Gold Nanoparticles Synthesis

Equipment

N.B. Since the different steps may be run at different moments, the necessary material for each step is written down just after the section caption.

Procedure

Sodium citrate solution

- sodium citrate
- scale (± 0,1 gr)
- measuring cylinder (50 ml)
- beaker (50 ml)
- spatula
- distilled water

1. Put a 50 ml beaker on the scale and set the weight to zero.
2. Weigh 0,5 gr of sodium citrate directly in the beaker.
3. Put 50 ml of deionized water in a measuring cylinder.
4. Add the deionized water to the sodium citrate; mix till you obtain a solution.

$HAuCL_4$ diluted solution

- lab googles
- latex gloves
- paper towels
- measuring cylinder (250 ml)
- measuring pipette (2ml)
- dark bottle (>250 ml)
- $HAuCL_4$ concentrated solution (2ml)
- deionized water

Careful! $HAuCL_4$ although diluted is still an acid!

1. With a measuring pipette take 2ml[1] of HAuCL4 concentrated solution .

[1]The dosage is thought for a concentrated solution obtained from 0,5 gr of HAuCl4*3H2O in 50 ml of deionized water.

2. Pour them in a dark 300 ml bottle.
3. Take 250 ml of distilled water with a measuring cylinder.
4. Pour the water into the dark bottle with the concentrated solution.
5. Seal with the cork and shake carefully.
6. The amount of solution that will not be used in the next step can be stored away in the well sealed bottle. As further precaution wrap the bottle in aluminium foil.

Gold nanoparticles synthesis

- latex gloves
- paper towels
- lab googles
- hot plate with magnetic stirrer + magnetic flyer
- measuring pipette (25ml)
- dark bottle (>250 ml)
- (20 ml) $HAuCL_4$ diluted solution
- temperature probe (optional)

1. Take 20 ml of gold diluted solution and pour them in an Erlenmayer flask.
2. Put the Erlenmayer flask on the hot plate of a magnetic stirrer. Put in the magnetic flyer and the temperature probe.
3. Turn the stirrer speed down to medium low and set temperature around 150/180°C .

> ***Careful! Too high a speed may result in the Erlenmayer flask flying away and spilling all its content!***

4. Bring to boiling point. When you see the first bubbles and the solution temperature reaches beyond 80/85° C add 2 ml of sodium citrate solution with a Pasteur pipette.
5. Turn the temperature knob down to 100 °C and wait for a few minutes. The solution will turn from pale yellow to red going through grey and violet. Note down your observations during the synthesis in the following table: write down the colour and the corresponding time.
6. **At the end of the synthesis** estimate the light wavelenght (nm) corresponding to both the reflected and the absorbed colour. You may consult a scheme of visible radiation in your textbook or on the Web.

The amount of diluted solution obtained is enough for 11-12 groups. Store away the remnants for future use.

	Color of HAuCL4 solution (*before the reaction*)	Color *immediately* after adding the sodium citrate	Intermediate color I	Intermediate color II	Final color
Color					
λ absorbed(nm)					
λ reflected (nm)					
T (sec) after adding the sodium citrate					

1. Explain why intermediate colors are noticed during the reaction.
   ……
   ……

2. What can you infer about the dimension of nanopartices and nanoclusters just basing on color?
   ……
   ……

Test to prove the formation of colloid – Tyndall effect.

- whole milk (a few drops)
- salt
- 2-3 clear glasses
- gelatine
- distilled water
- laser pointer

To test whether the sample you've previously synthetized really contains gold nanoparticles you may use a laser pointer. In fact, differently from solutions, colloids are made of particles which are small enough to be in suspension but large enough to diffuse light (Tyndall effect).
Compare what happens to the laser beam when it goes through the gold solution (pale straw yellow) and the nano gold (red). Watch the tube from one side: how does the beam look? Try to change your observation point and carefully observe what happens.

You may also test Tyndall effect on different substances (see table). Record your observations in the table below and find out which are colloids.

> ***Careful! NEVER shine the laser beam in anyone's eye. Watch out for reflected beams as well!***

Sample	Observed effect	Further comments	Is it a colloid?
Distilled water			
Gold solution (pale straw yellow)			
Gold synthtized in class (ruby red)			
Milk diluted in water[2]			
Salt dissolved in water[3]			
Gelatine			

1) Riassumete le principali differenze tra colloide, soluzione e sospensione.
   ……
   ……

2) Spiegate in modo dettagliato perché la presenza di nano particelle può essere confermata usando una penna laser.
   ……
   ……

3) Elencate alcuni colloidi naturali
   ……
   ……

4) Individuate nuovi esempi da aggiungere alla lista e proporre ai vostri compagni per il test.
   ……

[2] Two drops of milk in 150 mL of distilled water. What happens when you add more milk drops?

[3] After dissolving the salt and stirring the solution, let it rest for a few minutes so that there will not be any air bubble which may have an impact on Tyndalll effect

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5) Have you got any suggestions about how to improve the experimental protocol??
……
……

Waste disposal

At the end of the experiment the remnants of the colloid should be stored away in a dark bottle for future use.

Credits: This experimental protocol has been partially adapted from **Synthesis procedure for students - EXPERIMENT C: Nanogold colorimetric sensor** http://nanoyou.eu/

Exercises

Exercise 1

In the picture below you can see some colloidal solutions of silver nanoshells with gold inside. These solutions (the eight tubes on the right) according to nanoparticles dimension absorb different wavelengths of visible light. The last vial at the left containing the gold colloid (pink) is used as reference. Estimate the absorbed wavelength and the average shell dimension for each vial All'estrema sinistra viene utilizzato come termine di paragone il colloide d'oro (rosato).

Explain in detail your results.

(*Source: K.-T. Yong et al. / Colloids and Surfaces A: Physicochem. Eng. Aspects 290 (2006) 89–105)*