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Electrochemistry Bench and Field

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ABSTRACT

The study of chemical processes that result in electrical currents and potentials is known as electrochemistry. It has been widely applied in academic and industrial contexts, including biochemistry, energy storage and conversion, batteries, corrosion, materials research, deposition, and organic electrosynthesis. Two or three electrodes are placed in a tiny cell or vial to create an electrochemical cell setup. The cell has a working electrode (WE) and a counter electrode (CE) for a two-electrode setup. The potential difference between WE and CE is measured while the current is equal and in the opposite direction at the two electrodes. Similar to a 2-electrode setup, a 3-electrode setup also consists of a WE, CE, and reference electrode (RE). While the current is still equal and in the opposite direction at the WE and CE, the potential difference between the WE and RE is measured in this instance. The latter's capacity to separate or decouple current and potential measurements, which improves control over the electrochemical measurement, is a crucial advantage. As seen in Figure 1, several variables affect the signal generated in an electrochemical cell. Some of these parameters, like the electrode material and geometry, electrical variables (like the applied potential), and environmental factors (like the temperature), are easier for the end user to control than others, like mass transfer and solution variables (like diffusion, adsorption). Whatever the case, it is critical to fine-tune the controllable variables to get reliable and accurate electrochemical data. The success rate can be considerably increased by selecting an appropriate electrode material for the measurement being made and the study's intended use in general.

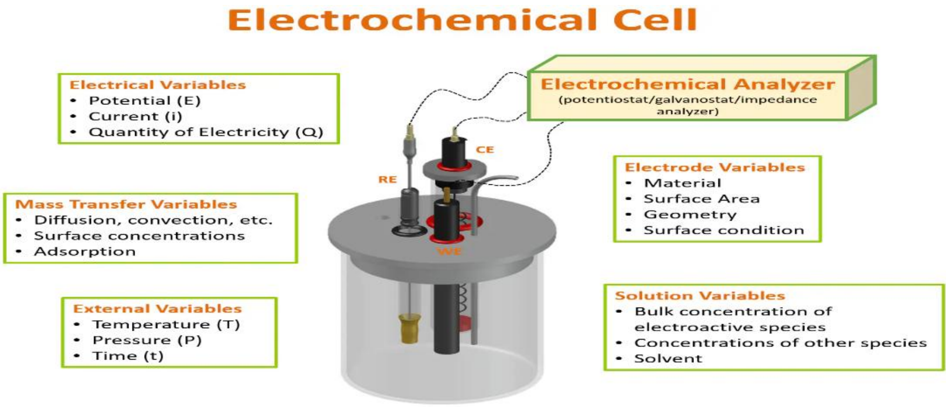


Figure 1 shows the electrochemical measuring setup with three electrodes and the variables that affect it.

INTRODUCTION

For Electrochemical Research Electrodes

Normative Electrodes

With extreme accuracy, reference electrodes (RE) measure and apply a voltage between electrodes. Due to its capacity to dissociate potential and current measurements in an electrochemical cell, using a reference electrode makes it possible to compare potential readings between studies accurately. Selecting the appropriate reference electrode for a particular experiment is critical because it must have crucial characteristics, including a stable and repeatable potential over time, non-polarizability, low solubility, and reversible coupling that complies with the Nernst equation.

Aqueous electrochemical studies reference electrodes

In an aquatic environment, common reference electrodes include hydrogen, silver/silver chloride (Ag/AgCl), and calomel (Hg/Hg2Cl2). Due to its stability, usability, easy fabrication, and disposal, the Ag/AgCl reference electrode is the most popular aquatic reference electrode. Electrolyte solutions containing 3M (or saturated) sodium chloride or potassium chloride can assemble this reference electrode. Although this RE has shortcomings, it exhibits the necessary qualities for a reference electrode. Liquid junction potentials, water in the solvent solution, and precipitation at the frit, which can generate noise in the electrical readout, are some of its drawbacks. By swapping out the CoralPor® frits and electrolyte solution in the reference compartment, the reference electrode can be cleaned and regenerate if the frit is compromised (e.g., blocked, discoloured, fractured, or chipped).

Product Suggestions:

Catalog Number	**Description**
BASMF2052-1EA	**Ag/AgCl (3M NaCl) Reference Electrode** Silver/silver chloride reference electrode with flexible connector (filled with 3M sodium chloride solution)
BASMF2056-1EA	**Ag/AgCl (3M KCl) Reference Electrode** Silver/silver chloride reference electrode with flexible connector (filled with 3M potassium chloride)
BASMF2064-1EA	**Replacement CoralPor® for Reference Electrodes** 1/8” chunks CoralPor® with 1/2” tubes Teflon heat shrink (5/pk)

There is specific research with organic, non-aqueous environments. Using a suitable, functional reference electrode in this environment is essential to the effectiveness of the measurement since the solution's composition and concentration of electroactive species present predominantly contribute to the electrochemical signal. The Ag/Ag+ reference electrode has become more popular among experts in the field, even though there are few commercially available reference electrodes for non-aqueous electrochemical experiments. In this process, a silver wire makes contact with an AgNO3, AgClO4, or other silver salt solution in the selected electrolyte.

The electrolyte in the reference compartment frequently matches the electrolyte in the solution. Tetrabutylammonium perchlorate (TBAP), tetraethylammonium perchlorate (TEAP), tetrabutylammonium hexafluorophosphate, and tetrabutylammonium fluoroborate are only a few of the salts that have been used in several research in the scientific literature. These organic salts are often dissolved using polar or dipolar aprotic solvents, such as acetonitrile (ACN), dimethyl formamide (DMF), dimethylsulfoxide (DMSO), and propylene carbonate (PC). When a solvent (such as methylene chloride or tetrahydrofuran) cannot sufficiently dissolve a salt, a more polar yet miscible solvent, such as acetonitrile, is advised.

Functional Electrodes

The conductive surface where the electrochemical reaction takes place is provided by working electrodes (WE). The electroactive surface of a particular working electrode allows for the transfer of electrons to and from the bulk liquid solution as the voltage is applied, resulting in a current response that produces an electrochemical readout. At the working and counter electrode surfaces, the resultant current from the applied voltage is equal and in the opposite direction. Depending on the application, a different working electrode must be chosen. To enable consistent and reliable electrochemical tests, it must be typically inert and conductive, have effective electron transfer kinetics, and have favorable thermodynamics.

Platinum, gold, and glassy carbon is the most well-known materials investigated and employed as working electrodes over time. Silver, nickel, zinc, and copper are other components. A working electrode must have a flawless surface with just the proper roughness for the electrochemical reaction. Imagine that the working electrode surface is contaminated with impurities, such as molecules diffusing from the bulk solution or a particle lodged in the electrode surface. In that instance, this significantly impacts the kinetics of electron transfer and the current responsiveness of redox-active species in the solution, leading to findings that cannot be replicated.

In this situation, the electrode may need to be cleaned and pretreated by being thoroughly rinsed with methanol and deionized water to remove any adsorbed species. Hand polishing with a diamond or alumina suspension applied to a micro cloth or polishing pad may be necessary for stubborn debris or organic particles. Always polish materials from coarse to fine, thoroughly washing and switching pads between each stage.
Product Suggestions:

Catalog Number	Description
BASMF2012-1EA	**Glassy Carbon (GC) Electrode for Voltammetry -** 3.0mm diameter
BASMF2014-1EA	**Gold Electrode (Au) for Voltammetry** - 1.6
BASMF2060-1KT	**PK-4 Electrode Polishing Kit** Kit includes: Electrode modifier - fine grit polishing pads (5) Diamond polishing pads (white, nylon) (10) Alumina polishing pads (tan, velvet) (5) Polishing glass plate (2) Electrode modifier - coarse diamond polish - 15 um (1) Electrode modifier - fine diamond polish - 3 um (1) Electrode modifier - ultrafine diamond polish - 1 um (1) Electrode modifier - polishing alumina (1)

Reverse Electrodes/Counter Electrodes

Counter electrodes (CE) are highly inert, conductive metal electrodes used as a source or sink of electrons in the cell and to "counter" or balance" the electrochemistry at the working electrode. The potential of the counter electrode is determined by the electrochemistry taking place at its surface.

A counter electrode should typically have a higher surface area than the working electrode in addition to the characteristics above to prevent overloading the current. Graphite rods, stainless steel, gold, silver, and platinum in various geometries (such as wire, flag, or gauze) have all been used as counter electrodes.

