Practical Final Exam 2025

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

G0-1

English (Official)

General Instructions

Instructions

- You have 3:00 h to solve this practical exam. Before starting, you will have 15 min to look through the exam without starting to work.
- Wait for the **START** signal before you begin with the laboratory work.
- Finish you work immediately once the **STOP** signal has been given.
- Write your name on every page. If there is insufficient space to solve an exercise on the front page, use the back page and indicate that you did so.
- Only legible answers clearly assignable to problems can be considered. Only materials properly submitted can be considered.
- Write all necessary equations, calculations, observations and data points legibly.
- Do not communicate with other participants during the exam. You may quietly talk with the laboratory assistants after raising your hand.
- 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, crushed ice, gloves and emptying of your waste beakers. Other than that, you have one free request to replace a piece of glassware or a chemical. Any further replacements will lead to a deduction of points.
- Hazardous or reckless behaviour may lead to expulsion from this exam.
- This exam has **2** problems.

Good luck!

Viel Erfolg!

Bonne chance!

Buona fortuna!

Periodic Table of Elements

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]

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Score Sheet

Problem	Title	Max. Points
P1	Basic Bleach Breaks Bonds	25.0 pt
P2	Advanced Amino Acid Assessment	25.0 pt
Total		**50.0 pt**

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Basic Bleach Breaks Bonds (25.0 Points)

In this problem, you will be investigating the reaction of *p*-methoxyacetophenone with bleach (NaOCl) under basic conditions. In order to check your reaction progress and to obtain insights on the product, you will be performing TLC analysis.

PREPARATION:

- Hand out
  - pH paper
  - Ruler
  - Ziplock bag, with the student's name
  - Aluminium foil
  - Filter paper
  - 2 stir bars (straight + olive)
  - Tweezers
  - Spatula
  - 15 plastic Pasteur pipettes
  - Pencil
  - Felt-tip marker
  - Rubber gasket
  - Keck clips (2)
- Set up the hotplate with water bath, including a straight stir bar.
- Give each student three small clamps and clamp holders.
- Using one of the clamps, attach the bubbler.
  - Fill one side of the bubbler with NOT QUITE half trap solution.
  - Connect the other side with some tubing, using glycerin to ensure a good seal.
  - Connect the other side of tubing to the 90° adapter, also using glycerin for a good seal.
- For TLC
  - Distribute 2 TLC plates
  - Distribute 4 micro capillaries
  - Distribute 2 small HPCL vials for the solutions (labelled)
- Distribute a Vigreux column
- Distribute a small (5 mL) vial labelled "product" and with the student's name.

- Trap solution (10 mL each, 9 parts 1 M NaOH, 1 part EtOH) -> bubbler
- Get a lot of ice.
- Weight out 500 mg of the *p*-methoxyacetophenone for each student and place it in a vial with an appropriate label. Note down the exact mass.
- Distribute 10 mL of a 1 mol L^{-1} NaOH solution in the smallest possible container.
- The morning before the exam, take out the bleach from the freezer/fridge and into vials, distribute 7.5 mL to each student. Place these back into the freezer and hand them out minutes before the exam.
- Hand out 7 mL of the 40% $NaHSO_3$ solution.
- Hand out 5 mL of the 2 mol L^{-1} HCl.
- Distribute 20 mL of the eluent, in a vial labelled "Eluent".

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P1-2

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Materials

- $1\times$ Hot plate
- $1\times$ Thermometer
- $4\times$ Capillary for TLC
- $1\times$ 250 mL beaker (TLC chamber)
- $1\times$ Magnetic stir bar
- $10\times$ Plastic pipette
- $3\times$ Clamp and clamp holder
- $1\times$ Pencil
- $1\times$ Ziplock bag
- Aluminium foil
- pH paper
- $1\times$ Vacuum flask
- $1\times$ Circular filter paper
- $1\times$ Laboratory stand
- $1\times$ Water bath with a stir bar
- $2\times$ Vial for TLC
- $2\times$ TLC plate
- $1\times$ 50 mL Round bottom flask
- $1\times$ Vigreux column
- $2\times$ Joint clip
- $1\times$ Vial for product
- $1\times$ Ruler
- $1\times$ Felt-tip marker
- Tweezers
- $1\times$ Vacuum tubing
- $1\times$ Büchner funnel
- $1\times$ Rubber gasket
- $1\times$ Bubbler, connected to a 90° adapter via tubing, attached via clamp

