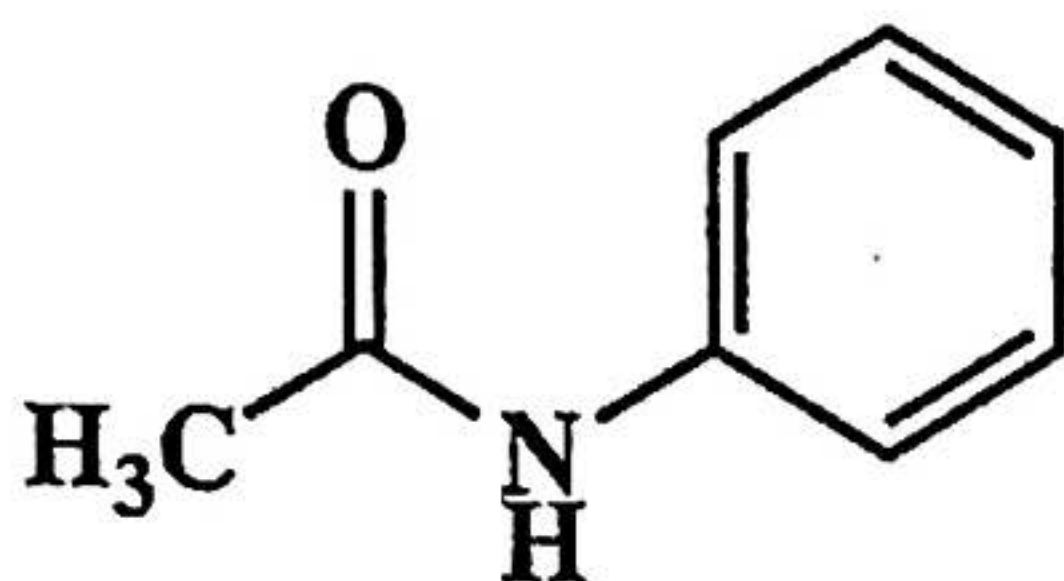


RECRYSTALLIZATION

Purification of Acetanilide by Recrystallization



Introduction

View the Recrystallization DVD in the LRC prior to this experiment. Especially pay attention to the section that describes how to prepare fluted filter paper.

General Methods of Recrystallization

For many years, solids have been purified by recrystallization from suitable solvents. Today this technique still stands as the most useful method for the purification of solids.

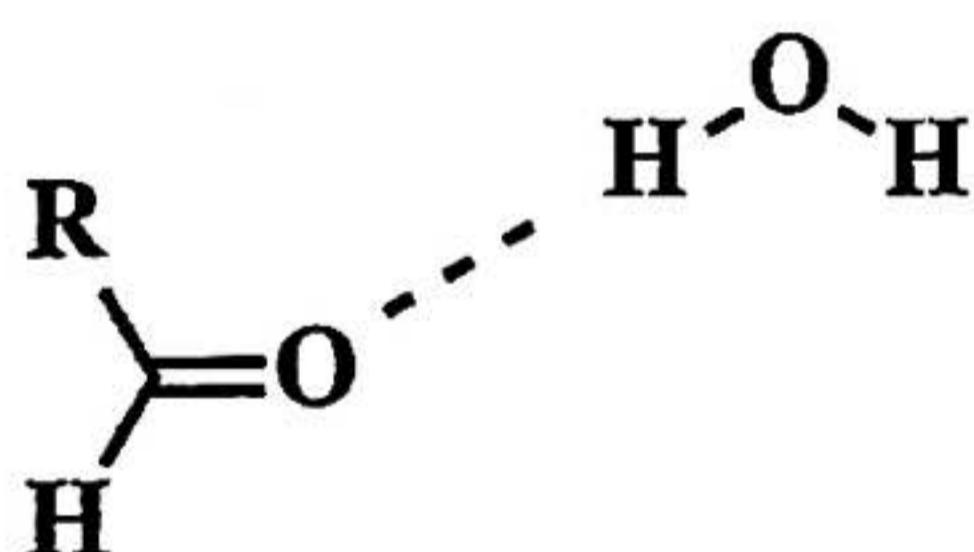
Purification by recrystallization depends upon the fact that most solids are more soluble in hot than in cold solvents. The solid to be purified is dissolved in the hot solvent at its boiling point, the hot mixture is filtered to remove all insoluble impurities and then crystallization is allowed to proceed as the solution cools. In the ideal case, all of the desired substance separates in nicely crystalline form and all the soluble impurities remain dissolved in the "mother liquor." Finally, the crystals are collected on a filter and dried. If a single recrystallization operation does not yield a pure substance, the process is repeated with the same or another solvent.

The great beauty of recrystallization as a purification technique lies in the fact that the orientation of molecules in a crystal lattice is an extremely delicate and selective process. Only infrequently do different substances crystallize in the same lattice. At times, the desired solid can be crystallized selectively from a solution also saturated with other solid impurities simply by the careful introduction of a tiny seed crystal. In such cases, the molecules of the desired compound leave the solution to take positions in the crystal lattice, while the mother liquor remains saturated, or even becomes supersaturated, with respect to the impurities.

- 1 The hydrogen bonds that are important with regard to the water solubilities of organic compounds are almost exclusively those in which hydrogen links oxygen atoms to oxygen or nitrogen atoms.
- 2 One may conveniently divide solutes into three classes: (1) those that are associated in the liquid state; (2) those that are not associated but can coassociate with water; (3) those that are neither associated themselves nor capable of coassociation with water.

Organic compounds such as alcohols, carboxylic acids, amides, and primary and secondary amines (RNH_2 and R_2NH , respectively), whose molecules possess hydrogen atoms capable of forming hydrogen bonds, are themselves associated in the liquid state and can also coassociate with water. Theoretically, at least, such coassociation can proceed through the hydrogen of water or through the hydrogen attached to oxygen or nitrogen in the organic molecule.

Aldehydes, ketones, esters, tertiary amines (R_3N) and similar oxygen- and nitrogen-containing compounds, can coassociate with water, even though they, themselves, are nonassociated because of the lack of a hydrogen-bonding hydrogen. In the coassociation of such non-associated compounds with water, the hydrogen-bonded hydrogen must be supplied by the water molecules. With aldehydes, for example:



Hydrocarbons and their halogenated derivatives neither are associated nor can they coassociate with water.

- 3 An Erlenmeyer flask is much preferred over a beaker. It is more convenient to handle, and loss of solvent by evaporation or boiling, with its attendant fire hazard, is reduced. Furthermore, an Erlenmeyer flask is ideally shaped so that solid material adhering to the walls of the flask can be removed with a spatula.
- 4 Calcium chloride, concentrated sulfuric acid, silica gel and sodium hydroxide are widely used to remove water. Sulfuric acid is effective also for removal of diethyl ether, and silica gel for removal of toluene, diethyl ether, carbon tetrachloride and chloroform.