Date _____

Expt. No. _____ Page No. _____

EXPERIMENT- 9.

AIM :- Spectrophotometric determination of iron (II) with 1, 10 - phenantroline

THEORY :- Iron (II) reacts with 1,10- phenanthroline to form an orange red complex $[(C_{12}H_8N_2)_3Fe^{+2}]$. The colour 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 515 nm. The variation in the conc. of a given coloured soln. changes the intensity of the transmitted light. The change in light intensity is measured by the instrument called photo colourimeter / colourimeter. When monochromatic light falls on a soln. sample, some light is absorbed and the intensity of the transmitted light is decreased. The decrease in intensity of light is prop. to the thickness of the absorbing medium and the conc. of soln. This may be expressed by Lambert - beers law -

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

where c is the conc. of the soln. 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 absorbance or optical density 'D' of solution, b is path length and related to transmittance ($T = I/I_0$)

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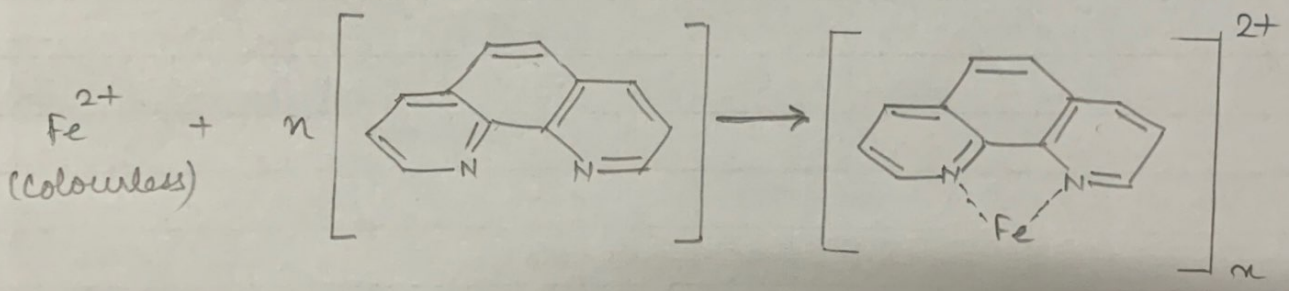
EXPERIMENT-9.

AIM :- Spectrophotometric determination of iron(II) with 1, 10-phenanthroline.

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

CHEMICALS REQUIRED: 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 :-



PHENANTHROLINE (COLOURLESS)

IRON-PHENANTHROLINE COMPLEX (REDDISH ORANGE COLOUR)

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

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Expt. No. _____ Date _____

Page No. _____

PROCEDURE :-

A) PREPARATION OF SAMPLES :-

1. Take 50ml volumetric flask and add 0, 1, 2, 3, 4, 5 ml. of FAS soln. in each flask. Lets name them as K, L, M, N, O, P.
2. Then add 2ml of 1, 10 - phenanthroline solution to each of these volumetric flasks.
3. Now dilute each volumetric flasks with deionised water to afford a total volume of 50ml. Stop and mix the contents well by shaking for few minutes. Allow the soln. to stand for 10 minutes.
4. The first flask to which 0 ml of FAS is added (ie. no Fe^{+2}) will serve as a blank.

B) TO DETERMINE λMAX

1. Get two cuvettes issued from lab.
2. Fill one of them with blank soln. (K) and another one of samples containing Fe. Lets say soln. P.
3. Light of single wavelength can be produced by selecting the filter on photocalorimeter usually the ranges goes from 410 nm – 700 nm.
4. Set the filter to 410 nm. Place the cuvette with blank solution, K, in the sample holder.
5. Set the absorbance to 0%.
6. Now place the second cuvette in the sample holder. Measure the absorbance of the solution. Now you have the absorbance at 410 nm for soln P.

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OBSERVATIONS :-

(i) ABSORBANCE OF THE SOLUTION AT THE HIGHEST CONCENTRATION (10X10S) AT VARIOUS WAVELENGTHS.

S.NO	WAVELENGTH	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 SOLUTION AT DIFFERENT CONCENTRATION AT λMAX.

S.NO.	CONCENTRATION(N)	ABSORBANCE
K	0	0
L	1.9×10^{-5}	0.04
M	3.9×10^{-5}	0.09
N	5.9×10^{-5}	0.13
O	7.9×10^{-5}	0.17
P	9.9×10^{-5}	0.20
X	unknown	0.12

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Date _____

Expt. No. _____ Page No. _____

7. By changing the filter to next wavelength each time, repeat steps 4-6. You need to set the absorbance to zero with K everytime you change λ with filter.

