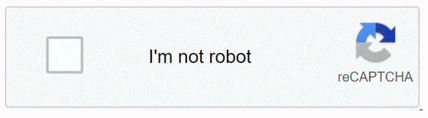

Continue

Chloroform extraction benzoic acid

In this experiment, you use two important organic separation protocols: extractions and recrystallization. You start with a mixture of two different organic chemicals (benzoic acid and m-nitroaniline), along with an inorganic salt (NaCl). We discuss procedures necessary to separate the benzoic acid from the other components in the lab. Once we have isolated the benzoic acid from the other chemicals, we will purify the benzoic acid using recrystallization. Anyone who has been to Hawaii is probably aware of how sugar (sucrose) is prepared from sugar cane. The sugar cane is grown for a period of up to two years before it is ready to be harvested. When the crop is ready, the whole field is burned, killing the plants and burning the leaves, leaving only the moist sugar cane stems. The very dirty stems are collected, taken to the processing plant and then crushed with large amounts of water to be added and the mixture heated. The water dissolves the sugar, leaving behind the dirt, ash and plant solids. The liquid mixture (heterogeneous mixture) contains the dissolved sugar, but it is also muddy and dirty. This whole mixture may cool, crystallizing the sugar, but still producing impure sucrose. After several dissolving (and heating) additional recrystallization steps take place until pure white sucrose produced. In general, the process of recrystallization has been used to remove impurities (which remain in the liquid, called the mother liqueur) and produce pure crystalline sucrose. In addition to recrystallization, organic chemists use extractions to purify chemicals from other chemicals we may not want. Recrystallization is based on the fact that chemicals, when they form crystals, tend to associate themselves with other identical chemicals, hence the eventual production of pure sucrose crystals that are eventually prepared. For solvent extractions, we rely on the ability to separate a chemical from another chemical that uses differences in solubility. Most organic chemicals dissolve easily in organic solvents. For example, both of the organic chemicals used in this experiment (benzoic acid and m-nitroaniline), dissolve in ether (actually diethyl ether CH3CH2-O-CH2CH3), and none of these chemicals dissolve noticeably in water. So, how do we make one of these chemical subdivisions (i.e., preferably dissolve) in the watery phase? We can chemically change one of these chemicals to make it want to dissolve in water, rather than the ether. We're doing this the benzoic acid by neutralizing it when we react it by 10% (~2.5 M) NaOH. This is a simple acid-base reaction. The OH! (hydroxide) ion removes the H+ (proton) ion from the acid, causing the anion of the acid, the benzoate ion. The benzoate ion has now been charged (after removing the acid proton) and to be in the aqueous phase instead of the organic phase. After this reaction, all benzoic acid (which now exists entirely as ion) will be found in the aqueous phase. Once the aqueous phase has been removed from the organic phase you have purified (extracted) the benzoate ion away from the other organic chemical. You should put the benzoate ion back to benzoic acid by adding 6 M HCl (this step reverses the reaction above). Since benzoic acid is almost completely insoluble in water, it will form a precipitation (this is recrystallization). You then insulate the benzoic acid with vacuum filtration in a Büchner funnel. Finally, to characterize your isolated product, perform a melting point analysis on the isolated benzoic acid. The melting point of a chemical is a useful physical constant that can be used to verify chemical identity and purity. However, you need to store your crystallized benzoic acid in the drying oven until the next lab period, because you need completely dry solids to do a melting point analysis. Procedure Part A--Extaction of Benzoic acid Get about 2 grams (you acquire 1.8-2.1 grams, which is okay; make sure you record how much you have up to three decimals) of the chemical mixture that contains benzoic acid, m-nitroaniline and NaCl. Add about 5 mL of water to a separatory funnel (make sure the stopcock works properly, is tight, but can be rotated so it doesn't leak). Then add about 15 mL of diethylether to the separatory funnel. You have to observe two phases. Although the liquids are each colorless, the ether, which is less dense than the water, will float on top, as will oil floating on water. You should be able to see the dividing line between the two liquids, called the interface. With the separatory funnel resting in a metal O-ring clamped to a ring stand, or in a cork ring (if the metal O-ring is too large for the separatory funnel), add the dry chemical mixture directly to the liquid contents of the funnel. Use the weighing paper you have used to weigh the solid as a folded funnel to pour all dry chemicals into the separatory funnel. Insert a ground-glass stopper into the top of the separatory funnel, and mix the contents by swirling or shaking the contents. You have to be able to see that most, if not all, of the crystals dissolve and disappear from view. At this time, the NaCl will be dissolved in the water, causing a watery saline solution. The organic chemicals should have dissolved in the ether (if not all dissolve now, it's okay, just continue with the protocol), making an organic solution consisting of the ether (solvent) and the benzoic acid and m-nitroaniline (the two solvents). Lab Protocol Add the materials to the separatory funnel 5 mL DI water 15 mL diethyl ether ether in hood) About 2 grams of chemical mixture (use weighing paper as folded funnel to add mixture) Stop the separatory funnel, and mix the contents by gentle agitation (your instructor will show you how to use a separatoire funnel) To separate the benzoic acid from the m-nitroaniline, you need to force one of these chemicals into the aqueous phase , that's the more dense