Experiment 6

Paper Chromatography

Chemicals and hazard statements

1. 0.5 M Manganese (II) chloride ($MnCl_2$).
   Harmful if swallowed. Harmful to aquatic life with long lasting effects. Wash skin thoroughly after handling. Do not eat, drink or smoke when using. Avoid release to the environment.
2. 0.5 M Iron (III) nitrate ($Fe(NO_3)_3$).
   May intensify fire, oxidizer. Causes skin irritation. Causes serious eye irritation. May cause respiratory irritation.
3. 0.5 M Cobalt (II) nitrate ($Co(NO_3)_2$).
   May intensify fire, oxidizer. Harmful if swallowed. May cause an allergic skin reaction. May cause allergy or asthma symptoms or breathing difficulties if inhaled. Suspected of causing genetic defects. May cause cancer. May damage fertility or the unborn child.
4. Nickel (II) nitrate ($Ni(NO_3)_2$).
   May intensify fire, oxidizer. Harmful if swallowed or inhaled. Causes skin irritation. May cause an allergic skin reaction. Causes serious eye damage. May cause allergy or asthma symptoms or breathing difficulties if inhaled. May damage fertility or the unborn child. Causes damage to organs through prolonged or repeated exposure if inhaled. Very toxic to aquatic life with long lasting effects.
5. Copper (II) nitrate ($Cu(NO_3)_3$).
   May intensify fire; oxidizer. Harmful if swallowed. Causes skin irritation. Causes serious eye damage. Very toxic to aquatic life with long lasting effects.
6. Acetone
   Highly flammable liquid and vapor. Causes serious eye irritation. May cause drowsiness or dizziness.
7. 12 M (~38%) Hydrochloric acid (HCl).
   May be corrosive to metals. Causes severe skin burns and eye damage. May cause respiratory irritation.
8. Concentrated ammonia (28% to 30% ammonium hydroxide, (NH_4OH)).
   Harmful if swallowed. Causes severe skin burns and eye damage. Very toxic to aquatic life with long lasting effects.
9. 0.25 M Sodium hydroxide (NaOH).
   May be harmful if inhaled. May cause respiratory tract irritation. May be harmful if absorbed through skin. May cause skin irritation. May cause eye irritation.

10. 1% Dimethylglyoxime in ethanol.
    Flammable liquid. May be fatal or cause blindness if swallowed. Causes severe eye irritation. May cause respiratory and digestive tract irritation. May cause skin irritation. May cause central nervous system depression. May cause kidney and liver damage. May cause reproductive and fetal defects.

Materials

1. 20 cm x 10 cm piece of chromatography paper
2. Ruler
3. Pencil
4. 600 mL beaker
5. Watch glass to cover 600 mL beaker
6. Heating lamp
7. Ammonia chamber
8. Cotton swabs
9. Capillary tubes

Procedure

1. Try to minimize handling of the chromatography paper and only touch the outside edges.
2. Use a 20 cm x 10 cm piece of chromatography paper to make the experiment's chromatogram.
3. Draw a spotting line 2 cm from the edge of the 20 cm side of paper (Figure 1).

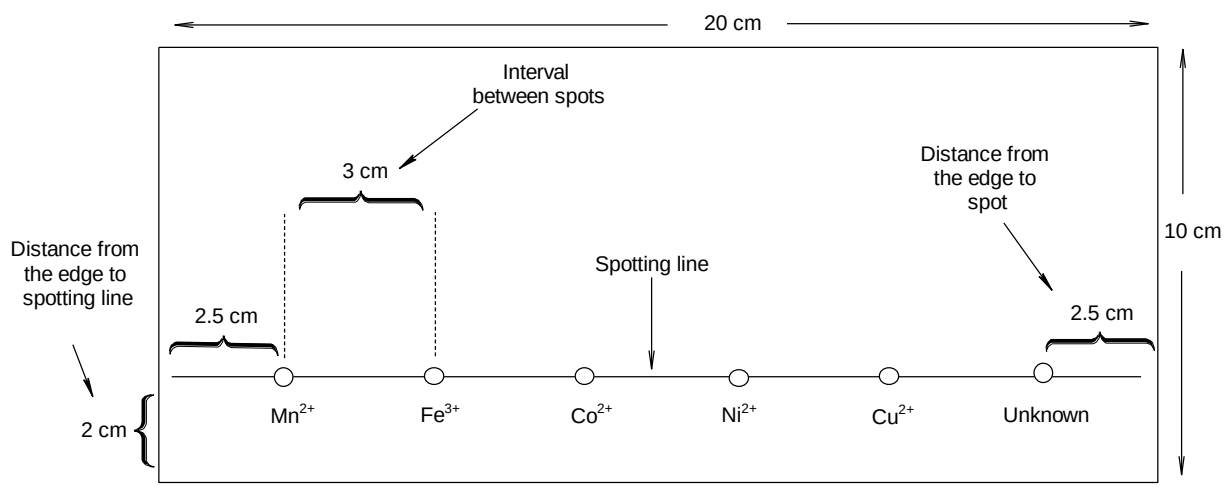

Figure 1. Parts of the chromatogram.

4. Make a mark along the spotting line 2.5 cm from one of the edges. Then make 5 more similar marks at 3 cm intervals along the line. The last spot should be about 2.5 cm from the edge (Figure 1).
5. Spot each of first 5 marks with the standard cations using a capillary tube.in the following order: Mn^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}. Spot the sixth mark with your unknown. You

may use one capillary to make two spots provided that you use each side of the capillary for only one of the samples. Make the spots by dipping the end of the capillary tube into the given solution and then touch the end on the corresponding spot on the paper. It may be helpful if you practice on a spare piece of paper before you do the final spotting. You want to keep the spots as small and as concentrated as possible. This is best achieved by making several small applications to the same spot and allowing each application to dry prior to making the next application.

6. After spotting, allow the spots to dry and take note of the color of each of the given spots. Write down what you see, even if you see no color.
7. Take the chromatogram and form a cylinder with it and keep the shape by stapling the two edges of the chromatogram at the ends and center. (Figure 2). Make sure the edges touch but do not overlap. Overlapping the edges makes the spots move unevenly.

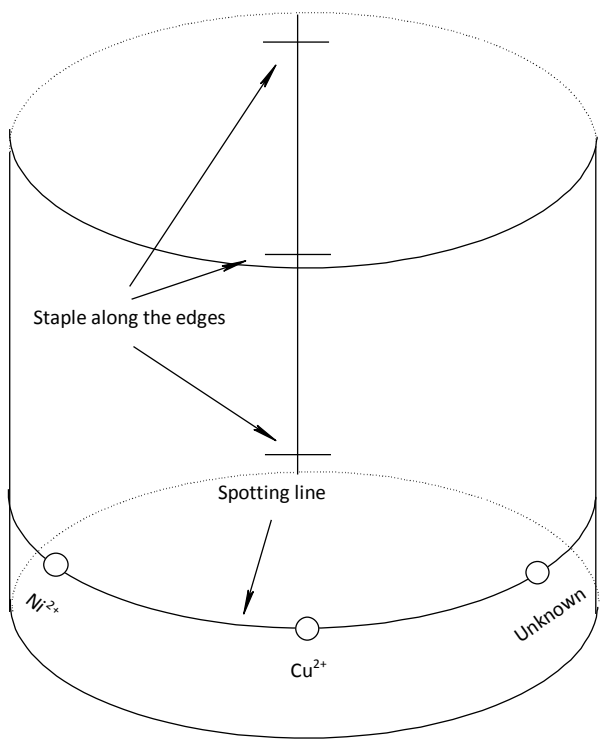

Figure 2. Chromtogram cylinder.

8. Inside a ventilation hood, prepare an eluent solution by mixing 34 mL of acetone with 7 mL of hydrochloric acid and 4 mL of water in a 600 mL beaker. Mix the solution by swirling the liquids inside the beaker. MAKE SURE YOU KEEP THIS MIXTURE INSIDE A VENTILATION HOOD. THE FUMES PRODUCED BY THE ELUENT ARE IRRITATING!!!
9. Place your cylindrical chromatogram with the spots positioned toward the bottom inside the 600 mL beaker containing the eluent you made, and let it rest there. Cover

the beaker with a watch glass to avoid the eluent from evaporating. DO NOT ALLOW THE CYLINDRICAL CHROMATOGRAM TO TOUCH THE EDGES OF THE BEAKER AND DO NOT MOVE YOUR SETUP ONCE YOU HAVE LET THE CHROMATOGRAM REST.

10. Allow the chromatogram to soak the eluent until it reaches about 1 cm from the top edge of the cylinder. This position is known as the solvent front (Figure 3). This may take about 15 to 20 minutes.

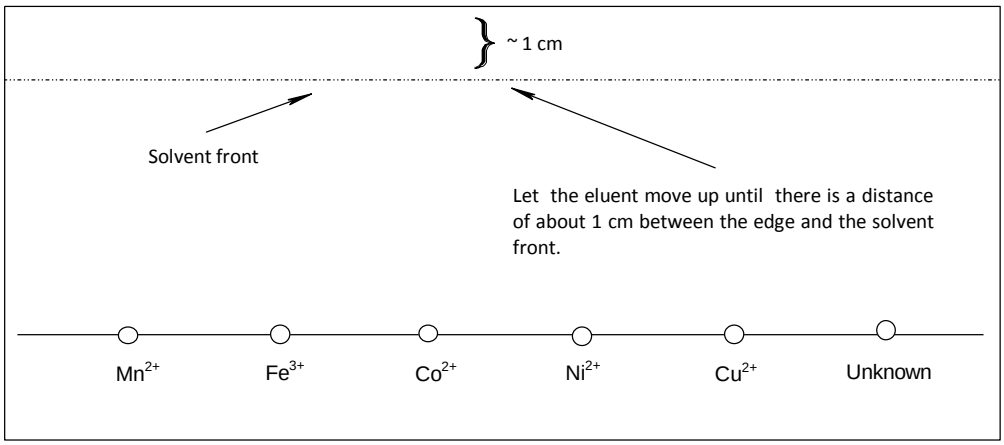

Figure 3. Chromatogram with solvent front.

11. Remove the chromatogram from the beaker and use a pencil to trace the solvent front.
12. Dry the chromatogram under a heat lamp. The lamp should be at about 30 cm above the chromatogram. Record your observations regarding the colors generated by each of your spots on the chromatogram.
13. Carefully remove the staples from your cylindrical chromatogram and lay it out flat. Observe any color changes that have occurred on any of your spots and record your observations.
14. Take your chromatogram and place it inside the ammonia chamber located in the ventilation hood. Do not let it touch the solution and keep it in the closed chamber for 2 to 3 minutes. Remove the chromatogram from the chamber and look for any color changes on it. You should be able to observe color changes for the sections where you spotted the paper with iron (III) (Fe^{3+}), cobalt (II) (Co^{2+}), and copper (II) (Cu^{2+}). Compare these sections with your unknown section and write down what you observe. You will need to perform some additional tests to be able to observe manganese (II) and nickel (II).
15. **Test for Manganese (II) (Mn^{2+}).**
    Wet a cotton swap with 0.25 M sodium hydroxide to streak the section were you spotted the paper with the manganese (II) standard. Streak the section up and down perpendicular to the spotting line. You should be able to see a dark brown spot appear. Repeat this procedure on your unknown by passing the cotton swab at the same

distance from the spotting line that the dark spot appeared on your Mn^{2+} standard. Record your observations. DO NOT STREAK ANY OTHER SECTIONS.

16. **Test for Nickel (II) (Ni^{2+}).**
    Wet a cotton swab with 1% dimethylglyoxime and repeat the procedure that you performed for manganese (II) on the section that contains your nickel (II) standard and your unknown. Remember to only streak on your unknown the section that is at the same height from the spotting line as in the section with your nickel (II) standard. The appearance of bright red indicates the presence of the nickel (II) cation. Write down your observations.
17. Place your chromatogram in the ammonia chamber for another minute in order to double check the colors that you observed previously. Compare your observations with the data that you wrote down.
18. Calculate the retention factor values (R_f) for your standards and then for your unknown. Compare the R_f values of your standards with the R_f value(s) of your unknown (Figure 4).

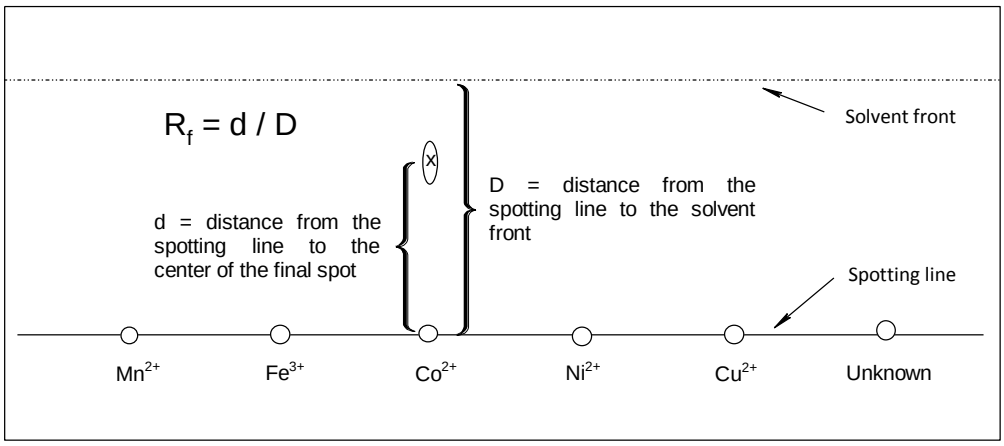

Figure 4. Parts of your chromatogram needed to calculate R_f

19. Determine and record the identity of your unknown by comparing the colors observed and the retention factors calculated. Record these final observations in your notebook. You may use the given report sheet as a guideline to the information that should be recorded in your notebook.