Why we use →

- silicone oil → expands on heating → thicke apparatus
- pure, known compounds to melt → to determine the accuracy of the thermometer and calibrate it

- boiling chips → to prevent the liquid from bumping due to superheating

- Grease → To reduce the friction and prevent the contamination
  To ensure a good seal

indentations → increasing surface area → its for fractional distillation

decolorizing carbon → to remove the colored impurity from solution before cooling.

Zn dust → catalyst.

drying agent → to remove traces of water.
↳ $MgSO_4$, Na_2SO_4, $CaCl_2$, NaCl.

Saturated solution of sodium chloride → (brine) increases the ionic strength of the water layer which helps force the organic material into the organic layer and no emulsion occur

NaOH
10% ~~Sulfuric acid~~ → removing the sulfuric acid.

Aluminum foil → for efficient heating

cotton plug → to make sure that the steam remain in the tube.

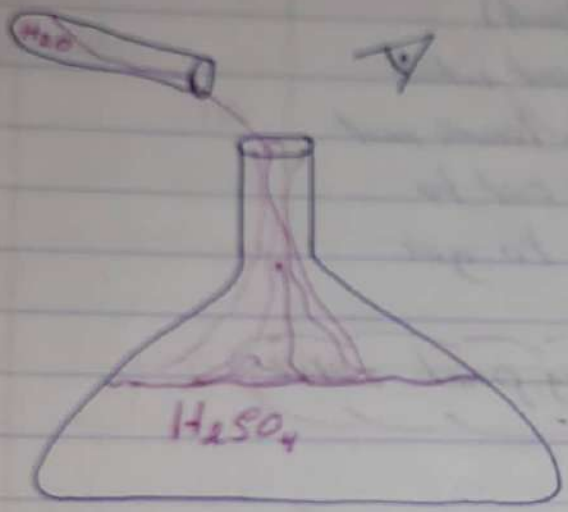
H_2SO_4 → protonates the hydroxyl group

Experiment "1"

Melting point and calibration of thermometer

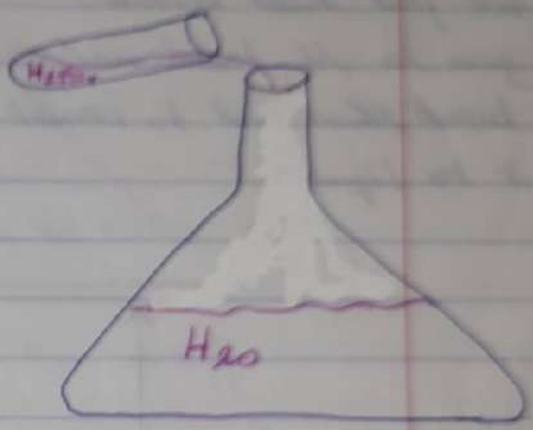
$H_2O + H_2SO_4$ → We have 2 methods to put $H_2O + H_2SO_4$ but only one we use because it's more safe

First Method



We don't put H_2O on the H_2SO_4 because the H_2SO_4 is hydrophilic (prefer H_2O) so it will attract to the H_2O and the droplets will go out the flask to our eyes Danger Method

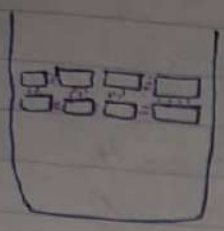
Second Method



Safe Method

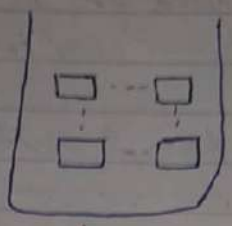
Objectives → First Melting point

Learning outcomes →
1. Measure and calibrate the thermometer.
2. Find Melting point for known materials.
3. Effect of the impurities.
4. Melting point for different unknown.
5. Mix
6. Eutectic composition.



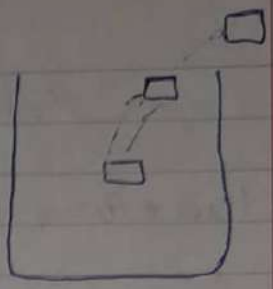
Solid

has a strong intermolecular forces (Bonds).
we put heat which goes to the bonds and breaks them out to convert it to Liquids.



