Experiment 9
Determination of an Unknown Anion

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Name:

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Key Objectives

1. Difference between qualitative and quantitative measurements.
2. Five signs that a chemical reaction has occurred.
3. Logical reasoning - inclusive, exclusive and indeterminate results.

Discussion

You accidentally forgot to label a bottle of clear liquid, how can you determine its contents? Or perhaps you find a mysterious bottle on the shelf, how can you identify it? We will explore using the chemical properties of compounds to identify an unknown liquid based on its characteristic chemical reactions. We will become familiar with the signs and observations that a chemical reaction has occurred. We will also explore several logic concepts including inclusive, exclusive, and indeterminate results.

Physical and Chemical Properties

Physical properties of substances is a characteristic of a substance that can be observed or measured without changing the identity of the substance, no chemical bonds are broken or formed. Physical properties include color, density, hardness, and melting and boiling points.

Chemical property describes the ability of a substance to undergo a specific chemical change. The substance is chemically changed, chemical bonds are broken and reformed resulting in a new substance with different physical and chemical properties.

In this experiment you will be given aqueous samples of sodium chloride (NaCl),sodium iodide (NaI), sodium hydrogen carbonate ($NaHCO_3$), sodium carbonate (Na_2CO_3), sodium hydrogen phosphate (Na_2HPO_4), and sodium sulfate (Na_2SO_4). You will test these compounds for characteristic reactions with nitric acid (HNO_3), barium nitrate ($Ba(NO_3)_2$), silver nitrate ($AgNO_3$), nitric acid (HNO_3), ammonium hydroxide (NH_4OH), and an acid-base indicator called thymol blue.

You will also be given an unknown sample that will be identical to one of the aqueous samples. After testing the known compounds, you will subject your unknown substance to those same tests. You will be able to determine the unknown sample's identity by matching its characteristic reactions with those of the known samples.

This experiment emphasizes the importance of accurate observations, properly recording them, and the use of deductive logic to draw conclusions from those observations. If you make accurate observations and record them faithfully, you will be able to identify the unknown compound without difficulty. Accurate observations are the only criterion for success. No prior knowledge of the chemistry that you will observe is necessary or assumed.

Signs of a Chemical Reaction

We will be observing the characteristic chemical properties of compounds through a series of tests with different reagents and observing if a chemical reaction occurs or not, and if so what specifically occurs. There are several signs that a chemical reaction has occurred. These signs are:

1. Color changes, that are not the result of simple dilution.
2. Evolution of a gas forming many bubbles rapidly.
3. Formation of a precipitate, normally a solid which will settle to the bottom of a test tube, but occasionally a finely divided precipitate that does not settle and often appears as only a milky color.
4. Disappearance of a precipitate.
5. Endothermic or Exothermic reactions (creation of heat or cold).

There are several cases where it is difficult to tell if a chemical reaction occurred. When dissolving a solid or precipitate, only a portion is dissolved, thus leaving some behind, and fooling a student into incorrectly reporting that no reaction occurred, or that a precipitate formed. This can be avoided by realizing that the original compounds are generally crystalline, and will sit at the bottom of a test tube (or settle out quickly), while a precipitate will have a milky appearance, and tend to spread evenly throughout the solution. A less common mistake is mistaking a lightening in color of a solution as a color change when it is simply due to the dilution of the original color. In general color changes will be fairly drastic, red to blue, green to yellow, not blue to light blue. Another common mistake is observing one or two bubbles due to the mixing or stirring of a liquid solution and reporting it as the formation of a gas. Generally the formation of a gas will be very rapid and vigorous, and should be very noticeable.

Standard nomenclature for these observations are shown below in Table 2.1. The exact nomenclature will vary from book to book, and person to person, but these general rules work well.

Observation	Nomenclature	Example
Color change	color 1 $\longrightarrow$ color 2	red $\longrightarrow$ blue
Gas formation	(g) - descriptor	(g) - vigorous
Formation of a precipitate	ppt - descriptor	milky white ppt
Disappearance of a precipitate	ppt dissolves (descriptor)	ppt dissolves (2 drops)
Exothermic or Endothermic	hot/cold	hot
No reaction	NR or x	NR or x

Table 9.1: Typical nomenclature used to describe chemical properties.

Inclusive and Exclusive Reasoning

When making observations and applying them to determining the identity of an unknown that may be a single chemical or a mixture of two or more chemicals, it is often necessary to determine whether a test provides an inclusive, exclusive or indeterminate answer. A test would be considered inclusive if the result indicated that a specific substance must be in the unknown mixture, an exclusive test would indicate that a specific substance must be excluded from the mixture. A third situation does exist which is when a test is indeterminate about whether a substance is in the mixture, neither including it or

excluding it. Consider the following set of results in Table 2.6.

Compound	Reagent 1	Reagent 2	Reagent 3
A	ppt occurs	no reaction	hot
B	ppt occurs	ppt occurs	hot
C	no reaction	ppt occurs	(g) - No temp change
D	(g)	no reaction	hot
Unknown	ppt occurs	ppt occurs	hot

Table 9.2: Reaction Data for Several Compounds

Assuming you have an unknown mixture of two of the compounds A, B, C, and D and mixed the unknown with each of the Reagents 1-3. Table 2.3 summarize the results. The results from reagent 1 are inclusive in that that you must have either compound A and/or B in your mixture, excludes compound D because no gas is observed and is indeterminate for compound C because it could be masked by A or B. The results from reagent 2 are inclusive in that you must have either B and/or C, but you can't exclude A or D being present because their presence could be masked by B or C. Reagent 3 is inclusive for A, B, and D but excludes C since no gas was formed. Combining our results we can state that our unknown can not contain C and D and therefore must be a mixture of compounds A and B.

Another way to view the results above would be to create a small table showing all possible combinations of the solutions and crossing off combinations that are excluded based on your observations. In this problem, there are 4 solutions, taken 2 at a time means that there are 6 possible combinations (as shown in the last column below). Combining the results from all 3 tests, the only solution not crossed off is the mixture of compounds A and B.

Reagent	Inclusive	Exclusive	Indeterminate	Combinations
Reagent 1	A and/or B	D	C	(a,b) (a,c) ~~(a,d)~~ (b,c) ~~(b,d)~~ ~~(c,d)~~
Reagent 2	B and/or C	none	A and D	(a,b) (a,c) (a,d) (b,c) (b,d) (c,d)
Reagent 3	A, B, D	C	none	(a,b) ~~(a,c)~~ (a,d) ~~(b,c)~~ (b,d) ~~(c,d)~~

Table 9.3: Inclusive, Exclusive, and Indeterminate results

Procedure

In this experiment you will be observing the reactions of 6 known compounds and one unknown compound with different reagents. Record all observations you make in the appropriate place. The tests are separated into 4 different tests (Parts 1-4), you may perform the tests in any order. You may work in pairs to perform Parts 1-4, but each individual should obtain a different unknown to identify.

Read the entire procedure before beginning the experiment.

Part 1 - Testing for Gas Evolution (Station 1)

1. Label 6 clean test tubes with the identity of each known compound and place 1 ml (about 20 drops) of each known liquid in each using a disposable pipet.
2. Add 5 drops of 3 M Nitric Acid to each test tube and record your observations. Be very careful with the Nitric Acid, it will eat holes in your clothes!
3. Discard the contents of the test tube in the sink and rinse with distilled water. You may use these same test tubes for the rest of the experiment.

Part 2 - Barium Nitrate and Nitric Acid Test (Station 2)

1. Label 6 clean test tubes with the identity of each known compound
2. Using the known solutions available in the laboratory place 1 mL (20 drops) of each known compound into your labelled test tubes.
3. Add 3 drops of ammonia to each test tube and stir with a clean stirring rod. Remove the stirring rod and touch a drop of each solution to a strip of red litmus paper. The paper should turn from red to blue, if it does not add drops of ammonia to the solution until it does. Do not record any results for this step, we are simply preparing the solution for the next step.
4. Add 5 drops of the Barium Nitrate solution to each test tube. Mix each tube gently (as demonstrated in class) to obtain a homogeneous solution. Record your observations in the column labelled Barium Nitrate.
5. The next step only applies to those test tubes that contained precipitates. Add 5 drops of 6 M Nitric Acid to each of these test tubes. Using a clean stirring rod, test each solution with a strip of blue litmus paper. If the paper does not turn from blue to pink, continue adding drops until it does. Examine each test tube carefully and note which of the precipitates has dissolved. Record your observations in the Barium Nitrate + Nitric Acid column.
6. Discard the solutions in the test tubes in the Waste Container labelled "Barium or Ba^{+2} Waste" which can be found in the hood.
7. Wash your test tubes and rinse them with distilled water.

Part 3 - Silver Nitrate and Nitric Acid Test (Station 3)

1. Label 6 clean test tubes with the identity of each known compound
2. Using the known solutions available in the laboratory place 1 mL (20 drops) of each known compound into your labelled test tubes.
3. Add 5 drops of 0.1 M Silver Nitrate solution to each test tube. Mix each tube gently (as demonstrated in class) to obtain a homogeneous solution. Record your observations in the column labelled Silver Nitrate.
4. The next step only apply to those test tubes that contained precipitates. Add 10 drops of 6 M Nitric Acid to each of these test tubes. Using a clean stirring rod, test each solution with a strip of blue litmus paper. If the paper does not turn from blue to pink, continue adding drops until it does. Examine each test tube carefully and note which of the precipitates has dissolved. Record your observations in the Silver Nitrate + Nitric Acid column.
5. Discard the solutions in the test tubes in the Waste Container labeled "Silver or Ag^+ Waste" which can be found in the hood.
6. Wash your test tubes and rinse them with distilled water.

Part 4 - Thymol Blue test (Station 4)

Thymol blue is a strong dying agent, it will stain clothing and fingers so be careful with it.

1. Label 6 clean test tubes with the identity of each known compound
2. Using the known solutions available in the laboratory place 1 mL (20 drops) of each known compound into your labeled test tubes.
3. Add 5 drops of thymol blue to each test tube.
4. Dilute each solution by adding 2.5 mL of water.
5. Record your observations.
6. Carefully add 1 drop of 6 M Nitric Acid to each test tube.
7. Stir each tube gently and record your observations in the column labeled Thymol Blue.
8. Discard the solutions in the test tubes in the sink.
9. Wash your test tubes and rinse them with distilled water.

Part 5 - Identifying an unknown solution

1. Perform each of the above tests (Parts 1-4) on your unknown solution and record your results.
2. You should be able to identify your unknown solution after comparing the results of your unknown with those of the known solutions. If any ambiguities occur or you are unable to identify your

unknown repeat as many of the tests as you find necessary. You may consult your instructor as to which tests are most likely to be in error.

3. The Thymol Blue test is very sensitive to the concentration of the solutions tested. If your results for your unknown do not match any of the known compounds, you may try adding a little more of your unknown to the solution to make it more concentrated, and/or add water to the solution to make it more dilute.

4. Identify your unknown, and provide an explanation as to why you believe it to be the compound you chose.

Results

	Station 1	Station 2	
Compound	**Gas Evolution**	**Barium Nitrate**	**Barium Nitrate + Nitric Acid**
NaCl			
NaI			
$NaHCO_3$			
Na_2CO_3			
Na_2HPO_4			
Na_2SO_4			
Unknown#			

Table 9.4: Results: Part 1-2

	Station 3		Station 4
Compound	**Silver Nitrate**	**Silver Nitrate + Nitric Acid**	**Thymol Blue**
NaCl			
NaI			
$NaHCO_3$			
Na_2CO_3			
Na_2HPO_4			
Na_2SO_4			
Unknown#			

Table 9.5: Results: Part 3-4

I was given unknown NUMBER _____I think it is _____because ...(Explain)

Post Lab Questions

1. An unknown sample contains two out of the following five compounds. Use information recorded below to determine the identity of the unknown. For each of the results below state which compounds are Inclusive (could be in your unknown), Exclusive (can't be in your unknown), or are Indeterminate (may or may not be in your unknown). Finally, cross out any combinations (solution) that are excluded by the test by crossing out the number/solution in the chart.

Compound	Reagent 1	Reagent 2	Reagent 3	Reagent 4	Reagent 5
1	ppt	NR	NR	(g)	NR
2	ppt	ppt	NR	NR	ppt
3	NR	ppt	Hot	(g)	NR
4	NR	NR	Hot	(g)	NR
5	ppt	NR	Hot	NR	ppt
Unknown	ppt	ppt	Hot	(g)	NR

Table 9.6: Reaction Data for Several Compounds

1(a) Reagent 1 Results: A white fluffy ppt was formed.

Inclusive: (1,2) (1,3) (1,4) (1,5)
Exclusive: (2,3) (2,4) (2,5) (3,4) (3,5)
Indeterminate: (4,5)

1(b) Reagent 2 Results: A chunky yellow ppt is formed.

Inclusive: (1,2) (1,3) (1,4) (1,5)
Exclusive: (2,3) (2,4) (2,5) (3,4) (3,5)
Indeterminate: (4,5)

1(c) Reagent 3 Results: The reaction mixture was warm to the touch.

Inclusive: (1,2) (1,3) (1,4) (1,5)
Exclusive: (2,3) (2,4) (2,5) (3,4) (3,5)
Indeterminate: (4,5)

1(d) Reagent 4 Results: Vigorous gas was evolved.

Inclusive: (1,2) (1,3) (1,4) (1,5)
Exclusive: (2,3) (2,4) (2,5) (3,4) (3,5)
Indeterminate: (4,5)

1(e) Reagent 5 Results: No reaction occured

Inclusive: (1,2) (1,3) (1,4) (1,5)
Exclusive: (2,3) (2,4) (2,5) (3,4) (3,5)
Indeterminate: (4,5)

2. Which 2 compounds are in the unknown? Explain.

3. Based on chemical properties, is it possible to be 100% sure that a chemical unknown you test which matches the properties of an existing compound is indeed that compound? Explain.

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Name: _____ Class: _____ Date: _____

Prelab Questions

1. What is the difference between an inclusive, exclusive and indeterminate test results?

2. List the five signs that a chemical reaction has occurred.

3. List two typical mistakes that a student might make when deciding whether a chemical reaction occurred or not.

4. When adding Thymol blue (a dark blue liquid) to a clear liquid, the resulting solution is light blue. Did a reaction occur? Explain.

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