

***Lab.4: Hydrolysis of Acetyl
Salicylic Acid Solution in
Sorenson Phosphate Buffer at
pH 8***

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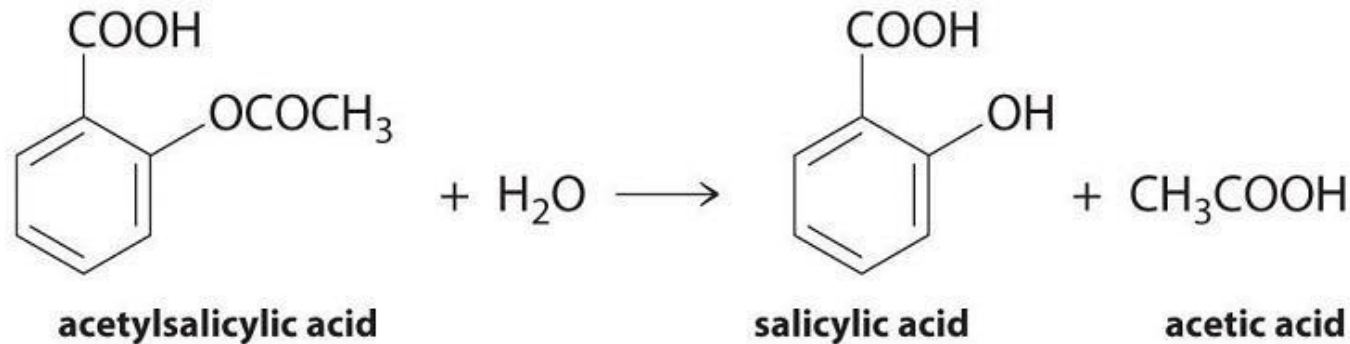
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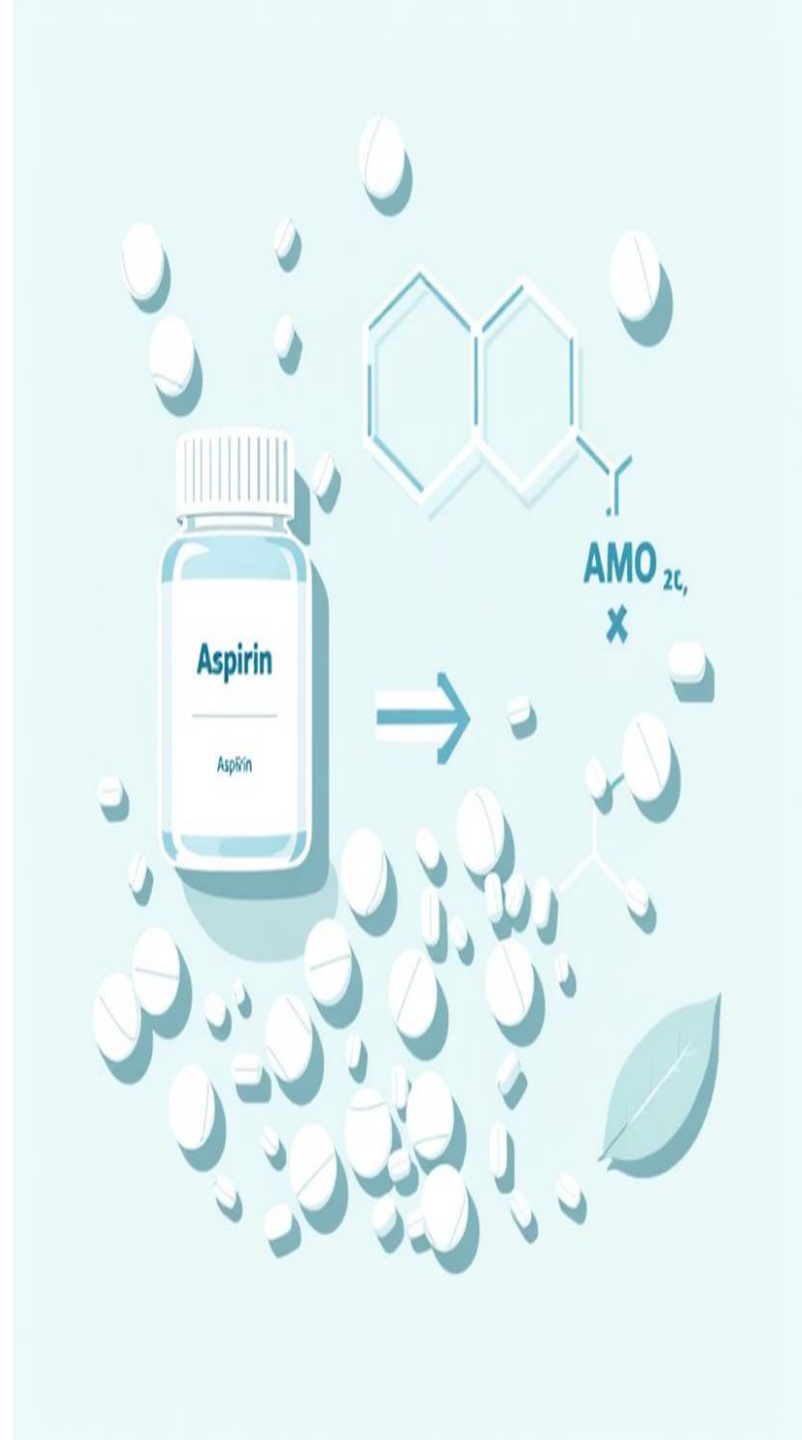
Introduction

