

# **Classic Qualitative Organic Analysis**

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**For year 4 students**

**First semester**

The classic method of qualitative organic analysis consists of six steps, each of which is discussed in the following subsections. The first four steps, which may be carried out in any order, should be completed before performing the qualitative tests for functional groups. The final step must always be the preparation of one or more solid derivatives.

1. Preliminary examination of physical and chemical characteristics.
2. Determining physical constants.
3. qualitative elemental analysis to determine the presence of elements other than carbon, hydrogen, and oxygen such as S, N and halogens.
4. Solubility tests in water, ether, dilute acid, and dilute base.
5. Functional group analysis using classification tests.
6. Derivatives

# 1-Preliminary Examination:

1.1 Physical State

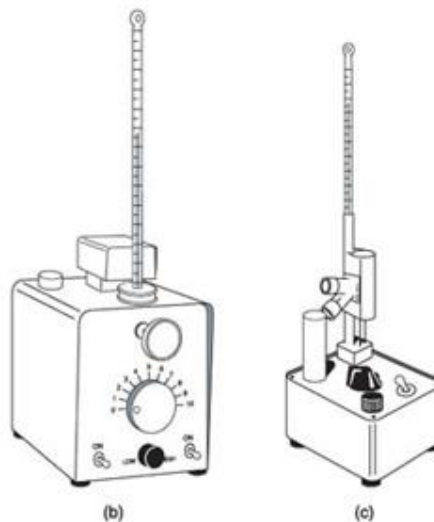
1.2 Color

1.3 Odor

1.4 Ignition Test

## 2- Determination of Physical Properties

2.1 Melting Points



2.2 Boiling Points

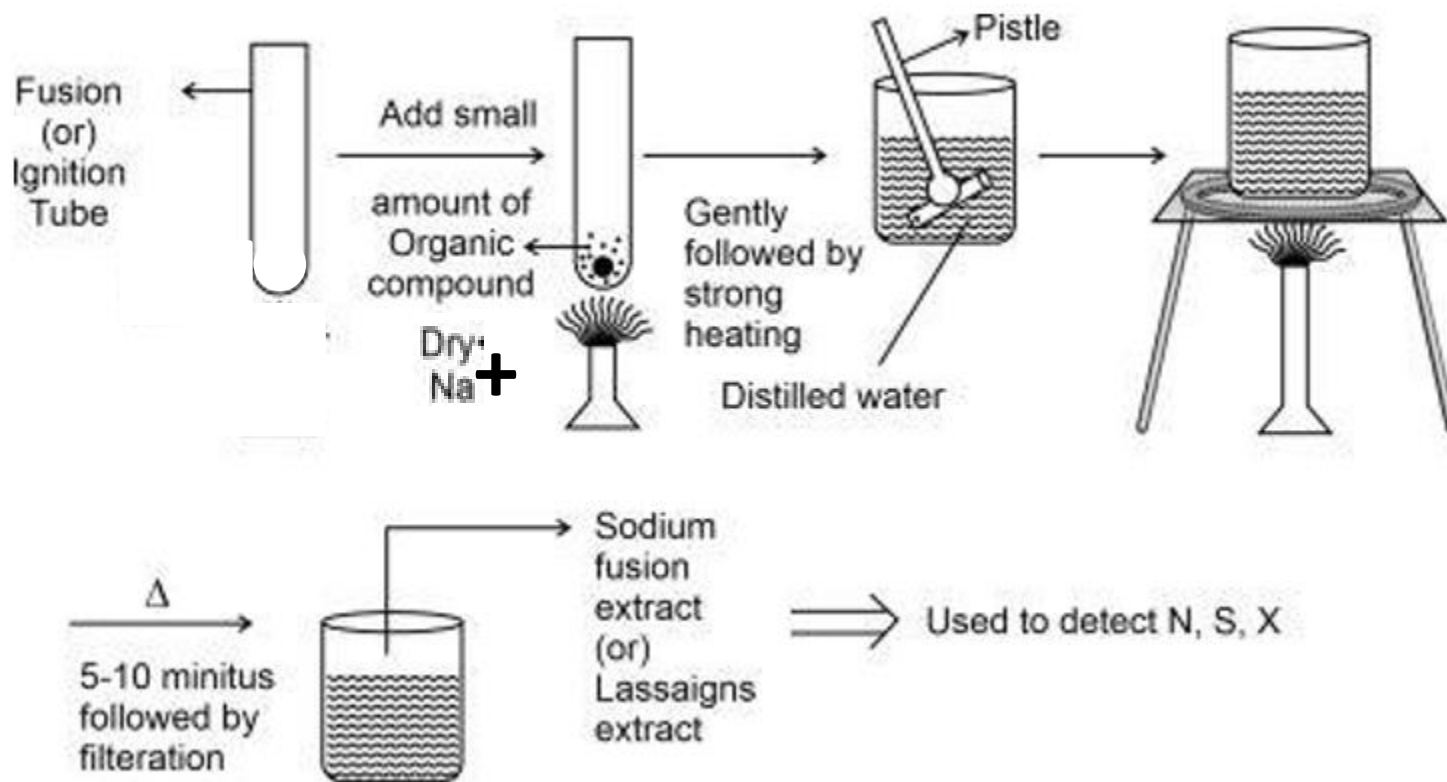
# **Qualitative Elemental Analysis**

## **Lassainge test**

## **Fusion with sodium (in the hood)**

- 1- Take a dry sodium fusion tube.
- 2- Place small and clean piece of sodium metal near the close end of the tube (the size of sodium is near the size of pea).
- 3-Catch the tube near the open end with a tong at an angle of 45 with the open end away from your body.
- 4-Heat the tube gently and carefully, on a burner ,to melt the sodium.
- 5-After melting of sodium, stop heating and catch the tube vertically. Add small amount of the unknown mixed with 50mg of crushed sucrose by means of spatula( in case of solid). In case of liquid also add few drop of the unknown with crushed sucrose by means of a dropper. Repeat this process few times to ensure complete fusion. In all cases be sure that the tube is away from your body.
- 6- After addition heat the tube to the glowing point.
- 7- Throw the glowing tube in a small beaker containing 15-20ml of D.W. (be careful).
- 8- Boil the containing of the beaker for 2 min..
- 9- Filter the hot solution. Use it for elements determination.
- 10- If the filtrate is colored, repeat the fusion.

**Figure 1: steps fusions with Na**



## Test for sulfur

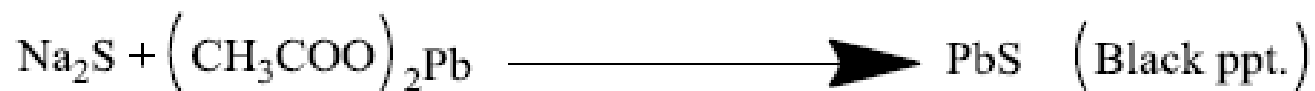
### 1. Lead acetate method

1- add 1 mL of alkaline filtrate.

2- acidify with acetic acid.

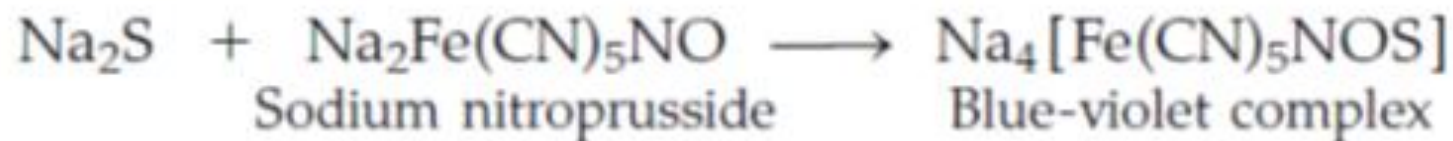
3- add one drop of 10%  $\text{Pb}(\text{OAc})_2$ .

black ppt indicates +ve test for sulfur.



## 2. Procedure for Sulfur

1. Add 1 mL of the alkaline filtrate in test tube
2. Now add 1 drop of dilute (2%) aqueous sodium nitroprusside solution. The formation of a deep blue-violet color is a positive test for sulfur:





# Test for Nitrogen

- 1- Add 1 mL of the alkaline filtrate.
- 2- Add 15-20 mg of  $\text{FeSO}_4$  and boil. upon boiling of the alkaline solution ferric produced by air oxidation.
- 3- Add conc.  $\text{H}_2\text{SO}_4$  (drop by drop) to dissolve ferrous and ferric



# Test for Halogen

## The general test

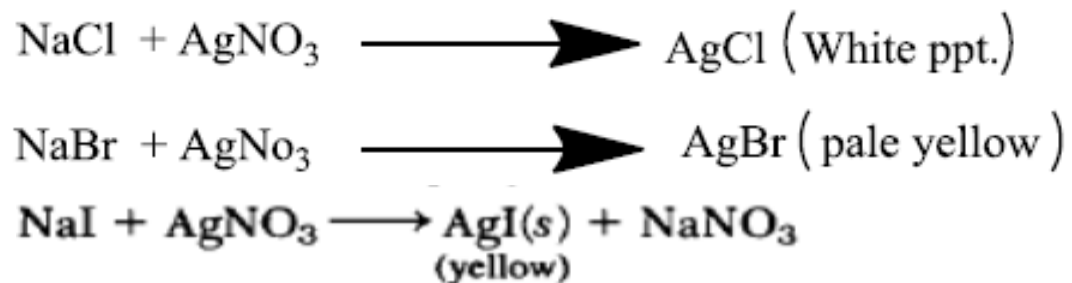
1-Add 1 mL of the alkaline filtrate.

