LASER DIFFRACTION AND NANOPARTICLES: MEASURING REALLY SMALL THINGS

In part A of this experiment you will determine the width of a strand of your hair using laser diffraction. In part B of this experiment you will determine the volume of potassium bromide added to your unknown silver nanoprism sample.

EQUIPMENT

You will need 1 red laser pointer (λ = 633 nm), a meter stick, tape, 5 strands of hair, 3 medium size binder clips, a small piece of cardboard, seven large test tubes, two cuvettes, and a spectronic 200 spectrophotometer.

CHEMICALS

You will need 10 mL of 0.0125 M sodium citrate solution, 25 mL of 0.000375 M silver nitrate solution, 25 mL of 0.050 M hydrogen peroxide solution, 1 mL of 0.0010 M potassium bromide solution, 15 mL of 0.0050 M sodium borohydride solution*, and an unknown. All solutions except the potassium broomide will be dispensed using repipetters.

WASTE DISPOSAL

Any solution containing silver (the silver nanoparticle solutions, silver nitrate, and washings of these) must go in the silver waste container.

SAFETY

Wear your goggles the entire time. Gloves should be worn during all of part B of this lab. Silver nitrate is corrosive and causes burns if it comes in contact with human tissue. Sodium borohydride is flammable and toxic; when wet it releases hydrogen gas, **DO NOT KEEP SODIUM BOROHYDRIDE SOLUTIONS IN TIGHTLY SEALED CONTAINERS** to avoid explosion of the container. Sodium citrate may act as an irritant to the skin, eyes, and respiratory tract. Hydrogen peroxide is corrosive and causes burns to the eyes, skin, and respiratory tract. Silver nanoparticle solutions should not be kept tightly capped due to the possible presence of unreacted borohydride. Do not point a laser pointer at anyone, especially not into their eyes.

PROCEDURE

Part A – Laser Diffraction

Take a small piece of cardboard and cut an opening in the middle if it does not already have one.

Tape one of your hairs across the middle of the opening in the cardboard vertically.

Use two binder clips to prop up the cardboard on a bench facing the wall.

Use another binder clip to support the laser pointer about 1 foot back from the cardboard pointing at the wall.

Make sure the laser beam is centered on the hair.

Place the cardboard just less than 1 meter from the wall.

The laser should be perpendicular to the wall.

It is important to keep the cardboard at the same distance from the wall the entire time, do not move it until you are done.

Measure the distance from the from of the cardboard to the center of the brightest light point (P_0) on the wall in cm. This distance is "**L**".

Measure the distance from the center of the bright spot to the center of the first dark region on one side of the diffraction pattern (P_1) in cm, this is "$\mathbf{y_1}$". Next measure the distance from the center of the bright spot to the center of the second dark region (P_2) on the same side of the bright spot as the first one in cm, this is "$\mathbf{y_2}$".

Repeat 4 more times, each with a different hair. You will measure "$\mathbf{y_1}$" and "$\mathbf{y_2}$" for each hair. If you don't move the laser pointer you only need measure "**L**" once. If you move the laser pointer then you should measure "**L**" again.

Part B - Nanoparticles

MAKE ABSOLUTELY SURE YOUR TEST TUBES ARE CLEAN AND DRY.

USE AN ACID BATH THE WEEK BEFORE PERFORMING THIS EXPERIMENT AS WELL AS THE DAY OF THE EXPERIMENT.

DO NOT USE SOAP. THIS EXPERIMENT WILL NOT WORK IF EVERYTHING IS NOT CLEAN.

Label 7 large test tubes as 10, 15, 20, 25, 30, Reference Standard, and Unknown.

To all test tubes add the following, in this order (use the solutions in the repipetters):

2.00 mL of 0.0125 M sodium citrate

5.00 mL 0.000375 M silver nitrate

5.00 mL of 0.050 M hydrogen peroxide

You are now ready to add the potassium bromide. Your professor or Matt will do this for you. Bring all test tubes to either your professor or Matt, making sure they are all clearly labeled.

To the test tube labeled 10 add 10 μL of 0.001 M potassium bromide with a micropipette.

To the test tube labeled 15 add 15 μL of 0.001 M potassium bromide with a micropipette.

To the test tube labeled 20 add 20 μL of 0.001 M potassium bromide with a micropipette.

To the test tube labeled 25 add 25 μL of 0.001 M potassium bromide with a micropipette.

To the test tube labeled 30 add 30 μL of 0.001 M potassium bromide with a micropipette.

To the test tube labeled Reference Standard a specific volume of 0.001 M potassium bromide will be added with a micropipette. You will be given the volume of potassium bromide added to the reference standard in lab.

To the test tube labeled Unknown an unknown volume of 0.001 M potassium bromide will be added with a micropipette.

To the test tube labeled “10”add 2.50 mL of freshly prepared (by Matt) 0.005 M sodium borohydride solution, immediately put the parafilm back over the top of the test tube, and gently invert several times. **Do not shake the test tube**.

DO NOT SHAKE THE TEST TUBES!

To the test tube labeled “15”add 2.5 mL of freshly prepared (by Matt) 0.005 M sodium borohydride solution, immediately put the parafilm back over the top of the test tube, and gently invert several times. **Do not shake the test tube**.

DO NOT SHAKE THE TEST TUBES!

To the test tube labeled “20”add 2.5 mL of freshly prepared (by Matt) 0.005 M sodium borohydride solution, immediately put the parafilm back over the top of the test tube, and gently invert several times. **Do not shake the test tube**.

DO NOT SHAKE THE TEST TUBES!

To the test tube labeled “25”add 2.5 mL of freshly prepared (by Matt) 0.005 M sodium borohydride solution, immediately put the parafilm back over the top of the test tube, and gently invert several times. **Do not shake the test tube**.

