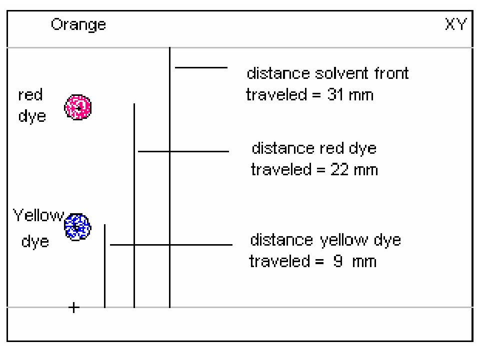
Chemistry 101
SEPARATION TECHNIQUES AND MELTING POINTS
Pre-Lab Exercises

Student:___________________________
Date:______________________________
Instructor:__________________________
Section:____________________________

1. What physical property determines how well paper chromatography can separate two different dyes in food coloring?

2. Why is the developing jar covered while performing paper chromatography?

3. Paper chromatography was performed for orange food coloring with the following resulting chromatogram. Calculate the R_f value for the yellow dye based upon the measurements that are recorded on the chromatogram.



R_f =

4. Based upon the procedure outlined for solvent extraction of salt from sand, how can you tell if a heated sample of sand is completely dry?

5. Solvent extraction was used to extract fat from butter. The original butter sample had a mass of 19.6 g. Following the fat extraction, the remaining sample (not the fat) had a mass of 4.2 g. Calculate the percent fat in the butter.

%

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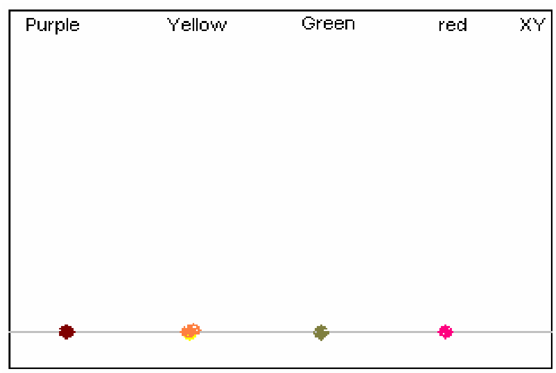
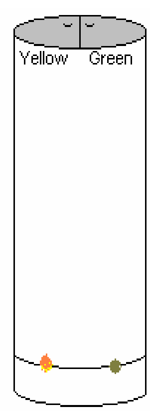
PROCEDURE AND REPORT

Separation of Food Coloring by Paper Chromatography

Food colorings are made of mixtures of dye molecules selected to obtain the desired color. In this section you will use paper chromatography to separate the dyes used in four different food colors. Each dye has unique physical properties. Paper chromatography works based upon the principle that molecules are attracted to other molecules that are similar in terms of the physical property called **polarity**. To perform the paper chromatography for this experiment, food colors will be placed on a piece of paper. The paper will be placed vertically in a mixture of water and *n*-propyl alcohol. This solvent mixture will move up the paper due to capillary action. The dye molecules will be carried up the paper by the rising solvent. The rate that a dye molecule moves up the paper relative to the movement of the solvent depends upon how strongly the molecule is attracted to the paper. Polar molecules that are strongly attracted to the polar cellulose fibers in the paper will move at a different rate than less polar molecules. The net result is that the dyes will move on the paper at different rates, due to differences in polarity, and be separated.



