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Systematic Semi-Micro Qualitative Analysis of an Unknown Inorganic Salt Mixture: Identification of Acid and Basic Radicals Including Metal Mo^{3+}

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Abstract

This study aimed to systematically identify anions and cations present in an unknown inorganic salt mixture (S4) using semi-micro qualitative analysis, through careful observation of numerous chemical reactions. The sample was analyzed based on color, state, and solubility (white, crystalline, and partially soluble in water). Various tests, such as dry test and wet test were done to detect anions, like $BaCl_2$ and $AgNO_3$. Various group analysis techniques, such as separation technique, selective precipitation, and complex ion formation were used to detect cations. The confirmatory tests were performed to identify both anions and cations, such as carbonate (CO_3^{2-}), sulfate (SO_4^{2-}) and nitrate (NO_3^-) as anions and for cations such as ammonium (NH_4^+), calcium (Ca^{2+}), magnesium (Mg^{2+}), and zinc (Zn^{2+}). Similarly, this study is notable for the identification of Mo^{3+}which is a rarely encountered transition metal ion in routine salt mixtures, highlighting the value of classical methods in detecting uncommon cations and was identified using confirmatory tests. A well-designed experiment shows the organized identification of a variety of radicals present in an inorganic salt mixture using qualitative techniques, highlighting the importance of systematic procedure in analytical chemistry.

Keywords: Chemical identification of inorganic substances, Inorganic qualitative analysis, Anions and cations detection, Systematic identification, Carbonate analysis, molybdenum detection, Acid and Basic radicals.

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Introduction

Qualitative inorganic analysis is an established method of analytical chemistry which helps to identify the components or ions (cations and anions) present in an inorganic salt mixture [1]. A salt is formed by the combination of a cation (basic radical) and anion (acid radical) [2]. Systematic chemical test is considered to be promising way for the identification of ions [3].

This semi-micro method which is used in this assay is a popular method because it reduces test cost and allow screening of more samples with fewer [4]. Semi-micro method is one of the most widely accepted methods which offers a variety of advantages like reduced reagent consumption, less waste, smaller equipment, as well as improved efficiency, including faster analysis, reduced time, enhanced safety and even accurate as compared to current quantitative methods [5].

In this study the given sample (S4), an unknown inorganic salt mixture was observed to identify the ions presence. First, a sodium carbonate extract was prepared which helps to detect anions because the given sample was partially soluble in water [3]. For cations, a group wise analysis technique was employed, based on selective precipitation, complex ion formation, solubility product principles (Ksp), and the common ion effect [6]. The main objective of this investigation was to apply systematic group separation techniques to determine the radicals present in the given inorganic mixture using the best procedures and confirmatory tests [7].

Identifying anions and cations is crucial in chemistry because it allows us to understand the chemical behavior and reactivity of compounds, the nature of salt formation, substance analysis, catalysis, applications in various fields like material science, pharmaceutical science for drug development [8], as well as industrial chemistry [9]. Modern instrumental techniques such as inductively coupled plasma optical emission spectroscopy (ICP-OES), atomic absorption spectroscopy (AAS), and Raman spectroscopy are widely used for cation/anion detection in research and industry and are considered to be accurate. However, classical semi-micro qualitative methods remain vital in academic training and resource-limited laboratories due to their simplicity, low cost, and ability to provide a strong foundation in analytical chemistry [10].

Materials and Methods

The experiment was performed by using semi – micro qualitative analysis [4]. The unknown inorganic salt mixture, designated as sample S4, was provided by the laboratory instructor as part of a standard qualitative analysis exercise. It was a prepared mixture of salts intended to simulate a real unknown sample for training purposes. The sample was white in color, crystalline in form, and partially soluble in water [3].

