CHEMISTRY.
OLYMPIAD.CH
CHEMIE-OLYMPIADE
OLYMPIADES DE CHIMIE
OLIMPIADI DELLA CHIMICA

SwissChO 2023 - Practical Final Exam

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G0-1
English (Official)

General Information

Instructions

- You have 2:30 h to solve this practical exam. Before that, you have 10 min to look through the exam. Wait for the **START** signal before you begin with any laboratory work.
- Finish your work immediately, once the **STOP** signal has been given.
- Write your name on each working page and number these.
- Use a new page for each problem. Clearly indicate what problem you are working on.
- Only **answers written on answer sheets** can be considered. Only **submitted materials** can be considered.
- Write all necessary calculations, observations and data points legibly.
- Do not communicate with other participants during the exam. You may quietly talk with the laboratory assistants.
- Stay at your designated desk. Only leave the working area if you have been given permission to do so.
- You may ask the laboratory assistants for more deionised water or gloves. Requesting any more materials will lead to deduction of points.
- Hazardous or reckless behaviour may lead to expulsion from this exam.
- This exam has **3** problems.

Viel Erfolg!

Bonne chance !

Buona fortuna!

Good luck!

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Periodic Table of the Elements

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1 H 1.008																	2 He 4.003
3 Li 6.94	4 Be 9.01											5 B 10.81	6 C 12.01	7 N 14.01	8 O 16.00	9 F 19.00	10 Ne 20.18
11 Na 22.99	12 Mg 24.31											13 Al 26.98	14 Si 28.09	15 P 30.97	16 S 32.06	17 Cl 35.45	18 Ar 39.95
19 K 39.10	20 Ca 40.08	21 Sc 44.96	22 Ti 47.87	23 V 50.94	24 Cr 52.00	25 Mn 54.94	26 Fe 55.85	27 Co 58.93	28 Ni 58.69	29 Cu 63.55	30 Zn 65.38	31 Ga 69.72	32 Ge 72.63	33 As 74.92	34 Se 78.97	35 Br 79.90	36 Kr 83.80
37 Rb 85.47	38 Sr 87.62	39 Y 88.91	40 Zr 91.22	41 Nb 92.91	42 Mo 95.95	43 Tc [98]	44 Ru 101.07	45 Rh 102.91	46 Pd 106.42	47 Ag 107.87	48 Cd 112.41	49 In 114.82	50 Sn 118.71	51 Sb 121.76	52 Te 127.60	53 I 126.90	54 Xe 131.29
55 Cs 132.91	56 Ba 137.33	57–71	72 Hf 178.49	73 Ta 180.95	74 W 183.84	75 Re 186.21	76 Os 190.23	77 Ir 192.22	78 Pt 195.08	79 Au 196.97	80 Hg 200.59	81 Tl 204.38	82 Pb 207.2	83 Bi 208.98	84 Po [209]	85 At [210]	86 Rn [212]
87 Fr [223]	88 Ra [226]	89–103	104 Rf [267]	105 Db [268]	106 Sg [269]	107 Bh [270]	108 Hs [270]	109 Mt [278]	110 Ds [281]	111 Rg [282]	112 Cn [285]	113 Nh [286]	114 Fl [289]	115 Mc [290]	116 Lv [293]	117 Ts [294]	118 Og [294]

57 La 138.91	58 Ce 140.12	59 Pr 140.91	60 Nd 140.24	61 Pm [145]	62 Sm 150.36	63 Eu 151.96	64 Gd 157.25	65 Tb 158.93	66 Dy 162.50	67 Ho 164.93	68 Er 167.26	69 Tm 168.93	70 Yb 173.05	71 Lu 174.97
89 Ac [227]	90 Th 232.04	91 Pa 231.04	92 U 238.03	93 Np [237]	94 Pu [244]	95 Am [243]	96 Cm [247]	97 Bk [247]	98 Cf [251]	99 Es [252]	100 Fm [257]	101 Md [258]	102 No [259]	103 Lr [266]

Ion Sheet

An ion sheet has been attached at the end of the exam.

