



**Miranda House
University of Delhi**

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**Colors of Gold and
Silver**

**Offered by:
Chemistry Department**



Chemistry: Colors of Gold and Silver

PREPARATION OF GOLD NANOPARTICLES

AIM

To prepare gold nanoparticles from a solution of gold chloride

REQUIREMENTS

Apparatus and equipment: One 100 mL conical flask; one hotplate cum magnetic stirrer

Reagents: Gold chloride; sodium citrate

PRINCIPLE

Materials have very different properties as compared to their macro state when present as nanoparticles, i.e. particles with diameter in the nanometre (10^{-9} m) range. Gold nanoparticles, for example, show many different colours – purple, red, yellow etc. depending on their size. This property of gold was utilised by artisans to make stained glass windows seen in old European churches. In this experiment, gold nanoparticles will be made from gold chloride in solution using the trisodium salt of citric acid (3-carboxy-3-hydroxy pentanedioic acid) as the reducing agent to reduce gold (III) to metallic gold. The citrate ions also act as stabilisers for the gold nanoparticles by creating a protective shield round the particles formed and preventing their agglomeration (coming together) to give bigger particles.

PROCEDURE

- Add 50 mL water to the 1% gold chloride solution provided in 100 mL conical flask placed on hotplate cum stirrer maintained at 40°C.
- Turn on the stirrer.
- Add 0.075 g sodium citrate provided to the diluted gold chloride solution in the flask.
- Allow stirring to continue for 3 minutes.
- Increase the hotplate temperature to boiling and allow the solution to boil for a few minutes.
- Switch off the hotplate when no further change takes place for 5 minutes.
- Add 10 drops of sugar solution to approximately 10 mL of gold nanoparticle solution taken in a test tube and observe changes, if any.
- Add 10 drops of salt solution to approximately 10 mL of gold nanoparticle solution taken in a test tube and observe changes, if any.

RESULTS AND DISCUSSION

The colour of the solution changes from almost colourless to purple to a deep wine red in about 20 minutes after the addition of sodium citrate. The purple nanoparticles are larger than the red ones. Continuous stirring ensures that the particle size is uniform throughout the solution. Salt changes the colour of the solution back to blue while sugar does not affect it. The formation of nanoparticles can be confirmed by determining the

wavelength of maximum absorbance (λ_{\max}) using a spectrophotometer. It should be about 520 nm for the red solution.

For silver nanoparticles:

A golden yellow silver colloid may be obtained by adding sodium citrate and hydrazine hydrate solution to a continuously stirred silver nitrate solution maintained at 40°C.

Nanoparticles

A nanoparticle may be defined as a particle having one or more dimensions of the order of 100 nm or less. Novel properties that differentiate **nanoparticles** from the **bulk material** typically develop at a critical length scale of under 100 nm. These "novel properties" are entirely dependent on the fact that at the nano-scale the properties of materials are different from the properties of the bulk material. This makes the size of particles or the scale of their features the most important attribute of nanoparticles. Nanoparticles are of great scientific interest as they are effectively a bridge between bulk materials and atomic or molecular structures.

Gold nanoparticles have been used since ancient times to colour glass in intense shades of red, yellow, and mauve. This is surprising since we normally think of gold as having a shiny golden colour and lustre. Metal nanoparticles, in general, and gold nanoparticles, in particular, are very attractive because of their size- and shape-dependent properties. Gold nanoparticles display many interesting electrical and optical properties. A solution of nano-sized particles of gold can appear to be a wide variety of colours depending on the size of the nanoparticles. You may be aware that metals (like the gold in the nanoparticles) are good conductors, which is why they are used in electronics and wiring. Metals are good conductors because their electrons are not bound to individual atoms instead forming a "cloud" around the atomic cores. This cloud of electrons is mobile allowing metal to transport charge (electrons) easily. Also, experience tells us that metals are shiny. This is because light is reflected off their surfaces back to the eye. The reason for this reflection has to do with the electron cloud that surrounds metals. Photons (individual units) of light cannot be absorbed by the atomic cores because they are blocked by the electron cloud. Consequently, photons are reflected back to the eye producing the sheen associated with metals.

However, we also know from quantum mechanics that electrons can behave as either a wave or a particle. If we imagine electrons in the electron cloud as a wave with a certain energy value, we can envision a situation where it is possible for light of the same wavelength to be absorbed by the electron cloud, producing resonance. This is similar to what happens on stringed instruments when a vibration occurs that matched the natural length of the string or one of its harmonics.

