

## Spectrophotometric Determination Of Nickel Piperazine Dithiocarbamate

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### Abstract:

Spectrophotometry is one of the important instrumental methods introduced in Analytical Chemistry which has its own significance even today inspite of various other sophisticated instruments developed and has been in common use in trace analysis<sup>1</sup>.

Spectrophotometric method for the determination of Ni(II) in the trace quantities was developed by using piperazine dithiocarbamate as ligand in sodium acetate - acetic acid medium at P<sup>H</sup>-6. The method developed was applied for the estimation of Ni(II) in microgram quantities in the water samples and it was found to be simple and sensitive.

**Key words:** spectrophotometry , nickel (II) , piperazine dithiocarbamate

### Introduction:

Nickel is a silver-white metallic element which occurs in nature in free state and its compounds are green in colour. The human body contains approximately 10mg of Nickel and is a dietary requirement for a number of organisms and therefore it is of vital importance to human beings also.

Several spectrophotometric methods for determining nickel based on organic reagents containing sulphur as a ligand atom have been known. The reaction in which nickel reacts with water quinoxaline-2, 3-dithiol to form a red colored complex in ammonical media has been reported by a number of investigators. Bis (di thio oxalate) chelate which can be extracted as an ion pair complex with the tri-phenyl methyl arsonium ion is studied by Cameron et.al<sup>2</sup> and pilipenko et al<sup>3,4</sup>. Di thizone and dithizone phenanthroline gives complexes with Ni, extractable into chloroform and carbon tetra chloride<sup>5,6,7</sup>.

In view of the wide range applications of the reagent (PDTC) in various instrumental methods reported<sup>8,9</sup> earlier the present work was taken up for determination of trace Ni(II) in the presence of piperazine dithiocarbamate as reagent in sodium acetate - acetic acid medium at P<sup>H</sup>-6.

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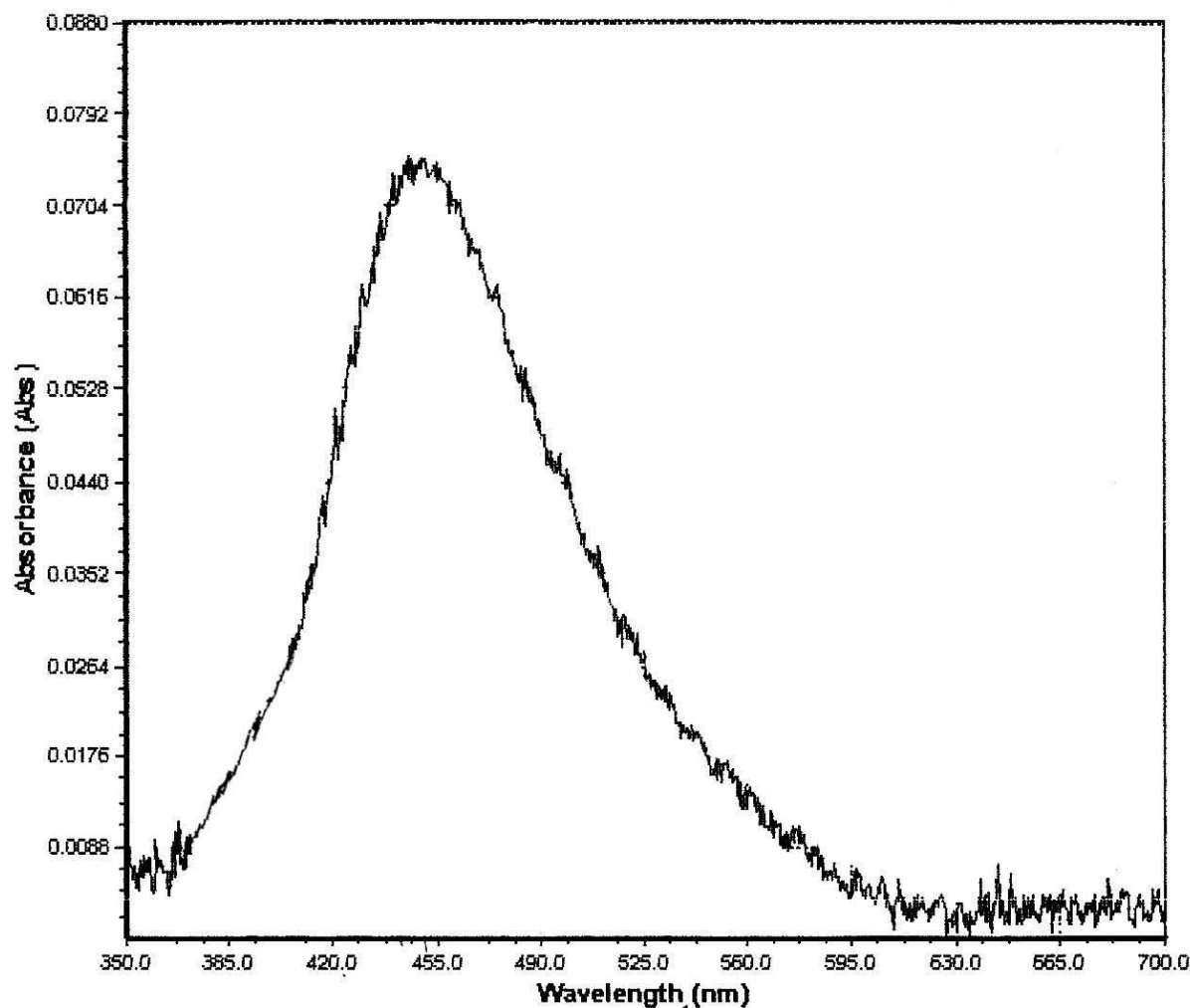
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## Experimental:

