

Spectrophotometric Determination Of Vanadium (V) With Schiff Base Derived From Pyridine-2-Carboxaldehyde And 2-Amino Pyridine By Preliminary Adsorption On Polyurethane Foam

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Abstract- The spectrophotometric determination of Vanadium (V) by adsorption of its pyridine-2-carboxaldehyde and 2-amino pyridine complex after adsorption on polyurethane foam is described. The complex is eluted from the foam with chloroform and absorbance is measured at 400 nm. Beer's law is obeyed in the concentration range 5-90 μg of vanadium. The molar absorptivity was found to be $2.3108 \times 10^4 \text{ Lmol}^{-1}\text{cm}^{-1}$ and sensitivity being $1.14 \times 10^{-2} \mu\text{g cm}^{-2}$ for the absorbance of 0.001. The effect of various parameters namely pH, reagent, adsorbant, shaking time and diverse ions have also been investigated.

Index Terms- Polyurethane foam, Schiff base, Spectrophotometric determination, Vanadium,

I. INTRODUCTION

Biological activities of Schiff bases are very well known. Some of them also show anticarcinogenic properties¹⁻². Schiff bases can be obtained from different amines and carbonyl compounds. These Schiff bases can form chelate complexes with different metal ions. These Schiff bases contain -C=N- imine linkage which is responsible for their biological activity and produce colour with metal ions by the formation of complex³⁻⁴. The following Schiff bases have been synthesized by respective carbonyl compound and amine in-

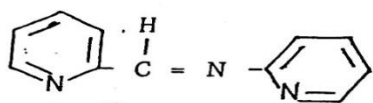


Figure. 1: SB derived from pyridine-2-carboxaldehyde and 2-amino pyridine.

Usually many organic compounds are used in analytical chemistry. Ample of investigation has been done for the development of new analytical organic reagents⁵⁻¹⁰. Due to the environmental contamination with vanadium in recent years it is the area of interest of chemists. This metal is widely involved in the enzymatic reactions and

nitrogen fixation.¹¹ It is a part of essential micronutrient in organisms which is responsible for health and disease.

Determination of vanadium is not easy from analytical point of view due to presence of interfering ions like iron(III). Hydroxyquinoline used for the determination of vanadium in bio samples has lack of selectivity and its pH range is not wide.¹² After this many reagents have been used for the determination of vanadium like reagent made by hydrogen peroxide, α -benzoinoxime and diaminobenzidine, Schiff base derived from 2-furfural-dehyde and p-aminophenyl mercaptoacetic acid and one more Schiff base derived from ethylenediamine and 4-benzoyl-1-phenyl-3-methyl-2-pyrazidine-5-one.¹³⁻¹⁵

Above mentioned Schiff bases are valuable towards transition metal ions. In this series new Schiff base derived from pyridine-2-carboxaldehyde and 2-amino pyridine has interesting analytical application.¹⁶ First row transition metals Vanadium (V) forms a stable water insoluble chelate complex with Schiff base derived from pyridine - 2 - carboxaldehyde and 2- amino pyridine. A new "Solid - Liquid Extraction" technique is used for the absorption of metal chelate on polyurethane foam.

II. MATERIALS AND APPARATUS

Various materials and arrangements were utilized in the spectrophotometric assurance:

A. SCHIFF BASE :-

Schiff was acquired from pyridine - 2 - carboxaldehyde and 2 - amino pyridine. Ethanolic arrangement of carbonyl compound pyridine-2-carboxaldehyde was blended in with 2-amino pyridine in ethanol in 1:1 molar proportion, refluxed over water shower for 3-5 hours. The subsequent Schiff base was isolated out. Blend was cooled, washed, dried in air and recrystallised by ethanol. Espresso earthy colored hued precious stones were acquired.

B. STANDARD METAL ION SOLUTION:-

A standard stock arrangement of vanadium (V) (1000ppm) was set up by dissolving sufficient measure of ammonium meta

vendate in refined water. Weakened arrangements of different focus were set up by weakening of this stock arrangement.

C. BUFFER SOLUTION:-

Two cushion arrangements of pH run 3-6 and 8-11 are set up by blending explicit measure of 1M acidic corrosive and 1M ammonium acetic acid derivation arrangement and 1M fluid smelling salts and 1M ammonium acetic acid derivation arrangement individually.

D. REAGENT SOLUTION:-

It was set up by dissolving 0.2 gm of Schiff base compound in 100 ml ethanol.

