

EXPERIMENT 12

PAPER CHROMATOGRAPHY OF SOME METAL CATIONS

PURPOSE:

To identify unknown cations by paper chromatography.

INTRODUCTION:

Chemical analyses may be accomplished in a variety of ways. Some employ wet chemistry techniques such as those used in qualitative analysis and some resort to the use of instruments like spectrophotometers. In this experiment use is made of chromatographic principles.

Chromatography can be defined as a technique for separating substances on the basis of differences in affinity for a stationary and a mobile phase. A substance may be soluble in a particular solvent, which implies an affinity for that solvent. The same substance in solution, when spotted onto a piece of paper, may adsorb onto the paper due to an attraction for the materials making up the paper fibers. The substance has an affinity for both solvent and paper. Many substances are like this in that they can be dissolved in solution and adsorbed onto paper. However, the affinities of different substances for the same solvent and the same paper vary, some with high affinities, some with low. Advantage can be taken of these differences to separate substances in a mixture and therefore identify them as individuals.

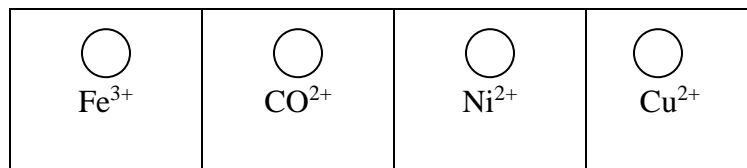
To illustrate the idea, consider a mixture of \underline{x} and \underline{y} in a solvent. A drop of that solution is placed on a strip of paper and allowed to dry in place. One end of the strip of paper is then placed in a solvent for which the \underline{x} and \underline{y} have affinities. The solvent is allowed to move through the paper from one end of the strip to the other by capillary action. When the traveling solvent comes in contact with \underline{x} and \underline{y} it will pick them up, depending on their affinities for that solvent, and carry them along through the paper. They will, however, be held back from traveling by their affinities for the stationary paper fibers. They will likely travel different distances because of their differing affinities and will wind up separated from each other on the paper. After separation, tests can be applied to identify them on the paper. The process just described is paper chromatography. Other types of chromatography make use of other stationary phases. Some types alter the mobile phase by using gases but in all types the principles are primarily the same. The experiment under consideration here will make use of paper chromatography to separate and identify some selected metal ion, Fe^{3+} , Co^{2+} , Ni^{2+} , and Cu^{2+} .

PROCEDURES:

1. **Ion identification:** As a first step, some method of identifying the ions as they exist on the paper must be established. This is so because the ions become difficult to observe and if they are moved on the paper by the mobile phase they can be totally lost. A developer can be used to make the ions more apparent. We will use

dimethylglyoxime (DMG) dissolved in alcohol as developer. The DMG reacts with metal ions to produce colored compounds, allowing the ions to be identified.

Cut a piece of chromatography paper with the approximate dimensions of 10 cm by 5 cm. Place a row of spots using aqueous solutions of the various metal ions on the paper as shown in Figure 3.1. Make the spots about one cm in diameter and label each with pencil.



Dry the spots on the paper by passing the paper back and forth slowly over a burner flame or use a hair dryer that you have brought from home. If you use the burner flame, diffuse the flame with wire gauze and keep the paper high enough over the appearance of the dried spots. Spray the paper lightly with DMG solution in the hood. Do not soak the paper, just spray enough to make it damp with the solution. Again dry the paper, then describe the appearance of the spots. Place the paper in a 600 mL beaker that contains a 10 mL beaker $\frac{3}{4}$ filled with concentrated ammonium hydroxide (NH₃(aq)). Avoid filling the lab with strong smelling NH₃ fumes by keeping the large beaker covered with clear plastic wrap. Leave the paper in the beaker of ammonia fumes for about five minutes, then remove and again record the appearance of the spots.

2. **Elution of Ions:** Elution is a term used to describe the process of moving a substance over a stationary phase with a mobile phase for the purpose of separation. Prepare a second piece of chromatography paper with dimensions of 10 cm by 15 cm. With a **pencil** (not pen) lightly sketch in five 3-cm wide columns. Two cm from the bottom of the paper sketch in a perpendicular line across the paper. Fold the paper along the column lines like an accordion.

Spot the paper on the perpendicular line in the middle of each column space with the four ions, one ion to a column, and one spot of a prepared mixture of all 4 ions. Again make the spots about 1 cm in diameter and label the columns at the top. Dry the paper then stand it in a 400 mL beaker of mobile phase solution made by mixing 10 mL of 7.2 M HCl and 30 mL of 2-butanone. Cover the beaker with clear plastic wrap to retain the vapor. Allow the paper to stand in the eluting solution until the solvent has moved to within 1 cm of the top of the paper. This will take many minutes. Keep a close eye on the paper. If the solution reaches the upper edge of the paper your results can be erratic. Do not allow the paper to touch the walls of the elution beaker and so not tip, shake, or lift up the beaker.

When the solvent gets to the 1 cm position, remove the paper and dry it. After the paper is dry, the spots may be developed by the same procedure of spraying with

DMG, drying, and exposing to ammonia fumes as used in Procedure 1. As before, record the appearance of the spots at each step in the development process.

It is possible to determine the R_f value for each ion from the developed spots. This value, which can be used to identify ions, is a ratio of how far ions have traveled up the paper compared to how far the solvent has traveled. The highest position the solvent has traveled is indicated by a light brown line on the developed paper. To determine an R_f value, measure how many cm the ion spot has traveled. Measure from the center of the spot to the line where the spot was originally placed. Next measure the distance traveled by the solvent, i.e. from the light brown line to original spot line.

$$\text{The } R_f \text{ value} = \frac{\text{distance traveled by ion}}{\text{distance traveled by solvent}}$$

Record the R_f values of each ion.

3. **Determination of an Unknown:** Prepare a piece of chromatography paper with the dimensions 10 cm by 6 cm. Divide the paper into two columns with a perpendicular line 2 cm from the bottom edge.

Your instructor will spot an unknown on the perpendicular line in one column. In the second column you will spot with a solution that contains all four ions. This column will provide direct comparison to the unknown column with exactly the same conditions as the unknown and will aid in identifying the ions in the unknown. Dry the spots, fold the paper in half, and place it in the same eluting solution used before. Allow the solution to travel to near the top. Develop the spots as in Procedure 2 and calculate R_f values for each ion present. There may be more than one ion in the unknown. Record the finding on the Report Sheet.

NAME: _____ SECTION _____

EMPERIMENT 12–PAPER CHROMATOGRAPHY REPORT SHEET

Table I – Effect of the DMG Developer on Metal Ions

Ion	Initial Appearance (Dry)	Developed Neutral	Appearance (Dry) Basic
Co ²⁺	_____	_____	_____
Cu ²⁺	_____	_____	_____
Fe ³⁺	_____	_____	_____
Ni ²⁺	_____	_____	_____

Table II – Effect of Eluting Solvent on Ions

Ion	R _f Value
Co ²⁺	_____
Cu ²⁺	_____
Fe ³⁺	_____
Ni ²⁺	_____

Table III – Analysis of Unknown Solution

Unknown Number _____

Ion	Present (Yes or No)	R _f Value
Co ²⁺	_____	_____
Cu ²⁺	_____	_____
Fe ³⁺	_____	_____
Ni ²⁺	_____	_____

Attach both “known” and “unknown” chromatograms to a separate page and label completely.