# EXPERIMENT 4
# THE N-BOTTLE PROBLEM

## INTRODUCTION

The purpose of this experiment is to use your knowledge about acid-base and precipitation reactions that occur in aqueous solutions to identify the ionic substances present in a set of solutions. These solutions will be labeled A through G, and you will be given the list of cations and anions that are present. All the solutions that you will be given are clear; hence you can already conclude that only cation/anion combinations that **do not react with each other** have been selected. Recall that when a cation and anion are mixed, a reaction occurs only if a precipitate is observed or a covalent compound (such as water or a gas) is formed

As a simple example, suppose that you are given three clear solutions X, Y and Z and are told that the solutions may contain the cations $Na^+$, $H_3O^+$ and $Ag^+$ (each ion used once) and the anions $CO_3^{2-}$, $NO_3^-$ and $Br^-$. Since the solutions are clear, you may conclude that one solution does *not* contain AgBr since AgBr is insoluble in water and would form a precipitate. Likewise, one solution does *not* contain $Ag_2CO_3$ since this compound is also insoluble. Thus, one solution must be $AgNO_3$. In a similar fashion, you know that none of the unknowns can contain both $H_3O^+$ and $CO_3^2$, because hydronium ion and carbonate ion react to form carbon dioxide gas and water according to the following equation:

$$CO_3^{2-}(aq) + 2\,H_3O^+(aq) \rightarrow H_2CO_3(aq) \rightarrow CO_2(g) + H_2O(l) \qquad (4\text{-}1)$$

Thus, the others must be $(H_3O)Br$ and $NaNO_3$. You could identify which is which by systematically mixing ~0.5 mL pairs of solutions and observing what happens. Suppose you mix X with Y and observe the formation of a pale yellow precipitate. Since $Na^+$ and $H_3O^+$ compounds are soluble, either X or Y must contain $AgNO_3$, and the reaction which occurs must be one of the following:

$$2\,Ag^+(aq) + CO_3^2(aq) \rightarrow Ag_2CO_3(s) \qquad (4\text{-}2)$$

$$Ag^+(aq) + Br^-(aq) \rightarrow AgBr(s) \qquad (4\text{-}3)$$

Suppose you now mix X with Z and also observe a yellow precipitate. Then, one of the above two reactions must have occurred; this identifies X as $AgNO_3(aq)$. The identity of Y and Z is still unclear. If you mix Y and Z, you will observe bubbles due the reaction described in **EQUATION 4-1**. We have no evidence at this point about $Na^+$ and $H_3O^+$. A flame test could be used to differentiate between these ions. Na gives a bright orange-yellow flame test, while $H_3O^+$ gives no discernable color in a flame. In addition, testing the unknown solutions with litmus paper can aid in our identification of the unknowns, as the presence of $H_3O^+$ will cause blue litmus to turn red.

Among the pieces of information you will need for this experiment is a set of solubility rules. Several statements of the rules are possible. However the set of rules given below is particularly useful because the rules are listed in hierarchical order; that is, Rule 1 takes precedence over Rule 2 and so on.

1. All $Li^+$, $Na^+$, $K^+$, and $NH_4^+$ salts are soluble.
2. $NO_3^-$, $C_2H_3O_2^-$ and $ClO_4^-$ salts are soluble.
3. $Pb^{2+}$ and $Hg_2^{2+}$ salts are insoluble.
4. $SO_4^{2-}$ salts are soluble except for those of $Ca^{2+}$, $Sr^{2+}$ and $Ba^{2+}$.
5. $Ag^+$ salts are insoluble.
6. $Cl^-$, $Br^-$ and $I^-$ salts are soluble.
7. $CO_3^{2-}$, $S^{2-}$, $O^{2-}$, $PO_4^{3-}$ and $OH^-$ salts are insoluble.

Thus, for example, using these rules we can predict that NaOH is soluble (Rule 1 takes precedence over Rule 7), that $Pb(C_2H_3O_2)_2$ is soluble (Rule 2 takes precedence over Rule 3) and that AgBr is insoluble (Rule 5 takes precedence over Rule 6).

## TECHNIQUE

For mixing solutions, you can use small (micro) test tubes and plastic disposable pipets with graduated volume markings on the barrel. The precise amounts of solutions you mix are not crucial; a good idea would be to use about 0.5 mL of each. You can hold the tube and flick it with your finger to achieve mixing. **It is never a good idea to put your finger over the mouth of the test tube and shake it!** After mixing solutions and observing any reaction, you may discard the contents of the test tube into the designated waste container.

You may work in pairs and discuss the experiments and interpretations with your partner. However, each person must submit an independent lab report. Alternatively, you may work alone if you so choose.

## EQUIPMENT NEEDED

- Alcohol burner
- red and blue litmus paper
- small test tubes
- nichrome wire
- dropper bottles
- glass stirring rod

## CHEMICALS NEEDED

unknown bottles labeled A-G (These unknowns contain the following cations and anions: $Ag^+$, $Ba^{2+}$, $Co^{2+}$, $H_3O^+$, $Li^+$, $Na^+$, $Sr^{2+}$ and $Cl^-$, $CO_3^{2-}$, $OH^-$, $NO_3^-$, $SO_4^{2-}$. Each solution contains a different cation, but more than one solution can contain the same anion. **Recall that a salt solution must contain both a cation and an anion.**)

## PROCEDURE

In order to help you interpret the results, you should prepare a chart detailing what **you predict** will happen when the various unknown solutions are mixed. This can be accomplished using the solubility rules given in the Introduction. For example, to determine whether $Na^+$ will form a precipitate with $CO_3^{2-}$, look for the first mention of either ion in the solubility rules. Since Rule 1 states that all $Na^+$ salts are soluble, $Na^+$ does not form a precipitate with $CO_3^{2-}$, and you should write NR (no reaction) on your chart.

If a reaction does occur for the mixing of another pair of ions, write the formula of the reaction product. A template for this chart can be found after the report sheet in this handout. Turn in the copy of this chart to your instructor, who will grade it as a portion of your lab score for this experiment and return it to you before the end of the lab period. Keep in mind that the mixing of some ions will result in the formation of a product that is a liquid or a gas such as in **EQUATION 4-1**. Also recall the important reaction that occurs between $H_3O^+$ and $OH^-$.

## Litmus Tests

Obtain about 10 mL of each solution A-G (about ½ of a bottle) in separate **clean** dropper bottles. **Make sure that the dropper tops are clean as well. Label the bottles carefully.** Test the acidity or basicity of each solution by placing a drop of the solution onto strips of red and blue litmus paper. An acidic solution ($H_3O^+$ present) will turn blue litmus red and a basic solution ($OH^-$ or $CO_3^{2-}$ present) will turn red litmus blue.

## Flame Tests

Obtain a piece of nichrome wire attached to a glass stirring rod. The wire should have a small loop on the end; if it does not, wrap the end around a pen or pencil. You will want to be sure that the wire itself is free of any material that could color the flame. Thorough rinsing of the wire with tap should be sufficient cleaning for this experiment. It will be necessary to clean the wire each time before you test a new solution. (***Why?***)

For each sample, dip the clean loop of the wire into the solution and thrust it into the flame.

Record the results, noting not only the color of the flame but also the intensity and duration. The colors of flames are as follows: Li–red; Ba–green; Na–orange-yellow; Sr–red. The other ions give no characteristic flame test color. As mentioned above, every solution that has been stored in a glass vessel will give a positive Na test due to the leaching of sodium from the glass. Thus, a positive flame test due to a dissolved unknown Na salt should exhibit a persistent and very intense orange-yellow color. As a corollary, the colors due to other cations are visible briefly immediately after the sample is introduced into the flame. Because you have other criteria besides flame tests for determining the ions present, no "known" solutions will be provided for comparison. Make some tentative hypotheses about which solution contains which cations based on the above results. **Since each of the possible cations is present in one of the unknown solutions, you should observe a single occurrence of each of the flame colors described above**.

