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Content: Recrystallization is a technique of purification; allows us to remove impurities in a sample. The idea is that you place solid impure in a liquid like water or ethanol. After heating for a short time, the solid will dissolve in the liquid (also known as solvent). When the solvent cools again, the solid falls out of the solution and leaves the impurities behind, still dissolved in the solvent. This should leave you with a purified solid. Let's take a look at more details as (and why) this works. How does re-cristallization work The details Solubility of most solids in solution increases as the solution temperature increases. For example, the solubility of acetanilid in ethanol at 0 oC is about 18g/100mL. This means that if you drop 50 grams of acetanilide in 100 mL of ethanol at 0 °C, about 18 grams will dissolve in ethanol and the rest (~32g) will remain suspended in the solution. But the solubility of acetanilid in ethanol increases to about 80g/100mL at 50 oC. This means that if we heated the same acetanilide-ethanol suspension at 50 oC, all acetalid would dissolve. In fact, we can add about 30 grams of acetanilid to this solution and it would fade again- but once we added more than 80g, further solid acetanilid would not dissolve any more, and we would have a suspension once again. At this point it is said that the solution is saturated. So let's say we had 50g of acetanilid dissolved in 100mL of ethanol at 50 oC. What would happen if we allowed the temperature to cool again at 0 °C? At this point, we have more acetanilid dissolved in ethanol that should be able to hold - 50g vs. 18g, so the solution is said to be supersaturated. This means that sooner or later, the ~32 extra grams of acetanilide should precipitate out of the solution (also known as crash out). Then we collect acethanylide by vacuum filtration (or gravity). The liquid that remains after filtration is known as the supernatant mother liqueur (or only mother liqueur for short). It's usually a good idea to save him. Since there have been relatively few impurities than relatively large amounts of acethanylide and ethanol used, these impurities should remain dissolved in the solution; the ~32 grams of acetanilid we collect should be pure. Key recrystalling concepts Choose a good recrystallization solvent A good solvent for re-crystallization is one that the compound not very soluble at low temperatures, but very soluble at higher temperatures. For example, if the solubility of your ethanol compound is 1g/100mL at 0 oC and 2g/mL at 50 oC, then you will not be able to purify much more than 1 gram! On the contrary, if the solubility of your ethanol compound is 10,000g/100mL at 0 oC and 20,000 g/100mL at 50 oC, this isuseless- when are you going to purify that much of your compound? (10kg) In this case, ethanol is too soluble. Soluble.is a good solvent to re-crystallize acethanylide due to the wide range of solubility - 18g/100mL at 0 oc and 80g/mL at 50 oc as dissolves as polar compounds are soluble in polar solvents such as water, methanol and ethanol. if the compound contains a polar group (see image below,) it is better to oate these solvents. non-polar compounds are soluble in non-polar solvents such as esane and diethyl ether. crystal size and cooling speed the speed at which a saturated solution is allowed to cool affects the size of the crystals that are formed! If you take a hot solution and bang it in an ice bath, you will get smaller crystals. if you allow a hot solution to cool without any added heat for 10-20 minutes, and then place it in an ice bath, you will get larger crystals. this makes a different important- if you have smaller crystals, they are more likely to intasate a filter! everyone likes to ocire from the wizard lab as soon as possible, but an inlaid filter could add a good 30-40 minutes to your procedure. it is worth not to put your solution in an ice bath at once; let cool near the room temperature before. removal of insoluble impurities we say that accidentally poured black pepper group into a solution of acethanyl and ethanol. the black pepper will not dissolve. So, how do you remove it? filtering the liquid- black pepper will be stuck in the filter paper, and you can continue to re-crystallize the liquid solution as normal. "My product does not crystallize!" sometimes happens. You probably smelled too much solvent, or you're just unlucky. the best way to get the product to crash is to add a seed crystal. this is just a small amount of pure material. your ta usually has a little useful (or see another lab group that finished the experiment before you.) you can also try to scratch the sides of the container with a mix bar. This works because you collect a small amount of solvent that evaporates and leaves behind a small amount of pure product. in other words, it is only another way to add a seed crystal. Save the mother's liquor! remember that there will still be some of your dissolved products in your mother's liqueur after the first re-cristallization. therefore, if you want, you can allow some of the mother liqueurs to evaporate and collect more product. Always save the mother's liqueur until ta tells you it's okay to throw it out. normal English procedure in this lab you are going to aniline acetylate. Acetylation is the addition of an acetyl group to an alcohol or amine. (this mechanism will not be covered until the spring semester. but you can see it here if you are interested.) in the text book says to oate a test tube. butbe more convenient to use a 25 mL Erlenmeyer flask, which has flat bottom, so it is easier to manage (can place it on a flat surface). Both aniline and acetic dioxide are liquid. You are going to pre-weight a vacuum test tube, add ~4 drops of aniline, and then weigh the test tube again forexactly how much aniline you added. You will need this number later to calculate a percentage return. After adding and weighing the aniline, add 6-7 drops of acetic dioxide. As always, do this stuff in the steam hood. Acetic anhydride becomes acetic acid when water