Student Code | CAN-1

Practical Problems

"Bonding the World with Chemistry"

49th INTERNATIONAL CHEMISTRY OLYMPIAD

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General Instructions:

- **Pages:** This document contains 36 pages for the practical exam tasks (including the answer sheets). The final two pages contain a periodic table and a table of ^{1}H NMR chemical shifts. **There are a total of three tasks: Task 1A, Task 1B, and Task 2.**

- **Exam Reading:** You will have 15 minutes to read this exam booklet before starting the experiments. The official English version of this examination is available on request only for clarification.

- **Exam Time:** You will have a total of 5 hours to complete all practical tasks. When planning your work, note that several steps require 20-30 minutes.

- **Start/Stop:** You may begin only after the **"Start Command"** is given and must stop ALL work immediately when the **"Stop Command"** is announced.

  - The supervisor will announce when there is 30 minutes remaining
  - If you do not stop working after the "**Stop Command**" has been given, you will receive a 0 on the practical
  - After the **"Stop Command"** has been given, place your exam papers in your exam envelope and wait at your station. The lab supervisor will pick up your exam paper and your submitted items.

- **Safety:** You must follow the safety rules given in the IChO regulations. While you are in the laboratory, you must wear laboratory goggles. Prescription safety glasses may be used if the supervisor approves. You **MAY** use gloves provided when handling chemicals.

  - If you break the safety rules given in the IChO regulations, you will receive only **ONE WARNING** from the laboratory supervisor. Any breaking safety rules after one warning will result in being dismissed from the laboratory and zero marks for the entire practical examination.
  - No eating or drinking allowed in the laboratory.
  - **Pipetting by mouth is strictly forbidden.**
  - Do not hesitate to ask your assistant or lab supervisor if you have any questions concerning safety issues. Inform your lab supervisor if you need to leave the laboratory for a restroom break or if having a drink or snacks.

- **Working space:** You are only allowed to work in the space assigned for you. Shared space and shared equipment must be cleaned after use.

- **Chemical Refills/Replacements:** Chemicals and lab equipment, unless noted, are not supposed to be refilled or replaced. Chemicals and equipment will be refilled or

replaced without penalty only for the first incident. Each further incident will result in the deduction of 1 point from your 40 practical exam points. You must ask the lab supervisor for any replacements you require. If you break equipment that you no longer need, you do not need to ask for a replacement.

- ☐ **Disposal:** Leave all chemicals and equipment on your working space. Chemical waste must be disposed of in the designated waste bottle for each task.

- ☐ **Answer sheets:** All results and answers must be clearly written in the appropriate area on the answer sheets for grading. **Only answers written in pen will be graded.**

  - Your student code should appear on every page.
  - Use only the pens provided for you.
  - Anything written outside the appropriate area on the answer sheets will not be graded. **You may use the backside of the sheets for rough work.**

- ☐ **Stay hydrated throughout the practical exam**. Drinks and snacks are provided outside the laboratory.

- ☐ **The UV-visible spectrophotometer is shared between you and another student**

  During the first two hours, you may use the spectrophotometer whenever it is free. If in use you must wait until the other student finishes. You cannot use the spectrophotometer for more than 1 hour in a row (longer than that and you will be asked to stop to allow the other student to use it).

  You may come back to the spectrophotometer if it is free. Organize your work so that you do not waste your time waiting.

Time	0900-1000	1000-1100	1100-1200	1200-1300	1300-1400
Slot	Free	Free	L	R	Free

L = student on the left side of the spectrophotometer

R = student on the right side of the spectrophotometer

You may work on the three tasks

in any order

Practical Exam

Task 1A

Chemicals and Equipment (Task 1A):

I. Chemical and materials (the actual labeling for each is given in **bold** font)

	Hazard Statements[a]
Instrument check solution, 80 mL in a plastic bottle	
2.00×10^{-4} M **Methyl orange indicator solution,** 30 mL in a wide mouth glass bottle	H301
1.00×10^{-3} M **Bromothymol blue indicator solution,** 30 mL in a wide mouth glass bottle	
Methyl red indicator solution, 10 mL in a wide mouth glass bottle	H225-H319-H371
1 M **HCl**, 30 mL in a plastic bottle	H290-H314-H335
1 M **NaOH**, 30 mL in a plastic bottle	H290-H314
buffer **solution A,** 110 mL in a plastic bottle	
Unknown solution X, 50 mL in a plastic bottle	
Unknown solution Y, 50 mL in a plastic bottle	
Unknown solution Z, 50 mL in a plastic bottle	

[a]**See page** 34 **for definition of Health Statements**

II. Equipment and glassware

Shared Equipment	**Quantity**
UV-visible spectrophotometer	1 per 2 students
Personal Equipment	**Quantity**
Beaker, 25 mL	2
Volumetric flask, 25.00 mL	9
Measuring pipette, 2.00 mL	2
Measuring cylinder, 10.0 mL	3
Pasteur pipette	6
Rubber bulb for Pasteur pipette	6
Pipette filler bulb (3-way)	1
Pipette tray	1
Test tube (13 x 100 mm)	6
Test tube rack	1
Plastic cuvette, **optical path length = 1.00 cm**	1
Waste bottle, 1 L	1
Sticker label set in a zipped bag	1

Task 1A 13%	a		b			c		Total
	a1	a2	b1	b2	b3	c1	c2	
Total	12	2	6	1	1	2	2	26
Score								

13% of Total Score

Task 1A: Acid-base Indicators and their Application for pH Measurement

Acid-base indicators are weak acids (or bases) exhibiting different colours when they are present in solution in their acidic form (HIn, colour 1) or in their basic form (In^-, colour 2). They undergo the following reaction in dilute aqueous solution:

$$HIn \rightleftharpoons H^+ + In^-$$

As the pH of a solution containing the indicator changes, the equilibrium will be driven either towards reactants (HIn), or products (In^-) causing the solution colour to change depending on the concentration of each form present. In strongly acidic solution, most of the indicator will be present in the HIn form (colour 1) and in strongly basic solution, most of the indicator will be in the In^- form (colour 2). At intermediate pH values, the solution colour will be a mix of colour 1 (absorption at wavelength 1) and colour 2 (absorption at wavelength 2), depending on the relative amounts of HIn and In^- present.

By monitoring the absorbance values at two wavelengths, the concentrations of HIn and In^- can be calculated using the following expressions:

$$\begin{aligned} A^{\lambda 1}{}_{total} &= A^{\lambda 1}{}_{HIn} + A^{\lambda 1}{}_{In^-} \\ &= \varepsilon^{\lambda 1}{}_{HIn}\, b[HIn] + \varepsilon^{\lambda 1}{}_{In^-}\, b[In^-] \\ A^{\lambda 2}{}_{total} &= A^{\lambda 2}{}_{HIn} + A^{\lambda 2}{}_{In^-} \\ &= \varepsilon^{\lambda 2}{}_{HIn}\, b[HIn] + \varepsilon^{\lambda 2}{}_{In^-}\, b[In^-] \end{aligned}$$

where b is the path length of solution (1.00 cm in this experiment) and ε is the molar absorptivity.

At a certain pH value, the relative amounts of HIn and In^- in solution are related to the acid dissociation constant (K_a) of the indicator:

$$K_a = \frac{[H^+][In^-]}{[HIn]}$$

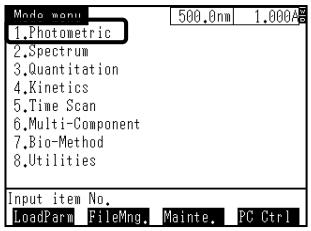
Therefore, for a given pH value, K_a of the indicator can be calculated when the relative amounts of HIn and In^- in solution are known.

Experimental Set-up

Instructions for using the spectrophotometer

1. Set up the spectrophometer to measure the absorbance at the desired wavelength following the procedure shown in the diagram on the next page.
2. Wipe the outside of a cuvette containing distilled water and insert it into the sample compartment.
3. Adjust the zero absorbance using water.
4. Remove the cuvette and replace the water with the sample solution to be analyzed. Make sure to tap out any bubbles and wipe the outside of the cuvette before placing it into the sample compartment.
5. Read the absorbance value of the sample.

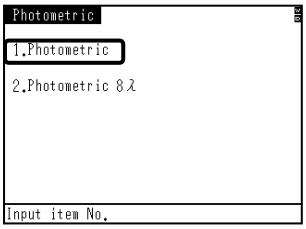
IMPORTANT: when changing the wavelength, make sure to adjust the zero absorbance using water in the cuvette.



Step 1: Press "1"

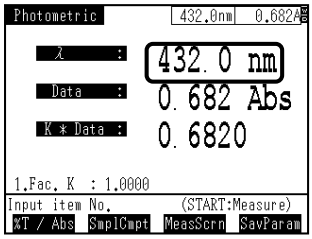
Press the icon on the keypad to select "Photometric" mode

Note: If the main menu as shown in the left picture is not displayed on the screen, press [return] on the keypad.



Step 2: Press "1"

Press the icon on the keypad to select Photometric mode "single wavelength mode"



Step 3: Set the wavelength

Press "**[GO TO WL]"** on the keypad to set the wavelength

Enter the wavelength number on the keypad

Note: For example, if the desired wavelength is 432, press "4" "3" "2" on the keypad.

Press **"[ENTER]"** on the keypad

Note: If the Abs is not displayed on the screen, press [F1] on the keypad to switch between %T and Abs

Rinse with deionized water

Fill the solution around ¾ of the cuvette height and wipe with paper

Step 4: Get the absorbance value

Place the cuvette **containing water** in the sample compartment and press "**[AUTO ZERO]**" on the keypad.

Place the cuvette **containing the sample solution** in the sample compartment to measure the absorbance.

Repeat Steps 3 and 4 to measure the absorbance at another wavelength

General Information

In 0.1 M HCl, all indicators are in the acidic form (HIn) only.

In 0.1 M NaOH, all indicators are in the basic form (In^-) only.

The answers in the dotted line boxes will not be marked – only the answers in the solid boxes will be graded.

NOTE:

Students are suggested to check the spectrophotometer before use by measuring the absorbance values of the instrument check solution at two different wavelengths, i.e., 430 nm and 620 nm.

Spectrophotometer No. _____ is used throughout the experiment.

