Face your challenge,
Be smart

PRACTICAL EXAMINATION

JULY 18, 2013
MOSCOW, RUSSIA

21934 characters in Problems Booklet

General Directions

- **safety rules** follow ones given in the Preparatory problems booklet, no eating or drinking in the lab.
- **violating safety rules** you get one warning only; offend again: you are disqualified.
- **problems and answers booklet** 24 pages (incl. cover sheet and Periodic table of elements) with 3 problems. Start with problem 1.
- **time** 5 h; 15 min for reading before start. 30 min warning before the end.
- **your student code** write this on **every page**.
- **answers** only in the answer boxes in the booklet, nothing else will be graded. Relevant calculations have to be shown.
- **use only the pen, pencil and calculator provided.**
- **burette** read it as accurately as possible.
- **more chemicals** needed? Ask your lab assistant. No penalty for this with an exception of the hereunder.
- **each extra aldehyde, 2,4-dinitrophenylhydrazine, 50 mL of HCl, EDTA titrant or portion of a polymer solution:** a penalty of 1point out of 40.
- **Be very careful! No replacement if you break your viscometer!**
- **questions** concerning safety, apparatus, chemicals, toilet break: **ask your lab assistant.**
- **chemical waste** put it only in the designated 800 mL beaker labeled “WASTE”.
- **official English version available** on request **for clarification only.** Ask your lab assistant.
- **after the stop signal** put your booklet and all graph paper in the envelope (don’t seal), leave at your table.
- **You must stop your work immediately after the stop signal has been given. A 5 min delay will result in zero points for the current task.**
- **During the Practical exam some of the glassware and plastics are expected to be used several times. Clean it carefully.**

List of Chemicals

Reagent	Quantity	Placed in	Labeled	Safety
Problem 1				
2,4-Dinitrophenylhydrazine	200 mg each, 2 vials	small screw neck vial	2,4-dinitrophenylhydrazine	H228, H302
Sulfuric acid, concentrated	1 mL each, 2 tubes	Plastic tube with screw neck	H_2SO_4 concentrated	H314
Aldehyde solution 1 mmol in ethanol	4 mL each, 2 bottles	30 mL small glass-stoppered bottle	Aldehyde 1 and Aldehyde2	H319 and H302
Ethanol	30 mL	glass-stoppered bottle	Ethanol	H225
NaOH solution (used in problems 1 and 2)	27 mL	60 mL glass-stoppered bottle	NaOH 2M	H314
Acetone	30 mL	amber glass screw neck vial	Acetone	H225, H319, H336
Problem 2				
EDTA, 0.0443M* standard solution	70 mL	125 mL glass-stoppered bottle	EDTA 0.05M	H319
HCl, 0.0535M* standard solution	70 mL	125 mL glass-stoppered bottle	HCl	H314, H335
Methyl orange, 0.1% in water	25 mL	dropping bottle	Methyl orange	H301
Murexide indicator, solid mix with NaCl (1:250 by mass)	in 10 mL bottle	small screw neck vial	Murexide	
Sample of water	500 mL	0.5 L plastic can	Water sample	
Problem 3				
Poly(vinyl) alcohol	40 mL each, 5 vials	amber glass screw neck vial	P1, P2, P3, P4 and X	
To be used in all problems				
Distilled water	500 mL	Plastic wash bottle	H_2O	
To be shared by students, on the common table				
Sodium hydrocarbonate	800 mL	800 mL beaker	$NaHCO_3$	

***The concentration indicated on the label is approximate. The exact values are indicated in the table.**

Labware and equipment

Item	Quantity
On every working place	
5 mL Plastic tube with screw neck labeled “1” with your student code	1
5 mL Plastic tube with screw neck labeled “2” with your student code	1
Lab stand	1
50 mL beaker	2
25 mL beaker	2
25 or 50 mL beaker	1
Magnetic stirrer	1
Stirring bar	2
Glass filter	2
Adapter	1
50 mL round bottom flask	1
Water-jet pump	1
2 mL pipette	2
5 mL pipette	2
Pipette filler	1
Spatula	2
500 mL plastic washer bottle	1
800 mL beaker for waste	1
10 mL measuring cylinder	1
Filter paper, round	2
Scissors	1
Filter paper	2
Glass rod	1
pH indicator papers (in a zipper-bag)	3
Viscometer	1
Stop-watch	1
30 mL rubber bulb	1
Ruler	1
Marker	1
25 mL burette	1
25 mL pipette	1
Plastic funnel	1
Erlenmeyer flask	2
Test strips for determining total dissolved solids content in zipper bag	1
Paper tissues (on the corner of each table, to be shared between 3 students)	1 package
Plastic basket	1
Graph paper	4 sheets
pH scale (in zipper bag)	1
On the tables for the common use	
Filter paper, round	
Filter paper	
Gloves	
Balances	
Bottle labeled “H_2O dist.”	
Thermometer immersed in H_2O	
Measuring cylinder 100 mL	
pH-meter	

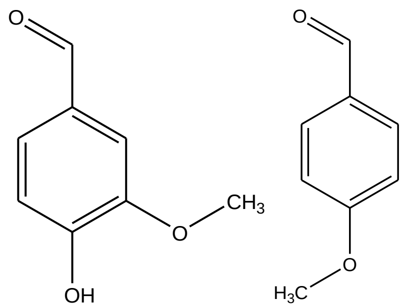
*If you need more filter paper, you can find it at the table of common use.

