

Synthesis of Colloidal Gold

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Purpose:

The purpose of this experiment is to synthesize gold nanoparticles, then use the concept of the Tyndall effect to confirm their presence.

Learning Objectives:

- 1) Distinguish between absorption versus reflection.
- 2) Recall and apply oxidation and reduction concepts.
- 3) Given specific ions student will be able to compare them to the ions in the experiment and predict the outcomes of such ions on the gold nanoparticles.

Introduction

A colloid is a mixture of particles smaller than the naked eye dispersed throughout another substance. There can be almost any combination of type of dispersed material and type of dispersed medium, except for a gas dispersed in a gas as shown below. The figure below is a good example of the types of colloids that are in existence.

Dispersed Material	Dispersed in Gas	Dispersed in Liquid	Dispersed in Solid
Gas (bubbles)	Not possible	Foams: soda pop; whipped cream; beaten egg whites	Solid foams: plaster; pumice
Liquid (droplets)	Fogs: mist; clouds; hair sprays	Emulsions: milk; blood; mayonnaise	butter; cheese
Solid (grains)	Smokes: dust; industrial smoke	Sols and gels: gelatin; muddy water; starch solution	Solid sol: pearl; colored glass; porcelain; paper

<http://www.scienceclarified.com/Ci-Co/Colloid.html>

Colloids also display the Tyndall effect. The Tyndall effect is the reflection of light off of small particles in some sort of medium. The Tyndall effect occurs in a colloid because the beam of light is scattered by the small particles.

In this experiment gold nanoparticles are produced in water. They are synthesized by the reduction of chloroauric Acid ($\text{H}[\text{AuCl}_4]$). Chloroauric acid is dissolved in water and heated to a boil while a reducing agent is added. The reducing agent reduces the gold ions to neutral gold atoms. If the colloidal solution becomes supersaturated then a gold nanoparticle precipitate forms. Throughout this entire heating process, the solution is stirred vigorously to ensure that all particles will be similar sizes. A stabilizing agent is

needed with the reducing agent to ensure that the gold nanoparticles do not aggregate together.

To prevent the particles from aggregating, some sort of stabilizing agent that sticks to the nanoparticle surface is usually added.

In this experiment sodium citrate is used to reduce the chloroauric acid. The surface of the gold nanoparticles later absorbs the citrate ions. The repulsion of the negative charged citrate ions stabilizes the colloid preventing the gold nanoparticles from sticking together. The larger the anion the further apart the nanoparticles are from one another. The smaller the anion the closer the nanoparticles are from one another. Based on the variety of distances between the particles, the particles reflect or absorb different wavelengths of light.

Materials:

- 1 M NaCl
- 1 mM hydrogen tetrachloroaurate: HAuCl_4
- 1% trisodium citrate
- Laser pointer
- 50 ml Erlenmeyer flask
- Rubber band
- 50 ml beaker

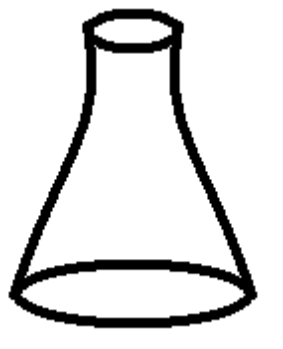
Safety Requirements:

Gloves and safety goggles

Procedure:

- 1) Add 20 mL of 1.0 mM HAuCl_4 to a 50 mL Erlenmeyer flask clamped to a ring stand and place it on a stirring hot plate. Add a magnetic stir bar to the flask. Turn on the hot plate and the stirring function and bring the solution to a boil. Note: The stirring function should be set between a medium to fast setting for vigorous stirring.
- 2) When boiling make sure there is adequate volume of liquid left in the flask.
- 3) Slowly add 2 ml of 1% trisodium citrate dehydrate $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ to the 50 ml flask, allowing the solution to continue stirring and boiling on the hot plate until a deep red color appears. The color will first appear as a dark purple, but over time will take on a deep red hue that is a little darker than the color of beets.
- 4) When the color is achieved, carefully remove the flask from the hot plate and place on wire gauze. Once it is cool enough to handle, pour contents of flask into a 50 ml beaker.
- 5) Take a laser pointer and wrap a rubber band around the on-switch so it is on continuously. Place the laser pointer to the edge of the beaker and pass the light

through the solution. Record your observations. Now rotate the laser pointer at 90° intervals and record any new observations.



