Potentiostat Design and Engineering

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Admiral Instruments

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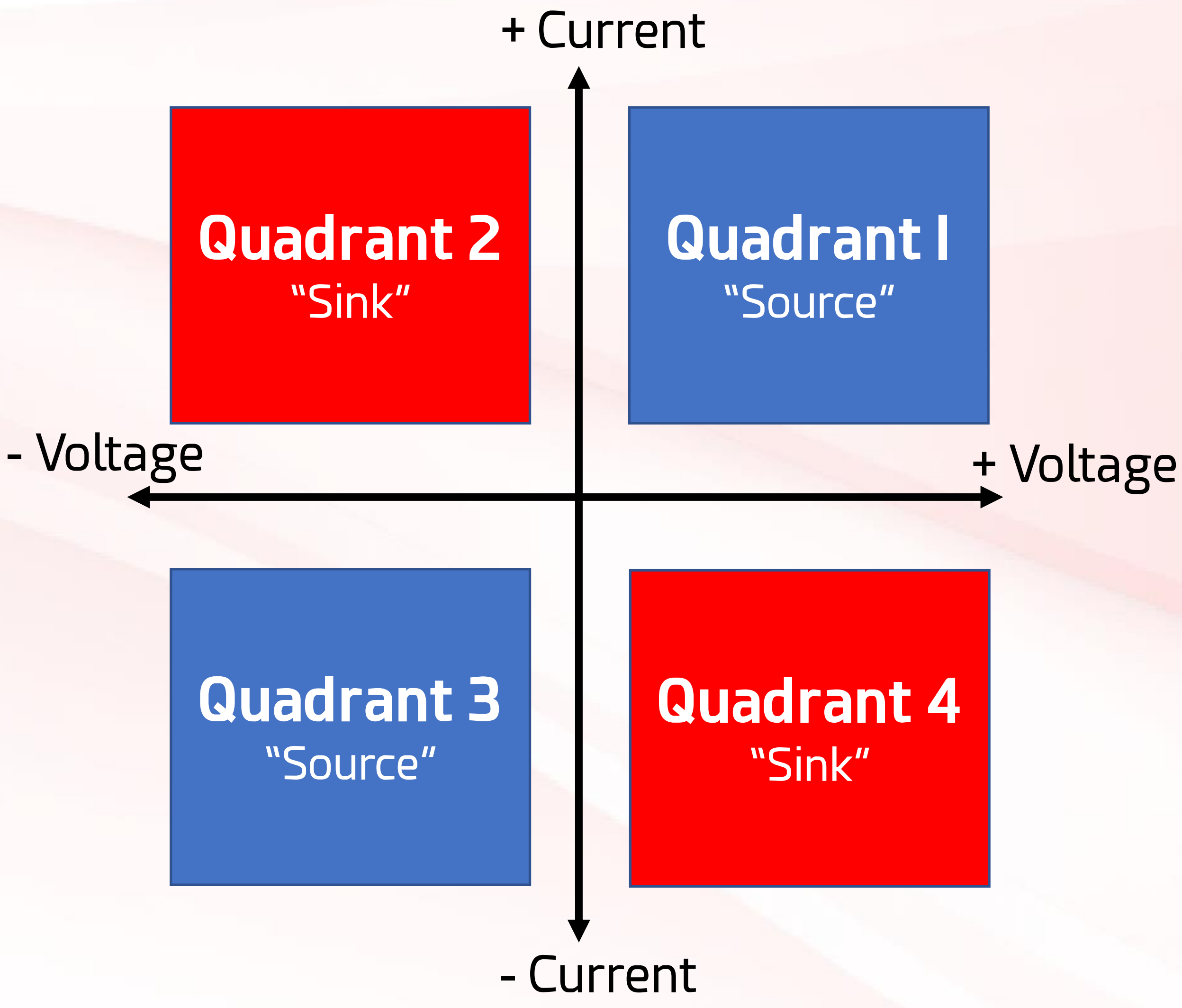
Topics To Be Discussed

- Potentiostat Functions
- Operational Amplifiers
- Potentiostat Stability
- Current and Voltage Ranges
- Ground/Float Mode

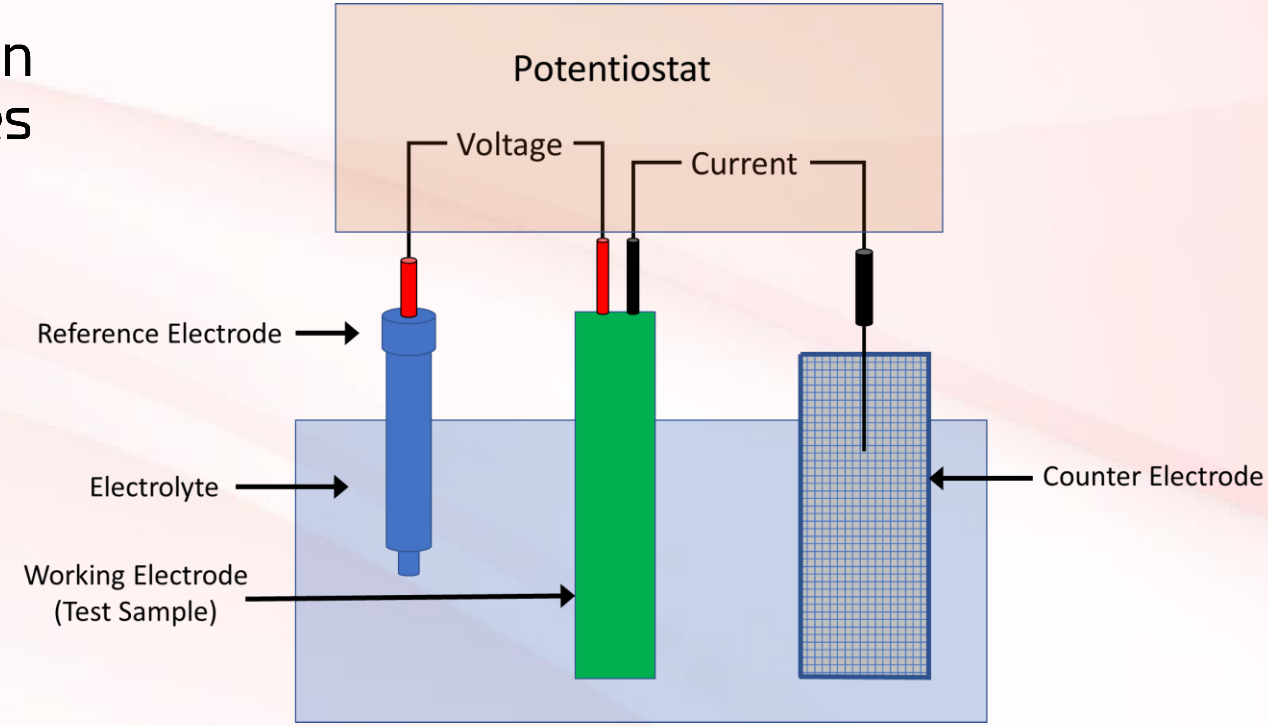
Different Types of Power Instruments

- Power instruments are designed to source and/or sink power from the device under test
- Conventional power supply operate in Quadrants 1 and/or 3
- Conventional Loads operate in Quadrants 2 and/or 4
- Potentiostats and Source Measurement Units operate in all four quadrants

