

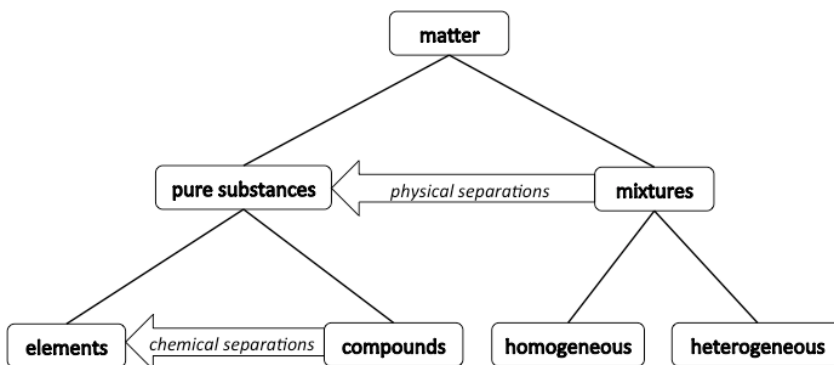
## Experiment #3. Physical Separations – Candy Chromatography

### Goals

1. To physically separate and identify dyes in candy by comparison to commercial food dyes using paper chromatography.
2. To become familiar with common lab equipment and techniques.

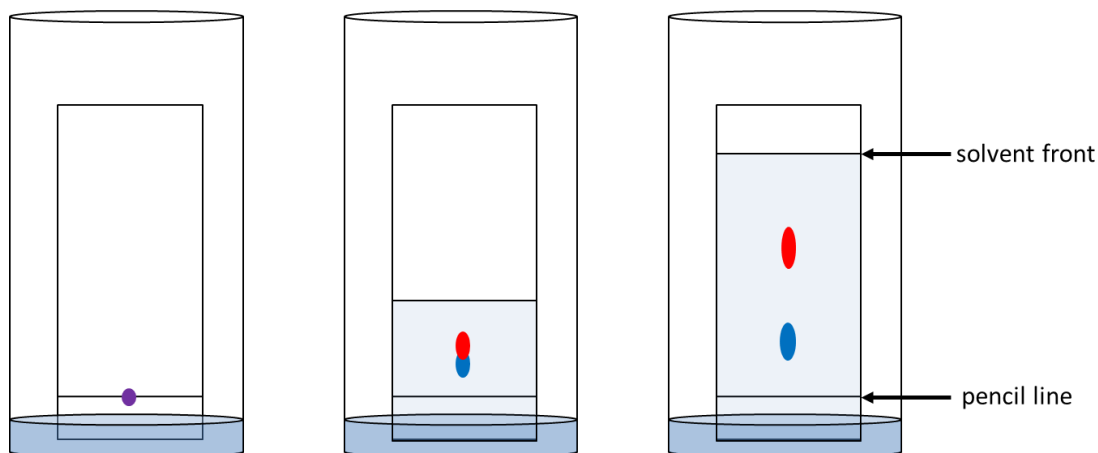
### Introduction

All matter can be classified as either a pure substance or a mixture. Pure substances, which have fixed compositions, can be elements such as gold and oxygen, or compounds such as water ( $\text{H}_2\text{O}$ ) or salt ( $\text{NaCl}$ ). Separating compounds into elements requires chemical reactions. Mixtures consist of two or more pure substances and can have variable compositions. Mixtures can be either homogeneous – uniform throughout down to the molecular level, such as a solution of salt in water, or heterogeneous – having distinct parts such as a slurry of sand and water. Mixtures can be separated into pure substances by physical methods; for instance, a sand and water mixture can be filtered and the water in a saltwater solution can be evaporated.



This lab will use several physical separation techniques, first to separate dyes from candy, then to separate the different dyes from each other. Chromatography is a technique that separates mixtures based on their attraction to other substances. Gas chromatography is widely used by forensic chemists to separate and identify mixtures found at crime scenes. In this lab we will use paper chromatography to separate dyes used in M&Ms, Skittles and food coloring. All of these products contain one or more dyes that can be separated.