Nickel forms a yellowish brown coloured complex with piperazine dithio carbamate in sodium acetate - acetic acid medium at  $P^H-6$  with a maximum absorption at 318nm and hence the experiment was carried out at the same wavelength.

SL 191, double beam u.v-visible spectrophotometer was used for recording absorption spectrum of the solutions. Digital pH meter ELICO L1 120 provided with temperature control knob was used throughout the work. Varian spectra AA-220 atomic absorption spectrophotometer was used for the analysis of samples. Analytical grade chemicals and double distilled water have been used.

### Absorption spectrum of Ni-PDTC complex



## Results and discussion:

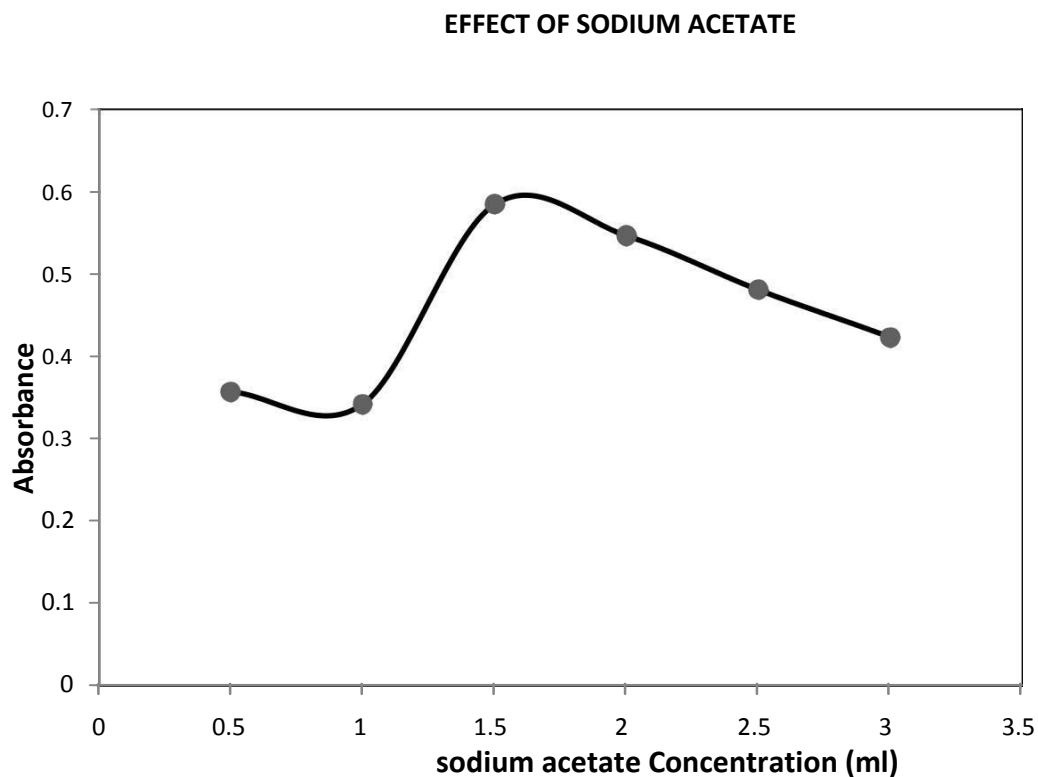
The experimental conditions for quantitative results have been developed by studying the effect of various parameters as discussed below.