## Mixing solutions

Now, begin to mix pairs of the solutions (~10 drops of each) together systematically in **clean** small test tubes and observe the results, being careful to observe any evidence of a reaction occurring, such as formation of a precipitate or **evolution of a gas (bubbles)**.

It is strongly recommended that you begin reaching your conclusions about which ions are present in which solutions before you leave the lab. Extra working time will not be allowed for this experiment, so be sure that you have done all the tests needed during your own lab period.

# EXPERIMENT 4
# REPORT SHEET

**Name:** ______________________________________ **Date:**__________

**Partner:**_____________________________________

**Unknown Number**_________________

**Results from Flame and Litmus Tests**

| Unknown | Flame Color | Litmus Results |
|---|---|---|
| A | | |
| B | | |
| C | | |
| D | | |
| E | | |
| F | | |
| G | | |

**Mixing Results**

| | A | B | C | D | E | F | G |
|---|---|---|---|---|---|---|---|
| A | xxxx | | | | | | |
| B | | xxxx | | | | | |
| C | | | xxxx | | | | |
| D | | | | xxxx | | | |
| E | | | | | xxxx | | |
| F | | | | | | xxxx | |
| G | | | | | | | xxxx |

# Mixing Prediction Table

Use the Solubility Rules table in the introduction to determine whether a reaction will occur when each cation and anion is mixed. If no product is formed, write NR. If a product forms, write the formula of the product. Note that some of the ions may form a covalent compound when mixed rather than a precipitate. Write down the formula of these compounds as well.

| | $Cl^-$ | $CO_3^{2-}$ | $OH^-$ | $NO_3^-$ | $SO_4^{2-}$ |
|---|---|---|---|---|---|
| $Ag^+$ | | | | | |
| $Ba^{2+}$ | | | | | |
| $H_3O^+$ | | | | | |
| $Li^+$ | | | | | |
| $Na^+$ | | | | | |
| $Co^{2+}$ | | | | | |
| $Sr^{2+}$ | | | | | |

**Notes**
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- **Do not put a pipet in any of the unknown bottles! Do not pour any leftover liquid back into the unknown bottles!**

- Be sure to write down your unknown number.

- The key step in this experiment is the systematic mixing of each unknown. Make careful observations (at least one reaction <u>will</u> form brief bubbles), and make sure the dropper bottles are labeled so you don't get them mixed up. Be sure your dropper bottles and test tubes are clean before starting, or you may get some false results.

- Remember, a precipitation reaction does not necessarily have to result in voluminous amounts of solid formed. <u>If the solution becomes cloudy, it contains a precipitate.</u>

- The unknowns may contain acids, bases, and other potentially hazardous materials. If you get any solution on you, wash it off immediately with plenty of tap water. (It is generally a good idea to do this any time you spill something on you and you don't know what it is.)

**Hints for deducing the identity of your unknown solutions:**

1) Each unknown contains an anion and a cation!
2) Draw conclusions from your flame results—remember that each cation is used once.
3) Draw conclusions based on your litmus tests.
4) If you have an unknown where you have narrowed down the possible ions to two, see what happens when you mix those ions with another ion that will give different results. For example, an unknown that turned red litmus blue contains either $OH^-$ or $CO_3^{2-}$. Which cation could you mix with these two ions that will give a different result? Hint—think bubbles!
5) The anion and cation in the same unknown **can't react with each other**. (If they did, you would see precipitate in the unknown bottles.) You can use this fact to narrow down the list of possible ions in an unknown where you already know one of the ions. For example, if you know that one of the unknowns contains $H_3O^+$, you can look at your mixing prediction table to see that the only possible anions in that same unknown are $Cl^-$, $NO_3^-$ or $SO_4^{2-}$.