

# **Teacher's Preparatory Guide**

# Electro-deposition of Copper on a Filter Template for Growing **Nanowires**

**Overview:** The electrochemical deposition process dates back to Alessandro Volta in the 1800s. With the advancement of modern technology, nanomaterial structures such as nanowires and nanotubes can be fabricated using the same electro deposition procedure. By applying a magnetic field, these nanowires can exhibit different behaviors based on differences at the nanoscale. Nanowires can have many practical applications such as vehicles for drug delivery and biological sensors for detecting glucose levels in diabetics. Also nanowires can conduct electricity faster in the materials used for computer chips and magnetic data storage systems, making the cost lower and the performance higher in all digital devices. General information on nanowires can be found at the end of the lesson.

**Purpose:** This lab is designed to help students understand the electro-plating process of growing nanowires. This experiment will use an electro-plating process to transfer metal ions in a solution to a template to produce nanowires. The electric field in the solution carries these ions and deposits them onto an electrode. In this experiment, an anode (platinum strip) will be used in a solution of copper sulfate. Copper will be plated out onto the pores of a filter at the cathode. Students will discover how nanowires can be created to further understand the reductionoxidation process, Ohm's law, and the mechanism of metal electroplating.

**Time Required:** The time required for this the lab is two 55-minute class periods on two different days, plus one pre-lab class and one post-lab group presentations class.

Level: Undergraduate chemistry and high school/AP Chemistry

**Big Ideas:** Structure of Matter; Forces and Interactions

**Teacher Background:** In this experiment, students will determine the amount of copper plated out from a solution of copper (II) sulfate into a filter template, forming copper nanowires. Using the calculated quantity of copper metal, students will predict the size of the nanowires formed, then compare with observations of the actual samples they made.

The formula of copper (II) sulfate is CuSO<sub>4</sub>. When this ionic substance dissolves in water, it dissociates into the Cu<sup>2+</sup> cation and the SO<sub>4</sub><sup>2-</sup> anion. A current, supplied by a dry cell battery, will be passed through the CuSO<sub>4</sub> solution. At the cathode, copper cation will be reduced to copper metal:

$$Cu^{2+}(aq) + 2e - \longrightarrow Cu(s)$$

Ouantitative measurements of copper deposited at the cathode indicate that there is a relationship that exists between the amount of charge passed through the anode and the chemical change at the electrodes. Based on Faraday's law, the mass of a substance produced by an electrode

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reaction in electrolysis is directly proportional to the quantity of electricity passed through the cell, and the masses of different substances produced by the same quantity of electricity are proportional to the equivalent weights of the substances.

The coulomb (symbol: C) is the SI unit of electric charge. It is defined as the charge transported by a steady current of one ampere (A) in one second (s):

$$1C = 1A \times 1s$$

The elementary charge, usually denoted as e, is the electric charge carried by a single proton, or equivalently, by a single electron. This charge has a measured value of approximately  $1.602176565(35) \times 10^{-19}$  coulombs.

The mole, abbreviated mol, is an SI unit equal to 6.022 x 10<sup>23</sup> atoms, or other elementary units such as molecules. For this experiment we want to deposit copper for about 5 minutes using a deposition current of about 0.5 amp. The numbers may vary among individual students, but that variation is okay; by precisely measuring both quantities, students will be able to accurately calculate the amount of copper deposited, and using knowledge of nanowire geometry, they will predict the final size of copper nanowires they have grown.

The calculation is done in the following steps. First, we calculate the amount of charge that has passed through the solution, and convert to the number of charged ions. The quantity of electricity that has passed through a solution may be determined by keeping the current constant at some measured value (0.5 amps, in this example) and recording the total time that the electrolysis is conducted (5 min in this case). The net amount of charge delivered (in coulombs) is then calculated by multiplying the current (in amps, a.k.a. coulombs/sec) by the total elapsed time (in seconds).

