**Exploring Nanotechnology : Synthesis and Characterization of Gold nanoparticles**

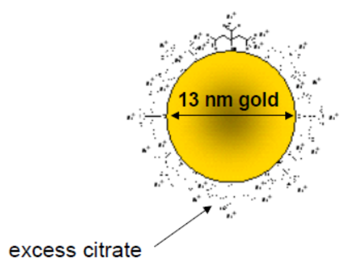
## Nanoparticulate (Colloidal) Gold[1]

Physical and chemical properties of certain materials are, to some extent, size-dependent. A sample of jewelry gold, for example, has the familiar yellow color, caused by its poor reflectance of light in the blue part of the spectrum. If the gold is divided into smaller and smaller particles, however, eventually the particles become similar in size to the wavelengths of visible light (400-720 nm). These nanoparticles interact with light in interesting ways – *e.g.*, Rayleigh or Mie scattering, electronic oscillations called surface plasmons – that are not important in bulk metal samples. The result is a range of brilliant colors, easily tuned by altering particle size or environment. While nanoparticulate gold has been utilized for centuries as pigments (*e.g.,* for stained glass), it is very much a modern material. One new application being investigated takes advantage of color changes induced when proteins adsorbed onto gold nanoparticles change their shape. The goal is to produce a sensor that physicians could use for quick identification of antibodies or other signs of infection in the blood stream.

Gold nanoparticles can be prepared by reaction of $HAuCl_4$ and sodium citrate, $Na_3C_6H_5O_7$. The citrate has two functions. First, it serves as a mild reducing agent, converting $Au^{3+}$ to Au (Equation 1).

$$\text{Au(III)} + \underset{\text{(citrate)}}{C_6H_5O_7^{3-}} \xrightarrow{\text{unbalanced}} \text{Au(0)} + \text{oxidized citrate}$$

Then, as the gold atoms cluster to form nanoparticles, excess citrate anion is adsorbed at the particle surface. This coating of anions keeps the nanoparticles separated in solution (Figure 1). Changing the ionic environment changes the tendency of the nanoparticles to aggregate, so that particle size and, in turn, color are affected.




The gold nanoparticles should have a dark red/purple color when synthesis is complete. To characterize the nanoparticles you will take an absorption spectrum. The larger the size of the gold nanoparticles the longer wavelength of light absorbed. As the size of the nanoparticles increase the SPR band shifts to longer wavelengths of light and the nanoparticles appear more blue (see table 1).

| **Diameter of nanoparticles** | **Peak SPR wavelength** |
|---|---|
| 5 nm -10 nm | 515-520 nm |
| 15 nm | 520 nm |
| 20 nm | 524 nm |
| 30 nm | 526 nm |
| 40 nm | 530 nm |

[1] Adapted from A.D. McFarland, et. al. ***J.Chem. Educ.*** 2004 81 544A. Additional background information from http://news-service.stanford.edu/news/2005/march30/gold-033005.html. www.mrl.ucsb.edu/~seshadri/2005_265/class05_nano_gold.pdf, www.primidi.com/2005/03/04.html.

The peak shape of the absorption spectrum also gives important information about the nanoparticles. A narrow peak indicates the nanoparticles are monodispersed, whereas a broad peak indicates the nanoparticles are polydispersed or aggregated. Gold nanoparticles that have aggregated will lose their characteristic red/purple color and appear a bluish-grey.

Finally, you will determine the effects of solutions of electrolytes and non-electrolytes on the color and SPR band of the gold nanoparticles.

**How to Prepare for lab**

Write a prelab (purpose and procedure) in your lab journal BEFORE coming to lab

Report sheets will be distributed in lab (one/pair) and handed in at the end of the lab period

No REPORT for this lab!

