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Recrystallisation is one of the most common techniques used in chemistry to purify a compound. Choosing your solvent When conducting a recrystallisation it is very important that you select the correct solvent. If you are following a procedure, it will most likely tell you which solvent to use. But when you are left to select the solvent yourself, there are several things to keep in mind. The ideal solvent will dissolve your product at higher temperature but not at room temperature. The idea is that the impurities will remain in the solvent after your product crystallises. A simple way to check if a solvent is suitable is to add a small amount of your product to a sample vial and add room temperature solvent. If the sample dissolves it is unsuitable. If you heat the sample vial you want the sample to eventually dissolve (although you may have to add more solvent). Common solvents for recrystallization include ethers, alkanes and alcohols. Performing the recrystallisation You've selected your solvent and you're ready to begin, now what? You'll need to set up a reflux condenser and add your sample to a round bottom flask. The liquid in the round bottom flask should not exceed the halfway mark so select the flask carefully. You want to dissolve the impure solid in the minimum amount of hot solvent. Start with a small amount in your flask and add more via the condenser until the solid has dissolved. Allow to reflux before cooling. The more slowly you allow the solvent to cool, the larger the crystals will be. Obtaining the product Filter off your solid and wash it with a small amount of cold solvent to remove any impurities that may be stuck to the crystals. Then leave this to dry – you may wish to do so overnight in a desiccator. +2 The principle behind recrystallization is that the amount of solute that can be dissolved by a solvent increases with temperature. In recrystallization, a solution is created by dissolving a solute in a solvent at or near its boiling point. At this high temperature, the solute has a greatly increased solubility in the solvent, so a much smaller quantity of hot solvent is needed than when the solvent is at room temperature. When the solution is later cooled, after filtering out insoluble impurities, the amount of solute that remains dissolved drops precipitously. At the cooler temperature, the solution is saturated at a much lower concentration of solute. The solute that can no longer be held in solution forms purified crystals of solute, which can later be collected. Recrystallization works only when the proper solvent is used. The solute must be relatively insoluble in the solvent at room temperature but much more soluble in the solvent at higher temperature. At the same time, impurities that are present must either be soluble in the solvent at room temperature or insoluble in the solvent at a high temperature. For example, if you wanted to purify a sample of Compound X which is contaminated by a small amount of Compound Y, an appropriate solvent would be one in which all of Compound Y dissolved at room temperature because the impurities will stay in solution and pass through filter paper, leaving only pure crystals behind. Also appropriate would be a solvent in which the impurities are insoluble at a high temperature because they will remain solid in the boiling solvent and can then be filtered out. When dealing with unknowns, you will need to test which solvent will work best for you. According to the adage "Like dissolves like," a solvent that has a similar polarity to the solute being dissolved will usually dissolve the substance very well. In general, a very polar solute will easily be dissolved in a polar solvent and will be fairly insoluble in a non-polar solvent. Frequently, having a solvent with slightly different polarity characteristics than the solute is best because if the polarity of the two is too closely matched, the solute will likely be at least partially dissolved at room temperature. There are five major steps in the recrystallization process: dissolving the solute in the solvent, performing a gravity filtration, if necessary, obtaining crystals of the solute, collecting the solute crystals by vacuum filtration, and, finally, drying the resulting crystals. Dissolving the solute in the solvent Add a small portion of boiling solvent to the beaker that contains the impure sample and a boiling chip. Heat the beaker containing the solute and continue adding boiling solvent incrementally until all of the solute has been dissolved. If additional solvent can be added with no appreciable change in the amount of solute present, the particulate matter is probably insoluble impurities. Hot Gravity Filtration 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 to beneath the boiling point of the solvent. Add a small amount of activated carbon (about the size of a pea) and mix the solution. If too much activated carbon is used, excessive loss of the desired product will result. Boil the solution containing the activated carbon for 5 to 10 minutes. A filter aid will need to be placed in the filter paper to remove the carbon in the following steps. Flute a piece of filter paper and place it inside of a stemless funnel. A funnel with a stem is prone to premature recrystallization inside the stem because the filtrate can cool as it passes through the stem. At these cooler temperatures, crystals are likely to form. Heat a beaker that contains some of your recrystallization solvent. Place the funnel and filter paper assembly in the beaker so that the rising vapors from the boiling solvent can heat the funnel and filter paper. Having the set up heated before filtration will prevent crystals from forming on the paper and in the funnel (see Figure 1 below). Figure 1. Hot gravity filtration. Keeping the set up hot prevents crystals from forming prematurely. Keeping the solution very hot so the solute stays dissolved, pour the solution through the funnel and filter paper assembly. As the filtrate begins to accumulate, heat the receptacle beaker; the resulting vapors will help to prevent any crystallization in the funnel or on the filter paper. If the funnel was properly heated before filtration, all of the solution will have passed through and no crystals will have formed on the paper or in the funnel. If crystals have formed, pouring a small amount of boiling solvent through the funnel will dissolve these. If the solution is still discolored after using activated carbon and filtering, either the color is from the compound and will not go away or you need to repeat the step with the addition of activated carbon. The solution should be allowed to cool slowly to room temperature. Gradual cooling is conducive to the formation of large, well-defined crystals. Vacuum Filtration (see Filtering Techniques, remembering these additional points) Agitate the crystals with a fire polished glass-stirring rod before pouring the mother-liquor along with the crystals through the Buchner funnel. Apply the maximum amount of suction possible using the aspirator. Some crystals may have been left behind in the beaker; there are two ways to effect a quantitative transfer of all of this material. Either use a portion of the filtrate to rinse the beaker or use a rubber policeman on the end of your stirring rod to scrape the remaining crystals into the Buchner funnel. When the crystals have been collected and washed, allow the aspirator to run for several minutes so that the crystals have an opportunity to dry. Drying the Crystals When the crystals have been dried as much as possible in the Buchner funnel, use a scoopula to remove them to a beaker or crystallizing dish. This will ensure that the crystals are not contaminated by filter paper fibers as they dry. After removing all the crystals from the filter paper, remove the filter paper and scrape any remaining crystals from the funnel. Spreading the crystals out in a beaker or a crystallizing dish will provide for the most efficient drying as the crystals will have a maximum of exposed surface area. When the crystals are dried, the purity of the sample can be measured by performing a melting point determination. What to do if crystals don't form If crystals don't form upon slow cooling of the solution to room temperature there are a variety of procedures 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 the slower crystals form, the more pure they are. Rate of crystallization slows as temperature decreases so cooling with an ice bath should only be used until crystals begin to form; after they do, the solution should be allowed to warm to room temperature so crystal formation occurs more slowly. If no crystals form even after the solution has been cooled in an ice bath, take a fire polished stirring rod and etch (scratch) the glass of your beaker. The small pieces of glass that are etched off of the beaker serve as nuclei for crystal formation. If crystals still do not form, take a small amount of your solution and spread it on a watch glass. After the solvent evaporates, the crystals that are left behind can serve as seeds for further crystallization. Both these methods of nucleation (i.e. etching and seed crystals) cause very rapid crystallization, which can lead to the formation of impure crystals. Crystals will not form if there is a large excess of solvent. If no crystals form with the methods already discussed, a portion of the solvent may need to be removed. This can be accomplished by heating the solution for a period of time in order to evaporate some solvent. The new, concentrated solution, should be cooled, and the previously mentioned methods to stimulate crystallization should again be attempted. Another potential problem in recrystallization is that the solute sometimes comes out of solution in the form of an impure oil instead of forming purified crystals. This usually happens when the boiling point of the solvent is higher than the melting point of the compound, but this is not the only scenario in which this problem presents itself. If this begins to happen, cooling 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. As the oil begins to form again, stir the solution vigorously to break up the oil. The tiny beads of oil that result from this shaking may act as the nuclei for new crystal formation. In this tutorial, you will learn about recrystallization analysis and the steps required to perform one. Growing Bismuth Crystals Tin Crystals From Tin Chloride Experiments The definition of recrystallization is a technique for the purification of compounds in which a compound is dissolved in a solvent and slowly cooled to form crystals, which are a purer form of the compound. Scientists use recrystallization to purify solids, typically products, from different chemical reactions. The process involves dissolving a solid into solution, then allowing the dissolved substance to gradually crystallize. This produces compounds high in purity, a quality which can be seen by the presence of uniform crystals. Recrystallization is a challenging procedure to carry out correctly. Researchers must carefully control all influential variables, including temperature and time, to ensure a successful recrystallization of their product. Although the procedures of crystallization and recrystallization display similarities, their respective definitions diverge. Firstly, crystallization is a separation technique. It involves precipitating crystals from solution through transitions in the solute's solubility conditions. The resulting crystals can be readily distinguished and subsequently filtered out from the solution. Solubility rules will tell you if a precipitate will form. Recrystallization, on the other hand, requires the purification of crystals already obtained via the crystallization method (hence the "re" prefix, which reinforces this sense of repetition). Scientists turn to this second process because crystals generated through crystallization often contain impurities. Recrystallizing the compounds facilitates a more effective removal of these impurities. This process can even be repeated multiple times, increasing the purity of the desired compound during each trial. We must now discuss another relevant discrepancy, the difference between precipitate formation and crystal formation. A precipitate is a solid mixture of compounds that originates from an oversaturated solution, falling out of it. However, precipitated substances may not necessarily be pure and can contain up to several compounds. In contrast, only one compound composes any given crystal. This results in a highly organized, structurally uniform product; in contrast, precipitates hold no apparent structure. Therefore, while precipitates occur relatively readily, procuring crystals is a far more difficult process. The mechanism of generating crystals involves heating a solution to a high temperature in order to saturate it. This target high temperature often falls near the solvent's boiling point. Then, you remove the heat source and allow the solution to drop back to room temperature. As the temperature decreases, so does the solubility of the compound in solution. Soon, the solution becomes supersaturated, prompting crystal formation. As discussed, these crystals then precipitate out of the solution at the cooler recrystallization temperature. Here is the complete procedure for seeing one of the most beautiful recrystallizations on the planet! As described, the recrystallization process further purifies the compounds of the crystallization process. The mechanisms explaining that include the simultaneous dissolving of both the compound and its impurities in the solvent. During this process, either the desired compound or the impurities can exit the solution and leave the other behind. Scientists filter these solutions to remove the insoluble impurities contained within. They can then evaporate the solvent, isolating the solid compound. To expedite crystallization and recrystallization processes, scientists can employ a technique called seeding. Seeding involves first dipping a small crystal, or seed, of solute into the saturated solution. Following this step, larger crystals can form and subsequently grow on this seed crystal. This practice boosts the efficiency of recrystallization because it eliminates any dependency on random molecular collisions. The seed crystal prompts more molecular interaction and facilitates crystal formation, speeding the overall process. This type of recrystallization is the most basic and, as a result, the most frequently employed. The process involves dissolving a compound, "A," and impurity, "B," in a heated solvent system. As this solution cools back to room temperature, the solubility of the compounds in the solution drops, prompting the desired compound to recrystallize from the mixture. This method of recrystallization closely resembles the first in terms of the procedure involved. However, multi-solvent recrystallization requires two, and sometimes more, solvents. The compound, "A," and impurity, "B," dissolve in the first solvent. Then, the addition of a second solvent prompts the insolubility and precipitation of either A or B. The other component of the mixture remains in solution. Scientists typically employ multiple solvents when no singular solvent meets all the criteria for that given recrystallization procedure. Each solvent pair contains a "soluble solvent," one in which the compound is soluble, and an "insoluble solvent," one in which the compound is insoluble. The soluble solvent and insoluble solvent must be miscible, so that the proportions used do not limit their solubility in solution. To work with these two solvents, you must first dissolve your compound in the hot soluble solvent. Next, you add hot insoluble solvent dropwise to this mixture until it becomes cloudy. You then add a small amount of hot solvent to clarify the mixture, then allow it to cool and crystallize, as you would during a single-solvent recrystallization. Below is a stepwise guide for completing a basic recrystallization procedure. 1. First, weigh the impure solvent and record that value. Then add the impure compound to a solvent system. 2. Heat this solvent system to your target temperature, or its boiling point. Be sure to raise the temperature in gradual increments to ensure that the rest of the process proceeds smoothly. 