Nimi: Kood:

Instructions (Task 1)

- This examination has 10 pages for practical Task 1 and answer sheets.
- You have 15 minutes to read this booklet before starting the experiments.
- You have **2 hours 15 minutes** to complete the practical **Task 1.**
- Begin only when the **START** command is given. You must stop your work immediately when the **STOP** command is announced. A delay in doing this by 5 minutes will lead to cancellation of your practical exam. After the **STOP command** has been given, **wait in your lab space**. A supervisor will check your lab space. The following items should be **left on your bench:**
- The problem / answer booklet (this booklet)
- You are expected to follow **safety rules** given in the IChO regulations. While you are in the laboratory, you must wear **safety glasses** or your own prescription safety glasses if they have been approved. You may use **gloves** when handling chemicals.
- You will receive only **ONE WARNING** from the laboratory supervisor if you break safety rules. On the second occasion you will be dismissed from the laboratory with a resultant zero score for the entire practical examination.
- Do not hesitate to ask your assistant if you have any questions concerning safety issues or if you need to leave the room.
- You are allowed to work only in the space allocated for you.
- Use only the pen provided, not a pencil, for writing the answers.
- Use the calculator provided.
- All results must be written in the appropriate areas on the answer sheets. Anything written elsewhere will not be graded. Use the backside of the sheets if you need scratch paper.
- Use the container labeled as "**Used Vials**" to dispose sealed vials with reaction solutions.
- Use the container labeled as "**Liquid Waste"** to dispose the waste solutions.
- Use the container labeled as "**Broken Glass Disposal"** to dispose the ampule fragments.
- Chemicals and lab ware will be **refilled or replaced** without penalty only for the first incident. Each further incident will result in the **loss of 1 point** from your 40 practical exam points.
- The official English version of this examination is available on request only for clarification.

Nimi: Kood:

Chemicals and Equipment (Task 1)

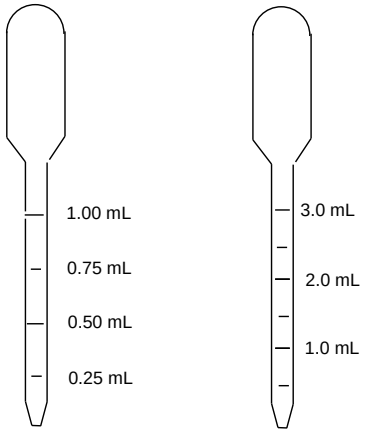
Chemicals (the actual labeling for each package is given in bold font)

	Risk Phrase[+]	Safety Phrase[+]
~2 M HCl,[*] solution in water, 50 mL in a bottle	R34, R37	S26, S45
~0.01 M KI_3,[*] solution in water, 10 mL in a bottle, labeled "**I_2**".		
Acetone, **$(CH_3)_2CO$**, M = 58.08 g mol^{-1}, density = 0.791 g mL^{-1}, 10.0 mL in a vial	R11, R36, R66, R67	S9, S16, S26
Acetone-d_6, $(CD_3)_2CO$, M = 64.12 g mol^{-1}, density = 0.872 g mL^{-1}, 3.0 mL in a pre-scored ampule	R11, R36, R66, R67	S9, S16, S26

[+] See page 3 for definition of Risk and Safety Phrases.
[*] The exact molarity is indicated on the label, with the concentration given before the name of the substance.

Equipment - Kit #1

- One glass bottle filled with distilled water
- Fifteen 20-mL screw-cap glass vials with Teflon-lined screw-caps
- Ten 1-mL polyethylene transfer pipettes graduated in 0.25 mL increments (see drawing in the right).
- Ten 3-mL polyethylene transfer pipettes graduated in 0.50 mL increments (see drawing in the right).
- One digital timer (stopwatch)



Risk and Safety Phrases (Task 1)

R11 Highly flammable

R34 Causes burns

R36 Irritating to eyes

R37 Irritating to respiratory system

R66 Repeated exposure may cause skin dryness or cracking

R67 Vapors may cause drowsiness and dizziness

S9 Keep container in a well-ventilated place

S16 Keep away from sources of ignition

S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice

S45 In case of accident or if you feel unwell, seek medical advice immediately

Nimi: Kood:

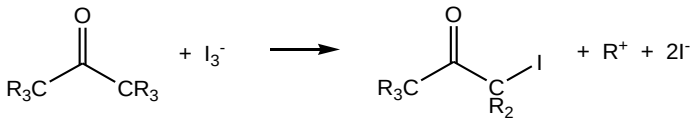
Task 1 18% of the total

a	b	c	d	e	f	g	Task 1	18%
10	2	10	12	16	12	8	70	

Kinetics, Isotope Effect, and Mechanism of Iodination of Acetone

Discoveries about the mechanisms of chemical reactions underlie advances in catalysis and synthesis. One of the most powerful tools for probing reaction mechanisms is the study of kinetics because the way in which reaction rates vary with reaction conditions follow directly from the mechanism of reaction. A second powerful tool is the study of isotopically substituted molecules. While isotopes impart similar reactivity, there are slight differences in reaction rates as a function of nuclear mass.

In this task you will use both kinetics and isotope effects to provide information about the iodination of acetone in acidic aqueous solution:



R = H or D

The reaction takes place with a rate law

$$\text{Rate} = k[\text{acetone}]^m[I_3^-]^n[H^+]^p$$

where the rate constant k and the integer reaction orders m, n, and p are for you to determine. You will also compare the reactivity of acetone with that of acetone-d_6, where the six atoms of protium (^{1}H) have been replaced by deuterium (^{2}H, D), to determine the isotope effect (k_H/k_D) of the reaction. From these data you will make inferences about the mechanism of this reaction.

Please read the whole description of this task and plan your work before you begin.

Procedure

Reaction rates are dependent on temperature. Record the temperature in the room you are working in (ask the room assistant):

	°C

Instructions for using the digital timer (stopwatch)

(1) Press the **[MODE]** button until the **COUNT UP** icon is displayed.

(2) To begin timing, press the **[START/STOP]** button.

(3) To stop timing, press the **[START/STOP]** button again.

(4) To clear the display, press the **[CLEAR]** button.

General Procedure

Measure the volumes of hydrochloric acid, distilled water, and potassium triiodide solution (labeled as "I_2") that you choose into the reaction vessel. The initial concentrations of the reagents in the reaction mixtures should be in the ranges given below (you need not explore the full ranges given, but your values should not be significantly outside these ranges):

$[H^+]$: Between 0.2 and 1.0 M

$[I_3^-]$: Between 0.0005 and 0.002 M

[acetone]: Between 0.5 and 1.5 M

To initiate the reaction, add the chosen volume of acetone to the solution containing the other reagents, quickly cap the reaction vessel, start the timer, shake the vial vigorously one time, then put it aside on a white background. Report the volumes of reagents that you use in the table provided in (a). When setting up and running a reaction do not hold or touch the vial below the level of liquid in it. The progress of the reaction can be monitored visually by watching the disappearance of the yellow-brown color of the triiodide ion. Record the time required for the color to disappear. When the reaction is complete, set aside the vessel, and leave it sealed so that you do not expose yourself to iodoacetone vapors.

Repeat as often as desired with different concentrations of reagents. Report the concentrations of the reagents that you use in the tables in (c) below. *Hint: change one concentration at a time.*

Once you have studied the rate of reaction of acetone, you should examine the rate of reaction of acetone-d_6. Note that while you have an ample supply of acetone, you will be given only 3.0 mL of acetone-d_6 because of the greater expense of the isotopically labeled material. Therefore, any refilling of acetone-d_6 will be accompanied by a one point penalty. **When you need to use this reagent, raise your hand and the lab supervisor will open the sealed ampule for you.** The reactions of deuterium-substituted compounds are generally slower than those of protium-substituted compounds. You would thus be well advised to use reaction conditions that promote faster reactions when working with $(CD_3)_2CO$.

When you are finished working:

a) empty the water bottle and place it along with any unused equipment back to the box labeled "Kit #1";
b) place used pipettes and sealed used vials in designated containers under the hoods;
c) Use a container labeled **Broken Glass Disposal** to dispose of all parts of the empty ampule.

You may clean up your area after the STOP command has been given.

Nimi: Kood:

a. Record your results for acetone, $(CH_3)_2CO$, in the table below. *You need not fill the entire table.*

Run #	Volume HCl solution, mL	Volume H_2O, mL	Volume I_3^- solution, mL	Volume $(CH_3)_2CO$, mL	Time to disappearance of I_3^-, s
1					
2					
3					
4					
5					
6					
7					
8					

b. Record your results for acetone-d_6, $(CD_3)_2CO$, in the table below. *You need not fill the entire table.*

Run #	Volume HCl solution, mL	Volume H_2O, mL	Volume I_3^- solution, mL	Volume $(CD_3)_2CO$, mL	Time to disappearance of I_3^-, s
1d					
2d					
3d					
4d					

c. Use the following tables to calculate concentrations and average rates for the reactions you studied. Assume that the volume of each reaction mixture is equal to the sum of volumes of its constituent solutions. **You need not use all of your runs in your calculation of *k* (parts e and f), but you must indicate which run or runs you used in your calculation by checking the appropriate box in the right-hand column.**

$(CH_3)_2CO$:

Run #	Initial $[H^+]$, M	Initial $[I_3^-]$, M	Initial $[(CH_3)_2CO]$, M	Average rate of disappearance of I_3^-, M s^{-1}	Run used in calculating k_H? **Yes**	**No**
1					☐	☐
2					☐	☐
3					☐	☐
4					☐	☐
5					☐	☐
6					☐	☐
7					☐	☐
8					☐	☐

$(CD_3)_2CO$:

Run #	Initial $[H^+]$, M	Initial $[I_3^-]$, M	Initial $[(CD_3)_2CO]$, M	Average rate of disappearance of I_3^-, M s^{-1}	Run used in calculating k_D? **Yes**	**No**
1d					☐	☐
2d					☐	☐
3d					☐	☐
4d					☐	☐

d. Give the integer reaction order in acetone, triiodide, and hydrogen ion.

