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, as in 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 separatory funnel. The various solutes in the mixture distribute themselves between the organic 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

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, non toxic, 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

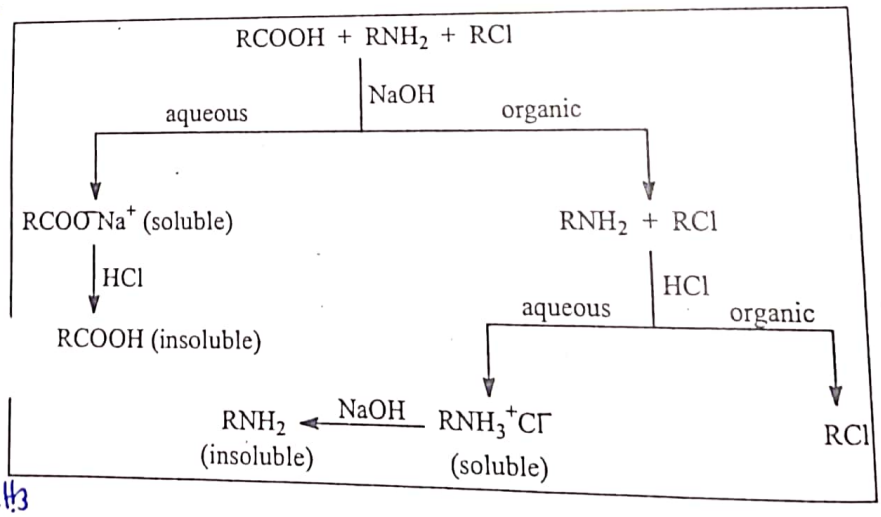
(e.g., when alkaline aqueous solutions are extracted with chloroform or dichloromethane). If an emulsion still forms one can often break it by:

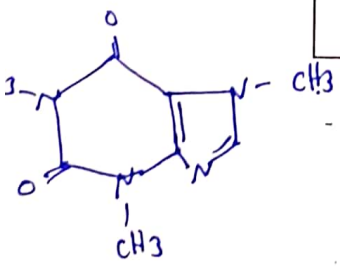
1. Stirring the emulsified layer gently with a glass rod.
2. Saturating the aqueous layer with a salt.
3. Centrifugation.

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.





caffeine

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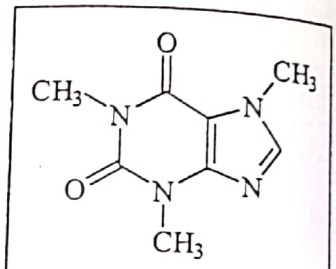
EXPERIMENTAL

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

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 and evaporate 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.

① 150mL water in 600mL beaker

② Tea bag 1.5 g

③ 10 g sodium carbonat ( )

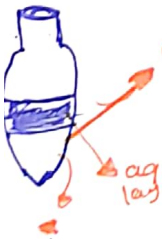
④ Boil mixture gently for 20 minutes.

⑤ cool the drak brown aqueous solution to room temperture.

⑥ Clamp + separatory funnle

⑦ Transfer the drak solution into separtory funnle

Don't forget:- to squeeze tea bag : to extract the organic layer fully before discarding it



⑧ Extract the solution twice 15 mL portions of dichloromethane

⑨ 15 mL dichloromethane.

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⑨ Drain the dichloro-methane layer into small flask

⑩ Repeat the extraction of solution with another 15 mL dichloro methane