Cyclic Voltammetry Basics: Reduction of Ferricyanide

This experiment is a great introduction to understanding cyclic voltammetry and how it can be used to study redox systems. Cyclic voltammetry is an essential technique for a chemist, and to use cyclic voltammetry effectively, students need to know how to assess performance and change the operational settings accordingly. In this experiment, students observe how changes in the instrument's settings alter the voltammogram and explore fundamental aspects of a voltammogram. This particular experiment gives students a qualitative overview of collecting proper voltammograms. For a more quantitative review, see Experiment 4, "Using RuHex in Quantitative Cyclic Voltammetry."

ESTIMATED TIME

We estimate that this experiment can be completed in one hour.

TIPS

1. In the Electronic Resources you will find PDF and word-processing files of the student experiment. You can print the PDF, distribute it to students electronically, or post the file to a password-protected class web page or learning management system. Edit the word-processing file if you would like to tailor the experiment to suit your equipment and students. Sign in to your account at **www.vernier.com/account** to access the Electronic Resources.

2. It is prudent to try this experiment with the solutions you have prepared prior to giving the solution to your students to ensure proper concentrations and experimental parameter setting.

3. If students get a signal greater than 2 μA for their 0.1 mM potassium nitrate solution, replace the SPE with a new one.

4. Here are some generic troubleshooting tips if the voltammogram has additional peaks or if signal saturation occurs:
   - While testing your solution before giving it to the students, make sure the recommended parameters in the student instructions are still appropriate. You may want to modify the Switching Potentials.
   - A fouled electrode cannot accurately measure the ferricyanide solution because the effective electrode area decreases. If you or your students start to see irregular peaks, it is best to switch out the SPE.
   - If the voltammogram stops plotting in the middle of a run, it is likely that your sample is too concentrated, dilute your sample and rerun.
   - If the voltammogram is too noisy, consider changing the current setting in the parameters.

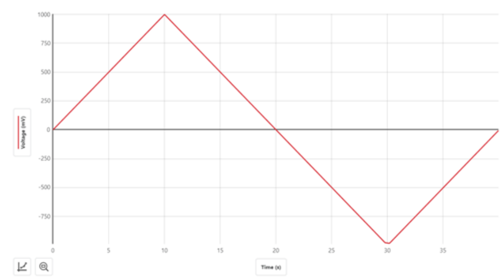
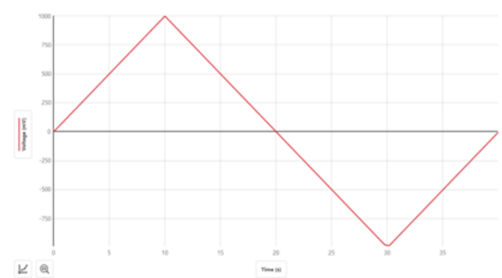
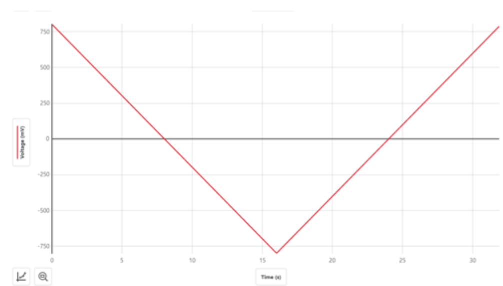
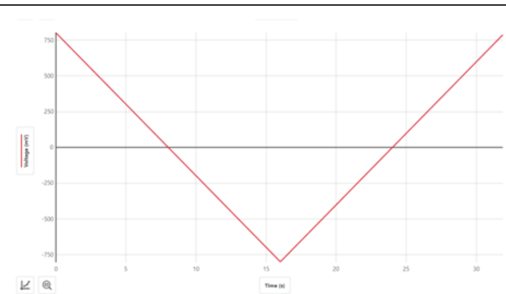
HAZARD ALERTS

The chemical safety signal words used in this experiment (**DANGER** and **WARNING**) are part of the Globally Harmonized System of Classification and labeling of Chemicals (GHS). Refer to the Safety Data Sheet (SDS) that came with the chemical for proper handling, storage, and disposal information. SDS can also be found online from the manufacturer.

Potassium ferricyanide, $C_6N_6FeK_3$: This chemical is considered non hazardous according to GHS classifications. Treat all laboratory chemicals with caution. Prudent laboratory practices should be observed.

Potassium nitrate, 1.0 M, KNO_3: This chemical is considered nonhazardous according to GHS classifications. Treat all laboratory chemicals with caution. Prudent laboratory practices should be observed.

DATA TABLE

Parameter modification	Observation	Sketch of voltage *vs.* time graph
Default parameters	Very noisy trace; it extends too far in the anodic direction and the cathodic direction.	
Current change	The trace line appears less noisy, but it still extends too far in the anodic direction and the cathodic direction.	
Segment and voltage change	The anodic and cathodic ends were appropriately captured. This change made the voltammogram appear much more duck-like.	
Scan rate change	The signal got smaller with decreased scan rate.	