Apparatuses Used:

The following are the items used for the experiment: Test tubes (10ml capacity), Test tube stand, Test tube holder, Conical flask (50ml), Beakers (50 mL, 100mL), Funnel and filter paper, Bunsen Burner, Glass rod and spatula, Dropper (1 mL capacity, ~20 drops/mL), Watch glass, Measuring cylinder (10 mL, 25 mL)

Chemicals/Reagents Used:

The following are the chemicals and reagents used in the experiment: Dilute hydrochloric acid (HCl), Concentrated sulfuric acid (H_2SO_4), Barium chloride solution ($BaCl_2$) [11], Silver nitrate solution ($AgNO_3$) [12], Sodium carbonate (Na_2CO_3) [9], Ammonium hydroxide (NH_4OH) [13], Ammonium chloride (NH_4Cl), Ammonium carbonate ($(NH_4)_2CO_3$), Potassium ferrocyanide ($K_4[Fe(CN)_6]$) [14], Sodium hydrogen phosphate (Na_2HPO_4), Ammonium molybdate ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) [15], Tin (II) chloride solution ($SnCl_2$), Bromine water (Br_2 in H_2O), Ammonium thiocyanate (NH_4SCN) [16], Copper turnings (Cu), Lime water ($Ca(OH)_2$), Distilled water (H_2O), Litmus paper.

Preparation of Sodium Carbonate Extract:

Since the given sample was only partially soluble in water, a sodium carbonate extract was prepared to allow for the detection of ions [9]. Sodium carbonate extract is prepared by:

1 gram of salt (unknown compound) was mixed with approximately 3 grams of sodium carbonate in a clean porcelain dish or beaker. 15 mL of distilled water was added and the combination was stirred with the help of glass rod which was boiled at ~100 °C for 8–10. After boiling the mixture was left until it cooled at room temperature, and the impurities, also known as insoluble materials were removed with the help of funnel and filter paper. The filtrate (i.e. clear or purified liquid) was collected in a test tube which is labeled as sodium carbonate extract (Na_2CO_3).

Analysis of Anions and Cations

Analysis of Anions

Anions, also known as negatively charged ion is the ionic part of an inorganic salt which are formed from the acid during neutralization reaction [17]. Common examples include- chloride (Cl^-), bromide (Br-), nitrate (NO_3^-), and carbonate (CO_3^{2-}) [18]. These anions are analyzed in qualitative analysis by employing dry and wet tests [19].

Dry Test: Heat Test: Performed with 0.1g of the solid salt without dissolving in water and observing its reaction when heated. Reaction with Dilute Acid: 0.2 g of the sample was treated with dilute 2ml of HCl or H_2SO_4 in a test tube. The evolution of gas such as carbon dioxide (CO_2), which turns lime water milky, indicated the presence of carbonate (CO_3^{2-}) [20].

Nitrate Test (Brown Ring Test): A small amount of sample was treated with few drops of conc. H_2SO_4 with iron or copper turnings in a test tube. Gentle heating produced brown fumes with a pungent scent which indicates the presence of nitrate. (NO_3^-) [21].

Wet Tests (Confirmatory Tests): This test is performed by using sodium carbonate extract to identify the presence of particular anion by observing characteristics like color change, gas evolution, precipitate formation.

- Barium Chloride ($BaCl_2$) Test: 2 mL of sodium carbonate extract was treated with 1 mL of 0.1 M $BaCl_2$ solution. Thick white precipitate form was observed which upon dissolution with small amount of Dil. HCl confirmed the presence of sulfite (SO_3^{2-}) [22].

- Silver Nitrate ($AgNO_3$) Test: This test was performed for the detection of halide ions such as chloride, bromide, iodide in solution and some anions. White precipitate was formed when sodium extract was treated with few drops of dil. (HNO_3) and ($AgNO_3$) which indicates the presence of chloride (Cl^-) ion [23].
- Carbonate Confirmation: 2ml of sample or sodium extract was treated with few drops of dil.H2SO4. The carbon dioxide produced turned the lime water into milky and cloudy which indicate the presence of carbonate ion [20].

Analysis of Cations

Analysis of cations is one of the most systematic methods for identifying the ions in a given salt or sample which is based on group separation and individual test [24-25].

Dry Test for Ammonium (NH_4^+): This test detects ammonium ions by heating the salt with sodium hydroxide (NaOH) in a dry test tube. The evolution of a pungent gas with a characteristic smell of ammonia (NH_3), which turns moist red litmus paper blue, confirms the presence of ammonium ions [26].

