**LABORATORY REPORT**

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| **SEPARATION AND PURIFICATION OF ORGANIC COMPOUNDS** |
| |  |  | | --- | --- | | Name: | Victor Kwansa | | Index Number: | 41350033 | | Class: | O.1.2.3 | | Demonstrator: | Ms. Flora Amarh | | Date: | 23rd February 2009 | |
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| **AIMS/OBJECTIVES:**   1. To isolate each component dry it and determine its weight. 2. To become acquainted with various separation methods. 3. To examine the solubility behavior of the various compounds in the mixture. |
| **INTRODUCTION/THEORY:**  In this experiment we would separate and purify organic compounds.  At the conclusion of a reaction the pure product must be isolated from the reaction mixture by a sequence of preparations collectively termed the work-up. As for example solvent this has been used as the reaction arising from alternative reaction pathway and so on. The planning of the isolation operations and the application to such complex mixtures is therefore an exaction test of the expertise of the chemist.  The disappearance from a reaction mixture of one of the reactants or the build-up of the reaction product, measured on a small aliquot portion removed at convenient time intervals from the bulk reaction mixture can yield valuable information on the progress of a reaction.  The adoption of a particular isolation procedure will depend to a large extent upon the physical and chemicals properties of the product whether it is a one phase (either solid or liquid) or a two phase (solid, liquid or liquid) system.  Some of the most challenging and time consuming laboratory procedures involve separation mixtures and purifying the components. Several common separation techniques are described. These are   1. Filtration 2. Crystallization 3. Distillation 4. Extraction 5. Chromatography   All these methods depend on the physical properties of the substances in the mixture and no chemical changes occur.  **Filtration:** separates the components of a mixture on the bases of difference in particle size. It is used most often to separate a liquid (smaller particles) from a solid (larger particles).  **Crystallization:** is the process of purification of compounds in which pure solid substances are obtained or isolated from impure solution. Crystals obtained can be recrystallised again thus increasing the degree of its purity.  **Distillation:** separates components through difference in volatility the tendency of a substance to become a gas.  Furthermore extraction is also based on difference in solubility. Therefore such a process can be used to separate organic substances from inorganic impurities simply by shaking on aqueous solution or suspension with suitable immiscible solvents such as benzene, dietheylether etc. To bring the separation process to an end, the combined organic phase is dried and the solvent is evaporated. There is sometimes the addition of electrolyte such as ammonium sulphate to the aqueous phase which helps to ensure that the organic layer separates clearly and also increases the extent of extraction into the layer.  All these methods depend on the physical properties of the substances in the mixture and no chemical changes occur  In choosing a suitable solvent, we need to find one in which the material to be extracted is readily soluble whereas the substance from which it is being extracted is not. The same consideration is applied not withstanding the fact that it is the substance being purified or one of its contaminants, that is taken into a new phase |
| **CHEMICALS & EQUIPMENT:**   1. Diethyl ether 2. NaOH solution 3. Distilled water 4. Separating funnel 5. Litmus paper 6. Filter paper and funnel 7. Beaker |
| **PROCEEDURE:**   |  |  | | --- | --- | | **PROCEDURE** | **OBSERVATION** | | A small amount of the given mixture was scooped into a beaker using a spatula. |  | | The mixture was then dissolved in water and then filtered into another beaker. | No observable reaction. | | The residue was washed into the beaker with NaOH and ten transferred into a separatory funnel. | Residue was insoluble in NaOH. | | Petroleum ether was added to the mixture in the separatory funnel. |  | | The separatory funnel was corked and shaken vigorously. The mixture was allowed to settle. | Three distinctively layers were formed. | |
| **DISCUSSION:**  From the result, the reaction mixture contained sodium salt, neutral organic compound, acid organic compound and inorganic compound. When water was added, the only component that dissolved in the water was the one which had quite strong intermolecular forces between them. Ionic compound such as salts and bases are attached to the water molecules through hydrogen bonding which is among the strongest of intermolecular forces. Hence, sodium salt can be said to have dissolved here since there was enough hydration energy to effect solubility.  NaOH and diethyl ether were used for easy dissolution of a particular solute; this method is known as selective solubility. The addition of NaOH specifically speaking formed a clear solution which settled at the bottom indicating the organic compound and therefore dissolved in diethyl ether. This is because petroleum ether is non-polar and so is the organic compound, thus they interact with each other. The insoluble inorganic compound which is quite denser than the soluble organic compound was also observed below this layer. |
| **ERROR ANALYSIS:** |
| **PRECAUTIONS:**   1. Separating funnel are very fragile and must be supported in an iron ring, pad the ring with rubber tubing to prevent breakage. 2. Always be certain that the stopcock is in a closed position before the funnel is returned to the normal vertical position that is securely seated, not floating loosely. 3. Always hold the stopper securely seated in the funnel. When the funnel is shaken vigorously to mix the two solvents, pressure inside the funnel increases as a results of the additive effect of the two particles vapour pressures of the two immiscible solvents. |
| **CONCLUSION:**  The bottom layer is the insoluble inorganic compound and on top of it was the soluble organic compound. The objectives of this experiment were successfully achieved. |
| **REFRENCES:**   1. CHEMISTRY: The Molecular Nature of Matter and Change by Martin Silberberg 2. Vogel’s Textbook of Practical Organic Chemistry. 3. CHEMISTRY: A contemporary Approach by Wesley. D. Smith and David. G. Lygre. |