تفريغ لاب ميديسنال

المحاضرة: Introduction
(melting Point, Recrystallization, Percentage yeild)

الصيدلاني/ـة: سارة جابر

Soul Academy

لجان الدُّفعات

MELTING POINT MEASUREMENT

M.P

9 — 16:35 min

Ali Salama

أول 8min بالفيديو الدكتور حكى عن

الـ Safety Rules والأدوات بشكل سريع

وأكد انه كل لاب لازم يكون معنا lab coat + face mask 🥼

🥽 + eye glasses

Save this Date

$\boxed{\text{9-Oct-2025}}$

Definition

لكل مادة درجة معينة

- The melting point is the specific temperature at which a solid turns into a liquid. At this stage, the intermolecular forces holding the crystal lattice together are broken, allowing the particles to move freely. The melting point of a substance depends largely on the type (1) and strength (2) of the forces within its structure, which explains why different molecules have different melting points.

كيف بتتحول؟

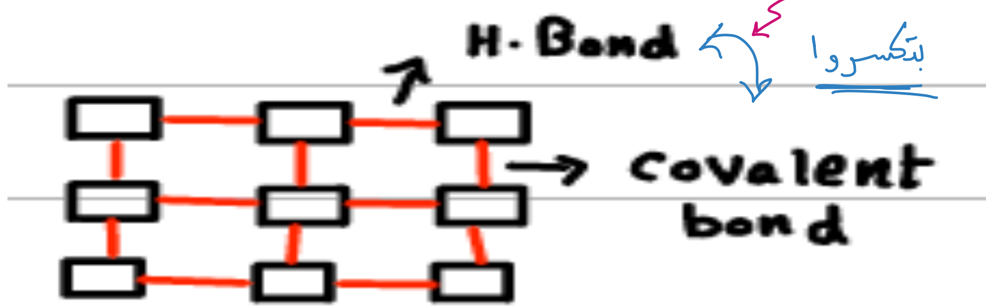
الروابط بين المواد بتتكسر بعد تعرضها للحرارة

لشو الي بحددها لكل مادة؟

الموضوع الي بخرب الـ Structure وبحولها للحالة السائلة

حرارة

بتكسروا



[H-Bond < Covalent] ← بحتاج حرارة اكبر لأكسر الـ Covalent ← فقدرة اني اعمل melting للمادة بتقل والـ M.P بتكون اعلى

على الجهاز بنقيس درجتين حرارة

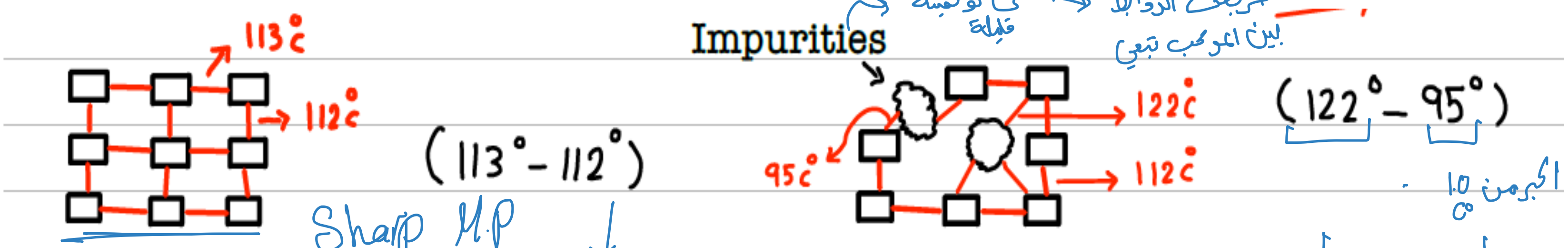
Characteristics of Pure Compounds

بتكون الجزيئات مرتبة

- A pure crystalline organic compound usually exhibits a sharp and narrow melting range, typically within 0.5–1.0 °C. When impurities are present, even in very small amounts, they disturb the lattice structure. This results in a lower melting point and a broader temperature range during melting. Therefore, impurities directly affect how precise and sharp the melting process appears.

