

**ELECTROCHEMISTRY: CORROSION**

**MATERIALS:** Cu, Zn strips; sandpaper; 20d bright common nails; 3 M HCl; 0.5 M NaCl; DC power supply; various electrical leads; plastic pipets; digital multimeter; phenolphthalein in dropper bottles; 50 mL (2), 400 mL beakers; 25 mL graduated cylinder

**PURPOSE:** The purpose of this experiment is to illustrate the principles and practical aspects of corrosion and corrosion prevention.

**LEARNING OBJECTIVES:** By the end of this experiment, the student should be able to demonstrate the following proficiencies:

1. Explain how “atmospheric corrosion” occurs, and describe specifically how it damages a metal object or structure
2. Identify the conditions for “chemical corrosion”.
3. Describe the use of galvanic protection and impressed voltage for corrosion prevention.

**PRE-LAB:** Complete the Pre-Lab Assignments before coming to lab.

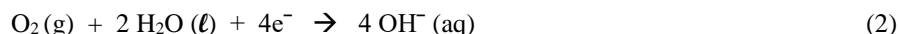
**DISCUSSION:**

Corrosion can be defined as the deterioration of metals by spontaneous electrochemical reactions between the metal and its environment. Oxidation of the metal during corrosion can lead to a loss of structural integrity. The more active (prone to oxidation) a metal is, to more susceptible to corrosion it will be. Our focus will be corrosion of iron, but the problem is not limited to iron-based structures. Aluminum and other important structural metals also corrode under the proper conditions.

Atmospheric corrosion and Chemical corrosion. In corrosion of metals, the anode half-reaction always involves oxidation of the metal. For the case of iron, the oxidation reaction is:



Corrosion reactions are divided into two classes depending upon the nature of the cathode (reduction) reaction. In **atmospheric corrosion** the anode half-reaction is coupled to the reduction of dissolved oxygen at the cathode of an electrochemical cell. In this case, the cathode half-reaction is:



Even if the anode and cathode regions of a cell are made from the same type of metal, atmospheric corrosion can still occur if there is a nonuniformity in the dissolved oxygen concentration, such as occurs with the hull of a ship where the dissolved oxygen concentration is greater near the water surface. These concentration differences are enough to cause the different regions of the same piece of metal to act anodically or cathodically.

**Chemical corrosion** occurs in acidic environments. For example, in chemical corrosion of iron, the oxidation (anode) reaction is the same as equation (1), but the reduction (cathode) reaction involves the formation of hydrogen gas:



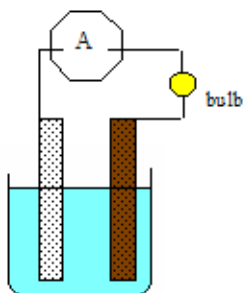
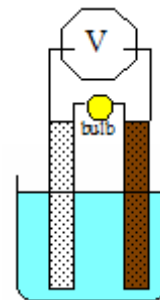
An example would be corrosion of iron by battery acid. For more information about naval applications of electrochemistry and corrosion, go to the website <https://intranet.usna.edu/ChemDept/files/documents/navapps/CURRENT/2018%20Documents/sc112-navapp-s18-ch19-corrosion-o2-production-FV.pdf>

*Corrosion Prevention.* There are a number of methods used to stop or slow down the spontaneous corrosion of iron. Barrier methods, such as coating the metal with paint or grease, are the simplest means to protect the iron. These work by preventing the three necessary reactants of atmospheric corrosion – iron, water and oxygen – from coming together. The use of a *less* active metal coating such as tin is another barrier method. Among the most important electrical methods for corrosion prevention, widely used in the Navy (and elsewhere), are **galvanic protection** and **impressed voltage**. With “galvanic protection” the Fe is electrically coupled to a *more* active element, typically Zn. The Zn corrodes sacrificially, protecting the Fe. Zinc plates are attached to the hulls of ships, both large and small, to perform this function. With impressed voltage, an electrical power supply is connected to the iron, and continually feeds electrons to it. This maintains the iron in a reduced state. This method of protection is typically used when ships are tied up in port.

