Separation of Metal Cations by Paper Chromatography

Name:

Partners:

PURPOSE
The purpose of this experiment is to identify the $R_f$ values of several metal cations and use that information to determine the identities of metal cations in an unknown mixture.

BACKGROUND
Chromatography, or color graphing, has its origins in the separation of plant pigments. Today, chromatography can also be applied to colorless compounds and ions. The chromatography process is comprised of a stationary phase, a mobile phase, and chemicals to be separated (analytes). The analytes are generally placed on the stationary phase, with which they have some attractive interactions (adsorption to the surface). A mobile phase is then introduced, for which they also have an attraction. As the mobile phase moves across the stationary phase, it will carry the analytes along with it. Those with greater interactions (affinity) for the mobile phase will move farther along the stationary phase in a given time than those with greater affinity for the stationary phase itself. For a system in stasis (same mobile/stationary phases and constant temperature), a characteristic of the analytes that can be calculated is the retention factor ($R_f$). The retention factor is calculated by measuring the distance the spot of a substance to be separated travels and dividing that value by the distance the spot travels in the same amount of time. In practice, the starting location of the spot is called the origin and the final position of the solvent is known as the solvent front. With this in mind, a formula for $R_f$ can be written as shown in equation 1 below.

Equation 1: $R_f = \frac{\text{Distance from origin to center of spot}}{\text{Distance from origin to solvent front}} = \frac{D_{\text{spot}}}{D_{\text{solvent}}}$

In this experiment, the stationary phase will be paper, the mobile phase will be a special acetone/HCl solution, and the analytes will be the following metal cations: Fe$^{3+}$, Cu$^{2+}$, and Co$^{2+}$. Each of these ions will form a different color when treated with a solution of ammonium hexacyano ferrate (II), ($\text{NH}_4)_4[\text{Fe(CN)}_6]$.

PROCEDURE
16mL of the previously prepared solvent mixture was measured out and placed in a large beaker (to be used as the mobile phase). Before leaving the fume hood, the beaker was tightly covered with plastic wrap. This allowed the atmosphere within the beaker to become saturated with solvent vapor and resulted in a more reliable chromatographic separation. A piece of chromatography paper that measured 24-25 cm in length and 12-14 cm in width was obtained. A pencil mark was drawn about 1.5 cm from the long edge of the paper to represent the origin. Also, a line about 1 cm long was drawn 2 cm from the top. A drop of each solution listed was transferred to the origin line. Each spot was applied evenly over the line, leaving a margin of roughly 3 cm from each short edge of the paper. Each spot was identified with a pencil directly beneath the spot. The solutions used were: (a) Fe$^{3+}$, (b) Cu$^{2+}$, (c) Co$^{2+}$, (d) solution of all three (Fe$^{3+}$, Cu$^{2+}$, and Co$^{2+}$), (e) unknown, (f) unknown. Then, the paper was dried in the fume hood. The paper was formed into a cylinder without overlapping the edges and fastened with staples to hold the shape. The beaker was then placed on the desk in a location where it remained undisturbed throughout this step. Taking care to make sure the
origin line remained above the solvent level, the paper cylinder was carefully placed into the beaker and the plastic wrap cover was replaced. When the solvent had risen above the short line, the cylinder was removed from the beaker and the solvent front position was marked. The staples were removed, and the paper was dried in the hood. Finally, the paper was sprayed with a solution of ammonium hexacyanoferrate(II), (NH₄)₄[Fe(CN)₆]. The presence of Fe³⁺ was shown by the spot turning a dark blue steel color. Cu²⁺ turned rust brown, while Co²⁺ turned a grayish purple color.

RESULTS

Table 1: Results and observations of solutions with known metal cations.

<table>
<thead>
<tr>
<th>Solution</th>
<th>Ion</th>
<th>Color (after spray)</th>
<th>Distance from origin (mm)</th>
<th>R_f</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Fe³⁺</td>
<td>dark blue</td>
<td>73</td>
<td>.97</td>
</tr>
<tr>
<td>B</td>
<td>Cu²⁺</td>
<td>rusty brown</td>
<td>60</td>
<td>.80</td>
</tr>
<tr>
<td>C</td>
<td>Co²⁺</td>
<td>periwinkle</td>
<td>38</td>
<td>.51</td>
</tr>
<tr>
<td>D</td>
<td>Fe³⁺, Cu²⁺, Co²⁺</td>
<td>blue, brown, blue</td>
<td>74, 61, 39</td>
<td>.99, .81, .52</td>
</tr>
<tr>
<td>E</td>
<td>Y, R_f = .99</td>
<td>Y, R_f = .55</td>
<td>N, R_f = ---</td>
<td></td>
</tr>
</tbody>
</table>

Figure 1: The developed chromatogram from the experiment.
ERROR ANALYSIS
One anomaly present in our experiment was a non-linear solvent front as we moved the beaker while the solvent was moving up the chromatography paper. This may have caused an uneven origin and nonlinear path travelled by the spots, which would result in an error in the calculation of the R$_f$ values.

Furthermore, looking at the relative polarities of the ions, it would be expected that Fe$^{3+}$ will have the highest R$_f$ value while Cu$^{2+}$ will have the lowest. In other words, Fe$^{3+}$ should have traveled the farthest, and Cu$^{2+}$ should have been nearest to the origin because it is the heaviest out of the three ions. Similarly, the least polar ion should have traveled the farthest and the most polar should have been nearest to the origin. However, it was observed that Fe$^{3+}$ has the highest R$_f$ value while Co$^{2+}$ has the lowest. These errors can be prevented in the future by keeping the paper level with the ground and the solvent inside, or by simply following the procedure closely so that the results will not be sacrificed.

CONCLUSION
Chromatography was successfully used in identifying the components of the unknown solutions, which were found to be Fe$^{3+}$ and Co$^{2+}$. These ions were identified using their respective R$_f$ values and the color of each spot. For example, Fe$^{3+}$ was observed to have a bluish spot with an R$_f$ value of 0.99. Co$^{2+}$, on the other hand, has a periwinkle spot. Although there were a few anomalies in the separation results, overall the experiment was successful in separating the three ions. In addition, the results also manifested the use of chromatography in non-colored analytes by adding a complexing agent to make them colored.