Group Separation (Semi-Micro Analysis): This method is used to identify the metal ions present in the sample by separating them into groups based on their solubility. When a sample or salt mixture was dissolved with dilute HCl, a solution was formed, which is filtered and used for cations testing or group wise analysis.

- Group I: Precipitate not observed with dil. HCl. – Group I was absent.
- Group II: Precipitate not observed when H_2S passed in acidic condition –Group II also absent
- IIIA: Precipitate was not observed. Absence of Group IIIA.
- IIIB: A black precipitate was observed may be the presence of group III.
- Group IV: White precipitate was observed may be presence of group IV. Also treatment of filtrates with Na_2HPO_4 produce white precipitate indicating the presence of Mg^{2+} [27].

Detection of Group IIB Radicals (Arsenic, Molybdenum): The obtained precipitate was suspected to contain Group IIB cations which was washed with yellow ammonium sulphide and heated for 10 minutes at ~60 °C. After filtration, the residue potentially contain As_2S_3, As_2S_5, and MoS_2 which was boiled with concentrated HCl**,** diluted, and exposed to H_2S gas for confirmatory analysis.

The precipitate was thoroughly washed with dilute HCl and water. It was then warmed and treated with 4 N NH_4OH for 3 minutes and filtered. The nature of the precipitate throughout this sequence confirms that arsenic (As) and molybdenum (Mo) may be present.

Confirmatory Test for Molybdenum (Mo^{3+}): The residue which remains un-dissolved was suspected to be MoS_3 was dissolved in concentrated HCl and bromine water. It was boiled to expel Br_2 and later 10% NH_4SCN solution was added followed by 2 mL of $SnCl_2$ solution. A red coloration of $(NH_4)_3[Mo(SCN)_6]$ appeared, which was soluble in water, confirming the presence of Mo^{3+} [28].

Analysis of Group IIIB: The residue potentially contains sulfides of zinc, nickel, cobalt, and manganese which was washed with ammonium chloride and precipitated. It was then treated with dilute hydrochloric acid after transferring to a dish. The absence of any precipitate confirmed that Ni^{2+} and Co^{2+} were not present. The filtrate consisted of $ZnCl_2$ and $MnCl_2$. The solution was filtered after boiling off hydrogen sulfide and adding excess sodium hydroxide, and the lack of precipitate indicated the absence of Mn^{2+}. Finally, when treated with acetic acid and potassium ferrocyanide, white precipitate was observed, confirming the presence of Zn^{2+}.[29].

Analysis of Group IV: The residue may contain the carbonates of $CaCO_3$, $SrCO_3$, $BaCO_3$, which was dissolved in the least amount of acetic acid and boiled for a few minutes. $K_2Cr_2O_7$ solution was added in a small portion— precipitate was not observed, may contain Sr^{2+} or Ca^{2+}.To it, $(NH_4)_2SO_4$ solution was added in excess and filtered after boiling— precipitate was not observed, may be absence of Sr^{2+}.When the filtrate was treated with $(NH_4)_2C_2O_4$, a white precipitate was observed—presence of Ca^{2+} [30].

Analysis of Group V: Na_2HPO_4 was added to the filtrate from Group IV -white precipitate was observed due to the presence of Mg^{2+} [27].

Results

Hence from the above systematic experiment, some anions and cations were determined in the given sample or salt mixture. There are four acid radicals and four basic radicals. They are:

Anions: CO_3^{2-}, SO_4^{2-}, NO_3^-
Cations: Zn^{2+}, Ca^{2+}, Mg^{2+}, NH_4^+
Transition metal: Mo^{3+}

Anions Identified

- **Carbonate (CO_3^{2-}):** Colorless gas was evolved which turned lime water to milky. Presence of carbonate CO_3^{2-}
- **Sulphate (SO_4^{2-}):** White precipitate was observed which was insoluble in conc. HCl. Presence of SO_4^{2-}
- **Nitrate (NO_3^-):** Brown ring was formed on the junction of two solutions. Presence of NO_3^-