## USE OF THE DIGITAL MULTIMETER

A multimeter can measure several important electrical properties, namely voltage, current and resistance. We will only be interested in the first two for this experiment. Because the instrument functions differently for these different measurements, it is important that it be set up properly to make them. Because it has multiple scales in each case, it is also important that you read it properly to get meaningful data. This sheet provides a brief description of proper use of the device.

**Making Voltage Readings** When the multimeter is set to one of the voltage scales, it acts as a potentiometer. This measures the voltage difference between two points of a circuit by creating an equal (but opposite) electromotive force and applying it to the circuit until the current going through the meter is reduced to zero. Thus, **when a multimeter is set to one of the voltage scales, there is no current flow in the meter**, and the electrochemical process under investigation is actually stopped. What you read is based on the voltage that was applied to stop current flow. For this to work properly, the two leads of the meter must be placed **ACROSS** the circuit, touching the two points of interest (usually the electrodes). See the drawing at right. Also, note that the **red wire** is attached to the **cathode** when the voltmeter displays a **positive** value. Make your connections to get positive voltages; knowing which electrode is the cathode will help you analyze the chemical behavior of the system.

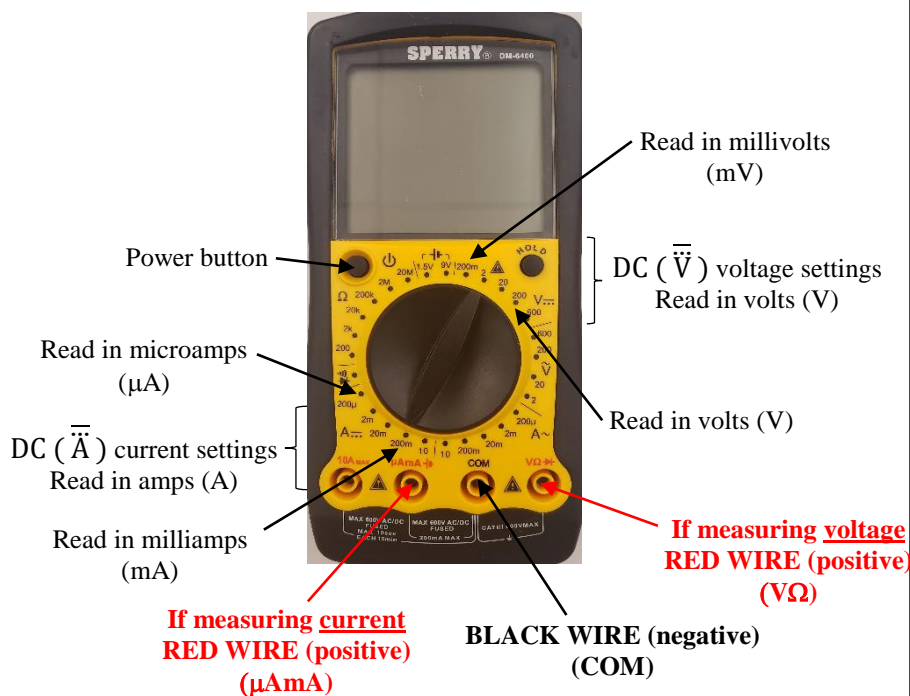


**Making Current Readings** When a multimeter is set to one of the current (amps) scales, the electrochemical process is not stopped by any opposing forces, as in the case when electrical potential is being measured. Instead, the current produced by the process is passed through the meter where it is measured. Thus for current readings, the meter must be **IN** the circuit. See the drawing at left.

**Reading Values of Voltage or Current** Because there are different scales on the meter, and even different units **within** the voltage scales or current scales, it is essential that you note what position you set the dial to, and to always include the units with your readings. Shown below is an image of the meter face.

The label at each setting (e.g. 200m) indicates the maximum reading on that scale.

