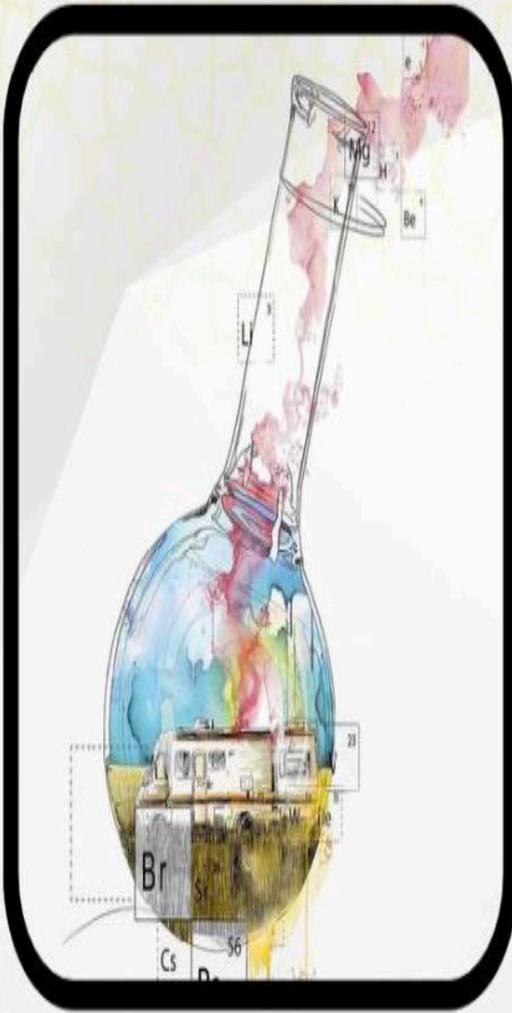


# تفريغ مختبر عضوية



اسر الموضوع: Exp : Carboxylic Acid

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رب اشرف لي هدي  
ويسر لي آمدي ❤️



لجان الرفعات

# EXPERIMENT 5: IDENTIFICATION OF CARBOXYLIC ACIDS

## General and Individual Identification Tests

### INTRODUCTION

Carboxylic acids are organic acids characterized by the presence of at least one carboxyl group. The general formula of a carboxylic acid is R-COOH. Carboxylic acids are Brønsted-Lowry acids; they are proton donors. They are the most common type of organic acid. Among the simplest examples are the formic acid H-COOH, that occurs in ants, and acetic acid CH<sub>3</sub>-COOH group, that gives vinegar its sour taste.

ROO<sup>-</sup> + Metals  
(Na<sup>+</sup>, K<sup>+</sup>, Li<sup>+</sup>, NH<sub>4</sub><sup>+</sup>)  
(oate)

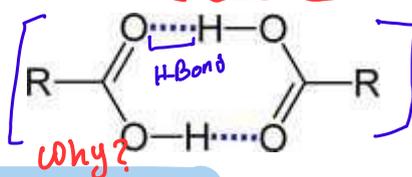
### Physical and Chemical Properties of Carboxylic Acids:



#### ✓ Solubility in water

Carboxylic acids usually exist as dimeric pairs in nonpolar media due to their tendency to "self-associate". But in the presence of water, the carboxylic acids don't dimerize. Instead, hydrogen bonds are formed between water molecules and individual molecules of acid. The solubility of the bigger acids decreases very rapidly with size. This is because the longer hydrocarbon "tails" of the molecules get between water molecules and break hydrogen bonds. In this case, these broken hydrogen bonds are only replaced by much weaker van der Waals dispersion forces.

Solubility of  
1  
molecular weight



#### ✓ Boiling Point

Carboxylic acids tend to have higher boiling points than water, not only because of their increased surface area, but because of their tendency to form stabilized dimers. Carboxylic acids tend to evaporate or boil as these dimers.

B.P of acid >  
B.P of water

what must happen

For boiling to occur? either the dimer bonds must be broken, or the entire dimer arrangement must be vaporized. Higher boiling points than similar alcohols due to dimer formation. (Acetic acid, b.p. 118 °C)

#### ✓ Melting Point

- Aliphatic acids with more than 8 carbons are solids at room temperature.

