12). If the applied potential is \( \leq E^0 \), then \( O \) is the dominant species within the diffusional limit of the electrode. As potential is stepped \( \geq E^0 \), \( O \) converts to \( R \) at an increasing rate as the potential step moves farther from \( E^+ \). The UV-vis spectral changes observed during the reduction and oxidation are a true response to applied electrode potential. Then changes in absorbance represent the changes in concentration of \( O \) and \( R \).

At a fixed wavelength, the relationship between \([0]/[R]\) and absorbance is a direct proportion (see: Equation 15).

\[
\frac{[0]}{[R]} = \frac{A_E - A_R}{A_O - A_E}
\]

(15)

where \( A_E \) is the absorbance of the solution at equilibrium for any applied potential, \( A_O \) is the absorbance of the fully oxidized species, and \( A_R \) is the absorbance of the fully reduced species. When sufficient absorbance spectra have been obtained at several values of applied potential (typically, at least ten data points), a plot of applied potential vs. In\([0]/[R]\) (as determined from Equation 15) reveals a linear relationship. From a linear regression of the data, one can find \( n \), number of electrons transferred in the reaction, from the slope, which is equal to \( RT/nF \), and \( E^+ \), standard reduction potential, from the intercept of the regression curve.

The experiment described here is a well established technique to study redox proteins, which often have intense absorption bands in the UV region. The main requirements for successful data acquisition with this type of experiment are as follows:

1. The electron transfer reactions are reasonable fast so that equilibrium conditions is reached within minutes.
2. The electrolysis time is comparable to the time to reach equilibrium.
3. The chemical species is stable, electrochemically reversible, and can be resolved spectroscopically.

### 3.4.3 Measurement of Electrochemical Kinetics

Electrochemical kinetics involves several physical processes that occur simultaneously. These processes include electron transfer at the electrode|electrolyte (heterogeneous reaction), chemical reactions proceeding or following an electron transfer step (homogenous reaction), surface effects (adsorption), and mass transport (such as diffusional transport) to the electrode surface.

Spectroelectrochemistry assists in the elucidation of kinetic rates, relative to the processes described previously. In general, a well established absorption peak, corresponding to a particular redox species, is measured as a function of time. When designing such an experiment, one has to trade off signal strength (absorbance) and with time resolution.
If a sufficiently negative (reducing) potential is applied to the electrode, then the electron transfer step is very fast. Conversely, a sufficiently positive (oxidizing) potential drives the electron transfer at a faster rate as well. In any system, exhaustive electrolysis will be achieved faster than bulk concentration equilibrium.

As absorbance is monitored at a specific wavelength, the absorbance vs. time plot reveals important pieces of information about how fast exhaustive electrolysis can be accomplished. Without electrical potential perturbation, the absorbance (at a fixed wavelength) is constant and the concentrations of $O$ and $R$ do not change. As potential is stepped (either positively or negatively), charge flows to balance the applied potential, resulting in some $O$ being electrolyzed to $R$ (see: Equation 11). The time to achieve exhaustive electrolysis of $O$ that is within the mass transport limit (diffusional limit) of the system is related to the gradual decrease or increase in absorbance. The diffusional time and exhaustive electrolysis time can be obtained from such a plot.
## 4 Instructions for Use

### Separate Reference (Background) and Sample Spectra may be Required

The instructions given in Section 4 are applicable to both obtaining a reference spectrum and a sample spectrum. Some users experimental conditions may require cleaning the cell between reference and sample spectra.

### 4.1 Cell Assembly

**Use Caution: Wear Gloves**

Do not touch optical surfaces.

---

**Prepare the cell cap and cuvette.**

The slot in the cap for the Honeycomb electrode should align with the slot in the quartz cuvette.

The photo shows alignment arrows and dashed lines to clearly show the orientation of the cap and cuvette.
Cap will fit loosely on the quartz cuvet.

The 3.5 mm OD hole in the cap (reference electrode access) should be positioned above the bulk well in the quartz cuvet.

4.2 Prepare Honeycomb Cell for a Measurement

Turn on the light source of your spectrometer to stabilize the light beam intensity.

Typically ~10 minutes is necessary.

Warm the Spectrometer Lamp(s)

Spectroelectrochemistry often involves measuring small changes in absorbance, so a very stable light source is essential.
Through the large hole on the cell cap, fill the quartz cell with test solution.