### Effect of pH:

The experiments were conducted by mixing volumes of 1ml of 0.001M Ni, 1.5ml of 2M sodium acetate solution with a pH range of 5-7. It was observed that absorbance values of the metal complex increase with increase in pH and reach a maximum at pH 6 and thereafter the values decrease. Since the complex formation is quantitative at pH 6, all the experiments were carried out at this pH only.

### Effect of sodium acetate concentration:

By keeping all the factors constant including pH, the volume of sodium acetate solution was changed from 0.5ml to 3ml and its effect was studied. It was observed that 1.5ml of 2M acetate buffer shows maximum absorbance and decreases thereafter.



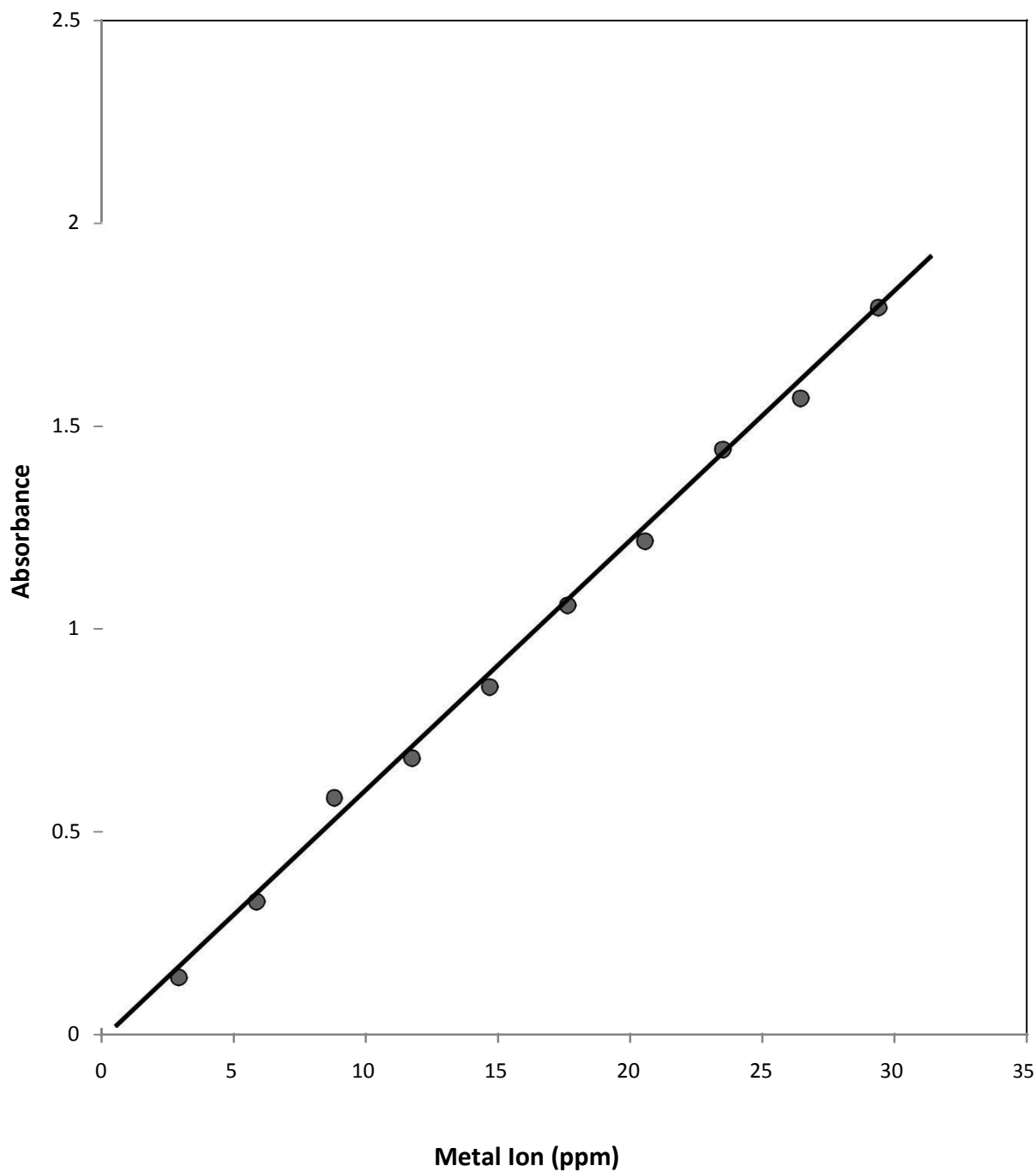
### Effect of reagent concentration:

A mixture containing 1ml of 0.001M metal ion, 1.5ml of 2M sodium acetate of pH 6 and the reagent concentration varying between 1.0-3.0 ml of 0.005M PDTTC was studied. It was observed that a maximum of 1.5ml of the reagent is necessary for the quantitative complexation of Nickel ion and hence the same volume was used in all the experiments conducted.

