

Experiment 21A

FV 08/03/17

FARADAY'S LAW

MATERIALS: Digital ammeter, J-shaped platinum electrode and holder, DC power supply, carbon electrode, electrical lead with alligator clips at ends, 10 mL graduated test tube, 100 mL beaker, 600 mL beaker, buret, thermometer, starch solution, 0.20 M KI, 1 M H₂SO₄, 0.0200 M Na₂S₂O₃.

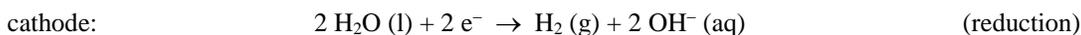
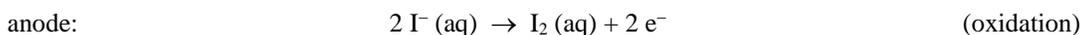
PURPOSE: The purpose of this experiment is to introduce quantitative electrolysis and determine values for the Faraday constant and Avogadro's number.

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:



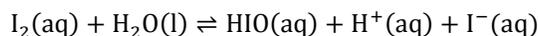
That the process is nonspontaneous (under standard conditions) can be confirmed by calculating the standard cell potential, E°_{cell} . For this system $E^{\circ}_{\text{cell}} = -1.36 \text{ V}$; the negative value indicates a nonspontaneous process.

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 (H₂) and molecular iodine (I₂) 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.

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

¹Iodine reacts with water according to the equilibrium below.



For this reason, the pH of the solution must be adjusted to insure that I₂ 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 ($\text{Na}_2\text{S}_2\text{O}_3$). Because of the overall stoichiometry, with 1 mole $\text{H}_2 = 1$ mole $\text{I}_2 = 2$ mol e^- , the number of moles of electrons that have passed through the cell can be obtained independent of the use of the Faraday constant. Using this experimental value of the number of moles of electrons, and the total charge in coulombs obtained from current and time measurements, an experimental value of the Faraday constant can be determined. 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:

Part A. Electrolysis and Quantity of Electrical Charge Used.

1. Arrange the apparatus as indicated in Figure 1. *Leave the power supply OFF and unplugged until your instructor has checked the electrical connections.*

