Ministry of Public Health of Ukraine

National O.O. Bohomolets Medical University

Medical and General Chemistry Department

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ANALYTICAL CHEMISTRY

SUBMODULE 1" QUALITATIVE ANALYSIS"

Student notebook

Kyiv- 2021

Course “Analytical chemistry”

for the students of Pharmaceutical faculty

MODULE 1" QUALITATIVE ANALYSIS"

Provide: Medical and General Chemistry Department

Availability: 2-nd academic year, Semester 3

Extent of education: lectures: 9 hrs;
practical lessons and tests: 40 hrs;
self-preparation -41 hrs

THEMATIC PLAN

of **lectures on analytical chemistry**

for the students of the second course pharmaceutical faculty

№ lectures	Topic
Semantic module 1. Ion equilibrium in analytical chemistry. Theory and practice of I - III analytical group cations analysis .	
1.	Object, task, role of analytical chemistry. Basic concepts, principles, methods of qualitative analysis. Analytical reactions, requirements to them.
2	Solutions of electrolytes in analytical chemistry.
Semantic module 2. Acid-base and complex formation equilibrium in analytical chemistry. Theory and practice of IV - VI of analytical group cations analysis .	
3	Ion equilibrium in analytical chemistry (dissociation of strong and weak electrolytes, hydrolysis etc).
4	Precipitation reactions in a chemical analysis.
5	Complex formation equilibrium and organic reagent in analytical chemistry.
6	Methods of separation and identification of cations.
Semantic module 3. The Reduction-oxidation equilibrium in analytical chemistry. Methods of separation and pre-concentration. Theory and practice of analysis of anions.	
7	Reduction-oxidation equilibrium in electrolyte’s solution. Redox reactions in analytical chemistry.
8	Methods of separation in analytical chemistry. Extraction.
9	Theory and practice of determining the qualitative composition of chemical compounds and drugs.

Main source:

[1] Analytical chemistry (Qualitative analysis). Part I. Edited by professor O. A. Ievtifieieva. Kharkiv,2014. https://www.researchgate.net/publication/301527607

[2] Analytical chemistry. Handbook. Edited by professor V. V. Bolotov. Kharkiv,2012. https://www.researchgate.net/publication/301542168

[3] Analytical chemistry. Tests collection. Edited by professor V. V. Bolotov. Kharkiv,2012. https://www.researchgate.net/publication/301282555

THEMATIC PLAN

of **practical lessons on analytical chemistry** for the students of the second course

pharmaceutical

№	Topic	
Semantic module 1. Ion equilibrium in analytical chemistry. Analysis theory and practice of I - III of analytical groups cations.		
1.	Rules of work and safety in a laboratory. Solution in analytical chemistry. Methods of solutions preparation. Acid-base classification of cations. 1-st group of cations (acid-basic classification of cations). Laboratory work "Analytical reactions of the 1-st analytical group of cations ". Test control.	4
2.	Second and Third group of cations. Laboratory work "Analytical reactions of the Second analytical group of cations ". Laboratory work "Analytical reactions of the III of analytical group of cations". Analysis of 1-3 groups of cations mixture. Semantic module 1 control: Writing control work and test.	
Semantic module 2. Acid-base and complex equilibriums in analytical chemistry. Analysis theory and practice of IV - VI of analytical groups cations.		
3.	Fourth group of cations. Laboratory work" Analytical reactions of IV analytical group of cations. Situational tasks.Analysis of Fourth group of cations mixture. Test control.	18
4.	Fifth group of cations. Laboratory work. "Analytical reactions of fifth analytical group of cations". Redox reactions.Writing of redox reactions based on the method of semireactions. Test control.	
5.	Sixth group of cations. Laboratory work "Analytical reactions of sixth analytical group of cations". Situational tasks and learning exercises. Test control.	
6.	Semantic modules 1- 2 control: the theory and practice of analysis of cations of I-VI analytical groups. Laboratory work "The analysis of cations I-VI analytical groups mixture". Writing control work and test.	
Semantic module 3. The Redox equilibrium in analytical chemistry. Methods of separation and pre-concentration. Theory and practice of analysis of anions.		
7.	Group reagents are in the analysis of anions. General characteristics of anions. Analytical reactions of anions I and II of analytical groups. Laboratory works "Analytical reactions of the First analytical group of anions "and "Analytical reactions of the Second analytical group of anions ".	

Test control.

8. Analytical reactions of anions of the III analytical group and anions of organic acids. Laboratory work "Reactions of anions of the III analytical group. Qualitative reactions of anions of organic acids ". Solving settlement and situational problems. Test control. Written control work.
9. Systematic analysis of compound of unknown composition. Dry salt analysis: cations identification
10 Systematic analysis of compound of unknown composition. Dry salt analysis: anions identification.
Submodule "Qualitative analysis" control: Test control. Written control work.

Practical Lesson 1

Rules of work and safety in a laboratory. Solution in analytical chemistry. Methods of solutions preparation. Acid-base classification of cations. 1-st group of cations (acid-basic classification of cations). Laboratory work "Analytical reactions of the 1-st analytical group of cations ". Test control.

Rules of Work and Safety in chemical-analytical laboratory

1. Before working in the chemical lab all students take instruction on safety and put his signature in the journal safety training.
2. In the chemical laboratory work in lab coats, each student should have a personal laboratory towel.
3. Before you perform the experiment, it is necessary to carefully read the instructions and follow specified methods perform the experiment. Do not pour excess reagents taken back into the glass.
4. Work with concentrated acids, alkalis, organic solvents should be performed in a fume hood. Heating the liquid in the tube, it should be kept away from the hole itself and working nearby colleagues.
5. Do not work with flammable substances (ether, alcohol, acetone, etc.) and store them near the lit torch. Flammable liquids should be heated only in a water bath.
6. The smell of the reaction products, such as NH3 or SO2, is determined by directing air from hand hole to the tube itself. Should be particularly careful when working with toxic and drastic substances; comply with all instructions of the teacher.
7. Do not take in a laboratory food. Do not try reagents to taste and mix them on your own.
8. Need economical use of reagents, electricity, gas, distilled water, care for chemical glassware and laboratory devices.
9. At the end of the work necessary to bring workplace in good order and pass to the teacher.

Theoretical questions

1. Solutions. Units that characterize quantitative composition of solutions.
2. Methods of solutions preparation.
3. Molecular, ionic and net ionic reactions.
4. Acid-basic classification of cations.
5. Analytical reactions of cations 1-st analytical group.

Practical questions

1. Write molecular, ionic and net ionic equation of the reactions presents in Laboratory work 1.
2. Determine the mass fraction of sodium hydroxide in solution is prepared by dissolving of 20g sodium hydroxide in 180g of water.

	m(NaOH),g	$m(H_2O)$,g	ω,%
1	20	180	
2	10	150	
3	25	145	
4	15	165	
5	5	195	
6	15	155	

3. You are asked to prepare 500 g of sodium chloride of 2.5% by mass. Calculate the mass of sodium chloride and water is required.

	m(solution),g	ω,%	m(NaCl),g	$m(H_2O)$,g
1	20	180		
2	10	150		
3	25	145		
4	15	165		
5	5	195		
6	15	155		

4. How many grams of sodium chloride must be dissolved in 1000 g of water to prepare a solution with mass fraction of sodium chloride 0.9%?

	m(NaCl),g	$m(H_2O)$,g	ω,%
1		1000	0.9
2		500	0.9
3		750	0.9
4		300	0.9
5		900	0.9
6		100	0.9

5. Calculate the molarity and normality of sulfuric acid solution, if mass of H_2SO_4 is 5.88 g and volume of solution - 300 mL.
6. How many grams of potassium hydroxide is in 200 mL of solution with the normality 0, 1 mol/L.
7. How many grams of SO_4^{2-} ions contain 1L of solution if molarity of these ions is 0, 2 mol/L.
8. Write the molecular, ionic and net ionic equations of the folowig reactions:

a) $Ba + H_2O \rightarrow$; b) $AlCl_3 + NaOH \rightarrow$; c) $Na_2CO_3 + Ca(OH)_2 \rightarrow$; d) $Ba(NO_3)_2 + H_2SO_4 \xrightarrow{T}$; e) $Zn + HCl \rightarrow$.	a) $NiSO_4 + NaOH \rightarrow$; b) $K_2SO_4 + Ba(OH)_2 \rightarrow$; c) $ZnO + Na_2CO_3 \rightarrow$; d) $MgO + Fe(OH)_3 \rightarrow$; e) $CoCl_2 + KOH \rightarrow$.

Laboratory work 1
THE 1-st ANALYTICAL GROUP OF CATIONS IDENTIFICATION
Reactions of K^+ cations.

Test 1. Sodium hydrotartrate $NaHC_4H_4O_6$ *(pharmacopeia's reaction)* react with Potassium ions and forms a white crystalline precipitate Potassium hydrotartrate:

$$K^+ + HC_4H_4O_6^- \rightarrow KHC_4H_4O_6\downarrow.$$

Precipitate is soluble in the mineral acids and alkalis. Solubility of precipitate increases undo heating.

To 3-4 drops of KCl solution add 3-4 drops of $NaHC_4H_4O_6$ solution. Cool the test tube under a water jet and rubs the wall-side of test tube by a glass stick till precipitate forms. Remember, if the tested solution is acidic, it is necessary to add Sodium acetate solution for immobilization of H^+ ions.

Observation:

Molecular equation: $KCl + NaHC_4H_4O_6 = KHC_4H_4O_6\downarrow + NaCl$

Ionic equation:

Net ionic equation:

Test 2. Sodium hexanitrocobaltate (III) of $Na_3[Co(NO_2)_6]$ *(pharmacopeia's reaction)* react with K^+ ions in a neutral or acetic-acid solution forms yellow crystalline precipitate of double salt $K_2Na[Co(NO_2)_6]$:

$$2K^+ + Na^+ + [Co(NO_2)_6]^{3-} = K_2Na[Co(NO_2)_6]\downarrow.$$

In a basic or an acidic solutions $Na_3[Co(NO_2)_6]$ (reagent) react with a base or an acid:

$$[Co(NO_2)_6]^{3-} + 3OH^- = Co(OH)_3\downarrow + 6NO_2^-.$$

$$[Co(NO_2)_6]^{3-} + 6H^+ = Co^{3+} + 6HNO_2.$$

It should be remembered that $Na_3[Co(NO_2)_6]$ is not stable in time. If its colour is rose (color of ions of Co^{2+}) such reagent can not apply for identification of K^+ ions.

Interfering ion is: NH_4^+ .

Place 1 drop of the tested solution on the slide plate and 2-3 drops of $Na_3[Co(NO_2)_6]$ solution near.

Use a glass stick to mix two solutions.

Observation:

Molecular equation: $2KCl + Na_3[Co(NO_2)_6] = K_2Na\,[Co(NO_2)_6]\downarrow + 2NaCl$

$$2K^+ + Na^+ + [Co(NO_2)_6]^{3-} = K_2Na[Co(NO_2)_6]\downarrow$$

Ionic equation:

Reactions of Na^+ cations.

Test 1. Potassium hexahydroxoantimonate $K[Sb(OH)_6]$ *(pharmacopeia's reaction)* react with the Sodium ions in a neutral or weak basic solution and form white precipitate:

$$Na^+ + [Sb(OH)_6]^- \rightarrow Na[Sb(OH)_6]\downarrow.$$

Precipitate does not appear in a strong basic solution.

The reagent is not stable in an acidic solution:

$$[Sb(OH)_6]^- + H+ \rightarrow H[Sb(OH)_6]$$

$$H[Sb(OH)_6] \rightarrow HSbO_3\downarrow + 3H_2O.$$

Precipitate does not form in dilute solutions.

Precipitation is slowed in presence of nitrate ions. Interfering ions are: NH_4^+, Mg^{2+}, Li^+.

To 3-4 drops of an investigated solution add 3-4 drops solution of reagent. Cool the test tube under the water jet and rubs the wall-side of test tube by a glass stick till precipitate forms.

Observation:

Molecular equation: $NaCl + K[Sb(OH)_6] = Na[Sb(OH)_6]\downarrow + KCl$

Ionic equation:

Net ionic equation:

Test 2. Flame test *(pharmacopeia's reaction)*.

To clean platinum wire: dip the platinum wire into a solution of HCl and heat it in a flame. Then dip the clean platinum wire into a solution of Sodium salt and heat it in a flame.

Observation:

Reactions of NH_4^+ cations.

Test 1. Solutions of alkalis (NaOH or KOH) *(pharmacopeia's reaction)* react with a solution of ammonium salt undo the heating results the ammonia:

$$NH_4^+ + OH^- = NH_3\uparrow + H_2O \text{ (Net ionic equation).}$$

Add 3-5 drops of an investigated solution to the test tube, some drops of NaOH or KOH and heat. Place a wet litmus paper above the test tube.

Observation:

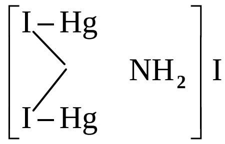
Molecular equation:

Test 2. Potassium tetraiodomercurate (II) $K_2[HgI_4]$ in presence of KOH (reagent of Nessler) with NH_4^+ ions forms red-brown precipitate:

$$NH_4Cl + 2K_2[HgI_4] + 2KOH = [NH_2Hg_2I_2]I\downarrow + 5KI + KCl + 2H_2O$$

$$NH_4^+ + 2[HgI_4]^{2-} + 2OH^- = [NH_2Hg_2I_2]I\downarrow + 5I^- + 2H_2O$$

The complex's $[NH_2Hg_2I_2]I$ structure is:



and name is bis-iodine bis-mercuryammonium iodide.

Interfering ions are: Fe^{3+}, Bi^{3+}, Cu^{2+}, Cd^{2+}, Ag^+, Pb^{2+}, As (V).

This reaction is sensitive and applicate for determination of trace concentration of the ammonium or ammonia.

Place 1 drop of the tested solution on the glass slide and 2-3 drops of Nessler's reagent near it. Use a glass stick to mix two solutions. Observation:

These questions are taken from the Test items for licensing examination Krok 1 PHARMACY

<u>The correct answer is first</u>

1-st group of cations

1) Reaction of sodium ions with potassium hexahydroxoantimonate (V) in neutral medium produces precipitate. Specify the color of this precipitate:
   A. White B. Red C. Yellow D. Green E. Blue
2) The solid residue obtained after evaporation of the sample solution makes the colorless flame of burner turn yellow, and when watched through a blue glass, it looks purple. What cations are present in the solid residue?
   A. Na^+, K^+ B. Ca^{2+}, K^+ C. Na^+ , Sr^{2+} D. Li^+, Ba^{2+} E. Na^+, Ca^{2+}
3) The ability of reagent to ensure a stable analytical effect in the interaction with the analyzed substance is characterized by:
   A. Reaction sensitivity B. Reaction selectivity
   C. Reaction specificity D. Reagent amount E. –
4) Some medications are produced by hydrolysis of corresponding neutral salts. From the salts listed below, select the one that WILL NOT succumb to hydrolysis:
   A. Na_2SO_4 B. $NaHCO_3$
   C. $AlCl_3$ D. $Bi(NO_3)_3$ E. Na_2SO_3
5) A pharmaceutical analyst has to identify potassium acetate. He can prove the presence of potassium cation in the analyzed substance by means of the following solution:
   A. Tartrate acid B. Sodium hydroxide

C. Potassium permanganate D. Iron (III) chloride E. Formate acid

6) What cations relate to the I analytic group according to the acid-base classification?

A. Sodium, potassium, ammonium B. Calcium, strontium, barium
C. Silver, lead, nickel D. Aluminium, magnesium, zinc
E. Potassium, barium, bismuth

7) What analytical effect is observed when potassium cation is being determined by the sodium hexanitrocobaltate (III) solution?

A. Yellow crystalline precipitate B. White crystalline precipitate
C. Yellow colouring of the solution D. Black crystalline precipitate
E. Red crystalline precipitate

Practical lesson 2

Second and Third group of cations. Laboratory works "Analytical reactions of the Second analytical group of cations ", "Analytical reactions of the III of analytical group of cations". Analysis of 1-3 groups of cations mixture. Semantic module 1 control: Writing control work and test

These questions are taken from the Test items for licensing examination Krok 1 PHARMACY

The correct answer is first

2-nd group of cations

1) Potassium dichromate solution was added into a solution obtained after the precipitate consisting of group II chloride cations was processed with hot water. Yellowprecipitate was produced; the precipitate is insoluble in acetic acid, but soluble in alkali. What cations were present in the solution under investigation?

A. **Lead (II)** B. Mercury (II) C. Barium D. Silver (I) E. Calcium

2) During reaction of silver cations identification first HCl and then ammonia solution have been added to the solution. What compound was produced as the result?

A. $\mathbf{[Ag(NH_3)_2]Cl}$ B. $[Ag_2(NH_3)_3]Cl$
C. AgOH D. AgCl E. $[Ag(NH_3)_3]Cl$

3) What anion of the 2nd analytic group produces black precipitate with the group reagent $AgNO_3$?

A. $\mathbf{S^{2-}}$ B. I^- C. Cl^- D. Br^- E. NCS^-

4) An analytical chemist performs qualitative analysis of cations of the II analytical group. The following solution is used to separate silver and mercury chlorides:

A. Ammonia B. Hydrochloric acid C. Sodium hydroxide

D. Sodium nitrate E. Potassium chloride

5) Burner's flame colors carminered in the presence of salts of an unknown cation. Name this cation:

A. Strontium B. Ammonium C. Sodium D. Potassium E. Iron

6) Silver mirror reaction can be characterized by:

A. Production of metal B. Smell C. Red colouring of precipitate

D. Blue colouring of solution E. Decolouration

7) What reagent is used to separate AgCl precipitate from AgI precipitate?

A. Aqueous solution of ammonia B. Concentrated nitric acid

C. Diluted nitric acid D. Concentrated solution of potassium chloride

E. Sulfuric acid solution

8) How to separate $PbSO_4$ from mixture of the 3rd analytical group cation sulphates in the process of systematic analysis?

A. Processing precipitate with 30% ammonium acetate solution B. Precipitate recrystallization

C. Processing precipitate with concentrated sulfate acid

D. Processing precipitate with acetate acid solution E. Processing precipitate with ammonia solution

9) Ammonia solution has been added to the solution being studied. Black precipitate has formed. That means the following cations are present in the solution:

A. Mercury (I) B. Copper (II) C. Iron (III) D. Iron (II) E. Silver (I)

10) A ground for separating lead (II) chloride from the other chlorides of the II analytical group (acid-base classification) is its different solubility in:

A. Hot water B. Hydrochloric acid C. Alkalis

D. Ammonia solution E. Sulfuric acid

11) To isolate the lead (II) chloride from the other cations of the II analytical group in the systematic analysis, the chloride precipitate should be processed with:

A. Hot water B. Ammonia solution C. Nitric acid solution

D. Acetate acid solution E. Alkali solution

12) The ability of reagent to ensure a stable analytical effect in the interaction with the analyzed substance is characterized by:

A. Reaction sensitivity B. Reaction selectivity

C. Reaction specificity D. Reagent amount E. –

13) Some medications are produced by hydrolysis of corresponding neutral salts. From the salts listed below, select the one that WILL NOT succumb to hydrolysis:

A. Na_2SO_4 B. $NaHCO_3$ C. $AlCl_3$ D. $Bi(NO_3)_3$ E. Na_2SO_3

14) Ammonia solution has been added to the solution under examination. A black precipitate fell out. This indicates the presence of the following cations in the solution:

A. Mercury (I) B. Copper (II) C. Iron (III) D. Iron (II) E. Silver (I)

15) 1M sulphuric acid solution was added to the solution under study. This resulted in formation of white sediment that was soluble in the alkalies. This indicated that the solution contains:

A. Plumbum cations B. Calcium cations C. Barium cations
D. Argentum cations E. Mercury (I) cations

16) Specify two compounds that can be present in a solution at the same time:

A. $Al(NO_3)_3$ and HCl B. $Ba(OH)_2$ and CO_2 C. NaOH and P_2O_5
D. $CuSO_4$ and $BaCl_2$ E. $AgNO_3$ and HCl

17) 1 M sulphuric acid solution was added to the solution under study. This resulted in formation of white sediment that was soluble in the alkalies. This indicated that the solution contains:

A. Plumbum cations B. Calcium cations C. Barium cations
D. Argentum cations E. Mercury (I) cations

18) After the diluted solution of hydrochloric acid had been added to the solution under examination, the white caseous precipitate settled down. This indicates presence of the following ions:

A. Silver B. Ammonium C. Iron (II) D. Barium E. Iodine

19) Diluted solution of hydrochloric acid was added to a solution under examination. This resulted in origin of white caseous deposition. This is the evidence of presence of following ions:

A. Silver B. Ammonium C. Iron (II) D. Barium E. Iodine

20) Adding of a diluted solution of chlorohydrogen acid to a solution under examination resulted in formation of white caseous sediment. It is the evidence of presence of the following ions:

A. Silver B. Ammonium C. Iron (II) D. Barium E. Iodine

Laboratory work 2

THE 2-nd ANALYTICAL GROUP OF CATIONS IDENTIFICATION

Reactions of Ag^{+} cations.

Test 1. To the test tube add 3 - 5 drops of $AgNO_3$ solution and 1 - 2 drops of HCl solution.
Observation:

Molecular equation: $AgNO_3 + HCl = AgCl\downarrow + HNO_3$

Ionic equation:

Net ionic equation:

Divide a mixture into two clean test tubes.
Add 3 - 5 drops HNO_3 to the first test tube.
Observation:

Add drop by drop of NH_3 solution to the second test tube up to precipitate dissolve.

Molecular equation: $AgCl + 2NH_3 = [Ag(NH_3)_2]Cl$.

Ionic equation:

Net ionic equation:

Test 2. *Silver mirror reaction*. To the test tube add 5 - 10 drops of $AgNO_3$ solution and drop by drop NH_3 solution to a precipitate formation. Continue adding of amonia solution till the precipitate dissolves.
Observation:

Molecular equation: $AgNO_3 + 2NH_3 = [Ag(NH_3)_2]NO_3$

Ionic equation:
Net ionic equation:

Then add 10 drops of formaldehyde solution and heat up to boiling.
Molecular equation:

$$4[Ag(NH_3)_2]NO_3 + CH_2O + H_2O = 4Ag\downarrow + 4NH_4NO_3 + 4NH_3 + CO_2$$

Ionic equation:

Net ionic equation:

Test 3. To the test tube add 3 - 5 drops of $AgNO_3$ solution and 1 - 2 drops of K_2CrO_4 solution.

Observation:

Net ionic equation: $2Ag^{+} + CrO_4^{2-} = Ag_2CrO_4\downarrow$.

Molecular equation:

Divide a mixture into two clean test tubes.

To one of them add HNO_3.

Observation:

Molecular equation: $2Ag_2CrO_4\downarrow + 4HNO_3 = 4AgNO_3 + H_2Cr_2O_7 + H_2O$.

Ionic equation:

Net ionic equation:

To the second test tube add NH_3 solution.

Observation:

Ionic equation: $Ag_2CrO_4\downarrow + 4NH_3 = 2\,[Ag\,(NH_3)_2]^{+} + CrO_4^{2-}$.

Molecular equation:

Reactions of Pb^{2+} cations.

Test 1. To the test tube add 1 drop of $Pb(NO_3)_2$ solution and 1 drop of KI solution and 0,5 – 1 ml of CH_3COOH solution. (*Note: CH_3COOH does not take part in reaction but promotes the formation of PbI_2 beautiful crystals*) Heat mixture up to boiling then cool the test tube under a water jet.

Observation:

Net ionic equation: $Pb^{2+} + 2I^{-} = PbI_2\downarrow$

Molecular equation:

Test 2. To the test tube add 3 - 5 drops of $Pb\,(NO_3)_2$ solution and 1 - 2 drops of K_2CrO_4 solution.

Observation:

Net ionic equation: $Pb^{2+} + CrO_{42}^{-} = PbCrO_4\downarrow$

Molecular equation:

Divide a mixture into two clean test tubes.
Add 3 - 5 drops of CH_3COOH solution to the first test tube (whether the precipitate is dissolved?)
Observation:

Add to the second test tube 10 - 20 drops of NaOH concentrated solution.
Observation:

Molecular equation: $PbCrO_4\downarrow + 4NaOH = Na_2[Pb(OH)_4] + Na_2CrO_4$

Ionic equation:

Net ionic equation:

Reactions of Hg_2^{2+} cations.

Test 1. To the test tube add 3 - 5 drops of $Hg_2(NO_3)_2$ solution and 1 - 2 drops of HCl solution.
Observation:

Net ionic equation: $Hg_2^{2+} + 2Cl^- = Hg_2Cl_2\downarrow$.
Molecular equation:

Test 2. To the test tube add 3 - 5 drops of $Hg_2(NO_3)_2$ solution and 1 drop of HCl solution, and then 3 - 5 drops of NH_3 solution.
Observation: the white precipitate which was formed first gradually blackens after addition of solution NH_3.
Molecular equation: $Hg_2(NO_3)_2 + 2HCl = Hg_2Cl_2\downarrow + 2HNO_3$
Ionic equation:

Net ionic equation:

Molecular equation: $Hg_2Cl_2\downarrow + 2NH_3 = [NH_2Hg]Cl\downarrow + Hg\downarrow + NH_4Cl$
($[NH_2Hg]Cl\downarrow$ - a white precipitate, $Hg\downarrow$ - black dispersed mercury precipitate.

Ionic equation:

Net ionic equation:

These questions are taken from the Test items for licensing examination Krok 1 PHARMACY

<u>The correct answer is first</u>

3-rd group of cations

1) What reagent should be chosen in order to detect presence of Ca^{2+} cation in a solution?

   A. $(NH_4)_2C_2O_4$ B. HCl C. HNO_3 D. KCl E. NaBr

2) In the qualitative analysis that involves precipitation of sulfates of the third analytical group cations (Ca^{2+}, Sr^{2+}, Ba^{2+}) the solubility of sulfates can be reduced by adding:

   A. Ethyl alcohol B. Distilled water C. Benzene D. Chloroform E. Amyl alcohol

21) Specify the reagent allowing to determine barium cations in the presence of calcium and strontium cations:

   A. Potassium dichromate B. Potassium chloride C. Potassium iodide D. Potassium nitrate E. Sodium hydroxide

3) In the qualitative analysis which involves precipitation of sulphates of the third analytical group cations (Ca^{2+}, Sr^{2+}, Ba^{2+}) the solubility of sulphates can be reduced by adding:

   A. Ethyl alcohol B. Distilled water C. Benzene D. Chloroform E. Amyl alcohol

4) How to separate $PbSO_4$ from mixture of the 3rd analytical group cation sulphates in the process of systematic analysis?

   A. Processing precipitate with 30% ammonium acetate solution
   B. Precipitate recrystallization C. Processing precipitate with concentrated sulfate acid
   D. Processing precipitate with acetate acid solution E. Processing precipitate with ammonia solution

5) Why is ethyl alcohol used along with the group reagent of the third analytical group?

   A. To ensure full precipitation of all cations of this group B. To further dissolve obtained precipitate
   C. For fractional precipitation of cations D. To change pH of medium
   E. To prevent complexing

6) To maintain a certain level of pHmedium, the buffer solutions are used. Specify a composition of substances that DOES NOT EXHIBIT buffer properties:

   A. NaOH + NaCl B. $CH_3COOH + CH_3COONa$ C. $NH_4Cl + NH_3 \cdot H_2O$

D. HCOOH + HCOONa E. NaH_2PO_4 + Na_2HPO_4

7) The ability of reagent to ensure a stable analytical effect in the interaction with the analyzed substance is characterized by:

A. Reaction sensitivity B. Reaction selectivity

C. Reaction specificity D. Reagent amount E. –

8) The pH of 0,001 M of hydrochloric acid solution is:

A. 3 B. 0 C. 10 D. 7 E. 5

9) Some medications are produced by hydrolysis of corresponding neutral salts. From the salts listed below, select the one that WILL NOT succumb to hydrolysis:

A. Na_2SO_4 B. $NaHCO_3$ C. $AlCl_3$ D. $Bi(NO_3)_3$ E. Na_2SO_3

10) Specify two compounds that can be present in a solution at the same time:

A. $Al(NO_3)_3$ and HCl B. $Ba(OH)_2$ and CO_2

C. NaOH and P_2O_5 D. $CuSO_4$ and $BaCl_2$ E. $AgNO_3$ and HCl

11) During identification of an unknown salt the colorless part of the burner flame turned yellow and green. What cation was the salt formed by?

A. Ba^{2+} B. Ca^{2+} C. Sr^{2+} D. Na^{+} E. K^{+}

12) What saturated heated solution is used for transformation of sulphates $BaSO_4$, $SrSO_4$, $CaSO_4$ to carbonates during the systematic analysis?

A. Na_2CO_3 B. $CaCO_3$ C. $(NH_4)_2CO_3$ D. $MgCO_3$ E. CO_2

13) Solution of potassium chromate was added to a solution under examination. As a result of it some yellow deposition settled down. This deposition cannot be dissolved in acetic acid. This means thatthe solution under examination contains cations of:

A. Barium B. Calcium C. Sodium D. Cobalt E. Magnesium

14) Cations of the third analytical group (acid-base classification) can be isolated in course of systematic analysis by means of the following group reagent:

A. 1 M solution of sulfate acid in presence of ethanol B. 1 M solution of potassium chromate

C. 0,1 M solution of sodium carbonate D. 0,1 M solution of ammonium oxalate

E. 1 M solution of ammonium carbonate

Laboratory work 3

THE 3-d ANALYTICAL GROUP OF CATIONS IDENTIFICATION

Reactions of Ca^{2+} cations

Test 1. To the test tube add 3 - 5 drops of $CaCl_2$ solution and 3 - 5 drops of Na_2CO_3 solution.
Observation:

Net ionic equation: $Ca^{2+} + CO_3^{2-} = CaCO_3 \downarrow$
Ionic equation:

Molecular equation:

Divide a mixture into two clean test tubes.
Add 3 - 5 drops HCl to the first test tube.
Observation:
Molecular equation:

Ionic equation:

Net ionic equation:

Add 3 - 5 drops CH_3COOH to the second.
Observation:

Molecular equation:

Ionic equation:

Net ionic equation:

Test 2. To the test tube add 3 - 5 drops of $CaCl_2$ solution and 1 - 2 drop of H_2SO_4 solution and 5 - 10 drops of ethanol.
Observation:

Molecular equation:

Ionic equation:

Net ionic equation:

Add 3 - 5 drops of HCl solution to precipitate (whether the precipitate is dissolved?).
Observation:

Test 3. To the test tube add 1 - 2 drops of $CaCl_2$ solution and 1 - 2 drop of $(NH_4)_2C_2O_4$ a solution.
Observation:

Ionic equation: $Ca^{2+} + C_2O_4^{2-} = CaC_2O_4\downarrow$.
Molecular equation:

Divide a mixture into two clean test tubes.
Add 3 - 5 drops HCl to the first test tube.
Observation:

Add 3 - 5 drops CH_3COOH to the second.
Observation:

Molecular equation: $CaC_2O_4\downarrow + 2HCl = CaCl_2 + H_2C_2O_4$
Ionic equation:

Net ionic equation:

Reactions of Sr^{2+} cations

Test 1. To the test tube add 3 - 5 drops of $Sr(NO_3)_2$ solution and 3 - 5 drops of Na_2CO_3 solution.
Observation:

Net ionic equation: $Sr^{2+} + CO_3^{2-} = SrCO_3\downarrow$
Molecular equation:

Divide a mixture into two clean test tubes .
Add 3 - 5 drops HCl to the first test tube.
Observation:

Add 3 - 5 drops CH_3COOH to the second.
Observation:

Molecular equation:

Ionic equation:

Net ionic equation:

Test 2. To the test tube add 3 - 5 drops of $Sr(NO_3)_2$ solution and 1 - 2 drops of H_2SO_4 solution and 5 - 10 drops ethanol.

Observation:

Molecular equation:

Ionic equation:

Net ionic equation:

To a mixture add 3 - 5 drops of HCl solution (whether the precipitate is dissolved?).
Observation:

Test 3. To the test tube add 3 - 5 drops of $Sr(NO_3)_2$ solution and $CaSO_4$ and leave for 3 - 5 minutes.
Observation:

$$Sr(NO_3)_2 + CaSO_4 = SrSO_4\downarrow + Ca(NO_3)_2$$

Test 4. On a filter paper put 1 drop of $Sr(NO_3)_2$ solution, then near put 1 drop of a sodium rodizonate on such distance that splashes from solutions have incorporated.
Observation:
Drop HCl solution on the painted place.
Observation:

Reactions of Ba^{2+} ion
Test 1. To the test tube add 3 - 5 drops of $Ba(NO_3)_2$ solution and $CaSO_4$. Pay attention, that Ba^{2+} ion gives effect immediately.
Observation:

Molecular equation: $Ba(NO_3)_2 + CaSO_4 = BaSO_4\downarrow + Ca(NO_3)_2$

Test 2. To the test tube add 3-5 drops of Ba $(NO_3)_2$ solution and 1 - 2 drop of H_2SO_4 solution.
Observation:

Ionic equation:

Net ionic equation:

Add 3 - 5 drops of HCl (whether the precipitate is dissolved?).
Observation:

Test 3. To the test tube add 3 - 5 drops of $Ba(NO_3)_2$ solution and 1 - 2 drop of $K_2Cr_2O_7$ solution and 3 - 5 drops of CH_3COONa solution.
Observation:

Molecular equation:

$2Ba(NO_3)_2+K_2Cr_2O_7+CH_3COONa+H_2O=2BaCrO_4\downarrow + 2CH_3COOH+2KNO_3+2NaNO_3$

Ionic equation:

Net ionic equation:

Divide a mixture into two clean test tubes.
Add 3 - 5 drops HCl to the first test tube.
Observation:

$$2BaCrO_4\downarrow + 4HCl = 2BaCl_2 + H_2Cr_2O_7 + H_2O$$

Ionic equation:

Net ionic equation:

Add 3 - 5 drops CH_3COOH to the second.
Observation:

Is precipitate $BaCrO_4$ dissolved after addition of CH_3COOOH?

Do the same tests with $CaCl_2$ and $Sr(NO_3)_2$ solutions.
Observation:

Test 4. On a filter paper put 1 drop of $Sr(NO_3)_2$ solution, then near put 1 drop of a sodium rodizonate on such distance that splashes from solutions have incorporated.
Observation:

Drop HCl solution on the painted place. What is observed?

Practical Lesson 3

Fourth group of cations. Laboratory work" Analytical reactions of IV analytical group of cations. Analysis of Fourth group of cations mixture. Test control.

These questions are taken from the Test items for licensing examination Krok 1 PHARMACY

<u>The correct answer is first</u>

4-th group of cations

1) Filter paper impregnated with solution of cobalt (II) nitrate and a solution under examination makes blue ash when burned down. This is the evidence of presence of the following ions:

A. Al^{3+} B. Cr^{3+} C. Ni^{2+} D. Sb^{3+} E. Zn^{2+}

2) What cation of the 4th analytical group is present in a solution, if it is known that the reaction with a group reagent causes formation of yellow precipitate?
A. Cr^{3+} B. Zn^{2+} C. Sn^{2+} D. Al^{3+} E. Sn(IV)

3) In order to identify the cations of zinc (II) an analytical chemist used the reagent solution of hexacyanoferrate (II) potassium (Pharmacopeia reaction). What colour precipitate is formed in this reaction?
A. White B. Yellow C. Black D. Green E. Red

4) A solution contains cations of zinc and aluminum. Specify the reagent that enables to detect cations of zinc in this solution:
A. Potassium hexacyanoferrate (II) solution B. Sodium hydroxide solution
C. Cobalt nitrate $Co(NO_3)_2$ D. The excess of 6M sodium hydroxide in presence of hydrogen peroxide
E. Sulfuric acid solution

5) Specify two compounds that can be present in a solution at the same time:
A. $Al(NO_3)_3$ and HCl B. $Ba(OH)_2$ and CO_2 C. NaOH and P_2O_5
D. $CuSO_4$ and $BaCl_2$ E. $AgNO_3$ and HCl

6) The fourth group of cations includes the following cations: Al^{3+}, Sn^{2+}, Sn(IV), As(V), As(III), Zn^{2+}, Cr^{3+}. The group reagent for the fourth group of cations is the solution of:
A. NaOH, H_2O_2 B. HCl C. NH_3, H_2O_2 D. $H_2C_2O_4$ E. H_2SO_4, H_2O_2

15) What cation of the 4th analytical group is present in a solution, if its reaction with the group reagent results in formation of yellow precipitate?
A. Cr^{3+} B. Zn^{2+} C. Sn^{2+} D. Al^{3+} E. Sn(IV)

16) Analysis of a dry substance always begins with preliminary tests. Sample under investigation is green in color, which allows to conclude the presence of:
A. Chrome (III) B. Manganese (II) C. Cobalt (II) D. Iron (III) E. Barium (II)

7) What cation of the 4th analytical group is present in a solution, if it is known that the reaction with a group reagent causes formation of green precipitate?
A. Cr^{3+} B. Zn^{2+} C. Sn^{2+} D. Al^{3+} E. Sn(IV)

8) Chemist-analyst should use the following reaction to detect chromium (III) ions during preliminary tests:
A. Reaction of peroxochromate acid formation after previous chromium oxidation
B. Apply sodium hydroxide
C. Apply potassium permanganate
D. Apply ammonia
E. Apply sodium hydroxide and hydrogen peroxide

9) A solution contains cations of zinc and aluminium. Specify the reagent that makes it possible to detect cations of zinc in this solution:
A. Potassium hexacyanoferrate (II) solution
B. Sodium hydroxide solution
C. Cobalt nitrate $Co(NO_3)_2$
D. Excess of 6M sodium hydroxide in presence of hydrogen peroxide
E. Sulfuric acid solution

10) The fourth group of cations includes the cations Al^{3+}, Sn^{2+}, Sn(IV), As(V), As(III), Zn^{2+}, Cr^{3+}. The group reagent for the fourth group of cations is the solution of:
A. NaOH, H_2O_2
B. HCl
C. NH_3, H_2O_2
D. $H_2C_2O_4$
E. H_2SO_4, H_2O_2

11) What reagents are used to separate the cations of the IV analytical group from the cations of the V and VI analytical groups in the analysis of their composition?
A. NaOH and H_2O_2
B. H_2SO_4
C. Dithizone
D. NH_4OH
E. Na_2S

12) To maintain a certain level of pH medium, the buffer solutions are used. Specify a composition of substances that DOES NOT EXHIBIT buffer properties:
A. NaOH + NaCl
B. $CH_3COOH + CH_3COONa$
C. $NH_4Cl + NH_3 \cdot H_2O$
D. HCOOH + HCOONa
E. $NaH_2PO_4 + Na_2HPO_4$

13) A solution contains cations of zinc and aluminum. Specify the reagent that allows to detect cations of zinc in this solution:
A. Potassium hexacyanoferrate (II) solution
B. Sodium hydroxide solution
C. Cobalt nitrate $Co(NO_3)_2$
D. Excess of 6M sodium hydroxide in presence of hydrogen peroxide
E. Sulfuric acid solution

14) The ability of reagent to ensure a stable analytical effect in the interaction with the analyzed substance is characterized by:
A. Reaction sensitivity
B. Reaction selectivity

C. Reaction specificity D. Reagent amount E. –

15) The pH of 0,0001 M of hydrochloric acid solution is:

A. 4 B. 0 C. 10 D. 7 E. 5

16) Some medications are produced by hydrolysis of corresponding neutral salts. From the salts listed below, select the one that WILL NOT succumb to hydrolysis:

A. Na_2SO_4 B. $NaHCO_3$ C. $AlCl_3$ D. $Bi(NO_3)_3$ E. Na_2SO_3

17) Filter paper impregnated with solution of cobalt (II) nitrate and a solution under examination forms blue ash when burned down. This is the evidence of presence of the following ions:

A. Al^{3+} B. Cr^{3+} C. Ni^{2+} D. Sb^{3+} E. Zn^{2+}

18) Sodium arsenate solution can be distinguished from the arsenite solution by means of the following reagent:

A. Magnesia mixture B. Potassium sulphate C. Potassium nitrate

D. Sodium chloride E. Sodium fluoride

19) During analysis of cations of the IV analytic group Zn cations can be detected under certain conditions with the following reagent:

A. Dithizone B. Ammonia solution C. Alkali

D. Alkali metal carbonates E. Dimethylglyoxime

17) Qualitative reaction for determination of Cr(VI) compounds is origination of chromium oxide-diperoxide that stains ether layer with blue. What is formula of this chromium compound?

A. CrO_5 B. CrO_3 C. Cr_2O_3 D. CrO E. $NaCrO_2$

18) You have to carry out a qualitative analysis. What substance will originate from chromium ions under the influence of group reagent excess (solution of sodium hydroxide) upon cations of the IV analytical group?

A. Sodium hexahydroxochromate (III) B. Chromium (III) hydroxide

C. Chromium (III) oxide D. Chromium (II) hydroxide

E. Chromium (II) oxide

19) Presence of the following ion of delements in solutions can be exploited by means of $K_4[F e(CN)_6]$:

A. Fe^{3+} B. Zn^{2+} C. Cr^{3+} D. Ni^{2+} E. Cu^{2+}

20) Under certain conditions of qualitative analysis $K_4[Fe(CN)_6]$ is a specific reagent to Fe^{3+} cations. What colour is the precipitate?

A. Blue B. White C. Brown D. Red E. Black

21) Solution with NaCl mass concentration of 0,95% is a part of a saline and can be used in case of significant blood loss. Name reaction of this solution's medium:

A. Neutral (pH = 7,0) B. Acidic (pH < 7,0)
C. Alkaline (pH > 7,0) D. Very acidic (pH = 1,0) E. Very alkaline (pH = 12,0)

22) Solution of a medicinal preparation under examination contains cations of magnesium (II) and aluminium (III). What reagent will help to separate these cations during analysis of this preparation?

A. Alkali solution B. Hydrogen peroxide in acidic medium C. Argentum nitrate solution
D. Hydrogen peroxide in ammoniac medium E. Chloride acid solution

Laboratory work №4
THE 4-th ANALYTICAL GROUP OF CATIONS IDENTIFICATION

Reactions of Al^{3+} cations

Test 1. To the test tube add 3 - 5 drops of $Al_2(SO_4)_3$ solution and 1 - 2 drops of NaOH solution.
Observation:

Molecular equation: $Al_2(SO_4)_3+6NaOH=2Al(OH)_3\downarrow+3Na_2SO_4$

Write ionic and net ionic equations:

Divide the test tube content into two parts.
Add 3 - 5 drops H_2SO_4 solution to the first test tube.
Observation:

Molecular equation: $2Al(OH)_3 + 3H_2SO_4 = Al_2(SO_4)_3 + 6H_2O$

Write ionic and net ionic equations:

Add 3 - 5 drops NaOH solution to the second.
Observation:

Molecular equation: $Al(OH)_3 + NaOH = Na[Al(OH)_4]$

Write ionic and net ionic equations:

To the resulting solution of $Na[Al(OH)_4]$ add concentrated NH_4Cl solution and heat the mixture.

Observation:

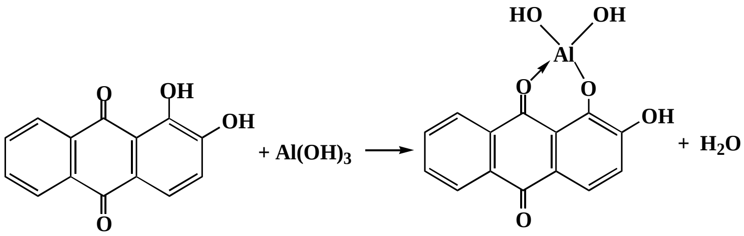
Molecular equation: $Na[Al(OH)_4] + NH_4Cl = Al(OH)_3\downarrow + NH_3 + H_2O + NaCl$

Write ionic and net ionic equations:

Test 2. Place 1 drop of $Al_2(SO_4)_3$ solution on the filter paper, dry it and add one drop of NH_3 solution. Then place one drop of Alizarine red S on the spot.

Observation:

Molecular equation: $Al_2(SO_4)_3 + 6NH_3+6H_2O = 2Al(OH)_3\downarrow + 3(NH_4)_2SO_4$



Test 3. Place 1 - 2 drops of $Al_2(SO_4)_3$ solution on a filter paper, dry it and add 1 - 2 drops of a $Co(NO_3)_2$ solution, dry and then burn filter paper.

$$2Al_2(SO_4)_3+2Co(NO_3)_2 \overset{T}{=} 2Co(AlO_2)_2+4NO_2\uparrow+O_2\uparrow + 6SO_3\uparrow$$

$$\overset{+5}{N} + 1e = \overset{+4}{N} \quad | \; 2 \; | \; 4$$

$$\overset{-2}{O} - 2e = \overset{0}{O} \quad | \; 1 \; | \; 2$$

2

Observation: cobalt aluminate $Co(AlO_2)_2$ is blue

Reactions of Sn^{2+} cations

Test 1. To the test tube add 3 - 5 drops of $SnCl_2$, 1-2 drops of NaOH solution.

Observation:

Net ionic reaction: $Sn^{2+} + 2OH^{-} = Sn(OH)_2\downarrow$

Write a molecular equation:

Divide the test tube content into two parts.
Add 5 - 10 drops of H_2SO_4 to the first test tube.
Observation:

Write molecular, ionic and net ionic equations:

Add 3 - 5 drops NaOH solution to the second tube till the precipitate dissolves.

Net ionic equation: $Sn(OH)_2 + 2OH^{-} = [Sn(OH)_4]^{2-}$

Write a molecular equation:

Test 2 (reduction properties of salts Sn^{2+}). To the test tube add 3 - 5 drops of $FeCl_3$ solution and 1 - 2 drops of $K_3[Fe(CN)_6]$ solution. To the mixture add 1 drop of $SnCl_2$ solution. Explain the formation of blue color precipitate.

Molecular equation: $2FeCl_3 + 2K_3[Fe(CN)_6] + SnCl_2 = 2KFe[Fe(CN)_6]\downarrow + SnCl_4 + 4KCl$

Write an ionic and a net ionic equation:

Reactions of cations Cr^{3+}

Test 1. To the test tube add 3 - 5 drops of $Cr_2(SO_4)_3$ solution, add NaOH solution to precipitate formation.

Observation:

Write molecular, ionic and net equations:

Divide the test tube content into two parts.
Add excess of NaOH solution to the first test tube.
Observation:

Write molecular, ionic and net ionic equations:

Add excess of H_2SO_4 solution to the second test tube.

Observation:

Write molecular, ionic and net ionic equations:

Reactions of Zn^{2+} cations

Test 1. To the test tube add 3 - 5 drops of $ZnCl_2$ solution; add 1-2 drops of NaOH solution.

Observation:

Net ionic equation: $Zn^{2+} + 2OH^- = Zn(OH)_2\downarrow$

Molecular equation:

The mixture obtained containes precipitate Zn $(OH)_2$ divide into *three* test tubes.

In the first test tube check the solubility of Zn $(OH)_2$ in NaOH solution.

Net ionic equation: $Zn(OH)_2 + 2OH^- = [Zn(OH)_4]^{2-}$

Write a molecular equation:

In the second tube check the solubility of Zn $(OH)_2$ in H_2SO_4 solution.

Net ionic equation: $Zn(OH)_2 + 2H^+ = Zn^{2+} + 2H_2O$

Write a molecular equation:

In the third tube check the solubility of Zn $(OH)_2$ in NH_3 solution.

Net ionic equation: $Zn(OH)_2 + 4NH_3 = [Zn(NH_3)_4]^{2+} + 2OH^-$

Write a molecular equation:

Test 2. To the test tube add 3 - 5 drops of $ZnCl_2$ solution, add 1 drop of K_4 [Fe $(CN)_6$] solution. Observe the formation of a white precipitate.
Molecular equation: $3ZnCl_2 + 2K_4[Fe(CN)_6] = K_2Zn_3[Fe(CN)_6]_2\downarrow + 6KCl$

Write ionic and net ionic equation:

Check solubility of precipitate in CH_3COOH. Note that the precipitate does not dissolve in CH_3COOH.

Test 3. Place 1 - 2 drops of $ZnCl_2$ solution on a filter paper, dry it and add 1 - 2 drops of a $Co(NO_3)_2$ solution, dry and then burn filter paper.

$2ZnCl_2 + 2Co(NO_3)_2 + 2H_2O = 2CoZnO_2 + 4NO_2\uparrow + O_2\uparrow + 4HCl\uparrow$ (Green ppt)

Observation:

Systematic analysis of a IV group cations

1. To a mixture of cations IV groups add a group reagent (2 mol/L NaOH and H_2O_2).
The solution 1 prepared in result of reaction **contains:**
SnO_3^{2-}, CrO_4^{2-}, AsO_4^{3-}, $[Al(OH)_6]^{3-}$, $[Zn(OH)_4]^{2-}$

2.Separation of Aluminium and Tin ions by precipitation**.**

To the **solution 1** add some dry ammonium chloride and heat.
In result the precipitate 1 of hydroxides $Al(OH)_3$, $Sn(OH)_4$ and a solution 2 of chromate, arsenate, tetrahydroxozincate are formed.

Precipitate 1	**Solution 2**
$Al(OH)_3$, $Sn(OH)_4$	CrO_4^{2-}, AsO_4^{3-}, $[Zn(OH)_4]^{2-}$

3. **Determination of Aluminium and Tin ions.**

The precipitate 1 dissolve in 6 mol/L hydrochloric acid solution. Identificate the Al^{3+} and Sn (IV) ions (see Reaction of identification)

4. In **the solution 2** detect the ions of Arsenic, Zinc and Chromium (as chromat ions) (see Reaction of identification).

Practical lesson 4

Fifth group of cations. Laboratory work. "Analytical reactions of fifth analytical group of cations". Redox reactions. Writing of redox reactions based on the method of semireactions. Test control.

These questions are taken from the Test items for licensing examination Krok 1 PHARMACY

<u>The correct answer is first</u>

5-th group of cations

1) Concentrated nitric acid and crystalline lead dioxide were added to a solution under examination. The solution turned crimson. This analytical effect indicates presence of:
   A. Manganese (II) B. Bismuth (III) C. Iron (III)
   D. Chromium (III) E. Tantum (II
2) What cations of the V analytical group can be detected by hydrolysis?
   A. Antimony and bismuth B. Manganese C. Iron (II)

D. Magnesium E. Iron (III)

3) Sodium hydroxide was added to a solution. Precipitation occurred. The precipitate was initially white and became brown later. It indicates the presence of the following in the solution:

A. Manganese (II) cations B. Lead (II) cations C. Potassium cations

D. Calcium cations E. Barium cations

4) Sodium hydroxide was added to a solution. Precipitation occurred. The precipitate was initially white and became brown later. It indicates the presence of the following in the solution:

A. Manganese (II) cations B. Lead (II) cations C. Potassium cations

D. Calcium cations E. Barium cations

5) In chemico-analytical laboratory a specialist studies the mixture of the 5th analytical group cations. When thiocyanate ions are added the solution becomes red-colored. This analytical effect indicates presence of the following cation:

A. Fe^{3+} B. Fe^{2+} C. Mg^{2+} D. Bi^{3+} E. Mn^{2+}

6) What reagents are used to separate the cations of the IV analytical group from the cations of the V and VI analytical groups in the analysis of their composition?

A. NaOH and H_2O_2 ` B. H_2SO_4 C. Dithizone D. NH_4OH E. Na_2S

7) Selective solvents are used in laboratories and factories to isolate and refine essential oils, alkaloids, antibiotics and other pharmaceutical substances. This process is called:

A. Extraction B. Sedimentation C. Coagulation D. Flocculation E. Flotation

8) The ability of reagent to ensure a stable analytical effect in the interaction with the analyzed substance is characterized by:

A. Reaction sensitivity B. Reaction selectivity C. Reaction specificity D. Reagent amount E. –

9) The pH of 0,001 M of hydrochloric acid solution is:

A. 3 B. 0 C. 10 D. 7 E. 5

10) Some medications are produced by hydrolysis of corresponding neutral salts. From the salts listed below, select the one that WILL NOT succumb to hydrolysis:

A. Na_2SO_4 B. $NaHCO_3$ C. $AlCl_3$ D. $Bi(NO_3)_3$ E. Na_2SO_3

11) After a solution had been heated with $(NH_4)_2S_2O_8$ in presence of $AgNO_3$, it turned crimson. What ions were present in the solution?

A. Mn^{2+} B. Fe^{3+} C. Fe^{2+} D. Co^{2+} E. Cu^{2+}

12) Specify two compounds that can be present in a solution at the same time:

A. $Al(NO_3)_3$ and HCl B. $Ba(OH)_2$ and CO_2 C. NaOH and P_2O_5

D. $CuSO_4$ and $BaCl_2$ E. $AgNO_3$ and HCl

13) Presence of which ion of d-elements in the solutions can be detected by means of $K_4[Fe(CN)_6]$?

A. Fe^{3+} B. Fe^{2+} C. Zn^{2+} D. Cr^{3+} E. Cu^{2+}

14) The analytical effect of reaction of potassium hexacyanoferrate (II) solution with iron (III) ions is:

A. Formation of blue precipitate B. Formation of white precipitate

C. Formation of blue precipitate and its dissolution in the excess of the reagent

D. Effervescence

E. Characteristic smell

15) A solution containing the cations of the V analytic group (acid-base classifi- cation) has been taken for the analysis. The solution of sodium hydroxostannite has been added to the composition which resulted in formation of black deposition. This is the evidence of presence of the following cation:

A. Bi^{3+} B. Fe^{2+} C. Sb^{3+} D. Fe^{3+} E. Mg^{2+}

23) A solution containing the cations of the V analytic group (acid-base classifi- cation) has been taken for the analysis. The solution of sodium hydroxostannite has been added to the composition which resulted in formation of black deposition. This is the evidence of presence of the following cation:

A. Bi^{3+} B. Fe^{2+} C. Sb^{3+} D. Fe^{3+} E. Mg^{2+}

24) In course of the systematic analysis separation of cations of the V and VI analytic groups (according to the acidbase classification) is carried out under the action of excess of:

A. Concentrated ammonia solution B. Sodium hydroxide solution

C. Hydrochloric acid solution D. Potassium hydroxide solution E. Sulphuric acid solution

25) After a solution had been heated with $(NH_4)_2S_2O_8$ in presence of $AgNO_3$, it turned crimson. What ions were present in the solution?

