Reearch paper

Environment, Water

Recrystallization Estopace, Edgie1, Polintan, Clarisse K. Professor Edgie Estopace, School of Chemical Engineering, Chemistry and Biotechnology, Mapua Institute ofTechnology; Clarisse Polintan, CHM145L/A21, School of Chemical Engineering, Chemistry and Biotechnology, Mapua Institute of Technology ABSTRACT This experiment is all about identifying the appropriate solvent for recrystallization and technique and to use the recrystallization technique in purifying a solid sample. Most organic substances are impure and require techniques in order to purify a sample. One of these techniques to make an impure sample pure is by recrystallization.

This experiment includes the determination of a good solvent for recrystallization for compounds such as: acetanilide, acetamide, aspirin, benzoic acid, naphthalene, and sucrose, for solvents such as: water, ethanol, benzene, and ethyl acetate. Also pure acetanilide is achieved. here are five major steps in the recrystallization process: dissolving the solute in the solvent, performing a gravity filtration, if necessary, obtaining crystals of the solute, collecting the solute crystals by vacuum filtration, and, finally, drying the resulting crystals giving us the pure sample of the compound.

The best solvent used for each of the compounds listed and the percentage recovery of the crude acetanilide. The physical properties of the compound were also determined. INTRODUCTION Differential solubility is defined as the differences in the amount of solid that can be dissolved in an appropriate solvent as affected by variations in temperature. Solubility is inversely proportional to its temperature, since most solids have solubilities that are lower in liquid solvents at low temperatures.

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Differences in solubilities are sometimes used in the process of obtaining pure compounds by dissolving the solid in hot solvent and allow the undissolved impurities to be filtered off. The filtrate would then be cooled down and recrystallized as a purer compound. There should be differences in the solubilities of the solid and the impurities in order for the recrystallization to be effective. Recrystallization only works when the proper solvent is being used.

The appropriate recrystallization solvent should: dissolve the entire compound at high temperature, dissolver very little or none of the compound at low temperature, have different solubilities for the compound and the impurities, have a boiling point below the melting point of the compound, have relatively low boiling point, be inert withrespectto the compound, and be relatively inexpensive. MATERIALS AND METHODS The first part of the experiment required the students figure out which solvent is appropriate for recrystallization technique. Approximately 0. 10 grams of acetamide was weighed and placed inside a test tube.

The acetamide was then dissolved in two millilitres of cold water. The solubility behaviour was then observed. If the acetamide did not dissolve, the mixture was then boiled and it’s solubility behaviour was again observed. These steps were then repeated but with cold ethanol, benzene, and ethyl acetate as the solvent instead of water. The above procedures were repeated, however, the students used different solutes this time

forobservation. The compounds used next were acetanilide, aspirin, benzoic acid, naphthalene, and sucrose, followed by the recording of data.

The second part of the experiment tackled the recrystallization process using impure acetanilide and water as a solvent to obtain a pure sample of acetanilide. This part used the Hot Gravity Filtration Set-up first, followed by the Vacuum Filtration Set-up. The Hot Gravity Filtration Set-up included the following apparatuses: a stemless funnel, a fluted ashless filter paper, an Erlenmeyer flask, a hot water bath, and a hot plate. The Vacuum Filtration Set-up included water suction, rubber tubing, a 500ml Erlenmeyer flask, a rubber stopper and a porcelain Buchner funnel.

Other apparatuses used include the Thomas-Hoover Melting Apparatus, micro test tubes, a test tube rack, capillary tubes, a beaker, and an oven. First, approximately 100 mL of water was heated and approximately 3. 50 grams of crude acetanilide was weighed. The impure acetanilide was then dissolved in 30 mL of water using 150-ml beaker. The mixture was slightly boiled using a hot plate. When the mixture started to boil, 2-ml portions of the previously heated water was added until the white solid had dissolved. A temperature of 90 C was maintained. The total volume of the water used was then recorded.

A small amount of activated carbon was then added. Using the hot gravity filtration set up, the solution was poured into the fluted filter paper that was in the stemless glass funnel. The filtrate should then be colourless, if not more activated carbon was added and the filtration process was repeated. The flask was then removed from the hot plate and the residue was discarded. FIGURE 1. Hot Gravity Filtration Setup FIGURE 2. Fluted Filter

paper The colorless filtrate was then cooled down to room temperature, placed in an ice bath, and was stirred continuously until crystallization was complete.