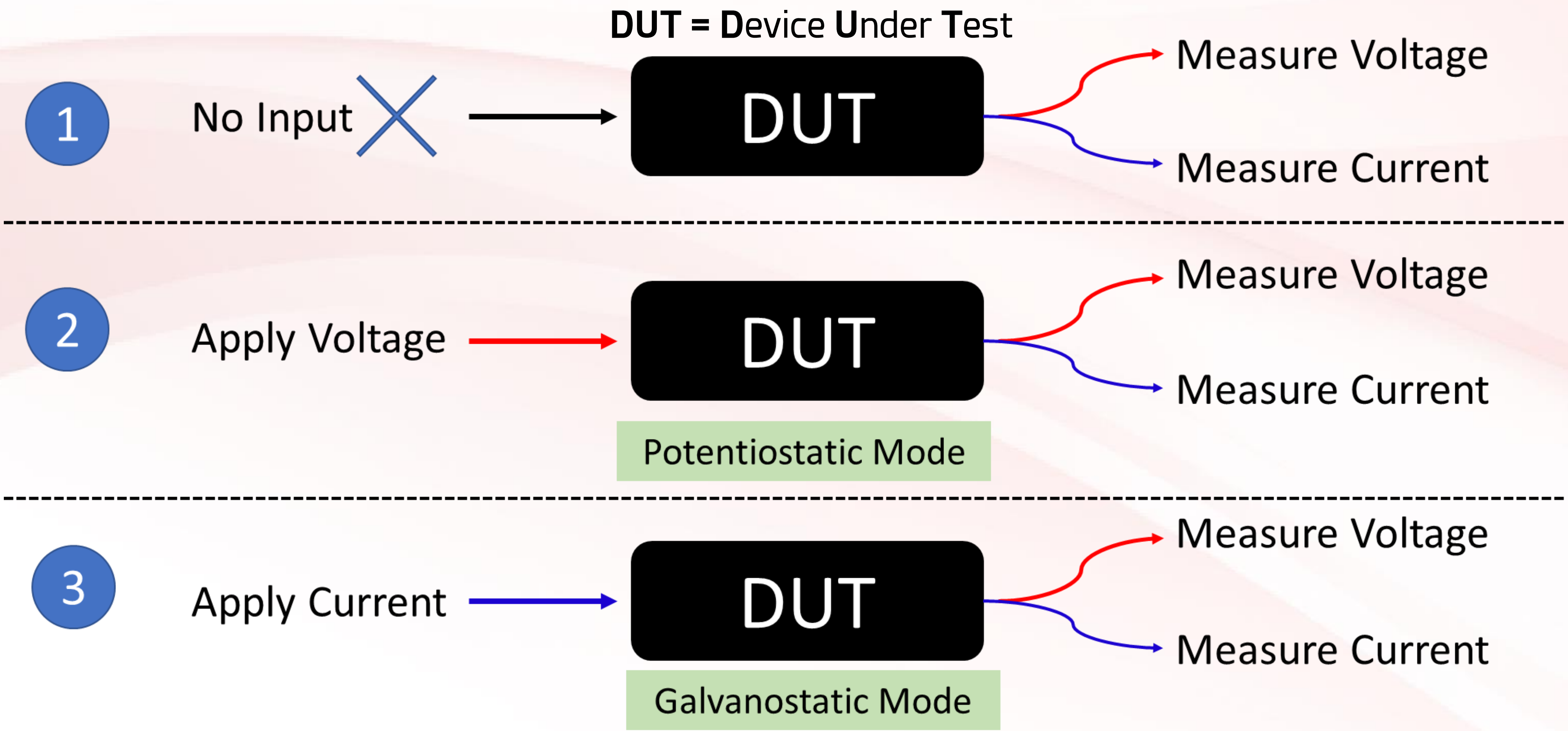


What is a Potentiostat?

- Runs 3-electrode potentiostatic experiments. Modern potentiostats run in both potentiostat and galvanostat modes
- A set voltage is applied on the Working Electrode (WE) with respect to the Reference Electrode (RE), while current flows between WE and RE
- Current does <u>not</u> flow through the RE
- Counter electrode can be at a different voltage than RE

