

Operating the potentiostat:

The AFRDE 5 Bi-potentiostat is a general multipurpose electrochemical instrument that can be used to generate a constant current (galvanostat), a constant potential (potentiostat) or a linear or cyclic voltage sweep. It can function as a traditional potentiostat that uses three electrodes (working, reference and counter) and also independently control an additional working electrode. The ferricyanide experiment requires that a linear potential sweep be impressed on the working electrode. The potential limits of the sweep are from 0.5 V to -0.1 V at a scan rate of 20 mV/sec. The following procedure describes the steps needed to achieve this goal.

1. Connect the potentiostat to the three electrodes: connect the **K1** lead to the rotator, **CE** to the platinum counter and **Ref** lead to the reference.
2. Switch on the potentiostat. You will notice that the **Electrode** LED (above the power button) is in the **dummy** position. The **dummy** position is an internal resistor of resistance $1\text{K}\Omega$. All operations in this mode are across this resistor and not across the external circuit. You will also notice that the LED marked **zero** is on and so is the LED marked **hold**. **Zero** forces the potential impressed to be zero volts and **hold** overrides all sweep commands and holds the potential at a constant value (in this case zero volts).
3. Set the **upper limit** potential to +0.5 V and the **lower limit** to -0.1 V. Note that the potential is in mV's.
4. Set the **sweep rate** to 20 mV/sec. There are two range settings in the sweep generator (i) 0 to 99.99 mV and (ii) 0 to 999.9 mV. Choose the 0-99.99 range and set the scan rate at 20.00 mV/sec.
5. Set the **off-set voltage** at zero volts and make sure that the **mode** is on **pot** (i.e. potentiostat).
6. The AFRDE displays the current generated in terms of a potential (in V). To find the current from this potential we have to multiply it by the **current converter per volt** setting. For instance, if the setting is in 1 mA per volt and the current display is 0.5 V, then the current is $0.5\text{ V} \times 1\text{mA/volt} = 0.5\text{ mA's}$. Note that if your experiment generates current in the order of μA and the current range is in 200 mA/volt, then the voltage displayed will be zero volts as the display has only 2 decimal places. For this experiment, 1mA/volt seems like an ideal setting for the currents generated.
7. Switch the **sweep voltage** on.
8. As the starting potential of the experiment is 0.5 V and the potentiostat is at present at 0 V, we have to sweep the potential up to 0.5 V before starting the experiment. This is done in dummy mode. Press the **stop on** button for the **upper potential limit**. This makes the potential stop when it reaches this point (0.5 V). Remove the **zero** button. Make sure that the **sweep direction** is up (meaning from 0 V to 0.5 V). Press the **hold** button to sweep the potential up to 0.5 V in dummy mode. The potential will stop at 0.5 V.
9. Press the **hold** button again to hold the potential at 0.5 V. Remove the **stop at limit** from the upper limit and turn it on for the lower limit. This ensures that the potential stops at the lower limit. Make sure that the **sweep direction** is down. The potentiostat is now set to sweep the potential from 0.5 V to -0.1 V.
10. Press the **dummy** button to turn it to the 'normal' mode. Now the potentiostat is impressing 0.5 V with respect to the reference to the RDE.

11. Press the **hold** button to turn the sweep on at any time to run the experiment.
12. Once the experiment is done, switch the potentiostat to the dummy mode and sweep the potential up to 0.5 V again. You can then repeat the experiment for a different rotation speed.