Experiment 5 – Introduction to Separation Techniques II

Objectives

1. To learn about solution, solubility concept and precipitation
2. To learn centrifuge technique
3. To learn how to separate Pb$^{2+}$ and Ag$^{+}$ ions from solutions.

Apparatus:

1. Centrifuge
2. Small test tubes
3. Test tube rack
4. Glass rod
5. Distilled water bottle

Solution:

1. 0.5 M lead (II) nitrate, Pb(NO$_3$)$_2$ solution
2. 0.1 M silver nitrate, AgNO$_3$, solution
3. 0.5 M potassium chromate, K$_2$CrO$_4$, solution
4. Dilute hydrochloric acid, HCl
Introduction

There are many methods that one can use to separate one substance from another. The method that one would choose will be dependent on the physical properties of the substances involved.

In this lab, we will focus on two separation techniques:

1. Precipitation
2. Centrifuge

Precipitation

If a solution contains ions, it is possible to select a reagent that will form an insoluble solid with the ions and separate them from the solution as a result of a chemical reaction. The insoluble solid is called a precipitate. During the process, the solution becomes murky and a suspension is formed. When the density of the precipitate is greater than the solution, over a period of time, the precipitate settles to the bottom. A technique known as centrifuging accelerates the settling process.

Separation of Pb (II) Ions

The separation of Pb$^{2+}$ ions from an aqueous solution will be carried out using the following reaction.

$$\text{Pb}^{2+}(aq) + \text{CrO}_4^{2-} (aq) \rightarrow \text{PbCrO}_4 (s) \text{ (yellow)}$$

The precipitating reagent that provides the CrO$_4^{2-}$ ions is potassium chromate, K$_2$CrO$_4$.

Separation of Ag$^+$ Ions
The separation of Ag$^+$ ions from an aqueous solution will be carried out using the following reaction.

$$\text{Ag}^+ \text{(aq)} + \text{Cl}^- \text{(aq)} \rightarrow \text{AgCl (s)} \quad \text{(white)}$$

The precipitating reagent that provides the Cl$^-$ ions is hydrochloric acid.

**Centrifuge Technique**

Centrifugation uses centrifugal force to separate mixtures. It works on the principle that the more-dense components of the mixture will migrate away from the axis of the centrifuge, while less-dense components of the mixture will migrate towards the axis. When the mixture to be centrifuged is comprised of a solution and a precipitate, the end result of centrifugation will cause the precipitate to settle toward the bottom of the test tube.

The solution that is on top of the precipitate is known as the supernate. When the supernate is decanted, the precipitate, which contains the ions, is separated from the original solution.

When using a centrifuge, always remember to balance the centrifuge by placing test tubes containing roughly the same volume of liquid into opposite compartments. An unbalanced centrifuge will cause the machine to operate poorly and vibrate violently.

**Decantation** is a process used to separate the liquid from the mixture to be filtered. To decant a liquid from a solid, in one hand hold the test tube that has the mixture in it. Hold a glass-stirring rod in the other hand. Touch the lip of the test tube to the glass rod and pour the mixture to be filtered. Use the glass rod as a guide to pour slowly to ensure that the solid is not carried along. This also prevents the liquid from running back along the outside of the test tube.
Part A – Separation of Pb(II) Ions from Solution as Lead (II) Chromate

Procedure:

1. Add about 20 drops of 0.5 M lead (II) nitrate, Pb(NO₃)₂ solution in a test tube. Record your observations on the data sheet.

2. Add a few drops of 0.5 M potassium chromate, K₂CrO₄ solution to the test tube. Mix well. Record your observations on the data sheet.

3. Centrifuge and separate the precipitate from the solution. Remember to balance the centrifuge with a second test tube containing approximately the same volume of liquid. Place the test tube in opposite compartments.

4. Decant the supernate. Record your observations on the data sheet.

Part B – Separation of Ag⁺ Ions from Solution as Silver Chloride

Procedure:

1. Add about 20 drops of 0.1 M silver nitrate, AgNO₃, solution in a test tube. Record your observations.

2. Add a few drops of dilute HCl solution to the test tube. Mix well. Record your observations on the data sheet.

3. Centrifuge and separate the precipitate from the solution. Remember to balance the centrifuge with a second test tube containing approximately the same volume of liquid. Place the test tube in opposite compartments.

4. Decant the supernate. Record your observations on the data sheet.
Observations

Part A – Separation of Pb(II) Ions from Solution as Lead (II) Chromate

<table>
<thead>
<tr>
<th>Step</th>
<th>Record Your Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>1 –</strong> Add 20 drops of 0.5 M Pb(NO₃)₂ solution in a test tube.</td>
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<tr>
<td><strong>2 -</strong> Add a few drops of 0.5 M K₂CrO₄ solution to the test tube.</td>
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<tr>
<td><strong>4 –</strong> Centrifuge and decant the supernate.</td>
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</table>
### Part B – Separation of Ag⁺ Ions from Solution as Silver Chloride

<table>
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<tr>
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</tr>
</thead>
<tbody>
<tr>
<td>1 – Add 20 drops of 0.1 M AgNO₃ solution in a test tube.</td>
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</tr>
<tr>
<td>2 - Add a few drops of dilute HCl solution to the test tube.</td>
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<tr>
<td>4 – Centrifuge and decant the supernate.</td>
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Questions:

1. Write the name, formula, and colour for the Pb$^{2+}$ precipitate and Ag$^+$ precipitate.

<table>
<thead>
<tr>
<th>Precipitate</th>
<th>Name</th>
<th>Formula</th>
<th>Colour</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pb$^{2+}$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ag$^+$</td>
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2. Explain how you would separate PbCl$_2$ (s) and AgCl (s).
   (Note: at the end of the separation you want pure PbCl$_2$ (s) and AgCl (s) in separate test tubes).