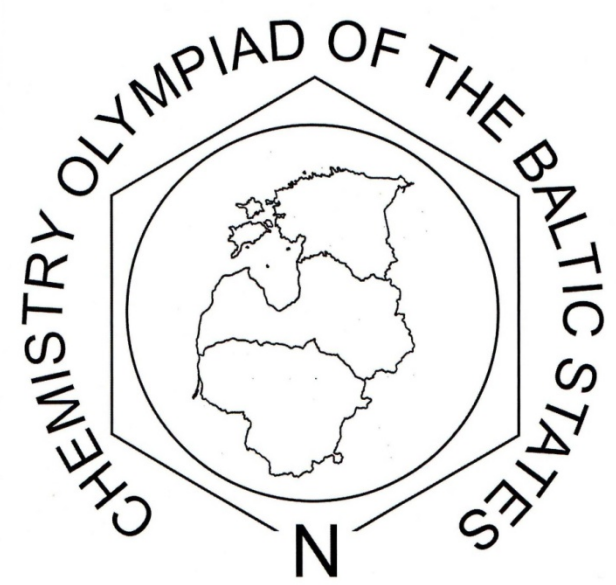
Student code: | | | |



23rd Chemistry Olympiad of the Baltic States

Daugavpils, Latvia

April 24-26, 2015

University of Daugavpils
http://du.lv/en

University of Latvia
http://www.lu.lv/eng/

JSC OlainFarm
http://olainfarm.lv/en/

PRACTICAL EXAMINATION, ENGLISH
SHORT MARKCHEME

" Scientia Vinces "

" Through knowledge you win "

Student code: | | | |

Problem 1. Analysis of hydrates (20 marks)

It was 24 marks for calculations and theoretical questions, 15 marks for determination of z value and 15 marks for titration of hydrate mixture.

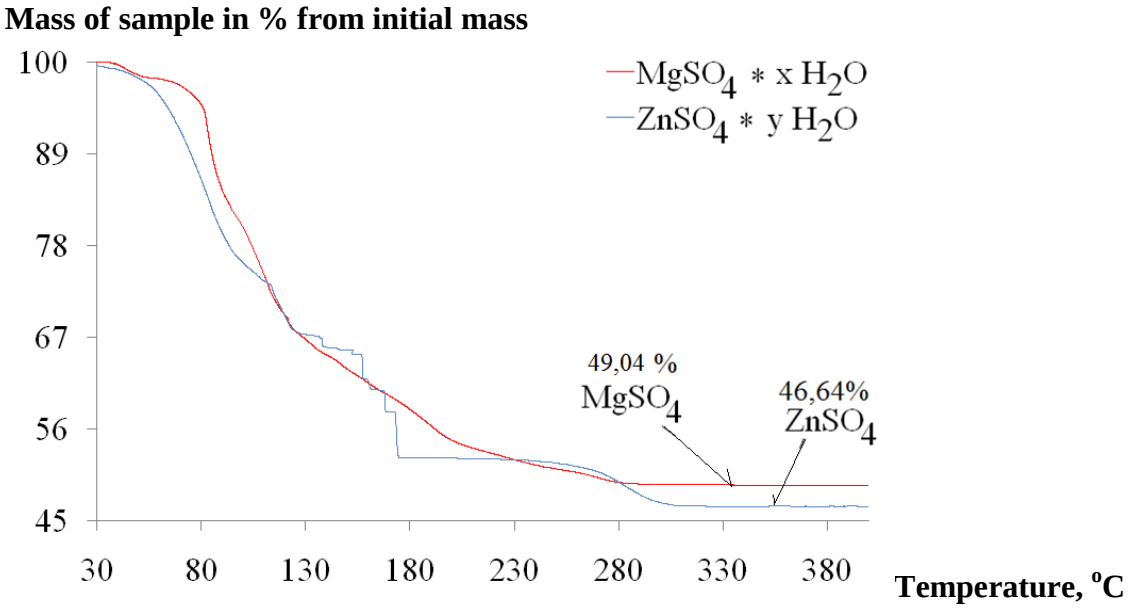
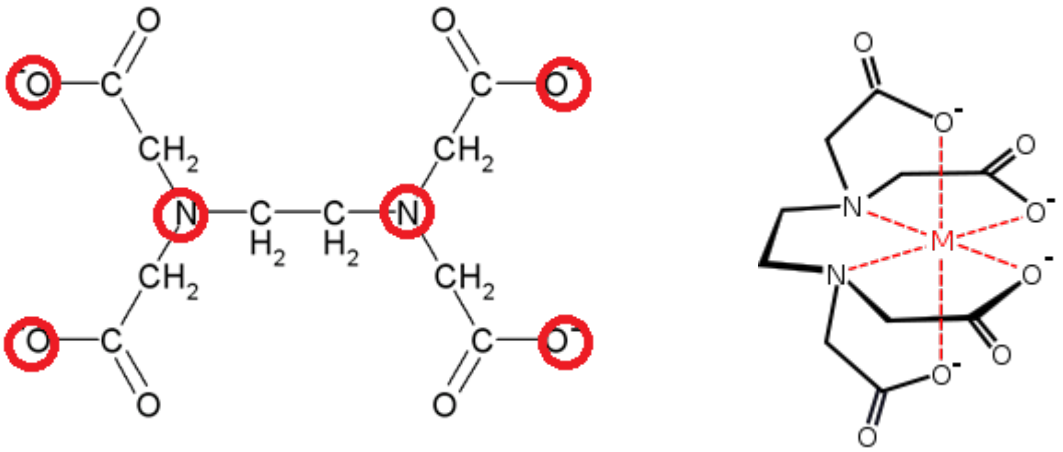


Figure 1. **Mass changes in % from starting mass during heating**

1.1. Use data from figure 1. and calculate composition of magnesium and zinc sulfate hydrates, find values of x and y. M($ZnSO_4$) = 161 g/mol; M($MgSO_4$) = 120 g/mol; M(H_2O) = 18 g/mol.

Composition of $MgSO_4 \cdot x\ H_2O$	Composition of $ZnSO_4 \cdot y\ H_2O$
x = 6.9	y = 10.2

Composition of zinc sulfate hydrate seems to be unrealistic (this composition is possible if hydrate is held in high humidity). So you have to analyze composition of zinc sulfate by titration with EDTA. EDTA is hexadentate ligand. It's structural formula is shown bellow.



1.2. In structure above circle atoms which binds to metal ions when EDTA anion reacts with metal ions.

Procedure for titration for determination of $ZnSO_4 \cdot z\ H_2O$ composition.

Student code: | | | |

1.4. Calculate average $z_{avg.}$ value (from three results) and random error for z abbreviated as Δz.

$$\Delta z = \frac{S_n \cdot 4{,}30}{\sqrt{3}}$$ ***, where S_n - standard deviation***

$$S_n = \sqrt{\frac{(z_1 - z_{avg.})^2 + (z_2 - z_{avg.})^2 + (z_3 - z_{avg.})^2}{2}}$$

Average value $z_{avg.}$	S_n	Δz
it should be approximately 6		

Some students were smart and wrote that all titration results are equal, so standart deviation is zero, as well as Δz = 0. It was acceptable.

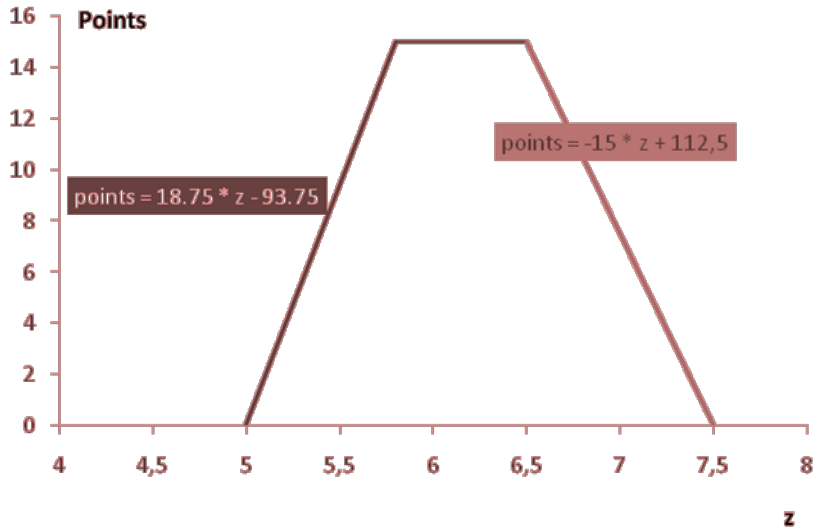
1.5. Calculate difference of z between your average value and value obtained from TG measurements.

Difference = | 10,2 - 6 | = ~ 4

1.6. Compare difference calculated in 1.5. and value of random error Δz and comment on possible systematic error:

There is systematic error if compare titration results with TG results: YES / NO (circle correct).

Allocation of marks for z determination. Acceptable interval was quite wide as hydrate composition may vary depending on relative humidity. Almost all students received good points.



Third part of this laboratory experiment is to determine composition of $MgSO_4 \cdot x\ H_2O$ (x value as calculated in 1.1.) and $ZnSO_4 \cdot z\ H_2O$ (z value as determined in previous titration) mixture. Here comes one problem with this practical problem, as magnesium sulfate hydrate composition was different from DTA/TG results. It made impossible calculations in task 1.8., but student titration results were recalculated using exact (not DTA/TG determined) composition.

Student code: | | | |

Procedure for titration for determination of mixture composition

1.7. Calculate average volume of EDTA solution used.

Average volume of EDTA solution used: _xx.xx mL

This volume was used for recalculation of hydrate mixture composition.

1.8. Calculate mass fraction of $ZnSO_4 \cdot z\ H_2O$ in mixture.

Only 3 marks for calculation method.

Mass fraction of $ZnSO_4 \cdot z\ H_2O$ in hydrate mixture is %

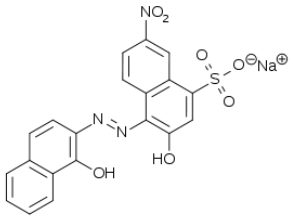
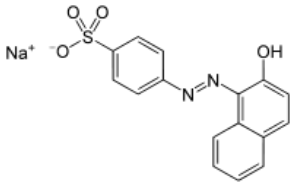
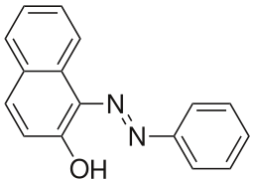
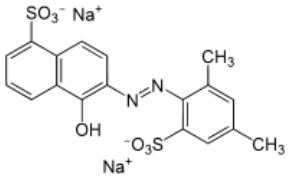
15 marks were allocated as follows:
as all students had hydrate mixture 50% : 50% (± 1 %) student titration results were tested on relative error of titration volume used
if absolute value of relative error was <2.5 %, then it was 15 points
if absolute value of relative error was <10 %, then it was some points

Three students received max marks; two of them had no 1st, 2nd or 3rd place prize and received special prizes from University of Daugavpils.

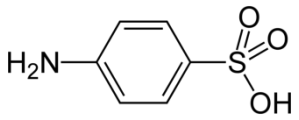
Student code: | | | |

Problem 2. Synthesis of β-naphtol orange (20 marks)

2.1. Choose correct structure for 2-naphthol orange from structures provided bellow. Mark correct structure with X.

			
	X (all students were right)		

2.2. Diazotization of Sulfanilic Acid

a) Calculation of sodium nitrite mass:
b) Write reaction equation for diazotization of sulfanilic acid: 

2.3. Orange II Synthesis

a) Calculation of 2-naphtol mass:
Reaction equation: β-naphtol (draw structural formula) +

1. If you are not able to do 2.4. part (due timing or other reasons), place filtered precipitate on preweighted Petri dish. Report mass of precipitate and ask lab assistant signature for conformation. Allow the moist dye to dry overnight. YOU DO NOT HAVE TO DO THIS IF YOU ARE ABLE TO DO 2.4. PART - RECRYSTALIZATION.

Mass of precipitate:	Lab assistant's signature:

2.4. Recrystallization of product

Mass of precipitate:	Lab assistant's signature:

Student code: | | | |

2.5. Additional questions

1. Calculate theoretical yield of product (in grams):

5 grams

2. Calculate percentage yield of product using mass from 2.3.5. or 2.4.2. questions:

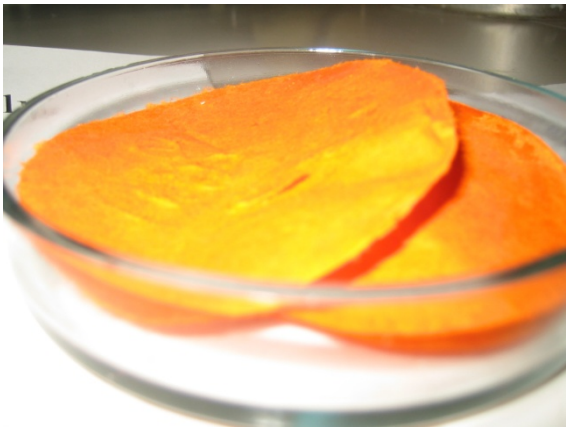
3. Temperature in this reaction is important factor as it must be kept bellow 10^{o}C. Why is diazonium salt so unstable, draw structure of compound which can be formed if temperature rises?

hydrolysis

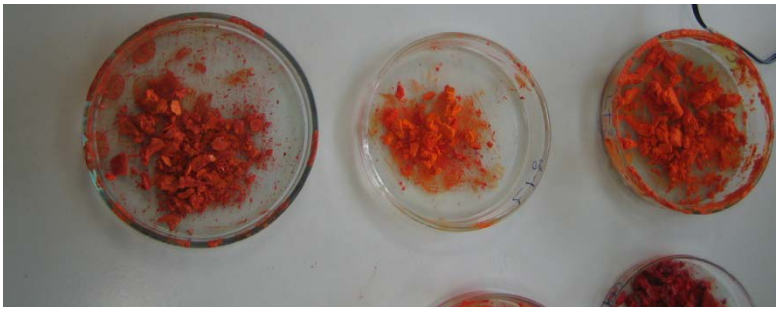
There was 25 marks for theoretical questions and calculations.

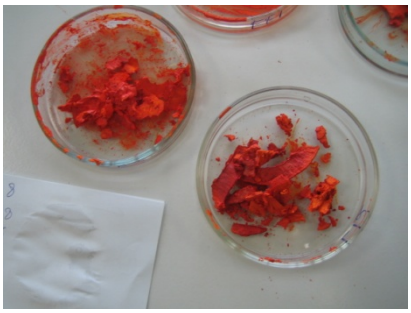
25 marks for mass of product obtained BUT:

- 25 marks are awarded if student recrystalize product and mass is 4-5 grams;
- if student obtain some product after recrystalization then number of points linearly decreases from 25 to 0 if mass obtained (after drying) is 4 grams to 0 grams. Best result in terms of mass was 2.4 grams after recrystalization.
- if student did not recrystalize their products then max number of points was 15 marks; this number of marks were awarded to students who had mass 4.5 till 5 grams (after drying; none of students had mass over 5 g); then number of marks linearly decreases from 15 to 0 it mass decreases from 4.5 grams to 1 gram. If mass was smaller than 1 g and recrystalization were not done then no marks were awarded.
- 3 students (EE4, LV1, LV7) did not obtain any product.

 <-- only colored filter paper obtained

Student code: | | | |





Some of obtained products.

Quality of products were graded depending on compound color by doc. Jelena Kirilova, author of this practical problem. Max marks 8 were awarded if product has light color, it was awarded to two students. If white sodium chloride crystals were seen in final product then no points were awarded.