P1. Synthesise and analyse (A new complex problem)

Equipment

Item	Quantity	Label
Part A		
Magnetic stirrer bar	1	
Steel spatula	1	
Sintered funnel	1	
Round bottom flask with ground-glass joint	1	
Vacuum pump with hose connected to a bubbler at the back	1	
Glass beaker, 100 mL	1	
Pre-weighed glass vial with cap for product	1	**A + student code**
Measuring cylinder, 10 mL	1	
Magnetic rod	1	
Yellow paper for product transfer	1	
Part B		
Graduated pipette, 10.0 mL	1	
Glass beaker, 50 mL	2	
Erlenmeyer flask, 100 mL	2	
Measuring cylinder, 10 mL	1	
Part C		
Plastic Pasteur pipette, 3 mL	12	
Eppendorf tube, 0.5 mL	15	

Experiment

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Q1-2

English (Official)

Chemicals

Name	State	Concentration	Quantity	Placed in	Label
Part A					
Solution **R1** (synthesis)	aq	To be determined	10.0 mL	Glass vial with cap, 30 mL	**R1-syn**
Solution **R2**	aq	1.03 M	10.0 mL	Glass vial with cap, 30 mL	**R2**
Pyridine in ethanol (synthesis)	sol	8.4 % v/v	10 mL	Plastic tube (15 mL) in plastic cup	**Py**
Part B					
Solution **R1** (analysis)	aq	To be determined	10.0 mL	Volumetric flask, 100 mL	**R1-an**
Ethylenediamine-tetraacetic acid disodium salt	aq	0.0770 M	50 mL	Bottle, 50 mL	**EDTA**
Ammonia buffer	aq	-	40 mL	Bottle, 50 mL	**NH_3 buffer**
Eriochrome Black T	s	1% in NaCl	0.2 g	Sample container	**Eriochrome**

Part C					
Complex **A**	s	-	10 mg	Eppendorf tube, 0.5 mL	**A**
Iron(III) nitrate	aq	0.4 %	0.5 mL	Eppendorf tube, 0.5 mL	**Fe^{3+}**
Copper(II) sulphate	aq	5%	0.5 mL	Eppendorf tube, 0.5 mL	**Cu^{2+}**
Pyridine in ethanol (analysis)	sol	8.4 % v/v	0.5 mL	Eppendorf tube, 0.5 mL	**Py-qual**
X^-	aq	0.1 M	0.5 mL	Eppendorf tube, 0.5 mL	**X^- – qual**
Unknown solvent	l	-	1 mL	Eppendorf tube, 1.5 mL	**Solvent**
Dithizone	s	-	1 mg	Eppendorf tube, 0.5 mL	**Dithiz.**
Dichloromethane	l	-	0.75 mL	Eppendorf tube, 1.5 mL	**CH_2Cl_2**

Experiment

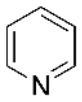
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Q1-3

English (Official)

14% of total											
Question	1.1	1.2	1.3	1.4	1.5	1.6	1.7	1.8	1.9	1.10	**Total**
Total	10	0	10	1	3	1	4	2	1	1	33

In this problem, you will synthesise and analyse complex **A** with molecular formula $[M(Py)_mX_n]$ where:

- M^{k+} is an unknown metal cation; - Py (pyridine); - X^- is either OCN^- or SCN^-; - *m* = 0, 1, 2, 3, ...; - *n* = 1, 2, 3, ...	*Py* = 

Part A. Synthesis

To synthesise complex **A**, solutions **R1-syn** and **R2** are used:

- Solution **R1-syn** contains 10.0 mL of an aqueous solution of a M^{k+} salt with unknown concentration.
- Solution **R2** contains 10.0 mL of an aqueous solution of potassium salt of X^- with a concentration of 1.03 M.

1. **Put** the stirrer bar in the 100 mL beaker.
2. **Transfer** the whole solution **R1-syn** to the same beaker.
3. **Rinse** the vial **R1-syn** with 1 mL of water using a Pasteur pipette and **transfer** the remains to the beaker.
4. **Add dropwise** the whole solution **R2** using a Pasteur pipette while stirring on the magnetic stirrer.
5. **Rinse** the vial **R2** with 1 mL of water using a Pasteur pipette and **transfer** the remains to the beaker.
6. **Add dropwise** the whole pyridine solution (**Py**) using a Pasteur pipette while stirring.
7. **Continue** stirring for 5 minutes.
8. **Connect** sintered funnel to a round bottom flask to assemble the vacuum filtration apparatus.
9. **Filter** the product using vacuum filtration and **wash** it three times with 10 mL portions of water.
10. Once you have finished washing, **ask** the lab assistant to collect the filtrate to avoid spills. For this, **raise** the green card with a question mark, **turn off** the pump and **disconnect** the vacuum hose. **Remove** the filter and **hand** the flask to the lab assistant. The lab assistant will discard the filtrate and **return** the empty flask.
11. **Reassemble** the filtration setup and **let** your product dry for **1 hour** with the vacuum turned on. During the drying, **it is important** to **use** your spatula **to break up** any aggregates of the product formed on the filter.
12. After complete drying, carefully **transfer** your product to the vial labelled "**A + Student Code**". For this you can use the yellow paper provided.

