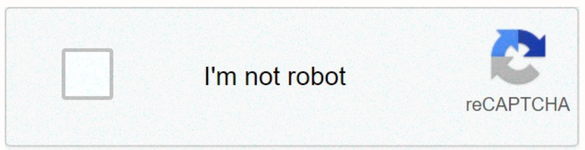

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Best solvent for recrystallization

last updated Friday, April 08, 2016 Since most reactions did not go to complete or allow byproducts, the raw product is more or less contaminated with other compounds. If the product is solid, recrystallization is a common way of cleaning the raw product. The main problem is to find a good solvent for this task. 1. Finding a good solvent and. The compound has a relatively low solubility at all temperatures T (°C) Solubility (mg/ml) 0 1.0 20 1.5 40 2.0 60 2.5 80 3.0 100 4.0 To dissolve a 100 mg compound, 25 ml of solvent is required at 100 oC. After cooling to 0 oC, the 75 mg compound is precipitated and 25 mg remains in the solution. Conclusions for this solvent: 1. 25 % of the compound is lost in solution, 75 % of the compound recovered as a precipitate. Not a good recovery! 2. A relatively large amount of solvent has been used which significantly increases the cost of this purification step. In addition, a lot of waste is also ingly edied! b The compound has a high solubility at all temperatures T (°C) Solubility (mg/ml) 00 50 20 52 40 54 60 56 80 58 100 60 ~100 mg of solvent is required to dissolve the 100 mg compound at 100 oC. After cooling to 0 oC, the 17 mg compound is precipitated and 85 mg remains in the solution. Conclusions for this solvent: 1. Approximately 85 % of the compound remains in solution and is lost, only 15 mg recovered. That's a very poor recovery rate. 2. The amount of solvent is much lower because the overall solubility of the compound is much higher, but due to the low slope of the curve, recovery is very weak. c. The compound displays high solubility at high temperature and low solubility at low temperature. Temperature (°C) Solubility (mg/ml) 00 1.7 10 2.1 20 2.9 25 3.4 30 4.2 40 6.0 50 9.5 60 12 0 70 17,7 80 27,2 90 45,5 95 68,0 To dissolve 100 mg benzoic acid, ~1,5 ml of water is required at 95 oC. After cooling to 0 oC, 97,5 mg of benzoic acid precipitate and 2,5 mg remain in solution. Conclusions: 1. The amount of solvent required is relatively small, saving costs 2. Most of the cleaned sample is recovered (here: 97.5 %) which is very desirable d. Solvent mixtures Although theoretically a single solvent should always be good for reconstallisation, the question is also whether it is readily available and reasonably priced. In many cases, it is also possible to source a mixture of two solvents in order to accomplish the same task. The boiling points of the two solvents should be very similar and the polarity difference should not be too extreme to prevent phase separation if a compound is added. Of course, these two solvents must be wrong in the first place and have different polarities. The following table shows the commonly used solvent mixtures. More polar solvent bp. (C) Less polar solvent bp. (C) Benzene 80 Cyclohexane Ethanol 78 Hexane 69 Ethyl acetate 77 Cyclohexane 81 Ethanol 78 Acetone 56 Chloroform 61 Petroleum ether ~50 Water 100 Ethanol 78 Aqueous acetone 56 However, the solvent mixture should not boil excessively to avoid a significant change in composition which may cause problems during reconstitution or part of the precipitation procedure. 2. Procedure There are different ways to perform a reconstallization. Which one is right for you depends on certain factors, i.e. quantity, amount of solvent required, equipment available for the task and last but not least, how similar the compounds are. A. This compound dissolves and the impurities do not dissolve the compound and remove the impurity by filtration. Strictly, it is not recrystallization, much more extraction. B. The impurities are dissolved and the material in question does not suspend the raw product in a suitable solvent (similar to dissolving) and then filter the mixture. Strictly, it's not really recrystallization, much more mining. c. Both compounds are somewhat similar in solubility The key to a clean sample here is to really dissolve everything. This should be done in an appropriate solvent (as set out in section 1) and at high temperature (solvent boiling point). In many cases, it takes time for all raw product to dissolve. The clock glass with some ice cubes on top of the Erlenmeyer flask allows you to gently reflux the mixture (less solvent evaporates during the heating phase). Do not forget to add a hob, boiling stone or spin bar (this of course should spin) when heating up. If necessary, the resulting mixture may be filtered hot with a short trace funnel and then cooled slowly to obtain clean crystals. The faster the precipitate forms, the more debris is usually trapped in the solid. Slow crystallization allows the compound to properly arrange its molecules in a solid phase and leave impurities in the solution. 