

Ministry of Health of the Russian Federation
Volgograd State Medical University

Department of Pharmaceutical and Toxicological
Chemistry

GENERAL PHARMACEUTICAL CHEMISTRY

**CHEMICAL METHODS OF PHARMACOPOEIAL
ANALYSIS - IDENTIFICATION OF INORGANIC
DRUGS**

**(Identification of anions of medicinal products of
inorganic nature)**

Lesson 3

V term

Discipline

GENERAL PHARMACEUTICAL CHEMISTRY

LESSON №3	Chemical methods of pharmacopoeial analysis
Chemical methods for the Identification of inorganic drugs.	- Identification of inorganic drugs. Identification of anions.

QUESTIONS FOR THE LESSON

1. Chemical elements included in the composition of medicinal substances of inorganic nature and ways of their detection.
2. Identification of inorganic medicinal substances – detection of anions.

IDENTIFICATION OF INORGANIC MEDICINAL SUBSTANCES – DETECTION OF ANIONS

Chlorides

Interaction with silver nitrate. Chloride solutions with a solution of silver nitrate form a white curd precipitate of silver chloride, insoluble in nitric acid but soluble in a solution of ammonia and ammonium carbonate.

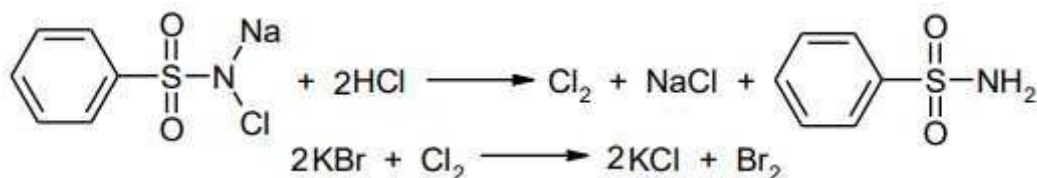


Technique:

ml of diluted nitric acid and 0.5 ml of silver nitrate solution are added to 1 ml of chloride salt solution; a white curd precipitate is formed, insoluble in diluted nitric acid, which dissolves in ammonia solution.

Bromides

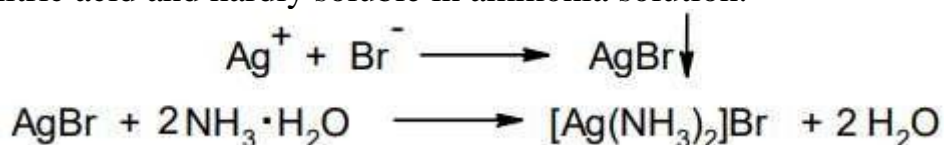
1. **Interaction with chloramine B.** Bromides are identified by the reaction of bromine release as a result of the redox reaction between bromide and chloramine in an acidic medium. The bromine released as a result of the reaction is extracted with chloroform. The chloroform layer turns yellow-brown:



Technique:

1 ml of diluted hydrochloric acid, 0.5 ml of chloramine B, 1 ml of chloroform are added to 1 ml of the bromide salt solution and shaken; the chloroform layer is colored yellow-brown.

2. **Interaction with silver nitrate.** Solutions of bromides with a solution of silver nitrate form a yellowish curd precipitate of silver bromide, insoluble in nitric acid and hardly soluble in ammonia solution.



Technique:

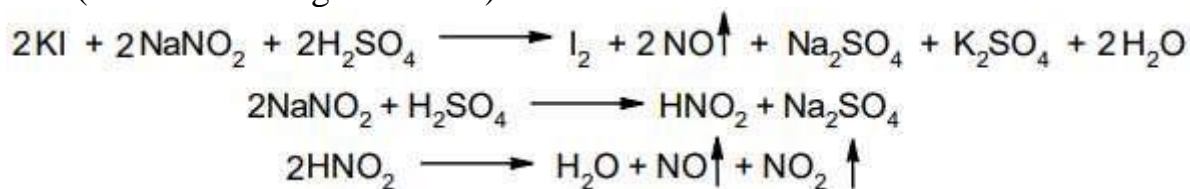
1 ml of diluted nitric acid and 0.5 ml of silver nitrate solution are added to 1 ml of the bromide salt solution; a yellowish-white curd precipitate is formed, insoluble in diluted nitric acid and hardly soluble in ammonia solution.