is added. It will smell of vinegar because it is this which is - vinegar is Latin for vinegar. Shake like a Polaroid for about five minutes. This should be... The reaction is done. Now is the time to purify your product... through re-cristallization. Add ~5 drops of water to the pipe and heat until the solid melts. Then let cool yourself for 5 minutes before putting an ice bath. Crystals should form. Collect the product using vacuum filtration through a funnel Hirsch. Use your solid on a pre-heavy drying dish. You will leave in the drawer to dry until next week, when you weigh it again and calculate a percentage yield. Questions will probably be asked Q: A student performed this experiment using 0.110 g aniline and collected 0.132 g acetanilid product. Calculate percentage performance. A: You will be asked to calculate the percentage performance on each lab you do for the rest of your life, and on many tests and quizzes as well. So this is something you have to learn to do. So, let's review what we learned in genchem: The first step is to understand how much product the student should have collected, assuming as 100% yield and without errors. To do this, first we convert the grams of aniline to aniline mole: Then we determine the size of aniline to acetanilide. From our reaction scheme, it is clear that for each 1 halve of aniline we use we produce 1 halves of acetanilid, so the ratio is 1-to-1: Finally, we convert the acetanilid moles into grams of acetanilid: We can (and should) put all this together in one calculation: Note that many units stand out. This is the way you know that you are setting the fractions properly; if it is not possible to cross units at the top and bottom of fractions, it means that you mix them somewhere. So the theoretical yield of acetanilide is 0.160g. If the student had a perfect lab day that he or she would collect 0.160g and have a 100% performance.percentage. But the student collected only 0.132g of acetanilid. To achieve the percentage performance, we calculate 0.132g as a percentage of 0.160g: D: What if you add too much solvent during re-crystallization? A: The goal is to add enough hot solvent to dissolve the product, and no more. Otherwise, more than your product will remain dissolved in the solvent when you cool it back down, and you will collect less product.lowers the yield of percent (also known as 100% recovery). Q: During filtration, why is it important to wash only solid with cold ice solvent? A: Remember that the product is soluble in the re-crystallization solvent. If washed with hot solvent, it would dissolve some (or all) of your product and you would have a yield of less percent. Q: Why should you bend and putin the filter paper before filtering? A: The folds increase the paper surface, allowing a faster filtration. D: What factors influence the solubility of a solid in solution? A: The first factor is summarized with “how it dissolves as”, said above. The second factor is the melting point of the solid: The higher the melting point, the higher the reticular energy, the lower the solubility. An example is given in Table 3.1 (page 24) of the laboratory manual: Note that the para isomer has the highest melting point and lowest solubility, although the polarities of all three isomers are similar. And this is all this week. The principle behind re-crystallization is that the amount of solute that can be dissolved by a solvent increases with temperature. In re-crystallization, a solution is created by melting a solute into a solvent or near the boiling point. At this high temperature, the solute has a considerably increased solubility in the solvent, so a much smaller amount of hot solvent is required than when the solvent is at room temperature. When the solution is then cooled, after filtering insoluble impurities, the amount of solute that remains dissolved drops precipitously. At the coldest temperature, the solution is saturated to a much lower concentration of solute. The solute that can no longer be held in forms of purified solute crystal solution, which can be collected later. Recrystalization only works when the correct solvent is used. The solute must be relatively insoluble in the solvent at ambient temperature but much more soluble in the solvent at higher temperature. At the same time, the impurities present must be soluble in the solvent at room temperature or insoluble in the solvent at high temperature. For example, if you want to purify a X compound sample which is contaminated by a small amount of Y compound, an appropriate solvent would be one where all Y compound dissolved at room temperature because impurities will remain in solution and pass through filter paper, leaving only pure crystals behind. Also appropriate would be a solvent where impurities are insoluble at high temperature because they will remain solid in the boiling solvent and can therefore be filtered. When it comes to strangers, you need to test which solvent will work best for you. According to the adage "Like dissolves as," a solvent that has a polarity similar to the solute that is dissolved usually dissolves the substance very well. In general, a very polar solute will be easily dissolved in a polar solvent and will be quite insoluble in a non-polar solvent. Often, having a solvent with slightly different polarity characteristics than the solute is better because if the polarity of the two is too closely matched,solute will probably be at least partially dissolved at room temperature. There are five main steps in the re-cristallization process: dissolve the solute in theperforming a gravity filtration, if necessary, obtaining solute crystals, collecting solute crystals for vacuum filtration, and finally drying the resulting crystals. Dissolve the solute into the solvent Add a small portion of boiling solvent to the beaker containing the impure sample and a boiling chip. Heat the beaker containing the solute and continue to add the boiling solvent in an incremental way until the whole solute has been dissolved. If the additional solvent can be added without any appreciable change in the