$$\text{rate} = -\frac{d[I_3^-]}{dt} = k[(CH_3)_2CO]^m[I_3^-]^n[H^+]^p$$

m =	n =	p =

e. Calculate the rate constant k_H for the reaction of acetone, $(CH_3)_2CO$, and indicate the units.

k_H =

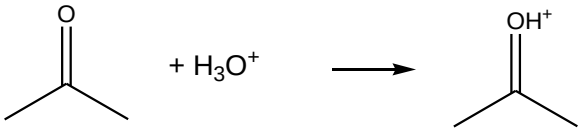
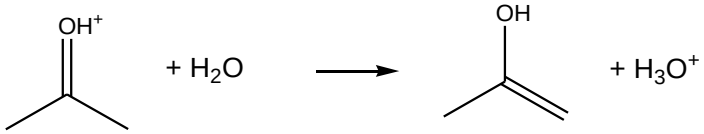
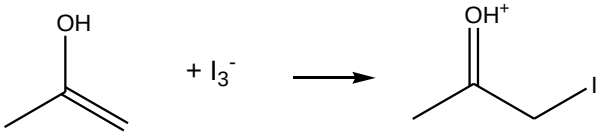
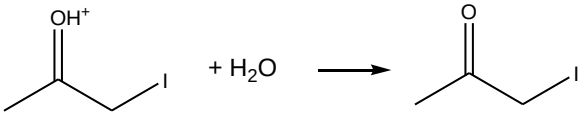
f. Calculate the rate constant k_D for the reaction of acetone-d_6, $(CD_3)_2CO$, and calculate the value of k_H/k_D (the isotope effect of the reaction).

k_D =

k_H/k_D =

g. From the kinetic and isotope effect data you may draw certain conclusions about the reaction mechanism. Shown below is a reasonable mechanism for the iodination of acetone. One reaction is the rate-determining step (R.D.S.), with all previous steps rapidly achieving an equilibrium that strongly favors the reactants.

In the box in the first column on the right next to each step, place a check mark (✔) if your *experimentally measured rate law* (part **d**) is **consistent** with that step being rate-determining and an **X** if your measured rate law is **inconsistent** with that step being rate-determining. In the box in the second column on the right next to each step, place a check mark (✔) if your *experimentally measured isotope effect* (part **f**) is **consistent** with that step being rate-determining and an **X** if your measured isotope effect is **inconsistent** with that step being rate-determining.

	R.D.S. consistent with rate law?	R.D.S. consistent with isotope effect?
		
		
		
		

Nimi: Kood:

Juhised (Eksperiment 2)

- Eksperiment 2 ja selle vastustelehed on esitatud **13** leheküljel**.**
- Teil on enne eksperimendi algust ülsande teksti lugemiseks aega 15 minutit.
- Teil on **Eksperimendi 2** tegemiseks aega **2 tundi 45 minutit.** Arvestage oma töö planeerimisel, et üks operatsioon nõuab 30 minutit aega.
- Alustage alles siis kui on antud käskuls **START**. Tea peate oma töö katkestama koheselt kui on antud käsklus **STOPP**. 5 minutilise viivituse puhul töö katkestamisel praktika töö tühistatakse. Peale **STOP käskluse andmist oodake oma töökohal**. Juhendaja kontrollib teie töökoha üle. **Töölauale tuleb jätta** järgmised asjad:

  Ülesannete/vastustelehtede tekst ()

  Üks TLC plaat suletud plastikkotis varustatud teie võistlejakoodiga

  Viaal sildiga “Product”

- Te peate järgima **ohtusreegleid**, mis on toodud IChO reeglistikus. Olees laboris peate te kandma **kaitseprille** või teie omi kaitseprille, kui need on juhendaja poolt lubatud. Kautage ainult teile antud **pipetitäitjat**. Te võite kasutada kemikaalidega töötamisel **kindaid.**
- Ohtustehnika reeglite rikkumise korral antakse teile juhendaja poolt ainult **ÜKS HOIATUS.** Teise rikkumise korral eemaldatakse teid laborist j ate saate kogu praktilise vooru eest null punkti.
- Pööeduge julgesti juhendaja poole kui teil on probleeme ohutusega või te soovite ruumist lahkuda.
- Teil on lubatud töötada ainult teile eraldatud tööpinnal.
- Kasutage vastuste kirjutamiseks ainult pastakat, mitte pliiatsit.
- Kasutage teile antud kalkulaatorit.
- Kõik tulemused tuleb kirjutada vastustelehtede vastavasse kohta. Mujale kirjutatud tulemusi ei hinnata. Kasutage teile antud lehtede tagakülgi mustandipaberina.
- Kasutage kasutatud viaalide jaoks sildiga **“Broken Glass Disposal**” varustatud konteinerit.
- Kasutage lahuste jääkide jaoks sildiga “**Liquid Waste” varustatud** konteinerit.
- Kemikaale ja laborivahendeid **antakse ilma** karistuspunktideta juurde **ainult üks kord.** Iga järgmise juhtumi korral võetakse teie 40 praktilise töö eest saadavast punktisummast 1 punkt maha.
- Te võite küsida ametlikku inglisekeelset versiooni ainult mõnede asjaolude selgitamiseks.

Nimi: Kood:

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	**1** 1.00794 **H** 0.28																	**2** 4.00260 **He** 1.40
2	**3** 6.941 **Li**	**4** 9.01218 **Be**											**5** 10.811 **B** 0.89	**6** 12.011 **C** 0.77	**7** 14.0067 **N** 0.70	**8** 15.9994 **O** 0.66	**9** 18.9984 **F** 0.64	**10** 20.1797 **Ne** 1.50
3	**11** 22.9898 **Na**	**12** 24.3050 **Mg**											**13** 26.9815 **Al**	**14** 28.0855 **Si** 1.17	**15** 30.9738 **P** 1.10	**16** 32.066 **S** 1.04	**17** 35.4527 **Cl** 0.99	**18** 39.948 **Ar** 1.80
4	**19** 39.0983 **K**	**20** 40.078 **Ca**	**21** 44.9559 **Sc**	**22** 47.867 **Ti** 1.46	**23** 50.9415 **V** 1.33	**24** 51.9961 **Cr** 1.25	**25** 54.9381 **Mn** 1.37	**26** 55.845 **Fe** 1.24	**27** 58.9332 **Co** 1.25	**28** 58.6934 **Ni** 1.24	**29** 63.546 **Cu** 1.28	**30** 65.39 **Zn** 1.33	**31** 69.723 **Ga** 1.35	**32** 72.61 **Ge** 1.22	**33** 74.9216 **As** 1.20	**34** 78.96 **Se** 1.18	**35** 79.904 **Br** 1.14	**36** 83.80 **Kr** 1.90
5	**37** 85.4678 **Rb**	**38** 87.62 **Sr**	**39** 88.9059 **Y**	**40** 91.224 **Zr** 1.60	**41** 92.9064 **Nb** 1.43	**42** 95.94 **Mo** 1.37	**43** (97.905) **Tc** 1.36	**44** 101.07 **Ru** 1.34	**45** 102.906 **Rh** 1.34	**46** 106.42 **Pd** 1.37	**47** 107.868 **Ag** 1.44	**48** 112.41 **Cd** 1.49	**49** 114.818 **In** 1.67	**50** 118.710 **Sn** 1.40	**51** 121.760 **Sb** 1.45	**52** 127.60 **Te** 1.37	**53** 126.904 **I** 1.33	**54** 131.29 **Xe** 2.10
6	**55** 132.905 **Cs**	**56** 137.327 **Ba**	**57-71** **La-Lu**	**72** 178.49 **Hf** 1.59	**73** 180.948 **Ta** 1.43	**74** 183.84 **W** 1.37	**75** 186.207 **Re** 1.37	**76** 190.23 **Os** 1.35	**77** 192.217 **Ir** 1.36	**78** 195.08 **Pt** 1.38	**79** 196.967 **Au** 1.44	**80** 200.59 **Hg** 1.50	**81** 204.383 **Tl** 1.70	**82** 207.2 **Pb** 1.76	**83** 208.980 **Bi** 1.55	**84** (208.98) **Po** 1.67	**85** (209.99) **At**	**86** (222.02) **Rn** 2.20
7	**87** (223.02) **Fr**	**88** (226.03) **Ra** 2.25	**89-103** **Ac-Lr**	**104** (261.11) **Rf**	**105** (262.11) **Db**	**106** (263.12) **Sg**	**107** (262.12) **Bh**	**108** (265) **Hs**	**109** (266) **Mt**	**110** (271) **Ds**	**111** (272) **Rg**	**112** (285) **Cn**	**113** (284) **Uut**	**114** (289) **Fl**	**115** (288) **Uup**	**116** (292) **Lv**	**117** (294) **Uus**	**118** (294) **UUo**

Atomic number → **1** 1.00794 **H** 0.28 ← Atomic weight; **Atomic symbol**; Covalent radius, Å

57 138.906 **La** 1.87	**58** 140.115 **Ce** 1.83	**59** 140.908 **Pr** 1.82	**60** 144.24 **Nd** 1.81	**61** (144.91) **Pm** 1.83	**62** 150.36 **Sm** 1.80	**63** 151.965 **Eu** 2.04	**64** 157.25 **Gd** 1.79	**65** 158.925 **Tb** 1.76	**66** 162.50 **Dy** 1.75	**67** 164.930 **Ho** 1.74	**68** 167.26 **Er** 1.73	**69** 168.934 **Tm** 1.72	**70** 173.04 **Yb** 1.94	**71** 174.04 **Lu** 1.72
89 (227.03) **Ac** 1.88	**90** 232.038 **Th** 1.80	**91** 231.036 **Pa** 1.56	**92** 238.029 **U** 1.38	**93** (237.05) **Np** 1.55	**94** (244.06) **Pu** 1.59	**95** (243.06) **Am** 1.73	**96** (247.07) **Cm** 1.74	**97** (247.07) **Bk** 1.72	**98** (251.08) **Cf** 1.99	**99** (252.08) **Es** 2.03	**100** (257.10) **Fm**	**101** (258.10) **Md**	**102** (259.1) **No**	**103** (260.1) **Lr**