3. Let the solution stand without disturbance. Allow the temperature to slowly drop, reaching room temperature. If you do not maintain a gradual temperature decrease, a precipitate may form instead of the desired crystals. Additionally, do not place the flask on a hard surface, which will cause "shock-cooling" of your solution. Instead, either place the flask in an insulated jar or clamp it to a steady, secure device. Recrystallization is a gradual process; do not become impatient as you wait and employ appropriate safety precautions. 4. To further lower the temperature, place the solution in an ice-water bath. This facilitates the formation of crystals by speeding the recrystallization reaction. Shortly after completing this step, you should observe many crystals. 5. Filter the crystals via vacuum filtration. 6. Air dry the crystals. Then weigh this recovered compound. 7. Calculate your percent recovery of desired compound. To accomplish this, divide the weight after purification, obtained in step six, by weight before purification, obtained in step one–then multiply this value by 100%. If your percent recovery falls within the range of 70-100%, you can view your procedure and the amount of recovered product as "very good." 8. Compliment your percent recovery quantification with a visual inspection. If the crystals appear uniform in shape and size, as well as display shiny surfaces that catch the light, you have successfully completed your procedure. You may wish to further distinguish the purity of your compound, which you can do by conducting a melting point analysis and comparing the obtained temperature to literature values. Although quite useful, recrystallization possesses some limitations and ramifications. Firstly, the compound in question must occupy a solid state at standard conditions. This means that substances including oils, greases, and waxes cannot be crystalized or recrystallized under standard conditions. The purity of your crude material also comes into play. The success of recrystallization processes depends directly on the solubilities of each respective component of your starting substance. But this substance must contain approximately 80% of your desired compound; in other words, it should be mostly pure. Otherwise, the recrystallization will not proceed smoothly. Lastly, for each recrystallization step, some of the desired compound will remain in solution even after cooling it. Because of this, each recrystallization step will decrease the overall yield of the desired compound. For small scale applications, like laboratory testing, this does not have a huge effect. However, for large scale applications like pharmaceutical production, even a small percentage loss represents a large mass of compound. These limitations all must be considered when deciding to use recrystallization for a given process. Recrystallization has applications that extend into the industrial, medical, and pharmaceutical industries. Techniques such as texture control, drug development, and treatment purification all involve the procedure. But the pharmaceutical industry actually makes the most use of recrystallization procedures. Purification and separation processes are key to the isolation of different active ingredients. These steps, in turn, inform the synthesis of many different drugs and medications. Recrystallization Theory Single Solvent Recrystallization Two Solvent Recrystallization Example of a Typical Experiment Practical Lab Tips FAQ: The solvent that we use to dissolve the sample for TLC, is that the solvent we will use for recrystallization? FAQ: How many boiling stones should I use? FAQ: When I tested the recrystallization solvent in a test tube it worked, but now my sample won't dissolve! FAQ: How long does it take for the crystals to grow? FAQ: So - once the solvent has reached its boiling point and my crude solid is NOT dissolved, do I add more solvent or do I let it boil longer? FAQ: If we add too much solvent do we just boil it off? FAQ: My sample has dissolved, but my solvent is just hot. Do I have to wait until it boils? FAQ: After the solution has cooled down to room temperature, how long should I let it cool in the ice bath? FAQ: Can I put my hot solution directly into the ice bath? FAQ: When we are collecting our crystals using vacuum filtration, what solvent do we use to wash our crystals? FAQ: I have a really lousy suction from that water aspirator. What can I do? FAQ: What should I put on the label when handing in my sample? FAQ: When using the two-solvent recrystallization method, why is it necessary to keep both solvents hot when adding? FAQ: Can we add the second solvent first? In the end, all are together anyway. FAQ: If I can choose between the 1-solvent or 2-solvent method, which one should I choose? Return to Top Recrystallization Theory Recrystallization is a purification technique. It works because: 1) different substances have different solubilities in the same solvent, and 2) only molecules of the same compound will fit easily into the crystal lattice of that compound. Impurities remain in solution or stick on the outside of the crystal lattice. In practice you purify by slowly cooling a hot, saturated solution of your compound. While cooling, molecules of the same type align in a crystal lattice, forming crystals. After cooling, crystals are collected by vacuum filtration and washed by rinsing with ice-cold solvent. Either the one-solvent or the two-solvent method for recrystallization can be used: Single-solvent method: The chosen recrystallization solvent will dissolve the compound when hot, but not at room temperature. Two-solvent method: The first recrystallization solvent will dissolve the compound at all temperatures. The second solvent will not dissolve the compound at any temperature. The two solvents must be miscible, i.e., soluble in one another, forming a single layer solution. Insoluble impurities can be filtered by hot gravity filtration. Decolorization is dealt with by adding decolorizing charcoal (Norit) and then performing a hot gravity filtration. Return to Top Single Solvent Recrystallization Single solvent recrystallization is the most basic and commonly used recrystallization method. An ideal solvent does NOT dissolve the solid at room temperature BUT dissolves the solid well in hot solvent. Process: 1. Use solubility tests to determine a suitable recrystallization solvent. 2. Heat the solvent and add a minimum of the hot solvent to your crude product to dissolve it (dropwise addition). 3. Hot gravity filter the hot solution if impurities are present. If your solution is colored, use decolorizing charcoal and then hot gravity filter. 4. Allow the hot, clear solution to slowly cool to room temperature (or 0 oC using an ice bath, if necessary). If crystallization does not occur, induce crystallization. 5. Collect crystals by vacuum filtration and wash the crystals using a minimal amount of cold solvent. 6. Allow the crystals to dry. Return to Top Two Solvent Recrystallization Two solvent recrystallization is an alternative and very useful recrystallization method to single solvent recrystallization. The first solvent should dissolve your crude product very well at room temperature (or in hot solvent). The second solvent should NOT dissolve your crude product at room temperature or in hot solvent. The two solvents should be completely miscible and preferably have similar boiling points. Process: 1. Use solubility tests to determine a suitable recrystallization solvent. 2. Heat the first solvent and add a minimum of the hot solvent to your crude product to dissolve it (dropwise addition). 3. Hot gravity filter the hot solution if impurities are present. If your solution is colored, use decolorizing charcoal and then hot gravity filter. 4. Add the second solvent slowly (with shaking) until the solution remains cloudy. Add one or two drops of the hot first solvent until the solution goes clear again. 5. Allow the hot, clear solution to slowly cool to room temperature (or 0 oC using an ice bath, if necessary). If crystallization does not occur, induce crystallization. 6. Collect crystals by vacuum filtration and wash the crystals using a minimal amount of cold solvent. 7. Allow the crystals to dry. Return to Top A Typical Experiment Crystal Line was working with her partner Bea Kurr to purify salicylic acid. They tested the solubility of this solid in several solvents both at room temperature and at the boiling point of the solvent. Crystal observed that the solid was insoluble in water at room temperature, but soluble in hot water. She also noted that the solid was insoluble in ethyl acetate at room temperature. After discussing these observations with Bea, Crystal decided to use the single solvent recrystallization method since the solvents which might be used in the two solvent method are not miscible and thus not suitable. In an Erlenmeyer flask Crystal dissolved about 1 g of the solid in about 5 mL of hot water by heating on a hot plate with swirling to make a fine slurry. After about 1 minute, solid remained and thus she added another 4 mL of hot solvent in portions, with swirling and heating. She continued to add hot water in 2 mL portions with swirling and heating every minute or so until all of the solid dissolved (total of 13 mL of solvent). Since the solution was not highly coloured, Crystal and Bea decided it was not necessary to decolourize it. However, Crystal noted a few insoluble coloured granules in the solution. Thus, she performed hot gravity filtration. She poured the hot solution into a fluted filter paper contained in a hot powder funnel. The receiving Erlenmeyer flask was covered by a beaker and kept hot by heating it in a steam bath. The filtrate was removed from the steam bath and allowed to cool to room temperature. Crystals of pure salicylic acid slowly began to appear in the flask. The flask and contents were cooled further in an ice-water bath for about 20 minutes. Bea set up the vacuum filtration apparatus: a Buchner funnel on top of a filter flask connected to a vacuum trap apparatus. The ice-cold flask and contents were swirled and poured onto the Buchner funnel under reduced pressure. After all crystals were in the funnel, Bea released the pressure and washed the crystals with a little bit of ice-cold solvent. Then she put the vacuum on again. When no more water was seen draining from the filter, she placed the crystals on several filter papers and covered the solid with more filter papers, crushing the acid and pressing firmly to remove as much water as possible. The top filter papers were removed and the product set aside to air dry. When the crystals were completely dry, Crystal crushed a small sample on a porous plate to prepare a sample for the melting point. Return to Top Practical Lab Tips Make sure, that the solvents you add are boiling or hot! It is very important that you add the minimum amount of boiling solvent in order to get a saturated solution. If you add too much solvent, the solution may be too dilute for crystals to form. It is important to slowly cool the flask first to room temperature and then in ice-water. A rushed crystal formation will trap impurities within the crystal lattice. Furthermore, the resulting crystals will be smaller. If none of the solvents tested is suitable for the single-solvent method, use the two-solvent method for recrystallization. In most cases, the single-solvent method is the recrystallization method of choice. If no crystals form, try: 1) scratching the inside of the flask with a glass rod at the interface of the solution or 2) concentrating your solution by boiling off some solvent. You may have too much solvent, i.e., your solution is not saturated, or 3) try the two-solvent recrystallization method. Do not move the flask during the crystal formation phase. Disturbing it can lead to the formation of small crystals and the incorporation of impurities in the crystal lattice. Use the water aspirator as a vacuum source in preference to the house vacuum line, because fumes and gases will dissolve in the water and be diluted and disposed of. The house vacuum line may be used if the water aspirator produces very little vacuum and no noxious gas has been involved in your previous experimental steps. When collecting crystals by vacuum filtration, release the vacuum, rinse your crystals with a little ice-cold solvent, then reapply the vacuum to remove impurities that might stick to the crystals. Return to Top FAQ's Q: The solvent that we use to dissolve the sample for TLC, is that the solvent we will use for recrystallization? This is not necessarily so, but it could be the case. Select a suitable recrystallization solvent by testing the solubility of your unknown solid sample in different hot and cold test solvents. After noting the solubility properties of the solid, you can choose the appropriate recrystallization method. Return to Top Q: How many boiling stones should I use? You want to use 1 or 2 boiling stones for about every 100 mL of liquid. Remember to remove them after recrystallization! Return to Top Q: When I tested the recrystallization solvent in a test tube it worked, but now my sample won't dissolve! Did you have water in your test tube? Did you use the proper ratio of solid (0.1 g) to solvent (1 mL)? Return to Top Q: How long does it take for the crystals to grow? It is impossible to know exactly how long it will take for the crystals to form. Until you have a little more experience, a good rule of thumb is to wait until your flask has slowly cooled to room temperature (touch the flask to check temperature). If no crystals have formed by then, try to induce crystallization by scratching the inner side of your flask at the interface of the solution with a glass rod and wait a few more minutes. When small crystals appear,cool your solution on ice about 15 minutes more. Your crystals should be formed by then. The most important factor affecting recrystallization time is ensuring that you have a saturated solution, obtained by adding the minimum amount of hot solvent to dissolve your crude solid. If you think you might have used too much solvent, you can concentrate your solution by boiling off some of your solvent. Return to Top Q: So - once the solvent has reached its boiling point and my crude solid is NOT dissolved, do I add more solvent or do I let it boil longer? Well, let's think about this. You can let it boil for a moment, but do not wait too long since. The boiling solvent will slowly evaporate, reducing the total volume of solvent added. This means that you need to add even more solvent the next time! The best method is to add hot solvent in timed intervals. This means, once you have added hot solvent, bring the solution to a boil, then wait about 20 seconds. If your crude solid has not dissolved, add more hot solvent, boil again, then wait another 20 seconds. Continue this process until all of your crude solid is dissolved. Return to Top Q: If we add too much solvent, do we just boil it off? Yes, you should reduce the overall volume by boiling off the excess solvent. Reduce the volume until you find that just a little more solvent needs to be added to completely dissolve the crude solid. By this process, you will obtain a saturated solution. If you have a large amount of excess solvent, you can speed up the process of boiling off the solvent by holding a side arm test tube over your Erlenmeyer flask. Connect the side arm of the test tube to a vacuum source, preferably the water aspirator or house vacuum. This process helps remove solvent vapors. Return to Top Q: My sample has dissolved, but my solvent is just hot. Do I have to wait until it boils? It is necessary to use hot solvent, but if your solid sample has already dissolved in hot solvent, this is enough. For some compounds, you might need to wait until the solution boils before your compound completely dissolves. Return to Top Q: After the solution has cooled down to room temperature, how long should I let it cool in the ice bath? You should let the solution cool in the ice bath for a minimum of 15 minutes to ensure that the bulk of the sample has recrystallized. Return to Top Q: Can I put my hot solution directly into the ice bath? No. You need to cool the solution first to room temperature before placing it in the ice-water bath. Besides reducing the risk of breaking your flask and loosing your product in the ice-water, you will get better and purer crystals if you let the solution cool slowly. Return to Top Q: When we are collecting our crystals using vacuum filtration, what solvent do we use to wash our crystals? When vacuum filtering, wash your crystals with the solvent you used to recrystallize your compound. However, use ice-cold solvent to ensure that you do not dissolve any of your crystals. Return to Top Q: I have a really lousy suction from that water aspirator. What can I do? Some possible reasons for little suction are: - The black filter vac adapter between the filtering flask and the Buchner or Hirsch funnel is missing. Make sure it is there and everything is sitting tight - The water trap is not closed to the atmosphere. Return to Top Q: What should I put on the label when handing in my sample? On your sample label, you should write: - your name, - experiment number (e.g., Exp 7), - the name of the product, - the product's melting or boiling point, - the weight of the sample, - the date. Return to Top Q: When using the two-solvent recrystallization method, why is it necessary to keep both solvents hot when adding? For the single-solvent and the two-solvents recrystallization method it is essential that you prepare a hot, saturated solution. To do this, all solvents must be hot before you add them. Heating the solvents decreases the kinetic energy necessary to dissolve the compound. This also means that less solvent is needed to dissolve the compound, which makes the solution more saturated. The more saturated the solution, the easier the crystals will form once the solution cools down. Return to Top Q: Can we add the second solvent first? In the end, all are together anyway. No. You must add a minimum amount of first hot solvent to dissolve your compound. Then you decrease the solubility of your solid by adding the second hot solvent to the first cloud. Reheat the solution to clear it again. This allows for a saturated solution and for crystallization to occur upon cooling. Return to Top Q: If I can choose between the 1-solvent or 2-solvent method, which one should I choose? The preferred method of recrystallization uses one solvent. When a suitable single solvent can not be found, the two-solvent method of recrystallization is used. Return to Top Supported by a grant from the National Science Foundation. NSF Funding {+} This material is based upon work supported by the National Science Foundation under Grant Number CHE-1565813. Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation. Safety Disclaimer {+} The Not Voodoo website is meant to support the education of students doing experimental organic chemistry. By the very nature of laboratory work, chemical experiments can lead to messy, highly toxic, explosive, corrosive, carcinogenic, or otherwise dangerous situations. You are expected to learn and adhere to the safety guidelines in your lab to ensure a safe laboratory environment for both yourself and the people you may be working near. No warranty, guarantee, or representation is made by Not Voodoo as to the accuracy and sufficiency of any information contained herein, and Not Voodoo assumes no responsibility in connection therewith. Furthermore, Not Voodoo is not responsible for the content of external pages that may be provided in the form of weblinks. Recrystallization is a method employed to purify solid compounds. It involves dissolving the impure solid in a heated solvent until the solution reaches saturation, followed by gradual cooling. As the solution cools, pure crystals form while impurities are left behind. These crystals are then collected, washed, and dried, resulting in a purified solid product. Recrystallization is a crucial technique for obtaining high levels of purity in solid compounds.The underlying mechanisms, dissolution, and recrystallization can also minimize the crystal's internal energy in order to reach a more global energy equilibrium resulting a stable polymorph. While recrystallization is usually applied deliberately to optimize crystals and processes, uncontrolled recrystallization can lead to the unwanted formation of hydrates and solvates or polymorph transformation.Crystallization proceeds through a series of interdependent mechanisms uniquely influenced by the choice of process parameters: nucleation, growth, oiling out, agglomeration, breakage, seeding, and polymorph transition.Often, these mechanisms occur simultaneously, making effective crystallization design challenging. Without mechanistic understanding, scientists rely on trial and error to adjust process parameters and optimize yield, purity, and crystal size. By understanding which mechanisms occur during crystallization, scientists can deploy strategies to deliver a crystal product with the desired properties.Read our 7 Crystallization Mechanisms Hiding Between Your Samples Guide to learn how mechanisms can influence the outcome of your crystallization process.This seven-step recrystallization guide provides essential information to design efficient recrystallization processes with optimal product and process performance.The first step of recrystallization is solvent selection. To identify the characteristics of a good recrystallization solvent, several factors should be considered:Solubility: The solvent should have high solubility for the compound to be purified at elevated temperatures, allowing for the efficient dissolution of the impure solid.Selectivity: The solvent should selectively dissolve the desired compound while leaving impurities insoluble or minimally soluble. This ensures effective separation during the recrystallization process.Temperature Range: The solvent should have a suitable boiling point and melting point range to facilitate dissolution at elevated temperatures and crystallization upon cooling.Inertness: The solvent should be chemically inert towards the compound and impurities, ensuring that it does not react or introduce additional impurities during the recrystallization process.Volatility: The solvent should possess adequate volatility to allow for easy removal after recrystallization, typically through evaporation, leaving behind pure crystals.Safety: The solvent should be non-toxic, non-flammable, and environmentally friendly to ensure safe handling during the recrystallization process.Depending on its molecular structure, a solute can be classified as soluble, partially soluble, or insoluble in these solvents or solvent mixtures. To understand this thermodynamic relationship, it is necessary to assess the solute's temperature-dependent solubility behavior in a wide range of different solvents. Solubility can be high or low at room temperature, a strongly increasing function of temperature, or almost independent of temperature at all. High throughput screening methods quickly determine solubility data for a large number of solute/solvent systems.A. Cooling Recrystallization A solute/solvent system with low solubility at low temperature, but with solubility being a strongly increasing function of temperature, is suitable for cooling recrystallization. A large amount of solute can be dissolved at high temperature and due to lower solubility at low temperature, controlled cooling can initiate recrystallization. Temperature sensitive solutes that decompose at elevated temperatures are not suitable for cooling recrystallization.B. AntiSolvent RecrystallizationSolute/solvent systems with high solubility at low temperature and the availability of a miscible antisolvent meet the requirements for antisolvent recrystallization. The controlled addition of antisolvent reduces solubility in the mixture and triggers recrystallization. Two common ways of operation are either antisolvent addition to product solution or product solution addition to antisolvent (reverse addition). The disadvantages of antisolvent recrystallization are introduction of an additional solvent, local high supersaturation at the point of addition, reduced volumetric productivity, and the requirement for solvent separation downstream.C. Evaporative RecrystallizationHigh solubility at low temperature and the unavailability of an antisolvent often requires evaporative recrystallization. The removal of solvent reduces solubility in the remaining mixture and once sufficient supersaturation is created, recrystallization occurs. Challenges in evaporative recrystallization are the introduction of gas bubbles, which can act as a source of nucleation, difficult to predict seeding points and unpredictable scale-up.D. Reaction (Precipitation) RecrystallizationWhen the desired solute is generated through a chemical reaction between two complex compounds or an acid/base