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Score Sheet

NOT TO BE FILLED IN BY PARTICIPANT

Name of participant:

Problem	Title	Maximum points	Achieved points	Pages
P1	Synthesis of (*E*,*E*)-Dibenzylideneacetone	20.0		3
P2	Love or Hate: Oxalate	18.5		2
P3	Qualitative Inorganic Analysis	18.0		2

Synthesis of (*E*,*E*)-Dibenzylideneacetone (20.0 Points)

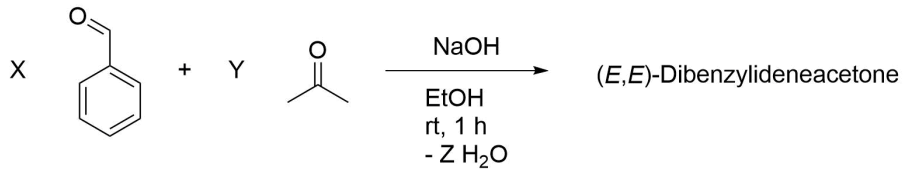

Figure 1: Reaction scheme for task 1.

Materials

- 250 mL two-necked round-bottom flask
- 250 mL one-necked round-bottom flask
- Magnetic stir bar
- Reflux condenser (already installed)
- Ice bath
- Addition funnel
- Water bath
- Hot plate
- Filter paper

Chemicals

- Ethanol
- Deionised water
- Sodium hydroxide
- Acetone, $\rho_{\text{Acetone}} = 0.784\,\text{g cm}^{-3}$
- Benzaldehyde, $\rho_{\text{PhCHO}} = 1.046\,\text{g cm}^{-3}$
- Hexane
- Ethyl acetate

Procedure

1. To a 250 mL two-necked round-bottom flask, add 50 mL of a 2.3 mol L^{-1} aqueous NaOH solution.
2. Add 33 mL of ethanol while stirring.
3. Cool the mixture to 0 °C using an ice bath.
4. Mix 4.6 mL of benzaldehyde and 1.7 mL of acetone in an addition funnel. Slowly add the mixture to the ethanol solution over 15 min while stirring.
5. Once the addition is complete, let the reaction stir at room temperature for 1 h.
6. Filter the mixture over vacuum and wash the solid three times with 50 mL deionised water.
7. Perform a TLC with the obtained solid.
8. Transfer the solid to a 250 mL one-necked round-bottom flask.
9. Add a little bit of ethanol and stir the solution.
10. Connect the flask to a reflux condenser and heat until boiling (approximately 78 °C) in a hot water bath. Add ethanol little by little until the solid is completely dissolved.
11. Once completely dissolved, stop heating the solution and remove the flask from the hot water bath.
12. Cool the flask to room temperature and then to 0 °C using an ice bath.
13. When all solid has crystallised, filter it over vacuum and wash three times with a small amount of ice cold ethanol.
14. Dry the product over vacuum for $2-3\,\mathrm{min}$. Disconnect from vacuum (remember to turn off the vacuum!) and let the product dry in air for at least $15\,\mathrm{min}$.
15. Perform another TLC with the dry product.
16. Transfer the product into your designated vial and hand it off to the lab assistant.

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P1-3

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Theoretical questions

1.1	**Determine** X, Y, and Z in the reaction scheme shown in **Fig. 1**.	1.5pt
1.2	**Draw** the structure of the expected product.	2.0pt
1.3	**Name** the type of reaction you performed in this task.	1.0pt
1.4	**Name** the potential driving forces for the elimination of water during the reaction.	2.0pt
1.5	What other methods could you use to favour the elimination of water in this synthesis? **Name** at least 2.	2.0pt
1.6	**State** the reason why the reaction (almost) exclusively forms the *trans* product.	1.0pt
1.7	What is the limiting reagent in the synthesis? **Explain** your answer.	1.5pt
1.8	According to the TLC you obtained immediately after the reaction, **state** whether your reaction has run to completion or not.	2.0pt
1.9	According to the TLC you obtained after purification, **make** an assertion about the purity of the product.	2.0pt
1.10	**Hand in** your product in the labelled vial with your TLCs for analysis.	5.0pt