To separate the dyes, we will put a small sample of a mixture on chromatography paper. The spot is put on a pencil line drawn to keep track of the starting point. The edge of the paper is placed in a liquid called the solvent. The solvent travels up the paper due to capillary action and as it does, it separates the dyes. The edge of where the solvent traveled is known as the solvent front.



Each dye will move up the paper at a different rate. Two factors are at work in the physical separation of the dyes: the dye's attraction to the paper and its solubility in the solvent. The more a dye is attracted to the paper, the less it will move up – you can think of it as the dye molecules getting stuck to the paper. Solubility also plays a role in how a

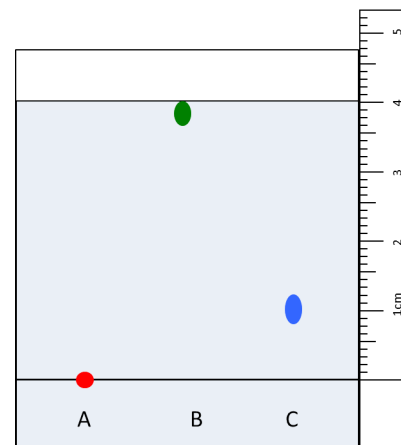
dye will move up the paper. The more soluble a dye is in the solvent, the farther it be carried up the paper. Since attraction and solubility are different for each dye, the dyes move different distances and get separated on the paper.

#### Calculating $R_f$

The retention factor ( $R_f$ ) is used to compare how far different spot travel up the paper. The  $R_f$  is calculated as follows:

$$R_f = \frac{\text{distance from pencil line to center of spot}}{\text{distance from pencil line to solvent front}}$$

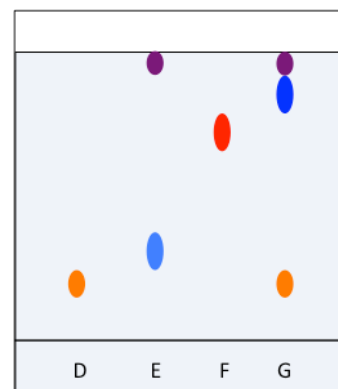
Note that all measurements taken from the spot's starting point – the pencil line. We divide the distance a spot traveled from its starting point, by the maximum distance it could have traveled, from the starting point to the solvent front. All  $R_f$  values will be between 0 and 1. If a spot did not move from the pencil line, it will have an  $R_f$  of zero, such as spot A in figure (2) – this spot is very attracted to the paper. If a spot moves with the solvent front, it will have an  $R_f$  of 1, such as spot B which is not very attracted to the paper. For spot C, the distance from the pencil line to the spot is 1.0 cm, from pencil line to the solvent front is 4.0 cm, making the  $R_f = 0.25$ .



#### Comparing Different Samples

The retention factor ( $R_f$ ) is used to determine if spots from different samples are the same compound. In the figure to the right, four samples (D-G) were developed with paper chromatography. Samples D & F were pure substances – one spot on the chromatography papers indicates that they contained only one dye. Sample E contained two dyes and sample G contained three dyes.

If spots have similar color and similar  $R_f$ , they are probably the same dye. The dye in D is likely the same as dye with the lowest  $R_f$  in G. The spot with the highest  $R_f$  in E and G are also probably the same dye. Currently, there are only 7 dyes approved for use in food by the Food and Drug Administration.



#### Laboratory Activity

<b>Materials</b>	M&Ms (not brown) <u>or</u> Skittles	test tube holder	ruler	watch glass
	1000 mL beakers	chromatography paper	aluminum foil	test tubes
	white household vinegar	10 mL graduated cylinder	stirring rod	
	clear household ammonia	food coloring	red litmus paper	
	10 cm piece of white un-dyed wool yarn	hot plate		

**Safety Hazards** – Caution should be exercised when working with boiling water baths and ammonia. Hot test tubes should be handled with test tube holders.

