AL- MUSTAQBAL UNIVERSITY College Of Health And Medical Techniques Prosthetic Dental Techniques Department Second Grade Second Semester



PRACTICAL ORGANIC CHEMISTRY

FOR Prosthetic Dental Techniques Department

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PART 2

EXP.1

(Separation And Purification of Organic Compounds)

By:

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Safety Rules:

- 1. Wear safety goggles. You may only remove your goggles if no one in the lab room is using chemicals or washing glassware.
- 2. Contact lenses must not be worn.
- 3. Wear protective clothing: You should be covered from the top of your shoulder to below your knees. A laboratory coat is suitable, especially if you are not well-protected by the clothes you have chosen to wear to lab. Wear closed-toed shoes. Gloves are highly recommended at all times.
- 4. Food and drink are not allowed in the lab room.
- 5. You may not use personal music players during your laboratory session.
- 6. No smoking.
- 7. Work in your student hood at all times. Cover containers of compounds during transport through the lab.
- 8. Prevent breakage by handling glassware with care at all times.
- 9. Prevent chemical spills and glassware breakage by clamping flasks containing chemicals whenever possible. This is especially true for reaction and vacuum flasks.
- 10. Place broken glassware in the proper receptacle.
- 11. Report frayed electrical cords to your TA.
- 12. Wrap cords firmly around equipment before you put it away to avoid trip hazards.
- 13. Use a Bunsen burner only when directed by your TA.
- 14. Place hazardous waste in the proper container.
- 15. When the fire alarm sounds, leave the building immediately.
- 16. Keep a clear pathway to the exits: Do not bring your bike or skateboard to lab and keep all personal items off the floor or close to the wall by the coat rack.
- 17. Never conduct experiments in the laboratory alone.
- 18. No unauthorized experiments.

COMMON ORGANIC LABORATORY APPARATUS



Fig.3. Common organic laboratory apparatus



Fig.4. Common organic laboratory apparatus



Fig.5. Common organic laboratory apparatus



Fig.6. Common organic laboratory apparatus





Fig.7. Common organic laboratory apparatus

COMMON ORGANIC LABORATORY APPARATUS



Test tube rack



thermometer adaptor



Rubber tubes



filtering funnels



Crystallizing dish



stir rod



Vials



graduated cylinders

Fig.8. Common organic laboratory apparatus

SEPARATION AND PURIFICATION OF

ORGANIC COMPOUNDS

The separation and purification of organic compounds is important way to get pure organic compounds. Products of organic reactions are seldom pure products as a result of side reactions. Pure compounds are also subject to partial decomposition on standing for some time or on exposure to light, air, heat, moisture, etc. (for example acetyl salicylic acid, commonly called aspirin, decomposes to salicylic acid). Therefore, the process of separation and purification of organic compounds becomes an important technique to get pure compounds.

Generally, organic compounds are separated and purified by the following methods:

- 1- Solution and filtration.
- 2- Crystallization.
- 3- Distillation.
- 4- Extraction.
- 5- Sublimation.
- 6- Chromatographic methods as TLC, GC.

Experiment 1

SOLUTION AND FILTRATION

When a solid or a liquid* (solutes can be solids or liquids, while solvents are liquids) dissolves, the structural units – ions or molecules, become separated from each other and the solvent molecules occupy the space between them.

The solubility of organic compounds can be divided into two major types;

1- Solubility in which the chemical reaction is the driving forces, for example, acid-base reactions:



2- Solubility in which only simple miscibility is involved, for example, ethyl ether in carbon tetrachloride.

The first type is used to identify the functional groups involved in the compound, while the second type is used to determine solvents for recrystallization and chemical reactions.

The filtration is an important procedure after completing the reaction either to separate the solid product (precipitate) or to get rid of insoluble impurities or reactant materials. The desired soluble compound is recovered from the filtrate by evaporating the solvent.

The liquid is poured into a filter paper fitted in a funnel and either the precipitate is collected or the filtrate that contains the desired soluble compound is collected. In this method we take the advantage that one compound in the mixture is readily soluble in a given solvent whereas the remainder of the mixture may be relatively insoluble.

Filtering Techniques:

1- Gravity Filtration:

• Flute filter paper. Fluting is desirable when a rapid filtration is necessary, as in the filtration of the hot solution during a recrystallization.



Fig.1: Fluting a filter paper (paper folded with many pleats to give a large surface)

• Place filter paper in the funnel. Place the funnel either directly on top of the beaker or on a ring stand above the beaker (see Fig. 4 below).



Fig.2. A beaker containing a stem less funnel. Note that the filter paper is fluted; however, that is not necessary for all applications.

• Wet the filter paper using a small amount of the liquid that is the solvent of the mixture being filtered.

• After the filtrate has been collected, pass a small amount of the wash liquid through the filter paper to wash the residue.

2- Vacuum Filtration with Buchner funnel:

• Select a Buchner funnel of appropriate size for the amount of residue you are collecting, not the amount of filtrate. Put a piece of filter paper in the funnel that fits exactly and wet the paper using a small amount of solvent.

• Place the funnel in a filter flask and connect the rubber tubing (see Fig. 4 below).



Fig. 3. A Buchner funnel in a filter flask, with rubber tubing connected to an aspirator.

• Using rubber tubing, the filter flask is connected to a trap, which in turn is connected to an aspirator, which provides the vacuum.

• Pour 15 ml of solvent through the funnel to wet the filter paper. Discard this water. Reconnect the flask to the aspirator.

• After the filtrate has been collected, pass a small amount of pure solvent through the filter to wash the residue.

3- Vacuum Filtration with Filter Crucible:

• To use a filter crucible, set up vacuum filtration by placing the filter crucible in a holder and placing that in a filter flask (see Fig. 3 below).



Fig. 4. A filter crucible in a crucible holder in a filter flask, with rubber tubing connected to an aspirator.

Procedure:

An impure mixture containing about 0.5 gm of benzoic acid and 0.5 gm of sugar (glucose) is to be dissolved in about 10 ml of distilled water. The sugar will dissolve in water while benzoic acid remains precipitated. Then perform filtration, by which the benzoic acid remains as the precipitate on the filter paper while the sugar goes with the filtrate as a solution. The sugar can be recovered from the filtrate by evaporating the solvent (water).

To test that the precipitate (on the filter paper) is the benzoic acid, sodium bicarbonate solution is to be added on this precipitate. The benzoic acid will be dissolved due to the formation of soluble sodium benzoate and bubbles of the evolved CO₂ gas will be seen.

Questions:

- If you have a mixture of urea and salicylic acid how can you separate them using solution and filtration method?
- After separation of urea from the mixture in the above question, give a way for

determining the efficiency of your method, i.e. the purity of your product.

• Why is fluted filter paper used in gravity filtration?

Thank You For Your Attention