Number of coulombs  $C = Amperes (A) \times seconds (s)$  $0.5A \times 300 \text{ s} = 150 \text{ coulomb}$ 

This can be converted to electronic charges as follows:  
Number of electron charge 
$$e^- = \frac{150 \ Coulomb}{1.602 \times 10^{-19} \ Coulomb/charge} = 9.36 \times 10^{20}$$
 charges

A convenient unit in electrochemistry is the Faraday (or *mole electron*), defined as the amount of charge (in coulombs) represented by one mole of electrons. This can be calculated by the following.

$$1.602 \times 10^{-16} Coulomb/e^{-} \times \frac{6.022 \times 10^{23}}{mole\; e-}\; e^{-}$$

$$=\frac{96,485 \text{ Coulomb}}{\text{mole } e^-} = 1 \text{ Faraday} = 1 \text{ mole electron}$$

So for our example of 150 Coulombs we can convert to the number of Faraday:

Number of Faraday = 
$$\frac{1 \ mole \ e^{-}}{1} \times \frac{150 \ Coulomb}{96485 \ coulomb} \times \frac{1 \ mole \ of \ Cu}{2 \ mole \ of \ e^{-}}$$

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This tells us how many moles of copper that this much charge will chemically react with. The last term tells us that two moles of electrons are required to react with one mole of Cu<sup>2+</sup>, since the copper ion is doubly positive charged.

The result of this equation is 0.0000773 mole of Cu. That is, our 150 Coulombs of charge will react with 0.0000773 moles of copper during the electro-plating process. This amount of copper will be electro-deposited at the cathode and into the pores of the Whatman filter that we are using as a deposition template. The filter pores are cylindrical, so the deposited copper nanowires will be too. We know the diameter of the pores (and resulting wires), but nanowire length will depend on how much copper we deposit.

To predict the size of nanowires, we'll need to know the total volume of copper deposited.

Using the molecular weight for Cu (63.5 g/mole) and the mass density (89.6g/cm<sup>3</sup>) we can calculate the mass and the volume of the deposited copper.

Mass of Cu deposit:

$$= \frac{0.000773 \, mole \, of \, Cu}{1} \times \frac{63.5 \, g}{1 \, mole \, of \, Cu} = 0.04936 \, g \, of \, copper \, deposited$$

Since mass density is equal to mass/volume, volume is equal to mass/ density. Then the total volume of Cu deposited is

Volume (cm<sup>3</sup>) = 
$$\frac{Mass(g)}{Density(g/cm^3)} = \frac{0.04936 g}{89.6 g/cm^3} = 5.5089 \times 10^{-4} cm^3 = 0.55089 \text{ mm}^3$$

This total volume of copper will be formed into many nanowires. To calculate the nanowire length, assume the following:

- a. Each filter is 25mm in diameter, so total area of the filter face is 490.9mm<sup>2</sup>.
- b. Each filter has a porosity of 50%, meaning the half face area is made up of pores that copper will deposit into.
- c. Assuming the copper plates evenly inside the pores, the expected length of each nanowire will be:

$$L = \frac{(total\ volume\ deposited)}{(Total\ pore\ area\ available)}$$

**Activity:** Students will perform the electroplating activity as described in the attached procedure. Different groups of students should use slightly different deposition times varying between 3 and 5 minutes. Each student measures and records the plating current (in amps or milliamps) and the total time elapsed during plating (in seconds). They then perform the calculation described above to make a prediction of copper nanowire length. Finally, they suspend their copper nanowires in a liquid and view them under a microscope to determine nanowire length.

## **Sources:**

1. "Fabrication and magnetic behavior of Co/Cu multilayered nanowires", Tan, L. and B. Stadler. *Journal of Materials Research*, v 21(11), 2006, pp 2870-2875.

