Rowan College at Burlington County

CHE 241

Lab 2: Recrystallization

Video Explanation of this lab

What is recrystallization?	Here's a lecture video This is the slideshow from the video Here's a second video (not me!) explaining the process
What is melting point determination?	Here's the video for that process This is the slideshow from that video
	This is a video of a version of the lab being done

In this lab you are going to do some of your own research and a little data interpretation. The purpose of the lab is to assess the purity and recovery of three different recrystallizations.

First, read through the procedure. This is the same procedure that is found in the lab supplement on Professor Sherlock's webpage.

Procedure - RECRYSTALLIZATION:

A. Benzil, $C_{14}H_{10}O_2$, from 95% ethyl alcohol

The material that is issued is pure to start with and is used to show the process in its simplest form.

	Water	Ethanol	Ether
Merck	i	s	s
HCP	i	v	v

Weigh approximately one gram of benzil (record actual weight in the Data Table) into a 125 ml Erlenmeyer flask, then dissolve in hot ethanol by gradually adding small quantities of the hot ethanol to the flask containing the benzil. After the initial addition of the hot ethanol, the flask containing the hot ethanol and benzil should also be kept warm until all the crystals have dissolved. The solution is then allowed to cool, to room temperature and then in ice, and the crystals are filtered by vacuum through a Buchner funnel2. (See demonstration apparatus for setup of the filtration apparatus). The crystals are removed from the funnel3, allowed to dry, and saved for Part II. of the experiment. The filtrate is placed in the waste ethanol container.

B. Acetanilide, C_8H_9NO, from water

The starting material is crude and must be decolored using charcoal.

	Water	Ethanol	Ether
Merck	1g/185ml ->	1g/3.4ml ->	1g/18ml ->
	0.5g/100ml	29g/100ml	5.6g/100ml
	1g/20ml boiling ->		
	5g/100ml		

Weigh approximately 1 gram of acetanilide into a 150 ml beaker, add an initial 20 ml of hot water then gradually add hot water to the acetanilide, keeping hot until the crystals just dissolve. When the acetanilide is dissolved, add a slight excess of hot solvent to prevent crystallization during decoloring. The solution is then removed from the hot plate, a "pinch" of charcoal is added to the solution, and the solution is returned to the hot plate for 5 minutes. Make sure that an excess of solvent is still present. To remove the charcoal, the solution is filtered through two fluted filter papers in a stemless funnel, which has been preheated, into a small beaker on the hot plate. Excess solvent is evaporated off if necessary. The filtrate is cooled and the crystals are vacuum filtered as before2. The crystals are removed from the funnel3, allowed to dry and saved for Part II of the experiment. The filtrate may be discarded down the sink drain.

C. 2-Naphthol, $C_{10}H_7OH$, from a mixed solvent

The pure compound is colorless, the technical grade purple. Recrystallization removes much of this color. Treatment with charcoal would remove even more, but is not required in this experiment.

The solubility of this compound in water (0.0876g/100ml of solution at 29.55oC) is too low to permit recrystallization from a convenient volume; the solubility in ethyl alcohol (55g/100ml at 5.5oC) and most other solvents is too high. The best answer is to use a mixture of water and ethyl alcohol.

Weigh approximately 1.0 gram of technical 2-naphthol into a 50 ml beaker, then dissolve in 5 ml of hot ethyl alcohol. Test the suitability of this solvent for recrystallization by cooling the solution. Rewarm the solution to room temperature and slowly add cold water until just reaching the "cloud point" (when the solution becomes slightly cloudy and remains cloudy upon swirling). Place the solution back on the steam bath until the cloudiness disappears. Remove the solution from the steam bath - cool immediately in an ice bath and scratch the sides and bottom of the beaker to promote crystallization. Vacuum filter the crystals as in previous procedures2. Leave the 2-naphthol crystals in the Buchner funnel to dry. The filtrate may be discarded down the sink drain. After drying at least three days, remove the crystals from the funnel3, and save them for Part II of the experiment.

Let's look at the details of each of the three recrystallizations.

The recrystallization of benzil is done from ethanol. Why was ethanol chosen as the recrystallizing solvent?

The acetanilide was “decolored” using charcoal. Why do you think this was done?

The last recrystallization was performed using a mixed solvent of water and ethanol. Why was a mixed solvent used?

In order to do some analysis, the melting point of each compound must be looked up. Record the literature melting points in the table below and give the source of that information.

Compound	Literature Melting point	Reference Source
Benzil		
Acetanilide		
2-Naphthol		

Here are the results from the lab:

Compound	% Recovery	Melting Point Range Trial 1	Melting Point Range Trial 2
Benzil	70	93.4 - 94.3	94.2 - 94.9
Acetanilide	35	112.2 - 112.9	111.9 - 112.7
2-Naphthol	55	120.0 - 121.6	119.7 - 121.3

Based on the information above, make some conclusions.

How well was benzil recrystallized? Support your answer with data from the lab. Make sure to comment on percent recovery and interpret both melting point ranges in the defense of your answer.

How well was acetanilide recrystallized? Support your answer with data from the lab. Make sure to comment on percent recovery and interpret both melting point ranges in the defense of your answer.

How well was 2-naphthol recrystallized? Support your answer with data from the lab. Make sure to comment on percent recovery and interpret both melting point ranges in the defense of your answer.