Implementation of Experimental Electrochemistry into the Undergraduate Laboratory Curriculum: It is Easier and More Cost-Effective than you Realize

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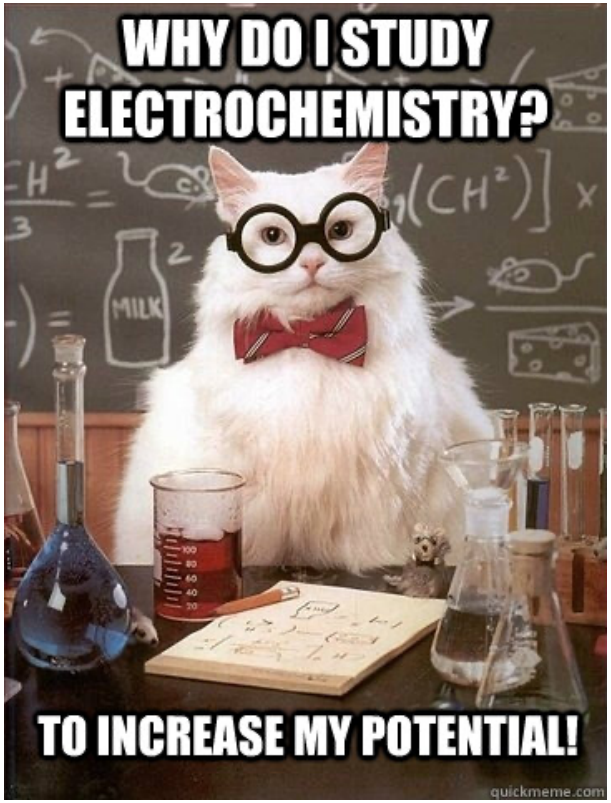
Presentation Overview

- Welcome
- Background
- Recognizing the importance of teaching electrochemistry
- Tools to conduct electrochemical based experiments
- Live Demonstration
- Why choose Pine / Instructor Resources
- Questions



PINE RESEARCH INSTRUMENTATION

2015 ACS CPT Guidelines

4.2 Instrumentation. … Approved programs must have a functioning NMR spectrometer on site that undergraduates use. The field strength and capabilities of the NMR instrumentation should support the instructional and research needs of the program. If the on-site instrument does not meet all of the program's research needs, stable arrangements must be made with proximal sites to provide ready access to appropriate NMR instrumentation. **In addition, instruments from at least four of the following five categories must be on site and used by undergraduates:**

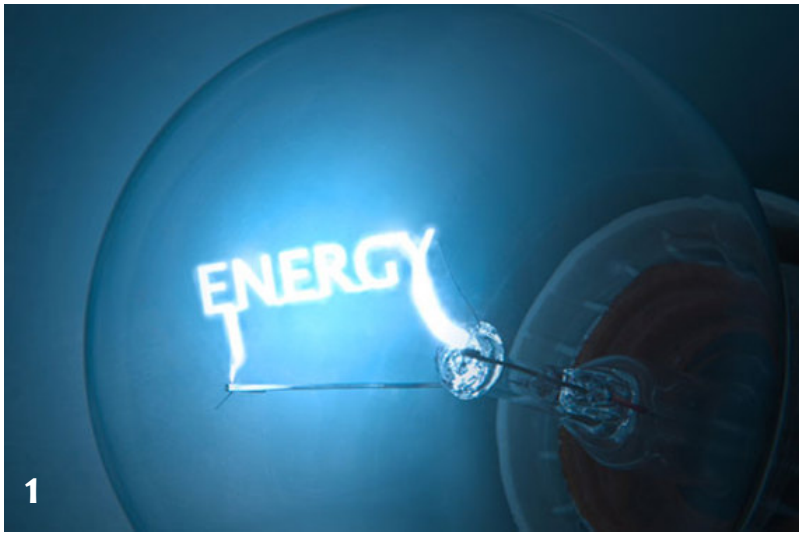
- optical molecular spectroscopy (e.g., FT-IR, fluorescence, Raman, UV-Vis)
- optical atomic spectroscopy (e.g., atomic absorption, ICP-atomic emission)
- mass spectrometry (e.g., MS, GC-MS, LC-MS)
- chromatography and separations (e.g., GC, GPC, HPLC, ion chromatography, capillary electrophoresis, SEC)
- **electrochemistry (e.g., potentiometry, amperometry, coulometry, voltammetry)**

ACS Chemistry for Life®

PINE RESEARCH INSTRUMENTATION

Electrochemistry

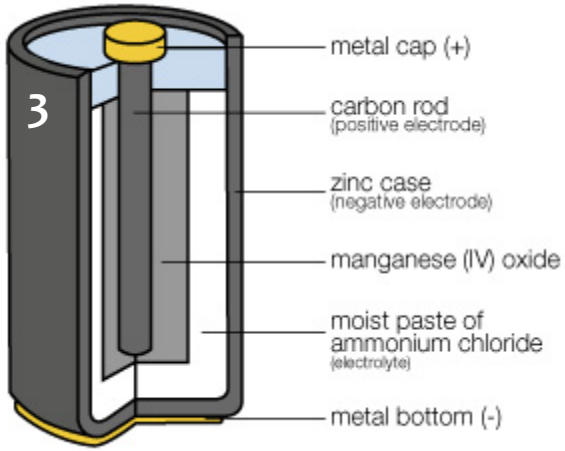
- It is more than balancing redox reactions and constructing electrolytic cells as commonly done in general chemistry
  - Heterogeneous and homogenous electron-transfer kinetics and mechanisms
  - Solution thermodynamics
  - Heterogeneous catalysis
  - Materials characterization
- Uses and applications are important and familiar



PINE RESEARCH INSTRUMENTATION

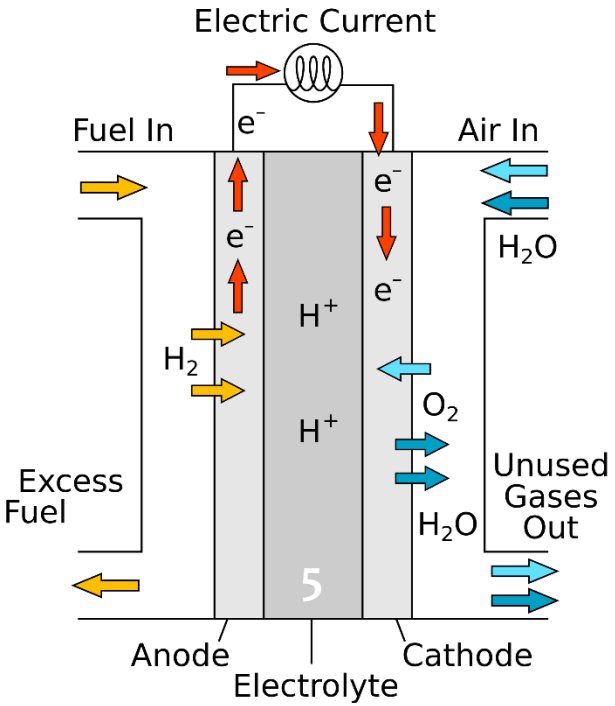
Importance of Electrochemistry

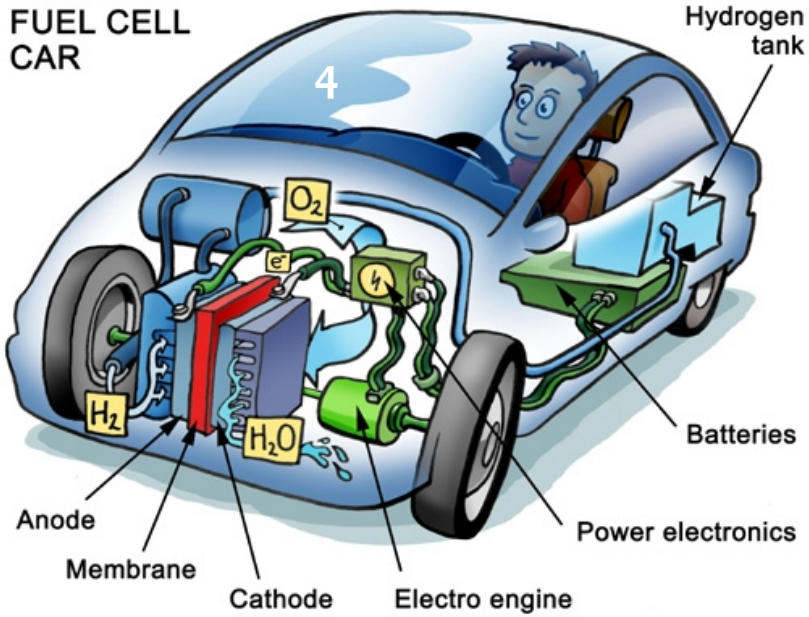
- Batteries
  - From cell phones to computers…



