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Examined
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Couch Benar College

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Expt. No. 1 Page No. 1

Determination of pH of soft drinks:-

Introduction:- The term pH refers to the measurement of hydrogen ion activity in the sol^n. Since the direct pH measurement is very difficult, specific electrodes are needed for quick and accurate pH determination. pH is measured on scale '0' to '14', with lower values indicating high H^+ (more acidic) and higher value indicating low H^+ ion activity (less acidic). A pH of '7' is considered as neutral. Every whole unit in pH represents a ten-fold increases in or decreases in hydrogen ion concentration. Most neutral water possess the pH value ranging from 5.0 to 8.5.

pH is measured using pH meter, which comprises a detecting unit consisting of a glass electrode refference electrode usually a calomel electrode connected by KCl bridge to pH sensitive glass electrode and an indicating unit which indicates the pH corresponding to the electromotive force is then detected. Before measurement, pH should be Calibrated by using at least two buffers sol^n.

Equipment: 1) pH meter.
2) pH electrode filled with KCl solution.

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3) Buffer sol^n of PH 4 and PH = 7
4) clean beakers.
5) Tissue papers.
6) Soft Drinks.

Procedure: ① Plug in the PH meter to Power source and let it warm up 5 to 10 mins.

② Wash the glass electrode with distilled water and clean slowly with a soft tissue.

③ Place the electrode in PH 7 buffer sol^n and set the value of 7 on the PH meter; turning the calibrate knob on the meter.

④ Take out the electrode, wash with D. W. and clean.

⑤ Deep the electrode in the PH 4 buffer sol^n, adjust the value of PH, repeat with PH 7 and PH 4 buffer till a correct and stable reading is displaced.

⑥ While moving and cleaning the electrode, Put the selector switch on stand by mode. Turn to PH mode for recording the PH.

⑦ Now we Place the electrode in the soft drinks Sample whose PH is to be determined.

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Result:

Solution (sample)	PH
Sprite	4.68
Thumps Up	4.27
Slice	4.61

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Expt. No. 2 Page No. 4

Determination of pH of soil:-

Aim: Measurement of soil pH using a potentiometer.

Principle: Using Potentiometer, the degree of acidity or alkalinity of a soil sample suspended in D.W and in 0.01M $CaCl_2$ sol^n is determined.

Apparatus: Analytical balance, pH meter, thermometer, magnetic stirrer, Stir bar, Centrifuge and tubes, Pipette, beakers (50ml - 100 ml), glass rod, Sieve, 1/4 inch mesh (6.35 mm) morter and Pestle.

Chemical Reagents: ① Buffer Solutions of pH 4.0, 7.0 and 10.0 (Suitably Prepared or, Commercially available)

② 0.01 M $CaCl_2$ Solution.

Sample Preparation:

① With water: Collect the soil sample and dry it. Breaks the bigger soil lumps and grind it with a morter Pestle. Separate the finer soil Particles into a 50 ml beakers and add 10ml of distill Water, using a glass rod, mix it throughly. Then stir the mixture using a magnetic stirrer until the soil is fully suspended in water, let the mixture stand for 10 mins to settle.

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(iii) With Calcium chloride Sol^n:- Follow the above Procedure to prepare another soil extract using 10ml 0.01M $CaCl_2$ aqueous Sol^n in place D.I. water.

Procedure:- Using a thermometer, measure the temperature of the soil suspension. Set this temperature (°C) in PH meter. According to the samples nature, Calibrate the pH meter using PH 4 and 7 buffer Sol^n. If the soil sample is basic in nature, PH meter must be Calibrated using PH 7 and 10 buffer Sol.^{n} on the other hand, if the soil sample is acidic in nature must be calibrate 4 to 7 buffer Sol^n. Take out electrode from buffer solution, rinse with D.I water and blot dry with a soft tissue. then place the electrode in the sample soil suspension. Record the pH of the soil sample in both water and 0.01M $CaCl_2$ aqueous solution.