Nimi: Kood:

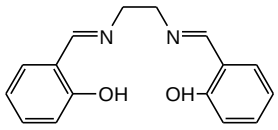
Kemikaalid ja seadmed (Eksperiment 2)

Kemikaalid ja materjalid (iga komplekti tähistus on antud rasvase kirjaga)

	Risk Phrase[+]	Safety Phrase[+]
(saleen)H_2,[a] ~1.0 g[b] viaalis	R36/37/38	S26 S28A S37 S37/39 S45
$Mn(OOCCH_3)_2$ $4H_2O$, ~1.9 g[b] viaalis	R36/37/38 R62 R63	S26 S37/39
Liitiumkloriidi lahus, LiCl, 1M lahus etanoolis, 12 ml pudelis	R11 R36/38	S9 S16 S26
Etanool, 70 ml pudelis	R11	S7 S16
Atsetoon, **$(CH_3)_2CO$**, 100 ml pudelis	R11 R36 R66 R67	S9 S16 S26
(saleen*)$MnCl_x$,[c] ~32 ml ~3.5 mg/ml[b] lahust pudelis		
KI_3, ~0.010 M vesilahus,[b] 50 ml pudelis, etiketiga "**I_2**".		
Askorbiinhape, ~0.030 M vesilahus,[b] 20 ml pudelis		
1% tärklis, vesilahus, 2 ml pudelis		
TLC plaat – üks 5 cm × 10 cm silikageeli plaat suletavas plastikkotis		

[+] Vaata leheküljel 14 toodud riski- ja ohutuslauseid.

[a] (saleen)H_2:



[b] Täpne väärtus on toodud etiketil.

[c] (saleen*)$MnCl_x$ (mõlemad R rühmad on igas ühendis samad ja võivad olla kas H, COOH või SO_3H):

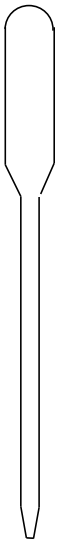
Seadmed

Üldiseks kasutamiseks: kaalud

- Kaks **statiivi** koos **käppadega** asuvad tõmbe all ja on tähistatuid teie koodiga
- Üks **kuumutatava plaadiga magnetsegaja**
- Üks **300 mm joonlaud**
- Üks **pliiats**

Kit #2:

- Kaks **250 ml Erlenmeyeri kolbi** (üks sünteesiks, teine kristallimiseks)
- Üks **mõõtsilinder**, 50 ml
- Üks **20 mm pikkusega munakujuline magnetsegaja pulk**
- Üks **Hirsch'i lehter**
- **Filterpaberi** kettad Hirsch'i lehtri jaoks ja TLC voolutusnõu jaoks
- Üks **125 ml imipudel vaakumfiltreerimiseks**
- **Kummiadapter** imipudeli jaoks
- Üks 0.5 l **plastikust jäävann**
- Üks **klaaspulk**
- Kaks 1 m plastikpipetti (vt paremal toodud joonist)
- Üks plastispaatel
- Üks tühi **4 ml snap-korgiga viaal** etiketiga "Product" reaktsiooniprodukti
- jaoks



- **Kit #3:**
- Kolm tühja **väikest keeratava korgiga viaali** (TLC lahuste jaoks)
- Kümme **lühikest kapillaari (100 mm)** TLC provide pealekandmiseks
- Üks **uuriklaas** (TLC voolutusnõu jaoks)
- Üks **250 ml keeduklaas,** TLC voolutusnõu

Kit #4:

- Üks komplekteeritud ja kasutamiseks valmis **25 ml bürett**
- Üks väike **plasticlehter**
- Neli **125 ml Erlenmeyeri kolbi**
- Üks **kummiballoon pipettide jaoks**

- Üks **10 ml mahtpipett**
- Üks **5 ml mahtpipett**

Nimi: Kood:

Riski- ja ohutuslaused (Eksperiment 2)

R11 Highly flammable

R36/37/38 Irritating to eyes, respiratory system and skin

R62 Possible risk of impaired fertility

R63 Possible risk of harm to the unborn child

R66 Repeated exposure may cause skin dryness or cracking

R67 Vapors may cause drowsiness and dizziness

S7 Keep container tightly closed

S9 Keep container in a well-ventilated place

S16 Keep away from sources of ignition

S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

S28A After contact with skin, wash immediately with plenty of water.

S37 Wear suitable gloves.

S37/39 Wear suitable gloves and eye/face protection.

S45 In case of accident or if you feel unwell, seek medical advice immediately

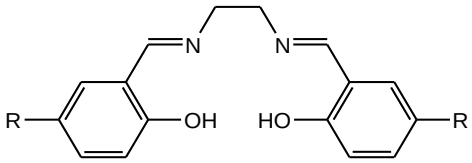
Nimi: Kood:

Eksperiment 2 22% summaarsest

Saleen-mangaankompleksi süntees ning x väärtuse ja produkti valemi määramine

A	B-i	B-ii	C-i	C-ii	Eksperiment 2	22%
10	15	4	4	2	35	

Bis(salitsülideen)etüleendiamiin (saleen) ligandidest saadud 3*d*-rühma elementide üleminekumetallide kompleksid on näidanud end efektiivsete redoksreaktsioonide katalüsaatoritena orgaanilises sünteesis.



(saleen)H_2, R = H

(saleen*)H_2, R = H, COOH, või SO_3H

Saleen-ligandide võime stabiliseerida kõrgemas oksüdatsiooniastmes olevaid of 3*d*-rühma elemente on keemias väga tähtis. +2 kuni +5 oksüdatsiooniastmetes olevaid mangaaniühendeid võib valmistada mangaan-saleenkompleksidena sõltuvalt reaktsioonitingimustest. Käesolevas eksperimendis tuleb teil valmistada mangaan-saleenkompleks (saleen)H_2 reaktsioonil Mn(II) atsetaadiga etanoolis õhu käes liithiukloriidi juuresolekul. Nendes tingimustes võite te saada kompleksi valemiga (saleen)$MnCl_x$, kus x = 0, 1, 2, või 3.

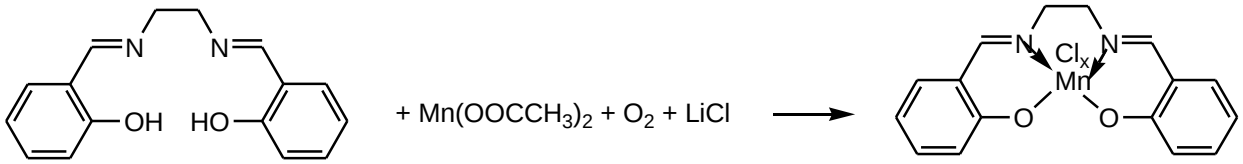
Teil tuleb: i) määrata produkti mass, ii) iseloomustada saadud produkti puhtust, kasutades plannarkromatograafiat (TLC), ja iii) määrata metalli oksüdatsiooniaste kompleksis, kasutades jodomeetrilist redokstiitrimist. Redokstiitrimiseks antakse teile eelnevalt valmistatud teie ühendiga analoogse ühendi, (saleen*)$MnCl_x$ lahus, kus mangaan on samas oksüdatsiooniastmes kui produktiski ja benseenitsükli R-asendaja on kas H, COOH, või SO_3H.

Palun lugege kogu selle eksperimendi kirjeldus läbi ja planeerige oma tööd enne alustamist. Mõningaid operatsioone tuleb teha paraleelselt, et jõuda tööga etteantud ajaks valmis.

Nimi: Kood:

Eeskiri:

A. (Saleen)$MnCl_x$ süntees



(salen)$MnCl_x$

1) Pange 2-3 (saleen)H_2 kristalli TLC analüüsiks väiksesse viaali.
2) Pange teile antud eelnevalt kaalutud ~1.0 g (saleen)H_2 ja magnetsegajapulk 250 ml Erlenmeyeri kolbi. Lisage 35 ml absoluutset etanooli.
3) Asetage kolb magnetsegajale. Kuumutage kolvi sisu segamisel kuni sademe lahustumiseni (tavaliselt on lahustumine lõppenud kui etanool on keema minemas). Reguleerige seejärel temperatuur selliselt, et see on keemistemperatuurist veidi madalamal. Ärge keetke segu, kolvi kael peab olema jahe. Kui kolb on käega hoidmiseks liiga kuum, siis kasutage hoidmiseks kokkuvolditud käterätipaberit.
4) Eemaldage kolb kuumutusplaadilt ja lisage selle sisule eelnevalt kaalutud ~1.9 g $Mn(OAc)_2 \cdot 4H_2O$. Segu värvub tumepruuniks. Asetage kolb kiiresti kuumutusplaadile; jätkake kuumutamist ja segamist 15 min. Ärge keetke segu, kolvi kael peab olema jahe.
5) Eemaldage kolb kuumutusplaadilt ja lisage selle sisule teile antud 1M LiCl lahus etanoolis (12 ml, liias). Pange kolb kuumutusplaadile tagasi; jätkake kuumutamist ja segamist 10 min. Ärge keetke segu, kolvi kael peab olema jahe.
6) Eemaldage seejärel kolb kuumutusplaadilt ja pange see produkti kristallumiseks 30 minutiks jäävanni. Kraapige kolvi siseseina iga 5 minuti järel klaaspulgaga (salen)$MnCl_x$ kristallumise kiirendamiseks. Esimesed kristallid võivad ilmuda kohe peale jahutamist või 10-15 minuti pärast.
7) Kasutage vaakumliini mis asub tõmbe all (vastav kraan on tähistatud "Vacuum") ja filtrige moodustunud kristallid Hirsch lehtri ja imipudeli abil vaakumis. Kasutage pipetti sademe pesemiseks mõne tilga atsetooniga ilma imipudelit vaakumliini küljest lahti ühendamata ja jätke sade filtrile (vaakumliinist lahtiühendamata) 10-15 min kuivama.
8) Viige tahke produkt eelnevalt kaalutud ja etiketiga "Product" varustatud viaali , määrake produkti mass m_p ja kirjutage alltoodud kasti. Kirjutage samuti üles järgnevate sünteesis kasutatud reagentide massid: (saleen)H_2, m_S, ja $Mn(OOCCH_3)_2 \cdot 4H_2O$, m_{Mn}.
9) Pange etiketiga viaal koos produktiga suletavasse plastikotti.