neutralization, the method is referred to as reactive recrystallization. The progressing chemical reaction increases supersaturation of the solute, which eventually recrystallizes. Creation of supersaturation can be extremely fast, leading to local high supersaturation at the point of mixing, extensive nucleation, poor process control, and difficult downstream handling.Understanding the solubility behavior of a compound is an important requirement for successful recrystallization process development. Knowledge about how much solute can be dissolved in a solvent and how much solute will remain in the mother liquor at the end is essential to assess recrystallization efficiency. For solution crystallization, the solubility is the maximum amount of solute that can be dissolved in a given amount of solvent at a specific temperature.A system is supersaturated when the concentration of dissolved solute exceeds the solubility limit at a given temperature. Depending on kinetics, the solution has the ability to remain supersaturated over a range of temperature and time before it recrystallizes. The elapsed time between the creation of supersaturation and the formation of the first crystals is called induction time. Increasing supersaturation reduces induction time to a point where the formation of crystals happens spontaneously as soon as supersaturation is further increased. This point is defined as the Metastable Boundary, the difference between solubility curve and Metastable Curve being the Metastable Zone Width.During recrystallization, tight control over supersaturation, nucleation, and growth is required to achieve a final product with desired physical properties. The help of seed crystals is often critical to start the recrystallization processes robustly and repeatedly. Unseeded processes often show uncontrolled spontaneous nucleation, which can lead to extreme process variation, especially during scale-up and large-scale manufacturing. Typically, the following seeding strategies are applied:Tiny (0.1-1 %): Hopefully avoiding oiling out, uncontrolled nucleation, or crashing out. It may be satisfactory in the laboratory, but is rarely effective or robust at larger scale.Small (1-5 %): Controlled nucleation but not enough to exclusively obtain crystal growth. Secondary nucleation along the process and bimodal distributions are likely.Large (5-10 %): Improved probability of crystal growth with the chance to suppress secondary nucleation to avoid bimodal distributions.Minimizing process time, energy consumption, and waste, optimal resource allocation and high process yield are important parameters to produce sellable crystal product at the lowest cost. Based on the solubility data one or more recrystallization methods (e.g. cooling, antisolvent, evaporative, reactive) are applied to reach an endpoint of high yield in the phase diagram.Careful control over the degree of supersaturation and an understanding what particle mechanisms crystals go through is essential to design efficient recrystallization processes with optimal downstream performance.In most recrystallization processes, the solid particles are the desired product, which need to be separated from the mother liquor by filtration. The basic requirements for an efficient filtration process are:Crystal suspension with a low number of fine particlesSuitable filter mediumDriving force (pressure or vacuum)Filtration equipment (for collecting the fluid and retaining the filter cake)After filtration, the cake is commonly washed with an easy to evaporate antisolvent to remove remaining mother liquor and to help with the drying process.The evaporative removal of moisture from the wet filter cake is often the final production step before a usable crystal product is obtained. Depending on the thermal and mechanical stability of the Active Pharmaceutical Ingredient (API) molecule and crystal, the solvent type and the risk of polymorphic transformation, a suitable drying method (atmospheric or vacuum) is applied.EasyViewer is a probe-based imaging tool that captures high-resolution images of crystals, particles, and droplets as they exist in process.Exceptional information-gathering capacity combined with excellent usability makes EasyViewer a compelling tool that scientists will enjoy using to accelerate decision making and speed process development. Perform all steps in a fume hood to prevent exposure to solvent fumes. 1. Selecting a Solvent Place 50 mg of the sample (N-bromosuccinimide) in an Erlenmeyer flask. Add 0.5 mL of boiling solvent (water). If the sample dissolves completely, the solubility in the cold solvent is too high to be a good recrystallization solvent. If the sample does not dissolve in the cold solvent, heat the test tube until the solvent boils. If the sample has not completely dissolved at this point, add more boiling solvent drop-wise, until all of the solid dissolves. If it takes more than 3 mL to dissolve the sample in the hot solvent, the solubility in this solvent is probably too low to make it a good recrystallization solvent. If the first choice of solvent is not a good recrystallization solvent, try others. If a single solvent that works cannot be found, try a two solvent system. If you cannot find a suitable single solvent system, then a solvent pair may be necessary. When identifying a solvent pair, there are several key considerations 1) The first solvent should readily dissolve the solid. 2) The second solvent must be miscible with the 1st solvent, but have a much lower solubility for the solute. As a general rule "likes dissolve likes" meaning that polar compounds tend to be soluble in polar solvents and non-polar compounds are often more soluble non-polar compounds. Common solvent pairs (Table 1) Make sure the solvent has a boiling point of at least 40 °C, so there is a reasonable temperature difference between boiling solvent and room-temperature solvent. Ensure that the solvent has a boiling point below about 120 °C, so it's easier to remove the last traces of solvent from the crystals. Also make sure the boiling point of the solvent is lower than the melting point of the compound, so the compound forms as solid crystals rather than as an insoluble oil. Confirm that the impurities are either insoluble in the hot solvent (so they can be hot-filtered out, once the compound is dissolved) or soluble in the cold solvent (so they stay dissolved during the entire process). 2. Dissolving the Sample in Hot Solvent Place the compound to be recrystallized in an Erlenmeyer flask. This is a better choice than a beaker, since the sloping sides help trap solvent vapors and slow the rate of evaporation. Place the solvent (water) in a separate Erlenmeyer flask, and add boiling chips or a stir bar to keep it boiling smoothly. Heat it to boiling on a hotplate. Add hot solvent to a flask at room temperature containing the compound in small portions, swirling after each addition, until the compound is completely dissolved. During the dissolution process, keep the solution hot at all times by resting it on the hotplate, too. Do not add more hot solvent than necessary – just enough to dissolve the sample. If a portion of the solid does not seem to dissolve, even after more hot solvent has been added, it is likely due to the presence of very insoluble impurities. If this happens, stop adding solvent and do a hot filtration before proceeding. To perform a hot filtration, fold a piece of filter paper into a fluted cone shape and place it into a glass stemless funnel. Add a 10-20% excess of hot solvent to the hot solution to allow for evaporation in the procedure. Pour the solution through the paper. If crystals begin to form at any time during the process, add a small portion of warm solvent to dissolve them. 3. Cooling the Solution Set the flask containing the dissolved compound on a surface that does not conduct the heat away too quickly, such as a paper towel set on a benchtop. Lightly cover the flask as it cools to prevent evaporation and to prevent dust from falling into the solution. Leave the flask undisturbed until it cools to room temperature. Once the crystals have formed, place the solution in an ice bath to ensure that the maximum amount of crystals is obtained. The solutions should be left undisturbed in the ice bath for 30 min to 1 h, or till the compound appears to have completely crystalized out of solution. If no crystal formation is evident, it can be induced by scratching the inside walls of the flask with a glass rod or by adding a small seed crystal of the same compound. If this still fails to work, then too much solvent was probably used. Reheat the solution, allow some of the solvent to boil off, then cool it. 4. Isolating and Drying the Crystals Set the cold flask containing the newly formed crystals on a benchtop. Lightly cover the flask to prevent evaporation and to prevent dust from falling into the solution. Isolate the crystals by vacuum filtration, using either a Büchner or Hirsch funnel (clamp the flask to a ring stand first). Rinse the crystals on the Büchner funnel with a small amount of fresh, cold solvent (the same solvent used for recrystallization) to remove any impurities that may be sticking to the crystals. To dry the crystals, leave them in the filter funnel and draw air through them for several minutes. Crystals can also be air-dried by allowing them to stand uncovered for several hours or days. More efficient methods include vacuum drying or placing in a desiccator. Polar Solvent Less Polar Solvent Ethyl acetate Hexane Methanol Methylene chloride Water Ethanol Toluene Hexane Table 1. Common solvent pairs. Recrystallization is a purification technique for solid compounds. To perform recrystallization, an impure solid compound is mixed with hot solvent to form a saturated solution. As this solution cools, the solubility of the compound decreases, and pure crystals grow from solution. Recrystallization is often used as a final step after other separation methods such as extraction, or column chromatography. Recrystallization may also be used to separate two compounds with very different solubility properties. This video will illustrate solvent selection for recrystallization, purification of an organic compound from solution, and will introduce a few applications in chemistry. Crystallization begins with nucleation. Solute molecules come together to form a stable small crystal, which is followed by crystal growth. Nucleation occurs faster on nucleation sites such as seed crystals, scratches, or solid impurities than spontaneously in solution. Agitation may also encourage rapid nucleation. However, rapid growth can lead to incorporation of impurities if not grown in optimal conditions. The solubility of a compound tends to increase with temperature, and is highly dependent on the choice of solvent. The greater the difference in solubility at high and low temperature, the more likely it is for the solute to come out of the solution as it cools, and form crystals. The solvent chosen should have a boiling point of at least 40 °C so there is a significant temperature difference between boiling and room temperature. The solvent's boiling point must also be below the melting point of the solute to enable crystallization. Rapid cooling of the solution induces the formation of many nucleation sites, thus favors the growth of many small crystals. However, slow cooling induces the formation of fewer nucleation sites, and favors larger and purer crystals. Thus, slow cooling is preferred. Additionally, a solvent can be selected to minimize impurities. If a solution impurity is more soluble than the solute itself, it can be washed off of the fully formed crystals with cold solvent. However, if an impurity is less soluble, it will crystalize first, and can then be filtered out of the heated solution, prior to recrystallization of the solute. If no single solvent has the necessary properties, a mixture of solvents can be used. For a solvent pair, the first solvent should readily dissolve the solid. The second solvent must have a lower solubility for the solute and be miscible with the first solvent. Common solvent pairs include ethyl acetate and hexane, toluene and hexane, methanol and dichloromethane, and water and ethanol. Now that you understand the principles of recrystallization, let's go through a procedure for purification of an organic compound by recrystallization. To begin this procedure, place 50 mg of the sample in a glass test tube. Add 0.5 mL of room temperature solvent. If the compound dissolves completely, the solubility in the cold solvent is too high to be used for recrystallization. Otherwise, heat the mixture in the test tube to boiling. If the compound does not dissolve completely in the boiling solvent, heat another portion of solvent to boiling. Add the boiling solvent dropwise to the test tube until the solid dissolves completely or until the test tube contains 3 mL of solvent. If the solid still does not dissolve, then its solubility in this solvent is too low. Confirm that impurities are either insoluble in the hot solvent so they can be filtered out after dissolution or soluble in the cold solvent so they remain in solution after recrystallization is complete. If a solvent meets all criteria, it is suitable for recrystallization. To start recrystallization, heat the solvent to boiling on a hot plate in an Erlenmeyer flask with a stir bar. Place the compound to be recrystallized in another Erlenmeyer flask at room temperature. Next, add a small portion of hot solvent to the compound. Swirl the mixture in the flask and then place it on the hot plate as well. Repeat this process until the sample has completely dissolved or until addition of solvent causes no further dissolution. Add a 10% excess of hot solvent to the solution to account for evaporation. Place filter paper in a Büchner funnel setup. Filter the solution to remove insoluble impurities. If crystals form during filtration, dissolve them with drops of hot solvent. Cool the solution on the benchtop. Cover the flask to prevent solvent loss to evaporation and to keep particulates out of the solution. Leave the flask undisturbed until it has cooled to room temperature. Agitation during cooling may cause rapid crystallization, yielding less pure crystals. If no crystal formation is evident upon cooling, induce crystallization by gently scratching the inside walls of the flask with a glass rod or adding a small seed crystal of the compound being recrystallized. If crystal formation cannot be induced, reheat the solution to boil off some of the solvent, and then cool the solvent to room temperature once more. Once crystals have formed, prepare an ice bath. Keeping the solution covered, cool the solution in the ice bath until crystallization appears to be complete. Clamp a filtration flask to a ring stand and connect the flask to a vacuum line. Set a Büchner funnel and adapter in the mouth of the flask. Pour the mixture of solution and crystals into the funnel and begin vacuum filtration. Rinse any crystals remaining in the flask into the funnel with cold solvent. Wash the crystals on the funnel with cold solvent to remove soluble impurities. Continue drawing air through the funnel to dry the crystals and then turn off the vacuum pump. If necessary, the crystals may be allowed to stand at room temperature to air dry or placed in a desiccator before storing the crystallized solid. The yellow impurities present in the crude compound have been removed, yielding an off-white solid. Based on the identity of the compound and the impurities, the purity of the crystals can be verified by NMR spectroscopy, melting point measurements, or visual inspection. Purification by recrystallization is an important tool for chemical synthesis and analysis. X-ray crystallography is a powerful characterization technique that identifies the three-dimensional atomic structure of a molecule. This requires a pure single crystal, which is obtained by recrystallization. Some classes of molecules such as proteins are difficult to crystallize, but their structures are extremely important for understanding their chemical functions. With careful selection of recrystallization conditions, even these classes of molecules can be analyzed by X-ray crystallography. To learn more about this process, see this collection's video on growing crystals for crystallography. Impure reactants can cause unwanted side reactions. Purifying reactants by recrystallization improves product purity and yield. Once a solid product has been isolated and washed, reaction yield can also be increased by removing volatiles from the filtrate and recrystallizing the product from the resulting solid. Antifreeze proteins, or AFPs, are expressed in many organisms that live in icy environments. AFPs hinder internal ice growth by binding to ice planes, inhibiting recrystallization into

