A Cost-Effective Two-Part Experiment for Teaching Introductory Organic Chemistry Techniques

ARTICLE *in* JOURNAL OF CHEMICAL EDUCATION · OCTOBER 2011

Impact Factor: 1.11 · DOI: 10.1021/ed101039m

3 AUTHORS, INCLUDING:

Brenna Brown
University of Alberta
4 PUBLICATIONS **4** CITATIONS
SEE PROFILE

Hayley Wan
University of Alberta
12 PUBLICATIONS **228** CITATIONS
SEE PROFILE

JOURNAL OF CHEMICAL EDUCATION

LABORATORY EXPERIMENT

pubs.acs.org/jchemeduc

A Cost-Effective Two-Part Experiment for Teaching Introductory Organic Chemistry Techniques

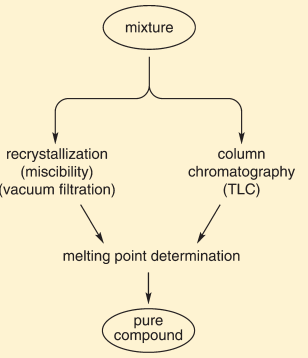
Christopher M. Sadek, Brenna A. Brown, and Hayley Wan*

Department of Chemistry, University of Alberta, Edmonton, Alberta, T6G 2G2, Canada

Supporting Information

ABSTRACT: This two-part laboratory experiment is designed to be a cost-effective method for teaching basic organic laboratory techniques (recrystallization, thin-layer chromatography, column chromatography, vacuum filtration, and melting point determination) to large classes of introductory organic chemistry students. Students are exposed to different methods for the separation of mixtures containing a nonpolar organic compound (e.g., biphenyl) and a polar organic dye (e.g., methyl orange). Students are also introduced to solubility and miscibility using common organic solvents such as methanol, dichloromethane, hexane, acetone, and water. This experiment requires two approximately 3-h laboratory periods.

KEYWORDS: First-Year Undergraduate/General, Second-Year Undergraduate, Laboratory Instruction, Organic Chemistry, Physical Chemistry, Hands-On Learning/Manipulatives, Chromatography, Microscale Lab, Physical Properties, Thin Layer Chromatography



This two-part experiment is designed to be a cost-effective method to introduce organic chemistry students to basic techniques such as recrystallization, vacuum filtration, thin-layer chromatography, column chromatography, and melting point determination. Cost is a major factor when designing experiments for beginning organic chemistry students that provide hands-on experience and successfully illustrate basic organic laboratory concepts and techniques.[1] The experiment has been successfully implemented during the first two laboratory periods in the introductory organic chemistry laboratory course, where student enrollment can reach over 1200 students per semester. At any one time, a maximum of 180 students performed this experiment, with a student/instructor ratio of 20:1. The experiment is designed so that it can be completed within two laboratory periods lasting 2 h and 50 min each. (All laboratory periods also include a 10−15 min laboratory lecture and a 10 min quiz.) Although there is sufficient time for students to complete the experiments while working alone, students can also work in pairs to help minimize the cost. The estimated cost per student for this two-part experiment is $0.84 (Canadian dollars), based on individual student work. In addition to the low cost of the experiment, the volume of solvent used is low, which makes the experiment safer and reduces the cost for chemical waste disposal.

The easily prepared solid samples for the experiment are used for both laboratory periods. The sample consists of a nonpolar organic compound such as naphthalene or biphenyl (Figure 1), mixed with a small quantity of a polar organic dye (e.g., methyl orange). The sample appears orange and consists of 700 mg of the nonpolar compound and 10 mg of the polar organic dye. Students use some of the mixture for recrystallization of the nonpolar component during the first laboratory and the remainder of the mixture for column chromatography during the second laboratory. Note that naphthalene has a strong odor; therefore, biphenyl is recommended for laboratory classes with a large number of students and minimal fume hood space.

EXPERIMENT

Solvent Miscibility and Recrystallization

In the first laboratory period, students are eased into the new laboratory environment by carrying out a series of miscibility tests between different solvents and water. Students are asked to determine the miscibility of methanol, acetone, dichloromethane, toluene, and hexane with water and predict which layer is the organic solvent (if immiscible). To predict the organic layer, the students are asked to add a few drops of the organic solvent to the liquid mixture and see which layer the drops add to.

