Advanced Chemistry with Vernier

4th Edition

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CHEM-A

Vernier

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Equipment Used in Experiments

X = Required
O = Optional

		Colorimeter or Spectrometer	Conductivity	Current Probe or Constant Current System	Drop Counter	Gas Pressure	ORP Sensor	pH	Radiation	Temperature	Voltage
1	The Determination of a Chemical Formula	No sensor									
2	The Determination of the Percent Water in a Compound	No sensor									
3	The Molar Mass of a Volatile Liquid					O				X	
4	Using Freezing-Point Depression to Find Molecular Weight									X	
5	The Molar Volume of a Gas					X				X	
6	Standardizing a Solution of Sodium Hydroxide				O			X			
7	Acid-Base Titration				O			X			
8	An Oxidation-Reduction Titration: The Reaction of Fe(II) and Ce(IV)				O		X				
9	Determining the Mole Ratios in a Chemical Reaction									X	
10	The Determination of an Equilibrium Constant	X									
11	Investigating Indicators				O			X			
12	The Decomposition of Hydrogen Peroxide					X				O	
13	Determining the Enthalpy of a Chemical Reaction									X	
14	(Parts A and B) Separation and Qualitative Analysis of Cations and Anions							O			
15	(Parts A and B) The Synthesis and Analysis of Alum									X	
16	Conductimetric Titration and Gravimetric Determination of a Precipitate		X		O						
17	Determining the Concentration of a Solution: Beer's Law	X									

X = Required O = Optional		Colorimeter or Spectrometer	Conductivity	Current Probe or Constant Current System	Drop Counter	Gas Pressure	ORP Sensor	pH	Radiation	Temperature	Voltage
18	Liquid Chromatography	No sensor									
19	Buffers							X			
20	Electrochemistry: Voltaic Cells										X
21	Electroplating			X							
22	The Synthesis and Analysis of Aspirin	X								X	
23	Determining the Ksp of Calcium Hydroxide							X			
24	Determining Ka by the Half-Titration of a Weak Acid							X			
25	The Rate and Order of a Chemical Reaction	X									
26	The Enthalpy of Neutralization of Phosphoric Acid									X	
27	Alpha, Beta, and Gamma								X		
28	Radiation Shielding								X		
29	The Base Hydrolysis of Ethyl Acetate		X							O	
30	Exploring the Properties of Gases					X				X	
31	Determining Avogadro's Number			X							
32	Potentiometric Titration of Hydrogen Peroxide				O		X				
33	Determining the Half-Life of an Isotope								X		
34	Vapor Pressure and Heat of Vaporization					X				X	
35	Rate Determination and Activation Energy	X								X	

An Oxidation-Reduction Titration: The Reaction of Fe(II) and Ce(IV)

A titration, as you recall, is a convenient method of learning more about a solution by reacting it with a second solution of known molar concentration. There are a number of ways to measure the progress of a titration. The method used in this experiment is called a potentiometric titration, in which the electric potential of a reaction is monitored. All acid-base titrations that are measured by a pH probe are potentiometric; thus, this method is not as unusual as it may seem.

You will conduct an oxidation-reduction reaction in this experiment in order to determine the amount of iron (II) ions in a solid sample of ferrous ammonium sulfate hexahydrate, $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$. The oxidizing agent for the sample will be ammonium cerium (IV) nitrate, $(NH_4)_2Ce(NO_3)_6$. The net ionic equation for the reaction is shown below.

$$Ce^{4+}(aq) + Fe^{2+}(aq) \rightarrow Ce^{3+}(aq) + Fe^{3+}(aq)$$

This experiment illustrates the electrical nature of chemical reactions, and offers practice with a process for observing and measuring an oxidation-reduction reaction.

OBJECTIVES

- Conduct the potentiometric titration of the reaction between ferrous ammonium sulfate hexahydrate and ammonium cerium (IV) nitrate.
- Measure the potential change of the reaction.
- Determine the molar concentration of iron (II) ions in a sample of ferrous ammonium sulfate hexahydrate.

CHOOSING A METHOD

If you choose **Method 1**, you will conduct the titration in a conventional manner. You will deliver volumes of Ce^{4+} titrant from a buret. You will enter the buret readings manually to store and graph each potential-volume data pair.