### **Applicability of Beer's law:**

To the solution containing different amounts of nickel of 0.001 M, 1.5ml of 2M sodium acetate at a pH 6 and 1.5 ml of 0.005 M PDTTC were added. The total volume was made up to 10ml by adding double distilled water. The plot between the concentration of nickel and absorbance values is linear ,passing through the origin ,obeying Beer's law in the concentration range of 2.93-29.34 ppm of nickel ion.

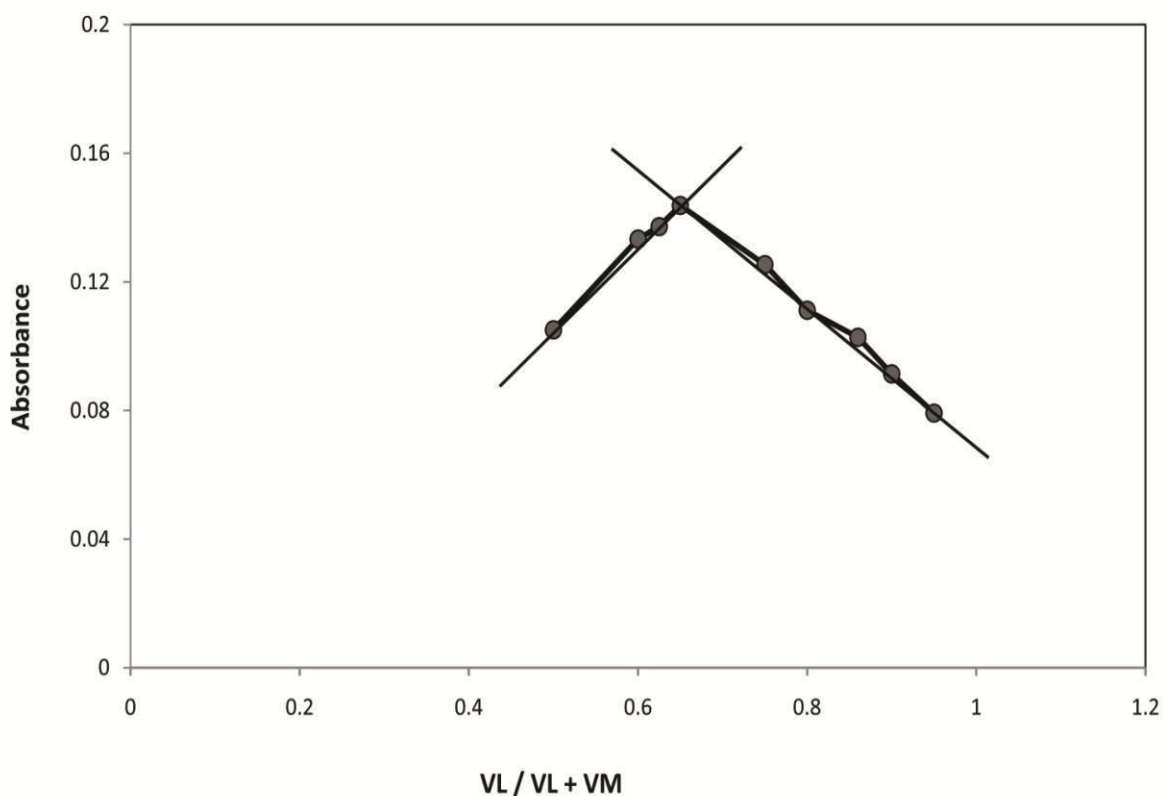
### Applicability of Beer's Law



## Composition of the complex :

### a) Job's method of continuous variation :

Equimolar solution of Ni (II) and PDTCC (0.001M) were prepared and the metal and the reagent solutions were mixed in different proportions keeping the total volume at 4ml. In each case 1.5ml of 2M  $\text{CH}_3\text{COONa}$  buffer of pH 6 was added and the total volume was maintained at 10ml. A graph was plotted between absorption and mole fraction of the ligand. From the graph the metal to ligand ratio in the complex is found to be 1:2 which means 1 mole of the metal ion reacts with 2 moles of the ligand.



