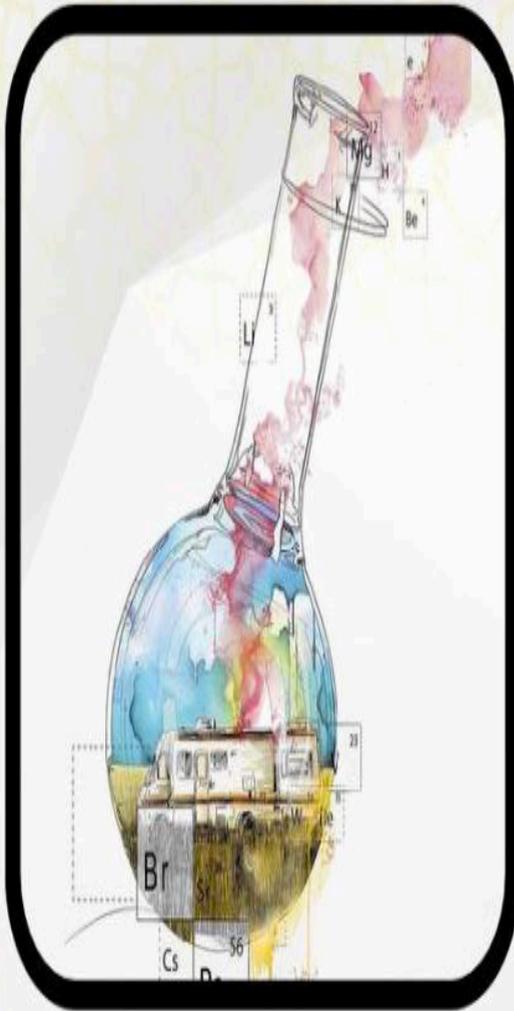


# تفريغ مختبر عضوية



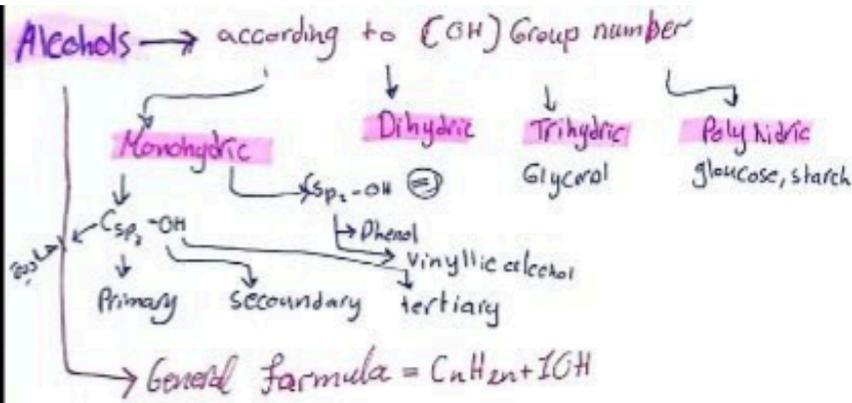
Exp 3 : اسر الموضوع :  
Alcohols & Phenols

Sara Taber إعداد الصيدلاني /ة:



لجان الرفعات

# المعلومات الإلهافية من الفيديو



⇒ Lucas Test ( $ZnCl_2/HCl$ )

## \* Reaction of Alcohols

$R-OH$	$C-OH$
- with Base	- dehydration & substitution Rxn
esterification	
	$OH^-$ → Protonation
	عشنة انقراط الحماض المركب
	لو قتل متحدة صعب الحماض المركب

## ⇒ Acid properties of Alcohol

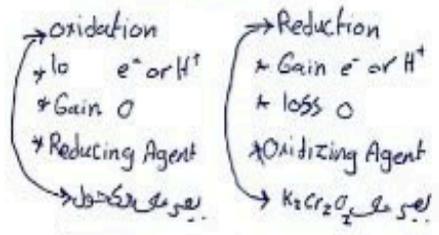
Alcohol → can show acidic & basic properties at O-H group

- ↳ Like water -
- ↳ slightly weaker acids than water
- $pK_a = 16-19$
- But, still able to react with strong bases ⇒ Sodium hydroxide

