Summary

ORGANIC LAB

Madar

ask.. believe and recieve

Melting point

Purpose :- Identification and Purity testing

* what is melting point?

Temp at which Solid and liquids phases are in equilibrium. at pressure of 1atm.

* we'll use organic Compounds in this expm why are organic compounds solids at room Temp?

Because of strong intermolecular forces.

* why different substances have different melting points?

nature and strength of intermolecular forces are responsible for the absorved differences in melting points

* Factors affect the melting point are :-

① Nature of bonds (stronger bonds → higher m.p)

② Molar mass ↑ m.p ↑

③ Branching ↑ كلما زاد m.p ↓
(molar mass مو لأنها زادت لنقص)

④ Impurities
→ Insolube (غير ذائبة) have no effect (glass, solid).
→ Soluble . ① lower melting point (and the greater amount of impurity, the greater is the melting point depression)

② Broaden melting point Range.

* Melting Point range ⇒ ($T_1 - T_2$)

T_1: Tem p at which substance starts melting

T_2: Temp at which substance is all in liquid state.

Our melting poin $T_{m.p}$ = avg of this range. $T_{mp} = \frac{T_1 + T_2}{2}$.

* Melting point range is affected by :-
① Particle size ② amount of material used ③ density of packing in the capillary tube ④ thickness of Cabillary tube ⑤ rate of heating of the liquid bath.

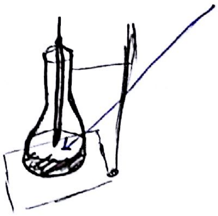
"مهم يأتي بال mid"

☒ Procedure :-

* What is the heat source used in expm?
(Direct flame, water bath, $\boxed{\text{Oil bath}}$)
answer

* Type of the oil used :- * high boiling *stable liquid like (parafin Oil
sillicon Oil)

* The thermometer bulb should be about 1cm above the bottom of flask.



to measure the right Temp (Temp of oil boiling) not the flask.

"لا شيءَ في هذا العالم،، فقط !" . بس حالياً خليك قاعد ادرس .. hhh

* In this expm we're dealing with two cases :-

① Usage of melting point measurment for purity testing.

m.p range ونقيس الذوبان عملية ونبدأ capillary tube بال المادة نحط [illegible] لك نجيب

Ⓐ We may have narrow m.p range. ← لاحظ

Soo ⇒ our substance is pure substance.

Ⓑ OR we can get wide range of m.p.

Soo ⇒ our substance is actually a (mixture) of other pure organic substances.

② Usage of melting point for Identification.

"Unknown part"

*اجراء التجربة على مادة غير معروفة ومقارنتها بدرجات m.p لمواد معروفة . (Table 1) manual

* نضع أسماء المواد المفضلة ان تكون هي Unknown (نستنتج ربما ذوبانها قريبة من التي حصلنا عليها في تجربتنا)

*سنأخذ خليط من Unknown والمادة المفضلة ونجري التجربة مرة أخرى (نقيس m.p)

اذا حصلنا على narrow range of m.p. ← فان Unknown ← هو هذه المادة المفضلة.

اذا حصلنا على wide range of m.p. ← فان كلا Unknown والمادة المفضلة مواد مختلفة

كأن كلاهما كـ (as impurity for each other)

⇒ فكرة ممكن "نستثمرها" من تجربة unknown انتوا ④

Compounds with Similar m.p doesn't mean they are the same Compound!

to be certain, we mix them and measure m.p for mix.
If
→ narrow range of m.p resulted (same compound)
→ wide range of m.p resulted (different compounds) (and below their m.p) الأصلية.

So2al Different compounds, compound A has 130°C m.p and compound B has also 130°C m.p. If mix of Both as (1:1) ratio has been prepared. What is m.p of that mix??

(a) > 130°c , $\boxed{\text{b) < 130°c}}$ ✓ c) = 130°c)

(سؤال) Wetted sample has a m.p (higher, [✓ lower]) than dried sample

Question) What effects on m.p measured would you expect in these cases?

① presence of pieces of glass in sample ⇒ No effect cuz It's insoluble material.

② presence of solvent within crystals ⇒ lower m.p.
لانه اصلا هدفنا انا نوصل من صلب ع سائل ← فبما يحوي جزء من اصله already سائل
→ we need less energy → low melting point.

③ Rapid heating during melting point determination.
raise m.p. because rapid heating won't provide enough energy to break bonds. we'll need more energy → more time heating → ↑ m.p.

Name of expm :- Boiling point and distillation.

Purpose :-. Identity and Purity of liquid organic Compounds.

*What is the boiling Point?.

Temp at which vapor pressure equals external pressure. at when vapor and liquid are in equilibrium.

* Boiling Point of Pure liquids depends on:- ① Nature of Inter-molecular forces.

H-bonding, dipole-dipole, London-forces.

← stronger bond- higher Bp ←

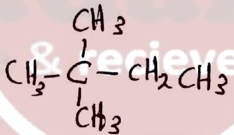
② Molarmass ↑ B.p ↑

③ shape. straight chained molecules have higher B.p than Branched ones.

$CH_3CH_2CH_2CH_2CH_2CH_3$

bp 68

n-hexane



$CH_3-C(CH_3)_2-CH_2CH_3$

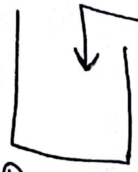
bp 50

2,2 dimethyl butane

* Boiling Point for Solutions.

Temp at which vapor pressure of that Solution equals the external atmospheric pressure.

to form a solution, we add substances into pure liquids and that affects (their Boiling point) depending on the nature of substances we add!



Pure liquid (A)

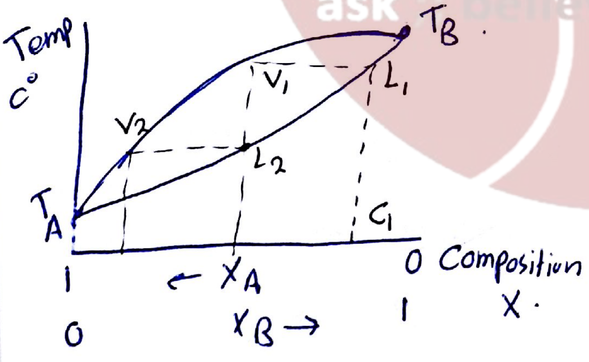
adding B
- → Soluble and volatile → affects b.p.
- → Soluble and non-volatile → affects b.p.
- → Non-Soluble and non-volatile → doesn't affect b.p.

*Applications of B.p measurments :-

① Identification.

measure B.p of Some unknown substance. then Comparing It. With Recorded data in tables.

② Purification and Separation of mixtures By distillation. ⇒ (process of heating a liquid to It's boiling point → condensing It's vapor. by cooling It → then collecting the liquid distillate.



* شرح curve يمثل ما ~~[illegible]~~ يحصل في Distillation

نبدأ ب mix من (A,B) تركيزه C_1

نسخنه لـ $T_{B.p}$ معينة وعندها يصبح 2-phases

L_1 , V_1

نبرد V_1 vapor phase بخفض حرارته

ليصبح liquid ← ثم نسخن liquid الى B.p

وهكذا نستمر الحلقة . [طب احنا شو استفدنا؟] ثم تبدأ عملية التسخين للغليان والتبريد للتقطير نتوجه الى محلول نسبة الأكثر فيه هي المادة more volatile يعني حققنا فصل المادة A عن B .

