Experiment - 9

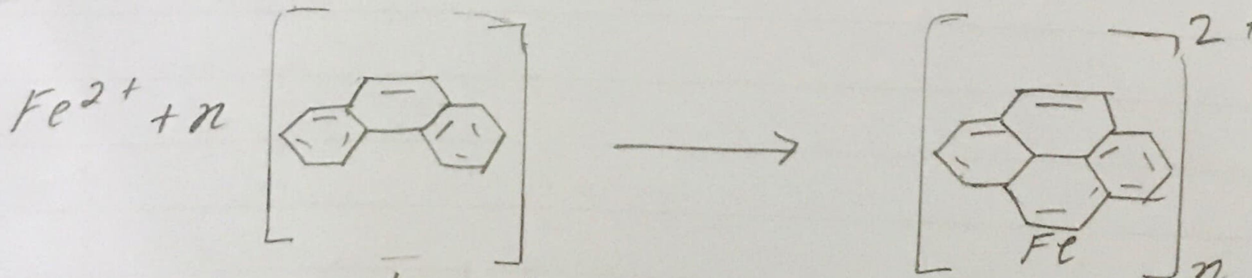
Aim :- Spectrophotometric determination of iron with 1, 10, Phenanthroline.

Apperatus: Burette, volumetric flasks (50 ml), curettes, funnel, burette, stand clamp and colorimeter.

Chemicals: Mohr's salt soln ($FeSO_4(NH_4)_2SO_4 6H_2O$) 1, 10 - phenanthroline, acetic acid solution acetate buffer of pH 4.5 and buffer of pH 4.5 and H_2SO_4

Indicator : Phenanthroline.

End color : Reddish - orange colour



colorless phenanthrolin (colourless) Iron - phenanthrolin comp (Reddish orange colour)

n = no. of phenanthroline molecules reacting with Fe^{2+}

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

Apparatus : Burette, volumetric flasks (50 ml), cuvettes funnel, burette stand, clamp and colorime

Chemicals : Mohr's salt solution (ferrous ammonium sulpate $FeSO_4 (NH_4)_2 SO_4 6H_2O$), 1, 10- phenan-throline, hydroxylamine hydrochlor acetic acid - sodium acetate buffer of PH 4.5 and sulphoric acid (H_2SO_4)

Theory : Iron (II) reacts with 1, 10 phenanthroline to form an orange red complex $[C_{12}H_8N_2]_3$ The colour intensity is independent of the acidity in pH range 2-9. If iron (III) is present it can be reduced with hydroxy-lamine hydrochloride. The absorbance is mentioned at a wavelength of 515 nm. The variation of 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 sample solv, some light is absorbed and the intensity of the transmitted light is decreased. The decrease

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Fe concentration in the flask.

K	0·0 N
L	$2·0 \times 10^{-5}$ N
M	$4·0 \times 10^{-5}$ N

N	$6·0 \times 10^{-5}$ N
O	$8·0 \times 10^{-5}$ N
P	$10·0 \times 10^{-5}$ N

Observation

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

S. No	Wavelength (nm)	Absorbance
P1	400	0·10
P2	420	0·19
P3	470	0·17
P4	500	0·20
P5	530	0·14
P6	620	0·01
P7	660	0·00
P8	706	0

$\lambda_{max} = 500$ nm

in intensity of light is proportional to the thickness of the absorbing medium and the concentration of solution. This may be expressed by Lambert Beer law.

where c is the concentration of the solution expressed in mol/l and 'ε' is a constant characteristic of the solute and the wave-length of light, ε is called the molar extinction cofficient. 'A' is absorbance or optical density (D) of solution, b is 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 these volumetric flasks as K, L, M, N, O and P

2. Then add 2ml of 1, 10 - phenonthroline solution to each of those volumetric flask.

3. Now dillute each volumetric flask with deionised water to afford a total volume of 50ml (by filling the these flasks upto the mark) stopper the flasks and mix the contents well by shaking

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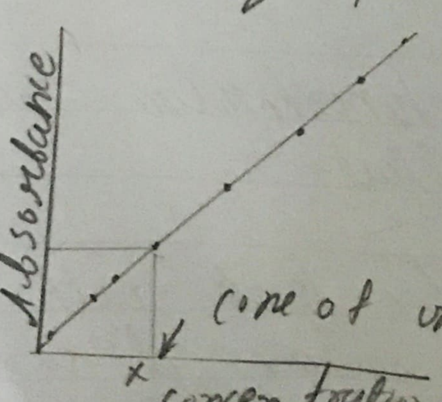
(iii) Absorbance of the Solution at different concentration at λ_{max}

S.no	Concentration (N)	Absorbance
K	0	0
L	1.7×10^{-5}	0.04
M	3.7×10^{-5}	0.09
N	5.7×10^{-5}	0.13
O	7.7×10^{-5}	0.17
P	9.7×10^{-7}	0.20
X	unknown	0.12

A Solution with Fe concentration of 2×10^{-5} N contains 10 ug of Fe (as used in this exp)



The concentration of unknown sample comes out to be 5.2×10^{-5} N

∴ The Fe content in the unknown / given sample is 26.1 ug of Fe $\left(i.e. \frac{10}{2 \times 10^{-5}} \times 5.2 \times 10^{-5}\right)$ mg of Fe 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)

B To determine the λ max

1. Get the two cuvettes issued from the labotary staff.

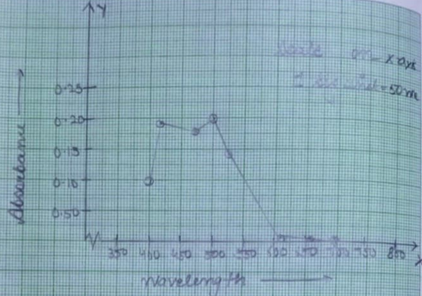
2. Fill one of them with the blank solution (K) and another one of the samples containing. Fe. let's say solution. P.

3. Light of single wavelength can be produced by selecting the filter on the photocolorimeter usually the range goes from 410 nm - 700nm

4. Set the filter to 410nm. Place the cuvette with blank solution, K, in the sample holder

5. Set the absorbance to 0 %

6. Now place the second cuvette filled with blank solution, K, in the sample holder measure the absorbance of the soln. Now you have the



Y
Scale on X axis
1 big unit = 50 nm
0.25
0.20
0.15
0.10
0.50
Absorbance
350
400
450
500
550
600
650
700
750
800
X
Wavelength

absoobance of 410 nm for Sol. P.

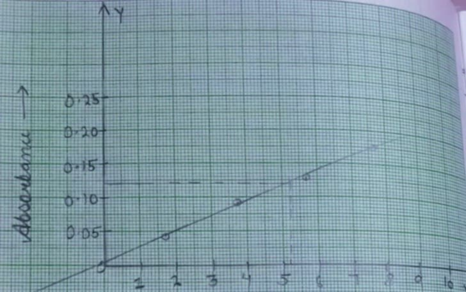
7. By changing the filter to marked wavelength each time, repeat steps 4-6. You need to set the absorbance to zero with blank (k) every time you change the λ with filter.

8. Now, you have absorbance of solution P, over a range of wavelength from 410 nm - 700 nm. You will notice that graph between the absorbance and wavelength takes an inverse prabola shape, with a maximium absorbance around 500 nm or 480 nm. This is λ max.

C Measurement for Absorbance for Solutions L to P at λ max.

1. Set the filter to λ max obtained in part 8.
2. Set the absorbance to 0 using your blank space (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 absoobance for an unknown sample provided to you.

5. plot absorbance vs concentration for samples L to P.


Y
Absorbance →
0.25
0.20
0.15
0.10
0.05
0
1
2
3
4
5
6
7
8
9
10

Connecting maximum points, draw a straight line ideally passing through origin.

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

GRAPH (OBSERVATION) : The concentration (x) can be determined from its absorbance value from the graph.

A solution with Fe concentration of 200×10^{-5} N contains 10.00 ug of Fe (as used in this exp) Hence knowing the Fe concentratio of unknown samples, their Fe content in ug can be calculated.

RESULT : The Fe content in the unknown/given sample is 26.1 ug of Fe.