If you choose **Method 2,** you will use a Vernier Drop Counter to conduct the titration. Ce^{4+} titrant is delivered drop by drop from the reagent reservoir through the Drop Counter slot. After the drop reacts with the reagent in the beaker, the volume of the drop is calculated and a potential-volume data pair is stored.

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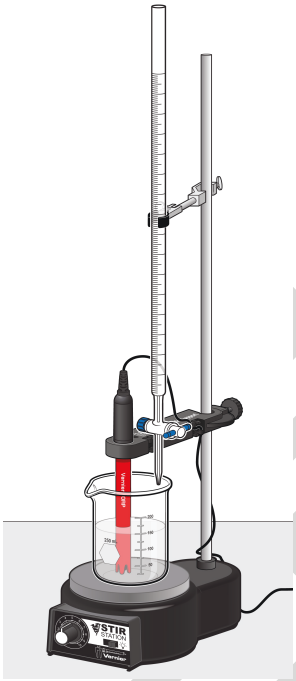

Figure 1

MATERIALS

Materials for *both* Method 1 (buret) *and* Method 2 (Drop Counter)

Vernier computer interface
computer
Logger *Pro*
ORP Sensor
0.100 M $(NH_4)_2Ce(NO_3)_6$ in 1 M H_2SO_4
$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ solution
50 mL graduated cylinder
Stir Station
magnetic stirring bar
wash bottle
distilled water

Materials required *only* for Method 1 (buret)

Electrode Support
50 mL buret
10 or 25 mL pipet and pump
buret clamp
150 mL beaker

Materials required *only* for Method 2 (Drop Counter)

Drop Counter
utility clamp
60 mL reagent reservoir
5 mL pipet
100 mL beaker
10 mL graduated cylinder
two 250 mL beakers

METHOD 1: MEASURING VOLUME USING A BURET

1. Obtain and wear goggles.

2. Measure out precisely 25.00 mL of a ferrous ammonium sulfate solution of unknown molar concentration and transfer it to a 150 mL beaker. **WARNING**: *Solid ferrous ammonium sulfate,* $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$: *May be harmful if swallowed. Causes skin and serious eye irritation. May cause respiratory irritation. Avoid breathing dust or fumes*.

3. Place the beaker of ferrous ammonium sulfate solution on a Stir Station and add a stirring bar.

4. Connect an ORP Sensor to Channel 1 of a Vernier computer interface. Connect the interface to the computer with the proper cable.

5. Start the Logger *Pro* program on your computer. Open the file "08a Potentiometric" from the *Advanced Chemistry with Vernier* folder.

6. Set up a Stir Station, buret clamp, and 50 mL buret to conduct the titration (see Figure 1). Rinse and fill the buret with 0.100 M Ce^{4+} solution. **DANGER**: *Sulfuric acid, concentrated,* H_2SO_4: *Causes severe skin burns and eye damage. Do not breathe mist, vapors, or spray. Maybe be harmful if inhaled. Harmful to aquatic life. Considerable heat generated when diluted with water*.

7. Use an Electrode Support on the Stir Station to hold the ORP Sensor in place during the titration. Position the ORP Sensor so that its tip is immersed in the Fe^{2+} solution but does not interfere with the movement of the magnetic stirring bar. Gently stir the beaker of solution.

8. You are now ready to begin the titration. The objective of your first trial is to determine the region of the titration curve near the equivalence point, and not to precisely determine the equivalence point.
   a. Before adding Ce^{4+} titrant, click Collect. Once the displayed potential reading has stabilized, click Keep. In the edit box, type "**0**" (for 0 mL added). Click OK to continue.
   b. Add 1 mL of the Ce^{4+} titrant. Stir the solution gently at all times. When the potential stabilizes, again click Keep. In the edit box, type the current buret reading. Click OK to continue.
   c. Add Ce^{4+} solution in 1 mL increments and enter the buret reading after each increment. Continue adding Ce^{4+} solution until the potential value remains constant.
   d. Click Stop when you have finished collecting data.
   e. Examine the titration curve and estimate the volume of Ce^{4+} solution used to reach the equivalence point of the titration. Record this value in your data table for Trial 1.

9. When you have completed the titration, dispose of the reaction mixture as directed. Rinse the ORP Sensor with distilled water in preparation for the second trial.

10. Repeat the necessary steps to conduct a second titration with a new sample of $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ solution.

