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Expt. No. 9

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EXPERIMENT: Spectrophotometric determination of Iron (II) with 1,10 - phenanthroline.

THEORY: Iron (II) reacts with 1,10-phenanthroline to form an orange red complex $[(C_{12}H_8N_2)_3Fe]^{+2}$. The color intensity is independent of the acidity in pH range 2-9. If Iron (III) is present it can be reduced with hydroxylamine hydrochloride. The absorbance is mentioned at a wavelength of 515nm. The variation in the concentration of a given colored solution, changes the intensity of the transmitted light. The change in light intensity is measured by the instrument called photocolourimeter / colourimeter. When monochromatic light falls on a solution sample, some light is absorbed and the intensity of the transmitted light is decreased. The decrease in increase of light is proportional to the thickness of the absorbing medium and the concentration of solution. This may be expressed by Lambert-Beer Law:

$$A = \log (1/T) = \log (I_0/I) = \varepsilon b c$$

where 'c' is the conc. of the solution expressed in mol/L and 'ε' is a constant characteristic of the solute and the wavelength of light, 'ε' is called the molar extinction coefficient. 'A' is the absorbance or optical density (D) of the solution; 'b' is the path length and is related to the transmittance ($T = I/I_0$).

PROCEDURE:

A. Preparation of Samples

1. Take six 50 ml volumetric flasks and add 0, 1, 2, 3, 4, 5 ml of FAS solution in each flask. Let's name the volumetric flasks as

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EXPERIMENT: Spectrophotometric determination of Iron (II) with 1,10-phenanthroline.

APPARATUS: Burette, volumetric flasks (50ml), cuvettes, funnel, burette stand, clamp and calorimeter.

CHEMICALS: Mohr's salt solution (Ferrous Ammonium Sulphate; $FeSO_4(NH_4)_2SO_4.6H_2O$), 1,10-phenanthroline, hydroxylamine hydrochloride, acetic acid-sodium acetate buffer of pH 4.5 and sulphuric acid (H_2SO_4).

CHEMICAL REACTIONS:

(Colourless). Phenanthroline (Colourless) Iron-phenanthroline complex (Reddish-orange colour)

n = number of phenanthroline molecules reacting with Fe^{2+}

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K, L, M, N, O and P.

2. Then add 2ml of 1-10 phenanthroline solution to each of these volumetric flasks.

3. Now dilute each volumetric flask with deionised water to afford a total volume of 50ml (by filling these flasks upto the mark). Stopper the flasks and mix the contents well by shaking vigorously for few minutes. Allow the solution to stand for 10 minutes.

4. The first volumetric flask to which 0ml of FAS is added (i.e. no Fe^{2+} ions), will serve as a blank (Solution K).

The Fe concentration in these flasks will be :

K	0.0N	N	6.0×10^{-5} N
L	2.0×10^{-6} N	O	8.0×10^{-5} N
M	4.0×10^{-5} N	P	10.0×10^{-5} N

B. To determine the λ_{max}

1. Get the two cuvettes.
2. Fill one of them with the blank solution (K) and another one with the one of the samples containing Fe. Let's say solution P.
3. Light of single wavelength can be produced by selecting the filter on the photocolorimeter. Usually, the range goes from 410nm - 700nm.
4. Set the filter to 410nm. Place the cuvette filled with blank solution, K, in the sample holder.
5. Set the absorbance to 0%.
6. Now place the second cuvette, with solution P, in the sample holder. Measure the absorbance of the solution. Now you have the absorbance at 410nm for solution P.

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OBSERVATIONS:

(i) Absorbance of the solution at highest concentration (10×10^{-5} N) at various λ

S. No.	Wavelength λ in nm	Absorbance
1.	400	0.10
2.	420	0.19
3.	470	0.17
4.	500	0.20
5.	530	0.14
6.	620	0.01
7.	660	0.00

(ii) Absorbance of the solutions at different concentrations at λ max

S. No.	Concentration (N)	Absorbance
K	0	0
L	2×10^{-5}	0.04
M	4×10^{-5}	0.09
N	6×10^{-5}	0.13
O	8×10^{-5}	0.17
P	10×10^{-5}	0.20
X	unknown	0.11

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7. By changing the filter to next wavelength each time, repeat steps 4-6. You need to set the absorbance to zero with blank (K) every time you change the wavelength with filter.
8. Now, you have absorbance of solution P, over a range of wavelength from 410nm - 700 nm. You will notice that graph between the absorbance and wavelength takes an inverse parabola shape, with a maximum absorbance around 500nm or 480nm This is your λ_{max}.

C. Measurement of Absorbance for Solutions L to P at λ_{max}

1. Set the filter to λ_{max} obtained in Part B (Step 8).
2. Set the absorbance to 0 using your blank sample (K).
3. Measure the absorbance for solutions L to P now at λ_{max}. Don't disturb the filter in between.
4. Now measure the absorbance for an unknown sample provided to you.
5. Plot absorbance vs. concentration for samples L to P. Connecting maximum points, draw a straight line ideally passing through origin.
6. Using absorbance value for the unknown, find out its conc.

RESULT: The Fe content in the unknown/given sample is 27.5 µg of Fe.

PRECAUTIONS: 1. Do not rinse the conical titration flask.
2. Use the colorimeter precisely taking blank solution as a reference for others.

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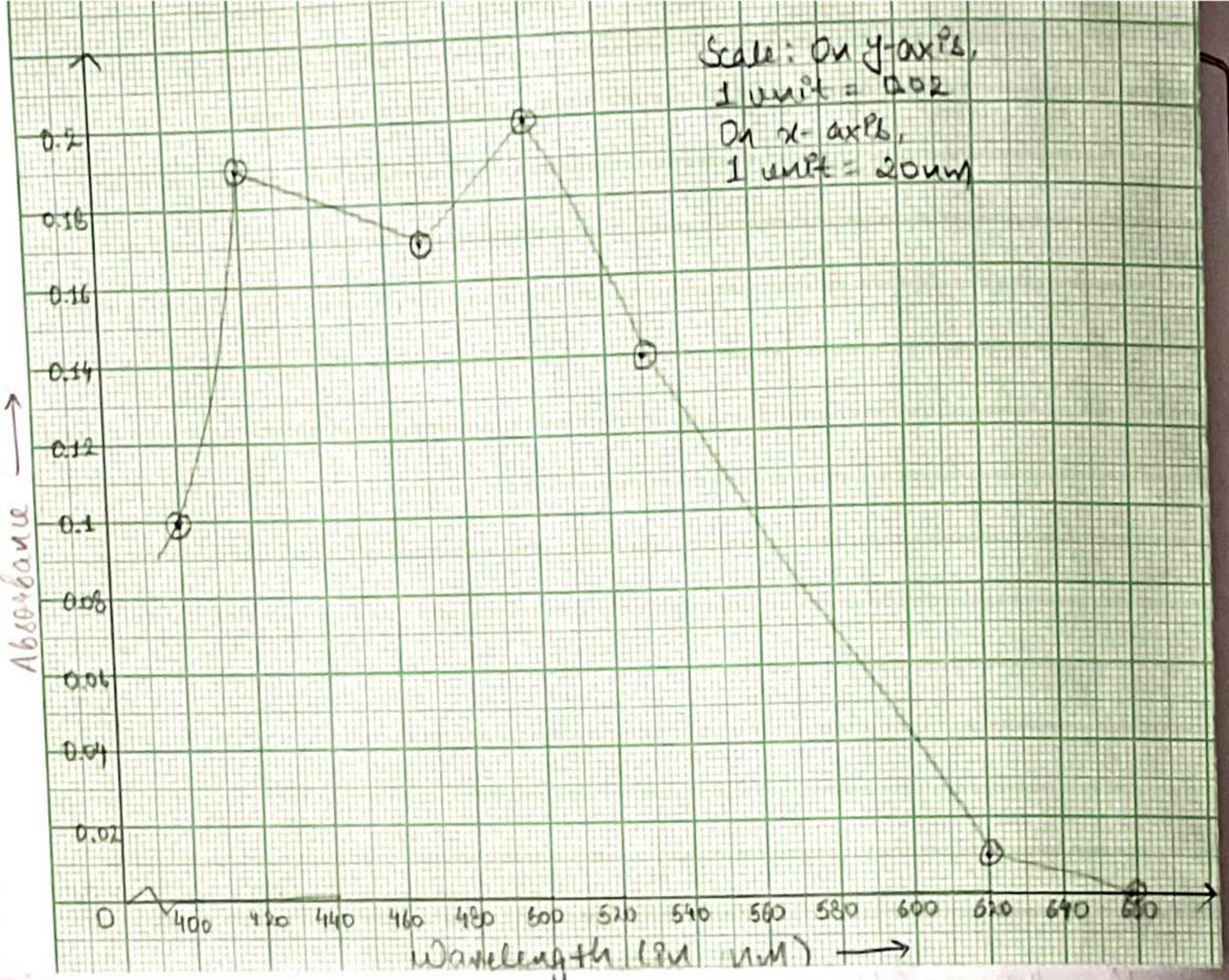
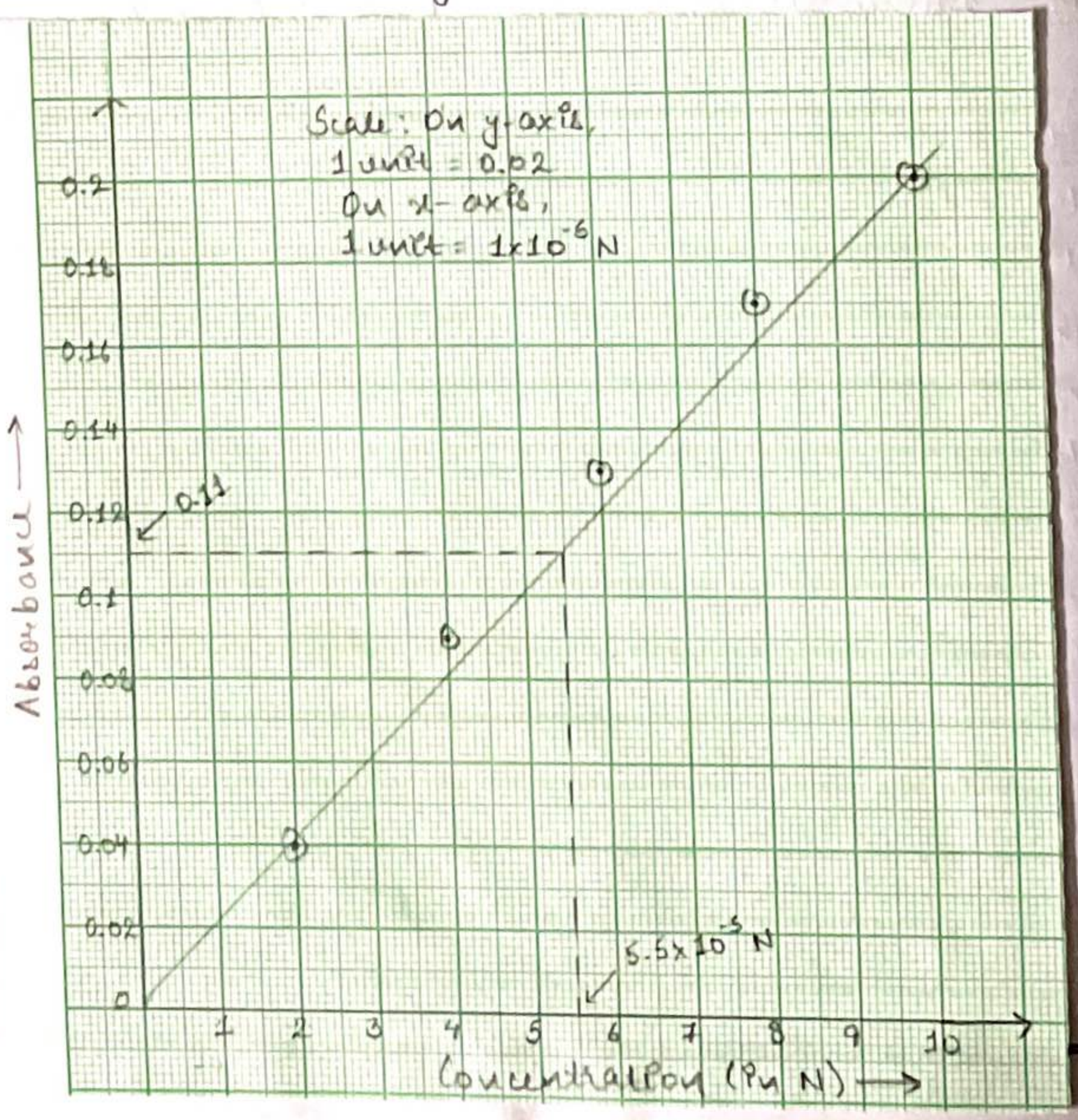
Scale: On y-axis,
1 unit = 0.02
On x-axis,
1 unit = 20nm



Scale: On y-axis,
1 unit = 0.02
On x-axis,
1 unit = 1×10^{-6} N



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