This took about ton to fifteen minutes. When the crystallization was complete, the solution was vacuum filtered and then the crystals were washed in 2 mL of cold water. The crystals were then dried and weighed. The percentage recovery was then computed for and the melting point of the purifying crystals was determined using the Thomas Hoover Melting Apparatus. FIGURE 3. Vacuum Filtration Setup RESULTS Table I. Choice of Solvent (S= Soluble , I= Insoluble) Compound| Water| Ethanol| Benzene| Ethyl Acetate| | Cold| Hot| Cold| Hot| Cold| Hot| Cold| Hot| Acetamide| S| S| S| S| I| S| S| S| Acetanilide| I| S| S| S| I| S| S| S|

Aspirin| I| S| S| S| I| S| S| S| Benzoic Acid| I| S| S| S| S| S| S| S| Naphthalene| I| S| I| S| S| S| S| S| Sucrose| S| S| I| S| I| I| S| S| Compound| Solvent Appropriate for Recrystallization| Acetamide| Benzene| Acetanilide| Water| Aspirin| Benzene| Benzoic Acid| Water| Naphthalene| Water| Sucrose| Ethanol| Table 1. 1 Table 1. 2 Table 2. Recrystallization of Impure Acetanilide Weight of crude acetanilide in grams| 3. 53 g| Weight of the filter paper in grams| 1. 23 g| Weight of the pure acetanilide in grams| 0. 79 g| Volume of water used in mL| 62 mL| Melting Point of pure acetanilide in | 130-190 |

Percentage Recovery| 29. 63 %| Step| Observation| Boiling of crude acetanilide| It boiled slowly and the boiling started at 90 C. White substance boiled as water was added; black substance remained | Addition of activated carbon| Impurities in water were absorbed. | Hot Gravity Filtration| Black substance was left on the filter paper and the white liquid went down and

was separated from the black substance. | Cooling in an ice bath| Dissolved acetanilide formed white crystals. | Vacuum Filtration| The acetanilide was filtrated and what was left on the filter was the crystals. DISCUSSION In the experiment, all six of the seven criteria were vividly exhibited. The fist criterion states that the solvent should dissolve all of the compounds at high temperature. The second criterion is that the solvent should dissolve very little, or none of the compound at low temperatures. The third criterion is that the solvent should have different solubility with the impurities and the compound. The fourth and fifth criterion states that the solvent should have a low boiling point and that it should be lower than the melting point of the compound.

The last criterion acquired from the experiment is that the solvent should be inert. In the first part of the experiment, we determined the most appropriate solvent for recrystallization of the compounds by checking whether the compound is insoluble in a cold solvent, and soluble when dipped in hot water bath. The results in Table 1. 2 are based on the data of Tables 1. 1, we chose such solvents because the compounds did not dissolve in the solvent at a low temperature, and it did at high temperature.

Using an appropriate solvent for recrystallization is necessary because it can save you time, moneyand other materials. Time because you are already sure that the solvent would be safe to use in dissolving the compound at certain temperatures, you wouldn’t need to experiment anymore. For the second part of the experiment, the recrystallization of impure acetanilide, we were conducted to separate the impurities of the acetanilide by using the

recrystallization process and by using water as the agent or the solvent for recrystallization.

During recrystallization, minimum amount of solvent is used to dissolve the solute. This is important because if the amount of solvent exceeded the volume needed to dissolve the solute, recrystallization would not be possible anymore. A fluted filter paper was used in the hot filtration set up because it has a larger surface area to catch the solid impurities, making it easier and more effective to separate the residues from the filtrate in the process.

A stemless funnel was used during the hot filtration set up because unlike a regular funnel, the stem where the filtrate would pass could recrystallize the solid immediately within its stem. If the funnel were stemless the filtrate would go straight down to the flask without crystalizing and compounds. The solution was not placed in an ice cold bath immediately after the hot filtration because the flask might have broke due to a drop in temperature. Vacuum filtration is used during the cold filtration step because it is asier to filter out the crystalized solid from the liquid since cold temperatures would already recrystallize the solid compounds. The cold crystals are washed with water to remove any impurities that are in the crystals. The main function of the activated carbon was to absorb any impurities in the water without reacting with it. Cooling the filtrate on an ice bath makes the recrystallization process faster because of the relatively low temperature. Based on the results on table 2, the percentage recovery was 29. 3%, which seems fairly low since 70. 37% of the crude acetanilide is impurities. Human error might have caused it to be that low and maybe some crystals might have been left in the apparatus such as flasks. CONCLUSIONS AND RECOMMENDATIONS

This experiment proves that the characteristics of choosing an ideal solvent for recrystallization are reliable and important. In the first part of the experiment, which had an objective of identifying the appropriate solvent for the recrystallization technique was achieved.

The second objective, which was to use the recrystallization technique in purifying a solid sample, was also achieved. I would like to recommend that future researchers experiment on other solutes and solvents, preferably common ones in order to determine which solvents are most appropriate for recrystallization. An understanding of these compounds can be better obtained. REFERENCES 1. Baluyut, J. Y. G. , and De Castro, K. A. , Organic Chemistry Laboratory Manual For Chemistry Students Part1. 2. Klein, D. , (2011) Organic Chemistry, 1st Ed. , Cold United States of America