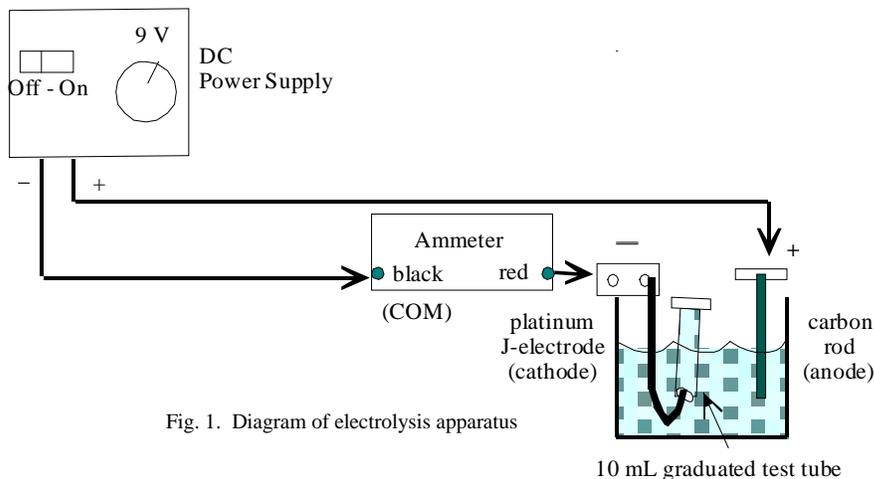


Fig. 1. Diagram of electrolysis apparatus

2. 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. Since it is the source of electrons for the reduction, it is attached (indirectly) to the negative side of the power supply. Current measurements require that the meter be placed *in* the circuit as shown. (This differs from the voltage measurements of other electrochemical experiments, where the meter leads are placed *across* the circuit.) Use the alligator clips on the electrical leads to make your connections. If necessary, *partly* unscrew the binding posts on the power supply to ensure that the alligator clips make good contact with the metal posts. For gripping the carbon rod, attach the alligator clip vertically, right at the top of the rod.
3. Remove the electrodes and fill the 600 mL beaker about three-quarters full with 0.20 M KI solution.
4. Completely fill a 10 mL graduated test tube with the same KI solution and place it mouth-downward in the beaker of KI solution. (Use a finger or thumb to seal the opening when you immerse the test tube. The KI salt solution is not harmful. You may want to practice inverting the graduated tube in some water near the sink until you perfect the technique.) Be sure no air bubbles are present in the inverted tube.
5. When the circuit has been properly constructed, place the platinum J-electrode and holder on top of the 600 mL beaker as shown in Figure 1. Loosen the white plastic screw and move the test tube clamping bar so that the inverted test tube can be placed in the recess of the clamp that is nearest to the J electrode. Adjust the position of the graduated test tube such that the mouth is just at the end of the Pt strip where the Pt meets the black insulation of the electrode. Roughly center the test tube mouth on the electrode and tighten the white screw just until the test tube is firmly held in place. Make sure that bubbles which will form at the tip of the J-electrode will be collected within the inverted cylinder. Place the carbon electrode in the beaker; do not insert it into the other recess of the clamping bar, as you will need to have it free for stirring.

Have your instructor check your apparatus before continuing.

_____ Instructor's initials

6. Set the ammeter to the mA or 200 mA setting, depending on the meter. Simultaneously close the switch and record the initial starting time. Record the current, including units, at one-minute intervals in the DATA SECTION. (Typical currents are ~100 mA or ~0.100 A.) 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 test tube, open the switch and record the time at the nearest 1 minute mark. Note that the gas level must be below the liquid level in the beaker but still on the scale of the graduated tube.

Part B. Moles of H₂ formed.

1. To equalize the pressure of the hydrogen gas inside the graduated test tube 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 tube, 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. Be sure to interpolate values if necessary. Record this value in the DATA SECTION, being sure to include units.

Part C. Moles of I₂ formed.

1. Remove the graduated test tube 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 support.
2. Add 10.0 mL of 1.0 M H₂SO₄ to the solution in the beaker.
3. Fill a buret with standard 0.0200 M Na₂S₂O₃. Record the initial buret reading in the DATA SECTION.
4. Using the carbon electrode as a stirring rod, titrate the solution of I₂ in KI with the Na₂S₂O₃ until the color due to I₂ fades to a paler yellow. (Do NOT let it go colorless.) 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 total volume of Na₂S₂O₃ 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 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

Trial 2

Initial Time _____

Initial Time _____

Final Time _____

Final Time _____

Current meter units: _____

Current meter units: _____

Average current (A) = _____

Average current (A) = _____

Total elapsed time (s) = _____

Total elapsed time (s) = _____

Observations of reaction: _____

Part B. Moles of H₂ formed.

	<u>Trial 1</u>	<u>Trial 2</u>
Volume of H ₂ (g) collected (mL)	_____	_____
Temperature of the solution (°C)	_____	_____
Barometric pressure (torr)	_____	_____
Vapor pressure of H ₂ O (g) at _____ °C (torr)	_____	_____
Pressure of <i>Dry</i> H ₂ (torr)	_____	_____

Part C. Moles of I₂ formed.

Initial buret reading (mL)	_____	_____
Final buret reading (mL)	_____	_____
Volume of 0.0200 M Na ₂ S ₂ O ₃ used (mL)	_____	_____

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: _____

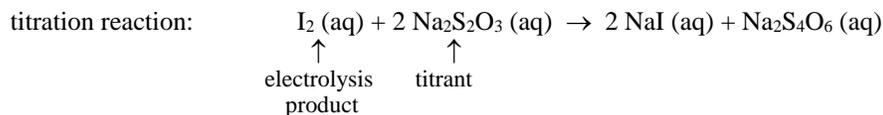
Part B. Moles of H₂ formed.

- (B.1) From the volume of H₂ gas collected and the pressure of the *dry* H₂, 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 I₂ formed.

- (C.1) From your Na₂S₂O₃ titration data and the reaction stoichiometry, calculate the number of moles of iodine formed during the electrolysis.



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 (A.1) 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, $1 F = 96485 \text{ C/mol } e^-$.

Trial 1: _____ Trial 2: _____

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 (E.1) 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,
 $N_A = 6.022 \times 10^{23} \text{ mol}^{-1}$.

Trial 1: _____ Trial 2: _____