E. POLYURETHANE FOAM :-

It was set up by the strategy for Hamon et al.¹⁷ Polyurethane froth pieces were cut in 1 cm³ size from a froth cushion which was gotten from a neighborhood departmental store . All froth pieces were cleaned by good cleaning strategies which were assessed previously. Right off the bat, the froth was more than once pressed in an enormous amount of 1M hydrochloric corrosive for certain minutes to a few hours to expel potential contaminants and afterward washed with refined water. They were next liberated from corrosive by crushing in water. Air dried in open at room temperature. No significant changes in polymeric properties because of these treatment was watched. Along these lines, perception uncovered that 1 hour pressing was

sufficient. Chow et al¹⁷ recommended that the froth pieces ought to be as uniform as could be expected under the circumstances.

F. APPARATUS:-

Spectrophotometer EC model (GS – 5701) was used for the spectrometric estimations. Systronics model (Sr. No. 5244) was used for pH estimations.

III. PROCEDURE

A fraction (1.0 ml) of standard vanadium(V) solution containing 15-90 µg of vanadium was taken in a beaker and 2.5 ml of 0.2% Schiff base solution was added to it. The pH was ranged for 3.0. After making volume 10 ml it was allowed to rest for 2 min for complete development of colour. Then, seven polyurethane pieces were added to this solution.¹⁸ The flask was shaken for 60 seconds to allow the metal complex formed to be adsorbed on the foam. The foam pieces then squeezed with a glass plunger. These foam pieces which contain vanadium ion complex were transferred to glass beaker. The complex was rinsed from the foam pieces by squeezing with two portion of 2.5 ml chloroform. To remove traces of water 2.0 gm anhydrous sodium sulphate was added. Absorbance was measured in the region of 370-560 nm wave length. Calibration curve was constructed under similar conditions.

IV. RESULT AND DISCUSSION

A. ABSORPTION SPECTRA :

A sample solution containing 60 µg of Vanadium (V), 2.5 ml of 0.2% reagent of Schiff base solution and 2.0 ml of the buffer solution was added to adjust the pH 3.0 . The absorbance of the complex elutes with chloroform was measured at wave length range between 370-560 nm. Following curve fig 2. represents the absorption spectra of vanadium (V) complex. The maximum absorption is observed at 400 nm, so absorbance measurements were carried out at 400 nm wave length.

