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Acid and Base Extraction

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Objective

The purpose of this laboratory exercise is to introduce the concept of separating organic compounds by utilizing the concept of acid/base chemistry in order to effect the separation.

Grading

You will be assessed on:

- Completion of separation of organic compounds
- Write-up in your Lab Notebook (see Lab Notebook Guidelines) and answers to the Post-Lab Questions
- TA evaluation of lab procedure

Introduction

Let us first begin with an introduction about extractions. As you learned so long ago, like dissolves like. Sodium chloride will dissolve in water (as an ionic species) but will not disolve in a nonpolar solvent. Nitrobenezene is insoluble in water but soluble in organic

solvents such as methylene chloride. This premise is very useful when you are separating salt from an organic compound but this lab will not be as straight forward as that.

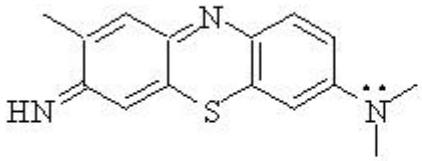
In this lab you will be separating a mixture of four organic compounds. All are soluble in an organic solvent. In order to separate these compounds you will have to use another concept, acid/base chemistry. As you learned in general chemistry a Bronsted-Lowry acid is a proton source and a base is an acceptor. The beauty of this chemistry is the proton transfer leaves you with a charged species (as seen below for HCl reacting with a generic organic base OrgB).

$$HCl + OrgB \rightarrow Cl^- + OrgBH^+$$

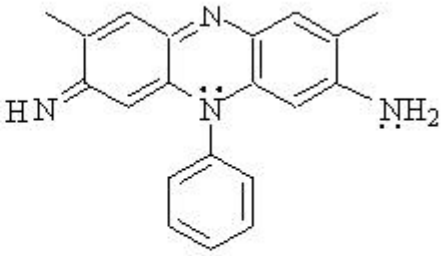
This ionic species is very soluble in water and poorly soluble in a typical organic solvent. You will use this principle in conjunction with extraction to isolate and identify your four compounds.

As mentioned before this will not be a mundane task. You will receive a mixture containing one strong acid, one weak acid, one base, and one neutral compound. You will then be asked to separate and identify these four compounds based on their solubility properties at various pH's. The compounds that you have been given can be identified by their pH dependent colors. When a separation has been achieved, you should have no trouble identifying the mixture. Your compounds will be limited to one of each of the following possibilities:

<u>Bases:</u>

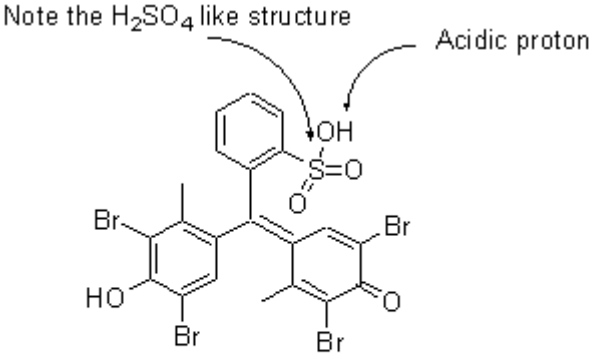


Toluidine blue
Dark blue salt solution
at pH < 7.0

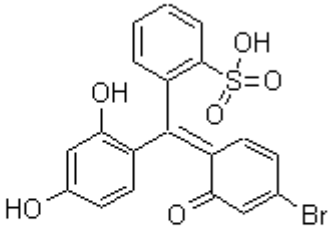


Safranin O
Magenta salt solution
at pH < 7.0

Strong Acids:

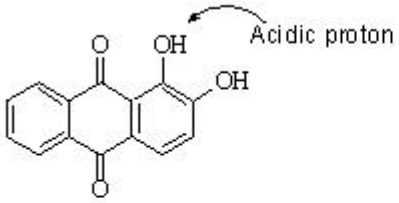


Bromocresol Green
Dark blue salt solution
at pH > 5.4

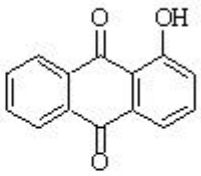


Bromocresol Purple
Magenta salt solution
at pH > 6.8

Weak Acids:

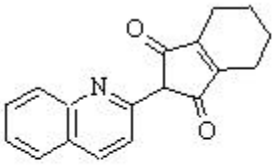


Alizarin
Bluish purple salt solution
pH > 12.1

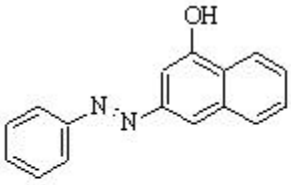


Anthrarufin
Orange salt solution
pH > 12.1

Neutral compounds:



Quinoline Yellow
Yellow 1
at pH < 4.0

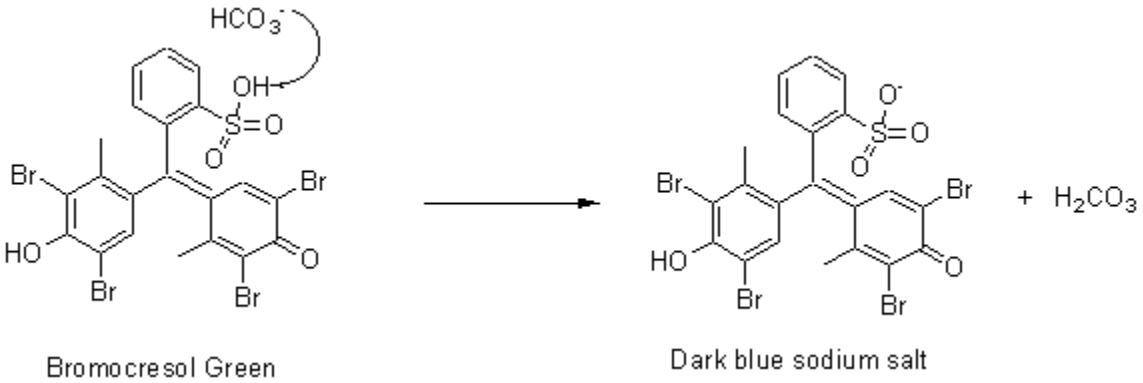


Sudan 1
Bright red-orange in low pH
at pH < 4.0

Experimental

The extractions you will run are very facile. The four unknown samples have been dissolved in methylene chloride, a relatively nonpolar solvent. If you were to add water to your sample you would find that it would separate into two layers: one water layer in which nothing had dissolved and one organic layer in which everything had remained. You will then selectively convert your organic acids and bases into ions that will be soluble in water. *A small hint, the methylene chloride is on the bottom--methylene chloride is one of the few organic solvents that is more dense that water*.

This selective formation of water soluble ions works in the following way. By adding a base, for example, to the sample mixture you receive, you will initiate a reaction between the base you added and an acid already in the mixture:

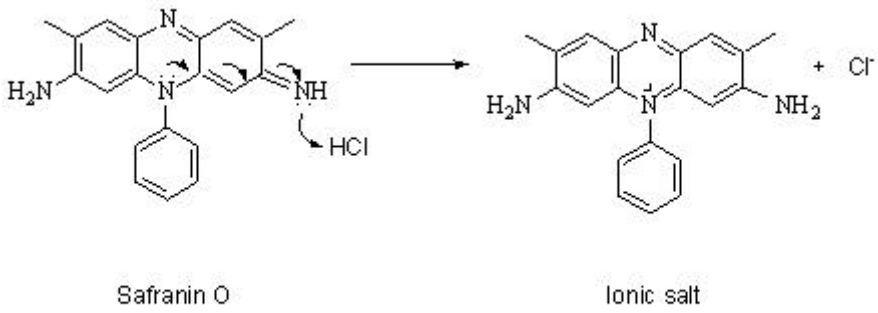


Bromocresol Green

Dark blue sodium salt

In the case shown above, where the acid is bromocresol green, the result is a dark blue salt that is readily soluble in water. When you add aqueous sodium bicarbonate (better known as baking soda) two layers, an aqueous and an organic layer, will form. The salt formed by the weak base and the strong acid will dissolve in the aqueous layer, and you will see a dark blue layer form on top. By removing this top aqueous layer and the compounds dissolved in it, you can remove all of the strong acid that was in the original mixture.

