

Synthesis of Colloidal Gold

This lab was adapted for use with the SALS Device by Lisa Goetter.

The experiment was obtained from the MRSEC website Video Lab Manuel.

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Introduction

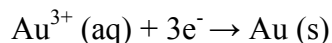
The formation of gold nanoparticles can be observed by a change in color since small nanoparticles of gold are red. A layer of absorbed citrate anions on the surface of the nanoparticles keep the nanoparticles separated. Switching to a smaller anion through using NaCl allows the particles to approach more closely and another color change is observed.

Learning Objectives

- To synthesize gold nanoparticles
- To understand the change in properties of gold nanoparticles
- To run an oxidation – reduction reaction
- To use the SALS Device

Reaction

The formation of colloidal gold occurs by using citrate to reduce gold (III) to gold solid. The reduction half reaction of gold is shown below:



Materials

Stock Solutions:

1.0mM hydrogen tetrachloroaurate – HAuCl_4 (s)

1% trisodium citrate – $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ (s)

1 M NaCl – NaCl (s)

Distilled water

Note: HAuCl_4 (s) can be purchased online through Sigma Aldrich – ten grams for \$112.50. www.sigmaaldrich.com. You may also want to contact the local university as they will usually have access to hydrogen tetrachloroaurate.

Trisodium citrate, commonly known as sodium citrate, is a common food additive that can be found at specialty food stores.

SALS Device

1 30mL beaker

Flea stir bar

2 10mL beakers

Stirring hot plate with white porcelain top

Ring stand with 3-prong clamp (optional)

Note: The white porcelain top on the hot plate is needed to obtain a good reading with the SALS Device.

Safety

Wear eye protection

Use care with the stir/hot plate

Thermal gloves

Procedure

Preparation of Stock Solutions

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

For 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.

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

(This procedure prepares enough stock solution for 25 batches.)

Synthesis

1. Using a graduated cylinder, measure and add 20mL of 1.0mM HAuCl₄ to a 30mL beaker. Move to a stirring hot plate, add a flea magnetic stir bar, and bring the solution to a boil. Keeping the beaker on the stirring hot plate, insert the SALS probe, play the tone, and save it in Memory 1. *Note:* All SALS readings MUST be done on the hot plate.
2. To the boiling solution, add 2 mL of a 1% solution of trisodium citrate dihydrate, Na₃C₆H₅O₇·2H₂O. Immediately, insert the probe and hold down the play button while the solution is still being heated and stirred. (The SALS probe can be set up with a ring stand and 3-prong clamp to remain in solution on its own, but it can be held in solution by you as well.) The gold sol gradually forms as the citrate reduces the gold (III). Continue to hold the play button the entire time the gold sol is forming. Listen carefully to the tone, when the pitch is no longer getting lower (approx. 1-2 minutes) remove the solution from the hot plate. CAUTION: Beaker will be hot. Be sure to wear thermal gloves when handling.
3. Pour the solution into two small (10mL) beaker. (Fill about ¾ of the way full). Set them on the stirring hot plate after turning both the heat and stirring controls off. Play the tone from the colloidal gold solution by inserting the probe into the beaker on the left. Save this in Memory 2. Add 10-15 drops of 1M NaCl to the beaker on the right. Stir this solution. Using the SALS Device play the tone of the solution comparing it to the initial solution in Memory 1 as well as the gold colloidal solution saved in Memory 2. Record your observations noting the change of tone of the solution as the nanoparticles get closer together.

References

A. D. McFarland, C. L. Haynes, C. A. Mirkin, R. P. Van Duyne and H. A. Godwin, "Color My Nanoworld," *J. Chem. Educ.* (2004) **81**, 544A.

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