CH 222 Winter 2025:
"Chromatography" *(in class)* ***Lab - Instructions***

Note: **This is the lab for section 01 and H1 of CH 222 only.**

- *If you are taking section W1 of CH 222*, please use this link:
  http://mhchem.org/r/1b.htm

Step One:

Get a printed copy of this lab! You will need a printed (hard copy) version of pages Ia-1-2 through Ia-1-6 to complete this lab. If you do not turn in a printed copy of the lab, there will be a 2-point deduction.

Step Two:

Bring the printed copy of the lab with you on Monday, January 6 (section 01) *or* **Wednesday, January 8 (section H1.)** During lab in room AC 2507, you will use these sheets (with the valuable instructions!) to gather data, all of which will be recorded in the printed pages below.

Step Three:

Complete the lab work and calculations on your own, then **turn it in** (pages Ia-1-5 through Ia-1-6 *only* to avoid a point penalty) **at the beginning of recitation to the instructor on Monday, January 13 (section 01)** *or* **Wednesday, January 15 (section H1.)** The graded lab will be returned to you the following week during recitation.

If you have any questions regarding this assignment, please email (mike.russell@mhcc.edu) the instructor! Good luck on this assignment!

Chromatography

Most of the substances we use everyday are mixtures of pure substances. Separating, detecting and identifying all of the components in a mixture is crucial for the successful chemist. Several techniques have been developed to do this all of which depend on the differing chemical and physical properties of the components in the mixture. Chromatography is a separation and identification technique that takes advantage of the difference in solubility of a pure substance in various solvents.

In a chromatographic separation, a mixture is deposited on a solid adsorbing substance called the *stationary phase*. The stationary phase can be a strip of filter paper, a thin film of silica gel on an inert surface, a column of silica gel, or a tube of small beads coated with a high molecular weight oil. A solvent is allowed to flow through the stationary phase either under pressure or by gravity or capillary action. As the solvent passes over the mixture, the components in the mixture dissolve in the solvent. A competition takes place between the adsorption of a component on the stationary phase and the dissolution of the component in the solvent (the *mobile phase*). The affinity of each of the components to the stationary phase or mobile phase will be different leading to a separation.

The name given to the various types of chromatography is based upon the type of stationary phase or the physical state of the mixture. Examples include column chromatography, paper chromatography, thin layer chromatography, vapor-phase chromatography, and high pressure liquid chromatography (HPLC).

In this experiment we will use **paper chromatography** to separate a mixture of metallic ions in an aqueous solution. A piece of filter paper is spotted with a drop of solution containing a mixture of ions, and the paper is allowed to dry. The paper is then suspended in a beaker (or jar) containing a solvent which moves up the paper by capillary action. Because each component of a mixture has its own characteristic affinities, each metal ion will travel up the paper at its own characteristic rate. If the paper is large enough, all the components will be separated and will appear as separate spots. If the components are highly colored the spots will be visible. You can convert weakly colored or colorless spots to highly colored ones by spraying them with substances that react to form colored compounds. The filter paper will now contain a vertical row of colored spots arranged according to their characteristic rate of ascent. The word *chromatography*, which is derived from to Greek words and literally means “written with color,” was coined to describe this phenomenon.

The distance traveled by a component ("spot"), $\mathbf{D_x}$, with respect to the distance traveled by the eluting solvent, $\mathbf{D_s}$, is called the **retention factor**, $\mathbf{R_F}$:

$$R_f = \frac{D_x}{D_s}$$

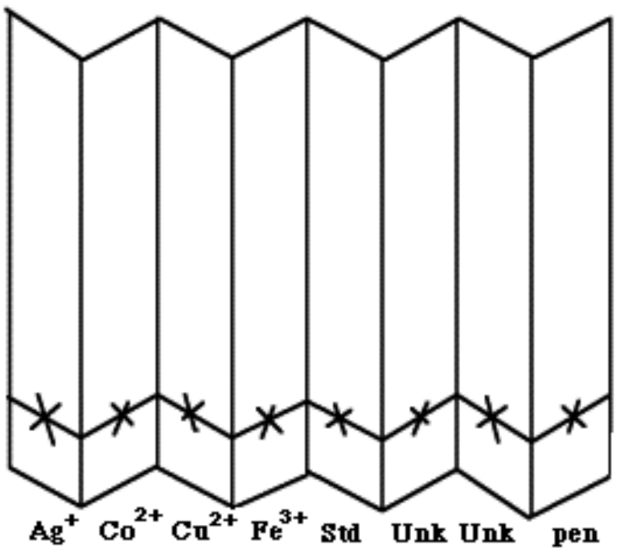
The R_F value is a characteristic property of a given component in a given solvent at a given temperature. Changing solvent or temperature implies a change in R_F. *Note* that the R_F will be a unitless number, and both D_x and D_s must be recorded in the same unit.

In this experiment you will use both the color of the spot and the calculated R_F value to identify the types of metal ions that are present in the solution.

PROCEDURE:

1. Obtain a piece of filter paper about 20 cm long by 12 cm wide. Placing the paper on a clean surface, draw a line using a straight edge and a pencil, 2 cm from one of the long edges.

2. Fold the filter paper so that the line that you have drawn is bisected. In the same manner fold the filter paper in half a second and third time. The line will have been divided into eight equal segments. Refold the paper so that it looks like an accordion, as shown below. Mark the center of each segment of the line with an X using pencil, and label each segment as shown below.



3. Obtain a small capillary tube. Using distilled water, practice spotting a scrap piece of filter paper. The maximum diameter of an acceptable spot is no larger than 0.5 cm.

4. When this procedure is satisfactory, use the appropriate known solutions of silver, cobalt, etc. to spot your chromatographic paper (2 drops each.) The standard (**Std**) should contain a spot from each of the four known solutions (four drops of liquid total). Be sure to allow each spot to dry before spotting it again. Hair dryers will facilitate a complete drying of the spots.

5. Each group will have two unknown (**Unk**) solutions to identify the ions present - be sure to write down their identity (letter) for the lab report. The unknown solutions are less concentrated than the metal ion solutions so you need to spot these two spots three times each.

6. For the last spot (**pen**), place a small dot from a felt pen on the X.

7. Add about 25 mL of the eluting solution (the mobile phase) to a 600 mL beaker and cover with plastic film (alternatively, a jar with a plastic lid can be used.) The eluting solution was made by mixing a solution of HCl with ethanol and butanol, two organic solvents known as alcohols.

8. When all spots are dry, carefully place the paper in the 600 mL beaker (or jar.) It is important that the solvent is below the 2 cm line on the paper. Cover the mouth of the beaker with plastic film (or put a lid on the jar) and allow the solvent to move up the paper.

9. **While the experiment is proceeding**, you can **test the effect of the staining reagent** on the metal ion solutions. Spot a piece of filter paper with each of the four known metal ion solutions and dry the paper as before. Some of the spots will be colored at this point; note the color of the unstained knowns in your lab. In a fume hood, place the filter paper on a paper towel and spray the paper evenly with the staining solution, getting the paper moist but not really wet. The staining reagent is a mixture of solutions of potassium ferrocyanide and potassium iodide and forms colored compounds with the metal ions. Note the **color** of each known metal ion spot in your lab.

10. When the eluting solution has risen to about 4 cm from the top of the chromatographic paper, remove the paper and immediately mark the paper with a line that is the **solvent front**. Dry the paper. Circle any cations that are visible by virtue of their colors. Place the paper on a paper towel and spray it with the staining reagent. Note the colors present. Dry the paper and **circle** the boundary of each spot since they may fade with time.

11. Measure the distance (to the tenth of a millimeter) from the straight line where you applied the spots to the solvent front. This distance is D_s, the distance traveled by the solvent. Measure the distance from the beginning line to the center of each spot. This distance is D_x for each metal cation. Calculate R_f for each of the metal cations. Be sure to include one sample calculation set up in your lab.

12. Report the R_f value for each metal cation as well as its color, the identity of the cations in your unknown solutions and any possible sources for error in this experiment.

13. Dispose of any remaining eluting solution in a waste bottle. Wash your hands before leaving the lab!

Chromatography

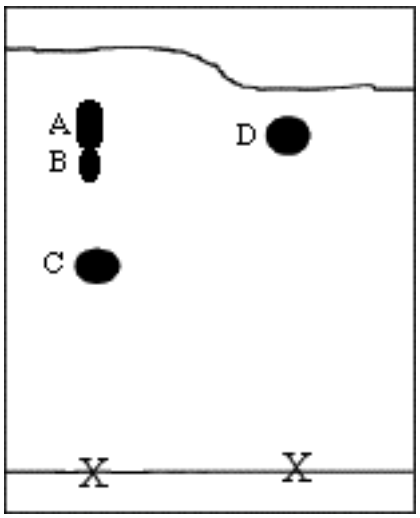
*include first **and** last names* **Name:**

Lab Partner(s):

	Ag^{+}	Co^{2+}	Cu^{2+}	Fe^{3+}
Known Solutions				
Colors (Dry)	_____	_____	_____	_____
Colors (After staining)	_____	_____	_____	_____
Distance solvent moved	_____	_____	_____	_____
Distance cation moved	_____	_____	_____	_____
R_f	_____	_____	_____	_____
Known Mixture (Std or Mix)				
Distance solvent moved	_____	_____	_____	_____
Distance cation moved	_____	_____	_____	_____
R_f	_____	_____	_____	_____
Unknown Mixture No. _____				
Color (Dry)	_____	_____	_____	_____
Color (After staining)	_____	_____	_____	_____
Distance solvent traveled	_____	_____	_____	_____
Distance cation traveled	_____	_____	_____	_____
R_f	_____	_____	_____	_____
Unknown Mixture No. _____				
Color (Dry)	_____	_____	_____	_____
Color (After staining)	_____	_____	_____	_____
Distance solvent traveled	_____	_____	_____	_____
Distance cation traveled	_____	_____	_____	_____
R_F	_____	_____	_____	_____

Chromatography Postlab Questions

1. Determine the R_f value for each spot on the following chromatogram.



$R_f(A)$ =

$R_f(B)$ =

$R_f(C)$ =

$R_f(D)$ =

2. Based on your calculations, is Compound D more likely to be identical to Compound A, Compound B, or Compound C? Explain.

3. Why are pencils used to mark the chromatographic paper rather than ink?

4. If a 1.0 cm^2 spot of silver solution contains 10. microliters of solution and the solution contains 11 g of Ag^+ ions per liter of solution, what mass of silver ions are in one 1.0 cm^2 spot?

Please note: The instructor will send you email throughout the term, so *please check your email several times each week!* The instructor will use your @saints.mhcc.edu address by default, but if you wish to use an alternate email address, send an email to mike.russell@mhcc.edu from your alternate email account and it will be changed promptly.