The fill volume should be 1.1 mL to 1.3 mL. The plastic pipettes have markings at the 1 mL interval.

Avoid Bubbles in Solution
Carefully fill the thin gap in the quartz cuvet to avoid introducing bubbles.

Pre-fill the channels of a clean Honeycomb electrode with the same electrolyte to be measured.

Check to be sure that no air pockets are present inside any of the channels.
Carefully Align Electrode in Quartz Cuvet and Cap

Ensure that the cell is assembled as designed. The Au or Pt pattern should face the shorter quartz notch, which is the same side the light beam should enter the cell.

Insert the Honeycomb electrode into the cell cap assembly.

The Au or Pt pattern should also face the incoming light for absorption measurements.

The face with printed Au or Pt pattern should point away from the fill hole in the cap (also, where the gel reference will be inserted).

The face with printed Au or Pt pattern should point away from the fill hole in the cap (also, where the gel reference will be inserted).
Insert the reference electrode (miniature Ag/AgCl gel or miniature pseudo Ag wire) and use the o-ring to adjust the depth of immersion.

**Install with Correct Orientation**

Ensure the reference electrode is installed behind the Honeycomb electrode. The reference electrode should be next to the bare Honeycomb electrode substrate surface, not the patterned side.

Insert the Honeycomb cell (fitted with cap, Honeycomb electrode, and reference electrode) into the cuvet holder of the UV-vis Spectrometer.

Determine the light beam path direction in your spectrometer. The diagram given below shows the directionality of the Honeycomb electrode and cuvet relative to the light beam source.

In the diagram below, the cuvet holder is a fiber optic cuvet holder. Your UV-vis spectrometer may not have fiber optic connection.
4.3 Connecting the Honeycomb Cell to a Potentiostat

**Use with Any Potentiostat**

There are two cables available for the Honeycomb cell so it can be used with any potentiostat, including the Pine WaveNow series.

To connect Honeycomb cell to a Pine WaveNow or WaveNano, use the reference breakout cable.

Connect the HD-15 potentiostat connector to the WaveNow or WaveNano and mini-USB connector to the top of the Honeycomb electrode.

A separate reference electrode breakout (white wire with barrel connector) should connect to the pin of the externally supplied reference electrode.
To connect Honeycomb cell to other potentiostats, use the USB-to-banana cable (generic cell cable).

Connect the working sense, working drive, and counter electrode banana jacks to appropriate potentiostat cell cable leads.

Connect the potentiostat reference electrode lead directly to the externally supplied reference electrode.

Separate Reference Electrode Connection

Note that the reference alligator clip from the potentiostat should be connected directly to the pin on top of the externally supplied reference electrode (gel Ag/AgCl or pseudo Ag). All other potentiostat leads (working drive, working sense, and counter) should be connected to the corresponding banana plugs on the generic cable.
The Pine Research Instrumentation cell connection color code is given below, in Table 4-4. When using the generic cable to connect the Honeycomb electrode to the potentiostat, connect potentiostat leads to the appropriate Pine color.

<table>
<thead>
<tr>
<th>Color</th>
<th>Description</th>
<th>Abbr.</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>GREEN</td>
<td>Counter Electrode</td>
<td>CE</td>
<td>Drive</td>
</tr>
<tr>
<td>WHITE</td>
<td>Reference Electrode</td>
<td>RE</td>
<td>Sense</td>
</tr>
<tr>
<td>RED</td>
<td>Primary Working Electrode</td>
<td>K1</td>
<td>Drive</td>
</tr>
<tr>
<td>ORANGE</td>
<td>Primary Working Electrode</td>
<td>K1</td>
<td>Sense</td>
</tr>
<tr>
<td>BLACK</td>
<td>Analog Ground/ DC Common</td>
<td></td>
<td>Ground</td>
</tr>
</tbody>
</table>

Table 4-3. Pine Research Instrumentation Electrode Lead Color Code.

Connect all components of the spectroelectrochemical experiment setup.

Below is an example setup showing a spectrometer connected to the Honeycomb cell by fiber optic cable, a potentiostat connected to the Honeycomb cell, and a computer to control and acquire data from both instruments.

**Figure 4-5. Spectroelectrochemical Component Setup.**
4.4 Obtain Reference (Background) Spectrum

To record an absorbance spectrum, first collect a background spectrum with an appropriate reference solution. The background spectrum will be subtracted from the sample spectrum to compensate for any reflection, scattering, and absorption by the cuvet, solvent, or electrolyte. Therefore, the true absorbance of the sample is collected. It is ideal to select optically transparent solvents and background electrolytes for spectroscopic experiments to minimize false changes in background absorbance.

**Important Information about the Reference Solution**

The nature of the user’s chemistry will dictate the composition of the reference solution. Some common reference solutions are:

- Solvent
- Solvent + background electrolyte
- Solvent + background electrolyte + sample (if one of the electrochemical forms is optically transparent at a select electrochemical potential)

With all components in the system connected, obtain a reference spectrum.

The Honeycomb electrode should be inserted into the cuvette during this measurement.

Save the reference spectrum.

Every spectrometer has its own unique software. The reference spectrum may have to be subtracted manually.

4.5 Obtain a Sample Spectrum

As discussed previously in Section 3.4, there are some common spectroelectrochemical experiments. Use of the Honeycomb cell requires method development by the user and the steps will be specific to the chemical system under interrogation. This user guide does not explain the steps of acquiring an absorption spectrum for each type of spectroelectrochemical experiment. In general, the spectrometer and potentiostat operate independently. There are several modes of operation possible. Here are three possible options:

- Obtain a sample spectrum without the potentiostat running. This type of spectrum is like a traditional UV-vis absorption measurement.
- Obtain a sample spectrum with the potentiostat at open circuit potential (OCP). This type of spectrum is like a traditional UV-vis absorption measurement. OCP is a reasonable potential to apply for the first experiment.
- Use the potentiostat to perform electrolysis (typically a potential step experiment) for some amount of time. After the electrolysis period, use the spectrometer software to collect a spectrum as desired (wavelength range or fixed wavelength).
5 Example Experiments

This section provides experimental data to highlight common uses of spectroelectrochemistry. Refer to Section 3.4 for a theoretical discussion of the experiments that follow. The Honeycomb cell was used in the following experiments.

5.1 Study of Methyl Viologen Radical Generated Electrochemically in situ

Methyl viologen (MV$^{2+}$) is a frequently employed electrochemical redox species. Due to its highly reversible one electron transfer, MV$^{2+}$ is often used in conjunction with biological systems as a rapid electron mediator between biological species in solution and the electrode surface. The highly conjugated nature of MV$^{2+}$ (see: Figure 5-1) lends itself to UV-visible spectroscopic interrogation as well as electrochemical studies.

![Figure 5-1. Chemical Structure of Methyl Viologen Dication.](image)

The first electron transfer reaction of MV$^{2+}$ reduces the dication to a cation radical intermediate (MV$^{2+}\*$, see: Equation 16). This first step is electrochemically reversible. A solution of MV$^{2+}$ is clear and colorless while its reduction product, MV$^{2+}\*$, is a violet blue color.

\[
MV^{2+} + e^{-} \rightarrow MV^{+}\star \quad E^\circ = -645 \text{ mV vs. Ag/AgCl} \quad (16)
\]

As electrode potential is held at an even higher reduction potential, a second electron transfer reaction converts the violet blue MV$^{2+}\*$ to the neutral charged and light yellow colored methyl viologen (MV, see: Equation 17). This second oxidation is irreversible, due to the insolubility and decomposition of MV. MV readily forms an adsorbed solid layer on working electrodes.

\[
MV^{+}\star + e^{-} \rightarrow MV_{(s)} \quad E^\circ = -1060 \text{ mV vs. Ag/AgCl} \quad (17)
\]

A solution of 0.1 mM MV$^{2+}$ in 100 mM KNO$_3$ served as the analytical solution for this experiment. The absorption spectrum of MV$^{2+}$ (see: Figure 5-2, green trace) was acquired at a potential (0.0 V vs. Ag/AgCl) where there was no faradaic (reduction) current. Notice the predominant absorbance peak for MV$^{2+}$ at 258 nm.

Upon a potential step to -1000 mV vs. Ag/AgCl, the absorption spectrum of MV$^{2+}\*$ (see: Figure 5-2, blue trace) shows a substantial change as compared to the MV$^{2+}$ spectrum. The potential step to -1000 mV vs. Ag/AgCl was sufficiently reductive to convert all the MV$^{2+}$ within the diffusional limit (inside the holes of the Honeycomb electrode) to MV$^{2+}\*$. Spectroscopically, this is evident by absence of the absorbance peak at 258 nm and appearance of peaks at 394 nm and 604 nm, not observed in the MV$^{2+}$ spectrum.
The experimental system was assembled as previously described (see: Figure 4-5). Data were acquired with 0.7 s integration time and 4 scans per spectrum using a UV-vis spectrometer system (Ocean Optics USB4000), CUV sample holder, fiber optics of 400 mm core diameter, and DT-mini-2-GS light source (3.8 watts deuterium, 1.2 watts tungsten halogen).

