What's In That Pill?

A Thin-Layer Chromatography Activity

for High School Chemistry Classes

CCMR Summer RET Experience
Summer 2002

Mary Kay Hickey

Support for Cornell Center for Materials Research RET Program is provided through
NSF Grant DMR-0097494

Acknowledgements

Development of this lab activity would have been impossible without the financial, technical and logistical support of the Cornell Center for Materials Research, particularly Nev Singhota, the Educational Programs Director for the Center.

Also essential to the project were Dr. Tyler McQuade of Cornell University who hosted my research experience in his lab, and his graduate student Steve Broadwater who trained me in some of the finer points of TLC.

Connection to NYS Regents Chemistry Core Guide

Key idea 3.1s
Mixtures are composed of two or more different substances that can be separated by physical means. When different substances are mixed together, a homogeneous or heterogeneous mixture is formed.

Key idea 3.1nn
Differences in properties such as density, particle size, molecular polarity, boiling and freezing points, and solubility permit physical separation of the components of the mixture.

Skill
Students should be able to describe the process and use of filtration, distillation, and chromatography in the separation of a mixture.

Students often have difficulty with the difference between a mixture and a compound. As a result, separation techniques are something of a mystery to them. Prior to completing my CCMR RET summer, my students were exposed to a filtration and distillation, but not to chromatographic separation. Since this is now one of the techniques that students are expected to be acquainted with, it seemed appropriate to develop an activity that highlights chromatography.

This lab activity fits nicely into the spring of the year, where it also provides an opportunity to review the following topics:

1. atomic spectroscopy – the concept of energy absorption and release by electrons is similar, although not identical to fluorescence
2. molecular polarity
3. solution chemistry – like dissolves like
4. organic nomenclature and structural formulas
5. vapor pressure

Support for Cornell Center for Materials Research RET Program is provided through NSF Grant DMR-0097494

Development of the Lab Activity

Much of the technology that I was exposed to during my RET summer is beyond the limited financial resources of my high school. Even TLC had previously seemed out of reach because of the cost of plates. When I saw how TLC was used everyday in the lab on a small scale, I realized that, with an engaging activity and plates cut to a small size, TLC could become a very doable project.

I found a number of TLC activities on the internet, primarily designed for college students. While some required fairly toxic materials and individual fume hoods, others seemed to lend themselves more to the high school setting. Given the popularity of forensic science among high school-aged television viewers, I chose an activity that involved identification of the active agents in over-the-counter analgesics. High school students require a completely different level of background and structure than college students, so I revised the college-level materials for my own students.

The student version of the activity was written following the summer RET experience. I ran it with my own 11th grade students during the '02-03 school year and made some additional revisions. In its current form, it should be usable in any high school chemistry class.

Helpful Information for High School Teachers – What I learned by Experience

Based on my personal experience with this activity and my own limited experience with TLC, the following may be useful.

1. Ideally, this activity should be run during an 80-minute block of time. In a 40-minute block, all students should be able to prepare samples, spot plates and start the development. The teacher will probably have to remove the plates from the developing chambers and mark the solvent fronts for slower students. The plates can be viewed under UV light during the next lab period. We had no problem with degradation of samples or plates over a period of several weeks.

2. Whenever you use organic solvents, make sure you have good ventilation. At present, I have a good ventilation system in a new lab. In the past, I would have waited until warm weather allowed for open windows in the lab. Also, since organic solvents are flammable, eliminate ignition sources.

Support for Cornell Center for Materials Research RET Program is provided through NSF Grant DMR-0097494

3. The directions call for use of beakers covered with foil. This works fine although, if you have them, using jars with screw cap lids would reduce evaporative loss of solvent. I found that the beaker and foil set-up was adequate for a 24-hour period without significant loss of developing solvent over the course of the day.

4. TLC plates are not cheap. The standard plates are 20 x 20 cm. By cutting a single plate into eight equal sized sections (5 x 10 cm each), the cost is greatly reduced and a single box of 25 plates will last for several years. In order to cut a plate, I used two thicknesses of cardboard covered by a clean sheet of paper. I placed the plate silica side down on the paper, marked the plastic backing with ink to guide cuts, then used a straightedge and utility knife to make the cut. A little damage to the silica layer occurred along the cut edges, but it didn't interfere with the experiment. Be careful not to touch the silica surface with your fingers. Oils will interfere with solvent movement. You could cut the plates into smaller pieces if you like. The size I chose to use is big enough to allow students to easily test three standards and at least two different OTC-medications. If the plates are too small, fewer tests can be run and there may be a tendency for the spots to run together. Make sure the plates are fluorescent – not all TLC plates are.

5. Use pencil to mark the silica surface during the experiment, not ink. Ink components are soluble in the solvent and will travel up the plate, ruining the results. A dull wooden pencil works best, leaving a visible mark without cutting into the silica layer. Use a light touch.

6. Making small spots seems to be the key to good results. Large spots tend to give smears rather than well-defined spots. Practicing helps students to get a feel for how to let a tiny sample out onto the plate. A single practice plate can be shared by all students. Used plates from other classes also make good practice plates.

7. In order to make the activity time-efficient, I assigned the task of solution preparation to some of my better-organized students while the others prepared developing chambers. One preparation of each medication is more than sufficient for an entire class.

8. I made the standard solutions myself, using aspirin, Tylenol and NoDoz, following the same procedure described in the student directions. Concentration is not a critical issue here.

9. Since the developing solution is simply ethyl acetate, you can use it for more than one experiment. I use the same solvent for all labs meeting on one day but use fresh solvent the next day, as I have read that small amounts of moisture in either the solvent or the plates will affect the results. Ethyl acetate, by the way, is the main ingredient in acetone-free commercial fingernail polish remover.

Support for Cornell Center for Materials Research RET Program is provided through
NSF Grant DMR-0097494

10. When it comes to viewing the results, I handle the UV light myself or closely supervise students who are using it. We use UV-protective goggles and work as quickly as possible. It only takes a few seconds for students to outline the spots with pencil.

11. When students share the same sample solutions and re-use ethyl acetate, very small volumes of solvent waste are generated. I pooled these and allowed them to evaporate in the fume hood. While this is not the ideal disposal technique, the quantity generated in running the activity with about 75 students was less than 200 mL. You may be able to dispose of waste materials through a local college, BOCES, or by incineration.

References

The following internet resources were used in development of this lab. All were active as of August 2002.

http://chemistry.mcmaster.ca/~chem2o6/labmanual/expt2/exp2a.html

http://www.terrificscience.org/lessonexchange/PACTPDF/DrugAnalysis.pdf

http://www.wsu.edu/~pkuzmic/chem240/lab/tlc.html

http://www.vscc.cc.tn.us/academic/math/CHE/122/Labs/TLC.html

http://aa.uncwil.edu/chmL211/F03%20ppts/211-5.ppt

Support for Cornell Center for Materials Research RET Program is provided through NSF Grant DMR-0097494