Importance of Electrochemistry

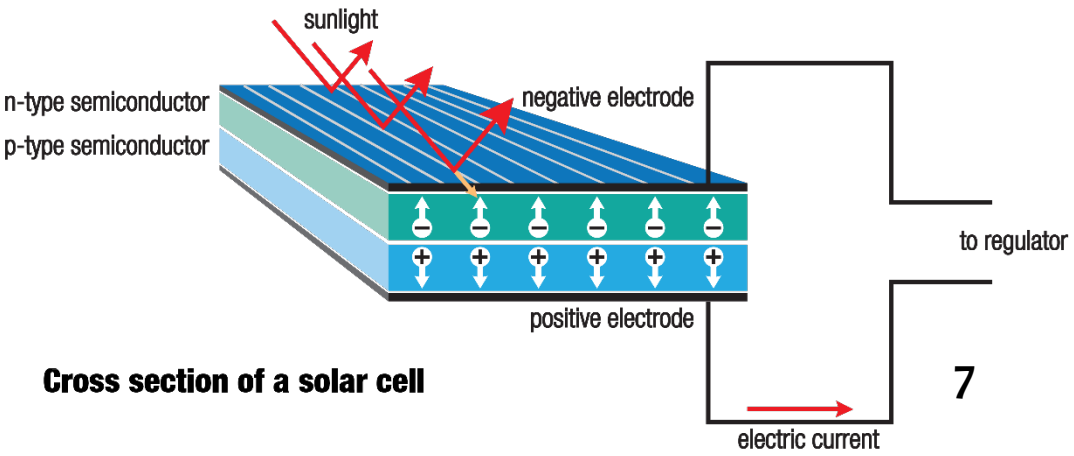
- Fuel Cells





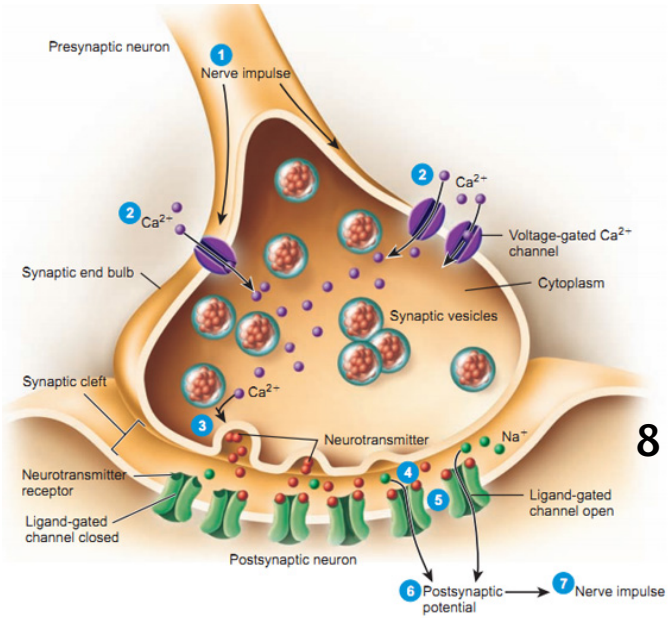
Importance of Electrochemistry

- Photovoltaics
  - Solar cells, dye-sensitized solar cells,



Importance of Electrochemistry

- Biological Signficance
  - Neuro[electro]chemistry, biosensing



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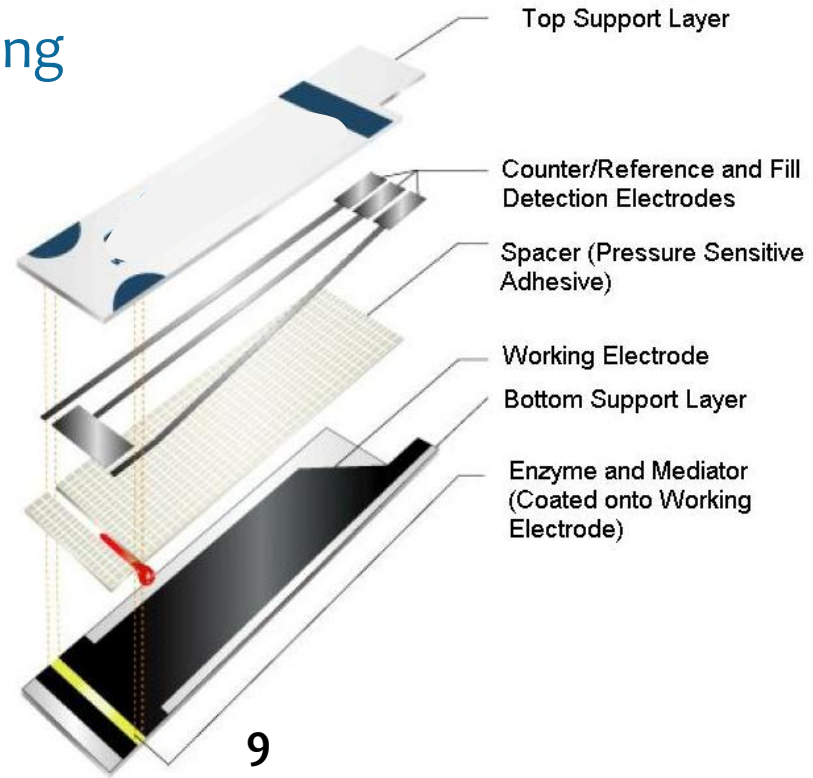

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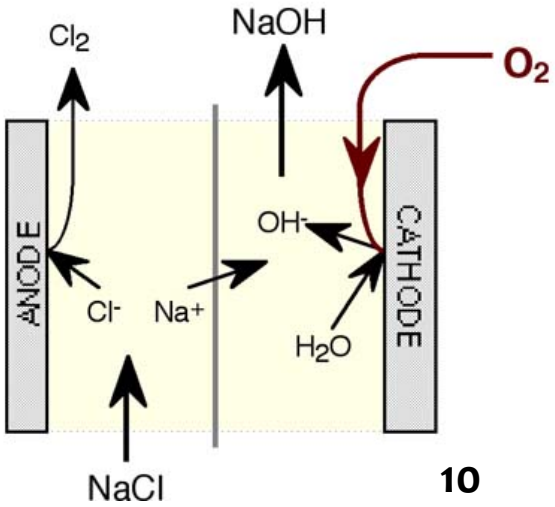
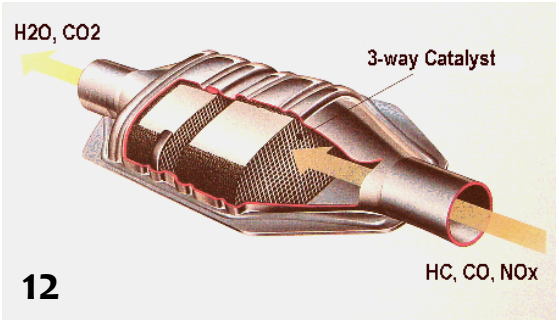
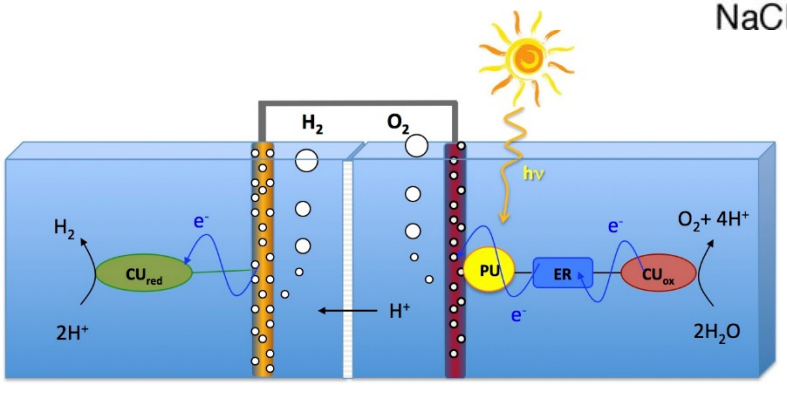
Other Important Electrochemistry

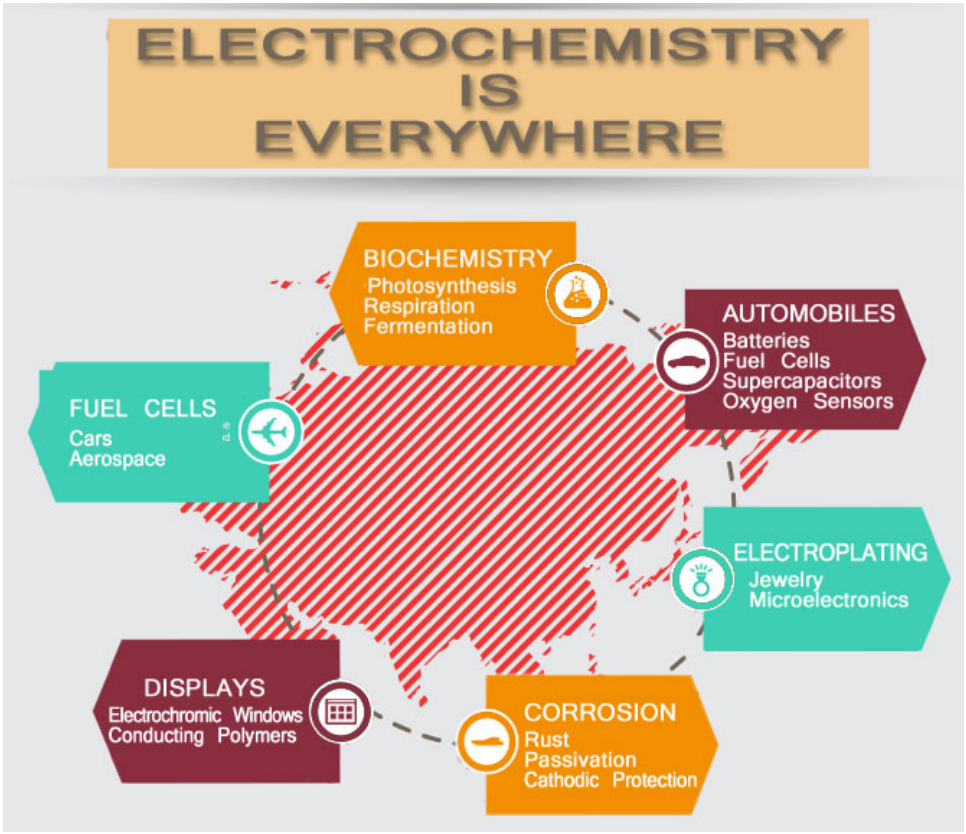
- Corrosion
- Catalysis
- Electroplating
- Electrosynthesis