A. Mn^{2+} B. Fe^{3+} C. Fe^{2+} D. Co^{2+} E. Cu^{2+}

26) During the quantitative analysis carried out under the primary conditions, a specific reagent to Fe^{3+} cations is $K_4[Fe(CN)_6]$. Their interaction gives a precipitate of the following colour:

A. Blue B. White C. Brown D. Red E. Black

16) What reaction is applied for detection of Fe^{3+} cation?

A. Complexing B. Precipitation C. Hydrolysis D. Neutralization E. Reduction

27) A drug solution under examination contains cations of magnesium (II) and aluminium (III). Which reagent can help to separate these cations during analysis of this drug?

A. Alkali solution B. Solution of hydrogen peroxide in acidic medium

C. Solution of silver nitrate D. Ammonia solution E. Solution of chloride acid

28) Solution with NaCl mass concentration of 0,95% is a part of a saline and can be used in case of significant blood loss. Name reaction of this solution's medium:

A. Neutral (pH = 7,0) B. Acidic (pH < 7,0) C. Alkaline (pH > 7,0)

D. Very acidic (pH = 1,0) E. Very alkaline (pH = 12,0)

29) Solution of a medicinal preparation under examination contains cations of magnesium (II) and aluminium (III). What reagent will help to separate these cations during analysis of this preparation?

A. Alkali solution B. Hydrogen peroxide in acidic medium C. Argentum nitrate solution

D. Hydrogen peroxide in ammoniac medium E. Chloride acid solution

Laboratory work №5
THE 5-th ANALYTICAL GROUP OF CATIONS IDENTIFICATION

Reactions of Bi^{3+} cations

Test 1. To the test tube add 5 - 10 drops of $Bi(NO_3)_3$ solution and 1-2 drops of KI solution.

Observation:

Net ionic equation: $Bi^{3+} + 3I^{-} = BiI_3\downarrow$

Molecular equation:

To the mixture prepeared add a solution of KI till the precipitate will dissolve.

$$BiI_3\downarrow + KI = K[BiI_4]$$

Write a ionic and net ionic equation:

Test 2 (the ability to recover ions of Bi^{3+} to metallic Bi).

First step - reductant (alkaline solution of $Na_2[Sn(OH)_4]$) preparation:

To the first test tube add 3 - 5 drops of $SnCl_2$ solution and some drops of NaOH solution till the precipitate $Sn(OH)_2$ that formed at the beginning will dissolve.

$$SnCl_2 + 4NaOH = Na_2[Sn(OH)_4] + 2NaCl.$$

Write ionic and net ionic equation:

Second step - To the second test tube add 5 - 10 drops of $Bi(NO_3)_3$ solution and 3 - 5 drops of NaOH solution.

Observation:

Molecular equation: $Bi(NO_3)_3 + 3NaOH = Bi(OH)_3\downarrow + 3NaNO_3$

To the mixture prepeared in the second test tube add the reductant prepeared in the first test. Observation:

Molecular equation: $3Na_2[Sn(OH)_4] + 2Bi(OH)_3\downarrow = 3Na_2[Sn(OH)_6] + 2Bi\downarrow$

Write ionic and net ionic equation:

Reactions of Mn^{2+} cations

Test 1. To the test tube add 5 - 10 drops of manganese salt solution and 3 - 5 drops of NaOH solution.

Observation:

Net ionic equation: $Mn^{2+} + 2OH^- = Mn(OH)_2\downarrow$.

Write a *molecular equation:*

The mixture prepeared divided into two tubes.

To the first tube add H_2SO_4 solution till the precipitate $Mn(OH)_2$ dissolves.

Write a *molecular equation:*

To the second tube add 3 - 5 drops of H_2O_2 .

Observation:

The *molecular equation:* $Mn(OH)_2\downarrow + H_2O_2 = MnO(OH)_2\downarrow + H_2O$

Reactions of Mg^{2+} cations

Test 1. To the test tube add 5 - 10 drops of magnesium salt solution and 3 - 5 drops of NaOH solution.

Observation:

Ionic equation: $Mg^{2+} + 2OH^- = Mg(OH)_2\downarrow$

Write a molecular equation :

The mixture prepeared divided into two tubes.

To the first tube add a solution of H_2SO_4 till the precipitate $Mg(OH)_2$ dissolves.

$$Mg(OH)_2\downarrow + H_2SO_4 = MgSO_4 + 2H_2O$$

Write ionic and net ionic equation:

To the second test tube add NH_4Cl solution till the precipitate $Mg(OH)_2$ dissolves.

Molecular equation: $Mg(OH)_2\downarrow + 2NH_4Cl = MgCl_2 + 2NH_3 + 2H_2O$

Write ionic and net ionic equation:

Test 2. To the test tube add 5 - 10 drops of magnesium salt, 3 - 5 drops of NH_4Cl and 2 -

3 drops of NH_3. Solution of NH_4Cl is added in order to prevent the formation of precipitate $Mg(OH)_2$. After mixing add 3 - 5 drops of Na_2HPO_4.

Observation:

Molecular equation: $MgCl_2 + NH_3 + Na_2HPO_4 = MgNH_4PO_4\downarrow + 2NaCl$.

Write ionic and net ionic equation:

***T**he mixture prepeared divided into two tubes*.

To the first tube add a solution of H_2SO_4 till the precipitate $MgNH_4PO_4$ dissolves.

Net ionic equation: $MgNH_4PO_4\downarrow + 3H^+ = Mg^{2+} + NH_4^+ + H_3PO_4$

Write a *Molecular equation:*

To the second tube add a solution of acetic acid till the precipitate $MgNH_4PO_4$ dissolves.

Molecular equation:

$$2MgNH_4PO_4 + 4CH_3COOH = Mg(H_2PO_4)_2 + 2CH_3COONH_4 + Mg(CH_3COO)_2$$

Write ionic and net ionic equation:

Reactions of Fe^{2+} cations

Test 1. To the test tube add 3 - 5 drops of salt solution of iron (II) and 3 - 5 drops of NaOH solution.

Observation:

Net ionic equation: $Fe^{2+} + 2OH^- = Fe(OH)_2\downarrow$

Write a *molecular equation:*

Shake a content of the test tube:

Observation:

Molecular equation: $4Fe(OH)_2\downarrow + O_2 + 2H_2O = 4Fe(OH)_3\downarrow$

The precipitate $Fe(OH)_3$ has a brown color.

Test 2. To the test tube add 3 - 5 drops of iron (II) salt solution and 1 - 2 drops of $K_3[Fe(CN)_6]$ solution.

Observation:

Net ionic equation: $Fe^{2+} + K^{+} + [Fe(CN)_6]^{3-} = KFe[Fe(CN)_6]\downarrow$

Write a *molecular equation:*

Reactions of Fe^{3+} cations

Test 1. To the test tube add 3 - 5 drops of salt solution of iron (III) and 3 - 5 drops of NaOH solution .

Observation:

Net ionic equation: $Fe^{3+} + 3OH^{-} = Fe(OH)_3$

Write a *molecular equation:*

To the mixture prepared add nitric acid:

Observation:

Write a *molecular equation:*

Test 2. To the test tube add 3 - 5 drops of iron (III) salt solution and 1 - 2 drops of $K_4[Fe(CN)_6]$ solution.

Observation:

Net ionic equation: $Fe^{3+} + K^{+} + [Fe(CN)_6]^{4-} = KFe[Fe(CN)_6]\downarrow$

Write a *molecular equation:*

Test 3. To the test tube add 3 - 5 drops of iron (III) salt solution and 1 - 2 drops of NH_4SCN solution. As a result the red solubile complexes formed. The composition of complex depends on the ratio of the components, for example: $Fe^{3+} + 3SCN^{-} = [Fe(SCN)_3]$. The following complex ions: $[Fe(SCN)_4]^{-}$, $[Fe(SCN)_5]^{2-}$, $[Fe(SCN)_6]^{3-}$ can form too.

Practical lesson 5

Sixth group of cations. Laboratory work "Analytical reactions of sixth analytical group of cations". Situational tasks and learning exercises. Test control.

These questions are taken from the Test items for licensing examination Krok 1 PHARMACY
The correct answer is first

6-th group of cations

1) What cations added to the solution of potassium iodide form orange-red deposition that is soluble in reagent excess and builds up a colourless solution?
   A. Mercury (II) B. Mercury (I) C. Bismuth D. Antimony (V) E. Lead
2) A compound undeer examination contains cations of iron (III) and copper (II). What group reagent can separate these cations?
   A. Concentrated ammonia solution B. Solution of sodium hydroxide and hydrogen peroxide
   C. Concentrated solution of hydrochloride acid D. Solution of sodium hydroxide
   E. Concentrated solution of sulfuric acid
3) Excess of ammonia was added to a solution under examination. The solution turned bright blue. This indicates presence of the following ions:
   A. Copper B. Silver C. Lead D. Bismuth E. Mercury (II
4) Dimethyl glyoxime entered into reaction with a solution that contained cations of the IV analytical group (acid-base classification). The deposition turned crimson. What cation caused this analytical effect?
   A. Nickel cation (II) B. Mercury cation (II) C. Copper cation (II)
   D. Cadmium cation (II) E. Cobalt cation (II)
5) At a chemical analytical laboratory, a technician examines a solution of the VI analytical group cations. After the addition of ammonium thiocyanate and amyl alcohol, the organic layer turned blue. What cation is present in the solution?
   A. Co^{2+} B. Ni^{2+} C. Cu^{2+} D. Hg^{2+} E. Cd^{2+}
6) In course of the systematic analysis separation of cations of the V and VI analytic groups (according to the acidbase classification) is carried out under the action of excess of:
   A. Concentrated ammonia solution B. Sodium hydroxide solution
   C. Hydrochloric acid solution D. Potassium hydroxide solution
   E. Sulphuric acid solution

7) In a solution containing cations of copper (II) and zinc, the copper cations can be identified by means of the excess of the following reagent:

A. 6M ammonia solution B. 2M sulfuric acid solution C. 6M potassium hydroxide solution

D. 2M hydrochloric acid solution E. 2M solution of ammonium carbonate

8) The ability of reagent to ensure a stable analytical effect in the interaction with the analyzed substance is characterized by:

A. Reaction sensitivity B. Reaction selectivity C. Reaction specificity

D. Reagent amount E. –

9) While detecting Co^{2+} ions in presence of Fe^{3+} the following ions should be added to the solution in order to mask Fe^{3+} ions:

A. Fluoride ions B. Chloride ions C. Bromide ions D. Nitrite ions E. Sulphate ions

10) What cation can be detected with Chugaiev's agent (Dimethylglyoxime)?

A. Ni^{2+} B. Ca^{2+} C. K^{+} D. Mn^{2+} E. Co^{2+}

11) Potassium iodide solution has been added to the solution containing cations of the sixth analytical group (acidbase classification). It resulted in red precipitate soluble in excess of reagent. What cations are present in the solution?

A. Mercury (II) B. Nickel C. Cobalt (II) D. Bismuth E. Cadmium

12) Cations Cu^{2+}, Co^{2+}, Ni^{2+}, Cd^{2+}, Hg^{2+} belong to the sixth group of cations. What is the group reagent for the sixth group of cations?

A. Excess of NH_3 B. Solution of H_2SO_4 C. Solution of NaOH

D. Excess of KOH solution E. Solution of HCl

13) An excess of concentrated ammonium hydroxide is a group reagent for the cations of the VI analytical group (acid-base classification), namely Co^{2+}, Ni^{2+}, Cd^{2+}, Cu^{2+}, Hg^{2+}. As a result of this reaction the following substances are formed:

A. Water-soluble ammonia complexes B. Hydroxides of acid-soluble cations

C. Stained water-insoluble compounds D. Hydroxides of alkali-soluble cations

E. Hydroxides of the cations insoluble in the excess of ammonium hydroxide

14) In a qualitative analysis, when an excess of the group reagent (NH_3 solution) reacts with the cations of the sixth analytical group (Cu^{2+}, Co^{2+}, Ni^{2+}, Cd^{2+}, Hg^{2+}), the following compounds are formed:

A. Metal ammine complexes B. Metal hydroxides C. Basic metal salts

D. Aqua complexes of metals E. Hydroxocomplexes of metals

15) What reagents are used to separate the cations of the IV analytical group from the cations of the V and VI analytical groups in the analysis of their composition?

A. NaOH and H_2O_2 B. H_2SO_4 C. Dithizone D. NH_4OH E. Na_2S

16) To maintain a certain level of pHmedium, the buffer solutions are used. Specify a composition of substances that DOES NOT EXHIBIT buffer properties:

A. NaOH + NaCl `B. CH_3COOH + CH_3COONa C. NH_4Cl + $NH_3 \cdot H_2O$

D. HCOOH + HCOONa E. NaH_2PO_4 + Na_2HPO_4

17) What reagent is used to separate the cations of copper (II) and mercury from the other cations of the VI analytical group?

A. Sodium thiosulfate B. Sodium sulfate C. Bromine water

D. Potassium sulfide E. Excess of the concentrated ammonia solution

18) An excess of concentrated ammonium hydroxide is a group reagent for the cations of the VI analytical group (acidbase classification) Co^{2+}, Ni^{2+}, Cd^{2+}, Cu2+, Hg^{2+}. In this case the following substances are formed:

A. Water-soluble ammonia complexes B. Hydroxides of acid-soluble cations

C. Stained, water-insoluble compounds D. Hydroxides of alkali-soluble cations

E. Hydroxides of the cations insoluble in the excess of ammonium hydroxide

19) Dimethyl glyoxime entered into reaction with a solution that contained cations of the IV analytical group (acid-base classification). The deposition turned crimson. What cation caused this analytical effect?

A. Nickel cation (II) B. Mercury cation (II) C. Copper cation (II)

D. Cadmium cation (II) E. Cobalt cation (II)

Laboratory work №6

THE 6-th ANALYTICAL GROUP OF CATIONS IDENTIFICATION

Reactions of Cu^{2+}cations

Test 1. To the test tube add 3 - 5 drops of solution copper salt, 1 - 2 drops of NH_3, mix and shake tube.

Observation:

Molecular equation: $2CuSO_4 + 2NH_3 + 2H_2O = (CuOH)_2SO_4\downarrow + (NH_4)_2SO_4$

Write an ionic and a net ionic equation:

The mixture prepeared divided into two tubes.

To the first tube slowly add drop by drop solution NH_3 to precipitate dissolve.

Observation:

$$(CuOH)_2SO_4\downarrow + (NH_4)_2SO_4 + 6NH_3 = 2[(Cu(NH_3)_4]SO_4 + 2H_2O$$

Write an ionic and a net ionic equation:

To the second tube add of H_2SO_4 till the precipitate dissolves.

Molecular equation: $(CuOH)_2SO_4\downarrow + H_2SO_4 = 2CuSO_4 + 2H_2O$

Write ionic and net ionic equation:

Test 2. To the test tube add 1 - 2 drops of copper salt solution, 5 - 10 drops of NaOH solution.
Observation:
Write a *molecular equation:*

The mixture in a test tube carefully heat.
Observation:

Molecular equation: $Cu(OH)_2 \overset{T}{=} CuO + H_2O$

Test 3. To the test tube add 1 - 2 drops of copper salt solution, 1 - 2 drops of H_2SO_4 solution and 5 - 10 drops of $Na_2S_2O_3$ solution. Then heat to boiling.
Observation:

Ionic equation: $2Cu^{2+} + 2S_2O_3^{2-} + 2H_2O = Cu_2S + S + 2SO_4^{2-} + 4H^+$
Write a *molecular equation:*

Test 4. To the test tube add 3 - 5 drops of copper salt solution and gently immerse an

iron nail. After 1 - 2 minutes get out a nail. Pay attention to the color of the surface of an iron nail that contacted with a solution of copper salt.
Observation:

$$CuSO_4 + Fe = FeSO_4 + Cu\downarrow$$

Write ionic and net ionic equation.

Test 5. To the test tube add 3 - 5 drops of solution copper salt and 1 - 2 drops of $K_4[Fe(CN)_6]$.

Observation:

Molecular equation: $2CuSO_4 + K_4[Fe(CN)_6] = Cu_2[Fe(CN)_6]\downarrow + 2K_2SO_4$
Write ionic and net ionic equation:

Reactions of Cd^{2+} cations

Test 1. To the test tube add 3 - 5 drops of cadmium salt solution, 1 drop of NH_3 solution.
Observation:

Ionic equation: $Cd^{2+} + 2NH_3 + 2H_2O = Cd(OH)_2\downarrow + 2\ NH_4^+$
Write a *molecular equation:*

Test 2. To the white precipitate $Cd(OH)_2$ prepared in an *Test 1* add a solution of NH_3 till precipitate dissolves.
Ionic equation: $Cd(OH)_2\downarrow + 4NH_3 = [Cd(NH_3)_4]^{2+} + 2OH^-$
Write a *molecular equation:*

Test 3. To the test tube add 2 - 3 drops of cadmium salt, 2 - 3 drops of of $Na_2S_2O_3$, heat mixture to boiling. Does observed changes in the solution? (Compare with similar experiments for $CuSO_4$).
Observation:

(Unlike of ions Cu^{2+} Cd^{2+} ions does not form a precipitate with solution of

$Na_2S_2O_3$.)

Reactions of Ni^{2+} cations

Test 1. To the test tube add 3 - 5 drops of solution nickel salt, add 1-2 drops of NH_3 solution to precipitate forms. What is the color of precipitate?

Molecular equation: $NiCl_2 + NH_3 + H_2O = NiOHCl\downarrow + NH_4Cl$.

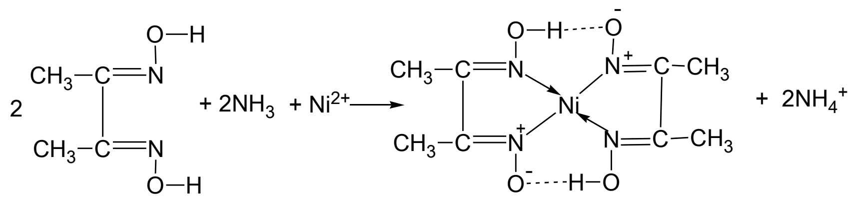
Write ionic and net ionic equation

Continue adding NH_3 till the precipitate dissolves. What is the color of solution formed?

Molecular equation: $NiOHCl\downarrow + NH_4Cl + 5NH_3 = [Ni(NH_3)_6]Cl_2 + H_2O$.

Write ionic and net ionic equation

Test 2. On a filter paper put 1 drop of nickel salts, put 1 drop of NH_3 solution and 1 drop of solution diacetyldihydroxyme.
Molecular equation:



Reactions of Co^{2+} cations

Test 1. To the test tube add 2 - 3 drops of cobalt salts solution; add 1-2 drops of NH_3 solution to the precipitate formation. What color of precipitate is?

Molecular equation: $CoCl_2 + NH_3 + H_2O = CoOHCl\downarrow + NH_4Cl$.

Write ionic and net ionic equation

Continue adding NH_3 solution to dissolve the precipitate. What color of the resulting solution?

Molecular equation: $CoOHCl\downarrow + NH_4Cl + 5NH_3 = [Co(NH_3)_6]Cl_2 + H_2O$.

Write ionic and net ionic equation:

To the solution obtained add 5 - 10 drops of solution H_2O_2.

Observe the cherry-red color of complex compound $[Co(NH_3)_5Cl]Cl_2)$:

Molecular equation:

$$2[Co(NH_3)_6]Cl_2 + H_2O_2 + 4HCl = 2[Co(NH_3)_5Cl]Cl_2 + 2NH_4Cl + 2H_2O.$$

Write ionic and net ionic equation:

Reactions of Hg^{2+} cations.

Test 1. To the test tube add 3 - 4 drops of salt solution of mercury (II), add a solution of NH_3 to the precipitate formation.
Molecular equation: $2Hg(NO_3)_2 + 4NH_3 + H_2O = [OHg_2NH_2]NO_3\downarrow + 3NH_4NO_3$.
Write ionic and net ionic equation:

To the mixture obtained add 3 - 4 drops of ammonium salt solution (NH_4Cl or NH_4NO_3). Continue adding of NH_3 solution till precipitate dissoves.

Molecular equation: $[OHg_2NH_2]NO_3\downarrow + 3NH_4NO_3 + 4NH_3 = 2[Hg(NH_3)_4](NO_3)_2 + H_2O$

Write ionic and net ionic equation

Test 2. To the test tube add 1 drop of mercury (II) salt solution; add 3 - 5 drops of NaOH solution. What color of precipitate is?
Molecular equation: $Hg(NO_3)_2 + 2NaOH = HgO\downarrow + 2NaNO_3 + H_2O$

Write ionic and net ionic equation

Test 3. To the test tube add 3 - 5 drops of mercury (II) salt solution; add 1 drop of dilute KI solution. What color of precipitate is?

Ionic reaction equation: $Hg^{2+} + 2I^{-} = HgI_2\downarrow$

Write a *molecular equation:*

Continue adding of KI solution till precipitate dissolves.

Observation:

Ionic reaction equation: $HgI_2\downarrow + 2I^{-} = [HgI_4]^{2-}$;

Write a *molecular equation:*

Test 4. The reaction of "copper coins". On a clean copper plate place 1 drop of mercury (II) salt solution.

Observation:

The equation reaction: $Hg(NO_3)_2 + Cu = Cu(NO_3)_2 + Hg\downarrow$.

Write ionic and net ionic equation

Practical lesson 6

Semantic modules 1- 2 control: the theory and practice of analysis of cations of I-VI analytical groups. Laboratory work "The analysis of cations I-VI analytical groups mixture". Writing control work and test.

Practical lesson 7

Group reagents are in the analysis of anions. General characteristics of anions. Analytical reactions of anions I and II of analytical groups. Laboratory works "Analytical reactions of the First analytical group of anions "and "Analytical reactions of the Second analytical group of anions ". Test control.

These questions are taken from the Test items for licensing examination Krok 1 PHARMACY

<u>The correct answer is first</u>

1-st group of anions

1) Choose the reagents for detection of the sulphate ions in a solution containing carbonate, sulphate and phosphate ions:

A. $Ba(NO_3)_2$, HCl B. $Ba(NO_3)_2$, NaOH C. $BaCl_2$, H_2O D. $CaCl_2$, NH_4OH E. $AgNO_3$,HNO_3

2) Silver nitrate solution has been added to the solution containing anions of the fi-rst analytical group. It resulted in yellow precipitate. That means the following are present in the solution:

A. Arsenite ions B. Arsenate ions C. Sulphate ions D. Iodide ions E. Bromide ions

3) A solution of magnesium mixture was added into solution with anions of the 1st analytical group. White crystalline precipitate was produced. What anions cause such analytical effect?

A. PO_4^{3-} and AsO_4^{3-} B. AsO_3^{3-} C. $S_2O_3^{2-}$ D. SO_3^{2-} E. $C_2O_4^{2-}$

4) Silver nitrate solution has been added to the solution containing anions of the first analytical group. If Silver nitrate solution has been added to the solution containing anions of the first analytical group. Itresulted in yellow precipitate. That means the following are present in the solution:

A. Arsenite ions B. Arsenate ions C. Sulphate ions D. Iodide ions E. Bromide ions

5) Selective solvents are used in laboratories and factories to isolate and refine essential oils, alkaloids, antibiotics and other pharmaceutical substances. This process is called:

A. Extraction B. Sedimentation C. Coagulation D. Flocculation E. Flotation

6) Sodium arsenate solution can be distinguished from the arsenite solution by means of the following reagent:

A. Magnesia mixture B. Potassium sulphate C. Potassium nitrate

D. Sodium chloride E. Sodium fluoride

7) In order to determine CO_2 in air the following substance can be applied:

A. Water solution $Ca(OH)_2$ B. Water solution NaOH C. CaO

D. $Fe(OH)_2$ E. Crystalline NaOH

8) Choose reagents for detection of sulphate-ions in a solution that contains carbonate-, sulfate-, thiosulfate-, phosphate-anions:

A. $Ba(NO_3)_2$, HCl B. $Ba(NO_3)_2$, NaOH C. $BaCl_2$, H_2O D. $CaCl_2$, NH_4OH E. $AgNO_3$, HNO_3

Laboratory work № 8

THE 1-st ANALYTICAL GROUP OF ANIONS IDENTIFICATION

Reactions of SO_4^{2-} anions

Test 1. To the test tube add 3 - 5 drops of salt contains SO_4^{2-} anions solution, 2 - 3 drops of $BaCl_2$ solution. Observation:

Net ionic equation: $SO_4^{2-} + Ba^{2+} = BaSO_4\downarrow$

Molecular equation:

Test 2. To the test tube add 1 - 2 drops of salt contains SO_4^{2-} anions solution, add 1 - 2 drops of $Pb(NO_3)_2$ solution. Observation:

Net ionic equation: $SO_4^{2-} + Pb^{2+} = PbSO_4\downarrow$

Ionic equation:

Molecular equation:

The mixture prepeared divided into two tubes.

To the first tube add 10 - 15 drops HNO_3 solution.

Observation:

To the second tube add 10 - 15 drops of NaOH solution and heat.

Observation:

Note that precipitate $PbSO_4$ dissolves only in NaOH solution and insoluble in nitric acid (***distinction from other anions of the first analytical group for which the lead salts are soluble in nitric acid***).

Molecular equation: $PbSO_4\downarrow + 4NaOH = Na_2[Pb(OH)_4] + Na_2SO_4$

Ionic and net ionic equations:

Reactions of SO_3^{2-} anions

Test 1. To the test tube add 1 - 2 drops of Na_2SO_3 solution, add 2 - 3 drops of HCl solution, heat, determine the presence of SO_2 by smell.

Observation:

Net ionic equation: $SO_3^{2-} + 2H^+ = H_2SO_3 \rightarrow SO_2\uparrow + H_2O$

Ionic equation:

Molecular equation:

Test 2. To the test tube add 3 - 5 drops of Na_2SO_3 solution, add 2 - 3 drops of H_2SO_4 solution and 1 drop of $KMnO_4$ solution. Observe discolour of *red-violet* solution of potassium permanganate.

Molecular equation:

$$5K_2SO_3 + 2KMnO_4 + 3H_2SO_4 = 6K_2SO_4 + 2MnSO_4 + 3H_2O$$

$$2 \mid \overset{+7}{Mn} + 5e = \overset{+2}{Mn}$$

$$5 \mid \overset{+4}{S} - 2e = \overset{+6}{S}$$

$$10$$

Ionic and net ionic equations:

Reactions of CO_3^{2-} anions

Test 1. To the test tube add 1 - 2 drops of Na_2CO_3 solution, add 1 drop of $MgSO_4$ or $MgCl_2$ solution.

Observation:

Net ionic equation: $CO_3^{2-} + Mg^{2+} = MgCO_3\downarrow$

Ionic equation:

Molecular equation:

To the precipitate add solution of HCl.

Observation:

Molecular equation:

Test 2. To the test tube add 3 - 5 drops of Na_2SO_3 solution, add 1 - 3 drops of HCl solution.

Observe the bubbles of gas CO_2 formation.

Ionic equation: $CO_3^{2-} + 2H^+ = CO_2 + H_2O$

Ionic equation:

Molecular equation;

Reactions of PO_4^{3-} anions

Test 1. To the test tube add 1 - 2 drops of salt of phosphoric acid, add 6 - 7 drops of concentrated HNO_3, 9 - 10 drops of concentrated ammonium molybdate solution and heat to a temperature ≈ 40-50ºC. Observe the appearance of the yellow color of the solution, and then formation of a yellow precipitate: $(NH_4)_3[PO_4(MoO_3)_{12}]$, or $(NH_4)_3[P(Mo_{12}O_{40})]$, or $(NH_4)_3H_4[P(Mo_2O_7)_6]$.

Net ionic equation:

$$PO_4^{3-} + 3NH_4^+ + 12MoO_4^{2-} + 24H^+ = (NH_4)_3[PO_4(MoO_3)_{12}] + 12H_2O$$

Ionic equation:

Molecular equation:

Net ionic equation:

$$PO_4^{3-} + 3NH_4^+ + 12MoO_4^{2-} + 24H^+ = (NH_4)_3H_4[P(Mo_2O_7)_6] + 10H_2O$$

Molecular equation:

Test 2. To the test tube add 1 - 2 drops of Na_3PO_4, add 1 drop of NH_3 and NH_4Cl solutions, mix and add 1 drop of $MgCl_2$ solution.

Observation:

Molecular equation: $Na_3PO_4 + MgCl_2 + NH_4Cl = MgNH_4PO_4\downarrow + 3NaCl$

Ionic equation:

Test 3. To the test tube add 3 - 5 drops of Na_3PO_4 solution, add 1 - 2 drops of $AgNO_3$ solution.

Observation:

Ionic equation: $3Ag^+ + PO_4^{3-} = Ag_3PO_4\downarrow$

Ionic equation:

Molecular equation:

The mixture prepeared divided into two tubes.

To the first tube add a solution of NH_3 till precipitate dissolves**.**

Net ionic equation: $Ag_3PO_4 + 6NH_3 = 3[Ag(NH_3)_2]^+ + PO_4^{3-}$

Ionic equation:

Molecular equation:

To the second test tube add a solution of HNO_3 till precipitate dissolves**.**

Net ionic equation: $Ag_3PO_4 + 3H^+ = 3Ag^+ + H_3PO_4$

Ionic equation:

Molecular equation:

Reactions of $S_2O_3^{2-}$ anions

Test 1. To the test tube add 3 - 5 drops of $Na_2S_2O_3$ solution, add 3 - 5 drops of HCl solution, mix by shaking the tube.

Observation:

Molecular equation: $Na_2S_2O_3 + 2HCl = 2NaCl + S\downarrow + SO_2\uparrow + H_2O$.

Ionic and net ionic equation:

Test 2. To the test tube add 1 drop of $Na_2S_2O_3$ solution and 1 - 2 drops of $AgNO_3$ solution. What color is the precipitate formed at the beginning?

Net ionic equation: $2Ag^+ + S_2O_3^{2-} = Ag_2S_2O_3\downarrow$

Ionic equation:

Observe gradual change of precipitate color from white to brow-black.

Molecular equation: $Ag_2S_2O_3\downarrow + H_2O = Ag_2S\downarrow + H_2SO_4$

Test 3. To the test tube add 1 drop of $Na_2S_2O_3$ solution, add 1 - 2 drops of $AgNO_3$ solution. To the precipitate add some drops of $Na_2S_2O_3$ solution till precipitate dissolves**.**

Net ionic equation: $Ag_2S_2O_3\downarrow + 3S_2O_3^{2-} = 2[Ag(S_2O_3)_2]^{3-}$

Ionic equation:

Molecular equation:

Test 4. To the test tube add 3 - 5 drops of I_2 solution; add some drops of $Na_2S_2O_3$ solution to discolouration of I_2.

Net ionic equation: $2S_2O_3^{2-} + I_2 = S_4O_6^{2-} + 2I^-$

Ionic equation:

Molecular equation:

Reactions of $C_2O_4^{2-}$ anions

Test 1. To the test tube add 2 - 3 drops of $(NH_4)_2S_2O_4$ solution, add 1 - 2 drops of $CaCl_2$ solution. Observation:

Net ionic equation: $C_2O_4^{2-} + Ca^{2+} = CaC_2O_4\downarrow$

Ionic equation:

Molecular equation:

The mixture prepeared divided into two tubes.

To the first tube add 3 - 5 drops of CH_3COOH.

Observation:

Molecular equation:

Ionic equation:

Net ionic equation:

To the second test tube add 3 - 5 drops of HCl.

Observation:

Molecular equation:

Ionic equation:

Net ionic equation:

Test 2. To the test tube add 3 - 5 drops of $(NH_4)_2S_2O_4$ solution, add 3 - 5 drops of H_2SO_4 solution and 1 drop of $KMnO_4$ solution. Pay attention to the fact that without heating the reaction practically does not takes place, and the pink color of $KMnO_4$ not disappears.

Observation:

Net ionic equation: $2MnO_4^- + 5C_2O_4^{2-} + 16H^+ = 10CO_2 + 2\ Mn^{2+} + 8H_2O$

Ionic equation:

Molecular equation:

Reactions borate anions

Test 1. In a porcelain cup place some crystals of $Na_2B_4O_7 \cdot 10H_2O$, add 5 - 6 drops of concentrated H_2SO_4 and 10 - 20 drops of C_2H_5OH. Mix dy glass stick.

The reactions: $Na_2B_4O_7 \cdot 10H_2O + H_2SO_4 = 4H_3BO_3 + Na_2SO_4 + 5H_2O$;

$H_3BO_3 + 3C_2H_5OH = (C_2H_5O)_3B + 3H_2O$.

Set fire to the alcohol. What is the color of the flame?

Molecular equation: $2(C_2H_5O)_3B + 18O_2 = B_2O_3 + 12CO_2 + 15H_2O$

Reactions of fluoride anions

Test 1. To the test tube add 2 - 3 drops of NaF solution, add 1 drop of $CaCl_2$ solution.

Observation:

Net ionic equation: $2F^{-} + Ca^{2+} = CaF_2\downarrow$

Ionic equation:

The mixture prepeared divided into two tubes. To the first tube add 5 - 10 drops of HCl solution.

Observation:

Molecular equation: $CaF_2\downarrow + 2HCl = CaCl_2 + 2HF$

Ionic and net ionic equations:

To the second test tube add 5 - 10 drops of CH_3COOH solution. Is there dissolution of the precipitate?

Observation:

Test 2. To the test tube add 1 drop of $FeCl_3$ solution and 1 drop of NH_4SCN solution.

Observation:

Dilute 2 - 3 times the solution prepeared and add dropwise NaF solution to bleaching of solution.

The reactions: $FeCl_3 + 6NH_4SCN = (NH_4)_3[Fe(SCN)_6] + NH_4Cl$

$(NH_4)_3[Fe(SCN)_6] + 6NaF = Na_3[FeF_6] + 3NaSCN + 3NH_4SCN$

Explanation: *The complex compound* $(NH_4)_3[Fe(SCN)_6]$ *(red color) transformed into more stable colorless complex compound* $Na_3[FeF_6]$*. So solution becomes colorless.*

These questions are taken from the Test items for licensing examination Krok 1 PHARMACY

The correct answer is first

2-nd group of anions

1) Argentum nitrate solution was added to a solution containing anions of the second analytical group. It resulted in generation of light yellow precipitate that was unsoluble in nitric acid and partly soluble in ammonium solution. What anions are contained in the solution?

A. Bromide-ions B. Iodide-ions C. Chloride ions
D. Sulfide-ions E. Arsenite-ions

2) Analytical indication of effect of potassium iodide solution upon unstained oxidizing anions in presence of chloroform is:
A. Brown stain of free iodine B. Settling down of white deposition
C. Change of aggregate state D. Emission of gas bubbles
E. Origination of deposition and its solution in reagent excess

3) A composition under examination contains ions of Cl , Br and I in equimolar quantities. The sequence of precipitate formation in course of argentometric titration will be determined by:
A. Solubility product of the corresponding silver halogenides
B. Value of oxidation-reduction potentials C. Way of titration - either back or direct
D. Value of corresponding ion mobility E. Ionic strength of solution

4) Solution under analysis received chloroform and, drop by drop, chlorine water. Chloroform layer colored orange, which indicates the presence of:
A. Bromide ions B. Iodide ions C. Sulfite ions D. Sulfate ions
E. Nitrate ions

5) What reagent can help distinguish between starch and glucose?
A. I_2 B. Br_2 C. $KMnO_4$ D. $K_2Cr_2O_7$ E. F eCl_3

6) What anion of the 2nd analytic group produces black precipitate with group reagent $AgNO_3$?
A. S^{2-} B. I^- C. Cl^- D. Br^- E. NCS^-

7) Medicines used in treatment of dental caries contain sodium fluoride. Which one of the compounds given below does NaF react with?
A. H_2SO_4 B. CO_2 C. NaCl D. KI E. CH_3COOH

8) What reagent is used to separate AgCl precipitate from AgI precipitate?
A. Aqueous solution of ammonia B. Concentrated nitric acid C. Diluted nitric acid
D. Concentrated solution of potassium chloride E. Sulfuric acid solution

9) What anion of the 2nd analytic group produces black precipitate with group reagent $AgNO_3$?
A. S^{2-} B. I^- C. Cl^- D. Br^- E. NCS^-

10) Group reagent of the second analytical group anions (Cl^-,Br^-,I^-, S^{2-}) is the solution of AgNO3 with the addition of the following substance:
A. Nitric acid B. Hydrobromic acid C. Acetic acid
D. Hydrochloric acid E. Hydrosulphuric acid

11) A solution contaning anions of the second analytical group has been blended with the solution of argentum nitrate. This resulted in formation of black

precipitate insoluble in the ammonia solution and soluble in the diluted nitric acid at heating. What anions are present in the solution?

