Practical Final Exam 2025

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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.

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

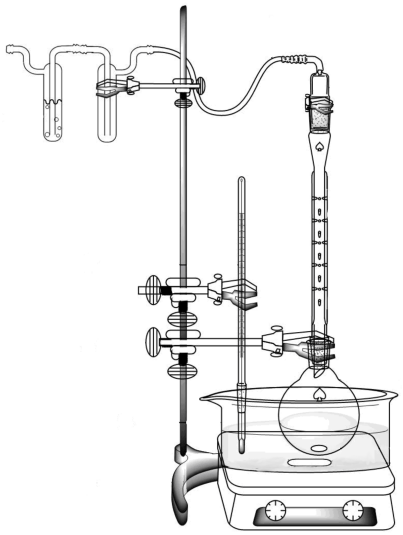
- *p*-Methoxyacetophenone
- Bleach (NaOCl solution, 14 w%)
- Sodium hydrogensulfite ($NaHSO_3$, 40 w%)
- Deionised water
- Eluent (Cyclohexane:EtOAc, 80 v%:20 v%)
- Sodium hydroxide solution (NaOH, 1 mol L^{-1})
- NaOH in EtOH + H_2O (trap solution in bubbler)
- Hydrochloric acid (HCl, 2 mol L^{-1})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

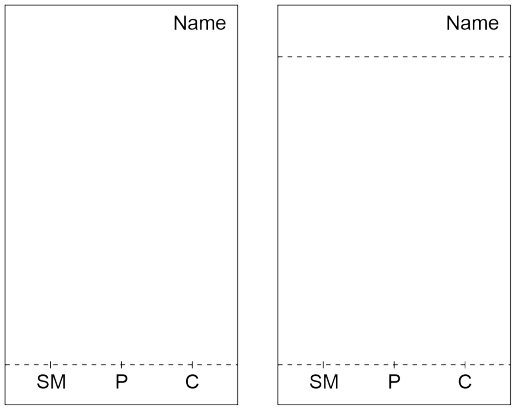
Synthesis



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.

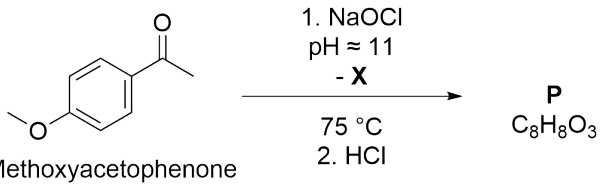
TLC Analysis



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

$R_{f,\mathbf{SM}} =$ _____ $R_{f,\mathbf{P}} =$ _____

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

☐ More polar ☐ Less polar

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

☐ Product is pure ☐ Product is not pure

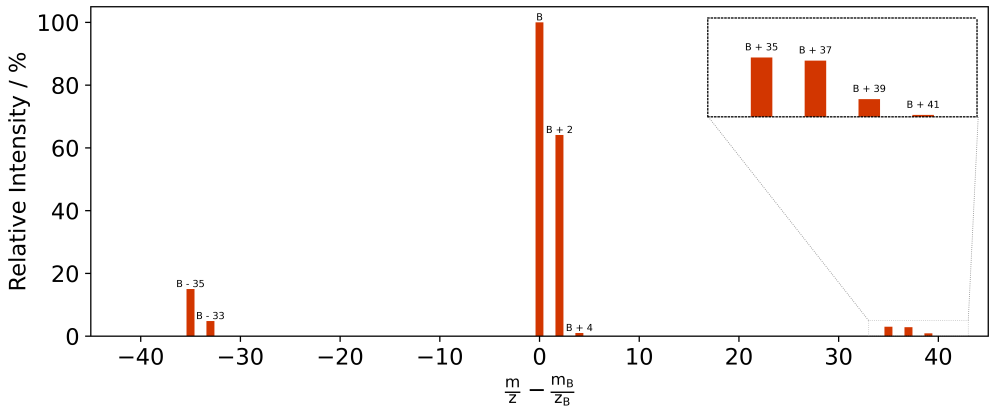
Justification:

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

n_C = _____

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

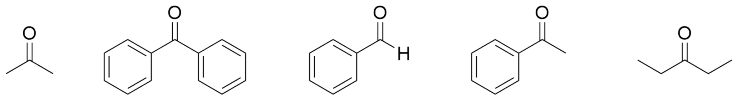
1.6 **Draw** the structure of the product **P**. 3.0 pt

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*** $M_{\mathbf{X}}' = 100\,\mathrm{g\,mol^{-1}}$.