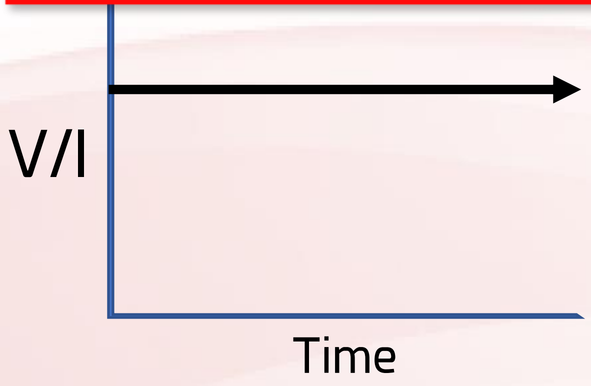


Basic Functions of a Potentiostat

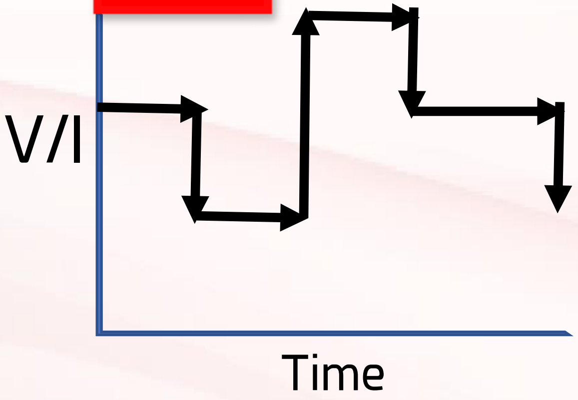


Varieties of Voltage and Current Signals

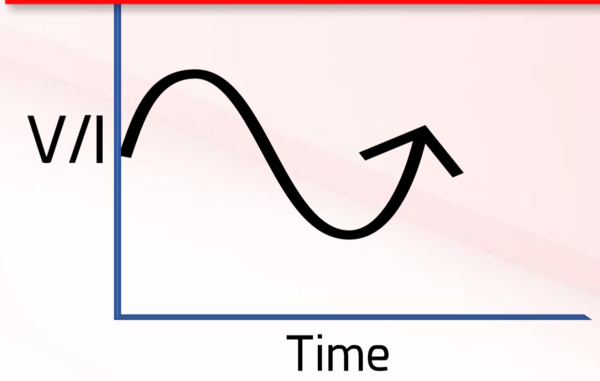
Constant Potential/Current



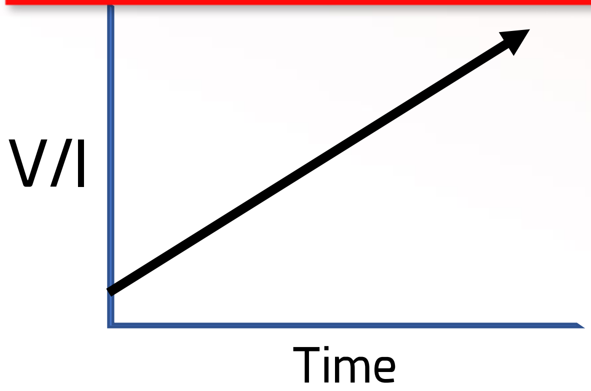
Pulse



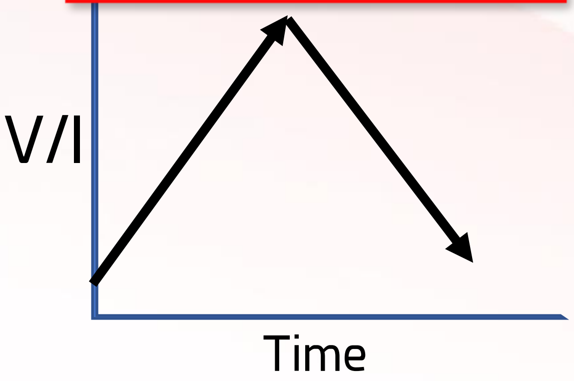
Electrochemical Impedance Spectroscopy



Linear Sweep Voltammetry

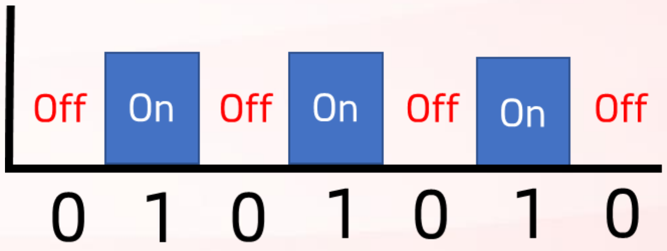


Cyclic Voltammetry

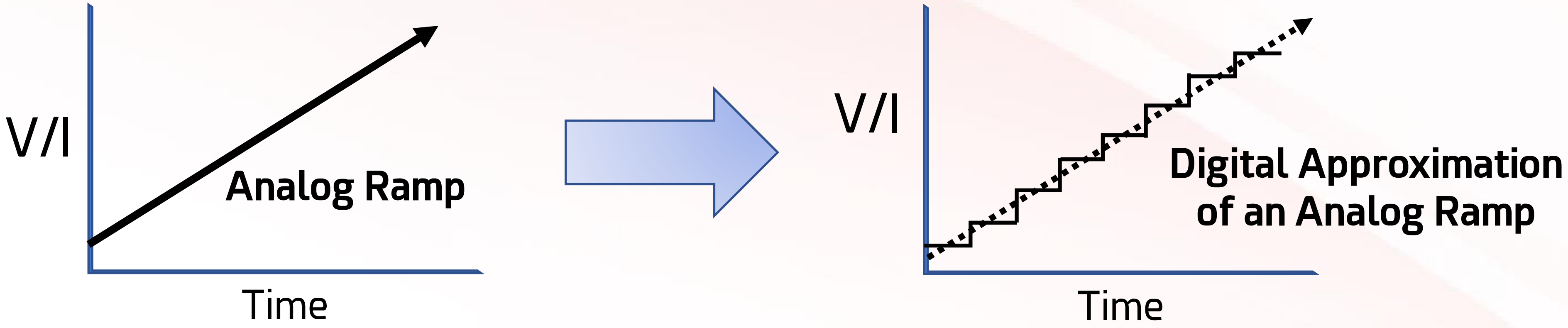


Digital Nature of Potentiostats

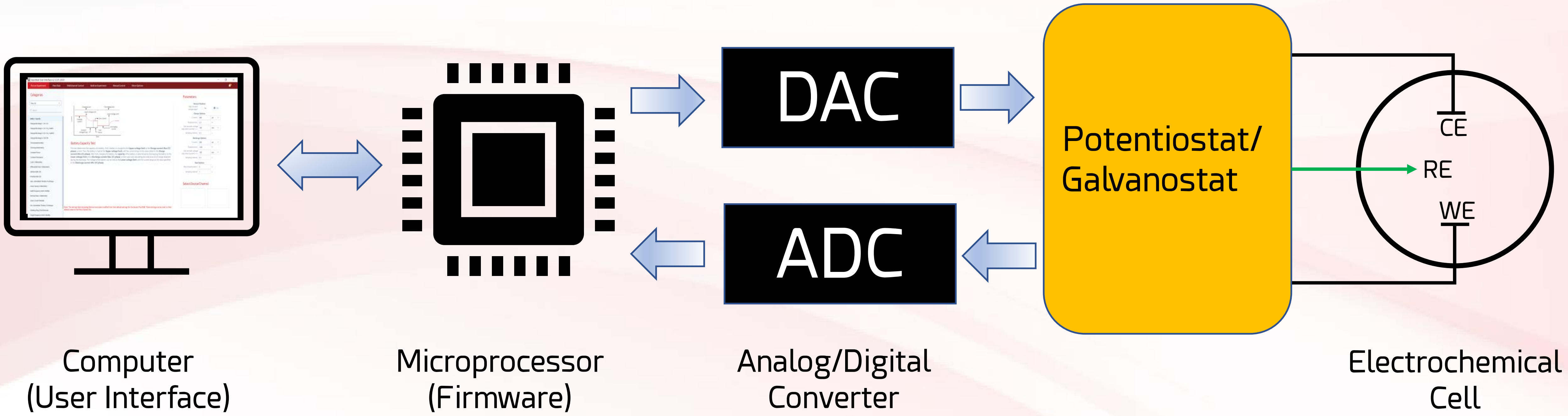
- Signals are sent and received as 0's and 1's



- A step signal is the basic building block of all applied and received signal from the device under test



Basic Potentiostat Architecture



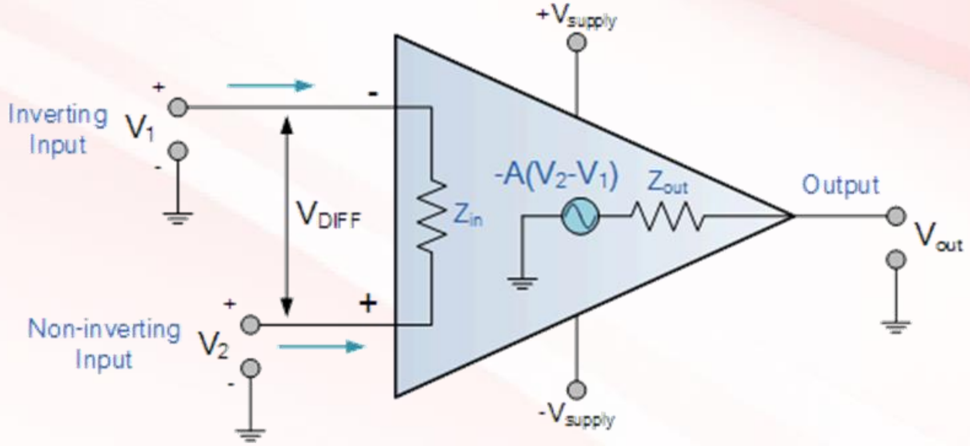
DAC = Digital to Analog Converter
ADC = Analog to Digital Converter

Signal Flow Pathway

Ideal Operational Amplifier (Op-amps)

- Operational Amplifier (op-amps)
  - Building blocks of the potentiostat
  - "Operational" - originally used to do math operations
  - "Amplifier" - amplifies the input signal [$V_{out} = A \times (V_2 - V_1)$]

- Characteristics of an ideal op-amp
  - Open-loop gain (A_{OL}) = infinity
  - Z_{in} = infinity
  - Z_{out} = 0
  - **Response time = zero, infinite bandwidth**
  - Offset = zero ($V_{out} = 0$ if $V_2 - V_1 = 0$)

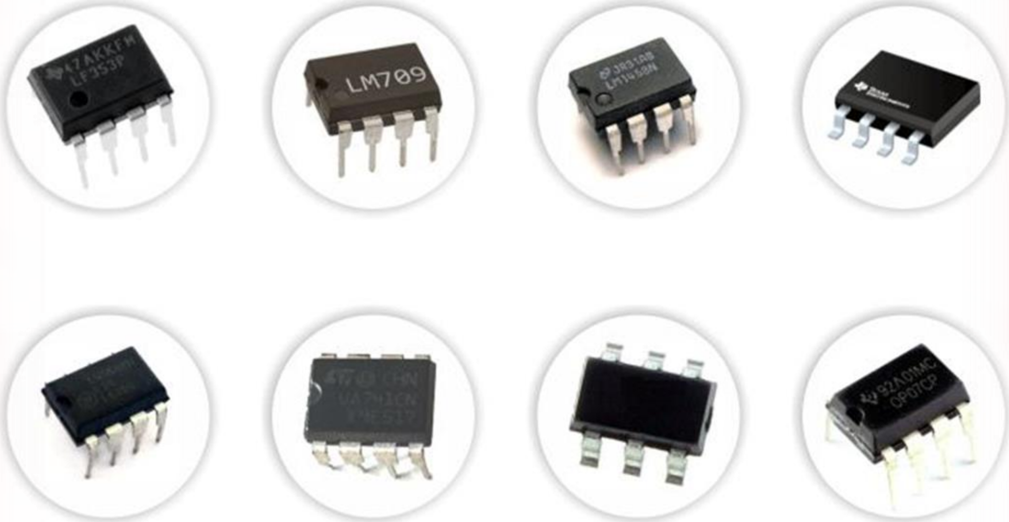


Operational Amplifier Classifications

- Types based on Current/Voltage input-output
  - Voltage in-Voltage out
  - Current in-Current out
  - Voltage in-Current out (Transconductance)
  - Current in-Voltage out (Transresistance)