2-acidify with dil.HNO<sub>3</sub>, drop by drop.

3-a. if N and or S are absent go to step 4

b. if N and S are present boil (in hood) to evaporate the solution to the half ( where CN<sup>-</sup> expelled as HCN and S<sup>-</sup> expelled as H<sub>2</sub>S). Then go to step 4

4- Add one drop of 5% AgNO<sub>3</sub> ,a white ppt indicates the presence of Br or Cl. If a ppt formed add NH<sub>4</sub>OH to the test tube and shake if the ppt disappear this indicate the presence of Cl otherwise it is Br.



# **Classification of Organic Compounds by Solubility**

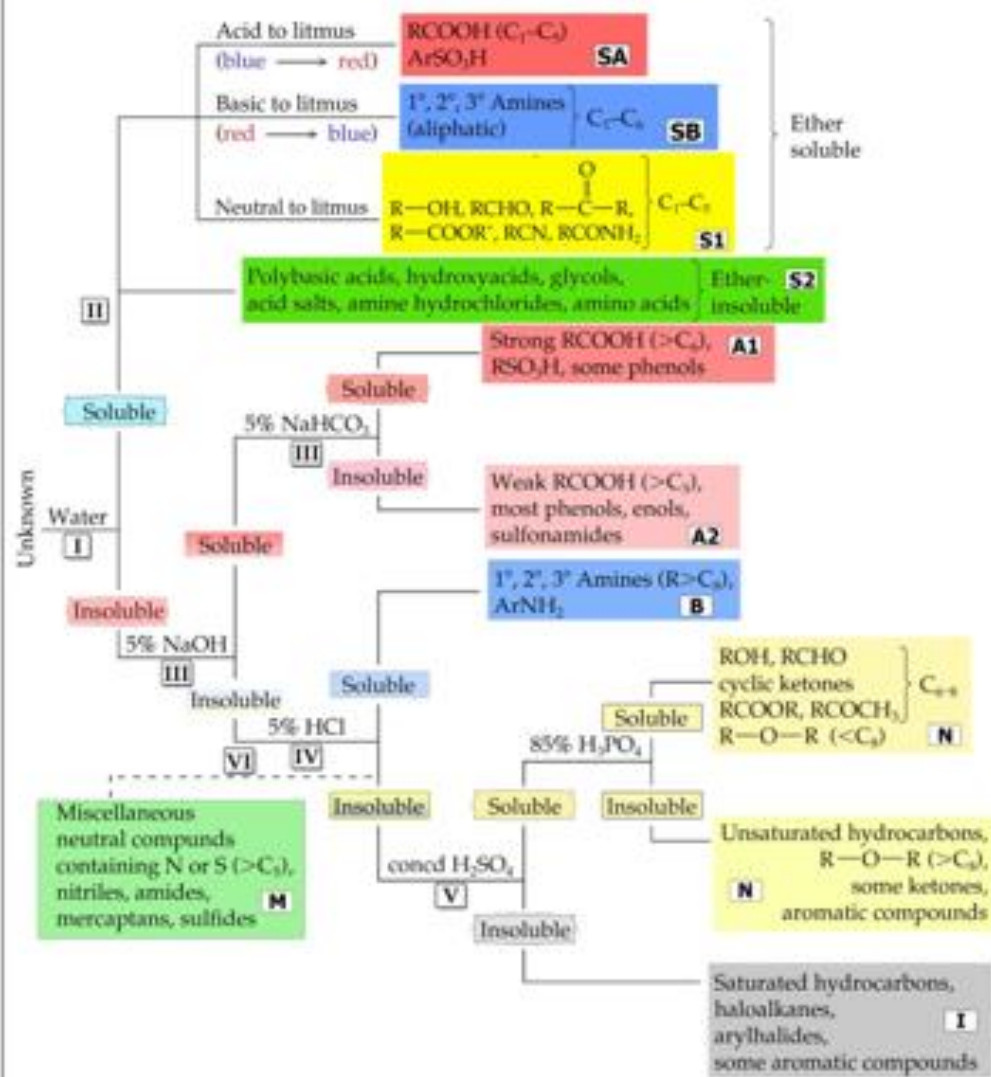
**TABLE 1: Organic Compounds Comprising the Solubility Classes of Figure**

<b>S<sub>2</sub></b>	Salts of organic acids (RCO <sub>2</sub> Na, RSO <sub>3</sub> Na); amine hydrochlorides (RNH <sub>3</sub> Cl); amino acid polyfunctional compounds with hydrophilic functional groups: carbohydrates (sugars), polyhydroxy compounds, polybasic acids, etc.
<b>S<sub>A</sub></b>	Monofunctional carboxylic acids with five carbons or fewer; aryl sulfonic acids.
<b>S<sub>B</sub></b>	Monofunctional amines with six carbons or fewer.
<b>S<sub>1</sub></b>	Monofunctional alcohols, aldehydes, ketones, esters, nitriles, and amides with five carbons or fewer.
<b>A<sub>1</sub></b>	Strong organic acids: carboxylic acids with more than six carbons; phenols with electron withdrawing groups in the <i>ortho</i> and/or <i>para</i> position(s); 1,3-diketones.
<b>A<sub>2</sub></b>	Weak organic acids: phenols, enols, oximes, imides, sulfonamides, thiophenols, all with more than five carbons; 1,3-diketones; nitro compounds with alpha-hydrogens.
<b>B</b>	Aliphatic amines with eight or more carbons; anilines (only one phenyl group attached to nitrogen); some ethers.
<b>M</b>	Miscellaneous neutral compounds containing nitrogen or sulfur and having more than five carbon atoms.
<b>N</b>	Alcohols, aldehydes, ketones, esters with one functional group and more than five but fewer than nine carbons, ethers ROR < C <sub>8</sub> and epoxides.
<b>N<sub>1</sub></b>	Alkenes, alkynes, some ether ROR > C <sub>8</sub> , some aromatic compounds (especially those with activating groups)
<b>I</b>	Saturated hydrocarbons, haloalkanes, aryl halides, other deactivated aromatic compounds, diaryl ethers.

\* Acyl halides and carboxylic acid anhydrides have not been classified because of their high reactivity.

# Solubility diagram

## مخطط الذوبانية



# Division Groups

## ***DIVISION (S<sub>1</sub>)***

### **1- Only C, H, O present:**

- a. Alcohols (Cerric Nitrate and/ or Xanthate test), if +ve then go to Lucas test
- b. Aldehydes and ketones (2,4-DNPH test), if +ve then go to Tollen's test
- c. Anhydride (Ferric hydroxamate test)
- d. Esters (Ferric hydroxamate test)
- e. Phenols (Ferric chloride test)

### **2- Nitrogen present:**

Amides (Ferric hydroxamate test and/ or Liberation of ammonia test)

## ***DIVISION (SA)***

Carboxylic acid (Iodate-iodide test)

## ***DIVISION (SB)***

### **Nitrogen present:**

Amines (Copper sulphate test), if +ve then go to (Hinsberg test)

## ***DIVISION (S<sub>2</sub>)***

### **1- Only C, H, and O present:**

- a. Carboxylic acid (Iodate-iodide test)
- b. Alcohols (Cerric Nitrate and/ or Xanthate test), if +ve then go to Lucas test
- c. Poly hydroxyphenols (Ferric chloride test)
- d. Poly hydroxyalcohols (Cerric Nitrate and/ or Xanthate test), if +ve then go to Lucas test

### **2- Nitrogen present:**

Amino acids (Ninhydrin test)

### **3- Halogen present:**

- a. Halo acids (Iodate-iodide test)
- b. Halo Alcohols (Cerric Nitrate and/ or Xanthate test), if +ve then go to Lucas test
- c. Acyl halides (Ferric hydroxamate test)

## ***DIVISION (A<sub>1</sub>)***

### **1- Only C, H, and O present**

- a. Acids (Iodate-iodide test)
- b. Anhydrides (Ferric hydroxamate test)

## **2- Nitrogen present**

- a. Nitro acids (Ferrous hydroxide test), if +ve then go to NaOH-Acetone colour test
- b. Poly nitro phenols (Ferric chloride test)

## **3- Halogen present:**

- a. Halo acid (Iodate-iodide test)
- b. Polyhalo phenols (Ferric chloride test)
- c. Acid halides (Ferric hydroxamate test)

## ***DIVISION (A<sub>2</sub>)***

### **1- Only C, H, and O present:**

- a. Phenols, Enols (Ferric chloride test)
- b. Acids (Iodate-iodide test)
- c. Anhydrides (Ferric hydroxamate test)

### **2- Nitrogen present:**

- a. Nitro phenols (Ferric chloride test)
- b. Aminophenol (Copper sulphate test), if +ve then go to (Hinsberg test)
- c. Trinitro aromatic hydrocarbons (Ferrous hydroxide test), if +ve then go to NaOH-Acetone colour test



### **3- Halogen present:**

- a. Halo phenols (Ferric chloride test)

### **4- Nitrogen and halogen present:**

- a. Poly nitro halogenated hydrocarbons (Ferrous hydroxide test), if +ve then go to NaOH-Acetone colour test
- b. Substituted phenols (Ferric chloride test)

### ***DIVISION (B)***

- a. Amines (Copper sulphate test), if +ve then go to (Hinsberg test)

### ***DIVISION (M)***

### **1- Nitrogen present:**

- a. Amides (Ferric hydroxamate test and/ or Liberation of ammonia test)
- b. Nitro aryl amines (Copper sulphate test), if +ve then go to (Hinsberg test)
- c. Nitro hydrocarbons (Ferrous hydroxide test), if +ve then go to NaOH-Acetone colour test
- d. Diaryl amines (Hinsberg test)
- e. Amino phenols (Ferric chloride test)
- f. Triaryl amines (Hinsberg test)

### **2- Nitrogen and halogen present:**

- a. Halogenated amines (Copper sulphate test), if +ve then go to (Hinsberg test)
- b. Halogenated amides (Ferric hydroxamate test and/ or Liberation of ammonia test)
- c. Halogenated nitro compounds (Ferrous hydroxide test), if +ve then go to NaOH-Acetone colour test

## ***DIVISION (N)***

- a. Alcohols (Ceric Nitrate and/ or Xanthate test), if +ve then go to Lucas test
- b. Aldehydes and ketones (2,4-DNPH test), if +ve then go to Tollen's test
- c. Esters (Ferric hydroxamate test)
- d. Anhydride (Ferric hydroxamate test)
- e. Ethers (comparison test)

## ***DIVISION (N<sub>1</sub>)***

- a. Unsaturated hydrocarbons (Baeyer test  $\text{KMnO}_4$ )
- b. ketones (2,4-DNPH test)
- c. Ethers (comparison test)

## ***DIVISION (I)***

### **Only C, H, and O present**

- a. Hydrocarbons ( $\text{CHCl}_3$ - $\text{AlCl}_3$  test)
- b. Diaryl ethers (Iodine test)

### **Halogen present**

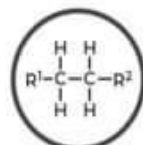
- a. Halogenated derivatives of hydrocarbons (Alcoholic silver nitrate test)

# **Chemical Tests for Functional Groups. Test tube Reactions**

# FUNCTIONAL GROUPS IN ORGANIC CHEMISTRY

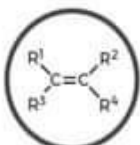
Functional groups are the characteristic groups in organic molecules that give them their reactivity. In the formulae below, R represents the rest of the molecule and X represents any halogen atom.

● Hydrocarbons 
 ● Halogen-containing groups 
 ● Oxygen-containing groups 
 ● Nitrogen-containing groups 
 ● Sulfur-containing groups 
 ● Phosphorus-containing groups



**ALKANE**

Naming: -ane  
e.g. ethane



**ALKENE**

Naming: -ene  
e.g. ethene



**ALKYNE**

Naming: -yne  
e.g. ethyne



**ARENE**

Naming: -yl benzene  
e.g. ethyl benzene



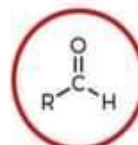
**HALOALKANE**

Naming: halo-  
e.g. chloroethane



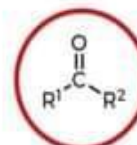
**ALCOHOL**

Naming: -ol  
e.g. ethanol



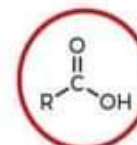
**ALDEHYDE**

Naming: -al  
e.g. ethanal



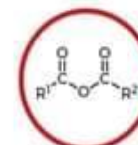
**KETONE**

Naming: -one  
e.g. propanone



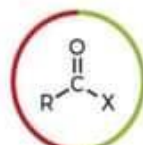
**CARBOXYLIC ACID**

Naming: -oic acid  
e.g. ethanoic acid



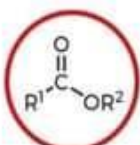
**ACID ANHYDRIDE**

Naming: -oic anhydride  
e.g. ethanoic anhydride



**ACYL HALIDE**

Naming: -oyl halide  
e.g. ethanoyl chloride



**ESTER**

Naming: -yl -oate  
e.g. ethyl ethanoate



**ETHER**

Naming: -oxy -ane  
e.g. methoxyethane



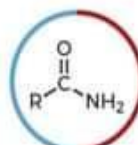
**EPOXIDE**

Naming: -ene oxide  
e.g. ethene oxide



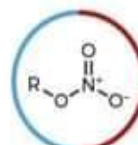
**AMINE**

Naming: -amine  
e.g. ethanamine



**AMIDE**

Naming: -amide  
e.g. ethanamide



**NITRATE**

Naming: -yl nitrate  
e.g. ethyl nitrate



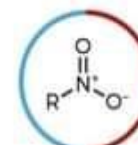
**NITRITE**

Naming: -yl nitrite  
e.g. ethyl nitrite



**NITRILE**

Naming: -nitrile  
e.g. ethanenitrile



**NITRO**

Naming: nitro-  
e.g. nitromethane



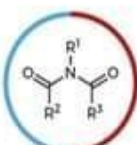
**NITROSO**

Naming: nitroso-  
e.g. nitrosoethane



**IMINE**

Naming: -imine  
e.g. ethanimine



**IMIDE**

Naming: -imide  
e.g. succinimide



**AZIDE**

Naming: -yl azide  
e.g. phenylazide



**CYANATE**

Naming: -yl cyanate  
e.g. methyl cyanate



**ISOCYANATE**

Naming: -yl isocyanate  
e.g. methyl isocyanate



**AZO COMPOUND**

Naming: azo-  
e.g. azoethane



**THIOL**

Naming: -thiol  
e.g. methanethiol



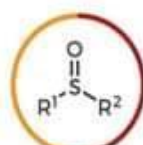
**SULFIDE**

Naming: sulfide  
e.g. dimethyl sulfide



**DISULFIDE**

Naming: disulfide  
e.g. dimethyl disulfide



**SULFOXIDE**

Naming: sulfoxide  
e.g. dimethyl sulfoxide



**SULFONE**

Naming: sulfone  
e.g. dimethyl sulfone



**SULFINIC ACID**

Naming: -sulfinic acid  
e.g. benzenesulfinic acid



**SULFONIC ACID**

Naming: -sulfonic acid  
e.g. benzenesulfonic acid



**SULFONATE ESTER**

Naming: -yl sulfonate  
e.g. methylmethanesulfonate



**THIOCYANATE**

Naming: thiocyanate  
e.g. ethyl thiocyanate



**ISOTHIOCYANATE**

Naming: isothiocyanate  
e.g. ethylisothiocyanate



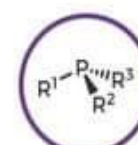
**THIAL**

Naming: -thial  
e.g. ethanethial



**THIOKETONE**

Naming: -thione  
e.g. propanethione



**PHOSPHINE**

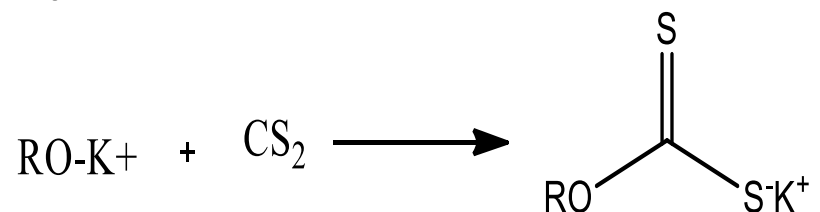
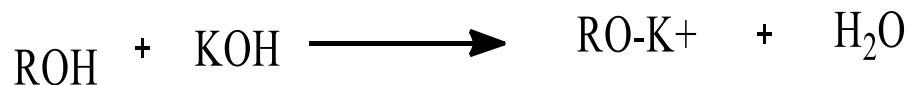
Naming: phosphane  
e.g. methylphosphane

**As you know there are many functional groups of compounds  
During the course, we will be familiar with some of them. And those  
may be:**

H.C. aliphatic	e.g. n-Hexane, cyclohexane
H.C. olefinic	e.g. cyclohexene
H.C. aromatic	e.g. toluene, naphthalene
Carboxylic acids	e.g. acetic, benzoic
Acid anhydride	e.g. acetic, phthalic
Amides	e.g. Benz amide
Esters	e.g. ethyl acetate
Amino acids	e.g. glycine
Alcohols	e.g. methanol
Ketones	e.g acetone
Aldehydes	e.g benzaldehyde
Phenols	e.g phenol
Nitro aromatics	e.g nitrobenzene
Amines	e.g aniline, pyridine
Alkyl and aryl halides	e.g benzyl chloride, chlorobenzene

# Test for alcohols

## 1. Xanthate



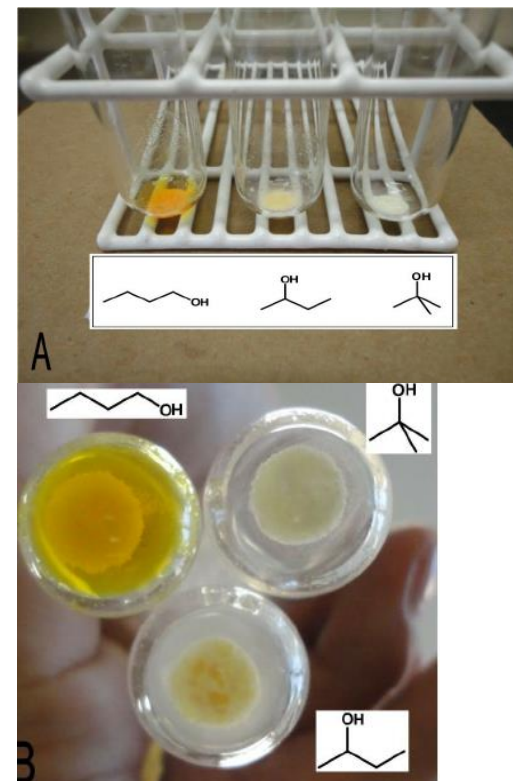
note: use dry test tube

pale yellow ppt.

1. add 1 pellet of KOH
2. add 2 drops of UN
3. heat on direct flame to dissolve KOH
4. cool and add Et<sub>2</sub>O
5. add drop by drop 5 drops of **CS<sub>2</sub> in the hood**

→ pale yellow ppt. +ve test


\* note: for water insoluble alcohols use dioxane then carry on with the same procedure




## 2. Ceric ammonium nitrate $(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6$ test for alcohol



(a) for water soluble alcohol

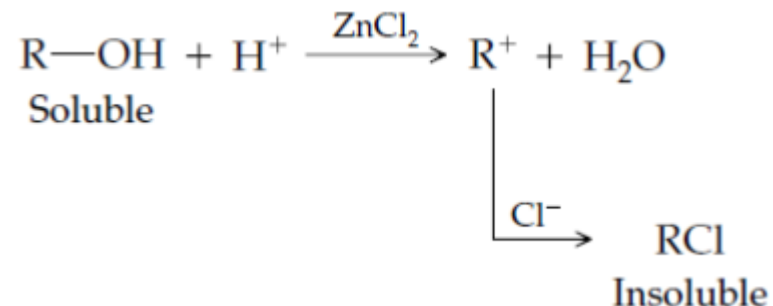
- 
1. add 5 drops of UN ( 0.1-0.2 g)
  2. add 2 ml of ceric ammonium nitrate solu.
  3. mix thoroughly
- note the formation of red color

(b) For water insoluble alcohol

- 
1. add 2 ml dioxane
  2. add 2 ml of ceric ammonium nitrate solu.
  3. divide the solu. Into tow halves
  4. add 5 drops of UN ,
- note and compare the color with the blank test tube
- red appear Ce ( IV) then disappear to colorless Ce(III) complex



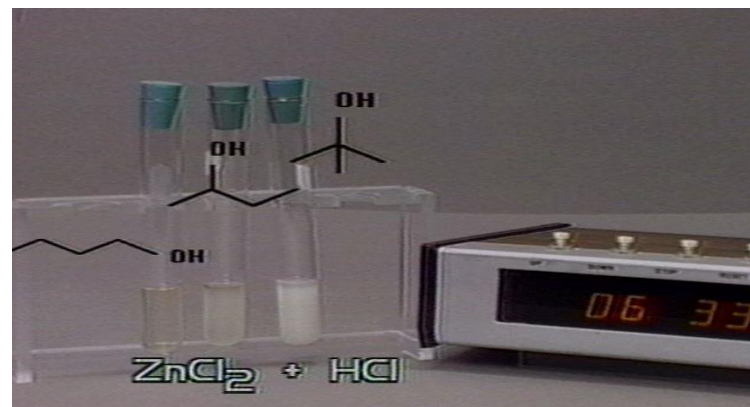
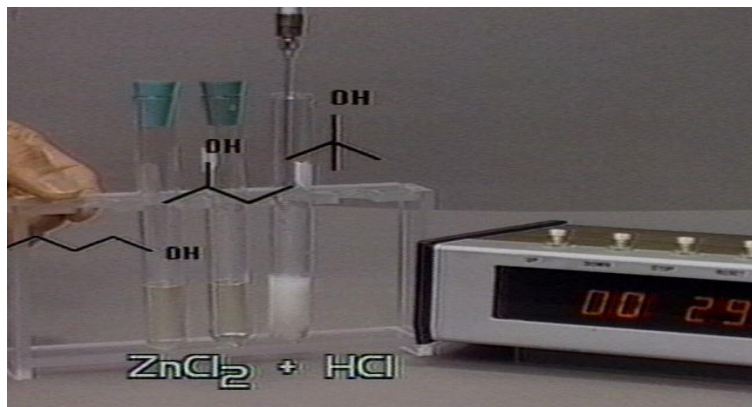
## Lucas test ( $\text{ZnCl}_2/\text{HCl}$ )



note: dry test tube

1. add 2 drops (0.2 g) of UN
  2. add 2 ml of Lucas reagent
  3. stopper the tube and shake well, allow to stand and record the formation of a turbid solu.
- 3  $\longrightarrow$  1-2 min.
- 2  $\longrightarrow$  5-6 min.
- 1  $\longrightarrow$  negligibly slowly or not at all

