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Experiment 10: 10-1	
Name: _____	
Date: _____	
Objective: To identify the cations and anions in a given sample using qualitative analysis.	
Theory: Qualitative analysis is the process of identifying the presence or absence of specific ions in a sample. It is based on the characteristic reactions of these ions with various reagents.	
Procedure: 1. Preparation of the sample: Weigh a small amount of the sample and dissolve it in distilled water. 2. Preliminary tests: Perform tests for the presence of carbonate, sulfide, and phosphate ions. 3. Cation analysis: Add dilute HCl to the solution and observe the formation of a precipitate. This indicates the presence of Ag ⁺ , Pb ²⁺ , and Hg ₂ ²⁺ . 4. Anion analysis: Add dilute H ₂ S to the solution and observe the formation of a precipitate. This indicates the presence of S ²⁻ , CO ₃ ²⁻ , and PO ₄ ³⁻ .	
Results: _____	
Conclusion: _____	

Introduction to Qualitative Analysis

Qualitative Analysis

Introduction

Purpose

The purpose of this experiment is to identify the cations and anions in a given sample using qualitative analysis.

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Procedure

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5. Results: _____

Conclusion

The purpose of this experiment is to identify the cations and anions in a given sample using qualitative analysis. The purpose of the experiment is to identify the cations and anions in a given sample using qualitative analysis.

Results

The results of the experiment are as follows: _____

Discussion

1. Preparation of the sample: Weigh a small amount of the sample and dissolve it in distilled water.
2. Preliminary tests: Perform tests for the presence of carbonate, sulfide, and phosphate ions.
3. Cation analysis: Add dilute HCl to the solution and observe the formation of a precipitate. This indicates the presence of Ag⁺, Pb²⁺, and Hg₂²⁺.
4. Anion analysis: Add dilute H₂S to the solution and observe the formation of a precipitate. This indicates the presence of S²⁻, CO₃²⁻, and PO₄³⁻.

Procedure 1 - Qualitative Analysis of Cations & Anions I

Sample 1	
Initial group	_____
Preliminary tests	_____
Preliminary identification	_____
1 st confirmatory test	_____
2 nd confirmatory test	_____
Final identification	_____
Initial test	_____
Final identification	_____
Sample 2	_____

Sample 2	
Initial group	_____
Preliminary tests	_____
Preliminary identification	_____
1 st confirmatory test	_____

The Solubility of Barium Salts in Neutral and Acidic Solutions

	A.1 Testing Recording in Neutral Solution		A.2 Testing Recording in Acidic Solution	
	Drops 0.1M Ba ²⁺ required for Precipitation in Neutral Solution	Precipitate dissolve in acid (y/n)?	Bubble formed in acid (y/n)?	Drops 0.1M Ba ²⁺ required for Precipitation in Acidic Solution
CO ₃ ²⁻				
SO ₄ ²⁻				
PO ₄ ³⁻				
NO ₃ ⁻				
Cl ⁻				
I ⁻				

If a precipitate did form when barium ions were added to a neutral solution, will that precipitate dissolve if the solution is made acidic? Add three drops of 6M HNO₃ to those test tubes that have a precipitate and stir the solution well. Record the observation in the right column under the "A.1.Neutral Solution" under the column "Precipitate dissolve in acid (y/n)?"

Which barium precipitates dissolved in nitric acid?

A.2) Now we shall test the solubilities of six barium salts in acidic solution. Again place 2 mL samples of the six known anion solutions individually in six clean test tubes so that the reaction of each anion may be observed. Make each of these six solutions acidic by adding 3 drops of 6M HNO₃. As you stir 6M HNO₃ in each test tube observe and record in the right column of the table above if a gas forms. Record bubble formation on the first column under "A.2.Acidic Solution".

Which anion reacts with acid to form tiny bubbles of an insoluble gas? What is the identity of the gas?

Now add drops of 0.1M BaCl₂ to each of the six test tubes. If no precipitate forms after adding 5 drops of 0.1M BaCl₂, you may assume that the anion does not form an insoluble barium salt in acidic solutions. Record your observation in the right column of the table above.

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Ariel Chen

Qualitative Analysis of Cations Lab Report

Step	Procedure	Known Solution		Unknown Solution (#1)	
		Results	Conclusion	Results	Conclusion
1	Add HCl to known solution in TT1. Centrifuge. Pour colored solution into TT2.	White ppt forms. Other ions present in solution in TT2	Ag ⁺ , Pb ²⁺ , and/or Hg ₂ ²⁺ present	White ppt forms. Other ions present in solution in TT2	Ag ⁺ , Pb ²⁺ , and/or Hg ₂ ²⁺ present
2	Add 2 mL of distilled water to the precipitate and place the test tube in a boiling water bath for three minutes. Stir. Centrifuge for a few seconds. Pour the supernatant liquid into another test tube. Immediately add two drops of K ₂ CrO ₄ to the liquid.	Yellow precipitate	Pb ²⁺ present	Yellow precipitate	Pb ²⁺ present
3	Add one mL of NH ₃ to the white precipitate from Step 2. Stir. Black residue. Centrifuge, then decant the solution into another test tube and discard the black residue.	Black residue	Hg ₂ ²⁺ present	No white ppt from Step 2	Hg ₂ ²⁺ and Ag ⁺ both absent
4	Add HNO ₃ to the solution from Step 3 until the solution is acid or a permanent white precipitate is formed.	White precipitate	Ag ⁺ present		
5	Neutralize the solution from procedure 1 (TT2) by adding NH ₃ solution until the solution is basic. Check with litmus paper after stirring thoroughly. Add one mL in excess. Add one mL of NH ₄ Cl. Colored ppt. Centrifuge and separate the solution into another test tube. Set the solution aside for Procedure 10. Treat the precipitate by Procedure 6.	Colored ppt	Fe ³⁺ and/or Al ³⁺ present	Colored ppt	Fe ³⁺ and/or Al ³⁺ present
6	Dissolve the precipitate from Procedure 5 by adding 6-M HCl dropwise. Stir after each addition. Make basic to litmus with 6-M NaOH. Stir. Then add 1-2 mL in excess. Precipitates. Centrifuge and separate the solution into another test tube. Set the solution aside for Procedure 8. Treat the precipitate by Procedure 7.	Precipitate forms	Precipitate: Fe(OH) ₃	Precipitate forms	Precipitate: Fe(OH) ₃
7	Dissolve the precipitate from Procedure 6 by adding dilute 6-M HCl dropwise. Stir. Add three drops	Deep red color: Fe(SCN) ²⁺	Fe ³⁺ present	Deep red color: Fe(SCN) ²⁺	Fe ³⁺ present

