

ANION ANALYSIS

OBJECTIVES FOR THE EXPERIMENT

The student will be able to do the following:

1. Write a general description of the qualitative analysis technique for identification of unknown substances.
2. Plan a procedure for the analysis of an unknown solution of an anion and describe that procedure with a flow chart.
3. Identify different anions using a qualitative analysis technique.

BACKGROUND

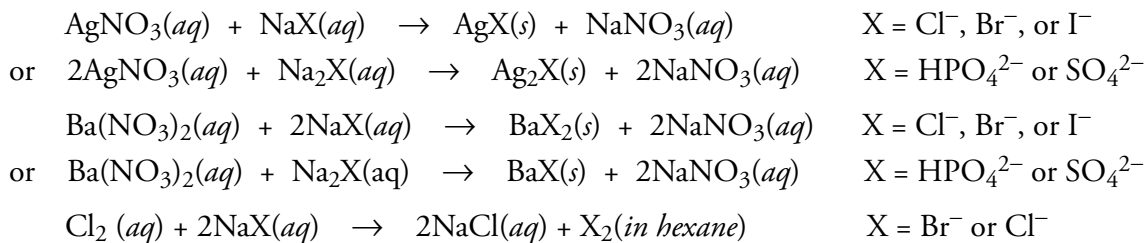
There are two main branches of analytical chemistry, qualitative analysis and quantitative analysis. Qualitative analysis involves the experimental determination of the nature of the substances in a sample. It answers the question, what's in there? Quantitative analysis involves the determination of how much of a substance is present in a sample. A chemist might test the water downstream from an out-fall of a chemical plant. The first step would be to do qualitative analysis techniques to determine whether it contains pollutants. The next step would be to use quantitative analysis techniques to find out if the concentrations were high enough to cause concern.

The goal of this experiment is to develop techniques for identifying unknown substances. You will identify unknown solutions as containing one of the following: Cl^- , Br^- , I^- , HPO_4^{2-} , or SO_4^{2-} .

You will do the following general steps.

1. You will first do tests with solutions that you know contain one of the anions.
 - a. You will determine how the anions react with silver nitrate and barium nitrate.
 - b. You will determine whether precipitates dissolve in acidic solutions.
 - c. You will determine how Br^- and I^- react with chlorine, Cl_2 .
2. Based on the observations made on these "knowns", you can devise a plan to analyze each of your unknowns. You will describe this plan with a flow chart. See below.
3. You will then do tests on three unknown solutions to determine which anion is in each solution.

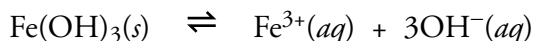
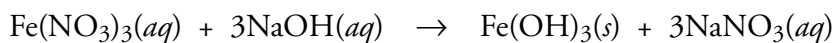
The general reactions in this experiment consist of two double displacement reactions and one oxidation-reduction reaction as shown below:



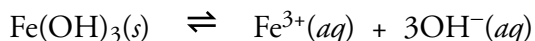
In the silver nitrate and barium nitrate tests, you will watch for the possible formation of the precipitates AgX , Ag_2X , BaX_2 , or BaX . The colors of these precipitates can be quite helpful. The appearance of any solids in these tests will show that a reaction has occurred. Even a slight cloudiness is an indication of a positive test.

If a precipitate forms in a silver nitrate or barium nitrate test, you will add nitric acid to see if the ionic compound will dissolve. If the anion is basic, the ionic compound should dissolve. When a solid ionic compound precipitates from a solution, the ions do not remain permanently in the ionic solid. Ions are constantly escaping from the solid into solution, and ions are constantly colliding with the solid and returning to the solid. These two changes occur at the same rate, so there is not normally any net change in the amount of solid in

the container or the amount of ions in solution. The following example describes this process for iron(III) hydroxide. $\text{Fe}(\text{OH})_3$ is insoluble in water, so it precipitates from a solution of iron(III) nitrate and sodium hydroxide. Iron(III) ions and hydroxide ions constantly escape from the solid and go into solution, and the ions that have escaped eventually collide with the solid and return to the solid form. Because these two changes take place at the same rate, we cannot see any change in the system.



When acid is added, the H^+ reacts with the basic anion to form an uncharged molecular substance. This stops the return of the ions to the solid form, but the rates at which the ions shift between solid to solution stays the same. Thus, the forward reaction proceeds, the reverse reactions stops, and the solid dissolves.



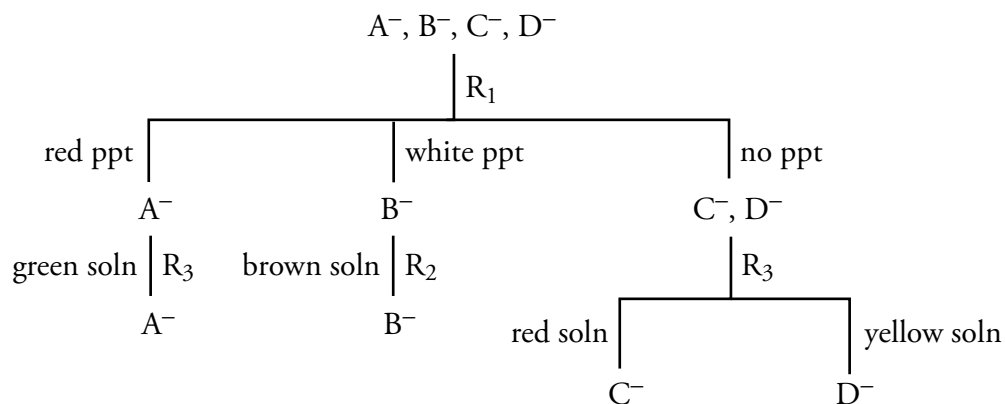
The ionic solid will not dissolve if the anion is a neutral anion, which will not react with H^+ .

In the chlorine water test, you will detect the formation of X_2 from X^- by dissolving the X_2 in hexane, C_6H_{14} . Some elemental halogens, X_2 , have very distinctive colors when dissolved in hexane. Thus, the color of the hexane layer will show whether a reaction has taken place and which halogen has been formed. Note that hexane is used only as a solvent and should not be considered when balancing the equations. The hexane is less dense than water, so it will be the top layer. In this test, you are also asked to add a drop of dilute sulfuric acid. The acid is used to generate fresh chlorine. Chlorine is not very soluble in water, so the chlorine water solution is actually a dilute bleach solution, NaOCl . The hypochlorite ion reacts with the H^+ from the sulfuric acid to generate Cl_2 .

To understand the general process, including flow charts, consider the following hypothetical analysis. Assume that we are trying to analyze an unknown that contains either A^- , B^- , C^- , or D^- . We are testing them with reagents R_1 , R_2 , and R_3 . This is a sample table of results.

	A^-	B^-	C^-	D^-
R_1	red ppt	white ppt	XXXXXXXXXX	XXXXXXXXXX
R_2	XXXXXXXXXX	brown ppt	white ppt	white ppt
R_3	green soln	XXXXXXXXXX	red soln	yellow soln

This is a sample flow chart flow chart derived from the results described on the previous page.



This is a sample table of results for an unknown analysis.

TESTS	OBSERVATIONS	CONCLUSIONS
R ₁ test	no ppt	C ⁻ , D ⁻
R ₂ test	white ppt	C ⁻ or D ⁻
R ₃ test	yellow solution	D ⁻

GENERAL CAUTIONS

1. Silver salts are poisonous. Wash your hands immediately after contact with either.
2. You should wear gloves.
3. Hexane is a euphoric substance. Avoid breathing the fumes.
4. Silver wastes, barium wastes and hexane wastes must be disposed of in the appropriate waste jars.
5. Avoid contact with the silver solutions. They can cause stains.

GENERAL PROCEDURES

1. The test tubes used may be wet, but they must be rinsed thoroughly with deionized water before use.
2. You do not need to do the known tests in a specific order. To avoid the trouble of competing for the same reagents, do whichever test is most convenient for you.

PROCEDURE

Silver nitrate test

1. Place about 1 mL of each sample to be tested in a small test tube.
 - a. All volumes used in this experiment can be estimated.
 - b. One mL equals about 20 drops.
2. Add three drops of the silver nitrate solution to each test tube, put a stopper in the tube, and shake briefly.
3. Record your observations.
 - a. If you get a precipitate, write its color and “ppt” on the table on the first page of your data sheet.
 - b. If there is no reaction, draw an “X” in the box.
4. If a precipitate has formed, add one mL of dilute nitric acid, put in a stopper, and shake it briefly.
5. Again, record your observations.
 - a. If the precipitates dissolve, write “dissolve” in the box on the data sheet.
 - b. If the precipitate does not dissolve, put an X in the box.
6. Discard all solutions in the heavy metal waste jar in the hood.

Barium nitrate test

Follow the same procedure as in the silver nitrate test, substituting barium nitrate solution for the silver nitrate solution. Discard all solutions in the heavy metal waste jar in the hood.

Chlorine water test

1. Run known tests with NaBr and NaI only.
2. Place one mL of each sample to be tested in a small test tube.
3. Add one mL of hexane, 1 drop of 3 M sulfuric acid and two drops of chlorine water.
4. Cap the test tube and shake it vigorously for half a minute.
5. Record the color of the hexane layer. The hexane will be the top layer.
 - a. If you get a change in color for your mixture, write the color of the solution and “soln”.
 - b. If there is no reaction, draw an “X” in the box.
6. Discard all solutions in the organic waste jar in the hood.

Analysis of the unknowns

1. After you have completed all the tests with known solutions, show your report sheet to your instructor.
2. Based on your known results, develop a plan for the analysis of your unknown and describe it with a flow chart.
3. Take your flow chart to your instructor for approval.
4. Upon approval, your instructor will give you three test tubes containing your unknowns.
5. Copy the unknown numbers onto your report sheet.
6. You have enough solution in each unknown test tube to do several tests.
7. Analyze your unknown by doing the tests that your flow chart suggests. Use about 1 mL of your unknown for each test.
8. Record your results on the report sheet.
9. Show your conclusions.

NAME _____

ANION ANALYSIS REPORT SHEET**KNOWN TEST RESULTS**

	AgNO_3	AgNO_3 with HNO_3	$\text{Ba}(\text{NO}_3)_2$	$\text{Ba}(\text{NO}_3)_2$ with HNO_3	Cl_2
NaCl					No Reaction
NaBr					
NaI					
Na_2HPO_4					Not Done
Na_2SO_4					Not Done

FLOW CHART

UNKNOWN A

TESTS	OBSERVATIONS	CONCLUSIONS

Unknown number _____

Conclusion for A _____

UNKNOWN B

TESTS	OBSERVATIONS	CONCLUSIONS

Unknown number _____

Conclusion for B _____

UNKNOWN C

TESTS	OBSERVATIONS	CONCLUSIONS

Unknown number _____

Conclusion for C _____