Types of distillation:-

① Simple ⇒ used only when boiling points of the components are 80 degree or more apart.

② fractional ⇒ when mixtures of liquids have Boiling points much less than 80 degrees.

③ Vaccum ⇒ used only for high boiling liquids, and for compounds that decompose at atmospheric pressure.

* for simple and fractional distillation:-

* Boiling flask should not be more than half full ??
to provide some space for the produced vapor.

* Boiling stones are added to the liquid ??
to prevent bumping.

* Each ground joint should be greased ??
to ensure a completely sealed system.

* Cooling water in the condenser should enter at the lower end and exit at the upper end ??
This ensures that the Condenser jacket is always full of water.

* The bulb of the thermometer should be below the opening of the side arm ?? to measure the Temp at which liquid and vapors are in equilibrium.

* Heat Sources used in distillation

- Water-bath
  used to distil low-boiling and flammable liquids.
- burner
  used for higher boiling and less flammable liquids

* During distillation process, we must make sure that there is a drop of liquid hanging from the bulb of thermometer. why is that??

to ensure that thermometer is reading the correct boiling point, Absence of this droplet indicates superheating.

GooD Luck

:)))

Name of expm :- Recrystallization.

Purpose :- Purification technique for solids.

* Types of impurities in a solid :-

① insoluble impurities ⇒ Can be removed by (hot filtration/gravity filtration)

② Soluble impurities ⇒ Can be removed by (cold filtration/suction filtration)

* Both procedures (hot/cold filtration) included in <u>Recrystallization</u>

* Solubility of a solid solute in a solvent is determined by 2 factors

① "Like dissolves like"

Polar solvents dissolves polar solute.
↓
water
methanol
ethanol

polar solute: which contain groups like OH, $COOH$, NH_2.

② Non polar solvents dissolves non-polar solutes
↓
Chloroform
Carbon tetrachloride
hexane
Petroleum ether

non-polar solutes: hydrocarbons.

② The lattice energy. the higher lattice energy ↑ ⇒ less soluble substance will be.

the higher lattice energy ↑ ⇒ the higher melting point ↑

For our expm we need solvent to dissolve the solute we need to purify, this solvent should possess the following properties:-

① high Temp Coefficient toward the Substance to be purified

يعني بذوب اعادي على حرارة مرتفعة فقط

② low Temp Coefficient toward the impurities.

الأفضل انه ما يذوب الشوائب.
ولكن اذا كان بذيبها. الأفضل على حرارة قليلة.

③ Not react with the substance to be purified

④ be relatively Volatile.

⑤ non-flammable, non-toxic and cheap.

* In Selection the suitable solvent

Cold hot
− +
لا يذوب يذوب

Solvents:-

مثال

	H_2O	CH_3OH	CH_3Cl
①	+ −	− −	− +
②	+ +	− −	− −

(good solvent هاد الشكل الذي نبحث عنه (عشان نعتبره

In case ① $\boxed{-+}$ هو المذيب المناسب CH_3Cl

In case ② نضطر للجوء لـ solvent-pair

نأخذ اثنين $\boxed{++}$ and $\boxed{--}$

ولكن يوجد اثنين $\boxed{--}$ فأيهما نضيف ؟؟

طبعي نوخذ الي بيتناسب مع $\boxed{++}$ ← ذائبين مع بعض (misicible)
H_2O , CH_3OH

* Crystal formation is a selective process, only molecules of the same substance can fit into the crystal lattice. excluding foreign molecules (impurities) which remain in solution

solid parts
impurities: يعني بناسب الاحجام (الترتيب الفراغي) هي المادة التي بنا لياها. الذائبة في المحلول ←

Recrystallization Procedure :-

* Dissolve the substance in aminnimum Portion of ahot suitable solvent

ليه → Why a slight excess of the solvent is usually added ?.

to Compensate for any losses (through evaporation) during hot filtration.

here → why we may add charcoal into the flask?

cuz It has a decolorizing effect. ~~against~~ against colored impurities.

* Do ahot filtration. (using a fulted filter paper and funnel).

ليه → this step is to get rid of insolube impurities.

* Now we have (our Substance and Soluble impurities) left.
in the mixture

Cool the mixture down until ⇢ large amount of crystals has formed.

* Cooling must be in a slow base?? to get good sized of Crystals.

* step 2 of Recrystallization is Cold filtration Using (Buchner funnel)

why Buchner funnel ⇒ to ensure rapid and Complete removal of the Solvent.

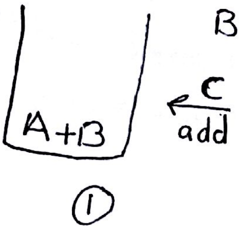
* Crystals we got must be washed with a few milliliters of fresh-ice-Cold solvent ?? to get rid of the last traces of solvent ($\overset{\text{soluble}}{\text{impurites}}$).

* Crystals then dried in an oven.

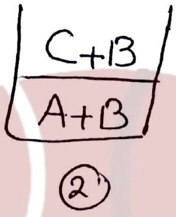
Name of expm :- Extraction.

Purpose :- Separation and Isolation technique.

* Extraction is the separation of a substance from a mixture. by means of a solvent that partily dissolves that substance.



B our desired Component, C is solvent that immiscible with A and pantily dissolves B.



two phases.
(Usually organic and aqueous)

* we separate the previous mixture by a separatory funnel.

* In that case, we can define a term called (distribution Coefficien)

$$\underbrace{K_D = \frac{C_o}{C_w} = \frac{S_o}{S_w}}_{\text{only at equilibrium.}}$$

$S_o \rightarrow$ organic phase.
$S_w \rightarrow$ water phase

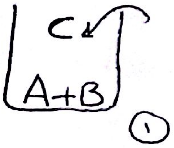
$C \rightarrow$ Concentration.
$S \rightarrow$ Solubility.

* when K_D is large. → single extraction would be enough to remove desired Component from the mixture. لانو ذائبية المركب بال organic عالية.

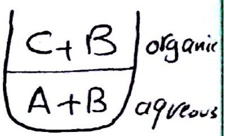
* when K_D is small → repeated extractions are required. to recover a compound from aqueous mixture. ذائبية المركب بال Two phases متقاربة.

(In general) It's better and more efficient to divide total volume of extracting solvent. over several extractions than to use the whole volume in single extraction.

Extraction ⇒



Substance to extract ⇒ B.
Solvent added ⇒ C



Choice of Solvent (C)

① Immiscible with the liquid (A) in which the Solute (B) is present.

احنا بناخذ أحد الطبقتين ونعالجهم . حتى نحصل على المادة العضوية B

② Readily dissolve the Solute to be extracted

③ extract little or none of impurities and other Compounds present in mix.

④ Volatile, easily removed from the Solute after extraction.