## ⇒ Chromic acid oxidation of alcohol

\* Rate of Reaction depend on :-

- 1- strength of reagent
- 2- pH
- 3- Temp
- 4- Concentration



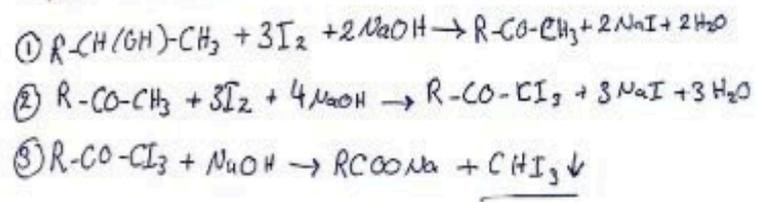
## ⇒ Solubility in water

$ROH$  ⇒  $R \leq 3$  → Miscible

$R = 4$  → slightly soluble

$R \geq 5$  → insoluble

## ⇒ Iodoform test ( $KI$ / water → $I_2$ )



# EXPERIMENT 3: ALCOHOLS AND PHENOLS

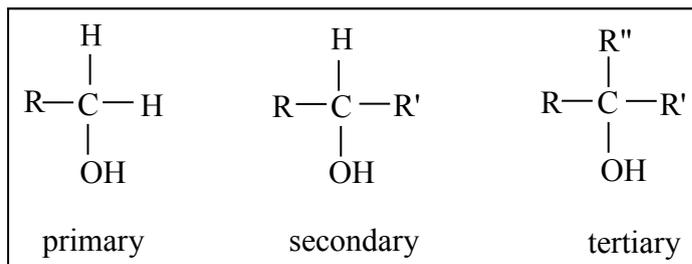
## Classification and Tests

Physical & Chemical Properties

① Solubility  
② Boiling Point  
③ Acidity

### I. ALCOHOLS

Alcohols are classified as primary, secondary and tertiary according to the number of alkyl groups directly attached to the carbinol carbon.



Reactions of alcohols involve the breaking of either of two bonds: the O-H bond as in reactions with bases and esterification reactions, or the C-OH bond leading to dehydration and substitution reactions. In breaking the C-OH bond, protonation of the -OH group is essential to convert it from a poor leaving group to a better one.

Some physical and chemical properties of alcohols are examined in the following tests.

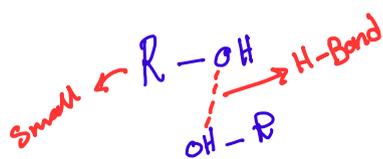
### EXPERIMENTAL

<b>MATERIALS NEEDED</b>	<u>Glassware:</u> 4 Test tubes. <u>Chemicals:</u> 1 mL each of: ethanol, 1-butanol, 2-butanol, 2-methyl-2-propanol, ethylene glycol, sodium metal, phenolphthalein indicator, 15 mL potassium dichromate (1%), 0.5 mL sulfuric acid, 6.0 mL Lucas reagent, 15 mL iodoform reagent, 6 mL NaOH (10%),
-------------------------	--

فرد جسد

#### 1. Solubility in Water

Alcohols of low molecular weight are water soluble due to their ability to form hydrogen bonds with water. Solubility in water decreases with



M.W.  $\downarrow$   
Solubility  $\uparrow$

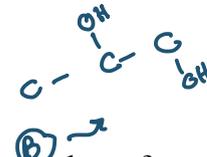
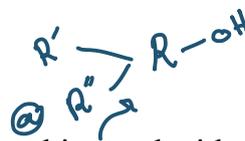
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Big M.W.  $\leftarrow$   $\text{R}-\text{OH}$   
 $\rightarrow$  Solubility  $\downarrow$



Identification Test

increasing molar mass but increases with branching and with the number of hydroxyl (OH) groups.



↑ Solubility

## PROCEDURE



**Glassware:** 4 test tubes

**The following alcohols to be tested:** 1- butanol, ethanol, ethylene glycol, or 2-methyl-2-propanol.

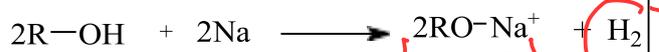
1. In each test tube, add **10** drops of one of the alcohols to be tested.
2. Add **2** mL of water to each test tube.
3. Shake very well.
4. Record your observations **and** result.

## 2. Acid Properties of Alcohols

Can show acidic or basic properties  
pKa = 16 - 19  
weaker acidic than water

Alcohols react with metallic sodium with the evolution of hydrogen. The relative acidities of alcohols and consequently their relative rates of reaction with sodium are in the order:

primary > secondary > tertiary.



Alkoxide  
RO-M<sup>+</sup>

## PROCEDURE



**Glassware:** 3 test tubes

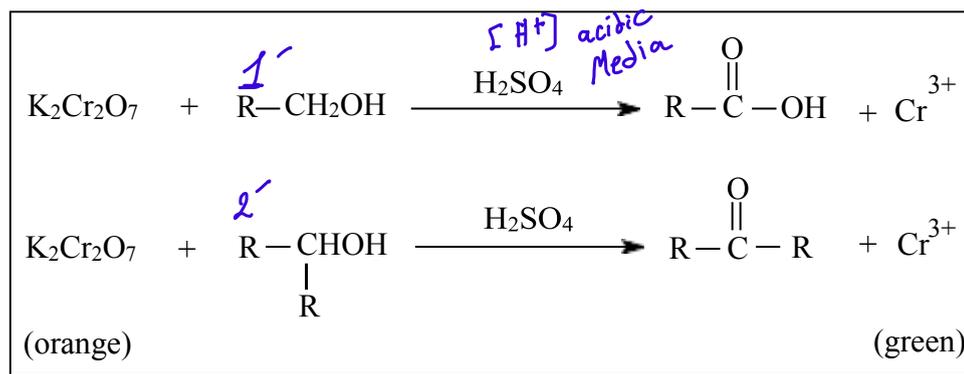
**The following alcohols to be tested:** 1- butanol, 2-butanol, or 2-methyl-2-propanol.

1. In each test tube, place a small piece of sodium.
2. Add **2** mL of one of the alcohols to be tested.
3. Add (up to **10** drops) of concentrated **sulfuric acid**.
4. Shake.
5. Compare the rates of evolution of hydrogen gas and record your results.
6. After all the sodium has reacted in the test tube containing the 1-butanol, add 3 drops of phenolphthalein indicator solution and observe the color change.
7. Record your observations **and** result.



### 3. Chromic Acid Oxidation of Alcohols

Primary and secondary alcohols are oxidized by chromic acid to the corresponding carboxylic acids and ketones respectively. Tertiary alcohols are generally unreactive under similar conditions. When alcohols are oxidized, they reduce chromium (VI) to Cr (III) changing the color of the solution from orange to green. Oxidation therefore offers a method for distinguishing primary and secondary alcohols from tertiary alcohols.



#### PROCEDURE

**Glassware:** 3 test tubes

**The following alcohols to be tested:** 1- butanol, 2-butanol, or 2-methyl-2-propanol.

8. In each test tube, place 5 mL of **Chromic Acid Reagent (1% potassium dichromate solution)**.
9. Add (up to 10 drops) of concentrated **sulfuric acid**.
10. Mix thoroughly and add 2 drops of one of the alcohol to be tested and shake.
11. Record your observations **and** result.

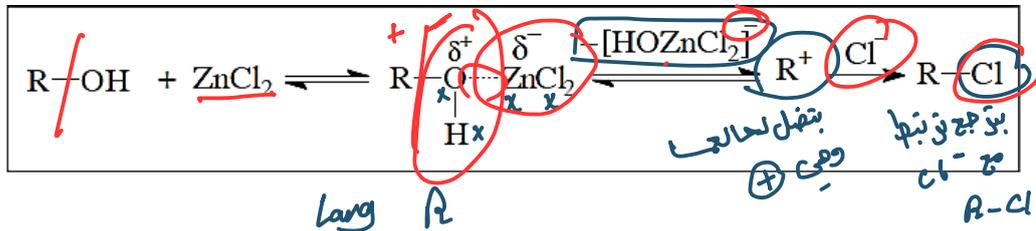
**Positive results → Green solution will be formed.**

#### 4. The Lucas Test

A solution of zinc chloride in concentrated hydrochloric acid (Lucas reagent) can be used to distinguish between primary, secondary and tertiary alcohols.



With this reagent the order of reactivity is typical of compounds reacting by the  $S_N1$  mechanism. The zinc chloride (a Lewis acid) assists in breaking the C-OH bond as illustrated in the equation below:



Alcohols (of no more than six carbons) are soluble in the Lucas reagent while the corresponding alkyl chlorides are not. Tertiary alcohols react rapidly with the reagent forming an insoluble alkyl chloride layer almost immediately. Secondary alcohols react within 5-10 minutes, while primary alcohols require several hours to react at room temperature ( $S_N2$  mechanism).

### PROCEDURE

**Glassware:** 3 test tubes

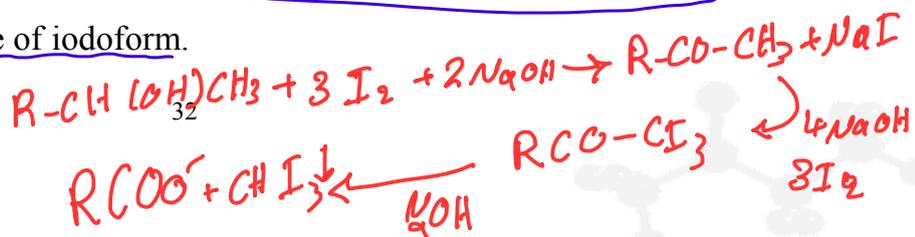
**The following alcohols to be tested:** 1- butanol, 2-butanol, or 2-methyl-2-propanol.

1. In each test tube, place 2 mL of Lucas' reagent
2. Add 6 drops of one of the alcohols to be tested.
3. Close the tubes with a piece of parafilm and shake well.
4. If no change occurs immediately, then place in water bath at (100°C) for 5-13 minutes.
5. Record your observations and result.

**Positive results** → White to cloudy mixture (immediately with 3° alcohols & within 5-10 min with 2° alcohols)

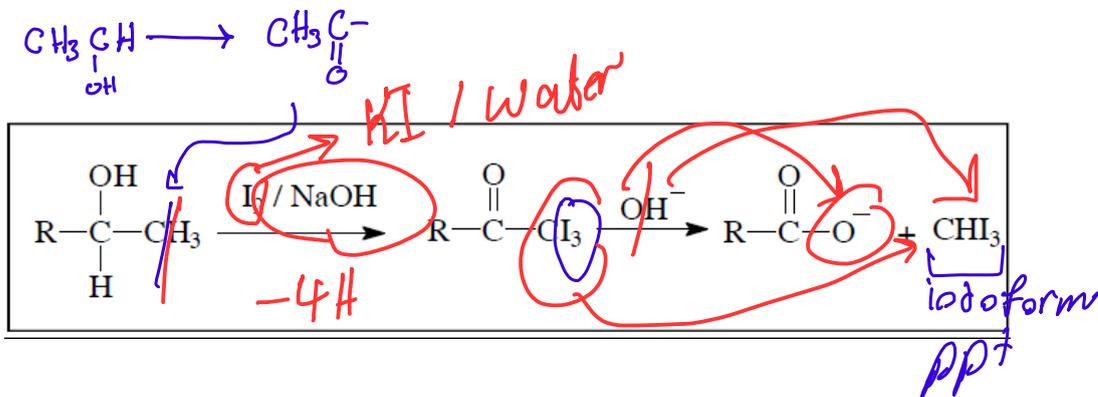
### 5. The Iodoform Test

is a test for methyl carbinols having the structure  $CH_3CHOH-$  and methyl ketones ( $CH_3CO-$ ). Methyl carbinols are first oxidized by the reagent to methyl ketones which become iodinated and then cleaved by base to give a bright yellow precipitate of iodoform.



2° alcohol  
 2° alcohol + methyl ketone  
 acetaldehyde  
 $CH_3C(=O)H$





## PROCEDURE

**Glassware:** 3 test tubes

**The following alcohols to be tested:** 1- butanol, 2-butanol, or 2-methyl-2-propanol.

- In each test tube, add 3 mL of 5% sodium hydroxide.
- Add 10 drops of one of the alcohols to be tested.
- Add 5-10 drops of iodine solution (or up to 0.5 mL) gradually.
- Shake very well.
- Allow to stand for 3-5 minutes.
- Record your observations **and** result.

**Positive results** → **Bright yellow precipitate**

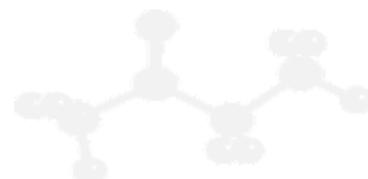
*Handwritten note:* 2° ROH

## II. PHENOLS

The most common reactions of phenols involve breaking the O-H bond and the usual electrophilic aromatic substitution at the aromatic ring.

$+H^+$  Protonation of the hydroxyl group and loss of a water molecule as in alcohols would give a phenyl cation which is very unstable and difficult to form. Since the aromatic nucleus is electron rich, direct attack by nucleophiles as in  $S_N1$  or  $S_N2$  reactions is not possible. Consequently, phenols do not undergo substitution of the hydroxyl group either by the  $S_N1$  or  $S_N2$  mechanisms.

The characteristic property that differentiates phenols from alcohols is acidity. Phenols are stronger acids than alcohols and react with sodium hydroxide, whereas alcohols do not. The reason for this difference is that the phenoxide ion is resonance-stabilized whereas the alkoxide ion is not.



## EXPERIMENTAL

<b>MATERIALS NEEDED</b>	<u>Glassware:</u> 4 Test tubes.
	<u>Chemicals:</u> cyclohexanol, phenol, <i>p</i> -cresol, 4 mL bromine water solution, 0.5 mL ferric chloride solution (1%), 6 mL of 10% NaOH solution .



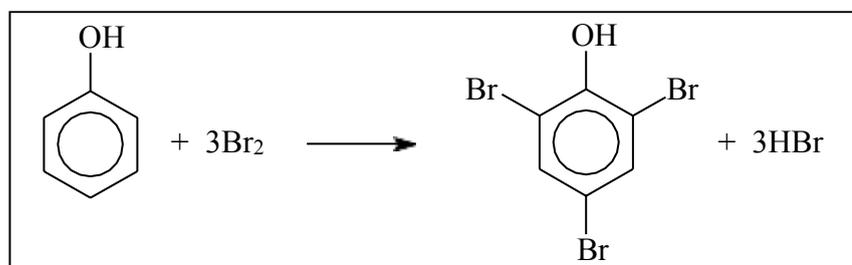
### 1. Acidity of Phenols

**Procedure** In each of three test tubes add 0.4 mL or 0.2 g of cyclohexanol, phenol, or *p*-cresol. Add 1 mL of water to each tube, shake and note whether the compound dissolves. If not add 2 mL of 10% NaOH solution and observe the result.

### 2. Bromination of Phenols with Bromine Water

The hydroxyl group strongly activates the aromatic ring towards electrophilic aromatic substitution. Phenol readily forms a tribromo derivative when treated with a solution of bromine-water at room temperature.

محبوب للبرومين  
مفعول موجب ← Br

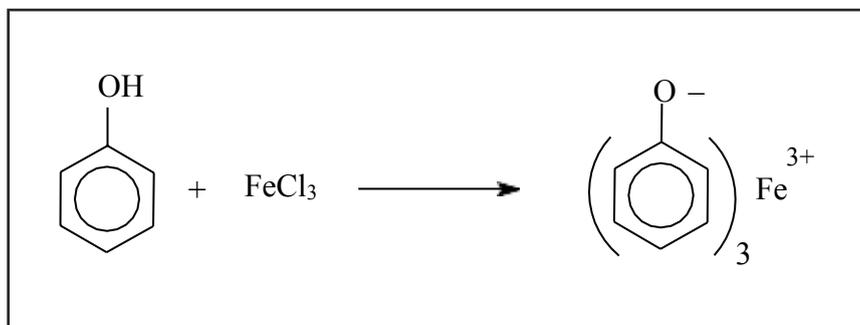


**Procedure.** In a test tube introduce 1 mL of water and about 0.2 g of phenol. Add enough bromine-water and shake until the yellow color persists. Observe the formation of a precipitate.



