Lab.4

Hydrolysis of Acetyl Salicylic Acid Solution in Sorenson Phosphate Buffer at pH 8

Presented By:

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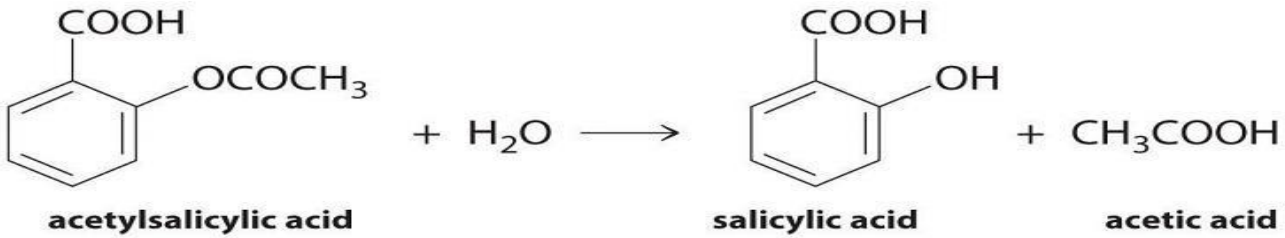
Introduction

Aspirin is a weak acid. It is soluble at 20 C° in 300 parts of water.

Stability:

1. It is unstable in aqueous solutions, degrading to salicylic acid and acetic acid

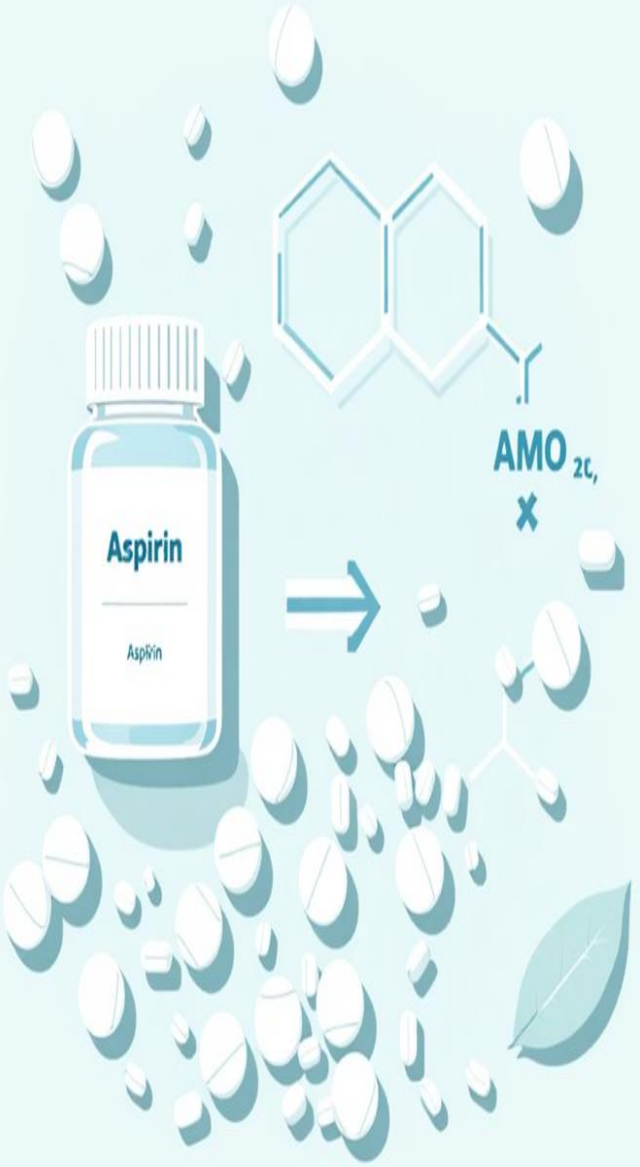


2. Aspirin degrades in solid dosage forms when exposed to moisture [should be stored in tightly closed containers and kept in a dry environment] .

The hydrolysis reaction is critical

It directly impacts the drug's effectiveness and shelf life.

shelf life.



Aim of Experiment

To study the **effect of temperature** on the hydrolysis of **aspirin**, and to calculate the **shelf life of aspirin**.

Shelf life:
It is the **time** required for the drug to **lose** 10% of its effectiveness.

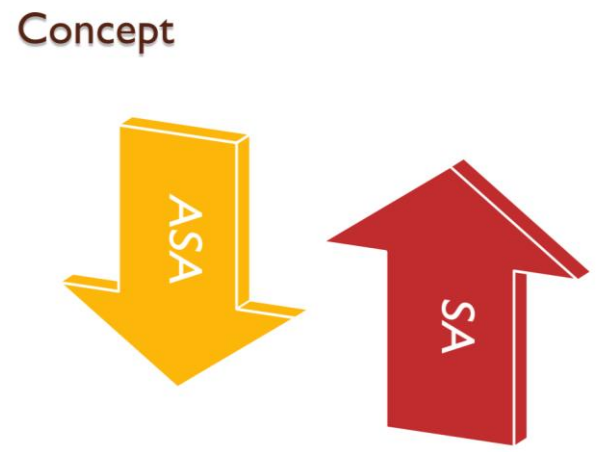
Procedure :

- 1. Dissolve 0.695 g of aspirin in 250 ml of phosphate buffer (use a volumetric flask)
- 2. place 200 ml of this solution in an erlenmeyer flask , then keep the flask in a water bath for (30 min) at required temperature. the temperature that will be used are(40, 60, 80 ℃)
- Withdraw (**5** ml) sample at the end of 30 mins , then continue withdrawing (**5** ml) sample at 15 min interval for 90 min.

Notes:

- Absorbance increases with time.

- Concentration of S.A at zero time is **ZERO** since ASA is not hydrolyzed yet, however, the initial concentration of ASA is 2.78 mg/ml ??



Is the initial conc. of ASA 2.78 mg/ml?

- We dissolved 0.695 **g** of Aspirin in 250 ml

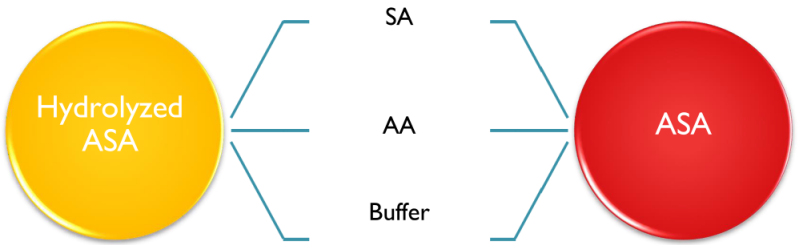
Therefore :

$\frac{0.695\ gram}{250\ ml}$ = 0.00278 gram per ml

Since 1 gm = 1000 mg

So :

0.00278 * 1000 = 2.78 mg / ml

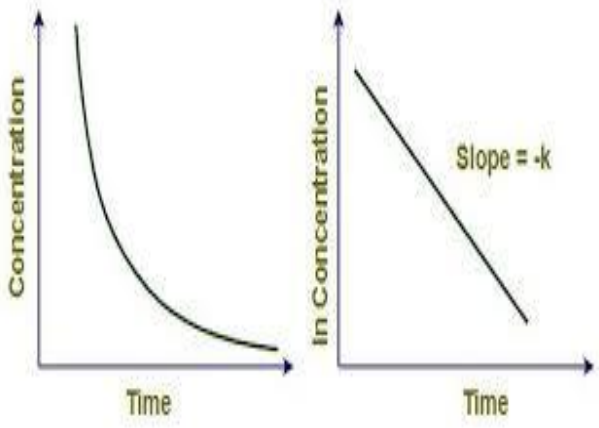


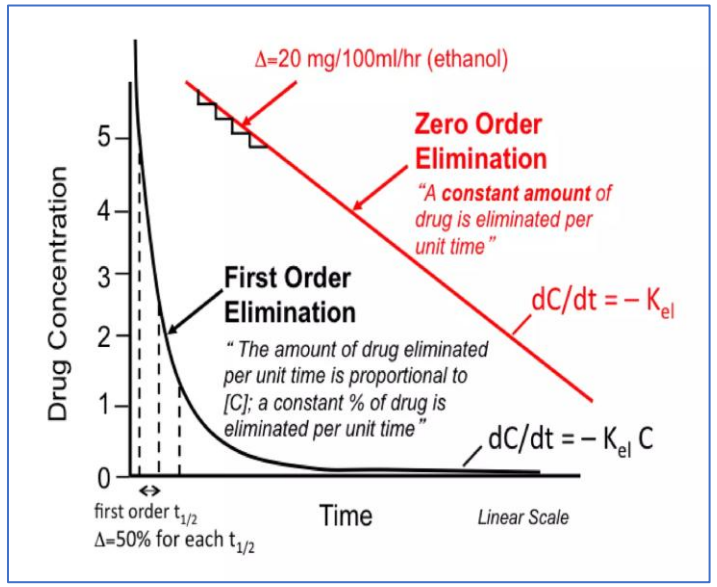
Sorenson phosphate buffer (pH 8)