- ▶ Aspirin is a weak acid. It is soluble at 20 C° in 300 parts of water.
- ▶ It is unstable in aqueous solutions, degrading to salicylic acid and acetic acid



- ▶ Aspirin also degrades in solid dosage forms when exposed to moisture and therefore, should be stored in tightly closed containers and kept in a dry environment.

The hydrolysis reaction is critical as it directly impacts the drug's effectiveness and 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** of any drug: 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 °C)
- ▶ 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 ??

Concept



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

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

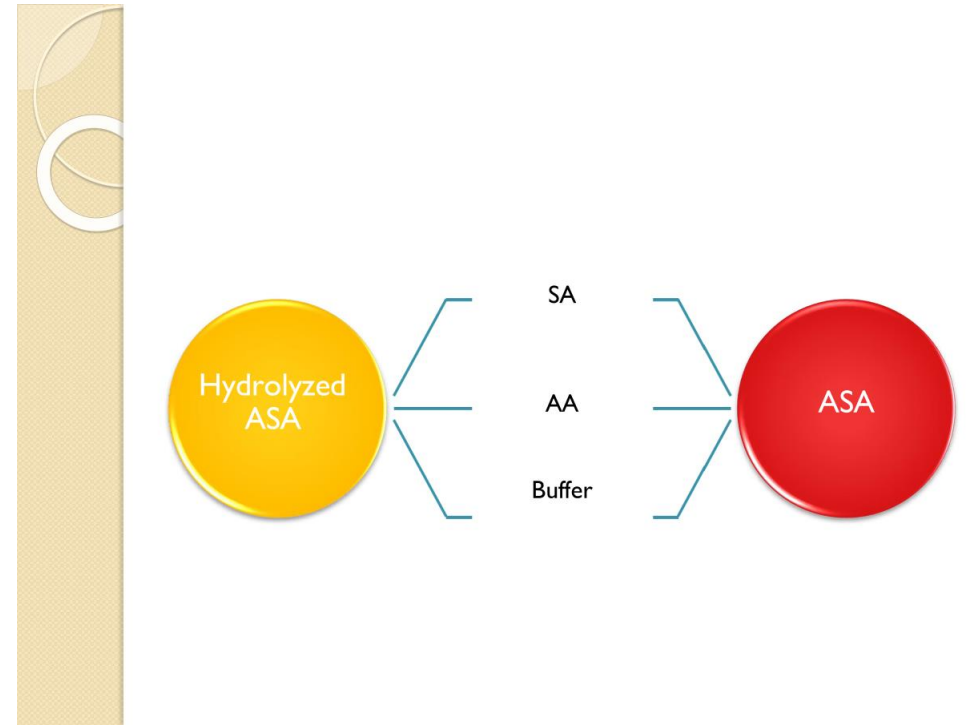
Therefore :

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

Since 1 gm = 1000 mg

So :

$$0.00278 * 1000 = \mathbf{2.78 \text{ mg / ml}}$$



Sorenson phosphate buffer (pH 8)

