CHAPTER-III

PHOTOMETRIC DETERMINATION OF COBALT (II) WITH 6-METHYL 2-CHLOROQUINOLINE 3-CARBALDEHYDE THIOSEMICARBAZONE

INTRODUCTION :

In the Sixteenth Century, the term Kobold, probably derived from the Greek κοβαλοℓ , goblins, was applied to certain ores in the Hartz Mountains which, when roasted, not only failed to yield copper but emitted troublesome and dangerous fumes from the associated aresenic. Modifications of this local designation soon spread over Europe, and the alchemical literature of that period has references to Kobolt, Cobalt etc. We cannot be certain, however, that these terms, in all cases, referred to Cobalt, since a good deal of confusion over Cobalt, zinc and arsenic minerals was exhibited even by that scholarly Rehaissance expositor of mining and metallurgy, Georgius Agricola[1].

When Cobalt ores were roasted to eliminate most of the sulfur and arsenic, they yielded a mixture of crude cobalt oxide and sand, called Zaffre. Fusion of the latter with potassium carbonate, or occasionally with glass, produced smalt, essentially a potash silica glass coloured blue with cobalt. For a long time, the alchemists believed the colour was due to arsenic or bismuth, but in 1742 Brandt showed that the effect was derived from a new demi-metal, cobalt rex. About 1780, Bergman and others studied the properties of cobalt and established its elemental character.

In the latter part of the Seventeenth Century, invisible inks attracted considerable attention, and it was inevitable that the colour changes of cobalt salts should be utilized for this purpose. In 1705, a colourless solution of bismuthiferous cobalt was used to give a green colour on heating. For a long time, the colour phenomena in these impure products were alleged to be caused by the presence of other metals, but finally the effects were correctly attributed to cobalt.

Although the beginning of cobalt chemistry and metallurgy may be said to date from the middle of the sixteenth century, the element has been found in some ancient blue glazes and glasses. A few statuettes have revealed that a cobalt base colouring agent was used by the Egyptians about 1300 B.C., and perhaps much earlier, but the blue of most old Egyptian glazes was due to copper. The blue of some early specimens of Persian glass beads, dating from about 2250 B.C. is due to cobalt; the element is also found in some specimens of ancient Roman and Venetian blue glass. It is certain that cobalt was used in pottery of Persia and Syria in the early centuries of the Christian Era. The use of cobalt blue in China has been authenticated for atleast as early as the T'ang Dynasty 618-906 A.D. Since ores of copper are more abundant and widely distributed on the earth's surface than those of cobalt, it is not surprising that most of the ancient blue glasses and glazes were coloured with copper compounds.

Towards the close of the nineteenth century, the small world output of cobalt, derived chiefly from various German, Norwegian and Hungarian deposits which has been worked for very long periods, was greatly augmented by the development of oxidized cobalt ores in New Caledonia. The latter deposits were discovered by Garnier in 1864, and the mines were opened about ten years later. New Caledonia remained the principal source of cobalt for some years until the Silver-Cobalt ores of Ontario, Canada were exploited, around 1903. It is probable that in the period 1880-1905, New Caledonia furnished about 1,00,000 tons of ore averaging 3.0 - 3.5 percent Co[2]. In 1958, after a lapse of nearly half a century, New Caledonia reappeared in world Cobalt production statistics.

The rich arsenide ores around the town of cobalt in northern Ontario yielded a rapid increase in production from 16 tons of cobalt in 1904 to a maximum of 1,553 in 1909, thereafter declining to 337 in 1917[3,4]. Canadian production in the period 1920-1940 ranged from about 150 to 550 tons per annum, depending on world economic conditions. During and immediately following World War-II, the Canadian output of cobalt was low; in recent years, however, it has shown a steady increase as a recovered by product of the nickel industry, and Canada is once again one of the principal world producers.

About 1920, Union Miniere du Haut Katanga began to extract cobalt from their Copper-Cobalt ores of Katanga province in the Belgian Congo and in 1926 became the leading world producer. The Belgian Congo still retains this position by a wide margin.

With the opening of the great Copper deposits of North ern Rhodesia, around 1930, it was found that Cobalt occurred in a small but fairly constant concentrations in the copper ores of one producer, Rhokano Corpo ration Limited, at Nkana. Since then, Northern Rhodesia, however, and this territory retains a large measure of local autonomy as a distinct political unit, the familiar name of Northern Rhodesia will continue to be used throughout this book, except in a few statistical tables.

Spectrographic procedures were originally developed for very small quantities of metals. This is still their greatest area of usefulness, but in recent years various techniques and equipment have been developed to extend the range of concentrations which can be profitably determined by spectrography.

Measurement of the Cobalt content of animal tissue, where amounts are in the millimicrogram range, possess unusual problems. Cobalt in human blood, about 1-part per billion, has been determined by ashing, removing iron and alkali chlorides, separating cobalt on the anion exchange resin Dowex 1-X8. eluting, and determining spectrographically[5].

Another procedure for determining Cobalt in animal tissue involves ashing; separation of Cobalt with α-nitroso β-naphthol; collection in a mixture of aluminium oxide, lithium chloride, and graphite as a constant base material for direct current are used in spectrography[6].

Strasheim and Camerer[7] found that the cathode layer arc excitation method was more suitable than the anode arc method for determining 0.1 = 2.9 ppm Cobalt in plant materials. The concentration procedure proposed by Metchell was employed.

Investigations on the spectrographic analysis of residual Cobalt in Steel[8] and stainless steel[9] have been reported. In the analysis of high purity iron[10], Cobalt was determined spectrographically on the aqueous layer after separation of iron by an ether extraction. Standen[11] has given the concentration of many elements which may interfere with the spectrographic determination of traces of Cobalt.

The detailed summary of reported photometric methods for Cobalt determination is given in Table 3.1.

Table 3.1 : Review of the Methods for the Photometric Determination of Cobalt(II)

Reagent	pH	MAX λmax nm	Molar extraction Coefficient, sensitivity range ppm	Interferences and remarks	Reference
1	2	3	4	5	6
O-(2-thizolyazo-2-Napthol)	5.0	618	ϵ = 24,000	-	12
Benzoyltrifluro acetone	6.0	390	-	Ca(II), Ni(II), Fe(II), interfere	13
8-Mercaptohydroxy-quinoline	7.3-8.2	352.7	-	Ni, Citrate, tartarate interfere	14
8-Mercaptohydroxy quinoline	-	470	-	Ni, Fe interfere	15
Di-2-Pyridyl keto-oxime	-	388	-	Ni(II), Cu(II), EDTA interfere	16
Acenophthene quinone monoxime	8.0-9.5	400	Sensitivity is 0.005 μg cm^{-2} 6-18 ppm	-	17
STTA	-	490	-	Ni, Cu, EDTA interfere	18
Thenoyltrifluoro acetone (TTA)	3.2-3.4	460	3-12 ppm	-	19

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1	2	3	4	5	6
2-(5-Chloro-2-Pyridytazo) -5-diethylaminophenol	5.9	580	ϵ = 106.000	-	20
Nitroso-R-Salt	-	-	-	-	21
β-Mercaptohydroxy and β-Mercaptocinnamanilide	9.0	640	10-100 ppm	Fe^{+3}, Cu^{+2}interfere	22
5-(2-hydroxy-1-naphthyl azo)Pyrazole-4-Carboxylic acid.	9.3	580	0.8 - 6.4 ppm	CN^-, EDTA, Citrate, oxalate, interfere	23
Ppcraminazo-4-Cyclohexyl resorcinol	2-3	555	ϵ = 28,000	-	24
Nitroso-R-Salt-4-diphenyl guanidine	7.8	520	-	Ni, Cu, Fe, Ti, Sn, In does not interfere	25
Picolinaldehyde 4-Phenyl-3-thiosemicarbazone	1	430	-	Ag,Pb,Fe,Cu(II),Ni,Zn, Hg,Cd,Au interfere	26
Furil α-monaxime	2.5-7.0	405-410	-	Ni,Cu,Mn,Pd,Pt interfere	27
Phenylazobenzoldoxime	-	530	0.6-3.5 ppm	Cu(II), Ni, Pd(II), EDTA interfere	28
2-Carboxy-2-hydroxy, 3'-5' dimethyl azobenzene -4-sulphonic acid (HDMAS)	7.5	630	-	Simultaneous Ni and Co determination	29
Eriochrome black-T	-	580	ϵ =50,000 S.sensitivity is 1.18 µg cm^{-2} 0.03-0.9 ppm	$C10_4^-$, No_3^-, EDTA interfere	30

1	2	3	4	5	6
4',5'-dibromo-2'-7'-dinitrofluorescein	4.6	560	ϵ = 50,000 0.06 - 1.68 ppm	-	31
5-(4-diethylamino-2-hydroxyphenylazo)-1,2,4-triazole 3-carboxylic acid	-	525-540	-	-	32
n-Butyl xanthalate	4.3-8.1	355	ϵ=9.43x10^{3} S.Sensitivity is 0.0062 ug cm^{-2}	Simultaneous determination of Ni and Co in alloy	33
n-butyl Xanthalate	4.3-8.1	355	ϵ=9.43x10^{3} S.Sensitivity is 0.0062 μg cm^{-2}	Simultaneous determination of Ni and Co in alloy	34
1-(2-Pyridylazo)-2-Naphthol (PAN)	-	580	Beer's law obeyed upto 0-25 ug/50 ml	-	35
5-({5-Chloro-O-2-pyridyl} azo)-2,4 diaminotoluene	-	570	1.20 x 10^{5} Beer's law obeyed upto 0-10 μg Co/25 ml	Desirable sensitivity and selectivity	36
1-Nitroso-2-Naphthol	6.8-8.4	-	-	-	37
0-(benzothiazol-2-ylaxo phenthren-g-01	6.7	560	-	Ni(II), Cu(II), Zn(II), Pd(II) and Cd(II) interfere.	38
N-N-dimethyl-4-(2-pyridyl-azo)-m-phenylene diamine	2.4	603	ϵ=1.14 x 10^{5} Beer's law obeyed upto 0.01-0.5ppm	Cd(II), Cu(II) interfere	39
Cyclohexane-1,3 dione bisthiosemicarbazone monohydrochloride	-	600	Beer's law obeyed upto 0.5 - 5 ppm	Several ions interfere	40

