



Mustansiriyah University
College of Dentistry

Practical Medical Chemistry
First Stage

Lab (11)

(Aldehydes and Ketones)

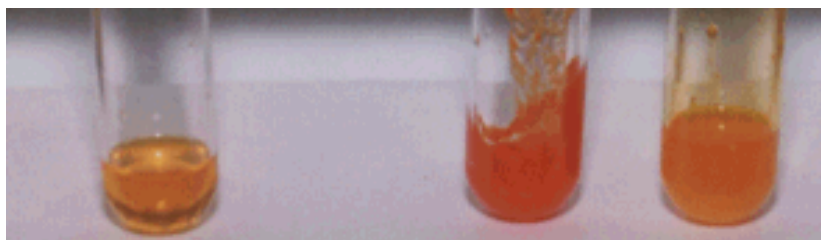
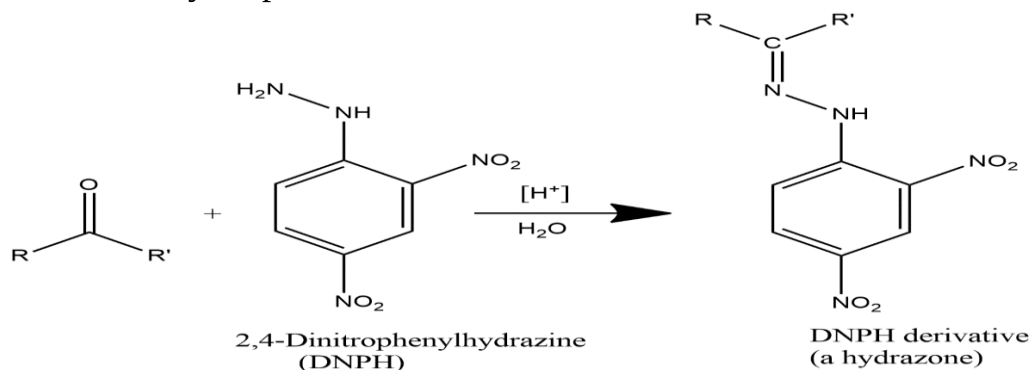
By

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Aldehydes and Ketones

Both aldehydes and Ketones contain the carbonyl group, hence a general test for carbonyl compounds will immediately characterize both classes of compounds. The preferred reagent is 2,4-dinitro phenyl hydrazine (**Brady's reagent**) which gives **insoluble phenyl hydrazone** with carbonyl compounds.



Reagent

Positive test

Brady's reagent. (2,4 dinitrophenyl hydrazine)

1. Add 0.5 g of 2,4-DNPH to about 12–13 cm³ of concentrated phosphoric acid. Do not use old phosphoric acid for the preparation of Brady's reagent.
2. Stir until the 2,4-DNPH has completely dissolved in the acid before moving on to Step 3. Dissolving may take 10–15 minutes.
3. Once the 2,4-DNPH has dissolved, make up to a total volume of 25 cm³ with ethanol. Stir to mix.

Brady's test for carbonyl group

1. To 1 drop of the sample, add 1 ml. of 2,4-dinitrophenyl hydrazine, wait for 10 minutes.
2. Orange precipitate will be formed.

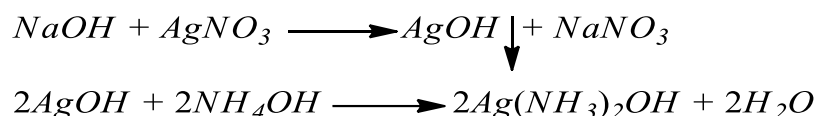
Aldehydes can be oxidized to carboxylic acids using oxidizing agents such as Tollens's and Fehling's reagents.

Tollens's test

Tollens' test, also known as silver-mirror test, is a qualitative laboratory test used to distinguish between an aldehyde and a ketone. It exploits the fact that aldehydes are readily oxidized, whereas ketones are not. Tollens' test uses a reagent known as Tollens' reagent, which is a colorless, basic, aqueous solution containing silver ions coordinated to ammonia. It is prepared using a two-step procedure.

Step 1: Aqueous silver nitrate is mixed with aqueous sodium hydroxide.

Step 2: Aqueous ammonia is added drop-wise until the precipitated silver oxide completely dissolves.



Tollens' reagent oxidizes an aldehyde into the corresponding carboxylic acid.

Preparation of Tollen's reagent: (freshly prepared)

Step 1 : In a clean test tube, add **2 mL** of **0.1M** or **(5%) Silver Nitrate** solution.

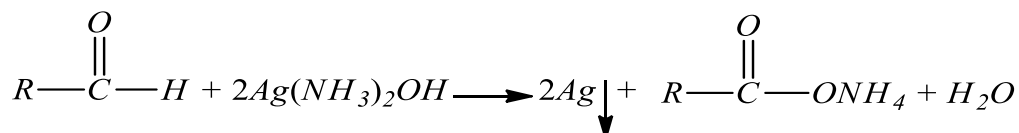
Step 2 : Add **2-1 drops** of **0.1 M** or **(10%) Sodium Hydroxide** solution. A **brown precipitate** of silver oxide will form immediately.

Step 3 : Add **dilute** NH_4OH (30% V/V) solution drop by drop while shaking the tube constantly.

Step 4 : Continue adding ammonia **only until** the brown precipitate completely dissolves and the solution becomes clear. This clear solution is the **Tollen's Reagent**

Procedure:

1. **In a clean test tube** add 2-3 drops of sample (aldehyde or ketone) to Tollen's reagent.
2. Mix the contents gently .Do not shake vigorously
3. Warm in hot Water bath for about 5-10 min.
 - a. **Positive Result** :A shiny Silver Mirror forms on the inner wall of the test tube (indicates an Aldehyde).
 - b. **Negative Result** :The solution remains clear or turns slightly cloudy (indicates a Ketone).



