

EXPERIMENT C- STUDENT SYNTHESIS PROCEDURE

COLORIMETRIC GOLD NANO-SENSOR

Below is the synthesis procedure for EXPERIMENT C included in the Experiment Module of the “NANOYOU Teachers Training Kit in Nanotechnologies” for students aged 14-18.

DISCLAIMER: The experiments described in the following training kit use chemicals which need to be used accordingly to MSDS specifications. Follow school lab safety guidelines. Personal protection must be taken as indicated. As with all chemicals, use precautions. Solids should not be inhaled and contact with skin, eyes or clothing should be avoided. Wash hands thoroughly after handling. Dispose as indicated. All experiments must be conducted in the presence of an educator trained for science teaching. All experiments will be carried out at your own risk. Aarhus University (iNANO) and the entire NANOYOU consortium assume no liability for damage or consequential losses sustained as a result of the carrying out of the experiments described.

MATERIAL NEEDED:

Chemicals:

- 0.1 g of $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ (Sigma Aldrich #G4022, 1g costs 144.50 Euro);
- 0.5 g of $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ (Sigma Aldrich #S4641, 25g costs 24 Euro),
- 1 litre of distilled water

Glassware/labware:

- Eye protection
- Latex or nitrile gloves
- Paper towels
- Cylinders: 10mL cylinder, 50mL cylinder and 500 mL cylinder
- Glass pipettes: 5 mL pipette and 25 mL pipette
- 50mL Erlenmeyer flask or beaker
- 4 disposable plastic capsules for measuring solids
- Spatula
- Glass bottles: bottle 500mL, 2 small bottles 25 mL or 2 beakers 25 mL

- Aluminium foil
- 1 laser pen
- 1 stirring hot plate
- 1 magnetic stir bar
- 1 oven glove

Also needed for laser test:

- 1 glass of milk

PRECAUTIONS

Use these materials with normal chemical precautions according to MSDS. Wear eye protection and gloves. Solids should not be inhaled and contact with skin, eyes or clothing should be avoided. Wash thoroughly after handling. **Be aware that $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ is corrosive and must be handled with caution.**

DISPOSAL OF THE GOLD COLLOID

After the experiment, dispose of any gold colloid remaining as follows: add enough NaCl solution to the colloid to induce precipitation. Leave the solution still for at least 30 minutes (a black residue will form). Filter the residue on filter paper, and then dispose of it with solid normal waste. Dispose of the clear liquid in the wash basin with plenty of water.

PROCEDURE:

A. Preparation of stock solutions

- **1.0 mM hydrogen tetrachloroaurate:** Dissolve 0.1 g $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ (orange solid) in 500 mL distilled water. Solution appears light yellow.
- **1% trisodium citrate:** Dissolve 0.5 g $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ in 50 mL distilled water
- **~1 M NaCl:** Dissolve 0.5 g of NaCl (or table salt) in 10 mL distilled water.
- **~ 1 M sugar:** Dissolve 2 g of sugar in 10 mL distilled water.

Your teacher might prepare those solutions for you. In that case, skip part A and go to part B.

B. Synthetic procedure of the gold colloid

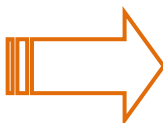
1) Add 20 mL of 1.0 mM HAuCl_4 stock solution to a 50 mL Erlenmeyer flask on a stirring hotplate (turned off). The solution appears light-yellow. Add a magnetic stir bar inside the flask; turn the stirring on and the temperature on (about 120 °C). Bring the solution to boiling point. To minimize the volume loss due to evaporation, once the solution is boiling, reduce the temperature to about 100 °C.



2) To the **boiling solution** add, all at once, 2 mL of a 1% solution of trisodium citrate dihydrate, ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$). The gold colloid gradually forms as the citrate reduces the gold (III). Observe the colour change as the citrate is added. Once the solution is ruby-red, turn the hotplate off and stop the stirring. Remove the flask from the hotplate. **WARNING:** the flask will be hot, so use an oven glove to avoid burning your fingers! Place the flask on a safe surface (e.g. table bench with a piece of aluminium foil).



Before reaction



End of reaction

CREDIT

This exercise was partly adapted from the experiment reported in: "Color my nanoworld", Journal of Chemical Education and from the experiment "Citrate synthesis of gold nanoparticles", University of Wisconsin-Madison, see: <http://www.mrsec.wisc.edu/Edetc/curriculum/index.html>, Vol. 81(4), 2004; a more detailed description of the synthesis of colloid gold is given in: Keating et al., Journal of Chemical Education 1999, Vol. 76, No. 7 pp. 949-955.

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