FTIR Educational Lesson Plan

Extraction lab with FTIR spectroscopy

Objective

- Use acid/base extraction to separate compounds in an unknown mixture. Then, use FTIR spectroscopy to analyze the purity of the extracted compound.

Materials Required

Boiling stick & hot plate	FTIR Spectrometer with ATR	Isopropyl alcohol
Unknown sample (mix of acid and neutral compounds)	Analytical balance	100 mL beaker
Graduated cylinder	Funnel	125 mL separatory funnel
Stand & ring	Ethyl acetate	1.5 M NaOH (aq)
DI water	Saturated NaCl solution	125 mL Erlenmeyer flask
Anhydrous Na_2SO_4	Filter paper	Vacuum funnel

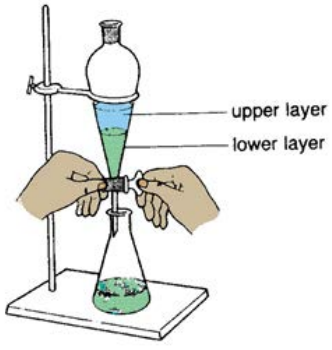
Introduction

- Extraction is the transfer of a solute from one phase/layer to another. Manipulating the acid/base chemistry can separate compounds into different layers. In this experiment, acid/base chemistry is used to manipulate which component is soluble in water, thus giving the ability to separate the components into an organic layer and an aqueous layer.

Discussion

- This experiment involves an unknown mixture comprised of a neutral compound and an acid compound. The neutral compound is uncharged, which means it will not dissolve in water, but will dissolve in organic solvents such as ethyl acetate. The acid compound in this experiment can also dissolve in the organic solvent. The acid compound can also be manipulated with acid/base chemistry to turn it into an ion by adding NaOH. Once the acid compound is ionized, it can dissolve in water. The acid compound can then be shifted back into the organic phase with the addition of HCl, which removes the charge on the compound.

- There are two layers in this experiment. The aqueous layer (composed of water) and the organic layer (composed of ethyl acetate). Density is a very important factor in layer ID. The denser solution will be on the bottom. It is important to have the densities of water and ethyl acetate written down for this experiment.



- Drying the extracts ensures all the water is removed from the organic phase. This step is done to prevent solutes in water from contaminating the final product. Saltwater is used to remove any leftover water through an osmotic effect. Then, anhydrous Na_2SO_4 is used as a drying agent to ensure that all traces of water are removed from the organic layer and the final product is as pure as possible.

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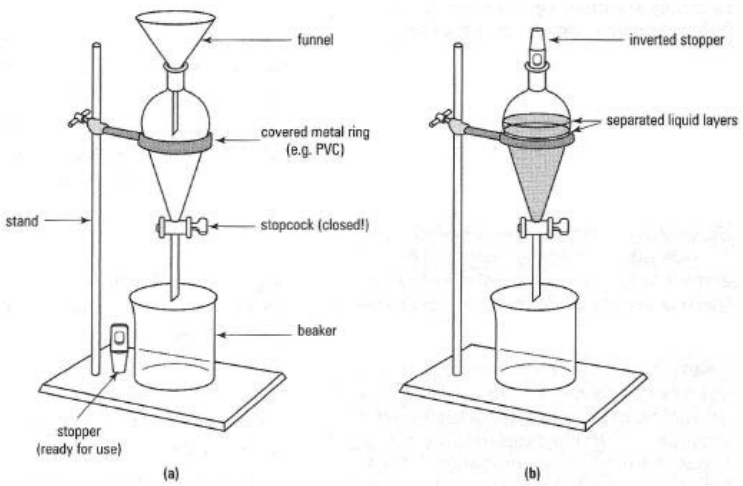
Experiment

First lab period

1. Obtain sample of unknown mixture.
2. Tare the analytical balance with a weigh boat on the balance. Add contents of vial to the weigh boat and record weight.
3. Transfer unknown to a 100 mL beaker.
4. Add 20 mL ethyl acetate to the beaker. Stir until unknown is fully dissolved.
5. Using a funnel, transfer the solution to a 125 mL separatory funnel. Rinse the beaker with two 10 mL portions of ethyl acetate and add to separatory funnel.