Record the absorbance values of the instrument check solution:

	A (at 430 nm)	A (at 620 nm)
Measured value	_____	_____
Guided value	0.220 – 0.260	0.450 – 0.510

If the measured values are within the guided values, students can proceed with further experiments. *If not, students should ask for assistance.*

Part a

Absorbance measurement of an acid-base indicator (methyl orange) in strong acid and strong base

1. Pipette **1.50 mL** of 2.00×10^{-4} M **methyl orange indicator** solution into a 25.00 mL volumetric flask.

   Add 2.5 mL of 1 M HCl into the flask and make up to the volume using distilled water. Record the absorbance at 470 nm and 520 nm.

2. Pipette **2.00 mL** of 2.00×10^{-4} M **methyl orange indicator** solution into a 25.00 mL volumetric flask.

   Add 2.5 mL of 1 M NaOH into the flask and make up to the volume using distilled water. Record the absorbance at 470 nm and 520 nm.

3. Calculate the molar absorptivities at 470 nm and 520 nm of the acidic and basic forms of **methyl orange**.

a1) Record the absorbance values of **methyl orange** in acidic and basic solutions

(You do not need to fill the entire table.)

methyl orange in acidic form	A (at 470 nm)	A (at 520 nm)
Trial 1		
Trial 2		
Trial 3		
Accepted value **(3 digits after decimal point)**	_____	_____

methyl orange in basic form	A (at 470 nm)	A (at 520 nm)
Trial 1		
Trial 2		
Trial 3		
Accepted value **(3 digits after decimal point)**	_____	_____

a2) Calculate the molar absorptivities of the acidic form and basic form of **methyl orange** (unit: L mol^{-1} cm^{-1})

Blank area for calculation

The molar absorptivities of **methyl orange** are as follows: (unit, L mol^{-1} cm^{-1})

	acidic form (HIn)		basic form (In^-)	
methyl orange	ε^{470}_{HIn}	ε^{520}_{HIn}	$\varepsilon^{470}_{In^-}$	$\varepsilon^{520}_{In^-}$
	_____	_____	_____	_____

Part b

Absorbance measurement of an acid-base indicator (bromothymol blue) in buffer solution

Bromothymol blue is an indicator which appears yellow when present in its acidic form (HIn) and blue when in its basic form (In^-).

The molar absorptivities of bromothymol blue in its acidic form are 16,600 L mol^{-1} cm^{-1} at 430 nm and 0 L mol^{-1} cm^{-1} at 620 nm.

The molar absorptivities of bromothymol blue in its basic form are 3,460 L mol^{-1} cm^{-1} at 430 nm and 38,000 L mol^{-1} cm^{-1} at 620 nm.

1. Pipette **1.00 mL** of 1.00×10^{-3} M **bromothymol blue indicator** solution into a 25.00 mL volumetric flask, and make up the remaining volume **using solution A**. (Note: solution A is a buffer solution, pH = 7.00)
2. Record the absorbance at 430 nm and 620 nm.
3. Calculate the concentrations of the acidic form and basic form of **bromothymol blue indicator** solution in the volumetric flask.
4. Calculate the acid dissociation constant of **bromothymol blue**.

b1) Record the absorbance values of **bromothymol blue** in buffer solution:

(You do not need to fill the entire table.)

bromothymol blue in buffer solution	A (at 430 nm)	A (at 620 nm)
Trial 1		
Trial 2		
Trial 3		
Accepted value **(3 digits after decimal point)**	_____	_____

b2) Calculate the concentrations of the acidic form and basic form of **bromothymol blue** indicator in the resulting solution:

Blank area for calculation

The concentrations of the acidic form and basic form of bromothymol blue in the resulting solution are as follows:

[HIn], mol L^{-1}	[In^-], mol L^{-1}
_____ (3 significant figures)	_____ (3 significant figures)

b3) Calculate the acid dissociation constant of **bromothymol blue** from this experiment:

Blank area for calculation

The acid dissociation constant of **bromothymol blue** from this experiment is as follows:

The acid dissociation constant = _____
(3 significant figures)

Part c

Determination of solution pH by using an acid-base indicator (methyl red)

Methyl red is an indicator which is reddish-pink in its acidic form (HIn) and yellow in its basic form (In^-).

The molar absorptivities of methyl red in its acidic form are 9,810 L mol^{-1} cm^{-1} at 470 nm and 21,500 L mol^{-1} cm^{-1} at 520 nm.

The molar absorptivities of methyl red in its basic form are 12,500 L mol^{-1} cm^{-1} at 470 nm and 1,330 L mol^{-1} cm^{-1} at 520 nm.

The pK_a of methyl red is 4.95.

Note: *there is no need to accurately measure the volumes used in this part, as it does not affect the accuracy of the results obtained.*

1. Fill a test tube one quarter full with solution of unknown pH (**X**). Add three drops of **methyl red** into the solution and mix thoroughly. Record the colour.
2. Fill a test tube one quarter full with solution of unknown pH (**Y**). Add three drops of **methyl red** into the solution and mix thoroughly. Record the colour.
3. Fill a test tube one quarter full with solution of unknown pH (**Z**). Add three drops of **methyl red** into the solution and mix thoroughly. Record the colour.