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- 2. "Electrochemical synthesis of magnetostrictive Fe-Ga/Cu multilayered nanowire arrays with tailored magnetic response", Reddy, S. M., Park, J. J., Na, S-M, Maqableh, M.M., Flatau, A.B., Stadler, B.J.H. *Advanced Functional Materials*, v 21(2011), p 4677-4683.
- 3. Electrodeposition: A Technology for the Future, by Walther Schwarzacher. http://www.electrochem.org/dl/interface/spr/spr06/spr06 p32-35.pdf

# **Materials and Equipment:**

## Materials

- Copper sulfate electrolyte solution
- Whatman alumina membrane filters (20 nm pore size, 25mm diameter)
- Copper tape
- Platinum-coated electrode (available through Amazon)
- Nail polish
- 10 ml 1.0 M sodium hydroxide
- 10ml 1.0M ethanol

# Equipment

- Safety goggles
- Nitrile gloves
- One pair of tweezers
- Power supply or battery (6.0-9.0 volts, capable of passing 0.60-1.0 amps)
- Two connecting wires with alligator clips
- 16-18 gauge copper wire
- Beakers: 25-mL; 50-mL; 250-mL
- Vial (~20mL)
- pipet
- Cotton swaps
- Plastic container to hold filter
- Hot stir plate to agitate the solution
- Cardboard square (approx. 15 cm on a side)
- Ammeter or multimeter
- Stopwatch
- Optical microscope
- Optical reticule (sometimes called a "stage micrometer") for the microscope, so that objects with lengths in the 2-100 micrometer range can be measured accurately.
- Optional: desktop SEM
- Optional: sonicator
- Optional: glass slide

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#### **Sources for Materials:**

Source/Website	Material
Sigma Aldrich Company www.sigmaaldrich.com	• Whatman Anodisc Al <sub>2</sub> O <sub>3</sub> Membrane Filter (20 nm pore size-25mm dia) Cat. No: Z694959
Ted Pella, Inc. www.tedpela.com	3M <sup>TM</sup> Copper Conductive Tapes Single Adhesive Surface
Household Goods or Drug Store	<ul><li>Battery</li><li>Nail polish</li><li>Cotton swabs</li><li>Acetone nail polish remover</li></ul>

# **Advance Preparation**

# 1. Prepare 1M copper sulfate solution

The molar mass of  $CuSO_4$  -  $5H_2O$  (copper sulfate pentahydrate ) is 249.68 g/mol. For a one molar solution, dissolve 124.08 of the pentahydrate salt in 500ml of water to produce 500 ml of 1.0 M of  $CuSO_4$ .

## 2. Prepare 1M sodium hydroxide solution

The molar mass of NaOH is 40g. Dissolve 2.0 g of solid NaOH in 50 ml of water to produce 50 ml of 1.0 M of NaOH

Safety Information: This lab makes use of a strong base solution (sodium hydroxide) and a volatile organic solvent (acetone). All these chemicals must be handled with care. Safety eyeware (full chemical goggles, not safety glasses) and nitrile gloves (not latex gloves) must be worn when handling the base and/or the solvent. Acetone should be handled under a fume hood if available, or in an area with good circulation, avoiding direct inhalation of the vapor. Before disposal, the base may be neutralized with a weak acid like vinegar. To neutralize a base with an acid (or vice versa), dilute the more concentrated solution with tap water first, then mix with the base and acid before disposal.

**Instructional Procedure:** This lab activity is divided into two periods so students can prepare and understand the concept of growing nanowires using the electroplating technique.

Time	Activity	Goal
Day 1	The day before the lab	
30 min	Introduce students to the topic of electrochemical plating, electrolysis, redox chemical reaction, and Ohm's law	To prepare students' understanding of oxidation-reduction half equations.  Calculation of the number electron

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		moles used in the reaction and the amount of copper metal deposited.
Day 2	The day of the student lab	
10 min	Students answer warm-up questions directed from the teacher	To ensure students understand the concept of electro chemical plating
30 min	Distribute <i>Student Worksheets</i> to students. Students follow the written procedure.	To allow students to work together to set up lab apparatus and carry out the experiment.
15 min	Clean up, make sure students dispose of chemicals properly and put their final product in a safe place.	To prepare workspace for next class.

# **Teaching Strategies:**

Students may need assistance visualizing the copper deposition process, especially the geometry of the filter and how it acts as template to form long thin copper wires. A diagram such as the one below can be used to discuss this deposition process, and what it is that the students are expected to calculate.