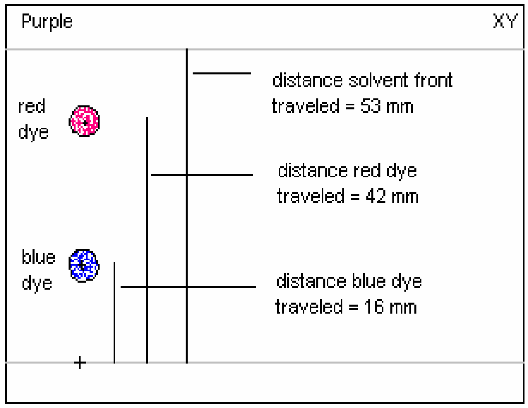
1. Obtain a sheet of chromatographic paper 11 cm x 7.5 cm. Draw a pencil line about 1.8 cm from and parallel to the 11 cm edge of the paper. On the line, place four pencil dots 2.5 cm apart. On the opposite edge of the paper above each dot label in pencil the color of the food coloring to be placed on the dot. Write your initials in the upper right hand corner of the paper.
2. You will separate four different food colorings. Using a glass capillary, transfer the appropriate food coloring to the appropriately labeled dot. Form a food coloring spot on the paper with a diameter no larger than 0.5 cm. Staple the two edges of the paper together forming a cylinder. Do not allow the edges to overlap.
3. Pour 2:1 *n*-propyl alcohol:water mixture into a developing jar to a depth of about 1 cm from the bottom. Check to be sure that the depth of the solution in the jar is only about half the height of the spots from the bottom of the paper.
4. Insert the paper cylinder into the developing jar with the spots at the bottom. Do not let the edge of the paper rest on the edge of the jar. Screw the lid on the jar to prevent the solvents from evaporating. Do not move or agitate the jar while the solvent is climbing up the paper.
5. When the solvent has almost reached the upper edge of the paper, remove the chromatogram from the jar. Immediately draw a pencil line to mark where the solvent front reached.



6. When the chromatogram has dried, outline each dye spot with a pencil and place a dot at the center of each spot.
7. In the table below, record the food colorings you used and the color of the dyes found in each food coloring. An example table has been included.
8. Measure the distance that each dye traveled above the original spot at the bottom of the chromatogram. Record the measured distances in the table below.

9. Measure the distance that the solvent traveled above the line at the bottom of the chromatogram.
10. The distance that a dye travels relative to the distance the solvent front travels is known as the R_f value. The R_f value is useful in determining the identity of a molecule. If different food colorings show spots with similar colors and R_f values, this is an indication that the same dye was used in each food coloring. Calculate the R_f value for each dye on the chromatogram. The R_f value is calculated using the equation:

$$R_f = \frac{\text{distance traveled by dye}}{\text{distance traveled by solvent front}}$$



Example Table

Food Color	Color of Dye	Spot Distance	Solvent Distance	R_f value
Purple	blue	16 mm	53 mm	0.30
	red	42 mm	53 mm	0.79

Food Coloring Paper Chromatography Results

Food Color	Color of Dye	Spot Distance	Solvent Distance	R_f value

Calculations:

Separation of Food Coloring by Paper Chromatography (Continued)

11. Now that you have analyzed your chromatogram and summarized the results in the table on the previous page, determine what different colored dyes are use to make the food colorings you analyzed. Do this by comparing R_f values and color. The same dyes should have approximately the same R_f value and color in your chromatogram. For instance the same yellow dye might be used in both orange and green food coloring, if this is the case, chromatograms of these food colorings should each yield yellow spots with similar R_f values and colors. Record the dye colors you observe and the average R_f value for each dye.

Color of Dye	Average R_f value

12. Attach the completed chromatogram to your lab report.

Distillation of Water

Distillation is a separation technique used to separate two liquids with different boiling points or a liquid from a nonvolatile (nonevaporating) solute such as NaCl or sugar. A distillation apparatus will be set up to separate water from the dissolved minerals (ions) in tap water.

1. Check the conductivity of a beaker of distilled water using a conductivity meter.

   Conductivity Result: ___________________

2. Check the conductivity of a beaker of tap water using a conductivity meter.

   Conductivity Result: ___________________

Compare and explain the conductivity observations for distilled water and tap water:

Separation of Salt from Sand by Solvent Extraction

Solvent extraction can be used to separate components of a mixture when one of the components will dissolve in a given solvent and one will not. In this section you will separate salt from sand by solvent extraction using water as the solvent. Salt obviously will dissolve in water and sand will not, making it possible to dissolve salt in water then remove the salt as a solution.

