Attention !

At all times while you are in the laboratory you must wear safety eye glasses or your own glasses if they have been approved, and use the pipette filler bulb provided. You will receive only **ONE WARNING** from the laboratory supervisor if you remove your glasses or fill a pipette by mouth.

A second infringement will be considered a major fault incompatible with further experimental work, and you will be dismissed from the laboratory with a resultant zero score for the entire experimental examination.

Do not hesitate to ask a demonstrator if you have any questions concerning safety issues.

- Please carefully read the text of each experimental task and **study the layout of the answer forms** before you begin your experimental work.

- Write your name and student code (posted at your workstation) on each answer sheet.

- You have 5 hours to complete all of the experimental tasks, and record your results on the answer sheets. In some steps, you have to ask for the demonstrator signature before proceeding to further step. You must stop your work immediately after the **STOP** command is given. A delay in doing this by 3 minutes will lead to cancellation of the current task and will result in zero points for that task.

- All results must be written in the appropriate areas on the answer sheets. Anything written elsewhere will not be marked. Do not write anything on the back of your answer sheets. If you need additional sheets or a replacement answer sheet, request it from the supervisor.

- When you have finished the examination, you must put all papers into the envelope provided, then you must seal the envelope and hand in to your demonstrator with your signature. Only papers in the sealed envelope will be marked.

- Do not leave the examination room until you are directed to do so. A receipt for your sealed envelope will be issued to you as you leave.

- Use only the pen and calculator provided.

- Use only the distilled water, and use the appropriate waste containers for disposal of chemicals and other waste materials.

- The number of significant figures in numerical answers must conform to the rules of evaluation of experimental errors. The inability to perform calculations correctly will result in penalty points, even if your experimental technique is flawless.

- This practical examination contains 2 envelopes. The first envelopes has 8 pages of Task 1 & 2 and 8 pages of answer sheet. The second envelope has 2 pages of answer sheet and 2 pages of spectra.

- Chemicals and/or laboratory wares can be requested if used up or broken. The penalty of each request will be the loss of 1 point.

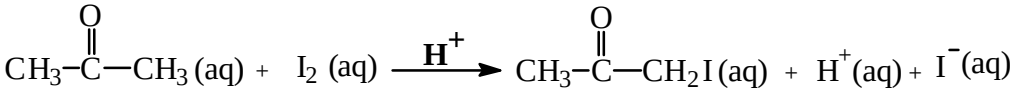
- The official English version of this examination is available for clarification only on request.

Do not start Laboratory Task 2 until you have finished Laboratory Task 1. The experimental part of Laboratory Task 1 can be completed in approximately 1.5 hours (calculation time not included).

A Kinetic Study of the Acid Catalyzed Reaction Between Acetone and Iodine in Aqueous Solution

INTRODUCTION

The reaction between acetone and iodine in aqueous solution is catalyzed by H^+.



In this experiment, the kinetics of the iodination is measured to determine the rate law of the reaction. The rate equation for the loss of I_2(aq) has been shown to have the form

$$\text{Rate} = -\frac{d[I_2]}{dt} = k[CH_3COCH_3]^x[I_2]^y[H^+]^z$$

where H^+ ions are the catalyst.

In order to determine the rate constant k and the kinetic orders x, y and z, the initial rate of reaction is measured.

$$\text{Initial rate} = k[CH_3COCH_3]_0^x[I_2]_0^y[H^+]_0^z$$

Where $[\ ]_0$ are the initial concentrations of acetone, I_2 and H^+, respectively.

If the initial rates are measured for various initial concentrations of the reactants then the order with respect to each reactant can be obtained.

The initial rate is obtained by measuring the decrease in the I_2(aq) concentration after a short time interval (7.0 min. in this experiment) after the start of the reaction. Aqueous sodium acetate solution is added to stop the reaction after 7 minutes. The acetate ion reacts immediately with the H^+ to produce acetic acid and so reducing the concentration of H^+. The reaction is thus stopped as there is no catalyst present.

Since the reaction does not come to a complete halt, the solution **should be titrated immediately after the addition of the sodium acetate solution.**

The remaining iodine I_2 (aq) is determined by titration with sodium thiosulfate, $Na_2S_2O_3$. As the end point of the titration is approached, starch indicator is added and the titration is continued until the blue color disappears.

Equipments

1.	Glass stoppered flask 250 mL	5
2.	Erlenmeyer flask 125 mL	3
3.	Burette 25 mL	1
4.	Pipette 5 mL	4
5.	Pipette 10 mL	3
6.	Pipette filler bulb with tip	1
7.	Beaker 100 mL	1
8.	Beaker 50 mL	3
9.	Beaker 250 mL (labeled waste disposal)	1
10.	Graduated cylinder 10 mL	1
11.	Wash bottle 500 mL	1
12.	Stop-watch	1
13.	Pen	1
14.	Label sheet	1

Chemicals

1.	Aqueous iodine solution in 0.4 M KI	80 mL
2.	0.100 M aq. HCl	50 mL
3.	0.50 M aq. CH_3COONa	80 mL
4.	Standard 0.02xxx M $Na_2S_2O_3$(aq) solution (the exact concentration will be announced at the beginning of Task 1)	200 mL
5.	Aqueous acetone (50% by volume) (density of pure acetone; 0.787 g/mL, MW. = 58.08)	50 mL
6.	Starch indicator	7 mL

Stop-watch Operation

A = Mode button (right bottom)
B = Start/Stop button (right top)
C = Split/Reset button (left top)

Mode is already set. **Do not touch the button A.**

1. Check that the display is 0.00$_{00}$. If not, call demonstrator.
2. To start, press **B.**
3. To stop, press **B**.
4. To reset, press **C**.

Procedure

A. Standardization of Iodine Solution

1. Pipet 5.00 mL of aqueous iodine into a clean 125 mL Erlenmeyer flask.
2. Add 10 mL of distilled water using graduated cylinder.
3. Titrate the iodine with the standard 0.02xxx M sodium thiosulfate solution until the colour of the solution is pale yellow.
4. Add 3 - 4 drops of starch indicator and continue the titration until the blue colour disappears.
5. Record the initial and the final volumes of the thiosulfate solution and the volume used in the answer sheet.
6. Repeat the titration as necessary (Steps 1 to 5)
7. Give the titre volume for calculation in the answer sheet.
8. Calculate the iodine concentration.

B. A kinetic study of acid catalyzed reaction between acetone and iodine in aqueous solution

1. Label the stoppered flasks as follows: Flask I, II, III and IV.
2. To each respective flask add the following volumes of distilled water, 0.100 M hydrochloric acid and 50% acetone:
   Stopper each flask immediately after addition of the solutions.

Flask No.	Volume (mL)		
	water	0.100 M HCl	50%acetone
I	5.00	5.00	5.00
II	0.0	5.00	5.00
III	0.0	5.00	10.00
IV	0.0	10.00	5.00

3. Measure out 10 mL of 0.50 M aq. CH_3COONa into the graduated cylinder.
4. Set the stop-watch to 0.00$_{00}$ display.
5. Pipet **5.00 mL** of iodine solution into the stoppered **Flask No. I**.
   Start the stop-watch as soon as the **first drop of iodine solution is added**.
6. Stopper the flask and swirl continuously.
7. Just before 7.0 min, remove the stopper, at **7.0 min**, immediately pour 10 mL of sodium acetate
   solution (from step 3) into the reaction flask. **Shake well.**
8. Titrate the remaining iodine with standard thiosulfate solution.
9. Record the volume of the thiosulfate solution.
10. Repeat the above steps (Steps 3 to 9) for Flask II, III and IV but add in step 5 the I_2(aq) solution to each flask as indicated:

Flask II: 10.00 mL I_2 solution
Flask III: 5.00 mL I_2 solution
Flask IV: 5.00 mL I_2 solution

Calculations

B-1. Calculate the initial concentrations (M) of iodine, acetone and HCl solutions in Flasks I to IV, assuming volumes are additive.
B-2. Calculate concentrations of iodine (M) remaining in Flasks I to IV at 7.0 minutes.
B-3. Calculate the initial reaction rate for Flasks I to IV in M s^{-1}.
B-4. The rate of reaction has the form

$$\text{Rate} = -\frac{d[I_2]}{dt} = k[CH_3COCH_3]^x[I_2]^y[H^+]^z$$

Calculate the reaction orders x, y and z from the initial rates and the initial concentrations of acetone, iodine and HCl. The values of x, y and z should be rounded off to the nearest integer and fill in the answer sheet. Write rate equation or rate law.
B-5. Calculate the rate constant, k, for Flasks I to IV with proper unit.
B-6. Give the mean value of the rate constant.