Next, the students are asked to identify a suitable recrystallization solvent for their sample by determining the solubility of the pure nonpolar component in both room temperature and hot solvents (methanol, acetone, dichloromethane, hexanes, and water). Students have previously been told that their sample consists of either a biphenyl−methyl orange mixture or a naphthalene−methyl orange mixture. The students are asked to put five clean, labeled test tubes into a test tube rack and place approximately 10 mg of the pure nonpolar component standard into each test tube. They then add a few drops of each solvent into the corresponding test tube and record the solubility of the solid at room temperature. Following this, the students heat each test tube using a steam bath and record the solubility of the solid in boiling solvent. Although students are told which recrystallization solvent should be used, they should attempt the solvent determination to see if their results are consistent with the

dx.doi.org/10.1021/ed101039m | J. Chem. Educ. XXXX, XXX, 000−000

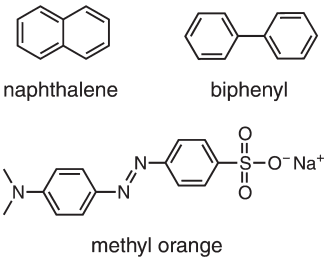


Figure 1. Structures of the compounds used in the student samples.

suggested recrystallization solvent. For smaller classes, the students could determine the recrystallization solvent to use.

The remainder of this first laboratory is dedicated to recrystallization of the provided student sample. The students are asked to transfer 0.5 g of their sample to a small Erlenmeyer flask and to slowly add hot hexanes over a steam bath until no more solid dissolves. They can then filter out the polar organic dye and evaporate the hexanes in a preweighed filter flask to yield crude biphenyl or naphthalene. The crude product is weighed and then recrystallized from methanol to yield pure crystals that can be collected by vacuum filtration. The recrystallized sample should be weighed and then saved for analysis during the following laboratory class.

Thin-Layer Chromatography, Column Chromatography, and Melting Point

During the second laboratory period, students learn how to perform thin-layer chromatography (TLC) analysis, separation by column chromatography, and melting point determination. First, students run a TLC of their original sample mixture against the pure product (biphenyl or naphthalene) that they obtained from recrystallization in the first laboratory period. Using UV light to visualize their plates, the students observe that the original sample has two spots and their recrystallized product has only one spot. Second, the students use 50 mg of their sample mixture and run a silica pipet column[2] using hexane/ethyl acetate (4:1) as the eluent. The students dry pack a pipet column by placing a small plug of cotton wool into the bottom of a long Pasteur pipet (~220 mm) and filling the pipet to two-thirds full with silica. This step is carried out in a fume hood. The column is first flushed with the eluent, and the sample (dissolved in two drops of acetone) is then loaded onto the surface of the silica. Instead of using a pipet bulb as a source of pressure for the column, the students use a syringe adapter, as reported by Baldwin.[3] The syringe adapter consists of a 10 mL plastic syringe connected to a 2 in. piece of $^1/_4$ in. diameter tubing. The open end of the tubing then connects to the top of the Pasteur pipet.

As the students run the pipet column, they collect at least three 1 mL fractions in separate test tubes. Once they have determined by TLC which fractions contain only the desired compound, they can combine the fractions and evaporate the solvent under reduced pressure, using a preweighed filter flask to obtain the pure product. The students can then compare the efficiency of purification via recrystallization to purification via column chromatography by comparing TLC plates, melting points, and yields.

Finally, the students carry out melting point analysis on (i) the original sample mixture, (ii) the recrystallized product, and (iii) the product purified by column chromatography. This teaches the students how to assess the purity of a sample using melting point data.

HAZARDS

Methanol, acetone, toluene, and hexane are flammable and should be kept away from open flames. Dichloromethane is a carcinogen and should be used in the fume hood. Silica gel for the pipet column can cause silicosis. Avoid breathing in the silica dust and load silica into the pipet in the fume hood. Biphenyl is a skin and eye irritant. Any spills on the skin should be washed off immediately with water. Naphthalene is a flammable solid and carcinogen. Keep away from open flames and avoid breathing in the vapors.

RESULTS AND DISCUSSION

In the first part of the laboratory, students are able to visualize and understand the terms miscible and immiscible with regard to different solvents. Dichloromethane is used as one of the solvents to illustrate solvent density. Following this, students learn that the first step of recrystallization is to carry out solubility tests of their compound. They perform solubility tests initially in room temperature solvents and then in hot solvents and compare their results with the suggested solvent.