amount of solute present, the particulate matter is probably insoluble impurities. Filtration of hot gravity This step is optional if there is no visible particulate matter and the solution is the expected color (most organic compounds are white or light yellow) If the solution is not the expected color, remove the boiling solution from the heat and allow it to cool under the solvent boiling point. Add a small amount of activated carbon (about the size of a pea) and mix the solution. If too much activated coal is used, the excessive loss of the desired product will result. Boil the solution containing the activated carbon for 5-10 minutes. To remove the carbon in the following steps, you must insert a help filter into the filter paper. Flute a piece of filter paper and place it inside a funnel without stem. A funnel with a stem is prone to premature recrystallization within the stem because the filter can cool while passing through the stem. At these colder temperatures, crystals are probably shaped. Heat a beaker containing some of your re-crystallization solvents. Place the funnel and filter the paper assembly into the beaker so that the rising vapors from the boiling solvent can heat the funnel and filter paper. Having the heated set up before filtration will prevent the crystals from forming on paper and imbute (see figure 1 below). Figure 1. Hot gravity filtering. Keeping the set up warm prevents crystals from forming prematurely. Maintain the solution very hot so that the solute remains dissolved, pour the solution through the imbute and assembly of filter paper. While the filter begins to accumulate, heat the beaker of the receptacle; The resulting vapours will help prevent any crystallization in the imbute or filter paper. If the imbute has been properly heated before filtration, the whole solution will be passed through and no crystal will be formed on paper or imbute. If the crystals are formed, pouring a small amount of boiling solvent through the imbute will dissolve them. If the solution is still discolored after the use of activated carbon and filtering, bothcolor is from the compound and will not go away or it is necessary to repeat the passage with the addition of activated carbon. The solution should be allowed to cool slowly at room temperature. Gradual cooling is favorable to the formation of large crystals well defined. Vacuum filtration (see filtering techniques, recalling these additionalShake the crystals with a glass barrel polished on fire before pouring the mother-liquor along with the crystals through the imbuto Buchner. Apply the maximum amount of suction possible using the vacuum cleaner. Some crystals may have been left behind in the beaker; there are two ways to carry out a quantitative transfer of all this material. Use a part of the filter to rinse the beaker or use a rubber cop at the end of your mixing bar to scrape the remaining crystals into the Buchner stock. When the crystals were collected and washed, they allow the aspirator to run for several minutes so that the crystals have the opportunity to dry. Dry the crystals When the crystals have been dried as much as possible in the Buchner imbute, use a scoopula to remove them to a beaker or a crystallizing dish. This will ensure that the crystals are not contaminated by filter paper fibers while drying. After removing all the crystals from the filter paper, remove the filter paper and scrape the remaining crystals from the imbute. Spread the crystals into a beaker or crystallizing dish will provide the most efficient drying as the crystals will have a maximum exposed surface. When the crystals are dried, the purity of the sample can be measured by determining the melting point. What to do if crystals are not formed If crystals do not form on the slow cooling of the solution at room temperature there are a variety of procedures that you can perform to stimulate their growth. First, the solution should be cooled in an ice bath. Slow cooling of the solution leads to slow formation of crystals and slower form of crystals, more pure are. The crystallization speed slows down when the temperature decreases so cooling with an ice bath should be used only until the crystals begin to form; after them, the solution should be allowed to heat at room temperature so that the formation of crystal occurs slower. If you do not form crystals even after the solution has been cooled in an ice bath, take a fire-polished mix bar and engraving (scratch) the glass of your beaker. The small pieces of glass that are extracted from the beaker serve as nuclei for the formation of crystal. If the crystals still do not form, take a small amount of your solution and spread it on a watch glass. After solvent evaporation, the crystals left behind can serve as seeds for further crystallization. Both these nucleation methods (etching and seed crystals) cause a very fast crystallization, which can lead to the formation of impure crystals. Ithey do not form if there is a large excess of solvent. If you do not form crystals with the methods already discussed, a part of the solvent may be necessary to remove. This can be achieved by heating the solution for a period of time in order to evaporate some solvents. The new concentrated solution should be cooled, and the methods mentioned above to stimulate crystallization should be again tempted. Another potentialin re-cristallization is that the solute sometimes comes out of the solution in the form of impure oil instead of forming purified crystals. This usually happens when the solvent boiling point is higher than the melting point of the compound, but this is not the only scenario where this problem arises. If this begins to happen, the cooling of the solution will not stimulate crystallization, it will make the problem worse. If an oil begins to form, heat the solution until the oil portion dissolves and let the whole solution cool. While the oil begins to form again, mix the solution energetically to break the oil. The tiny oil beads resulting from this trembling can act as the nuclei for the new crystal formation. training.