**Experimental Procedure**

***Safety:*** *Safety goggles must be worn at all times. The $HAuCl_4$ solution is corrosive, so gloves should be worn while preparing/using the nanoparticle suspension. Liquid nitrogen can cause serious freeze burns, so avoid prolonged contact with skin.*

***Waste Disposal:*** *The nanoparticle suspension and any excess $HAuCl_4$ solution should be placed in the appropriate recovery container. All other solutions used here may be disposed of in the sink.*

*Record all data you collect* ***directly*** *on your report sheets.*

**A. Preparation of Gold Nanoparticles**

*To avoid unwanted aggregation of the nanoparticles, make sure all glassware is very clean and rinsed well with distilled water.*

Put 20 mL of 1.0 mM $HAuCl_4$ in a 50 mL beaker, and add a stir bar. Use a permanent marker to mark the outside of the beaker at the solution line. Heat the solution to boiling on a stir/hot plate while stirring. Once the solution begins to boil, add 2 mL of 38.8 mM $Na_3C_6H_5O_7$. Continue to boil and stir the solution for ~10-15 minutes until it completely changes color. During this time, add distilled water as needed to keep the total solution volume near 22 mL (slightly above the mark). Remove the beaker from the hot plate, add ~20 mL of distilled water, and allow the solution to cool to near room temperature before using it in Part B.

B. **Characterization of Gold Nanoparticles**

Calibrate the spectrophotometer (Spectro-Vis) using a cuvette filled with distilled water as a blank. Take an absorption spectrum of a sample of the gold nanoparticles prepared in part A. You may need to dilute the gold nanoparticles if the absorbance is >1.100 at the maximum wavelength. Make sure to take a screenshot of the spectrum and save it as a png to include in your report! Determine the wavelength of maximum absorption (SPR band) and record in your lab notebook.

C. **Nanoparticles as Chemically Selective Sensors**

Clean five medium-sized test tubes and rinse them several times with distilled water. Add ~4 mL of the nanoparticle solution to each.

For the negative control add 20 drops of distilled water. Record your observations.

To one test tube, add up to ~20 drops 1M aqueous NaCl, 1-2 drops at a time, shaking the tube to mix well after each addition. Note any changes using one of the other three test tubes for color comparison. If no color change occurs at 20 drops you can stop adding the reagent. Record your observations.

In the remaining test tubes, perform similar tests using 1M aqueous solutions of sucrose (table sugar), acetic acid (vinegar) and 1 M hydrochloric acid.  What is happening to the nanoparticles as you add these solutions?  Why do some but not all of the test reagents cause these changes?  If you cannot discern trends, you may need to repeat some of your tests.

***Names:*****_____________________________________________ ____________________ *Lab Day*________________**

**Exploring Nanotechnology Report Sheets (turn in one copy per pair) (20 pts)**

A. Preparation of Gold Nanoparticles **(4 pts)**

Observations of the reaction:

B. Absorption Spectrum and size of nanoparticles **( 2 pts)**

What is the $\lambda$max of the gold nanoparticles? Based on this SPR absorption , what is the size of the gold nanoparticles?

C. Nanoparticles as Chemically Selective Sensors

1. Describe the forces that keep the nanoparticles apart in solution. **(2 pts)**

2. Test observations: **(4 pts)**

| | |
|---|---|
| Color of original nanoparticles | |

| Reagent added | Changes observed as reagent is added |
|---|---|
| | |
| | |
| | |
| | |

*Name*___________________________________________ ____________________ *Lab Day*________________

**Exploring Nanotechology Report Sheets**

C. Nanoparticles as Chemically Selective Sensors (continued)

3. In some of the tests, a color change is observed in the gold nanoparticle solution. What must be happening to the nanoparticles to cause the color change? **( 2pts)**

4. Group the four test compounds according to whether or not they caused a color change. What is similar about the compounds that caused a color change? What is similar about the compounds that had no effect? **( 4 pts)**

5. The nanoparticles synthesized in this lab can be used as chemical sensors, they can be used to distinguish different types of compounds. Summarize how your sensor works. **( 2 pts)**