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1	2	3	4	5	6
4-hydroxy-3-nitrosonaphthalene-1-sulphonic acid	-	307	ϵ=65,000 Beer's law obeyed upto 0.5-15 um	-	41
Sodium alizarin sulphonate	7.0	600	ϵ=6000 Beer's law obeyed upto 0-10.6 ppm	-	42
5-(4-diethylamino-2-hydroxyphenylazo)1,2,4-triazole-3-Carboxylic acid)	2.4 M HCl	510	Beer's law obeyed for 0.04-0.4 $\mu g\ ml^{-1}$	-	43
2-(5-Chloro-2-Pyridylazo) 5-dimethylamino aniline	6	570	ϵ= 114,000 Beer's law obeyed for 0 to 0.5 ppm	-	44
2,4-dichlorobenzyl-triphenyl phosphonium tetrathiocyanate	-	625	ϵ = 1800	Fe(III) interferes	45
4-(2-amino-3-hydroxy 4-pyridylazo)benzene sulphonic acid.	8-9.7	540	ϵ=34,100 S.Sensitivity is 1.7 $\mu g\ cm^{-2}$ Beer's law is obeyed for 0.16 to 1.5 ppm.	-	46
Salicylaldehyde thiosemicarbazone	5	400	ϵ =11,000 Beer's law is obeyed for 0.3 to 6 ppm.	-	47
3-(4-Sulphophenylazo chromotropic Acid (SPADNS)	5.5 to 6	580	ϵ= 14,000 S.Sensitivity is 0.06 $\mu g\ cm^{-2}$	Be, Zn, Cd, Zn, Al interfere	48
ethylene diamine-N.N' dipropionic acid	7.5-9	550	Beer's law is obeyed for 36-195 $\mu g\ ml^{-1}$	Cr(VI),Mn(II),Cu(II) Fe(III) interfere	49
2,6-diamino-3-(4-methyl 2-thiazolylazo)pyridine	-	610	Beer's law obeyed for 0.2 to 1.5 ppm	-	50

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1	2	3	4	5	6
4-Methyltetrahydrazo benzofurano(6,7-b)-Coumarin	8 or 10	380	ϵ=19.100 Beer's law obeyed for 2.0 to7.5 ppm of Co.	-	51
2-6'-Sulphobenzoyl) Pyridine thiosemicarbazone	-	420	Beer's law obeyed for 1 to 3.5 ppm	-	52
2-(5-bromo-2-Pyridylazo) 5-diethylamino-phenol	Acidic	580	ϵ= 1.03 x 10^5 Beer's law obeyed for 0-12.5 ug Co/25 ml.	Commonly other elements present do not interfere	53
Pyridine-2-Carboxaldehyde-2-pyridylhydrazone	-	350-390	-	-	54
Pyridoxal thiosemicarbazones	-	440	-	Pd(II),Au(III) interfere	55
Salicylaldehyde thiosemicarbazone	-	400	-	V(V) interfere	56
1-2Diaminoanthraquinone	-	690	-	Interference by 24 cations	57
di-2-pyridyl(ketone) pyrimidin-2-ylhydrazone	-	460	=29,500 Beer's law obeyed upto 2.1 ppm	-	58
		440	=31,500 Beer's law obeyed upto 2.2 ppm	-	
(4-(6-Chlorindazol-3-ylazo) -3-hydroxynaphthalene 2,7-disulphonic acid.	-	638	Sandell Sensitivity is $\simeq$ 2 µg cm^{-2}	Cu^{+2}, CN^- interfere	59

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1	2	3	4	5	6
2-amino-5-nitroso-1,4,5,6 tetrahydropyrimiline 4,6-dione	4.8 to 4.6	375	$\epsilon = 5.3 \times 10^4$ Beer's law obeyed upto 0 to 2 ug ml^{-1} Sandell sensitivity 1.11 ug cm^{-2}	Cu^{+2}, Fe^{+3} interfere	60
N-Methylaminothioformyl-N'-phenyl hydroxylamine	8	470	$\epsilon = 16,500$ Beer's law obeyed upto 6 to 60 um	Zn,Cd,Pb, do not interfere	61
3-hydroxy picolineal dehyde azine	4.3 to 4.6	545 570	$\epsilon = 3.04 \times 10^4$ $\epsilon = 2.66 \times 10^4$	-	62
Salicylaldehyde azine	7.8 to 8.1	530	$\epsilon = 1356$ Beer's law is obeyed for 4-11 ppm	-	63
Picolinealdehyde azine	7.4 to 9.5	400	$\epsilon = 3560$ Beer's law is obeyed for 3-11 ppm	-	64
Furidioxime	8	350	$\epsilon = 1.84 \times 10^4$	-	65
2,2',2"-Terpyridine	-	510	$\epsilon = 2900$ Beer's law is valid for 0.5 to 50 ppm	Cu,Ni,Fe,CN interfere	66
Sulphosalicylic acid	9.5 to 10.5	380	$\epsilon = 4265$ Beer's law is valid for 2.8 ppm	-	67
2,2'-dipyridylketone 2-Pyridylhydrazone (DPPH)	2	514	$\epsilon = 3.9 \times 10^4$ Sandell sensitivity 0.0015 ug/cm^2. Beer's law is obeyed for 4μg/ml.	-	68

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1	2	3	4	5	6
Isonitrosothiocamphor	-	350	$\epsilon = 1.7 \times 10^4$ Sandell sensitivity is 0.0025 ug cm^{-2}. Beer's law is obeyed for 0.2-1.5ppm	Tolerates thousand fold excess of diverse ions	69
Trifluro ethylaxanthalate	3.8 to 10	352	$\epsilon = 2.9 \times 10^4$ Beer's law is obeyed for 0.0-1.7 ug Co/ml.	-	70
3-Bromo-2-hydroxy-5-methyl acetophenone oxime (BHAMAO)	7.0 to 8.5	400	$\epsilon = 1190$ Beer's law is obeyed for 2-31 ppm. Sandell sensitivity 0.049 ug	-	71

The present work accounts for spectrophotometric determination of Cobalt(II). Cobalt(II) gives yellow coloured complex with 6-methyl, 2-chloro quinoline, 3-carbaldehyde, thiosemicarbazone. For cobalt(II) optimum pH is 6 and λ max. is 400 nm. Beer's law is obeyed upto 6 ppm for cobalt(II). The effect of pH, reagent concentration and diverse ions have been studied. The molar absorptivity and Sandell sensitivity are 2.626×10^{4} L $mole^{-1}$ cm^{-1} and 0.01 μg cm^{-2} respectively. For Cobalt(II) the dissociation constant of complex Co(II) 6-ME-QAT is 7.960×10^{-6}.