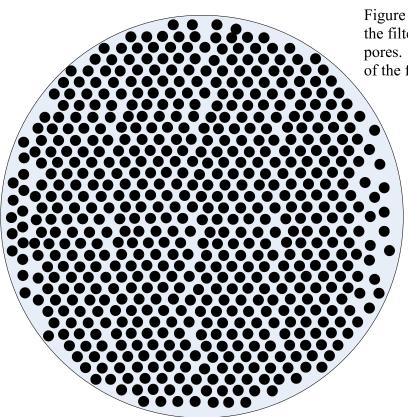


Figure 1. Idealized face view of the filter. The black dots are the pores. About 50% of the total area of the filter is covered by the pores

Figure 2. Actual microscopic view of the filter face

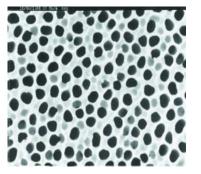


Figure 3. Cross sectional view of the filter, showing each pore partially filled with copper metal.

The math involved in converting the measured quantities (current, time) to those we need (volume of copper deposited) is straightforward, but students may need several run-throughs to understand each step and why they are employed. Be prepared to map this out as a flow chart on the classroom black/white board.

**Guided Dialog:** Before beginning the lab, review the meaning of these terms:

**Electrochemical plating** Electroplating is the application of electrolytic cells in which a thin layer of metal is deposited onto an electrically conductive surface. The current through the circuit is such that the rate at which the anode is dissolved is equal to the rate at which the cathode is plated.

**Nanowire** Solid rods of silicon, copper, nickel, or other materials that are only a few nanometers wide are called nanowires. A nanowire's length is much greater than its width and it behaves like a wire in which electrons can move, thus conducting an electric current. Nanowires can be used for many practical applications.

**Redox** Redox reactions are those dealing with the transfer of electrons between elements or molecules. Oxidation refers to the loss of electrons, while reduction refers to the gain of electrons.

**Electrocharge** There are  $6.022 \times 10^{23}$  elementary particles in a mole. If you had a mole of hydrogen atoms you would have  $6.022 \times 10^{23}$  electrons. If you had mole of neon (atomic number=10) you would have  $6.022 \times 10^{24}$  electrons (ten times as many, since each neon atom has 10 electrons).

Ask students questions to provoke thought and review what they already know: Can they balance the reduction reaction equation for Cu at the cathode?

## Cathode+

$$CuSO_4 \rightarrow Cu^{2+} + SO_4^2$$
  
 $Cu^{2+} + 2e^- \rightarrow Cu$ 

Next balance the oxidation reaction for Cu<sup>2+</sup> at the anode:

## Anode-

$$Cu \rightarrow Cu^{2+} + 2e^{-}$$
  
 $Cu^{2+} + SO_4^{2-} \rightarrow CuSO_4$ 

**Procedure:** This procedure is based on the unpublished paper titled "Tin Oxide Inverse Opals by Chemical Vapor Deposition" by Sun Sook Lee, Kang Hyun Baek, Anand Gopinath, and Bethanie Stadler from the departments of Electrical and Computer Engineering and Chemical Engineering and Materials Science at the University of Minnesota in Minneapolis, MN.

#### A. First Period

- 1. Cut the Cu tape into pieces about 6 to 8 cm in length.
- 2. Place the Whatman aluminum oxide filter sample near the end of one side of the tape. Apply nail polish as an insulator to cover the edge of the sample and half of the tape both sides and the edges.
- 3. Pour 150 ml of the copper plating solution into a 250 ml beaker. Place a stirring magnet in the beaker and put it on the hot plate.

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Rev: 12/13

- 4. Connect the sample to the one alligator clip. Make sure the unpainted nail polish part does not touch the solution. This sample acts as a cathode (the negative electrode, which attracts positive charges (cations)). The other alligator clip attaches to a platinum metal plate as an anode (the positive electrode, which attracts negative charges (anions)).
- 5. Connect the two ends of the alligator clips to the power source. Begin timing with a stopwatch or other timer.
- 6. Turn the hot plate on, adjust the temperature to warm and observe the experiment for 5 minutes.
- 7. Since the actual current may vary as the reaction proceeds, record the current (in milli amps) at regular 1 minute intervals. Find the mean of these current values. Also record the total reaction time (in seconds) in notebook.
- 8. Take the filter out and rinse with  $H_2O$ . Save the sample in a plastic container.

## **B.** Second Period

To observe and measure the nanowires, students will release the nanowires from the filter and examine the wires under an optical microscope, or if available, a scanning electron microscope.

To prepare the sample for microscopy, do the following.

- 1. Use acetone or nail polish remover to remove the nail polish from the filter.
- 2. Place the sample into a 25 ml beaker and pipet 15 ml ethanol, and leave it for 1 hour
- 3. Transfer the sample to a vial and separate the deposited nanowires using a sonicator or vigorous stirring.
- 4. Remove a small amount of liquid, which contains suspended nanowires, and place on a glass microscope slide, or if an SEM is available, an SEM sample stub. Let the liquid dry for one hour.
- 5. Now the nanowires can be further observed under the optical microscope or SEM. Students will measure the lengths of the 20-30 nanowires using the stage micrometer, and obtain an average wire length to compare with their predictions.
- 6. If insufficient numbers of nanowires are released using the ethanol, the alumina filter may be dissolved to release all the wires. To dissolve the filter, place the filter in 5 mL of 1..0M NaOH and leave for at least 20 minutes. Allow the wires to settle under gravity, pipette up a few drops from the bottom of the suspension, and transfer to a microscope slide. Repeat step 5.

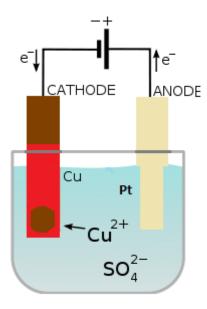


Figure 4. Experimental set up.

**Cleanup:** Transfer the copper sulfate solution to a sealed jar. It may be reused by sealing the jar with paraffin film or a watch glass Place in a safe place. Because of the environmental hazard, do not pour waste liquids in the sink but discard the acetone solution in a waste bottle for proper disposal with other solvents.

**Enhancing Understanding:** After lab activities, show students actual nanowire images (Google images) and compare the sizes of nanowires with small structures from the world of biology. e.g.. cilia of insects, the sound organs of crickets, etc., and explain how nanowires can be used for, among other things, mimicking cilia function in insects.

**Going Further:** Students who have a good grasp of the content of the lab can be further challenged with these questions:

1. What are the metals that can be deposited and what are the applications: *Metals such as zinc oxide, gallium oxide, silica, and tin oxide can be fabricated using a vapor-phase evaporation method*.

An application example: research shows that tin oxide can detect small amounts of gasphase carbon monoxide (CO) and carbon dioxide ( $CO_2$ ), making it a versatile gas sensor.

**Assessment:** Students' understanding will be assessed by

- Students' answers to the provided questions.
- Evaluation of student's calculations and their notes.
- direct observation of students during experiments

## **Resources:**

- Katherine Miller. "Faster nanowires may advance nanotechnological applications for detecting glucose, hormones or DNA" (accessed July, 2012)
- http://www.chem.purdue.edu/gchelp/howtosolveit/Electrochem/Electrolysis.htm
- http://web.centre.edu/che/che11 lab/electrolysis.htm

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- Electrodeposition: A Technology for the Future by Walther Schwarzacher. <a href="http://www.electrochem.org/dl/interface/spr/spr06/spr06">http://www.electrochem.org/dl/interface/spr/spr06/spr06</a> p32-35.pdf
- "Electroplating of metal nanotubes and nanowires in a high aspect-ratio nano template", Jie Fu, Serhiy Cherevko, and Chan-Hwa Chung, Department of Chemical Engineering, Sungkyunkwan University, Suwon 440-746, Republic of Korea (http://ecs.skku.ac.kr/papers/ful.pdf)
- <a href="http://www.nnin.org">http://www.nnin.org</a>
- <a href="http://mrsec.wisc.edu/Edetc/">http://mrsec.wisc.edu/Edetc/</a>