Record the colour change of indicator in sample solutions (no mark)

Indicator	Colour observed		
	in sample **X**	in sample **Y**	in sample **Z**
Methyl red			

c1) Select one solution from the three sample solutions for which the pH can be determined spectrophotometrically by using **methyl red** as an indicator:

☐ Sample **X** ☐ Sample **Y** ☐ Sample **Z**

4. Use a measuring cylinder to transfer 10 mL of the selected unknown solution into a beaker. Add three drops of **methyl red** indicator into the solution and mix thoroughly. Record the absorbance at 470 nm and 520 nm.
5. Calculate the concentration ratio of the basic form and the acidic form of **methyl red** in the solution.
6. Calculate the pH of the selected unknown solution.

Record the absorbance values of the resulting solution

selected unknown solution	A (at 470 nm)	A (at 520 nm)

c2) Calculate the concentration ratio of the basic and acidic forms of **methyl red** indicator in an unknown solution and the pH value of the unknown solution:

Blank area for calculation

Calculate the concentration ratio of the basic form and acidic form of methyl red indicator in an unknown solution and the pH value of the unknown solution:

sample	$[In^-] / [HIn]$	pH
	_____ **(2 digits after decimal point)**	_____ **(2 digits after decimal point)**

Practical Exam

Task 1B

Chemicals and Equipment (Task 1B)

I. Chemicals and materials (the actual labeling for each is given in bold font)

	Health Statements[a]
Solution A (KIO_3: 10.7042 g in 5.00 L), 60 mL in a plastic bottle	H272-H315-H319-H335
Solution B (Saturated $Ca(IO_3)_2$ solution), 50 mL in a plastic bottle	H272-H315-H319-H335
Solution C (Saturated $Ca(IO_3)_2$ in unknown dilute KIO_3 solution), 50 mL in a plastic bottle	H272-H315-H319-H335
Solution of **$Na_2S_2O_3$**, 200 mL in a plastic bottle	
KI 10% (w/v), 100 mL in a plastic bottle	H300+H330-H312-H315-H319-H335
HCl 1 M, 100 mL in a plastic bottle	H290-H314-H335
Starch solution 0.1% (w/v), 30 mL in a dropping glass bottle	
Distilled water, 500 mL in a wash bottle	
Distilled water, 1000 mL in a plastic container	

[a]See page 34 for definition of Risk and Safety Phrases

II. Equipment and glassware

Personal Equipment	**Quantity**
Beaker, 100 mL	2
Beaker, 250 mL	1
Erlenmeyer flask, 125 mL	9
Transfer pipette, 5.00 mL	2
Transfer pipette, 10.00 mL	1
Measuring cylinder, 10.0 mL	1
Measuring cylinder, 25.0 mL	2
Pasteur pipette	1
Rubber bulb for Pasteur pipette	1
Glass funnel, 7.5 cm diameter	2
Plastic funnel, 5.5 cm diameter	1
Filter paper in a zipped bag	3
Burette, 50.0 mL	1
Burette stand and clamp	1
O-ring with bosshead	2

Task 1B	a			b			c			Total
	a1	a2	a3	b1	b2	b3	c1	c2	c3	
Total	1	5	1	6	1	2	6	1	3	26
Score										

13% of Total Score

Task 1B: Calcium Iodate

Calcium iodate is an inorganic salt composed of calcium and iodate ions. $Ca(IO_3)_2$ is sparingly soluble in water. An equilibrium is established between the undissolved salt and a saturated solution of the salt:

$$Ca(IO_3)_{2\,(s)} \rightleftharpoons Ca^{2+}_{(aq)} + 2\,IO_3^-{}_{(aq)}$$

Titration data will be used to determine the concentration of iodate ions in a saturated solution of $Ca(IO_3)_2$ and then to determine the K_{sp} value for $Ca(IO_3)_2$.

The concentration of iodate ions will be determined by titration with a standard solution of sodium thiosulfate ($Na_2S_2O_3$), in the presence of potassium iodide (KI) and starch as the indicator.

Part a is associated with the standardization of $Na_2S_2O_3$. Part b is the determination of K_{sp} for $Ca(IO_3)_2$.

In Part c, solid $Ca(IO_3)_2$ is dissolved in an unknown dilute KIO_3 solution. After standing for 3 days, an equilibrium is also established between the undissolved salt and saturated solution of the salt. The concentration of iodate ion will be determined using the same titrimetric method, and then used to calculate the concentration of the dilute KIO_3 solution.

Part a

Standardization of $Na_2S_2O_3$

1. Fill the burette with the $Na_2S_2O_3$ solution.

2. Pipette 10.00 mL of the standard KIO_3 solution (provided as solution A, KIO_3: 10.7042 g in 5.00 L) into an Erlenmeyer flask. Add 10 mL of 10%(w/v) KI and 10 mL of 1 M HCl into the flask. The solution should turn dark brown as I_2 is formed.