EXPERIMENTAL

1. Standard Cobalt Solution :

Standard Cobalt solution 1 mg/ml was prepared from Cobaltous sulphate and the solution was standardized with distilled water volumetrically[76]. Further dilution for experimental purposes were made by diluting the stock solution with distilled water.

2. Reagent Solution :

0.055 gm 6-Me-QAT was dissolved in DMF and water (1:1) and diluted to 100 ml. The molarity of solution is 2.676 x 10^{-4} M. The solution is found to be stable for more than a week.

3. Recommended Procedure :

To an aliquot of solution containing upto 2.5 ppm of Cobalt, add 1.2 ml of 2.676 x 10^{-4} M reagent solutiion and a buffer solution of pH 6. Dilute this to 10 ml with DMF and water (3:2) in a volumetric flask. Measure the absorbance against reagent blank.

RESULTS AND DISCUSSION

Spectral Characteristics :

Figure 3.1 shows the absorption spectrum of the Cobalt(II) 6-Me-QAT complex containing 1.694 x 10^{-3} M of Cobalt and 2.676 x 10^{-4} M reagent at pH 6 using reagent blank Absorption measurements were made in the spectral region 370 nm to 700 nm. and recorded in table. From the group it was found 400 nm will be suitable wave length for the Cobalt determination. The molar absorptivity of the complex is 0.8971 x 10^{3} L $Mole^{-1}$ cm^{-1} at 400 nm.

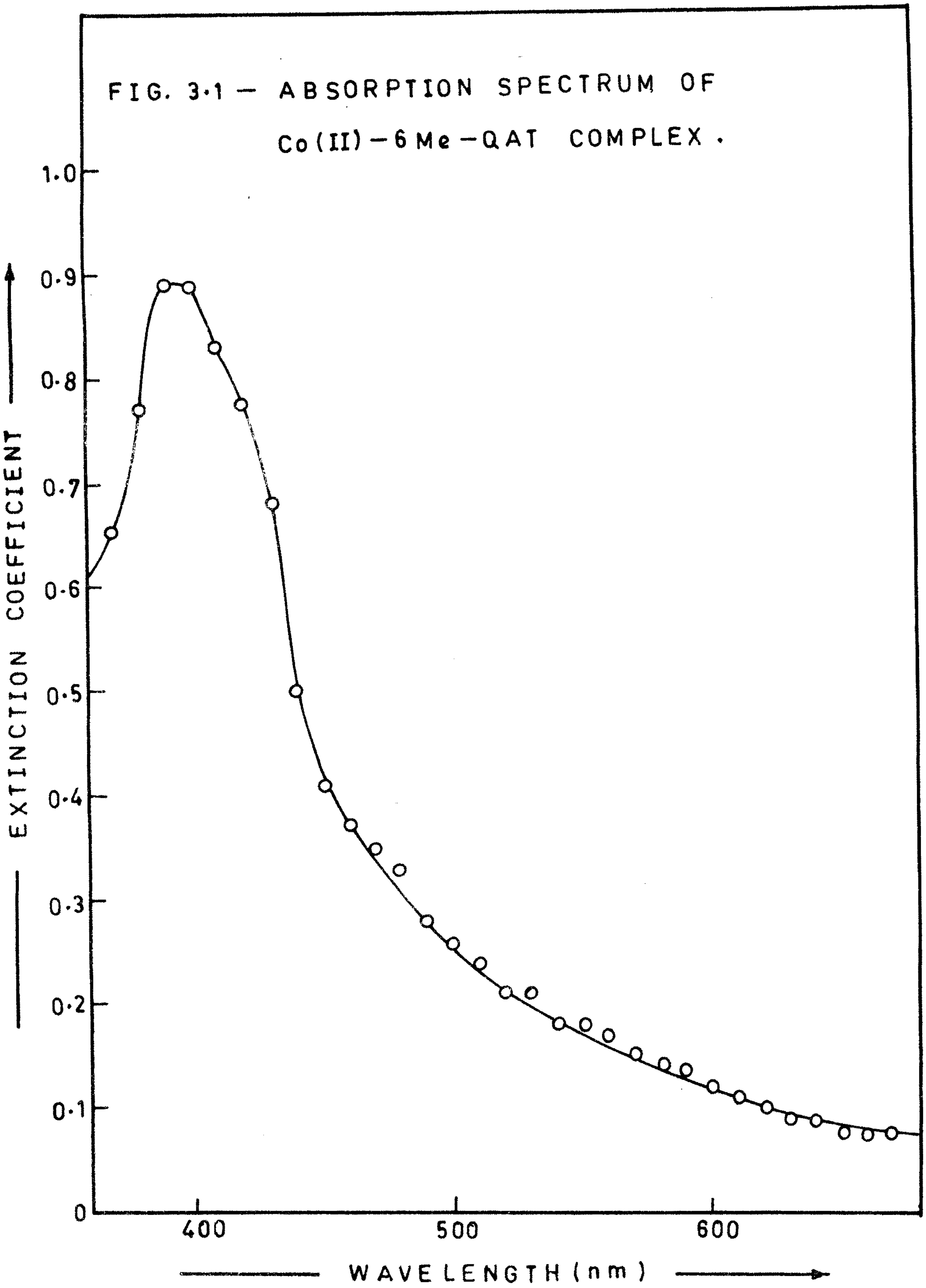
Table No. 3.2

Molar Extinction Coefficients of Co(II) 6-Me-QAT Complex

λnm	Molar Ext.Coefficient of Complex 10^{4} L $Mole^{-1}$ cm^{-1}
370	0.6578
380	0.7775
390	0.8971
400	0.8971
410	0.8373
420	0.7775
430	0.6877
440	0.5083
450	0.4186

nm	Molar Ext.Coefficient of Complex 10^4 L $Mole^{-1} cm^{-1}$
460	0.3708
470	0.3588
480	0.3349
490	0.2811
500	0.2691
510	0.2392
520	0.2153
530	0.2093
540	0.1854
550	0.1794
560	0.1674
570	0.1495
580	0.1435
590	0.1375
600	0.1235
610	0.1196
620	0.1136
630	0.1016
640	0.09569
650	0.09569
660	0.08971
670	0.08373
680	0.07775
690	0.07775
700	0.07775

FIG. 3·1 — ABSORPTION SPECTRUM OF Co(II)-6Me-QAT COMPLEX.



Effect of Time on Absorbance :

In order to study effect of time on the absorbance of Cobalt(II) 6-Me-QAT complex containing 1.694 x 10^{-3} M Co(II) at pH 6, the absorbance measurements were recorded at different time intervals at 400 nm. It was observed that there is instantaneous development of colour and the absorbance remains constant for 24 hours and decreases after that. Hence the complex is stable for atleast 24 hours.

Effect of Reagent Concentration :

Solutions containing the same amount of Cobalt (1.694 x 10^{-3} M) but different amounts of reagent varing from 0.2 to 1.0 ml of 2.676 x 10^{-4} M reagent solutions were prepared. The pH 6.0 buffer was added and the solution was made upto 10 ml with DMF : Water (3:2). Absorbance measurements were recorded at 400 nm against the simultaneously prepared reagent blank. The data given in Table (3.3, fig. 3.2) shows that 1.694 x 10^{-3} M Cobalt solution required minimum 1 ml of 2.676 x 10^{-4} M reagent solution for maximum complexation. At higher concentration of the reagent there was insignificant increase in absorbance. However, 1 ml of 2.676 x 10^{-4}M reagent solution was employed for further studies to ensure maximum colour intensity of the Cobalt complex.

Table 3.3

Effect of Reagent Concentration on the Absorbance of ~~Co(III)~~ 6-Me-QAT Complex

(Co) = 1.694×10^{-3} M, (QAT) = 2.676×10^{-4} M pH = 6.

Reagent ml	Absorbance at 400 nm
0.2	0.01
0.4	0.04
0.5	0.11
0.6	0.19
0.7	0.25
0.8	0.33
0.9	0.37
1.0	0.40

Effect of pH :

A series of solutions containing 1.694×10^{-3} M Co(II) and 2.676×10^{-4} M of the reagent, but varying in pH from 1 to 9 were prepared and the absorption measurements were recorded at 400 nm. The results are summarised in the 3.4. It was observed that maximum absorbance was obtained at the pH 6 The absorbance of the solutions decrease at higher or lower pH values than this which is shown in fig. 3.3. Hence the pH 6 was maintained in further studies.

Table 3.4

Effect of pH on the absorbance of Co(II) 6 Me-QAT Complex

Co(II) = 1.694×10^{-3}M   6Me-QAT = 2.676×10^{-4}M

pH	Absorbance at 400 nm
1	0.05
2	0.275
3	0.3
4	0.4
5	0.51
6	0.55
7	0.525
8	0.475
9	0.425

Validity of Beer's Law and Sensitivity :

The solutiions (final volume 10 ml) containing different amounts of Cobalt and the same amounts of reagents with the pH maintained at pH 6, were used for the study. The absorption measurements were recorded against reagent blank at 400 nm (Table 3.5, Fig.3.4) Beer's law is obeyed upto a concentration of 6 ppm of Cobalt (II). The Sandell sensitivity[77] of the reaction is 0.1 ng/cm^2 of Co(II). The Ringhom's[74] plot (Fig. 3.5) indicates that the optimum range is 1 to 3 ppm of Cobalt(II).

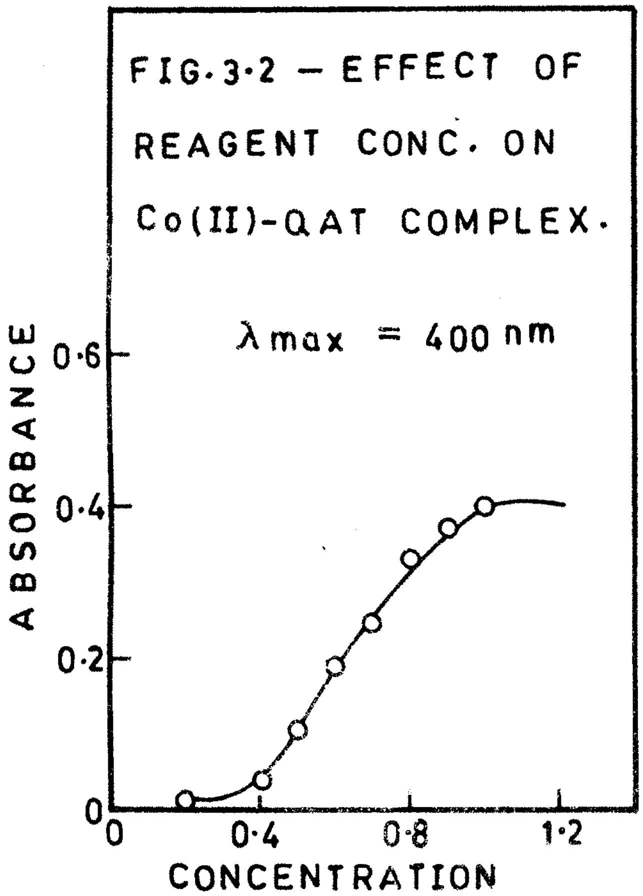


FIG. 3.2 – EFFECT OF REAGENT CONC. ON Co(II)-QAT COMPLEX.

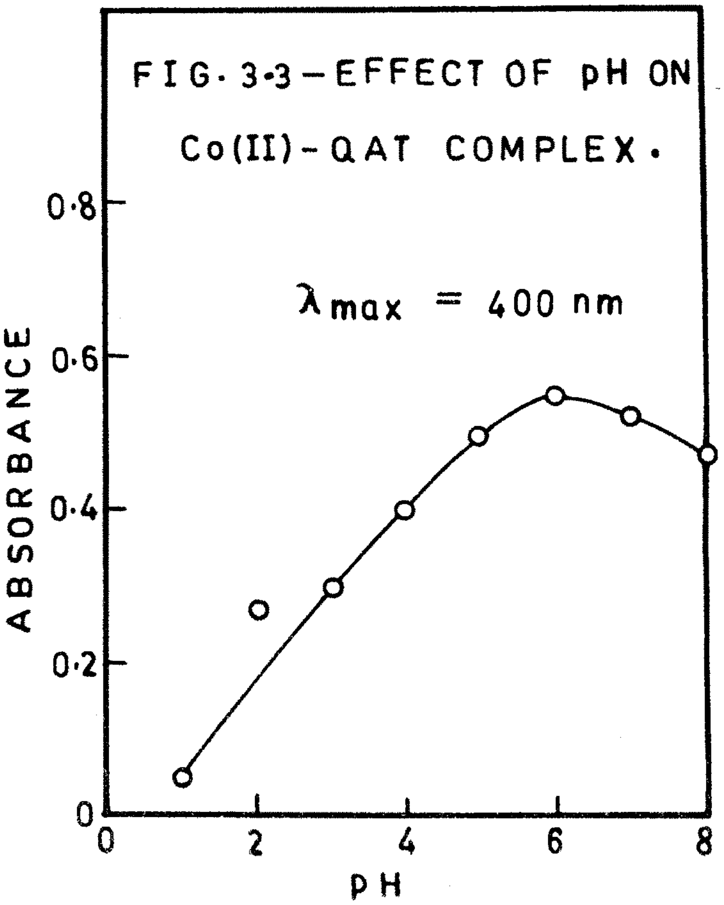


FIG. 3.3 – EFFECT OF pH ON Co(II)-QAT COMPLEX.

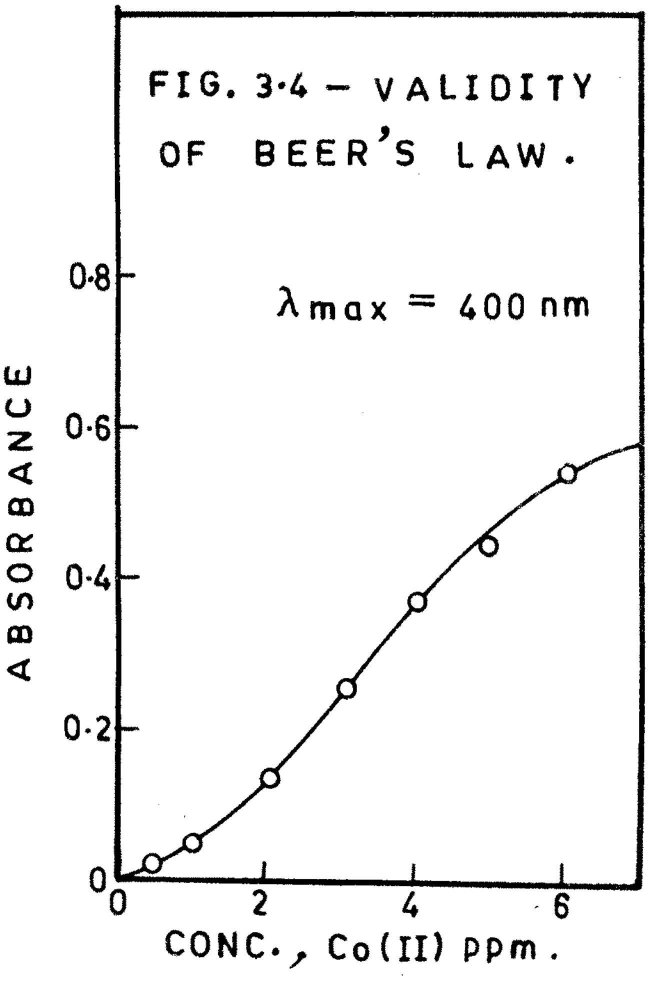


FIG. 3.4 – VALIDITY OF BEER'S LAW.

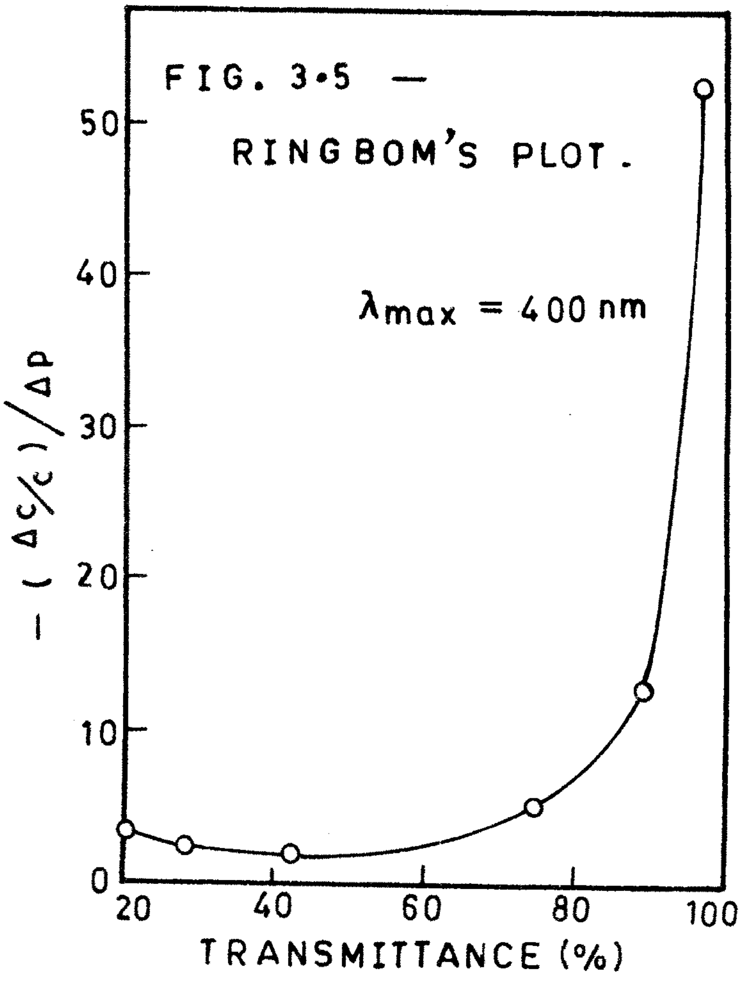


FIG. 3.5 – RINGBOM'S PLOT.

Table 3.5

Verification of Validity of Beer's Law

6-Me QAT = 2.676×10^{-4}M, pH = 6

Co taken ppm	Absorbance at 400 nm
0.5	0.01
1.0	0.05
2.0	0.13
3.0	0.26
4.0	0.37
5.0	0.54
6.0	0.65

Composition of the Complex :

The combining ratio of metal to reagent was ascertained by Job's method of continuous variations[72] and Mole ratio method[73]. For Job's method of continuous variation, a series of solution were prepared by mixing equimolar solutions of Cobalt(II) and the reagent (2.676×10^{-4}M). The pH of the solutions was adjusted to 6. The absorbance of the solutions after diluting to 10 ml in volumetric flask were recorded at 400 nm against simultaneously prepared reagent blank (Table 3.6, Fig.3.6). The plot indicates the existance of 1:1 complex with respect to metal and reagent represented as ML.

Table 3.6

Determination of the Formula by Job's method of Continuous Variation

(Co) = 6-Me QAT = 2.676×10^{-3}M, pH = 6

Metal ion ml	Reagent ml	Mole fraction of metal	Absorbance at 400 nm
0.1	0.9	0.1	0.2
0.2	0.8	0.2	0.37
0.3	0.7	0.3	0.46
0.4	0.6	0.4	0.57
0.5	0.5	0.5	0.60
0.6	0.4	0.6	0.54
0.7	0.3	0.7	0.38
0.8	0.2	0.8	0.22
0.9	0.1	0.9	0.12

The dissociation constant of the complex K is calculated from the mole ratio plot by using the following equation

$$K = \frac{\alpha C\ (n\alpha c)}{C\ (1-\alpha)}$$

where n = 1

C = Molarity of the solution of complex

and

α = degreee of dissociation defined by

$$\alpha = \frac{A_m - A_s}{A_m}$$

A_m is the maximum absorption obtained from horizontal portion of the curve and A_s is the absorbance of Stoichiometric molar ratio of reagent to Cobalt in the complex. The value of K comes out to be 7.960 x 10^{-6}.

Mole Ratio Method :

For mole ratio method solutions containing the same final metal concentration (2.676 x 10^{-3}M) and different amounts of reagent ranging from concentration were prepared keeping pH 6. Absorbance of the solutions were measured at 400 nm against reagent blank. The curve showed break at the metal to ligand ratio 1:1, confirming the results obtained by applications of Job's method of continuous variations (Table 3.7, Fig. 3.7).

Table 3.7

Determination of the formula of Co(II) 6-Me-QAT Complex by Mole Ratio Method

(Co) = 6-Me (QAT) = 2.676 x 10^{-3}M, pH = 6

Metal ion ml	Reagent ml	Reagent to metal ratio	Absorbance at 400 nm
0.4	0.2	0.5	0.38
0.4	0.4	1.0	0.66
0.4	0.8	2.0	0.87
0.4	1.2	3.0	0.97
0.4	1.4	3.5	1.02
0.4	1.6	4.0	1.07

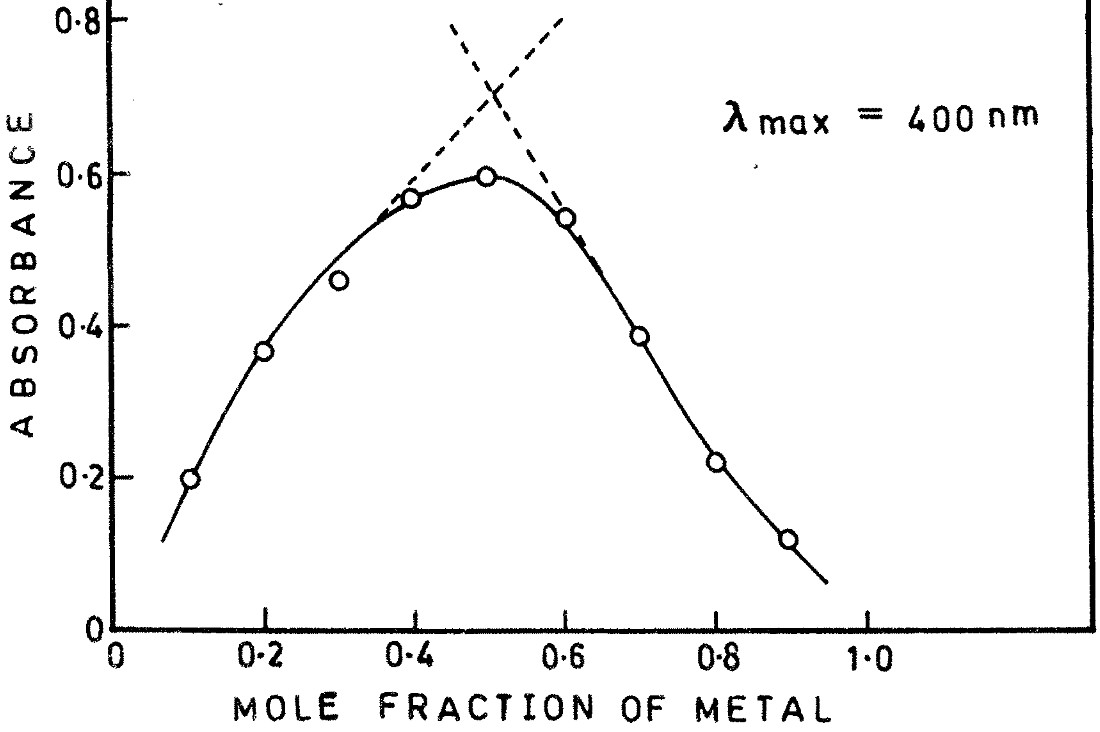


FIG. 3·6 — JOB'S PLOT FOR Co(II) – 6 Me – QAT COMPLEX.

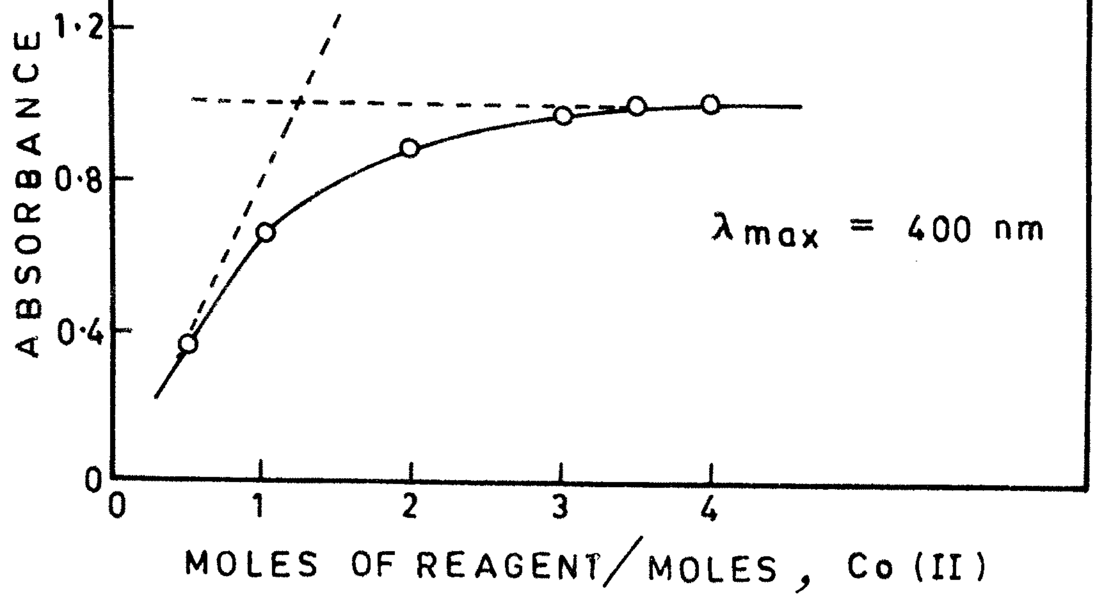


FIG. 3·7 — MOLE RATIO PLOT FOR Co(II) – 6 Me – QAT COMPLEX.

Effect of diverse ions :

The effect of diverse ions was studied using 2 ppm of Cobalt and 2.676×10^{-4}M reagent in a final volume of 10 ml at pH-6. The results indicated that Mn(II), Ni(II), Cu(II) and anions like citrate and cynide interfere strongly. The tolerance limit for various foreign ions tested has been shown in the Table 3.8.

Table No. 3.8

Effect of diverse ions

Co(II) = 2 ppm 6-Me-QAT = 2.676×10^{-4}M pH = 6

Metal ions	Tolerance limit ppm
Ca(III)	100
Mo(VI)	100
Ca(II)	100
Cu(II)	None
Mn(II)	None
Ni(II)	None
Fe(II)	None
CN^-	None
Acetate	1000
Oxalate	1000
Thiourea	1000
Citrate	None
F^-	1000
PO_4^{-3}	1000

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