## **Information on nanowires:**

How Nanowires Work: <a href="http://science.howstuffworks.com/nanowire.htm">http://science.howstuffworks.com/nanowire.htm</a>

Nanowires: http://uw.physics.wisc.edu/~himpsel/wires.html

Wikipedia Nanowire: http://uw.physics.wisc.edu/~himpsel/wires.html

Understanding Nano – Nanowire Applications: http://www.understandingnano.com/nanowires-

applications.html

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# **National Science Education Standards (Grades 9-12)**

Content Standard A: Science as Inquiry

• Abilities necessary to do scientific inquiry

Content Standard B: Physical Science

- Structure and properties of matter
- Chemical reactions

## **Next Generation Science Standards**

**HS-PS1.B** Chemical reactions

HS-PS1-7 Use mathematical representations to support claims that atoms, and therefore mas, are conserved during a chemical reaction

Name:	Date:	Class:	
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# **Student Worksheet**

(Teacher comments are in red)

# Electro-deposition of Copper on a Filter Template for Growing Nanowires

# Safety

This lab makes use of a strong base solution (sodium hydroxide) and a volatile organic solvent (acetone). All these chemicals must be handled with care. Safety eyewear (full chemical goggles, not safety glasses) and nitrile gloves (not latex gloves) must be worn when handling these chemicals. Acetone should be handled under a fume hood if available, or in an area with good circulation, avoiding direct inhalation of the vapor.

#### Introduction

This lab is designed to introduce you to the electroplating process of growing nanowires. Nanowires are nanoscale (1x10-9) objects that have large length to width ratios that can be greater than 20 to 1. That have a wide range of applications such as vehicles for drug delivery and biological sensors. This experiment will use an electro plating process to transfer metal ions in a solution to a template to produce nanowires. The electric field in the solution carries these ions and deposits them onto an electrode. In this experiment, an anode (platinum strip) will be used in a solution of copper sulfate. Copper will be plated out onto the pores of a filter at the cathode. You will discover how nanowires can be created to further understand the reduction-oxidation process, Ohm's law, and the mechanism of metal electroplating.

Question: What are the redox reactions at the anode and cathode?

Reduction reaction for Cu at cathode:  $CuSO_4 \rightarrow ?$ 

Oxidation reaction for  $Cu^{2+}$  at anode:  $Cu^{2+} + ? \rightarrow ?$ 

Answer:

<u> At Cathode,</u>

 $\underline{CuSO4} \rightarrow \underline{Cu2} + \underline{SO42}$ 

 $\underline{Cu2++2e-\rightarrow Cu}$ 

<u>At Anode,</u>

 $Cu \rightarrow Cu2 + + 2e$ 

 $Cu2++SO42-\rightarrow CuSO4$ 

## Materials

- Copper sulfate electrolyte solution
- Whatman alumina membrane filters (20 nm pore size, 25mm diameter)
- Copper tape
- Platinum-coated electrode
- Nail polish
- 10 ml 1.0 M sodium hydroxide
- 10ml 1.0M ethanol

# **Equipment**

- Safety goggles
- Nitrile gloves
- One pair of tweezers
- Power supply or battery (6.0-9.0 volts, capable of passing 0.60-1.0 amps)
- Two connecting wires with alligator clips
- 16-18 gauge copper wire
- Beakers 25-mL; 50-mL; 250mL
- Vial
- pipet
- Cotton swaps
- Plastic container to hold filter
- Hot stir plate
- Cardboard square (approx. 15 cm on a side)
- Ammeter or multimeter
- Stopwatch
- Optical microscope