حتى لو كمية قليلة

خربطت الروابط بين المركب تبعي



Crystalline

(113° – 112°)

Sharp M.P

0.5–1.0 C°

ال Range بينهم

(122° – 95°)

اكبر من 1.0 C°

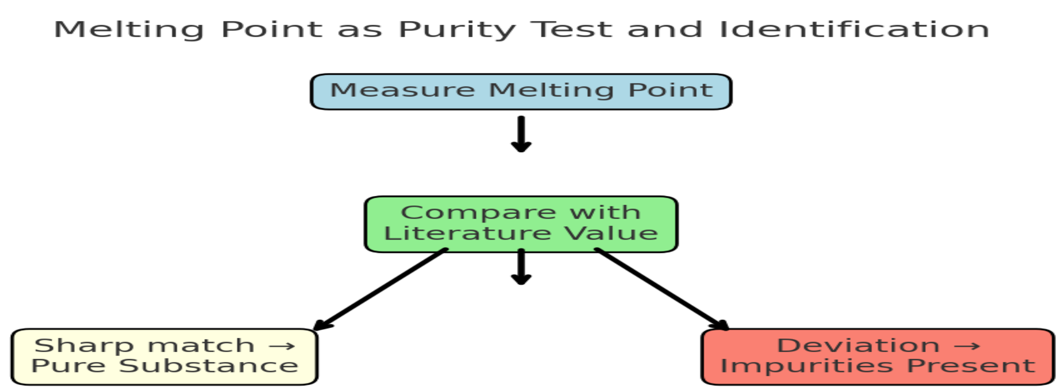
معناته المركب مش Pure 100%

لأنه عني Broadment بال Range

Importance of Measuring Melting Point

- Measuring the melting point is not only a way to identify compounds but also a practical test of purity. For researchers and chemists, a sharp melting point close to the literature value indicates a pure substance, while deviations suggest contamination or the presence of other compounds. Thus, it serves as both a quality control tool and a method of confirming identity.

لما يعطيني قيمة خطأ؟ يعرف المادة ملوثة أو غير نقية



لازم اعمل Purification
وحدة من الخطوات هي Recrystallization

Measurement Method

1

- To measure the melting point, a small quantity of powdered sample (about 3 mm) is packed into a thin-walled capillary tube that is sealed at one end. 2 This tube is attached to a thermometer and placed into a heating bath. The bath is heated slowly, and the 3 observer records two key temperatures: the a) point where the first drop of liquid appears, and the point where the entire b) sample has melted.

[a-b]
Range
0.5-1.0 C° → Pure ✓

Sample Preparation and Accuracy

- Accurate melting point measurement requires careful preparation. The sample should always be finely crushed to increase its surface area, allowing heat to penetrate evenly. Thin-walled tubes are preferred since they conduct heat efficiently and avoid delays that may broaden the melting range. Finally, a fresh capillary should be used each time, as heating can cause decomposition and alter the results.

