Introduction to Qualitative Analysis

Introduction

Qualitative analysis is a way inorganic chemists have to determine what [ions] is [are] present in a chemical sample. Qualitative analysis tells the chemist only what is present, NOT how much is present (the latter used to be called quantitative analysis and is now called analytical chemistry).

For all intents and purposes, "Qual" takes advantage of the various solubility rules by manipulating the solubilities of various ions in different solvents, at different pH's and/or as complex ions.

Anions are derived from the nonmetals in the upper right of the periodic table. Cations are from the metals below and to the left of the non-metals in the periodic table. There are five classical groups of cations -- NOT to be confused with the GROUPS on the periodic table. These cation groups are based solely on the manner in which they may be identified, table, below, or removed from a mixture containing all of the cations in the table, below:

Cation Groups [1]				
Group 1	Group 2	Group 3	Group 4	Group 5
HCl Group	Acidic Hydrogen Sulfide Group	Basic Hydrogen Sulfide Group	Ammonium Carbonate Group	Soluble Group
Silver (I)*	Mercury (II)	Aluminum (III)	Calcium (II)	Sodium (I)
Mercury (I)	Lead (II)	Chromium (III)	Strontium (II)	Potassium (I)
Lead (II)	Bismuth (III)	Iron (II & III)	Barium (II)	Magnesium (II)
	Copper (II)	Manganese (II)		Ammonium ion
	Cadmium (II)	Cobalt (II)		
	Arsenic (III & V)	Nickel (II)		
	Antimony (III & V)	Zinc (II)		
	Tin (II & IV)			

*Roman Numeral following the name of the metal indicates the positive charge on the ion, e.g., Silver(I) = Ag^{+}.

The anions, above, are not grouped according to reactivity with different solvents as are the cations.

Although this experiment is a descriptive introduction to "Qual", it must, nevertheless, be remembered that "Qual" is based also in ionic equations and numerical calculations. This lab experiment will not attempt to cover these latter two bases. This lab, however, WILL cover some simple techniques in Qual" -- you will be expected to learn what the final reaction products are of each experimental method.

Experimental

Supplies and Chemicals			
1-spot plate	$NaNO_2$	Disposable test tubes	$AgNO_3$
1-spatula	Na_2SO_4	Magnesia mixture	Cotton
Na_2S	6, 9 or 10 M H_2SO_4	Disposable pipets	Red litmus paper
Na_2CO_3	KCl	1-glass stirring rod	Devarda's alloy
KNO_3	6 M HCl	6 M NaOH	$FeCl_3$
7.4 M NH_3	$NaHCO_3$ solution	$CuSO_4$	Test tube rack

Anions: Part 1 [2]

Do this part in the hood AFTER you have placed a crystal or two of Na_2S, Na_2CO_3, $NaNO_2$ and Na_2SO_4 in each of 4 wells in the spot plate so that you have 4 wells with one crystal each. Once you have taken this to the hood, add a drop of the sulfuric acid to each crystal and observe each reaction one well at a time. Describe the odor or appearance of the reaction product of each anion in the table below (NOTE: waft the vapors towards you instead of directly inhaling them over the spot plate):

Observations for Anions: Part 1		
Substance (Anion)	Crystal before reaction with sulfuric acid	After reaction with sulfuric acid
Na_2S (S^{2-})		
Na_2CO_3 (CO_3^{2-})		
$NaNO_2$ (NO_2^-)		
Na_2SO_3 (SO_3^{2-})		

Anions: Part 2 [2]

Place a test tube in a test tube rack. Label it #1. Place about the size of a third of a small pea of the following crystals into the respective tube:

Tube #1
KCl

To the test tube, above, add about 3 cm of distilled water and mix. Now add, in order, to the tube as follows in the table, below:

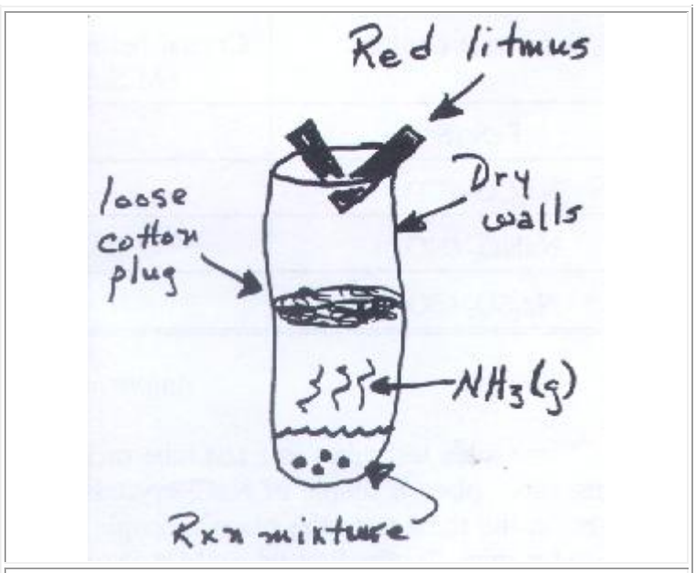
Tube #1	Observations before reaction	Observations during and after reaction
$AgNO_3$, drop-by-drop		

Anions: Part 3 [2]

Obtain a DRY test tube and place about a third the size of a small pea of crystals of KNO_3 in the bottom of the tube with 0.5 mL of deionized water containing 0.5 mL 6 M NaOH. Transfer the two liquids (the solution of water and NaOH) with a disposable pipet so that the walls of the test tube do not get wet. Place about a quarter the size of a small pea of crystals of Devarda's alloy in the solution, place a loose cotton plug in the test tube (Figure, below) and then place a moistened piece of red litmus paper in the tube in the shape of a "V". This reaction requires patience, as it may begin immediately or it may take 15-20 minutes to begin.

CAUTION: this reaction can get very hot! Do NOT hold this in your hand after the reaction has started.

The red litmus ought to turn blue as a gas exits the mixture in the test tube. CAREFULLY waft some of the vapors towards your nose. What is the gas?



Sketch of apparatus for nitrate determination from KNO_3.

The end products of the anion analysis are as follow in this table:

Reaction Products from Anion Analysis					
Anion	Product	Observation	Anion	Product	Observation
S^{2-}	H_2S	Vile	Cl^{-1}	AgCl	White ppt
CO_3^{2-}	CO_2	Odorless, fizzing	Br^{-1}	Br_2	Orange to amber in CCl_4
NO_2^{-1}	NO_2	Brown, sharp	I^{-1}	I_2	Violet in CCl_4
SO_3^{2-}	SO_2	Sharp, white	NO_3^{-1}	NH_3	Litmus turns blue from red

Cation Analysis

Obtain a test tube rack and 20 disposable test tubes. Set up your test tube rack as in the table below, remembering that rows run left to right and columns run top to bottom (or front to back in the rack), placing the reagents as indicated in the tubes, below:

Tube Set-Up for Cation Analysis					
	Column 1	Column 2	Column 3	Column 4	Column 5
Row 1	5 drops 2% $CuSO_4$	5 drops 2% $CuSO_4$	5 drops 2% $CuSO_4$	5 drops 2% $CuSO_4$	5 drops 2% $CuSO_4$
Row 2	5 drops $AgNO_3$	5 drops $AgNO_3$	5 drops $AgNO_3$	5 drops $AgNO_3$	5 drops $AgNO_3$
Row 3	5 drops $FeCl_3$	5 drops $FeCl_3$	5 drops $FeCl_3$	5 drops $FeCl_3$	5 drops $FeCl_3$
Row 4	5 drops Magnesia mixture	5 drops Magnesia mixture	5 drops Magnesia mixture	5 drops Magnesia mixture	5 drops Magnesia mixture

One tube and one column at a time, add 4 drops of the following reagents to each tube in the column as indicated, below:

Reagent Dispensation Table				
Column 1	Column 2	Column 3	Column 4	Column 5
6 M HCl	H_2SO_4	6M NaOH	7.4 M NH_3	$NaHCO_3$ solution

And record your observations in the table, below:

Tube Set-Up for Cation Analysis Observations					
	Column 1	Column 2	Column 3	Column 4	Column 5
Row 1					
Row 2					
Row 3					
Row 4					

The end-products of the cation analysis are shown in the table, below:

End-Products of Cation Analysis		
Cation	Product	Observation
Ag^{+1}	AgCl	White ppt
Fe^{3+}	Complex	Brown ppt
Cu^{2+}	$CuCO_3$	Light blue ppt
Mg^{2+}	$Mg(OH)_2$	White ppt

Questions

Complete the questions on a separate piece of paper and attach to this experiment for turn-in.

1. Based upon your results, design a method in flow chart form from which each ion could be separated from a solution containing all of the cations above.
2. Based on the following data, construct a separation scheme in flow chart form from a mixture of the cations listed, below:

Cation →	Ag^{+1}	Cu^{2+}	Fe^{3+}	Sr^{2+}
Reagent 1	HCl	HCl	HCl	HCl
Result	White ppt	No reaction (NR)	Yellow solution	NR
Reagent 2	NaOH	NaOH	NaOH	NaOH
Result	Brown ppt	Powder blue ppt	Red ppt	NR
Reagent 3	$(NH_4)_2CO_3$	$(NH_4)_2CO_3$	$(NH_4)_2CO_3$	$(NH_4)_2CO_3$
Result	NR	NR	NR	White ppt
Reagent 4	H_2S with acid	H_2S with acid	H_2S with acid	H_2S with acid
Result	Black ppt	Brown ppt	NR	NR
Reagent 5	H_2SO_4	H_2SO_4	H_2SO_4	H_2SO_4
Result	NR	White ppt	NR	White ppt

3. Write, using chemical symbols and notation, the ions described in the first table of this whole experiment.

References

1. King, E.J.: Ionic Reactions and Separations: Experiments in Qualitative Analysis. (Harcourt Brace Jovanovich: Austin) © 1973, pp. 129, 143, 163, 183, 198.
2. Ibid. pp. 61, 64, 66.

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