Experimental No. (6)   **Extraction**

Extraction

Purpose of experimental

To purify samples of organic compounds that are solids at room temperature

Theory part of experimental

Extraction is a method often used by organic chemists for the rapid crude separation of mixtures containing acidic and/or basic compounds often in the presence of neutral materials. The separation uses acid-base chemistry and two mutually insoluble layers of solvent (water and t-butylmethylether).

Extraction is the recovery of a substance from a mixture by bringing it into contact with a solvent, which dissolves the desired material.

Like recrystallization and distillation, **extraction** is a separation technique frequently employed in the laboratory to isolate one or more components from a mixture. **Unlike** recrystallization and distillation, it does not yield a pure product; thus, the former techniques may be required to purify a product isolated by extraction. In the technical sense **extraction is based on the principle of the equilibrium distribution of a substance (solute) between two immiscible phases, one of which is usually a solvent**. The solvent need not be a pure liquid but may be a mixture of several solvents or a solution of some chemical reagent that will react with one or more components of the mixture being extracted to form a new substance soluble in the solution. The material being extracted may be a liquid, a solid, or a mixture of these.

Extraction is accomplished by shaking the solution in a **separatory funnel** with a second solvent that is immiscible with the one in which the

compound is dissolved, but dissolves the compound more readily. Sometimes not the entire product is extracted in a single operation and the process must be repeated once or twice more to assure a clean separation. It has been found that when two immiscible solvents are shaken together, the solute distributes itself between them in a ratio roughly proportional to its solubility in each. The ratio of the concentration of the solute in each solvent at equilibrium is a constant called the **distribution ratio(d) or partition coefficient (Kd).**

$$Kd = [solute]_{org.}/ [solute]aq. = C_{org.} / C_{aq.}$$

The larger the value of Kd, the more solute will be transferred to the ether with each extraction, and the fewer portions of ether will be required for essentially complete removal of the solute.
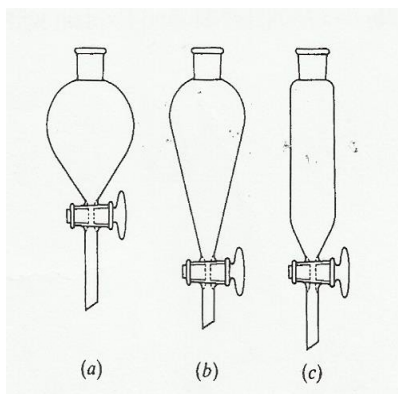
(a)   (b)   (c)   **seperatory funnel**

Properties of an organic extraction solvent :

(1) It must readily dissolve the substance being extracted but must not dissolve to any appreciable extent in the solvent from which desired substance is being extracted.

(2) It should extract neither the impurities nor other substances present in the original mixture.

(3) It should not react with the substance being extracted.

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(4) It should be readily separated from the desired solute after extraction.
Some of the solvents commonly used for extracting aqueous solutions or
mixtures include diethyl ether, methylene chloride, chloroform, carbon

tetrachloride, benzene, n-pentane, n-hexane, and various mixtures of
saturated hydrocarbons from petroleum (petroleum ether, ligroin, etc.).
Each of these has a relatively low boiling point so that it may be fairly
easily separated from the solute by evaporation or distillation. Methanol
and ethanol are not good solvents for extracting aqueous solutions or
mixtures because of their solubility in water; however, if an aqueous
solution can be saturated with potassium carbonate without affecting the
solute, ethanol can be used to extract polar solutes from the solution.

Chemical and Apparatus

benzoic acid, CH_2Cl_2 (dichloromethane), water, separatory funnel ,
beaker, $CaCl_2$, boiling chip, hotplate, balance, filter paper, funnel

Procedure of experimental

Part A

1- Weigh out 0.5 mg of benzoic acid and dissolve it in separatory funnel
containing 1.6 mL CH_2Cl_2.

2- Add 1.6 mL distilled water and mix thoroughly.

3- Alternatively, you may shake the separatory funnel after sealing with
a stopper.

4- Set the separatory funnel aside and allow the layers to separate.

5- Remove the aqueous layer without removing any organic layer.

6- Add anhydrous $CaCl_2$ to the CH_2Cl_2 layer

7- After 5 minutes of drying time, remove the organic layer and place in a clean, dry, pre-weighed 10 mL Erlenmeyer flask with a boiling chip.

8-Evaporate the solvent on a hotplate in the hood to dryness.

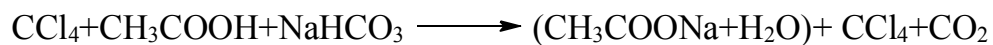9- Weigh the flask with boiling chip and benzoic acid. Use this mass to determine the weight of benzoic acid.

