PRACTICAL PROBLEMS

PROBLEM 1P (Practical)

The oxidation of iodide by iron(III) ions – a kinetic study based on the thiosulfate clock reaction

Clock reactions are commonly used as demonstrations by chemical educators owing to their visual appeal. Oxidation of iodide by iron(III) ions in a weakly acidic medium is a reaction that can be transformed into a clock reaction. In the presence of thiosulfate and starch, chemical changes in this clock reaction can be presented by the following equations:

$Fe^{3+}(aq) + S_2O_3^{2-}(aq) \leftrightarrows [Fe(S_2O_3)]^+(aq)$ (1) fast

$2\ Fe^{3+}(aq) + 3\ I^-(aq) \rightarrow 2\ Fe^{2+}(aq) + I_3^-(aq)$ (2) slow

$I_3^-(aq) + S_2O_3^{2-}(aq) \rightarrow 3\ I^-(aq) + S_4O_6^{2-}(aq)$ (3) fast

$2\ I_3^-(aq) + \text{starch} \rightarrow \text{starch} - I_5^- + I^-(aq)$ (4) fast

Reaction (1) is a fast reversible equilibrium which occurs in the reaction mixture giving a reservoir of iron(III) and thiosulfate ions. After being produced in reaction (2), iodine in the form of triiodide ion (I_3^-), is immediately consumed by thiosulfate in reaction (3). Therefore, no iodine accumulates in the presence of thiosulfate. When thiosulfate is totally depleted, the triiodide ion accumulates and it may be detected by use of starch indicator according to reaction (4).

The kinetics of reaction (2) is easily investigated using the initial rates method. One has to measure the time elapsed between mixing the two solutions and the sudden color change.

For the oxidation of iodide by iron(III) ions (reaction 2), the reaction rate can be defined as:

$$v_0 = -\frac{d[Fe^{3+}]}{dt} \quad (5)$$

The initial reaction rate can then be approximated by:

$$v_0 = -\frac{\Delta[Fe^{3+}]}{\Delta t} \quad (6)$$

with $\Delta[Fe^{3+}]$ being the change in the concentration of iron(III) ions in the initial period of the reaction. If Δt is the time measured, then $\Delta[Fe^{3+}]$ is the change in iron(III) ion concentration from the moment of mixing to the moment of complete thiosulfate consumption (assume that the reaction rate does not depend on thiosulfate concentration). Therefore, from the reactions' stoichiometry it follows:

$$-\Delta[Fe^{3+}] = [S_2O_3^{2-}]_0 \quad (7)$$

and consequently:

$$v_0 = -\frac{[S_2O_3^{2-}]_0}{\Delta t} \quad (8)$$

The initial thiosulfate concentration is constant and significantly lower than that of iron(III) and iodide ions. The above expression enables us to determine the initial reaction rate by measuring the time required for the sudden color change to take place, Δt.

The rate of reaction is first order with respect to $[Fe^{3+}]$, and you will determine the order with respect to $[I^-]$. This means the initial reaction rate of reaction can be expressed as:

$$v_0 = k\,[Fe^{3+}]_0\,[I^-]_0^y \quad (9)$$

where k is the rate constant and y is the order with respect to $[I^-]$.

We assume that the reaction rate does not depend on the thiosulfate concentration, and that the reaction between Fe^{3+} and $S_2O_3^{2-}$ is negligible. You have to observe carefully the color changes during the clock reaction and to determine the reaction order with respect to $[I^-]$, and the rate constant of clock reaction.

Experimental part

Equipment

- 1 hotplate stirrer
- 1 magnetic stirring bar
- 1 plastic wash bottle filled with distilled water
- 1 glass beaker for inorganic waste liquid (ca. 1 dm^3)
- 1 conical flask for organic waste liquid, 250 cm^3
- 1 graduated pipette, 1 cm^3
- 1 graduated pipette, 5 cm^3

- 1 graduated pipette, 5 cm^3, labeled 'MeOH' (for Problem 2)
- 1 graduated pipette, 10 cm^3
- 1 volumetric pipette, 10 cm^3
- 1 graduated pipette, 25 cm^3
- 2 Pasteur pipettes and bulb
- 2 glass spatula spoon
- 1 cleaning brush
- 1 large glass stirring rod
- 1 glass funnel
- 1 bag of paper towels
- 1 digital thermometer
- 1 three-way pipette bulb with a little rubber adapter for bigger pipettes
- 1 ceramic Büchner funnel with fitted rubber bung
- 1 Büchner flask
- 1 digital stop watch
- 6 glass beakers (100-cm^3)