### 3. Ferric Chloride Test

The presence of a phenolic (or enolic group) in a compound is indicated by the formation of a violet (or red) iron complex when treated with a ferric chloride solution.



#### PROCEDURE

**Glassware:** 1 test tube

**The following alcohols to be tested:** phenol, and cyclohexanol.



1. In a test tube, place **3 mL** of **water**.
2. Add **5** drops of the **Unknown**.
3. Add **1-2** drops of **1% ferric chloride solution**.
4. Shake well and allow to stand for **1-2** minutes.
5. Record your observations **and** result.

**Positive results → violet solution will be formed**

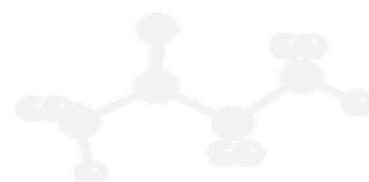
#### Useful links

Alcohols (Classification and Tests)

<https://www.youtube.com/watch?v=dinNjEiqxcg>

Organic Chemistry Experiment: Solubility of Alcohol and Phenol

<https://www.youtube.com/watch?v=iVEmFOweVA>



# EXPERIMENT 3

## ALCOHOLS AND PHENOLS

### Report Sheet

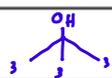
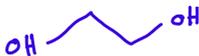
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➤ **OBJECTIVES:**

- Determine the chemical properties of alcohols & Phenols
- Identify the chemical properties of the unknown

➤ **ALCOHOLS:**

#### I. Solubility of Alcohols in Water

Alcohol	Structure	Solubility
ethanol	 $C_2H_6O$	✓
1-butanol	 $C_4H_{10}O$	X
2-methyl-2-propanol	 $C_4H_{10}O$	✓
ethylene glycol	 $C_2H_6O_2$	✓

What general conclusions can you draw concerning the solubility of alcohols in water?

- \* Solubility  $\propto \frac{1}{M.W}$
- \* Solubility  $\propto$  number of Branches
- \* Solubility  $\propto$  number of Hydroxyl Groups

#### II. Oxidation of Alcohols with Chromic Acid

Alcohol	Result (+ or -)	Observations (color, ppt,...)
1-butanol	+	Color Change to Green
2-butanol	+	
2-methyl-2-propanol	-	No Color Change

#### III. Lucas Test

Alcohol	Result (+ or -)	Observations (color, ppt,...)
1-butanol	+	Nothing
2-butanol	+	Cloudy & white after heating
2-methyl-2-propanol	-	Cloudy & white mixer

Arrange the three alcohols according to their rates of reaction with the Lucas reagent:

2-methyl-2-propanol > 2-Butanol > 1-Butanol  
 - 3° alcohol - immediately      - 2° alcohol - 5-10 min with heating      - 1° alcohol - take several hours

#### IV. Iodoform Test

2° alcohol & ethanol ⇒ Give (+) result

Alcohol	Result (+ or -)	Observations (color, ppt,..)
1-butanol	-	Nothing
2-butanol	+	Bright yellow ppt
2-methyl-2-propanol	-	Nothing

### ➤ PHENOLS

#### I. Ferric Chloride Test

Alcohol	Result (+ or -)	Observations (color, ppt,..)
Cyclohexanol <i>ألكحول سيكلوهكسانول</i>		
Phenol	+	Violet color

### ➤ Unknown Alcohol Determination:

According to your unknown

Unknown ID:		
Test used	Observation	Result
Chromic Acid Oxidation		
Lucas Test		
Iodoform test		
Ferric Chloride		

- Based on your results, what is your unknown alcohol type?
- Draw the expected alcohol structure of your alcohol showing the main function group: