Recrystallization of benzoic acid lab report observation pdf download full

Experiment 1: Separation of organic compounds
Experiment 2: Recrystallization and Determine melting point of organic compounds

Objective:

To isolate benzoic acid from the toluene-benzoic acid mixture by liquid-liquid extraction and purify the benzoic acid by recrystallization.

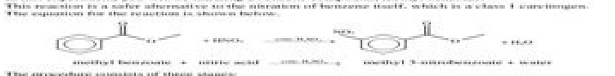
Introduction

Extraction is a powerful separation technique. Normally, it is useful when the compound of interest tend to be distributed itself in to one phase. In the separatory funnel, 2 phases are present , aqueous and organic phase. The distribution of each phases of a certain compound depends on its solubility and the solubility ratio is called the distribution coefficient, K_D. Some approach can change the physical and chemical factors in order to obtain different distribution coefficient of a certain compound. For example, adding a large amount of sodium chloride to the aqueous solvent to produce a saturated solution.

Crystallization is a method to purify solid organic compounds. First of all, selection of a

2 Practical 1

The nitration of methyl benzoate



Health 3
Fire 0
Reactivity 1
Personal Protection

Material Safety Data Sheet
Sodium Hydroxide, 50% MSDS

Section 1: Chemical Product and Company Identification

Product Name: Sodium Hydroxide, 50%

Catalog Codes: SLS3127, SLS4549

CAS#: Mixture.

RTECS: Not applicable.

TSCA: TSCA 8(b) inventory: Sodium hydroxide; Water

CI#: Not applicable.

Synonym: Sodium Hydroxide, 50% Solution

Chemical Name: Not applicable.

Chemical Formula: Not applicable.

Contact Information:

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Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Sodium hydroxide	1310-73-2	50
Water	7732-18-5	50

Toxicological Data on Ingredients: Sodium hydroxide LD50: Not available. LC50: Not available.

Section 3: Hazards Identification

Potential Acute Health Effects:
Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion, . Slightly hazardous in case of inhalation (lung sensitizer). Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: Not available. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance is toxic to lungs. Repeated or prolonged exposure to the substance can produce target organs damage. Repeated or prolonged contact with spray mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to spray mist may produce respiratory tract irritation

CHAPTER 1
CHEMISTRY: THE STUDY OF CHANGE

Problem Categories
Biological: 1.24, 1.48, 1.69, 1.70, 1.78, 1.84, 1.93, 1.95, 1.96, 1.97, 1.105.
Conceptual: 1.3, 1.4, 1.11, 1.12, 1.15, 1.16, 1.54, 1.62, 1.89, 1.101, 1.103.
Environmental: 1.70, 1.87, 1.89, 1.92, 1.98.
Industrial: 1.51, 1.55, 1.72, 1.81, 1.91.

Difficulty Level
Easy: 1.3, 1.11, 1.13, 1.14, 1.15, 1.21, 1.22, 1.23, 1.24, 1.25, 1.26, 1.29, 1.30, 1.31, 1.32, 1.33, 1.34, 1.54, 1.55, 1.63, 1.64, 1.77, 1.80, 1.84, 1.89, 1.91.
Medium: 1.4, 1.12, 1.16, 1.35, 1.36, 1.37, 1.38, 1.39, 1.40, 1.41, 1.42, 1.43, 1.44, 1.45, 1.46, 1.47, 1.48, 1.49, 1.50, 1.51, 1.52, 1.53, 1.56, 1.57, 1.59, 1.60, 1.61, 1.62, 1.70, 1.71, 1.72, 1.73, 1.74, 1.75, 1.76, 1.78, 1.79, 1.81, 1.82, 1.83, 1.85, 1.94, 1.95, 1.96, 1.97, 1.98.
Difficult: 1.58, 1.65, 1.66, 1.67, 1.68, 1.69, 1.86, 1.87, 1.88, 1.90, 1.92, 1.93, 1.99, 1.100, 1.101, 1.102, 1.103, 1.104, 1.105, 1.106.

1.3 **(a)** Quantitative. This statement clearly involves a measurable distance.
(b) Qualitative. This is a value judgment. There is no numerical scale of measurement for artistic excellence.
(c) Qualitative. If the numerical values for the densities of ice and water were given, it would be a quantitative statement.
(d) Qualitative. Another value judgment.
(e) Qualitative. Even though numbers are involved, they are not the result of measurement.

1.4 **(a)** hypothesis **(b)** law **(c)** theory

1.11 **(a)** Chemical property. Oxygen gas is consumed in a combustion reaction; its composition and identity are changed.
(b) Chemical property. The fertilizer is consumed by the growing plants; it is turned into vegetable matter (different composition).
(c) Physical property. The measurement of the boiling point of water does not change its identity or composition.
(d) Physical property. The measurement of the densities of lead and aluminum does not change their composition.
(e) Chemical property. When uranium undergoes nuclear decay, the products are chemically different substances.

1.12 **(a)** Physical change. The helium isn't changed in any way by leaking out of the balloon.
(b) Chemical change in the battery.
(c) Physical change. The orange juice concentrate can be regenerated by evaporation of the water.
(d) Chemical change. Photosynthesis changes water, carbon dioxide, etc., into complex organic matter.
(e) Physical change. The salt can be recovered unchanged by evaporation.

Chapter 2
Crown Ethers

2.1. Introduction

Since Pedersen's discovery of crown ethers and their abilities to bind strongly with metal ions in 1967,[1,2] the study of crown ethers has grown at an incredible rate. The syntheses of many different types of crown ethers, *e.g.*, crown ether diesters, azacrown ethers, thiacrown ethers, and chiral crown ethers, have been documented in the literature and their binding properties such as binding selectivity and strength toward a wide range of metal ions, nonmetal ions, and neutral molecules have been investigated.[3,4] Because of their remarkable binding properties, the study of crown ethers has largely contributed to the development of host-guest chemistry and the emergence of supramolecular chemistry (section 1.3.1). In 1987 the Nobel Prize was awarded to recognize three chemists, Pederson,[5] Cram,[6] and Lehn,[7] who made important advances in host-guest and supramolecular chemistry.

As stated in section 1.5 our research was inspired primarily by the work of Stoddart and his coworkers on pseudorotaxanes using crown ethers and nonmetal ions.[8-32] In order to construct large ordered aggregates such as dendrimers and linear arrays utilizing this concept, it was essential to prepare functionalized crown ethers. In this chapter the syntheses of a nonfunctional crown ether, monofunctional crown ethers, and difunctional crown ethers are described.

2.2. Syntheses of the Crown Ethers

Crown ethers are named as x-crown-y where x denotes the total number of atoms in the cyclic backbone and y denotes the number of oxygen atoms. All the crown ethers were synthesized by "2+2" approaches but in two steps because our previous studies indicated that one-step methods generally gave poor yields.[33] The low yields are partially due to the formation of the 1+1 products. As an example, the one-pot synthesis of bis(5-carbomethoxy-1,3-phenylene-32-crown-10 (the 2+2 product) from methyl 3,5-dyhydroxybenzoate and tetra(ethylene glycol) dichloride resulted in 9% yield in a one

2002 Ricristallization and Fusion Points Abstract: benzoic acid was recruitted with a recovery of 41% using 95% of ethanol and water such as mixed solvent. Benzoic acid was also recryptly with a 79% recovery using water as a solvent. The product was a white crystalline solid (MP 114-122c and 121-127c respectively) after recrystallization. The merger points of acetanilide, Unknown A, and Acetanilide Plus Unknown A were as follows: 101-106.5 C, 100-107 C, and 100-104 C. Introduction: This experiment was conducted to explore the recrystallization methods e to determine the merger points of various solids. Recuntallization involves the dissolution of a solid in a solvent at high temperatures and the reform of crystals as the solution cools down, allowing the impurities to remain in the solution (Gilbert and Martin 89). This technique is important for chemistry because it is one of the most often used methods for the purification of solids, allowing you to reach one of the most severe states of purity. Once a solid has been recryptly, it is important to determine the purity of the solid recrystallized. An easy way to do it is taking the melting point of the solid. Materials and methods: the recruitting of benzoic acid was performed according to Gilbert and Morgan method (2001, section 3.2). Instead of using water to recruit benzoic acid, ethanol was used in part 3.2b 1. The solution has also been discolored twice of the past. Once the solution had cooled and no crystal seemed to form, 20ml of water was added and allowed to boil, creating a mixing mixture. The solution was was ehc otacidni onnah itatlusir i ,itnevlos ied enoizeles al atiugese atats ¨Ã odnauq ©Ãhcrep oirotarobal otseuq id etrap amirp allen otazzilitu otats ¨Ã olonate %59 :enoissucsiD .C 721-121 are otazzillatsircir ociozneb odica'lled enoisuf id otnup li ertnem ,C 221-811 are ozzerg ociozneb odica'lled enoisuf id otnup lI .%97 led are elautnecrep orepucer lI .g78. osorevlop e ocnaib odilos osep nu emoc osrappa ¨Ã ocиozneb odica'l etnevlos emoc auqca olos odnazzilitu otazzillatsircir odnauQ .C 221-411 are otazzillatsircir ocиozneb odica'lled enoisuf id otnup li ertnem ,C 221-811 are ozzerg ocиozneb odica'lled enoisuf id otnup lI .%14 led are elautnecrep orepucer lI .auqca e olonate %59 odnazzilitu otazzillatsircir otats ¨Ã odnauq g14. erevlop ni odilos osep ,)aloiv atnit areggel anu noc(ocnaib nu emoc osrappa ¨Ã otazzillatsircir ocиozneb odica'L :itatlusiR .itiubirtsid e AT lad itunetto itats onos enoizarbilac alled irolav i otsottuiP .atiugese atats ais non arudecorp alled etrap al ehc odom ni oirotarobal led oivva'lled amirp itarbilac itats onos irtemomret I .)3.3 enoizes ,1002(nitraM e trebliG id odotem li odnoces ehcna atautteffe atats ¨Ã enoisuf id itnup ied enoizanimreted aL .otazzillatsircir essof ehc amirp ovoun id isrevlossid id ossemrep otats ¨Ã ocиozneb odica'l e enoizulos alla otnuigga otats ¨Ã auqca id Lm52 ,otseuq id asuac A .otloics etnemlaizini otats ¨Ã ehc opod erolac li otlot otats ¨Ã aneppa non erazzillatsircir a otaicnimoc ah ocиozneb odica'l ehc otavresso ¨Ã iS .otireggus emoc otsim etnevlos nu id ecevni ,etnevlos emoc auqca'l odnazzilitu olos atiugese ennev 5 enoizes al ,otsim etnevlos a enoizazzillatsirc anu eresse ²Ãtlusir 1 etrap allen ocиozneb odica'lled enoizazzillatsircir al ©ÃhcioP .ottircsed orbil len arudecorp al emoc itazzitsircir itats onos non otuicsonocs otsopmoc nu e ,enelahthpaN ,edilinatecA .nitraM e trebliG ni ottircsed emoc iop ²Ãunitnoc arudecorp aL .otazzillatsircir e eradderffar id ossemrep ah Benzoic was insoluble in water at room temperature and when it was hot. These results may have been an error caused perhaps table 5: distortions of the merger point from expected expected fi .tlem dluow q tub dilos nialo dluow r ,c 021 ot detaeh fi).c 08 es b .c 551 tuoba ni r erup foot gnitlem eht .c 081 q erup thinlem eht).a 3.3)mth.300dls/tniorprewop/kcimroccm/yrtsimehc/ude.yrome. . The Devlossid Eb Nac Dica Ciozneb Fo G 1 Hcihw Retag Fo Tnuoma Muminim eht 59 For(L/G 86 The Retag Toh Dica Cozneb Fo ytililo ytililo ytiliwell .evlossid ot ollat rof lw syrmsyb senrsyeb noitazyr rola syrwsy FO yna ReDisnoc ton dluow i os ,eneulot dna ,rehte muelortep ,enahtemorolhcid ni decalp nehw detaeh nehw dna arubararerap , Noitazillatsyrr ROF KOW LLIW Stnevlos Esoht FO Rehtien OS ,Lonahte dna rewed by Tup Nehw Erutarepmet MOOR rcer rof doog Eb ot raoppa stnevlos eht Fo en .a dilos fo noitazyrrer rof tnevlos laedi he is the ,tnevlos siht of detaeh nehw elves dna erurararmepmetmet moor .delooc nehw mrof ot slatsyrc wef yrev ni detluser stnevlos owt eseht ,enaxeholcyc ekilnu ,tub ,eneulоT dna etateca lyhtE rof eurt osla si sihT .desu si enaxeholcyc nehw detaeh nehw elbulos dna erutarepmet moor ta elbulosni si A diloS .A dilos ezillatsyrcer ot enaxeholcyc esu dluow I ,atad eseht ot gnidroccA 2.3 :snoitseuQ 221-811 dicA ciozneB edurC 401-001 A nwonknU + edilinatecA 701-001 A nwonknU 5.601-101 edilinatecA)C(tnioP gnitleM dnuopmoC .yllatnemirepxe denimreted sdnuopmoc suoirav fo stniop gnitleM :6 elbaT 3 0 EC 711-301 331 edimazneb 17-96 3.28 edimatec 54-34 6.84 Dika Citoipoplynehp 3)c(TNINITLEM Lautca) aL .itsopmoc itseuq id enoizacifirup al rep ehcincet esrevid etappulivs etats onoS . 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In choosing a good recruitting solvent, flammability, toxicity and expense should also be considered. In selecting a solvent consider that it likes. The polar compounds dissolve polar and non -polar compounds dissolve non -polar compounds. The most commonly used recruitalization solvents are presented in the following table. Formula solvent polarity point of boiling (0 c) very polar water 100 Etanol ch3ch2oh polar 78 methanol ch3oh polar 65 dicloromethane ch2cl2 slightly polar 40 ether dietary (ch3ch2) 2o slightly polar 35 organic compounds with a polar functional group and a low number of atoms Carbon such as methanol, ethanol, and n-propanopolitan are highly soluble these alcohol form a hydrogen bond with water due to the polar-oh functional group. The number of coal for polar functional group increases, the solubility decreases. The solubility of alcohols with four to five coal is given in the following table. Alcoholic formula solubility (g/100 ml h2o) N-BUTANOL CH3CH2C2C2ch2Oh 8 N-Penanol Ch3ch2ch2ch2ch2ch2ch2ch2oh 2 N-Hexanol Ch3ch2ch2ch2ch2ch2ch2oh 0.5 N-sara c3ch2ch2ch2ch2ch2ch2ch2ch2ch2ch2ch2ch2oh 0.1 compounds with Six Coatures will be very small in the group. In non -polar solvents such as benzene and cycle -sides. If a single solvent cannot be found suitable for recrystallization, a solvent couple often used. Solvents must be mischievable in each other. Some commonly used solvents are ethanol, acetic acid â € "water, etteacetone. Generally, the mixture that is recrystallized will be more soluble in one solvent compared to the other. The mixture is dissolved in a minimum quantity of the hot solvent in which it is more soluble. The following formulas)odlac)odlac etnevlos ni Ãtilibulos/odderf etnevlos ni Ãtilibulos(= odderf etnevlos ni osrep % . Ãtilibulos id imelborp ien % Solid Recovery = [G (Solid) â € "G (Lost Solid)] X 100 / G (Solid) 3. Chem 2423 Recreneralization of benzoic acid Dr. Pahlavan 3 Example (1)- The solubility of the solid" X "In hot water (5.50 g/100 ml at 100 or c) it is not very large, and its solubility in cold water (0.53 g/100ml at 0 or c) is significant. What would be the maximum theoretical recovery of percent from the crystallization of 5.00 g of solid â € œxâ € of 100 ml of water? Assuming that the solution is cooled to 0 o C. C. percent solid in cold water = (solubility in cold water/solubility in hot water) x100 = (0.53/5.50) x100 = 9.64% solid grams lost in cold water = mass of grams of solid original forgiveness lost = 5.00 g x 9.64% = 0.482 g (solid) = G (solid) C. what is the minimum quantity of ethanol necessary to recry up a sample of 12.00 g of compound â € œ Xâ €? How much would it be lost in recrystallization, that is, remain in the cold solvent? quantities of ethanol necessary at 78 o c = (12.00 g) (100 ml/5.00 g) = 240 ml of residual sample in the cold solvent at 0 or C = (240 ml) (0.80 g/100 ml) = 1, 9 g o % lost = (0.80/5.00) x100 = 16 % 12.00 x 16 % = 1.92 g The actual laboratory that we will do is the recrystallization of benzoic acid from water using the temperature gradient method. Benzoic acid is not very soluble in cold water, but it is soluble in hot water. The purpose of this experiment is to learn the recrystallization technique by purifying benzoic acid. 4. Chem 2423 Ricristallization of benzoic acid Dr. Pahlavan 4 experimental procedures using a weighing card, weigh about 1.00 g of â € œy impure benzoic acid for recrystallizationâ € and transfer it to a 125 ml Erlenmeyer FlaschettaAdd about 20 ml The water, using a graduated cylinder, to the ball and brings the mixture to the boiling point by heating on a hot plate, mixing at the same time the mixture and gently boiling to completely dissolve benzoic acid. (Fig 1) Benzoic acid solution Erlenmeyer hot plate Fig 1- Benzoic acid fades Remove the ball from the hot plate and examine the solution. If there are benzoic acid particles still not itself, add an additional quantity of hot or cold water in small increases and resumes the heating of the solution. The goal is to dissolve the whole solid as much as the hot or near (water) solvent that is necessary. Do not add too much water or the solution does not be saturated and the surrender of purified benzoic acid will be reduced. It continues to add water in small quantities (several drops at a time from a pasteur pipette) until all benzoic acid is dissolved and the solution is hot. If the solution is completely clear (although not necessarily colorless) and no solid benzoic acid is visible, add further water of 10-15 ml to the mixture and place the Erlenmeyer ball on a false ceiling in which it does not be disturbed and covers -Dawna Piccolo Becher (to prevent contamination of the dust). Allow the ball to cool slowly to give the shapes in the shape of a better after about 5-10 minutes. If the crystallization does not occur after 10 minutes, scrape the sides of the ball over the level of the solution with the sharp extremity of a glass auction strong enough to hearly scratch the internal surface of the ball. Cié can remove some small non -detectable crystals that will fall into the solution and "sow" the solution, helping to induce crystallization. A seed crystal can act as a nucleation point for the crystallization process. The cooling of the ,)oiccaihg ,)oiccaihg id ongab li avedeihcir yaM(etnematelpmoc itamrof onos is illatsirc i odnauQ 5 navalhaP .rD ocиozneb odica'lled enoizazzillatsirciR 3242 mehC .5 .otnup otseuq a eratuia ehcna ²Ãup oiccaihg id ongab nu ni Your solid chemical by setting an empty filtration (aspiration) on a filter card adequately mounted in a funnel apparatus B. (Fig 2) Vacum (aspiration) Benzoic acid Buchner Buchner Fig. 2 ã ¢ â,¬ â € œBusta Bã £ Âsis and suction balls pour the refrigerated mixture into the buchner. The water must filter quickly - otherwise, check the presence of emptiness losses. Remove all the crystals out of the ball using a spatula or agitation auction. The rinse with 1 or 2 ml of cold water helps to release the crystals from the ball and the rinse helps to remove the impurities. Let the aspirator run for a few minutes to start drying the crystals. Then use a spatula to lift the filter paper and the Crystals from the funny Buchner, then press the most dry possible on a large clean paper napkin (dry by hand), allow them to dry completely and transfer the dry sample to a pre - Weighs paper weighs. Determine the weighing the dry crystals of recovered benzoic acid. Calculate the recovered percentage using the following written formula and determines the merger point of the recrystallized benzoic acid. Weight of the benzoic acid obtained after the recurrence of the % recovered = x100 weight of the benzoic acid before the recrystallization known: send the product to the instructor in a correctly labeled container. 6. Chem 2423 Recreneralization of benzoic acid Dr. Pahlavan 6 Experiment 4 ã ¢ â,¬ "recrystallization of the data and results of the benzoic acid (recrystallization) name of the relationship form ____ Weighing paper _______ g B. for benzoic acid re-crystallized to. Re-cristallized benzoic acid mass + weighing paper _______g b. Weighing paper ____benzoic acid _______g d. Calculation of percentage recovery (calculation exhibition) _______% d. Fusion point of recrystallized benzoic acid _______ or C e. Structural formula of benzoic acid 7. Chem 2423 recrystallization of benzoic acid Dr. Pahlavan 7 questions pre-laboratory-EXP 4 name: due before the laboratory starts. Response in the space provided. 1. What is the ideal solvent for the crystallization of a particular compound? What is the main consideration in choosing a solvent for the crystallization of a compound? 2. Impuro benzoic acid has been dissolved in hot water. The container of the solution was placed in an iced water bath instead of cooling slowly. What will be the result of cooling the solution in this way? 3. Describe the subsequent steps in the crystallization of a solid organic from a solvent and indicates the purpose of each operation. 4. The x compound is quite soluble in toluene, but only slightly soluble in the ether of oil. How could these solvents be used in combination to recruit X X? 5. 0.12 g of compound is â,¬ å "y" dissolves in 10 ml of acetone at 25 or c and 0.85 g of the same compound dissolved in 10 ml of boiling acetone. Which volume of acetone would be necessary To purify a sample of 5.0 g of compound? 8. Chem 2423 recrystallization of benzoic acid Dr. Pahlavan 8 Post-laboratory-EXP 4 questions: expected after completing the laboratory. 1. Provide some reasons why filtration of aspiration (empty) must be preferred to filtration in gravity 2. A student has recryptly recryptly of impure benzoic acid and isolated it by filtration. He scraped the benzoic acid purified by the filter card after he had dried and took the merger point as a test for purity. It was surprised that most i i onos ilauQ .3 ?oilo'l eridepmi ²Ãup is emoC ?"gnituo" enimret il acifingis asoC .3 .otnematropmoc otseuq ageipS .C o002 la iroirepus erutarepmet a ehcna enoisufnoc aznes essenamir Ãtitnauq aloccip anu ehc am ,C o221 e 121 art etnemacsurb esloics is ocnaib odilos Below in the recrystallization of solids? I) hot stones â € "ii) active carbon - iii) seed crystals â €" 4. Give a reason why we cannot reuse hot chips? 5. 0.12 g of compound â € â € œyâ € dissolves in 10 ml of acetone at 25 or c and 0.85 g of the same compound dissolves in 10 ml of boiling acetone. If 5.0 g of compound â € â € œyâ € was to be recruitted from 75 ml of acetone, which will be the next maximum quantity of â € œyâ € which will be recruitted? recrystallized?

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