ANION ANALYSIS

Much of the work you will be doing in the Chemistry 112 laboratory will be concerned with identifying positive and negative ions, that is, cations and anions, in solutions whose composition is unknown. This procedure is called QUALITATIVE ANALYSIS.

The modern chemist frequently wishes to identify the constituents in a very small amount of substance, and so he depends heavily on instrumental methods of analysis. While the procedures you will use do not use fancy and expensive instruments, your methods are still very effective in determining the major components of systems containing common inorganic ions. Because anion analysis is somewhat simpler than cation analysis, we shall begin our work in qualitative analysis with methods of identifying four common anions in solution:

PO_4^{3-}, phosphate	**Cl^-, chloride**
SO_4^{2-}, sulfate	**NO_3^-, nitrate**

After having determined the chemical reactions of the individual ions, you will be asked to identify the ions present in an unknown mixture.

CHEMICAL REACTIONS OF INDIVIDUAL ANIONS

1. The $BaCl_2$ Test

Take a set of four small test tubes. After cleaning them, label them 1 through 4, and place 4-5 drops of one of the known solutions in each tube as follows:

Be sure to record the results of your tests in your notebook.

Test tube	Known Solution
1	PO_4^{3-}, phosphate
2	SO_4^{2-}, sulfate
3	Cl^-, chloride
4	NO_3^-, nitrate

Next, make each solution slightly basic by adding 5 M ammonia (NH_3) dropwise. Making sure the solution is thoroughly mixed, test the basicity of the solution with litmus paper as demonstrated by your instructor.

The ammonia bottle may be labeled either with the formula NH_3 or, less correctly, as NH_4OH.

When the solutions are basic, note any changes that have occurred, and enter your observations in your lab book. Next, add 2-3 drops of 0.2 M $BaCl_2$ to form precipitates between Ba^{2+} and some of the anions.

*To test for a basic solution, use red litmus paper. It will turn blue if the solution basic. Just remember: **blue = base**.*

$$Ba^{2+}(\text{aqueous}) + \text{anion}(\text{aqueous}) \rightarrow [Ba(\text{anion})](\text{solid})$$

Record observations on the color and texture or appearance of the precipitates in your notebook. It is best to draw a table in your notebook something like that below.

Some of the precipitates you have formed will dissolve in acid. In each case where a precipitate has formed with $BaCl_2$, make the solution acidic with 6 M HCl (blue litmus paper should turn red in acid). Be sure to mix the solution well after adding acid! (***The most common error made in qualitative analysis laboratory is to fail to mix solutions completely!***) Record your observations. (If you had made a table as described above, you can add your observations on

Anion	PO_4^{3-}	SO_4^{2-}	NO_3^-	Cl^-
Color and texture of precipitate				
Precipitates that dissolve in HCl				

acid solubility to this table.) Discard the solutions from the tests above and clean the test tubes thoroughly.

Blue litmus paper turns red in acid solution.

2. The $AgNO_3$ Test

Once again prepare four test tubes, each containing 4-5 drops of one of the known solutions. Dilute each solution with about 1 mL of distilled water and then add 2 drops of silver nitrate, $AgNO_3$, solution. Now you should see some of the anions combine with silver ion to again produce insoluble precipitates.

$$Ag^+(aq) + anion(aq) \rightarrow [Ag(anion)](s)$$

Once again record your observations in a table such as that suggested above.

After observing the precipitates that may form with some anions, attempt to dissolve these precipitates in acid. This time, however, you must use nitric acid, HNO_3, rather than hydrochloric acid, HCl. (Why?) Add 4 drops of 3 M HNO_3 to each precipitate with silver ion, mix well, and note the results. Again record your observations in the table of results.

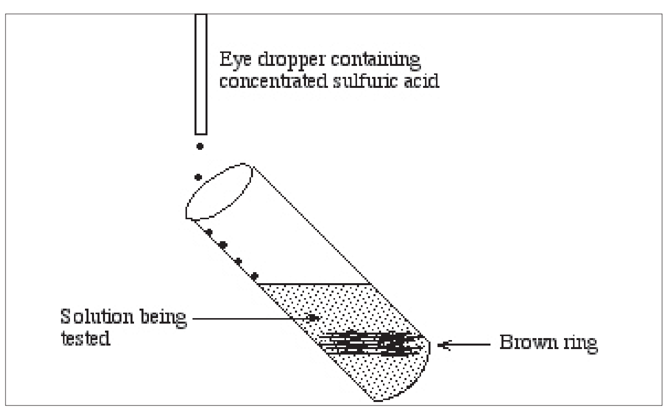
3. The Brown Ring Test For Nitrate Ion

As you may have observed in your tests thus far, the nitrate ion does not form precipitates with either Ba^{2+} or Ag^+. Therefore, we have to have some independent way of testing for the ion. The test that has been used for many years is called the ***brown ring test***, and it is specific for NO_3^-.