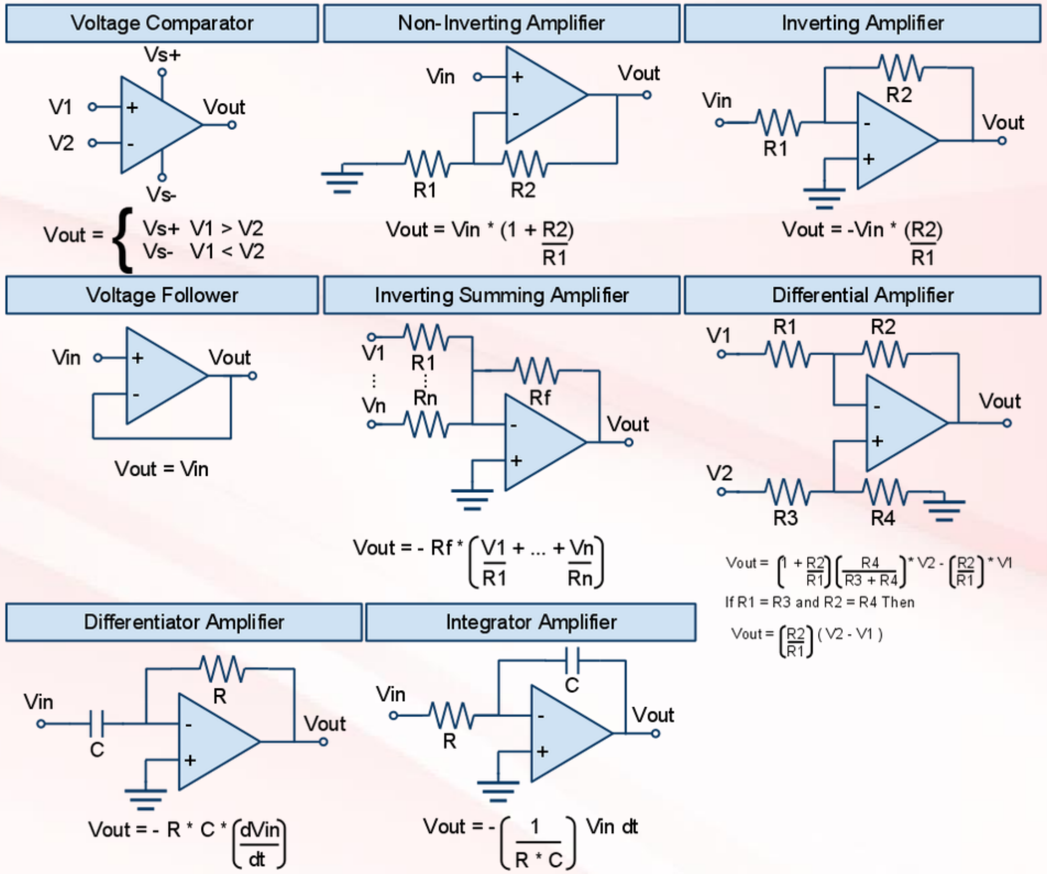
- Types based on specifications
  - Universal
  - High resistance
  - Low temperature drift
  - High speed
  - Low power
  - High Voltage High Current
  - Programmable

- Other types
  - Differential
  - Instrumentation



Some Basic Types of Op-Amps Circuits

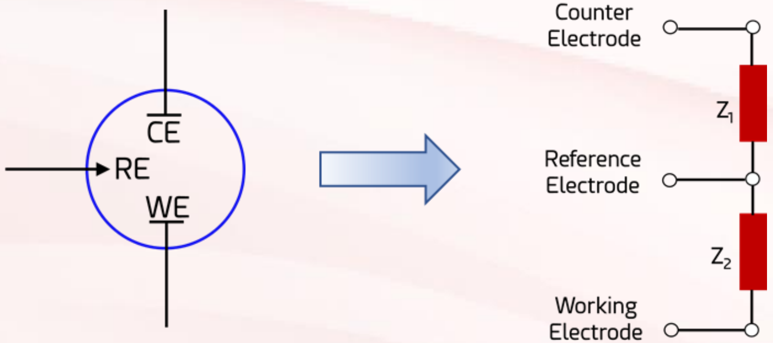
- The configuration of op-amps are limitless (logic, mathematic, filter, etc)
- Negative-feedback loop (the output is connected to the inverting input) is the most common configuration
- In fact, an op-amp is rarely used in open-loop (no feedback loop) configuration



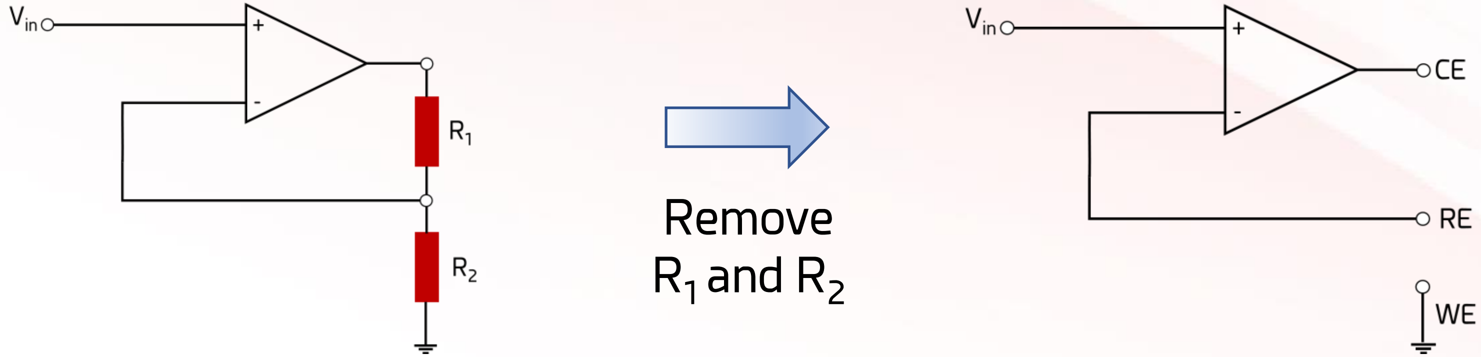
Basic Operational Amplifier Configurations

Making Potentiostats Using Op-Amps

- An electrochemical cell can be simulated with resistors and capacitors



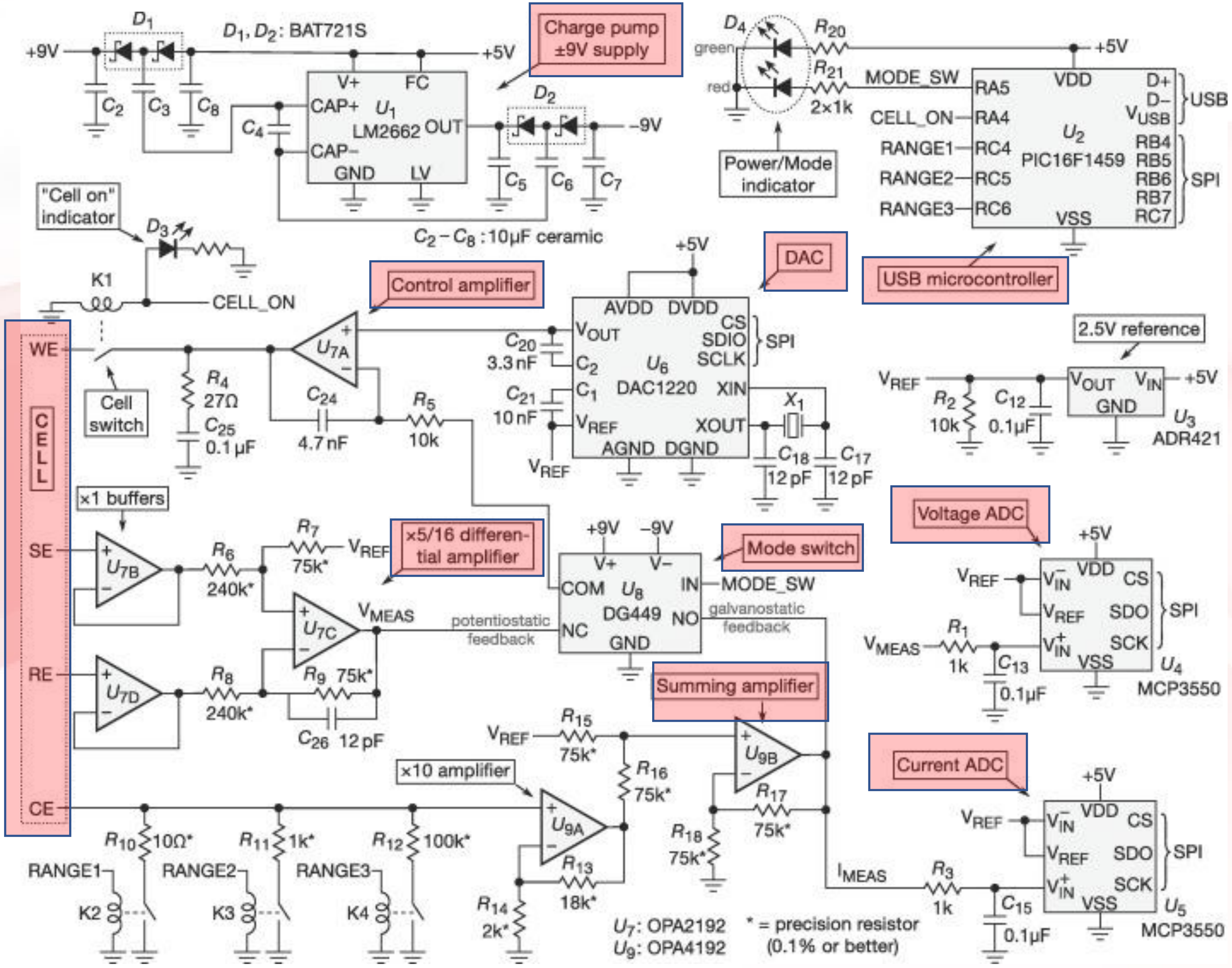
- A simple potentiostat can be created using one of the basic op-amp circuits

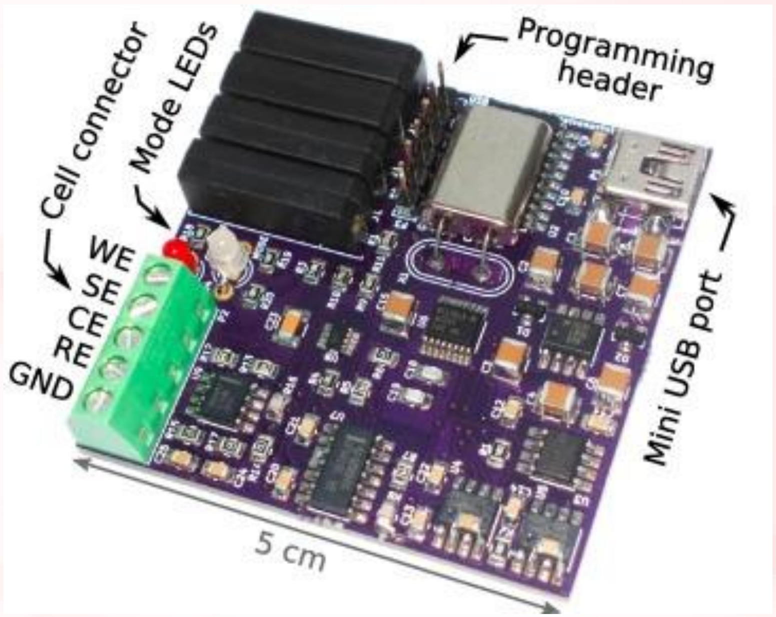


Non-inverting Amplifier

A Simple Potentiostat

A Real Potentiostat is Complex!

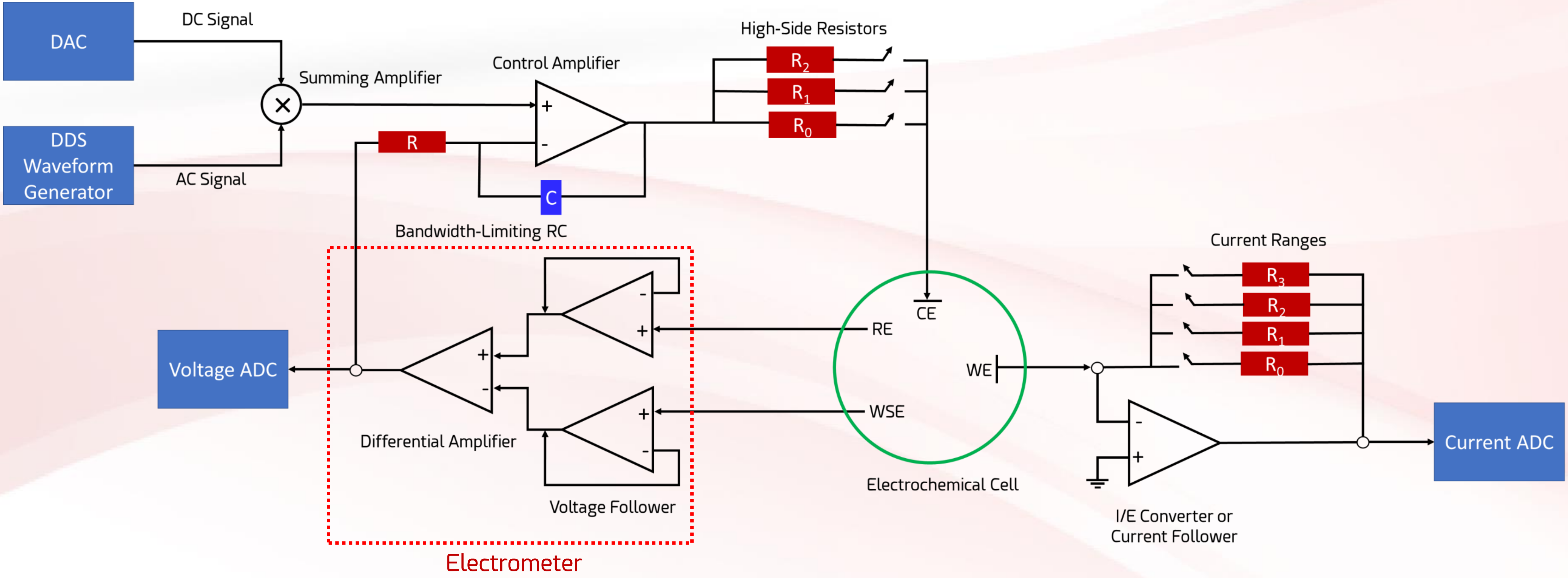




"A USB-controlled potentiostat/galvanostat for thin-film battery characterization"

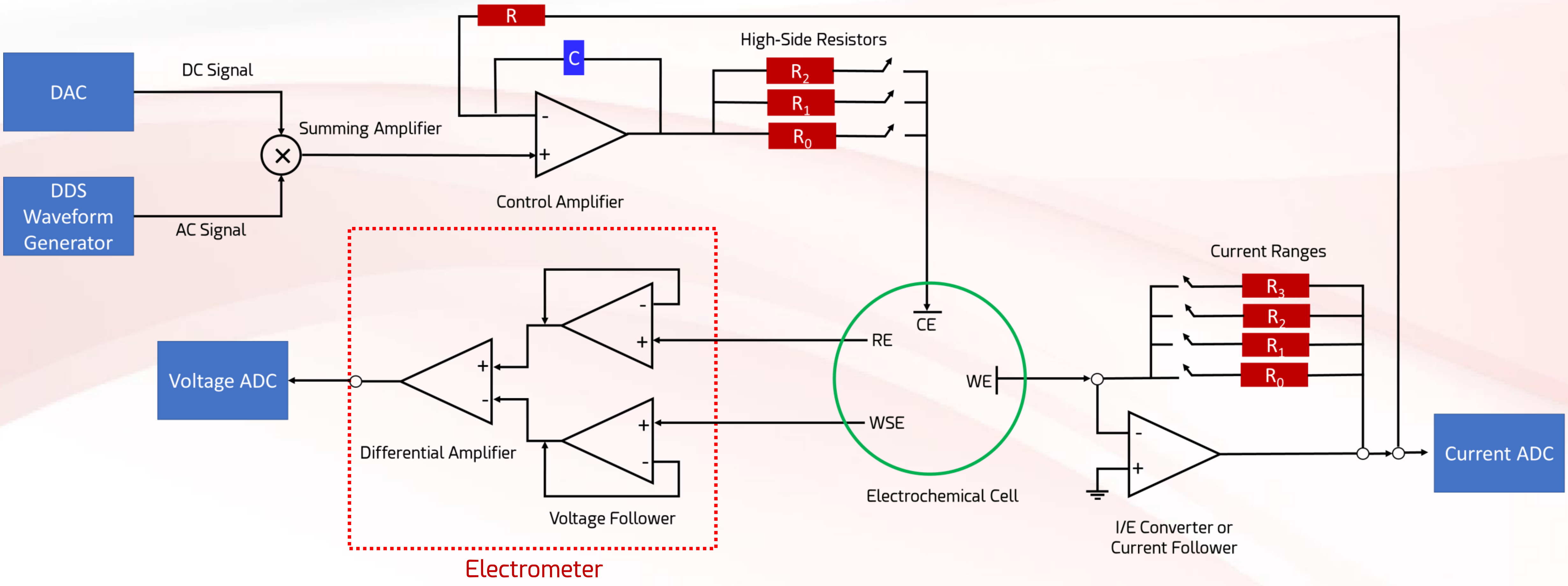
Dobbelaere, et al. Hardware X 2 (2017) 34-49