Chemicals

- KI, aqueous solution, c = 0.100 mol dm^{-3}, 120 cm^3
- solution **A1** in a glass bottle containing KI, $Na_2S_2O_3$, and starch indicator in distilled water, 40 cm^3
- solution **B1** in a glass bottle containing $Fe(NO_3)_3$, HNO_3 in distilled water, 40 cm^3
- solution **A2-1** in a glass bottle containing $Na_2S_2O_3$ ($c = 5.883\times10^{-4}$ mol dm^{-3}), KNO_3 and starch indicator in distilled water, 360 cm^3
- solution **B2** in a glass bottle containing $Fe(NO_3)_3$ (c = 0.1020 mol dm^{-3}) and HNO_3 in distilled water, 100 cm^3
- distilled water, 1 dm^3

P r o c e d u r e

Solution **A** (containing $Na_2S_2O_3$, KI, KNO_3 and starch) is first placed in the beaker and is stirred using the magnetic bar. The rate of stirring is set at level 8 as indicated in

Figure 1. Solution **B** (containing $Fe(NO_3)_3$ and HNO_3) is quickly added into solution **A** and the stopwatch is simultaneously started. The time is recorded at the moment the solution suddenly turns dark blue. The temperature of the solution is recorded using the digital thermometer.

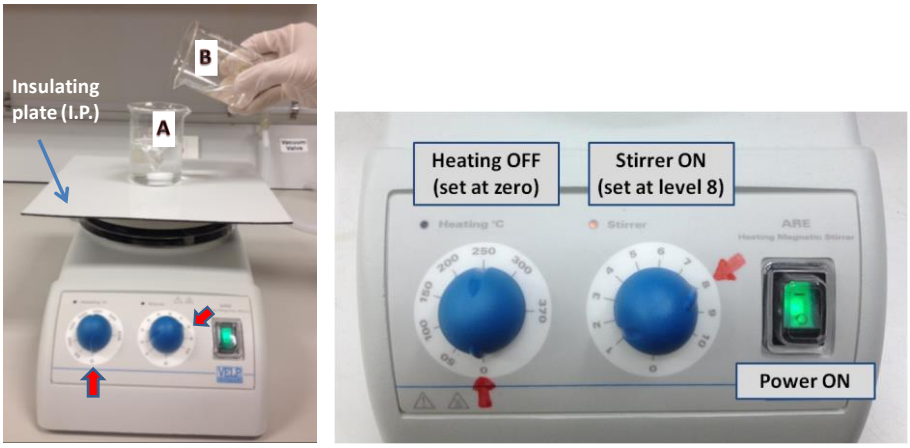


Figure 1. The apparatus employed for kinetic study of the clock reaction.

1. Practice run to observe the color changes

There is no need to measure accurately the volumes used in this part – just use the marks on the beaker as a guide.

- Pour ca. 20 cm^3 of solution **A1** (containing KI, $Na_2S_2O_3$, and starch in water) to a 100 cm^3 graduated beaker containing a magnetic stirrer bar. Place the beaker on top of the insulating plate on the magnetic stirrer.
- Pour ca. 20 cm^3 of solution **B1** (containing $Fe(NO_3)_3$ and HNO_3 in water) in another 100 cm^3 graduated beaker.
- Quickly pour the solution **B1** into solution **A1** and start stopwatch simultaneously. Stop stopwatch when the color of the mixture changes. There is no need to record this time. Answer the following questions.

1.1 Write down the molecular formula of the limiting reactant for the given clock reaction.

1.2 What are the ions or compounds responsible for the colors observed in this experiment? Tick the appropriate box.

Color	Compound
Purple	☐ Fe^{3+} ☐ $[Fe(S_2O_3)]^+$ ☐ Fe^{2+} ☐ starch-I_5^- ☐ I_3^-
Dark blue	☐ Fe^{3+} ☐ $[Fe(S_2O_3)]^+$ ☐ Fe^{2+} ☐ starch-I_5^- ☐ I_3^-

2. Determination of the order with respect to $[I^-]$ (y), and the rate constant (k)

In this section, Δt is determined for different initial concentrations of KI according to the table below. The experiment is repeated as necessary for each concentration of KI.

Hint: Use 25 cm^3 graduated pipette for solution **A2-1**, 10 cm^3 graduated pipette for KI, 5 cm^3 graduated pipette for solution **B2**, and one of the burettes for water (you will need to refill the burette from the wash bottle for each measurement).

- Prepare 55 cm^3 of solution **A2** in a 100 cm^3 beaker containing a magnetic stirrer bar and place it on top of the insulating plate on the stirrer. Solution **A2** contains solution **A2-1**, KI, and distilled water (see the table below for the volume of each component).
- Add 5 cm^3 of solution **B2** in another 100 cm^3 beaker.

Quickly pour prepared solution **B2** into solution **A2**. Determine the time (Δt) necessary for the color change by a stopwatch. The temperature of the solution is recorded.

1.3 Record the time (Δt) for each run in the table below. (You DO NOT need to fill all three columns for the runs.) For each concentration of KI, record your accepted reaction time ($\Delta t_{accepted}$) and temperature. You will be only graded on your values of $\Delta t_{accepted}$ and $T_{accepted}$.

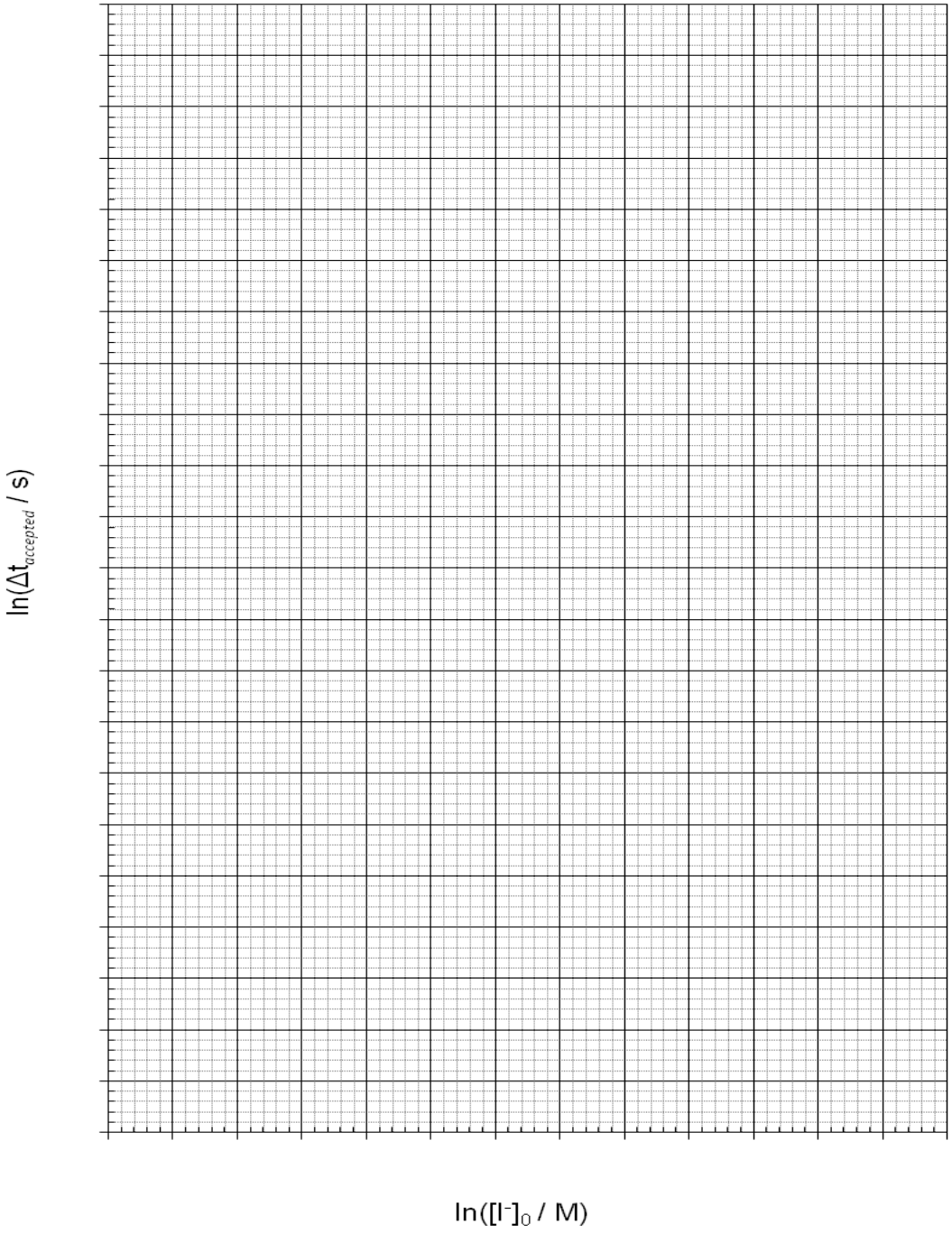
No	**55** cm^3 **of solution A2**									$\Delta t_{accepted}$ (s)	$T_{accepted}$ (ºC)
	A2-1 (cm^3)	H_2O (cm^3)	0.100 mol dm^{-3} KI(cm^3)	Run 1		Run 2		Run 3			
				Δt (s)	T (ºC)	Δt (s)	T (ºC)	Δt (s)	T (ºC)		
1	20.4	31.6	3.0								
2	20.4	30.1	4.5								
3	20.4	28.6	6.0								
4	20.4	27.4	7.2								
5	20.4	25.6	9.0								

When you are satisfied you have all the necessary data for Problem 1. Before continuing with the analysis, it is strongly recommended that you start the practical procedure for Problem 2 since there is a reaction time of one hour in that Problem.

