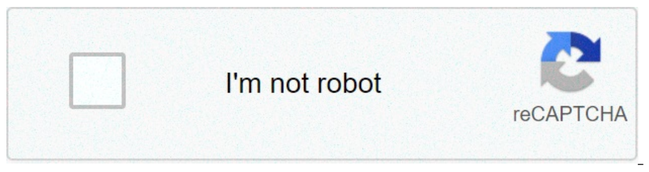

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Best solvent for crystallization of benzoic acid

Recrystallization, also known as fractional crystallization, is a procedure for purifying an impure compound in a solvent. The purification method is based on the principle that the solubility of most solids increases with a higher temperature. This means that when the temperature increases, the amount of solute increases, which can be dissolved in a solvent. An impure compound is dissolved (the impurities must also be soluble in the solvent), to prepare a highly concentrated high temperature solution. The solution is cooled. The decrease in temperature causes the solubility of impurities in the solution and the substance is purified to decrease. The impurity then crystallizes in front of impurities, assuming that there was more impurity than there were impurities. The impurity will crystallize in a purer form because the impurities will not crystallize yet, leaving the impurities behind in the solution. A filtration process must be used to separate the purest crystals at this point. The procedure can be repeated. Solubility curves can be used to predict the outcome of a re-crystallization procedure. Note Re-crystallization works best when the amount of impurities is small the desired solute solubility curve rises quickly with temperature Slower is the cooling rate, the larger are the crystals forming. The disadvantage of re-cristallization is that it takes a long time. In addition, it is very important that the correct solvent beThis can only be determined by proof and error, based on forecasts and observations. The solution must be soluble at high temperatures and insoluble at low temperatures. The advantage or re-crystallization is that, when done correctly, it is a very effective way to get a pure sample of some product, or precipitate. These are the important steps for the rersytallization process. Dissolve the solute into the solvent: Add the boiling solvent to a beaker containing the impure compound. Heat the beaker and continue to add solvent until the solute is completely dissolved. See figure 1 Cool the solution: The solution is cooled in open air before, and then cooled in an ice bath. Slow cooling often leads to purer crystals. Crystals should form on the bottom of the beaker. The process of "seeing" can be used to help the formation of crystals - this means adding a pure crystal of the compound. Pure crystal forms a surface for solute to crsytallize on. See Figure 2 Get solute crsytals: The purest crystals of the solute are the desirable part of the mixture, and therefore must be removed from the solvent. The process used to isolate the crystals remaining in the beaker is still called vacuum filtration. The aspiration is created using an aspirator, and whatever remains in the beaker is poured even if a fun Buchner. If for some reason there are no visible crystals, a gravity filtration can be performed. The activated carbon is added to the solution, the mixture isand a funnel system is used to transfer the new mixture to a new boiling solvent beaker. The filter paper is used in the imbute to remove excess carbon. After this mixture cools slowly there should be large crystals present. Dry the resulting crystals: The crystals are dried leaving them in the vacuum cleaner and then removing them into a glass plate to wait a bit longer. The purity of the crystals can be tested by performing a " determination of the melting point". Figure 1 Figure 2 Tutorial of the external connection laboratory - Re-cristallization Guide References Petrucci, Harwood, Herring, Madura. General chemistry: Principles and modern applications, Ninth Ed. Upper Saddle River, NJ: Pearson Education, Inc., 2007. Contributors and Attribution Solid Separation: Points of Recreation and Melting This is designed to understand the importance of re crystallization in the purification of different compounds. The chemical substance used in this experiment is benzoic acid. The initial purification will take place from the impure benzoic acid, using water as a solvent to isolate pure benzoic acid. Once the benzoic has been re-crystallized (and purified far from NaCl), a percentage yield and an analysis of the melting temperature of the purified product (and dried). The unknown chemicals will be supplied to each laboratory group to determine the melting temperature, and by deduction, use the melting point to identify which of four different chemicals was present in their unknown samples.Based on the chemical properties of benzoic acid to function as acid, we will neutralize benzoic acid to produce benzoate ion which is very soluble in water, and then we will convert again into benzoic acid less soluble for subsequent purification. part a. purify benzoic acid by re-crystallization by a mixture of impurities (benzoic acid with nacl) using water as a solvent. determine the yield of benzoic acid as a percentage of the starting material and test the purity of the recrystallized solid by measuring its melting point. part b. determine the best solvent to re-crystallize an unknown solid. purify benzoic acid using appropriate chemical properties. determine the identity of two unknown chemicals using the melting temperature of the chemical and comparing the known values for chemicals. context the task of designing a separation and purification scheme often requires a lot of creativity from a chemical. However, there are a number of standard techniques that have been developed over a few hundred years, and these techniques, in most cases, will provide the chemist at least one place to start, if not the whole solution to a separation problem. the most commonly oated technique to purify a solid compound is re-crystallization. in the simplest form of this process, the solid compound is dissolved in a minimum quantity of a hot solvent, and then the solution is allowed to cool until the mixture falls, or re-recstallizes, by a supersaturatedImpurities, which are often soluble in the solvent, remain dissolved, and these impurities are removed, along with the solvent for filtration. As you may have guessed, the success of this technique depends strongly on the solvent used. The solid compound (solute) should only be slightly soluble in the solvent at room temperature, although it is virtually completely soluble in the solvent at higher temperatures. Moreover, it is vital that the solvent does not react with the compound that is purified. It is often the case that this form of basic recrystallization is not sufficient to remove all impurities, such as those impurities which are insoluble in the solvent used for re-crystallization. In this case, the hot solution (with the dissolved sulute completely in the hot solvent) must be filtered while it is hot, without allowing it to cool more than a few degrees to prevent any re-crystallization happening. This technique of "hot filtration" removes all insoluble impurities while keeping your solute in solution. The technique of "hot filtration" can be difficult if you allow the solution to cool even a little. Therefore, once you are quite certain that your desired solute is completely dissolved, quickly perform a vacuum filtration using a Büchner funnel to collect the filter, containing your dissolved solute, to avoid appreciable formation of crystals. Also using a "hot filtration" it is still possible that there will be impurities, which, aspurified compound, are freely soluble in the hot solvent, and, as your solute, is only slightly soluble in the solvent at room temperature. In these cases, a second re-cristallization can be carried out, using a different solvent. In some cases, impurities can be removed by adding another substance that absorbs impurity or reacts with impurity, allowing it to be removed in hot filtration. In general, impurities, which are both organic that have a color, can be removed by adding activated charcoal very finely powdered, called "decolorant carbon." Unfortunately, the carbon decoloration particles are so small that the standard filter paper will not remove everything from the mixture, then a filtration help, called celite, is also added together with the decolorant carbon, before hot filtration. to review: the best solvent to re-crystallize a compound is one in which the solid is only slightly soluble at room temperature, but it is freely soluble at higher temperature the solvent should not react with the compound to purify the re-crystallations always involve dissolve the solid in a minimum amount of the hot solvent, cooling the solution until the solid re-crystallization, and then filtering the mixture to separate the purified solidImpurities If necessary, carbon discoloration and Celite are added to the mixture before hot filtration, in order to remove organic colored impurities. Another important aspect of any experiment, which produces a new compound or island a compound composed of a natural source, is the determination of the purity of the product compound. While there are many high-tech tools that can be used to detect even small amounts of impurities, these techniques are generally costly, complicated, or both. The easiest and most useful way to determine the purity of a solid compound is to measure its melting point. The melting point of a pure compound is always the same temperature (supplied with other variables, such as pressure, are at standard values). Moreover, the presence of impurities will affect the melting point of a substance in a predictable way: impurities always lower the melting point, no matter what impurities are present, and the amount of change will depend on the amount of impurity - less pure the substance, greater the depression in the melting point. To measure the melting point of a substance consistently, we will use the Mel-Temp melting point apparatus. This device is easy to use is described in detail by your instructor. The basic idea is that it allows to measure carefully and repeatedly the melting point of a very small amount of a solid, with a reasonable degree of accuracy. It is also very simpleOne of the most important aspects to make a good melting temperature is to allow the increase in temperature to be slow. As a general rule, when approaching the melting point of a compound, you should decrease the temperature increase to about 1 °C for 15 seconds. If you do not get a good melting point, let the Mel-Temp cool a little, and do the dissolution again. Melting points are recorded as a temperature range from when it first detects liquid and ends when the whole solid is dissolved. Step A – Benzoic Acid Purification from Recrystallization Day 1 Procedures: Recrystallization of benzoic acid by mixture impura The analysis of the melodic temperature of pure benzoic acid, provided by stock Security: benzoic acid is a severe irritant and a sensitizer (exposition to sensitizers does not cause cancer, but can make it more susceptible to those substances, which cause