Problem 1	Student code________	Quest.	1	2	3	4	5	Total
		Marks	3.5	1.5	1	3	35	44

Problem 1. Synthesis of 2,4-dinitrophenylhydrazones (13 points)

Hydrazones belong to the class of *imines*, which contain a nitrogen-nitrogen single bond adjacent to a carbon-nitrogen double bond. Hydrazones are formed when NH_2-containing hydrazine reacts with aldehydes or ketones under appropriate conditions. Because the hydrazone derivatives of the carbonyl compounds are often stable, crystalline, highly colored solids, they are used to confirm the identity of aldehydes and ketones.

In this task you will have to identify two substituted benzaldehydes (shown below) by studying the products of their reactions with 2,4-dinitrophenylhydrazine.



Procedure

Preparation of 2,4-dinitrophenylhydrazones

Attention! Do not carry out two syntheses simultaneously, otherwise a beaker can fall from the magnetic stirrer, and you will lose your reaction mixture.

Equip one 50 mL beaker with a magnetic bar. Fix the beaker on the stirrer using the metal ring attached to the stand. Place the content of vial (200 mg of 2,4-dinitrophenylhydrazine) into the beaker and start stirring carefully. ***Only in the presence of your lab assistant,*** carefully pour one sample of concentrated sulfuric acid (1 mL) onto the solid. Using pipettes add 1.6 mL of water and 4 mL of ethanol to the reaction mixture. Then using a pipette add dropwise the content of the aldehyde solution bottle (either "**aldehyde 1**" or "**aldehyde 2**", each contains 1.00 mmol of the aldehyde). Bright precipitate starts forming at once. Continue stirring for 10 min, then add 10 mL of water and stir for another 3 min.

Separation and purification of the product

Using scissors carefully cut out a filter paper circle, *ca.* 1 cm bigger in diameter than that of the glass filter. Wet the filter circle with water, and carefully put it on the filtering surface. The paper filter should fit evenly and tightly. **If you fail to cut out an even circle, take a new filter from the table of common use and repeat cutting out**. Assemble the filtering apparatus. Remove the stirring bar from the beaker using the spatula and transfer the reaction product onto the filter. Turn on the water-jet pump (**seek for help from your lab assistant if you experience difficulties**) and filter out the precipitate. Put a little amount of water in the beaker and transfer the leftover product onto the filter. Wash the solid on the filter with water until the pH of the drops coming out the funnel are neutral. (Use the **WASTE** beaker to pour the round-bottom flask). Then wash the solid twice with ethanol using no more than 3 mL each time (**Note: Hydrazone is slightly soluble in ethanol**). Dry out the solid on the filter with working water-jet pump, loosening and squeezing the product with a glass rod from time to time. After *ca.* 20-30 min carefully transfer the dried powder into the self-made filter paper box for the final drying in the air. Put the box with the product in a safe place (e.g. on the shelf). **Turn off the water-jet pump when you do not use it!** As soon as your products seem dry, we advise you weigh them to avoid queuing at the balances. To collect the products, use the plastic tubes with your student code. Fill in the answer box below. **Note:** The products you synthesized will be further re-examined by lab staff.

Repeat the above procedures with the other aldehyde.

Plastic tube **1**	Plastic tube **2**
Mass of empty tube________________ mg	Mass of empty tube ________________ mg
Mass of tube with product___________ mg	Mass of tube with product___________ mg
Mass of product ___________________ mg	Mass of product ___________________ mg

Lab assistant's signature ________________

1.1. Write down the structures of 2,4-dinitrophenylhydrazine and both products.

Problem 1	Student code________	Quest.	1	2	3	4	5	Total
		Marks	3.5	1.5	1	3	35	44

1.2. What kind of stereoisomerism (if any) is possible for these hydrazones? Tick the appropriate box

☐ R/S ☐ E/Z ☐ threo/erythro ☐ manno/gluco ☐ D/L

2.1. What is the role of sulfuric acid in 2,4-dinitrophenylhydrazone formation? Tick the appropriate box.

☐ stoichiometric reagent ☐ catalyst ☐ reducing agent ☐ oxidizing agent

2.2. How would the rate of the reaction change, if the synthesis is carried out in neutral medium? Tick the appropriate box.

☐ highly increase ☐ slightly increase
☐ not change ☐ the reaction would proceed very slow

2.3. How would the rate of the reaction change, if it is carried out in alkaline medium? Tick the appropriate box.

☐ highly increase ☐ slightly increase
☐ not change ☐ the reaction would not proceed

Characterization

Place **small** amount ("on the tip of a spatula") of each product in an individual 25 mL beaker. Add 10 mL of acetone to each beaker. **The best result will be obtained if the color and color intensity in each beaker are similarly yellow.** Pour 5 mL of $NaHCO_3$ solution into each beaker. Stir the resulting mixtures with the glass rod using different ends.