> 8 carbons

↳ Solids at R.T

- Double bonds (especially cis) lower the melting point.
- Note these 18-C acids:

M.P. عالية ← No Cis double Bond

- Stearic acid (saturated): 72°C
- Oleic acid (one cis double bond): 16°C
- Linoleic acid (two cis double bonds): -5°C

low m.p.

### ✓ Acidity of carboxylic acid

Carboxylic acids are typically weak acids, meaning that they only partially dissociate into H<sup>+</sup> cations and RCOO<sup>-</sup> anions in neutral aqueous solution. For example, at room temperature, only 0.02 % of all acetic acid molecules are dissociated. Electronegative substituents give stronger acids.

[acidity ↑, pKa ↓] dissociation will be low in water

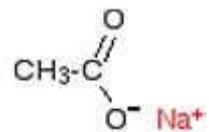
Carboxylic Acids	pKa
Formic acid (HCO <sub>2</sub> H)	3.77
Acetic acid (CH <sub>3</sub> COOH)	4.76
Chloroacetic acid (CH <sub>2</sub> ClCO <sub>2</sub> H)	2.86
Dichloroacetic acid (CHCl <sub>2</sub> CO <sub>2</sub> H)	1.29
Trichloroacetic acid (CCl <sub>3</sub> CO <sub>2</sub> H)	0.65
Trifluoroacetic acid (CF <sub>3</sub> CO <sub>2</sub> H)	0.5
Oxalic acid (HO <sub>2</sub> CCO <sub>2</sub> H)	1.27
Benzoic acid (C <sub>6</sub> H <sub>5</sub> CO <sub>2</sub> H)	4.2

pKa = -log Ka

Ka ↑, acidity ↑, pKa ↓

### ❖ Salts of Carboxylic Acids

When the acids form salts, this is lost and replaced by a metal. Sodium ethanoate, for example, has the structure:



sodium ethanoate → لو بيدي ارجعه لـ acid بفاعله مع strong acid

- Sodium hydroxide removes a proton to form the salt.
- Adding a strong acid, like HCl, regenerates the carboxylic acid.

### ➤ Properties of acid Salts

- Usually solids with no odor.
- Carboxylate salts of Na<sup>+</sup>, K<sup>+</sup>, Li<sup>+</sup>, and NH<sub>4</sub><sup>+</sup> are soluble in water.
- Soap is the soluble sodium salt of a long chain fatty acid.
- Salts can be formed by the reaction of an acid with NaHCO<sub>3</sub>, releasing CO<sub>2</sub>.
- The bond between the sodium and the ethanoate is ionic.

# كحل لكاربوكسيك اسيد

## GENERAL TESTS FOR CARBOXYLIC ACIDS:

تجريبية (بتبين اذا موجود بالعينة او لا)

### 1. Sodium Carbonate test (Demonstration Only)

They are soluble in both dilute sodium hydroxide and sodium carbonate

(Na<sub>2</sub>CO<sub>3</sub>) or sodium bicarbonate solutions (NaHCO<sub>3</sub>). Sodium hydrogen

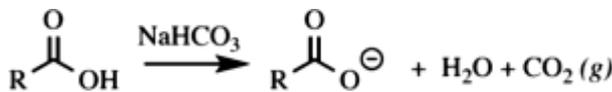
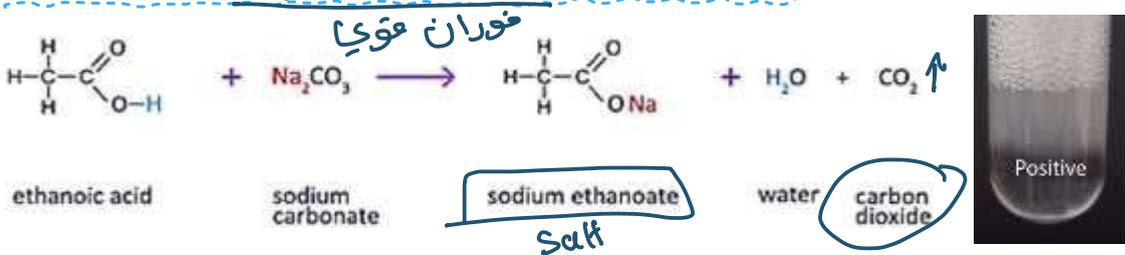
carbonate (NaHCO<sub>3</sub>) or sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) reacts with carboxylic

