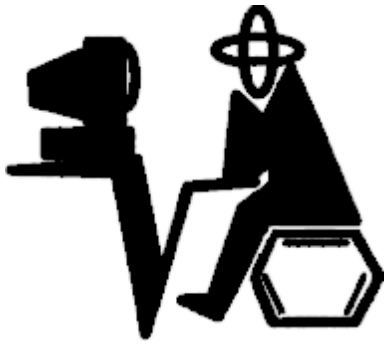
The 52nd INTERNATIONAL CHEMISTRY OLYMPIAD

April 23-29, 2018

Minsk



EXPERIMENTAL EXAMINATION

Minsk

2018

General directions

1. Total duration of the exam is 5 hours. You will be warned 30 minutes before the end of the exam. After the STOP command you should immediately stop your work and hand over the Answer Sheets to your lab assistant.

2. Wearing your lab coat and goggles (or your own correcting glasses) is obligatory during the whole experimental exam.

3. Take care when handling the acid and ammonia solutions!

4. Fill pipettes using rubber bulb only. It is absolutely prohibited to suck solutions into the pipette.

5. All participants have individual sets of glassware. However, all solutions will be shared between two adjacent participants. Please take this into consideration and do not spend more than half of the given solution.

6. You are supplied with clean burettes, do not spend solutions and time for rinsing when filling the burettes with the titrant for the first time.

7. To fill the tip of the burette, open the valve very slowly. This will allow you removing the air bubble out of the tip. If the problem persists, approach your lab assistant immediately. Remember that the amount of the titrant given is limited.

8. The burettes you are given have been thoroughly tested. However, some of the burettes may start leaking during the experiment. To be on a safe side, you are provided with two burettes. In case both of these are leaking, approach your lab assistant.

9. Pour used solutions into nearest sink (larger or smaller).

10. When working do not interfere with other Olympiad participants. Keep your working place in order.

11. Use the back side of the Answer sheets for your draft work.

Practical exam	Name	Country	Place #

Qualitative and quantitative analysis of antacid preparations

Antacids are drugs protecting stomach mucous membrane by lowering the gastric juice acidity.

You will have to identify substances found in antacids in the first part of the work. In the second part you will study the neutralizing capacity of an antacid (modeling of the physiological effect of the preparation in stomach). In the third part you will check the antacid quality determining titrimetrically its quantitative composition.

You can perform experimental work in any sequence of parts.

The lists of equipment and reagents are given separately for each part.

Part 1. Identification of antacids

Equipment and reagents

Hydrochloric acid, 1 M (labeled "HCl 1M")
Acetic acid, 1 M (labeled "CH_3COOH 1M")
Sodium hydroxide, 1 M (labeled "NaOH 1M")
Ammonium sulfate, 2 M (labeled "$(NH_4)_2SO_4$ 2M")
Lead nitrate, 0.0500 M (labeled "$Pb(NO_3)_2$ 0.0500 M", to be also used in Part 3)
Ammonia, 1 M (labeled "NH_3 1M")
Dropper with Methyl Orange, 0.1% (labeled "Methyl orange", to be also used in Part 2)
Glass test tubes, 8 pcs.
Glass beaker
Glass rod
Pasteur pipettes, 3 pcs.
Paper filters, 3 pcs.
Funnels, 2 pcs.

Practical exam	Name	Country	Place #

Samples of substances found in antacid preparations are placed in four test-tubes labeled **A**, **B**, **C,** and **D**. These substances are among the following listed: $NaHCO_3$, MgO, $CaCO_3$, $Mg(OH)_2{\cdot}MgCO_3$, $Al(OH)_3$, $Al_2(HPO_4)_3$.

An active ingredient (either a mixture or a double salt formed by the substances from the above list) of an antacid is placed in the test-tube labeled «**1**».

Using the reagents available:

1.1. Identify substances in the test-tubes **A**, **B**, **C**, and **D**. Fill in the Table and write down the reactions allowing the substances identification.

Label	Reactions (use "=" if the reaction occurs and "≠" if no reaction)	Conclusion about the composition
A		
B		
C		
D		

1.2. Determine the composition (cations, anions) of the sample in the test-tube «**1**». Support your choice by corresponding reactions.

Notes.
You can use filtration to separate precipitates from supernatants.
Remember that crystalline precipitates do not form immediately.
Heating may be required to dissolve some substances. Use water bath for heating.

Label	Reactions	Conclusion
1		Cations: Anions:

Practical exam	Name	Country	Place #

Part 2. Determination of neutralizing capacity of an antacid drug

Hydrotalcite, aluminum magnesium hydroxycarbonate $Mg_nAl_m(CO_3)_x(OH)_y \cdot 4H_2O$, is an efficient antacid.

The so-called neutralizing capacity is used to check an antacid quality. You will have to determine this value modeling the antacid effect of the preparation in stomach. In this part you will study the time dependence of neutralization of hydrochloric acid with hydrotalcite.

Equipment and reagents

Hydrochloric acid, 0.10 M (labeled "HCl 0.10 M")
Sodium hydroxide, 0.033 M (labeled "NaOH 0.033M")
Dropper with Methyl Orange, 0.1% (labeled "Methyl orange", to be also used in Part 1)
Burette
Cylinder, 50 mL
Beaker, 150 mL
Conical flasks, 2 pcs
Pipette
Wall-mounted clock

Procedure

Before you start working, write down the mass of the sample **2**. You will find the mass on the 150 mL beaker labeled «Sample **2**»:

m = _____ g.

Place 30 mL of distilled water into the 50 mL cylinder. Wet the weighted amount of the preparation (Sample **2**) with 2-3 drops of water and paste up with the glass rod. Keep adding water from the cylinder drop-wise, pasting up the mixture with the glass rod every time a new droplet is added. You are expected to finally get a yogurt-like mixture. *Only after this* pour out the rest of the water from the cylinder into the beaker. Using the glass rod, carefully break all the preparation lumps, if any. Then measure by burette 80.0 mL of 0.10 M HCl into the conical flask. Transfer the acid solution into the beaker and write down the time in the Table (record time with the 10 s accuracy). A stable opalescent suspension is expected to be formed.

2 min after the addition of hydrochloric acid, transfer by pipette 10.00 mL of the suspension into the conical flask, and add 1 drop of the Methyl Orange solution. Note that the solution to be titrated must be of *light-pink* color. An excess of the indicator complicates observation of the color change!). Titrate with 0.033 M NaOH solution till pure yellow color of the indicator appears. It is recommended to prepare the reference solution in the other conical flask. To do so, add 1 drop of each of the alkali and indicator solutions to the corresponding volume of water. Take the moment when the titration is completed as the experiment time. Write down the time in the Table (record time with the 10 s accuracy).

Practical exam	Name	Country	Place #

To study the time-dependent neutralization of the acid by the antacid, perform the titrations three more times making 5-7 min breaks between the end of each titration and beginning of the subsequent one. Record the results in the Table.

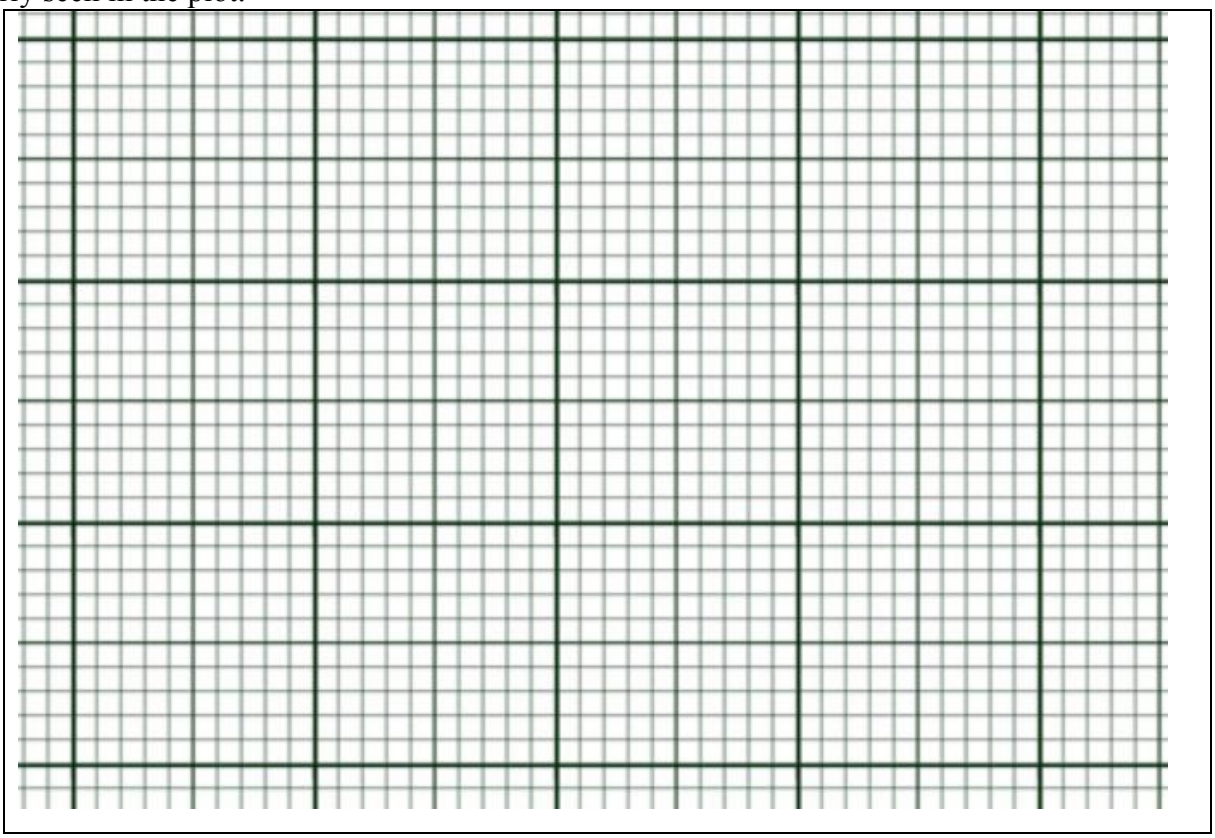
Note. Be sure the valve is tightly embedded in the burette, otherwise the solution can be leaking.

Time on the wall-mounted clock at the moment of HCl addition					
Time on the wall-mounted clock at the end of the titration					
Time since the experiment start, min					
The initial burette reading, mL					
The final burette reading, mL					
V(NaOH), mL					
The amount of the reacted HCl, mmol					
Neutralizing capacity, mmol/g					

Practical exam	Name	Country	Place #

2.1. Calculate the neutralizing capacity of the antacid as the amount (in mmol) of hydrochloric acid reacted with it. Reduce the results to the sample mass. Write down the results in the Table.

2.2. In the hereunder box, plot the curve of the hydrochloric acid neutralization in the coordinates: *time, min – neutralizing capacity, mmol HCl/g of the preparation*. Experimental points must be clearly seen in the plot.



Answer the questions:

2.3. Encircle the final pH value of the excessive acid titration providing for the maximum neutralizing capacity of the preparation:

a) 1; b) 2; c) 3; d) 4.

2.4. Write down the reaction equation explaining the difference in the neutralizing capacity of the preparation depending on the medium acidity.

Practical exam	Name	Country	Place #

Part 3. Determination of aluminum and magnesium content in the preparation.

Dissolution of the sample

Sample: ground tablets of a hydrotalcite containing preparation, labeled "**3**".

Equipment and reagents

Hydrochloric acid, 5 M (labeled "HCl 5M")
Beaker, 100 mL
Water bath
Volumetric flask, 100 mL

Procedure

In the 100 mL beaker, dissolve the weighted amount labeled "Sample **3**" in 6.0 mL of 5 M hydrochloric acid, heating the beaker on the water bath for 2 min.

Notes.

Some students will find their heaters under the fume hood.

It is impossible to dissolve the sample completely, since there are insoluble components in the preparation.

Cool down the beaker (you can do it under tap water), quantitatively transfer the suspension into the 100 mL volumetric flask, and bring up to the mark with distilled water. Mix carefully.

Titration 1

Reagents and equipment

EDTA disodium salt, 0.0500 M (labeled "EDTA 0.0500 M")
Lead nitrate, 0.0500 M (labeled "$Pb(NO_3)_2$ 0.0500 M")
Urotropine, 0.7 M (labeled "$(CH_2)_6N_4$")
Xylenol Orange, 0.2% solution (labeled "Xylenol orange")
Titration flask
Pipette
Beaker
Water bath
Burette

Procedure

Transfer by pipette an aliquot (10.00 mL) of the analyzed suspension into the titration flask. If the pipette cannot be placed inside the volumetric flask, use the beaker. Add ca. 40 mL of water, and then add from the burette 7.00 mL of 0.0500 M EDTA. Heat the mixture for 2–3 min using the water bath, and then cool down under tap water. Add 5 mL of 0.7 M urotropine solution. Add 3 drops of Xylenol Orange as the indicator. Titrate the excess of EDTA with 0.0500 M lead nitrate solution till the indicator coloration *irreversibly* changes from yellow-orange into pink (you can use a reference solution).

Practical exam	Name	Country	Place #

3.1. Write down the results into the Table:

The burette reading, mL		The consumed volume, mL
Initial	Final	

Your accepted volume (Titration 1): _____ mL.

Answer the questions

3.2. What metal is determined in the Titration 1? _____

3.3. Calculate the content of this metal in 1 g of the preparation:

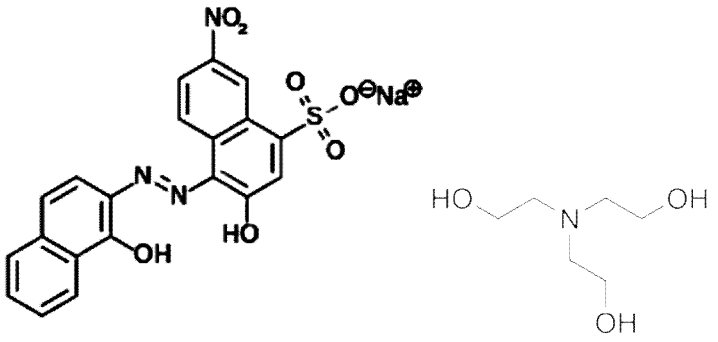
Calculations

The metal **1** content in 1 g of the preparation _____ g

Titration 2

Equipment and reagents

EDTA disodium salt, 0.0500 M (labeled "EDTA 0.0500 M")
Ammonia buffer solution, prepared by mixing of 1 volume of 1.5 M NH_4Cl and 2 volumes of 1.5 M NH_3 (labeled "Ammonia buffer solution")
Triethanolamine (see the structure below), a 1:3 aqueous solution, (labeled "Triethanolamine")
Eriochrome Black T (see the structure below), a solid mixture with NaCl, 1:100 by mass (labeled «Eriochrome Black T»)
Titration flask
Spatula



The structures of Eriochrome Black T and triethanolamine.

Practical exam	Name	Country	Place #

Procedure

Transfer an aliquot (10.00 mL) of the analyzed suspension into the titration flask, add 50 mL of water, 8 mL of aqueous triethanolamine solution as the masking agent, 15 mL of the buffer solution, the indicator mixture (with spatula), and immediately titrate with 0.0500 M EDTA till *complete* transition of the crimson-purple color of the indicator-containing complex into *pure* blue color of the free indicator (use the overtitrated solution as the reference one!).

Note:

Try adding the same quantity of the indicator to get coinciding results.

3.4. Write down the results into the Table.

The burette reading, mL		The consumed volume, mL
Initial	Final	

Your accepted volume (Titration 2): _____ mL.

Practical exam	Name	Country	Place #

Answer the questions

3.5. What metal is determined in the Titration 2? _____

3.6. Write down the reaction of the indicator transition in the equivalence point. Denote the indicator form shown in the figure above as H_2Ind^-, and that in the complex with the metal as $Met^{2+}Ind^{2-}$

3.7. Why the titrated metal is determined selectively? Write down the scheme of the other metal masking

3.8. Calculate the content of the titrated metal in 1 g of the preparation:

Calculations

The metal **2** content in 1 g of the preparation _____ g

3.9. Calculate the molar ratio *n*:*m* (aluminum:magnesium) in hydrotalcite $Mg_nAl_m(CO_3)_x(OH)_y \cdot 4H_2O$

Calculations

n:*m* = _____