Development of a Two-Week Discovery-Based Technique Lab for Undergraduate Organic Chemistry

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Abstract

The purpose of this experiment is to develop a discovery-based undergraduate organic chemistry lab focusing on technique. The lab, which is intended to be taught early in the organic course, teaches students to use the following techniques: thin-layer chromatography (TLC), liquid-liquid extraction, and recrystallization. As understanding of these techniques is important in organic chemistry, this lab is not a step by step procedure, meaning it requires the students to use their knowledge of chemistry to progress throughout the lab. Students will be given an unknown mixture containing two compounds, a neutral and either an acid or an amine base. These will come from a list of 9 possible compounds. The students will then use TLC to propose the identity of the compounds in the mixture. Next, liquid-liquid extraction will be used to separate the two compounds, with separate procedures for acids and amine bases. The separated components will then be recrystallized so that a melting point may be determined. Finally, a portion of the crystals will be used to make a TLC plate to compare the purified products to a standard.

Goal

To replace traditional organic technique labs (crystallization, TLC, liquid-liquid extraction, use of rotary evaporators) with a single two-week discovery based lab to be performed at the beginning of a semester.

Background

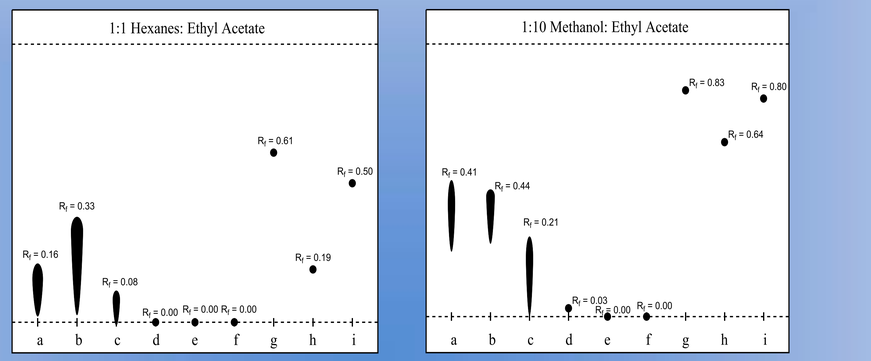
Discovery-based labs or guided-inquiry labs are replacement labs for the more traditional "cookbook" labs often seen in chemistry courses. They are viewed as more practical than other replacements for traditional laboratories, as they do not require the student to develop their own procedure and have a set outcome unlike open-inquiry.[1] Students in discovery-based labs have reported they feel they have a better understanding of the material presented in the lab.[2]

Thin Layer Chromatography (TLC) and liquid – liquid extraction (LLE) are common procedures for both research and undergraduate teaching labs. Despite these procedures' importance, in many cases, one or both do not have an organic lab dedicated to themselves. In an attempt to express the importance of them, it has been proposed that three weeks be dedicated to each in an extended discovery based lab.[3,4] Although this is a step in the correct direction, many do not have the ability to dedicate six weeks to learning TLC and LLE, as other topics must be taught in the introductory organic courses. As such we have developed a two week guided- inquiry lab dedicated to teaching students techniques, including LLE, TLC, and recrystallization.

Experiment

Week 1

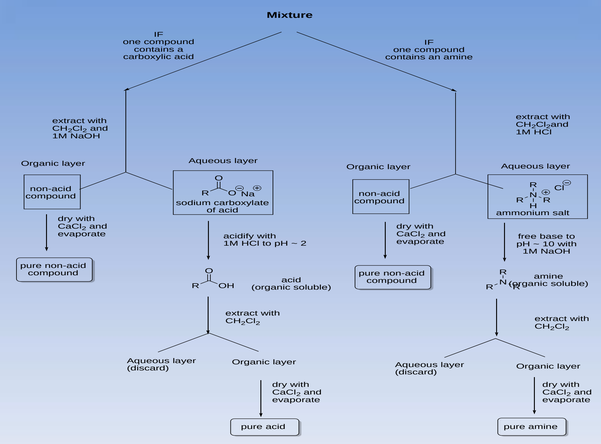
1. The students obtain an unknown containing 100mg an acid or amine and 100mg of a neutral compound.
2. The students run TLC plates of standards and their unknown in: 1:1 Ethyl Acetate:Hexane and 1:10 Methanol: Ethyl Acetate
3. The TLC plates are viewed by UV and then stained in PMA; the amines are stained in ninhydrin.
4. The students then propose if the mixture contains an acid or an amine.



a: mandelic acid (light blue UV, Green PMA)
b: salicylic acid(Dark UV, light brown PMA)
c: aspirin (light Blue UV)
d: quinidine (Blue UV, Dark Brown Ninhydrin)
e: pseudoephedrine (UV, brown ninhydrin, purple PMA)
f: atropine (faint blue UV, Brown Ninhydrin)
g: desoxyanisoin (UV, Black PMA)
h: phenacetin (UV, pink PMA)
i: dihydroxynaphthalene (UV, Black PMA)

Week 2

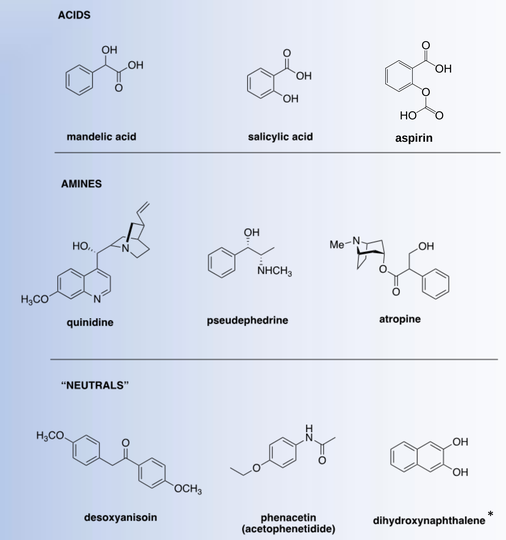
1. Following the chart below, the students separate their compounds using liquid-liquid extraction.



2. The compound obtained from the extraction will then be isolated by evaporating solvent in a rotary evaporator.
3. The weight of the compound will be determined so that percent recovery may be calculated.
4. A portion of the compounds will be recrystallized in appropriate solvents.
5. The melting range will be determined, and in combination with previously collected data, the student will propose what compounds made up the unknown.

Results

Compounds that may be used in the unknown



* Cannot be used in an unknown containing an acid

- 9 compounds were identified to be used in this experiment: 3 Acids, 3 Amines, and 3 Neutrals.
- 11/ 18 potential combinations have been tested.
- It is expected that the lab will be tested in a teaching lab in the near future.

Problems encountered

- Dihydroxynaphthalene is too acidic to be separated via liquid-liquid extraction, which reduces the potential combinations to 16.

References

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2. Allen J.B. Guided inquiry laboratory. *J. Chem. Educ.* **1986**, 6, 533-534
3. Wu, N Adapting Meaningful Learning Strategies for an Introductory Laboratory Course: Using Thin-Layer Chromatography to Monitor Reaction Progress. *J. Chem. Educ.* **2019,** 96, 1873-1880
4. Wu, N Adapting Meaningful Learning Strategies to Teach Liquid -Liquid Extractions. *J. Chem. Educ.* **2020**, 97, 80-86

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