DO NOT SHAKE THE TEST TUBES!

To the test tube labeled “30”add 2.5 mL of freshly prepared (by Matt) 0.005 M sodium borohydride solution, immediately put the parafilm back over the top of the test tube, and gently invert several times. **Do not shake the test tube**.

DO NOT SHAKE THE TEST TUBES!

To the test tube labeled Reference Standard add 2.5 mL of freshly prepared 0.005 M sodium borohydride solution, immediately put the parafilm back over the top of the test tube, and gently invert several times. **Do not shake the test tube**.

DO NOT SHAKE THE TEST TUBES!

To the test tube labeled “Unknown”add 2.5 mL of freshly prepared 0.005 M sodium borohydride solution, immediately put the parafilm back over the top of the test tube, and gently invert several times. **Do not shake the test tube**.

DO NOT SHAKE THE TEST TUBES!

Wait for the colors to finish developing (about 5 minutes).

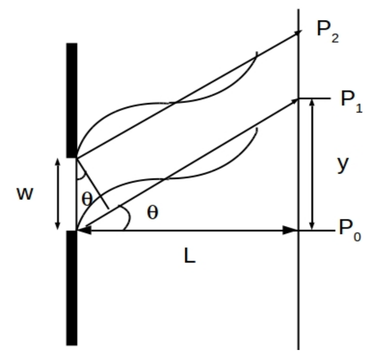
Place the Parafilm back over the test tubes and invert each test tube **gently**, 3 times each.

Using a spec 200 find the wavelength of maximum absorption (λ_{max}) for each solution between 400 nm and 900 nm. Record the wavelength of maximum absorption for each solution (in nm) in your data table.

CALCULATIONS

Part A:

The hair over the laser acts the same as a single slit through which the light passes.



Here w is the width of the hair, L is the distance from the hair to the wall, and y is the distance from the bright spot on the wall to the middle of the first dark area (node). This will be your $\mathbf{y_1}$. Your $\mathbf{y_2}$ will be the distance from the middle of the bright spot to the middle of the second dark area.

When $w \sin(\theta_n) = \lambda$ the path difference between the top and bottom light rays equals one wavelength (λ). That is, they are in phase.

This means that the path difference between the top ray and the ray just below the midpoint of the slit (w/2), is half a wavelength ($\lambda/2$).

Here the top wave, and the wave just below the midpoint of the slit, are out of phase, and therefore cancel each other out.

That means that for there to be destructive interference the following is true:

$$w \sin(\theta_n) = n\lambda \qquad \textbf{(Equation 1)}$$

where n = 1, 2, 3, …

If we know θ_n and the wavelength of the light (633 nm) we can find w, the slit width (or for us, the thickness of your hair!).

Looking at the larger right triangle we can see that

$$\tan(\Theta_n) = \frac{\mathrm{y_n}}{\mathrm{L}} \textbf{ (Equation 2)}$$

It ends up that:

$$\tan(\Theta_n) = \frac{\sin(\Theta_n)}{\cos(\Theta_n)} \quad \textbf{(Equation 3)}$$

Also, when θ_n is really small $\cos(\Theta_n) \approx 1$. This means that we can say that

$$\tan(\Theta_n) \approx \sin(\Theta_n) \quad \textbf{(Equation 4)}$$

Combing Equation 1, Equation 2, and Equation 4 we get:

$$\frac{\mathrm{y_n}}{\mathrm{L}} = \frac{\mathrm{n}\,\lambda}{\mathrm{w}} \quad \frac{\mathrm{y_n}}{\mathrm{L}} = \frac{\mathrm{n}\lambda}{\mathrm{w}} \text{ or}$$

$$\mathrm{w} = \frac{\mathrm{n}\lambda\mathrm{L}}{\mathrm{y_n}} \textbf{ (Equation 5)}$$

In Equation 5 n=1 for the first spot you measured and n=2 for the second spot (for each hair).

Find w (the thickness of the hair) for all 5 hairs. You will have 2 values for w for each hair, one from y_1 and one from y_2. That gives you 10 values for w. Find the average of these 10 values, and the standard deviation (using a spreadsheet of your choice)

Part B:

Using a spreadsheet plot λ_{max} **on the y-axis** versus the volume of potassium bromide added, in μL, for the test tubes labelled 10, 15, 20, 25, and 30.

Do not graph your reference standard or unknown!

Have your spread sheet give you the equation of the curve (2^{nd} order) for your data. The equation will have the form

$$\mathrm{y} = ax^{-b}$$

The λ_{max} for the reference standeard and your unknown is **y** in this equation, **solve for x**, which is the volume of potassium bromide added to the reference standard and your unknown, in μL.

Calculate the percent error for the reference standard. The true value will be given in lab.

CONCLUSION

Part A:

Report the average width of your hair in millimeters and the standard deviation for your data.

Determine and analyze one source of potential experimental error. Please read "How to Determine and Analyze a Source of Experimental Error".

Part B:

Report your unknown number. Report λ_{max} for each test tube.

Report the λ_{max} and volume of potassium added, in μL, for the reference standard.

Report your percent error for the reference standard.

Report the λ_{max} and volume of potassium added, in μL, for your unknown.

Determine and analyze one source of potential experimental error. Please read "How to Determine and Analyze a Source of Experimental Error".

[1] Andrew J. Frank, Nicole Cathcart, Kenneth E. Maly, and Vladimir Kitaev. Synthesis of Silver Nanoprisms with Variable Size and Investigation of Their Optical Properties: A First-Year Undergraduate Experiment Exploring Plasmonic Nanoparticles. *J. Chem. Ed. 2010; 10: 1098 – 1101*

[*] The sodium borohydride solution should be made up the morning of the experiment.