acids to give the sodium salt of the acid and liberates carbon dioxide. If the

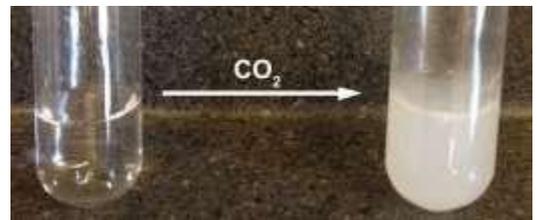
acid is insoluble in water and the reaction is sluggish dissolve the acid in

methanol and add carefully to a saturated sodium hydrogen carbonate

solution, when a vigorous effervescence will be observed.



Limewater turns milky



### 2. Ferric chloride test

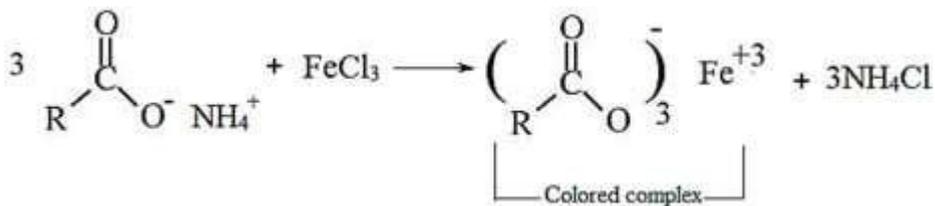
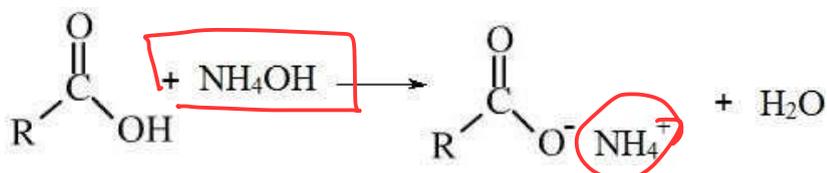
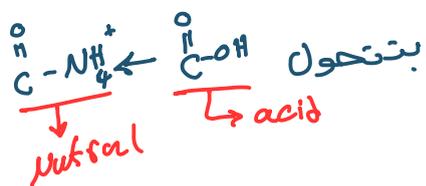
Neutral carboxylic acids form colored complexes with ferric chloride

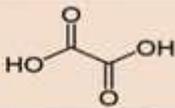
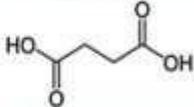
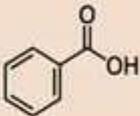
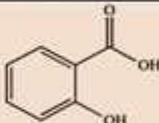
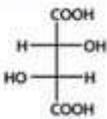
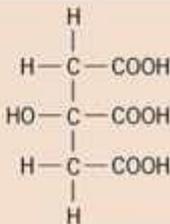
solution. The carboxylic acids are first neutralized using ammonia. The

color of the complex depends on the kind of acid.

كيف بخلع  
Neutral?

بصيف امونيا



Acids	Structure	Use/Presence	Result
Formic	HCOOH	Ants and venoms السموم	Red sol > brown ppt with heating
Acetic	CH <sub>3</sub> COOH	Vinegar	Red sol > brown ppt with heating
Oxalic		Cleaning, bleaching, removal of rust	Faint yellow ppt
Succinic		Sweetener	Brick red ppt
Benzoic		Preservative	Buff to brown ppt
Acids	Structure	Use/Presence	Result
Salicylic		Keratolytic, antifungal	Violet sol
Tartaric		Sour taste	Yellow sol
Citric		Citrus fruit	Yellow sol

### PROCEDURE



- Place 2 mL of each of the following neutral acid solutions in separate test tubes; Sodium acetate solution, Sodium succinate solution, Sodium benzoate solution, Sodium salicylate solution, Sodium oxalate solution, Sodium citrate solution, Sodium tartarate solution and Formic acid.
- Add excess Ammonium hydroxide solution till the solution is just alkaline to litmus paper.
- Boil the solution till the odor of ammonia is completely removed.
- Add few drops of neutral Ferric chloride solution.
- Note the results.

*Remember same test with phenol give violet color.*

## Identification Tests for Carboxylic acids

### 2. Ester Formation Test

- The reaction of carboxylic acid with alcohol is known as the **Fischer esterification** reaction. A mineral acid, usually sulfuric acid is used as catalyst.



- Positive Result : nice odor

لكل واحد منهم تفاعل خاص بكشف عن وجودهم

### Individual reactions of Carboxylic acids

#### Outline:

#### 1. Formic acid

- Sulfuric acid test (Carbon monoxide test): *Blue flame*
- Reducing property with Mercuric chloride test: *white ppt*
- Reducing property with acidic permanganate: *Colourless, light pink*

#### 2. Acetic acid → No Reducing power at all

#### 3. Oxalic acid

- Calcium chloride test
- Sulfuric acid test

#### 4. Succinic acid

- Fluorescein test

#### 5. Tartaric acid

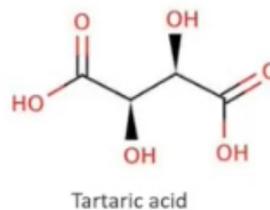
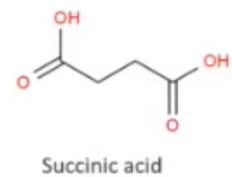
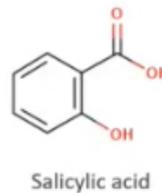
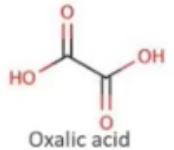
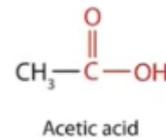
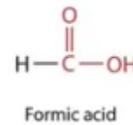
- Sulfuric acid test
- Reducing property with Tollens reagent
- Fentons reagent

#### 6. Citric acid

- Sulfuric acid test

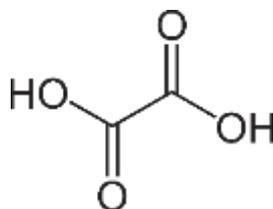
#### 7. Salicylic acid

- Phthaline formation



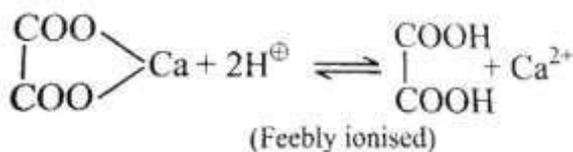
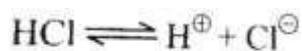
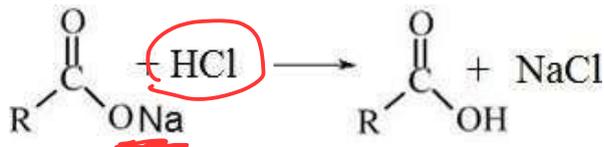
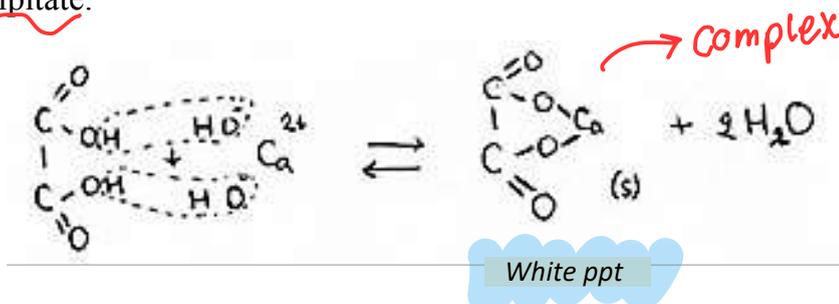
## ❖ INDIVIDUAL REACTIONS OF CARBOXYLIC ACIDS:

### ➤ Oxalic Acid



#### 1- Calcium chloride test

Carboxylic acid salts react with strong mineral acids w liberate free organic acid. If the freed carboxylic acid is water insoluble, it will precipitate.



