Experiment 5 ELECTROCHEMISTRY AND CORROSION

INTRODUCTION

A. Electrochemistry

Electrochemistry is the branch of chemistry that deals with the relationship between chemical reactions and electrical energy. It covers a wide field of subjects such as batteries, corrosion, reactivity of metals, and electroplating. The chemical reactions involved in electrochemistry are of the oxidation-reduction (redox) type.

Oxidation-reduction or redox reactions involve changes in the oxidation states of the reactants. Substances that lose electrons (increasing the oxidation number) during a chemical reaction are said to be oxidized. Those that gain electrons (decreasing the oxidation number) are said to be reduced. If one reactant gains electrons, another one must lose an equal number of electrons. Thus, oxidation and reduction must occur simultaneously. When the flow of electrons constitutes a current of electricity, the reaction is said to be an electrochemical reaction. These reactions are carried out in a device called electrochemical cells.

Electrochemical reactions that are spontaneous can generate electricity. The type of cell where this process takes place is called a voltaic or galvanic cell. The two halves of the redox reaction are separated, requiring electron transfer to occur through an external circuit. In this way, useful electrical energy is obtained. An example of a voltaic cell is a battery.

By reversing the process, electricity can be used to make nonspontaneous redox reactions happen. This process is called electrolysis. An electrolytic cell consists of a container for the reaction material with electrodes immersed in the reaction material and connected to a source of direct current. Inert electrodes are often used so they do not become involved with the reaction.

B. Corrosion

Corrosion is the partial or complete wearing away, dissolving, or softening of any substance by electrochemical reaction with its environment. The term corrosion specifically applies to the gradual action of natural agents such as air or salt water on metals.

The most familiar example of corrosion is the rusting of iron. It is a complex chemical reaction in which the iron combines with both oxygen and water to form hydrated iron oxide. The oxide is a solid that retains the same general form as the metal from which it is formed but porous and somewhat bulkier. It is relatively weak and brittle.

The oxidation of metals occurs most readily at points of strain (where the metals are most "active"). A point of strain in a steel object acts as an anode where the iron is oxidized to iron(II) ions.

Fe
$$\rightarrow$$
 Fe²⁺ + 2e⁻ (oxidation, anode)

The electrons produced then flow through the nail to areas exposed to O_2 . These act as cathodes where oxygen is reduced to hydroxide ions, OH.

$$^{\cdot}\text{O}_2 + ^{2}\text{H}_2\text{O} + ^{4}\text{e}^{^{-}} \rightarrow ^{4}\text{OH}^{^{-}}$$
 (reduction, cathode)

At the same time, the Fe^{2+} ions migrate through the moisture on the surface from the anode toward the cathode region where they combine with OH^- ions to form iron(II) hydroxide. Iron is further oxidized by oxygen to the +3 oxidation state. The material we call rust is a complex hydrated form of iron(III) oxides and hydroxides with variable water composition.

2Fe(s) +
$$3/2O_2(g)$$
 + $xH_2O \rightarrow Fe_2O_3 \cdot xH_2O$

In this experiment, the electrochemical nature of corrosion of iron will be studied. To locate the part of the iron where oxidation and reduction take place, an indicator consisting of potassium ferricyanide and phenolphthalein is used. Potassium ferricyanide reacts with ferrous ion to form a blue precipitate; thus, the appearance of a blue color establishes the presence of the ferrous ion and therefore locates the oxidation site (anode). Phenolphthalein forms a pink color in the presence of hydroxide ion. The appearance of a pink color locates the reduction site (cathode).

C. Corrosion Protection

There are several methods for protecting metals against corrosion. The most widely used are the following:

- 1. plating the metal with a thin layer of a less easily oxidized metal
- 2. connecting the metal directly to a sacrificial anode, a piece of another metal that is more active and therefore is preferentially oxidized
- 3. allowing a protective film such as a metal oxide to form naturally on the surface of the metal
- 4. galvanizing or coating steel with zinc (a more active metal)

5. applying a protective coating such as paint

OBJECTIVES

- 1. To assemble a voltaic cell
- 2. To illustrate the electrochemical nature of corrosion of iron
- 3. To demonstrate cathodic protection

LIST OF CHEMICALS

50 mL	1M zinc Sulfate	15 gram sodium chloride
50 mL	1M cupric sulfate	0.5 gram agar-agar
2 mL	phenolphthalein	4 pieces iron nail
1 mL	0.01M potassium ferricyanide	1 piece paper clip
5 drops	0.1M ferrous sulfate	a strip of zinc (1.5cm x 15 cm)
5 drops	0.1M sodium hydroxide	1 strip of copper (1.5 cm x 15 cm)
. •	cotton	distilled water

LIST OF APPARATUS

Bunsen burner, 2 micro test tubes, test tube rack, 10-mL graduated cylinder, 50-mL graduated cylinder, stirring rod, two 150-mL beakers, iron stand, wire gauze, 2 Petri dishes, iron ring, electronic balance, 10-cm plastic tube, DC voltmeter (1-5 volts), 15-cm copper wire cables (red and black) with alligator clips at each end

SAFETY PRECAUTION: When skin is in contact with sodium hydroxide, immediately wash it with water. Sodium hydroxide is a skin irritant.

NOTE: Clean all metals with sandpaper before using them. This will remove any oxide and dirt on the surface of the metals.

