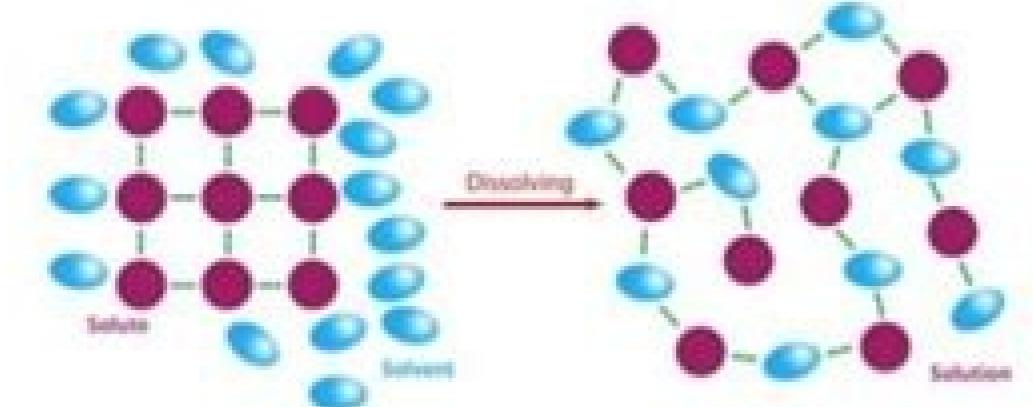


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the mp of A unless of course the unknown is A.¹³ If the two mixed samples were different, the melting point would have been lower. Compounds have unique melting ranges due to the varying intermolecular forces between the atoms. As the forces increase in strength, the boiling point also increases.⁷ Another common mistake in recrystallization is heating the compound too quickly when taking its melting point so ramp rates of 5°C and 2°C were used.¹⁴ Sagging can also occur before melting. It gives a false lower end of the melting range because the crystalline structure sags but there is not actually any liquid yet. The lower end of the melting range should only be recorded when liquid forms.¹⁵

In part 4 of the experiment, the goal was to recrystallize an impure sample of p-dibromobenzene. The contaminant was successfully separated from the product. An orange impurity was left behind and a white product was obtained using an ethanol-water solvent. Incorrectly pairing the mixed solvents could occur as a common mistake in this process. Crystallization occurred using this solvent. This proved that a mixed solvent could be successfully used for recrystallization. It is known that, "compounds having similar structural features will be soluble in one another". Examples of these features are polarity and the interaction of forces.¹⁶ This is demonstrated in the images below (Figure 1, Figure 2). Using ethanol by itself as a solvent for p-dibromobenzene would not work because the substance was soluble in cold and hot ethanol. The use of water by itself as a solvent for p-Dibromobenzene would not work as well because the compound had a low solubility in hot and cold water. Kathleen Armstrong of Foothill College Chemistry stated that, "When a suitable solvent cannot be found, search instead for two miscible solvents that together provide the appropriate solubility properties."⁶ Ethanol and water were capable of being mixed together. A certain mixture of these two caused the compound to dissolve when heated but be insoluble when cold.

Figure 1



*Demonstration of solubility

Procedure for recrystallization of benzoic acid. Introduction of benzoic acid. Purpose of recrystallization of benzoic acid.

This PaperA short summary of this paper& Full PDF's related to this paperDownloadPDF Pack Recrystallization (To separate benzoic acid and acetanilide impurities by the technique recrystallization and to calculate the percent recovery of benzoic acid and acetanilide after recrystallization.) TheoryAs chromatography, recrystallization is a technique that is used to purify solid compounds. It is the process of a crystalline solute being dissolved in a hot solvent and then returned to its solid state when cooled in a solvent by crystallizing. Crystal formation is a selective process. During recrystallization, the solute dissolved in a hot solvent destroying impurities. Once the heat has been given to it, it is cooled in a cold solvent selectively producing pure crystals. The size of the crystals determines how pure the compound is as larger crystals are purer than smaller crystals. The factor in recrystallization is polarity. The solute should have maximum solubility in the hot solvent and minimum solubility in the cold one. The solvent used to dissolve the compound being used should have a similar polarity. For the higher polarity compounds, the solvents typically used are ethanol and methanol but non-ionic compounds generally dissolve in water. MaterialsWater/Charcoal/Acetanilide (C 8H 9NO) Benzoic Acid (C 7H 6O 2) Digital Melting Point Apparatus/Capillary Tubes/Hot Plate/Polymer Funnel/Weighing Paper/Vacuum Tubing/Buchner Filter/Erlenmeyer Flasks/Thermometer/Procedure 1. 2 g of crude benzoic acid, 2 spatulas of activated charcoal, 3 boiling chips, and 30 mL of distilled water weighed and placed into a 125 mL Erlenmeyer flask.2. Benzoic Acid is heated until dissolved in 50 mL of distilled water was heated on a hot plate to 60°C. The ceramic funnel was rinsed with a blow dry and the weighed filter paper was placed into the funnel and saturated with hot water. The hot solution was then filtered. The benzoic acid solution was heated and filtered again to remove the remaining impurities and was allowed to cool to room temperature.6. The Solution was then cooled in an ice bath. Vacuum filtered through the Buchner funnel. Crystals remaining in the funnel are washed out and placed on weighed filter paper. The filtered crystals were dried to dry weight and were allowed to cool to room temperature.7. Add to the 125-mL Erlenmeyer flask with 2 g of crude acetanilide, 2 spatulas of activated charcoal, 3 boiling chips, and 30 mL of distilled water was weighed and added to the flask. The above process is repeated with acetanilide. Purpose/introduction The process of recrystallization is an important method of purifying a solid organic substance using a hot solution as a solvent. This method will allow the separation of impurities. We will analyze Benzoic Acid as it is dissolved and recrystallized in water and in a solvent of Methanol and water. Reaction/Summary In Experiment One we will be recrystallizing Benzoic Acid from water. In Experiment Two we will be recrystallizing Benzoic Acid using a solvent pair made up of Methanol and Water. The Seven step process of recrystallization consists of adding a solid organic substance into a solvent, then dissolving the chosen solute, decolorizing the solution, filtering solids, then recrystallizing the solute by slowly cooling...show more content...It is faster due to the filter funnels surface area. Results/Observations Experimental data resulted as expected because it was found that on experiment one, Benzoic Acid could recrystallize with a better recovery percentage than the solvent pair in experiment two. The mass recovered in experiment one was 0.048g while experiment two had a mass recovery of 0.045g. Solvent(s) Used Mass of "Crude" (g) Mass of Recovered (g) Amount of Solvent Used (mL) Percent Recovered (%) Experiment One Water (H2O) 0.051 0.048 1.049.1 Experiment Two Methanol (MeOH) And Water (H2O) 0.049 0.045 Methanol – 1.5 Water – 0.5 9.92 Conclusion Both experiments were of fair solubility, but in the case of recrystallization of Benzoic Acid, Water was the best solvent to recrystallize acid the most. Experimental data determined that there was a difference of .003g between using the single solvent in comparison to the paired solvent. Data suggests that Water is the best solvent that will allow for better saturation and the best recrystallization of benzoic acid, this is largely due to water being a polar molecule whose properties allow for carboxylic acid groups, such as the one found in benzoic acid, to disassociate and donate protons to the water...show more content...You are given a known solid substance with its melting point which has high solubility in hot water and low solubility in cold water. You are assigned the following task: i. Determine if the substance is pure ii. If impure, find a suitable method for its purification by using the given information about the substance How would you proceed, clearly explain your rationale? i. To find if it is a pure substance we see if there is a change in melting point. ii. If impure, perform recrystallization procedure to remove the impurities. Then calculate Percent Recovered on crystals formed, and perform melting point procedure. 2. You find that a solid substance you are trying to purify is very soluble in ethanol, but not very soluble in water. You decide that you are going to try to recrystallize it from a solvent pair consisting of ethanol and water; is this decision based on solid rationale? Comment briefly. 3. The use of ethanol and water in a solvent pair is perfect do due to ethanol having a high solubility while the water has low 1. CHEM 2423 Recrystallization of Benzoic Acid Dr. Pahlavan 1 EXPERIMENT 4 - Purification - Recrystallization of Benzoic acid Purpose: a) To purify samples of organic compounds that are solids at room temperature b) To dissociate the impure sample in the minimum amount of an appropriate hot solvent Equipment / Materials: hot plate 125-mL Erlenmeyer flask ice stirring rod spatula Büchner funnel impure benzoic acid weighing paper digital scales rubber tubing (hose) benzoic acid boiling stones (chips) filter paper 25 mL graduated cylinder 50 mL beaker Mel-temp apparatus Discussion: The products of chemical reactions can be impure. Purification of your products must be performed to remove by-products and impurities. Liquids are customarily purified by distillation, while solids are purified by recrystallization (sometimes called simply "crystallization"). Recrystallization is a method of purifying a solid. There are two types of impurities: those more soluble in a given solvent than the main component and those less soluble. (If there are any impurities that have the same solubility as the main component, then a different solvent needs to be chosen.) When organic substances are synthesized in the laboratory or isolated from plants, they will obviously contain impurities. Several techniques for purifying these compounds have been developed. The most basic of these techniques for the purification of organic solids is recrystallization, which relies on the different solubilities of solutes in a solvent. Compounds, which are less soluble, will crystallize first. The crystallization process itself helps in the purification because as the crystals form, they select the correct molecules, which fit into the crystal lattice and ignore the wrong molecules. This is of course not a perfect process, but it does increase the purity of the final product. The solubility of the compound in the solvent used for recrystallization is important. In the ideal case, the solvent would completely dissolve the compound to be purified at high temperature, usually the boiling point of the solvent, and the compound would be completely insoluble in that solvent at room temperature or at zero °C. In addition the impurity either would be completely insoluble in the particular solvent at the high temperature, or would be very soluble in the solvent at low temperature. In the former case, the impurity could be filtered off at high temperature, while in the latter case the impurity would completely stay in solution upon cooling. In the real world, this will never happen and recrystallization is a technique that has to be practiced and perfected. Regardless of crystallization method, the purity of the solid can be verified by taking the melting point. A good (suitable) recrystallization solvent will dissolve a large amount of the impure compound at temperatures near the boiling point of the solvent. Small amount of compound being purified should remain in solution at low temperatures, between approximately 25 and 5 °C. Low solubility at low temperatures minimizes the amount of purified compound that will lose during recrystallization. 2. CHEM 2423 Recrystallization of Benzoic Acid Dr. Pahlavan 2 A suitable recrystallization solvent should also be partially volatile in order to be easily removed from the purified crystals. The solvent should not react with the compound being purified and it should have the boiling point below the melting point of the compound being purified because solid melts before dissolves (oiling out). In selecting a good recrystallization solvent one should also consider flammability, toxicity, and expense. In selecting a solvent consider that like likes like. Polar compounds dissolve polar compounds and non-polar compounds dissolve non-polar compounds. The most commonly used recrystallization solvents are presented in the following table. solvent formula/polarity boiling point (O) C) water H2O very polar 100 ethanol CH3CH2OH polar 78 methanol CH3OH polar 65 dichloromethane CH2Cl2 slightly polar 40 diethyl ether (CH3CH2)2O slightly polar 35 Organic compounds with one polar functional group and a low number of carbon atoms such as methanol, ethanol, and n-propanol are highly soluble (miscible) in water. These alcohols form hydrogen bond with water due to the polar -OH functional group. As the number of carbons per polar functional group increase, solubility decreases. The solubility of alcohols with four to five carbons is given in the following table. alcohol formula/Solubility (g/g) 100 mL H2O) n-butanol CH3CH2CH2CH2OH 2-n-pentanol CH3CH2CH2CH2CH2CH2OH 0.5 n-pentanol CH3CH2CH2CH2CH2CH2CH2OH 0.5 Compounds with six or more carbons for each polar group will not be very soluble in polar solvents but will be soluble in non-polar solvents such as benzene and cyclohexane. If a single solvent cannot be found that is suitable for recrystallization, a solvent pair often used. The solvents must be miscible in one another. Some commonly used solvent pairs are water-ethanol, acetic acid - water, ether-acetone. Typically, the compound being recrystallized will be more soluble in one solvent than the other. The compound is dissolved in a minimum amount of the hot solvent in which it is more soluble. The following formulas used in solubility problems. % lost in cold solvent = (solubility in cold solvent/solubility in hot solvent) x100 % recovery of solid = (g solid) - (g solid lost) / (solid recovered) x 100 / g (solid) = (4.52/5.00) x100 = 90.4 % Example (2) - The solubility of compound "X" in ethanol is 0.80 g per 100 mL at 0 °C and 5.00 g per 100 mL at 78 °C. What is the minimum amount of ethanol needed to recrystallize a 12.00 g sample? What would be the maximum theoretical percent recovery from crystallization of 5.00 g of solid "X" from 100 mL of water? Assuming the solution is chilled at 0 °C. 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