EXPERIMENT 3

Separation of Cations by Paper Chromatography

Performance Goals

3–1 Separate a mixture of cations by paper chromatography and calculate their R_F values.

3–2 Analyze an unknown mixture of cations by paper chromatography.

CHEMICAL OVERVIEW

Chromatography, which means ''the graphing of colors,'' gets its name from the early experiments of Tswett, who, in 1906, succeeded in separating a mixture of colored pigments obtained from leaves. A solvent mixture, carrying the pigments, was allowed to pass through a glass column packed with chalk. At the end of the experiment, the pigments were separated in colored bands at various distances from the starting level. This method is now known as column chromatography.

Chromatography may now be applied to colorless compounds and to ions. Paper chromatography is a more recent and much faster separation technique than column chromatography. It may be used for the separation of substances by a solvent moving on sheets or strips of filter paper. The filter paper is referred to as the **stationary phase,** or **adsorbent.** The mixture of solvents used to carry the substances along the paper is called the **mobile phase,** or **solvent system.**

In practice, a sample of the solution containing the substances to be separated is dried on the paper. The end of the paper is dipped into the solvent system so that the sample to be analyzed is slightly above the liquid surface. As the solvent begins to soak the paper, rising by capillary action, it transports the sample mixture upward. Each component of the mixture being separated is held back by the stationary phase to a different extent. Also, each component has a different solubility in the mobile phase and therefore moves forward at a different speed. A combination of these effects causes each component of the mixture to progress at a different rate, resulting in separation.

In a given solvent system, using the same adsorbent at a fixed temperature, each substance can be characterized by a constant **retention factor,** R_F. By definition,

$$R_F = \frac{\text{Distance from origin to center of spot}}{\text{Distance from origin to solvent front}} \tag{3.1}$$

where the **origin** is the point at which the sample was originally placed on the paper and the **solvent front** is the line representing the most advanced penetration of the paper by the solvent system. The R_F value is a characteristic property of a species, just as the melting point is a characteristic property of a compound.

In this experiment you will separate a mixture of iron(III), copper(II), and cobalt(II) ions, Fe^{3+}, Cu^{2+}, and Co^{2+}, respectively. Each ion forms a different colored complex when sprayed with a solution containing potassium hexacyano ferrate(II), $K_4[Fe(CN)_6]$.

SAFETY PRECAUTIONS AND DISPOSAL METHODS

Acetone is *extremely flammable.* Its vapors can ignite even when the liquid is a considerable distance from an open flame, so be sure no such flame is operating in the vicinity of your work area. Fumes of acetone and concentrated hydrochloric acid are objectionable and, to some degree, harmful. These chemicals should be used in the hood. Be sure to wear safety glasses.

After you have finished the experiment, dispose of the solvent mixture in a stoppered bottle.

PROCEDURE

1. Using a graduated cylinder and working in the hood, prepare the following solvent system: 19 mL acetone; 4 mL concentrated hydrochloric acid, HCl; 2 mL water or use 25 mL of a pre-prepared solvent mixture, if available. Pour the solvent mixture into an 800- or 1000-mL beaker and cover it tightly with a plastic film (e.g., Saran wrap). This allows the atmosphere within the beaker to become saturated with solvent vapor and helps to give a better chromatographic separation.
2. Obtain a piece of chromatography paper 24 to 25 cm long by 11 to 14 cm wide. Draw a pencil line about 1 cm from the long edge of the paper. (You must use an ordinary pencil for this line. Ink or colored pencil often contains substances that may be soluble in the solvent, producing chromatograms of their own.) This line will indicate the origin (see Figure 3.1). Also draw a line about 1 cm long, 6 cm above the center of the penciled line.
3. Using a different capillary tube for each solution (do not mix them!) transfer a drop of each solution listed below to the penciled line, as shown in Figure 3.1. Apply the spots evenly over the line, leaving a margin of about 3 cm from each short edge of the paper. Use a separate, clean capillary tube for each solution; or, if the solutions are to be

obtained from beakers in which a capillary tube is provided, *be sure to return the tube to its proper beaker.* With a pencil, identify each spot by writing on the paper directly beneath the spot. The solutions are:

A. Fe^{3+} solution

B. Cu^{2+} solution

C. Co^{2+} solution

D. Solution containing all three ions, Fe^{3+}, Cu^{2+}, and Co^{2+}

E. Any of the unknowns furnished (be sure to record its number)

F. Another unknown (again, record the number).

4. Dry the paper under a heat lamp or air blower.
5. Form the paper into a cylinder without overlapping the edges. Fasten the paper with staples, as shown in Figure 3.2.
6. Place the beaker in a position on your desk where it will remain undisturbed throughout this step. Taking care that the origin line remains above the solvent, carefully place the cylinder into the beaker,

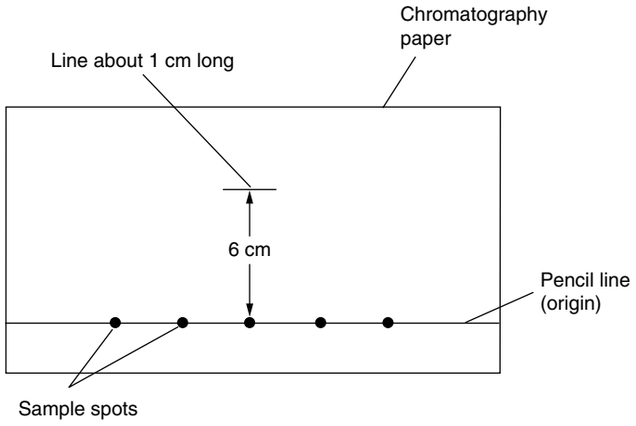


Figure 3.1
Preparing chromatography paper

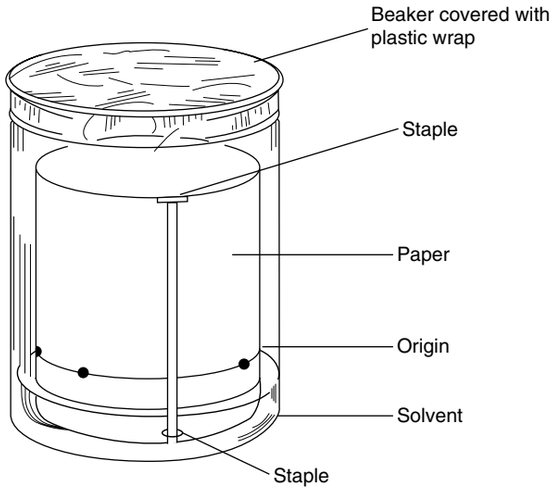


Figure 3.2
Development of chromatogram

as shown in Figure 3.2. Replace the plastic film and wait as the solvent moves up the paper. Do not move the beaker or the solvent front will be uneven.

7. *NOTE: In this and all remaining steps, when the paper is wet, be sure not to lay it down on any surface that is not clean.* When the solvent has risen above the short line drawn 6 cm above the origin in Step 2, remove the cylinder from the beaker and quickly mark the solvent front position with a pencil. Remove the staples and dry the paper under a heat lamp.
8. Spray the paper with a solution of potassium hexacyano ferrate(II), $K_4[Fe(CN)_6]$.*

   The presence of Fe^{3+} is shown by the spot turning a dark steel blue color. Cu^{2+} turns a rust brown, and Co^{2+} turns grayish purple (rose).

RESULTS AND CALCULATIONS

Observe and record on the work page the colors of spots produced by the three ions in each of the chromatograms of solutions A through D.

Measure and record in millimeters the distance between the origin and the solvent front (X in Figure 3.3). Next, measure and record the distance between the origin and the center of each spot in the chromatograms for solutions A through D. Calculate the R_F value for each ion, using Equation 3.1. Record that value as a decimal fraction to two decimals (i.e., 0.82, 0.75).