Consists of two solutions:

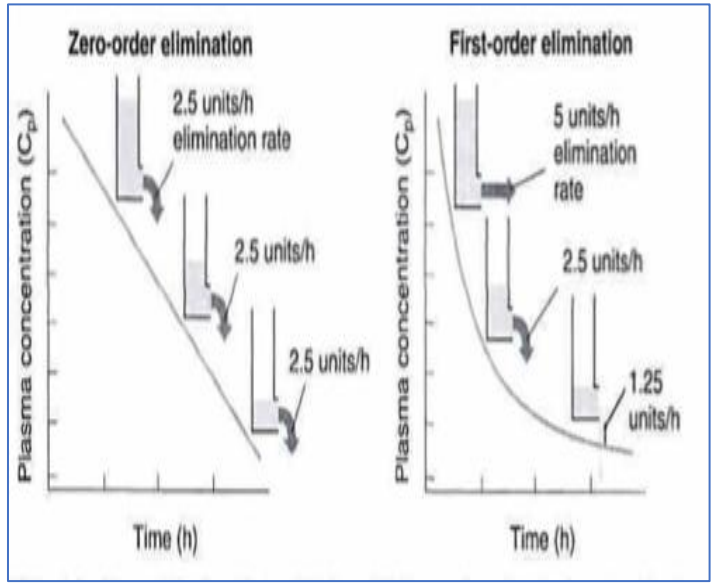
A- 1/15M Monopotasium phosphate (KH2PO4)

B- 1/15M Disodium phosphate Na2HPO4.2H2O

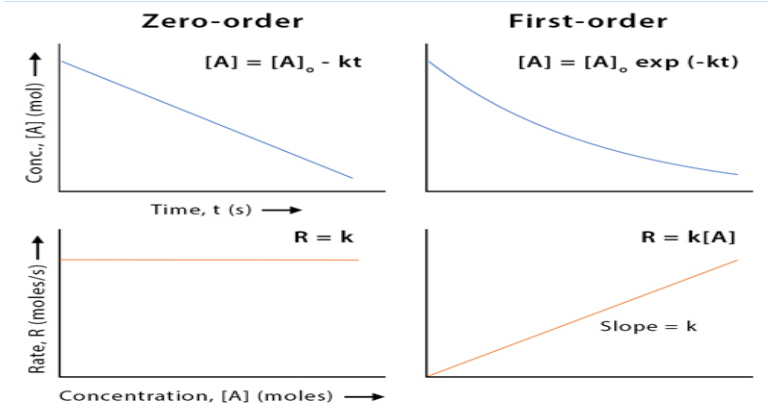




- The rate of ASA hydrolysis follows a first-order reaction



- **Use the calibration curve equation**
- **Y= C + bX (Put the equation for others)**



We are seeking ASA, not SA

Since each MOLE of aspirin give 1 mole of salicylic acid and 1 mole of acetic acid