3.1. Record your observations of the solutions color change in the box.

☐ The color does not change in either beaker
☐ Color changes significantly in both beakers
☐ Color changes significantly only in one beaker

Add 2 mL of NaOH solution to each of the resultant mixtures from the question **3.1**. Stir the reaction mixtures with the glass rod.

3.2. Record your observations of the solutions color change in the box.

☐ The color does not change in either beaker
☐ Color changes significantly in both beakers
☐ Color changes significantly only in one beaker

Problem 1	Student code________	Quest.	1	2	3	4	5	Total
		Marks	3.5	1.5	1	3	35	44

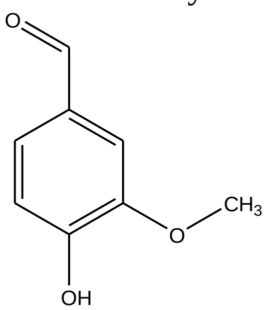
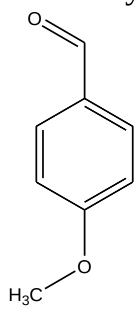
4.1. What structural features of your products explain the color change in the reaction with $NaHCO_3$? Tick the appropriate box.

☐ presence of MeO group at position 4 in the benzene ring;
☐ presence of MeO group at position 3 in the benzene ring;
☐ presence of the OH group at position 4 in the benzene ring;
☐ presence of both MeO and OH groups.

4.2. Which of the listed processes is responsible for the color change observed in the reaction of 2,4-dinitrophenylhydrazones with aqueous NaOH? Tick the appropriate box.

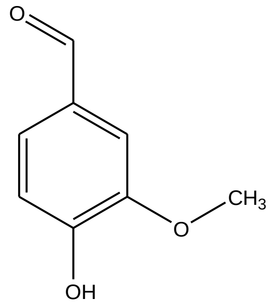
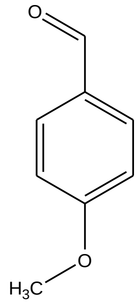
☐ alkaline hydrolysis ☐ dehydration ☐ hydration
☐ deprotonation ☐ dehydrogenation

4.3. Draw the structures of the main organic species present in each test reaction medium in the answer box below.

Initial aldehyde: 	Initial aldehyde: 
Solution of $NaHCO_3$	Solution of $NaHCO_3$
Solution of NaOH	Solution of NaOH

Problem 1	Student code________	Quest.	1	2	3	4	5	Total
		Marks	3.5	1.5	1	3	35	44

5. Put the numbers **1** or **2** under each structure. Calculate the percent yields of both hydrazones

 Number:______________ Yield calculation:	 Number:______________ Yield calculation:
Yields: Number 1__________________ % Number 2_________________ %	

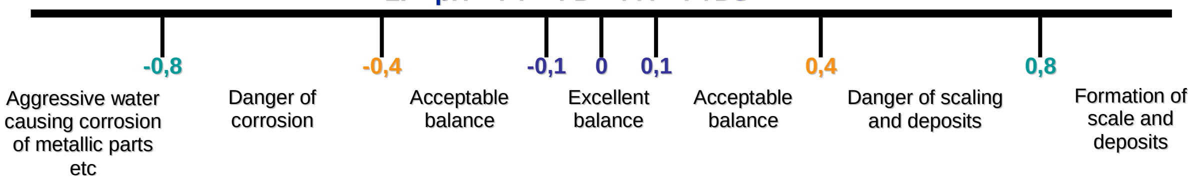
Replacement or extra chemicals	Lab assistant signature	Penalty
________________________ ________________________ ________________________	________________________ ________________________ ________________________	___________ ___________ ___________

Problem 2	Student code_______	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	27	0	5	25	0	4	8	1	9	79

Problem 2. Determination of the Langelier Saturation Index of a pool water (12 points)

The Langelier Saturation Index (LI) is a measure of a swimming pool water corrosivity as well as its ability to dissolve or deposit calcium carbonate. If LI is approximately zero, the water is considered "balanced". If the LI is a positive number, the water tends to deposit calcium carbonate and is scale-forming. If the LI is a negative number, the water is corrosive and dissolves calcium carbonate. The LI is a combination of the physical values factors taken from Table 1 and can be calculated by the formula:

LI = pH + FT + FD + FA – FTDS



pH : pH value
FT : Temperature factor
FD : Calcium hardness (CH) factor
FA : Total alkalinity (TA) factor
FTDS : Total dissolved solids (TDS) factor

Table 1. Values and corresponding factors

Temperature, °C	FT	Calcium hardness (CH), mg/L $CaCO_3$	FD	Total alkalinity (TA), mg/L $CaCO_3$	FA	Total dissolved solids (TDS), mg/L NaCl	FTDS
0	0.0	5	0.3	5	0.7	0	12.0
3	0.1	25	1.0	25	1.4	-	-
8	0.2	50	1.3	50	1.7	1000	12.1
12	0.3	75	1.5	75	1.9	-	-
16	0.4	100	1.6	100	2.0	2000	12.2
19	0.5	150	1.8	125	2.1	-	-
24	0.6	200	1.9	150	2.2	3000	12.25
29	0.7	250	2.0	200	2.3	-	-
34	0.8	300	2.1	300	2.5	4000	12.3
41	0.9	400	2.2	400	2.6	-	-
53	1.0	600	2.35	800	2.9	5000	12.35
-	-	800	2.5	1000	3.0	-	-
-	-	1000	2.6	-	-	6000	12.4

Problem 2	Student code_______	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	27	0	5	25	0	4	8	1	9	79

In this task you will have to determine the LI value of the given water sample. Note that hardness is expressed as the equivalent to the concentration of $CaCO_3$ (expressed in mg/L). Total alkalinity being the acid equivalent to the total amount of carbonate and hydrocarbonate, also expressed in mg/L of $CaCO_3$, whereas TDS is recalculated as NaCl concentration (mg/L).

Procedures

Calcium hardness is determined by complexometric titration with EDTA (Na_2H_2Y). This is performed in a strongly alkaline medium to mask magnesium (large amounts of Mg^{2+} interfere due to the co-precipitation of calcium with $Mg(OH)_2$; moreover, the complexometric indicator is also adsorbed on $Mg(OH)_2$, which impairs the observation of its color change). When the alkali is added, titration should be carried out immediately to avoid the deposition of $CaCO_3$.

1.1. Write down equation of the reaction occurring during titration with Na_2H_2Y:

Procedure for calcium determination

a) Put the standard solution of **EDTA** (exact concentration of 0.0443 M) in the burette.
b) Pipette a 20 mL aliquot of the **Water sample** into an Erlenmeyer flask.
c) Add 3 mL of 2M NaOH solution with the 10-mL measuring cylinder.
d) Add murexide indicator with spatula to obtain noticeably pink solution.
e) Within few minutes titrate the mixture with EDTA until the indicator color changes from pink to purple.

1.2. Fill in the table 2.

Table 2

Calcium titration	**Titration No**			
Initial reading of the burette, mL				
Final reading of the burette, mL				
Consumed volume, mL				

Accepted volume, mL_____

2. Calculate the hardness of the water sample in mg/L $CaCO_3$. Write down the result in Table 4 (see question 7).

Your work:

Problem 2	Student code_______	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	27	0	5	25	0	4	8	1	9	79

Measurement of pH. Locate a pH meter in the lab (or ask your lab assistant).

a) Place about 70-90 mL of the water sample into a clean Erlenmeyer flask.

b) Remove the protective cap from the pH-meter (keep the cap standing, since there is solution in it).

c) Rinse the electrode with distilled water using a plastic wash bottle.

d) Turn the meter on by sliding the ON/OFF switch.

e) Immerse the meter in the solution to be tested and stir gently by swirling the flask.

f) Place the flask on the table and wait until the reading stabilizes (not more than 1 min).

g) Read and record the pH value.

h) Switch the meter off, rinse the electrode with distilled water and place the protective cap back (in case of queuing, pass over the meter to the next student).

3.1. Write down the pH value in Table 4 (see question 7).

3.2. Which form of carbonic acid predominates in your water sample?

Confirm your choice with calculation and tick one box.

Note. The dissociation constants of carbonic acid are: $K_1 = 4.5\cdot10^{-7}$; $K_2 = 4.8\cdot10^{-11}$.

Your work:

☐ Carbonate ☐ Hydrogen carbonate ☐ Carbonic acid

3.3. Write down the ionic equation of the predominant reaction of titration of the water sample with HCl.

Determination of total alkalinity. To obtain the value of the total alkalinity the water sample should be titrated to H_2CO_3. An acid-base indicator used is methyl orange, which starts changing its color from yellow to orange at pH of about 4.5.

Problem 2	Student code_______	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	27	0	5	25	0	4	8	1	9	79

a) Rinse the burette with distilled water and fill it with the standard HCl solution (exact concentration of 0.0535 M).

b) Pipette a 50.0 mL aliquot of water sample into an Erlenmeyer flask and add 3 drops of methyl orange solution.

c) If the sample is orange prior to addition of the acid the total alkalinity is zero. If the solution is yellow titrate it with the standard acid solution until the first noticeable color change towards orange is observed. Record the volume of the titrant used.

4.1. Fill in the Table 3.

Table 3

Alkalinity determination	**Titration No**			
Initial reading of the burette, mL				
Final reading of the burette, mL				
Consumed volume, mL				

Accepted volume, mL______

4.2. Calculate the total alkalinity (in mg/L $CaCO_3$). Write down the result in Table 4 (see question 7).

Your work:

5. Temperature measurement. Read the thermometer located at the table of common use and write down the value into Table 4 (see question 7).

6. TDS determination in the water sample with the test strip.

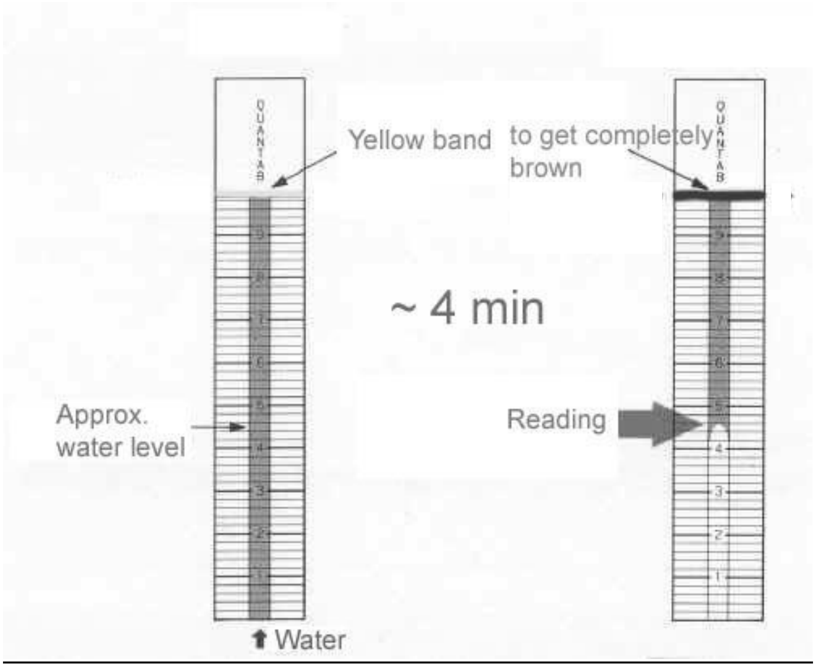
a) Fill a beaker with the water sample to a level of about 3 cm of height. Immerse the strip into water; be sure that the yellow band on the top of the strip does not touch the liquid.

b) Wait for 3–4 min until the yellow band turns completely brown. Take the reading as shown in the picture hereunder, reading result to one decimal digit.

c) Report the reading:

Problem 2	Student code________	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	27	0	5	25	0	4	8	1	9	79

d) Find your TDS concentration as that of NaCl, mg/L in the table to the right of the picture.
e) Write down the concentration of NaCl in Table 4 (see question 7).



Reading	NaCl conc., mg/L
1.4	360
1.6	370
1.8	420
2.0	430
2.2	470
2.4	530
2.6	590
2.8	660
3.0	730
3.2	800
3.4	880
3.6	960
3.8	1050
4.0	1140
4.2	1240
4.4	1340
4.6	1450
4.8	1570
5.0	1700

7. Fill in all the blank boxes in the Table 4. Calculate LI and write down the result in Table 4. Take the values of the factors to the accuracy of two decimal digits.

Your work:

Problem 2	Student code_______	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	27	0	5	25	0	4	8	1	9	79

Table 4. Calculation of LI of the water sample

Water sample Number ______					
CH, mg/L $CaCO_3$	TA, mg/L $CaCO_3$	*t*, °C	pH	TDS, mg/L NaCl	**LI**
FD	FA	FT		FTDS	

Theoretical questions. Water balance correction.

If LI significantly deviates from zero, it is needed to be adjusted to zero.

Imagine you are given a sample of pool water analyzed as you have done above. The results of the analysis are: CH = 550 mg/L, FD = 2.31, TA = 180 mg/L, FA=2.26, t° = 24°C, FT = 0.6; TDS = 1000 mg/L , FTDS = 12.1, pH = 7.9, LI = 0.97.

The pool serviceman added 10 mL of 0.0100 M solutions of reagents ($NaHCO_3$, NaOH, $NaHSO_4$, $CaCl_2$, EDTA (disodium salt dihydrate) and HCl) to different pool water samples 200 mL each (one reagent for one sample).

8. Decide whether $CaSO_4$ is deposited upon addition of $NaHSO_4$.

Note: $CaSO_4$ solubility product is $5 \cdot 10^{-5}$. Assume no precipitate of $CaCO_3$ is formed upon addition of any of the above reagents.

Your work:
Your answer (tick one) Yes ☐ No ☐

Problem 2	Student code_______	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	27	0	5	25	0	4	8	1	9	79

9. Fill in the hereunder table by showing the trends of changes resulting from addition of each reagent to this particular water sample (use "+" if the factor increases, "–" if it decreases, and "0" if it does not change).

Table 5

Reagent	pH	FA	FD	FTDS	LI
$NaHCO_3$					
NaOH					
$NaHSO_4$					
$CaCl_2$					
Na_2H_2Y					
HCl					

Replacement or extra chemicals	Lab assistant signature	Penalty
________________________ ________________________ ________________________	________________________ ________________________ ________________________	___________ ___________ ___________

Problem 3	Name_______________ Student code_________	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	3	2	0	27.5	5	0	19.5	4	1	64

Problem 3. Determination of molecular mass by viscometry (15 points)

Viscosity coefficient is a measure of fluid resistance to flow. It can be determined by measuring the rate of liquid flow through a thin capillary. Polymer solution viscosity grows with increasing concentration. At constant concentration, stronger solvent-polymer interactions result in more expanded polymer coils, and therefore, in higher viscosity.

Provided the density of the diluted solution of a polymer is equal to that of the solvent, the reduced viscosity η_{red} of the polymer solution with concentration c (g/mL) is defined as follows:

$$\eta_{red} = \frac{t - t_0}{t_0 c} \quad [mL/g],$$

where t and t_0 are the flow times of the solution and pure solvent, respectively.

Reduced viscosity for dilute polymer solutions depends on concentration as follows:

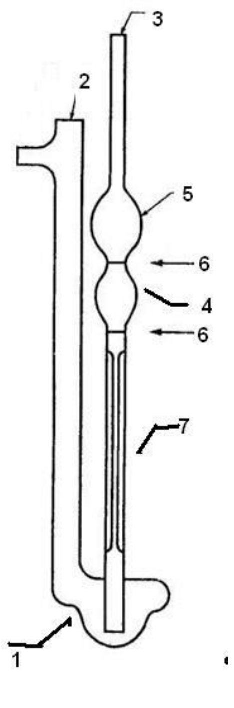
$$\eta_{red}(c) = [\eta] + kc,$$

with k, a parameter (mL^2/g^2) and $[\eta]$, intrinsic viscosity (mL/g). The intrinsic viscosity $[\eta]$ is determined by extrapolation of the reduced viscosity to zero polymer concentration. In general, the intrinsic viscosity is related to the molecular mass M of the polymer according to the Mark-Kuhn-Houwink equation:

$$[\eta] = KM^{\alpha},$$

where K and α are the constants for a particular solvent-polymer pair at a certain temperature. Thus, M can be derived from the Mark-Kuhn-Houwink equation using experimentally determined $[\eta]$ and reference data for K and α.

How to work with viscometer



1 – Collection vessel
2, 3 – Supplementary tubing
4 – Measurement vessel
5 – Collection vessel
6 – The match marks
7 - Capillary

Problem 3	Name_______________ Student code_________	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	3	2	0	27.5	5	0	19.5	4	1	64

a) Mount the viscometer so that its tubing (3) is vertical, and the collection vessel (1) stands on the lab stand basement. Adjust the fixing clamp as low as possible.
b) Put 10 mL of the liquid to be analyzed into the collection vessel (1) through the tubing (2) using a pipette.
c) Place the pipette filler or rubber bulb on top of the tubing (3) and suck the liquid into the measurement vessel (4) so that the liquid is drawn into the collection vessel (5). When sucking the liquid, avoid the air bubbles in the capillary (7) and the vessels (4, 5), as these can cause significant experimental errors. The liquid meniscus should be about 10 mm above the upper mark (6).
d) Zero the stopwatch, and remove the pipette filler or bulb out of the tube (3). The liquid starts flowing down to the collection vessel (1).
e) Measure the **flow time**: start the stopwatch when the liquid meniscus passes the upper match mark (6) and stop the stopwatch when the liquid meniscus passes the lower match mark (6).

ATTENTION: Handle the viscometer with great care!
There will be no replacement if you have broken your viscometer!
If you do break your viscometer tell the lab assistant. You may then attempt to do the experiment using the 25 mL pipette and beaker in place of viscometer.

Clean the viscometer three times with tap water and once with distilled water before you pass over to a new polymer sample. To do this, first wash it with tap water, and then rinse with distilled water. There is no need to wash it with the polymer solution, the error can occur but it is negligible.
You are NOT requested to fill in all table cells in the Answer Boxes. Perform as many measurements as you prefer for accurate averaging.

Procedure
You are provided with a set of aqueous solutions of polymers (0.01 g/mL, stock solutions). Three of P1-P4 are solutions of poly(vinyl alcohol), whereas the fourth one is that of a partially hydrolyzed poly(vinyl acetate) containing *ca*. 10% of non-hydrolyzed units. It is unknown which of the P1-P4 solutions is partially hydrolyzed poly(vinyl acetate). Molecular masses of the polymers P1-P4 are given in the Table.

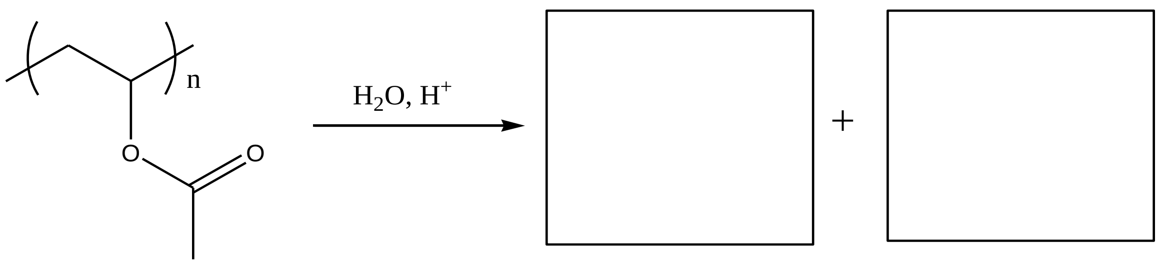
Approximate molecular mass	Sample code
26650	P2
50850	P1
65300	P4
91900	P3

Problem 3	Name_______________ Student code_________	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	3	2	0	27.5	5	0	19.5	4	1	64

Sample X is poly(vinyl alcohol) of an unknown molecular mass.
In this task you will have to identify which of P1-P4 is the solution of partially hydrolyzed poly(vinyl acetate) and determine the molecular mass of polymer X.

1. Write down the reaction scheme of poly(vinyl alcohol) preparation by hydrolysis of poly(vinyl acetate).

Reaction scheme:



2. Choose (tick appropriate box) which polymer shows the stronger interaction with water and compare the viscosities of aqueous solutions of fully and partially hydrolyzed poly(vinyl acetates). Assume that the concentration of the solutions and the molecular masses of the polymers are the same.

Poly(vinyl alcohol) ☐
Partially hydrolyzed poly(vinyl acetate) ☐
Compare the viscosities:

η poly(vinyl alcohol) ______ η partially hydrolyzed poly(vinyl acetate) (put either <. >, or ≈)

3. Measure the flow time of the pure solvent (distilled water). You are not requested to fill all the boxes below

Accepted value: ___________ s

4. Measure the flow times of the stock solutions of P1-P4, and that of X. Calculate the reduced viscosities. You are NOT requested to fill in all table cells in the Answer Boxes. Perform as many measurements as you prefer for accurate averaging.

Problem 3	Name_______________ Student code________	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	3	2	0	27.5	5	0	19.5	4	1	64

Sample→	**P2** (26650)	**P1** (50850)	**P4** (65300)	**P3** (91900)	**X**
Flow time, s					
Accepted flow time:	_____ s	_____ s	_____ s	_____ s	_____ s

Calculations:

Sample→	**P2** (26650)	**P1** (50850)	**P4** (65300)	**P3** (91900)	**X**
Reduced viscosity of the stock solutions, mL/g					

5. Encircle the solution out of P1-P2-P3-P4 which is the sample of partially hydrolyzed poly(vinyl acetate). **Hint:** Take into account the given molecular masses of the polymers P1-P4.

P1	P2	P3	P4

DO NOT USE THIS POLYMER IN THE NEXT PART OF THE EXPERIMENT.

6. To determine the parameters of the Mark-Kuhn-Houwink equation and calculate the unknown molecular mass of X choose and encircle two most appropriate solutions of poly(vinyl alcohol) with different molecular masses. Assume that the absolute error of intrinsic viscosity determination does not depend on the sample molecular mass.

P1	P2	P3	P4

7. Using appropriate measuring glassware to prepare the solutions, measure the flow time of a number of diluted solutions of three poly(vinyl alcohol) samples: that of unknown molecular mass (X), and the pair of poly(vinyl alcohols) chosen in i. 6, and calculate the corresponding reduced viscosities. When calculating the diluted solutions concentration, assume density of the polymer solutions is equal to that of water. Determine the intrinsic viscosities for each of the

Problem 3	Name______________ Student code________	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	3	2	0	27.5	5	0	19.5	4	1	64

examined samples. Submit the graph paper with your plots together with the booklet. **Note:** if you would like to plot the data referring to different samples on the same plot, make sure you use clearly distinguishable symbols for each dataset. You are NOT requested to fill in all table cells in the Answer Boxes.

Sample: ___

Concentration, g/mL:					
Stock solution, mL					
Water, mL					
Flow time, s:					
Accepted flow time, s					
Reduced viscosity, mL/g					
Intrinsic viscosity [η], mL/g					

Sample: ___

Concentration, g/mL:					
Stock solution, mL					
Water, mL					
Flow time, s:					
Accepted flow time, s					
Reduced viscosity, mL/g					
Intrinsic viscosity [η], mL/g					

Problem 3	Name_______________ Student code________	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	3	2	0	27.5	5	0	19.5	4	1	64

Sample: ___					
Concentration , g/mL:					
Stock solution, mL					
Water, mL					
Flow time, s:					
Accepted flow time, s					
Reduced viscosity, mL/g					
Intrinsic viscosity [η], mL/g					

Summary of experimental results (only fill in the measured values)

Sample→	P__	P__	X
Concentration (c), g/mL:	0.01	0.01	0.01
Reduced viscosity (η_{red}), mL/g			
c (1st dilution), g/mL:			
η_{red}, mL/g			
c (2nd dilution), g/mL:			
η_{red}, mL/g			
c (3rd dilution), g/mL:			
η_{red}, mL/g			
c (4th dilution), g/mL:			
η_{red}, mL/g			
c (5th dilution), g/mL:			
η_{red}, mL/g			

Problem 3	Name_______________ Student code_________	Quest.	1	2	3	4	5	6	7	8	9	Total
		Marks	3	2	0	27.5	5	0	19.5	4	1	64

8. Write down the form of equation you would use to determine K and α.

Derive the *K* and α values for the aqueous solution of poly(vinyl alcohol).

K = _________ mL/g	α = __________

9. By using the obtained *K* and α values, as well as the intrinsic viscosity of the X solution, calculate the molecular mass of the polymer X. If you have failed to determine *K* and α, use *K* = 0.1 mL/g and α = 0.5.

Your work. M (X) = ______________

Replacement or extra chemicals	Lab assistant signature	Penalty
Broken viscometer ______________________ ______________________	______________________ ______________________ ______________________	__0________ __________ __________

IUPAC Periodic Table of the Elements

Key:

atomic number
Symbol
name
standard atomic weight

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1 **H** hydrogen [1.007; 1.009]																	2 **He** helium 4.003
3 **Li** lithium [6.938; 6.997]	4 **Be** beryllium 9.012											5 **B** boron [10.80; 10.83]	6 **C** carbon [12.00; 12.02]	7 **N** nitrogen [14.00; 14.01]	8 **O** oxygen [15.99; 16.00]	9 **F** fluorine 19.00	10 **Ne** neon 20.18
11 **Na** sodium 22.99	12 **Mg** magnesium 24.31											13 **Al** aluminium 26.98	14 **Si** silicon [28.08; 28.09]	15 **P** phosphorus 30.97	16 **S** sulfur [32.05; 32.08]	17 **Cl** chlorine [35.44; 35.46]	18 **Ar** argon 39.95
19 **K** potassium 39.10	20 **Ca** calcium 40.08	21 **Sc** scandium 44.96	22 **Ti** titanium 47.87	23 **V** vanadium 50.94	24 **Cr** chromium 52.00	25 **Mn** manganese 54.94	26 **Fe** iron 55.85	27 **Co** cobalt 58.93	28 **Ni** nickel 58.69	29 **Cu** copper 63.55	30 **Zn** zinc 65.38(2)	31 **Ga** gallium 69.72	32 **Ge** germanium 72.63	33 **As** arsenic 74.92	34 **Se** selenium 78.96(3)	35 **Br** bromine 79.90	36 **Kr** krypton 83.80
37 **Rb** rubidium 85.47	38 **Sr** strontium 87.62	39 **Y** yttrium 88.91	40 **Zr** zirconium 91.22	41 **Nb** niobium 92.91	42 **Mo** molybdenum 95.96(2)	43 **Tc** technetium	44 **Ru** ruthenium 101.1	45 **Rh** rhodium 102.9	46 **Pd** palladium 106.4	47 **Ag** silver 107.9	48 **Cd** cadmium 112.4	49 **In** indium 114.8	50 **Sn** tin 118.7	51 **Sb** antimony 121.8	52 **Te** tellurium 127.6	53 **I** iodine 126.9	54 **Xe** xenon 131.3
55 **Cs** caesium 132.9	56 **Ba** barium 137.3	57-71 lanthanoids	72 **Hf** hafnium 178.5	73 **Ta** tantalum 180.9	74 **W** tungsten 183.8	75 **Re** rhenium 186.2	76 **Os** osmium 190.2	77 **Ir** iridium 192.2	78 **Pt** platinum 195.1	79 **Au** gold 197.0	80 **Hg** mercury 200.6	81 **Tl** thallium [204.3; 204.4]	82 **Pb** lead 207.2	83 **Bi** bismuth 209.0	84 **Po** polonium	85 **At** astatine	86 **Rn** radon
87 **Fr** francium	88 **Ra** radium	89-103 actinoids	104 **Rf** rutherfordium	105 **Db** dubnium	106 **Sg** seaborgium	107 **Bh** bohrium	108 **Hs** hassium	109 **Mt** meitnerium	110 **Ds** darmstadtium	111 **Rg** roentgenium	112 **Cn** copernicium		114 **Fl** flerovium		116 **Lv** livermorium		

57 **La** lanthanum 138.9	58 **Ce** cerium 140.1	59 **Pr** praseodymium 140.9	60 **Nd** neodymium 144.2	61 **Pm** promethium	62 **Sm** samarium 150.4	63 **Eu** europium 152.0	64 **Gd** gadolinium 157.3	65 **Tb** terbium 158.9	66 **Dy** dysprosium 162.5	67 **Ho** holmium 164.9	68 **Er** erbium 167.3	69 **Tm** thulium 168.9	70 **Yb** ytterbium 173.1	71 **Lu** lutetium 175.0
89 **Ac** actinium	90 **Th** thorium 232.0	91 **Pa** protactinium 231.0	92 **U** uranium 238.0	93 **Np** neptunium	94 **Pu** plutonium	95 **Am** americium	96 **Cm** curium	97 **Bk** berkelium	98 **Cf** californium	99 **Es** einsteinium	100 **Fm** fermium	101 **Md** mendelevium	102 **No** nobelium	103 **Lr** lawrencium

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Notes

- IUPAC 2009 Standard atomic weights abridged to four significant digits (Table 4 published in *Pure Appl. Chem.* 83, 359-396 (2011); doi:10.1351/PAC-REP-10-09-14). The uncertainty in the last digit of the standard atomic weight value is listed in parentheses following the value. In the absence of parentheses, the uncertainty is one in that last digit. An interval in square brackets provides the lower and upper bounds of the standard atomic weight for that element. No values are listed for elements which lack isotopes with a characteristic isotopic abundance in natural terrestrial samples. See PAC for more details.

- "Aluminum" and "cesium" are commonly used alternative spellings for "aluminium" and "caesium."

- Claims for the discovery of all the remaining elements in the last row of the Table, namely elements with atomic numbers 113, 115, 117 and 118, and for which no assignments have yet been made, are being considered by a IUPAC and IUPAP Joint Working Party.

For updates to this table, see iupac.org/reports/periodic_table/. This version is dated 1 June 2012.