Iodides

1. **Interaction with iron chloride or sodium nitrite.** Weak oxidizing agents isolate molecular iodine from iodide solutions, detectable by the characteristic color and blueness of the starch solution. The State Pharmacopoeia of the Russian Federation recommends using a solution of iron (III) chloride or sodium nitrite:



Iodine is extracted by chloroform, in which it has a purple color. The reaction with a solution of sodium nitrite should be carried out under traction (release of nitrogen oxides!).

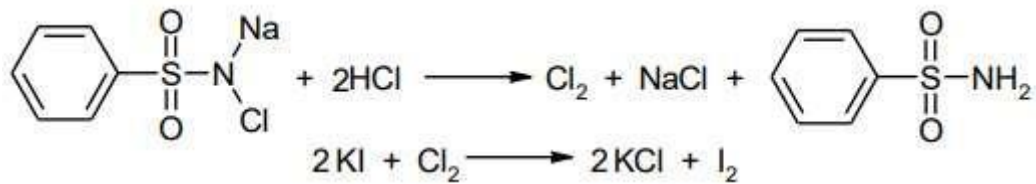


Technique:

0.2 ml of diluted sulfuric acid, 0.2 ml of sodium nitrite solution or iron (III) chloride solution and 2 ml of chloroform are added to 2 ml of iodide solution and shaken; the chloroform layer turns purple.

2. **Interaction with chloramine B.** Iodides are identified by the reaction of

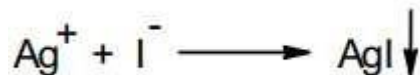
iodine release as a result of the redox reaction between iodide and chloramine in an acidic medium. The iodine released as a result of the reaction is extracted with chloroform, which turns purple:



Technique:

1 ml of diluted hydrochloric acid, 0.5 ml of chloramine B, 1 ml of chloroform are added to 1 ml of iodide salt solution and shaken; the chloroform layer is colored purple.

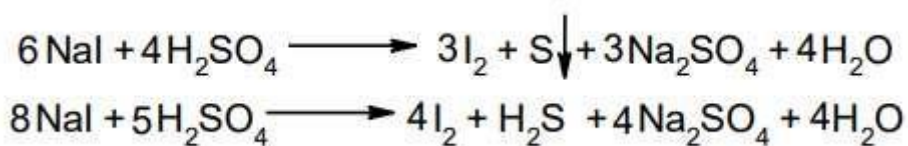
3. **Interaction with silver nitrate.** Solutions of iodides with a solution of silver nitrate form a yellow curd precipitate of silver iodide, insoluble in nitric acid and ammonia solution:



Technique:

0.5 ml of diluted nitric acid and 0.5 ml of silver nitrate solution are added to 2 ml of iodide solution; a yellow curd precipitate is formed, insoluble in diluted nitric acid and ammonia solution.

4. **Interaction with sulfuric acid.** The reducing properties of iodides are manifested when interacting with concentrated sulfuric acid; in this case, iodides are oxidized to free iodine, and H_2SO_4 to free sulfur or even to H_2S :

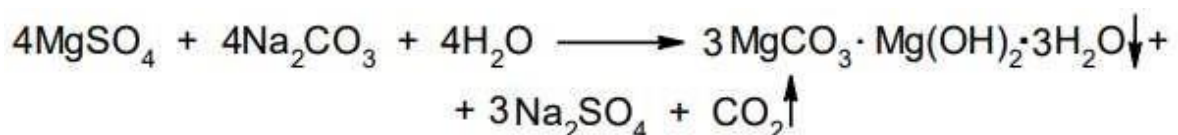


Technique:

When heating 0.1 g of the drug with 1 ml of concentrated sulfuric acid, purple iodine vapors are released.

Carbonates (bicarbonates)

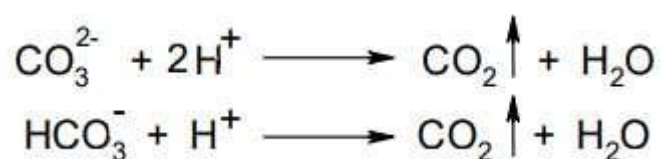
1. **Interaction with magnesium sulfate.** Carbonates with a saturated solution of magnesium sulfate, a white precipitate is formed, unlike hydrocarbonates.