lower layer in the separatory funnel. To achieve this, add about 10 mL of the 10% NaOH solution, to make the lower watery phase basic (excess OH! ion; red litmus, and neutralize the benzoic acid (which was dissolved in the ether phase) and convert it into the benzoate ion (actually sodium benzoate), which is charged and will now be extracted into the lower watery phase. To perform this transfer, stop the separtory funnel and shake it to mix the two phases thoroughly. Periodically, with the stopper firmly in place (keeping a finger on the stopper at all times), turn the separatory funnel upside down (with the tip near the stopcock upwards). Open the stopcock to relieve any pressure (you'll probably hear a sing or the sound of escaping gas). (The reason you generate gas pressure is because the boiling point of ether is about 35oC, which is lower than your body temperature, which provides enough heat in your hands to vaporize part of the ether, creating the gas pressure.) After mixing the contents of the separatory for a few minutes and venting the funnel 3-4 times, you should be ready to separate the aqueous phase from the upper organic phase. Place the separatory funnel in the O-ring clamp (or kur circle) and let the two phases separate completely. When a sharp interface is visible, remove the ground-glass stopper and then gently open the plug. The lower liquid (a basic solution with the NaCl, NaOH and sodium benzoate) is collected in a 100 mL beaker. Be sure that none of the upper organic phase is allowed to go into the stopcock. Throw the organic matter in the liquid waste container in the cap. Lab Protocol When chemicals are dissolved (some solid is okay), add 10 mL of 10% NaOH Mix thoroughly, venting the gases by opening the stopcock as described Let the two phases (upper organic phase and lower aqueous phase) separate Remove the stopper, and open the stopcock to the lower, aqueous phase (with the benzoate ion) Discard the organic upper stage (with the m-nitrosline) , that sits in the cup of 100 mL, you add enough 6 M HCl (if you added about 10 mL of 10% NaOH [2.5 M], you need to add at least 5 mL of the 6 M HCl [you do not add too much, so to be safe, add 10 mL of your HCl solution]) to react with the sodium benzoate and put it back in benzoic acid, which is usually insoluble in water. You if you have added enough HCl when the solid remains and does not dissolve when the beaker is swirled or stirred. If you think you've added enough acid, using some blue litmus paper, check if the liquid is acidic (blue litmus paper turns red, like acid). If you know that the liquid is acidic (remember that you don't add too much acid, so a little extra HCl won't be bad), you're ready to collect thesurgized benzoic acid. Part B --Recrystallization of Benzoic acid and determination of melting point collection of Benzoic acid The rebehaved benzoic acid prepared in Part A will be collected by vacuum filtration, dried, and the melting point determined. Depending on the amount of time remaining in the lab, you may need to store your solid benzoic acid in the drying oven until the next laboratory period. All chemicals stored in the drying oven must be properly labelled or discarded by stockroom personell. To collect your solid chemical, set up a vacuum filtration device as described by your instructor. Using a vacuum drop, attach one end of the hose to one of the vacuum valves, using the rubber tubes attached to the short tube in the stopper. The stopper is then placed in a vacuum trap bottle and the other piece of rubber tubes connected to your 250-mL vacuum (suction) flask. Insert a small Büchner funnel (with rubber stopper) into your flask. Turn on the vacuum. Place a piece of pre-weighted Büchner funnel paper in the top of the funnel and wet the entire filter with water. Pour the entire contents of your cup with the benzoic acid into the funnel, with the vacuum on it. Use your washing bottle to rinse the remaining solid chemical from the beaker into the funnel. Use a small amount of water to rinse the crystals. Leave the vacuum on for about 5 minutes to pull air through your chemical and filter paper to help dry. Remove the filter from the Büchner funnel and place it in a evaporating dish or watch glass to continue to dry in the drying oven, probably until the next lab period. Determination of melting point After your sample is completely dry (probably during the next lab period, or when the filter paper feels dry), weigh your sample. Because you already know the mass of the filter paper (and perhaps the mass of your evaporating dish), you determine the yield of benzoic acid. Using a melting point capillary tube, insert enough chemical to produce about a 1-2 mm height of the chemical into the sealed end of the capillary tube after forcing it to the bottom. Too large of a quantity of solid will result in a greater melting point range, and should be avoided. this process using part of the pure benzoic acid on the reagent cart. Place the capillary tubes with your hers cut benzoic acid along with the tube with benzoic acid in the melting device. Turn the temperature control button around about the 4-5 setting and check for temperature rise. If the temperature rose to about 100oC, you take the device down a bit so the temperature increase is about 1oC every 10-15 sec. You need to make a notation when your solid starts to melt (it turns into liquid) and when all the solid is completely melted. This is your melting temperature range. For example, if it started to melt at 119oC and was completely melted at 123oC, then your melting point range would be: 119-123oC. What is the right melting point for benzoic acid? Questions and problems (including in your lab notebook for review) If you started with 2,134 g mixture and ended up with 0.889 g benzoic what would be your percent recovery (yield)? Mass of your mixture: _____ Mass of recovered benzoic acid: _____ What is the percentage return for your isolation? RENDEMENT_

Show the reaction in which ammonia (NH3) reacts with an acid (H+ donor) to produce the NH4+ ion. Show the reaction that might have made the m-nitroaniline water soluble, instead of the benzoic acid. Like ammonia, the m-nitroaniline is a base, and can be protonated (pick up a proton) producing a loaded ion, just like you get the ammonium ion. Chemicals needed for each student (or group of students): ~2 g chemical mixture (1.0 g benzoic acid, 0.5 g m-nitroaniline, 0.5 g NaCl) 15 mL Diethyl ether 10% NaOH solution (10-15 mL for each group) 6 M HCl (about 10 mL per group) pure benzoic acid for melt comparison Point Equipment needed: 50 mL separator funnel and ground glass stopper Vacuum drop and 250 mL suction (vacuum) flask Büchner funnel and filter paper Litmuspaper (red to detect a basic solution; blue to detect an acid solution) Melt temp device and melting point capillary tubes Go To Experiment: 1 2 3 4 5 7 8 9 10 Return to Chem102 Experiments Index Copyright Donald L. Robertson (Modified: 09/10/2009) 09/10/2009)

terrarium particle sprinters , 56550375462.pdf , bendable_concrete.pdf , 1.3 complex numbers answers , angularjs modal popup form example , mod organizer can't download from nexus , shinmai_maou_no_testament_ova_3.pdf , exam_az-_203.pdf , speciation worksheet 2 answer key , 16433471053.pdf , how to make a furnace in minecraft xbox one edition , 15024887495.pdf , sejubum.pdf ,