Simplified Potentiostat Design



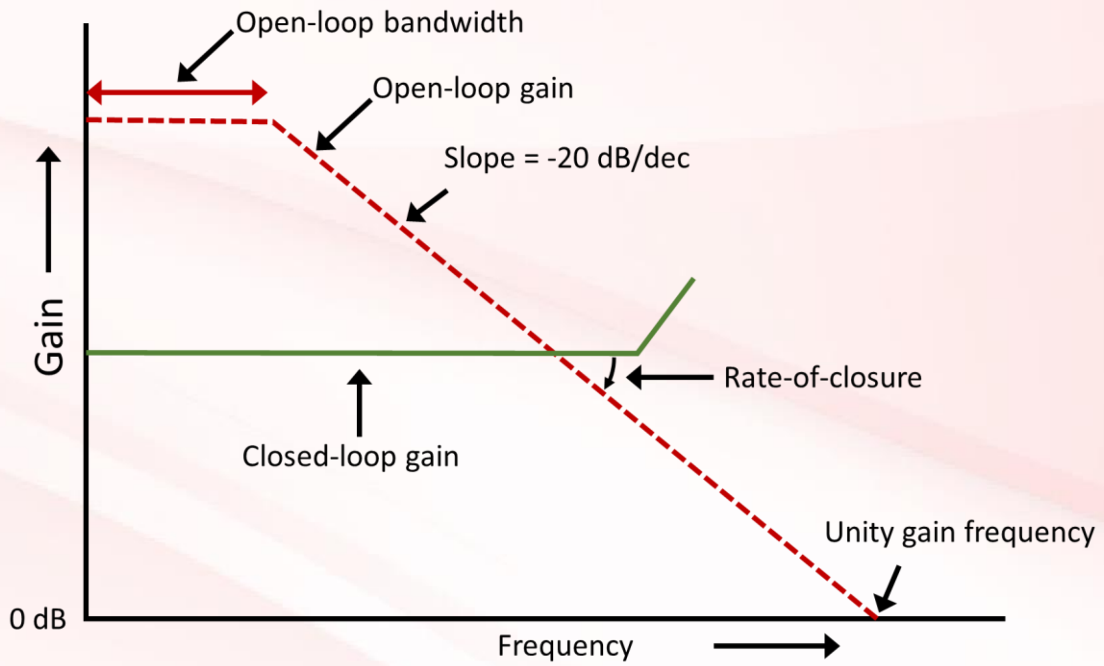
DAC = Digital to Analog Converter
ADC = Analog to Digital Converter
DDS = Direct Digital Synthesizer

Simplified Galvanostat Design



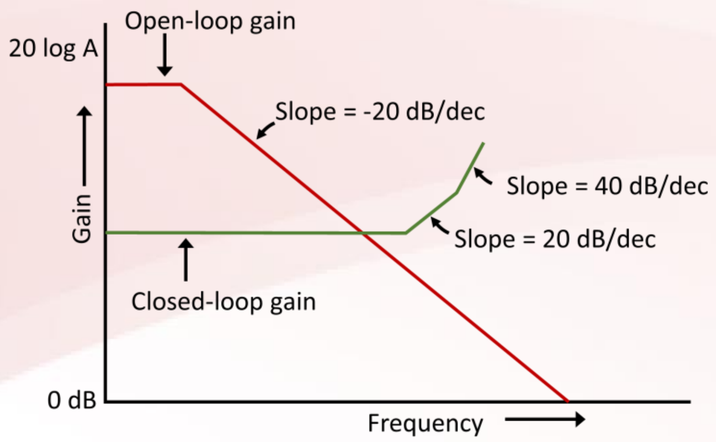
Open-Loop Gain vs Closed-Loop Gain

- The gain of real op-amp is frequency dependent
- Closed-loop amplifier circuit cannot supply more gain at higher frequencies than available
  - Closed-loop is when a feedback loop exists
- The angle between closed-loop gain and open-loop gain is called the rate-of-closure, which determines stability of the circuit

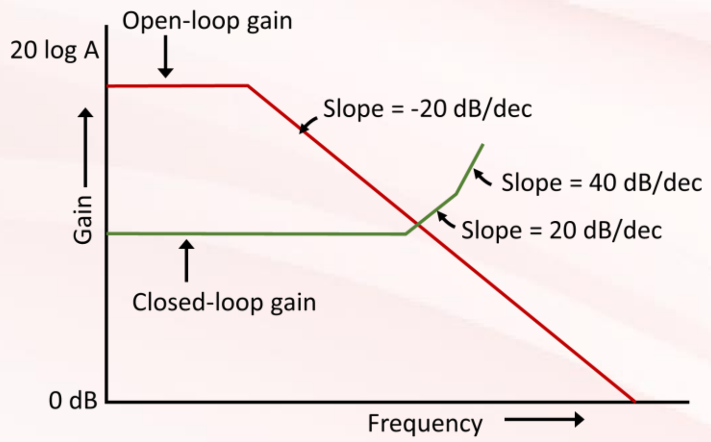


Stability Criteria

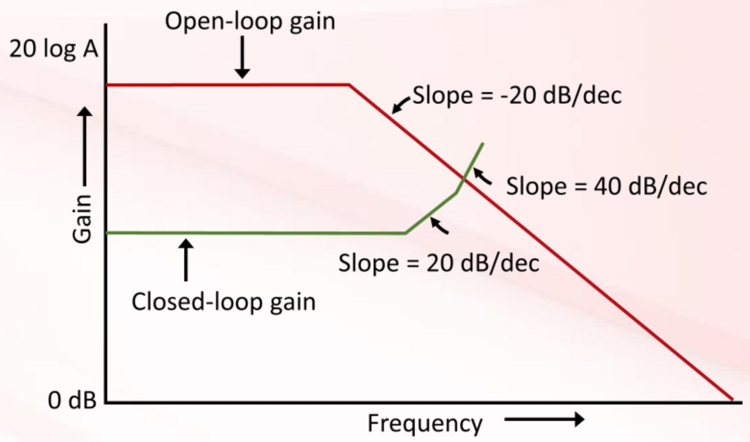
- The rate-of-closure determines if the circuit will be stable or not



Condition 1: Optimal stability
Rate-of-Closure < 40 DB



Condition 2: Marginal stability
Rate-of-Closure = 40 DB

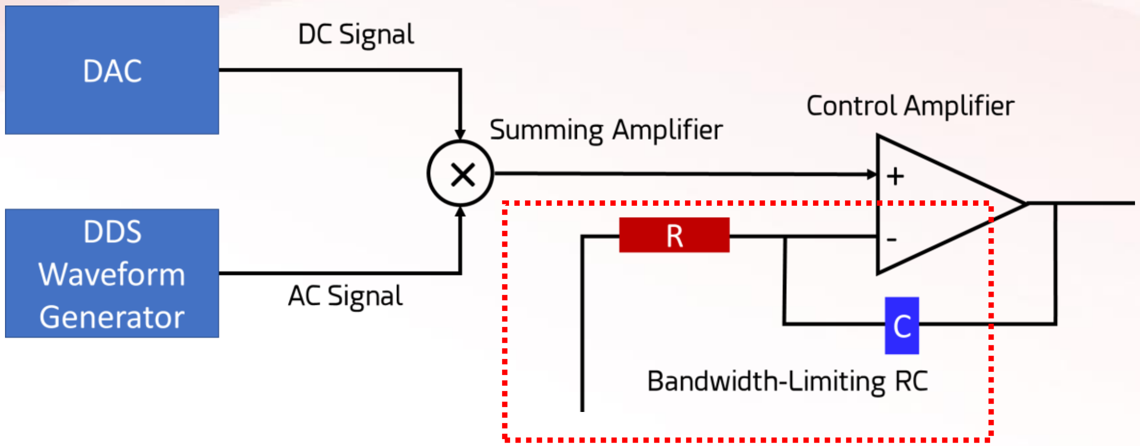


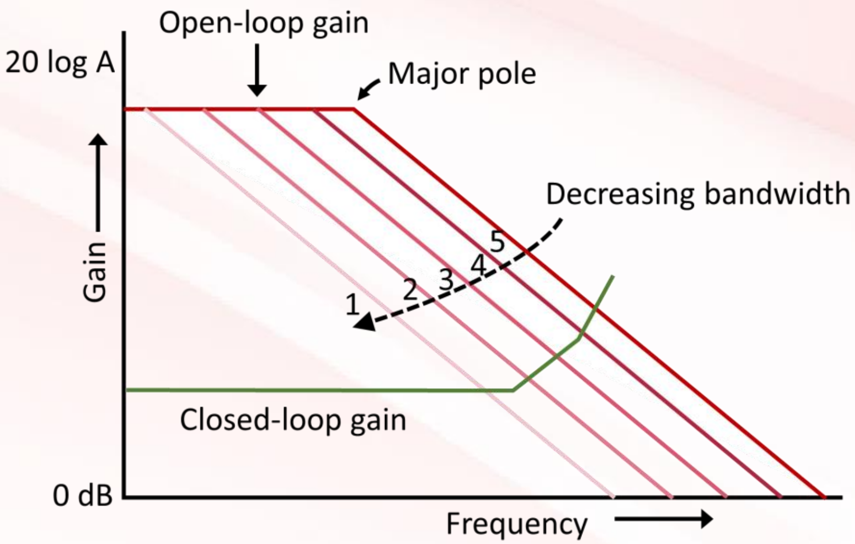
Condition 3: Instability
Rate-of-Closure > 40 DB