Maximum yield of:

P: _____ mg **X**: _____ mg

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

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

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

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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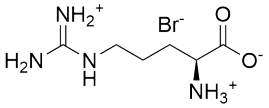
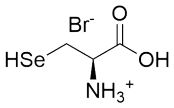
Advanced Amino Acid Assessment (25.0 Points)

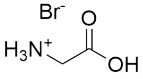
In this problem, you will be identifying an unknown amino acid hydrobromide from the list of eight candidates below. To do so, you will be titrating your samples with two types of titrations.



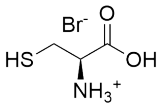
Arginine hydrobromide, **R** · **HBr**
pK_a = 9.00, 12.10
255.11 g · mol^{-1}



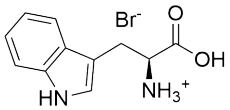
Selenocysteine hydrobromide, **U** · **HBr**
pK_a = 1.90, 5.23, 10.00
248.98 g · mol^{-1}



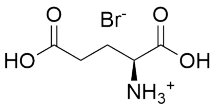
Glycine hydrobromide, **G** · **HBr**
pK_a = 2.34, 9.58
155.98 g · mol^{-1}



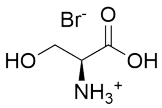
Cysteine hydrobromide, **C** · **HBr**
pK_a = 1.91, 8.14, 10.28
202.06 g · mol^{-1}



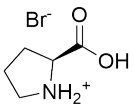
Tryptophan hydrobromide, **W** · **HBr**
pK_a = 2.38, 9.34
285.14 g · mol^{-1}



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}

Materials

- $1\times$ 50 mL Burette
- $2\times$ 200 mL Erlenmeyer flask
- $1\times$ 10 mL Volumetric pipette
- $1\times$ 5 mL Volmetric pipette
- $1\times$ 100 mL Volumetric flask
- $1\times$ 25 mL Volumetric pipette
- $5\times$ Plastic pipettes
- $1\times$ 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.

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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.

Titration Run	1	2	3	4	5
V_{NaOH}/mL					

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.

Titration Run	**1**	**2**	**3**	**4**	**5**
V_{KSCN}/mL					

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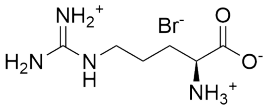
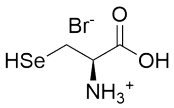
Theoretical Questions

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.

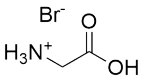
Note: *For the argentometric titration, all precipitates can be assumed to be completely insoluble.*



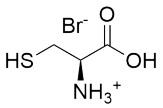
Arginine hydrobromide, **R** · **HBr**
pK_a = 9.00, 12.10
255.11 g · mol^{-1}



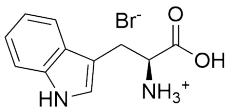
Selenocysteine hydrobromide, **U** · **HBr**
pK_a = 1.90, 5.23, 10.00
248.98 g · mol^{-1}



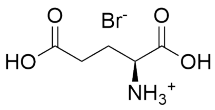
Glycine hydrobromide, **G** · **HBr**
pK_a = 2.34, 9.58
155.98 g · mol^{-1}



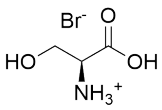
Cysteine hydrobromide, **C** · **HBr**
pK_a = 1.91, 8.14, 10.28
202.06 g · mol^{-1}



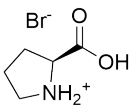
Tryptophan hydrobromide, **W** · **HBr**
pK_a = 2.38, 9.34
285.14 g · mol^{-1}



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

R: _____ **U**: _____ **G**: _____ **C**: _____

W: _____ **E**: _____ **S**: _____ **P**: _____

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

V_{NaOH} = _____ mL

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

$N_{H^+} = 1$: _____ g mol^{-1} 2: _____ g mol^{-1} 3: _____ g mol^{-1}

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

Addition of $AgNO_3$:

Addition of KSCN:

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

V_{KSCN} = _____ mL

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

Molecular weight: _____ $g\,mol^{-1}$

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

C · HBr:

U · HBr:

Driving force:

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

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

☐ **R** ☐ **U** ☐ **G** ☐ **C**

☐ **W** ☐ **E** ☐ **S** ☐ **P**

Explanation: