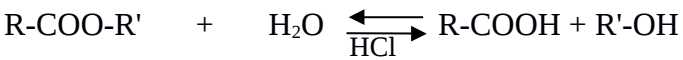
CB101: Experiment 2: Study of kinetic of hydrolisis of ester

I- INTRODUCTION

Oxidation of iron under earth's atmosphere can take years, whereas combustion of cellulose lasts a fraction of second. The rate of a reaction defines how fast or slow a reaction takes place. The investigation of how different experimental conditions (temperature, composition for example) influence the speed of a reaction yield to understanding its microscopic mechanism and can allows to control a reaction.

We will study the hydrolysis of an ester, catalysed* by hydrochloric acid:



For example, if the ester is methyl acetate, the products of this reaction will be acetic acid and methanol.

*Catalyst: substance that speeds up a reaction without being consumed by it.

This reaction is first order with respect to ester concentration, i.e. the reaction rate can be expressed: $v = \frac{-dC}{dt} = k \times C$, where k is the reaction constant, and C is the ester concentration.

Objective: Determine the rate constant k of hydrolysis of an ester by following the concentration of acid in the reaction mixture with time.

The concentration of acid (HCl + R-COOH) will be determined by titration procedure of several samples pipetted at different time using NaOH solution 0.05 mol/L as a titrant, and phenolphtalein as indicator.

II- PRELIMINARY WORK

a) Show that the reaction rate constant in this case can be determined from the relation: $k \times t = \ln\left(\frac{C_0}{C_t}\right)$ where C_0 is the initial ester concentration, and C_t the ester concentration left at a later time t.

b) Read carefully the lab manual.

c) Given V_0, V_t, and V_∞ the volumes of titrant solution added to reach the endpoint at the beginning of the reaction (when actually no reaction has occurred), after time t, and after completion of the reaction, respectively. Show that the reaction constant can be expressed:

$k = \frac{1}{t} \ln\left(\frac{V_\infty - V_0}{V_\infty - V_t}\right)$ *Hint: two acids will be present in the mixture : the catalyst HCl and the product R-COOH*

d) What is the simplest graph to be plotted to determine the rate reaction constant k?

Try to plot the graph between $\log(V_\infty - V_t)$ vs t. From the graph calculate the value of k

III- PROCEDURE

Chemicals required:

- Methyl acetate, ester, CH_3COOCH_3 
- Hydrochloric acid HCl solution 0.5 mol/L 
- Sodium hydroxide NaOH solution 2.50 mol/L 
- Phenopthalein solution 

Apparatus and laboratory glassware required: See appended.

1) Preparation of titrant solution

- From the solution of sodium hydroxide 2.50 mol/L provided, 250.0 mL of sodium hydroxide 0.050 mol/L is prepared. Then, the burette is filled with it.

2) Get use to the micropipette with water :

Set it to 2.0 mL volume by turning the plunger.

The plunger will stop at two different positions when it is pressed. Use first position to slowlydraw up the liquid into the pipette. Use second position when delivering the liquid to expel the last drops.

3) How to stop a reaction?

- A significant amount of very cold water (ice temperature) is prepared in a 500 mL beaker, along with ice.

- About 25 mL of the so prepared ice cold water is prepared in a conical flask. *Why ? Dilution and low temperature will freeze the reaction in order to be able to titrate the reaction medium.*

4) Start the reaction

- 50 ml of supplied HCl solution is poured in a conical flask.

- 2.0 mL of ester is pipetted from the so-called labelled beaker and added to the conical flask at the same time than a timer is started. If the ester had to be added in two steps, the timer would be started at the first step. The pipette is carefully rinsed in a beaker full of distilled water after use.

We now want to titrate the reactionnal medium at 5 or 6 different times : for example t = 5 min, 15 min, 25 min, 35 min, 45 min, 55 min. We also want to titrate the reactionnal medium when the reaction is finished.

5) To obtain V_∞

- 2.0 mL of the reactionnal medium is pipetted and taken in a test tube. The test tube is closed and kept in a water bath at 70°C for at least 45 min for the reaction to complete. Then, with help of distilled water, the full amount of the tube is taken in the conical flask and titrated. *Why ? High temperature will accelerate the reaction so it can be completed in a shorter time*

6) Study of the reaction medium at a certain time t.

- At a certain time t i.e. at the interval of every 10 min (for example t = 5 min, 15 min, 25 min, 35 min, 45 min, 55 min), a sample of 2.0 mL of the reactionnal medium is pipetted from the reaction medium and taken in the conical flask where about 25 mL was poured .

- Few drops of phenolphtalein (about 10) are added, and it is titrated with NaOH solution.

Note: the titration reaction is also slown down because of cold. Make sure that you have or have not reached the end point after every drop by mixing long enough.

Apparatus and glassware required:

- timer
- burette on stand
- magnetic stirrer
- 500 mL beaker
- four 250 mL conical flasks
- 10 mL measuring cylinder
- 50 mL measuring cylinder
- two 100 mL (or 250 mL) beakers
- funnel
- 1 dropper
- 2mL micropipette
- 5 mL pipette
- 250 mL volumetric flask
- small test tube.

APPENDED: