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Practical Methods for Separating and Detecting Permanganate, Nitrite and Hypochlorite Ions in Analytical Chemistry Education

Métodos Práticos para Separação e Detecção dos Íons Permanganato, Nitrito e Hipoclorito no Ensino de Química Analítica

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The qualitative analysis of cations and anions constitutes a fundamental topic in analytical chemistry, as undergraduate chemistry students must acquire the ability to identify and separate distinct chemical species. For cations, analytical procedures rely on their classification into groups based on shared precipitation reagents, simplifying the identification of individual ions. Anion analysis presents methodological challenges, particularly in mixtures where chemical similarities or incompatibilities between species hinder their identification. This study aimed for a didactic methodology for the separation and identification of the anions permanganate (MnO_4^-), nitrite (NO_2^-), and hypochlorite (ClO^-), intended for application in analytical chemistry laboratory courses. The ClO^- ion can be separated from the mixture via precipitation with lead nitrate. The resulting precipitate was characterized by heating in a water bath, yielding a dark brown insoluble salt consistent with lead oxide. The NO_2^- ion identification was achieved through a displacement reaction by adding silver nitrate to the supernatant, forming a white solid identified as silver nitrite ($AgNO_2$). The MnO_4^- ion was detected via a redox reaction with hydrogen peroxide, evidenced by solution decolorization due to electron transfer between species. The developed methodology proved effective and suitable for separating and identifying permanganate, nitrite, and hypochlorite anions in qualitative analytical chemistry.

Keywords: Systematic separation; anions; qualitative analysis; separation techniques.

1. Introduction

The origins of chemical analysis trace back approximately 2.000 years, when the Roman naturalist Gaius Plinius Secundus conducted the first wet chemical test. From this period through the Middle Ages and into the Modern Age, analytical procedures underwent refinement and were widely adopted by the scientific community.[1] As a result, numerous advancements were seen in analytical chemistry and such innovations played a pivotal role in the development of methodologies that enable environmental monitoring, ensuring quality control in industrial processes, safeguarding food safety protocols and validating pharmaceutical compounds with precision.[2,3]

Building on Bergman's legacy with the detailed analytical procedure for water analysis, subsequent contributions by german chemists Heinrich Rose (1829) and Carl Remigius Fresenius (1841) refined qualitative chemical analysis into the structured discipline recognized today. In his seminal text, Rose established a systematic framework for qualitative analysis, while Fresenius categorized the most prevalent elements of his era into analytical groups defined by the solubility of their sulfides.[4] The cation classification system and analytical protocols they formerly devised for cation identification remain fundamentally unchanged in modern instructional frameworks, underscoring their enduring relevance to analytical chemistry education to the present day.[5,6]

In analytical chemistry, cations are organized into five distinct groups according to their shared chemical behavior. With the exception of group V, all remaining groups undergo precipitation reactions with a specific precipitating agent, resulting in insoluble compounds. This precipitation step allows the isolation of the cations, allowing their identification.[7] To characterize the ions, it is taken into observation the physical or chemical changes that occur during reactions, such as shifts in color, the release of gases, or the formation of solids, alongside sensory cues like odor. These methods remain foundational to qualitative analysis.[8,9]

Given the inherent challenges in categorizing anions and separating/characterizing anionic mixtures, coupled

with the pedagogical need for students to visualize and grasp theoretical principles of qualitative analytical chemistry, Souza *et al.* devised a practical methodology for separating and identifying chloride (Cl^-), bromide (Br^-), and iodide (I^-) anions in laboratory settings using silver (Ag^+) as a precipitating agent.[5] Expanding on this approach, Anconi *et al.* introduced a novel protocol for isolating and characterizing carbonate (CO_3^{2-}), phosphate (PO_4^{3-}), and chromate (CrO_4^{2-}) anions via selective precipitation with Ba^{2+} ions.[10]

To elucidate and discuss fundamental concepts in chemical equilibria, including precipitation dynamics, acid-base interactions, liquid-liquid extraction, solubility behavior, solute phase distribution and species distribution diagrams, this study introduces a novel methodology for the separation and identification of nitrite (NO_2^-), permanganate (MnO_4^-) and hypochlorite (ClO^-) anions. The approach integrates principles of selective precipitation and redox reactivity to address challenges in distinguishing these anions within complex mixtures.[11]

Nitrite is a widely employed substance as an additive in processed foods such as meats, vegetables, herbs and dairy products.[12] However, its use raises significant health concerns, as nitrite can react with amines to form nitrosamines, compounds classified as potential carcinogens due to their ability of inducing malignant cell proliferation.[13]

The hypochlorite anion (ClO^-) is widely employed in domestic, industrial, and medical settings, primarily for surface disinfection, sterilization procedures and sanitation.[14,15] However, improper application of hypochlorite-containing products poses significant risks to human health and ecosystems.[16] Chronic exposure has been linked to arthritis, neurodegenerative disorders, and cardiac pathologies due to its reactive oxygen species generation and cellular toxicity.[17] Similarly, the permanganate ion (MnO_4^-) serves as a potent oxidizing agent in wastewater treatment systems.[18] Its application mitigates eutrophication processes by limiting nutrient availability, thereby suppressing exponential algal proliferation in potable water reservoirs and industrial effluents, but its accumulation can disrupt natural processes and negatively affect microorganisms and plant life, thereby impacting ecological balance.[19]

Therefore, given the widespread utilization of nitrite, hypochlorite, and permanganate anions across industrial and domestic sectors, this study proposes the development of a novel simultaneous methodology for identifying these ions within the framework of Qualitative Analytical Chemistry. The proposed method offers a simple, low-cost alternative that can be explored in analytical chemistry courses, with the objective of reinforcing multidisciplinary concepts in practical laboratory instruction and thereby enhancing pedagogical effectiveness by encouraging student-driven correlations between theoretical chemical principles and experimental observations.

2. Experimental

2.1. Materials and reagents

The experiments were conducted using the following reagents: potassium permanganate ($KMnO_4$, ≥99.0%), potassium nitrite (KNO_2, ≥96.0%), sodium hypochlorite (NaClO, 4.00–4.99%), lead nitrate ($Pb(NO_3)_2$), ≥99.0%), silver nitrate ($AgNO_3$, ≥99.0%), sulfuric acid (H_2SO_4, 95.0–98.0%), and hydrogen peroxide (H_2O_2, 30% v/v). pH measurements were conducted using universal indicator paper (J-Prolab, pH 1–14).

2.2. Instrumentation

Liquid-solid phase separation was achieved using a Quimis® Q-222™ centrifuge. Precipitate heating was performed in a water bath (Fisatom® Model 005502), with temperature monitoring via a high-precision decimal thermometer (Incoterm® Model 5096).

2.3. Methods

The separation and identification of NO_2^-, MnO_4^-, and ClO^- were conducted using ion-specific reactions. In a test tube, 10 drops of 0.2 mol L^{-1} $KMnO_4$, 0.5 mol L^{-1} KNO_2, and 0.5 mol L^{-1} NaClO solutions were combined. The pH of the reaction medium was measured using universal indicator paper. Subsequently, 20 drops of 0.5 mol L^{-1} $Pb(NO_3)_2$ solution were introduced, resulting in the formation of a light brown precipitate. The test tube was centrifuged at 3000 rpm for 60 seconds. Following centrifugation, the supernatant was transferred to a separate test tube. The precipitate-containing tube was heated in a water bath at 90 °C for 10 minutes. Post-heating, a distinct color transition from light brown to dark brown was observed in the precipitate, confirming thermal stability changes.

To the test tube containing the supernatant, 20 drops of 0.5 mol L^{-1} silver nitrate ($AgNO_3$) solution were introduced, yielding a white precipitate. The mixture was subsequently centrifuged at 3000 rpm for 60 seconds. The clarified supernatant was transferred to a third test tube, where 10 drops of 0.05 mol L^{-1} H_2SO_4 and 4 drops of 30% H_2O_2 were added, resulting in a colorless solution. All waste generated during the separation of nitrite (NO_2^-), hypochlorite (ClO^-), and permanganate (MnO_4^-) anions, was collected in a designated and labeled container for subsequent treatment, in accordance with institutional safety and waste management guidelines. For guidance on the

handling and proper treatment of hazardous waste, readers may refer to specialized references.[20-22]

2.4. Experimental conditions

For the execution of the experiments the following solutions were prepared: 0.2 mol L^{-1} $KMnO_4$; 0.5 mol L^{-1} KNO_2; 0.5 mol L^{-1} NaClO; 0.5 mol L^{-1} $Pb(NO_3)_2$; 0.5 mol L^{-1} $AgNO_3$; 0.05 mol L^{-1} H_2SO_4; 30% (v/v) H_2O_2. Also, the pH was measured (using universal indicator paper) after the addition of each anion-containing solution to the test tube.

2.5. Data collection and analysis

Regarding the qualitative approach of the present study, each step of the analytical method was visually analyzed and validated against reference literature for classification of the obtained species.

2.6. Manuscript translation from original language

Portions of the text originally drafted in Portuguese (PT-BR) were translated into English using DeepSeek (DeepSeek-R1-Lite-Preview, DeepSeek Inc., 2024), an artificial intelligence (AI) based tool. The authors rigorously reviewed and refined the translated content to ensure alignment with the original meaning. The use of AI was restricted to the translation, and no alteration nor enhancement of the collected data was performed whatsoever.

3. Results and Discussion

A common precipitating agent could not be employed for the group due to their divergent chemical reactivity. The distinct electron transfer kinetics and coordination preferences of these species require tailored analytical protocols to account for their unique redox and solubility behaviors.[8]

The reaction medium containing the permanganate (MnO_4^-), nitrite (NO_2^-), and hypochlorite (ClO^-) anion solutions exhibited a pH of 10. This alkalinity stems from the presence of NO_2^- and ClO^-, which function as conjugate bases of the weak acids nitrous (HNO_2) and hypochlorous (HClO), respectively. In contrast, MnO_4^- derives from a neutral inorganic salt and thus does not contribute to the solution's basicity. The elevated pH also stabilizes the oxidation state of manganese (VII) (Mn^{7+}) in permanganate, as even mildly acidic conditions trigger its reduction by nitrite to manganese (II) ions (Mn^{2+}), accompanied by electron transfer and solution decolorization.[19,23]

Figure 1 presents the species distribution diagram obtained with CurTiPot.[24]

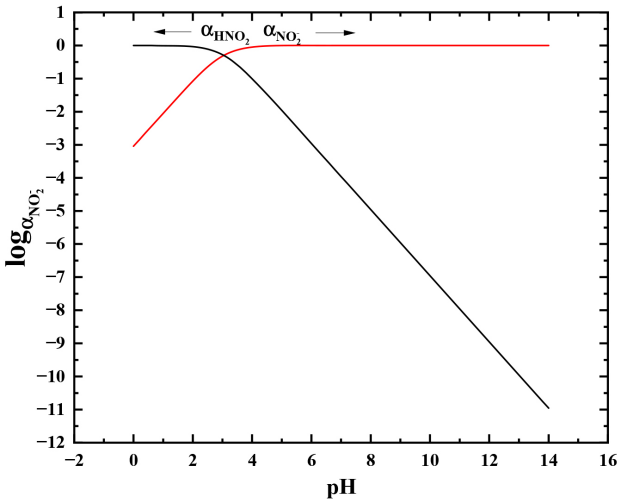

Figure 1. Distribution diagram of NO_2^- species in aqueous solutions, relating the concentration of each component in the system to the pH

Nitrite's distribution equilibrium is directly influenced by the acid-base equilibrium between nitrous acid and nitrite as shown in Reaction 1:

$$HNO_2\,(aq) \rightleftharpoons H^+\,(aq) + NO_2^-\,(aq) \quad \text{(Reaction 1)}$$

Nitrous acid (HNO_2) has a pK_a of 3.3. Consequently, at pH values below 3.3, the protonated form (HNO_2) predominates and can be identified as the primary species. At pH 3.3, equilibrium is established between the protonated (HNO_2) and deprotonated (NO_2^-) forms, with both species present in comparable concentrations. For pH values exceeding 3, the conjugate base nitrite (NO_2^-) becomes the dominant species in solution, reflecting the pH-dependent speciation typical of weak acid-base systems.[7]

Similarly, the pH-dependent distribution equilibrium of the hypochlorite anion (ClO^-) is governed by the acid-base equilibrium between hypochlorous acid (HClO) and hypochlorite, as described by Reaction 2. This pH-dependent speciation behavior dictates the relative concentrations of protonated and deprotonated forms, with implications for redox activity and disinfection efficacy in aqueous systems.

$$HClO\,(aq) \rightleftharpoons H^+\,(aq) + ClO^-\,(aq) \quad \text{(Reaction 2)}$$

Hypochlorous acid (HClO) exhibits a pK_a of 7.5. Consequently, at pH values below 7.5, the protonated form (HClO) predominates in solution. At pH 7.5, the acid-base equilibrium results in equal concentrations of hypochlorous acid (HClO) and its conjugate base, hypochlorite (ClO^-). When the pH exceeds 7.5, the deprotonated hypochlorite anion becomes the dominant species, reflecting the pH-dependent speciation inherent to weak acid-conjugate base systems.[25] These observations can be obtained from Figure 2.

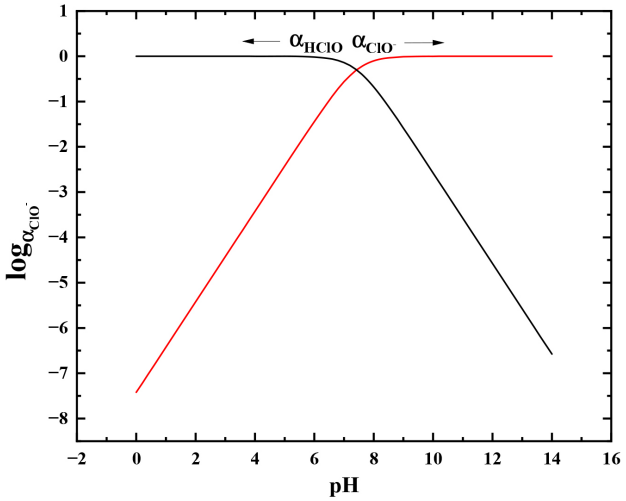

Figure 2. Distribution diagram of ClO^- species in aqueous solutions, relating the concentration of each component to the pH, there are two lines, the black line and the red line, the former representing the concentration of HClO and the latter the concentration of hypochlorite

A 0.5 mol L^{-1} $Pb(NO_3)_2$ solution was initially employed to precipitate and characterize the ClO^- ion, as can be seen on Figure S1. As the permanganate ion exhibits an intense purple color in aqueous solution, its presence dominates the visual appearance of the system when the anions are added simultaneously, resulting in a predominantly purple/violet coloration. Owing to hypochlorite's rapid decomposition into chlorine gas and water, the working solution was freshly prepared immediately prior to analysis.[26] The addition of $Pb(NO_3)_2$ to the solution induced the formation of a light brown precipitate consistent with lead (II) hypochlorite ($Pb(ClO)_2$), as outlined in Reaction 3.

$$ClO^-(aq) + Pb^{2+}(aq) \rightarrow Pb(ClO)(s) \quad \text{(Reaction 3)}$$

Upon heating, chloride ions are solubilized and the lead (II) hypochlorite precipitate is converted into lead dioxide, a dark brown precipitate (Reaction 4).

$$Pb(ClO)(s) + H_2O(l) \rightarrow PbO_2(s) + 2H^+(aq) + Cl^-(aq) \quad \text{(Reaction 4)}$$

Thus, the ClO^- ions were characterized by the formation of an insoluble brown precipitate in an aqueous solution of lead (II) dioxide (Reaction 5).

$$2ClO^-(aq) + Pb^{2+}(aq) \rightarrow PbO_2(s) + 2Cl^-(aq) \quad \text{(Reaction 5)}$$

Lead dioxide is extensively utilized in battery manufacturing and electrochemical processes due to its high oxidative potential.[27] This property arises from the compound's elevated reduction potential, wherein lead ions in the +4 oxidation state (Pb^{4+}) readily accept electrons from other substances, reducing their oxidation state to +2 (Pb^{2+}) as illustrated in Half-reaction 1.

$$PbO_2(s) + 4H^+(aq) + 2e^- \rightleftharpoons Pb^{2+}(aq) + 2H_2O(l)$$
$$E^0 = +1.455\ (V) \quad \text{(Half-reaction 1)}$$

The separation and identification of the nitrite ion was achieved by adding 0.5 mol L^{-1} $AgNO_3$ solution to the supernatant. The illustrated reaction can be consulted in Figure S2. Silver ions in the reagent reacted with NO_2^- ions, producing a white crystalline precipitate of silver nitrite ($AgNO_2$), consistent with the stoichiometry outlined in Reaction 6.

$$Ag^+(aq) + NO_2^-(aq) \rightarrow AgNO_2(s) \quad K_{ps} = 7.2 \times 10^{-4} \text{ at } 25\ °C \quad \text{(Reaction 6)}$$

Silver nitrite ($AgNO_2$) exhibits a solubility of 0.415 g per 100 mL of water at 25 °C, significantly lower than that of silver nitrate ($AgNO_3$), which dissolves at 213 g per 100 mL under identical conditions. The addition of a concentrated $AgNO_3$ solution triggers a single-displacement reaction, wherein nitrate ions (NO_3^-) are substituted by nitrite ions (NO_2^-). This exchange selectively precipitates silver nitrite ($AgNO_2$), the only water-insoluble species within the nitrite group, due to its markedly reduced solubility compared to other nitrites.[7] Unlike other systems involving silver salts, $AgNO_2$ does not form stable soluble complexes with nitrite ions, even when an excess of reagent is added.[7]

The final purple supernatant was used to identify MnO_4^- ions. To this end, 10 drops of 0.05 mol L^{-1} sulfuric acid (H_2SO_4) and 4 drops of 30% (v/v) H_2O_2 were added to the test tube. Higher volumes of sulfuric acid, or a solution at higher concentration than 0.05 mol L^{-1} can oxidize the manganese ion into a precipitate of MnO_2.[7] The identification of MnO_4^- relied on a redox reaction with hydrogen peroxide, characterized by rapid solution decolorization due to the reduction of Mn (VII) to Mn (II). The spectrum of manganese-containing species generated during this process can be consulted in Figure 3.

In the presence of dilute sulfuric acid (H_2SO_4), hydrogen peroxide (H_2O_2) decolorized the MnO_4^- solution while releasing molecular oxygen (O_2), as detailed in Reaction 7. This decolorization occurs because the violet-colored permanganate ion (MnO_4^-) undergoes reduction to the colorless manganese (II) ion (Mn^{2+}) during its redox reaction with hydrogen peroxide. The reaction is illustrated in Figure S3. The electron transfer from H_2O_2 to Mn (VII) drives this transformation, accompanied by the oxidation of peroxide to oxygen gas.[7]

$$2MnO_4^-(aq) + 5H_2O_2(aq) + 6H^+(aq) \rightarrow 5O_2(g) + 2Mn^{2+}(aq) + 8H_2O(l) \quad \text{(Reaction 7)}$$

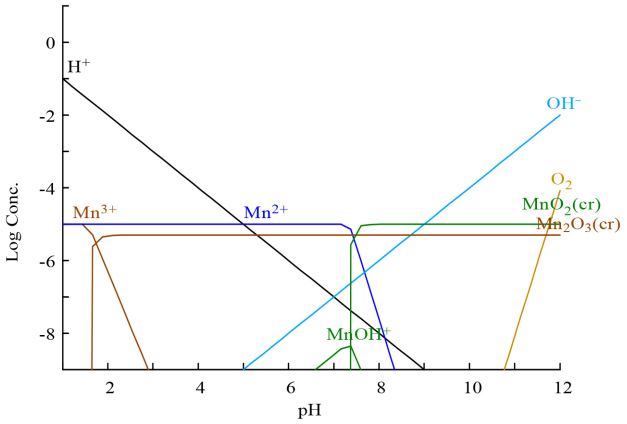

Figure 3. Sillén diagram for the distribution of species involving manganese, highlighting the dependence of the chemical equilibria in solution, with the pH

The reaction proceeds as a result of the high reduction potential exhibited by MnO_4^- in acidic medium (Table I), which drives the oxidation of hydrogen peroxide *via* a five-electron transfer mechanism.

Table 1. Half-reactions and reduction potentials[a] involving MnO_4^- and hydrogen peroxide at 25 °C

Half-reactions	E^0 (V)
$MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$	+ 1.51
$H_2O_2(aq) + 2H^+(aq) + 2e^- \rightarrow 2H_2O(l)$	+1.17

[a]Source: Skoog *et al.*[25]

The final solution was discarded in a designated, labeled waste container. Figure 4 outlines the analytical workflow employed for the separation and identification of permanganate (MnO_4^-), nitrite (NO_2^-), and hypochlorite (ClO^-) anions, including detailed procedures and reagents utilized throughout every step of the study.

4. Conclusion

Reagents containing permanganate (MnO_4^-), nitrite (NO_2^-), and hypochlorite (ClO^-) anions are widely used for product development, domestic use or water treatment. The development of qualitative methodologies still remains important to the present date, hence the present study proposes a methodology that allows the separation and identification of permanganate, nitrite and hypochlorite through an easy and simple procedure. By leveraging precipitation reactions, equilibrium displacement and oxidation processes, the chemical properties of these anions were systematically explored. The implementation of the procedure is straightforward and the results are readily observable, rendering the experiment both educational and engaging for students. Furthermore, the reliance on common, low-cost reagents ensures the methodology's practicality for use in instructional laboratory settings.

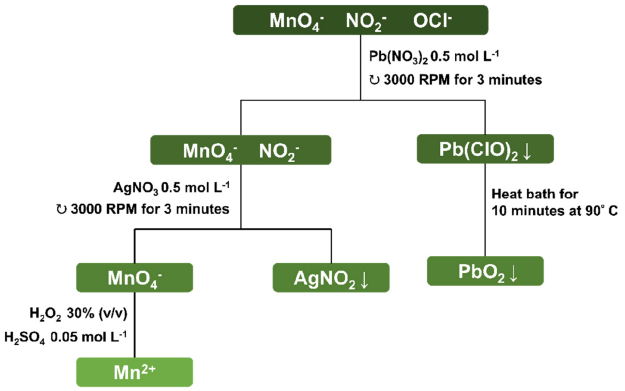

Figure 4. Separation flowchart for MnO_4^-, NO_2^- and ClO^- anions, designed to establish a simple methodology and summarize each step of the analytical procedure

Supplementary Information

Supplementary information is available free of charge at https://rvq.sbq.org.br/pdf/AR2026-4997-MS.

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Author Contributions

Ms. Giovanna Victoria Vieira: Conceptualization (Lead), Investigation (Lead), Formal analysis (Lead), Methodology (Lead), Writing - original draft (Lead), Writing - review & editing (Lead); Mr. Pedro Augusto Santos: Investigation (Lead), Methodology (Lead), Supervision (Lead), Writing - original draft (Lead), Writing - review & editing (Lead); Ms. Laura Paula: Formal analysis (Supporting), Methodology (Supporting), Writing - original draft (Supporting); Dr. Adelir Saczk (*Corresponding Author*): Conceptualization (Lead), Project administration (Lead), Resources (Lead), Supervision (Lead), Writing - review & editing (Lead).

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