Love or Hate: Oxalate (18.5 Points)

Materials

- pH paper
- 50 mL Burette
- Glass pipettes
- 3×200 mL Erlenmeyer flasks
- 1×250 mL volumetric flask

Chemicals

- Potassium permanganate solution ($KMnO_4$, 0.01 mol L^{-1}), strongly acidified
- Oxalic acid (ethanedioic acid) sample
- Sodium hydroxide solution (0.025 mol L^{-1})
- Deionised water
- Phenolphthalein solution

Procedure

Acid-base titration

1. You have received a 250 mL volumetric flask with an unknown amount of oxalic acid. Fill the volumetric flask up to the mark with deionised water.
2. Take 25 mL of the solution and transfer it into a 200 mL Erlenmeyer flask. Fill the flask up to approximately 100 mL with deionised water.
3. Add two drops of the provided phenolphtalein solution to the Erlenmeyer.
4. Titrate the solution with 0.025 mol L^{-1} NaOH solution until the indicator changes to pink.
5. Repeat steps 2-4 at least **two** more times.

Permanganometric titration

1. Transfer 25 mL of the oxalic acid solution from the volumetric flask to a 200 mL Erlenmeyer flask.
2. Add deionised water until a volume of approximately 100 mL is reached.
3. Heat the solution to approximately 50 °C.
4. Take the solution off the hot plate and add around 3 mL of the permanganate solution from the burette and wait until the colour has disappeared.
5. Titrate the solution with the remaining potassium permanganate solution until the pink colour does not disappear anymore.
6. Repeat steps 1-5 at least **two** more times.

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Theoretical questions

2.1	**State**, which deprotonation is responsible for the colour change in the indicator.	1.0pt
2.2	Observe, that the discolouration of the added permanganate is very slow at first but the reaction rate increases, the more permanganate has already reacted. **State**, which process and species is responsible for this phenomenon.	1.5pt
2.3	**Write** the balanced reaction equations for the half-cell reactions and the full redox reaction involving the permanganate ions.	3.0pt
2.4	A gas is created when the permanganate solution is added. **Name** the gas and **give** its molecular formula.	1.0pt
2.5	From the acid-base titrations, **calculate** the unknown mass of oxalic acid you were given. **Show** your working and obtained values.	6.0pt
2.6	From the permanganometric titrations, **calculate** the unknown mass of oxalic acid you were given. **Show** your working and obtained values.	6.0pt

Qualitative Inorganic Analysis (18.0 Points)

Materials

- Plastic pipettes
- pH paper
- Mortar and pestle
- Test tubes

Chemicals

- Three unknown salts
- Possible cations: Cu^{2+}, Mn^{2+}, Mg^{2+}, Ba^{2+}, Fe^{3+}, NH_4^{+}
- Possible anions: CO_3^{2-}, Cl^{-}, SO_4^{2-}, NO_3^{-}, CH_3COO^{-}, $HCOO^{-}$, OH^{-}
- Deionised water
- Ammonia
- Potassium hexacyanoferrate
- Sulfuric acid
- Potassium hydroxide
- Ammonium chloride
- Sodium hydrogen phosphate
- Hydrochloric acid
- Sodium sulfate
- Potassium thiocyanate
- Silver nitrate
- Barium chloride
- Isopropanol
- Ammonium acetate
- Iron(II) sulfate
- Potassium hydrogen sulfate

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Procedure

1. You have received three different salts in a vial.
2. Analyse the colour of your salt mixture and write down your findings.
3. Test the salts according to their solubility in deionised water and note your findings.

