Chem2211L

Lab Report

Recrystallization

September 27, 2016

Experiment #3: Recrystallization

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Objective: To separate benzoic and acetanilide impurities by the technique recrystallization and to calculate the percent recovery of benzoic acid and acetanilide after recrystallization.

Theory:

As the temperature increases, the amount of solute dissolved in the solvent increases also.

Introduction:

Pure homogeneous compounds consist of the same molecules and have similar structures. These compounds may not be totally pure due to possible amounts of contaminants. The process of recrystallization can be used to purify a solid and remove the impurities. Recrystallization heats or cools a solvent then filters the impurities. Any impurity present in a solid sample will remain in the aqueous solution and only pure solute will be on the filtered paper.

Table of Reagents:

Compound	Structure	Molar mass (g/mol)	Density g/ml	Melting Point^{0}C	Boiling Point^{0}C	Color	Solubility in water	Odor
Benzoic Acid	$C_7H_6O^2$	122.12	1.32	122.00	249.20	White	Soluble in hot water	faint
Acetanilide	C_8H_9NO	135.16	1.12	114.30	304.00	White	Slightly in water	none

Recrystallization:

Compound	Structure	Molar mass (g/mol)	Density g/ml	Melting Point^{0}C	Boiling Point^{0}C	Color	Solubility in water	Odor
Deionized Water	H_2O	18.02	1.00	0.00	100.00	-	-	-

Procedure:

2.0 g of crude benzoic acid, 2 spatulas of activated charcoal, 3 boiling chips, and 30 mL of distilled water were weighed and placed into a 125-mL Erlenmeyer flask and the benzoic acid is heated until dissolved. 50 mL of distilled was heated to boiling in a beaker on a hot plate to use during experiment. The ceramic funnel was heated with blow dryer and the weighed filter paper was placed into funnel, and saturated with hot water. The hot solution was then filtered through the filter. The benzoic acid solution was heated and filtered again to remove remaining impurities and was allowed to cool to room temperature. The solution is then cooled in an ice bath after 15 minutes and vacuum filtrated through the Buchner funnel. Crystals remaining in the funnel is scooped out and placed unto weighed filtered paper. The filtered crystals spread out to dry evenly and allowed to air dry overnight. A separate 125-mL Erlenmeyer flask with 2.0 g of

crude acetanilide, 2 spatulas of activated charcoal, 3 boiling chips, and 30 mL of distilled water was weighed and added to the flask. The above process is repeated with acetanilide.

Results & Calculations:

MW of benzoic acid ($C_7H_6O_2$) = 122.12g, MW of acetanilide (C_8H_9NO) = 135.16g, MW of deionized water (H_2O) = 18.02g.

Weight of crude benzoic acid: 2.0g

Weight of filter paper: 0.18g

Actual weight of benzoic acid: 1.82g

Weight of crude acetanilide 2.0g

Weight of filter paper 0.18g

Actual weight of acetanilide 1.82g

After recrystallization:

Weight of benzoic acid 1.70g

Weight of filter paper 0.18g

Weight of acetanilide: 1.78g

Weight of filter paper 0.18g

Limiting Reactant:

Benzoic acid $2.0\text{ g } C_7H_6O_2 \times \frac{1\text{ mol } C_7H_6O_2}{122.12\text{ g } C_7H_6O_2} \times \frac{18.02\text{g } H_2O}{1\text{ mol } H_2O} = 0.295\text{ mol } C_7H_6O_2$

Acetanilide $2.0\text{ g } C_8H_9NO \times \frac{1\text{ mol } C_8H_9NO}{135.16\text{ g } C_8H_9NO} \times \frac{18.02\text{g } H_2O}{1\text{ mol } H_2O} = 0.266\text{ mol } C_8H_9NO$

Theoretical Yield (T.Y.):

Benzoic acid $1.52\ g \times \frac{1\ mol\ C_7H_6O_2}{122.12\ g\ C_7H_6O_2} \times \frac{18.02g\ H_2O}{1\ mol\ H_2O} = 0.224g\ C_7H_6O_2$

Acetanilide $1.60\ g \times \frac{1\ mol\ C_8H_9NO}{122.12\ g\ C_8H_9NO} \times \frac{18.02g\ H_2O}{1\ mol\ H_2O} = 0.236g\ C_8H_9NO$

% Yield:

Benzoic acid $\frac{0.224g}{0.295g} \times 100\% = 75.9\%$

Acetanilide $\frac{0.236g}{0.266g} \times 100\% = 88.7\%$

Percent recovery:

Benzoic acid $\frac{1.52}{1.82} \frac{(1.70 - 0.18\text{(filter paper)})}{(2.0 - 0.18\ \text{(filter paper)})} \times 100 = 83.5\%$

Acetanilide $\frac{1.60}{1.82} \frac{(1.78 - 0.18\text{(filter paper)})}{(2.0 - 0.18\ \text{(filter paper)})} \times 100 = 87.9\%$

Discussion and Conclusion:

The technique recrystallization was used because it is a simple method to purify a solid compound. The percentage yield for benzoic acid and acetanilide acid being high indicated the initial sample had a low concentration of impurities. With the melting point being in a specific range it is concluded that benzoic acid and acetanilide is a pure substance after recrystallization was done.