11. When you conduct the second trial, carefully add the Ce^{4+} solution drop by drop in the region near the equivalence point so that you can precisely identify the equivalence point of the reaction.

12. Follow the steps below to find the *equivalence point*, which that is the largest increase in potential upon the addition of a very small amount of Ce^{4+} solution. A good method of determining the precise equivalence point of the titration is to examine the second derivative of the potential-volume data, a plot of Δ^2potential/Δvol^2.
    a. View a plot of the second derivative on Page 3 by clicking Next Page, .
    b. Analyze the second derivative plot and record the volume of Ce^{4+} at the equivalence point.

13. At the direction of your instructor, conduct a third trial. Use your titration data from the second (or third) trial to determine the equivalence point of the reaction.

METHOD 2: MEASURING VOLUME WITH A DROP COUNTER

1. Obtain and wear goggles.

2. Add about 20 mL of distilled water to a 100 mL beaker. Use a pipet bulb (or pipet pump) to transfer 5.0 mL of the ferrous ammonium sulfate solution of unknown molar concentration into the 100 mL beaker with distilled water. **WARNING**: *Solid ferrous ammonium sulfate,* $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$: *May be harmful if swallowed. Causes skin and serious eye irritation. May cause respiratory irritation. Avoid breathing dust or fumes*.

3. Connect the ORP Sensor to CH 1 of the computer interface. Lower the Drop Counter onto a Stir Station (see Figure 2) and connect it to DIG/SONIC 1.

4. Start the Logger *Pro* program on your computer. Open the file "08b Potentiometric (Drop)" from the *Advanced Chemistry with Vernier* folder.

5. Obtain the plastic 60 mL reagent reservoir. Close both valves by turning the handles to a horizontal position. Follow the steps below to set up the reagent reservoir for the titration.
    a. Rinse the reagent reservoir with a few mL of the 0.100 M Ce^{4+} solution and pour it into an empty 250 mL beaker. **DANGER**: *Sulfuric acid, concentrated,* H_2SO_4: *Causes severe skin burns and eye damage. Do not breathe mist, vapors, or spray. Maybe be harmful if inhaled. Harmful to aquatic life. Considerable heat generated when diluted with water*.
    b. Use a utility clamp to attach the reservoir to the Stir Station.
    c. Fill the reagent reservoir with slightly more than 60 mL of the 0.100 M Ce^{4+} solution.
    d. Place the 250 mL beaker, which contains the rinse Ce^{4+} solution, beneath the tip of the reservoir.

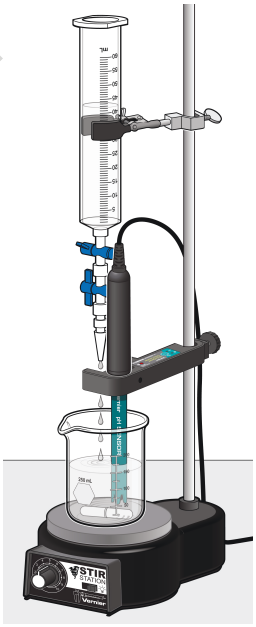

Figure 2

e. Drain a small amount of the Ce^{4+} solution into the 250 mL beaker to fill the reservoir's tip. To do this, turn both valve handles to the vertical position for a moment, then turn them both back to horizontal.

f. Discard the drained the Ce^{4+} solution in the 250 mL beaker as directed.

6. Calibrate the drops that will be delivered from the reagent reservoir. **Note**: If you are using the stored calibration (28 drops per mL), skip this step.
   a. Choose Calibrate ► DIG 1: Drop Counter from the Experiment menu.
   b. Proceed by one of these two methods:
      - If you have previously calibrated the drop size of your reagent reservoir and want to continue with the same drop size, select the Manual button, enter the number of Drops/mL, and click OK. Then proceed directly to Step 7.
      - If you want to perform a new calibration, select the Automatic button, and continue with this step.
   c. Place a 10 mL graduated cylinder directly below the slot on the Drop Counter, lining it up with the tip of the reagent reservoir.
   d. Open the bottom valve on the reagent reservoir. Keep the top valve closed.
   e. Click the Start button on the dialog box.
   f. Slowly open the top valve of the reagent reservoir so that drops are released at a slow rate (~1 drop every two seconds). You should see the drops being counted on the computer screen.
   g. When the volume of the Ce^{4+} solution in the graduated cylinder is between 9 and 10 mL, close the bottom valve of the reagent reservoir.
   h. Enter the precise volume of Ce^{4+} solution (read to the nearest 0.1 mL) in the edit box.
   i. Click OK. Discard the Ce^{4+} solution in the graduated cylinder as directed and set the graduated cylinder aside.