SAMPLE VOLTAMMOGRAMS

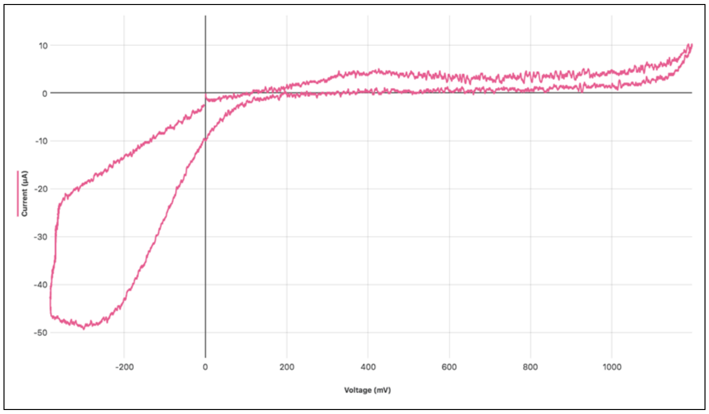


Figure 1 CV of 2.5 mM ferricyanide solution, default parameters

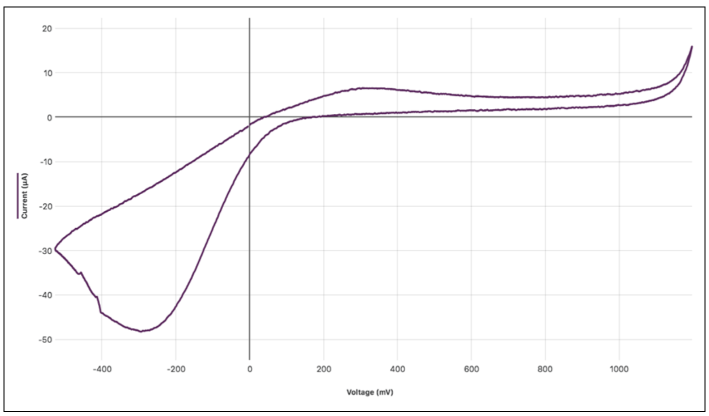


Figure 2 CV of 2.5 mM ferricyanide solution, current optimized

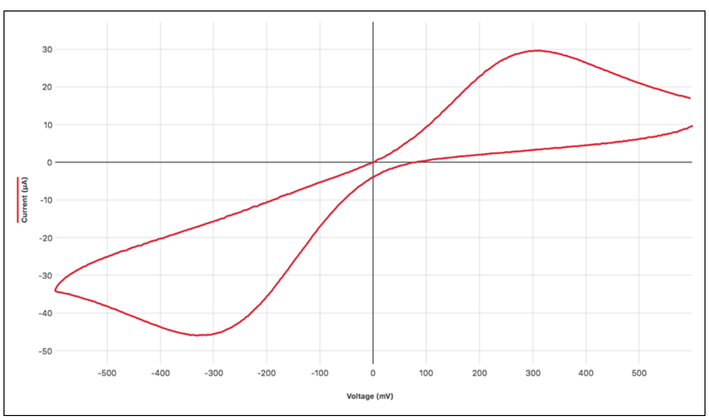


Figure 3 CV of 2.5 mM ferricyanide solution, segment and potential optimized

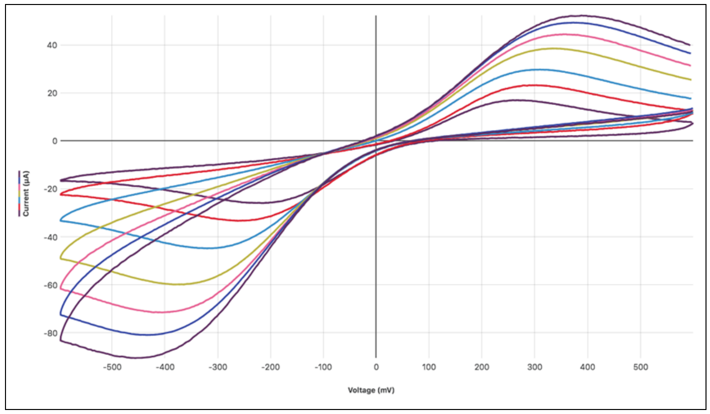


Figure 4 CV of 2.5 mM ferricyanide solution at various scan rates

ANSWERS TO ANALYSIS QUESTIONS

1. Results will vary. The sample data were collected with the following parameters:

Number of segments	2
Initial potential (mV)	600
Switching potential 1 (mV)	–600
Final potential (mV)	600
Sweep rate (mV/s)	25
Current range	Medium (±100 µA)

2.

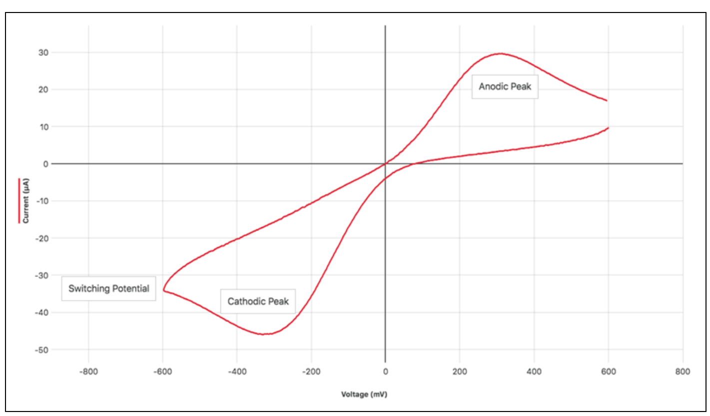


Figure 5