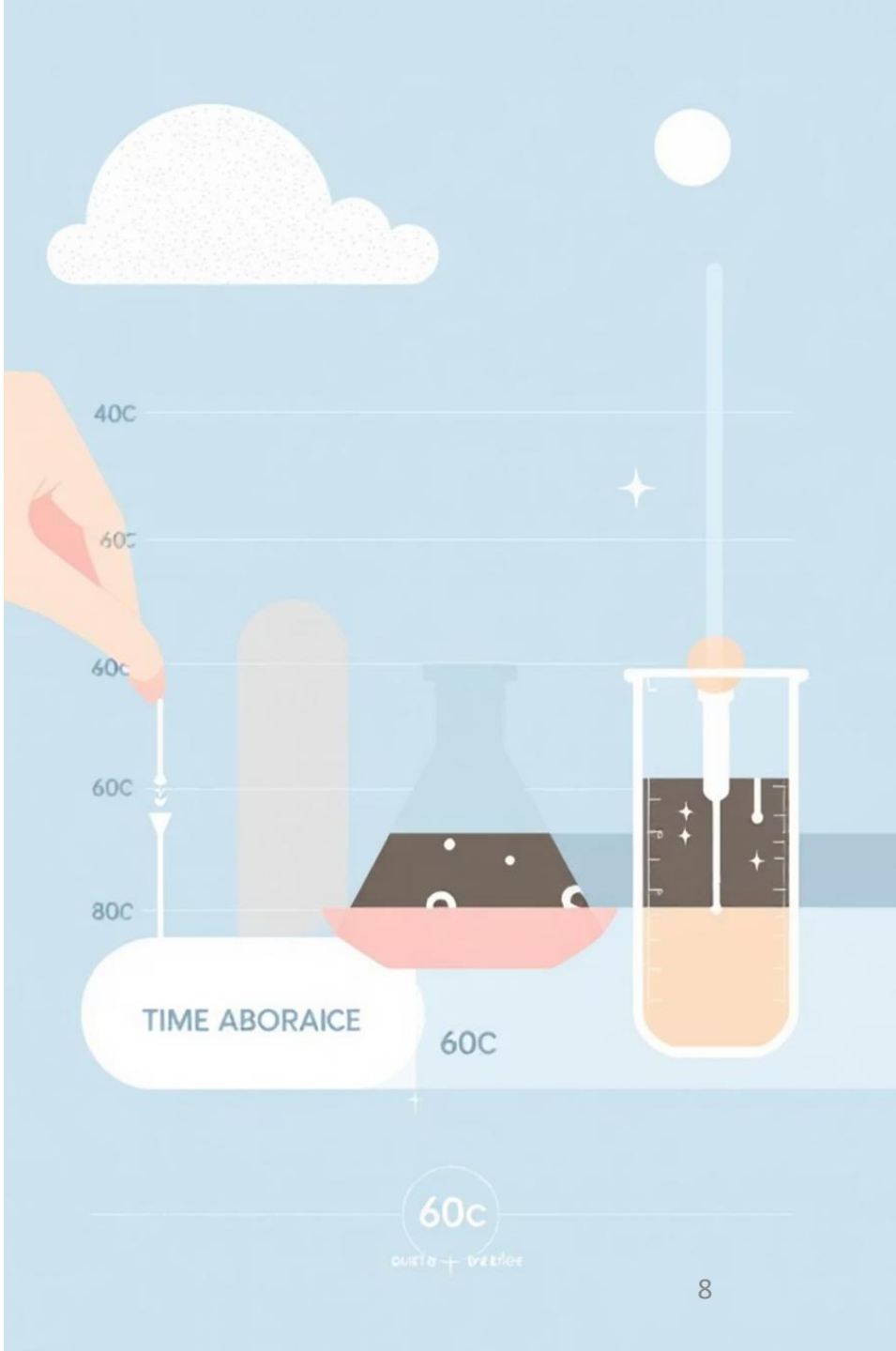
$$\text{Mole} = \frac{Wt}{M\,Wt}$$

Therefore

$$\frac{wt\ of\ SA}{M\,wt} = \frac{Wt\ of\ ASA}{M\,wt}$$

So : 0.35 (from the example) * 180 =X * 138

X= 0.456 ≈ 0.46



- Calculate remaining ASA from:

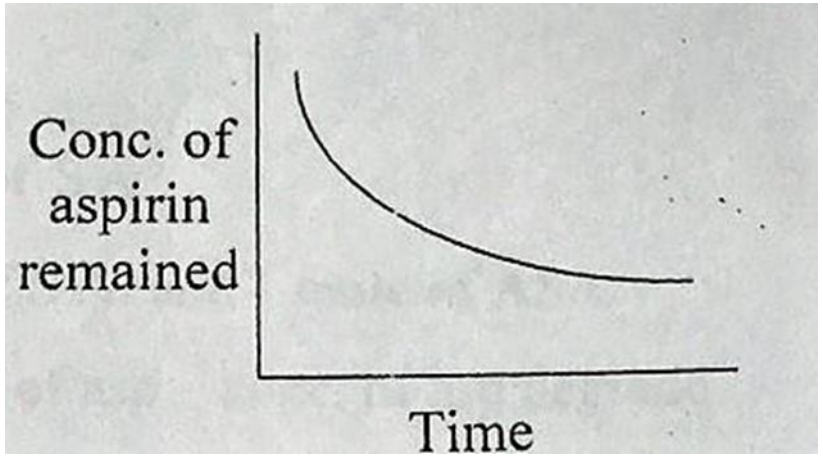
Initial conc. of ASA – conc. of ASA degraded

