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ANALYSIS OF A MIXTURE OF HYDROXIDE CATIONS INSOLUBLE IN NAOH AND KOH SOLUTIONS (FIFTH ANALYTICAL GROUP OF CATIONS)

Damen NURGALIEVA*, Ainur KENZHEBAEVA[1], Akzhan KABYLKANOVA[2], Amadey MOISSEYENKO[3], Akulpa SATYBALDY[4]

* *Astana International University, Kazakhstan*
[1] *Astana International University, Kazakhstan*
[2] *Astana International University, Kazakhstan*
[3] *Astana International University, Kazakhstan*
[4] *Astana International University, Kazakhstan*

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ABSTRACT

The article provides a detailed review of methods for the qualitative analysis of cations of the 5th analytical group, including iron (II) (Fe^{2+}), iron (III) (Fe^{3+}), magnesium (Mg^{+}) and manganese (Mn^{2+}). Their individual chemical properties, behaviour in various reaction environments and the importance of specific analytical techniques for their accurate identification are analysed. The authors paid considerable attention to fractional analysis, including the step-by-step separation and identification of cations in complex mixtures. Approaches to sequential analysis with minimisation of cross-reactions are described, which ensures the reliability of the results. Recommendations are given for the practical implementation of analytical procedures in laboratory conditions, and the main errors and their prevention are indicated. The aim of the study is to ensure high accuracy and reproducibility of the analysis of group 5 cations.

1. Introduction

Group reagents for cations of the fifth analytical group are alkalis (e.g., NaOH or KOH) and ammonium hydroxide (NH_4OH). When interacting with these reagents, cations form metal hydroxides that do not dissolve in excess alkali. These hydroxides precipitate as solids, allowing the corresponding metals to be isolated. The cations of the fifth group include magnesium (Mg^{2+}), iron (II) (Fe^{2+}), iron (III) (Fe^{3+}) and other elements, whose precipitation from solution serves as the basis for their further separation and identification.

According to N. Ya. Loginov's description [1, pp. 16–17], two types of analysis are used in analytical chemistry: systematic and fractional. The systematic course of analysis is largely associated with the precipitation of cations by a group reagent and the subsequent step-by-step detection of cations. It involves dividing the mixture into groups and subgroups using group reagents, and then detecting individual ions within these groups using specific reactions. This process allows for a complete analysis of the sample, detecting each ion after all interfering ions have been removed, but this type of work requires a huge amount of time and effort, which is not always possible. Therefore, we will consider the option of fractional analysis of the mixture of the fifth analytical group of cations.

As already mentioned, fractional analysis differs from systematic analysis in the speed of reactions and the detection of ions regardless of the presence of others.

2. Practical part

Fractional analysis of the mixture of the fifth analytical group of cations

Fractional analysis of ions is carried out with separate portions of the solution or dry sample in the presence of all sample components. For fractional analysis, characteristic or specific reactions are used that are unique to a given ion or substance.

Reagents: potassium hexacyanoferrate (III) ($K_3[Fe(CN)_6]$, potassium hexacyanoferrate (II) ($K_4[Fe(CN)_6]$, distilled water (dH_2 O), hydrochloric acid (HCl), sodium hydrophosphate ($NaHPO_4$), ammonium hydroxide (NH_4OH), ammonium chloride (NH_4Cl), nitric acid (HNO_3), lead minium (Pb_3O_4) .

Chemical glassware and equipment: test tube rack, conical flat-bottom flask, spirit burner, light microscope, pipette, microscope slide, test tube holder, porcelain dish, water bath.

Procedure:

Discovery of the Fe^{2+}cation.

Experiment conditions:

1) The reaction must be carried out at pH = 12-13.

2) The presence of ammonium salts prevents the formation of a precipitate.

3) Oxygen in the air causes colour changes due to partial oxidation of Fe^{2+}ions.

When opening the cation, several steps are performed. First, we took 3 drops of the control task and 3 drops of distilled water, 2 drops of potassium hexacyanoferrate (III) - $K_3[Fe (CN)_6]$ into a test tube. The presence of Fe^{2+}cations in the solution of group 5 cations we were studying was indicated by the formation of a blue precipitate, shown in Figure 1.

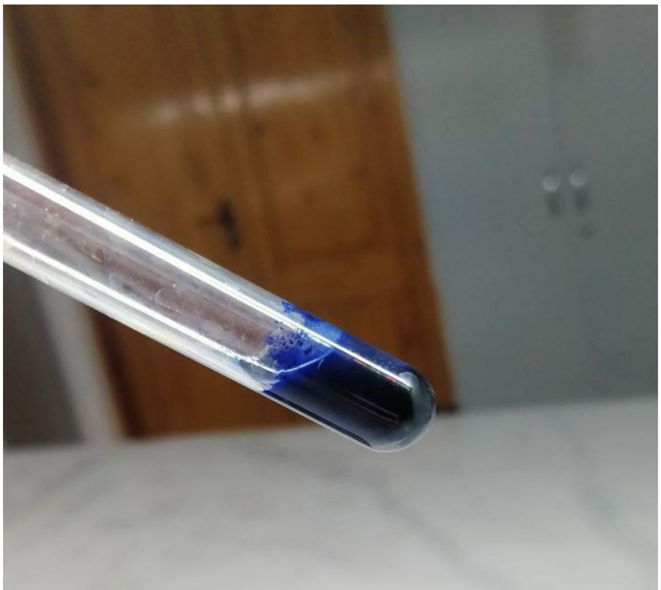

Figure 1 - Turnbull's blue

$3FeSO_4 + 2K_3[Fe(CN)_6] \rightarrow Fe_3[Fe(CN)_6]_2 + 3K_2SO_4$.

$3Fe^{2+} + 3SO_4^{2-} + 6K^+ + 2[Fe(CN)_6]^{3-} \rightarrow Fe_3[Fe(CN)_6]_2 + 6K^+ + 3SO_4^{2-}$

$3Fe^{2+} + 2[Fe(CN)_6]^{3-} \rightarrow Fe_3[Fe(CN)_6]_2$

Discovery of the Fe^{3+}cation.

Experiment conditions:

1) The reaction is carried out at $pH < 3$.

2) Free alkalis decompose Prussian blue.

3) Iron (II) and other cations do not interfere with the discovery of Fe^{3+}cations.

4) An excess of $K_4[Fe(CN)_6]$ is undesirable, as it can cause the formation of a soluble form of Prussian blue.

In one test tube, we took 3 drops of k/z, 3 drops of distilled water, 2 drops of 2n hydrochloric acid solution - HCI, and added 2 drops of potassium hexacyanoferrate (II) solution - $K_4[Fe(CN)_6]$. The presence of Fe^{3+}cations in the test solution was indicated by the appearance of a blue colour - Prussian blue, shown in Figure 2. To distinguish Turnbull's blue from Prussian blue, simply hold the test tubes up to the light. Then the colour in the test tube with the Fe^{2+}cation will be dark blue, and in the test tube with the Fe^{3+}cation the colour will be a lighter shade of blue, and sometimes even greener. It is also possible to distinguish between them by the physical properties of the solution. In our opinion, the solution of Turnbull's blue is denser than that of Prussian blue, which is confirmed by the research of Glushchenko V.A. [2, p. 8].

Figure 2 - Prussian blue

$2Fe_2(SO_4)_3 + 3K_4[Fe(CN)_6] \rightarrow Fe_4[Fe(CN)_6]_3 + 6K_2SO_4$.

$4Fe^{2+} + 6SO_4^{2-} + 12K^+ + 3[Fe(CN)_6]^{3-} \rightarrow Fe_4[Fe(CN)_6]_3 + 12K^+ + 6SO_4^{2-}$

$4Fe^{2+} + 3[Fe(CN)_6]^{3-} \rightarrow Fe_4[Fe(CN)_6]_3$

Discovery of the Mg^{2+}cation.

Experimental conditions:

1) The reaction is carried out in an ammoniacal environment at pH=8.

2) An excess of NH_4Cl prevents the precipitation of $MgNH_4PO_4$, so an excess of hydrochloric acid should not be used.

3) Cations of all analytical groups, except for the first one, may interfere with the reaction.

We took a microscope slide and placed one drop of k/z on it. Next, we placed two NH_4Cl crystals and one drop of Na_2HPO_4solution on this drop. Then, we held the microscope slide over ammonia vapours, which had been poured into a porcelain cup beforehand, for about one minute. After that, the microscope slide was held over the burner flame until it was completely dry. We examined the resulting crystals under a microscope and observed the characteristic shape shown in Figure 3.

$MgSO_4 + Na_2HPO_4 + NH_4OH \rightarrow MgNH_4PO_4 + Na_2SO_4 + H_2O$

$Mg^{2+} + SO_4^{2-} + 2Na^+ + HPO_4^{2-} + NH_4OH \rightarrow MgNH_4PO_4 + 2Na^+ + SO_4^{2-} + H_2O$

$Mg^{2+} + HPO_4^{2-} + NH_4OH \rightarrow MgNH_4PO_4 + H_2O$

Figure 3 - $MgNH_4PO_4$crystals

Discovery of the Mn^{2+}cation.

We took 3 drops of c/t, 6 drops of distilled water and 5 drops of concentrated nitric acid - $HNO_{3(conc.)}$ into a test tube and added a small amount of lead minium - Pb_3O_4. Then we heated the test tube in a water bath until it boiled. In the presence of Mn^{2+}cations in the solution manganic acid is formed, which decomposes when heated or settled, forming MnO_4^- as indicated by the appearance of a crimson colour in the solution we obtained (Figure 4).

Figure 4 - Manganic acid ($HMnO_4$)

$2Mn(NO_3)_2 + 5PbO + 6HNO_3 \rightarrow 2HMnO_4 + 5Pb(NO_3)_2 + 2H_2O$

$Mn^{2+} + 6H^{+} + 5PbO + 6NO_3^{-} \rightarrow 2MnO_4^{-} + 5Pb^{2+} + 10NO_3^{-} + 2H_2O$

$Mn^{2+} + 6H^{+} + 5PbO \rightarrow 2MnO_4^{-} + 5Pb^{2+} + 2H_2O$

For more details on all qualitative reactions to fifth group cations, see the table below

Table. Qualitative reactions to fifth group cations

Cation	Reagent	Analytical effect
Mg^{2+}	Sodium hydrogen phosphate Na_2HPO_4 in the presence of NH_3 and NH_4Cl	White crystalline precipitate $MgNH_4PO_4$
	8-hydroxyquinoline $C_9H_6N(OH)$ in NH_3 (aq)	Yellow-green crystalline precipitate of magnesium oxyquinolinate
Mn^{2+}	Oxalic acid $H_2C_2O_4{\cdot}2H_2O$ in an alkaline environment	Pink solution of $Na_3[Mn(C_2O_4)_3]$
	Bismuthate sodium $NaBiO_3$ in HNO_3 (diluted)	Raspberry-purple solution of $HMnO_4$
Fe^{2+}	Potassium hexacyanoferrate (III) $K_3[Fe(CN)_6]$	Formation of a dark blue precipitate ("Turnbull's blue")
	Dimethylglyoxime $C_4H_6N_2(OH)_2$ in NH_3 (aqueous) medium	Alo-red solution $[Fe(C_4H_6N_2O_2)_2]{\cdot}xNH_3$
Fe^{3+}	Potassium hexacyanoferrate (II) $K_4[Fe(CN)_6]$	Formation of a dark blue precipitate ("Berlin blue")
	Potassium thiocyanate KSCN	Red solution of thiocyanates $[Fe(SCN)]^{2+}$, etc.
	Salicylic acid $HCO_2C_6H_4OH$ in NH_3 (aqueous)	Red-purple solution of iron(III) salicylate

3. Conclusion

In conclusion, we can conclude that in fractional analysis of fifth group cations, each cation can be determined by a specific reaction regardless of the presence of other cations in the mixture. This confirms the speed and accessibility of fractional analysis.

Fractional analysis, a method of qualitative chemical analysis that allows the detection of individual ions in a solution without their preliminary sequential separation, is based on the use of highly sensitive selective reagents, with the help of which the desired ion can be detected in the presence of others.

The advantage of this method is that it uses small amounts of solution and the analysis time is short. The method is highly sensitive: the minimum detectable concentration of the ions of interest can reach 0.05–0.001 μg.

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