Chemicals

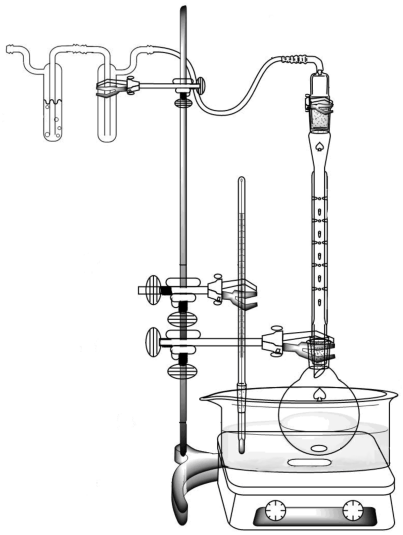
- *p*-Methoxyacetophenone
- Bleach (NaOCl solution, 14 w%)
- Sodium hydrogensulfite ($NaHSO_3$, 40 w%)
- Deionised water
- Eluent (Cyclohexane:EtOAc, 80 v%:20 v%)
- Sodium hydroxide solution (NaOH, 1 mol L^{-1})
- NaOH in EtOH + H_2O (trap solution in bubbler)
- Hydrochloric acid (HCl, 2 mol L^{-1})
- Crushed ice (provided upon request)

Safety

- Sodium hydroxide (NaOH) is caustic, causing chemical burns and blindness. **Make sure** that you are not wearing contact lenses and that your eyes and skin are properly shielded.
- Sodium hypochlorite/bleach (NaOCl) is a strong oxidising agent, capable of causing chemical burns and blindness. **Never acidify** solutions containing NaOCl and **never heat** solutions containing NaOCl without a laboratory assistant having approved of your setup. **Make sure** to protect your eyes and skin from NaOCl.
- Sodium hydrogensulfite ($NaHSO_3$) can cause irritation and will release sulfur dioxide (SO_2) upon strong acidification. Only gently **acidify** solutions containing $NaHSO_3$ according to the instructions given, and **inform** your laboratory assistants if you **detect** the smell of SO_2 (similar to a fresh-struck match; sulfury) coming from your mixture.
- The eluent containing ethyl acetate (EtOAc) and cyclohexane (C_6H_{12}) is volatile and very flammable. **Keep** it away from electrical equipment and sources of heat and sparks. The mixture can be irritating to skin and eyes and may cause drowsiness upon inhalation. Furthermore, cyclohexane can be acutely toxic in large doses. **Keep** exposure time to a minium and always **seal** containers containing the eluent.
- Hydrochloric acid (HCl) is a strong acid and irritant to skin and eyes. Exposure of HCl to your eyes can cause permanent damage and blindness. **Make sure** you are suitably protected with lab coats, closed shoes, gloves and goggles and that you are not wearing contact lenses. Addition of HCl to NaOCl will produce highly toxic chlorine gas. **Make absolutely sure** that you have first quenched all NaOCl with $NaHSO_3$, before adding any HCl.