![Absorption Spectra of Methyl Viologen and its Cation Radical.](image)

**Figure 5-2. Absorption Spectra of Methyl Viologen and its Cation Radical.**

### 5.2 Study of Ferricyanide/Ferrocyanide Equilibrium

Potassium Ferricyanide ($K_2Fe(CN)_6$) is a commonly available, inexpensive, and well-behaved electrochemical redox chemical (see: Figure 5-3).

![Structure of Potassium Ferricyanide.](image)

**Figure 5-3. Structure of Potassium Ferricyanide.**

The oxidized form is called ferricyanide, [$Fe(CN)_6^{3-}$] and the reduced form is called ferrocyanide, [$Fe(CN)_6^{4-}$]. The ferric/ferrocyanide system is generally reversible and is soluble in water. Ferricyanide undergoes a one electron reduction to ferrocyanide,
which corresponds to a visual color change as well, from yellow to optically transparent (see: Equation 18).

\[ [\text{Fe(CN)}_6]^{3-} + e^{-} \rightleftharpoons [\text{Fe(CN)}_6]^{4-} \quad E^\circ = 260 \text{ mV vs. Ag/AgCl} \]  (18)

A solution of 0.1 mM \([\text{Fe(CN)}_6]^{3-}\) in 100 mM \(\text{KNO}_3\) served as the analytical solution for this experiment. The absorption spectrum of \([\text{Fe(CN)}_6]^{4-}\) (see: Figure 5-4, green trace) was acquired at a potential (-700 mV vs. Ag/AgCl). At -700 mV, all \([\text{Fe(CN)}_6]^{3-}\) within the diffusional limit of the electrode was electrolyzed to the clear and colorless form, \([\text{Fe(CN)}_6]^{4-}\). At this potential, no significant absorbance peaks are observed.

Upon a potential step to 800 mV vs. Ag/AgCl, the absorption spectrum of \([\text{Fe(CN)}_6]^{3-}\) (see: Figure 5-4, blue trace) shows a substantial change as compared to the \([\text{Fe(CN)}_6]^{4-}\) spectrum. The potential step to -1000 mV vs. Ag/AgCl was sufficiently oxidative to convert any the \([\text{Fe(CN)}_6]^{4-}\) to the pale yellow form, \([\text{Fe(CN)}_6]^{3-}\). Spectroscopically, this is evident by the appearance of an absorbance peak at 418 nm.

The experimental system was assembled as previously described (see: Figure 4-5). Data were acquired with 0.7 s integration time and 4 scans per spectrum using a UV-vis spectrometer system (Ocean Optics USB4000), CUV sample holder, fiber optics of 400 mm core diameter, and DT-mini-2-GS light source (3.8 watts deuterium, 1.2 watts tungsten halogen).

![Absorption Spectra of Ferricyanide and Ferrocyanide](image)

Figure 5-4. Absorption Spectra of Ferricyanide and Ferrocyanide.
5.3 Measurement of Ferricyanide/Ferrocyanide Electrochemical Kinetics

As discussed (see: Sections 3.4 and 5.2), the kinetics of ferricyanide/ferrocyanide can be interrogated with spectroelectrochemical experiments. From typical absorbance spectrum at reductive and oxidative overpotentials (see: Figure 5-4) the spectral signature of of $[Fe(CN)_6]^{3-}$ shows two significant peaks at 302 nm and 418 nm.

A

**Plot of Applied Potential**

Square potential wave applied to the electrochemical cell. The dominant species of the redox pair is stated on the curve.

B

**Absorbance vs. Time at a fixed Wavelength**

Fixed wavelength measure of absorbance vs. time, which can be decoupled for kinetic data.

Figure 5-5. Absorbance Transients of Ferricyanide/Ferrocyanide Redox Species.
To estimate the electron transfer kinetics of the ferricyanide reaction (see: Equation 18), one must monitor absorbance vs. time at a fixed wavelength, during a potential step experiment (see: Figure 5-5).