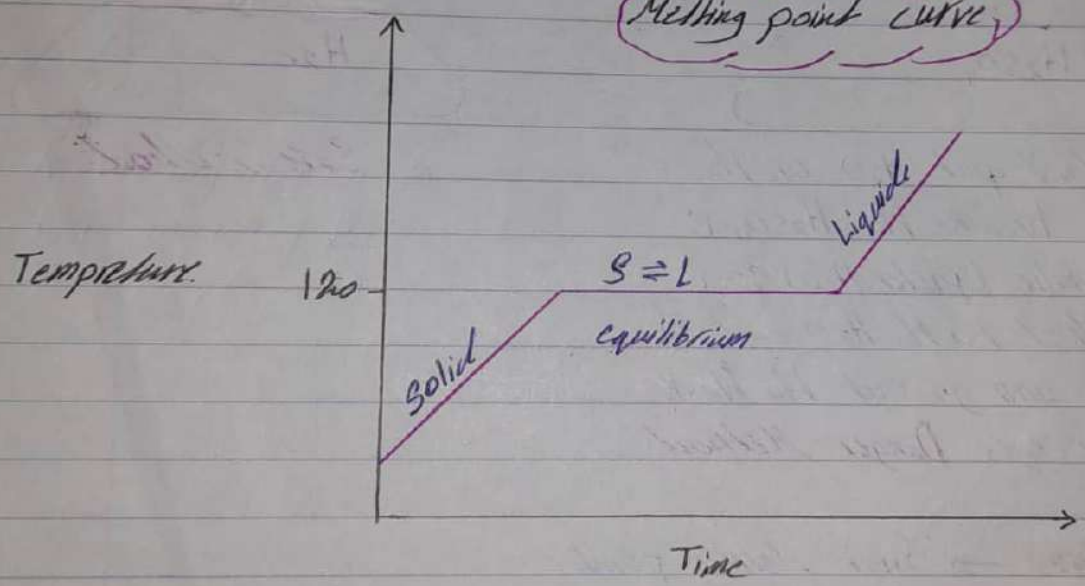
Liquids

Less intermolecular forces (Bonds)
we put heat which goes to the bonds and breaks them out to convert it to gas.



Gas

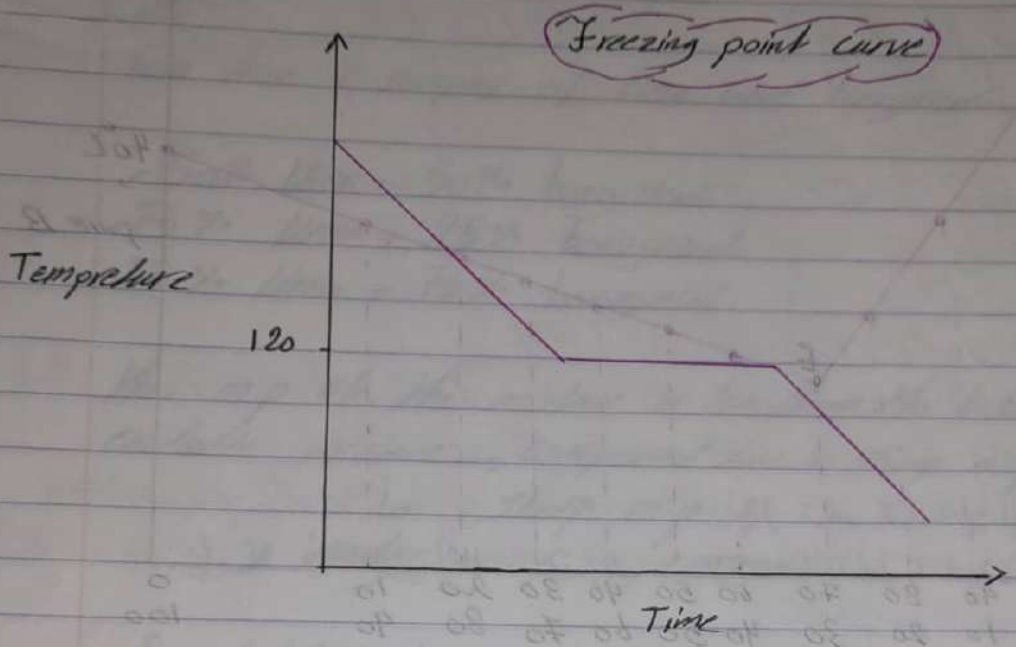
Melting point curve



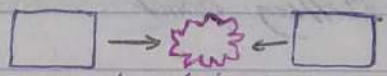
Why the temprecture don't increase in S ⇌ L stage?!
Because the temprecture is being used in breaking down the bonds not to the thermometer. & after breaking all the bonds, the temprecture of thermometer will increase ✓.