A. Sulphide ions B. Iodide ions C. Chloride ions

D. Bromide ions E. Arsenite ions

12) Analytical indication of effect of potassium iodide solution upon unstained oxidizing anions in presence of chloroform is:

A. Brown stain of free iodine B. Settling down of white deposition C. Change of aggregate state

D. Emission of gas bubbles E. Origination of deposition and its solution in reagent excess

Laboratory work № 9

THE 2-nd ANALYTICAL GROUP OF ANIONS IDENTIFICATION

Reactions of Cl^- anions

Test 1. To the test tube add 3 - 5 drops of solution containing anions Cl^- , add 3 - 5 drops of HNO_3 solution and 1 - 2 drops of $AgNO_3$ solution.
Observation:

Net ionic equation: $Cl^- + Ag^+ = AgCl\downarrow$

Ionic equation:

Molecular equation:

Check the solubility of the precipitate formed in nitric acid and ammonia solution. Pay attention on fact that AgCl insoluble in nitric acid but the NH_3 solution forms soluble complex salt $[Ag(NH_3)_2]Cl$.
Write the reaction of AgCl with NH_3

Test 2. To the test tube add 1 - 2 drops of salts containing anions Cl^- solution, add 3 - 5 drops of concentrated sulfuric acid and 2 - 4 drops of $KMnO_4$ solution. Heat the mixture carefully. The discoloration of solution and the formation of gaseous chlorine are observed. Free chlorine formed can be determined by ***iodine-starch*** paper or because of characteristic odor.
Net ionic equation: $10Cl^- + 2MnO_4^- + 16H^+ = 2Mn^{2+} + 5Cl_2 + 8H_2O$

Ionic equation:

Molecular equation:

Explanation: I_2, formed in result of interaction of molecular chlorine and ***iodine-starch*** paper contains KI ($Cl_2 + 2KI = 2KCl + I_2$) react with starch and blue color appear.

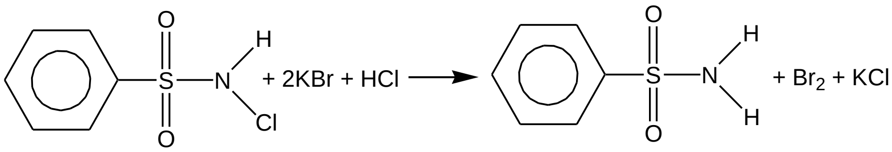
Ionic equation:

Net ionic equation:

Reactions of Br^- anions

Test 1. To the test tube add 2 - 3 drops of KBr solution, add 2 - 3 drops of HCl solution and the same solution of ***chloramine***.

The equation of the reaction:



To the mixture add 5 - 10 drops organic solvent, shake mixure. Observe the appearance of a yellow-brown color of the organic solvent. Explain the analytical effect:

Test 2. To the test tube add 5 - 10 drops of KBr solution, add 2 - 3 drops of $AgNO_3$ solution. Observation:

The mixture prepeared divided into two tubes.

To the first tube add 10 - 15 drops of NH_3 solution.

Observation:

To the second tube add 10 - 15 drops of $Na_2S_2O_3$ solution.

Observation:

AgBr slightly soluble in NH_3 and easily soluble in $Na_2S_2O_3$:

$$AgBr\downarrow + 2Na_2S_2O_3 = Na_3[Ag(S_2O_3)_2] + NaBr$$

Ionic equation:

Net ionic equation:

Reactions of I^- anions

Test 1. To the test tube add 3 - 5 drops KI solution, add 5 - 10 drops KNO_2 and the same solution of H_2SO_4

$$2KI + 2KNO_3 + 2H_2SO_4 = I_2 + 2NO + 2K_2SO_4 + 2H_2O$$

Ionic equation:

Net ionic equation:

To the solution prepeared add 5 - 10 drops of organic solvent and shake the mixture. Observe the red-violet color of the organic solvent. Explain this effect:

Test 2. To the test tube add 3 - 5 drops of KI solution, add 1 - 2 drops of $AgNO_3$ solution.

Observation:

Net ionic equation: $I^- + Ag^+ = AgI\downarrow$

Ionic equation:

Molecular equation:

The mixture prepeared divided into two tubes.

To the first tube add 10 - 15 drops of NH_3 solution.

Observation:

To the second tube add 10 - 15 drops of $Na_2S_2O_3$ solution.

Observation:

Write a molecular equation: (a soluble complex compound $Na_3[Ag(S_2O_3)_2]$ forms)

Ionic equation:

Net ionic equation:

Reactions of SCN^- anions

Test 1. To the test tube add 3 - 5 drops of NH_4SCN solution, add 1 drop of $FeCl_3$ solution. What is the color of the solution?

Molecular equation: $6NH_4SCN + FeCl_3 = (NH_4)_3[Fe(SCN)_6] + 3NH_4Cl$

Ionic equation:

Net ionic equation:

Practical lesson 8

Analytical reactions of anions of the III analytical group and anions of organic acids. Laboratory work "Reactions of anions of the III analytical group. Qualitative reactions of anions of organic acids ". Solving settlement and situational problems. Test control. Written control work.

These questions are taken from the Test items for licensing examination Krok 1 PHARMACY
The correct answer is first

3-rd group of anions

1) Choose reagents for detection of nitrite ions in presence of nitrate ions contained in a pharmaceutical under examination:
   A. Antipyrin and chlorohydrogen acid (diluted)
   B. Iron (II) sulfate (diluted) and potassium iodide
   C. Iron (III) sulfate (concentrated) and potassium bromide
   D. Iron (II) chloride E. Iron (III) chloride
2) A solution under examination was added to the solution of $FeSO_4$ in presence of concentrated H_2SO_4. Generation of a brown ring indicates presence of:
   A. Nitrate ions B. Acetate ions C. Carbonate ions
   D. Oxalate ions E. Phosphate ions
3) Solution of potassium iodide was added to the solution acidated with sulfate acid that contained anions of the third analytical group. Release of free iodine is observed. What anion are present in the solution?
   A. Nitrite ion B. Carbonate ion C. Sulfate ion
   D. Bromide ions E. Acetate ions
4) Nitrite ions in presence of nitrate ions can be detected with:

A. Crystalline antipyrine in presence of diluted HCl B. Crystalline sodium thiosulfate

C. Dimethylglyoxime D. Crystalline iron (III) sulfate E. Diphenylcarbazone

5) In a chemical analytical laboratory a chemist investigates a solution of anion mixture. When antipyrin solution is added it colors emerald-green. This analytical effect signifies presence of the following anions:

A. Nitrite B. Nitrate C. Acetate D. Tartrate E. Citrate

6) Nitrite ions in presence of nitrate ions can be detected by means of:

A. Crystalline antipyrine in presence of dilute HCl B. Crystalline sodium thiosulfate

C. Dimethylglyoxime D. Crystalline iron (III) sulfate E. Diphenylcarbazone

7) What anions form brown ring with iron (II) salts in the presence of concentrated sulfuric acid?

A. Nitrate ions B. Acetate ions C. Bromate ions

D. Citrate ions E. Thiocyanate ions

8) In a chemico-analytical laboratory a dispensing chemist studies the solution of anion mixture. When antipyrin solution is added to the solution, it becomes emeraldgreen in colour. This analytical effect signi- fies presence of the following anions:

A. Nitrite B. Nitrate C. Acetate D. Tartrate E. Citrate

9) In the chemico-analytical laboratory the dispensing chemist studies solution of anion mixture. When antipyrin solution is added it becomes emerald-green colored. This analytical effect signifies presence of the following anions:

A. Nitrite B. Nitrate C. Acetate D. Tartrate E. Citrate

10) Identical analytical effect is observed when NO^{3-} and NO^{2-} ions interact with:

A. Diphenylamine and concentrated H_2SO_4 B. Solution of $KMnO_4$

C. Solution of I_2 y KI D. Solution of $AgNO_3$ E. Solution of $BaCl_2$

11) Nitrite ions in presence of nitrate ions can be detected by means of:

A. Crystalline antipyrine in presence of dilute HCl B. Crystalline sodium thiosulfate

C. Dimethylglyoxime D. Crystalline iron (III) sulfate E. Diphenylcarbazone

12) For determination of nitrate ions diphenylamine was added to the solution under examination. The following changes were observed:

A. Generation of blue solution B. Generation of yellow deposition

C. Generation of blue deposition D. Generation of brown gas
E. Emergence of a typical smell

13) A solution under examination was added to the solution of $FeSO_4$ in presence of concentrated H_2SO_4. Formation of a brown ring indicates presence of:
A. Nitrate ions B. Acetate ions C. Carbonate ions D. Oxalate ions E. Phosphate ions

14) Qualitative determination of the following compound is accompanied by blue stain of the ether layer:
A. H_2O_2 B. Cl_2 C. Na_2HPO_4 D. $MnSO_4$ E. $FeSO_4$

15) For determination of nitrate ions diphenylamine was added to the solution under examination. The following changes were observed:
A. Generation of blue solution B. Generation of yellow deposition
C. Generation of blue deposition D. Generation of brown gas
E. Emergence of a typical smell

THE 3-d ANALYTICAL GROUP OF ANIONS IDENTIFICATION

Reactions of NO_3^- anions

Test 1. To the test tube add 2 - 3 drops of KNO_3 solution, add 5 - 10 drops of H_2SO_4 (conc). Immerse a copper wire into a mixture. Carefully heat test tube. What color is gas formed?

Complete the reaction and balance:

$$K\overset{+5}{N}O_3 + H_2SO_4 + \overset{0}{Cu} = \overset{+4}{N}O_2\uparrow + \overset{+2}{Cu}SO_4 + K_2SO_4 + ...$$

$$\begin{array}{c|l} 2 & \overset{+5}{N} + 1e = \overset{+4}{N} \\ 1 & \overset{0}{Cu} - 2e = \overset{+2}{Cu} \\ \hline & \quad 2 \end{array}$$

Test 2. To the test tube add 2 - 3 drops of $NaNO_3$ solution, add 10 - 15 drops of NaOH (conc). Immerse a pellet of Zn in a mixture. Heat. Identify the smell of gas formed.
The reaction equation

$$Na\overset{+5}{N}O_3 + 4\overset{0}{Zn} + 7NaOH + 6H_2O = 4Na_2[\overset{+2}{Zn}(OH)_4] + \overset{-3}{N}H_3\uparrow$$

$$\begin{array}{c|l} 4 & \overset{0}{Zn} - 2e = \overset{+2}{Zn} \\ 1 & \overset{+5}{N} + 8e = \overset{-3}{N} \\ \hline & \quad 8 \end{array}$$

Ionic equation:

Net ionic equation:

Test 3. To the test tube add 2 - 3 drops of KNO_3 solution, add 1 - 2 drops of $KMnO_4$ solution. Observation:

Do the same test with KNO_2 solution.

Observation:

Compare the analytical effect:

Reactions of NO_2^- anions

Test 1. To the test tube add 3 - 5 drops of KNO_2 solution, add excess of H_2SO_4 solution. Heat. Observation:

$$2KNO_2 + H_2SO_4 = 2HNO_2 + K_2SO_4$$

$$2HNO_2 = NO_2\uparrow + NO\uparrow + H_2O$$

Test 2. To the test tube add 2 - 3 drops of KNO_2 solution, add 2 - 3 drops of NH_4Cl solution. Heat. Observe the bubbles of gas N_2.

Net ionic equation: $NO_2^- + NH_4^+ = N_2\uparrow + 2H_2O$

Molecular equation:

These questions are taken from the Test items for licensing examination Krok 1 PHARMACY

The correct answer is first

Organic anions

1) To detect anions in a solution by fractional method a reaction with iron (III) chloride was performed in acid medium. The solution coloured red-violet. What anion is the cause of such analytical effect?
   A. Salicylate B. Chloride C. Nitrate D. Bromate E. Phosphate
2) Selective solvents are used in laboratories and factories to isolate and refine essential oils, alkaloids, antibiotics and other pharmaceutical substances. This process is called:
   A. Extraction B. Sedimentation C. Coagulation

D. Flocculation E. Flotation

3) Pharmacopoeia test reaction for determination of benzoate ions is the interaction with the following solution:
A. Iron (III) chloride B. Potassium chloride C. Resorcinol
D. Acetic anhydride E. Diphenylamine

LABORATORY WORK "THE ANIONS OF ORGANIC ACIDS IDENTIFICATION"

Acetate ions (salts of acetic acid)

Test 1. To the test tube add 3 - 5 drops of sodium acetate, add 3 - 5 drops of ethanol and 10 - 15 drops of sulfuric acid (conc), heat. Pay attention to the product formed (acetic ethyl ester $CH_3COOC_2H_5$), which has a characteristic fruity odor. The equation of the reaction:

$$CH_3COOH + C_2H_5OH \xrightarrow{\text{конц.}H_2SO_4} CH_3COOC_2H_5 + H_2O$$

Test 2. To the test tube add 3 - 5 drops of sodium acetate and 3 - 5 drops of ferric chloride (III). Observation:

Molecular equation: $FeCl_3 + 3CH_3COONa = Fe(CH_3COO)_3 + 3NaCl$
Solution heat to boiling.

Observation:

Molecular equation: $Fe(CH_3COO)_3 + 2H_2O = Fe(OH)_2CH_3COO\downarrow + 2CH_3COOH$.

Ionic equation:

Net ionic equation:

Tartrate ions (salts of tartaric acid)

Tartrate anions is acid residue of tartaric acid $H_2C_4H_4O_6$.

Test 1. To 1 ml of solution of tartaric acid add 1 - 2 crystals of potassium chloride, 3 - 5 drops of ethanol and 1 - 2 drops of sodium acetate CH_3COONa. *Cool the test tube under a water jet and rubs the wall-side of test tube by a glass stick till precipitate forms.* Pay attention to a formation of a white crystalline precipitate.
Molecular equation: $H_2C_4H_4O_6 + KCl + CH_3COONa = KHC_4H_4O_6\downarrow + NaCl + CH_3COOH$.
Ionic equation:

Net ionic equation:

Citrate ions (salts of citric acid)

Citrate anions is acid residue of citric acid $H_3C_6H_5O_7$.

Test 1. To the test tube add 3 - 5 drops of sodium citrate, add 3 - 5 drops of calcium chloride solution.

Observation:

Heat to boiling.

Observation:

Pay attention to the fact that the precipitate formed after boiling the solution.
Molecular equation: $2Na_3C_6H_5O_7 + 3CaCl_2 = Ca_3(C_6H_5O_7)_2\downarrow + 6NaCl$

Benzoate ions (salts of benzoic acid)

Benzoate anion is acid residue of benzoic acid C_6H_5COOH.
Test 1. To the test tube add 3 - 5 drops of sodium benzoate solution and 3 - 5 drops of iron (III) chloride solution.
Observation:

$$3C_6H_5COONa + FeCl_3 = Fe(C_6H_5COO)_3\downarrow + 3NaCl$$

(pink-yellow precipitate)

Ionic equation:

Net ionic equation:

Salicylate ions (salts of salicylic acid)

Salicylate anion is acid residue of salicylic acid: $HO–C_6H_4–COOH$.

Test 1. To 3 - 5 drops of sodium salicylate solution add 3 - 5 drops of iron (III) chloride solution.

Observation:

Salicylate anions in a reaction with iron (III) ions can forms different composition and color (from blue- purple to red-purple) complex compounds depend on proportion of the reagents.

$$HO–C_6H_4–COONa + FeCl_3 = [Fe(O–C_6H_4–COO)]Cl + HCl + NaCl,$$

or

$$2HO–C_6H_4–COONa + FeCl_3 = Na[Fe(O–C_6H_4–COO)_2] + 2HCl + NaCl,$$

or

$$3HO–C_6H_4–COONa + FeCl_3 = Na_3[Fe(O–C_6H_4–COO)_3] + 3HCl.$$

The mixture prepeared divided into two tubes.

To the first tube add 3 - 5 drops of acetic acid.

Observation:

To the second tube add 3 - 5 drops of hydrochloric acid.

Observation:

Practical lesson 9

Systematic analysis of compound of unknown composition. Dry salt analysis: cations identification

Laboratory work 10

Dry salt analysis: "Cation Identification"

Unlike covalent compounds, which can be identified using physical properties like boiling point and refractive index, ionic compounds are more appropriately identified with their chemical properties. In the qualitative analysis procedure, the chemical properties of an unknown substance are determined by systematically reacting the unknown with a number of different reagents. By predetermining what the particular reaction will produce if a specific ion is present, the ions that actually are in the solution can be identified. For example, if a reaction is known to produce a precipitate if ion A is present and a precipitate is formed when the reaction is run, then ion A may be present in solution (there may be, and usually are, other ions that will also precipitate with a particular reagent). If no precipitate is formed when the reaction is run, then ion A is clearly not present in the unknown solution and a different reaction will have to be run to determine what ions are present.

There are two general situations in which qualitative analysis is used - in the identification of a simple salt, or the identification of multiple cations in a solution.

Identifying a Simple Salt

The basic testing procedure for identifying a salt is as follows.

1. **Appearance of compound**
2. Predicting the color of a compound can be extremely complicated. Some examples include: Cobalt chloride is pink or blue depending on the state of hydration (blue dry, pink with water) so it is used as a moisture indicator in silica gel. Zinc oxide is white, but at higher temperatures becomes yellow, returning to white as it cools.

Name	**Formula**	**Color**
Copper(II) sulfate	$CuSO_4$	White

Copper(II) sulfate pentahydrate	$CuSO_4 \cdot 5H_2O$	Blue
Copper(II) benzoate	$C_{14}H_{10}CuO_4$	Blue
Cobalt(II) chloride	$CoCl_2$	Deep blue
Cobalt(II) chloride hexahydrate	$CoCl_2 \cdot 6H_2O$	Deep magenta
Manganese(II) chloride tetrahydrate	$MnCl_2 \cdot 4H_2O$	Pink
Copper(II) chloride dihydrate	$CuCl_2 \cdot 2H_2O$	Blue-green
Nickel(II) chloride hexahydrate	$NiCl_2 \cdot 6H_2O$	Green
Lead(II) iodide	PbI_2	Yellow

3. The compound will most likely be in solid form. Note the color and shape of the crystals. Ionic compounds formed from the representative elements tend to be white or colorless, while ions of transition elements tend to be colored.

The following is the colors of metal ions in solution with NO_3^-:

Co^{2+}- rose; Co^{3+}- violet; Cr^{3+}- violet; Cu^{2+}- blue; Fe^{2+}- pale green, pale violet; Fe^{3+}-yellow-brown; Mn^{2+}- pale pink; Ni^{2+}- blue-green.

4. **Heating effect**

Heating a compound can cause a liquid to condense on the inside of the test tube. This is probably water, indicating that the compound is a hydrate. If a gas is given off, note the color and odor of the gas. The nitrate, carbonate, and sulfite ions may decompose, as illustrated by the reactions:

$2\ Pb(NO_3)_2(s) + heat \rightarrow 2\ PbO(s) + O_2(g) + 4\ NO_2(g, brown)$

$CaCO_3(s) + heat \rightarrow CaO(s) + CO_2(g, colorless, odorless)$

$CaSO_3(s) + heat \rightarrow CaO(s) + SO_2(g, colorless, pungent)$

Some bromides and iodides decompose to give Br_2(g, orange-brown) and I_2(g, purple).

5. **Flame test**

Solutions of ions, when mixed with concentrated HCl and heated on a nickel/chromium wire in a flame, cause the flame to change to a color characteristic of the atom.Visible colors occur with the following ions:

Sodium - Bright yellow (intense, peristent); Potassium- Pale violet (slight, fleeting)

Calcium- Brick red (medium, fleeting); Strontium- Crimson (medium)

Barium- Light green (slight); Lead- Pale bluish (slight, fleeting)

Copper- Green or blue (medium, persistent)

6. **Solubility in water**

Place one small spatula of the compound in 1 mL of water. If the compound is soluble this amount will dissolve after considerable stirring. If the compound is moderately soluble, some of this amount will dissolve. If the compound is insoluble, even a very small amount will not dissolve.

General solubility rules:

- All nitrates are soluble.
- Practically all sodium, potassium, and ammonium salts are soluble.

○ All chlorides, bromides, and iodides are soluble except those of silver, mercury(I), and lead(II).
○ All sulfates are soluble except those of strontium, barium, and lead(II), which are insoluble, and those of calcium and silver which are moderately soluble.
○ All carbonates, sulfites, and phosphates are insoluble except those of sodium, potassium, and ammonium.
○ All sulfides are insoluble except those of the alkali metals, the alkaline earth metals, and ammonium.
○ All hydroxides are insoluble except those of the alkali metals. The hydroxides of calcium, strontium, and barium are moderately soluble. Ammonium hydroxide does not exist; ammonium hydroxide is a misnomer for aqueous ammonia, NH_3(aq).

7. **Reaction with nitric acid**

Add nitric acid to the compound and observe any reaction that occurs. If the compound dissolved in water, it should dissolve in nitric acid. If it did not dissolve in water, but appears to be dissolving in nitric acid, it is undergoing a chemical reaction. In general, compounds that contain anions that are the conjugate bases of weak acids will react (unless the compounds are very insoluble). For example:

$CaCO_3(s) + 2\ H^+(aq) \rightarrow Ca_2^+(aq) + H_2O(l) + CO_2$(g, colorless, odorless)

$NiS(s) + 2\ H^+(aq) \rightarrow Ni_2^+(aq) + H_2S$(g, colorless, rotten egg smell)

$Ca_3(PO_4)_2(s) + 6\ H^+(aq) \rightarrow 3\ Ca_2^+(aq) + 2\ H_3PO_4(aq)$

The remaining tests must be perfomed on a solution of the compound. If the compound is insoluble in water, dissolve it in nitric acid. Otherwise, dissolve in water.

8. **Reaction with sodium hydroxide**

Add NaOH dropwise to the solution, stir or shake the solution, and observe any reaction (if the compound was dissolved in nitric acid, the first several drops will neutralize the acid so be sure to check the pH with litmus paper). Look for a precipitate (refer to the solubility rules for hydroxides). If a precipitate forms, continue adding NaOH. Some metal hydroxides are amphoteric and will form a complex ion and redissolve. The following ions are amphoteric:

Species	**Acidic Solution**	**Slightly Basic Solution**	**Basic Solution**
Al^{3+}	$Al^{3+}(aq)$	$Al(OH)_3(s)$	$Al(OH)_4^-(aq)$
Cr^{3+}	$Cr^{3+}(aq)$	$Cr(OH)_3(s)$	$Cr(OH)_4^-(aq)$
Pb^{2+}	$Pb^{2+}(aq)$	$Pb(OH)_2(s)$	$Pb(OH)_4^{2-}(aq)$
Zn^{2+}	$Zn^{2+}(aq)$	$Zn(OH)_2(s)$	$Zn(OH)_4^{2-}(aq)$
Sn^{4+}	$Sn^{4+}(aq)$	$Sn(OH)_4(s)$	$Sn(OH)_6^{2-}(aq)$

9. **Reaction with ammonia**

Add NH_3 dropwise to the solution, stir or shake the solution, and observe any reaction. If a metal hydroxide precipitate forms, continue adding ammonia. Some metal hydroxides fom a complex ion and redissolve. See Figures 4, 5, and 6 for an example of this reaction. The following ions form ammonia complexes:

Acid Solution	**Basic Solution**	**Solution with Excess NH_3**	**Color of Complex**

$Ni^{2+}(aq)$	$Ni(OH)_2(s)$	$Ni(NH_3)_6^{2+}(aq)$	violet
$Cu^{2+}(aq)$	$Cu(OH)_2(s)$	$Cu(NH_3)_4^{2+}(aq$	blue
$Zn^{2+}(aq)$	$Zn(OH)_2(s)$	$Zn(NH_3)_6^{2+}(aq)$	colorless
$Ag^{+}(aq)$	$Ag_2O(s)$	$Ag(NH_3)_2^{+}(aq)$	colorless
$Cd^{2+}(aq)$	$Cd(OH)_2(s)$	$Cd(NH_3)_4^{2+}(aq)$	colorless

10. **Reaction with hydrochloric acid**

Add HCl dropwise until solution tests acidic to litmus paper and observe any reaction. A precipitate will form with any cation that forms an insoluble chloride (refer to the solubility rules). For example:

$Pb^{2+} + 2Cl- \rightarrow PbCl_2(s)$

11. **Reaction with sulfuric acid**

Add H_2SO_4 dropwise until solution is acidic and observe any reaction. A precipitate will form with any cation that forms an insoluble sulfate (refer to the solubility rules). For example:

$Ba^{2+} + SO_4^{2-} \rightarrow BaSO_4(s)$

12. **Reaction with silver nitrate**

Add HNO_3 dropwise until solution is acidic (unless of course it was dissolved in nitric acid), then add a few drops of $AgNO_3$ and observe any reaction. A precipitate will form with certain cations that form insoluble silver compounds, but because of the acidic environment, some insoluble silver salts (e.g. salts containing CO_3^{2-}, S^{2-}, and PO_4^{3-} ions) are "destroyed." Cl^-, Br^-, and I^- form insoluble compounds, while SO_4^{2-} forms a moderately insoluble compound.

$Ag^+ + Cl^- \rightarrow AgCl(s)$

13. **Reaction with barium nitrate**

Add HNO_3 dropwise until solution is acidic, boil the solution for two minutes, then test with litmus paper. Continue adding and boiling until solution remains acidic after boiling. Cool the solution and add a few drops of $Ba(NO_3)_2$ and observe any reaction. A precipitate will form with anions that form an insoluble barium compound (except the ones destroyed by acid as in the above test).

14. **Specific Tests**

Sometimes the above tests can not definitively confirm the presence of a specific ion. In these cases, it is necessary to do specific tests for a particular ion.

Specific Tests
Cation Identification Tests

1. Generally Soluble Cations

- **Ammonium Ions:**

Take a small amount of the material to be tested and place it in a 50-mL beaker. Add 6 M NaOH and smell cautiously. The odor of ammonia indicates the presence of ammonium ions. If you do not smell ammonia, warm the beaker and again smell the emitted vapors. The liberated ammonia will also change the color of a moistened strip of red litmus paper held at the entrance of the test tube.

$NH_4^+(aq) + OH^-(aq) \rightarrow NH_3(g) + H_2O$

This test is very reliable. It should be performed whenever the generally soluble cations, NH_4^+, Na^+, and K^+, are suspected.

- **Sodium Ions:**

The most common method of identification of Na^+ is the flame test. Sodium imparts a brilliant, long lasting, yellow flame that masks colors from other ions. The test may be performed on a small sample of the unknown treated with concentrated HCl or a few drops of solution unknown treated with concentrated HCl. The flame should be bright and it should last as long as that of 0.1 M NaCl. Sodium is a common impurity and traces will be found in almost any unknown. You must learn to distinguish between an unknown that has sodium ion as the cation and an unknown that has sodium ion as an impurity.

- **Potassium Ions**:

The most common method of identification of K^+ is the flame test. The test may be performed on a small sample of the unknown treated with concentrated HCl or a few drops of solution unknown treated with concentrated HCl. The violet flame is not intense but it is clearly visible in the absence of sodium ions. Cobalt glass filters yellow light from sodium impurities and allows the violet flame to be seen. Do not confuse the glowing wire for the potassium flame.

2. Cations That Form Insoluble Chlorides

- **Silver Ions:**

Although Ag^+, Pb^{2+}, and Hg_2^{2+} all form insoluble white chlorides, $Ag^+$is the only one of these cations that forms an ammonia complex. Therefore, AgCl dissolves readily in aqueous NH_3. When the resulting solution is acidified with HNO_3, AgCl reprecipitates.

$AgCl(s) + 2NH_3(aq) \rightarrow Ag(NH_3)^{2+}(aq) + Cl^-(aq)$

$Ag(NH_3)^{2+}(aq) + Cl^-(aq) + 2H_3O^+ \rightarrow AgCl(s) + 2NH_4^+ + 2H_2O$

Add 3 M HCl dropwise to the solution being tested. If a white precipitate is formed, centrifuge and remove the supernatant liquid. Add 6 M NH_3 solution to the precipitate. If the precipitate dissolves, add 6 M HNO_3. Formation of a white precipitate indicates Ag^+.

- **Lead Ions:**

Although $PbCl_2$ is insoluble at room temperature, its solubility is increased dramatically at higher temperatures; it dissolves readily in boiling water. Pb^{2+} also forms an insoluble white sulfate, which dissolves in a solution containing acetate ion due to the formation of the weak electrolyte, $Pb(CH_3COO)_2$. The addition of chromate ion to this lead acetate solution yields a precipitate of yellow lead chromate.

$Pb^{2+}(aq) + SO_4^{2-}(aq) \rightarrow PbSO_4(s)$

$PbSO_4(s) + 2CH_3COO^-(aq) \rightarrow Pb(CH_3COO)_2(aq) + SO_4^{2-}(aq)$

$Pb(CH_3COO)_2(aq) + CrO_4^{2-}(aq) \rightarrow PbCrO_4(s) + 2CH_3COO^-(aq)$

To the solution to be tested add 3 M HCl dropwise. (A large excess of HCl must be avoided because of the formation of the soluble chloro complex, $PbCl_4^{2-}$.) Centrifuge and remove the supernatant from the white precipitate ($PbCl_2$). Add hot water to the precipitate and stir. If the precipitate dissolves, Pb^{2+} is indicated. Add 3 M H_2SO_4 to the hot solution. Centrifuge and remove the supernatant liquid from the white precipitate ($PbSO_4$). To the precipitate add 3 M $NH_4(CH_3COO)$ and stir. If the white precipitate was $PbSO_4$, it will dissolve. To confirm, add a few drops of 0.5 M K_2CrO_4 to the resulting solution. A yellow precipitate of $PbCrO_4$ indicates the presence of Pb^{2+}.

- **Mercury(I) Ions:**

When Hg_2Cl_2 is treated with aqueous NH_3 a reaction occurs in which free mercury and amidochloromercury(II) are formed.

$Hg_2Cl_2(s) + NH_3(aq) \rightarrow Hg(l) + HgNH_2Cl(s) + HCl(aq)$

The $HgNH_2Cl$ is a white solid, while the Hg in a finely divided state appears black. The resultant mixture is gray to black.

Add 3 M HCl to the solution to be tested for Hg_2^{2+}. If a white precipitate forms, centrifuge and remove the supernatant liquid. To the precipitate, add 6 M NH_3 and stir. The appearance of a gray to black precipitate is positive for Hg_2^{2+}.

3. Cations That Form Insoluble Sulfates

Identification tests for Pb^{2+} and Ag^+ (Ag_2SO_4 is moderately soluble) are described above (Cations that form Insoluble Chlorides). Ba^{2+}, Sr^{2+}, and Ca^{2+} form moderately soluble sulfates.

The alkaline earth ions Mg^{2+}, Ca^{2+}, Sr^{2+}, and Ba^{2+} are one of the best examples of a periodic relationship among the elements of a family. Solubilities of their compounds are graduated nicely and the separations (qualitatively) can be accomplished readily. Flame tests are very important.

- **Barium Ions:**

Barium ions can be identified by precipitation of its insoluble yellow $BaCrO_4$ salt. If Ca^{2+} or Sr^{2+} are present they will also precipitate in the presence of high concentrations of CrO_4^{2-}. However, the chromates of Ca^{2+} and Sr^{2+} are moderately soluble; their precipitation can be prevented by addition of acetic acid. This weak acid provides sufficient hydronium ions to lower the CrO_4^{2-} concentratiion enough to keep $CaCrO_4$ and $SrCrO_4$ in solution but to allow the $BaCrO_4$ to precipitate.

$2CrO_4^{2-}(aq) + 2H^+(aq) \rightarrow Cr_2O_7^{2-}(aq) + H_2O$

The flame test on the solid chromate is important for confirmation.

To about 1 mL of solution add 10 drops of 6 M CH_3COOH. Then add a few drops of 0.5 M K_2CrO_4 solution. The appearance of a yellow precipitate indicates the presence of Ba^{2+}. To confirm, dissolve the precipitate in concentrated HCl and flame test.

- **Strontium Ions:**

Strontium can be identified, in the absence of calcium, by precipitating its sulfate. To the solution add 0.1 M H_2SO_4 dropwise. The formation of a finely-divided, crystalline, white precipitate indicates the presence of Sr^{2+}. (Ba^{2+} must be absent, of course.) To confirm, dissolve the precipitate in concentrated HCl and flame test.

- **Calcium Ions:**

If Ba^{2+} and Sr^{2+} are absent, Ca^{2+} may be precipitated as the oxalate from neutral or alkaline solutions. Test the acidity of the solution with litmus paper. If it is acidic, add 3 M NH_3 until basic. Then add 0.2 M $(NH_4)_2C_2O_4$ solution. The formation of a white precipitate indicates the presence of Ca^{2+}. Confirm by adding a few drops of concentrated HCl and flame testing.

4. Cations That Form Ammonia Complexes

- **Cadmium Ions:**

Cadmium forms a yellow precipitate with sulfide ion either from a neutral solution containing free Cd^{2+} or from an ammoniacal solution of $Cd(NH_3)_4^{2+}$. Since most sulfides are insoluble, and many of them are black, the presence of other metal ions may make it difficult to detect the yellow color of CdS. Therefore, separations must be as complete as possible before testing for Cd^{2+}.

$Cd^{2+}(aq) + S^{2-}(aq) \rightarrow CdS(s)$

$Cd(NH_3)_4^{2+}(aq) + S^{2-}(aq) \rightarrow CdS(s) + 4NH_3$

To a solution of Cd^{2+} or to a solution thought to contain $Cd(NH_3)_4^{2+}$add 0.1 M Na_2S solution dropwise. The formation of a yellow precipitate confirms the presence of Cd^{2+}.

- **Copper(II) Ions:**

The very distinct deep blue color of the copper ammonia complex can be used to identify Cu^{2+}. This identification can be carried out in the presence of other cations which form either colorless ammonia complexes or white precipitates. Thus, Zn^{2+}, Cd^{2+}, Al^{3+}, among others, will not interfere.

In relatively dilute solutions the color of the ammonia complex may not be intense enough to give an unqualified identification, and some other test for confirmation must be used. Cu^{2+} forms a very insoluble reddish-brown hexacyanoferrate(II).

$2Cu^{2+}(aq) + Fe(CN)_6^{4-}(aq) \rightarrow Cu_2Fe(CN)_6(s)$

Other cations that react with this reagent to form highly colored precipitates must be absent (Co^{2+} and Fe^{3+} for example). Acidify the test solution with acetic acid. Then add a few drops of 0.1 M potassium hexacyanoferrate(II) solution ($K_4Fe(CN)_6$). A red-brown precipitate confirms the presence of Cu^{2+}.

- **Nickel(II) Ions:**

Nickel(II) is one of the easiest cations to identify. Ni^{2+} forms a red precipitate with dimethylglyoxime in a buffered acid solution. Palladium(II) is the only other cation which forms a precipitate with this reagent. However, a few other cations can interfere. Cobalt(II) preferentially forms a dark brown solution with dimethylglyoxime, and excess reagent must be used in its presence.