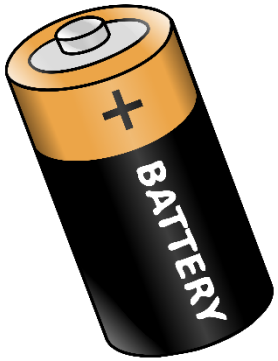






Electrochemistry is Relatable





PINE RESEARCH INSTRUMENTATION

Affordable Potentiostat

- **WaveNow Potentiostat/Galvanostat**
  - $4,335 AfterMath (software) included
  - Free software download
  - Free software updates
  - Low current (WaveNano) and extended voltage (WaveNow XV) options

PINE RESEARCH INSTRUMENTATION

Scale Up Economically

- **Bipotentiostat/Galvanostat System**
  - $6,800
  - For dual-electrode work - like rotating ring-disk (RRDE)
  - Larger current and voltage ranges
  - Auxiliary input/output
  - Advanced filters

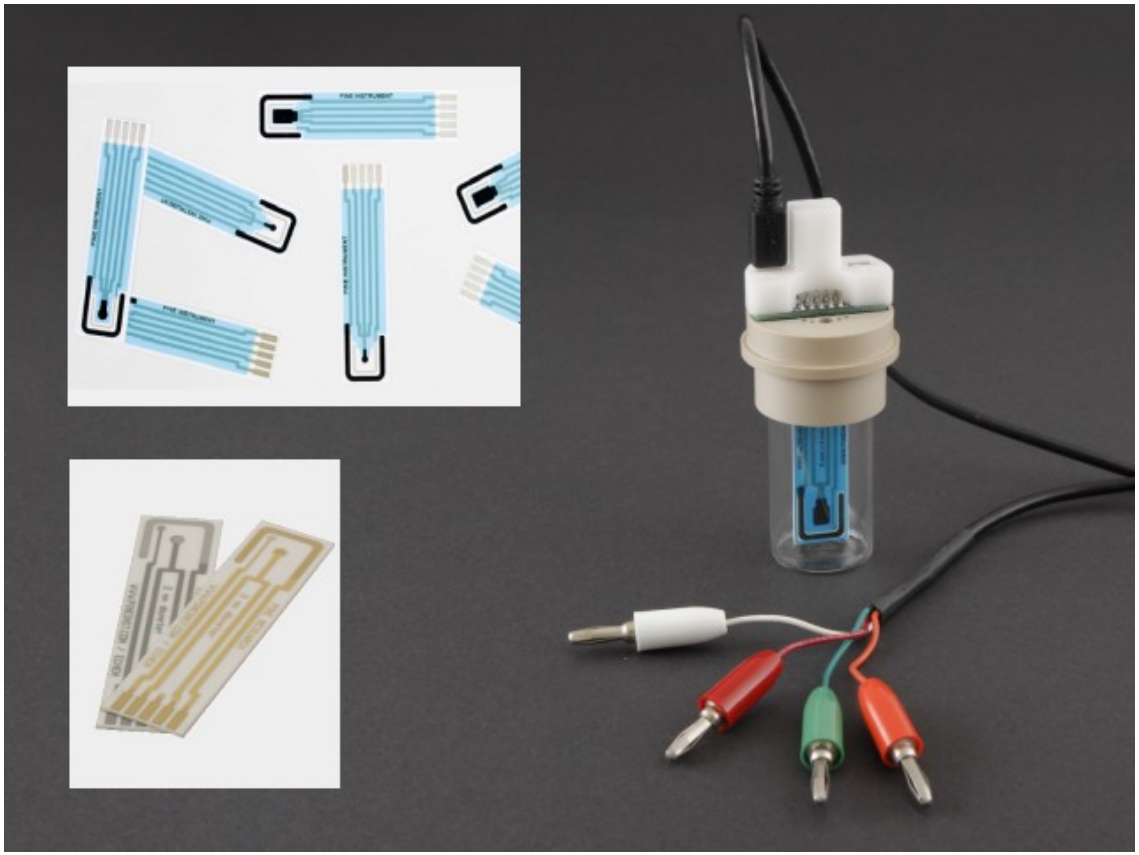
wavedriver

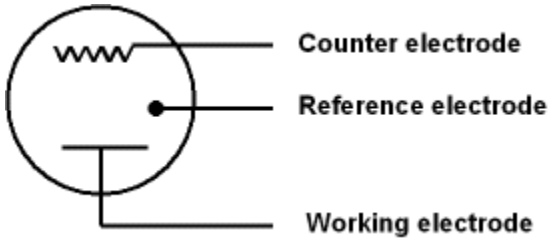
USB COMPATIBLE

aftermath COMPATIBLE

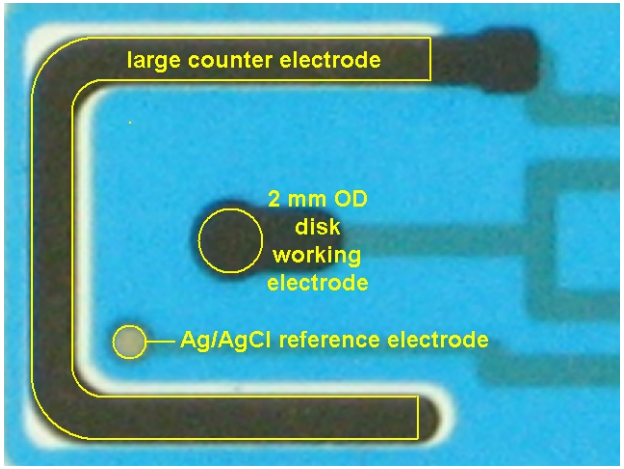
PINE RESEARCH INSTRUMENTATION

Patterned Electrodes





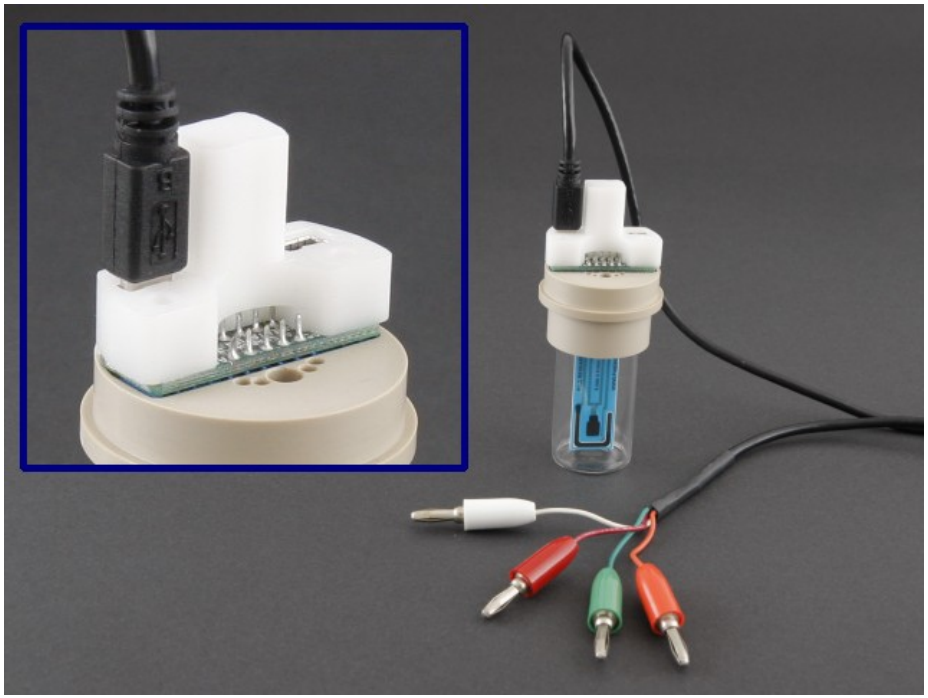
USB-style Connection



PINE RESEARCH INSTRUMENTATION

Compact Voltammetry Cell Kit

- $410
- No more expensive electrodes
- No more polishing
- Ideal platform for biosensing, solution-based studies, air sensitive (glovebox) work
- Low volume, large volume, jacketed cell options