Nimi: Kood:

Tühja produktiviaali mass: _____ g

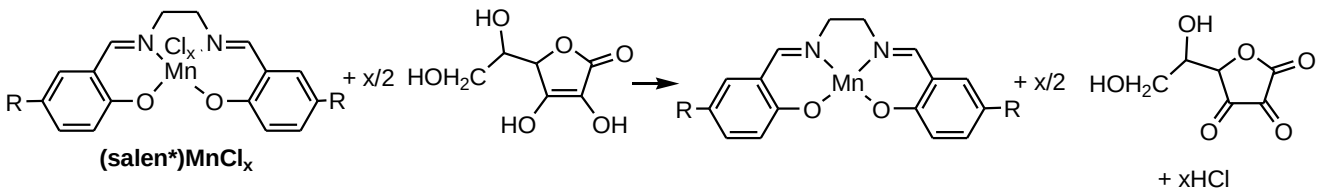
Produktiviaali mass koos kuiva produktiga: _____ g

Produkti mass, m_p: _____ g

(Saleen)H_2 mass viaali etiketilt (kirjuta etiketilt), m_S:
_____ g

$Mn(OOCCH_3)_2 \cdot 4H_2O$ mass viaali etiketilt (kirjuta etiketilt), m_{Mn}:
_____ g

B. Volumetric analysis of a sample of (salen*)$MnCl_x$ provided



R = H, COOH, or SO_3H

Using squeeze bulb

1) Attach the bulb to a pipette
2) Squeeze firmly the rubber bulb
3) Squeeze the up arrow button to suck in some solution into pipette attached
4) Squeeze the down arrow button to release some solution from the pipette into a target flask

Note: The pipettes and burette are ready to use and need not be conditioned.

1) Dispense 10.00 mL of the provided (salen*)$MnCl_x$ solution into a 125 mL Erlenmeyer flask using the volumetric pipette.
2) Add 5.00 mL of the ascorbic acid solution to this solution and mix well. Allow the solution to stand for 3-4 minutes.
3) To avoid oxidation of ascorbic acid with O_2 do not delay and titrate the solution immediately with the KI_3 solution using 5 drops of a 1% starch solution as indicator. The blue or blue-green endpoint should persist for at least 30 sec.
4) If time permits, perform 1-2 replicate titrations to improve the accuracy of your determination.

   Place results of your titration experiment(s) in the table below:

#	Initial volume reading in burette of KI_3 solution, mL	Final volume reading in burette of KI_3 solution, mL	Volume of KI_3 solution consumed, mL
1			
2			
3			

i. Indicate the volume (selected or averaged) of KI_3 solution consumed in mL that you will use for calculations of molar mass of (salen*)$MnCl_x$:

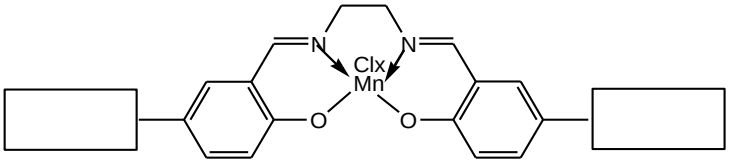
Volume of KI_3 solution used in calculations: _____ mL

Concentration of (salen*)$MnCl_x$ (from label on the bottle): _____ mg/mL

Concentration of ascorbic acid (from label on the bottle): _____ M

Nimi: Kood:

ii. From your titration data and referring to the table below deduce the value of *x*, oxidation state of manganese and the identity of the substituent on the salen ligand (R = H, COOH, SO_3H). Show them in the template below:



x = _____

Manganese oxidation state: _____

R	x	(Theoretical molar mass)/x, g/mol
H	1	357
H	2	196
H	3	143
COOH	1	445
COOH	2	240
COOH	3	172
SO_3H	1	517
SO_3H	2	276
SO_3H	3	196

