تفريغ مختبر عضوية

اسم الموضوع: Exp 8 : Caffeine extraction

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رب اشرح لي صدري
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لجان الدفعات

التجربة طويلة شوي

- فيها تجميع من Manual + Slides + Vedio + Report sheet → حريصة الحل والتطبيق حسب الأرقام اللي بتطلع محكم

بِسمِ اللهِ الرَّحمنِ الرَّحيمِ

EXPERIMENT 8: EXTRACTION

A Separation and Isolation Technique

INTRODUCTION

Extraction is the separation of a substance from a mixture by means of a solvent that preferentially dissolves that substance. If the substance is extracted from a solid phase, the process is called solid-liquid extraction, asin the isolation of caffeine from tea leaves by means of hot water. Extraction of a substance from a liquid phase is called liquid-liquid extraction. The most common applications of this latter technique are:

a. The recovery of an organic product from a reaction mixture containing excess unreacted materials and by-products.

b. Isolation of an organic substance from its natural source, such as a plant.

Liquid-Liquid Extraction. This is the most common type of extraction. It involves shaking the liquid mixture with an immiscible solvent which preferentially dissolves the desired compound. On standing, the two immiscible phases (usually organic and aqueous) form two separate layers (upper and lower) that can be separated by means of a separatoryfunnel. The various solutes in the mixture distribute themselves between theorganic and aqueous phases according to their relative solubilities in each solvent. At equilibrium, the ratio of the concentration (C) or solubility (S)of the substance in the organic phase, (C_o or S_o) to that in the aqueous phase(C_w or S_w) is called the distribution coefficient (K_D).

$$K_D = \frac{C_o}{C_w} = \frac{S_o}{S_w}$$

A large distribution coefficient implies that the compound is much more soluble in the organic phase than in the aqueous one and, in this case, a single extraction suffices to remove the desired compound from the mixture. When K_D is small, it means that the compound distributes itself

more evenly in both phases, so that repeated extractions are required to recover such a compound from the aqueous mixture.

In general, it is more efficient to divide the total volume of extracting solvent over several extractions than to use the whole volume in a single extraction.

GENERAL EXPERIMENTAL CONSIDERATIONS

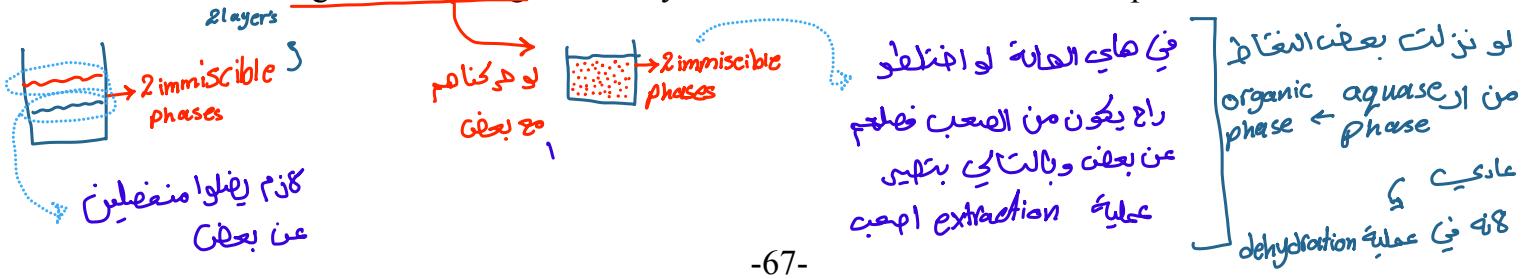
Choice of Solvent. A solvent used for extraction should have the following characteristics:

1. Immiscible with the liquid in which the solute is present.
2. Readily dissolve the solute to be extracted.
3. Extract little or none of the impurities and other compounds present in the mixture.
4. Non-flammable, nontoxic, cheap and easily removable from the solute after extraction (*i.e.*, volatile).

Salting-out. Extraction of organic compounds from aqueous mixtures is usually improved by saturating the aqueous phase with a salt such as NaCl or Na_2CO_3. This phenomenon is called salting-out and has the following effects:

1. Decreases the solubility of organic compounds in the saturated aqueous phase.
2. Decreases the solubility of the organic and aqueous phases in each other, thus improving their separation. This is particularly useful in breaking up emulsions.

Emulsions. In certain cases, the two immiscible phases do not separate cleanly into two distinct layers; instead, they form an emulsion which, once formed, is usually difficult to break. It is therefore advisable to prevent the formation of emulsions during extraction. This is best achieved by avoiding vigorous shaking of the layers whenever an emulsion is expected to form



(*e.g.*, when alkaline aqueous solutions are extracted with chloroform or dichloromethane). If an emulsion still forms one can often break it by:

1. Stirring the emulsified layer gently with a glass rod.
2. Saturating the aqueous layer with a salt.
3. Centrifugation.

Drying Agents. The organic phase often shows turbidity due to the presence of traces of water from the aqueous phase. Anhydrous $CaCl_2$, $MgSO_4$, or Na_2SO_4 may be used as drying agents which absorb the traces of water present in the organic phase. When dry, the organic phase becomes clear.

ACID-BASE EXTRACTION

Mixtures of organic acids and bases are commonly separated by acid-base extraction. Such compounds are converted to their salts by treatment with acid or base. Unlike the original compounds, the corresponding salts are usually soluble in water, thus enabling their transfer from the organic phase to the aqueous layer. After separation of the layers, the organic acid or base is recovered by neutralization of the aqueous layer. Since the acid or base is insoluble in water it precipitates out, and is collected by filtration. A flow diagram for such a separation is shown in Figure 16.

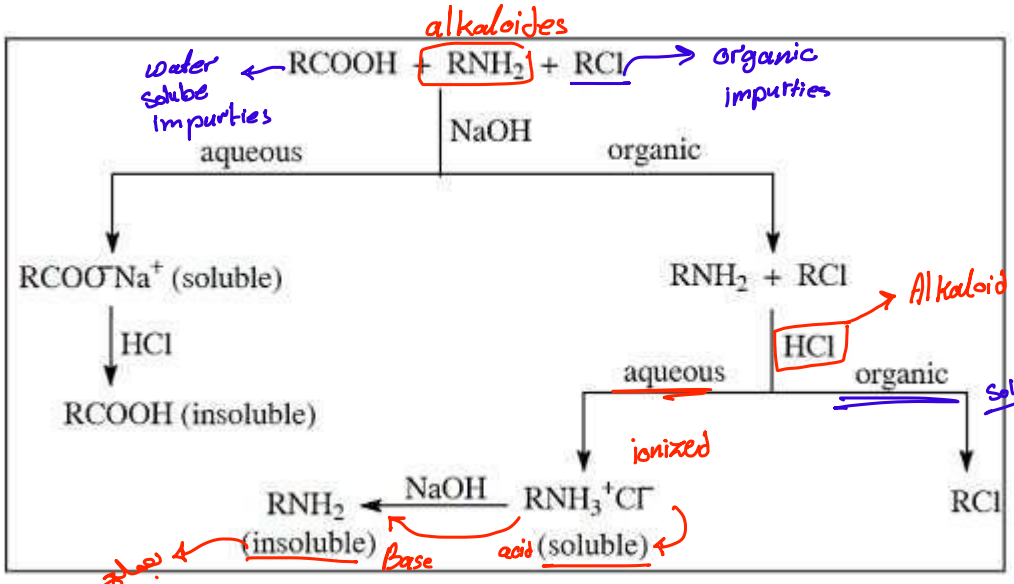


Figure 16. Flow chart for separation of a mixture by acid base extraction.

APPARATUS AND PROCEDURE

The set-up for an extraction using the separatory funnel is shown in Figure 17. The stopcock and stopper should be greased before use. The separatory funnel is held upside down in both hands, such that the stopper is firmly held in the left hand, while the right hand controls the stopcock (Figure 17). The funnel is shaken gently at the beginning and vented periodically through the stopcock to release excess pressure which builds up inside. Once there is no more pressure build-up in the funnel, shaking may be more vigorous to ensure goodextraction.

Figure 17. Correct position for holding a separatory funnel.

The mixture is left standing for some time until the two layers are well separated. The lower layer is drained through the stopcock, while the upper layer is poured through the top of the funnel into a separate container.

To determine whether a given layer is organic or aqueous, place a few drops of it on a watch glass containing a few milliliters of water and check the solubility. As a precaution, never discard any layer before you get your product.

OBJECTIVES

1. Isolating caffeine from tea leaves.
2. Separating a two-component mixture (acid and neutral).

المعلومات الإضافية عن الكافين

Introduction

- The active ingredient that makes tea and coffee valuable to humans is **caffeine**. Caffeine is an **alkaloid**, a class of naturally occurring compounds containing nitrogen and having the properties of an organic amine base. Caffeine is found in over 60 plant species. Caffeine belongs to a family of naturally occurring compounds known as **xanthines**. The xanthines, which come from plants, are possibly the oldest known stimulants. Caffeine is the most powerful xanthine in its ability to (1) increase alertness, (2) put off sleep and to (3) increase ones capacity for thinking. Caffeine is a vasodilator (relaxes the blood vessels) as well as a diuretic (increases urination).

1,3,7-trimethyl xanthine

BackBone

مُدِرّ البول

* Relaxed air passage → improve Breathing

* ease of Muscle Contraction

- Caffeine does not exist alone in tealeaves; the leaves are mainly cellulose, pigments and chlorophylls, and tannins. Tannins are **phenolic** compounds of high molecular weight. The acidic phenols of tannins can complex with metals like calcium to form solid precipitates.

Vasoconstriction in prephral

Vasodilator in Brain

نحكي شوي فسيولوجي

Caffiene → Sympathatic Stimulation

Relaxation ← peripherally

Constriction ← Centrally

- Some of the better-known plant sources are coffee and cocoa beans, tea leaves, and kola nuts. While coffee and tea are both popular products containing caffeine, the amounts vary widely in a single serving. To further confuse the matter, coffee beans contain less caffeine than tea leaves when measured dry. However, a serving of coffee contains roughly twice the caffeine of tea. Much of the flavor of coffee and tea comes from tannins and other flavoring agents.

مُر

- Caffeine has a slightly bitter flavor. As a result, decaffeinating coffee beans and tea leaves will leave the flavor slightly changed even if no other compounds are lost

* معلومة خارجية *

Caffeine can trigger a headache.

When caffeine is consumed regularly, the body becomes dependent on its effects. And because caffeine narrows the blood vessels that surround the brain, when consumption is stopped, the blood vessels enlarge. This causes an increase in blood flow around the brain and pressures surrounding nerves. This can then trigger what is known as a caffeine withdrawal headache. These headaches can last for a couple of weeks because it takes the body a while to adjust to not having caffeine in its system.

- Caffeine's main effect on your body is to make you feel more awake and alert for a while, but it can also cause problems.

- Many studies confirm caffeine's (if it consumed properly) ability to enhance mood and, exercise performance, the speed at which information is processed, awareness, attention, and reaction time.

effect of Caffiene
لو استخدمناه بشكل ✅

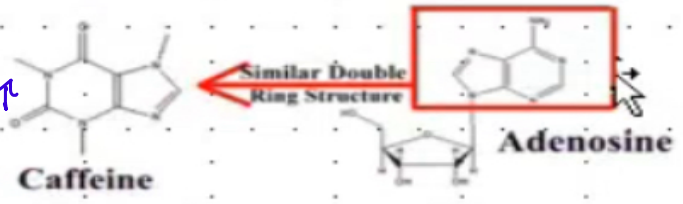
- Non proper consuming of caffeine can make you shaky, make it hard to fall asleep, your heart beat faster, raise your blood pressure, cause headaches, nervousness, In massive doses, caffeine is lethal.
- A fatal dose of caffeine is more than 10 grams (about 170 mg/kg body weight).

effect of Caffiene
لو تم استخدامه بطريقة خطأ ❌
الجرعة الكبيرة قد تكون قاتلة

Mechanism of action

- Adenosine is a central nervous system neuromodulator that has specific receptors.
- When adenosine binds to its receptors, neural activity slows down, and you feel sleepy. Adenosine thus facilitates sleep and dilates the blood vessels.
بشتغل عكس الكافين
- Caffeine acts as an adenosine receptor antagonist. This means that it binds to these same receptors, but without reducing neural activity. Fewer receptors are thus available to the natural "braking" action of adenosine, and neural activity therefore speeds up.
- Caffeine also causes the pituitary gland to secrete hormones that in turn cause the adrenal glands to produce more adrenalin so it increases your attention level and gives your entire system an extra burst of energy.

Receptor ↓ → Neural activity ↑



عدد الـ Receptors مختلف عند الأشخاص
- بيعمل اختلاف بالاستجابة لـ Caffiene

- Caffeine ($C_8H_{10}N_4O_2$) is an alkaloid. **Alkaloids** are bitter tasting, natural nitrogen-containing compounds found in plants. Alkaloids are often found to have potent physiological activity. Some better known examples are morphine, heroin, lysergic acid (LSD), cocaine, quinine, strychnine, and nicotine.

- The basic property of alkaloids come from the lone pair of electrons found on at least one nitrogen. The basic N in caffeine can be used to increase or decrease its water solubility. Acidic conditions will form the conjugate acid salt giving caffeine increased water solubility as a cation. On the other hand if caffeine is in a basic environment it takes the neutral form and is only somewhat polar.

- In order to successfully extract any substance from one solvent into another, we must maximize differences in solubility.

- 1. Adding NaCl to the caffeine in water solution:

The water will be more attracted to the very polar NaCl and less attracted to caffeine thus "salting out" the caffeine from water solution.

- 2. Adding $Ca(OH)_2$ or $CaCO_3$ to a caffeine in water solution:

a. This makes the solution basic so puts caffeine in its least polar form and so more readily solvated in organic solvents and less attracted to water.

b. The water solution contains much more than just caffeine, and some of these compounds are also soluble in organic solvents. Basic $Ca(OH)_2$ reacts with tannic acids to form insoluble tannin salts which precipitate and so can be removed from the solution before the caffeine is extracted.

EXPERIMENTAL

MATERIALS NEEDED	Glassware: Beaker (600 mL), 2 beakers (100 mL), 2 Erlenmeyer flasks (100 mL), stand, wire gauze, clamp, ring, clamp holder, graduated cylinder (10 mL), separatory funnel (100 mL), cheesecloth 20x20 cm. Chemicals: 15 g Tea leaves, 10 g sodium carbonate, 30 mL dichloromethane, 1.0 g anhydrous sodium sulfate, 1 g benzoic acid, 1 g p-dichlorobenzene, 25 mL ether, 40 mL of 10% sodium hydroxide, 20 mL concentrated hydrochloric acid, anhydrous calcium chloride, blue litmus paper.

➢ *EXTRACTION OF CAFFEINE FROM TEA LEAVES*

Caffeine is an organic compound present in the fruit and bark of some plants, as well as in tea leaves, coffee, cocoa and cola beans. The caffeine content in dried tea leaves is about 3-4%.

Caffeine belongs to a family of basic, nitrogen-containing, cyclic compounds called alkaloids. It is a mild stimulant and is used as such in many drugs and analgesics. The solubility of caffeine at room temperature is 2.2 g/100 mL of water and 18 g/100 mL of chloroform.

In this experiment, you will extract caffeine from tea leaves with hot water. This treatment also extracts tannins, a class of acidic organic compounds, also present in the leaves. Sodium carbonate is used to remove the acidic tannins by converting them to water-soluble salts.

Procedure. You will be provided with a large tea bag containing about 15 g dry tea leaves. Place the tea bag in a 600 mL beaker, add 10 g of sodium carbonate and 150 mL of water, and boil the mixture gently for 20 minutes. Cool the dark brown aqueous solution to room temperature and

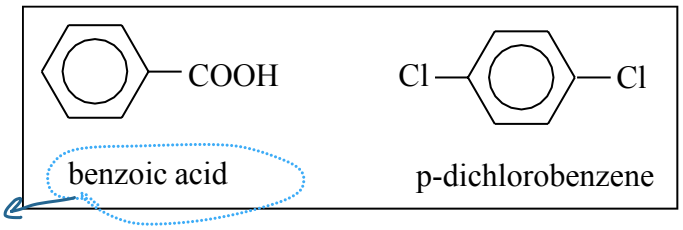
squeeze the tea bag to extract the liquid fully before discarding the bag.

Transfer the dark solution to a separatory funnel and extract twice with 15 mL portions of dichloromethane.

Avoid vigorous shaking of the funnel since emulsions may form readily; instead, swirl the funnel gently or turn it upside down several times. After each extraction drain the denser dichloromethane layer into a small flask. Dry the combined organic extracts with anhydrous sodium sulfate until the solution is clear. Decant the dichloromethane into a small beaker andevaporate to dryness over a water bath in the fume hood. Do not heat the residue any longer than necessary since caffeine decomposes readily. Weigh the crude caffeine and calculate its percentage in the tea leaves. Determine the melting point of your product.

➢ *SEPARATION OF A TWO COMPONENT MIXTURE*

In this part, a mixture of benzoic acid and *p*-dichlorobenzene (or any other neutral compound such as naphthalene, *p*-dimethoxybenzene or diethoxybenzene) will be separated into its components by means of extraction.



water soluble

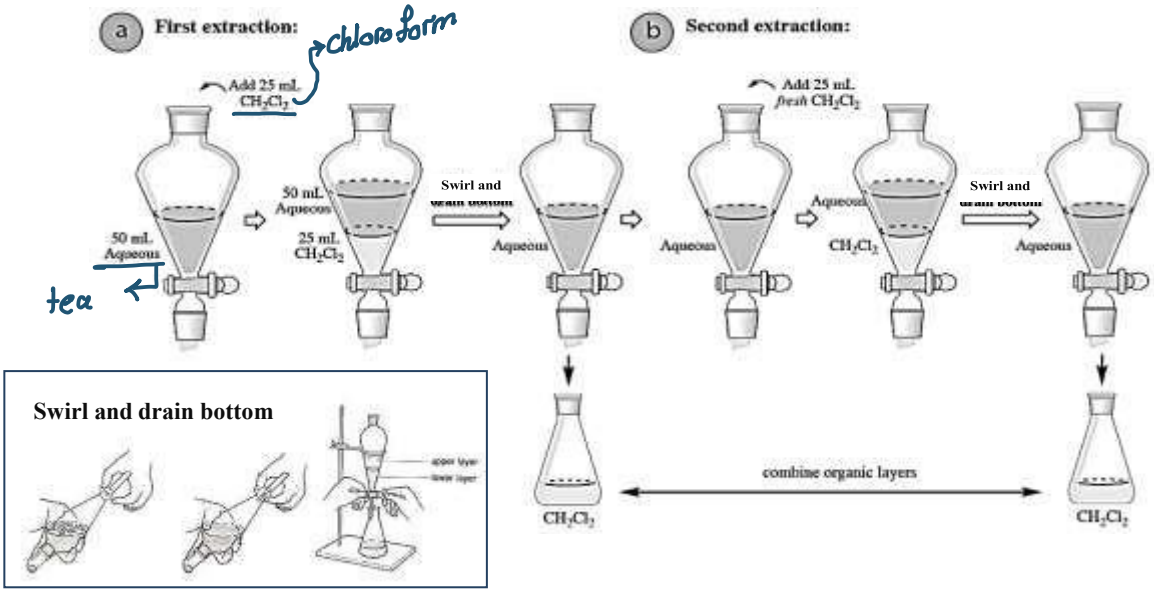
Procedure. Obtain from your instructor a 2.0 g sample of the two-component mixture, dissolve it in 25 mL of ether, and pour the solution into a 100 mL separatory funnel. To extract the benzoic acid from the mixture, shake the ether solution with 20 mL of 10% NaOH solution.

Draw off the lower (aqueous) layer into a flask and repeat extractionof the ether solution with another 20 mL of 10% NaOH solution followed by 10 mL of water. Combine the three aqueous extracts and cool in an ice

bath. Neutralize the cold aqueous phase by adding concentrated HCl until the solution is acid to blue litmus paper (about 8 mL). Collect the precipitated benzoic acid by suction filtration.

Dry the product, determine its weight and its melting point. Pour the remaining ether solution (which contains the neutral component) into a small flask and add enough anhydrous calcium chloride to remove any traces of water. Decant the dry ether solution into a small, weighedbeaker and evaporate the solvent in the fume hood (use a low temperature water bath since *p*-dichlorobenzene may sublime). Determine the weight of yourproduct.

PROCEDURE SUMMARY DIAGRAM



Useful links

مهمين و بسهلوا شغل المختبر وفهم الـ Procedure

Extraction of Caffeine from tea leaves:
https://www.youtube.com/watch?v=5K1t4-1TDdo

How to use the separatory funnel:
https://www.youtube.com/watch?v=EFiFPoOzqtk&t=161s

Experiment 8

Caffeine extraction

Name: Section No.

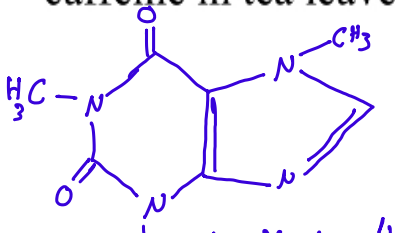
The objective of the experiment

Isolating caffiene from the tea leaves

Weight of tea bag:

Prelab work:

Draw the structure of caffeine and find the expected amount of caffeine in tea leaves?



1,3,7-trimethylxanthine

Fill the following table

A)	**Weight of empty beaker used to dry the solvent in final step**	
B)	**Weight of beaker and caffeine without the solvent**	
C)	**Weight of caffeine**	B − A
	Percentage of caffeine in tea leaves	$\frac{\text{weight of caffeine}}{\text{weight of tea bag}}$