3. Problems sensing recrystallization If the compound does not crystallize again from the solution there may be several things you can do: Problem Fix Samples will not dissolve the right solvent or solvent mixture (correct ratio if mixed solvent?) Double-check the solvent Heat the mixture to a boil to dissolve the sample? If not, then you will probably need a lot of solvent. Boil a significant amount off. Sample does not crystallize Have you used too much solvent (see above)? If so, boil part of the solvent from the solution is oversauated Use a seed crystal or scratch the inside of the flask with a glass rod. 4. Adding materials a. See Survival Kit reader: Recrystallization, Vacuum Filtration b. Video sources: Theory, procedure This is a sketch of the reconstallation 1.) Remove the solvent. In the initial organic laboratory course, the solvent for recrystallization is usually intended for you. The criteria used to select an appropriate recrystallisation solvent include: (a.) finding a solvent with a high temperature coefficient. The solvent must not dissolve the compound at low temperatures (including room temperature), but must dissolve the compound at high temperatures. The dissolved substance must be dissolved to remove the bars of impurities, but must not remain dissolved at room temperature (after all, solid regeneration is necessary!). b.) solvent that dissolves impurities easily or at all. If the solvent easily dissolves impurities (even at room temperature), then impurities are not trapped in evolving crystal lattices, but remain dissolved in the solvent. If impurities do not dissolve (even at elevated temperatures), then they can be easily removed by gravity filtration. (see section on gravity filtration) c.) solvent insurance will not react with a spiral. As already mentioned, recrystallization does not chemically alter the molecule. No chemical bonds may be broken in the molecule of the distinguishing process. The crystal lattice dissolves at elevated temperatures, but this involves only overcoming intermolecular attractive forces. d.) using a solvent that is non-flammable, cheap and volatile. Solvents with low boiling points (i.e. volatile) can be easily removed from the resulting crystals by simply allowing the solvent to evaporate. 2.) Dissolve the dissolution. Remember that dissolution should only be dissolved when the solvent is heating up. Therefore, the solvent is heated to boiling point (remember to use boiling stones!) and then slowly added to dissolve. If too much solvent is added, the solution will not become satiated after cooling and crystals will not form. Dissolution of the dissolution generally involves adding a small volume of hot solvent, swirling the flask (or mixing the solution) and monitoring whether the dissolution of the dissolution is dissolved. 3.) Discolour the solution. If the dissolution is to be white in its pure solid state (most organic solids are) and the solution is coloured after dissolving all the solute, it will be necessary to add decolorizing carbon to the solution. This causes colored molecules to adsorb carbon discoloration on the surface, thereby depriving the solution of these impurities. If these impurities remain in the solution, they may be trapped in the developing crystal during cooling. Read the carbon discoloration material. 4.) Filter all solids from the hot solution. If carbon discoloration has been used (as in step or undissolved impurities remain in the hot solution, gravitational filtering of the solution should be filtered while it is still hot. Learn about hot gravity filtration and carbon discoloration. Under no circumstances should the hot solution be vacuum filtered with a Buchner funnel. This leads to the premature development of crystals, since the solution passes through a vacuum filter. (Vacuum reduces pressure, but also temperature.) The dirt will be trapped in the crystal grille and steps 1 to 3 will have to be repeated! 5.) Crystallize solute. This includes allowing the hot solution with the dissolved scent to slowly return to room temperature. The slower the cooling process, the less likely it is to catch impurities in evolving crystal lattices. Allow the solution to reach room temperature. If the crystals have not been formed by the time the solution reaches room temperature, further steps may be needed to induce nucleation. Try the following a.) Take a clean, glass rod and scratch the inner surface of the Erlenmeyer flask. This provides a small stain of glass on which nucleation may occur. b.) Ask a classmate who has recovered a clean greeting to 'donate' a small amount of greeting. Add a small sample of pure frame to the flask. Again, it is thought to provide a place for nucleation. c.) Place the Erlenmeyer flask in an ice water bath. This will dramatically reduce the temperature of the solution. At this lower temperature, saturation conditions can be met, allowing crystals to form. 6.) Collect and wash crystals. The resulting crystals generated by this process may be collected by vacuum filtration, provided that the solution is at room temperature and no further growth of the crystals is apparent. To transfer all the crystals to buchner funnel, add a small amount of cold recrystallization solvent. Remember that solubility is not soluble in cold solvent, so it is safe to use this solvent for crystal transfer. Wash the crystals with a small amount of cold solvent to rinse the dirt from the surface of the crystal. 7.) Dry the crystals. Usually the melting point is taken directly after the cleaning process, so it is necessary to quickly dry the crystals. This is achieved by keeping the crystals in the Buchner funnel and keeping the vacuum for a few minutes. Alternatively, if more time is allocated, the crystals can be safely stored and can sit for several days, allowing the solvent to evaporate over time.

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