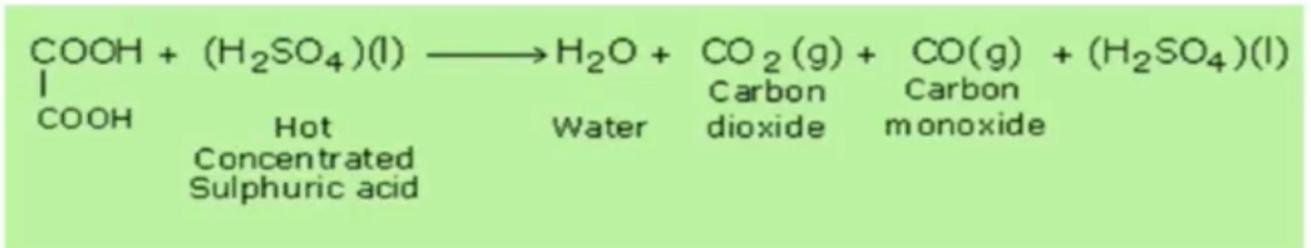
### PROCEDURE

Add Calcium chloride solution to Oxalate solution. Try the solubility of the ppt in acetic acid and dilute HCl.



### 3. Oxalic acid

#### 2. Sulfuric acid test

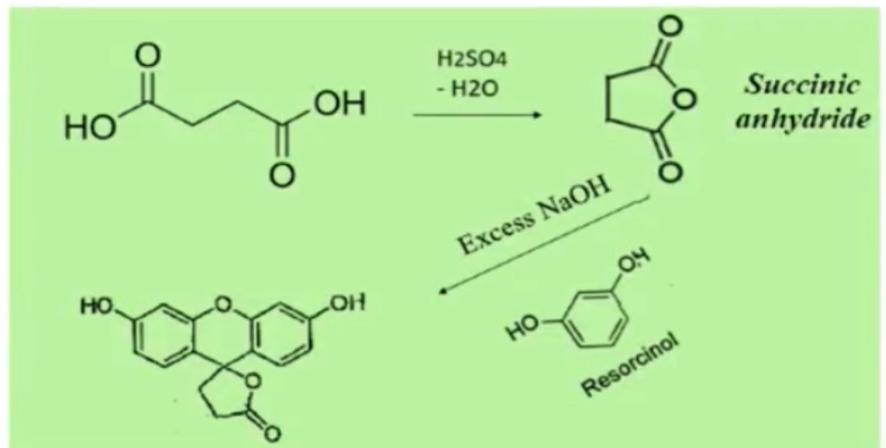


Carbon monoxide (CO) detected by **blue** flame.

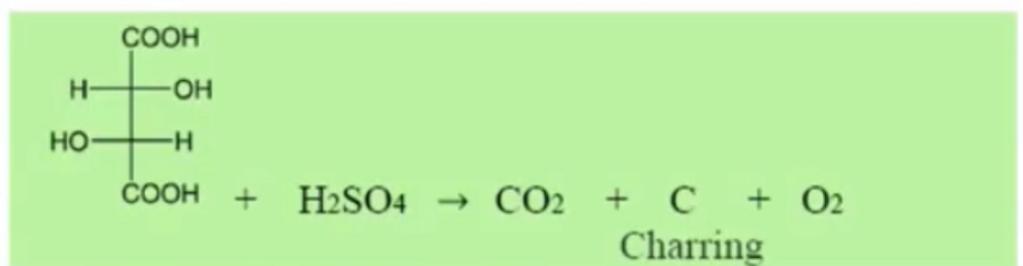
Carbon dioxide (CO<sub>2</sub>) detected by lime water.

### 4. Succinic acid

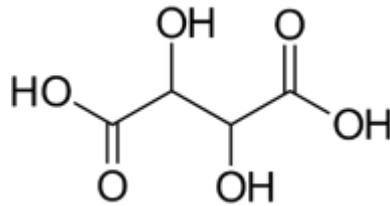
- Succinic acid forms coupling product with phenols, e.g. resorcinol.
- This product shows very brilliant color in alkaline medium, due to the formation of a special ion. at the same time there is a typical fluorescence.



### 1-Sulfuric acid test

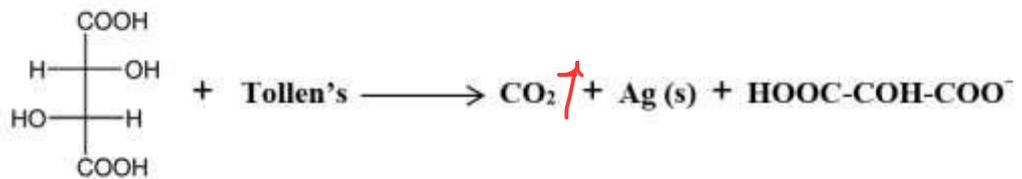


➤ **Tartaric Acid**



2- **Tollen's test**

Reducing property of tartaric acid is tested with Tollen's reagent (Reduction of Ammonical Silver nitrate).

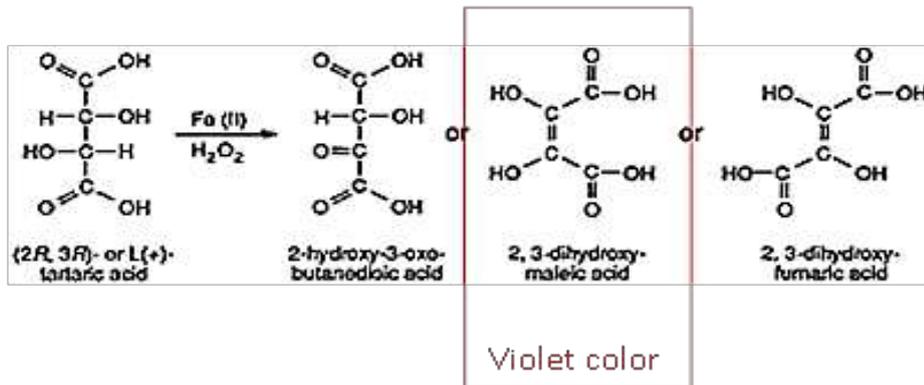


**PROCEDURE**

Prepare the Tollen's reagent then add to 5 mL of that reagent, few drops of neutral tartarate solution and place the mixture in a warmwater bath. Note the formation of a mirror.

بكون صحت  
NH<sub>3</sub>

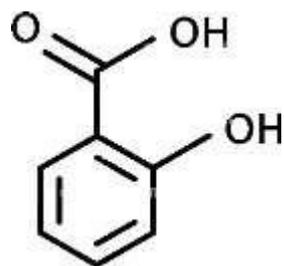
3- **Reaction with Fenton's reagent**



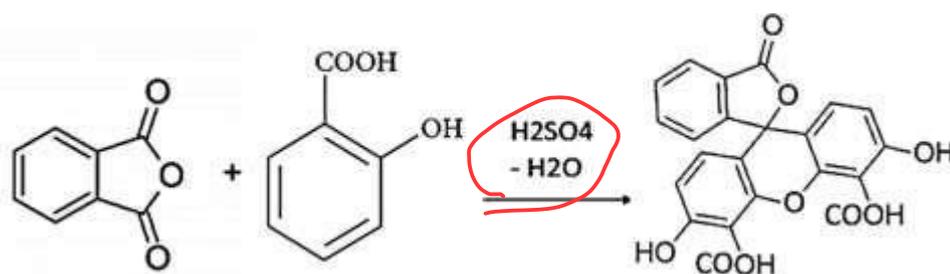
**PROCEDURE**

Add to few mLs of a tartarate solution one drop of freshly prepared Ferrous sulphate and 2 drops of Hydrogen peroxide solution followed by excess Sodium hydroxide. Notice the color.

➤ *Salicylic Acid*



➤ *Phthalein formation*



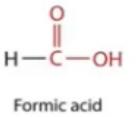
***PROCEDURE***

Prepare In a dry test tube fuse together few crystals of Salicylic acid with the same amount of Phthalic anhydride and few drops of conc. Sulfuric acid, cool then dissolve in water and add excess Sodium hydroxide. Notice the color.

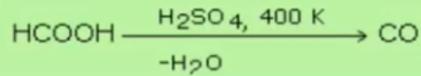


# Individual reactions of Carboxylic acids

## 1. Formic acid

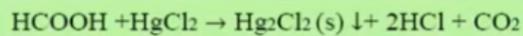


### 1. Sulfuric acid test (Carbon monoxide test):



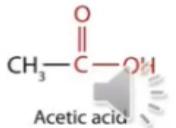
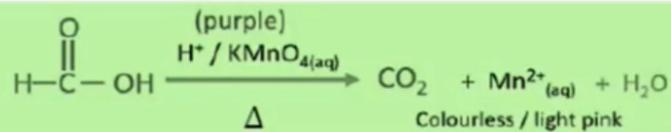
Carbon monoxide give blue flame

### 2. Reducing property with Mercuric chloride test



White ppt

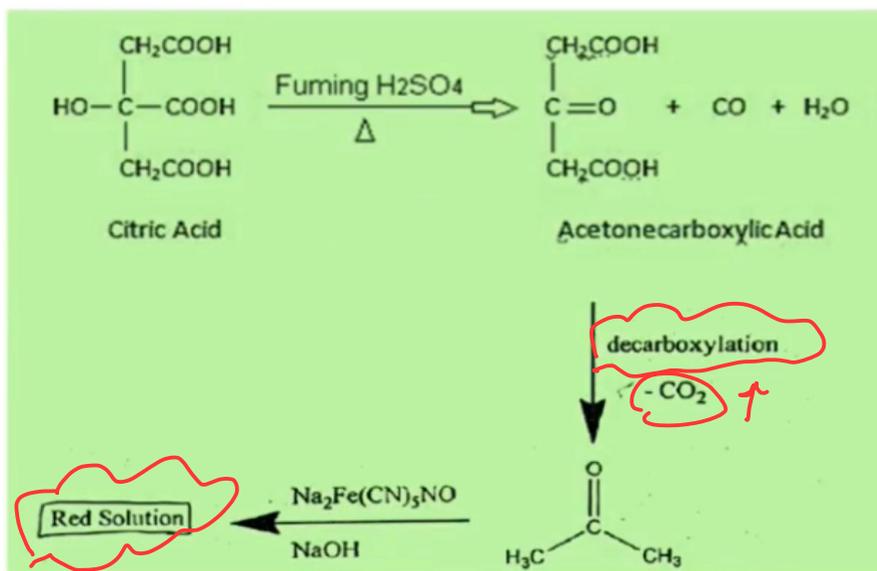
### 3. Reducing property with acidic permanganate



2. Acetic acid : This acid has no reducing power at all

## 6. Citric acid

### 1. Sulfuric acid test



**Note:** The appearance of red coloration shows the presence of ketone.

EXPERIMENT 5  
**IDENTIFICATION OF CARBOXYLIC ACIDS**  
Report Sheet

Group number		Section no:	
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➤ OBJECTIVES:

Determine chemical & physical properties  
of given carboxylic acids.

➤ IDENTIFICATION TESTS:

I. Solubility of Acids in Water

as given samples.

Acid Type	Form (salt or as is)	Solubility

II. Oxalic Acid

Test name	Observations (color, ppt.,)
Calcium chloride test	Cloudy - white ppt

III. Tartaric Acid

Test name	Observations (color, ppt.,)
Tollen's Mirror Test	Give silver mirror
Reaction with Fenton's reagent	Give Brown ppt