1. Obtain a vial containing a sand-salt mixture and record the unknown number in the table below.
2. Measure the mass of a clean dry 50 mL beaker. Record the mass to at least 0.01 g.
3. Transfer the vial contents to the beaker. Measure the mass of the beaker and mixture.
4. Calculate the mass of the mixture by subtraction.
5. Add about 10 mL of distilled water to the beaker to dissolve the salt. Swirl to facilitate dissolution. Allow the sand to settle and decant the clear liquid. Wash the sand with at least two additional rinses to make sure all the salt has been removed. Discard the liquid portions.
6. Dry the sand in the beaker in an oven set at 110 ^{o}C for about 12 minutes. After heating, allow the beaker to cool to room temperature then obtain the mass of the beaker and sand. Reheat the beaker and sand for an additional 2 minutes, cool and weigh the sand and beaker again. Repeat this process until the mass of the sand and beaker remains constant.
7. Calculate the mass of the sand.
8. Calculate the mass of the salt.
9. Calculate the percent sand in the original sand-salt mixture.
10. Calculate the percent salt in the original sand-salt mixture.

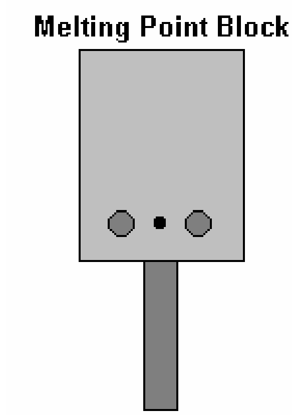
1. Unknown Number	
2. Mass of Beaker	
3. Mass of Beaker and Sand-Salt Mixture	
4. Mass of Sand-Salt Mixture	
6. Mass of Beaker and Sand	
7. Mass of Sand	
8. Mass of Salt	
9. Percent Sand in Mixture	
10. Percent Salt in Mixture	

Calculations:

Identification of Unknown Compounds by Melting Point Analysis

The melting point of an unknown compound is a physical property that can be used to help identify the compound. When completing this portion of the experiment, you will find the melting points for four compounds by looking in reference books. You will then experimentally measure the melting points for two of these compounds. However, in the laboratory the compounds will only be marked with numbers. You will be required to identify the two unknown compounds by comparing the experimental melting points that you measure with the accepted melting points that you obtained from reference material.

1. Using a Merck Index, CRC Handbook or other reference source, find the melting points for the four compounds listed in the table below. Record the melting points in the table.
2. Obtain two vials containing unknown compounds to be used in the melting point analysis. Record the unknown numbers in the table below.
3. Obtain an aluminum melting point block, a hot plate, and a digital thermometer. Turn the thermometer on and set it to measure ^{o}C. Turn the hot plate on to a high setting. **Caution: The hot plate will become hot very quickly!!**
4. You will use the side of the aluminum block with two small indentations and a thermometer hole between these indentations. You will not use the side with several holes. Using a clean spatula, transfer a small amount of the first unknown compound to one of the small indentations on the melting point block. Wipe off the spatula and transfer a small amount of the second unknown compound to the second indentation on the melting point block. Be sure to remember which compound has been placed in each indentation.
5. Place the thermometer in the thermometer hole and place the block on the hot plate.
6. Monitor the compounds and the temperature. Record the melting temperature for each compound in the table below next to the appropriate letter.
7. After the compounds have melted, use tongs to remove the aluminum block from the hot plate. Wipe the melted compounds from the indentations on the block using paper towels. <u>Place several thicknesses of paper towel over your finger as you clean the block so that you are not burned during the cleaning process</u>. It is important that no compound remains on the block after this step. **Do not leave the block on the hot plate longer than necessary!**
8. Identify the unknown compounds by comparing your experimental melting points with the accepted melting points you obtained during step #1. Your melting points will probably not be exactly the same as any of the literature values but they should be close enough to identify the compounds.



Compound	Melting Point (^{o}C)
Acetamide	
Acetanilide	
Benzophenone	
Benzoic Acid	

Unknown Number	Melting Point (^{o}C)	Compound Identity