1.4 Fill in the table below and plot the results in the graph.

Hint: Make sure your data is graphed as large as possible in the provided space.

No.	1	2	3	4	5
$\ln([I^-]_0$ / M)	– 5.30	– 4.89	– 4.61	– 4.42	– 4.20
$\Delta t_{accepted}$ (s)					
$\ln(\Delta t_{accepted}$ /s)					



1.5 Draw the best fit line on your graph and use it to determine the order with respect to $[I^-]$ (y).

y = ...

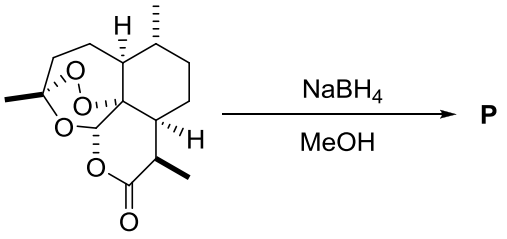
1.6 Complete the table below and calculate k for each of the concentrations of iodide. Report your accepted value for the rate constant, giving the appropriate unit. Remember that the order with respect to $[Fe^{3+}]$ is equal to one.

No	$\Delta t_{accepted}$ (s)	$[Fe^{3+}]_0 \times 10^{-3}$ (mol dm^{-3})	$[I^-]_0 \times 10^{-3}$ (mol dm^{-3})	$[S_2O_3^{2-}]_0 \times 10^{-3}$ (mol dm^{-3})	k
1			5.0		
2			7.5		
3			10.0		
4			12.0		
5			15.0		
$k_{accepted}$ = ...					

PROBLEM 2P (Practical)

Synthesis of a derivative of artemisinin

Artemisinin (also known as Quinghaosu) is an antimalarial drug isolated from the yellow flower herb *Artemisia annua* L., in Vietnam. This drug is highly efficacious against the chloroquine-resistant *Plasmodium falciparum*. However, artemisinin has a poor solubility in both oil and water so that one needs to prepare its new derivatives to improve the applicability of this drug. The reduction of artemisinin is an attractive method to synthesize new derivatives of artemisinin as shown in Scheme 1.



artemisinin
Molecular Weight: 282.3

Scheme 1

In this practical exam you are going to reduce artemisinin to product **P** and check its purity using Thin-Layer Chromatography (TLC).

Experimental part

Glass and equipment

- 1 hotplate stirrer, 1 magnetic stirring bar, 1 plastic wash bottle filled with distilled water, 1 glass beaker for inorganic waste liquid (ca. 1 dm^3), 1 conical flask for organic waste liquid, 250 cm^3, 1 graduated pipette (1 cm^3), 1 graduated pipette (5 cm^3), 1 graduated pipette (5 cm^3, labeled 'MeOH'), 1 graduated pipette (10 cm^3), 1 volumetric pipette (10 cm^3), 1 graduated pipette (25 cm^3), 2 Pasteur pipettes and bulbs, 2 glass spatula spoons, 1 cleaning brush, 1 large glass stirring rod, 1 glass funnel, 1 bag of paper towels, 1 digital thermometer, 1 three-way pipette bulb with a little rubber adapter for bigger pipettes, 1 ceramic Büchner funnel with fitted rubber bung, 1 Büchner flask;
- 1 graduated measuring cylinder, 5 cm^3
- 2 graduated measuring cylinders, 50 cm^3
- two-neck round bottom flask with plastic stopper (in ice bath), 100 cm^3

- conical (Erlenmeyer) flask, 100 cm^3
- hair dryer
- Petri dish with cover containing 1 TLC plate, 2 capillaries in paper holder
- Plastic pot for ice bath
- stand & clamp
- TLC developing chamber with glass lid
- 2 tweezers
- metal spatula
- 2 very small test tubes for TLC in container,
- zipper store bag (containing cotton wool, round filter paper, watch glass
- Petri dish with cover

Chemicals

- Artemisinin, 1.000 g,
- Sodium borohydride, $NaBH_4$, 0.53 g
- CH_3OH, 20 cm^3
- *n*-Hexane, 30 cm^3
- cerium staining reagent for TLC, 3-5 cm^3
- Ceri reagent
- CH_3COOH, 1 cm^3, in vial
- Ethyl acetate, 5 cm^3
- Bag of NaCl for salt (ice) bath
- $CaCl_2$ in drying tube, 5-10 g

Experimental Set-up

- The experimental set-up is shown in Figure 2.1.
- By moving the finger clamp, you can adjust the position of the two-neck round-bottom flask

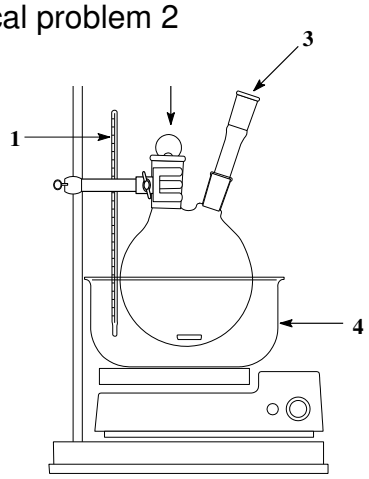
Procedure

Step 1. Synthesis of a Derivative of Artemisinin

1. Prepare an ice bath with a temperature between –20 and –15 ^oC by mixing ice and sodium chloride in the plastic pot (approximate ratio of NaCl : crushed ice = 1 scoop :

3 scoops). Use the digital thermometer to monitor the temperature. Place the bath on the magnetic stirrer. Put a layer of three tissues between the bath and the stirrer.

Fig. 2.1
Apparatus for practical problem 2



1: digital thermometer;
2: plastic stopper;
3: $CaCl_2$ drying tube;
4: ice bath

2. Place a magnetic stirring bar into the dry round-bottom flask and set up the reaction system onto the clamp-stand so that the system is immersed in the ice bath. Monitor the temperature using the digital thermometer.
3. *Setting aside a tiny amount* (ca. 2 mg) of artemisinin for TLC analysis, open the stopper and add the 1 gram of artemisinin through the bigger neck.
4. Use the glass funnel to add 15 cm^3 of methanol (measured using the 50-cm^3 graduated cylinder). Close the stopper and turn on the magnetic stirrer. (Set the magnetic stirrer to level 4). Start the stopwatch to keep track of the time.
5. After *ca.* 5 min stirring, open the stopper and add carefully 0.53 g of $NaBH_4$ in small portions over 15 min using a spatula. Close the stopper in between addition. *(Caution: Adding $NaBH_4$ rapidly causes side-reactions and overflowing).* Keep stirring for 50 min. Maintain the temperature of the ice bath below –5 ^oC; remove some of the liquid and add more NaCl-crushed ice mixture if necessary. Cool the vial containing 1 cm^3 of acetic acid in the ice bath.
7. Prepare 50 cm^3 of ice-cold distilled water *(cooled in the ice bath)* in the 100 cm^3-conical flask. Measure *ca.* 20 – 22 cm^3 *n*-hexane in the 50 cm^3 measuring cylinder and cool it in the ice bath. After the reaction is complete, keep the reaction flask in the ice bath below 0 ^oC. Remove the $CaCl_2$ tube, open the stopper, and add gradually *ca* 0.5

cm^3 of the cold acetic acid from the vial into the reaction flask until the pH is between 6 and 7. (Use the glass rod to spot the reaction mixture on to the pH paper.) With stirring, slowly add the 50 cm^3 of ice cold water over 2 min. A white solid precipitates in the reaction flask.

8. Assemble the vacuum filtration apparatus. Put a filter paper onto the Büchner funnel, wet the filter paper with distilled water and open the vacuum valve. Transfer the reaction mixture on to the filter and remove the stirring bar from the reaction flask using the spatula. Wash the product three times with portions of 10 cm^3 ice-cold water *(cooled in the ice bath).* Wash the product two times with portions of 10 cm^3 ice-cold *n*-hexane *(cooled in the ice bath).* Continue to use the pump to dry the solid on the filter. After *ca.* 5 min, carefully transfer the dried powder on to the watch glass labeled with your code and put into the labeled Petri dish. Turn off the vacuum valve when you do not use it!

   Note: Your sample will be collected, dried and weighed later by the lab assistant.

Task 2.1 The recording of your yield – will be performed after the exam by the lab assistant.

Step 2. TLC Analysis of the product

1. Check your TLC plate before use. Unused damaged plates will be replaced upon request without penalty. Use the pencil to draw the start front line, and the line where the solvent front will be run to **exactly as shown in Figure 2.2**. Write your student code on the top of the TLC plate in pencil.