why ①

Why

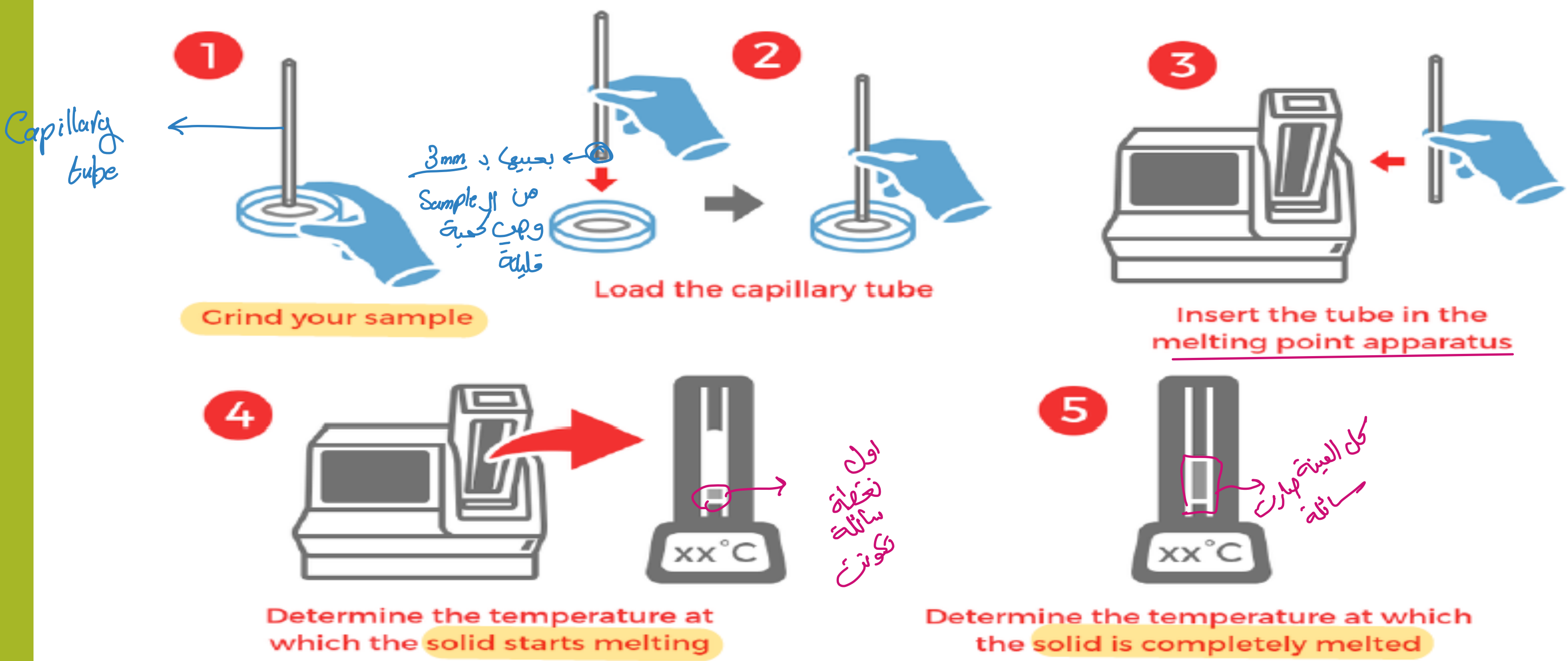
② عشان اقدر ادخل ال Sample بال capillary tube

① ال Sample لازم تكون مطحونة صغير واعبيها بكمية قليلة

② ال wall تبع ال Capillary لازم يكون نحيف

③ كل مرة لازم استخدم Capillary جديد

Measurement Method



Conclusion

- The melting point is a reliable fingerprint for identifying and testing the purity of organic solids. A sharp melting point indicates a pure compound, while impurities cause depression and broadening of the range. By following proper preparation and measurement techniques, scientists can ensure accurate and reproducible results, making melting point analysis one of the most useful tools in organic chemistry.

RECRYSTALLIZATION

16:36 –23:04 min Ali Salama

Definition

- Recrystallization is the most widely used method for purifying solid organic compounds. In this technique, an impure solid is dissolved in a solvent and then allowed to slowly crystallize as the solution cools. The impurities remain dissolved in the solvent while the compound forms a pure, ordered crystal lattice.

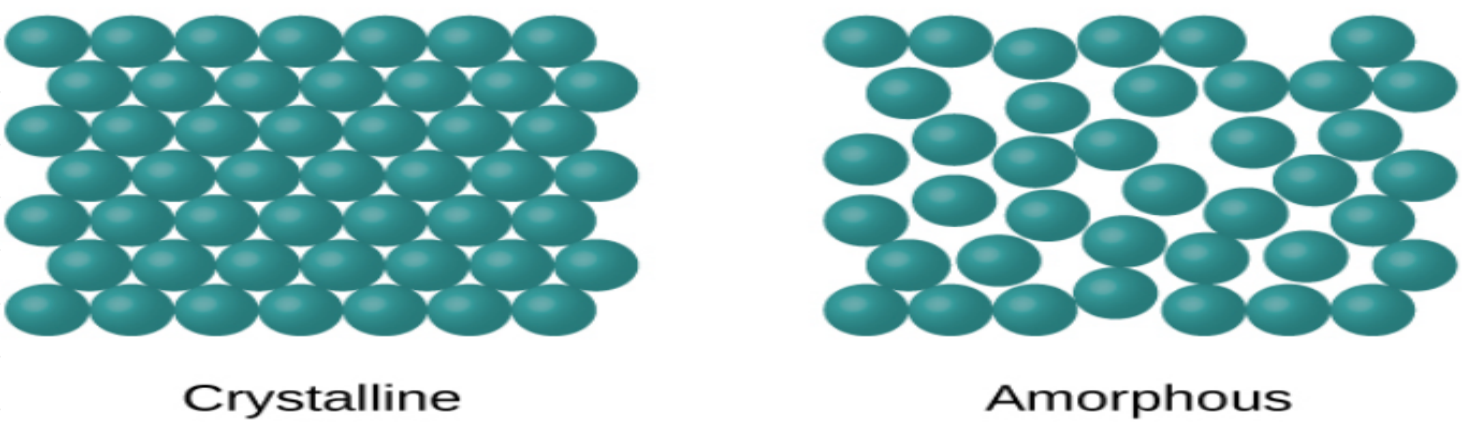
Crystallization vs Precipitation

مختلفين

Precipitation :- مواد صلبة بترسب بالقاع

- Crystallization differs from precipitation. Crystallization involves a (1) slow, (2) selective formation of an ordered crystal structure that excludes impurities, producing a pure compound. In contrast, precipitation happens (1) rapidly and (2) often produces an amorphous solid with many trapped impurities inside the lattice.

المادة الي بختار افصلها عن الباقي



الجزيئات مرتبة

جزيئات عشوائية وغير مرتبة

Principle of Recrystallization

يعتمد على

- The process depends on solubility changes with temperature. Most solids are more soluble in hot solvent and less soluble at lower temperatures. By heating the solvent, the impure solid dissolves completely; as the solution cools, the desired compound crystallizes while impurities remain in solution.

* ادرسوا الرسمة بالأول *

Steps of Recrystallization

كيف ؟!

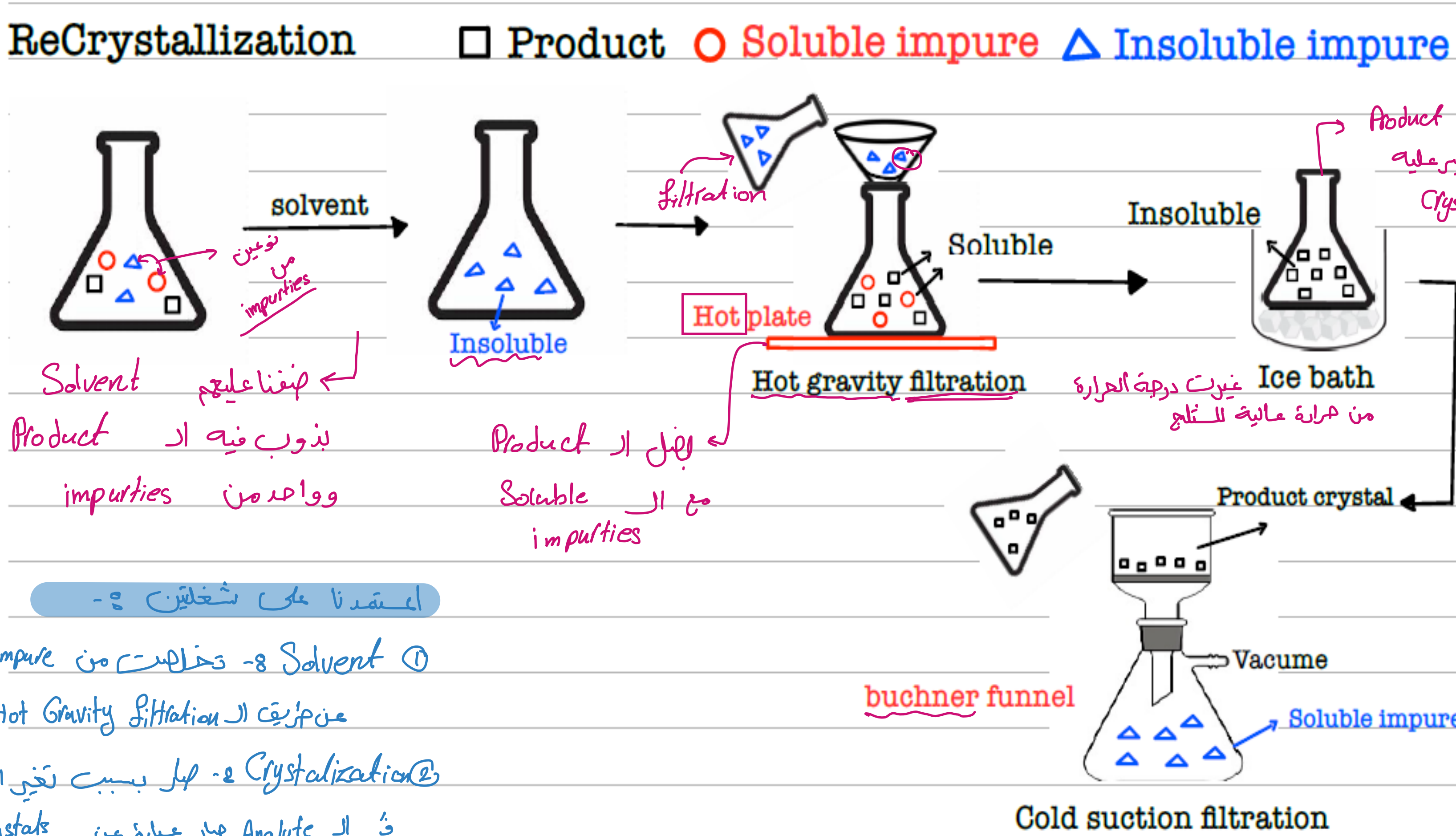
1. Choose a suitable solvent. موجود تحت

ليه؟

لو ضفت كمية كبيرة ممكن ال insoluble impure ممكن جزء منها يذوب وهون راح احصل على منتج غير نقي

2. Dissolve the impure solid in a [minimal amount] of hot solvent.
3. Filter out insoluble impurities.
4. $\boxed{\text{Slowly cool}}$ to allow crystals to form.
5. Filter and collect the purified crystals.
6. Wash the crystals to remove any residual impurities.

Steps of Recrystallization



اعتمدنا على شغلتين :-

① Solvent :- تخلصت من Insoluble impure

عن طريق ال Hot Gravity filtration

② Crystalization :- صار بسبب تغير الحرارة

فَ ال Analyte صار عبارة عن Crystals وقدرت

افصله بـ Buchner funnel

Choosing the Right Solvent

- The compound should be highly soluble in hot solvent but sparingly soluble at room temperature.
- Impurities should either (1 remain dissolved at room temperature or (2 be insoluble in hot solvent.
- The solvent must not react with the compound.
- The solvent should be volatile so it can be easily removed after crystallization.

Conclusion

- Recrystallization is a reliable purification method for solid compounds. By exploiting differences in solubility, it separates the desired product from impurities. Careful solvent selection and slow crystallization are essential for obtaining pure, crystalline compounds suitable for further analysis or use.

23:08 – 30:00 min

المهم يحل ال Report

Percent Yields Calculations

Many synthesis require more than one reaction, with each reaction yielding an isolated compound before the final product is reached. Each individual step in the sequence has a percent yield, and the total synthesis has an **overall yield** calculated from the steps yields

- **Limiting reagent** : the reagent that is present in the smallest equivalent stoichiometric amount and limits how much product will be produced. It determines the maximum amount of product(theoretical yield) that will be formed.

وزن الواقعي من الميزان

مهمين وراح يمروا معنا بكل التجارب

- **Percent yield** = $\frac{\text{actual mass yield}}{\text{theoretical yield}}$ *100%

وزن متوقع من الحسابات

- **Overall yield** = $\frac{\text{\%yield for step1}}{100} * \frac{\text{\%yield for step2}}{100} * \frac{\text{\%yield for step3}}{100}$