Acidify the solution to be tested with 6 M CH_3COOH. Then add about one mL of 0.2 M $NaOOCCH_3$ solution. Add dimethylglyoxime solution dropwise. A bright red precipitate is positive for Ni^{2+}.

- **Zinc Ions:**

Zinc forms one of the few insoluble white sulfides. It is precipitated from a solution of the ammonia complex. Small traces of cations that form dark colored sulfides will obviously interfere.

Add an excess of 3 M NH_3 to the test solution, so that any zinc present is in the form of $Zn(NH_3)_4^{2+}$. Then add a few drops of 0.1 M Na_2S solution. A white precipitate indicates the presence of Zn^{2+}.

5. Cations That Form Amphoteric Hydroxides

- **Aluminum Ions:**

Aluminum is generally identified by making use of the amphoteric property of its hydroxide and the red color of the "lake" $AlOH_3$ forms with the reagent, aluminon. Aluminon is a dye (an organic molecule, usually fairly large, that absorbs visible light). As the $Al(OH)_3$precipitates the dye is adsorbed on the $Al(OH)_3$ particles. The adsorption of the dye is called "laking." Aluminum is a fairly common impurity and care must be taken that trace quantities are not reported. Since most laboratory manipulations are carried out in glass containers, silica gel, which physically resembles aluminum hydroxide, is also a common impurity.

Adjust the pH of about 1 mL of the test solution (with 3 M NaOH and 3 M HNO_3) to precipitate the hydroxide. Centrifuge the mixture. Remove the mother liquor with a capillary pipet and wash the precipitate with distilled water. Centrifuge the mixture. Remove the mother liquor with a capillary pipet and wash the precipitate with distilled water. Centrifuge the mixture and remove the mother liquor with a capillary pipet. These repeated washings remove other ions from the precipitate. Dissolve the precipitate in 3 M HNO_3. If any precipitate does not dissolve in the nitric acid, remove the supernatant to a clean test tube and discard the residue. Add two drops of aluminon reagent (avoid any excess). Add 3 M NH_3(aq) until the solution is basic. Centrifuge. A red, gelatinous precipitate (sometimes called a red lake) indicates Al^{3+}.

Any precipitate that remains after the addition of the nitric acid is probably silica gel, $SiO_2 \bullet xH_2O$. Silica gel is present in many solutions; it is leached from glass containers. Any silica gel present must be removed before the addition of the aluminon and the ammonia because silica gel will also give a red lake.

Do not confuse traces of red-brown ferric hydroxide for the red lake. Other precipitates will also form colors with the reagent. The supernatant liquid will be an intense blue-purple color if too much reagent has been added. This color has nothing to do with the presence of aluminum. The color of the reagent is sensitive to changes in pH, (the reagent is an acid-base indicator).

- **Chromium(III) Ions:**

Chromium can be taken through a series of colored tests which leaves no doubt as to its identity. Chromium(III) forms a steel green hydroxide which dissolves in excess strong base to give a deeply green colored solution of the hydroxy complex. Treating this complex with 3% hydrogen peroxide gives the yellow solution of the chromate ion, which upon acidification with dilute nitric acid gives the orange color of dichromate. Treatment of the cold solution of dichromate with 3% hydrogen peroxide gives the intense blue color of a peroxide of chromium. (The actual composition of this peroxide is not known, but it is believed to have the empirical formula CrO_5.) This peroxide readily decomposes to the pale violet color of the original hydrated chromium(III) ion. In low concentrations of dichromate the blue color is fleeting, and attention must be focused on the test tube during the addition of the hydrogen peroxide to avoid missing the color change.

$Cr(OH)_4^-$ (green) + (H_2O_2)+(OH^-)→ CrO_4^{2-} (yellow)

CrO_4^{2-} +(H^+)→ $Cr_2O_7^{2-}$ (orange)

$Cr_2O_7^{2-}$ +(H_2O_2)+(HNO_3)→ CrO_5 (blue)→ $Cr(H_2O)_6^{3+}$ (violet)

The following color changes are all indicative of Cr^{3+}. Add an excess of 6 M NaOH to about one mL of test solution. To this green solution add 10 drops of 3% H_2O_2. Heat the test tube in the water bath until the excess H_2O_2 is destroyed as indicated by the cessation of bubbles. Acidify the yellow solution with 3 M HNO_3. Cool the resulting orange solution in an ice bath. To the cooled solution add a drop or two of 3% H_2O_2 and observe the immediate fleeting blue color.

○ **Tin(IV) Ions:**

Sn^{4+} is most conveniently identified by reduction of Sn^{4+} to Sn^{2+} with iron. The Sn^{2+} solution is treated with $HgCl_2$ solution, whereupon $Sn^{2+}$is oxidized to Sn^{4+} and, simultaneously, $HgCl_2$ is reduced to Hg_2Cl_2 (a silky, white precipitate). The Hg_2Cl_2 is further reduced by Sn^{2+} to Hg, which appears black.

$Sn^{4+}(aq) + Fe(s) \rightarrow Sn^{2+}(aq) + Fe^{2+}(aq)$

$Sn^{2+}(aq) + 2HgCl_2(aq) \rightarrow Sn^{4+}(aq) + Hg_2Cl_2(s) + 2Cl^-(aq)$

$Sn^{2+}(aq) + Hg_2Cl_2(s) \rightarrow Sn^{4+}(aq) + 2Hg(l) + 2Cl^-(aq)$

Add some concentrated HCl to the solution to be tested for Sn^{4+}. Place an iron brad (or small iron wire) in this solution and heat in a water bath for 5 minutes. Take the clear solution (filter if necessary) and add $HgCl_2$ solution dropwise. The appearance of a silky, white precipitate, which then turns black, confirms the presence of tin.

6. Other Cations

○ **Manganese(II) Ions:**

Manganese is easily identified by oxidation of Mn^{2+} to purple MnO_4^-using sodium bismuthate ($NaBiO_3$). Heat must be avoided to prevent the decomposition of permanganate ion to brown, insoluble manganese dioxide. Chloride ion must be absent, because it reduces permanganate ion to either manganese dioxide or manganese(II) depending upon the conditions.

Acidify the test solution with 3 M HNO_3. Add solid $NaBiO_3$ and stir. Centrifuge. If the supernatant has the characteristic purple color of MnO_4^-, Mn^{2+} was present.

$2Mn^{2+} + 14H^+ + 5NaBiO_3 \rightarrow 5Bi^{3+} + 5Na^+ + 7H_2O + 2MnO_4$

○ **Bismuth(III) Ions:**

Bismuth(III) forms a highly insoluble hydroxide which upon treatment with the hydroxy complex of tin(II) is immediately converted to free bismuth, a black precipitate.

$3Sn(OH)_4^{2-}(aq) + 2Bi(OH)_3(s) \rightarrow 2Bi(s) + 3Sn(OH)_6^{2-}(aq)$

Precipitate Bi^{3+} from the test solution with 3 M NaOH and centrifuge the precipitate. Then, to a solution of tin(II) chloride add with stirring 6 M sodium hydroxide until the precipitate of tin(II) hydroxide which first forms just redissolves. This solution is then added dropwise to the precipitate of bismuth(III) hydroxide. The rapid formation of a black color confirms bismuth.

○ **Iron(III) Ions:**

The Fe^{3+} ion is readily identified in a dilute nitric acid solution through the blood red color of its thiocyanate complex. A large excess of reagent should be avoided.

$Fe^{3+}(aq) + SCN^-(aq) \rightarrow Fe(SCN)^{2+}(aq)$

Acidify the solution with 3 M HNO_3. Then add a few drops of 0.1 M NH_4SCN solution. The solution turns red if Fe^{3+} is present.

Practical lesson 10

Systematic analysis of compound of unknown composition. Dry salt analysis: anion identification. Submodule "Qualitative analysis" control: Test control. Written control work.

Laboratory work 11

Dry salt analysis: Anion Identification Tests

- **Carbonate Ions:**

The most characteristic reaction of carbonate is the formation of carbon dioxide upon treatment with acid:

$CO_3^{2-}(aq) + 2H^+(aq) \rightarrow CO_2(g) + H_2O(l)$

The colorless, odorless carbon dioxide can be identified by bubbling it through a saturated solution of barium hydroxide, with which it forms a white precipitate of barium carbonate.

$CO_2(g) + Ba^{2+}(aq) + 2OH^-(aq) \rightarrow BaCO_3(s) + H_2O(l)$

Assemble a gas-liberation apparatus from a small test tube and a section of bent tubing. Dissolve or suspend a portion of your compound in a small amount of water and place it in the small test tube. Add about 0.5 mL of 6 M HCl and quickly fit the tube into the small test tube, allowing the gas liberated to bubble into a 6" test tube of saturated $Ba(OH)_2$ solution. The formation of a white precipitate in the large test tube (if the gas liberated is odorless) is a positive test for carbonate. It is imperative to test the gas-liberation apparatus by adding HCl to Na_2CO_3.

- **Sulfide Ions:**

When treated with nonoxidizing acids (HCl, CH_3COOH) sulfides react to liberate H_2S gas (rotten-egg odor). If the sulfide is very insoluble liberation of the gas may require concentrated acids (indeed some sulfides, HgS, CuS, are so insoluble that dissolution requires special treatment). The gas is generally identified by its odor and its precipitation of colored sulfides of various metal ions. Sulfides or hydrogen sulfide also are oxidized to elemental sulfur and sulfate by oxidizing agents such as permanganate, nitric acid, sulfuric acid, Fe(III), etc.

$3H_2S(aq) + 2H^+(aq) + 2NO_3^-(aq) \rightarrow 2NO(g) + 4H_2O + 3S(s)$

Acidify a sample with 6 M hydrochloric acid and warm. Cautiously smell the gas evolved and also test the gas with a piece of filter paper moistened with lead acetate solution. A foul smelling gas which turns lead acetate paper black constitutes a positive sulfide test.

- **Sulfate Ions:**

Sulfate is conveniently identified by precipitation of $BaSO_4$. Other insoluble barium salts contain anions of weak acids (CO_3^{2-}, $SO_3^{2-}$and PO_4^{3-}). Precipitation of these anions is prevented by acidifying the solution.

Acidify the test solution with 6 M HCl, and add a few drops of 0.2 M $BaCl_2$ solution. A white precipitate indicates the presence of SO_4^{2-}.

- **Nitrate Ions:**

The most notable feature of the chemistry of the nitrate ion is its oxidizing ability as illustrated by the following reactions:

$3Fe^{2+}(aq) + 4H^+(aq) + NO_3^-(aq) \rightarrow NO(g) + 2H_2O + 3Fe^{3+}(aq)$

In the last reaction the nitrogen oxide reacts with excess Fe^{2+} to give the brown complex ion $Fe(NO)^{2+}$. It is the formation of this brown complex that is used to identify NO_3^- (called the brown ring test).

Acidify about 2 mL of the test solution with 3 M H_2SO_4 and then dissolve one-half spatula full of solid $FeSO_4.7H_2O$ in the acidified solution. Cool the solution and then carefully introduce about 0.5 mL of concentrated H_2SO_4 by allowing it to flow down the side of the tilted test tube. Allow the solution to sit undisturbed so that the sulfuric acid forms a definite layer. The formation of a brown color at the interface of the layer constitutes a positive test for nitrate.

- **Phosphate Ions:**

The precipitation usually used to identify phosphate is the formation of yellow ammonium molybdophosphate from ammonium molybdate in acidic solution.

$12MoO_4^{2-} + 3NH_4^+ + PO_4^{3-} + 24H^+ \rightarrow (NH_4)_3[P(Mo_{12}O_{40})] + 12H_2O$

Acidify the sample with concentrated nitric acid and add several drops in excess. Then treat the solution with ammonium molybdate reagent and warm. The formation of a yellow crystalline precipitate confirms the presence of phosphate.

- **Chloride, Bromide, and Iodide Ions:**

All three of these anions form insoluble silver salts. Although the precipitates are of different colors (AgCl white, AgBr cream, AgI yellow) the colors are difficult to distinguish, and confirmatory tests are necessary.

Silver chloride, the most soluble of the three, dissolves readily in 6 M NH_3 solution because of formation of the ammonia complex. Furthermore, when the solution of the ammonia complex is acidified, AgCl reprecipitates. Neither AgBr nor AgI will dissolve readily in 6 M NH_3, a much higher concentration of NH_3 being required to form the complex.

$Cl^-(aq) + Ag^+(aq) \rightarrow AgCl(s)$

$AgCl(s) + 2NH_3(aq) \rightarrow Ag(NH_3)^{2+}(aq) + Cl^-(aq)$

$Ag(NH_3)^{2+}(aq) + Cl^-(aq) + 2H^+(aq) \rightarrow AgCl(s) + 2NH_4^+(aq)$

Bromide and iodide are usually identified by oxidation to the free elements with chlorine. The elements thus formed are extracted into carbon tetrachloride and identified by their color.

$2Br^-(aq) + Cl_2(g) \rightarrow Br_2(g) + 2Cl^-(aq)$

$2I^- (aq) + Cl_2(g) \rightarrow I_2(s) + 2Cl^-(aq)$

- **Chloride:**

Acidify the test solution with 3 M HNO_3. Then add several drops of 0.1 M $AgNO_3$. If a white precipitate forms, centrifuge and remove the supernatant. To the precipitate add 6 M NH_3 with stirring. If the precipitate dissolves, add 6 M HNO_3 to the solution. A white precipitate will form if the original test solution contained Cl^-.

- **Bromide and Iodide:**

Acidify the sample with several drops of 6 M HCl and add 4-5 drops of carbon tetrachloride. Then add about 0.5 mL of chlorine water and shake. Appearance of an orange-brown carbon tetrachloride layer indicates the presence of bromide. Formation of a purple layer indicates iodide.

Example Unknown Salts

Sample 1 had the following characteristics:

- Visual test: white crystalline powder   Heat test: brown gas given off

- Flame test: no color identified Solubility in water: soluble
- Nitric acid: soluble
- Hydroxide: formed an insoluble white precipitate, then dissolved with excess NaOH
- Ammonia: formed an insoluble white precipitate which did not redissolve in excess NH_3
- Hydrochloric acid: formed an insoluble white precipitate

Analysis of observations:

1. The brown gas given off during the heat test indicates presence of the NO_3^- ion, since the NO_3^- ion reacts to form brown NO_2 gas as shown below:

$2\ Pb(NO_3)_2(s) + heat \rightarrow 2\ PbO(s) + O_2(g) + 4\ NO_2(g)$

2. The white precipitate which formed and then redissolved with the addition of sodium hydroxide indicates the presence of an amphoteric cation. The possibilities are Al, Cr, Pb, Zn, and Sn. The reaction occurs as follows:

$Cr^{3+}(aq) + 3\ OH^-(aq) \rightarrow Cr(OH)_3(s)$

$Cr(OH)_3(s) + OH^-(aq) \rightarrow Cr(OH)_4^-(aq)$

3. Since the cation does not form an ammonia complex, it eliminates Zn from the list of possible cations established above.
4. There are only three cations which form precipitates with hydrochloric acid, Pb^{2+}, Ag^+, and Hg_2^{2+}. Of these three, only Pb^{2+} is amphoteric. The reaction with chloride occurs as follows:

$Pb^{2+} + 2\ Cl^- \rightarrow PbCl_2(s)$

Conclusion: Sample 1 is $Pb(NO_3)_2$

Sample 2 had the following characteristics:

Visual test: white crystalline powder Heat test: no reaction

Flame test: green color observed

<u>Specific barium test</u> (precipitation with CrO_4^{2-}): formed an insoluble yellow precipitate

Solubility in water: insoluble Nitric acid: produced odorless, colorless gas

Analysis of observations:

1. The green color observed during the flame test indicates the presence of barium. Although the remaining tests could be done to confirm the presence of barium, none is specific for just the Ba^{2+} ion. Therefore, the specific barium test is used.
2. The yellow precipitate produced by the barium test confirms the presence of barium. The reaction occurs as follows:

$Ba^{2+}(aq) + CrO_4^{2-}(aq) \rightarrow BaCrO_4(s)$

3. The odorless, colorless gas produced by the addition of nitric acid is CO_2. This indicates the presence of the CO_3^{2-} ion.

Conclusion: Sample 2 is $BaCO_3$

Sample 3 had the following characteristics:

Visual test: yellow/brown solid

Heat test: no reaction   Flame test: no reaction

Solubility in water: soluble   Nitric acid: soluble

Sodium hydroxide: formed an insoluble white precipitate which did not redissolve in excess NaOH

Ammonia: formed an insoluble white precipitate which did not redissolve in excess NH_3

Hydrochloric acid: no reaction

Specific iron(III) test (formation of a thiocyanate complex): solution turned blood red

Silver nitrate: formed an insoluble white precipitate

Specific chloride, bromide, iodide test: precipitate dissolved with addition of 6 M ammonia

Analysis of observations:

0. The color of the solid is characteristic of the Fe^{3+} ion.

1. Since the compound forms an insoluble precipitate with hydroxide, it eliminates the alkali metals, calcium, strontium, and barium. Since the cation is not amphoteric, it also eliminates aluminum, chromium, lead, zinc, and tin.

2. The cation does not form an ammonia complex, which eliminates nickel, copper(II), silver, and cadmium.

3. Since there is no reaction with Cl^-, Hg_2^{2+} is also eliminated. Of the ions which have not been eliminated (Fe^{3+}, Mn^{2+}, Bi^{2+}), Fe^{3+} is the most likely. A specific ion test is needed to confirm the presence of Fe^{3+}.

4. The red solution produced by the iron(III) test confirms the presence of iron(III). The reaction occurs as follows:

$Fe^{3+}(aq) + SCN^-(aq) \rightarrow Fe(SCN)^{2+}(aq)$

5. The precipitate formed by the addition of silver nitrate indicates the presence of either chloride, bromide, or iodide. Although the colors are different (AgCl white, AgBr cream, AgI yellow), they are difficult to distinguish and a specific ion test is needed to determine which one is present.

6. The specific ion test indicated that the anion present is Cl^-. The reaction occurs as follows:

$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$

$AgCl(s) + 2\ NH_3(aq) \rightarrow Ag(NH_3)_2^+(aq) + Cl^-(aq)$

Conclusion: Sample 3 is $FeCl_3$