Results:

Soil (Sample)	PH
1. ~~Sol~~ Soil with H_2O	6.41
2. Soil with $CaCl_2$ Sol^n	6.17

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Determination of the Composition of Fe^{3+}-salicylic acid Complex in a solution by Job's Method :-

Theory : By using a method of continuous variation, a technique is first developed by Job, allows us to find the formula for the complex in solution. In this method, several solutions are prepared in which concentration of the metal ion and ligand are varied but the sum of the concentrations is kept constant. Using this solutions absorbance are calculated and consequently the mole fractions are calculated. In this experiment, the composition of Fe^{3+}-salicylic acid ion to form a violet colour complex as follows:-

$$x Fe^{3+} + y\, C_6H_4(OH)COOH \rightleftharpoons Complex$$

the complex is formed by the anion of salicylic acid with Fe^{3+} ion and thus the stability constant of the solution varies with the pH.

The experiment is carried out at pH = 2.6 to 2.8 at which the phenolic group of acid is undissociated and the carboxylic acid partly dissociated. The pH range is achieved by using Fe^{3+} sol^n and salicylic acid in 0.002(M) mineral acid solution.

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the optical absorbance of the complex is given by Lambert-Beers law:

$$A = \log I_0/I = \epsilon c l.$$

where, A = absorbance
I_0 = Intensity of incident light.
I = Intensity of transmitted light.
ϵ = extention Co-efficient.
c = Concentration.
l = Path length of the sample.

Requirments :- i) Spectrometer.
ii) Curettes
iii) standard-flask (100ml)
iv) volumetric flask (100ml)
v) Graduated pipette
vi) Weighting bottle
vii) Salicylic acid
viii) Sulphuric acid
ix) Ferric sulphate.

Solution Preparation:

i) 0.002 (M) H_2SO_4 Preparation:
50 ml 1 (M) H_2SO_4 = 3.9 ml H_2SO_4 + 46.1 ml Water.
and 500 ml 0.002 (M) H_2SO_4 = 1 (M) H_2SO_4 1 ml + 499 ml water.

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ii) 250ml 0.002 (M) Salicylic acid :-

$$\text{Molarity} = \frac{W \times 1000}{M \times V}$$

$$\Rightarrow 0.002 = \frac{W \times 1000}{138 \times 250}$$

$$\Rightarrow W = 0.069 \text{ gm Salicyclic acid.}$$

iii) 250 ml 0.002 (M) Ferric Sulphate Solution:

$$\text{Molarity} = \frac{W \times 1000}{M \times V}$$

$$\Rightarrow 0.002 = \frac{W \times 1000}{400 \times 250}$$

$$\Rightarrow W = 0.2 \text{ gm}$$

Hence, 0.2 gm Salt + 250ml 0.002 (M) H_2SO_4.

Procedure: Firstly Prepared 0.002 (M) Salicylic acid Sol^n in 0.002 (M) H_2SO_4. Secondly, Prepare 0.002 (M) Fe^{3+} Sol^n in 0.002 (M) H_2SO_4. Then Prepare the following table to dissolved the Salicylic acid Sol^n. the solution may be gently heated for 5 minute.

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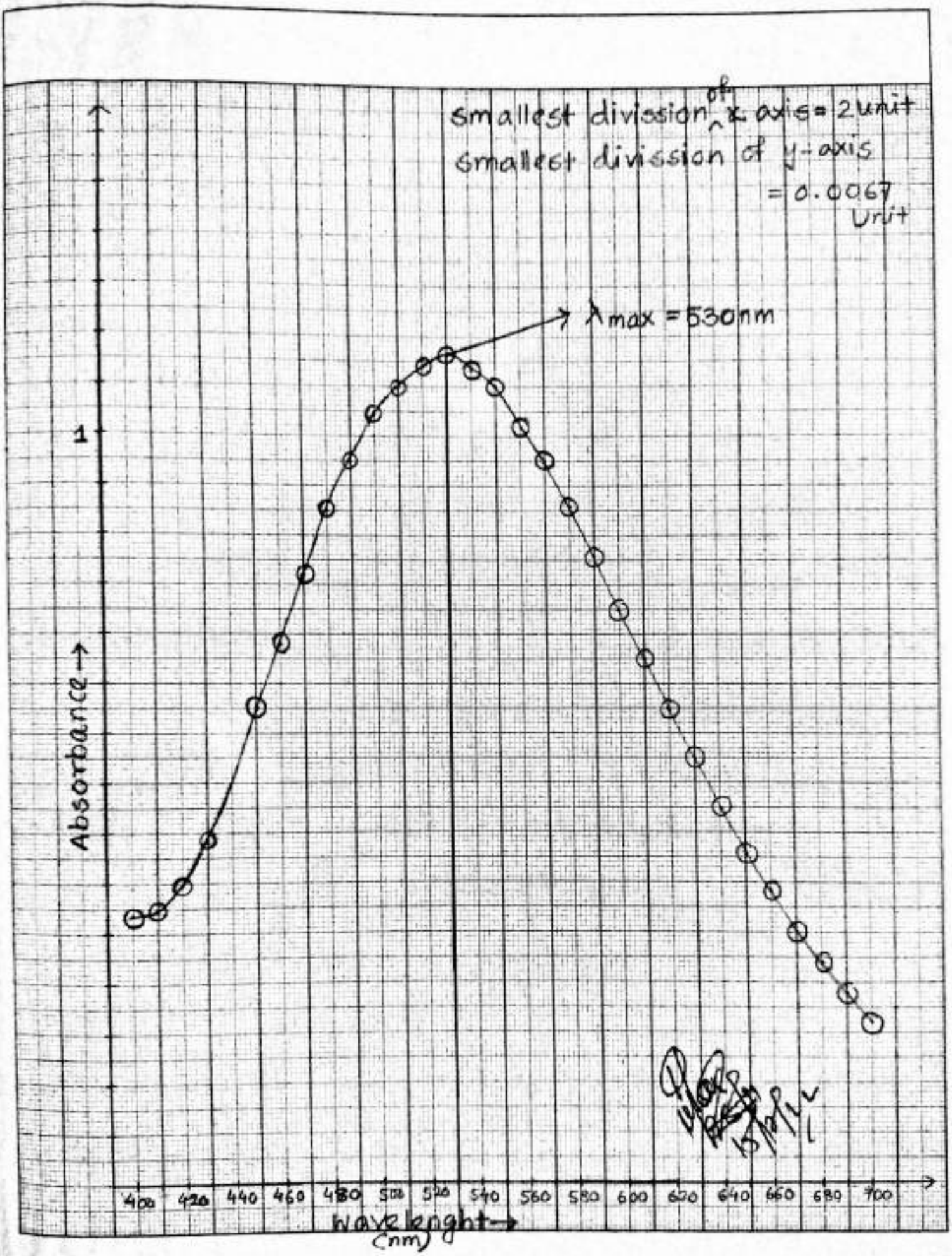
Smallest division of x-axis = 2 unit
Smallest division of y-axis
= 0.0067
Unit
λmax = 530nm
Absorbance →
1
400
420
440
460
480
500
520
540
560
580
600
620
640
660
680
700
wavelenght →
(nm)

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Table-1

SL. NO.	Fe^{3+} sol^n (mL)	salicylic acid sol^n (mL)
1	9	1
2	8	2
3	7	3
4	6	4
5	5	5
6	4	6
7	3	7
8	2	8
9	1	9

Determination of λmax using one of the above sol^n say 5:5, λmax record the absorbance of each sol^n. plote the graph between the absorbance vs volume of Fe^{3+} ions sol^n in the mixture. Mark the maximum plot in the curve which corresponds to the Composition of the complex. In this case. It is 1:1 Complex.

Table-2

Determination of λmax of Fe^{3+} - salicylic acid complex.

SL NO.	λmax	Absorbance (A)
1	400	0.358
2	410	0.362

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Sl. No.	λmax	Absorption(A)
3	420	0.399
4	430	0.460
5	440	0.591
6	450	0.639
7	460	0.727
8	470	0.819
9	480	0.905
10	490	0.978
11	500	1.036
12	510	1.077
13	520	1.099
14	530	1.105
15	540	1.093
16	550	1.066
17	560	1.025
18	570	0.975
19	580	0.915
20	590	0.846
21	600	0.777
22	610	0.708
23	620	0.638
24	630	0.572
25	640	0.508
26	650	0.448
27	660	0.392
28	670	0.341
29	680	0.295
30	690	0.254
31	700	0.218

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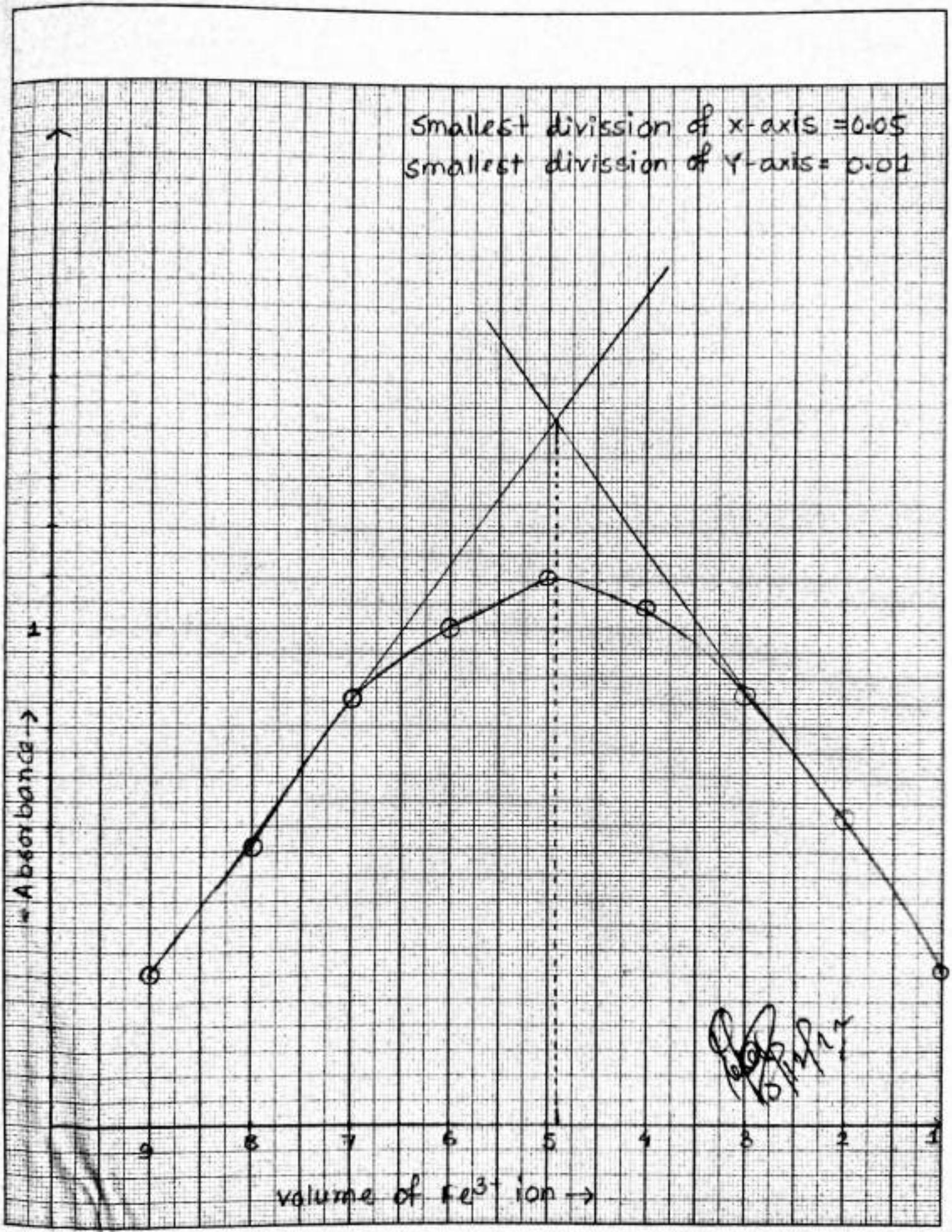
Smallest division of x-axis = 0.05
smallest division of Y-axis = 0.01
Absorbance →
9
8
7
6
5
4
3
2
1
volume of Fe^{3+} ion →

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Table-3

Determination of Composition of Complex:

Fe^{3+} ion Soln (mL)	salicylic acid Soln (mL)	Absorbance at 530 nm
9	1	0.307
8	2	0.558
7	3	0.862
6	4	1.027
5	5	1.103
4	6	1.043
3	7	0.869
2	8	0.620
1	9	0.311

Result: the Composition of Fe^{3+}-salicylic acid Complex in solution by Job's method is found to be 5.15 : 4.85

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Expt. No. 4 Page No. 12

Determination of spectroscopic identification and determination of benzoic acid in soft drinks.

Principle: the commercial soft drinks contain several ingredients such as carbonated water, high fructose, commercial syrup, caramel colour, sodium benzoate etc. Benzoic acid is a food preservative which is widely used in acidic food and generally add as salt, sodium benzoate. To avoid any kind of interference by some other UV active molecules like aspartame or caramelized sugar (darkly coloured drinks), white colour non-diet soft drinks (like sprite etc. are choosen for this experiment. Pure benzoic acid absorbance maximum at 229 and 272 nm respectively. Soft drinks containing benzoic acid will also record at this absorbance maxima. By comparing the intensities with reference solutions the content of benzoic acid is sample soft drink is calculated.

Apparatus: i) Beaker
ii) Measuring cylinder
iii) Spectrophotometer (single beam)
iv) Test tube.

Sample Preparation:

1) 0.1 M HCl 1000 ml Preparation:
(4.1 ml Conc. HCl + 495.9 ml H_2O)

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2) 100 mg/L benzoic acid Sol^n Preparation:

0.10 g (100 mg) benzoic acid dissolved in 1000 ml distilled water.

Chemical Reagent:

i) 0.1 M HCl 1000 ml
ii) Standard benzoic acid solutions.

Prepare 100 mg/L aqueous stocks Sol^n of benzoic acid and then make a set of standard solutions containing 2, 4, 6, 8 and 10 mg/L benzoic acid in 0.01 M HCl. To prepare these standard solutions take 2, 4, 6, 8 and 10 ml of benzoic acid in stock Sol^n, respectively in 100 ml beaker. Diluted each solution upto 100 ml water (DL).

Procedure:

Record the absorption spectrum of the soft drinks Sol^n. From the absorbance value of sample Sol^n at 272 nm. find the concentrations of benzoic acid in original soft drinks.

Sample Preparation:

Table-1

Amount of benzoic acid stock Sol^n (mL)	Amount of 0.1 (M) HCl Sol^n	Amount of water (mL)
2	10	88
4	10	86