larger ice crystals. Different AFPs bind to different types of ice crystal planes. Investigating AFP binding mechanisms involves adsorbing them onto single ice crystals. Proper growth of a single ice crystal is essential for clear and informative results. These proteins have applications from the engineering of cold-resistant crops to cryosurgery. You've just watched JoVE's introduction to purifying compounds by recrystallization. You should now be familiar with the principles of the technique, a purification procedure, and some applications of recrystallization in chemistry. Thanks for watching! Written by J.A Dobado | Last Updated on April 22, 2024What is recrystallization?In a typical laboratory experiment, a solid that separates from the reaction crude is usually accompanied by impurities, so it is necessary to carry out purification (recrystallization).By means of a technique called recrystallization, many solids can be purified using pure solvents or solvent mixtures.Recrystallization is based on the different solubility of a solid substance in a solvent at room temperature or when the solvent is hot.The recrystallization process is carried out with loss of product, thus affecting the overall reaction yield.The crucial point in the crystallization process is the choice of solvent which must comply with the following properties:Total solubility of the substance to be purified at high temperatures.Low capacity to dissolve impurities that contaminate the product in any temperature range at any temperature range.Absence of chemical reaction with the product to be purified.Generation of good crystals of the product to be purified.Easy extraction.For practical purposes, two types of recrystallization can be distinguished: in water and in organic solvents.Recrystallization in waterMany organic compounds are insoluble in water at room temperature, but soluble when heated. For this purpose, a suspension of the solid in the minimum amount of water is prepared in a beaker or Erlenmeyer flask, and the mixture is brought to a boil.If the solid does not dissolve under these conditions, small amounts are added and the water is boiled again until the compound dissolves.It should be noted that suspended particles corresponding to part of the insoluble impurities should not be dissolved.If the solid is observed to be dark in color, the addition of small amounts of activated carbon (activated charcoal) (before proceeding to hot filtration) will retain most of these colored impurities.Procedure in waterTransfer the solid to be recrystallized to a beaker.Dissolve the substance in the minimum amount of hot solvent.If the initial crystals exhibit an intense color due to the presence of impurities, add a little activated carbon to remove them (before hot filtering), as the colored impurities are retained in the activated carbon.Heat the mixture to boiling on a hot plate, verifying that the product to recrystallize has completely dissolved. Magnetic stirring (or add a piece of porous plate) should be performed to avoid sudden boiling and the formation of solid splashes.Disconnect the hot plate.Step 1 – Dissolve the solid in water (beaker) Step 2 – Add activated carbon Step 3 – Hot gravity filtration Step 4 – External cool Step 5 – Vacuum filtrationGravity filter the hot solution using a conical funnel and a pleated filter to remove insoluble impurities and activated carbon (discarding the solid residue, composed of activated carbon and insoluble impurities, on the filter paper). Forceps should be used to handle the beaker to avoid burns when handling hot glass vessels.In case the product solidifies in the funnel, add hot water to try to recover as much of it as possible.As the solution cools, crystals of the corresponding product will form. External cooling aids this process.Finally, after the filtrate is completely cooled, the crystals obtained are vacuum filtered and washed (with the cold solvent used in the recrystallization) in a büchner to wash away the adhering product and then dried to remove solvent residues.In case the crystallization is incomplete, concentrate the filtrate (by heating and evaporating half of the solvent) and repeat the process to obtain more of the desired solid product.Recrystallization in organic solventsIn most cases, the organic compound to be purified cannot be recrystallized in water, it does not present a suitable solubility. It is necessary to find a suitable organic solvent.When a volatile organic solvent is used instead of water, the heating part of the solution is performed with a reflux assembly to prevent flammable volatile organic solvent vapors from causing fires.ProcedureA round-bottomed flask with a clamp and nut attached to a metal rack or support is placed on a hot plate.The solid to be recrystallized is transferred to the flask using a solid funnel.We add the solvent and boiling chips (or a stir bar if a magnetic stirrer is available).We connect a reflux condenser to the flask, connecting it to the water circuit.The mixture is heated to reflux to dissolve the solid. Once dissolved, turn off the hot plate and allow to cool to stop refluxing.While the flask is still hot, add activated carbon if necessary and gravity filter the contents of the flask with the help of beacker tongs.Cool the filtrate to room temperature and allow to stand until it crystallizes from the solid.When the filtrate is completely cooled, the crystals obtained are vacuum filtered and washed (with the cold solvent used in the recrystallization) in a büchner to wash away the adhering product and then dried to remove solvent residues.Step 1 – Use reflux for volatile organic solvents Step 2 – Add activated carbon Step 3 – Hot gravity filtration Step 4 – External cool Step 5 – Vacuum filtrationCrystallization is sometimes facilitated by adding a few small crystals of the product or by scraping the bottom of the container with a glass rod. In both cases, crystallization nuclei are generated, which accelerate the process.Main sources of errorThe wrong solvent is chosen for recrystallization.The wrong amount of solvent is used to dissolve the recrystallization solid.No precipitation is observed when the solution is cooled.Formation of an oily substance instead of obtaining a solid precipitate.References Recrystallisation is one of the most common techniques used in chemistry to purify a compound. Choosing your solvent When conducting a recrystallisation it is very important that you select the correct solvent. If you are following a procedure, it will most likely tell you which solvent to use. But when you are left to select the solvent yourself, there are several things to keep in mind. The ideal solvent will dissolve your product at higher temperature but not at room temperature. The idea is that the impurities will remain in the solvent after your product crystallises. A simple way to check if a solvent is suitable is to add a small amount of your product to a sample vial and add room temperature solvent. If the sample dissolves it is unsuitable. If you heat the sample vial you want the sample to eventually dissolve (although you may have to add more solvent). Common solvents for recrystallization include ethers, alkanes and alcohols. Performing the recrystallisation You've selected your solvent and you're ready to begin, now what? You'll need to set up a reflux condenser and add your sample to a round bottom flask. The liquid in the round bottom flask should not exceed the halfway mark so select the flask carefully. You want to dissolve the impure solid in the minimum amount of hot solvent. Start with a small amount in your flask and add more via the condenser until the solid has dissolved. Allow to reflux before cooling. The more slowly you allow the solvent to cool, the larger the crystals will be. Obtaining the product Filter off your solid and wash it with a small amount of cold solvent to remove any impurities that may be stuck to the crystals. Then leave this to dry – you may wish to do so overnight in a desiccator. +2