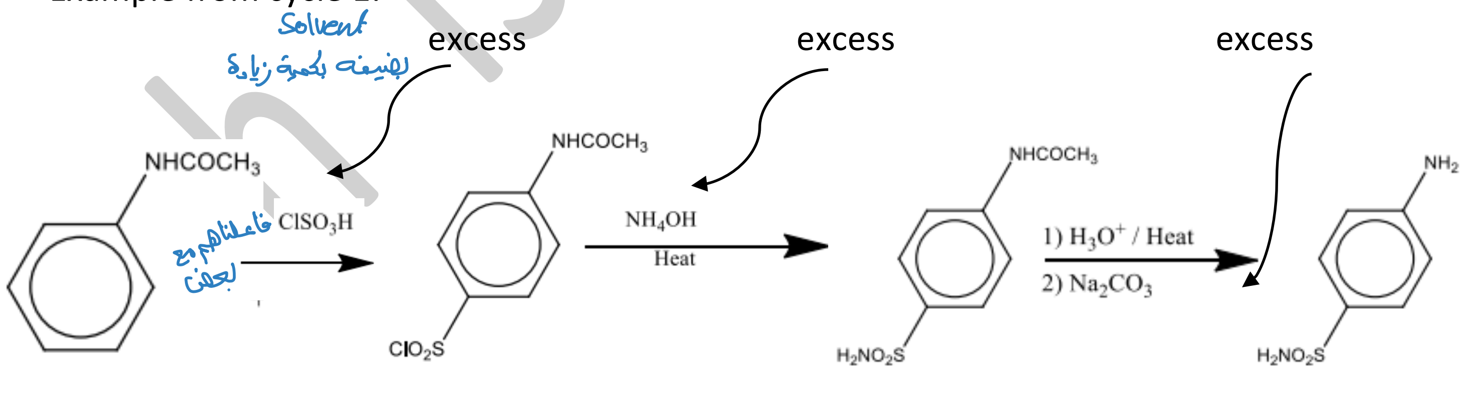
عشان الدقة بنحسب yeild % لكل خطوة لبعدين بنحسب ال overall

عنا ٣ حالات بنحسب فيها yield

- Each individual step has a relatively high efficiency, but the overall efficiency is low. Each subsequent reaction further reduces the actual amount of product that is formed from the initial starting material.

① عنا Certien amount of solid + excess Solvent(liq)

Example from cycle 1:



Acetanilide p-acetaminobenzene sulfonylchloride p-acetaminobenzene sulfonamide sulfanilamide

1. You have 3 steps for this reaction, so you have to find percent yield for each step then multiply them to find the overall yield for the synthesis.

2. In this cycle the other reactants were used in excess so your main reactant will be the rate limiting reactant.

For Step 1:

لـ هكاكي اضيف وزن معين منه + انا بعرف الـ M.W = بحسب المولات

Acetanilide + chlorosulfonic acid $\longrightarrow$ p-acetaminobenzene sulfonylchloride

Product

فعواكيد مش الـ * Rate limiting step *

	Acetanilide	chlorosulfonic acid
*Amount used	15 g	excess
*No. of moles (wt/m.wt)	(15/135.17)	excess(no need to calculate)
	$\boxed{(0.111\ \text{moles})}$	excess

بالتالي مابقدر احسب المولات تبعته

So rate limiting reactant is acetanilide

*كمية اقل، هو الي بحدد كمية التفاعل ⇐ يعني 100% عدد مولاته = الـ Product مولات

No. of moles of product =No. of moles of acetanilide (ratio 1:1) =0.111 moles

Theoretical yield = 0.111*233.67(m.wt of product) = 25.93 g

عدد مولات Product * M.W

- % yield = $\dfrac{\text{actual mass yield}}{\text{theoretical yield}}$ *100%

Let's assume that when you weighed your product after drying in the next lab and was 16 g (this 16 g is your actual yield)

actual/theoretical

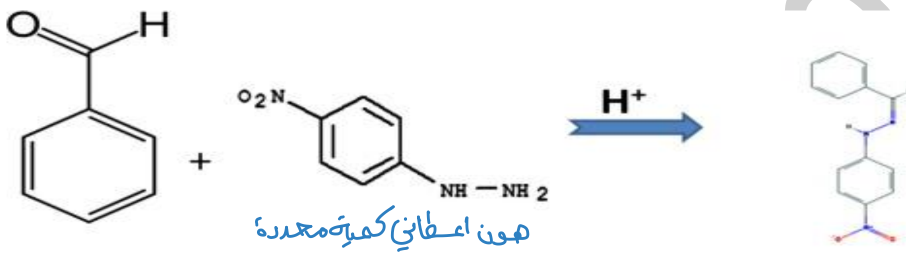
So % yield for this step is equal to (16/25.93) *100% = 61.7%