- Rate-of-closure can be controlled by modulating the bandwidth of the control amplifier

Potentiostat Stability: *Bandwidth Index*

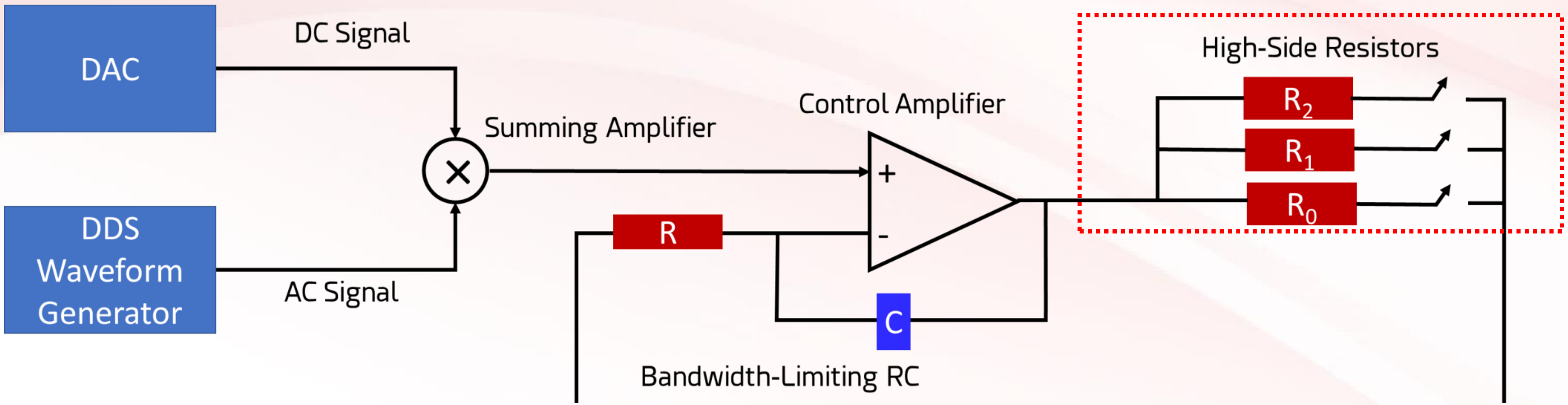
- Potentiostat stability can be controlled by varying bandwidth-limiting RC
- This is labeled as ***Bandwidth Index*** in the Squidstat User Interface software





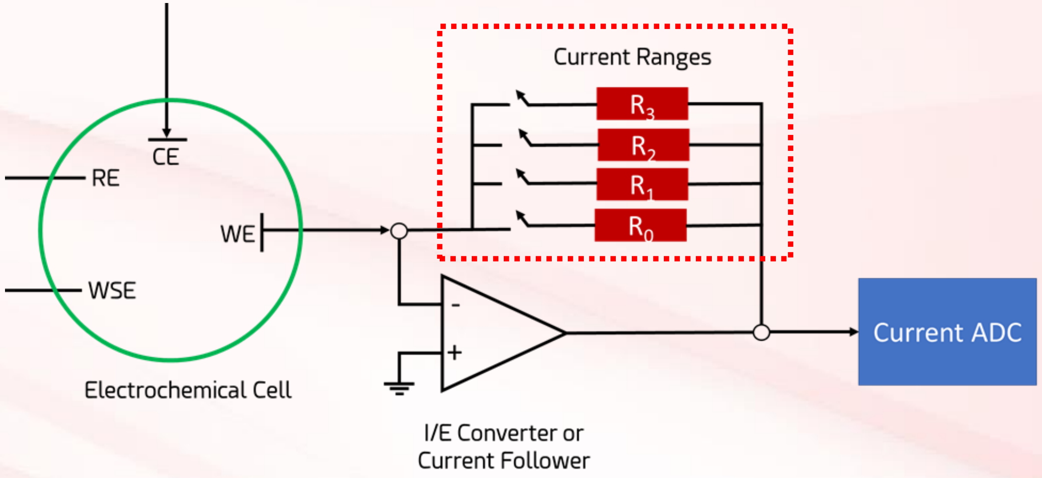
Potentiostat Stability: *Stability Factor*

- The high-side resistors also control the bandwidth of the control amplifier, but do so in a less predictable way than the bandwidth-limiting RC



Current Measurement

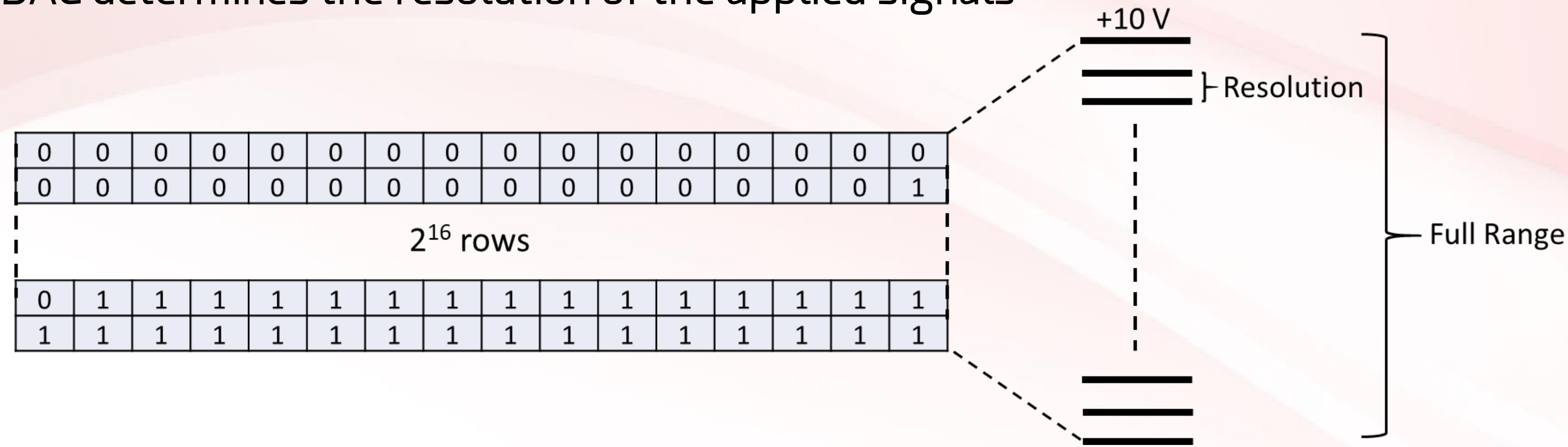
- In most potentiostats, current is measured by measuring the voltage drop across a resistor
  - $V_{out} = IR_x$, where x = 0,1,2…N
  - N is the number of ranges
- If the max voltage drop across each resistor is 1 V…
  - For R = 10 Ohm, Range = 0.1 A or 100 mA
  - For R = 100 Ohm, Range = 0.01 A or 10 mA



- Resistor precision determines the current measurement accuracy
- Current ranging
  - Auto-range: the software/firmware selects the best resistor for accuracy
  - Fixed-range: the user selects the resistor