Technique:

5 drops of saturated magnesium sulfate solution are added to 2 ml of carbonate solution (1:10); a white precipitate is formed (bicarbonate forms a precipitate only when the mixture is boiled).

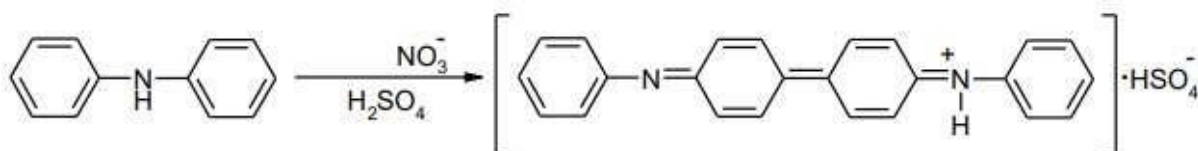
2. **Interaction with acids.** When acting on carbonates and bicarbonates of acids, carbon dioxide bubbles are released due to the decomposition of the released unstable carbonic acid:



3. **Interaction with phenolphthalein.** The carbonate solution (1:10) with the addition of 1 drop of phenolphthalein solution turns crimson (unlike bicarbonates).

Nitrates

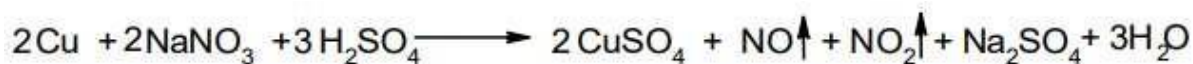
1. **Reaction with diphenylamine.** The reaction is based on the oxidation of diphenylamine to diphenyldiphenylquinoxaline diimine hydrosulfate, colored blue.



Technique:

2 drops of acidified diphenylamine solution are added to 1 ml of the drug solution; blue staining appears.

2. **Interaction with metals.** Nitrates are strong oxidizing agents. In an acidic medium, they oxidize metals with the formation of brown vapors – nitrogen reduction products:



Technique:

2-3 drops of water and concentrated sulfuric acid, a piece of metallic copper are added to 1 ml of the drug solution and heated; brown nitrogen dioxide vapors are released.

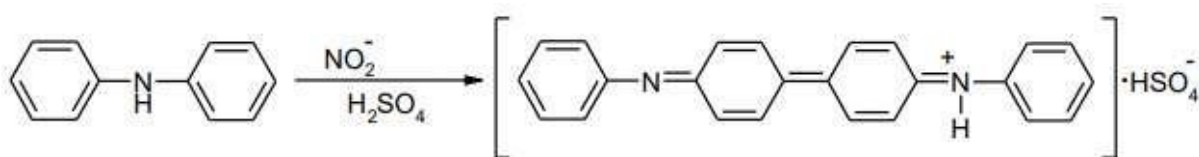
3. **Don't discolor the potassium permanganate solution.** The nitrogen atom in nitrates is in the highest degree of oxidation, therefore they cannot reduce Mn^{+7} and Fe^{+3} (unlike nitrites).

Technique:

Nitrates do not discolor a solution of potassium permanganate acidified with diluted sulfuric acid.

Nitrites

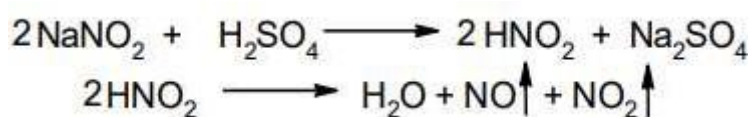
1. **Oxidation of diphenylamine in an acidic medium.** A blue coloration appears.



Technique:

2 drops of acidified diphenylamine solution are added to 1 ml of the drug solution; blue staining appears.

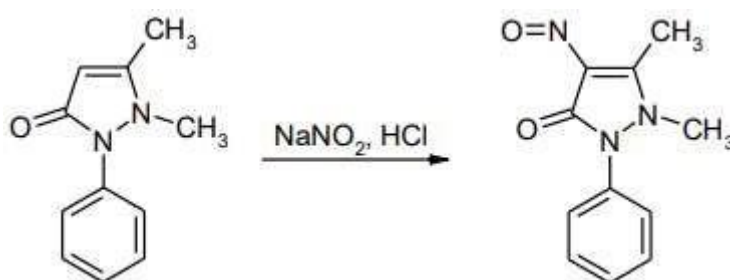
2. **Reaction with sulfuric acid.** When a solution of sulfuric acid acts on nitrite, nitrous acid is displaced, which decomposes to form nitrogen oxide (II) and nitrogen oxide (IV):



Technique:

1 ml of concentrated sulfuric acid is added to 1 ml of the drug solution, yellow-brown vapors of nitrogen oxides are released (unlike nitrates).

3. **Interaction with antipyrine.** Nitrites nitrosate the antipyrine at position C4. An emerald-green color appears:



Technique:

Several crystals of antipyrine are dissolved in a porcelain cup in two drops of diluted hydrochloric acid, 2 drops of nitrite solution are added: green staining appears.

Sulfates

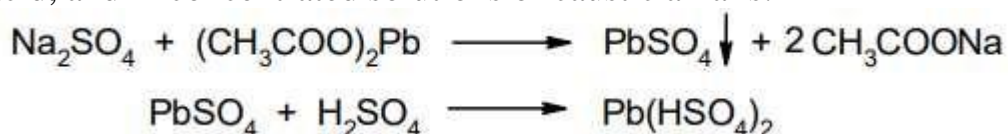
1. **Reaction with barium chloride.** Sulfates are precipitated by barium chloride; the resulting white precipitate BaSO_4 is insoluble in water and in dilute acids even when boiling:



Technique:

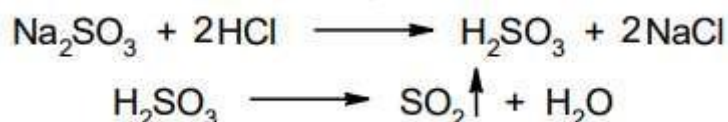
0.5 ml of barium chloride solution is added to 2 ml of sulfate solution; a white precipitate is formed, insoluble in diluted mineral acids.

2. **Interaction with lead acetate.** Sulfates are precipitated by lead salts in the form of a white precipitate of lead sulfate, soluble in concentrated sulfuric acid, and in concentrated solutions of caustic alkalis:



Sulfites

1. **Interaction with strong mineral acids.** From sulfides, when strong mineral acids act on them, sulfurous acid is displaced, which is an acid of medium strength and easily decomposes into sulfurous gas (odor) and water.



Technique:

2 ml of diluted hydrochloric acid is added to 2 ml of the sulfate solution and shaken; sulfur dioxide is gradually released, detectable by a characteristic pungent smell.

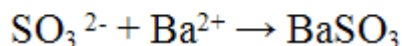
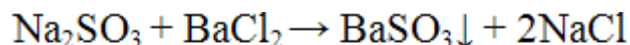
2. **Oxidation to sulfates.** Sulfites are strong reducing agents, while they themselves are easily oxidized to sulfate ions, which can be detected by precipitation (see "Sulfates"):



Technique:

When adding a few drops of iodine solution to the sulfite solution, the reagent discolors.

3. **Reaction with barium chloride.** Sulfides are precipitated by a solution of barium chloride. The released white precipitate of BaSO_3 is easily dissolved in dilute hydrochloric acid (unlike sulfates).



Technique:

ml of barium chloride solution is added to 2 ml of sulfate solution; a white precipitate is formed, soluble in diluted hydrochloric acid (unlike sulfates).

Phosphates

1. **Reaction with silver nitrate.** A yellow precipitate is formed, soluble in dilute nitric acid and ammonia solution.



Technique:

To 1 ml of phosphate solution, neutralized to a pH of about 7.0, add a few drops of silver nitrate solution; a yellow precipitate is formed, soluble in dilute nitric acid and ammonia solution (divide the precipitate into two test tubes and add dilute nitric acid to one and ammonia solution to the other).

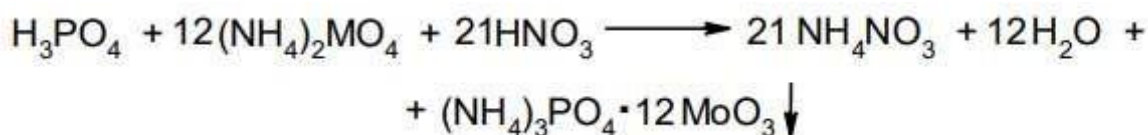
2. **Interaction with magnesium salts in the presence of ammonia.** A white crystalline precipitate of magnesium-ammonium phosphate, soluble in acetic acid, is formed:



Technique:

1 ml of ammonium chloride solution, 1 ml of ammonia solution and 0.5 ml of magnesium sulfate solution are added to 1 ml of phosphate solution; a white crystalline precipitate is formed, soluble in diluted mineral acids.

3. **Interaction with ammonium molybdate.** Solutions of phosphates in dilute nitric acid give a yellow staining with ammonium molybdate when heated, when standing, a yellow crystalline precipitate of ammonium phosphorolybdate is released:

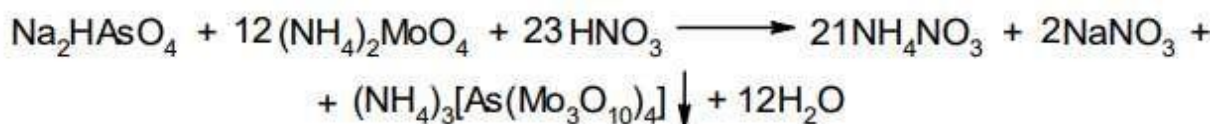


Technique:

2 ml of ammonium molybdate solution is added to 1 ml of phosphate solution in diluted nitric acid and heated; a yellow crystalline precipitate is formed, soluble in ammonia solution.

Arsenates

1. **Interaction with ammonium molybdate in a nitric acid medium.** Mixture $(\text{NH}_4)_2\text{MoO}_4$ with HNO_3 is called "molybdenum liquid". A yellow crystalline precipitate of a complex salt is formed:



Technique:

2 ml of ammonium molybdate solution and 3 ml of nitric acid solution are added to 1 ml of arsenate solution; a yellow crystalline precipitate is formed.

2. **Reaction with silver nitrate.** When arsenates interact with a solution of silver nitrate, a chocolate-colored silver arsenate precipitate is formed:



Technique:

0.5 ml of silver nitrate solution is added to 1 ml of arsenate solution; a chocolate-colored precipitate is formed.

3. **Reaction with potassium iodide.** If potassium iodide is added to arsenate in an acidic medium, then the iodide will be oxidized to free iodine and the solution will turn yellow:

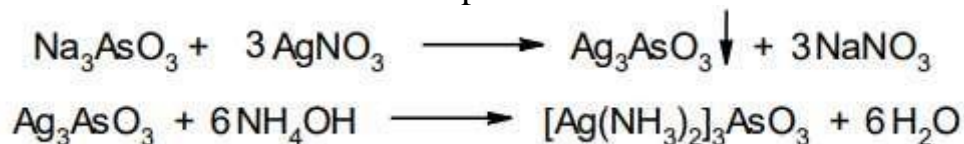


Technique:

1 ml of potassium iodide solution and 1 ml of nitric acid solution are added to 1 ml of arsenate solution; the solution acquires yellow coloring. If you add 2 ml of chloroform, the chloroform layer will turn purple.

Arsenites

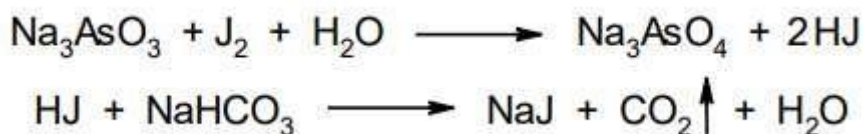
1. **Interaction with a solution of silver nitrate.** A precipitate of yellow silver arsenite is formed. The precipitate is soluble in nitric acid and in ammonia solution. The dissolution of the precipitate in excess of NH_4OH is explained by the formation of a soluble complex salt:



Technique:

0.5 ml of silver nitrate solution is added to 1 ml of arsenite solution; a yellow precipitate is formed. The precipitate is divided into two test tubes: add 2 ml of nitric acid solution to one, and 2-3 ml of ammonia solution to the other – the precipitate will dissolve in both test tubes.

2. **Interaction with iodine solution.** If an iodine solution is added to arsenite, the solution will discolor due to the reduction of iodine to iodide. The reaction is carried out in a bicarbonate medium to bind the resulting HI, which prevents the reversibility of the reaction:



Technique:

0.5 ml of iodine solution and 1 ml of sodium bicarbonate solution are added to 1 ml of arsenite solution; the solution is discolored.