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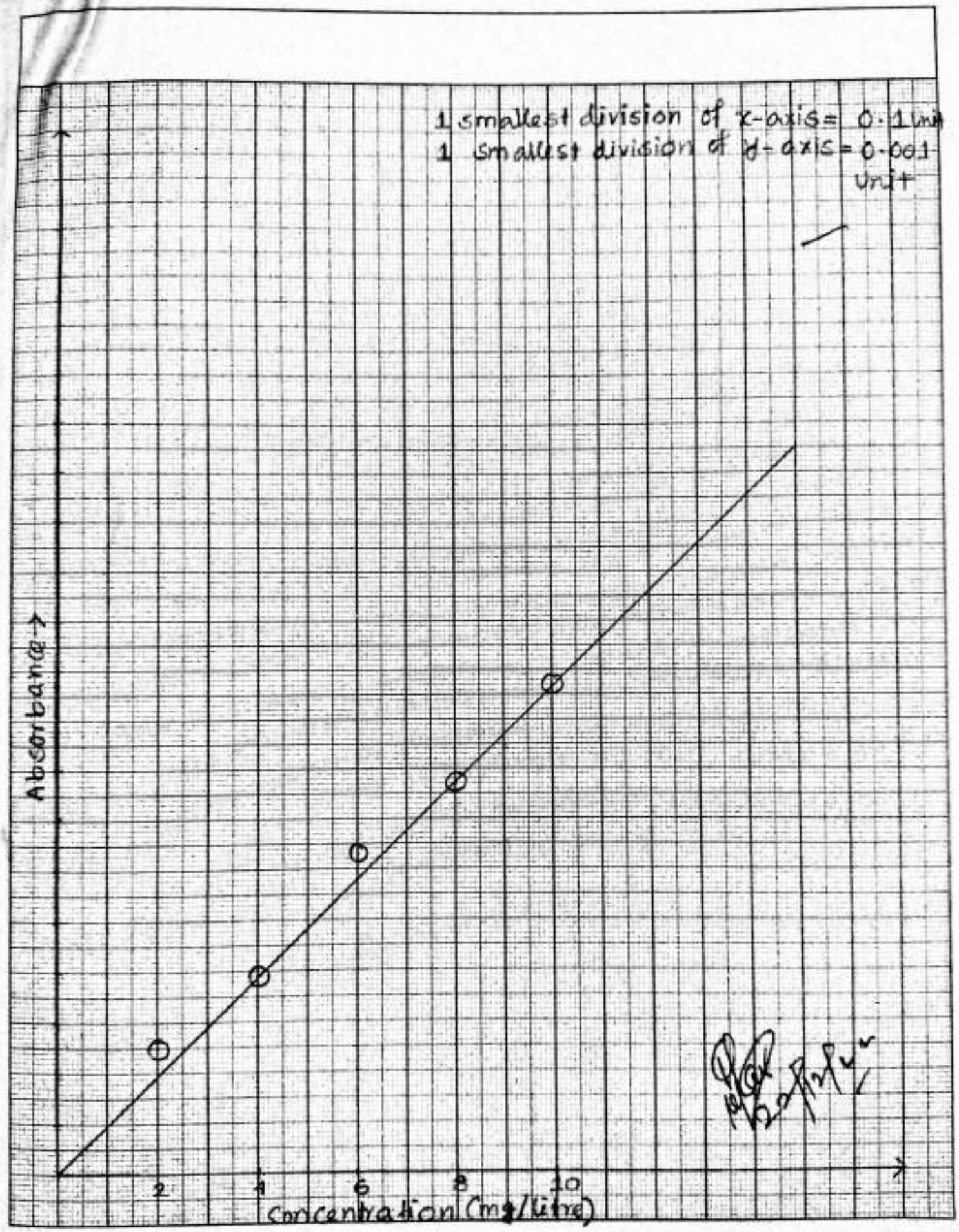
1 smallest division of x-axis = 0·1 unit
1 smallest division of y-axis = 0·001 unit
Absorbance →
2
4
6
8
10
Concentration (mg/litre) →

6	10	84
8	10	82
10	10	80

Results:

Sl. No.	Amount of Benzoic acid stock sol^n (mL)	Amount of 0.1(M) HCl sol^n	Amount of water	Absorbance
1	2	10	88	0.025
2	4	10	86	0.039
3	6	10	84	0.063
4	8	10	82	0.077
5	10	10	80	0.097
Unknown	4	10	86	0.0538

Calculation:

$\lambda_{max} = 272$ nm.

This is a linear (straight line) passing through origin we found from graph. equation of straight line, $y = mx$

$$\therefore m = \frac{y}{x} = \frac{\Delta y}{\Delta x} = \frac{0.097 - 0.039}{10 - 4}$$

$$= \frac{0.058}{6}$$

$$= 9.66 \times 10^{-3}$$

For, unknown sol^n we know, $y = 0.538$

$$\therefore m = y/x \Rightarrow x = y/m = 0.538 / 9.66 \times 10^{-3}$$

$$= 55.69$$

Hence, In unknown solution benzoic acid = 55.69 mg/L.

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