**Here are some general rules for reading the meters.  
Follow these to get consistent results.**



1. Always write down the **UNITS** with your values.
2. For the **SPERRY** multimeter:
  - a. When reading DC voltage, use the DC ( $\overline{\text{V}}$ ) setting.
    - i. Set the function/range switch to the appropriate DC V range. When reading voltage, start with the 200mV setting, if needed you can go to the next higher scale (2V). You will never need the 200V or 600V settings!
  - b. When reading DC amperage, use the DC ( $\overline{\text{A}}$ ) setting.
    - i. Set the function/range switch to the appropriate DC A range. When reading voltage, start with the 200 $\mu$ A setting, if needed you can go to the next higher scale (2mA). You will never need the 200mA settings!
  - c. Insert the black (negative) test lead into the COM input terminal.
  - d. Insert the red (positive) test lead into the V input terminal OR into the  $\mu$ mA input terminal when measuring current.
  - e. Read the value of the measurement displayed. If the leads are reversed a “-“ indicator will appear on the display.
  - f. The instrument will adjust the scale automatically unless the load is too large, then a “1.” will appear and you will need to switch to the next LARGER scale.

Name \_\_\_\_\_

Section \_\_\_\_\_

Partner \_\_\_\_\_

Date \_\_\_\_\_

**PROCEDURE, DATA AND IN-LAB QUESTIONS**  
**Experiment 21H**

**Part 1: "Atmospheric Corrosion"**

*In each of these experiments, it's important not to let the electrodes touch each other, in solution. Hold the electrodes upright to ensure that the metal clips connecting the wires to the electrodes do NOT contact the solution directly.*

**a) Corrosion cell in pure water**

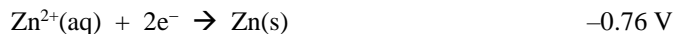
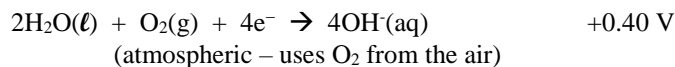
Fill 400 mL beaker ½ full with deionized (DI) water and place it on a white- or light-colored paper. Place Cu & Zn electrodes in beaker (not touching). Attach the voltmeter such that a (+) voltage is obtained and record the voltage and identify which electrode is attached to the red and black wire:

Red wire (cathode) is: \_\_\_\_\_

Black wire (anode) is: \_\_\_\_\_

Voltage: \_\_\_\_\_ V

Four possible cathode half-reactions, and E° values :



Any reaction that occurs is based on one of these four possible half-reactions. Which chemical species are available in significant amounts in the beaker? (circle all that apply)

H<sub>2</sub>O(ℓ)    O<sub>2</sub>(g)    OH<sup>-</sup>(aq)    Cu<sup>2+</sup>(aq)    Cu(s)    Zn<sup>2+</sup>(aq)    Zn(s)    H<sub>2</sub>(g)

You can only have a chemical reaction when the reactants are available. Recognizing that fact,

Which is the most likely reduction half-reaction occurring? \_\_\_\_\_

most likely oxidation half-reaction? \_\_\_\_\_

Why did you choose these reactions?

Add a drop of phenolphthalein indicator around each electrode, one at a time:

Color change around Zn? (Y/N) \_\_\_\_\_ Describe. \_\_\_\_\_

Color change around Cu? (Y/N) \_\_\_\_\_ Describe. \_\_\_\_\_

Switch multimeter to current (mA) setting and move the test lead as necessary:

Record observations of color change: \_\_\_\_\_

Record current, with units: \_\_\_\_\_

**b) Corrosion cell in salt water**

Dispose of the previous solution in the beaker, clean off the electrodes, then rebuild cell the same as above, but use 0.5 M NaCl solution in the beaker instead of water. Attach the voltmeter to obtain a (+) voltage. Record the following:

Voltage: \_\_\_\_\_

Add phenolphthalein indicator around each electrode, one at a time:

Color change around Zn? (Y/N) \_\_\_\_\_ Describe. \_\_\_\_\_

Color change around Cu? (Y/N) \_\_\_\_\_ Describe. \_\_\_\_\_

Switch multimeter to current (mA) setting and move the test lead as necessary:

Record observations of color change. \_\_\_\_\_

Record current, with units: \_\_\_\_\_

How did the voltage compare to the pure water case? \_\_\_\_\_

How did the current compare to the pure water case? \_\_\_\_\_

Why? Explain the observed differences (if any) in voltage and current for cell set up in pure water vs. salt water.

**c) Effect of non-uniform O<sub>2</sub> concentration**

Dispose of solution and clean off the electrodes. Use the small plastic container in the student drawer and fill it to a depth of ~ 1/2 inch with NaCl solution. Immerse two Zn strips in the solution on opposite sides of the container. (You can lay them flat on the bottom.) Attach the voltmeter to obtain a (+) voltage. Once you have it all connected, avoid agitating the solution.

Voltage: \_\_\_\_\_

Using a plastic disposable pipet, gently and repeatedly squeeze bubbles of air right next to the surface of one of the Zn strips. Try for a long steady stream of bubbles. (Again, agitate the solution as little as possible before/while bubbling.)

What happens to the voltage? \_\_\_\_\_

Now, slowly squeeze out air bubbles next to the other Zn strip.

What happens to the voltage now? \_\_\_\_\_

Your actions above created locally higher concentrations of O<sub>2</sub>(aq) wherever you bubbled the air. What effect does that have on the cathodic reaction of atmospheric corrosion? (Note that the cathodic (reduction) reaction is the same one you identified on the previous page.)

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Which strip of Zn will be the cathode? (Consider the possible half-reactions.) (circle)

- i. Zn where the bubbles were added                      ii. Zn strip where bubbles were NOT added

Which strip of Zn will experience a higher rate of corrosion? (Consider the possible half-reactions.) (circle)

- i. Zn where the bubbles were added                      ii. Zn strip where bubbles were NOT added

**Part 2: “Chemical Corrosion” (Effect of HCl acid on Zn metal and Fe metal (nail))**

Add 25.0 mL of 3.0 M HCl into a 50 mL beaker. Set up another beaker, full of DI water, right next to the beaker of HCl solution. Take a 4 inch nail and sand the bottom half clean. Also sand the bottom half of a Zn(s) strip. Wipe away all sanding residue, rinse in DI water and completely dry the metals with a paper towel.

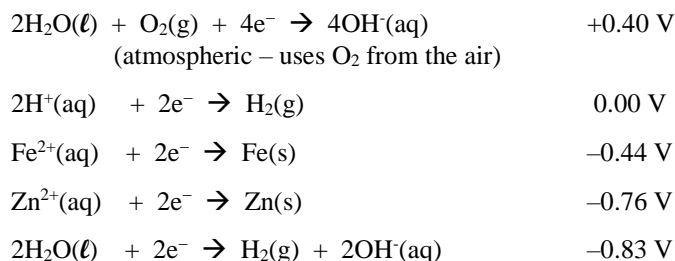
Go to one of the **analytical (4 decimal place) balances**. Zero the balance with a 100 mL beaker in place on the balance pan. (Make sure all the doors are closed.) One at a time, place the Zn strip and nail in the beaker and record the initial mass of each. *In this and all subsequent parts of the lab, make all before and after measurements on the SAME analytical (4 decimal place) balance! Make sure the object is DRY!*

Zn: \_\_\_\_\_ g                      Nail \_\_\_\_\_ g

**(In this step DO NOT let electrodes touch!)** Simultaneously immerse the two metals (cleaned ends) into the HCl solution, for 3.0 minutes. Observe the sample and agitate periodically. After 3.0 minutes have elapsed, remove both metals from the HCl beaker and swish them around in the beaker of water to remove traces of acid and reaction products.

Observations: \_\_\_\_\_

Possible half-reactions:



Any reaction that occurs is based on these possible half-reactions. Which chemical species are available in significant amounts in the beaker? (circle all that apply)

$\text{H}_2\text{O}(\ell)$      $\text{O}_2(\text{g})$      $\text{OH}^-(\text{aq})$      $\text{H}^+(\text{aq})$      $\text{Fe}^{2+}(\text{aq})$      $\text{Fe}(\text{s})$      $\text{Zn}^{2+}(\text{aq})$      $\text{Zn}(\text{s})$      $\text{H}_2(\text{g})$

You can only have a chemical reaction when the reactants are available. Recognizing that fact,

Which are the two most likely oxidation half-reactions occurring? (1) \_\_\_\_\_

(2) \_\_\_\_\_

Bubbles at Zn? (Y/N) \_\_\_\_\_ Identify the gas: \_\_\_\_\_

Bubbles at Nail? (Y/N) \_\_\_\_\_ Identify the gas: \_\_\_\_\_

Which is the most likely reduction half-reaction occurring? \_\_\_\_\_

More bubbles at one electrode than the other? (Y/N) \_\_\_\_\_ Which had more? \_\_\_\_\_

Think of the spontaneous electrochemical cells based on the oxidation and reduction reactions you just identified. Use them to explain why one electrode bubbled more than the other.

Remove the metals from the water beaker, rinse them with DI water, THOROUGHLY dry them and reweigh on the **same analytical balance** as before. Record final mass of each:

Zn: \_\_\_\_\_ g                      Nail: \_\_\_\_\_ g

### **Part 3: "Galvanic Protection" (Effect of coupling a more active metal to a less active one)**

Discard used HCl solution. Obtain a fresh 25.0 mL HCl sample, and a fresh beaker of water. This time you will see what happens when the metals are connected electrically with a jumper wire.

Re-sand the metal strips; clean, DRY and reweigh them (**analytical balance**), recording initial mass of each:

Zn: \_\_\_\_\_ g                      Nail: \_\_\_\_\_ g

Use a jumper wire with alligator clips on each end to connect the **unsanded** parts of the two metals. Simultaneously immerse the two metals (cleaned ends) into the HCl solution, for 3.0 minutes. Observe the sample and agitate periodically. After 3.0 minutes have elapsed, remove both metals from the HCl beaker and swish them around the beaker of water to remove traces of acid and reaction products.

Bubbles at zinc? (Y/N) \_\_\_\_\_ Identify the gas: \_\_\_\_\_

Bubbles at nail? (Y/N) \_\_\_\_\_ Identify the gas: \_\_\_\_\_

More bubbles at one electrode than the other? (Y/N) \_\_\_\_\_ Which had more? \_\_\_\_\_

Which electrode is more readily oxidized? (Consider the half-reactions on the preceding page.) \_\_\_\_\_

That electrode will be the (circle)      anode      cathode

Remove the metals from the water beaker, rinse them with DI water, THOROUGHLY dry them and reweigh on the same **analytical balance**. Record final mass of each:

Zn: \_\_\_\_\_ g                      Nail: \_\_\_\_\_ g

### **Part 4: "Impressed Voltage" Protection - Effect of applied voltage on same electrodes**

Discard the HCl solution in the 50 mL beaker and replace it with 25.0 mL of **0.5 M NaCl** solution. Prepare two 4 inch nails by sanding the lower halves; then clean and dry the nails thoroughly. Mark the head of one nail to identify it as nail #1. Carefully weigh both nails on the **analytical balance**. Record the initial mass of each:

nail #1 (marked): \_\_\_\_\_ g                      nail #2: \_\_\_\_\_ g

With no wires attached, turn on the power supply, adjust the upper knob to give a 3.0 V setting, and then turn off the power supply. Use jumper wires to attach #1 nail to the (+) terminal and #2 nail to the (-) terminal of the power supply.

Immerse both nails simultaneously into the NaCl solution (do not let them touch). Turn on the power supply. Let it run for 3.0 minutes. (If nothing happens, adjust the lower (current) knob until the red LED turns green.)

(+) Bubbles around nail #1? (Y/N) \_\_\_\_\_                      (-) Bubbles around nail #2? (Y/N) \_\_\_\_\_

After 3.0 minutes, Turn off the power supply – leave nails undisturbed, answer questions:

Record any color change around nail #1: \_\_\_\_\_ Note that yellow or yellow-green indicate  $\text{Fe}^{2+}(\text{aq})$ .

Identify the half-reaction that produced this species: \_\_\_\_\_

Add a drop of phenolphthalein indicator to the area around #2 nail, and record color change: \_\_\_\_\_. You have used phenolphthalein before; a pink color is indicative of what ion? \_\_\_\_\_.