When a metal absorbs light of a resonant wavelength it causes the electron cloud to vibrate, dissipating the energy. This process usually occurs at the surface of a material (as metals are not usually transparent to light) and is therefore called **surface plasmon resonance**. Plasmons are the name for the oscillations of the electron cloud.

What this means in real terms is that there are certain wavelengths for metal where photons are not reflected, but instead are absorbed and converted into surface plasmon resonance (electron cloud vibrations). For “normal” metals, like gold jewelry, these wavelengths occur in the infrared portion of the spectrum (wavelength > 800 nm). These wavelengths fall outside the visible range that can be seen with the eye, and the metal therefore appears to reflect most light and is shiny.

But nanoparticles have extremely high proportions of their substance at their surfaces. If you were to compare the proportion of surface in a nanoparticle to a gold stud earring you would find that it has two million times more surface area compared to its volume than the earring. More surface area means more potential for surface plasmon resonance. Nanoparticles can experience surface plasmon resonance in the visible portion of the spectrum. This means that a certain portion of visible wavelengths will be absorbed, while another portion will be reflected. The portion reflected will lend the material a certain colour. Small nanoparticles absorb light in the blue-green portion of the spectrum (~400-500 nm) while red light (~700 nm) is reflected, yielding a deep red colour (particle size 30 nm). As particle size increases, the wavelength of surface plasmon resonance related absorption shifts to longer, redder wavelengths. This means that red light is now absorbed, and bluer light is reflected, yielding particles with a pale blue or purple colour (size 60 nm). As particle size continues to increase toward the bulk limit, surface plasmon resonance wavelengths move into the IR portion of the spectrum and most visible wavelengths are reflected. This gives the nanoparticles clear or translucent colour (90 nm).

Individual small gold nanoparticles appear red; however, when particles aggregate together the plasmon resonances can combine. The particle will appear as one large particle rather than two separate ones. Plasmon resonance associated absorption wavelengths will shift from blue to red, and reflected light will shift from red to blue. Therefore particle colour will change from red to blue on aggregation.

Nanoparticles may be synthesized in several different ways. The present method is called **chemical reduction**. The metal salt (hydrogen tetrachloroaurate) is taken and reduced and stabilized with sodium citrate. What happens is that the gold salt is converted by the sodium citrate into gold particles a few

nanometres across. The sodium citrate then serves another purpose by acting as a charge **stabilizer** as it surrounds the gold particles and prevents them from touching each other and sticking together (like repels like, so when the negatively charged citrate ions come close, they repel each other like magnets and the gold particles don't stick together – the colour is stable).

CHEMICAL CHAMELEON

AIM: To make colours with chemistry – a 'chemical chameleon'.

REQUIREMENTS

- Potassium permanganate
- Sodium hydroxide solution
- Sugar (sucrose)
- Distilled water
- Beaker or glass
- Spatula



THEORY

When a very dilute solution of potassium permanganate is slowly reduced to colloiddally dispersed manganese (IV) oxide, then a beautiful range of colours is traversed, starting from purple/violet and ending in yellow/brown. It is amazing to see the solution change colour. That's why permanganate is sometimes called the 'chemical chameleon' due to its extraordinary ability to change colours when changing oxidation states. In this particular experiment, diluted potassium permanganate is added to a solution of sugar, water, and sodium hydroxide. Sugar (sucrose) is an organic compound, which contains many –OH groups. These groups reduce the permanganate ion, which leads to the colour changes. This experiment is easy to perform, fun to watch and a great way to visibly see the effects of oxidation and reduction in chemical experiments. It involves a

redox reaction, i.e. one which involves exchange of electrons between chemical species. Here, the potassium permanganate is **reduced**, meaning it gains electrons, and the sugar is **oxidized**, meaning it loses electrons.

PROCEDURE

- Dissolve about one teaspoon of sugar in sodium hydroxide solution (solid sodium hydroxide + distilled water).
- Then slowly pour half a test tube of potassium permanganate into the above solution.
- Then let the beaker stand for a while and watch the colour change from purple to yellow/brown.

OBSERVATIONS

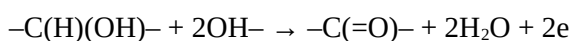
After adding potassium permanganate to the solution, the colour changes are observed from purple/violet to yellow/brown. This takes a few minutes, the exact period depending on temperature and concentration of the reactants. The photographs of all changes are given below. Between the pictures, there is an interval of time of approximately 10 seconds, but precise times cannot be predicted easily. In another run, the change of colours may go faster or slower, but the order and type of colours always will be the same. The colour first darkens, then it goes to green and gradually it shifts towards yellow/brown.



RESULTS AND DISCUSSION

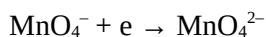
Permanganate is slowly reduced by sugar in alkaline environments. Sugar is an organic compound, having many -OH groups, attached to carbon atoms, which also have a hydrogen atom attached directly to it. Such organic compounds, containing -C(H)(OH)- structures (secondary alcohol groups) are easily oxidized.

The oxidation of the -C(H)(OH)- structure is as follows, where the alcohol group is oxidized to a ketone group:

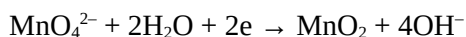


This reaction requires hydroxide ions. The observed speed of the reaction indeed is strongly dependent on the concentration of sodium hydroxide. When a lot of sodium hydroxide is used, then the first part of the reaction only takes a few seconds instead of ten seconds or more.

In alkaline environment, permanganate ion first is reduced to manganate ion:



The left is deep purple, the right is deep green. When both are present, then light in the red end of the spectrum is absorbed by the green manganate, and at the same time, light at the blue end of the spectrum is absorbed by the violet permanganate. This combination of absorptions make the solution almost appear black, hence the darkening at the start of the experiment. When almost all permanganate is reduced to manganate, then the liquid looks beautifully deep green. When there is excess sugar, then the manganate in turn is reduced further as follows:



At the very low concentrations used in this experiment, the MnO_2 does not precipitate, but a colloidal solution of hydrous MnO_2 is formed, which remains clear. Hydrous MnO_2 is brown, but at the low concentrations used in this experiment, it is more yellow than brown.

PRECAUTIONS AND SAFETY

- Potassium permanganate is a strong oxidizer. However, in this experiment a very dilute solution is used, so there is no real risk.
- Sodium hydroxide is very corrosive to human tissue. Be careful with the solid and concentrated solutions. If any of this solid comes in contact with the skin, then rinse with water, until the slippery feeling is gone.
- DISPOSAL AND CLEAN UP: The waste of this experiment only contains mg quantities of manganese and as such it can be flushed down the drain. If any brown stains of manganese dioxide remain on the glassware after the experiment, then rinse with dilute hydrochloric acid (around 5%) to which a small amount of sodium sulphite or hydrogen peroxide is added. This liquid removes brown stains of manganese dioxide at once.

CHEMICAL FIRE

AIM: To observe fire as potassium permanganate oxidizes glycerine.

REQUIREMENTS

Potassium permanganate (KMnO₄), glycerine, sand, petri dish or sand bath.

PRINCIPLE

Oxidation is the loss of electrons or an increase in oxidation state by a molecule, atom, or ion. Anything that oxidizes another substance is called an oxidizing agent. KMnO₄ is a strong oxidizing agent. A few drops of glycerine are placed into a hollow in a pile of potassium permanganate. Since, Potassium permanganate is a strong oxidizing agent and glycerine is an easily oxidized substance, consequently a redox type reaction is expected between these two substances. An exothermic reaction does occur with the glycerine boiling giving off smoke and eventually producing a flame that spreads around the pile of potassium permanganate. Eventually all the glycerine is consumed by the permanganate.

The reaction that occurs is:



PROCEDURE

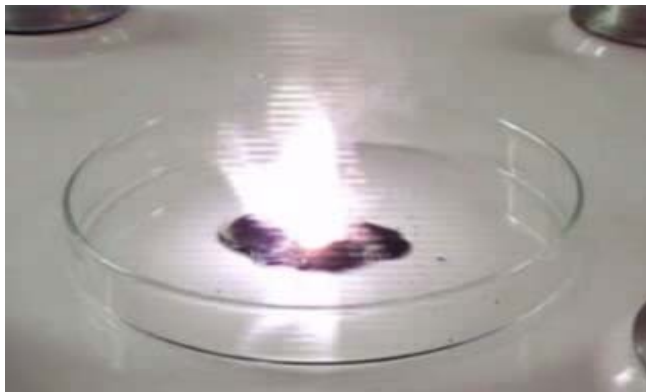
- Grind about a teaspoon of the KMnO₄ into a powder and place in a pile in the sand bath or petri dish.
- Make a small dimple in the pile with a piece of chalk, and add three drops of glycerine into the KMnO₄.
- Observe the reaction. Observations suggest a chemical change occurred.