Under these conditions the $\text{[Fe(SCN)}^{\text{-}}\text{]}^{\text{2+}}$ will decompose. A dark red solution upon the addition of $\text{[Fe(NO}_3\text{)}_3\text{]}^{\text{3+}}$. Now add 2 drops of 0.1 M $\text{[Fe(NO}_3\text{)}_3\text{]}^{\text{3+}}$. You will then perform the same test on 1 mL of your unknown solution which may or may not contain the anion in the control solution. Loading PreviewSorry, preview is currently unavailable. Name: _____ Date: _____ Lab Partner: _____ Lab Section: _____ Indicate your observations in the table below. Ion Control Diluted Control Unknown $\text{[CO}_3^{\text{-}}\text{]}^{\text{2-}}$ $\text{[SO}_4^{\text{-}}\text{]}^{\text{2-}}$ $\text{[PO}_4^{\text{-}}\text{]}^{\text{3-}}$ $\text{[SCN}^{\text{-}}\text{]}^{\text{-}}$ $\text{[Cl}^{\text{-}}\text{]}^{\text{-}}$ $\text{[NO}_3^{\text{-}}\text{]}^{\text{-}}$ Unknown ID number: _____ Ions present in your unknown: _____ Write the net-ionic equation for the chemical reaction that occurs in each of the following spot tests. Test for the presence of phosphate ion, $\text{[PO}_4^{\text{-}}\text{]}^{\text{3-}}$ Add 1mL of 0.5 M $\text{[Na}_2\text{HPO}_4\text{]}^{\text{-}}$ (or your unknown) and 1 mL of 6 M $\text{[HNO}_3\text{]}^{\text{-}}$ to a small test tube. Add 1 mL of 0.5 M $\text{[NaNO}_3\text{]}^{\text{-}}$ (or your unknown) and 1 mL of 6 M $\text{[NaOH]}^{\text{-}}$ to a small test tube. Important: If your unknown contains $\text{[SCN}^{\text{-}}\text{]}^{\text{-}}$ it will interfere with the test for the $\text{[NO}_3^{\text{-}}\text{]}^{\text{-}}$. Add the $\text{[Fe(NO}_3\text{)}_3\text{]}^{\text{3+}}$ to the supernatant solution. Next add 2 to 3 drops of 0.1 M $\text{[AgNO}_3\text{]}^{\text{-}}$. $\text{[Ag}^{\text{+}}\text{]}^{\text{+}}$ (aq) + $\text{[SCN}^{\text{-}}\text{]}^{\text{-}}$ (aq) $\rightarrow$ [AgSCN(s)] (label(2)) In this case put 1 mL of your unknown sample into a small beaker (30 mL or 50 mL) and add 1 mL of 6 M $\text{[HNO}_3\text{]}^{\text{-}}$. Suppose that a series of spot tests on an unknown solution yields the following results. Then add a few granules of aluminum metal and put the test tube in the hot water bath. Important: If your unknown contains $\text{[SCN}^{\text{-}}\text{]}^{\text{-}}$ it will interfere with the test for the $\text{[Cl}^{\text{-}}\text{]}^{\text{-}}$ since it will form a white precipitate with $\text{[Ag}^{\text{+}}\text{]}^{\text{+}}$. A yellow precipitate of indicates the presence of $\text{[PO}_4^{\text{-}}\text{]}^{\text{3-}}$. If the red litmus paper turns uniformly blue (due to $\text{[NH}_3\text{]}^{\text{-}}$ vapor coming out of the test tube) then it can be concluded that $\text{[NO}_3^{\text{-}}\text{]}^{\text{-}}$ is present in the unknown. You can download the paper by clicking the button above. For the unknown if no effervescence is observed, place the test tube in the hot water bath before concluding that $\text{[CO}_3^{\text{-}}\text{]}^{\text{2-}}$ is not present. Waste Disposal All waste generated by this lab is toxic and must be disposed of in the hazardous waste container! Unused sample of the unknown should be discarded in the hazardous waste container. In this experiment you will use qualitative analysis to identify the various anions in a sample. Wash the empty test tube and return it to your instructor's bench. Place the test tube in a hot water bath for about 2 minutes. Next add a 1 mL of 0.5 M $\text{[Fe(NO}_3\text{)}_3\text{]}^{\text{3+}}$ and stir thoroughly. In the concentrated control solution you should see effervescence for at least a few seconds. Centrifuge the mixture and decant the supernatant solution into another small test tube. The reaction between $\text{[Al]}^{\text{3+}}$ and $\text{[NaOH]}^{\text{-}}$ will produce $\text{[H}_2\text{]}^{\text{0}}$ gas which will reduce the $\text{[NO}_3^{\text{-}}\text{]}^{\text{-}}$ to $\text{[NH}_3\text{]}^{\text{-}}$. A finely divided, white precipitate indicates the presence of the $\text{[SO}_4^{\text{-}}\text{]}^{\text{2-}}$ ion. $\text{[Fe}^{\text{3+}}\text{]}^{\text{3+}}$ + $\text{[PO}_4^{\text{-}}\text{]}^{\text{3-}}$ (aq) $\rightarrow$ $\text{[FePO}_4\text{(s)]}$ (label(1)) In this case it will be necessary to remove all of the $\text{[PO}_4^{\text{-}}\text{]}^{\text{3-}}$ before any conclusion can be made concerning the presence of $\text{[SCN}^{\text{-}}\text{]}^{\text{-}}$. Next add a few drops of 1 M $\text{[BaCl}_2\text{]}^{\text{-}}$. Indicate whether each of the following is present, not present, or undetermined. Place the test tube in the hot water bath and continue to stir. Boil the solution very gently (directly over the open flame) until the volume has decreased by about one half. In the previous two experiments, you have used qualitative analysis to determine the identity of various cations in a sample. Now pour this solution into a small test tube, add 1 mL of 6M $\text{[HNO}_3\text{]}^{\text{-}}$ and 2 to 3 drops of 0.1 M $\text{[AgNO}_3\text{]}^{\text{-}}$. The formation of a white, curdy precipitate indicates the presence of $\text{[Cl}^{\text{-}}\text{]}^{\text{-}}$. A white precipitate upon the addition of $\text{[AgNO}_3\text{]}^{\text{-}}$. The solid may again be discarded in the waste. A white precipitate upon the addition of $\text{[BaCl}_2\text{]}^{\text{-}}$ (aq) No yellow precipitate upon the addition of $\text{[Fe(NO}_3\text{)}_3\text{]}^{\text{3+}}$. Specifically you will test for the presence of each of the following anions: $\text{[CO}_3^{\text{-}}\text{]}^{\text{2-}}$, $\text{[SO}_4^{\text{-}}\text{]}^{\text{2-}}$, $\text{[PO}_4^{\text{-}}\text{]}^{\text{3-}}$, $\text{[SCN}^{\text{-}}\text{]}^{\text{-}}$, $\text{[Cl}^{\text{-}}\text{]}^{\text{-}}$, $\text{[NO}_3^{\text{-}}\text{]}^{\text{-}}$. The methodology used in identifying the anions will be different than that which was used to identify cations. The production of the aqueous $\text{[Fe(SCN)}^{\text{-}}\text{]}^{\text{2+}}$ complex ion in the spot test for thiocyanate, $\text{[CO}_3^{\text{-}}\text{]}^{\text{2-}}$, $\text{[SCN}^{\text{-}}\text{]}^{\text{-}}$, $\text{[SO}_4^{\text{-}}\text{]}^{\text{2-}}$, $\text{[Cl}^{\text{-}}\text{]}^{\text{-}}$, $\text{[PO}_4^{\text{-}}\text{]}^{\text{3-}}$, $\text{[NO}_3^{\text{-}}\text{]}^{\text{-}}$. The production of the white precipitate in the spot test for sulfate. The diluted anion solutions can be prepared by adding 3 drops of deionized water to 9 drops of the anion solution. Effervescence indicates the presence of $\text{[CO}_3^{\text{-}}\text{]}^{\text{2-}}$. The $\text{[NH}_3\text{]}^{\text{-}}$ can be detected by placing a piece of moistened red litmus paper directly above (but not in contact with) the mouth of the test tube. Important: If your unknown contains $\text{[PO}_4^{\text{-}}\text{]}^{\text{3-}}$ it will interfere with the test for the $\text{[SCN}^{\text{-}}\text{]}^{\text{-}}$ since it will form a precipitate with $\text{[Fe}^{\text{3+}}\text{]}^{\text{3+}}$. Test for the presence of carbonate ion, $\text{[CO}_3^{\text{-}}\text{]}^{\text{2-}}$ Add 1 mL of 1M $\text{[Na}_2\text{CO}_3\text{]}^{\text{-}}$ (or your unknown) and 1 mL of 6M $\text{[HCl]}^{\text{-}}$ to a small test tube. If more precipitate forms, centrifuge and decant a second time. Please be careful when handling them. To the supernatant solution add 1 mL of 1 M $\text{[Na}_2\text{CO}_3\text{]}^{\text{-}}$. Add 1 mL of 0.5 M $\text{[KSCN]}^{\text{-}}$ (or your unknown) and 1 mL of 6 M acetic acid ($\text{[HC}_2\text{H}_3\text{O}_2\text{]}^{\text{-}}$) to a small test tube. The production of carbon dioxide gas in the spot test for carbonate. In these situations the interfering ions must be removed before the test can be performed. Test for the presence of sulfate ion, $\text{[SO}_4^{\text{-}}\text{]}^{\text{2-}}$ Add 1mL of 0.5 M $\text{[Na}_2\text{SO}_4\text{]}^{\text{-}}$ (or your unknown) and 1mL of 6M $\text{[HCl]}^{\text{-}}$ to a small test tube. Based on your observations indicate which anions are present in your unknown sample. In this case add 1 mL of your unknown to 1 mL of 1 M $\text{[CuSO}_4\text{]}^{\text{-}}$ in a small test tube. The solid may be discarded in the waste. A white curdy precipitate indicates the presence of $\text{[Cl}^{\text{-}}\text{]}^{\text{-}}$. Now add $\text{[Fe(NO}_3\text{)}_3\text{]}^{\text{3+}}$ to the supernatant solution. General Instructions For each anion you will perform a positive control test on 1 mL of solution of the anion, followed by a positive control experiment on 1 mL of a diluted solution of the anion. Here you will use a small portion of the unknown mixture (1 mL) to perform a spot test for each anion individually. In some cases the test for a particular ion will be complicated by the presence of other ions in the mixture that will interfere with the test. Chemicals 1 M $\text{[Na}_2\text{CO}_3\text{]}^{\text{-}}$, 0.5 M $\text{[Na}_2\text{SO}_4\text{]}^{\text{-}}$, 1 M $\text{[BaCl}_2\text{]}^{\text{-}}$, 0.5 M $\text{[Na}_2\text{HPO}_4\text{]}^{\text{-}}$, 0.5 M $\text{[Fe(NO}_3\text{)}_3\text{]}^{\text{3+}}$, 0.5 M $\text{[KSCN]}^{\text{-}}$, 0.1 M $\text{[Fe(NO}_3\text{)}_3\text{]}^{\text{3+}}$, 0.5 M $\text{[NaCl]}^{\text{-}}$, 0.1 M $\text{[AgNO}_3\text{]}^{\text{-}}$, 0.5 M $\text{[NaNO}_3\text{]}^{\text{-}}$, 1 M $\text{[CuSO}_4\text{]}^{\text{-}}$, 6 M $\text{[HCl]}^{\text{-}}$, 6 M $\text{[HNO}_3\text{]}^{\text{-}}$, 6 M acetic acid, 6 M $\text{[NaOH]}^{\text{-}}$. Aluminum granules Equipment 8 small test tubes, glass stirring rod, small 10-mL graduated cylinder, 250-mL beaker, stand, ring clamp, wire gauze, small watch glass, dropper pipets, blue litmus paper, red litmus paper, wash bottle filled with deionized water, Bunsen burner, centrifuge Safety There are quite a few concentrated acids and bases in this experiment. In the diluted control solution it may be necessary to place the test tube in a hot water bath in order to observe the effervescence. Place the test tube in the hot water bath and use red litmus paper to test for the presence of $\text{[NO}_3^{\text{-}}\text{]}^{\text{-}}$ as described in the previous step. Add 1 mL of 0.5 M $\text{[NaCl]}^{\text{-}}$ (or your unknown) and 1 mL of 6 M $\text{[HNO}_3\text{]}^{\text{-}}$ to a small test tube. This will yield approximately 1 mL of a diluted solution. The $\text{[PO}_4^{\text{-}}\text{]}^{\text{3-}}$ can be removed by centrifuging the mixture and decanting the supernatant solution. Note that small blue spots produced on the red litmus paper are the result of spray from the basic solution in the test tube and do not necessarily indicate the presence of nitrate. A dark red solution indicates the presence of $\text{[SCN}^{\text{-}}\text{]}^{\text{-}}$. Centrifuge the mixture and decant 1 mL of the supernatant solution into another small test tube. To the supernatant solution in the test tube add 1 mL of 6 M $\text{[NaOH]}^{\text{-}}$ and a few granules of aluminum metal.

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