Identify the half-reaction that produced this species: \_\_\_\_\_

Now, rinse, thoroughly dry and re-weigh each nail on the **same analytical balance**. Record final masses:

nail #1 (marked): \_\_\_\_\_ g                      nail #2: \_\_\_\_\_ g

## POST-LAB QUESTIONS

(1) Calculate the mass lost by the Zn and Fe metals in the “unconnected” experiment (Part 2). Comment on the differences. Why did one metal corrode (lose mass) more than the other?

(2) Calculate the mass loss for each metal (Zn and Fe) in the “connected” experiment (Part 3). How do the mass differences demonstrate the concept of sacrificial anode or galvanic protection?

(3) In Part 4 you connected each nail to the + or – terminals of the power supply. Calculate the mass loss for each nail.

Mass loss #1 (marked) : \_\_\_\_\_ (+ terminal)

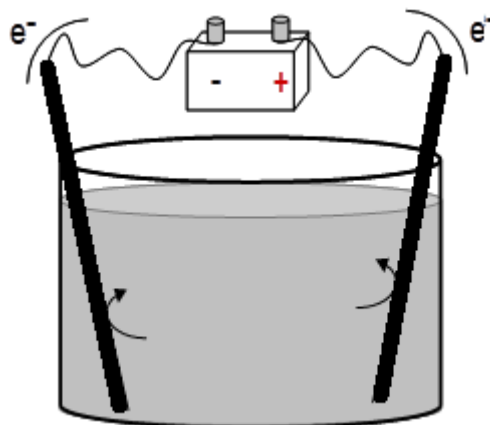
Mass loss #2: \_\_\_\_\_ (– terminal)

Does this make sense? Explain the observed changes in terms of oxidation and reduction processes.

Describe the “impressed voltage” cell of Part 4. Fill in half-reactions, add heads to upper arrows in the figure to show direction of electron flow, label electrodes as #1 or #2. (NOTE- pay attention to polarity at the power supply!)

nail (electrode) # \_\_\_\_\_

half-reaction:  
\_\_\_\_\_



nail (electrode) # \_\_\_\_\_

half-reaction:  
\_\_\_\_\_

Name \_\_\_\_\_

Section \_\_\_\_\_

Date \_\_\_\_\_

**Pre-Lab Exercises**  
**Experiment 21H**

1 a. The type of corrosion which results when battery acid is spilled on a wrench is:

- i. atmospheric corrosion      ii. chemical corrosion      iii. pitting corrosion      iv. stress corrosion

b. The type of corrosion which results when a wrench is left in damp grass is:

- i. atmospheric corrosion      ii. chemical corrosion      iii. pitting corrosion      iv. stress corrosion

2. Common methods to limit or prevent corrosion include (1) use of corrosion-resistant materials; (2) applying impervious coatings; (3) galvanic protection; and (4) impressed voltage. Which method best describes the following circumstances?

a. Gold (Au) is used for electrical contacts.

- i. corrosion-resistant material      ii. impervious coating      iii. galvanic protection      iv. impressed voltage

b. The hull is kept attached to a power supply when in port.

- i. corrosion-resistant material      ii. impervious coating      iii. galvanic protection      iv. impressed voltage

c. The bulkhead is painted gray.

- i. corrosion-resistant material      ii. impervious coating      iii. galvanic protection      iv. impressed voltage

d. A metal can is plated with tin (Sn).

- i. corrosion-resistant material      ii. impervious coating      iii. galvanic protection      iv. impressed voltage

3. In the corrosion of iron, the iron would act as the \_\_\_\_\_ and have a \_\_\_\_\_ electrode sign (polarity).

- i. anode\_\_positive      ii. anode\_\_negative      iii. cathode\_\_positive      iv. cathode\_\_negative

4. In the corrosion of iron, the rust that forms will often appear \_\_\_\_\_. (Check the Naval Applications module [https://intranet.usna.edu/ChemDept/\\_files/documents/navapps/ADDITIONAL-INFO/Corrosion%20Chapter.pdf](https://intranet.usna.edu/ChemDept/_files/documents/navapps/ADDITIONAL-INFO/Corrosion%20Chapter.pdf) if you are not sure.)

- i. at the site of the anode      ii. at the site of the cathode      iii. somewhere between anodic and cathodic regions  
iv. only when water is excluded.