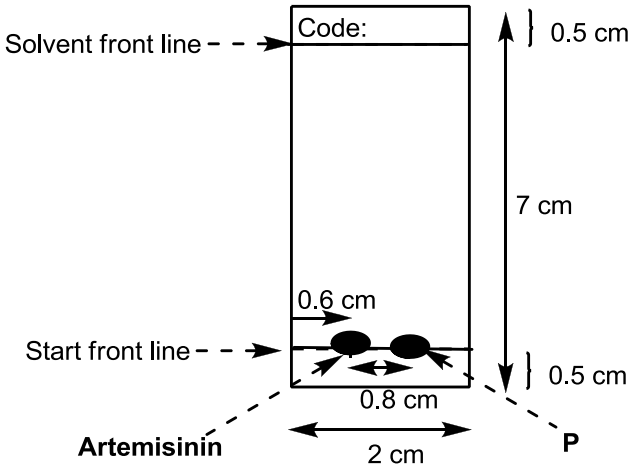


Figure 2.2. Instruction of TLC plate preparation

2. Dissolve *ca.* 1 mg of artemisinin *(a spatula tip)* in *ca.* 0.5 cm^3 of methanol in the labeled very small test tube (use the labeled 5 cm^3 graduated pipette). Dissolve *ca.* 1 mg of the product in *ca.* 1 cm^3 of methanol in the labeled test tube.
3. Spot the artemisinin solution and the product solution on the TLC plate using two different glass capillary spotters so the finished plate is as shown in Figure 2.2.
4. Prepare the TLC developing chamber. Use the 5 cm^3 graduated cylinder to make 5 cm^3 of a mixture of *n*-hexane/ethyl acetate (7/3, v/v) as the solvent system. Pour tme mixture of *n*-hexane/ethyl acetate into the chamber *(Note: The solvent level should not reach the spots on the plate if prepared as shown).* Cover and swirl the chamber and allow it to stand for 2 min.

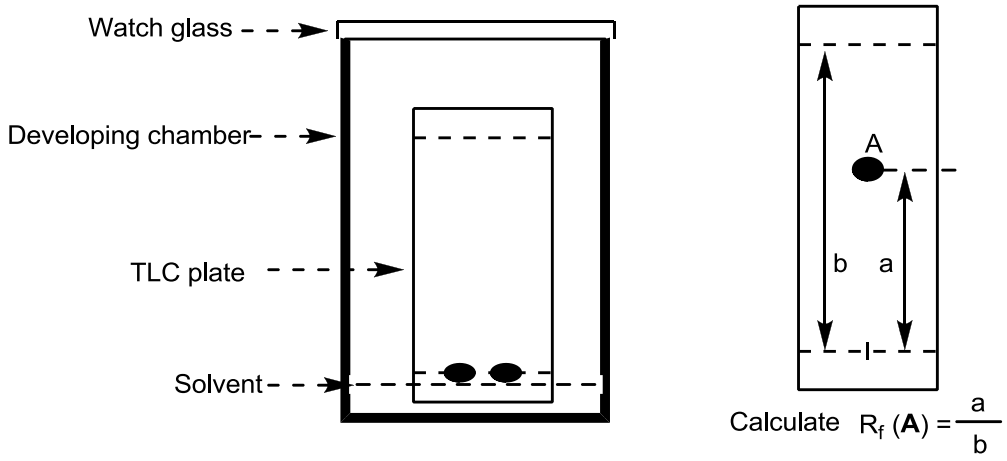


Figure 2.3. A TLC plate placed in the TLC developing chamber and instruction for R_f calculation of compound **A**

5. Insert the TLC plate upright into the TLC developing chamber. Wait until the solvent system reaches the pre-drawn solvent front line. (*Note: You are advised to work on some question below while you wait for the TLC to run.*)
6. When the solvent front reaches the line, remove the TLC plate using the tweezers and then dry the solvent using the hair dryer set at level 1.
7. Dip the piece of cotton wool into the cerium staining reagent, taking care not to let the tweezers come into contact with the solution since the metal stains the plate. Carefully apply the stain to the whole TLC plate.
8. Heat the TLC plate using the hair dryer set at level 2 (*Attention: DO NOT set the hair dryer to COLD*) until the blue spots of artemisinin and the product appear on the TLC plate.
9. Ask the lab assistant to take a photo of your final TLC plate together with your student code.
10. Circle all the visualized spots and calculate the R_f values of both artemisinin and the product (*See instruction in Fig. 2.3*). Store your TLC plate in the Petri dish.

