Care of Computer-Controlled Potentiostats





What is a Potentiostat?

Potentiostat

 An electronic instrument that <u>measures and controls the</u> <u>voltage difference</u> between a Working Electrode and a Reference Electrode.

 It <u>measures the current flow</u> between the Working and Counter Electrodes.



What is Potential?

Potential or Voltage (E, sometimes V):

- Unit: Volt
- The Potential is the driving force for the redox reaction.
- The potential is related to the thermodynamics of the system:

 $\Delta G = -n \mathbf{F} \Delta E$ (negative ΔG is spontaneous)

- Potential is always measured versus a Reference Electrode.
- A positive voltage is oxidative and a negative voltage is reductive.
- O Volts <u>is not nothing</u>! Redox reactions happen at 0 volts that do not happen at +1 volt.

There is no correlation between the thermodynamics of the chemical system and the kinetics (rate) of the reaction.



What is Current?

Current (i):

- Unit: Ampere
- Electron flow is the result of a redox reaction.
- Current measures the rate of the reaction (electrons per second).
- Zero current <u>is</u> nothing, i.e., if the current is zero, no redox reactions are occurring (that's not quite true in corrosion!).
- Anodic (oxidation) and cathodic (reduction) currents have different polarity (signs).
- Current may be expressed as current or current density.



An Analogy of Potential and Current as a Flowing Water Circuit





Electrodes

A Potentiostat works with three electrodes immersed in a conductive electrolyte.

- Working Electrode
 A sample of the corroding metal being tested.
- Reference Electrode
 An electrode with a constant electrochemical potential.
- Counter Electrode
 A current-carrying electrode that completes the cell circuit.



Why does a Potentiostat have three electrodes?

We would like to study the electrochemical events taking place at one specific electrode...the Working Electrode. The use of a three-electrode potentiostat with a separate Reference and Counter Electrode allows the potential at the WE and the current at the WE to be measured with little or no "interference" or "contribution" from the other electrodes.



Pay Special Attention to the Reference Electrode!

A Potentiostat needs a low impedance Reference Electrode!

- Use large junction reference electrodes
- Replace isolation frits regularly
- Avoid narrow Luggin Capillaries

Potentiostats are less forgiving of high-impedance Reference Electrodes than pH Meters!

If there's an problem with the cell, it's almost always the Reference Electrode!



The Analog Potentiostat





Three Primary Components of a Potentiostat

- Control Amplifier: Supplies the power to maintain the controlled potential between the Working and Reference Electrodes.
- Electrometer: Measures the potential difference between the Reference and Working Electrodes.
- Current-to-Voltage Converter: Measures the current between the Working and Counter Electrodes.



A Potentiostat is a feedback/servo device...measure the voltage (electrometer), apply power from the Control Amp...repeat until the experiment is finished.

- What happens if the feedback is too slow in our Potentiostat...uhh, shower?
- Skin = Electrometer
- Hand = Control Amp
- Water is too hot
 - Turn the knob to COLD
 - 2 seconds later, you're freezing !
 - Turn the water to HOT
 - 2 seconds later, you're scalded !
 - Turn the knob to COLD
 - Repeat
- Then an electrochemical reaction occurs by flushing the toilet!





- Do you need to know how a Potentiostat works?
- No.
- Do you need to be able to recognize when something is wrong?
- Yes!
- Why would something go wrong?
- Because the performance of the Potentiostat is affected by the electrical characteristics of the sample...or something in the cell is causing a problem...or the Potentiostat is busted!



To Evaluate Your Electrochemical Data... Look At It!

- Electrochemical data is <u>always</u> a collection of individual data points...one followed smoothly by another.
- Noisy data is bad.
- Flat-lined data is bad.
- Overloads are bad.
- It is <u>very</u> rare to collect bad data that looks good.



















My Data Is Bad. Now What Do I Do?

- 1. Calibrate the Potentiostat.
- If calibration is successful, check the Potentiostat by running a dummy cell (a network of resistors/capacitors that give a known result).
- 3. If the instrument is OK, then check the cell. Check the Reference Electrode first!
- 4. If the cell is OK, then it's something in your sample chemistry. Do you need a Faraday Cage?
- 5. At some point, you should contact your Potentiostat supplier for technical support.



The One Equation You Need to Know

- Ohm's Law
- E = iR
- If I apply 100 mV to a 1000 ohm resistor, I should measure a current of...
- 100 µA



Pstat Check-Out: PolRes on a 2000 Ohm Resistor





Pstat Check-Out: EIS on a Dummy Cell





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