3. Titrate with $Na_2S_2O_3$ solution until the solution has turned pale yellow. Add 2 mL of 0.1%(w/v) starch solution. The solution should turn dark blue. Titrate carefully to the endpoint (colourless). Record the volume of the $Na_2S_2O_3$ solution.

a1) Balance the relevant chemical equations:

..... $IO_3^-{}_{(aq)}$ + …… $I^-{}_{(aq)}$ + …... $H_3O^+{}_{(aq)}$ → …… $I_{2(aq)}$ + …… $H_2O_{(l)}$

……$I_{2(aq)}$ + …… $S_2O_3^{2-}{}_{(aq)}$ → …… $I^-{}_{(aq)}$ + …… $S_4O_6^{2-}{}_{(aq)}$

a2) Record the volume of the $Na_2S_2O_3$ solution:

(You do not need to fill in the entire table)

	Titration no.		
	1	2	3
Initial reading of the burette of $Na_2S_2O_3$ solution, mL			
Final reading of the burette of $Na_2S_2O_3$ solution, mL			
Consumed volume of $Na_2S_2O_3$ solution, mL			

Accepted volume, mL: V1 =

a3) Calculate the concentration of the $Na_2S_2O_3$ solution:

Concentration of $Na_2S_2O_3$ (M): _____ (answer in 4 digits after decimal point)

(If you cannot find the concentration of $Na_2S_2O_3$, use a concentration of 0.0700 M for further calculations.)

Part b

Determination of the K_{sp} of $Ca(IO_3)_2$

1. You are provided with the filtrate of the filtered saturated solution of $Ca(IO_3)_2$ (Solution B).

2. Pipette 5.00 mL of the filtrate (solution B) into an Erlenmeyer flask. Add 10 mL of 10% (w/v) KI and 10 mL of 1 M HCl into the flask.

3. Titrate with $Na_2S_2O_3$ solution until the solution has turned pale yellow. Add 2 mL 0.1% (w/v) starch solution. The solution should turn dark blue. Titrate carefully to the endpoint (colourless). Record the volume of $Na_2S_2O_3$ solution.

b1) Record the volume of the $Na_2S_2O_3$ solution.

(You do not need to fill in the entire table)

	Titration no.		
	1	2	3
Initial reading of the burette of $Na_2S_2O_3$ solution, mL			
Final reading of the burette of $Na_2S_2O_3$ solution, mL			
Consumed volume of $Na_2S_2O_3$ solution, mL			

Accepted volume, mL: V2 =

b2) Calculate the concentration of the IO_3^- in solution B.

Concentration of IO_3^- (M): _____ (answer in 4 digits after decimal point)

b3) Calculate value of K_{sp} for **$Ca(IO_3)_2$**:

K_{sp} for $Ca(IO_3)_2$: _____ (answer in 3 significant figures)

(If you cannot find the K_{sp} value, use the value of 7×10^{-7} for further calculations.)

Part c

Determination of the concentration of an unknown dilute KIO_3 solution

1. You are provided with the filtrate of the filtered saturated solution of $Ca(IO_3)_2$ dissolved in the unknown dilute KIO_3 (provided as solution C).

2. Pipette 5.00 mL of the filtrate (solution C) into an Erlenmeyer flask. Add 10 mL of 10% (w/v) KI and 10 mL of 1 M HCl into the flask.

3. Titrate with $Na_2S_2O_3$ solution until the solution has turned pale yellow. Add 2 mL 0.1% (w/v) starch solution. The solution should turn dark blue. Titrate carefully to the endpoint (colourless). Record the volume of $Na_2S_2O_3$ solution.

c1) Record the volume of the $Na_2S_2O_3$ solution:

(You do not need to fill in the entire table)

	Titration no.		
	1	2	3
Initial reading of the burette of $Na_2S_2O_3$ solution, mL			
Final reading of the burette of $Na_2S_2O_3$ solution, mL			
Consumed volume of $Na_2S_2O_3$ solution, mL			

Accepted volume, mL: V3 =

c2) Calculate the concentration of the IO_3^- in solution C:

Concentration of IO_3^- (M): _____ (answer in 4 digits after decimal point)

c3) Calculate the concentration of the unknown dilute KIO_3 sample:

Concentration of KIO_3 (M): _____ (answer in 4 digits after decimal point)

Practical Exam

Task 2

Chemicals and Equipment (Task 2):

I. Chemicals and materials

Chemicals	Labeled as	Health Statements [a]
3-Pentanone (MW 86.13), ~0.86 g[b] in a vial	**A**	H225-H319-H335-H336
4-Chlorobenzaldehyde (MW 140.57), ~3.5 g[c] in a vial	**B**	H302-H315-H319-H335
Ethanol, 200 mL in a wash-bottle	**Ethanol**	H225-H319
2 M **NaOH** solution in water (**labelled as 2N NaOH**), 25 mL in a bottle	**2N NaOH**	H290-H314

[a] **See page 34 for definition of Health Statements**
[b] You will need to weigh the vial containing 3-pentanone right before using. The exact value can be calculated based on the information given on the label.
[c] The exact value is indicated on the label.