#### A. Removing the coating from the candy

1. Put five M&Ms or Skittles all of the same color and type (i.e. five green Skittles) into a large test tube. Add about 5 mL of vinegar, or enough to cover the candies completely. The added vinegar should dissolve the colored coating. Avoid dissolving the interior of the candies.
2. Carefully decant (pour the liquid off the solids) the solution into a clean test tube. Avoid transferring the candies or any solid. The solution contains mostly FD&C dyes and sugar.
3. Dispose the candies in the trashcan immediately to prevent later sticking.

### B. Separating the dyes from the dissolved sugar

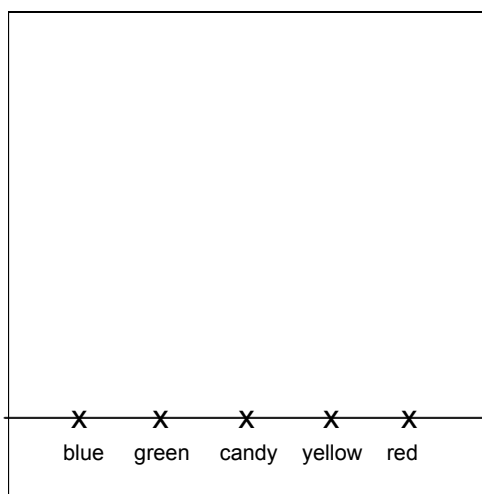
1. Completely submerge a piece of woolen yarn in the test tube containing the dye solution. Heat the test tube in a boiling water bath for 5 minutes; stir occasionally with a glass stirring rod.
2. Remove the now colored yarn from the test tube and rinse the yarn with a little water. The dyes are now chemically attached to the yarn, while the sugar remains in the solution.
3. Dispose of the vinegar solution in the sink.

### C. Re-dissolving and concentrating the dye solution

1. Place the yarn in a clean test tube with about 5 mL of ammonia.
2. Verify that the solution is basic. Dip a glass stirring rod in the solution, then touch the rod to red litmus paper. If the paper turns blue, the solution is basic. If it does not turn blue, add an additional 1 mL of ammonia and retest.
3. Heat the tube with the yarn and ammonia in a boiling water bath for 5 minutes, stirring occasionally with a glass rod. Most of the dye will come off of the yarn and dissolve in the ammonia.
4. Concentrate the dye solution by pouring the solution from on a watch glass which has been placed a hot water bath. The solution on the watch glass should reduce to almost half its original volume. Do not allow the solution to completely evaporate. Add a drop or two of water if the dish has dried out.
5. Dispose of the yarn in regular waste.

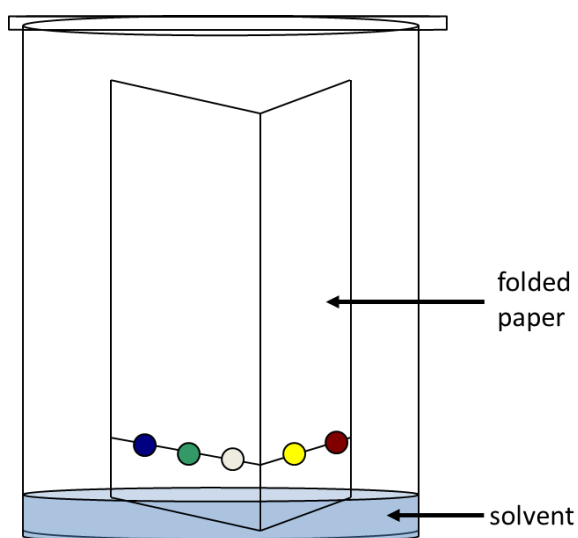
### D. Preparing the chromatography paper

1. Make two identical papers for separation. Obtain two pre-cut 8 x 8 cm pieces of chromatography paper. With a pencil draw a line 1 cm from one of the edges on each paper.
2. Mark 5 x's along the pencil line and label them blue, green, candy, yellow, red, as in the diagram below.
3. Using a capillary tube, spot some the candy solution and each of the food coloring solutions on the appropriate x. Small spots work the best – practice making very small spots on a paper towel before using chromatography paper. You may need to spot the candy dye more than once to get it dark enough.
4. Fold the papers so that it will stand up in your beaker without touching the edges. Test it before you add solvent.