As this is the first time that the students are performing a recrystallization, they are told which solvent to use. The students add hot hexanes to their sample until no more solid dissolves. The biphenyl and naphthalene dissolves readily in hot hexanes but the polar organic dye does not. The insoluble dye is then filtered off. The solvent is evaporated to leave crude biphenyl or naphthalene and the students are instructed to recrystallize the product from methanol. Both biphenyl and naphthalene crystallize readily from methanol. At the beginning of the laboratory class, the teaching assistant demonstrates how to make a hot saturated solution of the crude sample and how to collect the pure product crystals by vacuum filtration.

In the second part of the laboratory, students run a TLC of their original sample mixture against a sample of their recrystallized product. Using hexane/ethyl acetate (4:1) as the eluent, the organic dye remains close to the baseline (R_f = 0.03) and biphenyl and naphthalene follow the solvent front (R_f = 0.77 and 0.83, respectively). This large separation in R_f values between the organic dye and the nonpolar compound helps students better grasp the concept of polarity and ensures that students can successfully and quickly separate the compounds by column (pipet) chromatography.

Using hexane/ethyl acetate (4:1) mixture, the students can easily separate the nonpolar compound from the polar organic dye. By using a dye as the polar impurity, students are given visual evidence of a polar compound being retained by the silica.[4] Students obtain further practice in TLC analysis when they have to analyze each fraction to determine which fractions can be combined. Once the students have evaporated the solvent and obtained the yield of the product, they are able to compare the efficiency of purification by recrystallization and column chromatography.

Finally, the students learn how to determine the melting point of their crude and pure products. Pure biphenyl and pure naphthalene have melting points of 68−70 and 80−82 °C, respectively.[5] The melting point of the crude sample cannot be determined as the nonpolar compound has a much lower melting point than the polar organic dye. Methyl orange has a melting point of greater than 300 °C (but the thermometers used in our laboratories cannot exceed a temperature of greater than 300 °C).[5]

dx.doi.org/10.1021/ed101039m | J. Chem. Educ. XXXX, XXX, 000–000

CONCLUSION

This experiment teaches students that impure samples can be purified by recrystallization or column (pipet) chromatography and that purity of a sample can be determined by TLC and melting point determination. The use of a colored polar organic dye is helpful for the students especially during pipet chromatography as they are able to see movement of the colored band and hence not contaminate their pure fractions. This experiment is a cost-effective method of teaching basic organic laboratory techniques to large numbers of chemistry students. For less than $1.00 per student, this two-part experiment teaches recrystallization, vacuum filtration, column chromatography, TLC, and melting point determination. Additionally, this two-part laboratory uses relatively small volumes of solvent, which is safer and reduces the amount of chemical waste.

ASSOCIATED CONTENT

Supporting Information

List of chemicals and apparatus; student handout. This material is available via the Internet at http://pubs.acs.org.

AUTHOR INFORMATION

Corresponding Author

*E-mail: hayley.wan@ualberta.ca.

ACKNOWLEDGMENT

The authors would like to thank Dennis Hall and Nada Djokic for helpful feedback and proof-reading. The help and support from the Chemistry Storeroom staff (Andrew Yeung, Marcel Munroe, and Matthew Kingston) in preparing the student samples and supplying the syringe adapter for each student was greatly appreciated. The authors also thank the 2010 spring session teaching assistants and students for testing the experiment and providing feedback.

REFERENCES

(1) Bradley, J. D.; Durbach, S.; Bell, B.; Mungarulire, J.; Kimel, H. *J. Chem. Educ.* **1998**, *75*, 1406–1409. Chen, C.; Dyer, J. M.; Strawbridge, P. B. *J. Chem. Educ.* **1996**, *73*, A236–237. Neckers, D. C.; Duncan, M. B.; Gainor, J.; Grasse, P. B. *J. Chem. Educ.* **1977**, *54*, 690–692.

(2) Wigman, L. S.; Kelsch, C. T. *J. Chem. Educ.* **1992**, *69*, 991–992.

(3) Baldwin, B. W. *J. Chem. Educ.* **2003**, *80*, 1182.

(4) Heumann, L. V. *J. Chem. Educ.* **2008**, *85*, 525.

(5) Sigma-Aldrich Home Page. http://www.sigmaaldrich.com/canada-english.html (accessed Jun 2011).