The same general principle applies in removing the weak acid and the basic compound. The weak acid is weak enough that it will not react to form a salt with $NaHCO_3$ but will react with the stronger base, NaOH. Therefore, adding $NaHCO_3$ will not extract the weak acid while NaOH will. To remove the base, we will add an acid, HCl, which will also initiate an acid-base reaction to form a water soluble salt.



Safranin O Ionic salt

Once the base, the strong acid, and the weak acid have been removed from your mixture, you will ideally be left with a pure sample of your neutral compound dissolved in methylene chloride. Remember that this neutral compound has no tendency to react with either acids or bases and will not form salts that allow it to dissolve in water. It will remain permanently in the organic layer, where you will be able to identify it by its color. In reality, some of the acids and bases you extracted will probably be left in the mixture as well, since the reactions may not have gone to completion. As a result you should expect a darker color in the neutral compound than would normally occur. Fortunately, the two compounds you will be given have very strong and bright colors in low pHs, and it is easy to tell the difference between the two by adding a small amount of concentrated acid to these neutral compounds.

Remember to note all the colors you see in this lab, or you'll be unable to identify your mixture.

Procedure

! SAFETY PRECAUTIONS !

! WASTE COLLECTION: All test tubes and pipettes are to be disposed of in the SHARPS CONTAINER and NOT in the garbage!!

1) Obtain 2 ml of your unknown in a 10x 100 mm test tube.

2) Obtain 5 ml of 1M HCl, 5 ml of saturated $NaHCO_3$ and 5 ml of 1M NaOH in separate 10x 100 mm test tubes. Label each.

3) Using a Pasteur pipet, add about 1 ml of 1M HCl to your unknown

4) Mix the sample by pipetting up and down repeatedly

5) Let the tube sit undisturbed until two distinct layers form.

6) Note the color of the aqueous layer. This (upper) layer should indicate which base was in your mixture. Use a **CLEAN** Pasteur pipette to remove all of the aqueous layer and transfer to a clean, labeled test tube.

7) Repeat steps 3-6 two more times. By the third extraction the aqueous layer should be nearly clear. If it is not, continue to repeat the extraction steps until very little color remains in the top layer.

8) Make sure that all of the aqueous layer has been removed and add 1-2 ml of saturated $NaHCO_3$. Mix the sample as before, repeating steps 4-6. Be sure to note the color of the aqueous layer. This will indicate your strong acid. Repeat two more times.

9) Once the strong acid has been removed, use the same procedure with NaOH to extract the weak acid. In this step some of you may have an orange layer on top and an orange layer on the bottom. These are still two different compounds, they just happen to be the same color. You may have to hold the test tube up to the light or view it against a solid background to see the interface of two phases, but it will be visible if you are careful. Again, be sure to note the color of the aqueous layer, as it will tell you the identity of the weak acid.

10) You are now left with just the neutral compound. In order to positively identify it, you may want to place about 0.5 ml in a small test tube and add 5-10 drops of concentrated HCl. This will lower the pH enough so that there will be no ambiguity between the distinct red-orange of Sudan 1 and the light yellow of Quinoline yellow.

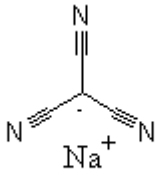
Caution: Concentrated hydrochloric acid is corrosive. Do not let it come in contact with your skin or clothing. If it does, wash it extensively with running water.

11) Using the colors you noted in the aqueous layers, identify the base, strong acid, and weak acid in your solution. The color of your acidified organic layer will allow you to identify the neutral compound. Check to see that you obtained the correct results, and if not explain why this might have happened.

Post Lab Questions:

1) What were the compounds contained in your unknown?

2) If you were given a mixture of trifluoroethanol, sodium methanetricarbonitrile salt (drawn below), para-benzoquinone and trifluoroethanoic acid and were to follow the same procedure as in the experiment (1 M HCl, saturated $NaHCO_3$, 1 M NaOH) what organic compound would come off in the aqueous layer in each extraction? Include your reasoning.



sodium methanetricarbonitrile salt