### E. Separating the dyes

1. Obtain two 1000 mL beakers. Place 5 mL of ammonia in one and 5 mL of vinegar in the other (there should be enough solvent in each beaker to cover the bottom of the beaker entirely). Put the prepared chromatography papers in each of the beakers being careful that they do not touch the sides of the beaker. Cover the beakers with aluminum foil or a watch glass.
2. As the solvent rises up the paper, it will separate the dyes. When the solvent front is approximately 1 cm from the top of the paper, remove the paper from the beaker. Mark where the solvent front is with a pencil after you pull it out.
3. Calculate the  $R_f$  for all dyes in the food coloring and in the candy. Some spots will be long streaks – approximate the center of the spot and use that point for the  $R_f$  calculation.
4. If the solvent line keeps rising above the 1 cm mark, use the pencil line as the distance travelled by the solvent front.
5. After you have noted down all the measurements, the chromatography papers can be thrown into the regular waste.



### Waste Disposal

- liquids – sink
- candy, string, litmus paper, chromatography paper – regular waste
- capillary tubes – glass waste box

Name \_\_\_\_\_

Team Name \_\_\_\_\_

## CHM111 Lab – Physical Separations – Grading Rubric

Criteria	Points possible	Points earned
<b>Lab Performance</b>		
Printed lab handout and rubric was brought to lab	3	
Safety and proper waste disposal procedures observed	2	
Followed procedure correctly without depending too much on instructor or lab partner	3	
Work space and glassware was cleaned up	1	
<b>Lab Report</b>		
Sketch of both chromatography papers drawn with colors indicated.	2	
All $R_f$ values correctly calculated	3	
Post Lab: Question 1	1	
Post Lab: Question 2	2	
Post Lab: Question 3	3	
<b>Total</b>	20	

Subject to additional penalties at the discretion of the instructor



Type of candy used:		Color of candy used:	
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1. Sketch your chromatography papers and label the color of each developed spot.
2. In the tables write the color of each spot, the distance the spot traveled and the calculated  $R_f$ . Write a separate entry for each spot on paper. If the source only contained one dye, leave one line blank.

blue green candy yellow red

Vinegar			
Distance from pencil line to solvent front: _____			
Dye Source	Color of Spot	Distance spot traveled	$R_f$
Blue Food Coloring			
Green Food Coloring			
Candy			
Yellow Food Coloring			
Red Food Coloring			

blue green candy yellow red

Ammonia			
Distance from pencil line to solvent front: _____			
Dye Source	Color of Spot	Distance spot traveled	$R_f$
Blue Food Coloring			
Green Food Coloring			
Candy			
Yellow Food Coloring			
Red Food Coloring			

1. If you let the experiment run for less time, removing the chromatography paper from the beaker when the solvent front was 6 cm from the top of the paper, what would be the likely result? Would any problems arise?

2. Based on your observations, classify your spots as mixtures or pure substances. (Did they contain one dye or more than one dye?)

blue food coloring \_\_\_\_\_ red food coloring \_\_\_\_\_

green food coloring \_\_\_\_\_ candy spot \_\_\_\_\_

yellow food coloring \_\_\_\_\_

3. Spots with the same color and similar R<sub>f</sub>s are likely to be the same dye.

Were any dyes used in your candy also used to make one or more food colorings? Yes or No \_\_\_\_\_

Explain in detail how you came to your conclusion. Cite specific colors and R<sub>f</sub>s from your data sheet to support your answer.