Freezing point and Melting point is identical its only different in the energy.

Freezing point → energy taken
Melting point → energy supply

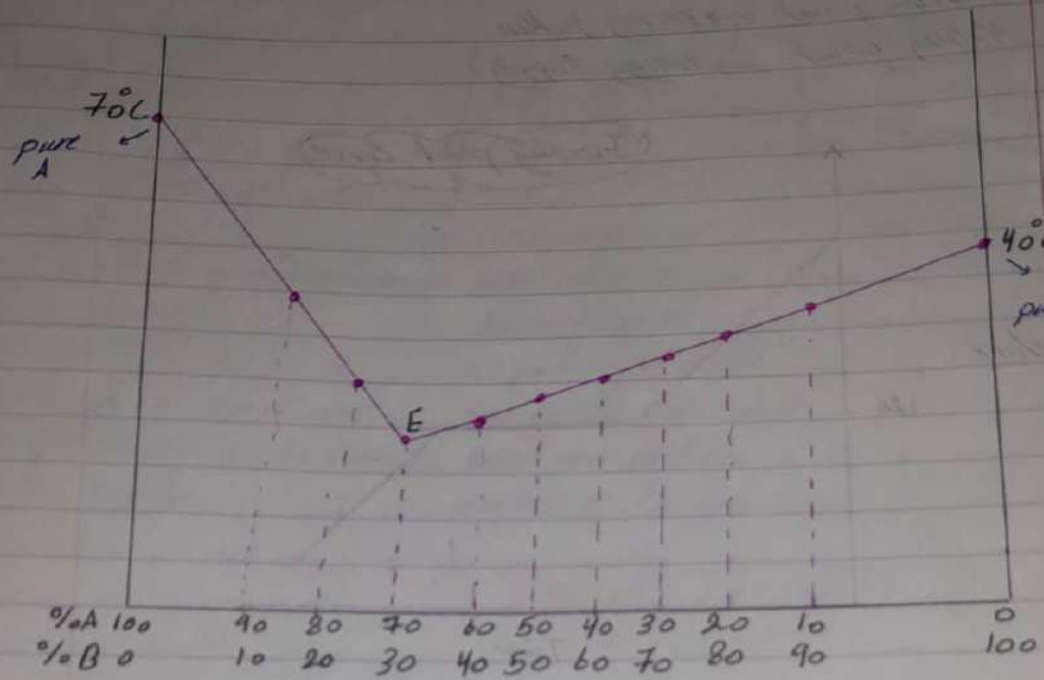


When we have impurities the freezing point decreases to help it become closer → because freezing and melting are identical. the melting will also decrease.



The impurity will prevent the substance molecules from coming closer which let us lower the temprehure to make it come closer barely so the freezing point will decrease.

Discussion for the curve below ?!
Every dot present a melting point which has an equilibrium ratio in solid - liquid phase.
X-axis presents the percentage of every solid is needed for each melting point.



A is impurity for B
B is impurity for A
E → is the eutectic point which is the percentage of solidus mixture for the lowest Melting point.

why we don't determine Freezing point by measuring it?
because we are afraid that it will do super cooling so we miss the Freezing point

the substance have to be a dry solid so we can degrade it

We use melting point in

- examine the impurities
- test the unknown material
- test the new compound

presence of impurities → widening range → not sharp melting point
→ decrease melting point.

we have 3 samples of Urea and benzymide.

50% Urea + 50% benzymide
75% Urea + 25% benzymide
25% Urea + 75% benzymide.

the m.p of the mixture is less than the both substances
eutectic mixture → compound has a sharp m.p
لما يكون عندها مادة نقية إلها sharp m.p
بس لما يتلف الـ compound مع يكون الثاني impurity للأول.

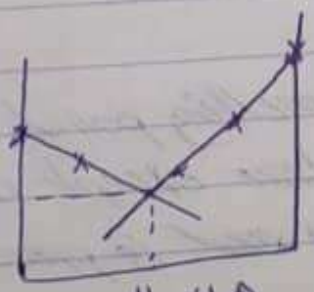
Ex → (65% benzymide + 35% Urea) → eutectic point
any composition different from it cause one substance to be impurity to the other.

higher percentage إلي ما بدوب هو إلي إله

التسخين يكون 1 degree/min
start melting ↓ ↓ total melting.
$\frac{63 + 55}{2}$ = average

التسخين السريع بعمل depression و wider range

هيك الرسم:



Biol 243 ... Chem 241

Questions;

①

a) Heating the sample too quickly (lag between real temp and what thermometer reads).
- Using an uncalibrated thermometer
- Measuring a wet sample
- Measuring a sample with impurity

b) - Using too large a sample size
- packing a sample too loosely in the calibrating tube
- Using an uncalibrated thermometer
- Measuring a sample with significant quantities of an impurity with much higher m.p than the sample itself.

c) - Measuring an impure sample
- Measuring a wet sample
- Heating the sample too quickly
- Using too large a sample size.
- Measuring a sample with large crystals.

True / false ⇒

- An impurity always lower the melting point of an organic compound (F, Usually true except if the impurity has a significantly higher mp and is present in large quantities).

- A sharp m.p for crystalline organic ~~compound~~ substance always indicated a pure signal compound (F, Usually true with the exception of eutectic mixture).

Phar 243 analog 2
Phys 121

- If the addition of a sample of compound A to compound X does not lower the m.p of X, X must be identical to A (T)

- If the addition of a sample of compound A to compound X does lower the m.p of X, X ~~must be~~ and A cannot be identical (T)

mixed melting point → the temp of a mixture of 2 components that in the case of 2 different substances is usually lower than that of either component

Q.3 →

2. Briefly define the following terms:

a. vapor pressure as applied to melting
b. melting point or melting-point range
c. mixture or mixed melting point
d. eutectic point
e. eutectic mixture

3. Describe on a molecular level the difference between the two physical changes "melting" and "dissolving."

4. Answer the following questions about melting points.

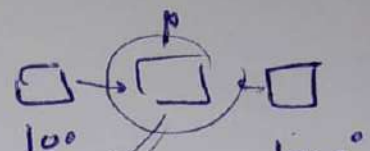
a. Why is the melting "point" of a substance actually a melting "range" and therefore should never be recorded as a single temperature? Because the actual m.p of a substance is measured starting from the temperature at which the first droplet of liquid can be detected to the temperature where all solid has completely changed to liquid which gives a range so there is no m.p

b. In theory, does a melting "point" exist? Explain your answer.

5. Explain how a eutectic mixture could be mistaken for a pure substance, and comment on whether encountering a eutectic mixture would be a frequent or infrequent occurrence.

because both have a sharp m.p

↓ uncommon encounter because m.p mixture higher than mp range



6. Compound A and compound B have approximately the same melting point. State two ways in which a mixed melting point of these two compounds would be different from the melting point of either pure A or pure B. The m.p of a combination of A & B is normally lower and the m.p range is wider than either each material alone because each one will act as an impurity

7. Filter paper is usually a poor material on which to powder a solid sample before introducing it into a capillary melting-point tube because small particles of paper may end up in the tube along with the sample. Why is this undesirable, and how might the presence of paper in the sample make the melting-point determination difficult? an extension of 2 is that the cellulose could affect the m.p due to the mixed melting point.

8. Some solids sublime before they melt, making a determination of a melting point impossible using a standard melting-point capillary tube. How could you modify your capillary tube to obtain a melting point for such a compound?

9. Some solids, in particular many amino acids, decompose upon melting. These compounds are often reported in the literature with the term "dec" following their melting-point range.

   a. How might the melting process appear different for this type of compound?

   b. Look up and record the melting point and structure for a compound that decomposes upon melting. Use a chemical handbook or a chemical catalog as the source of this information.

0. Criticize the following statements by indicating whether each is *true* or *false*, and if false, explain

a capillary melting-point tube because small particles of paper may end up in the tube along with the sample. Why is this undesirable, and how might the presence of paper in the sample make the melting-point determination difficult? an extension of 2 is that the cellulose could affect the m.p due to the mixed melting point.

8. Some solids sublime before they melt, making a determination of a melting point impossible using a standard melting-point capillary tube. How could you modify your capillary tube to obtain a melting point for such a compound?

9. Some solids, in particular many amino acids, decompose upon melting. These compounds are often reported in the literature with the term "dec" following their melting-point range.

   a. How might the melting process appear different for this type of compound?

   b. Look up and record the melting point and structure for a compound that decomposes upon melting. Use a chemical handbook or a chemical catalog as the source of this information.

10. Criticize the following statements by indicating whether each is *true* or *false*, and if false, explain why.

   a. An impurity always lowers the melting point of an organic compound. F (except if impurity has a higher m.p and is present in high quantities).

   b. A sharp melting point for a crystalline organic substance always indicates a pure single compound. F (Usually T with exception of eutectic mixture)

   c. If the addition of a sample of compound *A* to compound *X* does not lower the melting point of *X*, *X* must be identical to *A*. T

   d. If the addition of a sample of compound *A* lowers the melting point of compound *X*, *X* and *A* cannot be identical. T

11. The melting points of pure benzoic acid and pure 2-naphthol are 122.5 °C and 123 °C, respectively. Given a pure sample that is known to be either pure benzoic acid or 2-naphthol, describe a procedure you might use to determine the identity of the sample.

12. A student used the Thiele melting-point technique to determine the melting point of an unknown and reported it to be 182 °C. Is this value believable? Explain why or why not.

prepare 2 new samples (A and B), A with a mix of pure sample and B with a mix of pure sample and a sample

why.

a. An impurity always lowers the melting point of an organic compound. F (except if impurity has a higher m.p and is present in high quantities).

b. A sharp melting point for a crystalline organic substance always indicates a pure single compound. F (Usually T with exception of eutectic mixture)

c. If the addition of a sample of compound *A* to compound *X* does not lower the melting point of *X*, *X* must be identical to *A*. T

d. If the addition of a sample of compound *A* lowers the melting point of compound *X*, *X* and *A* cannot be identical. T

11. The melting points of pure benzoic acid and pure 2-naphthol are 122.5 °C and 123 °C, respectively. Given a pure sample that is known to be either pure benzoic acid or 2-naphthol, describe a procedure you might use to determine the identity of the sample.

12. A student used the Thiele melting-point technique to determine the melting point of an unknown and reported it to be 182 °C. Is this value believable? Explain why or why not.

11- prepare 2 new samples (A and B), A with a mix of pure sample and sample of Benzoic acid, and B a mix of pure sample and a sample of 2-naphtol. Measure both m.p. if the melting point of the sample is broad and depressed. then that sample (A or B) is likely a mixture. If the melting point of the sample is sharp and the same temp then the sample is likely a pure sample.

Phar 242

Experiment "2"
Boiling point and distillation methods

Boiling point under reduce pressure so it didn't reach decomposition → Low Boiling point.

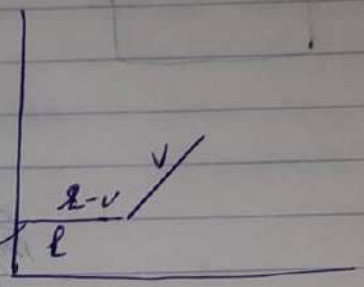
* purity → Distillation temperature

For non-volitile

volitile → Low boiling point in room temp

↓

Frictional distillation



The More volitile substance, it will be distilled first because it has a low b.p and can easily be converted from liquids to gas.

HETP

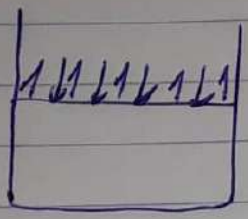
كل ما كانت ال ~~distillation~~ أقل، كل ما كان ال distillation أفضل وال exchange أعلى وبتغير تركيب ال Liquid & بخار

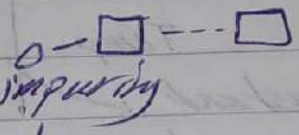
إذا ال sample size قليل ما بنفصل يكون طوله كبير

~~Ho~~ More volitile gives percentage of pressure higher than the non-volitile

A water bath is used to distil acetone (low boiling and flammable liquid)

multi gram → simple distillation.





تضعف الرابطة
فنقلل الـ boiling point.

glass to see if it will burn. Record the data obtained by each of your partners and compare the three sets of data. Collect the first fraction in the waste bottle labeled as waste acetone and get rid of the remaining two fractions in the sink at the end of your bench.

QUESTIONS

1. Summarize the data for the distillation of the acetone-water mixture obtained by members of your group. Explain the differences in the results.

2. Why does not all of the liquid in the distilling flask vaporize at once when the boiling point is reached? additional heat must be supplied for a phase change to occur.

3. Why should a distilling flask not be filled much more than half full? because when the liquid boils, that headspace is needed because the drops of the boiling liquids are propelled out and they could contaminate the condenser and get into the distillate.

4. What is the disadvantage of using a distilling flask whose capacity is four or five times or more the volume of the liquid being distilled? ① lower the yield of distillation. ② reduce the rate of distillation and more time is necessary

5. Draw a general temperature-composition diagram, but with a boiling point of 56.6° for component *x* and of 100° for component *y*. This diagram now represents, at least approximately, the acetone-water system. At approximately what temperature will a 1:1 molar mixture of acetone and water begin to distill? A 3:1 molar mixture? A 1:3 molar mixture? What is the approximate composition of an acetone-water mixture which begins to distill at 70°? At 80°?

6. If liquids *x* and y both have a boiling point of 160° and do not form an azeotrope, what will be the boiling point of a mixture of *x* and y? In a general sense, how does this fact limit the value of boiling points as a criterion for determining the purity of liquids?

7. If the thermometer bulb is not kept moist with condensate during a distillation, will the boiling-point reading tend to be high or low? Explain. low, because vapor is condensing below the position you are measuring

10. Define the following terms:

a. fractional distillation
b. head temperature
c. pot temperature
d. Raoult's law
e. ideal solution
f. mole fraction
g. height equivalent to a theoretical plate (HETP)
h. temperature gradient
i. Dalton's law
j. reflux ratio

11. Specify whether a simple distillation or a fractional distillation would be more suitable for each of the following purifications, and briefly justify your choice.

a. Preparing drinking water from sea water.
b. Separating benzene, bp 80 °C (760 torr), from toluene, bp 111 °C (760 torr).
c. Obtaining gasoline from crude oil.
d. Removing diethyl ether, bp 35 °C (760 torr), from *p*-dichlorobenzene (s), mp 174–175 °C.

12. Sketch and completely label the apparatus required for fractional distillation.

13. Explain why a packed fractional distillation column is more efficient than an unpacked column for separating two closely boiling liquids.

14. If heat is supplied to the stillpot too rapidly, the ability to separate two liquids by fractional distillation may be drastically reduced. In terms of the theory of distillation presented in the discussion, explain why this is so.

15. Explain why the column of a fractional distillation apparatus should be aligned as near to the vertical as possible.

16. Explain the role of the stirbar normally added to a liquid that is to be heated to

13. Explain why a packed fractional distillation column is more effective in separating two closely boiling liquids.

14. If heat is supplied to the stillpot too rapidly, the ability to separate two liquids by fractional distillation may be drastically reduced. In terms of the theory of distillation presented in the discussion, explain why this is so.

15. Explain why the column of a fractional distillation apparatus should be aligned as near to the vertical as possible.

16. Explain the role of the stirbar normally added to a liquid that is to be heated to boiling.

17. The top of the mercury bulb of the thermometer placed at the head of a distillation apparatus should be adjacent to the exit opening to the condenser. Explain the effect on the observed temperature reading if the bulb is placed
(a) below the opening to the condenser or (b) above the opening.

18. Calculate the mole fraction of each compound in a mixture containing 15.0 g of cyclohexane and 5.0 g of toluene.

19. In the fractional distillation of your mixture of cyclohexane and toluene, what can be learned about the efficiency of the separation on the basis of the relative volumes of fractions?

Experiment 4

لازم نفل نفتح الصمام عشان نفك الـ pressure release to the

solubility → concentration unit

When we do extraction and let it settle

وبيه موجود الـ organic وبيه موجود الـ aqueous بعتمدوا على الـ density

Diethyl ether → ما بتفاعل مع المواد بس بتقدر تذوبها
$CH_3CH_2OCH_2CH_3$

لازم يصير واحد واحد عشان كلهم مع بعض

solubility → بتحدد اذا بتذوب بالماء
density → بتحدد موقع المادة ,

or we can use H_2O + ether
$H_2O + CH_2Cl_2$

solubility tests to determine if its aqueous or organic

Originally, all perfumes came from ... tremendous development of organic chemistry, have led to synthetically perfumes and the development of organic chemistry, have led to synthetically perfumes from coal tar or petroleum products. The most expensive perfumes are extracted or steam distilled from fresh flowers (the yield may be as little as a pint oil from a ton of flowers).

Perfumes today consist of a blend of ingredients diluted in alcohol and water to a concentration. Perfumes may have as few as 10 ingredients or as many as 200. The composition is a closely guarded secret by the perfumers.

β-Naphthol → limiting reactant

↓

will be change to β-Naphthoate.

↓

emulsion (liquid).