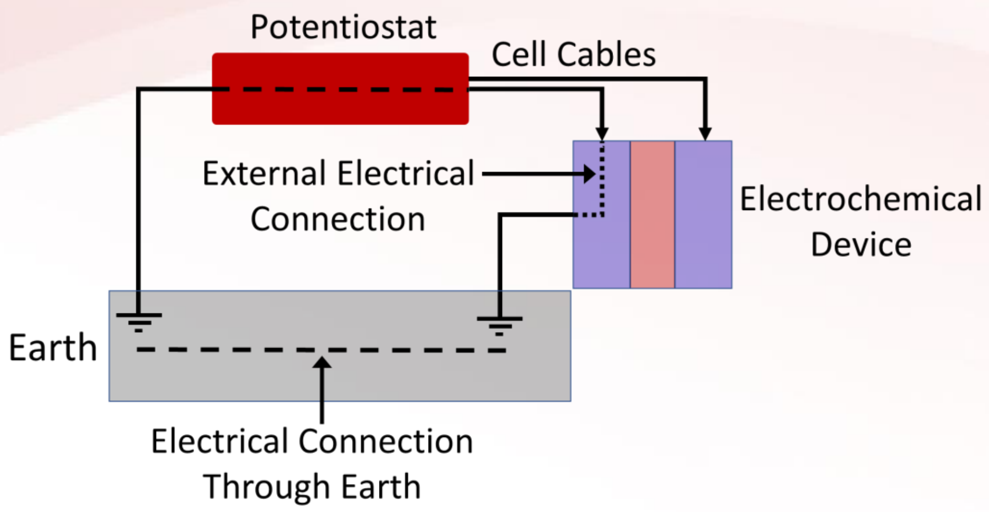
Voltage and Current Ranges

- ADC determines the resolution of the measured signal
  - 16-bit ADC means there are 2^{16} binary numbers to represent a voltage or current range
- DAC determines the resolution of the applied signals

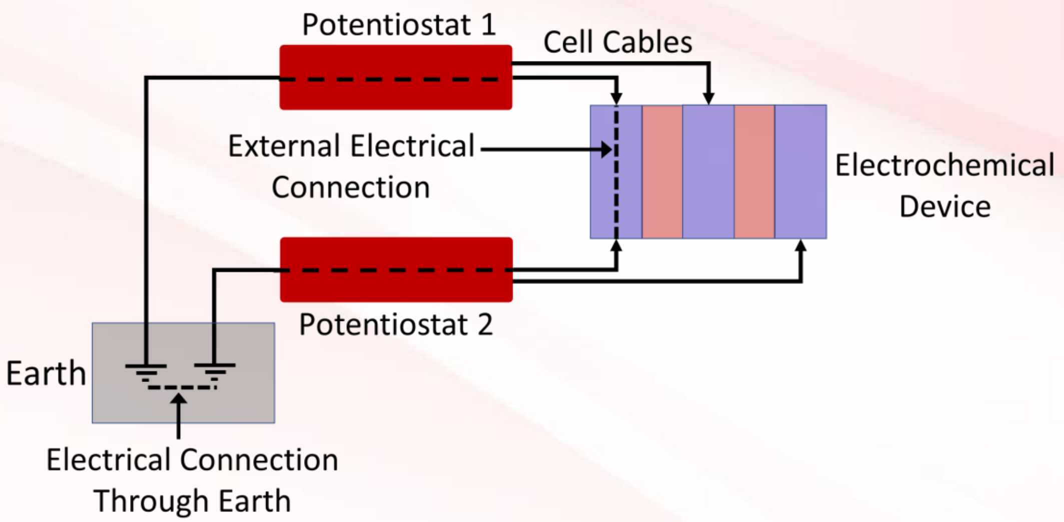


Ground Loops

- "Ground" acts as a voltage reference point and current sink
- A ground loop occurs when two or more instrument are connected via "ground"
- A ground loop introduces noise and instability



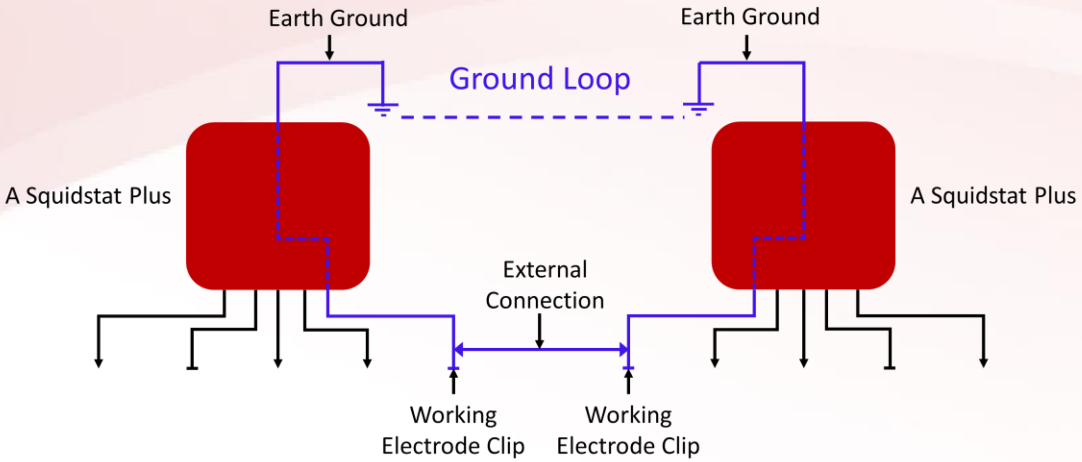
Ground Loop Through a Grounded Electrochemical Device



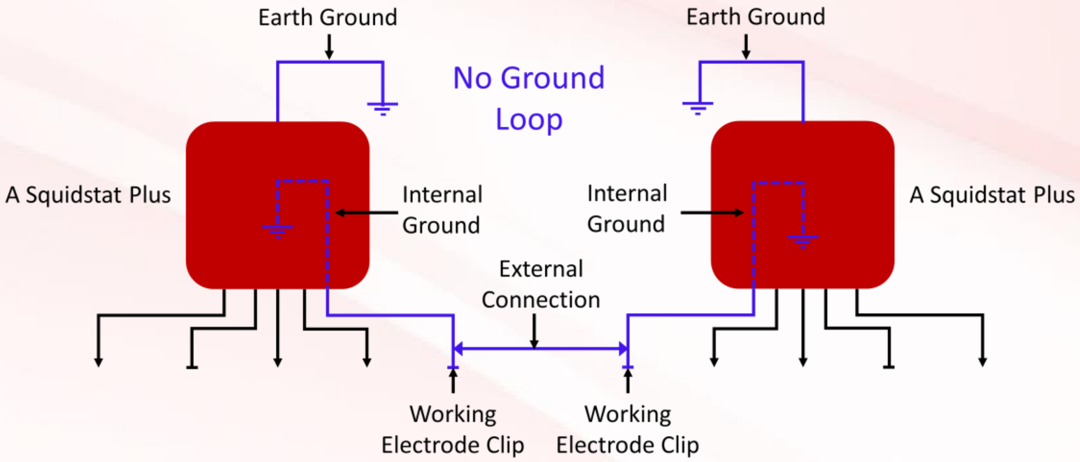
Two Potentiostats In Ground Loop Through Common Connection

Floating Mode

- When an instrument is disconnected from “Ground”, it is in a floating mode



Ground Mode



Float Mode

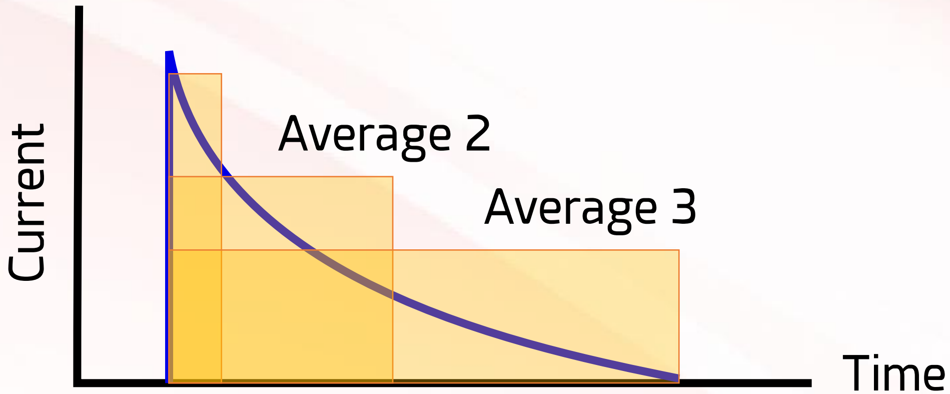
Some Final Points to Consider

- Noise management
  - Thermal noise
  - Atmospheric noise
  - Shot noise
  - Flicker noise
  - Burst noise
  - Transit-time noise
  - Filter
- Issues caused by digital approximation of analog signals
- Data sampling

<u>Thermal Noise</u>

$$E = (4\ R\ k\ T\ \Delta F)$$

E is RMS noise in voltage
R is resistance
k is Boltzmann's constant
T is temperature
ΔF is frequency range



Capacitive response to a voltage step

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