## **Procedure:**

## First period.

- 1. Cut the copper tape into pieces about 6 to 8 cm in length.
- 2. Place the Whatman aluminum oxide filter near the end of one side of the tape. Apply nail polish as an insulator to cover the edge of the filter and half of the tape on both sides and along the edges.
- 3. Pour 150 ml of the copper plating solution into 250 ml beaker. Place a stirring magnet in the beaker and put it on the stirrer/hot plate.
- 4. Connect the copper tape to one alligator clip. Make sure the unpainted portion of the tape does not touch the solution. This tape acts as a cathode (negative electrode, which attracts positive charges (cations)). The other alligator clip attaches to a platinum metal plate as an anode (positive electrode, which attracts negative charges (anions)).
- 5. Connect the cathode to the negative (minus) pole on the power source, using an alligator clip.
- 6. Connect the anode to the negative (minus) pole on the ammeter or multimeter. This is usually black in color. Prepare to start timing.
- 7. Connect the positive (plus) pole on the ammeter (usually red in color) to the positive terminal of the power source. Start timing *immediately* when this connection is made.
- 8. Check to see that the ammeter is measuring a current of around 500 milliamps (mA) or less. If no current is flowing, check your connections and try again.

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- 9. Turn the hot plate on, adjust the temperature to warm and observe the experiment for 10 minutes or as advised by your instructor.
- 10. Record the current (in milliamps) every minute in your notebook. At the end of the deposition, record the total time that current has flowed (in seconds).
- 11. Take the sample out of the plating bath and rinse it with water. Save the sample in a plastic container.

# Second period.

To observe and measure the nanowires, you will release the nanowires from the filter and examine the wires under an optical microscope, or if available, a scanning electron microscope.

To prepare the sample for microscopy, do the following:

- 1. Use acetone or nail polish remover to remove the nail polish from the filter.
- 2. Place the sample into a 25 ml beaker and pipet 15 ml ethanol, and leave it for 1 hour
- 3. Transfer the sample to a vial and separate the deposited nanowires using a sonicator or vigorous stirring.
- 4. Remove a small amount of liquid, which contains suspended nanowires, and place on a glass microscope slide, or if an SEM is available, an SEM sample stub. Let the liquid dry for one hour.
- 5. Now the nanowires can be further observed under the optical microscope or SEM. You will measure the lengths of the 20-30 nanowires using the stage micrometer, and obtain an average wire length to compare with their predictions.
- 6. If insufficient numbers of nanowires are released using the ethanol, the alumina filter may be dissolved to release all the wires. To dissolve the filter, place the filter in 5 mL of 1..0M NaOH and leave for at least 20 minutes. Allow the wires to settle under gravity, pipette up a few drops from the bottom of the suspension, and transfer to a microscope slide. Repeat step 5.

## **Record Your Observations.**

Measurements (Example values shown)

	Ampere (mA)	Second (s)	Coulombs	Charge
Current	470			
	458			
	462			
	440			
	432			
Mean Current	452.4			
Time		300 s	135.72	
Mole electron				0.0000703 e <sup>-</sup>

# Analysis.

The calculation is done in the following steps. First, calculate the amount of charge that has passed through the solution, and convert to the number of charged ions. The quantity of electricity that has passed through a solution is equal to the current (for example, 500 mA =

0.500 amps) multiplied by the total time that the electrolysis is conducted (for example, 5 minutes = 300 seconds.

The amount of charge is given in coulombs.

Number of coulombs  $C = Amperes(A) \times seconds(s)$ 

If you ran the current for 5 minutes (300 seconds), then the calculation would be

$$0.5A \times 300 \text{ s} = 150 \text{ coulomb}$$

This can be converted to electronic charges as follows:

Number of electron charge 
$$e^{-}=\frac{150 Coulomb}{1.602\times10^{-19}}=9.36\times10^{20}$$

A convenient unit in electrochemistry is the Faraday (or *mole electron*), defined as the amount of charge (in coulombs) represented by one mole of electrons. This can be calculated by the following.

$$\frac{1.602 \times 10^{-16}}{e^{-}} Coulomb \times \frac{6.022 \times 10^{23}}{mole \ e^{-}} \ e^{-}$$

$$= \frac{96,485 \text{ Coulomb}}{mole e^{-}} = 1 \text{ Faraday} = 1 \text{ mole electron}$$