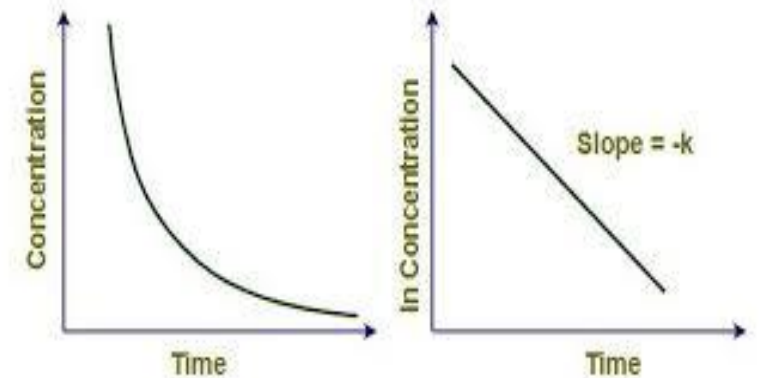
Consists of two solutions

A-1/15 M Monopotassium phosphate KH_2PO_4

B- 1/15 M Disodium phosphate
 $\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$

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

- **Use the calibration curve equation**
- **$Y = C + bX$**



We are seeking ASA, not SA

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

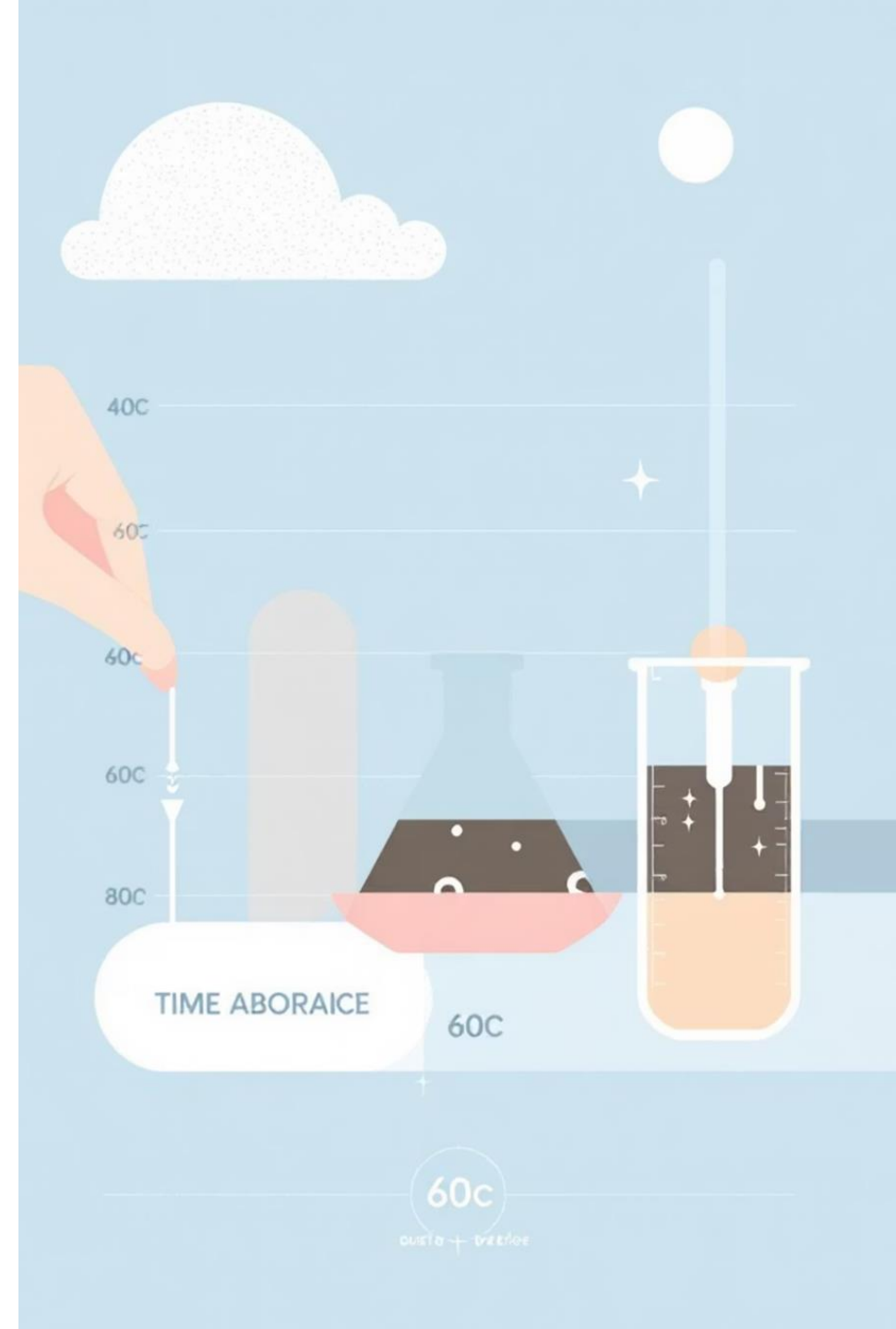
$$\text{Mole} = \frac{Wt}{M Wt}$$

Therefore

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

$$\text{So : } 0.35 \text{ (from the example) } * 180 = X * 138$$

$$X = 0.456 \approx 0.46$$



- Calculate remaining ASA from Initial conc. Of ASA – conc. Of ASA degraded

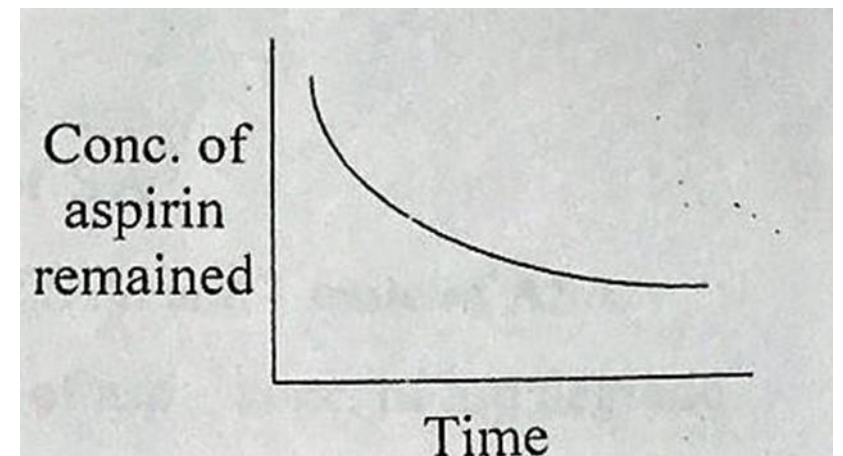
$$2.78 - 0.46 = 2.38 \text{ mg}$$

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					



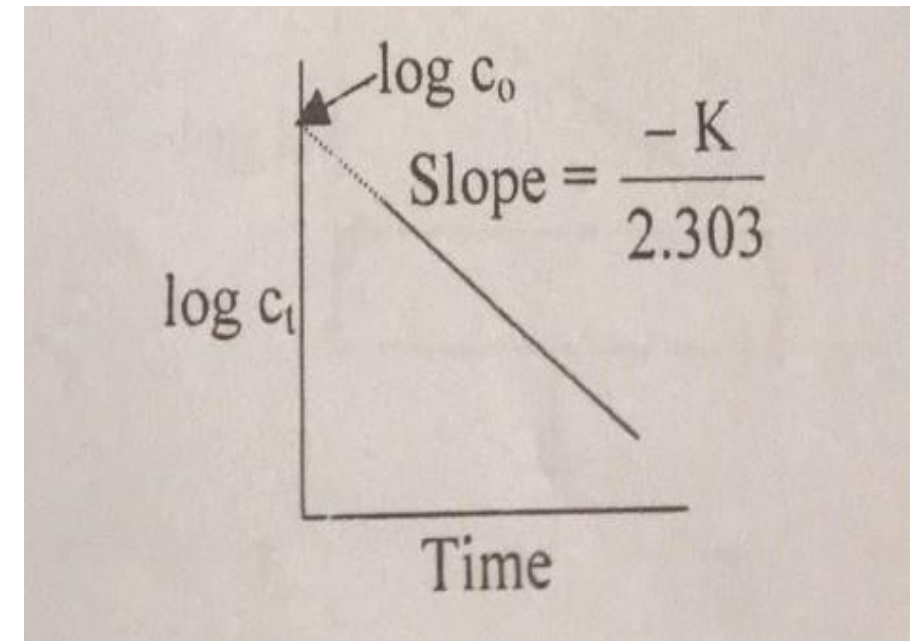
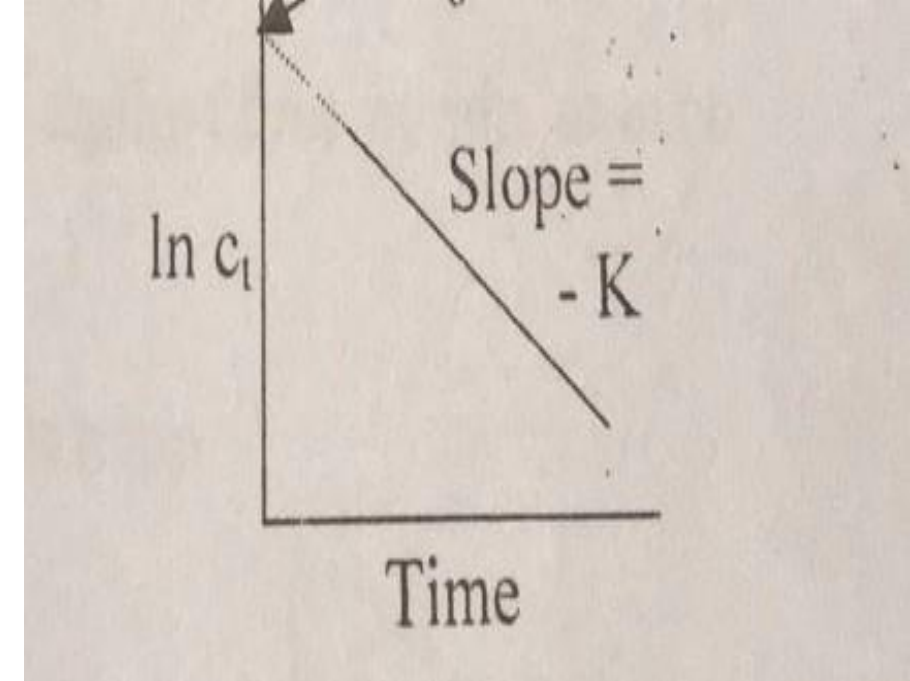
First order Kinetics

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



Data Analysis: Plotting and Calculating :

- ▶ $\int_{c_0}^{c_t} \frac{dc}{c} = -k \int_0^t dt$
- ▶ $-(\ln C_t - \ln C_0) = kt(t-0)$
- ▶ $\ln C_t - \ln C_0 = -kt$
- ▶ $\ln C_t = \ln C_0 - kt$
- ▶ Since $\ln = \log \times 2.303$
- ▶ $\log C_t * 2.303 = \log C_0 * 2.303 - kt$
- ▶ $\log C_t = \log C_0 - kt/2.303$



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.

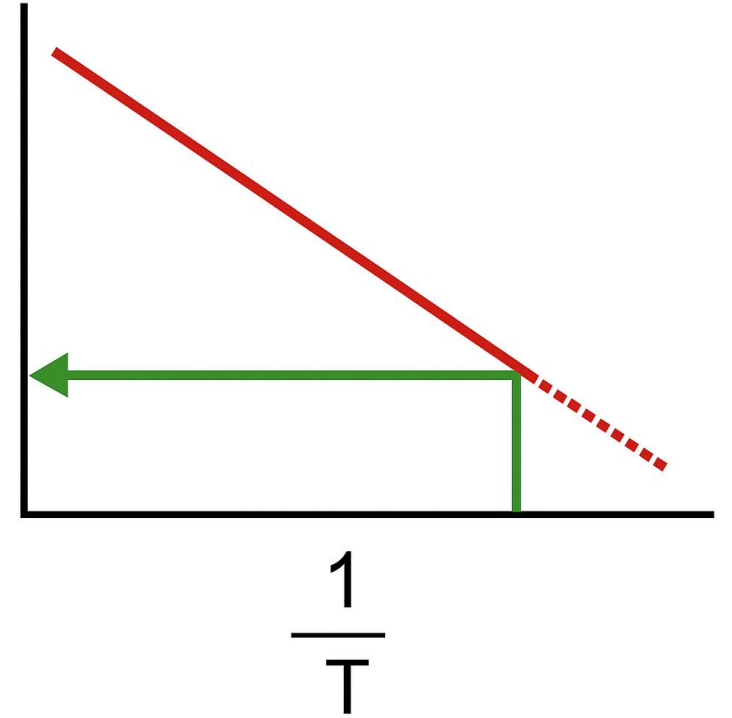
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4-Take log K and plot $1/T$

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

- ▶ Arrhenius equation :
- ▶ $\text{Log } K = \text{log}A - (E_a)/(2.303 R) * 1/T$
- ▶ Where $A =$ frequency factor ,
- ▶ $E_a =$ energy of activation , $T =$ absolute temp.(temp.+273)
- ▶ $R =$ gas constant .
- ▶ $t_{10\%} = 0.105/ K_{25^\circ\text{C}}$

log K



Report to have done according to the results

- 1- Calculate K for each temp.**
- 2- Calculate the Aspirin shelf life**



Best Regards!

Thank you!