OBSERVATIONS

A fire is ignited in the petri dish (or sand bath). The reaction produces both flames and sparks.

PRECAUTIONS

- Be careful to stand away from the dish after adding glycerine. Bits of hot solids are often expelled several centimetres.



GREEN FIRE

AIM: To produce 'green fire' using easily available reagents.

REQUIREMENTS

Boric Acid; methanol; metal sand bath or porcelain dish; lighter or matches

WHAT IS HAPPENING?

The green colour is produced because of the presence of boric acid. When we take a boron compound and energize it in a flame, that energy is absorbed by an outer electron. The electron is energized to a higher shell and when it returns to a lower shell, it loses energy as it falls back and emits the energy that it absorbed earlier as visible light. The path taken by the electron to return varies in compounds of different elements and so does the colour associated with them. It is because of this that we see a green flame with boron compounds. In this case, the methanol used as fuel forms volatile trimethoxy borate $B(OCH_3)_3$ which is easy to ignite.

PROCEDURE

- Pour some methanol into the dish. How much you use will determine how long your fire will burn. A quarter of a cup of methanol is sufficient for approximately 5 minutes of fire.
- Sprinkle some boric acid into the liquid and swirl it around to mix it up (1-2 teaspoons of powder). It won't all dissolve, so don't worry about some powder at the bottom of the container.
- Set the container on a heat-safe surface and ignite it with a lighter.

OBSERVATIONS

A brilliant green fire is observed.

PRECAUTIONS:

- Boric acid is a relatively safe household chemical. You can rinse the residue remaining in the container down the drain.
- Methanol is **flammable so be cautious** in its use. It is also **toxic**.
- Be sure to set your container on a heat-safe surface.



It is fairly easy to make other **coloured flames** in the laboratory by adding these chemicals:

Chemicals Used to Colour Flames

1. Red - strontium salts, most easily found in road flares
 2. Orange - calcium chloride (laundry bleaching agent)
 3. Yellow - sodium nitrate (common in chemistry lab)
 4. Green - barium salts, such as barium nitrate (common in chemistry lab)
 5. Greenish-Blue - copper sulphate (common in a chemistry lab, also found in many algacides for pool treatment)
 6. Blue - copper chloride (common in chemistry lab)
 7. Purple - potassium permanganate (common in a chemistry lab, also used in sewage or water treatment)
 8. White - magnesium sulphate (Epsom salts, found in a laundry or a pharmacy)
- Use caution when considering other metals salts in making coloured flames, as some compounds may produce toxic smoke.

PURPLE VAPOUR CLOUD

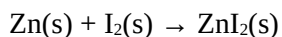
AIM: To see the action of zinc dust on solid iodine and produce a purple vapour cloud

REQUIREMENTS: Zinc dust; solid iodine; sand bath or tin lid; mortar and pestle; water and dropper



THEORY

The chemical properties of iodine are very similar to those of bromine and chlorine. However, its reactions are far less vigorous. It can also act as an oxidant for a number of elements such as phosphorus, aluminium, zinc and iron, although increased temperatures are generally required. Oxidation of finely powdered zinc with iodine can be initiated using drops of water. The reaction is strongly exothermic, and the excess iodine vaporises, forming a deep violet vapour. The reaction is:



PROCEDURE

- Finely grind 1 teaspoon of iodine in the mortar.
- Carefully mix the iodine with approximately 1/2 teaspoon of zinc dust and place the mixture on the tin lid or sand bath as a mound.
- Put one or two drops of water onto the top of the mound using the dropper. There can be an induction period before the reaction starts but if there appears to be nothing happening add one or two more drops of water.
- When the reaction starts, clouds of purple iodine vapour are released as heat is generated.

RESULT

Purple iodine vapour cloud is observed, leaving a white residue.

Note:

1. The resulting slurry is to be disposed off with plenty of water.
2. Preferably do this experiment outside your lab or do it using a fume cupboard.

WRITING WITH INVISIBLE INK

AIM: To write a secret message with invisible ink and read it after ‘developing’

REQUIREMENTS: For heat- activated inks: Milk, baking soda, vinegar, soapy water, sucrose, diluted aerated drink; paper, brush and oven

WHAT IS HAPPENING?