7. Assemble the apparatus.
   a. Insert the ORP Sensor through the large hole in the Drop Counter.
   b. Adjust the positions of the Drop Counter and reagent reservoir so they are both lined up with the center of the Stir Station.
   c. Lift up the ORP Sensor, and place the 100 mL beaker containing the ferrous ammonium sulfate solution onto the Stir Station. Lower the ORP Sensor into the beaker.
   d. Place a magnetic stirring bar in the beaker, and adjust the position of the ORP Sensor so the sensor will not be struck when the stirring bar is stirring.
   e. Adjust the reagent reservoir so its tip is just above the Drop Counter slot.

8. Turn on the Stir Station so that the stirring bar is stirring at a fast rate.

9. You are now ready to conduct the titration. Click Collect. No data will be collected until the first drop goes through the Drop Counter slot. Fully open the bottom valve, but do not touch the top valve. After the first drop passes through the Drop Counter slot, check the data table to see that the first data pair was recorded.

10. Watch your graph to see when a large increase in potential takes place. This will be the equivalence point of the reaction. When this jump in potential occurs, let the titration proceed for several more milliliters of titrant, then click ■ Stop. Close the bottom valve of the reagent reservoir. Dispose of the reaction mixture as directed.

11. Follow the steps below to find the *equivalence point*, which is the largest increase in potential upon the addition of a very small amount of Ce^{4+} solution. A good method of determining the precise equivalence point of the titration is to examine the second derivative of the potential-volume data, a plot of Δ^2potential/Δvol^2.
    a. View a plot of the second derivative on Page 3 by clicking Next Page, .
    b. Analyze the second derivative plot and record the volume of Ce^{4+} at the equivalence point.

12. Repeat the titration with a second ferrous ammonium sulfate solution. Analyze the titration results in a manner similar to your first trial and record the equivalence point.

DATA TABLE

	Trial 1	Trial 2
Volume of Fe^{2+} solution (mL)		
Volume of Ce^{4+} solution used to reach equivalence point (mL)		

DATA ANALYSIS

1. Calculate the molar amount of Ce^{4+} used to reach the equivalence point of the reaction.

2. Calculate the molar amount of iron in the sample of ferrous ammonium sulfate solution.

3. Calculate the molar concentration of the ferrous ammonium sulfate solution.

4. The ferrous ammonium sulfate solution that you tested was prepared by dissolving 40.0 g of solid $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ in 1.00 liter of solution. This substance is often impure.
    a. Calculate the theoretical percent Fe in a pure sample of $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$.
    b. Calculate the percent Fe in the sample that you tested.
    c. Compare your experimental percent Fe to the theoretical percent Fe. How pure was your sample? Use a calculation to support your assertion.

Electroplating

In this experiment, you will conduct, observe, and measure the process of electroplating. This process is used to deposit a layer of metal, such as chromium, copper, or gold, onto another metal. As a commercial process, electroplated coatings are used to improve appearance, resist corrosion, or improve hardness of metallic surfaces. This experiment describes one method of producing a copper coating on a brass key or other suitable metallic object.

You will prepare an electrochemical cell by using a copper strip and a brass key as the electrodes. The electrodes are immersed in a solution containing acidified copper (II) sulfate. As you apply a potential to the electrodes, you will be transferring Cu atoms to the surface of the key.

This process is expressed in equation form as

$$\text{Mass deposited at an electrode} = \frac{I \times t \times (MM)}{96{,}500 \times \text{n}}$$

I is the current in amperes; *t* is the time that the current is applied, in seconds; *MM* is the molar mass of the element that is deposited; n is the number of moles of electrons/mol; and 96,500 is *F*, the Faraday constant.

