Experiment 21A

WBH 04/6/2023

FARADAY'S LAW

MATERIALS: Digital ammeter, J-shaped platinum electrode and holder, carbon electrode, electrical leads (3), 10 mL graduated cylinder, 10 mL graduated centrifuge tube, 50 mL beaker (2), 400 mL beaker, buret, thermometer, starch-KI paper, starch dropper, 0.20 M KI, 1 M H_2SO_4, 0.0200 M $Na_2S_2O_3$.

PURPOSE: The purpose of this experiment is to determine values for the Faraday constant and Avogadro's number.

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

1. Construct an electrolytic cell from a diagram.
2. Determine the number of moles of products formed in a redox reaction from experimental data.
3. Determine the total charge that has passed through an electrolytic cell.
4. Calculate values for the Faraday constant and Avogadro's number from experimental data.

DISCUSSION:

Forcing an electrical current through an electrolytic cell can cause a nonspontaneous chemical reaction to occur. For example, when direct current is passed through a solution of aqueous potassium iodide, KI, the following reactions occur at the electrodes:

anode:	$2\ I^- (aq) \rightarrow I_2 (aq) + 2\ e^-$	(oxidation)
cathode:	$2\ H_2O (l) + 2\ e^- \rightarrow H_2 (g) + 2\ OH^- (aq)$	(reduction)
overall redox:	$2\ I^- (aq) + 2\ H_2O (l) \rightarrow I_2 (aq) + H_2 (g) + 2\ OH^- (aq)$	

Electrons can be treated stoichiometrically like the other chemical species in these reactions. Thus, the number of moles of products formed is related to the number of moles of electrons that pass through the cell during the electrolysis. Iodine is formed at the anode in this electrolysis and dissolves in the solution upon stirring.[1] Hydrogen gas is formed at the cathode and will be collected in an inverted graduated cylinder by displacement of water. In this reaction, because the same number of electrons must pass through each electrode, the number of moles of iodide ion oxidized at the anode must equal the number of moles of water reduced at the cathode (i.e., in redox equations, electrons gained = electrons lost). Thus, equimolar quantities of hydrogen gas (H_2) and molecular iodine (I_2) will be produced by the electrolysis.

The current, or rate of flow of electricity, is measured in amperes, A. The ampere is the SI unit of current and corresponds to 1 coulomb of charge flowing for 1 second. Therefore, the total charge passing through the circuit, in coulombs, is equal to the product of the current in amperes and the time of current flow in seconds.

$$C = A \times t \text{ (in sec)}$$

[1]Iodine reacts with water according to the equilibrium below.

$$I_2 (aq) + H_2O (l) \leftrightarrow HIO (aq) + H^+ (aq) + I^- (aq)$$

For this reason, the pH of the solution must be adjusted to ensure that I_2 is the predominant species in solution. For a more detailed explanation see: a) I. M. Kolthoff and R. Belcher, Volumetric Analysis, New York, Interscience (1957), Vol. 3, pp 214-215 and b) W. C. Bray and E. L. Connolly, *J. Am. Chem. Soc.* **33** (1911), 1485.

In this experiment the following quantities will be obtained: (1) the total charge that has passed through the cell obtained from the average current and the elapsed time; (2) the number of moles of H_2 determined from the volume of gas collected; and (3) the number of moles of I_2 found by titration of the iodine with sodium thiosulfate ($Na_2S_2O_3$). The number of moles of electrons that have passed through the cell can be obtained from the moles of hydrogen or the moles of iodine using stoichiometry. The total charge in coulombs that has passed through the cell can be obtained from the average current data. The value of the Faraday constant (F) can then be calculated from the total charge used in the electrolysis and the number of moles of electrons. Remember that 1 F is the electric charge, in coulombs, on 1 mole of electrons.

Avogadro's number can also be determined using the following procedure. The value of Avogadro's number is the number of units in one mole. For the electrolysis, the required quantities for this calculation are: (1) the number of electrons and (2) the moles of electrons flowing through the electrolytic cell. As before, the moles of electrons can be determined stoichiometrically. The number of electrons can be determined from the total charge used, knowing that the charge on a single electron is 1.60×10^{-19} coulombs.

PROCEDURE: *Inspect all the glassware for cracks and other defects prior to starting the experiment. Replace all defective glassware.*

Part A. Electrolysis and Quantity of Electrical Charge Used.