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Economical Consumables

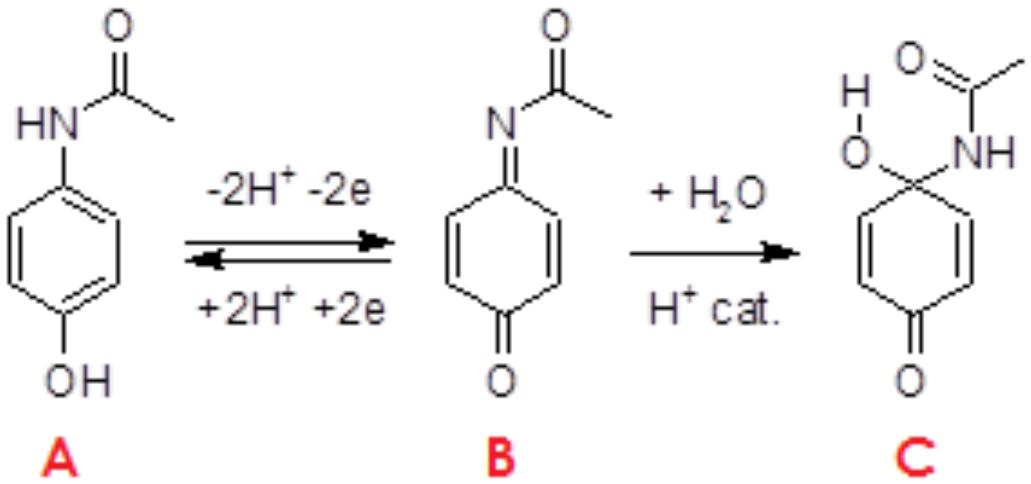
- Carbon screen printed electrodes
  - Disposable
  - $2
- Gold/Platinum patterned electrodes
  - Reusable
  - $40
- Disposable glass cells
  - 20 mL scintillation vials

AfterMath Data Organizer

- Control software for Pine potentiostats
- Workup data in the same program
- Easy to import and export
- Excellent (and cost-effective) licensing
  - Install on as many computers as many times as you wish
  - Download the software from our website
  - Free Updates
- Archive parent-child file structure
- Easy-to-follow GUI

Live Chemical Demonstration

- pH-dependent voltammetric analysis of Children's Tylenol® Suspension (4-acetamidophenol)



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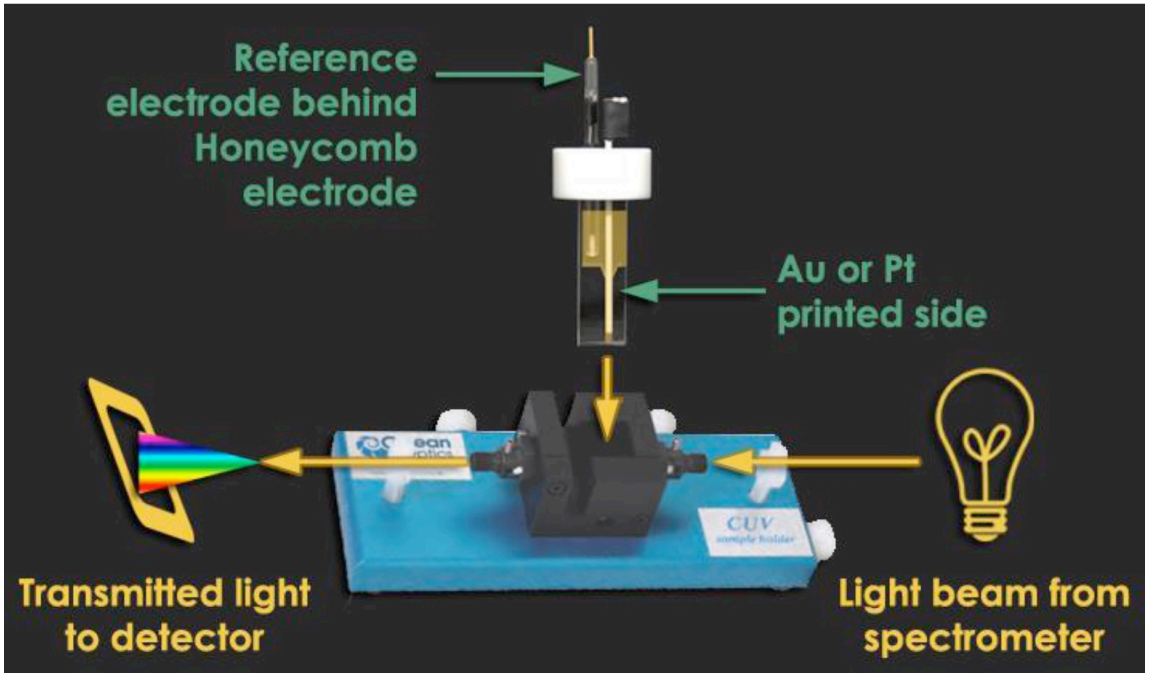
Spectroelectrochemistry

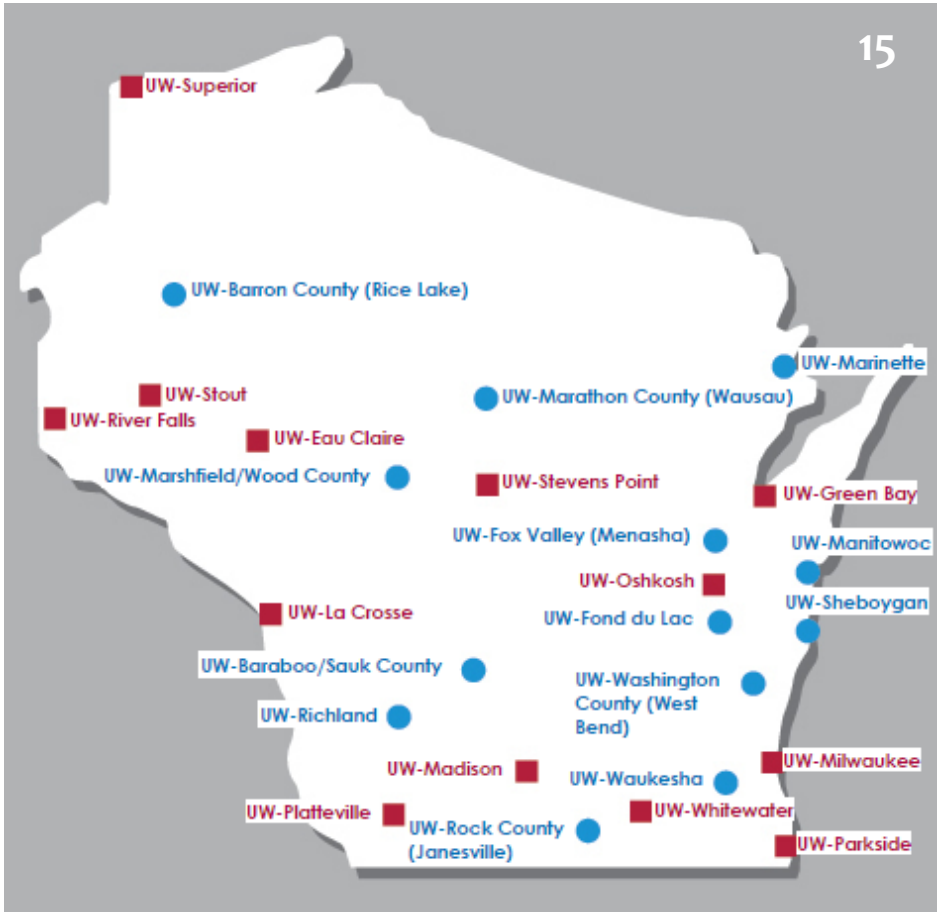
- Species concentrations are determined spectroscopically at various solution potentials
- Intermediate, radical species identification and quantification