Invisible ink is any substance that can be used to write a message that is invisible until the ink is revealed. The ink is used by writing the message with it using a cotton swab, dampened finger, fountain pen, brush or

toothpick. A normal message may be written on the paper so that it doesn't appear to be blank and meaningless. How you reveal the message depends on the ink you used. Most invisible inks are made visible by heating the paper. Ironing the paper or holding it over a 100-watt bulb are easy ways to reveal these types of messages. Some messages are developed by spraying or wiping the paper with a second chemical. Other messages are revealed by shining an ultraviolet light on the paper.

PROCEDURE

- Use the solutions as 'ink' by dipping a stick or paintbrush and writing on paper.
- Allow the paper to dry.
- When you are ready to read your invisible message, hold the paper up to sunlight, a light bulb (recommended), or other heat source.
- The heat will cause the writing to darken to a pale brown, so your message can now be read.

OBSERVATIONS

The ink became visible after heating it in an oven for some time. The best form of invisible ink was obtained using baking soda.

PRECAUTIONS: Avoid using lined paper or any other ink since their ink could run into your invisible ink.



OSCILLATING CHEMICAL REACTION

AIM: To perform an oscillating chemical reaction (Briggs-Rauscher reaction) and observe the colour changes

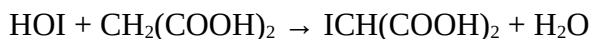
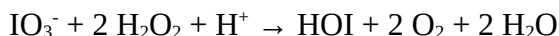
REQUIREMENTS: 30% H₂O₂; soluble starch; potassium iodate (KIO₃); deionized water; 1 M H₂SO₄; manganese (II) sulphate (MnSO₄•H₂O); malonic acid; large beaker; graduated cylinders (3); stir plate / stir bar

THEORY: An **oscillating chemical reaction** also called **chemical clock** is a complex mixture of reacting chemical compounds in which the concentration of one or more components exhibits periodic changes, especially in colour, or where sudden changes occur after a predictable induction time.

PROCEDURE:

- Solution A: Prepare 100 mL of 9% H₂O₂ by diluting 30 mL of 30% H₂O₂ with 70 mL of deionized water.
- Solution B: Prepare an acidified 0.2 M KIO₃ solution by adding 10 mL of 1.0 M H₂SO₄ to 80 mL of deionized water. Dissolve 4.3 g KIO₃ in this solution and dilute to 100 mL.
- Solution C: Prepare starch solution by dissolving 0.1 g of soluble starch in 90 mL of boiling deionized water. When cool, add 1.5 g malonic acid, 0.4 g MnSO₄•H₂O, stir and dilute to 100 mL.
- Add 50 mL of Solution A to a clean beaker fitted with a stir bar. Next add 50 mL of Solution B and let solutions mix thoroughly. Once complete, add 50 mL of Solution C and let reaction stir. Upon addition of the final solution, bubbles should appear. The solution will turn yellow then blue, then colourless. This reaction will oscillate for 5-10 minutes.

DISCUSSION: The three colours observed are a result of an increase in the concentrations of I₂ (yellow), starch-iodine complex (blue), and I⁻ (colourless) in order of appearance during the demonstration. The BR reaction can be summarized by two chemical reactions:



When the reaction begins, the solution becomes yellow as I⁻ (generated from reaction of HOI with H₂O₂, not shown) reacts with HOI under acidic conditions to produce I₂. The solution suddenly changes to dark blue as the accumulated I⁻ reacts with I₂ in the presence of starch to generate the penta-iodide ion (I₅⁻) which is encased in the amylose present in solution. The reaction proceeds by consuming I₂ at a rate greater than it is being produced thus removing the presence of I₅⁻ and increasing the concentration of I⁻ (colourless). The oscillations continue until malonic acid or IO₃⁻ is completely consumed.

PRECAUTIONS: Wear proper protective equipment including gloves and safety glasses when preparing and performing this demonstration. Concentrated hydrogen peroxide can cause burns. KIO₃ is an oxidizer. Sulphuric acid is severely corrosive to eyes, skin and other tissue. Malonic acid solution is moderately toxic and corrosive to eyes, skin and respiratory tract. The reaction produces

iodine which is toxic by inhalation and irritating to eyes, skin, and respiratory tract. Perform the demonstration in a well-ventilated room.

Disposal: Dispose of final solution in an appropriate aqueous waste container.