Because the electron transfer reaction is fast at a high overpotential, the decay in absorbance is related to the electron transfer rate (see: Figure 5-5). In this experiment, potential was held constant for 100 s (where absorbance was measured at both wavelengths, 302 nm and 418 nm). Then, a potential step (see Figure 5-5, plot A) stepped to 800 mV vs. Ag/AgCl. The potential step induced reduction of $[Fe(CN)_6]^{3-}$ to $[Fe(CN)_6]^{4-}$ and a simultaneous decay in absorbance at both 302 nm and 418 nm (see Figure 5-5, plot A).

The decay in absorbance begins to approach a zero slope after about 60 seconds. Thus, the time to reach exhaustive electrolysis is about 60 seconds. Therefore, the diffusional time can be estimated as 30 seconds.

### 5.4 Determination of Ferricyanide Standard Reduction Potential

From Section 3.4, the standard reduction potential for chemical species can be determined with spectroelectrochemistry. As discussed in Section 5.2, the $E^\circ$ for ferricyanide (260 mV vs. Ag/AgCl) is the potential about which faradaic current will arise (oxidative current when $E > E^\circ$ and reductive current when $E < E^\circ$). From the Nernst Equation (see: Equation 11) the ratio of ferricyanide concentration ($[Fe(CN)_6]^{3-}$, $[O]$) to ferrocyanide concentration ($[Fe(CN)_6]^{4-}$, $[R]$) is a function of applied potential.

In a spectroelectrochemical setup, the ratio of ferrocyanide concentration ($[O]$) to ferrocyanide concentration ($[R]$) relates to a change in optical absorbance, as a function of applied potential (see: Equation 15).

Here, applied potential was first stepped to 400 mV vs. Ag/AgCl. 400 mV is significantly oxidative of $E^\circ$; therefore, it is reasonable to have achieved exhaustive electrolysis and all ferri/ferrocyanide in the honeycomb electrode should be in the $[Fe(CN)_6]^{3-}$ form. The Honeycomb cell was held at 400 mV vs. Ag/AgCl for approximately 1 minute, after which time a UV-vis absorbance spectrum was obtained. From previous experiments (see: Sections 5.2 - 5.3) the peak at 418 nm corresponds to $[Fe(CN)_6]^{3-}$. The rate of ferrocyanide oxidation is higher at greater overpotential, where absorbance is high and the time to reach exhaustive electrolysis is small. As overpotential decreases (applied potential increases in a negative direction toward $E^\circ$), absorbance at 418 nm also decreases (see: Figure 5-6). These data confirm the relationship between the Nernstian ratio, $\ln([O]/[R])$, and optical absorbance (see: Equation 15 and 11).
Figure 5-6. Absorption Spectra of Ferricyanide at Different Applied Potential.

The absorbance value at 418 nm for each spectrum as a function of applied potential is tabulated below (see: Table 5-4). A plot of applied potential vs. ln([O]/[R]) shows a linear agreement (see: Figure 5-7), as predicted by the Nernst Equation (see: Equation 11). The y-intercept is 262.85, which agrees well with the reported standard reduction potential for ferricyanide in 1 M KNO₃ (263 mV vs. Ag/AgCl).

<table>
<thead>
<tr>
<th>Absorbance</th>
<th>Potential (mV)</th>
<th>[O]/[R]</th>
<th>ln [O]/[R]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.772</td>
<td>400</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>0.736</td>
<td>350</td>
<td>20.3</td>
<td>3.01</td>
</tr>
<tr>
<td>0.584</td>
<td>300</td>
<td>3.85</td>
<td>1.35</td>
</tr>
<tr>
<td>0.431</td>
<td>275</td>
<td>1.41</td>
<td>0.34</td>
</tr>
<tr>
<td>0.259</td>
<td>250</td>
<td>0.544</td>
<td>-0.61</td>
</tr>
<tr>
<td>0.151</td>
<td>225</td>
<td>0.257</td>
<td>-1.36</td>
</tr>
<tr>
<td>0.091</td>
<td>200</td>
<td>0.142</td>
<td>-1.95</td>
</tr>
</tbody>
</table>

Table 5-4. Ferricyanide/Ferrocyanide Absorbance as a Function of Applied Potential.
Figure 5-7. Linear Relationship between Applied Potential and Concentration.

6 Care and Storage

Do not Traditionally Polish the Honeycomb Electrode

The ceramic Honeycomb electrodes cannot be cleaned with traditional electrode polishing methods that include aluminum oxide slurries. The electrode will be damaged with alumina slurries.