1. $A = \varepsilon bc$

2. $E = E^o + \frac{RT}{nF} ln \frac{C_o}{C_R}$



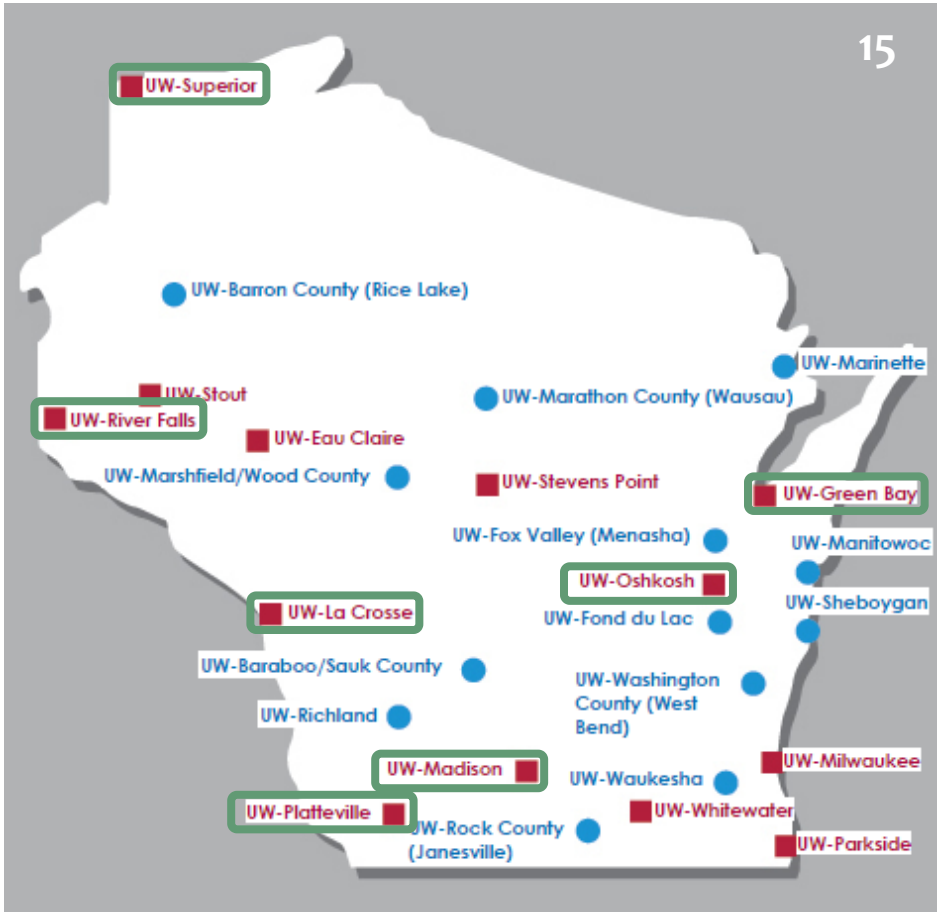
Why Choose Pine Research?



PINE RESEARCH INSTRUMENTATION

Why Choose Pine Research?

- **Local colleagues** with instrumentation familiarity
  - Green Bay
  - La Crosse
  - Madison
  - Oshkosh
  - Platteville
  - River Falls
  - Superior



Typical Instrumentation Costs

- Gas Chromatograph (starting at $20,000)
- FT-IR Spectrometer (starting at $25,000)
- FT-NMR Spectrometer (>100 MHz, starting at $200,000)
- UV/Vis Spectrometer (starting at $10,000)
- Mass Spectrometer (starting at $20,000)
- High Performance Liquid Chromatography System (starting at $15,000)
- Capillary Electrophoresis System (starting at $40,000)
- PLUS detectors, columns, ATR accessories, vacuum systems, etc.

PINE RESEARCH INSTRUMENTATION

Why Choose Pine Research?

- **Cost** – for about $5,000
  - Potentiostat
  - Electrodes
  - Cells
  - Software
- Appropriate for use in several laboratory courses
  - General Chemistry
  - Analytical Chemistry
  - Instrumental Analysis
  - Inorganic Chemistry
  - Physical Chemistry
  - Biochemistry

aftermath
DATA ORGANIZER

PINE RESEARCH INSTRUMENTATION

Support for Educators

Electrochemical Based Enzymatic Determination of Glucose in Beverages

A laboratory for quantitative determination by selective electrochemistry

This laboratory seeks to quantitatively determine glucose concentration in common beverages. The method employed is enzyme catalyzed oxidation of glucose, quantified by chronoamperometry. With an inexpensive patterned electrode and low cost glucose oxidase enzyme, an electrochemical sensor is constructed to selectively measure glucose in complicated beverages which can present as analytically challenging matrices. This lab briefly discusses conceptual information about sugars used to sweeten common beverages, enzyme-substrate interactions, glucose mutarotation, enzyme kinetics, and electroanalysis.

PINE RESEARCH INSTRUMENTATION

Support for Educators

Electrochemical Based Enzymatic Determination of Glucose in Beverages

A laboratory for quantitative determination by selective electrochemistry

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Highly Sensitive Electrochemical Determination of Lead in Tap Water

Anodic Stripping Voltammetry with Disposable Screen Printed Carbon Electrodes

This experiment demonstrates the use of anodic stripping voltammetry to determine lead (Pb) in tap water. Inexpensive and disposable screen-printed carbon electrodes are first coated with mercury micro-droplets (microns in diameter) by reduction of mercury acetate (Hg^{2+}) at a constant electrode potential. Then, lead ions (Pb^{2+}) in a sample are reduced and collected into the mercury droplets. This pre-concentration step allows determination of very low concentrations (ppb) of lead when anodic differential pulse voltammetry (DPV) is used as the method of detection and standard addition as the method of calibration.

Support for Educators

Electrochemical Based Enzymatic Determination of Glucose in Beverages

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Highly Sensitive Electrochemical Determination of Lead in Tap Water

Anodic Stripping Voltammetry with Disposable Screen Printed Carbon Electrodes

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Exploring Faraday's Law Using Inexpensive Screen-Printed Electrodes

A simple electroplating experiment for the general chemistry laboratory

This note describes an experimental procedure for the general chemistry laboratory student that conveniently illustrates the electroplating process. Students plate a thin film of nickel on to a screen-printed carbon electrode. The plating process is accurately controlled using a traditional three-electrode electrochemical cell arrangement. Each screen-printed electrode pattern includes all three required electrodes (working, reference, and counter). Nickel is plated on to

PINE RESEARCH INSTRUMENTATION

Support for Educators

Electrochemical Based Enzymatic Determination of Glucose in Beverages

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Highly Sensitive Electrochemical Determination of Lead in Tap Water

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Exploring Faraday's Law Using Inexpensive Screen-Printed Electrodes

A simple electroplating experiment for the general chemistry laboratory

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Determination of Acetaminophen in a Children's Pain Relief Elixir

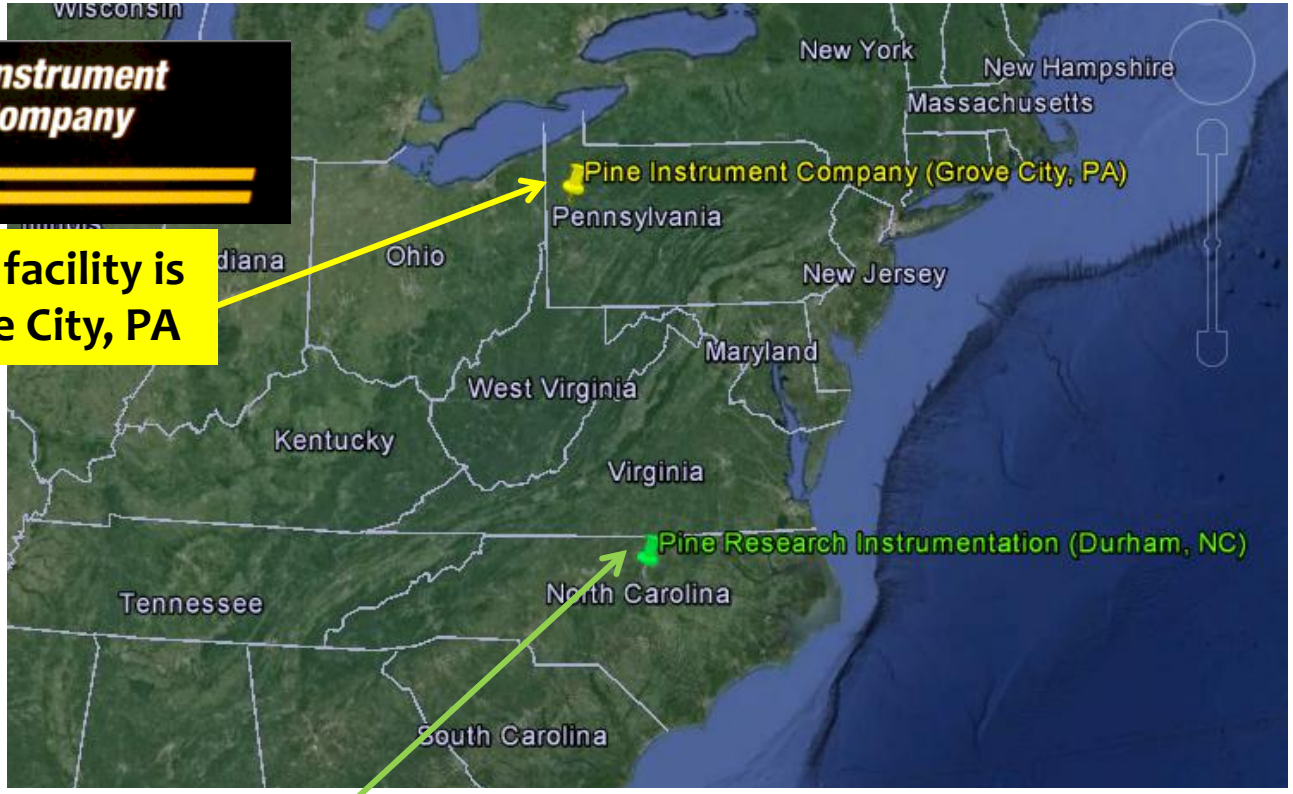
An inexpensive experiment using disposable screen-printed carbon electrodes

PINE RESEARCH INSTRUMENTATION

Where is Pine Located?

PINE Instrument Company

Our production facility is located in Grove City, PA



Sales, R&D, and Software office is located in Durham, NC

PINE RESEARCH INSTRUMENTATION

Facts About Pine

- Our instruments are made in the USA (Grove City, PA)
- Instead of service contracts, we make ourselves available to customers
  - On site visits
  - Call our phone number, talk to a human
  - Continued support for the lifetime of the instrument
- Research and Educational support

PINE RESEARCH INSTRUMENTATION

Contact Us

- By phone
  919.782.8320
- Technical support, service, electrochemical questions
  - Tim Paschkewitz
    tpaschkewitz@pineinst.com
- Sales, quotes, orders
  - pinewire@pineinst.com

PINE RESEARCH INSTRUMENTATION

Presentation References

1. www.shutterstock.com
2. http://www.sofreshandsogreen.com/2010/10/21/dont-throw-out-those-batteries/
3. http://www.ecokids.ca/pub/eco_info/topics/batteries/index.cfm
4. http://www.hydrogen-fuelcells.com/2012/08/cars-with-fuel-cells.html
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6. http://greatbrooksolar.com/
7. http://www.google.com/imgres?imgurl=&imgrefurl=http%3A%2F%2Fwww.redarc.com.au%2Fsolar%2Fabout%2Fsolarpanels%2F&h=0&w=0&tbnid=6r7DvNWW1JkpDM&zoom=1&tbnh=143&tbnw=353&docid=XV5EWMe0-EYn9M&tbm=isch&ei=8-k3VILiI4fOggS_yILABg&ved=0CAQQsCU0AA
8. http://antranik.org/synaptic-transmission-by-somatic-motorneurons/
9. http://knowledge.electrochem.org/encycl/art-g01-glucose.htm
10. http://www.lanl.gov/orgs/mpa/mpa11/chlor.htm
11. http://gotayloronline.com/index.php/the-costs-of-corrosion/
12. http://www.aa1car.com/library/converter.htm
13. http://mariesircoglou.wordpress.com/
14. http://www.cvs.com/shop/product-detail/Tylenol-Pain-&-Fever-Liquid-Cherry-Flavor?skuId=876684
15. www.Wisconsin.edu

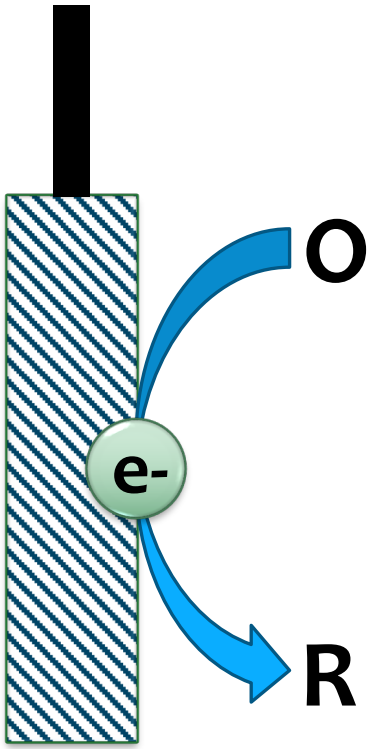
PINE RESEARCH INSTRUMENTATION

Resource Slides

- The slides that follow are for your reference.

What is Electrochemistry?

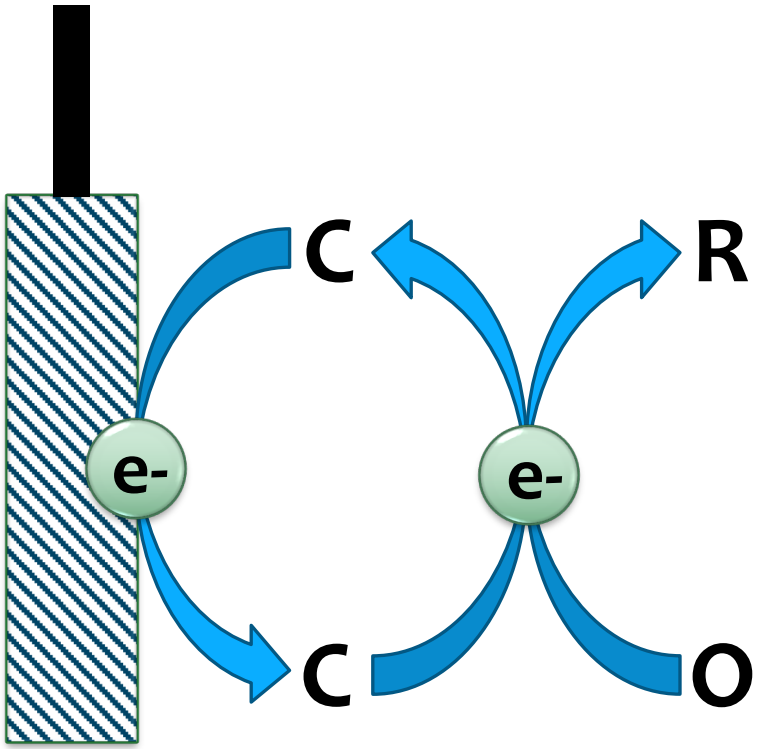
- Interrelation of electrical and chemical effects
  - Electron transfer in solution, at interfaces, between molecules
  - Relationship of mass (concentration) to electrical parameters (current and potential)
  - Kinetics and thermodynamics of redox reactions
  - $O + ne \rightleftharpoons R$ at a specific potential, E



Direct electron transfer

What is Electrochemistry?

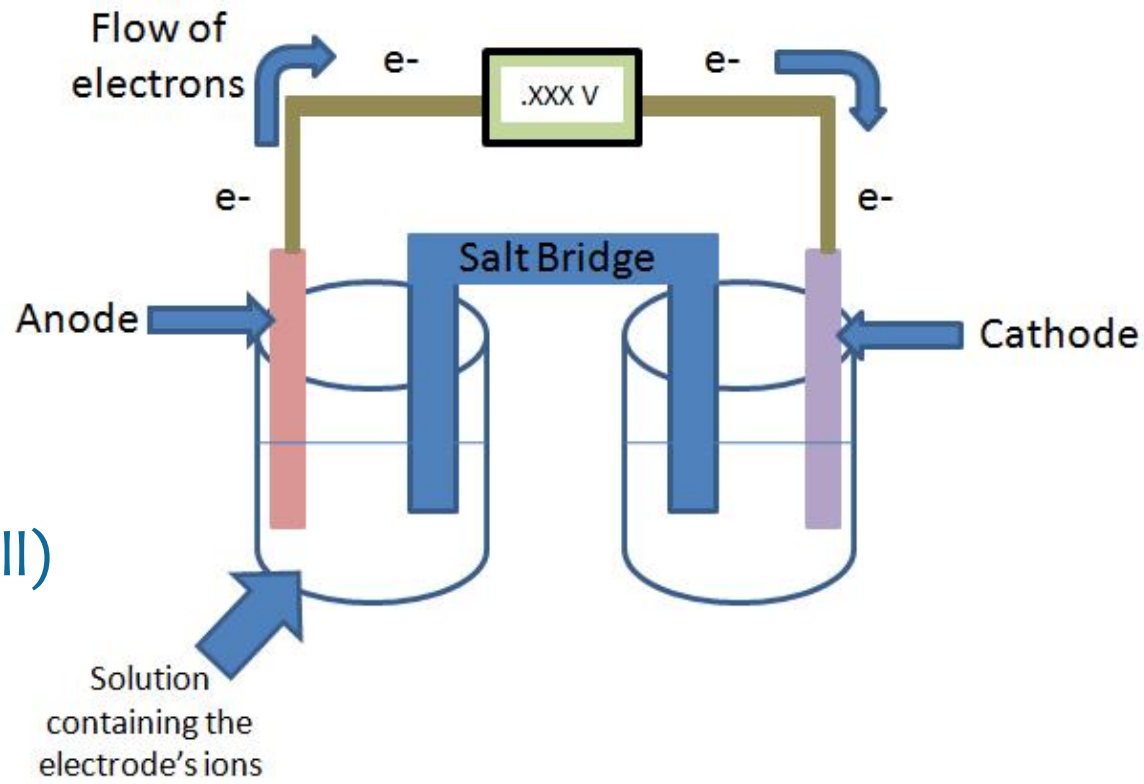
- Interrelation of electrical and chemical effects
  - Electron transfer in solution, at interfaces, between molecules
  - Relationship of mass (concentration) to electrical parameters (current and potential)
  - Kinetics and thermodynamics of redox reactions
  - $O + ne \rightleftharpoons R$ at a specific potential, E