** Make the same calculations for the second two steps, then multiply them to find the $\boxed{\text{overall yield.}}$

tow liquids

Example 2 (if the reactant not in excess)

Calculate the % yield if 2 ml of benzaldehyde(density =1.043, m.wt =106) were reacted with 4 ml of phenylhydrazine ((density =1.095, m.wt=108) in order to synthesize phenylhydrazone (m.wt=196)



هون اعطاني كمية محددة

Benzaldehyde + p-nitrophenylhydrazine

Benzaldehyde 4-nitro phenyl hydrazone

مش excess

فبقدر احسب ال moles

1. Convert volumes to no. of moles

Density

- weight (vol*density)	2*1.043 =2.080	4*1.095 =4.38
- #moles (wt/m.wt)	2.080/106 =0.019	4.38/108 =0.04

Ratios to find rate limiting reactant: الخطوة المهمة

1 mol of benz. → 1 mol of hydrazone
0.019 mole → ? = 0.019moles of hydrazine(product)

مولات اقل → Limiting Reactant.

1 mol of hydrazine → 1 mol of hydrazone
0.04 mole → ? = 0.04moles of hydrazine(product)

So benz. Is the rate limiting reactant(lower no. of moles of product). so no. of moles of product =0.019 moles

Theoretical yield = 0.019 *196(m.wt of product)= 3.72g

Assume that you weighed your product and was equal to 2g

So

%yield = 2/3.72 *100% =53.76%

* مثال ضافه الدكتور

① نوازن المعادلة

$2Al + 3Br_2 \rightarrow 2AlBr_3$

%yield ??

$Al = 1.62g$, $M.wt = 27 g/mol$ | $Br_2 = 6.392 gm$, $M.wt = 159.8 g/mol$ | $AlBr_3$ $wt = ??$, $M.wt = 293.7 g/mol$ | Actual wt = 7gm

② بوجد عدد المولات ووجد Limiting كيف؟

#mol Al = wt / M.wt

$1.62/27 = 0.06 mol$

#mol Br_2 = wt / M.wt

$6.392/159.8 = 0.04$

$2Al$	$3Br_2$
$\frac{0.06}{2}$	$\frac{0.04}{3}$
$= 0.03$	$> = 0.0133$

Factor ← 2

Limiting reactant

هاي الخطوه ← بتقسم عدد المولات على Factor

خطوه لتحديد LR

(الرقم يلي جنب العنصر بالمعادلة)

الأقل هو الـ Rate Limiting step

So $3Br_2 \rightarrow 2AlBr_3$

$0.04 \rightarrow X$

$X = 0.0267 mol AlBr_3$

wt = #mol × M.wt

$0.0267 \times 293.7 = 7.8339 gm$

① المعادلة لازم تكون موزونة

$2Al + 3Br_2 \longrightarrow 2AlBr_3$

بعد الموازنة بنلاحظ انه الـ Ratio مش 1:1

القوانين

$$\text{The number of moles (n)} = \frac{\text{weight of substance(w)}}{\text{molar mass of substance(M)}}$$

$$\text{Density} = \frac{\text{mass}}{\text{volume}}$$

$$\text{Percent yield} = \frac{\text{Actual Yield}}{\text{Theoretical Yield}} \times 100\%$$

$$\text{Overall yield} = \frac{\text{\%yield for step1}}{100} * \frac{\text{\%yield for step2}}{100} * \frac{\text{\%yield for step3}}{100}$$

آخر اشي : تغير توزيع العلامات

Grade Distribution		
Assessment	**Grade**	**Date**
1. Quiz	15% ~~10%~~	weekly كل اسبوعين في كويز
2. Midterm exam	30%	To be arranged
3. Report	10%	weekly
4. Evaluation	5% ~~10%~~	weekly
5. Final Examination	40%	To be arranged

كل عملية تصنيع
دوا الها Report
بجمع كل الخطوات

التجربة فرغتها بتاريخ 9-Oct-25

بيوم وقف الحرب على غزة

دعائكم الهم ربنا يجبر قلوبهم ويرحم الشهداء
ويتقبلهم