*

عادةً المادة العضوية بعد أن يتم الحصول عليها ذائبة في (aqueous mix) . عن طريق استخدام (separatory funnel) . ممكن أن تتشرب جزء من طبقة organic للمزيج النهائي . كيف نعالج ذلك ؟

* Drying agents ($CaCl_2$, $MgSO_4$, Na_2SO_4) absorbs traces of water in organic phase.

* By (Salting-out) :- Saturating the aqueous phase with a salt. Such as NaCl or Na_2CO_3.

What are the effects of previous procedure :-

① Decrease Solubility of organic Compounds in saturated aqueous phase

② Decrease Solubility of organic and aqueous phase in each other

* فخلال تجربة extraction ممكن يصير في خلل في انفصال (mix) الطبقتين org / aqu وذلك يسمّى ب (Emulsion)

How to prevent emulsion :-

By avoiding vigorous shaking of the layers whenever an emulsion is expected to form.

* كيف نتخلص من Emulsion اذا صار، وقلّله ؟؟ .

① Stirring the emulsified layer gently with a glass rod.

② Saturating the aqueous layer with Salt.

③ Centrifugation.

Extraction of Caffine from tea leaves :-

Caffeine → organic Compound . belongs to alkaloids family

In this expm , you will extract Caffeine from tea leaves .

* why we add Sodium Carbonate ??

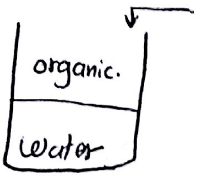
Because this treatment also extracts tannis , (class of acidic organic compounds) also present in the leaves . so to remove It . we add Soduim Carbonate . and converting them to water Soluble salt .

* Solvent used in extraction Caffeine ⇒ dichloromethane .

* why the funnel is shaken gently at the beginning and vented periodically ??? to release excess pressure which builds up inside

معظم الأسئلة تأتي بهالشكل :-

(Example) Find mass of Caffeine in each Phase :-



10g caffeine .
Total .
متوزعة بين
الاثنين .

Given :- Volume of water = 100ml
Volume of organic solvent added = 50mL.
$K_D = 10$ مثلاً

Answer :- let mass of Caffeine in water $10 - X$.
" " " in organic X

$$K_D = \frac{C_o}{C_w} \quad \frac{mass/V\ o}{mass/v\ w} = \boxed{\frac{X/50}{(10-x)/100} = 10}$$

$X = 8.333\text{ g}$.

Name of expm:- Steam distillation.

Purpose:- *Purification and *Separation of liquids and solids.

Steam distillation ⇒ separation of ① *slightly volatile ② *water insoluble *from non-volatile materials by means of steam. at low Temp. distillation.

عادةً بنستخدم للمواد التي درجة غليانها عالية جداً. فالفكرة انو بنسخنها مع water
بعدين نفصلهم عن بعض.

*Applications of this method:-

① Separation of such compounds from mixtures containing non-volatile impurities

الشروط التي سبق ذكرها بالأعلى.

② isolation of steam-volatile organic compounds from natural sources

Example:- isolation of essential oils. from the plant tissue.

*[Part1] Isolation of essential Oils. from Cloves. القرنفل

System used → same as simple distillation. except, we need large distillation flask. Containing large volume of water ?? why.

To provide steam. [لأنو اساسها the. اصلا بحاجة لل steam مشان ما رح تطلع ماء.] ^^

⇒

* The volatile essential oils in some plants are generally complex mixtures of what??

Hydrocarbons, alcohols and carbonyl compounds.

* Oil of cloves is rich In (eugenol) which is Used for what ??

Used medicinaly as a dental antiseptic and analgesic.

* Unlike ordinary distillation, steam distillation is Vigorous "شديد", So we should take care to avoid [material boiling over into the receiver]

* In which case do we use an air Condenser instead of water Condenser?.

In Case the substane being distilled is a solid, It often solidifies and accumulates in side Condenser.

* After we Collect the distillate in the process of [Isolation of essential-oils] what processes shall come next ???

- Adding Sodium chloride to the distillate we've collected.
- Cooling It to room Temp.
- Extraction using separatory funnel and with 15ml of dichloromethane.
- Collecting the organic layer and Drying It with anhydrous sodium Sulfate.
- Evaporate the solvent on the steam bath, in fume hood.

* Steam distillation always takes place at Temp below boiling point of water. SO It is *considered best method to distill high-boiling steam-volatile, organic compound at Temp below 100°c.

*يعني بدون steam distillation بنواجه صعوبة في توفير حرارة عالية جداً لأنه في درجة غليان بعض المواد العضوية
من اجل نفصلها بهي طريقة (normal distillation)

So this method $\longrightarrow$ *can replace vacuum distillation.

$\downarrow$ *by It we can avoid possible decomposition of such composition.

"all (*) marked sentences are advantages of this method"

Dis advantages :-

Steam distillation is not enough alone to get pure substance.

It requires Extraction to separate the desired substance from water.

* In the process of evaporation, we finally get to equilibrium point. at that point we can apply these relationships:-

$$\frac{\text{moles of A}}{\text{moles of B}} = \frac{\text{mass of A} / \text{molecular weight of A } (MW_A)}{\text{mass of B} / \text{molecular weight of B } (MW_B)} = \frac{P_A^o \text{ vapor pressure}}{P_B^o \text{ vapor pressure}}$$

$$\frac{n_A}{n_B} = \frac{(\text{mass}/MW)_A}{(\text{mass}/MW)_B} = \frac{P_A^o}{P_B^o}.$$

Name of exp:- Chromatography.

Purpose:- * Purification * separation * Identification * Purity testing

Types of chromatography:- ① TLC [Thin layer chromatography]

② PC [Paper Chromatography]

③ gc [gas Chromatography].

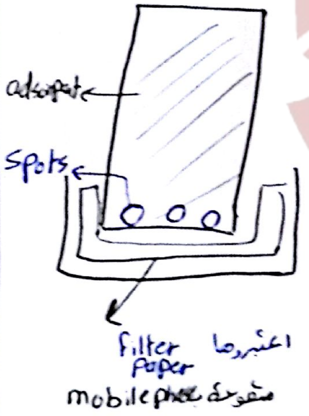
* Two principles are basically involved in Chromatography:-

① In (TLC) ⇒ adsorption

② In (PC) ⇒ Partition

* [TLC] we'll be dealing with two phases:-

* Stationary phase. (adsorbent) (Solid and polar)



*usually we use Silicagel ($SiO_2 . x H_2O$)

alumina ($Al_2O_3 . x H_2O$).

*(In expm) we'll use homogeneous slurry of those adsorbents in volatile organic solvent (chloroform or dichloromethane)

* we dip the microscope slides in the slurry and hold them vertically to air-dry..

بالأخير بتصير السلايد مغطاة بمادة (adsorpant) على شكل صلب

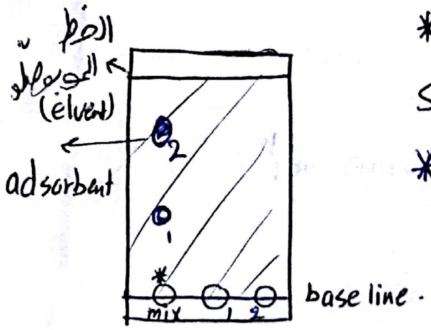
* why do we shake the jar of adsorbent before each use??

to homogenize the slurry.