   Very carefully, you may also add little amounts of dilute acid or base.
4. You are also permitted to use the following reagents and/or tools to characterise your salt mixture:

   a) pH paper
   b) Ammonia
   c) Potassium hexacyanoferrate solution
   d) Concentrated sulfuric acid
   e) Potassium hydroxide
   f) Ammonium chloride
   g) Sodium hydrogen phosphate solution
   h) HCl
   i) Sodium sulfate solution
   j) Isopropanol
   k) Potassium thiocyanate solution
   l) Silver nitrate solution
   m) Barium chloride solution
   n) Iron(II) sulfate solution
   o) Potassium hydrogen sulfate

Theoretical questions

3.1	**State** your observations on the colours inside your salt mixture. **Elaborate** on which of your salts has which colour.	4.5pt
3.2	**Give** your observations from the solubility tests you performed. **State**, which of your salts are soluble in water or dilute acid/base and which are insoluble.	1.5pt
3.3	**Determine** the composition of your salt mixture. **Write** down how you were able to identify/verify their presence and **write** down the balanced reaction equations for their detection.	12.0pt

Ion alone	OH^-	NH_3	CO_3^{2-}	additional info
Ag^+	↓ brown	↓ brown, xs. sol.	↓ cream	+ Cl^- ↓ in NH_3 sol. / + S^{2-} ↓
Ba^{2+}	↓ clouding	-	↓	+ SO_4^{2-} ↓ + $C_2O_4^{2-}$ ↓ + CrO_4^{2-} ↓ + $Cr_2O_7^{2-}$ ↓ ↓ insol. in HOAc
Cu^{2+}	↓ blue	↓ turquoise xs.:°deep blue	↓ turquoise	+ $K_4[Fe(CN)_6]$ ↓ + S^{2-} ↓
Cu^+	↓ dark red			+ SCN^- ↓
Fe^{2+}	↓ olive green turns brown	↓ green-brown turns brown	↓ green	$[Fe(CN)_6^{3-}]$:↓ dark blue ox.: brown + S^{2-} ↓
Fe^{3+}	↓ orange-brown	↓ red-brown	↓ brown	$[Fe(CN)_6^{4-}]$:↓ dark blue SCN^-: blood red I^-: yellowish brown +S^{2-} ↓
H_3O^+			↑ CO_2	pH paper: acidic
NH_4^+	T↑: smell pH paper	-	-	
Ni^{2+}	↓green	↓green xs.: °blue	↓green	+ S^{2-} ↓
Al^{3+}	↓, xs. sol.	↓	↓	
Bi^{3+} acidic	↓, (T↑: yellow)	↓, (T↑: yellow)	↓, (T↑: yellow)	I^- :↓black xs.: °orange +S^{2-} ↓orange
Ca^{2+}	↓	-	↓	+ $C_2O_4^{2-}$ ↓ + SO_4^{2-}maybe clouding
Co^{2+}	↓ blue	↓ blue	↓purple	+ S^{2-} ↓
Cr^{3+}	↓ grey-green xs.: °green	↓ grey-green	↓blue-grey	+S^{2-} ↓ blue-grey
Mg^{2+}	↓	↓	↓	
Mn^{2+}	↓ turns black		↓ light pink	+ S^{2-} ↓ pink
Pb^{2+}	↓, xs. sol.	↓	↓	+ I^- ↓ yellow + CrO_4^{2-} ↓ yellow sol. in HOAc + Cl^- ↓ +SO_4^{2-} ↓ + S^{2-} ↓
Sr^{2+}	↓	-	↓	+ SO_4^{2-} ↓ +CrO_4^{2-} ↓yellow
Zn^{2+}	↓, xs. sol.	↓, xs. sol.	↓	+ S^{2-} ↓

Legend:

↓	white precipitate	↓ colour	coloured precipitate
°colour	solution coloured	-	no rxn
xs.	in excess	sol.	soluble
T	apply heat	↑	gas evolution

Ion alone	H^+ (H_2SO_4) maybe T	Ag^+	Ba^{2+}	other rxns
F^-	DON'T DO THIS	-	↓ ?	If you add acid to this, write your testament.
Cl^-	-	↓ UV: turns dark insol. in HNO_3 sol. in dil. NH_3	-	
Br^-	-	↓ light yellow insol. in HNO_3 sol. in conc. NH_3	-	
I^-	-	↓ yellow insol. in HNO_3 insol. in NH_3	-	+ Fe^{3+}: brown (I_2) + Cu^{2+}: brown + ↓ white
HCO_3^-	↑			T: ↑, gas into $Ca(OH)_2$ solution: clouding
CO_3^{2-}	CO_2 ↑	↓ white, turns yellow sol. in HNO_3	↓ powdery	+ Ca^{2+}: clouding
CH_3COO^-	vinegar smell	↓, dissolves in dil. HNO_3	TOXIC	
S^{2-}	stinks like hell	↓ blackish-grey		+ $Pb(OAc)_2$: ↓ black
SO_4^{2-}	-	-	↓ fine	
OH^-	-	↓ brown	maybe clouding	pH paper basic
NO_3^-	-	-	-	ring test
CrO_4^{2-}	°orange	↓ brown-red	↓ yellow	
$Cr_2O_7^{2-}$	-	↓ brown-red	↓ yellow-orange	
MnO_4^-	-	-	-	oxidises Fe^{2+}, H_2O_2, $C_2O_4^{2-}$
PO_4^{3-}	-	↓ yellow sol. in HNO_3	↓ fine flaky	
SCN^-	-	↓ fine UV: turns purple insol. in HNO_3 sol. in dil. NH_3	-	+ Fe^{3+} blood-red
$C_2O_4^{2-}$	-	↓, sol. in acid	↓, sol. in acid	Ca^{2+}: ↓, sol. in acid +MnO_4^- + H^+ + heat: discolours

Legend:

↓	white precipitate	↓ colour	coloured precipitate
°colour	solution coloured	-	no rxn
xs.	in excess	sol.	soluble
T	apply heat	↑	gas evolution

Special detection reactions

Formate ($HCOO^-$)

Prepare a reagent solution consisisting of 0.5 g citric acid monohydrate + 10.0 g acetamide in 100 mL ^{i}PrOH. Prepare a sodium acetate solution by dissolving 30.0 gNaOAc in 100 mLH_2O. Mix 0.5 mL sample with 1.0 mL reagent solution, 1 drop NaOAc solution, and 3.5 mL acetic anhydride. If a raspberry-red colour develops, the test is positive for formate.

Nitrite ($NO_2{}^-$)

Acidify solution with conc. HOAc. Add 2-3 drops sulfanilic acid + 2-3 drops 1-naphthylamine. If the solution turns a deep red colour, the test is positive for nitrite. Attention: test reaction is very sensitive to other ions such as Br^-, I^-, $ClO_3{}^-$, $IO_3{}^-$, S^{2-}, $SO_3{}^{2-}$, $S_2O_3{}^{2-}$, SCN^-, $CrO_4{}^{2-}$, $[Fe(CN)_6]^{4-}$, $[Fe(CN)_6]^{3-}$.

Sulfite ($SO_3{}^{2-}$)

Mix 10.0 mL of a solution of $KMnO_4$ in HOAc and 10 drops dil. $BaCl_2$ solution. Quickly add the sample. If a white precipitate of $BaSO_4$ forms, the test is positive for sulfite. Careful: once the testing solution of permanganate and barium chloride is prepared, it must be immediately used, since it degrades very quickly. When in doubt, repeat experiment.

Ring test (for $NO_3{}^-$)

Transfer your sample to a test tube. Add a few drops $FeSO_4$ solution and a few drops dil. H_2SO_4 to your sample solution. Then tilt your test tube about 45° and slowly add 2-3 drops conc. H_2SO_4 along the wall of the tube. This will allow the denser concentrated acid to slip underneath the solution. If a brownish/purple ring forms at the interface, the test is positive for nitrate.

Special detection reaction for Mg^{2+}

Acidify your test solution with hydrochloric acid. Add a solution of Na_2HPO_4. If crystals form upon addition of ammonia, the test is positive for magnesium. Calcium ions interfere negatively with this reaction.