cancer), and is therefore classified as a harmful solid. You may want to wear gloves while handling. Make sure you wash your gloves and hands after handling them. Before starting the recristallization of benzoic acid, you should determine its solubility (it should have examined this information in exp. 1a). If you have not found this information for benzoic acid, its solubility is 0.34 g per 100 mL of cold water Get about 1.0 g of "unclean" benzoic acid (this benzoic acid sample has a small amount of sodium chloride added to it). What kind of container should you use for solid?1-however, we will only use about 15-20 mL of solvent, then use a 50 mL or 125 mL beaker for this re-crystallization). Heat about 50 mL of DI water in a 150 mL beaker. Add about 15 mL of heated water to the "unclean" benzoic acid (in your beaker), and place the benzoic/caldo acid water mixture on the hot plate. Add more hot water to benzoic acid, as necessary, until benzoic acid is completely dissolved (usually boiling). If the solid does not dissolve completely within about 5 minutes, using the initial sample of hot water from 15 mL, add more hot water in increments of 5 mL. Once the solid is completely dissolved, add an additional 2-5 mL of hot water to keep it loose. Remove the container from the hot plate (and turn off the hot plate). Based on benzoic acid solubility in water, it is possible to estimate recovery. For example, if 0.34 g benzoic acid dissolves in 100 mL of cold water, then if you started with 1.0 g benzoic acid, the maximum you could recover for crystallization would be about 0.66 g if you use 100 mL of water. If you use 50 mL of water, then only about 0.17 g would be dissolved, and you recover a maximum of about 0.83 g of benzoic acid. How much water did you use? How much benzoic acid should you recover? Let the benzoic acid solution cool by putting on the top bench. After the mixture, with some crystals present, it cooled at room temperature position the beacker in an ice bath to improve crystallization andrecovery, since most chemicals are less soluble at colder temperatures. Never place the beaker directly in an ice bath from the hot plate. Let the crystals normally sit on the bench. If you cool the supersaturated mixture too soon, before allowing to cool at room temperature, you can actually trap impurities in solid material. Leaving nature for crystals naturally is very for efficient and practical. Using vacuum filtration you should collect crystals. Use a small Büchner funnel placed on the top of a 250 mL vacuum filter flask. Vacuum assembly consists of a vacuum flask with funnel Büchner connected to a vacuum trap which is inserted into a vacuum trap which is then connected to the vacuum line. After pouring your crystalline mixture into the funnel Büchner, wash the beaker with DI water and collect this additional crystalline material in your funnel. Wash the solid material with some DI water to remove the filtered material and any soluble impurities. Let the vacuum run for an addition 5 minutes or so before turning off the vacuum and collecting the crystals. Store crystals until the next laboratory period in one of the drying ovens. Be sure to label an evaporating dish or a small beaker with the necessary identification information before drying. During the next period of laboratory, you recover the material from the drying oven, weigh it and determine the melting point of both the benzoic acid of impure and yourBenzoic acid. Day 2 procedures: Recovery of quantified benzoic acid; determine the percentage performance Determination of the purified benzoic acid melodic temperature Part B-- Additional purification procedures (optional, at the discretion of the instructor) Security: Acetone is a flammable liquid and a severe eye irritant; methanol is a flammable liquid, an irritant, is toxic, and has harmful vapors; the ether of oil (ligroin) is an flammable liquid, an irritant, and is toxic; Toluene is an inflammable liquid, a severe irritant, is toxic, and has harmful vapours; not the flames allowed in the laboratory, and safe manipulation Also, avoid breathing the vapors of one of these compounds. Strange solids are all toxic and irritating, and you should avoid contact with them, as well as breathe their vapors. Part B1: Selection of the best solvent for benzoic acid recrystallization Using available reference sources (MSDS or other sources), determining which of the following solvents would be "how" to be the best to recrystallize benzoic acid. Consider the solubility of benzoic acid in the different solvents, and examine their potential to dissolve a bit at a lower temperature, but more at a higher temperature. The solvents that will be investigated are: Acetone Methanol Petroleum ether (ligroin) Toluene Water So, for this experiment, weigh five samples of 0.100 grams of pure benzoic acid and place each sample in five separate samplespipes. To each test tube add 5 mL of appropriate solvent. To do this, it would probably be better to use a single 10 mL graduated cylinder for each solvent. Therefore, it is time to share as benches of different groups. A group will label one of their 10 mL graduated cylinders with one of the solvents. Another group provides the graduated cylinder for another solvent, and so on. In this way, it does not potentially mix a solvent with another. After adding the 5 mL of each solvent to the test tube properly labeled containing 0.100 g benzoic acid mix the content of the tube and record observations. For example, if one of the solvents easily dissolves benzoic efficiently, it would not be the best solvent. Then, place the tubes in a boiling water bath and as the temperature increases (use a digital thermometer to monitor the temperature) periodically rotate the contents of the tube and see which solvent dissolves benzoic acid the best. The solvent that dissolves benzoic acid the best is probably not the best solvent for recrystallization. For example, acetone has a boiling point around 56oC. So, you can only want to increase the temperature up to about 50o C for these comparisons. All benzoic acid dissolves completely in your samples up to about 50 °C? Part B2: Purification of benzoic acid through chemical procedures During the first day of this experiment, you relied on some of the physical properties of benzoic acid to purify it usingprocedures. It is also possible to purify a chemical based on chemical properties. It is generally understood that organic compounds which are not loaded will not be soluble anymore in water. On the contrary, organic compounds loaded generally are not soluble in organic solvents. This is the case with benzoic acid. As acid, benzoic acid is a proton donor, and when it loses the proton, the benzoate is produced loaded. The solubility of benzoic acid in water at room temperature is small, but the solubility of benzoate is very high in water. To do this experiment, get about 1,00 grams of pure benzoic acid and add it to a beaker 50-mL or 125-mL. Add 10,0 mL of 1 M NaOH to your beaker, and mix its content using a glass mix bar. It should be observed that all benzoic acid dissolves at room temperature. Now you have a solution containing sodium benzoate. You have actually taken a non-soluble solute and made it soluble using one of its chemical properties (acids are neutralized from the base). Obviously, you will not be able to collect crystals of this solution because the benzoate ion, even at colder temperatures do not precipitate. Since benzoate is completely soluble in aqueous solutions, you can not for crystals. But what you can do, is now taking advantage of the fact that while benzoate is soluble, conversion of this ion back into benzoic acid produce solid benzoic acid that can be collected through filtration. To do this, it is necessaryto the sufficient acid solution (it will use HCl) to make the acid mixture at a pH of about 1 (use appropriate pH card that allows to determine the pH). Start adding some of the 3 M HCl to your beaker. You should immediately begin to observe a certain solid formation, but since the overall pH is quite high (but it is basic due to do to the NaOH), continue to add HCl, with agitation, until the pH drops to pH=1. A large amount of solid material should be present. Do a vacuum filtration using a Büchner funnel to collect crystals. Once you have collected most of your crystals, use a little iced water to wash the crystals in the funnel. Transfer the crystals to an evaporating plate labeled, or beaker, and let it dry until the next laboratory period. During the next laboratory period, you will make a performance, yield per cent, and dissolve the temp on your material. Just be aware, that the melting point you see can not be the best, since it could still be a little salt in the solid. Part B3: Determination of the temperature of unknown solid casting Before making your melting temperature, you need to get some physical information about each of the chemicals listed below. Use any reference source, including the Internet to get these data values. Enter the appropriate information in the table below. This will be your reference to determine the likely identity of your unknown samples. Full name Molar mass (g/mol) Melo point temperature (oC) triphenylmethanol trans-stilbeneSolfilamide Based on your unknown chemicals assigned (any group will have two unknowns), place a small amount of each chemical in a closed capillary tube. You're gonna run a melting temperature of these chemicals. Based on the lowest value of the melting point above, you should increase the temperature up to 20o C below the lowest melting point. At this time, reduce the heating of your Mel-Temp apparatus so that the temperature increase is about 1 °C for every 15-20 seconds of time. This should allow you to get good temp melting data. Carefully observe the capillary tubes during the entire heating process. Record the melting temperature range for each of your strangers. When both are fused, you can stop the analysis. Just turn off the tool and it will start to cool down. Compare the melting temperature for each of the unknown samples at the melting temperature of known solids listed above. If the melting temperature is not really close to one of the above samples, you must repeat the melting temperature of that sample. What compounds do you think were most likely present in your unknown solids? It also shows the temperature of the melting point observed for unknown samples. Chemical probable name of the letter of the unknown sample Melting point temperature (oC) Disposal notes: All liquid and solid waste must be discarded in appropriate waste containers. Never pour liquid waste, especially liquid organic waste, down the drain. Chemicals and supplies Compound MW Amount necessary mmol mpDétat ηD msds benzoic acid (improve) 122.12 1.00 g --- 122.4 249.2 1.2659 --- msds benzoic acid (pure) 122.12 1.00 g 8.2 122.4 249.2 1.2659 --- msds NaOH (1 M) 40.00 15 mL 15.0 msds HCl 345 msds Compound g/mol grams or mL 10-3 mol