Questions for discussion

1- Why must you remove the stopper from the separatory funnel before draining?

2- What is the purpose of $CaCl_2$?

3- What is the purpose of the boiling chips?

Part B

1- Place 5 mL of CCl_4 contamination with acetic acid in separatory funnel .

2- Add 15 mL of (15%) sodium carbonate solution   and mix thoroughly.

3- Alternatively, you may shake the separatory funnel after sealing with a stopper.

4- Set the separatory funnel aside and allow the layers to separate.

5- Remove the aqueous layer without removing any organic layer.

6- After 5 minutes of time, remove the organic layer and place in a clean, dry, pre-weighed 10 mL Erlenmeyer flask

7- Measure the volume of organic layer and calculate the percentage volume

$$\text{The percentage volume \%} = \frac{\text{practical volume}}{\text{theoretical volume}} \times 100$$

Experimental No. (6)   **Extraction**

$$CCl_4 + CH_3COOH + NaHCO_3 \longrightarrow (CH_3COONa + H_2O) + CCl_4 + CO_2$$

Questions for discussion

1. Why we added sodium carbonate solution ?
2. Define the extraction
3. What is the principle of extraction

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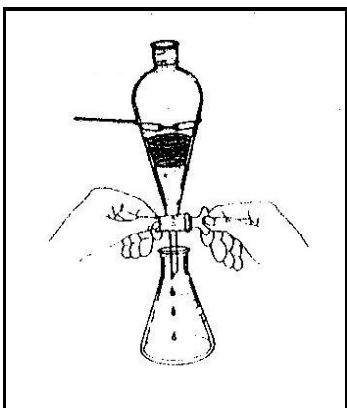
Experimental No. (7)   **Extraction of caffeine from tea**
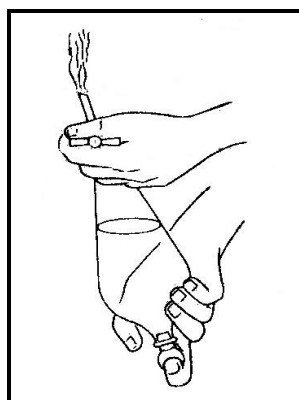
Extraction of caffeine from tea

Purpose of experimental

1. To demonstrate the isolation of a natural product.
2. To learn the techniques of extraction.

Theory part of experimental

Many organic compounds are obtained from natural sources through extraction. This method takes advantage of the solubility characteristics of a particular organic substance with a given solvent. In the experiment here, caffeine is readily soluble in hot water and is thus separated from the tea leaves. Caffeine is one of the main substances that make up the water solution called tea. Besides being found in tea leaves, caffeine is present in coffee, kola nuts, and cocoa beans. As much as 5% by weight of the leaf material in tea plants consists of caffeine.

The caffeine structure is shown below. It is classed as an alkaloid, meaning that with the nitrogen present, the molecule has base characteristics (alkali-like). In addition, the molecule has the purine ring system, a framework which plays an important role in living systems. Caffeine is the most widely used of all the stimulants.
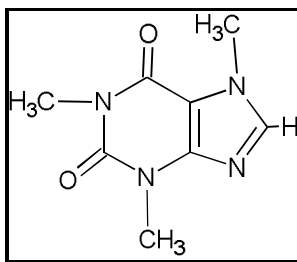
Structure of Caffeine

Experimental No. (7)   **Extraction of caffeine from tea**

Tea leaves consist primarily of cellulose; this is the principle structural material of all plant cells. Fortunately, the cellulose is insoluble in water, so that by using a hot water extraction, more soluble caffeine can be separated. Also dissolved in water are complex substances called tannins. These are colored phenolic compounds of high molecular weight (500 to 3000) that have acidic behavior. If a basic salt such as Na_2CO_3 is added to the water solution, the tannins can react to form a salt. These salts are insoluble in organic solvents, such as chloroform or dichloromethane, but are soluble in water.

Although caffeine is soluble in water (2 g/100 g of cold water), it is more soluble in the organic solvent dichloromethane (14 g/100 g). Thus caffeine can be extracted from the basic tea solution with dichloromethane, but the sodium salts of the tannins remain behind in the aqueous solution. Evaporation of the dichloromethane yields crude caffeine; the crude material can be purified by sublimation.

Chemical and apparatus

  Boiling chips, Cold finger condenser, Filter paper, fast flow

Hot plate, 125-mL separatory funnel with stopper, Melting point capillaries, adapter, Tea leaves, rubber stopper, Anhydrous sodium sulfate Na_2SO_4, Anhydrous sodium carbonate Na_2CO_3, Dichloromethane CH_2Cl_2,

Procedure of experimental

1. Into a 150-mL beaker, place the tea leaves so that they lie flat on the bottom. Add 30 mL of distilled water and 2.0 g of anhydrous Na2CO3; heat the contents with a hot plate, keeping a *gentle* boil, for 20 min. While the mixture is boiling, keep a watch glass on the beaker.

2. Decant the hot liquid into a 50-mL Erlenmeyer flask, carefully pressing the tea bag with a glass rod; add this wash water to the tea extract. (If any solids are present in the tea extract, filter them by gravity to remove.) Cool the combined tea extract to room temperature. Transfer the cool tea extract to a 125-mL separatory funnel that is supported on a ring stand with a ring clamp.

3. Carefully add 5.0 mL of dichloromethane to the separatory funnel. Stopper the funnel and lift it from the ring clamp By holding the stopper in place with one hand, invert the funnel.

4. Return the separatory funnel to the ring clamp, remove the stopper, and allow the aqueous layer to separate from the dichloromethane layer. You should see two distinct layers form after a few minutes, with the dichloromethane layer at the bottom. Sometimes an emulsion may form at the juncture of the two layers. The

   emulsion often can be broken by gently swirling the contents or by gently stirring the emulsion with a glass rod. Separation of the aqueous layer and the dichloromethane layer in the separatory funnel.

5. Carefully drain the lower layer into a 25-mL Erlenmeyer flask. Try not to include any water with the dichloromethane layer.

6. Repeat the extraction with an additional 5.0 mL of dichloromethane. Combine the separated bottom layer with the dichloromethane layer obtained from step no. 5.

7. Add 0.5 g of anhydrous Na_2SO_4 to the combined dichloromethane extracts. Swirl the flask. The anhydrous salt is a drying agent and will remove any water that may still be present.

8. Weigh a 25-mL side-arm filter flask containing one or two boiling stones. Record this weight (2). By means of a gravity filtration, filter

the dichloromethane–salt mixture into the pre-weighed flask. Rinse the salt on the filter paper with an additional 2.0 mL of dichloromethane.

9. Remove the dichloromethane by evaporation *in the hood*. Be careful not to overheat the solvent, since it may foam over. The solid residue which remains after the solvent is gone is the crude caffeine. Reweigh the cooled flask (3). Calculate the weight of the crude caffeine by subtraction (4) and determine the percent yield (5).

10. Take a melting point of your solid. First, scrape the caffeine from the bottom and sides of the flask with a microspatula and collect a sample of the solid in a capillary tube. Pure caffeine melts at 238 °C. Compare your melting point to the literature value.

Calculation

$$\% \text{ Yield } = \frac{\text{Weight of product}}{\text{Weight of sample}} \times 100$$

Plan of work

Experimental No. (7)   **Extraction of caffeine from tea**
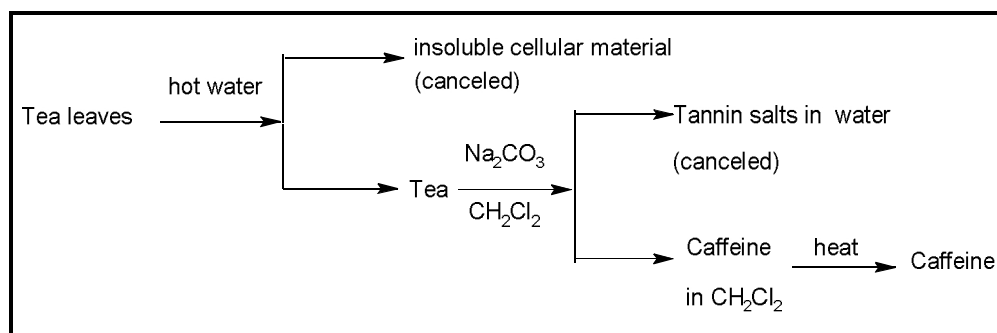
Questions for discussion

1. Is methylene chloride more or less dense than water? What evidence do you have to support your answer?
2. Was your melting point the same as the reported melting point? If not, why not?
3. Why was sodium carbonate added to the coffee solution?
4. Explain why the caffeine that you obtained in this experiment may not have been white in color.
5. What method is used to obtain caffeine for tea leaves?
6. Why is caffeine classed as an alkaloid?
7. Why might an individual use a product containing caffeine?
8. Besides caffeine, what other compounds are found in tea leaves?