PROCEDURE

A. Zinc-Copper Voltaic Cell

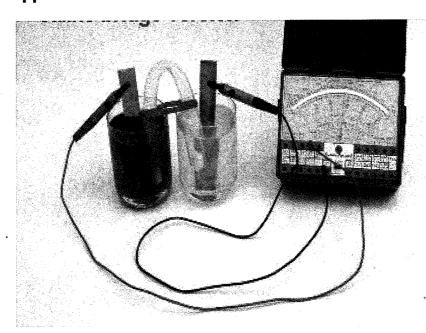


Figure 1. Zinc-Copper Voltaic Cell

- 1. Construct the set-up shown in Figure 1. Pour 50 mL of 1M cupric sulfate in a 150-mL beaker with the copper electrode.
- 2. Pour 50 mL of 1M zinc sulfate in a 150-mL beaker with the zinc electrode.
- 3. Prepare the salt bridge. It is made by using a U-shaped tube (plastic tube) filled with saline solution. Fill the tube with sodium chloride dissolved in distilled water (10 grams of NaCl per 100 mL of water). Plug the ends with cotton so that the saline solution will remain in the bridge and not mix with the electrolytes.
- 4. Attach the alligator clip of the red cable of the voltmeter to the copper electrode and the clip of the black cable to the zinc electrode.
- 5. Measure the voltage produced between the two strips.
- 6. **Waste Disposal:** Clean the copper and zinc strips with tap water and return to the laboratory assistant. Place the cupric sulfate and zinc sulfate solutions in labeled containers. Dispose the sodium chloride solution in the sink with plenty of water.

B. Tests for the Products of Corrosion of Iron

- 1. Place 5 drops of 0.1M ferrous sulfate in a micro test tube. Add one drop of 0.01M potassium ferricyanide. Observe the color that will be produced. This is the color indicating the presence of ferrous ions (Fe²⁺).
- 2. Place 5 drops of 0.1M sodium hydroxide in a micro test tube and add one drop of phenolphthalein. Observe the color that will be produced. This is the color indicating the presence of the hydroxide ions (OH⁻).
- 3. **Waste Disposal:** The solutions can be disposed down the drain with plenty of water.

C. Oxidation and Reduction Sites and Cathodic Protection

Observe the set-ups very carefully. Your observations should be tied up with what you saw in part C. An oxidation site is the area where Fe²⁺ ions are formed while a reduction site is where OH⁻ ions are formed. Other metals coupled with the iron nails may enhance or retard the rate of corrosion.

- 1. In a Petri dish, place 2 iron nails: one nail is bent at right angle and the other nail is just as it is. Keep the two nails well separated. Label this Petri dish A.
 - 2. Wrap the head of a third iron nail with copper wire. Get another iron nail and wrap its head with a piece of magnesium ribbon. Place these nails in another Petri dish and label this dish B.
 - 3. Heat to boiling 100 mL of distilled water in a 150-mL beaker. Add 0.5 gram of agar-agar. Stir well until the entire agar-agar has dissolved and a colloidal suspension is formed. Turn off the burner. Add 5 grams of sodium chloride, 1 mL of 0.01M potassium ferricyanide, and 2 mL of phenolphthalein. Mix well. Allow the mixture to cool slightly.
 - 4. Pour the lukewarm agar-agar mixture to the prepared Petri dishes until the metals are sufficiently covered.
 - 5. Observe the locations of the blue and pink areas in the nails in dish A.
 - 6. Describe all the changes that occurred and compare the locations of the blue areas in both nails in dish B.

7. **Waste Disposal:** Wash the iron nails with tap water, dry, and return to the laboratory assistant. Dilute the agar-agar mixture with tap water and dispose down the drain.

REFERENCES

Brady, James E. (2004). Chemistry: Matter and Its Changes, 4th Edition. John Wiley and Sons, Inc, NJ.

Chang, R. (2008). General Chemistry: The Essential Concepts, 5^{th} Edition. McGraw-Hill, New York.

GRO	UP	NC).	

Name:	Date Performed:		
Course & Section:	Date Submitted:		
Program & Year:	Professor:		

Experiment 5 ELECTROHEMISTRY AND CORROSION

A. Zinc-Copper Voltaic Cell

Which of the two electrodes was the anode? the cathode?

What happened to the zinc electrode?

Write the half-cell reaction that took place in the zinc electrode.

What happened to the copper electrode?

Write the half-cell reaction that took place in the copper electrode.

Write the overall cell reaction for this voltaic cell.

What was the cell emf (voltmeter reading) for this voltaic cell?

Write the cell notation for this voltaic cell.

B. Tests for the Products of Corrosion of Iron

What was the color produced when Fe²⁺ ion reacted with potassium ferricyanide?

What was the color produced when phenolphthalein was added to the solution containing OH ions?

C. Oxidation and Reduction Sites and Cathodic Protection

Draw the two Petri dish set-ups. Show where the colors appeared. Identify the anode and the cathode.

Answer t	the	following	questions	based	on	the	results	obtained	from
Petri dish	۱ A.								

Petri dish A.
At what part(s) of the iron nail did oxidation take place?
At what part(s) of the iron nail did reduction take place?
Make a generalization on the effect of metal strain to the corrosion of iron.
Write half-cell reactions occurring in the iron nail.

Answer the following based on the results obtained from Petri dish B.

If cathodic protection is to be used to prevent the corrosion of iron, which of the two metals, magnesium and copper, can be used to protect the iron? Why?

Write half-cell reactions occurring in the iron nail with magnesium ribbon.