Remember to use blue litmus paper to test for acidity.

Place 10 drops of the solution to be tested in a clean, well-rinsed test tube. Make the solution acidic by adding 3 M H_2SO_4 as needed. Next, add 5 drops of a freshly prepared, saturated solution of iron(II) sulfate ($FeSO_4$) and mix gently. Incline the test tube at a 45° angle, and, as shown in the sketch, carefully add 5 drops of concentrated H_2SO_4 so the drops roll down the side of the test tube and slide gently onto the top of the solution. **DO NOT MIX** the solutions!

Two separate liquid layers will be observed in the test tube. If NO_3^- is present, a *very faint* brown ring will be observed near the bottom of the test tube, thereby confirming the presence of nitrate ion. Record your observations in your notebook.



QUALITATIVE ANALYSIS OF AN UNKNOWN MIXTURE

The goal of this experiment is to be able to analyze a solution that may contain a mixture of any or all of the following anions: PO_4^{3-}, SO_4^{2-}, NO_3^-, Cl^-. Based on the tests you have done above, you should be able to work out a foolproof scheme that will allow you to do this analysis. Therefore, at this point you should sit back for a moment, think about your results, and construct a scheme for the analysis of the unknown solution. As usual, you will find it helpful to construct a table, such as that below, to organize your results.

The reaction that produces the brown ring is not understood at all. It is difficult to write a balanced equation for the process.

Anion	PO_4^{3-}	SO_4^{2-}	NO_3^-	Cl^-
Summary of test results				

Once you have worked out an approach to analyzing an unknown, it is always good to try it on a solution that you have made up yourself. For example, put a few drops of each of the known solutions of PO_4^{3-}, NO_3^-, SO_4^{2-}, and Cl^- in a single test tube and see if your method of analysis will allow you to identify their presence unequivocally.

When you are satisfied with your method of analysis, work up your courage and ask your instructor for an unknown. (*He or she will want to see your notebook* at this point to make certain you have been recording your tests and observations properly. The instructor may also ask you what method of analysis you have chosen.) Determine the anion content of your unknown in the remaining time of the laboratory period, being certain to write down all of your procedures and observations. Summarize your observations in a table, and then list the results as follows:

Anions probably present: _________________________

Anions probably absent: _________________________

Show your instructor your results before leaving the laboratory, and they will be checked for you.

Very carefully write down your observations as you work through the unknown. If you have made an error, your lab instructor will be able to help you find the mistake.

WRITING UP THE NOTEBOOK

As you went through the experiment you described your observations and intermediate conclusions. There is a final portion to the experimental write-up that can be done outside of the laboratory. This consists of writing balanced equations for at least some of the reactions you observed and answering some other questions regarding the experiment. *Your grade on the laboratory book depends on the overall quality of your write-up and on your answers to the final questions.*

We normally grade the results of the experiment BEFORE you leave the laboratory.

For this experiment, you must write answers to the following questions in your laboratory book.

1. Write the balanced, net ionic equation for the reaction occurring when
   (a) Ba^{2+} is added to the SO_4^{2-} containing solution
   (b) Ba^{2+} is added to the PO_4^{3-} containing solution

2. Write the balanced, net ionic equation for the reaction occurring when
   (a) Ag^+ is added to the Cl^- containing solution
   (b) Ag^+ is added to the PO_4^{3-} containing solution

3. When some precipitates dissolve in HCl or HNO_3, the reason is that a more “stable” or less strong acid than either HCl or HNO_3 is formed. For example, barium phosphate, which is normally insoluble in water, dissolves in HCl because the weaker acid H_3PO_4 (phosphoric acid) is formed.

   $$Ba_3(PO_4)_2(s) + 6\ HCl(aq) \rightarrow 2\ H_3PO_4(aq) + 3\ BaCl_2(aq)$$

   Write a balanced equation to show how insoluble silver(I) phosphate, Ag_3PO_4, can dissolve in the strong acid HNO_3 for a similar reason.