Task 2.2 Fill the values of R_f in Table below.

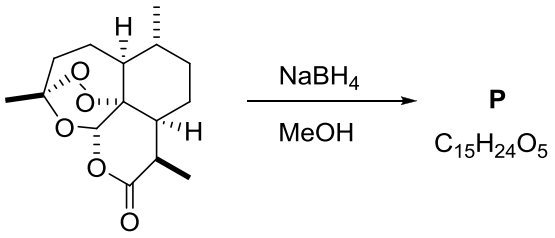
$R_{f,\ \text{Artemisinin}}$	$R_{f,\ \text{Product}}$	$R_{f\ Artemisinin}$ / $R_{f\ Product}$
----------	----------	----------

Task 2.*3* Check the total number of developed spots on the TLC plate:

1 ☐	2 ☐	3 ☐	4 ☐	5 ☐

Step 3. Identifying the reaction product P

The reduction of artemisinin leads to the formation of two stereoisomers (**P**). Comparing the ^{1}H-NMR spectrum (in $CDCl_3$) of one of these isomers with the spectrum of artemisinin shows an extra signal at δ_H = 5.29 ppm as a doublet, and also an extra signal as a broad singlet at δ_H = 2.82 ppm.



artemisinin
$C_{15}H_{22}O_5$

Task 2.4 Suggest the structure for P.
(You do not need to draw the stereochemistry of the compounds).

Task 2.5 P is mixture of two stereoisomers. What is their stereochemical relationship? Tick the appropriate box below.

Z/E Isomers	Enantiomers	Diastereomers	Constitutional Isomers
☐	☐	☐	☐

PROBLEM 3P (Practical)

Analysis of a hydrated zinc iron(II) oxalate double salt

Zinc iron(II) oxalate double salt is a common precursor in the synthesis of zinc ferrite which is widely used in many types of electronic devices due to its interesting magnetic properties. However, such double salts may exist with different compositions and different amount of water depending on how the sample was synthesized.

You will analyze a pure sample of hydrated zinc iron(II) oxalate double salt (**Z**) in order to determine its empirical formula.

Reagents

- ~ 30 % H_2SO_4, solution in water, 40 cm^3, in a plastic bottle,
- $KMnO_4$,, aqueous solution , $c = 1.00\times10^{-2}$ mol dm^{-3}, in 50 cm^3 bottle,
- EDTA, aqueous solution, $c = 2.00\times10^{-3}$ mol dm^{-3}, 40 cm^3 in a bottle,
- Buffer aqueous solution, $NH_4Cl + NH_3$, $pH = 9 – 10$, 40 cm^3 in a bottle
- NaOH, 20 % aqueous solution, 20 cm^3 in a plastic bottle,
- H_3PO_4, solution in water, c = ~3 mol dm^{-3}, 15 cm^3 in a bottle
- Indicator: ETOO, solid in KCl , *ca.* 0.5 g in a plastic bottle

Chemical glass and equipment

- 2 glass beakers, 50-cm^3 (for transferring EDTA and $KMnO_4$ solutions to burettes)
- 1 burette with BLUE graduation marks, 25-cm^3
- 1 burette with BROWN graduation marks, 25-cm^3
- 2 glass beakers, 250 cm^3
- 2 Erlenmeyer flasks, 250 cm^3
- 2 volumetric flasks with stopper, 100 cm^3
- 1 glass graduated measuring cylinder, 10 cm^3
- 1 glass graduated measuring cylinder, 100 cm^3
- burette stand & clamp
- pH paper
- round filter paper for the glass funnel

Procedure

The concentration of the standard $KMnO_4$ is posted on the lab walls.

Bring a clean 250 cm^3 beaker to the lab assistant who will be waiting by the balance. You will receive a pure sample of Z for analysis. Accurately weigh between 0.7 – 0.8 g of the pure sample Z onto the weighing paper (*m*, grams). This should then be immediately quantitatively transferred into your 250 cm^3 beaker for analysis, and its mass recorded in table below.

Task 3.1 Record the mass of the sample of pure Z taken.

Mass of sample, *m* (gram)	Lab assistant's signature
----------	----------

Analysis of Z

- Using the 100 cm^3 graduated measuring cylinder, measure *ca.* 30 cm^3 of 30 % H_2SO_4 solution and add it into the 250 cm^3 beaker containing your accurately weighed pure sample of **Z**. To speed up the dissolving of your sample you may use the hotplate stirrer to warm up the mixture, **but be careful not to boil** it. *You should not use the digital thermometer as the acid may damage it*. After the solid has dissolved, remove the beaker from the hotplate stirrer and cool it to close to room temperature. After the solution has cooled, quantitatively transfer it into the 100 cm^3 volumetric flask. Add distilled water up to the 100 cm^3- mark. We will now call this solution **C.**
- Use an appropriately labeled beaker to transfer the standardized $KMnO_4$ solution into the burette graduated with **brown** marks.
- Use another appropriately labeled beaker to transfer the standardize **EDTA** solution into the burette graduated with **blue** marks.

Titration with the $KMnO_4$ solution

a) sing the 5 cm^3 graduated pipette add 5.00 cm^3 of the solution **C** into a 250 cm^3 conical flask.

b) To this conical flask add about 2 cm^3 of 30 % H_2SO_4 solution, about 3 cm^3 of H_3PO_4

solution (c = 3.0 mol dm^{-3}), and about 10 cm^3 of distilled water. Heat the mixture on the hot plate stirrer until hot, **but be careful not to boil** it.

c) Titrate the hot solution with the standardized $KMnO_4$ solution, recording your burette readings in the table below. At the end point of the titration, the pink color of the solution appears. Repeat the titration as desired and report your accepted volume of $KMnO_4$ solution consumed (V_1, cm^3) in the table.

Task 3.2 Record volumes of standardized $KMnO_4$ solution consumed

(You DO NOT need to fill in the entire table)

	Titration No			
	1	2	3	4
Initial reading of the burette of $KMnO_4$, cm^3				
Final reading of the burette of $KMnO_4$, cm^3				
Consumed volume of $KMnO_4$, cm^3				

Accepted volume, V_1 = _____ cm^3

Task 3.3 Can aqueous HCl or HNO_3 be used instead of H_2SO_4 for the dissolving of sample Z and the subsequent analyses?

HCl:	Yes ☐	No ☐
HNO_3:	Yes ☐	No ☐

Titration with EDTA

i) Clean both the 250 cm^3 beakers ready for the next part of the experiment. Pipette 10.00 cm^3 of solution **C** into a 250 cm^3 beaker. Heat and stir the solution on the hotplate stirrer, but be careful not to boil it. Add *ca.* 15 cm^3 of 20 % NaOH solution to the beaker and keep it on the hotplate for *ca. 3-5* min in order to complete the precipitation of iron hydroxide, and to convert all Zn^{2+} ions into the ionic complex $[Zn(OH)_4]^{2-}$.

ii) Using a glass funnel and the large quantitative filter paper, filter the hot suspension directly into the 250 cm^3 conical flask. From this point take care with the volumes as you will be preparing a standard solution of exactly 100 cm^3 from the filtrate. As it is filtering, prepare some warm distilled water in a 250 cm^3 beaker (*ca.* 50 cm^3). Wash the precipitate on the filter paper (at least 5 times) with small portions (*ca.* 5 cm^3) of the warm distilled water. Cool the filtrate down and then quantitatively transfer it into the 100 cm^3 volumetric flask via a glass funnel. Add distilled water to make up to the 100 cm^3 mark. This will now be referred to as solution D.

iii) Pipette 10.00 cm^3 of solution **D** into a 250 cm^3 conical flask. Add *ca.*10 cm^3 ammonia buffer solution (*pH* = 9 – 10) and a small quantity of the ETOO indicator using the glass spatula spoon. Mix well to obtain a purple solution. Titrate the solution with the standardized EDTA solution ($c = 2.00\times10^{-3}$ mol dm^{-3}), recording your burette readings in table below. At the end point, the color of the solution turns blue. Repeat the titration as desired and report your accepted volume of EDTA solution consumed (V_2, cm^3) in the table.

Task 3.4 Record the volumes of EDTA solution consumed

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

	Titration No			
	1	2	3	4
Initial reading of the volume of EDTA, cm^3				
Final reading of the volume of EDTA, cm^3				
Consumed volume of EDTA, cm^3				

Accepted volume, V_2 = _____ cm^3

Determination of the empirical formula of Z

Task 3.5: Calculate the amount of substance (mol) of Zn^{2+} present in 100 cm^3 of solution C.

Task 3.6: Give the ionic equations for the reduction-oxidation reactions taking place in the titration with $KMnO_4$.

Task 3.7: Calculate the amount o substance (in mol) of Fe^{2+} present in 100 cm^3 of solution C.

Task 3.8: Calculate the amount o substance (in mol) of $C_2O_4^{2-}$ present in 100 cm^3 of solution C.

Task 3.9: Calculate the amount o substance (in mol) of water in tme original sample of Z in tme original sample of Z taken for analysis.

Task 10: Give tme empirical formula of Z.