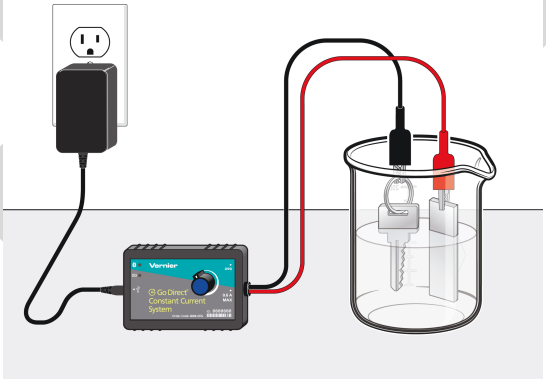

Figure 1

OBJECTIVES

- Prepare and operate an electrochemical cell to plate copper onto a brass surface.
- Measure the amount of copper that was deposited in the electroplating process.
- Calculate the amount of energy used to complete the electroplating process.

MATERIALS

Chromebook, computer, **or** mobile device
Graphical Analysis 4 app
Go Direct Constant Current
steel wool
two 250 mL beakers
distilled water
electrolyte solution ($CuSO_4$ in H_2SO_4)
vinegar
1 cm × 10 cm copper strip
brass key
solid sodium chloride, NaCl
analytical balance
bare copper wire, 20–22 gauge

PROCEDURE

1. Obtain and wear goggles.

2. Use steel wool to clean a brass key and a strip of copper, which will be the electrodes of the electrochemical cell.

3. Mix 3 g of NaCl with 15 mL of vinegar in a 250 mL beaker. Wash the key and the copper strip in this salt/vinegar solution. Rinse the key and copper strip with distilled water and dry each metal piece. **WARNING**: *Sodium chloride solution,* NaCl: *May be harmful if swallowed. Skin and eye irritant*. **Caution**: *Vinegar (acetic acid, 5%): Treat as a non-food-grade chemical. Prudent laboratory practices should be observed.*

4. Use an analytical balance to determine the mass of the key and the mass of the copper strip. Record these two masses in your data table.

5. Fill a 250 mL beaker about 3/4 full with the electrolyte solution. **DANGER**: *Copper sulfate electrolyte solution, containing sulfuric acid: Do not eat or drink when using this product—toxic if swallowed. Causes skin and serious eye irritation.*

6. Attach a 7 cm length of bare copper wire to the brass key to act as a handle, so that it is easier to completely immerse the key in the electrolyte solution.

7. Gently turn the dial of the Constant Current System counterclockwise to confirm that it is in the minimum current position. Clip the wire handle on the key and the copper strip to the proper leads (See Figure 1). **Important**: You will not place the electrodes in the cell until Step 10.

8. Plug the Constant Current System into a powered electrical outlet. Launch Graphical Analysis. Connect the Constant Current System to your Chromebook, computer, or mobile device.

9. Place the key and the copper strip into the electrolyte solution in the beaker. Make sure that the key is completely immersed in the solution, and keep the two electrodes as far apart as possible.

10. Click or tap Collect to start data collection. Allow two data points to be collected to establish a baseline. Adjust the current to the 0.2–0.3 A range. Observe the electrolysis. Note the slow deposition of copper on the surface of the key. Data collection will run for 30 minutes.

11. After data collection has run for ~28 minutes, turn off the power supply. Allow the data collection to run to completion. After data collection stops, carefully remove the copper strip and the key from the electrolyte solution. Rinse the two metals with distilled water. Gently dry the copper strip and key so as not to remove copper.

12. Measure and record the mass of the dry copper strip and key.

13. Determine the average current applied during the experiment.
    a. Click or tap Graph Tools, , and choose Statistics.
    b. Record the mean (average) current in your data table.

14. Analyze the graphed data to determine the average current applied during the experiment. Record the average current in your data table.

15. Discard the electrolyte solution and take care of the electrochemical cell as directed.

DATA TABLE

Initial mass of copper electrode (g)	
Final mass of copper electrode (g)	
Initial mass of key (g)	
Final mass of key (g)	
Average current (A)	
Time of current application (s)	

DATA ANALYSIS

1. Calculate the number of coulombs of charge that passed through the electrolytic cell. (**Hint**: Make sure you are using the proper unit of time for your calculation.)

2. Use your current *vs*. time data to calculate the theoretical number of moles of copper that should have plated out onto the brass key.

3. Use your data from measuring the mass of the electrode and the key to calculate the actual number of moles of copper that plated out.

4. Calculate the percent yield of copper.
5. Suggest the sources of error in your experiment.
6. Write the oxidation and reduction half-reactions for this process.