II. Equipment and glassware

Shared equipment	Quantity
Balance	Shared 12 per room
Water aspirator	Shared 2 per bench
Foam bucket filled with ice	Shared 1 per row (an ice refill can be requested with no penalty)
Personal Equipment	**Quantity**
Hotplate stirrer with temperature probe	1
Stand	1
Clamps	2
100 mL round-bottomed flask	1
Measuring cylinder, 25 mL	1
Measuring cylinder, 50 mL	1
Air condenser	1
Crystallizing dish, 250 mL	1
125 mL Erlenmeyer flask	2
Suction flask, 250 mL	1
Buchner funnel, 25 mL	1
Watch glass	1
Pasteur pipettes (droppers)	5
Rubber bulbs	2
Suction rubber	1
Rubber support ring	1
Magnetic bar	1
Filter papers	3 (pack in 1 zipped bag)
Spatula	1
Stirring Rod	1
Forceps	1
Plastic joint clips	1
Wash Bottle (filled with ethanol)	1 (can be refilled)
Nitrile gloves	2 (exchange size if needed)
Towels	2
Paper clip	1
“Waste Task 2”, 500 mL glass bottle	1
Vial labeled “Student code” for submitting product.	1
Goggles	1

Task 2	A			b	Total
	a1	a2	a3	b1	
Total	2	2	2	18	24
Score					

14% of Total Score

Task 2: Elaborating a Carbon Framework

The core structure of organic molecules is mostly based on a carbon-carbon skeleton. Synthetic transformations to efficiently achieve carbon-carbon bond formation have long been of interest. In this experiment, you are required to react 4-chlorobenzaldehyde and 3-pentanone to form a product, and to deduce its structure.

Important Notes:

- Ethanol can be refilled with no penalty.
- All weighing processes require verification from the lab supervisor. The supervisor will need to sign on your answer sheet for grading. **No points will be given for unverified values.**
- A total of 18 points of this task score will be based on the quality and quantity of the product submitted. **No points will be awarded if the product is not submitted for grading.**
- ^{1}H NMR and melting point determination techniques will be used by the grader to verify the quality of your product.

Part a

1. Take the vial containing 3-pentanone (**A**) (Code Axxx, e.g.: A305) and remove the parafilm. Weigh the vial with the cap. Record the weight on the answer sheet (question a1).
2. Set up a water bath in a 250 mL crystallizing dish and heat to **55±2°C**. Add a paper clip to the water bath and let it stir.
3. Ensure a stir bar is in the 100 mL round-bottomed flask. Transfer the pre-weighed 3-pentanone (labeled as **A**) and 4-chlorobenzaldehyde (labeled as **B**) to the flask. Add 50 mL ethanol and swirl to dissolve the solid.
4. Add 15 mL of 2 M NaOH (labeled as 2N NaOH) to the reaction mixture. **Be careful not to wet the ground glass joint with the NaOH solution.**

5. Set up the reaction as shown in **Figure 1**. Place the reaction flask in the heated water bath. Attach the air condenser with the plastic joint clip. Heat the reaction mixture while stirring **for 30 minutes**.

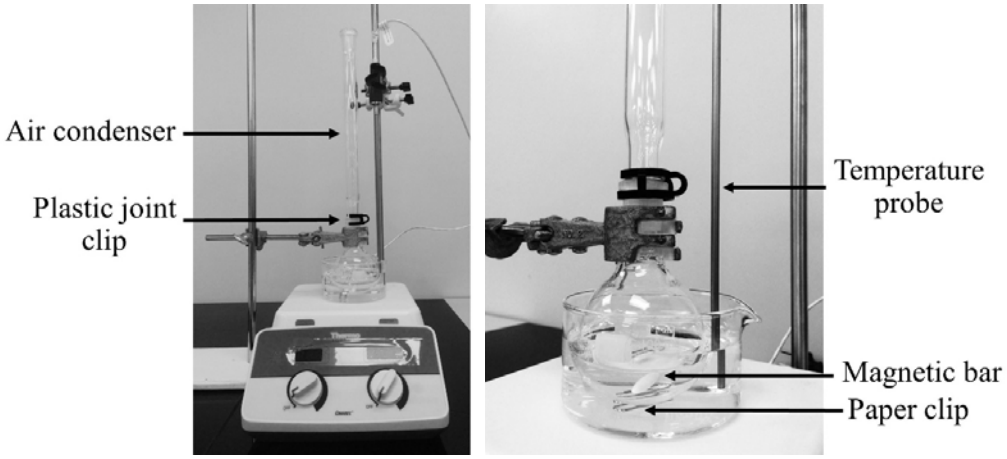


Figure 1: Set up needed for heating the reaction.

6. Remove the flask from the water bath. Place the flask on the rubber support ring.

7. **VERY IMPORTANT: detach the probe from the hotplate/stirrer at this point.** After you detach the probe, inform the supervisor to check it.

8. Place the reaction flask in an ice bath. A precipitate should form. (**suggestion:** if you do not observe any solid within 5 minutes, use a stirring rod to scratch the side of the flask).

9. Cool the flask for approximately 20 minutes to allow complete precipitation.

10. Set up the suction filtration equipment (**Figure 2**). Connect the suction flask to the water aspirator. Place a Buchner funnel fitted with a rubber adapter onto the suction flask. Place a wetted filter paper at the center of the funnel. Filter the precipitate and wash it with a small amount of **COLD** ethanol. Dry the precipitate on the funnel for 2-3 minutes.

Figure 2: Set up needed for suction filtration.

11. Remove the tubing from the suction flask (before turning off the water aspirator). Bring your equipment back to your space. Remove the precipitate from the funnel and transfer it to the Erlenmeyer flask. **Do not scrape the paper too hard as you may contaminate your product with filter paper**.