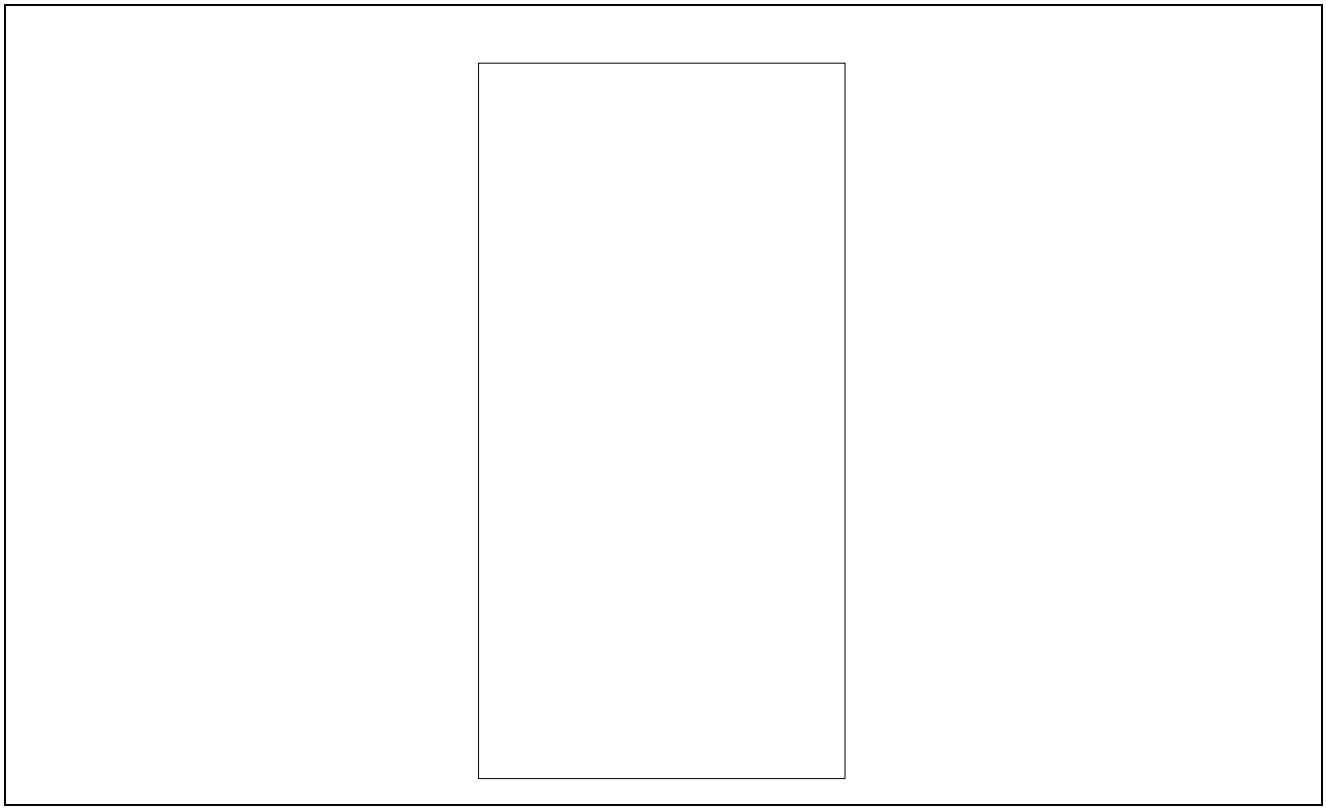
Nimi: Kood:

C. (Saleen)$MnCl_x$ TLC analüüs

1) Lahustage mõned kristallid teie sünteesitud (saleen)$MnCl_x$ mõnes tilgas absoluutses etanoolis, kasutades väikest viaali ja etanooli plastikpipetti.
2) Lahustage mõned kristallid (saleen)H_2 mõnes tilgas absoluutses etanoolis, kasutades teist väikest viaali.
3) Kui TLC plaati on tarvis voolutuskambrisse mahutamiseks parajaks lõigata, siis kasutage selleks kääre (saate juhendajalt kui küsite).
4) Voltige või lõigake suur filterpaberiketas sobivalt ja pange selliselt voolutusnõusse, et see ulatub peaaegu kogu voolutusnõu kõrguseni. See on vajalik voolutusnõu kiiremaks küllastamiseks etanooli aurudega. Valage etanooli voolutusnõusse selliselt, et filterpaber saaks märjaks ja voolutusnõu põhi oleks kaetud 3-4 mm paksuse etanooli kihiga. Sulgege voolutusnõu uuriklaasiga.
5) Tähistage ära stardijoon.
6) Kandke mõlema proovi lahused kapillaaride abil TLC plaadile.
7) Voolutage TLC plaati uuriklaasiga kaetud voolutusnõus 10-15 min.
8) Tähistage pliiatsi abil TLC plaadil solvendifront ja samuti värvilised laigud.
9) Kuivatage TLC plaat õhu käes ja pange tagasi suletavasse plastikkotti.
10) Arvutage (saleen)H_2 ja (salen)$MnCl_x$ laikude R_f väärtused.

Nimi: Kood:

i. Joonistage TLC plaat oma vastustelehele



ii. Arvutage (saleen)H_2 ja (saleen)$MnCl_x$ R_f väärtused ja kirjutage alltoodud kasti

R_f, (saleen)H_2: _____

R_f, (saleen)$MnCl_x$: _____

Kui olete töö lõpetanud:

a) Pange vedelad jäägid etiketiga **Liquid Waste** konteinerisse.

b) Pange kasutatud viaalid etiketiga **Broken Glass Disposal** konteinerisse.

c) Pange kasutatud klaasnõud tagasi etikettidega “Kit #2”, “Kit #3” ja “Kit #4” kastidesse.