* Mobile phase (eluent) (developing Solvent)

* In this expm we'll use dichloromethane as mobile phase.

* The mobile phase moves up the solid adsorbent, the adsorbed components of the mix get absorbed and carried along at diff rates by moving solvent



* spots of different substances (Mixture or pure substance to Compare R_f values later).

* num of spots that appear on surface of plate when the sepration is over indicates num of the components inside mix.

* efficiveness of separation depends on:-

① Kind and activity of the adsorbent.

② Polarity of eluent (mobile phase)

③ chemical nature of the componets of the mix.

بشكل عام (mobile phase) سوف تحرك أفراد معه ال Components للمزيج. وحركة Eluent و Components
كلها معتمدة على ما سبق.

① and ③ ⇒ The more strongly absorbed a given compound is, slower it is transported by mobile phas. $R_f \downarrow$

② ⇒ The greater Polarity of the eluent, the greater Its ability to dislodge a compound from the surface of the adsorbent. $R_f \uparrow$

* The plate is ~~developed~~ placed in developed chamber (covered beaker) Containing the solvent and lined with filter paper soaked in the solvent.

why do we use a covered beaker ?? to help saturate the atmosphere with solvent vapors.

*R_F value (retardation factor):-

(num(s) less than 1). It's a characteristic of a compound for a given adsorbent and developing.

$$R_F = \frac{\text{distance traveled by the compound}}{\text{distance traveled by the solvent}}.$$

R_F values depend on a) nature of solute

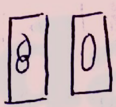
كلما كان عندي قابلية للامتصاص من قبل (adsorbent) رح يمشي مع الـ Components لأعلى أكثر.
(elvent)

R_F ↑

b) Nature of mobile phase. when polarity ↑ R_F ↑

* Problems we can face in this expm:-

① Trailing and overlapping



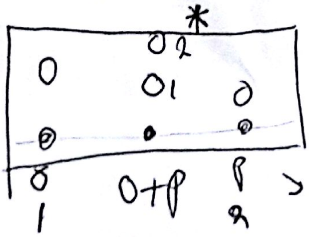
how can we avoid it?? By using less amount of mix during spotting

② locating compounds on the plate (Spots).

→ for colored spots → by eye
→ for non-colored spots → UV light
non-visible → I_2 vapor
→ H_2SO_4

③ How can this method be used for Identification process

نقارن R_F value لتحديد المواد الغريبة المجهولة



1 O → ortho
2 P → para.

* mix بـ R_F value للمواد التي تم فصلها من

"Similar R_F values similar compounds"

Name: [redacted]

6.5

1) Soluble Impurities were removed during the recrystallization Exp. by cold filtration (suction). 2

2) Charcoal was added to the hot solution to remove colored impurities. 2

3) Give example of drying agent $CaCl_2$ / $MgSO_4$ / Na_2SO_4

the purpose of this agent during the extraction exp. is to remove any ~~ef~~ effect of mother liqoure (saturated solution). and separate it from our compound that we want to purify. 0.5

4) Aqueous solution of sodium carbonate was added into a solution of tea leaves in water since It works as salting agent to prevent organic and aqueous substances from mixing.

5) The K_D (C_{ether}/C_{H_2O}) for an organic substance X is 10. what relative volumes of ether to water should be used for the extraction of 90% of X from a water?

w .9
eth .1

$$K_D = \frac{C_{ether}}{C_{H_2O}} = \frac{\frac{\text{mass of ether}}{V_{ether}}}{\frac{\text{mass of } H_2O}{V_{H_2O}}}$$

1

$$10 = \frac{\frac{.1}{V_{ether}}}{\frac{.9}{V_{H_2O}}} = \frac{.1 * V_{H_2O}}{V_{ether} * .9} = 10.$$

$$\frac{V_{H_2O}}{V_{ether}} = \frac{10 * .9}{.1} = \frac{90}{1} \qquad \frac{V_{ether}}{V_{H_2O}} = \frac{1}{90} = 0.01$$

Organic Lab Quiz

⑦

Name [redacted]

1) The m.p of Compound (A) 130°C and Compound (B) 130°C ⟹ mixture of (A) and (B) (1:1) ratio has a m.p a) >130°C [✓ b) <130°C] c) = 130°C

1

2) Wetted sample has a m.p (higher or [✓ lower] or =) than a dried sample.

← الجواب

1

3) The m.p range is affected by:

1 a) purity only b) amount of material used only in the Capillary tube [✓ c) A+B]

4) The applications of m.p experiment are:

1 a) Identification

b) Purity test

5) Pure sample has a narrow m.p range (narrow or wide).

1

6) Boiling stones are used in the distillation to prevent bumping

1

7) Mixture of (water _ acetone) was heated during the exp. by: [✓ water_bath] or direct flame.

2

8) Vacuum distillation could be used if the substance is ~~has~~ high boiling and ~~maybe~~ because of Its decomposition under external pressure.

1

9) B.P of liquid (A) 70°C and for liquid (B) 110°C ⟹ Simple distillation will be effective for separation ([✓ T] or F).

2

10) "Sugar in water" soln has a b.p. a) >100°C b) <100°C [✓ c) = 100°C]

2

11) As Atmospheric pressure decreases ⟹ b.p of liquid decreases

1

⑨ Dehydration of alcohols :- / Formation of alKenes.

loss of water molecule.

Dehydration of alcohols requires an acid catalyst and heat.

H_3PO_4 / H_2SO_4.

معلومة

لا نستخدم HBr/HCl
لانّه Cl و Br هيكونو
strong Nucliphile.
يعني ممكن يستبدلوا الـ OH -
ويحولوا الـ alcohol الى alkyl halide.
مش الكين -

Tertiary alcohols dehydrate most easily while primary ~~and secondary~~ are the most difficult to dehydrate.

معلومة

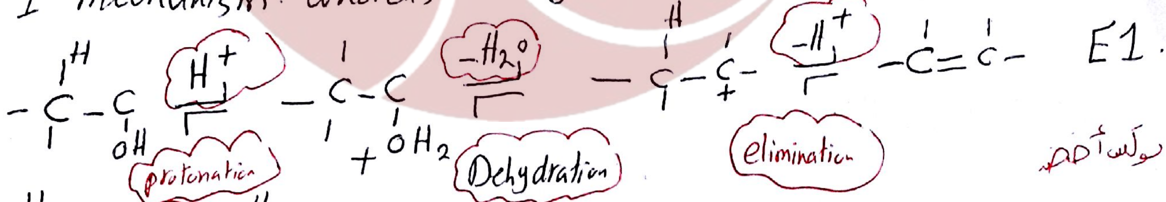
معلومة.

لذلك العوامل المساعدة (heat and acid) تكون أقلّ عند حدوث dehydration for 3°.