1. Before you connect anything to the power supply, turn both knobs to the left (fully counterclockwise). Turn on the power supply and adjust the lower knob (current) until the red LED just goes out. Then adjust the upper knob (voltage) until the window reads 9.0 V. Turn off the power supply main switch and connect the wires.

2. Arrange the apparatus as indicated in Figure 1. The negative of the power supply is connected to the COM port of the meter. The J electrode is connected to the uA-mA port of the meter. *Do not turn on the power supply again until your instructor has checked the electrical connections.*
   The carbon rod is the anode and the platinum J-electrode is the cathode. Platinum serves both as an electrical conductor and as a catalyst for the formation of hydrogen gas.

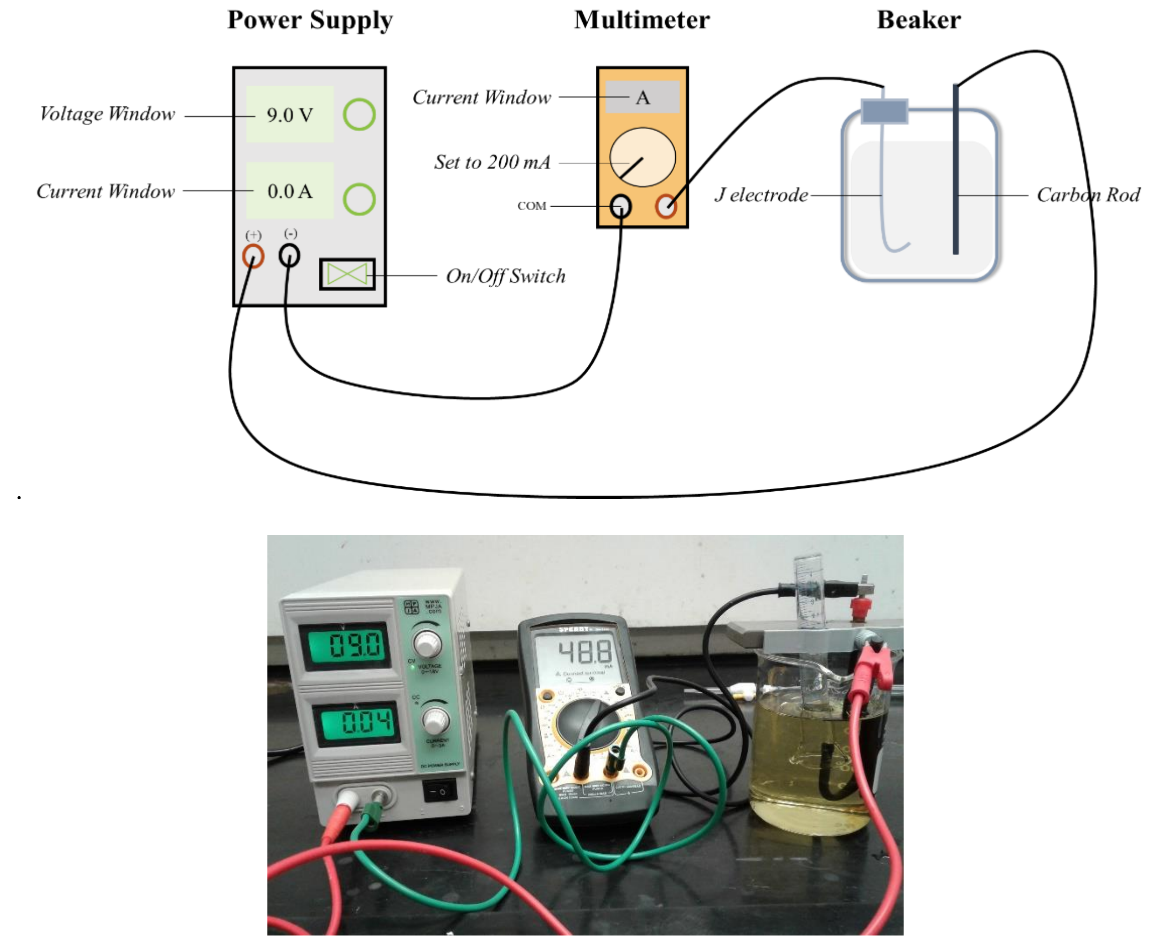

Figure 1. Diagram of electrolysis apparatus (schematic and picture)

3. Fill a 400 mL beaker half full with 0.20 M KI solution.

4. Completely fill a 10 mL graduated cylinder with the same KI solution and place it mouth-downward over the J-shaped electrode. Be sure *no air bubbles* are present in the inverted graduated cylinder. (You may want to practice inverting the graduated cylinder in some water near the sink until you perfect the technique.)