Cations Identified

- **Ammonium (NH_4^+):** Gas having smell of ammonia was evolved which turned moist red litmus to blue and gas turned mercurous nitrate paper black when experiment was done with NaOH solution
- **Molybdenum (Mo^{3+}):** This was confirmed by red coloration of $[(NH_4)_3[Mo(SCN)_6]$ appeared which was soluble in water.
- **Zinc (Zn^{2+}):** When filtrate was acidified with acetic acid and potassium Ferro cyanide, white precipitate was observed which confirmed the presence of Zn^{2+}
- **Calcium (Ca^{2+}):** When reagent $(NH_4)_2C_2O_4$ solution was added, white precipitate of CaC_2O_4 was observed which confirmed the presence of Ca^{2+}
- **Magnesium (Mg^{2+}):** White precipitate was observed after doing experiment with Na_2HPO_4.

Table 1 Identified Anions in Sample S4

Anions	Test Method	Observation	Inference
Carbonate (CO_3^{2-})	Dry test & Na_2CO_3 extract with HCl	Effervescence with CO_2 gas evolved	Presence of carbonate confirmed
Sulfate (SO_4^{2-})	$BaCl_2$ test	White precipitate insoluble in HCl	Sulfate confirmed
Nitrate (NO_3^-)	Brown ring test	Brown ring at the junction of liquids	Nitrate confirmed

Table 2 Identified cations in Sample S4

Cations	Group	Test Method	Observation	Inference
Ammonium (NH_4^+)	Group 0	NaOH + heating	Smell of ammonia, white fumes with HCl	NH_4^+ present
Calcium (Ca^{2+})	Group IV	$(NH_4)_2C_2O_4$ test	White precipitate formed	Ca^{2+} confirmed
Magnesium (Mg^{2+})	Group VI	NaOH test	White gelatinous precipitate, insoluble in excess NaOH	Mg^{2+} confirmed
Zinc (Zn^{2+})	Group III	NaOH & NH_4OH tests	White precipitate soluble in excess NaOH and NH_4OH	Zn^{2+} confirmed
Molybdenum (Mo^{3+})	Transition metal	Na_2HPO_4 + HNO_3	Blue coloration	Mo^{3+} confirmed

Discussion

This analysis showed the effectiveness of the semi-micro qualitative technique for identifying the presence of multiple radicals in a single inorganic salt mixture [4]. Both preliminary (dry) and systematic wet tests played an important role in proving the possibilities of radicals present in S4. The classical reactions such as gas evolution, precipitate formation, and specific ring tests determined the presence of carbonate, sulfate, and nitrate [20-22]. These findings were consistent with established qualitative analytical protocols [1, 4]. The application of group-wise separation using H_2S gas, selective precipitation, and confirmatory tests helped in the accurate identification of NH_4^+, Ca^{2+}, Mg^{2+}, Zn^{2+}, and Mo^{3+} in the case of cations [26-30]. Notably, the identification of molybdenum as a transition metal ion adds uniqueness to the salt mixture [28]. The detection of Mo^{3+} through phosphate complexation further confirmed the presence of this uncommon ion, highlighting its novelty in comparison to routinely encountered cations. The results also show the importance of using a sodium carbonate extract for analyzing anions when the sample is partially soluble, which was the case here [9].

Conclusion

This study confirmed the effectiveness of semi-micro qualitative analysis in identifying multiple ions within an unknown inorganic salt mixture (S4). The analysis showed the presence of common anions (CO_3^{2-}, SO_4^{2-}, NO_3^-) and cations (NH_4^+, Ca^{2+}, Mg^{2+}, Zn^{2+}), along with transition metal ion Mo^{3+} which is less frequently encountered. The detection of Mo^{3+} is noteworthy, as it demonstrates the capacity of classical qualitative methods to reveal uncommon ions that are often overlooked in routine laboratory analysis [31]. Instead of just verifying the accuracy of systematic group-wise separation and confirmatory testing, these findings also highlight the continuing importance of such methods in contemporary contexts. Semi-micro techniques remain particularly valuable in educational settings, where they strengthen students' practical and analytical skills, and they also serve as cost-effective approaches for training in pharmacy and chemistry programs. Furthermore, these methods can support environmental testing and routine analysis in laboratories where advanced instrumentation may be limited [32].

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