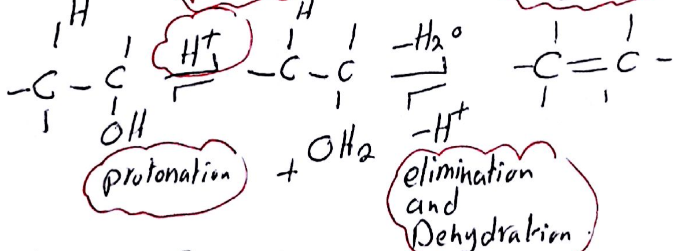
~~$R-CH_2OH \xrightarrow[140°C]{95\% H_2SO_4}$~~

انظر بوكس ص 83 في manual.

Dehydration of 3°/2° alcohol is faster than 1° due to its E1 mechanism whereas 1° by E2 mechanism.



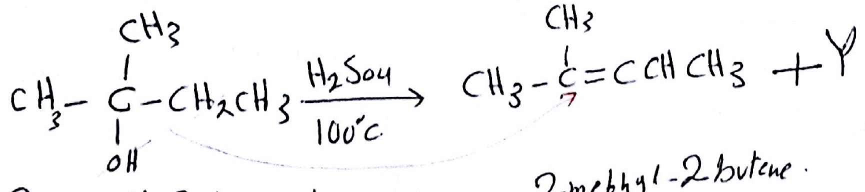
بوكس أحمر



مش فقط بل افهم المعادلات. كل خطوة وليش صارت

Experiment ① Preperation of 2-methyl-2-Butene

Equation

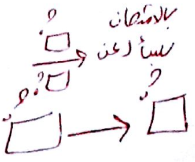


$$CH_3-\overset{CH_3}{\underset{OH}{C}}-CH_2CH_3 \xrightarrow[100°C]{H_2SO_4} CH_3-\overset{CH_3}{C}=CCHCH_3 + Y$$

2-methyl-2-butanol

2-methyl-2-butene.

بوكس أخضر



٢ سؤال أجا بالفاينل يقصد في H_2O الي طلع لما كونت الألكين

⁂ How we get rid of H_2O in mix ?

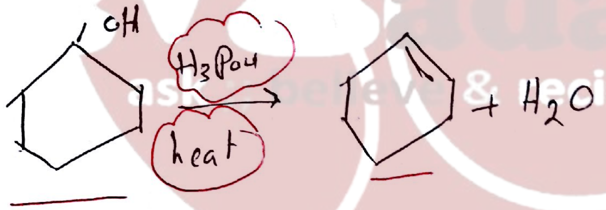
by separatory funnel and washing it with NaOH solution then dry it over anhydrous calcium chloride.

⁂ How we separate the alkene product from the mix :-

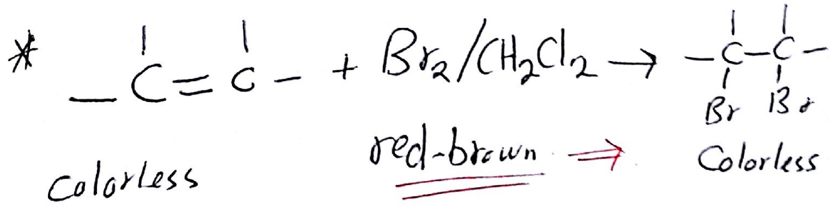
Using Distillation.

⁂ we Continue distillation of the Crude alkene until thermometer indicates a rise in Temp above 50°

⁂ ② Cyclohexene from Cyclohexanol.

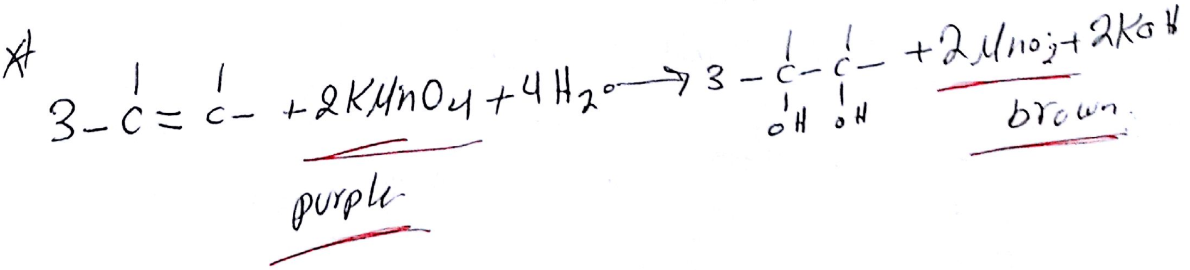


③ Tests for alkene.



$$-\overset{|}{C}=\overset{|}{C}- + Br_2/CH_2Cl_2 \longrightarrow -\underset{Br}{\overset{|}{C}}-\underset{Br}{\overset{|}{C}}-$$

colorless red-brown ⇒ Colorless.



$$3-\overset{|}{C}=\overset{|}{C}- + 2KMnO_4 + 4H_2O \longrightarrow 3-\underset{OH}{\overset{|}{C}}-\underset{OH}{\overset{|}{C}}- + 2MnO_2 + 2KOH$$

purple brown

⑪ Nucleophilic substitution / preperation of alkyl halides

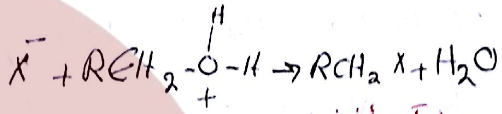
$R-OH + \bar{X} \rightarrow R-X$ ببساطة

بوكس أخضر -
معلومات مهمة لازم تفهم
ممكن تيجي بالامتحان .
(موجود بالمانيوال)

* for this reaction to occur alcohols are treated with HX (hydrohalic acids), (PX_3, PX_5) phosphorous halides, $SOCl_2$ thionyl Chloride.

* Reactivity of alcohols towards hydrohalic acids follows the order

$3° > 2° > 1°$ → by S_N2 mechanism.

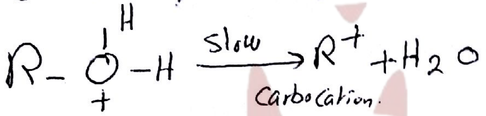


$$\bar{X} + RCH_2-\overset{H}{\underset{+}{O}}-H \rightarrow RCH_2X + H_2O$$

بوكس أخضر

"مش حفظ فهم"

↓ by S_N1 mechanism



$$R-\overset{H}{\underset{+}{O}}-H \xrightarrow{slow} \underset{\text{Carbocation}}{R^+} + H_2O$$

$$R^+ + \bar{X} \xrightarrow{fast} R-X$$

بوكس أخضر

* # معلومة

السرعة اللي في التفاعل
لا تحتاج اي
في الـ 2 ، 1° تحتاج الى

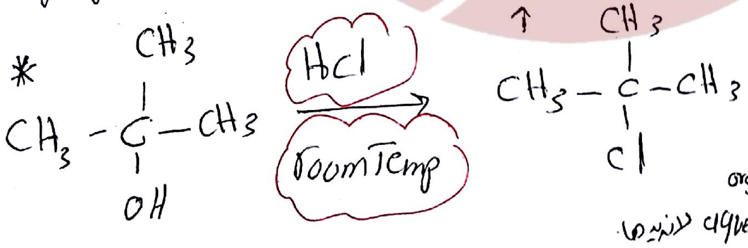
3°

special Conditions

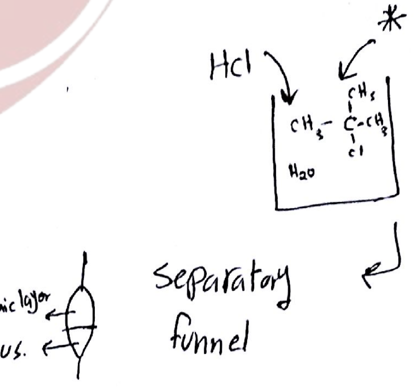
Catalyst

$ZnCl_2$.

① preperation of 2-Chloro-2-methyl propane.



$$CH_3-\overset{CH_3}{\underset{OH}{C}}-CH_3 \xrightarrow[\text{room Temp}]{HCl} CH_3-\overset{CH_3}{\underset{Cl}{C}}-CH_3$$



Wash it with H_2O [to remove excess HCl] → organic layer (beaker)

Distillation ← Decantation ← add anhydrous Calcium Chloride. ←

② preperation of 1-Bromobutane

"بوكس أخضر"

$$2NaBr + H_2SO_4 \rightleftharpoons 2HBr + Na_2SO_4$$ تحضير HBr

$$CH_3CH_2CH_2CH_2OH + HBr \rightleftharpoons CH_3CH_2CH_2CH_2Br.$$

* Relative Rates of alkyl halide.

ethanol and Ag^+. نعمل تفاعل مع

نميز بين أنواع alkyl halid. 1° 2° 3°

observation: production of silverhalide AgX.

The most reactive is Tertiary halides. 3° > 2° > 1°.

$$R-X + Ag^+ \rightarrow R^+ + \boxed{AgX.}$$

$$R^+ + CH_3CH_2OH \rightarrow R-\underset{\displaystyle |\atop H}{\overset{+}{O}}-CH_2CH_3 \rightleftharpoons \boxed{R-O-CH_2CH_3} + H^+.$$

"مو لازم معجزة كونيّة تخلّيك تبتسم"

:)))

①

Electrophile aromatic Substitution

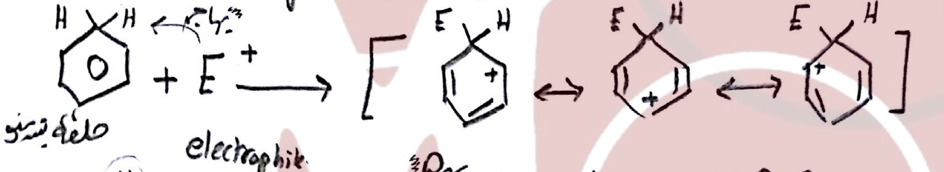
+ve compounds
تفتقدالى الالكترونات
فتهاجم حلقة البنزين
ذات π bonds
الغنيه بالالكترونات

من عائلة البنزين

استبدال H
بمجموعة أخرى

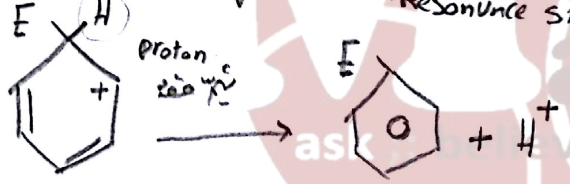
Examples of these reactions :- nitration, halogenation, Sulfonation, Friedal alkylation and acylation. *

Mechanism of electrophilic .. : *



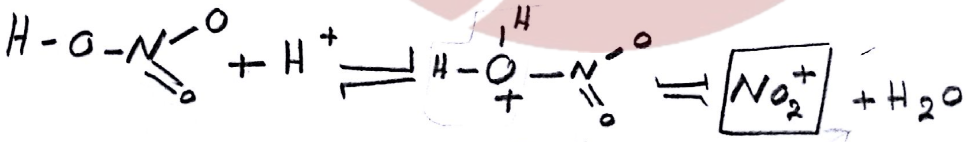
بوكس أحمر

* Resonance structures of Benzonium Ion.



Equations to generate nitronium / bromonium Ion :-

بوكس أحمر

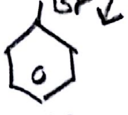


$$H-O-NO_2 + H^+ \rightleftharpoons H-\overset{H}{\underset{+}{O}}-NO_2 \rightleftharpoons \boxed{NO_2^+} + H_2O$$

$$Br-Br + FeBr_3 \rightleftharpoons \boxed{Br^+} + [FeBr_4]^-$$

Our experiment :- part I Nitration of BromoBenzen.

NO_2^+ سنستخدم as our E^+



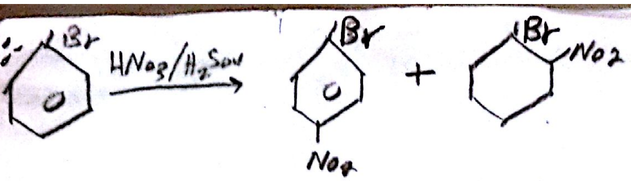
ماذا سنستخدم كعامل مساعد للتفاعل : Concentrated HNO_3/H_2SO_4

Both. (nitric acid sulfaric acid)

لتحضير NO_2^+ الالكتروفايل بحت ^^

Eqn:-

②



p- bromonitrobenzen
mp 127

O - bromo nitro benzen
mp 43

[illegible] *

ملاحظتين :-

* Molecular weight for O and P isomers ar the same

* mp for $\boxed{O-}$ is lower

* يعني شو؟ - يعني هي الي تبقى ذائبة في المحلول تحت
تجمده سنيا $\boxed{P-}$ mp تبعها أعلى يعني بدها
حرارة أعلى عشان تتحول من solid ← liquid للأشوفه
اثناء التفاعل فتبقى solid.

procedure:- من الكتاب على شكل أسئلة

*why we use cold water bath for mixing H_2SO_4/HNO_3 in the beginning of reaction ?

because their reaction will cause Temp to rise.

*when doing exp Temp must be maintained at ? 60°c

سؤال قصير

*mixture of bromobenzen and (catalyst) is heated by ?
(HNO_3/H_2SO_4 above catalyst)

Direct flame / or boiling water bath

* which product remains soluble in mix and which 1 appears as ppt ?

O- soluble
P- precipitate

* how the precipitate p-bromonitrobenzen is processed ?

1) Filter It by suction (Buchner Funnle)
2) washing It with cold water
3) Recrystallize using ethanol

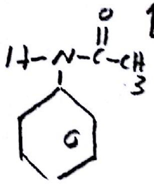
Part II Relative Rates of Bromination :- → ③

اضافة E^+ (Br^+)

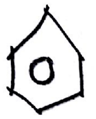
الى Benzen ring ←

أو مشتقاتها

phenol



acetanilide



+ Br_2 → X no reaction.

