

Lab report for Experiment #2: Extraction

Your Name

Name of TA
Lab Section

Title: Experiment #2: Extraction

Observations:

Weighed out 3.2568 grams of chemical mixture

Dissolved dry chemicals in 38 ml CH_2Cl_2 with gentle heating.

Poured solution into sep funnel

Added 10 ml CH_2Cl_2 to flask to rinse, poured solution into sep funnel

Added 15 ml 3 M HCl, gently mixed and vented

Allowed layers to separate

Upper layer determined to be aqueous by density: lower layer removed. Upper layer placed into separate flask labeled "A" for acid extract

Lower layer placed into sep funnel and reextracted with 15 ml 3 M HCl.

Combined aqueous layers in flask A

Reextracted CH_2Cl_2 layer with 15 ml 3 M NaOH

Upper layer in flask labeled "B" for base extract

Re-extract organic layer with 15 ml 3M NaOH

Combined base extracted materials in flask "B"

Dried organic layer with anhyd Na_2SO_4 until free flowing salt was observed (about 3 grams)

Filtered the organic solution into a tared 100 ml round-bottom flask

Rinsed flask and funnel with 10 ml of CH_2Cl_2

Rotovaped off remaining solvent and weighed flask

Weight of flask and biphenyl = 106.1784 g

Weight of empty flask = 104.7368 g

Weight of recovered biphenyl = 1.4416

Cooled flask A in a water bath

Added 18 ml 6 M NaOH and pH tested. Still acidic.

Added 1.5 ml more 6 M NaOH; alkaline solution cloudy

Cooled in ice bath for 21 minutes

Collected p-nitroaniline using vacuum filtration

Washed with cold water and allowed to dry for 12 minutes

Dry solid p-nitroaniline and watch glass = 18.9372 g

Watch glass = 18.0512 g

Weight of recovered p-nitroaniline = 0.8660 g

Cooled flask B in an ice water bath
 Added 16 ml 6 M HCl and pH tested. Still alkaline.
 Added 0.5 ml more 6 M HCl; acidic solution cloudy
 Cooled in ice bath for 25 minutes
 Collected benzoic acid using vacuum filtration
 Dry solid benzoic acid and watch glass = 17.5631 g
 Watch glass = 16.9512 g
 Weight of recovered benzoic acid = 0.6119 g

Discussion:

By adding up the weights of the various components without any loss, I should have recovered 3.2568 g of starting material.

Weight of biphenyl	= 1.4416 g
Weight of p-nitroaniline	= 0.8660 g
Weight of benzoic acid	= 0.6119 g
Total solid recovered	= 2.9195 g

Total recovered mass	= 2.9195 g
Mass of starting material	= 3.2568 g
% recovered	= 89.6 %

I recovered 89.6% of my starting material. Because of solubility issues (difficulty dissolving all of the starting material) I know that some of the starting material never left the first flask. There was a green film all over the glassware indicating an incomplete transfer.

Post-laboratory Questions

1. Why is it better to extract twice with 15 ml portions of solvent 2 instead of once with a 30 ml volume of solvent 2? Assume $K_{2/1} = 4$ and start with 10 grams in 100 ml of solvent 1.

2 15 ml washes remove more material from the solution than 1 30 ml wash.

$15 \text{ ml} \times (4) / 100 \text{ ml} (1) = 0.6/1.0$ for 10 grams of material
 $1 + 0.6 = 1.6$ $10/1.6 = 6.25$ remains in solvent 1 and 3.75 g transfers into solvent 2.

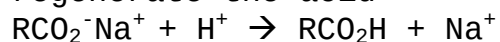
The second extraction $6.25/1.6$ transfers 2.34 g into solvent 2 for a total for two washes of 6.09 g

$30 \text{ ml} \times (4) / 100 \text{ ml} (1) = 1.2/1.0$ for 10 grams of material
 $1.2 + 1 = 2.2$ $10/2.2 = 4.54$ remains in solvent 1 and 5.45 g transfers into solvent 2.

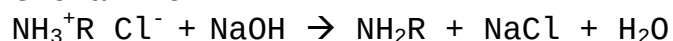
It is obvious that more material was removed with two 15 ml extractions (6.09 g) than in one 30 ml extraction (5.45 g).

2. Write equations for the acid-base reactions involved in this experiment.

$\text{NaOH} + \text{RCO}_2\text{H} \rightarrow \text{RCO}_2^-\text{Na}^+ + \text{HOH}$ This step transfers the benzoic acid from the organic layer when it can be treated with H^+ to regenerate the acid



$\text{H}^+ + \text{NH}_2\text{R} \rightarrow \text{NH}_3^+\text{R Cl}^-$ This step transfers p-nitroaniline the from the organic layer when it can be treated with OH^- to regenerate the amine.



Conclusion:

The implication of "like dissolves like" has implications in daily life from washing dishes and hair all the way to oil spills in the North Atlantic. In this lab exercise we took advantage of the properties of the materials to move them into and out of various phases by using the acid base properties of the compounds. Benzoic acid is soluble in CH_2Cl_2 until it is deprotonated and becomes highly water soluble, but neither the acid nor the base changes the solubility of biphenyl. This is not surprising since biphenyl doesn't contain the kinds of functional groups that would be affected by adding or removing protons.