So for our example of 150 Coulombs we can convert to the number of Faraday:

Number of Faraday = 
$$\frac{1 \, mole \, e^{-}}{1} \times \frac{150 \, Coulomb}{96485 \, coulomb} \times \frac{1 \, mole \, of \, Cu}{2 \, mole \, of \, e^{-}}$$

This tells us how many moles of copper that this much charge will react with. The last term tells us that two moles of electrons are required to react with one mole of Cu<sup>2+</sup>, since the copper ion is doubly positive charged.

The result of this equation is 0.0000773 mole of Cu. That is, our 150 coulombs of charge will react with 0.0000773 moles of copper during the electro-plating process. This amount of copper will be electro-deposited at the cathode and into the pores of the filter that we are using as a deposition template. The filter pores are cylindrical, so the deposited copper nanowires will be too. We know the diameter of the pores (and resulting wires) from the filter manufacturer, but nanowire length will depend on how much copper we deposit.

To predict the size of nanowires, we'll need to know the total volume of copper deposited. Using the molecular weight for Cu (63.5 g/mole) and the mass density (89.6g/cm<sup>3</sup>) we can calculate the mass and the volume of the deposited copper in our example.

Mass of Cu deposit:

$$= \frac{0.0007773 \, mole \, of \, Cu}{1} \times \frac{63.5 \, g}{1 \, mole \, of \, Cu} = 0.04936 \, g \, of \, copper \, deposited \, in \, this \, example.$$

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Then the total volume of Cu deposited in this example is

Volume (cm<sup>3)</sup> = 
$$\frac{Mass(g)}{Density(g/cm^3)} = \frac{0.04936 g}{89.6 g/cm^3} = 5.5089 \times 10^{-4} cm^3 = 0.55089 \text{ mm}^3$$

This total volume of copper will be formed into many nanowires. To calculate the nanowire length for this example, assume the following:

- a. Each filter is 25mm in diameter, so total filter face area can be calculated (what is the formula you will use?).
- b. Each filter has a porosity of 50%, meaning the half face area is made up of pores that copper will deposit into. So the total pore area is equal to 0.5 x the filter face area.
- c. Assuming the copper plates evenly inside the pores, the expected length of each nanowire will be:

$$L = \frac{(total\ volume\ deposited)}{(Total\ pore\ area\ available)}$$

Now, repeat this analysis using your actual measured values for mean current (in amperes) and deposition time (in seconds). You may use all other assumptions used in the above example.

# Data Analysis Table (Example values shown)

	Diameter (mm)	Thickness (µm)	Area (mm²)	Volume (cm³)	Mass (g)
Anodisc	25mm		491 mm <sup>2</sup> (50% porosity)		
Nanowires	0.02µm		$245  \text{mm}^2$		
Copper deposited				$5.51x10^{-4}$ $cm^3$	0.0494 g
Predicted Nanowire Length	2.4 µm				
Mean measured length	3.5 µm				
Difference					

## Step by step calculations.

1. What is the volume of copper deposited in the pores of the filter template after the depotion is complete, using the measured value of current?

Students must repeat the above calculation to derive the number of moles of Cu produced, transform this to the mass of copper deposited (in g), and divide by the mass density of copper (89.6 g/cm³) to get a volume (in cm³)

- 2. What is the predicted length of the copper nanowires deposited in the 20 nm diameter pores? Divide the volume of the copper deposited by the total pore area, given by 50% of the filter surface area (since it has 50% porosity). This gives the rough average length of the nanowires. In the sample calculation above, this works out to 2.4 µm.
- 3. When you measure at least 20 of the nanowires that you made under a microscope, what is the mean length?

Collect data from class and compare the different groups' outcomes.

4. What is the difference between the predicted and measured values? Calculate the difference using the following formula:

% Difference = 
$$\frac{\text{Measured value - predicted value}}{\text{predicted value}} \times 100$$

Collect data from class and compare the different groups' outcomes.

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