8. Now, you have absorbance of soln. P, over a range of wavelength from 410 nm – 700 nm. You will notice that graph b/w absorbance and wavelength takes an inverse parabola shape with λ max around 500 or 480 nm.

c) MEASUREMENT FOR ABSORBANCE FOR SOLUTIONS L to P AT λMAX.

1. Set filter paper to λmax obtained at B.

2. Set absorbance to zero.

3. Measure absorbance for solutions L to P now at λmax. Don't disturb filter in between.

4. Now measure absorbance of unknown sample provided to you.

5. Plot absorbance Vs concentration for samples L to P. Connecting max points draw a straight line passing through origin.

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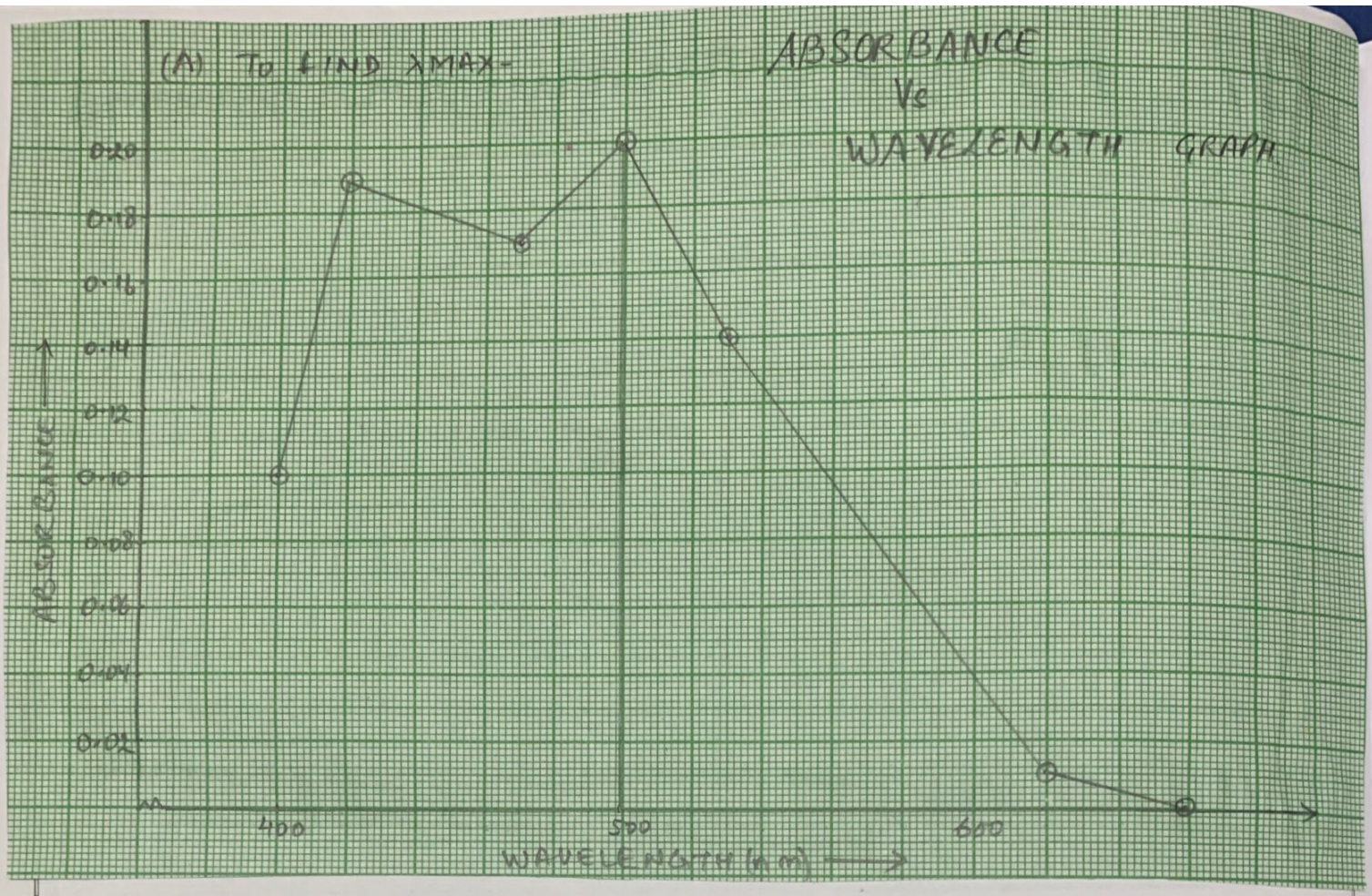
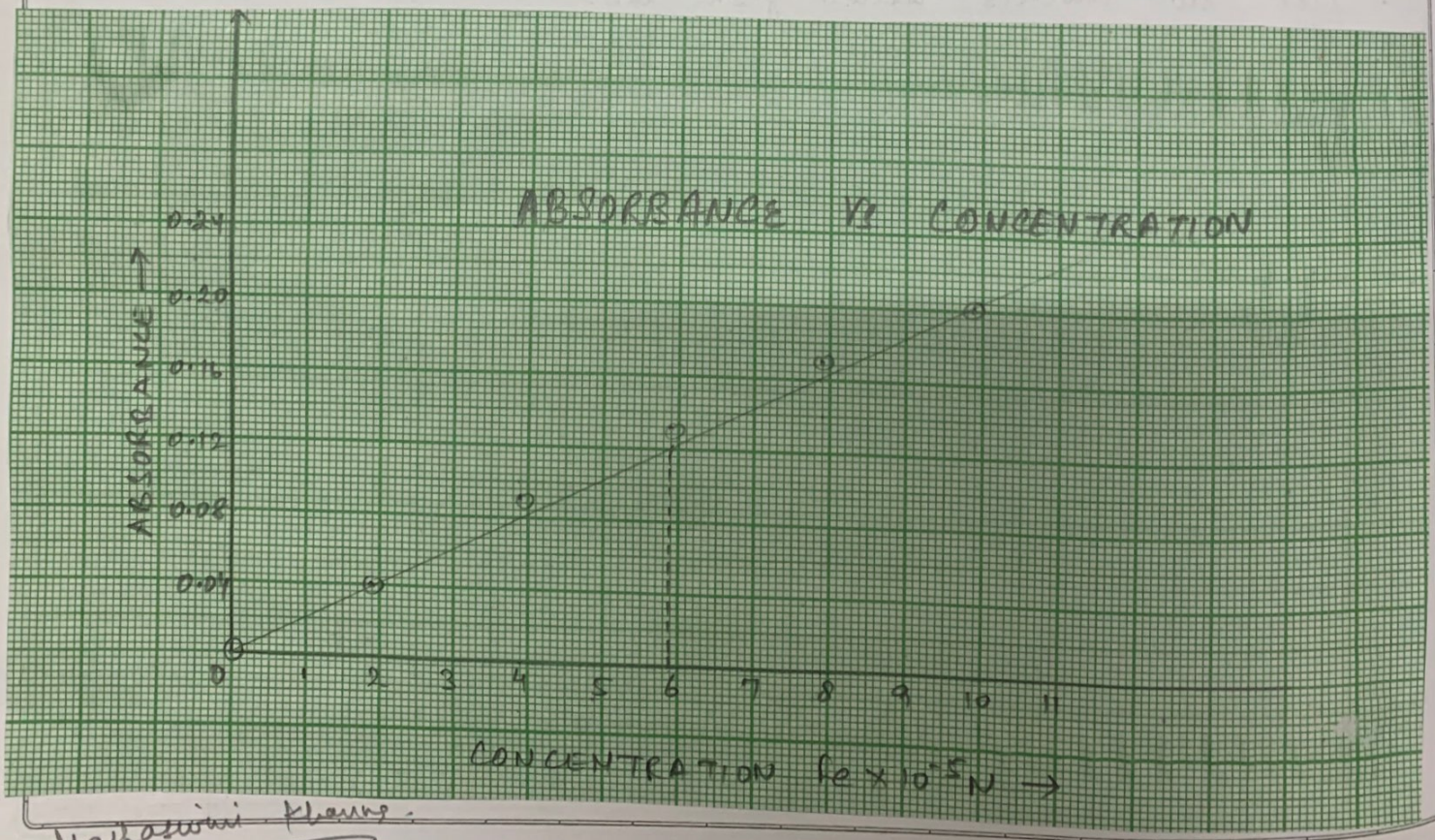
(A) TO FIND λMAX-



(B) TO FIND UNKNOWN CONCENTRATION-



Yashaswini Khanna

Date _____

Expt. No. _____ Page No. _____

6. Using absorbance value for unknown find out its concentration.

GRAPH (OBSERVATION) :-

The conc (x) can be determined from its absorbance value from the graph. A soln. with Fe concentration of 2×10^{-5} N contains 10 ug of iron. Hence the content in ug can be calculated.

RESULT :- Fe content in the unknown sample is 29.5 ug of Fe.

PRECAUTIONS :-

1. Remove the funnel after fitting the burette with FAS solution.
2. Stopper the flasks and mix the contents well by shaking vigorously for few minutes.
3. Set the absorbance to zero with blank (K) everytime you change the wavelength with filter.
4. Place the cuvette in one particular direction only each time.

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RESULT:-

The Fe content in the unknown sample is

μg of Fe 29.5 μg of Fe.

(i.e., $\frac{10}{2 \times 10^{-5}} \times 5.9 \times 10^{-5}$) μg of Fe.

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