Indirect (mediated) electron transfer

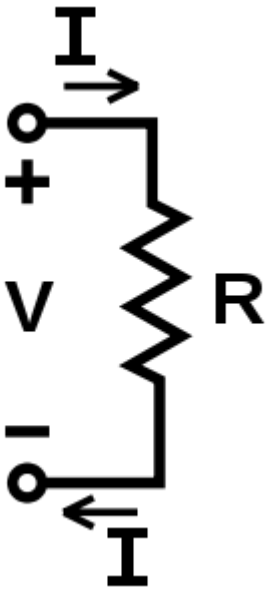
Electrochemistry in Brief

- For $O + ne \rightleftharpoons R$
- $E = E^o + \frac{RT}{nF} \ln \frac{C_o}{C_R}$
- Mass transport by diffusion
- Voltaic (spontaneous cell)
- Electrolyte background



Imagine a Circuit

- Electronic component – like a circuit
  - Ohm's Law (from Physics), but expanded
  - $V = iR$
  - Resistance is a variable in electrochemistry
- Relate current that flows to mass/concentration
  - Faraday's Law
  - $it = nNF \; where \; it = Q$
  - Q = charge, related to moles of a substance times Faraday's constant (96,485 Coulombs/mole)



AfterMath Experiments

Simple Methods
Open Circuit Potential (OCP)
Constant Potential Electrolysis (BE)
Constant Current Electrolysis (BE)

Voltammetric Methods
Cyclic Voltammetry (CV)
Linear Sweep Voltammetry (LSV)
Staircase Voltammetry (SCV)
Chronoamperometry (CA)
Double Potential Step Chronoamperometry (DPSCA)
Cyclic Step Chronoamperometry (CSCA)
Differential Pulse Voltammetry (DPV)
Square-Wave Voltammetry (SWV)
Normal Pulse Voltammetry (NPV)

Galvanostatic Methods
Chronopotentiometry (CP)
Current Ramp Chronopotentiometry (CRP)
Cyclic Chronopotentiometry (CCP)
Cyclic Step Chronopotentiometry (CSCP)

Stripping Voltammetry
Anodic & Cathodic Stripping Voltammetry (ASV)
Differential Pulse Stripping Voltammetry (DPSV)
Square-Wave Stripping Voltammetry (SWSV)

Rotating Disk Methods
Rotating Disk Electrode (RDE)
Koutecky-Levich Series (KL-RDE)
Rotating Disk Electrolysis (BE-RDE)
Rotating Disk Chronopotentiometry (CP-RDE)
Rotating Disk Cyclic Chronopotentiometry (CCP-RDE)

Rotating Ring-Disk Methods
Rotating Ring-Disk Voltammetry (RRDE)
Rotating Ring-Disk Koutecky-Levich (KL-RRDE)
Rotating Ring-Disk Electrolysis (BE-RRDE)

Dual Electrode Methods
Dual Electrode Electrolysis (DEBE)
Dual Electrode Voltammetry (DECV)

Glassware

- Don’t have a glassblower handy? Consider Pine.

Spectroscopy Analogy

Electroanalysis

- Signal is a function of energy (electrical potential)
- Energy levels interrogated by interaction of electricity and electroactive molecules
- Chemical species respond to specific electrical potentials
- Diffusion coefficient (D) proportionality constant for specific molecule
- Advanced study requires a potentiostat

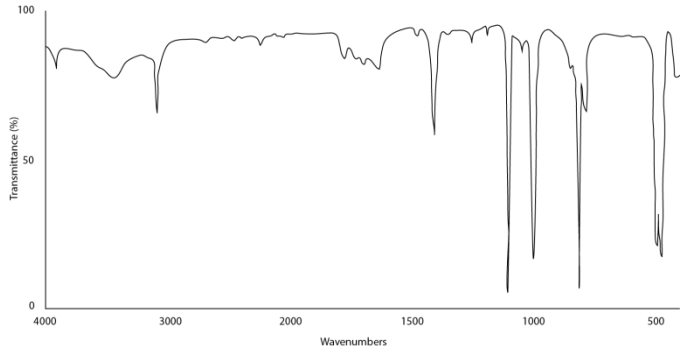
Spectroscopy

- Signal is a function of energy (wavelength)
- Energy levels interrogated by interaction of light and molecules
- Chemical species respond to specific wavelengths
- Molar absorptvity (ε) proportionality constant for specific molecule
- Advanced study requires a spectrometer

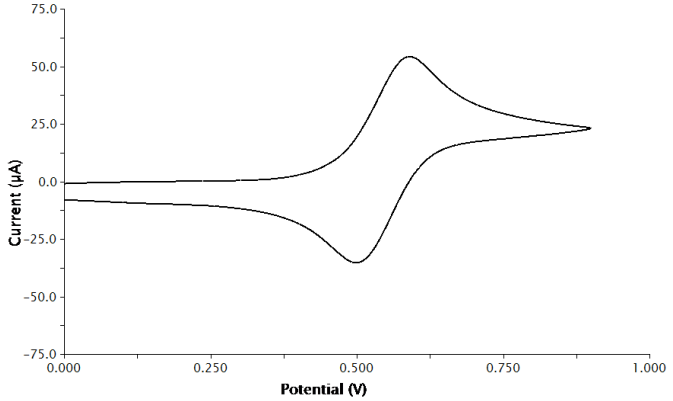
Electrochemical Data Analysis

- Like spectroscopic and chromatographic data, electrochemical data are information rich

- Peak position (wavenumber)
- Peak height (stretching intensity)
- Peak separation (symmetry)
- Slope and tailing (solvent effects, overlapping energy bands)



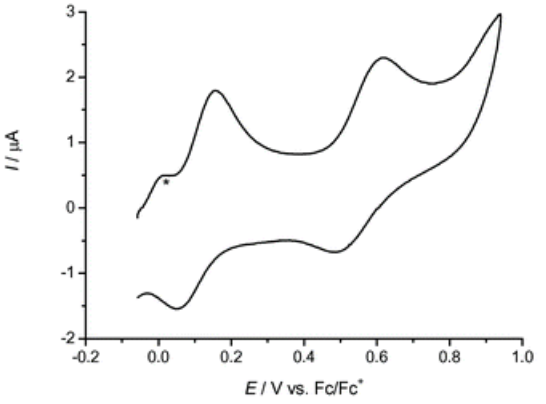
- Peak position (potential)
- Peak height (current)
- Peak separation (reversibility)
- Slope and tailing (kinetics)

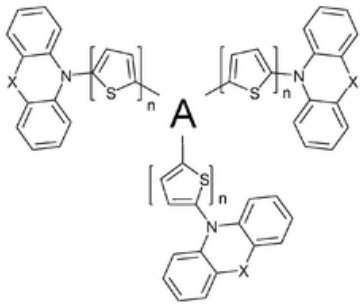


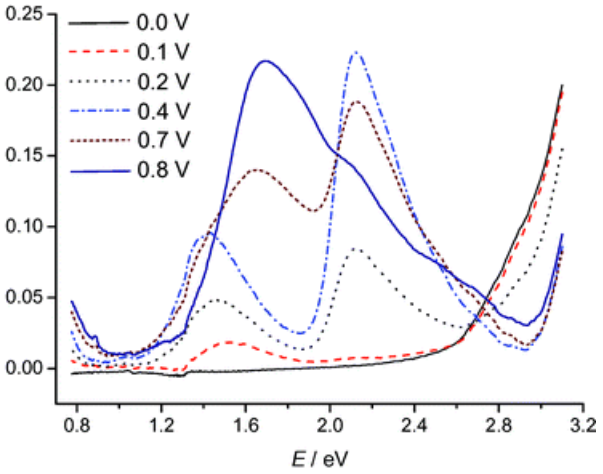
IR spectra and cyclic voltammogram of ferrocene

Spectroelectrochemistry

- Identify electroactive species or products of redox reactions
- Under potential control, spectroscopic information (electronic absorption, vibrational modes, scattering, etc.) about in situ electrogenerated species readily obtained







2-diarylaminothiophene CV and potential-dependent UV

Electrochemical Biosensors

- Use electrochemical methods to convert chemical concentration information (biological) to electrical signals (electrochemical)
- Biorecognition interface – label + electrode
- Measure current/potential directly or indirectly

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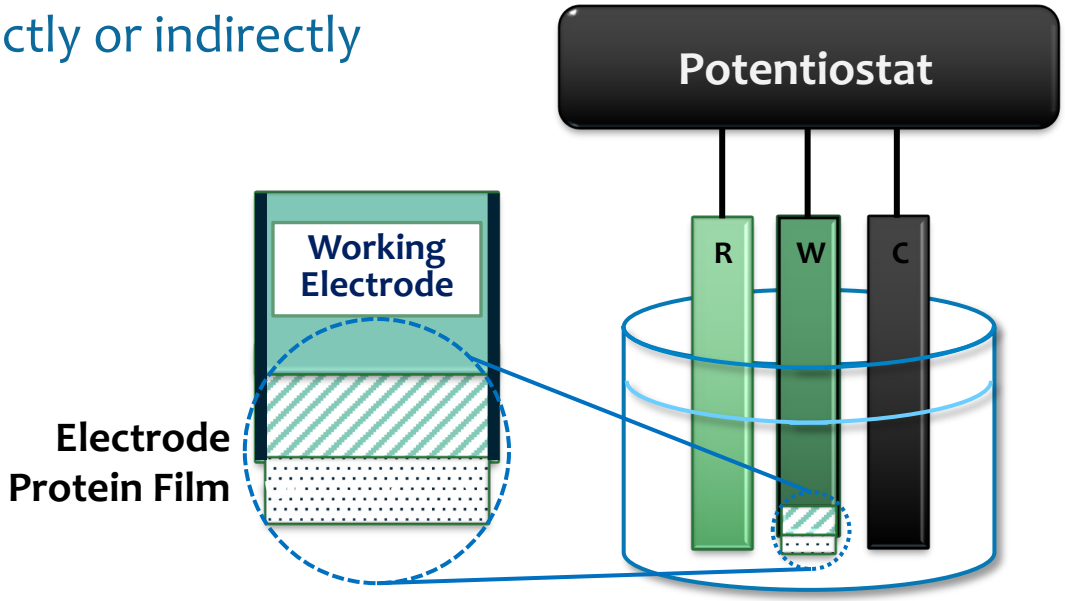