ونقارن ما بينهما مين الأسرع في التفاعل ؟

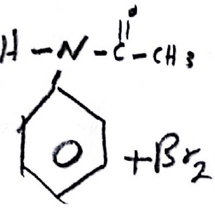
+ Br_2 → بيتفاعل بسرعة. Fast to decolorize

طب كيف بدنا نعرف مين الأسرع ؟

مؤشرات حدوث التفاعل ← لون → اختفاء

← راسب

← غاز



+ Br_2 → Moderate to decolorize

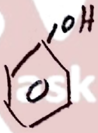
لون Br^+ orange.

اذا صار تفاعل بيصير Colorless.

ح يختفي.

Observation: time to decolorize.

* Reason that



is the Fastest when Reacting with Br_2 ?

−OH is activating group (electron releasing) لتفهم "ما بنسحب الالكترونات

من حلقة البنزين ولذلك تجذب E^+ الفقير بالالكترونات لأنه غني وبيرتبط بالحلقة

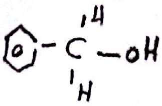
بسهولة. عكس H−N−C−CH_3

أكيد متذكرينها من Organic Course.

⑪ Alcohols and phenols

R-OH

بحيث OH- متصله directly لحلقة البنزين



!!الإضلال

هاي مش فينول

Alcohols :-

تصنيفها :-

$R-CH_2OH$ 1° $R-\overset{H}{\underset{OH}{C}}-R'$ 2° $R-\overset{R'''}{\underset{OH}{C}}-R'$ 3°

في علاقات تختص بكسر الروابط :.

① O ⋛ H (bases and esterification reactions)

② C ⋛ OH (Dehydration and subtitution)

رح نشرحهم بالتجربة (1) و (2)

Tests :- our experiment ① Solubility in water

للتمييز بين alcohols 1° 2° 3°

3 factors must be taken into Consideration:-

① #.of-OH (hydroxyl) groups ↑ تزداد s↑ كلما تزداد

ليش؟ لأنه Polar- OH والماء Polar -

② MW ↑ كلما زادت ↓ Solubility تقل

لأنه زيادة MW يعني C-C-C جزء Non-Polar بزيد فيقلل ذلك ذائبيته بالماء

لأنه ما عنده H-bonding.

③ branching ↑ كلما زاد s↑ تزداد   C-C-C-C $C-\underset{C}{C}-C$ ليش؟

لأنه مساحة سطح الجزء Non-polar تقل وتصبح أقل تعرضاً للماء كأنها تصبح الشكل الكروي.

1-butanol $C-C-C-\overset{OH}{C}$ ⟶ Insoluble

2-methy-2 propanol $C-\overset{CH_3}{\underset{OH}{C}}-C$ ⟶ soluble

حسب exprm

cyclo hexanol  ⟶ Insoluble

ethylene glycol $\overset{OH}{C}-\overset{OH}{C}$ ⟶ soluble

② Acidic Properties of alcohols . Reagent = metalic sodium Na .

Equation :- $2R-OH + 2Na \rightarrow 2R\bar{O}Na^{+} + H_2$ بوكسا خض

observation :- evolution of hydrogen gas $H_2\uparrow$

$1° > 2° > 3°$ ترتيب alcohols حسب شدة حامض

primary tertiary .

③ Oxidation of alcohols Reagent :- $K_2Cr_2O_7$ with H_2SO_4 .

oxidizing agent (orange) .

Equation :-

$$K_2Cr_2O_7 + R-CH_2OH \xrightarrow{H_2SO_4} R-\overset{O}{\overset{\|}{C}}-OH + Cr^{+3}$$

$$\underset{orange}{K_2Cr_2O_7} + R-\underset{R}{\underset{|}{C}}HOH \xrightarrow{H_2SO_4} R-\overset{\|}{C}-R + \underset{green}{Cr^{+3}}$$

بوكس أخضر

$$K_2Cr_2O_7 + R-\overset{R}{\overset{|}{\underset{R}{\underset{|}{C}}}}-OH \xrightarrow{H_2SO_4} \text{No reaction} .$$

Observation :- تحبذ لون البرتقالي الى green بعد ين 1°, 2° alcohols

بينما 3° لا تشتغل .

※ تذكر لاحظوا تحول [1° alcohol ← Carboxylic acid / Ketone ← 2° alcohol] والاكسدة

④ Lucas test Reagent [$ZnCl_2 + HCl$]

بوكس أخضر

Equation : $R-OH + ZnCl_2 \rightleftharpoons R-\underset{H}{\underset{|}{\overset{+}{O}}}-ZnCl_2 \xrightleftharpoons{-[HOZnCl_2]^{-}} R^{+} \xrightarrow{Cl^{-}} \underline{R-Cl}$

ثم فقدص

Insoluble alkyl halide

لون تكون دايب "ولكن عند تكوّن R-Cl يتعكّر .

observation :- Turbidity (العكورة) / cloudy appearence .

$3° > 2° > 1°$ تزداد شدة التعكّر

$\longleftarrow$

Phenols . مايميز Phenols عن alcohols بال Tests عملي [مهم]
⑥ acidity.

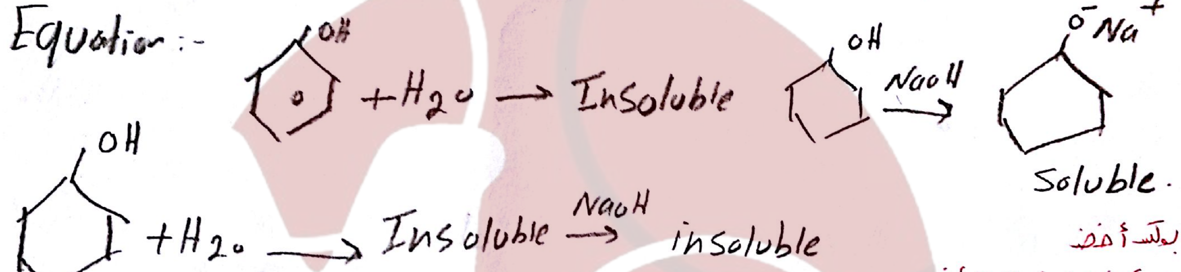
phenols are stronger acids than alcohols and react with NaOH whereas alcohols do not.

لمشي statement

cause phenoxide Ion is resonance - stabilized whereas the alkoxide ion is not.

Tests ① Acidity of phenols Reagent NaOH

Equation :-

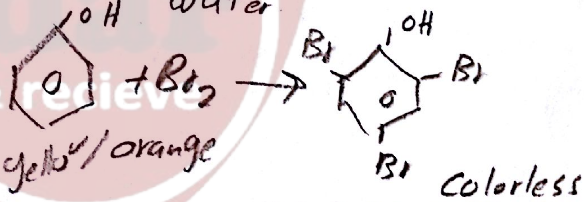


مش بوكس أخضر
مجرد توضيح للي كاتبنا بالتفاعل
يعني انه phenol تفاعل وذاب
بس cyclohexanol ما ذاب

Observation : وبان phenol بصير متجانس المحلول

② Bromination of phenols with Bromine water

Reagent Br_2 , Equation



بوكس أخضر

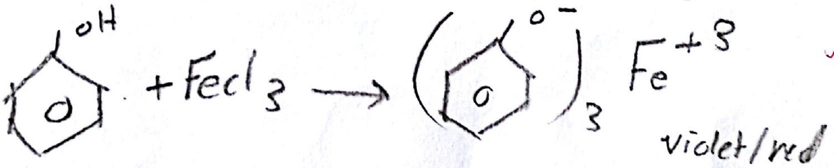
أو انه بيختفي اللون

2,4,6

observation:- production of tri bromophenol and Removal of yellow Color.

③ Ferric Chloride Test Reagent :- $FeCl_3$

Equation

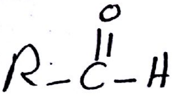


بوكس أخضر

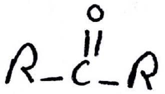
Observation:- Violet/red Iron Complex indicates the present of phenolic or enolic group

AlDehydes and Ketones

①



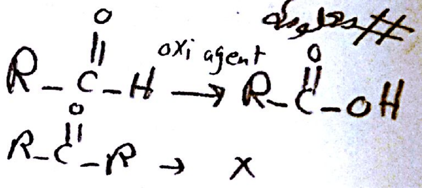
$R-\overset{O}{\overset{\|}{C}}-H$



$R-\overset{O}{\overset{\|}{C}}-R$

Both have carbonyl groups $-\overset{O}{\overset{\|}{C}}-$

معلومة



$R-\overset{O}{\overset{\|}{C}}-H \xrightarrow{\text{oxi agent}} R-\overset{O}{\overset{\|}{C}}-OH$

$R-\overset{O}{\overset{\|}{C}}-R \rightarrow X$

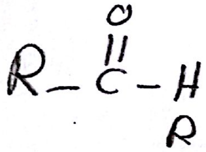
No reactio

للتميز بينهم ممكن استخدام Oxidizing agent

معلومة ##

2,4-Dinitrophenyl hydrazine is commonly used to distinguish Carbonyl Compounds from non-Carb

Derivatives of Both aldehydes and Ketones :-



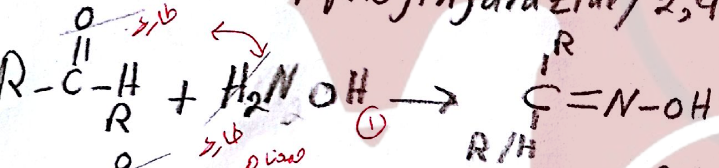
$R-\overset{O}{\overset{\|}{C}}-\underset{R}{H}$

يعني انت جم تعامله الالدهيد كيتون

ج كترمن

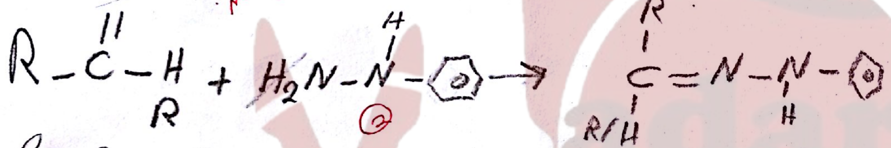
hydroxylamine/pheylhydrazine/2,4-dinitro phenyl hydrazine/semicarbazide

① ② ③ ④



$R-\overset{O}{\overset{\|}{C}}-\underset{R}{H} + H_2NOH \longrightarrow \overset{R}{\underset{R/H}{C}}=N-OH$ ①

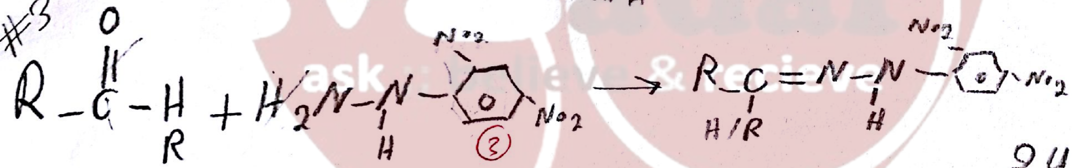
oxime



$R-\overset{O}{\overset{\|}{C}}-\underset{R}{H} + H_2N-\overset{H}{N}-C_6H_5 \longrightarrow \overset{R}{\underset{R/H}{C}}=N-\underset{H}{N}-C_6H_5$ ②

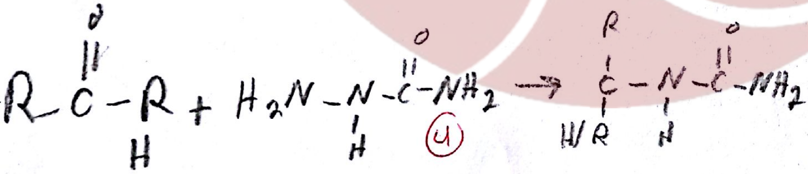
phenylhydrazone.

#3



$R-\overset{O}{\overset{\|}{C}}-\underset{R}{H} + H_2N-\underset{H}{N}-C_6H_3(NO_2)_2 \longrightarrow R-\underset{H/R}{C}=N-\underset{H}{N}-C_6H_3(NO_2)_2$ ③

2,4 Dinitrophenyl hydrazone.



$R-\overset{O}{\overset{\|}{C}}-\underset{H}{R} + H_2N-\underset{H}{N}-\overset{O}{\overset{\|}{C}}-NH_2 \rightarrow \overset{R}{\underset{H/R}{C}}-\underset{H}{N}-\overset{O}{\overset{\|}{C}}-NH_2$ ④

SemicarbaZones

هون مش مطلوب أحفظ ولكن ضروري تعرف مين بطلع مين ؟ أي Compound بيعطي أي Derivative

"مش حفظ المعادلة."

Tests:- ① 2,4 Dinitrophenyl hydrazine

Reagent : ↗ نقاش

Equation :

3# نقاش

Observation:- Orange precipitate

①② Tollen's Test Reagent $[Ag(NH_3)_2]^+OH^-$

بوكس أخضر

Equation $R-\overset{O}{\overset{\|}{C}}-H + 2[Ag(NH_3)_2]^+OH^- \rightarrow R-\overset{O}{\overset{\|}{C}}-\bar{O}\,NH_4^+$
$+ 2Ag + 3NH_3$
$+ H_2O$.

observation:- Formation of silver mirror only for aldehydes

③ Fehling's and Benedicts Reagent $[Cu\ Complex]^{+2}$

$R-\overset{O}{\overset{\|}{C}}-H + 2[Cu\ Complex]^{+2} \rightarrow R-\overset{O}{\overset{\|}{C}}-\bar{O} + Cu_2O$ بوكس أحمر

راسب أحمر

Observation :- Cu_2O راسب أحمر

④ Iodo Form test reagent I_2/OH^- بوكس أخضر

Equation $R-\overset{O}{\overset{\|}{C}}-CH_3 + I_2 + OH^- \rightarrow R-\overset{O}{\overset{\|}{C}}-\bar{O} + CHI_3$

راسب أصفر

Observation production of Iodoform CHI_3. راسب أصفر

الألدهايد مابتفاعل

هاد Test بتشتغل فقط للي compound يحوي

$CH_3-\overset{O}{\overset{\|}{C}}---$

$CH_3-\underset{H}{\underset{|}{\overset{OH}{\overset{|}{C}}}}---$

The End

:D :)))