Q1.1	The lab assistant **will collect** your product at the end of the exam. Both you and the lab assistant should **sign** your answer sheet. The organisers will further dry your product in an oven before measuring the mass, however **it is important you have vacuum dried the product for 1 hour and broken up any aggregates** to remove the vast majority of the water before putting it in the vial. (If the product is **not transferred** to the vial, it will **not be collected** and **ZERO points** will be awarded for the synthesis.)	10 pt

Part B. Quantitative Analysis

You will use a complexometric titration to determine the concentration of M^{k+} in solution **R1-an**. The volumetric flask **R1-an** contains 10.0 mL of M^{k+} solution with the same concentration as in **R1-syn** in **Part A**.

1. **Dilute** the **R1-an** solution in the volumetric flask with H_2O up to the mark and **mix** thoroughly.
2. **Fill** the burette with 0.0770 M **EDTA**.
3. **Add** a 10.0 mL aliquot of the diluted solution **R1-an** using a graduated pipette, 5 mL of ammonia buffer solution (**NH_3 buffer**), and a tip of the plastic spatula of indicator (**Eriochrome**; the spatula is inside the sample container) to an Erlenmeyer flask. **Close** the bottle of **NH_3 buffer** after using it.
4. **Titrate** the mixture with 0.0770 M **EDTA** solution until the colour turns a stable blue.
5. **Repeat** steps 3-4 if necessary.

Q1.2	**Record** the observed volumes (V_0, mL – initial burette reading; V_f, mL– final burette reading; T, mL - titre). This table is for your convenience and will not be graded.	0 pt

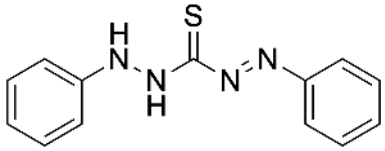
Q1.3	**Record** your accepted final titre of **EDTA** (V_{EDTA}, mL).	10 pt

Q1.4	**Calculate** the ratio of X^- and M^{k+} ($n_{X^-}/n_{M^{k+}}$) used in the synthesis according to your result. **Round** your answer to two decimal places.	1 pt

Part C. Qualitative Analysis

In this part, you need to perform different chemical tests to identify the components of complex **A**.

1. **Prepare** a 0.4 mL solution of complex **A** in the Eppendorf tube by adding the solvent with a Pasteur pipette from the Eppendorf tube labelled **Solvent** until the 0.4 mL mark. If the sample has not dissolved completely, shake the tube and use the supernatant for the tests below.
2. **Prepare** a 0.4 mL solution of the indicator Dithizone (**Dithiz.**) in the Eppendorf tube by adding the solvent with a Pasteur pipette from the Eppendorf tube labelled **Solvent** until the 0.4 mL mark. The structure of the indicator is given below.



3. **Take** four empty Eppendorf tubes and **transfer** 1 drop of the solution of **A** obtained to each.
4. **Perform** the individual tests **T1–T4** by **adding** the following reagents to each of the four Eppendorf tubes:

- **T1**: 2 drops of **Fe^{3+}**(acidified $Fe(NO_3)_3$)
- **T2**: 2 drops of the prepared **Dithizone** solution
- **T3**: 2 drops of **Cu^{2+}**($CuSO_4$)
- **T4**: 2 drops of **Cu^{2+}**($CuSO_4$) followed by 3 drops of **CH_2Cl_2** and **shake**

You may perform additional tests; for this you are provided with extra equipment and chemicals (e.g., solutions of Py and X^-). *Additional information:* $\rho(CH_2Cl_2) = 1.32$ g/cm^3

Q1.5 **Tick** the correct box(es) corresponding to your observation(s) in **T1 - T3**. 3 pt

(a) No visible change
(b) Formation of precipitate
(c) Solution became colourless
(d) Solution became red or pink

Q1.6 **Tick** the correct box(es) corresponding to your observation(s) in **T4**. 1 pt

(a) The aqueous phase changed colour
(b) The aqueous phase did not change colour
(c) The CH_2Cl_2 phase changed colour
(d) The CH_2Cl_2 phase did not change colour

Q1.7 The above tests **T1–T4** are designed to identify the components of the complex **A**. **Tick** the box(es) that correctly describe(s) the result(s) of each test. 4 pt

(a) Identifies presence of metal M^{k+}
(b) Identifies presence of ligand X^-
(c) Identifies presence of ligand Py
(d) Identifies absence of ligand Py

Q1.8 **Identify** ligand X^- and metal M^{k+} with its charge in complex **A**. 2 pt

Q1.9 From the list below, **choose** the correct description of M^{k+}/X^{-} stoichiometry in the synthesis based on your reported results. Consider as little as an additional 1 mol% as an excess of the reagent. 1 pt

(a) M^{k+} and X^{-} were used in a stoichiometric amount
(b) M^{k+} was used in excess
(c) X^{-} was used in excess

Q1.10 **Write** the molecular formula of complex **A** assuming it follows the 18-electron rule. 1 pt