R = reference W = working C = counter

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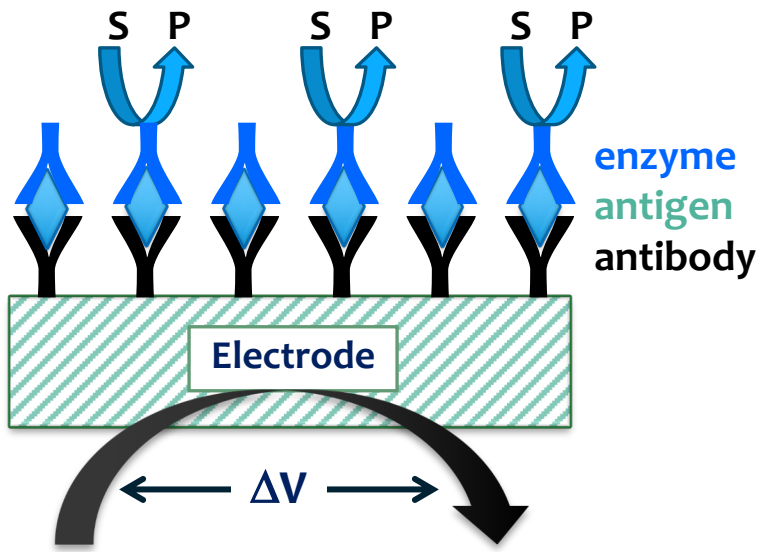
Biological Assays



- Antibody/Antigen Assay
  - Increased specificity
  - High sensitivity
  - Rapid test (measure current transient)
- Prostate specific antigen (PSA) serum screen
- Enzyme Linked Immunosorbent Assay (ELISA) for HIV screening

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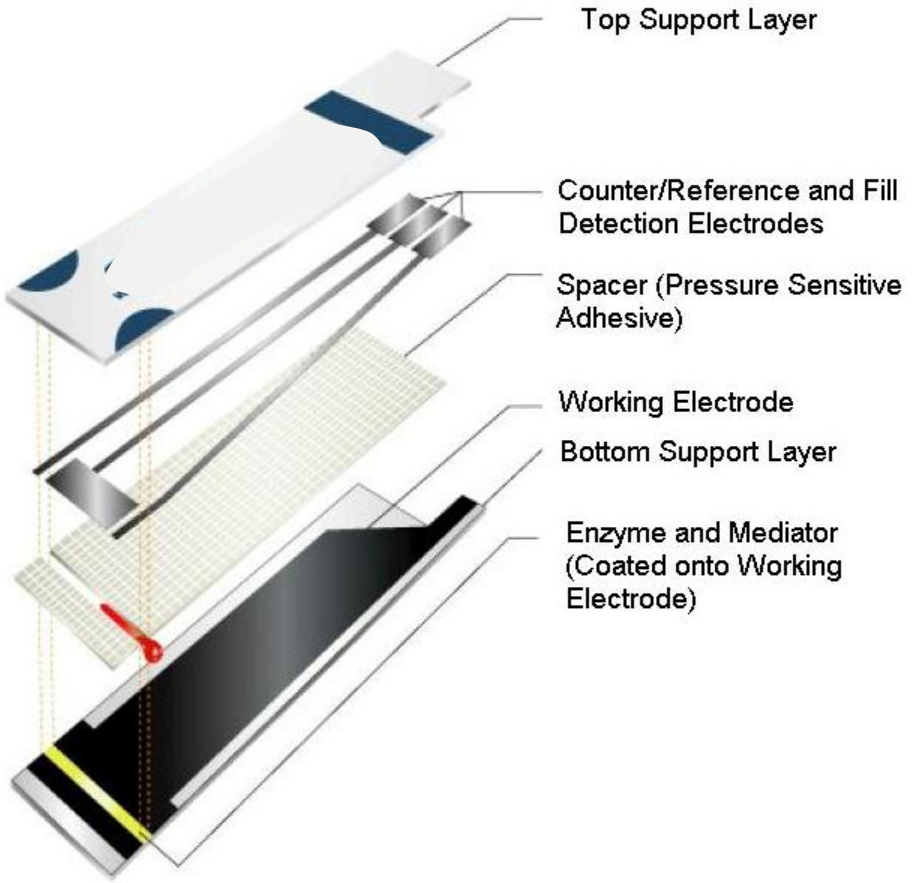
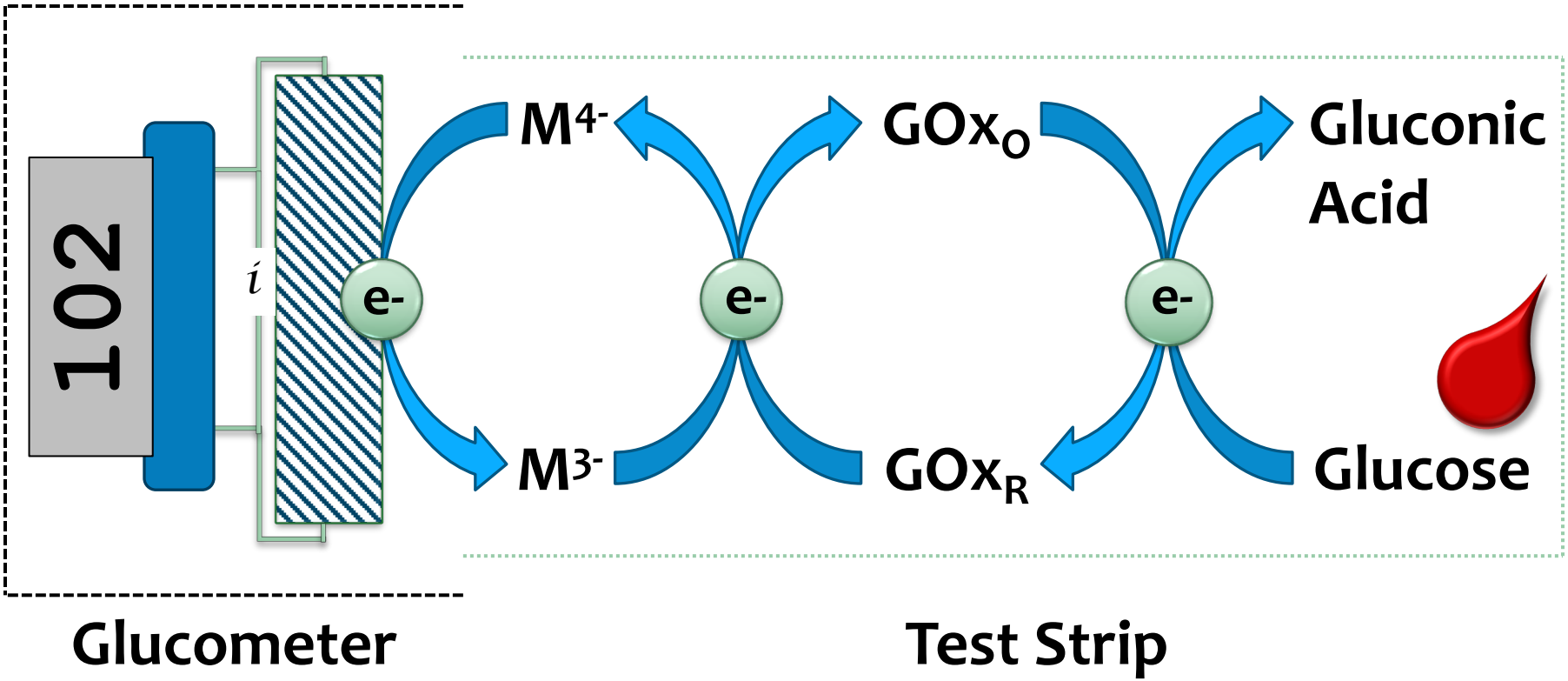
Blood Glucose Monitoring

- Blood Glucose Monitoring
  - Enzymes
    - Glucose Oxidase
    - PQQ, NAD, and FAD-Glucose Dehydrogenase
  - Chemical Mediator
    - Ferricyanide
    - Ferrocene
    - 1,10-Phenanthroline



PINE RESEARCH INSTRUMENTATION

Electrochemistry of Test Strips



Gox = glucose oxidase, M = mediator