Product Suggestion:

Catalog Number	Description
BASMW1032-1EA	**Platinum Auxiliary Electrode**

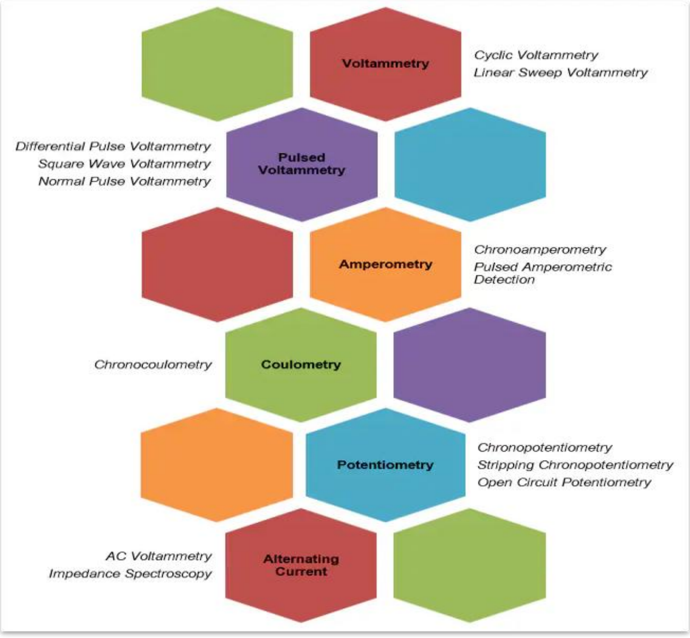
ELECTROCHEMICAL RESEARCH TECHNIQUES

Electrochemical Methodologies

Since electrochemistry was first employed in academic and industry studies, numerous electrochemical techniques have been created and applied. Depending on the end-user, the needed quantitative data determines which electrochemical approach is preferred over another.

Voltammetry, amperometry, coulometry, potentiometry, and impedance spectroscopy are typical methods for obtaining electrochemical measurements (Figure 2). However, cyclic voltammetry is the most common fundamental electrochemical technique.

Electrochemical methods, Figure 2.



An effective first characterization method is cyclic voltammetry (CV), which delivers valuable data on the kinetics, thermodynamics, stability, and adsorption of redox-active species in the solution. The CV can identify real-world samples like acetaminophen, as shown in the example below.

Electrochemistry in the Field: Cyclic Voltammetry-Based Small Molecule Sensing: The main component of Tylenol, a commonly used painkiller, is acetaminophen (N-acetyl-p-aminophenol, or APAP). When given in high quantities, one side effect is the potential for liver and renal damage. The oxidation of the medication is one of the liver's main metabolic processes. Therefore, it is advantageous to research acetaminophen's oxidation (redox) chemistry and its metabolites. It functions electrochemically because it is a mono-substituted aminophenol. The voltammetricbehavior indicates the 2-electron oxidation at moderate voltage with significant but not perfect reversibility (Figure 3). The experiment's solution pH and scan rate can be changed to get data that can be compiled to explain the mechanism.

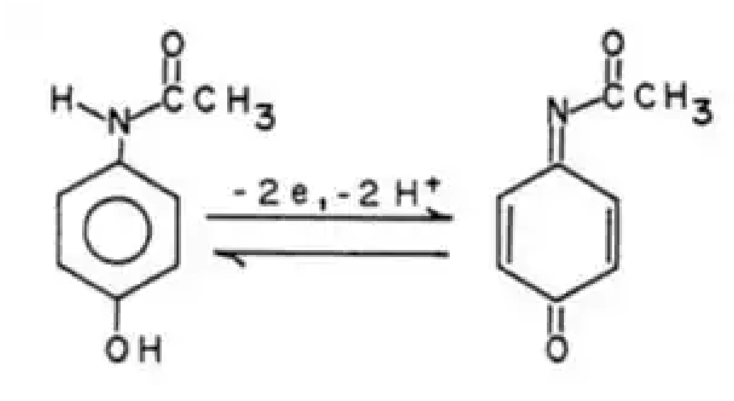


Figure 3 Shows the oxidation of acetaminophen by two electrons and two protons to an electrochemically active intermediate.

This illustration shows how CV determines quantitative concentration and mechanistic information in a real-world setting. CV measurements can be utilized quantitatively to ascertain unknown acetaminophen content in an acetaminophen tablet and qualitatively to show mechanistic information, offering an alternative to more traditional approaches in detecting some small molecule medicines.

The glassy carbon working electrode (GCE) (BASMF2012-1EA), platinum wire auxiliary electrode (BASMW1032-1EA), and Ag/AgCl reference electrode (BASMF2052-1EA) were placed in the solution to be studied to form the electrochemical cell in this investigation.

In order to create a calibration curve (Figure 4) and determine if acetaminophen saturating concentrations were present (Figure 5), CVs were acquired for five standard solutions.

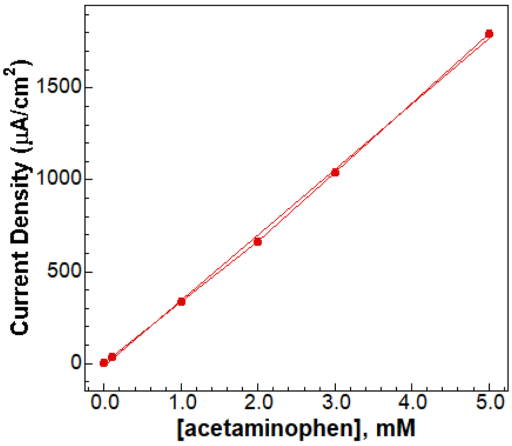


Figure 4: Acetaminophen Standards Calibration Curve

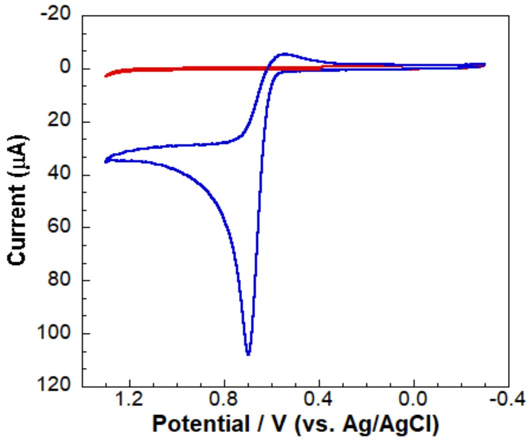


Figure 5 Shows the Cv of Gce In Both the Presence (Blue) and Absence (Red) of Acetaminophen.

The cathodic and anodic peaks are single and relatively well-defined, indicating only two electroactive species. In Figure 5, the cathodic wave depicts the reduction of acetaminophen to an anodic wave, reflecting the acetaminophen's oxidation. The solution is acidic enough to protonate the oxidized species at lower pHs, producing a relatively unstable but electrochemically active intermediate (Figure 3). As seen in Figure 5, this intermediate quickly hydrates to produce an electrochemically inactive species. Any intermediate that the oxidation forms quickly undergoes a chemical process to yield an electrochemically inactive species, as shown by the lack of a clearly defined cathodic current. The estimated values of the concentrated and dilute forms of the unknown acetaminophen tablet are 4.15 1.49 mM and 1.99 0.15 mM, based on the voltammograms obtained for the unknown (Figure 6) and calibration curve (Figure 4), respectively.

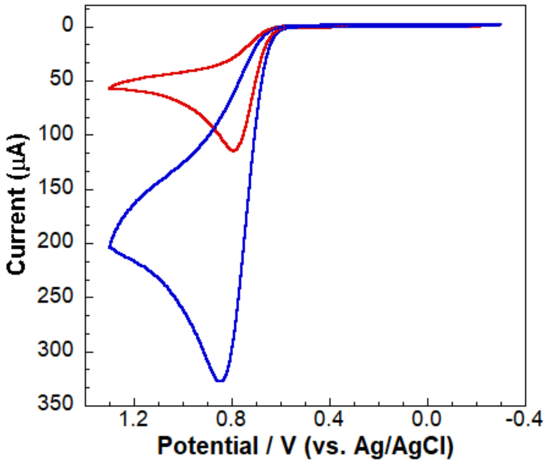


Figure 6 Shows the GCE's CV for Unknown Diluted (Red) and Concentrated (Blue) Solutions.

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