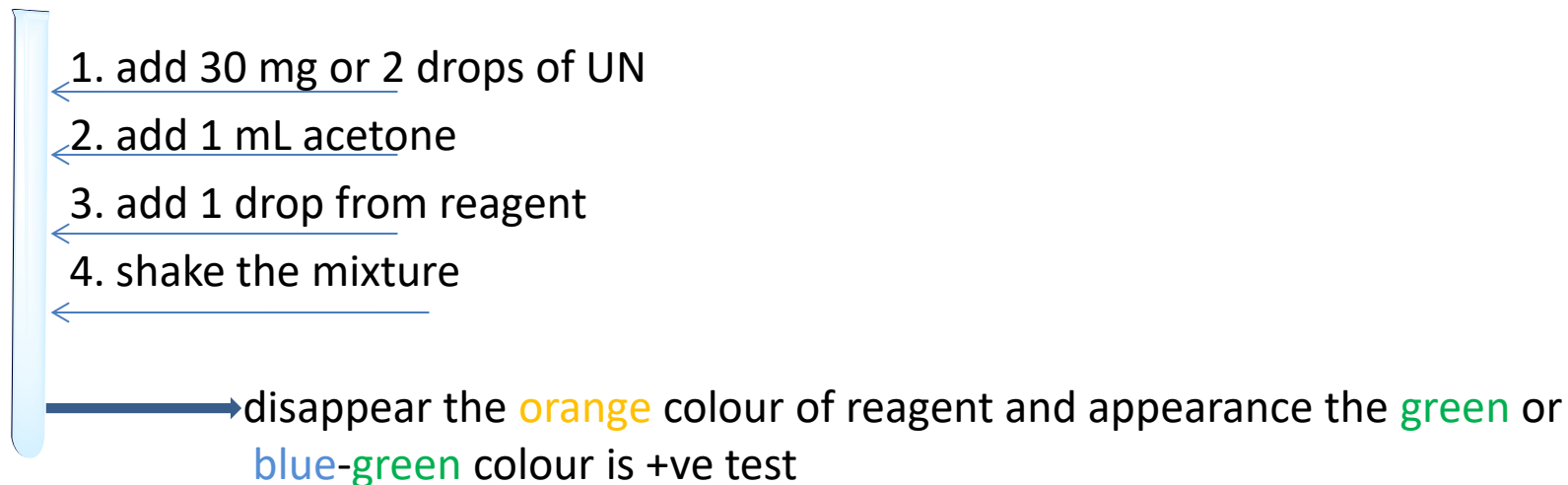
Jones reagent does react with 3





# Chromic acid (Jones) test

note: use dry test tube



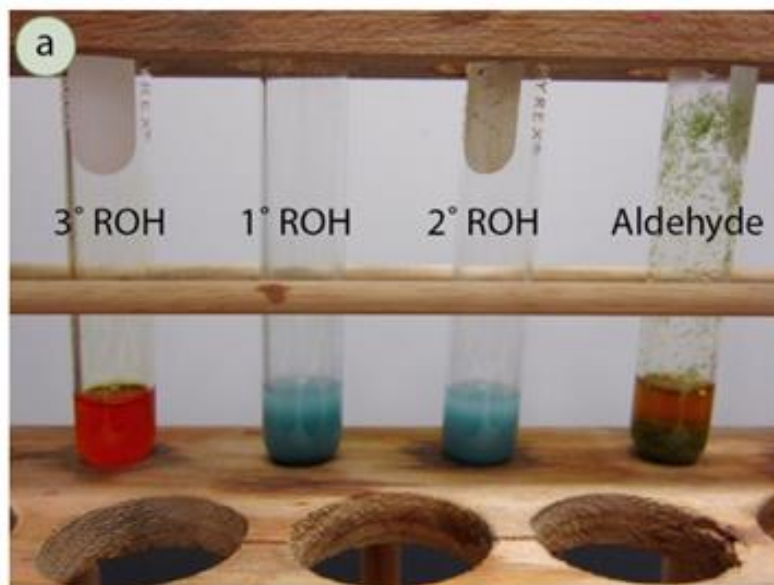
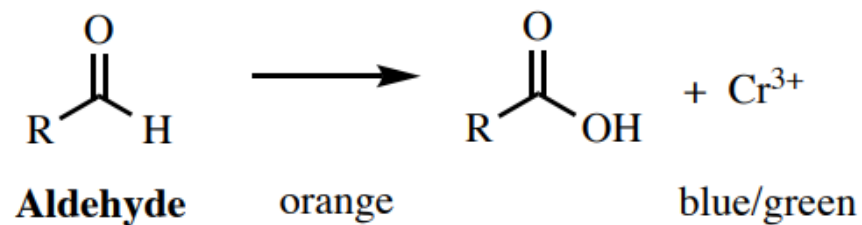
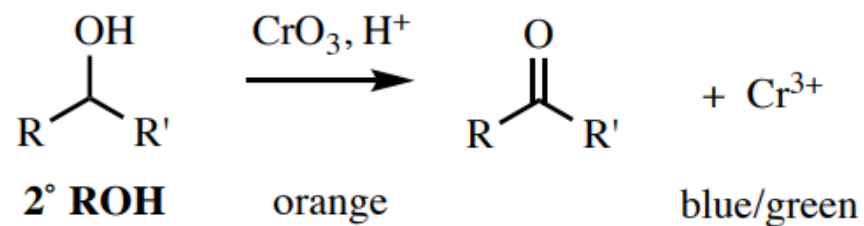
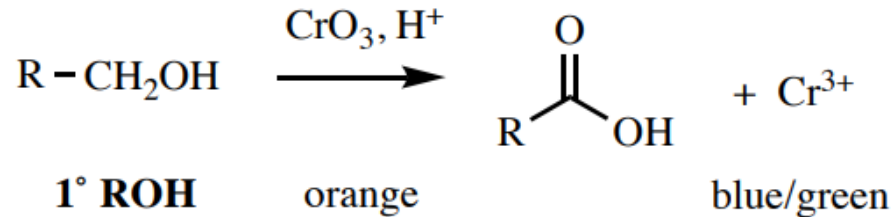
A diagram of a test tube with four numbered steps listed inside it, each preceded by a blue arrow pointing left. A blue arrow points from the bottom of the test tube to the text 'disappear the orange colour of reagent and appearance the green or blue-green colour is +ve test'.

1. add 30 mg or 2 drops of UN
2. add 1 mL acetone
3. add 1 drop from reagent
4. shake the mixture

disappear the orange colour of reagent and appearance the green or blue-green colour is +ve test

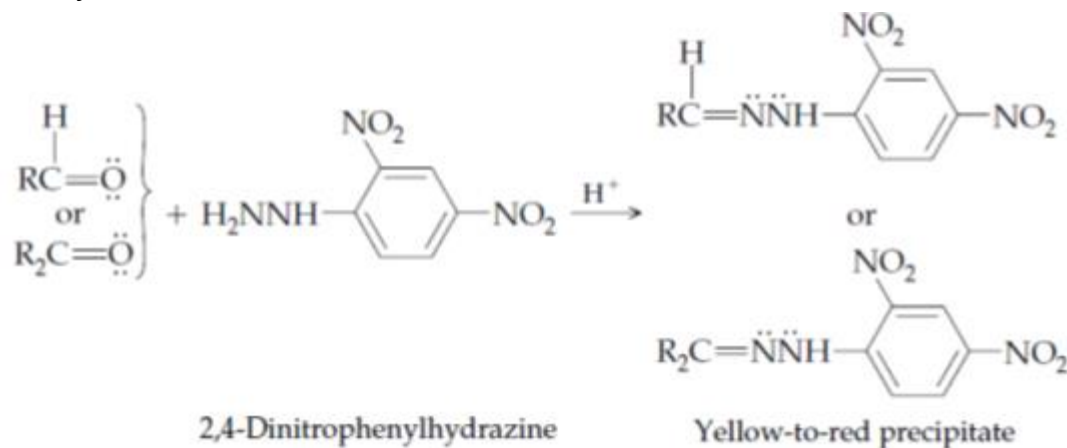
## Note\*

- The reagent prepared from dissolving 1 gm of chromium oxide ( $\text{Cr}_2\text{O}_3$ ) in 1 mL conc.  $\text{H}_2\text{SO}_4$  and diluted with 3 mL water.
- Primary and secondary alcohols react to give green colour or ppt during 10 min.
- Aldehydes give +ve test within few min.
- Some phenols, enols, aromatic amines give colour dark ppt.



## Detection of carbonyl group: Brady's reagent (2,4-Dinitrophenylhydrazine (2,4-DNPH))

General test for aldehydes and ketones



note: use dry test tube

1. add 2 drops (50 mg) of UN

2. add 3 ml of reagent, 2, 4-DNPH, shake

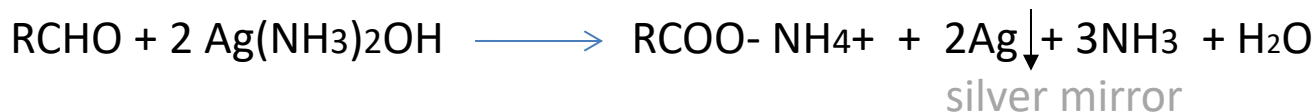
if no ppt. form allow to stand for 15 min.

yellow, orange, or red ppt. +ve test



note: cinnamyl alcohol or some primary or secondary alcohols give +ve test.

## Tollen's Test to differentiate aldehydes from ketones



Reagent is freshly prepared

1. add 1 ml of 5%  $\text{AgNO}_3$
  2. add 5%  $\text{NaOH}$ , shake ( ppt.  $\text{AgOH}$ )
  3. add drop by drop  $\text{NH}_4\text{OH}$  to dissolve  $\text{AgOH}$
- avoid excess  $\text{NH}_3$  solu.

1. add 1ml of freshly prepared reagent
  2. add 1 drop (few crystals) of UN
  3. leave the tube for 10 min. ,if no reaction
- happen warm on steam bath for 5 min.

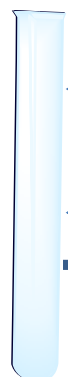
→ Silver mirror

-acetone, formaldehyde, enols, diphenylamine  
some aromatic amines as well as 1-naphthols  
and some phenols



# Test for unsaturated hydrocarbons (Alkene & Alkyne)

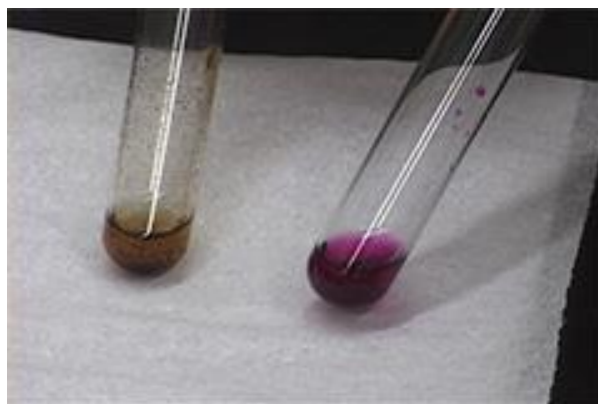
## 1. $\text{KMnO}_4$ Solution (Permanganate test), Bayer test

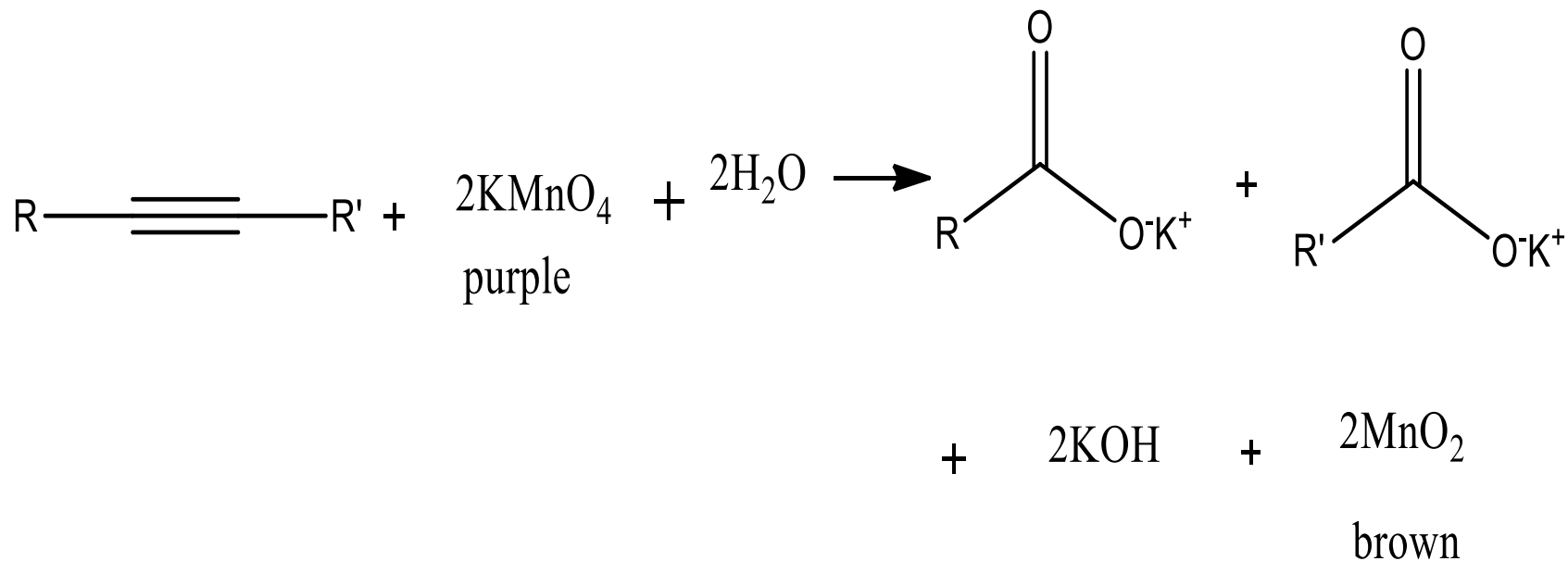
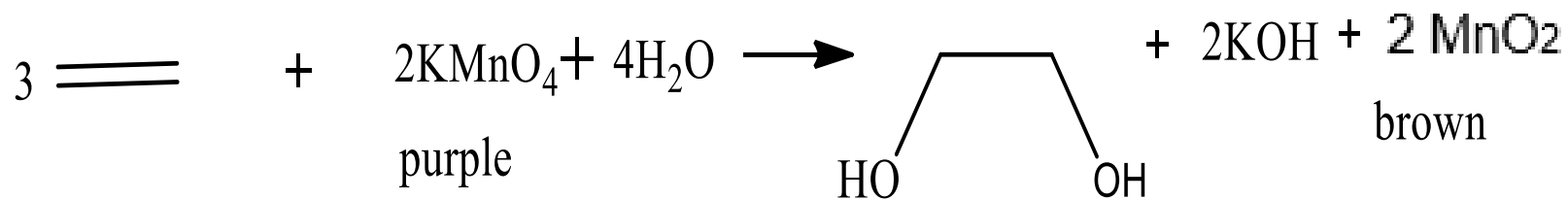