6. Add 10 mL of 1.5 M NaOH (aq) to the separatory funnel and shake. Remove the aqueous layer. **SAVE** and label beaker "Aqueous Layer." Repeat again.
7. Add 10 mL of DI water to organic layer in the separatory funnel and shake. Remove aqueous layer. Combine aqueous layer with aqueous solution from Step 6.
8. Add 15 mL of saturated NaCl solution to the organic layer in the separatory funnel to dry the layer. Drain lower aqueous layer and discard.
9. Drain the organic layer from the separatory funnel into a 125 mL Erlenmeyer flask. Add anhydrous Na_2SO_4 drying agent until there is no clumping of the Na_2SO_4.
    a. Filter out the Na_2SO_4 with filter paper and a vacuum flask into a pre-weighed 100 mL beaker.
    b. Rinse the Erlenmeyer flask with 3-5 mL of ethyl acetate and pour into the same filter.
10. Remove the solvent by placing a boiling stick in the 100 mL beaker.
11. Place the beaker on a hot plate and boil off organic layer (solvent).

Aqueous layer

1. Add concentrated HCl dropwise to the labeled aqueous layer beaker until the solution tests acidic on litmus paper. Then, add a few drops of HCl in excess. Check for complete precipitation.
2. Add 30 mL ethyl acetate and extract (shake separatory funnel).
3. Drain aqueous layer and discard.
4. Add 15 mL of saturated NaCl solution to the organic layer in the separatory funnel to dry the layer. Remove the lower aqueous layer and discard.
5. Drain the organic layer from the separatory funnel into a 125 mL Erlenmeyer flask. Add anhydrous Na_2SO_4 drying agent until there is no clumping of the Na_2SO_4.
    a. Filter out the Na_2SO_4 with filter paper and a vacuum flask into a pre-weighed 100 mL beaker.
    b. Rinse the Erlenmeyer flask with 3-5 mL of ethyl acetate and pour into the same filter.
6. Remove the solvent by placing a boiling stick in the 100 mL beaker.
7. Place the beaker on a hot plate and boil off the organic layer (solvent).
   SAVE both solid components in separate labeled vials for next lab.

Second lab period

Initial analysis without search

Summit with Everest ATR accessory connected to network

1. Obtain a small amount of the initial unknown.
2. Clean the ATR crystal with isopropyl alcohol. To prevent scratching the crystal, do not use a Kimwipe to clean the crystal. Use paper towel instead.
3. Make sure the Paradigm software is open in desktop mode. On the dashboard, go to “New measurement” and enter a Measurement name
4. Set final format, sample scans, and resolution to class specifications.
5. Select “None” for analysis type.
6. Click on “Preview and Measure Background” and then click on “Start Background Measurement.”
7. Once the background measurement is complete, place the initial unknown on the crystal, ensuring there is enough sample to fully cover the crystal.
    a. Lower the pressure tower by rotating the knob clockwise until you hear a “click.”
8. Click on “Measure Sample” to begin analysis of the sample.
9. Click on File and select “Export.” Save to your class network folder.
10. Remove the unknown sample from the ATR crystal and clean the crystal surface with isopropyl alcohol and paper towel.
11. Repeat Steps 1–10 with the solid neutral component and the solid acid component until you have three spectra total.

After obtaining spectra

Data interpretation – OMNIC Paradigm

- In OMNIC Paradigm, spectral data is automatically saved in the “Measurements” section on the Dashboard after acquisition.
- The goal is to overlay the neutral and acid component spectra with the initial unknown spectrum to see a rough estimate of the purity of the components.
    – At the Dashboard, the Measurements section is in the middle. Find your three spectra (neutral component, acid component, and unknown). Hold Control while selecting your spectra. Right click on the selected spectra and click “Open Selected Measurements.”
    – The spectra will overlay. On the right side click the eye icon to select or deselect a spectrum.
- Identify functional groups and structures in the spectra of the components to help determine the identity of the components.
- State your assumptions about the identify and purity of each component.

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Utilizing search for data interpretation

1. From the Dashboard, scroll to “Measurements.” Select the the measurement corresponding to the solid neutral component and open it by double clicking.
2. Once opened, the spectrum will be in the spectral view. From here, go to the “Identify” menu and select “Correlation Search.”
3. Once the search is completed the spectral pane will display the sample spectrum as well as the best matched spectrum from a library. The best matched spectrum should be the identity of the component.
4. Repeat Steps 1–3 for the solid acidic component.
5. After the components have been matched, go to the Dashboard.
6. From there, select the measurement corresponding to the initial unknown and open it.
7. Select “Identify” and click on “Multi-Component Search”
8. Once the search is completed the spectral pane will display the sample spectrum as well as the best matched composite set of spectra.

After search is complete

- What is the identity of each of the components?
- Did the multi-component search results match the ID of your extracted components?
- Do the components appear to be pure or are there other contaminants? Are the components still somewhat mixed together?
- What is total percent recovery?
  - (Total % recovery = %A + %B)
    - %A = (weight A/starting weight of unknown) * 100

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