12. Place ethanol in a separate Erlenmeyer flask and heat it gently on a hotplate (you may set the temperature mark at 100-120°C).

    Recrystallize the product from the minimum amount of hot ethanol. After cooling to room temperature, be sure to place the flask into an ice bath to complete crystallization.

13. Collect the recrystallized product *via* suction filtration (see step 10 for suction filtration protocol) and wash the product with a small amount of **COLD** ethanol. Dry the precipitate on the funnel for 2-3 minutes. Air-dry the product on the benchtop for at least 15 minutes.

14. Weigh the vial labeled with your student code (without the cap). Record the value on the answer sheet (question a1).

15. Transfer the recrystallized product to the pre-weighed vial. Determine and record the mass of the purified product on the answer sheet (question a1).

16. Complete the information on the label of the product vial. Place the product-containing vial on the benchtop. **The supervisor will collect your vial and sign on your answer sheet** after the "Stop" command. **YOU MUST SIGN THE ANSWER SHEET (QUESTION B) FOR GRADING.** Once both signatures are collected, place the vial into the zipped bag and submit for grading.

    The following items should be left on your bench:

    - the exam/answer booklet (this booklet) placed in an envelope
    - the vial labeled "Student Code" with all information completed

Student Code | CAN-1

The supervisor will place a label here:

Axxx (For example: A567) =	Code of vial containing 3-pentanone
Tared (with cap):	Mass of (vial + label + cap) **before** adding 3-pentanone
Bxxx (For example: B567) =	Code of vial containing 4-chlorobenzaldehyde
Net:	Mass of 4-chlorobenzaldehyde

a1) Use the information provided in the label above along with your experimental data for your calculation. Write down all the results in this table:

Mass of 3-pentanone + the vial provided (must weigh with cap) = _____

*Signature of the supervisor is required for grading

Mass of 3-pentanone = _____

Mass of 4-chlorobenzaldehyde (copy from the label) = _____

Mass of empty vial for product (without cap) = _____

*Signature of the supervisor is required for grading

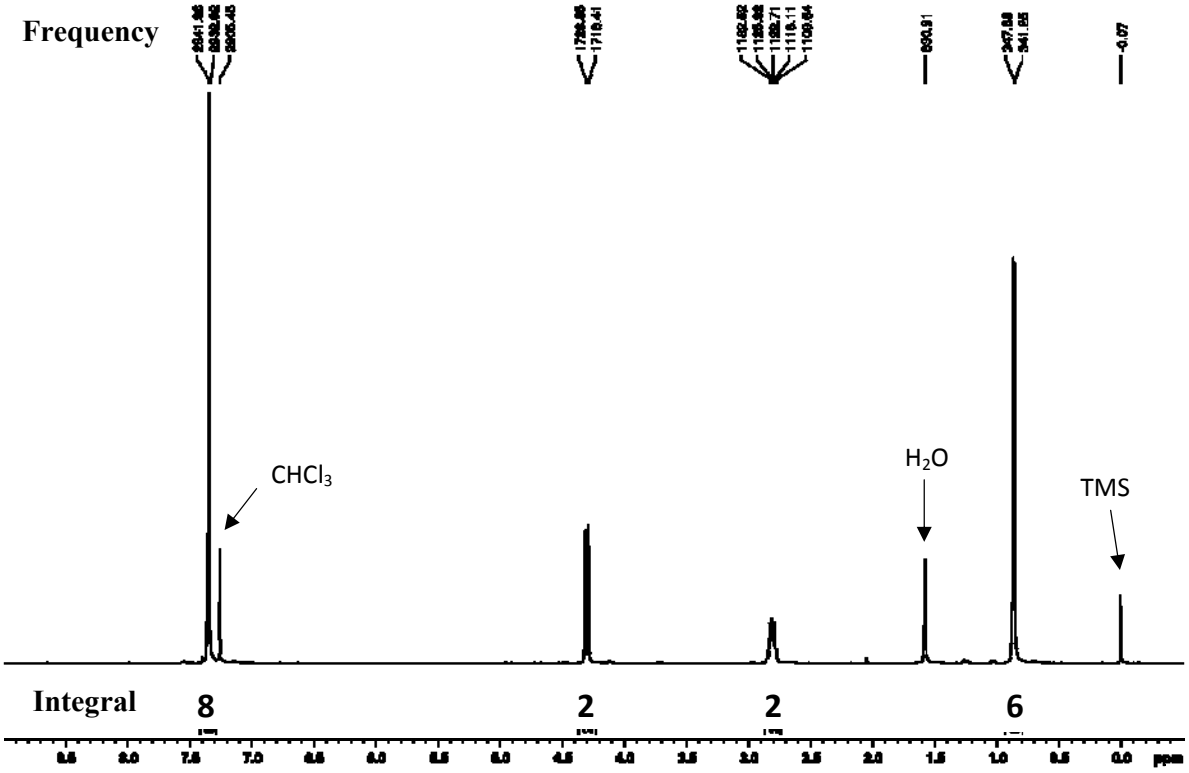
Mass of the vial (without cap) containing the recrystallized product = _____

*Signature of the supervisor is required for grading

Mass of the recrystallized product = _____

a2) Draw the structure of 4 possible aromatic compounds that may be formed in this reaction (not including stereoisomers):

a3) Given the 400MHz ^{1}H NMR (in $CDCl_3$) of the actual product formed, draw the structure of the product. Integrals are for all protons present in the molecule. **NOTE:** the absolute frequency values are NOT required to solve the structure.