5. When the circuit has been properly constructed, place the electrodes into a 400 mL beaker as shown in Figure 1. Make sure that bubbles, which will form at the tip of the J-electrode will be collected **within** the inverted graduated cylinder

Have your instructor check your apparatus before continuing. ____________ Instructor's initials______

6. **Set the ammeter to the 200 mA DC setting (lower left side of the dial).** Turn on the power supply and record the initial starting time. Record the current, including units, at one-minute intervals in the DATA SECTION. *Read the current from the METER display, not from the power supply window.* Stir the solution frequently with the carbon electrode to dissolve the iodine, which deposits on the electrode. Record your observations of the reaction in the cell in the DATA SECTION.

7. When approximately 7 to 8 mL of hydrogen gas has been collected in the inverted graduated cylinder, turn off the power supply and record the time. Note that the gas level must be below the liquid level in the beaker but still on the scale of the graduated cylinder.

Part B. Moles of H_2 formed.

1. To equalize the pressure of the hydrogen gas inside the graduated cylinder with atmospheric pressure outside the beaker, raise or lower the graduated cylinder until the liquid levels inside the cylinder and in the beaker are the same. By carefully reading the scale on the *inverted* graduated cylinder, record the volume of hydrogen gas collected in the DATA SECTION.

2. Record the temperature of the solution and the barometric pressure in the DATA SECTION. Obtain the vapor pressure of water at this temperature from an appropriate reference source, such as the CRC Handbook. Record this value in the DATA SECTION, being sure to include units.

Part C. Moles of I_2 formed.

1. Remove the graduated cylinder and the J-electrode from the beaker. Rinse each into the beaker with a little distilled water. Do not remove the J-electrode from the wooden block.

2. Add 10 mL of 1.0 M sulfuric acid (H_2SO_4) to the solution in the beaker.

3. Fill a buret with standard sodium thiosulfate ($Na_2S_2O_3$). Record the concentration (provided by instructor) and initial buret reading in the DATA SECTION.

4. Using the carbon electrode as a stirring rod, titrate the solution of I_2 in KI with the $Na_2S_2O_3$ until the color due to I_2 fades to a pale yellow. Then add ***two full droppers*** of starch solution to the solution being titrated. The solution will become dark blue due to the formation of a starch-iodine complex. If not, add more starch.

5. Carefully continue the titration until the blue color just disappears. Record the final buret reading in the DATA SECTION. (Note that the <u>total</u> <u>volume</u> of $Na_2S_2O_3$ solution from the titration is needed, not just the volume after the addition of starch.)

6. Do a second trial of the entire procedure if so directed by your instructor. Start completing the calculations on page E21A-6.

Clean-up:

1. With the plug disconnected, disassemble the circuit.

2. All aqueous solutions may be disposed of in the sink. Clean all glassware items and return them to their proper locations.

Name ________________________________ Section

Partner ______________________________ Date

DATA SECTION
Experiment 21A

Report all data with the proper number of significant figures and units.

Part A. Electrolysis and Quantity of Electrical Charge Used.

Trial 1

Initial Time _____________

Final Time _____________

Time	Current

Trial 2

Initial Time ______________

Final Time ______________

Time	Current

Average current (A) = ____

Total elapsed time (s) = ____

Observations of reaction: ____

Part B. Moles of H_2 formed.

	Trial 1	Trial 2
Volume of H_2 (g) collected		
Temperature of the solution (ºC)		
Barometric Pressure		
Vapor pressure of H_2O (g) at ________ (ºC)		
Pressure of Dry H_2 (g)		

Part C. Moles of I_2 formed.

	Trial 1	Trial 2
Initial buret reading (mL)		
Final buret reading (mL)		
Volume of Sodium Thiosulfate used (mL)		
Concentration of Sodium Thiosulfate (M)		

DATA TREATMENT
Experiment 21A

Show your work for all calculations. Include the proper number of significant figures and units in your final answers. Try to complete these calculations before you leave lab.

Part A. Electrolysis and Quantity of Electrical Charge Used.

(A.1) Using the average current and the total elapsed time for the electrolysis, calculate the total electrical charge (*i.e.*, number of coulombs) that passed through the cell during the electrolysis.

Trial 1: ____________________________ Trial 2: ____________________________

Part B. Moles of H_2 formed.

(B.1) From the volume of H_2 gas collected and the pressure of the *dry* H_2, use the Ideal Gas Law to calculate the number of moles of hydrogen gas formed during the electrolysis.

Trial 1: ____________________________ Trial 2: ____________________________

Part C. Moles of I_2 formed.

(C.1) From your $Na_2S_2O_3$ titration data and the reaction stoichiometry, calculate the number of moles of iodine formed during the electrolysis.

titration reaction: $\underset{\substack{\uparrow \\ \text{electrolysis} \\ \text{product}}}{I_2\ (aq)} + 2\ \underset{\substack{\uparrow \\ \text{titrant}}}{Na_2S_2O_3\ (aq)} \rightarrow 2\ NaI\ (aq) + Na_2S_4O_6\ (aq)$

Trial 1: ____________________________ Trial 2: ____________________________

Part D. Calculating the Value of the Faraday.

(D.1) From the moles of hydrogen gas formed at the cathode and the appropriate reaction stoichiometry on page E21A-1, calculate the moles of electrons that passed through the cell during the electrolysis.

Trial 1: ____________________________ Trial 2: ____________________________

(D.2) From the moles of iodine formed at the anode and the appropriate reaction stoichiometry on page E21A-1, calculate the moles of electrons that passed through the cell during the electrolysis.

Trial 1: ____________________________ Trial 2: ____________________________

(D.3) Using the values from (D.1) and (D.2), calculate the *average* number of moles of electrons that passed through the cell.

Trial 1: ____________________________ Trial 2: ____________________________

(D.4) Use the *average* moles of electrons that passed through the cell and the *total* charge that passed through the cell to calculate the Faraday, i.e., the number of coulombs per mole of electrons.

Trial 1: ____________________________ Trial 2: ____________________________

(D.5) Calculate the percent error between the value of the Faraday determined in this experiment and the accepted value. (The source for the accepted value must be properly referenced.)

Trial 1: ____________________________ Trial 2: ____________________________

Reference: ____________________________________

Part E. Calculating the Value of Avogadro's Number.

(E.1) Use the charge on a single electron, as determined by Millikan, and the total charge that passed through the cell to calculate the number of electrons used during the electrolysis.
(Charge on a single electron = 1.60×10^{-19} C/electron)

Trial 1: ____________________________ Trial 2: ____________________________

(E.2) From the number of electrons and the average number of moles of electrons (from (D.3)), calculate a value for Avogadro's number.

Trial 1: ____________________________ Trial 2: ____________________________

(E.3) Calculate the percent error of this value from the accepted value of Avogadro's number.

Trial 1: ____________________________ Trial 2: ____________________________

QUESTIONS
Experiment 21A

1. Explain why the moles of electrons calculated in (D.1) and (D.2) should be approximately equal.

2. The moles of electrons that passed through the cell during the electrolysis were calculated from two different measurements in (D.1) and (D.2). Which method of measurement might have more sources of experimental error? Explain. (In your explanation, consider the types of glassware and the techniques involved.)

3. How would the calculated value of the Faraday be affected (larger or smaller) if the pressure of the hydrogen gas were not corrected for the presence of the water vapor? Support your answer (mathematically or otherwise).

Name ____ Section _________

Date ___________

Pre-Lab Exercise
Experiment 21A

1. In this experiment, you will be studying the electrolysis reaction occurring in an aqueous solution of potassium iodide:

$$2\ I^{-}\ (aq) + 2\ H_2O\ (l) \rightarrow I_2\ (aq) + H_2\ (g) + 2\ OH^{-}\ (aq)$$

   a. Using Table A6.1 (on page APP-30 in the textbook), determine the standard cell potential for this reaction.

   b. Is this reaction spontaneous as written (under standard conditions)? YES NO

   c. If the electrolysis process was carried out for 12 minutes at 120 mA, what was the total charge (in C) that passed through the cell?

2. Hydrogen gas is collected during the electrolysis of aqueous potassium iodide. If 9.00 mL of gas was collected *over water* at standard temperature and pressure, how many moles of hydrogen gas were collected? The vapor pressure of water at this temperature is 23.8 mm Hg. *Assume ideal gas behavior.*
(For a review of collecting a gas over water, see page 425 of your textbook.)