The electrochemical cleaning method can remove trace organic impurities adsorbed on the surface and is effective at removing a monolayer of impurity. Some applications, such as protein electrochemistry, may require post-cleaning steps such as chemical modification of the electrode surface to increase the heterogeneous electron transfer rate.

Users have Reported More Aggressive Methods

Some users have reported to us that a more aggressive cleaning protocol may be necessary to refresh the Honeycomb electrodes. The reader is directed to the scientific literature to consider alternative cleaning methods appropriate for glass/ceramic substrates with thin metal tracings.
6.1 Cleaning the Honeycomb Electrodes

Caution! Sulfuric Acid is Corrosive!

Sulfuric Acid (H₂SO₄) is a corrosive acid. Wear appropriate personal protective equipment when handling sulfuric acid. Wear gloves. Do not get sulfuric acid in contact with your skin, eyes, or mucous membranes. Rinse any affected area with copious running water.

With the electrochemical cleaning method, one can qualitatively assess whether or not the surface is clean by reviewing the resultant cyclic voltammograms.

To electrochemically clean the Honeycomb electrode, follow these steps:

1. Prepare a solution of 0.5 M H₂SO₄.
2. Use the plastic pipette to fill the working electrode holes in the Honeycomb electrode.
3. Place the electrode in a cell (or small beaker) filled with the sulfuric acid.
4. Perform cyclic voltammetry repeatedly with the parameters given in Table 6-1.
5. Continue to repeat CV segments until the electrode is clean (see below).
6. Remove the Honeycomb card from solution and rinse the working electrode holes with DI water using the plastic pipette.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Gold Honeycomb Electrode</th>
<th>Platinum Honeycomb Electrode</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of Segments</td>
<td>At least 20*</td>
<td>At least 20*</td>
</tr>
<tr>
<td>Initial Potential</td>
<td>-375 mV</td>
<td>-300 mV</td>
</tr>
<tr>
<td>Upper Potential</td>
<td>1800 mV</td>
<td>1700 mV</td>
</tr>
<tr>
<td>Lower Potential</td>
<td>-375 mV</td>
<td>-300 mV</td>
</tr>
<tr>
<td>Final Potential</td>
<td>-375 mV</td>
<td>-300 mV</td>
</tr>
<tr>
<td>Sweep Rate</td>
<td>500 mV/s</td>
<td>500 mV/s</td>
</tr>
<tr>
<td>Electrolyte</td>
<td>0.5 M H₂SO₄</td>
<td>0.5 M H₂SO₄</td>
</tr>
</tbody>
</table>

Table 6-1. Electrochemical Cleaning Parameters for Honeycomb Electrodes.

*Depending on the chemical nature of use, more segments may be required to regenerate the surface.

The number of segments (or repeated cyclic voltammograms) is variable and depends on the condition of the Honeycomb electrode prior to cleaning. Start with the parameters given (see: Table 6-1).
A typical cyclic voltammogram (CV) of a gold Honeycomb electrode after electrochemical cleaning can be used for qualitative determination of electrode cleanliness (see: Figure 6-1). The electrode was cleaned by continuous voltammetric cycling in 0.5 M H$_2$SO$_4$ electrolyte at 500 mV/s sweep rate. The total time varies from 2 min to 15 min, depending on the condition of the Honeycomb electrode prior to cleaning.

While cleaning, one will see certain features in the voltammogram grow or decay, eventually stabilizing to a reproducible trace. At that time (typically 5 minutes or about 10-15 CV cycles) when new CV traces lay exactly on top of previous CV traces, the electrochemical cleaning process has completed. Clean Au surface typically shows an onset of Au oxidation around 1100 mV (vs. an Ag/AgCl/KCl gel reference electrode) and a sharp reduction peak around 900 mV.

![Cyclic Voltammogram](image)

*Figure 6-1. Electrochemical CV Signature of an Au Honeycomb Electrode in Acid.*

### 6.2 Cleaning the Quartz Cell

The quartz cell can be cleaned by rinsing it with water or ethanol, depending on the solubility of the chemicals analyzed in the cell. Over time, contamination can build up on the optical surface.
6.3 Cleaning the Cell Cap

The cap is made of PET plastic, and it can be cleaned by rinsing it with water or a common organic solvent, followed by drying it under a stream of nitrogen or compressed air.

6.4 Component Storage

All components should be cleaned and stored dry after each use.