

**XXIV МЕЖДУНАРОДНАЯ ОЛИМПИАДА ШКОЛЬНИКОВ «ТУЙМААДА»**

**XXIV INTERNATIONAL SCHOOL OLYMPIAD «TUYMAADA»**

**ХИМИЯ**

**CHEMISTRY**

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**II (экспериментальный) этап**

**Second (experimental) round**

**Младшая лига**

**Junior league**

**Yakutsk, 2017**

**Safety information**

**Please, observe the following rules when working in a chemical laboratory:**

* You must plan your work. Any experiments, which is not given in a problem, are prohibited.
* Keep work area neat and free of any unnecessary objects.
* Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron, secure long hair.
* Do not eat and drink in a laboratory!
* Never touch, taste, or smell any reagents.
* Wash hands before and after work.
* All experiments with smelling and toxic substances must be carried out in a fume cupboard.
* Always use a spatula or scoopula to remove a solid reagent from a container. Do not directly touch any chemical with your hands.
* Weigh out or remove only the amount of chemical you will need. Do not return the excess to its original container, but properly dispose of it in the appropriate waste container.
* Use a holder at heating of solutions or solid substances.
* Never point the open end of a test tube containing a substance at yourself or others.
* Never lean over dish where liquid is boiling or is heating.
* When it is necessary to smell chemicals in the lab, the proper technique is to cup your hand above the container and waft the air toward your face.
* If it is necessary, determine smell only in air flow from open dish.
* Add concentrated acid to water slowly. Never add water to a concentrated acid.
* Place chemical waste in appropriately labeled waste containers.
* With all the emerging issues immediately contact with the members of the jury.
* Immediately report any spills, accidents, or injuries to a teacher.

**Reagents and equipment for Problem 1**

**Reagents**

|  |  |  |
| --- | --- | --- |
| **Reagents** | **Set** | **Volume per 1 set, ml** |
| 10% aqueous-spirituous solution of K4[Fe(CN)6] | 1/2 | 10 |
| 0.2% solution of dithisone in CCl4 | 1/2 | 10 |
| 0.2 % aqueous-spirituous solution of dimethylglyoxime | 1/2 | 10 |
| 5% aqueous solution of KI | 1/2 | 10 |
| Saturated solution of KSCN | 1/2 | 10 |
| 5% Na2S | 1/2 | 10 |
| 0,2% solution of benzidine in 0.5 M HCl | 1/2 | 10 |
| 2M solution of NaOH | 1/2 | 10 |
| 25% ammonia solution | 2 for room | 10 |
| Acetone | 2 for room | 10 |
| 1% spirituous solution of alizarin  | 1/2 | 10 |
| Acetone : water : HCl (87:5:8) | 1/1 | 10 |

**Laboratory glassware and equipment**

|  |  |  |
| --- | --- | --- |
| **Glassware and equipment** | **Set** | **Amount per 1 set** |
| 150 ml graduated beaker (height not less than 12 cm) | 1/1 | 1/1 |
| Petri dish | 1/1 | 1/1 |
| Filter paper 2.5x12 cm | 1/1 | 2/1 |
| Capillaries (paperclips)  | 1/1 | 1/1 |
| Drying cabinet | 2 for room |  |
| Test tubes | 1/1 | 3/1 |
| Plastic beakers with red lids for system  | 1/1 | 1/1 |
| Vial for visualizing agents | 8 per 2 persons | 8/1 |
| Jar for ammonia solution | 2 for room |  |
| Jar for acetone | 2 for room |  |

**Reagents and equipment for Problem 2**

**Reagents**

|  |  |  |
| --- | --- | --- |
| **Reagent** | **Set** | **Volume per 1 set** |
| X solution | 1/1 | 100 ml |
| 0.05 M EDTA | 1/1 | 100 ml |
| Indicator methyl red | 1/4 | 10 ml |
| Ammonia solution | Shared | 100 ml |
| Ammonium acetate | 1/6 | 36 g |
| Salycilic acid | 1/6 | 12 g |
| 0.1 M FeCl3 | 1/1 | 50 ml |
| 0.1M H2SO4 | 1/1 | 25 ml |
| Dist. H2O | Shared | 400 ml |

**Laboratory glassware and equipment**

|  |  |  |
| --- | --- | --- |
| **Glassware and equipment** | **Set** | **Amount per 1 set** |
| Beaker | 1/1 | 1 |
| 10 ml volumetric pipette | 1/1 | 1 |
| Graduated tube | 1/1 | 1 |
| Paper filter | 1/1 | 5 |
| Erlenmeyer flask | 1 | 3 |
| 25 ml burette | 1 | 1 |

**Problem 1**

**Paper chromatography**

Mixture of four different cations from the list: Mn2+, Ni2+, Cd2+, Co3+, Al3+, Pb2+, Cu2+ и Zn2+ is given. Conduct separation of the mixture by paper chromatography and identify individual components.

**Technique**

Separation of the listed cations is conducted on a filter paper strip. It is recommended to save time conducting **parallel** chromatographic experiments.

1. Take a filter paper strip with dimensions 2x12 cm. Mark start line with pencil in a distance of 1 cm from the bottom edge and mark finish line in a distance of 0.5 cm from the top edge.

2. Transfer filter paper on a Petri dish. Gently apply a drop of the mixture to be analyzed on the start line with the tip of the paper clip. The drop must be spread out in the form of a circle with a diameter of not more than 4-5 mm. Cut the left and right corners of the bottom edge for uniform lifting of liquid on the filter paper.

3. Carefully, without soaking the walls of the dish, pour out the system of acetone, water and hydrochloric acid in a volume ratio of 87: 5: 8 into a graduated beaker so that the thickness of the solvent layer does not exceed 1 cm.

4. Lower filter paper strip with the drop of analyzed mixture vertically into a beaker and close it by a Petri dish. *Ensure that the eluent is lifted evenly and strictly vertically through the filter paper!* *The spot of analyzed mixture must not be touch the solvent!*

5. Duration of chromatography is 30 – 40 minutes. Process should be stopped after solvent reaches finish line. After that, gently take out paper from the beaker and transfer into a drying cabinet for 3 – 5 minutes for complete evaporation of solvent. Every cation is characterized by retardation factor:

$$R\_{f}=\frac{l}{L}$$

where l – distance traveled by cation; L – distance traveled by solvent.

|  |  |  |  |
| --- | --- | --- | --- |
| Cation | Rf | Cation | Rf |
| Ni2+ | 0.05 | Co3+ | 0.71 |
| Al3+ | 0.02 | Cu2+ | 0.95 |
| Mn2+ | 0.41 | Zn2+ | 0.096 |
| Pb2+ | 0.83 | Cd2+ | 1.0 |

6. With the values of *Rf* for each cation, moisten cotton with solutions of visualizing agents and apply solutions to certain parts of the filter paper to visualize colored spots. Make a conclusion about the presence of a particular cation in the mixture given to you:

|  |  |  |
| --- | --- | --- |
| Cation | Visualizing agent | Spot color |
| Ni2+ | dimethylglyoxime, ammonia vapor | red |
| Al3+ | alizarin, ammonia vapor | Pink |
| Mn2+ | benzidine, 2M NaOH | Blue |
| Pb2+ | solution of KI | yellow |
| Co3+ | solution of KSCN, drop of acetone | Blue |
| Cu2+ | Solution of K4[Fe(CN)6] | Red-brown |
| Zn2+ | dithisone | red |
| Cd2+ | Na2S solution | Green/Black |

Tasks:

1. Determine composition of the given solution.

2. Explain principles of paper chromatography.

**Problem 2**

**Determination of Pb2+ concentration in a mixture of Pb2+ and Zn2+**

**Determination of the total concentration of cations in analyzed solution**

Transfer 10 ml of analyzed solution with volumetric pipette into a flask for titration. Then pour 10 ml of EDTA with known concentration into the flask, add 2 – 3 drops of indicator methyl red and neutralize with 1-2 ml of ammonia solution until yellow color appears. Transfer 0.5 g of ammonium acetate and 0.1 g of salicylic acid into the neutralized solution and thoroughly mix until salts are dissolved. Titrate excess of EDTA with FeCl3 solution until appearing stable brown color of solution.

Repeat titration until three converging results are obtained and calculate average volume of FeCl3 solution spent on titration. Calculation of total concentration of cations are conducted using formula:

$$С\left(cations\right)=\frac{С\_{EDTA}V\_{EDTA}-С\_{FeCl3}V\_{FeCl3}}{V\_{aliquot}},$$

where *C(cations)* – total concentration of cations, mol/L; VEDTA – volume of EDTA, used for titration, ml; CFeCl3 – concentration of FeCl3 solution, mol/L; VFeCl3 - volume of FeCl3 solution, spent on titration, ml; Valiquot – volume of the aliquot of analyzed solution.

**Determination of Pb2+ concentration in analyzed solution**

Transfer 10 ml of analyzed solution with volumetric pipette into a beaker. Then add 1.5 ml of sulfuric acid with 0.1 M concentration and filter obtained solution into an Erlenmeyer flask using paper filter. The filter must be rinsed with 10 ml of distilled water. Then pour 10 ml of EDTA with known concentration into the flask, add 2 – 3 drops of indicator methyl red and neutralize with 1-2 ml of ammonia solution until yellow color appears. Transfer 0.5 g of ammonium acetate and 0.1 g of salicylic acid into the neutralized solution and thoroughly mix until salts are dissolved. Titrate excess of EDTA with FeCl3 solution until appearing stable brown color of solution.

Repeat titration until three converging results are obtained and calculate average volume of FeCl3 solution spent on titration. Lead concentration is calculated using formula:

$$С\left(Pb\right)=\frac{С\left(cations\right)V\_{aliquot}-(С\_{EDTA}V\_{EDTA}-С\_{FeCl3}V\_{FeCl3})}{V\_{aliquot}},$$

where C(Pb) – lead cations concentration, mol/L; *C(cations)* – total concentration of cations, mol/L; VEDTA – volume of EDTA, used for titration, ml; CFeCl3 – concentration of FeCl3 solution, mol/L; VFeCl3 - volume of FeCl3 solution, spent on titration, ml; Valiquot – volume of the aliquot of analyzed solution.

**Tasks:**

1. Determine total concentration of cations.

2. Determine concentration of lead in the solution.

3. Why is salicylic acid added to the titration flask?

4. Write the equations of reactions that occur during titration.