Recrystallization of benzoic acid

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The objective of this experiment was to observe multi-step purification of benzoic acid after performing the extraction from a mixture containing benzoic acid, cellulose, and methyl orange.

Recrystallization was done to remove impurities from the sample. The amount of BA recovered during recrystallization is much less than the extracted amount of BA. The difference between the pure and impure samples was observed by comparison of melting points. It was found that impure samples will have a lower and wider melting point range. The experiment performed was important because it provides important knowledge about the chemical nature and reactivity’s of various compounds that can be used in everyday life. Introduction Now that the desired compound is extracted, it still contains impurities. These impurities can be removed through a process called recrystallization.

Recrystallization is a method for purifying solid compounds which are frequently the products of organic reactions. This theory is based on three important concepts. The first is that a compound is always more soluble in hot solvents than in cold solvents. Secondly, molecules have unique solubility properties. Lastly, a growing crystal will only accept similar entities into its lattice. This is related to the crystal lattice theory which states crystal formation is anti-entropic based on the equation ? G =? H-T? S. There are three stages of solubility: collision, dissociation, and solvation.

In order to raise the probability of collision between the solute and solvent, heat is added to the system. When this occurs, the solute will attack the crystalline structure of the solute and start dissociation. As dissociation

continues until all the molecules are free, solvation occurs. Choosing the right solvent is important when considering recrystallization. The solute must have a high temperature coefficient in the solvent: it must be soluble at high temperatures and insoluble at low temperatures, satisfying the first principle stated above. Also the boiling point of the solvent must be lower than the melting point of the solute. This will ensure that the solute dissolves in the solvent and does not melt.

The solvent must also be inert to prevent chemical interactions with the solute. Impurities must either be readily dissolved in the solvent or be insoluble. The solvent must also be volatile, and finally, it would be beneficial to the experimenter if the solvent is relatively inexpensive. Once the color and most impurities are removed, the crystals can be formed in the solution through a process called nucleation. Nucleation can be induced by slowly cooling the solution to room temperature. It is important to do this slowly so that small solute resembling impurities do not enter the crystal lattice, as described by the third principle of recrystallization. Chemists often add already pure crystals of the solute to the solution in order to provide a pre-formed lattice for other molecules to enter.

This is called seeding. Scratching the glass container in which the solution is contained with a glass stir rod will also induce nucleation on microscopic glass particles. The concept of scratching is similar to that of seeding. Finally, nucleation can be induced by cooling the solution farther in an ice bath; however, this is only a last resort option due to its least efficiency to bring forth crystals. The crystallized solute can then be collected by vacuum

filtration. There is a difference observed in the physical properties of the pure compound and the impure mixture. One of the tests of purity is melting point.

Melting point is an intrinsic, or intensive, property; the value is independent of the quantity of the substance. Melting point is the temperature at which a substance changes physical state from solid to liquid. Because the substance will have a certain shape and surface area exposure, there will be a time interval in which the melting occurs. Thus, it is more accurate to refer to this as melting range. Melting range encompasses the temperature at which the first particles can be seen turning from solid to liquid until all the sample is in the liquid state. Purer samples have smaller melting ranges. Perfectly pure compounds will have ranges of only 1-2°C.

Impurities lower melting point below literature values and also widen the range between which melting occurs. New techniques and equipment used were used during both parts of the purification process. Activated charcoal was used to adsorb impure colored products. Gravity filtration was used to remove insoluble products which were impurities. Fluted filter paper was used to catch more of the impurities. A heating mantel was introduced during recrystallization. It is a heat source used for solutions.

For determining the melting point of benzoic acid, a Mel-Temp apparatus was use. It is used for determining when a small amount of a solid begins to melt and when the melting ends while recording the temperatures, respectively. Experimental: Recrystallization Lab: Initially, took a small amount of impure BA to the side for following week’s lab. The impure BA

crystals were weighed, using an analytical balance, and then placed in a 250 mL Erlenmeyer flask. 200 mL of water was boiled (with boiling stones) in a 250 mL round bottom flask using a heating mantel. The boiled water was added slowly to the benzoic acid crystals. Only enough water was added to dissolve the BA crystals.

For the rest of the lab the Erlenmeyer flask was kept on a steam bath. Charcoal was added to the BA solution and then gravity filtered using a funnel with fluted filter paper. The filtrate was collected in a 250 mL Erlenmeyer flask. If color in the BA solution still remained then charcoal should be added again. The BA solution was removed from the steam bath and cooled, preferably on a window sill. If crystals did not form while cooling on window sill, use another method to induce nucleation to the start the formation of crystals. The crystals were collects by vacuum filtration and placed on a watch glass to dry.

Melting Point of Benzoic Acid: A melting point tube was loaded with BA crystals by inverting the tube into the BA crystals on the watch glass. The tube was packed by dropping through a funnel. The Mel-Temp apparatus was used to determine the melting point range. A rapid melting range experiment was done first and then a slow melting range experiment was carried out to get a better range. Results: Recrystallization lab: Observations: Not much charcoal was used to adsorb the colored molecules The BA crystals were small, shiny, flaky, and white. Almost comparable to texture of dandruff. Data: Mass of extracted Benzoic Acid = 2.

3 g Mass of recrystallized Benzoic Acid = 1. 84 g Calculations: Percent (%) recovery =(weight after recrystalization)/(mass crude BA) ? 100 Percent (%) recovery =(1. 84 g)/(2. 43 g) ? 100= 75. 72% Melting Point lab: Observations: Once the benzoic acid began to melt, it happened very quickly. It was hard was difficult to watch the melting and recording the start and end temperature. The crude BA had a lower and wider melting point range.

Data: Melting Point Ranges RunCrude (Impure) Benzoic Acid (°C)Purified Benzoic Acid (°C) Fast 110. 2-118. 4 at 4. 5 selectivity (Range: 8. 2)115. 4-118. 5 at 4 selectivity(Range: 3.

) Slow113. 2-117. 3 selectivity (Range: 4. 1)117. 5-120. 0 selectivity (Range: 2. 5) Discussion ; The goal of recrystallization and determining melting point labs were to explore the techniques used to obtain pure samples from desired compounds.

After the extraction the goal was to recrystallize the benzoic acid and determine the percent recovery. Then the goal was to determine the melting point range of the recrystallized benzoic acid and compare it to the melting range of the small sample of the impure benzoic acid. 1. 84 grams of pure benzoic acid was obtained, which was calculated to be 75. 2% from the 2. 43 grams of impure benzoic acid. The percent recovery from the impure sample shows that about three fourths of the sample was able to be collected as pure benzoic acid, and that 0.

59 g of the impure benzoic acid was impurities. From this the experimenter can infer that the pure benzoic acid accounted for most of the mass or that little benzoic acid was lost with the removal of the impurities. Also, there is always a chance that some of the benzoic acid may have remained in the solution instead of crystallizing. Also, using an ice-bath to induce nucleation could have lowered my results or recrystallization because it was the last resort to crystalize the solute. Maybe adding too much charcoal could have adsorbed some of the solute. The charcoal was used to adsorb any of the cellulose (which is the reason for the color) that was left behind. Not too much charcoal was actually used; however, my benzoic acid solution did not have much color so charcoal may not have been needed.

Note that the initial value for percent recovery is slightly less than the extracted mass because of the impure benzoic acid set aside for the melting point comparison. The total composition from the very first starting benzoic acid mixture is only 44. 2% (1. 84 grams / 4. 17 grams). Considering practical purposes of recrystallization, one comes to realize how important it is to make this processes as efficient as possible to savemoneyand gain as much product as possible, for example with ibuprofen purification for the pharmaceutical industry. The purity of the sample is shown by the melting range.

As expected, the pure benzoic acid had the smallest melting point range of 2. 5°C. From this, one can tell that this sample was not perfectly pure because pure compounds have melting point ranges of less than 1°C. There may have been some impurities left in the sample since activated charcoal

was not used for this sample. Human error while scraping the impure mixture from the funnel and also from the Erlenmeyer flask would result in a decreased yield. Also, the nucleation may have had some impurity particles collect in the lattice when recrystallizing. After performing the rapid and slow experiment, the temperatures (especially for the pure BA) were not similar.

This was probably due to what is called thermometer lag. If the Mel-Temp apparatus' selectivity was higher than the temperature readings were not as accurate as the slow experiment. The actual value for the melting range coincides with the literature value of 122. 4°C given in the reagent table. The impure sample, however, had a significantly larger melting range of 4. 1°C. Also, the impure sample had a lower temperature.

This is because impurities disrupt lattice forces and decrease the energy required to break the intermolecular bonds between benzoic acid molecules. One way that this experiment could have been improved is to have more space to perform the various steps in the lab. A lot of the mistakes and errors committed (i. e. pillage) were due to crowding of various equipment that were closely packed next to each other. Overall, theses labs were a success be the experimenter was able to achieve thegoalsof each lab. The benzoic acid was able to recrystallize and the melting point was fairly close to the literature value even with possible errors performed in lab.

Works Cited Wikipedia: The Free Encyclopedia. Wikimedia Foundation, Inc. 22 July 2004. Thurs. 7 Oct. 2010. Zubrick, James.

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