2.78 – 0.46 = 2.38 mg

Time	Absorbance	Conc. of S. A	Conc. of aspirin hydrolyzed	Conc. Of aspirin remined (ct)	Log ct
0	0	0	0		
30		Y= c + bx	X *180/138	2.78 - Ct	
45					
60					
75					
90					

First order Kinetics

- $\frac{dc}{dt} \propto C$
- $-\frac{dc}{dt} \propto C$ (since conc. Of aspirin decrease with time as hydrolyzed)
- $-\frac{dc}{dt} = KC$



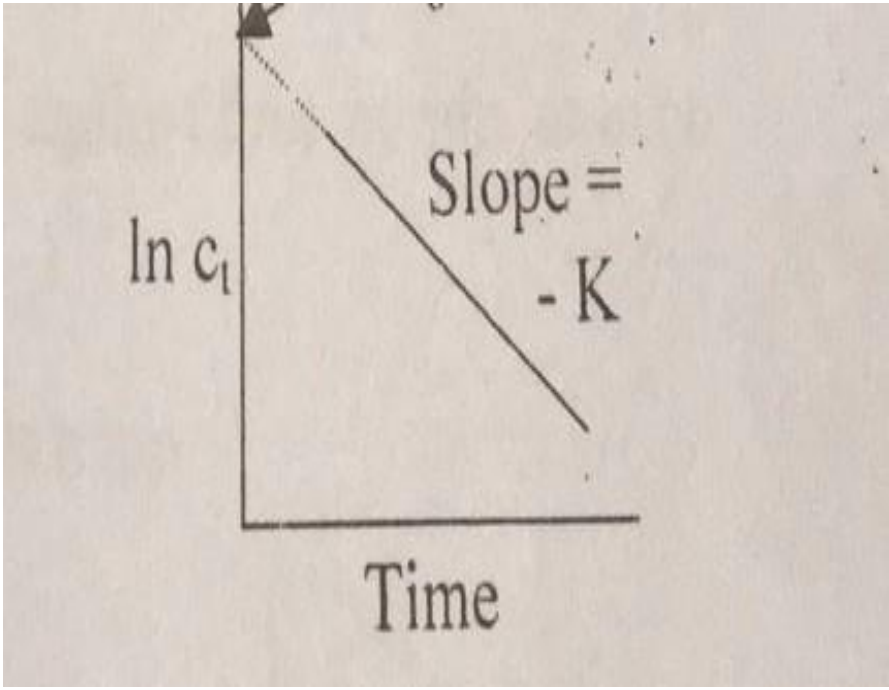
Data Analysis: Plotting and Calculating:

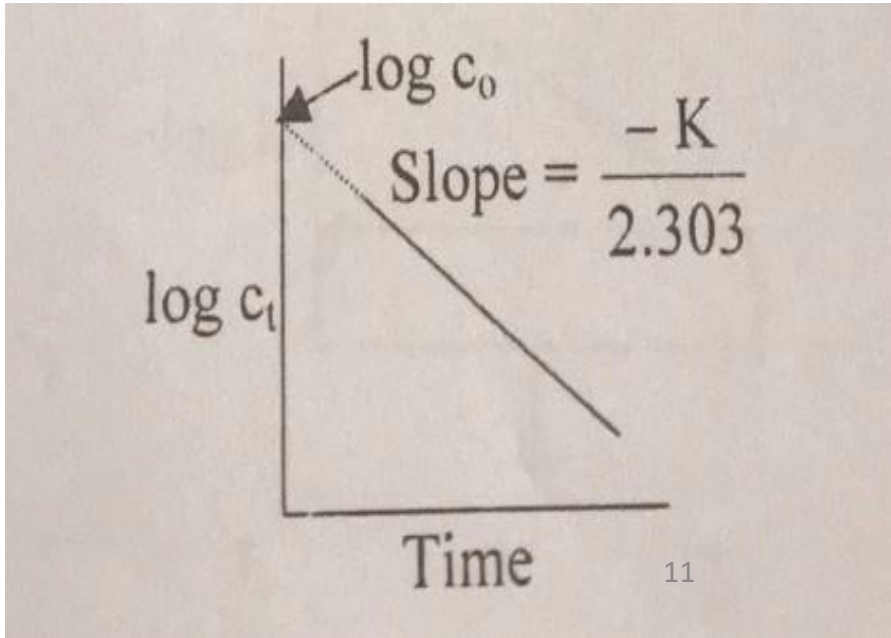
Calculating:

- $\int_{c0}^{ct} \frac{dc}{c} = -k \int_0^t dt$
- $-(\ln Ct - \ln Co) = kt(t-0)$
- $\ln Ct - \ln Co = -kt$
- $\ln Ct = \ln Co - kt$ ★
- Since $\ln = \log \times 2.303$
- $\log Ct * 2.303 = \log Co * 2.303 - kt$
- $\log Ct = \log Co - kt/2.303$ ★

Y= C + bX

Log Ct = log Co – kt/2.303





What to do with data ?

1-take the Log, or Ln, for each calculated conc.

2-Plot the log/ ln of conc. against time in min for each temp.

You will have 3 plots

3-Calculate K for each temp.

4-Take log K and plot $1/T$

5-(draw Arrhenius plot) to find K at 25 °C

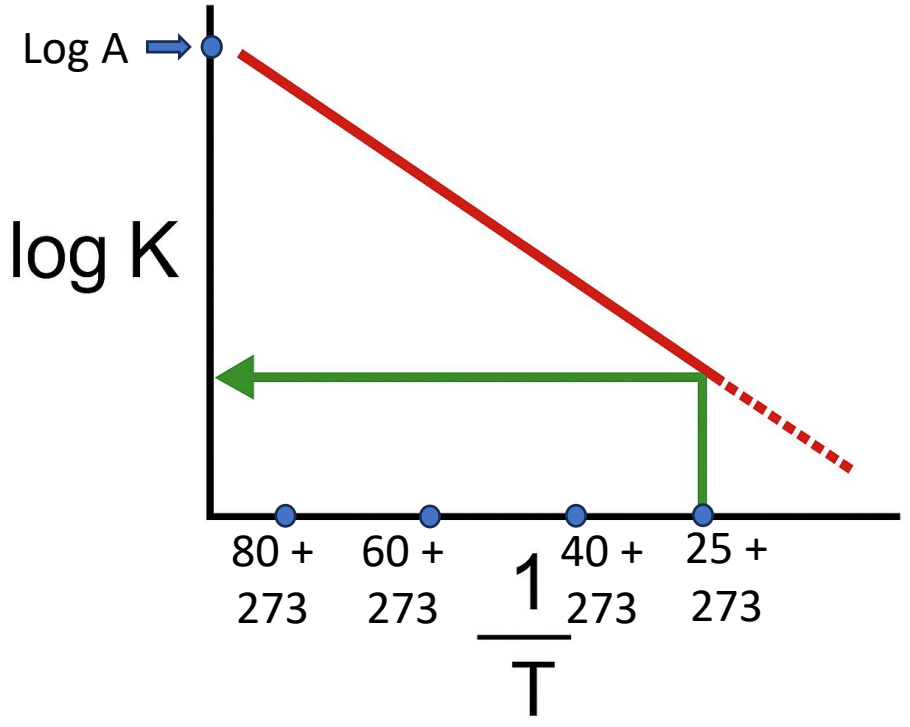
- Arrhenius equation :

$$\text{Log } K = \log A - \frac{(Ea)}{(2.303\ R)} * 1/T$$

Y — Intercept — Slope — x

Where:

- A= frequency factor,
- Ea = energy of activation,
- T= absolute temp.(temp.+273)
- R= gas constant .
- **t10%= 0.105/ K25℃**



Repot to have done according to these results

1- Calculate K for each temp.

2- Calculate the Aspirin shelf life

The results for ASA hydrolysis in pH 8 was as the follow

Time in min	Absorbance at 40 C	Absorbance at 60 C	Absorbance at 80 C
0	0	0	0
30	0.095	0.125	0.33
45	0.118	0.2	0.5
60	0.15	0.27	0.64
75	0.2	0.31	0.76
90	0.23	0.4	0.76

Knowing that the calibration curve was $y = x + 0.0897$

Note: the calibration curve was in mg/ml


Best Regards!
Thank you!