Safety & Cleaning

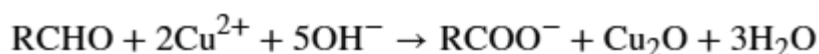
- **Immediate Cleaning** :After the test, immediately wash the test tube with **dilute Nitric Acid** to dissolve the silver mirror and prevent the formation of explosive silver nitride.
- **Disposal** :Never store the reagent; always dispose of the residue by neutralizing it with acid and rinsing with plenty of water.

Fehling 's test

Fehling's test is a chemical analysis used to distinguish between reducing sugars (e.g., glucose, lactose) and non-reducing sugars, as well as to identify aldehyde functional groups. It acts as a redox test, where a positive result produces a brick-red precipitate of copper(I) oxide (Cu_2O) from a deep blue copper(II) solution upon heating

The test relies on the oxidation of an aldehyde to a carboxylate ion by copper(II) ions under alkaline conditions.

Aldehydes (but not most ketones) reduce the ions in the complex to red copper(I) oxide



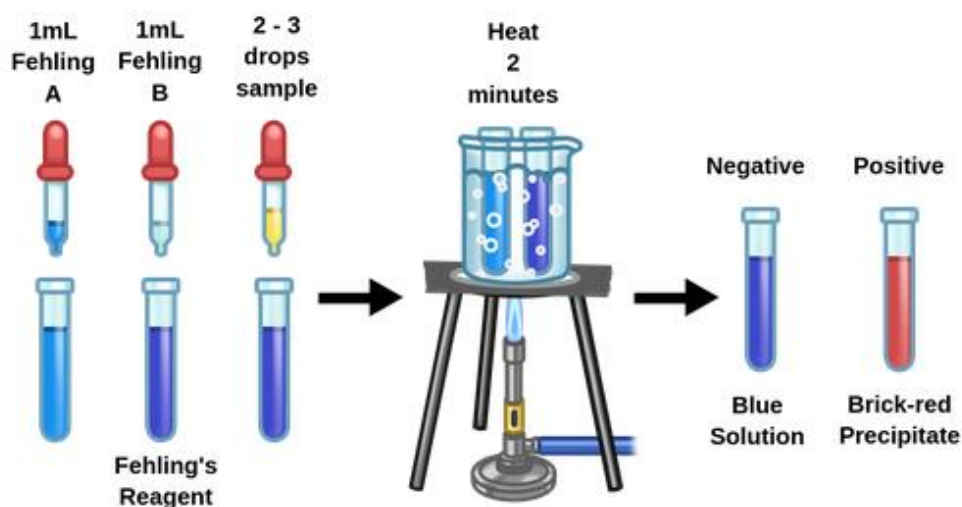
Fehling 's solution (Freshly prepared)

Fehling's solution is prepared by mixing equal parts of **Fehling's A** (aqueous copper sulfate, blue) and **Fehling's B** (colorless potassium sodium tartrate and sodium hydroxide) to create a deep blue copper tartrate complex.

1. **Fehling's A** : Dissolve 7 g of CuSO_4 in 40 ml of distilled water, add 2-3 drops of c. H_2SO_4 , complete the volume to 100 ml with distilled water.
2. **Fehling's B** : Dissolve 10g of NaOH and 35g of sodium potassium tartartate in 100 ml distilled water, filter if necessary
3. mix exactly equal volumes of(1) and (2) immediately before use.

Procedure:

1. Place 2 ml. of freshly prepared Fehling's solution in a test tube.
2. Add 2-3 drops of a sample and boil for 2-3 minutes.
3. A brick red precipitate of Cu_2O is formed.



Iodoform Test:

The iodoform test is a specific example of the haloform reaction, where a methyl ketone $\text{CH}_3\text{-CO-R}$ reacts with iodine in the presence of a strong base to produce iodoform (triiodomethane, CHI_3) and a carboxylate salt.

The formation of iodoform, a pale yellow solid with a characteristic "antiseptic" or "medical" odor, provides a **positive test**. The reaction is highly specific for compounds containing the R-CO-CH_3 group or those that can be oxidized to this structure under the reaction conditions, such as ethanol ($\text{CH}_3\text{CH}_2\text{OH}$) and secondary alcohols of the type $\text{CH}_3\text{-CHOH-R}$. Only one aldehyde CH_3CHO gives a positive haloform test. A few methyl ketones fail to give a positive reaction.

General Procedure:

1. Dissolve approximately 0.1 g of a solid sample or 4-5 drops of a liquid sample in 2 mL of water or dioxane (if insoluble in water or pure methanol: acetone and with n-butyraldehyde) in a clean test tube.
2. Add 2 mL of 5% aqueous sodium hydroxide (NaOH) solution.
3. Add a 10% iodine-potassium iodide (I_2/KI) solution dropwise while shaking, until a dark brown color of excess iodine persists (indicating an excess of iodine).
4. Allow the mixture to stand for 2-3 minutes at room temperature.
5. If no precipitate forms, gently warm the test tube in a water bath at approximately 60°C for 2-3 minutes.
6. If the brown color fades, add more I_2/KI solution until the color persists for at least two minutes of heating.
7. Neutralization: Add a few drops of 5% NaOH solution to remove excess iodine, until the solution is colorless or pale yellow.
8. Dilute the mixture with an equal volume of water and allow it to stand for 10-15 minutes.
9. Result: The formation of a pale yellow, crystalline precipitate of iodoform (CHI_3) with a characteristic antiseptic smell indicates a positive test. It is prudent to confirm the identity of the crystals by collecting them with suction, drying and taking the melting point (119°).
10. Before using a solvent it should be tested to see if iodoform-producing impurities are present.