Procedure

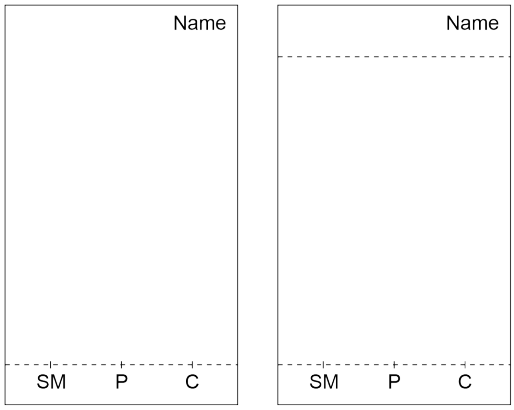
Synthesis



1. **Transfer** a tiny amount of *p*-methoxyacetophenone to a TLC vial and **set** it **aside**.
2. To a 50 mL round-bottom flask, **add** the remaining *p*-methoxyacetophenone (500 mg).
3. **Start** stirring slowly.
4. **Add** 7 mL of the 1.0 mol L^{-1} sodium hydroxide solution.
5. While stirring vigorously, slowly **add** the entire contents of the vial containing bleach (7.5 mL).
6. **Lower** the reaction vessel into the water bath.
7. To the reaction flask, **attach** a Vigreux column. **Secure** the Vigreux column with a clamp. To the Vigreux column, **attach** the 90° adapter leading to the bubbler. **Secure** all joints with a clip.
8. **Attach** a thermometer as shown in the figure above. **Make sure** the stir bar will not damage the thermometer.
9. **Check** that your setup looks identical to the one presented above. Once verified, **raise** your hand. A laboratory assistant will check your setup before you continue.
10. **Start** heating. Throughout the entire synthesis, **monitor** the temperature of the water bath. It should neither drop below 70 °C nor exceed 80 °C.
11. **Let** the reaction run for 70 min.
12. Once 70 min have elapsed, **remove** the reaction flask from the water bath. **Do not remove** the Vigreux column. The solution should still be mixing.
13. **Stop** the heating.
14. **Raise** your hand. A laboratory assistant will exchange your water bath for an ice bath.
15. **Cool** the reaction flask in the ice bath while stirring for ca. 7 min.

16. **Remove** the Vigreux column.
17. With the reaction flask in the ice bath, slowly **add** 5 mL of the $NaHSO_3$ solution. A white precipitate should form.
18. With the 2.0 mol L^{-1} HCl, dropwise **adjust** the pH of the solution, until it reaches pH $\approx$ 1-2. Continuously **check** the pH with pH paper.
19. **Filter** your product with vacuum filtration. **Do not dry** your product on the filter just yet.
20. **Wash** the product thoroughly twice with water (2×10 mL).
21. **Dry** the product quickly by pulling a vacuum, for a maximum of 3 min.
22. **Set aside** a small amount of your product in a different TLC vial.
23. **Transfer** the remaining product into the designated product vial, and **place** it onto the shelf. The vial will be picked up by a laboratory assistant at the end of the exam.

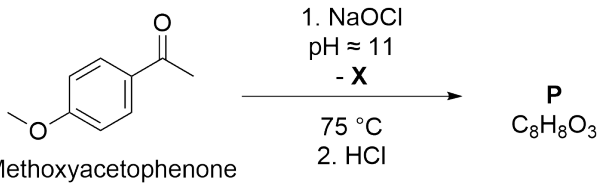
TLC Analysis



1. **Prepare** your TLC chamber by **adding** a bit of eluent into your 250 mL beaker. **Cover** the beaker with aluminium foil and **wait** 3 min.
2. **Perpare** your samples by **dissolving** the solids in around 0.5 mL eluent in their respective TLC vials.
3. On your TLC plate, **draw** a horizontal line around 1 cm above the bottom of the plate.
4. **Label** and **mark** three spots on your baseline.
   (a) *SM* (for your starting material)
   (b) *P* (for your product)
   (c) *C* (for your cospot for both of the above)
5. **Using** a capillary, **spot** the required TLC solutions for each spot.
6. At the very top of your plate, **write down** your name. Your TLC plate should now look like the first TLC plate pictured above.
7. **Using** tweezers, gently **lower** the TLC plate into your TLC chamber and **cover** with the aluminium foil.
8. **Wait** until the eluent front has reached around 2 cm below the top of the plate.

9. **Using** tweezers, gently **remove** the plate and immediately **mark** the eluent front on the plate. Your TLC plate should now look like the second TLC plate above.
10. **Raise** your hand. A laboratory assistant will escort you to the UV lamp. There, gently **circle** all spots.
11. **Place** your TLC plate into the provided ziplock bag and **place** said bag next to your product vial on the shelf of your laboratory workspace.

Theoretical Questions



p-Methoxyacetophenone

1.1 **Calculate** the R_f value of the starting material (**SM**) and your product (**P**). 2.0 pt

SOLUTION:

$R_{f,\mathbf{SM}}$ = 0.1 - 0.2

$R_{f,\mathbf{P}}$ = 0.4 - 0.5

1.0 pts for each R_f within accepted bounds. 2.0 pts total.

1.2 Based on your observations, **state** whether product **P** is more or less polar than the starting material. 1.0 pt

SOLUTION:

☒ More polar ☐ Less polar

1.0 pts for the correct box ticked. 0 pts for multiple ticks or none. 1.0 pts total.

1.3 Based on your TLC, **assert** whether product **P** is pure or not. **Justify** your choice. 2.0 pt

SOLUTION:

Individual answer.

Justification:

Individual answer. If only one spot (distinct from the starting material) is visible in the product lane, it is pure. If however this is not the case, it is not pure.

1.0 pts for correct tick box, which matches the TLC (check under UV again!), regardless of purity. 1.0 pts for correct justification, matching the TLC. 2.0 pts total.

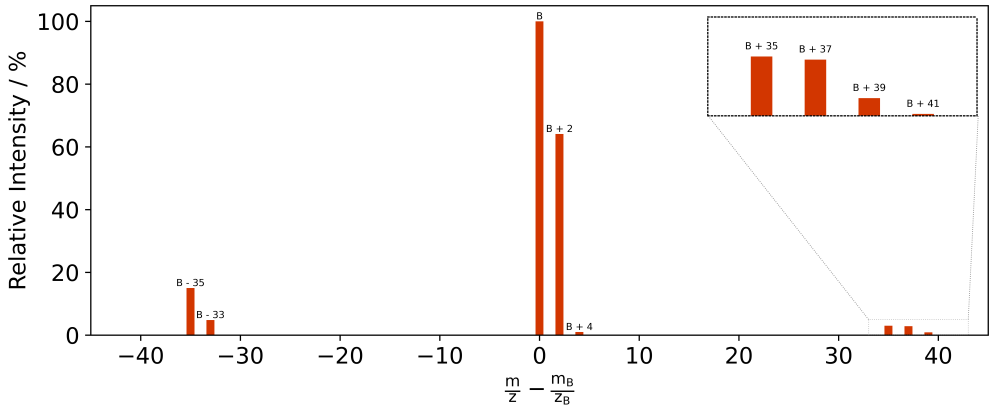
A by-product **X** is formed during the reaction. Let us investigate this molecule more closely.

1.4 **Determine** the number of carbon atoms n_C contained in the by-product **X** cleaved off in the reaction. 0.5 pt

SOLUTION:
Seeing that the sum formula of the starting material is $C_9H_{10}O_2$ and that of the product (given) is $C_8H_8O_3$, it is evident that one carbon went missing.
$n_C = 1$

0.5 pts for the correct value. 0.5 pts total.

The following is a section of a simplified mass spectrum of **X**. The base peak is labelled "B", and all other peaks are given relative to the base peak. The peaks corresponding to the highest $\frac{m}{z}$ values are on the right, however there are more peaks corresponding to lower $\frac{m}{z}$ values not shown. A table of the abundances of the relevant isotopes of all possible elements is given at the end of this exercise.



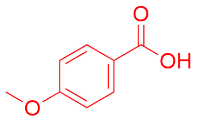
1.5 **Draw** the structure of the by-product **X**. 2.0 pt

SOLUTION:
The by-product **X** is chloroform, $CHCl_3$ This can either be seen by simply knowing the haloform reaction or via the mass spectrum. Looking at the isotopic distribution of Cl, the peaks line up perfectly with all possible fragments.
Any correctly drawn structure counts.

2.0 pts for a correct structure of chloroform. 1.0 pts for dichloromethane and carbon tetrachloride (off by 1 chlorine). 2.0 pts total

1.6 **Draw** the structure of the product **P**. 3.0 pt

SOLUTION:



3.0 pts for the correct structure. 3.0 pts total.

1.7 **Calculate** the maximum theoretical yield of **P** and **X** in mg, if *p*-methoxyacetophenone is the limiting reagent. 2.0 pt

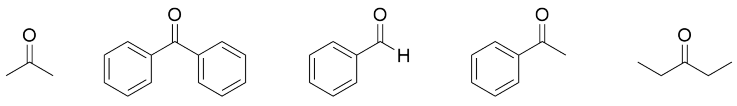
Note: *if you could not determine **X**, **assume*** $M_{\mathbf{X}}' = 100\,\mathrm{g\,mol^{-1}}$.

SOLUTION:

The amount of starting material they were given is 500 mg. This corresponds to 3.33 mmol.
Per equivalent of the starting material, one equivalent of **P** and **X** are produced.
Therefore, the maximum yields are
P: 507 mg
X: 398 mg (333 mg for $M_{\mathbf{X}}'$, 280 mg for CH_2Cl_2 and 513 mg for CCl_4)

1.0 pts for each correct numerical value. 2.0 pts total.

1.8 The studied reaction can only occur if the formation of **X** is possible. From the list below, **select** for which substrates the reaction would proceed or not. 2.5 pt



1 2 3 4 5

SOLUTION:

Substrate	1	2	3	4	5
Reaction proceeds	☒	☐	☐	☒	☐
Reaction doesn't proceed	☐	☒	☒	☐	☒

For the reaction to proceed, the substrate must be a methyl ketone. Anything else will not be able to generate the trihalomethyl group.

0.5 pts for each correctly assigned substrate. 0.0 pts for no ticks and two ticks. 2.5 pts total.

1.9 A laboratory assistant will pick up your TLC and product vial at the end of the exam. The yield and purity will be graded. 10.0 pt

SOLUTION:

qNMR analysis with subsequent yield/purity analysis is performed in DMSO-d6, with mesitylene as an internal standard.

7.0 pt awarded for yield of the actual product (as determined by NMR). Anything above 40% will give 7.0 pt, decreasing linearly to 0.0 pt at 0%.

3.0 pt awarded for purity of product in the submitted vial (as determined by NMR). Anything above 75% gives 3.0 pt, decreasing linearly to 0.0 pt at 0%.

Table of Natural Abundances of Relevant Isotopes

Element	Isotopes		
H	^{1}H 99.986%	^{2}H 0.014%	^{3}H *trace*
C	^{12}C 98.840%	^{13}C 1.160%	^{14}C *trace*
O	^{16}O 99.776%	^{17}O 0.019%	^{18}O 0.205%
Cl	^{35}Cl 75.820%	^{36}Cl *trace*	^{37}Cl 24.180%

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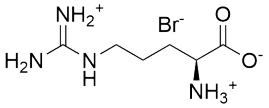
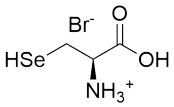
Advanced Amino Acid Assessment (25.0 Points)

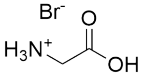
In this problem, you will be identifying an unknown amino acid hydrobromide from the list of eight candidates below. To do so, you will be titrating your samples with two types of titrations.



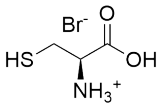
Arginine hydrobromide, **R** · **HBr**
pK_a = 9.00, 12.10
255.11 g · mol^{-1}



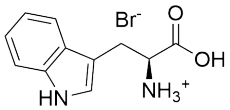
Selenocysteine hydrobromide, **U** · **HBr**
pK_a = 1.90, 5.23, 10.00
248.98 g · mol^{-1}



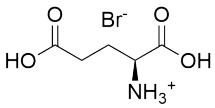
Glycine hydrobromide, **G** · **HBr**
pK_a = 2.34, 9.58
155.98 g · mol^{-1}



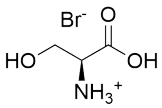
Cysteine hydrobromide, **C** · **HBr**
pK_a = 1.91, 8.14, 10.28
202.06 g · mol^{-1}



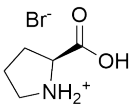
Tryptophan hydrobromide, **W** · **HBr**
pK_a = 2.38, 9.34
285.14 g · mol^{-1}



Glutamic acid hydrobromide, **E** · **HBr**
pK_a = 2.16, 4.15, 9.58
228.04 g · mol^{-1}



Serine hydrobromide, **S** · **HBr**
pK_a = 2.13, 9.05
186.00 g · mol^{-1}



Proline hydrobromide, **P** · **HBr**
pK_a = 1.95, 10.47
196.04 g · mol^{-1}

PREPARATION:

- Weight out the AA · HBr exactly and note down their masses on the student's exams. The masses need to be in the range between 575 mg and 625 mg.
- Each student needs 150 mL of a 0.0250 mol L^{-1} NaOH solution. Distribute in a 200 mL plastic bottle.
- Each student needs 170 mL of a 0.0150 mol L^{-1} KSCN solution. Distribute in a 200 mL plastic bottle.
- Each student needs 150 mL of a 0.0200 mol L^{-1} $AgNO_3$ solution. Distribute in a 200 mL plastic bottle and cover with aluminium foil.
- Each student needs half of a small vial filled with a bromothymol blue indicator solution. Prepare said solution by dissolving 0.2 g of the indicator in 100 mL of EtOH.
- Each student needs 30 mL of a 30 wt% HNO_3 solution. Distribute in small plastic bottles.
- Distribute 5 mL of a 10 w% $Fe(NO_3)_3 \cdot 9\,H_2O$ solution to each student in a small vial.
- Add a bit of a very basic solution to their waste beaker to ensure that the traces of SCN^- are never in an acidic medium (10 g NaOH should be enough per student. Dissolve in a bit of water and add a few drops of bromothymol blue.).

Materials

- 1× 50 mL Burette
- 2× 200 mL Erlenmeyer flask
- 1× 10 mL Volumetric pipette
- 1× 5 mL Volmetric pipette
- 1× 100 mL Volumetric flask
- 1× 25 mL Volumetric pipette
- 5× Plastic pipettes
- 1× Schott flask labelled "P2" (for waste, contains NaOH)

Chemicals

Sodium hydroxide (NaOH, 0.0250 mol L^{-1})
Silver nitrate ($AgNO_3$, 0.0200 mol L^{-1})
Potassium thiocyanate (KSCN, 0.0150 mol L^{-1})
Unknown amino acid hydrobromide (AA · HBr)
Bromothymol blue indicator solution
Nitric acid (HNO_3, 30 wt%)
Iron(III) nitrate ($Fe(NO_3)_3$, 6 wt%)

Safety

- Sodium hydroxide (NaOH) is caustic, causing chemical burns and blindness. **Make sure** that you are not wearing contact lenses and that your eyes and skin are properly shielded.
- Silver nitrate ($AgNO_3$) can irritate your skin and may cause blindness. **Protect** yourself properly by wearing goggles and gloves, etc. **Keep** the solution containing $AgNO_3$ protected from light, as it is light-sensitive. Contact with your skin will stain the skin for prolonged amounts of time.
- Potassium thiocyanate (KSCN) is an irritant. Free SCN^- ions should never be in an acidic solution, as toxic gasses may be produced. Therefore, **add** only a little KSCN at a time, as to not have an excess SCN^- in solution. **Remember** to add the indicator, to see when your SCN^- is no longer being consumed (colour change).
- Nitric acid (HNO_3) is a strong acid and potent oxidising agent. **Protect** your eyes and skin properly and **do not mix** it with flammable organic material.

Practical Final Exam 2025

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P2-4

English (Official)

Procedure

1. In your 100 mL volumetric flask, have received a sample of an unknown amino acid hydrobromide (AA · HBr), dissolved in some deionised water. The exact mass of your sample is noted below.
2. **Fill up** the volumetric flask with deionised water to the 100.00 mL mark. **Cap** and **shake** the flask thoroughly.

Student name: _____

Exact AA · HBr mass: _____ mg

Acid-Base Titration

1. **Transfer** 10.00 mL of the AA · HBr solution into an Erlenmeyer flask and **dilute** it to approximately 100 mL with deionised water.
2. **Add** a few drops of the bromothymol blue indicator solution.
3. **Titrate** the resulting solution with 0.0250 mol L^{-1} NaOH solution. The endpoint is reached once a green colour presists.
4. **Dispose** of the titrated sample into the flask labelled "P2".
5. **Repeat** steps **1**-**4** as needed.

Argentometric Titration

1. **Transfer** 5.00 mL of your AA · HBr solution into an Erlenmeyer flask.
2. **Add** 25.00 mL (an excess) of the $AgNO_3$ solution to the Erlenmeyer flask. A fine precipitate should turn the solution milky.
3. **Dilute** the solution to an approximate volume of 100 mL with deionised water.
4. **Add** 5.0 mL of 30 w% HNO_3.
5. **Add** three drops of the Fe^{3+} solution.
6. **Titrate** the excess Ag^+ ions with the 0.0150 mol L^{-1} KSCN solution. The endpoint is reached once a very faint orange colour persists.
7. **Dispose** of the mixture into the flask labelled "P2".
8. **Repeat** steps **1**-**7** as needed.

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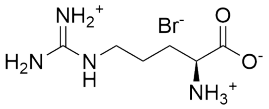
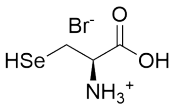
Theoretical Questions

Below, the eight possible amino acid hydrobromides are shown together with their names, their one-letter codes, their pK_a values (in ascending order), and their molecular weights.

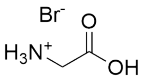
Note: *For the argentometric titration, all precipitates can be assumed to be completely insoluble.*



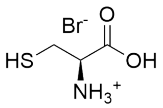
Arginine hydrobromide, **R**·**HBr**
pK_a = 9.00, 12.10
255.11 g·mol^{-1}



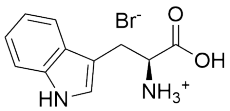
Selenocysteine hydrobromide, **U**·**HBr**
pK_a = 1.90, 5.23, 10.00
248.98 g·mol^{-1}



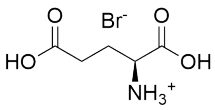
Glycine hydrobromide, **G**·**HBr**
pK_a = 2.34, 9.58
155.98 g·mol^{-1}



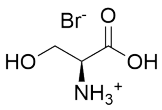
Cysteine hydrobromide, **C**·**HBr**
pK_a = 1.91, 8.14, 10.28
202.06 g·mol^{-1}



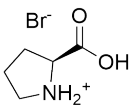
Tryptophan hydrobromide, **W**·**HBr**
pK_a = 2.38, 9.34
285.14 g·mol^{-1}



Glutamic acid hydrobromide, **E**·**HBr**
pK_a = 2.16, 4.15, 9.58
228.04 g·mol^{-1}



Serine hydrobromide, **S**·**HBr**
pK_a = 2.13, 9.05
186.00 g·mol^{-1}



Proline hydrobromide, **P**·**HBr**
pK_a = 1.95, 10.47
196.04 g·mol^{-1}

2.1 For each amino acid hydrobromide, **give** the number of protons N_{H^+} that are deprotonated in the acid base titration. The colour change of bromothymol blue from yellow to green occurs at pH = 7.1. 2.0 pt

SOLUTION:

R: 0 **U**: 2 **G**: 1 **C**: 1

W: 1 **E**: 2 **S**: 1 **P**: 1

This is due to the fact, that a proton can be "seen" in a titration, if its pK_a is approximately 2 units below that of the indicator. In essence, we are simply counting the number of protons with that property.

2.0 pts for eight correct assessments. -0.5 pts for each mistake, for a minimum of 0.0 pts. 2.0 pts total.

2.2 **Give** your accepted value of the volume of base V_{NaOH} in mL needed to reach the end point in the acid-base titration. 5.0 pt

SOLUTION:

The theoretical volume used is dependent on the exact mass given.

$V_{NaOH} = \frac{m_{sample} \cdot V_{aliquot} \cdot N_{H^+}}{M_{AA \cdot HBr} \cdot V_{sample} \cdot c_{NaOH}}$

For the given quantities, this simplifies to $V_{NaOH} = 28.065\,mL\,g^{-1} \cdot m_{sample}$

5.0 pts for any result within 0.5% of the theoretical result, decreasing linearly to 0.0 pts at ± 5%.

NOTE:

This point distribution may need to be changed in testing.

2.3 From your acid-base titration data, you can calculate the molecular weight of the unknown AA · HBr. Your result is dependent on the variable N_{H^+}. Based on your data, **calculate** the determined molecular weights of AA · HBr in g mol^{-1} for $N_{H^+} = 1, 2, 3$. **Show** your working and the formulae you use. 2.5 pt

SOLUTION:

As can be derived from the formula above $M_{AA \cdot HBr} = \frac{m_{sample} \cdot V_{aliquot} \cdot N_{H^+}}{V_{NaOH} \cdot V_{sample} \cdot c_{NaOH}}$

Plugging in the correct values gives

$M_{AA \cdot HBr} = 114.018\,g\,mol^{-1} \cdot N_{H^+}$, i.e. for

$N_{H^+} = 1$: $114.018\,g\,mol^{-1}$

$N_{H^+} = 2$: $228.040\,g\,mol^{-1}$

$N_{H^+} = 3$: $342.054\,g\,mol^{-1}$

The values will be different depending on the given V_{NaOH}.

*0.5 pts for expressing $M_{AA \cdot HBr}$ **proportionally** in terms of N_{H^+} no matter of the correctness of the formula. Another 0.5 pts for the correct formula. 0.5 pts for each correct numerical result (depending on result above). 2.5 pts total.*

2.4 In the argentometric titration, a fine precipitate turns the sample cloudy upon the addition of $AgNO_3$. A second precipitate is formed upon the addition of KSCN. For both reactions, **provide** a balanced reaction equation. 1.0 pt

SOLUTION:

All silver (pseudo-)halides are insoluble, giving:

Addition of $AgNO_3$:

$Ag^+ + Br^- \longrightarrow AgBr\downarrow$

Addition of KSCN:

$Ag^+ + SCN^- \longrightarrow AgSCN\downarrow$

0.5 pts for each correctly balanced reaction. 1.0 pts total.