- 6) Take two test tubes or disposable cuvettes. Place 4 ml of the colloidal gold into each one. To one tube add 5-10 drops of NaCl until the color of the liquid changes. What is the color change that is observed?

- 7) Record the UV absorption spectrum of both of the cuvettes. Attach spectrum to lab.

Questions:

- 1) Describe a colloid. What type of colloid was made during the course of this lab?
- 2) Summarize why the Tyndall effect proves that there are nanoparticles in the solution.
- 3) What happens to the solution when the sodium citrate is added?
- 4) What was the initial oxidation state of the gold?
- 5) What was the Final oxidation state of the gold?
- 6) What is the purpose of adding the Sodium Chloride?
- 7) What would happen if sugar were added instead of salt? Would there be a color change? Why?
- 8) What keeps the nanoparticles from aggregating together in the original colloidal gold suspension?
- 9) Elaborate on the wavelengths of light that are absorbed and reflected.
 - a. What wavelength of light is absorbed in the first colloid suspension? What color does this correspond to?
 - b. What wavelength of light is reflected in the first colloid suspension? What color does this correspond to?

References:

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Teacher Preparations:

Materials:

Stock Solutions for 25 batches

1.0 mM hydrogen tetrachloroaurate: Dissolve 0.1 g HAuCl_4 in 500 mL distilled water. This stock solution of gold (III) ions can be prepared in advance if stored in a brown bottle.

1% trisodium citrate: Dissolve 0.5 g $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ (sodium citrate) in 50 mL distilled water.

1 M NaCl: Dissolve 0.5 g of NaCl in 10 mL distilled water.

Suggestions for placement of the lab:

This lab can be placed in any general chemistry course at the high school or undergraduate level. Questions can be changed to accommodate material covered in a given course knowledge levels.

Questions or concerns about labs:

We could not observe the polarizing effect of the colloidal gold as observed in the original lab. The procedure was done a multitude of times and similar results were obtained each time. The shape of the laser may account for the diminished light that was observed and not the polarization. When a circular beam was used verses a oblong beam, no polarizing effect was observed.

Answers

Questions:

- 10) Describe a colloid. What type of colloid was made during the course of this lab?

Colloid occurs when there are small particles dispersed throughout a continuous medium. The colloid in this lab is a solid particle dispersed in a liquid medium, normally called a solution or gel.

- 11) Summarize why the Tyndall effect proves that there are nanoparticles in the solution.

The scattering of the light beam inside the solution indicates that there are nanoparticles in the solution.

- 12) What happens to the solution when the sodium citrate is added?

The tetra aurate is reduced. The gold ions are reduced to neutral gold particles.

- 13) What was the beginning oxidation state of the gold?

3+

- 14) What was the ending oxidation state of the gold?

0

- 15) What is the purpose of adding the Sodium Chloride

To show that if a strong electrolyte is added the particles will become closer together and at some point fall out of colloidal suspension.

- 16) What would happen if sugar were added instead of salt? Would there be a color change? Why?

Nothing would happen, because sugar is not a strong electrolyte and there would be no ion exchange in the solution.

- 17) What keeps the nanoparticles from aggregating together in the original colloidal gold suspension? How?

The sodium citrate keeps the particles from aggregating together in the original colloidal suspension by keeping the particles electrostatically further apart from one another.

- 18) Elaborate on the wavelengths of light that are absorbed and reflected.

- a. What wavelength of light is absorbed in the first colloid suspension? What color does this correspond to?

Green light is absorbed, which has a wavelength corresponding to around 510nm

- b. What wavelength of light is reflected in the first colloid suspension? What color does this correspond to?

Red light is reflected which corresponds to a wavelength of about 650 nm.