### JOB'S METHOD

### b) Mole ratio method :

1ml of nickel solution of 0.001M was treated with different known volumes of ligand of 0.5 to 5ml of the 0.001M in the presence of 1ml of 2M  $\text{CH}_3\text{COONa}$  buffer at a pH 6. The mixture was diluted to 10ml and absorbance values were noted at 318nm.A

graph was plotted between the values of volume of the reagent and absorbance. From the graph it is observed that 1mole of nickel chelates with 2 moles of PDTTC.

### **Asmus method :**

The experimental part for asmus method is same as performed in mole ratio method. A linear plot is obtained between  $1/m$  and  $1/v^2$  confirming the ratio of metal to ligand as 1:2. The instability constant of the nickel complex calculated is found to be  $5.2083 \times 10^{-7}$ .

### **Estimation of nickel in drinking water samples:**

1 litre of water samples collected from Krishna River, Bore water on hill and from well water of Vijayawada town were pre concentrated to 100ml and 5ml of these samples were taken for analysis.

### **Leafy Vegetables:**

Except curry leaves, all other leafy vegetables each of 5 grams grown in the nearby villages of Vijayawada town, Krishna District were collected, digested by dry ash method and brought into solution by dissolving in 500ml double distilled water where as the curry leaves sample was dissolved in 50ml. The leafy samples are *Mentha spicata* (Mint leaves), *Piper beetle* (Beetle leaves) and *Murraya Koenigii* (curry Leaves).

Aliquots of the above solutions were taken into beakers and the experimental conditions developed as already mentioned were maintained. The results obtained are further supported by atomic absorption spectrophotometric method. The values are compared with AAS method and the results obtained from Spectrophotometric method are presented in the following tables 1 & 2.

**Table 1 : Determination of nickel(II) in water samples of Vijayawada town:**

I.Krishna river water

II.Bore water on hill

III.Well water

Sodium Acetate: 2M

PDTC: 0.005M

P<sup>H</sup> : 6

Sample	S.No.	Ni(II) added (ppm)	Ni(II) found (ppm)	% recovery	AAS method	
					Ni(II) found (ppm)	% recovery
I	1	1.0	1.0	100.00	0.98	98.00
	2	1.2	1.2	100.00	1.20	100.00
	3	1.6	1.58	98.75	1.60	100.00
	4	1.8	1.8	100.00	1.80	100.00
	5	2.0	1.98	99.00	1.98	99.00
				Average	99.55	Average
II	1	1.0	0.98	98.00	1.00	100.00
	2	1.2	1.18	98.33	1.18	98.33
	3	1.6	1.6	100.00	1.58	98.75
	4	1.8	1.8	100.00	1.80	100.00
	5	2.0	2.0	100.00	1.99	99.16
				Average	99.26	Average
III	1	1.0	1.00	100.00	1.00	100.00
	2	1.2	1.19	99.16	1.18	98.33
	3	1.6	1.58	98.75	1.59	99.37
	4	1.8	1.80	100.00	1.80	100.00
	5	2.0	1.98	99.00	1.99	99.16
				Average	99.38	Average



**Table 2: Determination of Ni (II) in Leafy Vegetables:**

I. Mentha Spicata (Mint Leaves)

II. Piper Beetle (Beetle Leaves)

III. Murraya Koenigii (Curry Leaves)

Sodium Acetate: 2M

PDTc: 0.005M

P<sup>H</sup> : 6

Sample	S.no	Ni (II) PPm		Ni(II), PPm found AAS Method
		Added	Found	
I	1	1.0	1.825	1.826
	2	1.0	1.822	1.820
	3	1.0	1.820	1.823
II	1	1.0	1.408	1.406
	2	1.0	1.404	1.402
	3	1.0	1.405	1.404
III	1	1.0	1.298	1.295
	2	1.0	1.296	1.296
	3	1.0	1.290	1.292

### Conclusion

The results in the table indicate that the water samples analysed are free from nickel content and the % recovery values obtained are comparable and in good agreement with atomic absorption spectrophotometric data. The Nickel content present in leafy vegetables are in agreement with the standard values reported<sup>10</sup>.

The method developed for Ni (II) in the presence of sodium acetate buffer medium using PDTC is found to be sensitive, selective, specific, rapid and may be successfully applied for the determination of low concentration of nickel present in water samples and leafy vegetables.

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