2.5 **Give** your accepted value of the volume of titrant KSCN V_{KSCN} in mL needed to reach the end point in the argentometric titration. 5.0 pt

SOLUTION:

Assuming quantitative precipitation of Br^- by Ag^+ the remaining V_{SCN^-} that is needed for full precipitation is given by:

$$V_{SCN^-} = \frac{V_{Ag^+,added} \cdot c_{Ag^+,added} - \left(\frac{V_{aliquot}}{V_{sample}} \cdot \frac{m_{sample}}{M_{AA \cdot HBr}}\right)}{c_{SCN^-}}$$

For the given quantities, this simplifies to:

$V_{SCN^-} = -11.694\,\text{mL g}^{-1} \cdot m_{sample} + 26.667\,\text{mL}$

5.0 pts for any result within 1% of the theoretical result, decreasing linearly to 0.0 pts at ± 10%.

NOTE:

This point distribution may need to be changed in testing.

2.6 From the argentometric titration, **calculate** the resulting molecular weight of $AA \cdot HBr$ in g mol^{-1}. **Show** your working. 2.5 pt

SOLUTION:

$n_{AA \cdot HBr}$

$n_{AA \cdot HBr} = n_{Ag^+} - n_{SCN^-}$, therefore

$n_{AA \cdot HBr} = c_{Ag^+,added} \cdot V_{Ag^+,added} - c_{SCN^-} \cdot V_{SCN^-}$

$n_{AA \cdot HBr} = 4 \times 10^{-4}\,\text{mol} - 0.015\,\text{mol L}^{-1} \cdot V_{SCN^-}$

For $m_{sample} = 0.745\,\text{g}$ (see above), this becomes $n_{AA \cdot HBr} = 0.131\,\text{mmol}$.

Molecular weight:

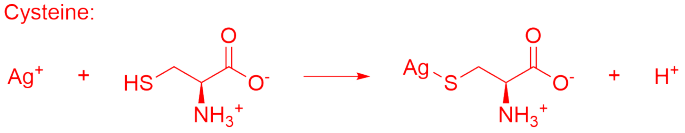
This is now simply $M_{AA \cdot HBr} = \frac{m_{sample}}{n_{AA \cdot HBr}} = 228.04\,\text{g mol}^{-1}$ for the ideal case.

1.0 pts for seeing that this is a back titration (i.e. subtracting the n). 1.0 pts for correct mathematical reasoning leading to the molecular weight. 0.5 pts for a "correct" numerical value (depending on their accepted value)

2.7 Under basic conditions, both C·HBr and U·HBr could react with Ag^+ in unintended reactions. **Write** balanced chemical equations for those reactions. **Provide** the driving force for those reactions. 3.0 pt

SOLUTION:

Cysteine:



Selenocysteine:

(ionic formulae (with no Ag—S or Ag—Se bond) are also accepted)

Explanation: Both S and Se have very high affinity for Ag^+ (or vice versa). This can be seen e.g. in the insolubility and stability of Ag_2S and Ag_2Se. Another driving force is the insolubility of these compounds, removing them from the equilibrium. (Soft-soft interactions being very covalent are the driving force, but this is not expected to be known.)

1.0 pts for each chemical equation fulfilling the spirit of the answer (we neglect acid-base criteria here). 1.0 pts if they mention high affinity of the chalcogenides to silver and/or the insolubility of the products.

2.8 **List** the two species involved in the colour change to orange in the argentometric titration. No balanced equation is needed. 1.0 pt

SOLUTION:

The two species involved are Fe^{3+} and SCN^-, which form a deep red complex.

0.5 pts for each correct species. 0.0 pts for mentioning more than two species. 1.0 pts total.

2.9 **State** which AA · HBr was in your flask. **Explain** your decision. 3.0 pt

SOLUTION:

☐ **R** ☐ **U** ☐ **G** ☐ **C**

☐ **W** ☒ **E** ☐ **S** ☐ **P**

Any founded explanation is accepted, though it will most likely be based on molecular weight, as

- of the possible molecular weights based on N_{H^+}, only it matches up
- the molecular weight calculated from the argentometric titration matches that of E · HBr
- a combination of the above

2.0 pts for the correct identity of the amino acid. +1.0 pts for any sound, evidence-based reasoning (even if amino acid is wrong). If the correct amino acid is identified, however, no explanation is given, 1.0 pts will be awarded. 3.0 pts total.