Part b

b1) Your submitted product will be characterized and graded for its yield and purity. Provide information for the product you submitted:

Status: ☐ Solid ☐ Liquid

Signature of Supervisor: _____(signed when submitted)

Signature of Student: _____(signed when submitted)

Health Statements

H225	Highly flammable liquid and vapor
H272	May intensify fire; oxidizer
H290	Maybe corrosive to metals
H300	Fatal if swallowed
H301	Toxic if swallowed
H302	Harmful if swallowed
H314	Causes severe skin burns and eye damage
H315	Causes skin irritation
H319	Causes serious eye irritation
H330	Fatal if inhaled
H335	May cause respiratory irritation
H336	May cause drowsiness or dizziness
H371	May cause damage to organs

Characteristic ^{1}H NMR Chemical Shifts

Type of Hydrogen (R=Alkyl, Ar=Aryl)	Chemical Shift (ppm)	Type of Hydrogen (R=Alkyl, Ar=Aryl)	Chemical Shift (ppm)
$(CH_3)_4Si$	0 (by definition)		
R**H**$_3$	0.9	R**H**=O	9.5-10.1
R**H**$_2$R	1.2-1.4	RCOO**H'**	10-13
R$_3$C**H**	1.4-1.7	RCOC**H**$_3$	2.1-2.3
R**H**$_2$I	3.2-3.3	RCOC**H**$_2$R	2.2-2.6
R**H**$_2$Br	3.4-3.5	RCOOC**H**$_3$	3.7-3.9
R**H**$_2$Cl	3.6-3.8	RCOOC**H**$_2$R	4.1-4.7
R**H**$_2$F	4.4-4.5	R$_2$C=CRC**H**R$_2$	1.6-2.6
R**H**$_2$NH$_2$	2.3-2.9	R2C=C**H**$_2$	4.6-5.0
R**H**$_2$OH	3.4-4.0	R$_2$C=C**H**R	5.0-5.7
R**H**$_2$OR	3.3-4.0	RC≡C**H**	2.0-3.0
R**H**$_2$CH$_2$OR	1.5-1.6	Ar**H**$_3$	2.2-2.5
R$_2$N**H**	0.5-5.0	Ar**H**$_2$R	2.3-2.8
RO**H**	0.5-6.0	Ar**H**	6.5-8.5

Periodic table of elements

1 1A																		18 8A
1 **H** 1.008	2 2A												13 3A	14 4A	15 5A	16 6A	17 7A	2 **He** 4.003
3 **Li** 6.941	4 **Be** 9.012												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	3 3B	4 4B	5 5B	6 6B	7 7B	8 8B	9 8B	10 8B	11 1B	12 2B		13 **Al** 26.98	14 **Si** 28.09	15 **P** 30.97	16 **S** 32.07	17 **Cl** 35.45	18 **Ar** 39.95
19 **K** 39.10	20 **Ca** 40.08	21 **Sc** 44.96	22 **Ti** 47.88	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** 65.39		31 **Ga** 69.72	32 **Ge** 72.61	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** 101.1	45 **Rh** 102.9	46 **Pd** 106.4	47 **Ag** 107.9	48 **Cd** 112.4		49 **In** 114.8	50 **Sn** 118.7	51 **Sb** 121.3	52 **Te** 127.6	53 **I** 126.9	54 **Xe** 131.3
55 **Cs** 132.9	56 **Ba** 137.3	57 **La** 138.9	72 **Hf** 178.5	73 **Ta** 180.9	74 **W** 183.8	75 **Re** 186.2	76 **Os** 190.2	77 **Ir** 192.2	78 **Pt** 195.1	79 **Au** 197.0	80 **Hg** 200.6		81 **Tl** 204.4	82 **Pb** 207.2	83 **Bi** 209.0	84 **Po** (209)	85 **At** (210)	86 **Rn** (222)
87 **Fr** (223)	88 **Ra** (226)	89 **Ac** (227)	104 **Rf** (261)	105 **Db** (262)	106 **Sg** (263)	107 **Bh** (262)	108 **Hs** (265)	109 **Mt** (266)	110 **Ds** (281)	111 **Rg** (272)	112 **Cn** (285)		113 **Nh** (286)	114 **Fl** (289)	115 **Mc** (289)	116 **Lv** (293)	117 **Ts** (294)	118 **Og** (294)

58 **Ce** 140.1	59 **Pr** 140.9	60 **Nd** 144.2	61 **Pm** (145)	62 **Sm** 150.4	63 **Eu** 152.0	64 **Gd** 157.3	65 **Tb** 158.9	66 **Dy** 162.5	67 **Ho** 164.9	68 **Er** 167.3	69 **Tm** 168.9	70 **Yb** 173.0	71 **Lu** 175.0
90 **Th** 232.0	91 **Pa** 231.0	92 **U** 238.0	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** (262)