From the spots above your solutions E and F, indicate by a check mark in the table the ions present in the unknowns. Be sure to list the identification numbers of the unknowns.

If the center of a spot is right on the solvent front, the R_F value equals 1.00.

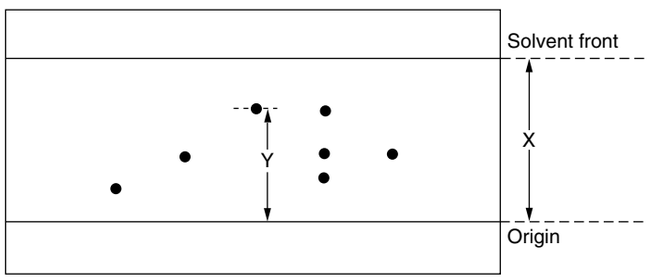


Figure 3.3
Developed chromatogram

*Also called potassium ferrocyanide.

Name *Date* *Section*

Experiment 3

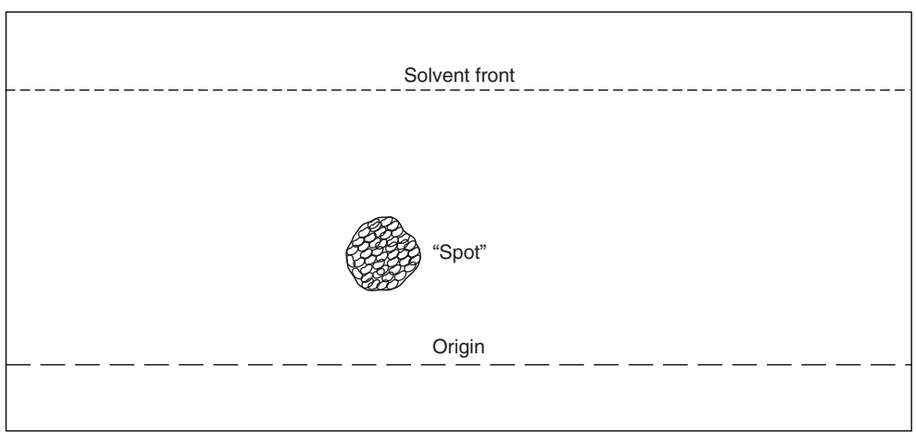
Advance Study Assignment

1. What would you observe if you used a ballpoint pen, instead of a pencil, to mark the chromatography paper?

2. Why do you have to cover the beaker while the solvent is moving up the paper?

3. What problem would be caused by moving the beaker during the development of the chromatogram?

4. Make the necessary measurements (in millimeters) and calculate R_F for the following chromatogram:



Name *Date* *Section*

Experiment 3

Work Page

Distance between origin and solvent front: _____ mm

Solutions of Known Ions

Solution	*Ion*	*Color (After spraying)*	*Distance from Origin (mm)*	R_F
A	Fe^{3+}			
B	Cu^{2+}			
C	Co^{2+}			
D	Fe^{3+}			
	Cu^{2+}			
	Co^{2+}			

Solutions of Unknown Ions

Put a check mark in the three right-hand columns to indicate the presence of each respective ion; leave blank if no ion is present. Be sure to enter the identification number of your unknown.

Solution	*Unknown No.*	*Fe^{3+}*	*Cu^{2+}*	*Co^{2+}*
E				
F				

Name *Date* *Section*

Experiment 3

Report Sheet

Distance between origin and solvent front: _____ mm

Solutions of Known Ions

Solution	*Ion*	*Color (After spraying)*	*Distance from Origin (mm)*	R_F
A	Fe^{3+}			
B	Cu^{2+}			
C	Co^{2+}			
D	Fe^{3+}			
	Cu^{2+}			
	Co^{2+}			

Solutions of Unknown Ions

Put a check mark in the three right-hand columns to indicate the presence of each respective ion; leave blank if no ion is present. Be sure to enter the identification number of your unknown.

Solution	*Unknown No.*	*Fe^{3+}*	*Cu^{2+}*	*Co^{2+}*
E				
F				