1. 0.1 g or 2 drops of UN

2. 1-2 drops of 1%  $\text{KMnO}_4$

the disappearance of purple color or formation of brown ppt +ve test






If the KMnO<sub>4</sub> color is not changed in 0.5-1 min, allow the tube to stand for 5min with Occasional vigorous shaking.

## 2. Iodine test for ethers and alkenes

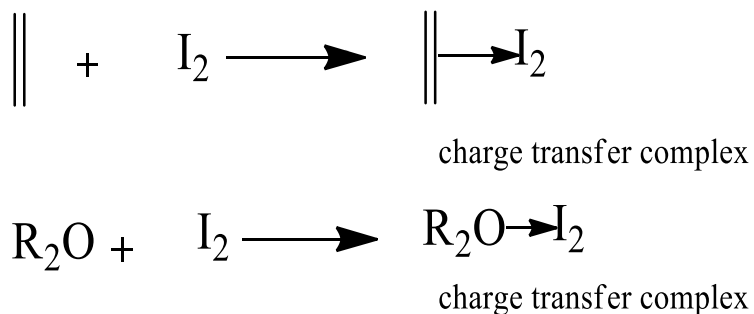
note: use dry test tube



1. add 2 drops or 0.25 g of UN  
2. add 5 drops of I<sub>2</sub>/CH<sub>2</sub>Cl<sub>2</sub>


ether (purple → tan)  
alkene (purple → tan solid → purple)

Aromatic H.C., sat. H.C. or chlorinated H.C. does not react.



## Test for ethers

note: use dry test tube

- 
1. add 3 drops of UN
  2. add 5 drops of conc.  $\text{H}_2\text{SO}_4$
  3. add 5 drops of  $\text{I}_2$

————→ formation of brown colour refer to presence ether or oxygens solvents



## Ethanol silver nitrate test

To differentiate between alkyl halide and aryl halide



1. add 2 ml of  $\text{AgNO}_3$ / EtOH solu.
2. add one drop or crystals of UN
3. let stand for 5 min.
4. if no ppt. formed heat to boiling

\* **Note:** tertiary halides react directly at r.t.

Secondary and primary halides react slowly or do not react at r.t. but they react after heating

$\text{ArX}$  does not give a ppt.



## Test for amide (Alkaline hydrolysis)

1. add 50 mg of UN

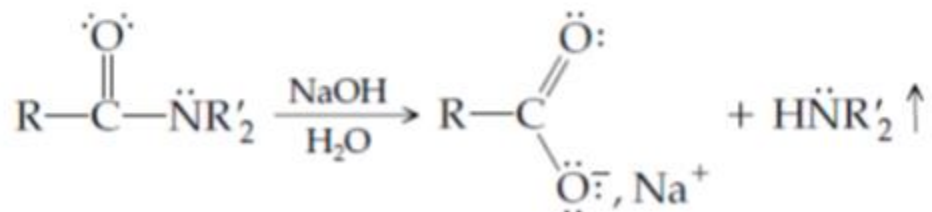
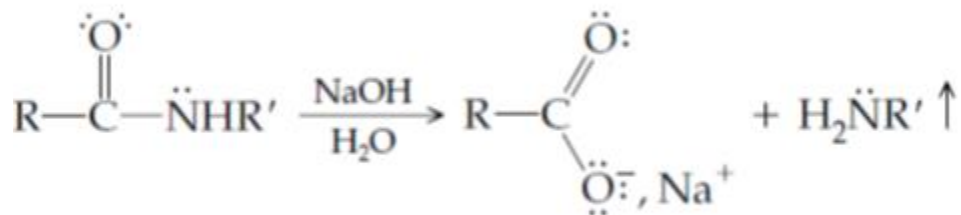
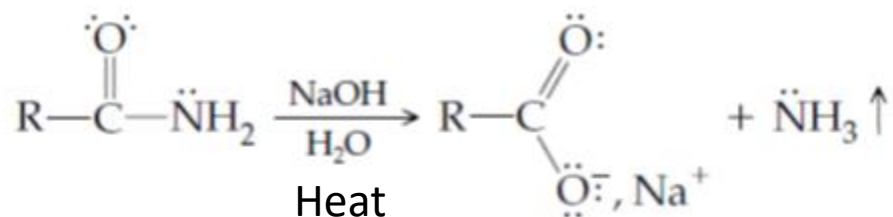
2. add 3-4 pellets of solid NaOH

3. add 1 mL of H<sub>2</sub>O

4. place moist red litmus paper on the top of the tube

5. boil for 1 min.

note the change of litmus **red** to **blue** +ve test (indicate the presence of amide)



# Test for amines

## Copper Ion test (General test)

1. add 20 mg (2 drops) of UN

For water insoluble amines add 5 drops of EtOH

2. add 0.5 ml of 10% Copper sulphate solution

Blue-green coloration is a +ve test



## Nitrous acid test to distinguish between amines



1. Add 2 drops of UN

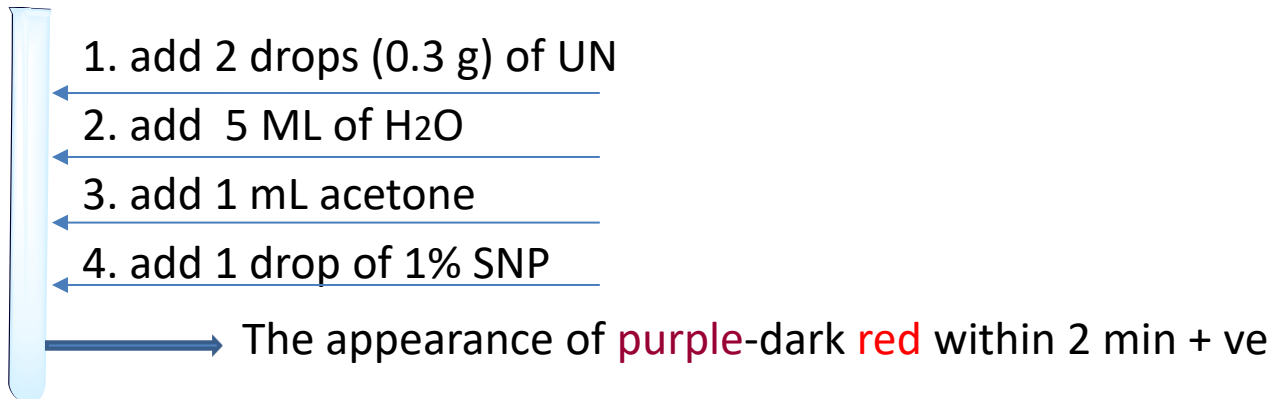
2. Add 10% HCl to dissolve the UN

3. Add 5 drops of 20% NaNO<sub>2</sub> drop by drop till effervescence

Formation clear **yellow** solution of diazonium salt refer to presence of 1 amine  
2 amine give oily **yellow** compounds of N-nitroso amine  
3 amine give **red** solid compounds of *p*-nitroso in the presence of NaOH  
convert to **green** crystals.

**Note:** NaNO<sub>2</sub> added and the test tube in an ice bath

## Rimini test (special test for primary aliphatic amines)



1. add 2 drops (0.3 g) of UN


2. add 5 ML of H<sub>2</sub>O

3. add 1 mL acetone

4. add 1 drop of 1% SNP

→ The appearance of purple-dark red within 2 min + ve

## Simon test (special test for secondary alkyl amines)

- 
1. add 2 drops (0.3 g) of UN
  2. add 5 mL of H<sub>2</sub>O
  3. add 1 mL of 5% AcH
  4. add 2 drop of 10% SNP
  5. add 2 drops of 10% NaHCO<sub>3</sub>

→ The appearance of blue colour within 3 min  
converting to green then to yellow with time + ve

# Hinsberg's test to distinguish between amines

1. add 3 drops (0.3 g) of UN
2. add 5 ML of 10%NaOH
3. add 40 mg PTSC (make sure that the solu. is alkaline)
4. stopper the tube and shake vigorously until all PTSC has reacted

note if ppt. formed

- |            |   |         |         |
|------------|---|---------|---------|
| 1. no ppt. | $\xrightarrow{10\% \text{ HCl}}$          | ppt.    | 1 amine |
| 2. ppt.    | $\xrightarrow{\hspace{1cm}}$              | ppt.    | 2 amine |
| 3. no ppt. | $\xrightarrow{\hspace{1cm}=\hspace{1cm}}$ | no ppt. | 3 amine |

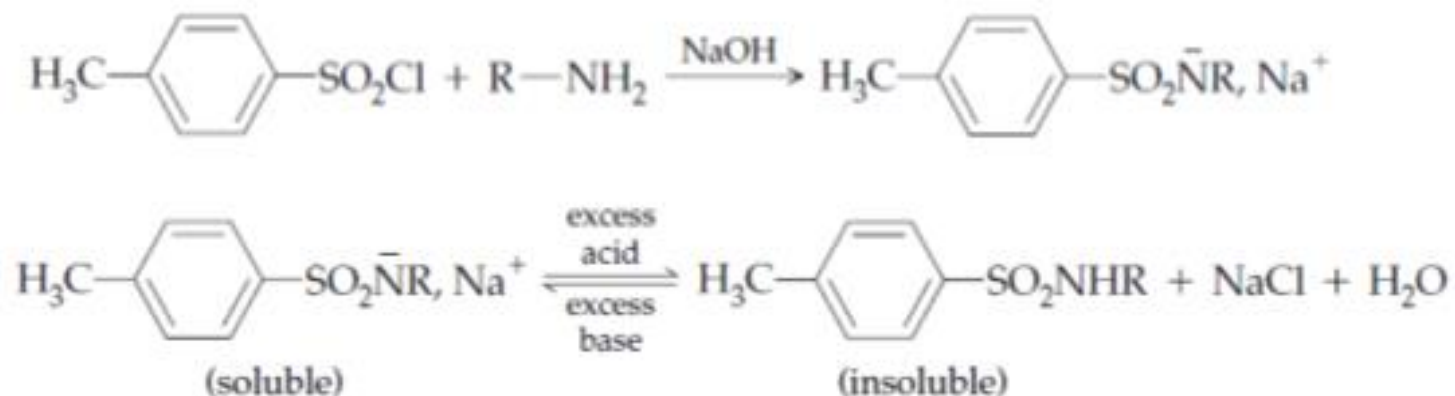
## Alternative procedure (in the hood)

1. add 2drops or 30 mg of UN
2. add 2 ml of pyridine
3. add 8 drops of 2% NaOH
4. add 30mg of PTSC and shake well

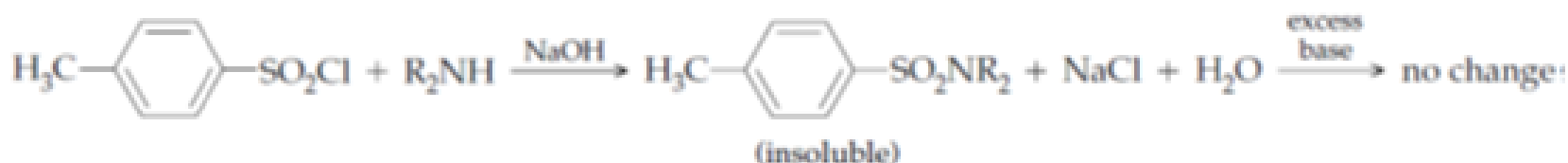
note the color: yellow 1, orange 2, deep red, or purple 3

note: some compounds other than amines gave colored solu.

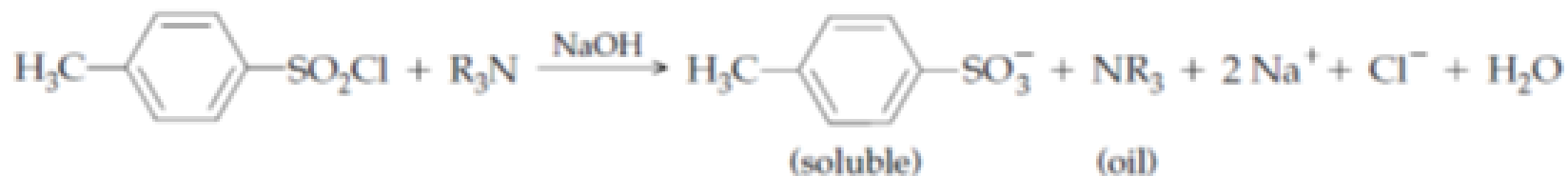
Primary amines with fewer than seven carbon atoms form a sulfonamide that is soluble in the alkaline solution. Acidification of the solution results in the precipitation of the insoluble sulfonamide:



Secondary amines form an insoluble sulfonamide in the alkaline solution:



*Tertiary* amines normally give no reaction under these conditions:





# Test for carboxylic acids

## 1. Iodate-Iodide test



1. add 3 drops or crystals of UN

2. add 2 drops of 5% KI

3. add 2 drops of 5% KIO<sub>3</sub>

4. Stopper the tube and hold it in a water bath for 1 min

5. Cool the tube and add 5 drops of starch

Blue, violet formation +ve test for carboxylic acid

Brown / Yellow



**Negative Test** (starch absent)

Blue / Purple



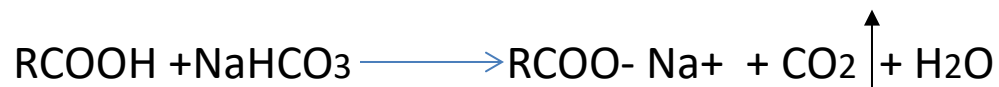
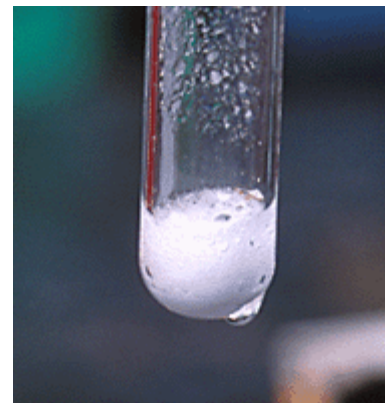
**Positive Test** (starch present)

# Test for carboxylic acids


## 2. Sodium bicarbonate



1. add 1 ml MeOH
  2. add 3 drops or crystals of UN
  3. add 1 ml of saturated NaHCO<sub>3</sub>
- evolution of CO<sub>2</sub> gas indicate +ve test



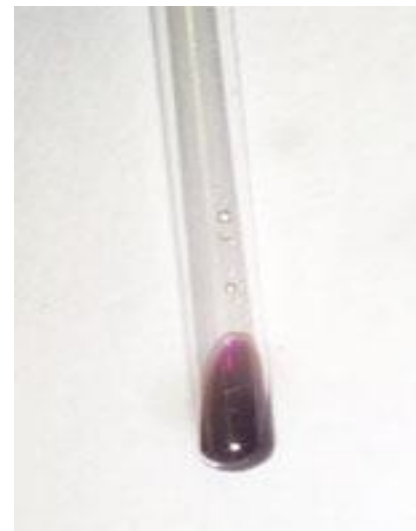
## Test for Ester (Ferric Hydroxamate)

- 
1. add 0.5 ml of 1N  $\text{NH}_2\text{OH} \cdot \text{HCl}$  / MeOH
  2. add 30 mg or 3 drops of UN
  3. add drop by drop 2N  $\text{KOH}$  / MeOH to render the solu. basic to litmus
  4. add additional 4 drops of  $\text{KOH}$  / MeOH
  5. heat to boil
  6. cool and then add with shaking drop by drop 2N  $\text{HCl}$  to PH 3
  7. add 1 drop of 10%  $\text{FeCl}_3$

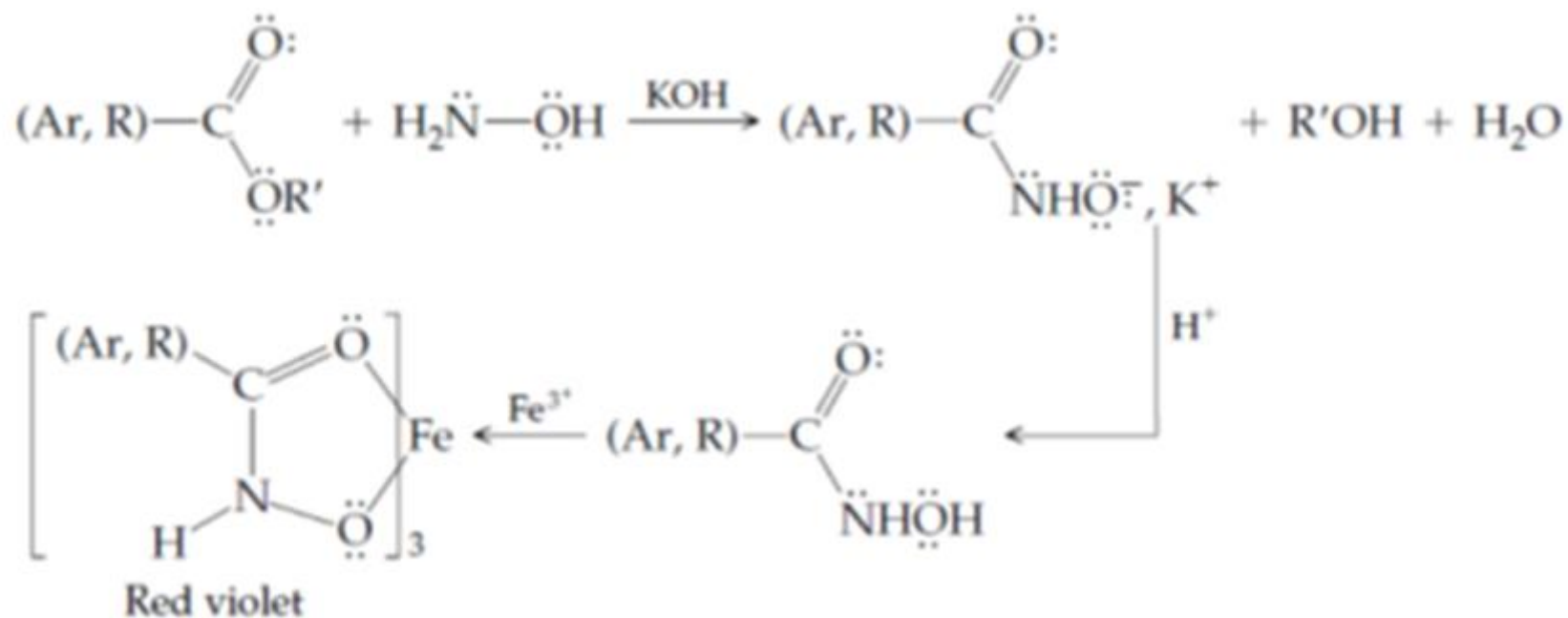
—————→ Reddish-blue or bluish- red



reddish-blue



Reaction:



## Ferrous hydroxide test for nitro aromatics



blue-green



red-brown ppt

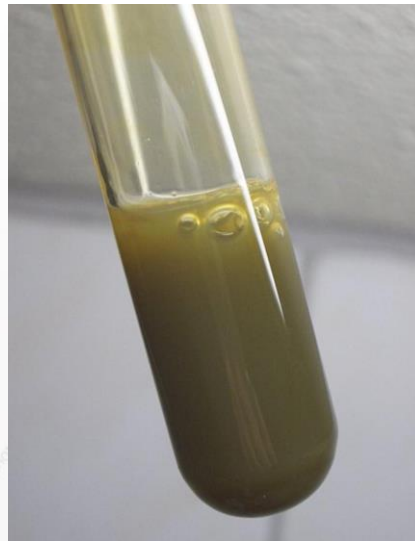
1. add 20 mg (2 drops) of UN

2. add 1.5 ml of 5% aqueous ferrous ammonium sulfate or (freshly prepared)

3. add 1 drop of 3M H<sub>2</sub>SO<sub>4</sub>


4. add 1ml of 2M KOH in MeOH

, stopper the tube and shake vigorously, vent, then allow it to stand over a 5-min period, the formation of red-brown, light brown precipitate is +ve test for a nitro group.



# Test for determining the number of nitro groups on the aromatic structure


## Sodium hydroxide colour test



1. add 20 mg of UN
2. Add 1mL of NaOH/EtOH

→ note the colour of the solution (no colour or light yellow refer to mono nitro, blue refers to dinitro, red refers to trinitro)

## Alternative procedure



1. add 20 mg of UN
2. Add 1 mL of acetone
3. Add 1 pellet of NaOH
4. Add 1mL of EtOH with shaking

→ note the colour of the solution (no colour or light or dark yellow refer to mono nitro, blue refers to dinitro, red refers to trinitro)

## Ferric chloride $\text{FeCl}_3$ (test for phenols)

1. add 4-5 drops or 30-50 mg of UN

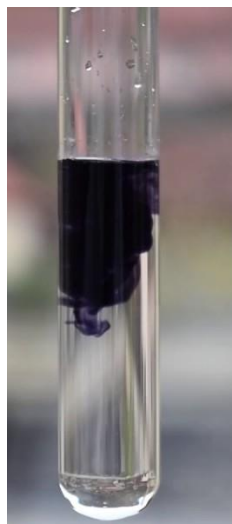
2. add 2 drops of water or (ethanol for water insoluble phenols)

3. add 3 drops of 2.5%  $\text{FeCl}_3$  (light yellow in colour)

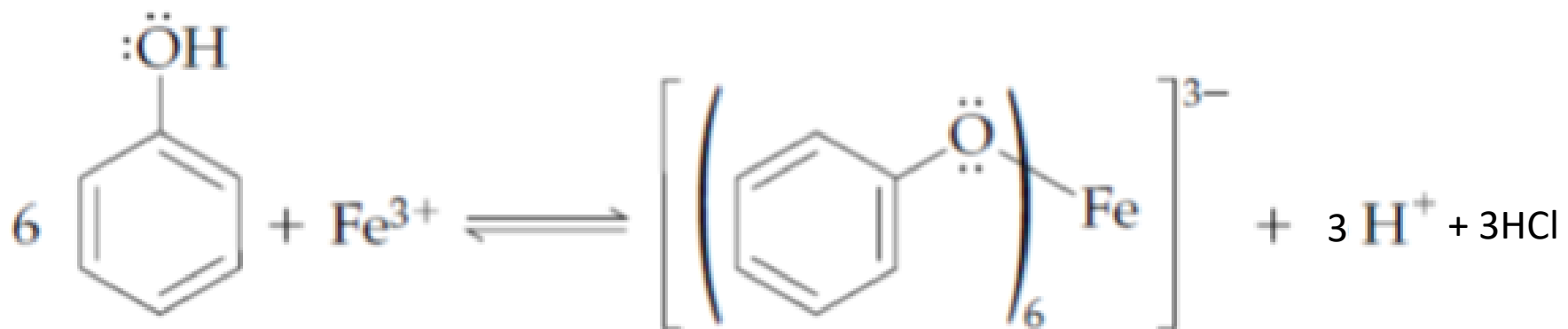
→ note the colour of the solution (blue, violet, purple, green, red-brown)

- enols, oximes give +ve test

- hydroquinone, nitrophenols, and m- and p- hydroxybenzaldehydes give –ve test



Most phenols and enols form colored complexes in the presence of ferric ion,  $\text{Fe}^{3+}$





## Test for aromatic structure

### Chloroform- $\text{AlCl}_3$ test

note: use dry test tubes



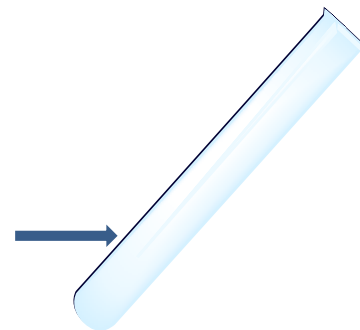
1

- 1. add 8 drops of dry  $\text{CHCl}_3$
- 2. 10-20 mg (3 drops) of UN



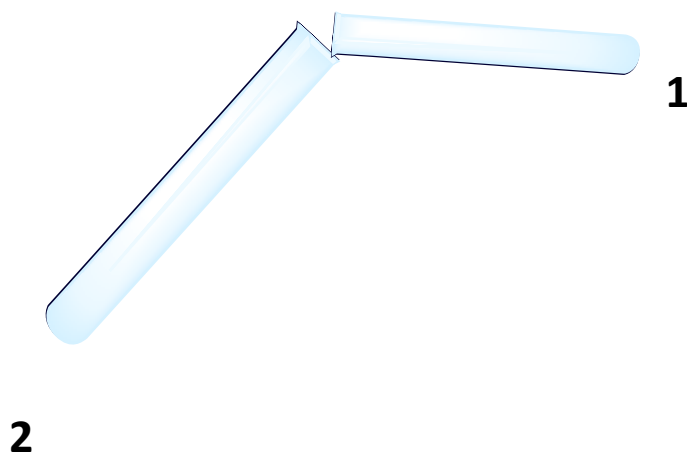
2

- 1. add 100 mg of  $\text{AlCl}_3$



2

heat the on burner to sublime  
the  $\text{AlCl}_3$  on the  
on the wall of the tube



- pour the solu. of UN in  $\text{CHCl}_3$  on the wall of test tube **2** so that it touch the sublimed  $\text{AlCl}_3$ .

- benzene, toluene, xylenes, mesitylene gave red to orange color
- aryl halides orange to red also
- naphthalene blue
- biphenyl purple
- phenanthrene purple
- anthracene green




**Benzene and aryl halides**



**Naphthalene**

## Ninhydrin (test for amino acid)

- 
1. add 1-5 mg (1-5 drops) of UN
  2. add 1-3 ml of H<sub>2</sub>O
  3. add 1 ml of 0.1% ninhydrin and boil for 1-2 min.

—————→ blue- blue-violet +ve test

orange or red -ve test