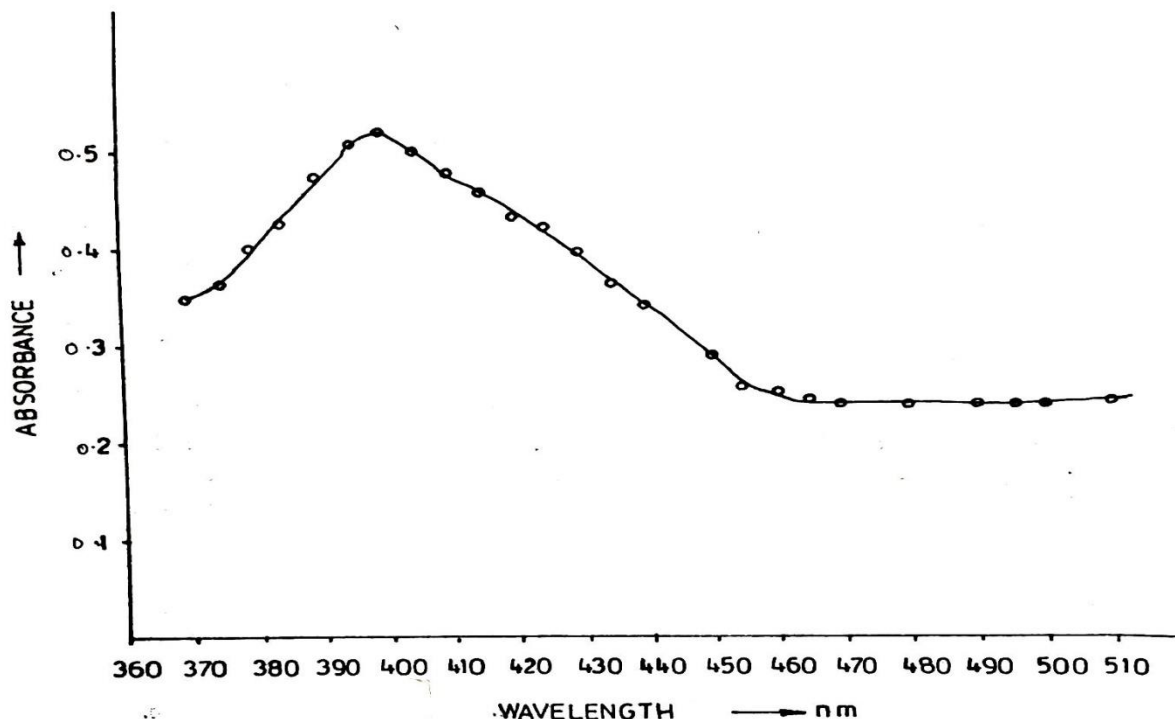


Figure .2 : Absorption Spectra of Vanadium

[VANADIUM (V) :60 µg, pH : 3.0, 0.2% REAGENT SOLUTION: 2.5ml, POLYURETHANE FOAM : 7 PIECES, SHAKING TIME :120 sec]

B. EFFECT OF pH

The effect of Ph on the absorbance of vanadium(V) complex having 60 µg vanadium ion in the solution was investigated at 400 nm in the pH range 1-10. The maximum and almost constant absorbance was obtained between the pH range 2.0 – 6.0 . So, for all absorbance measurements pH of solution was taken to be 3.0 shown in fig. 3 and table 1.

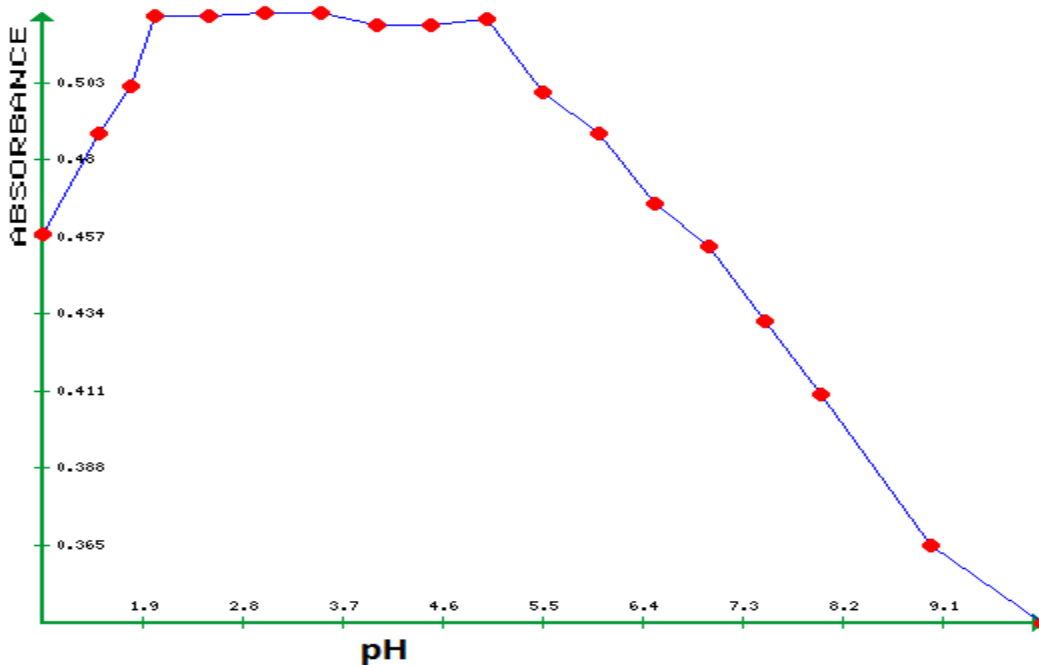


Figure. 3: Effect of pH on absorbance

(VANADIUM (V) : 60 µg, WAVELENGTH : 400 nm, 0.2% REAGENT SOLUTION : 2.5 ml, POLYURETHANE FOAM : 7 PIECES, SHAKING TIME : 120 sec.)

TABLE (1) : Effect of pH

pH	Absorbance at 400 nm
1.0	0.458
1.5	0.488
1.8	0.502
2.0	0.523
2.5	0.523
3.0	0.524
3.5	0.524
4.0	0.520
4.5	0.523
5.0	0.522
5.5	0.500
6.0	0.488
6.5	0.467
7.0	0.454
7.5	0.432
8.0	0.410
8.5	0.389
9.0	0.365
10.0	0.342

Vanadium (V): 60 μ G
0.2% Reagent : 2.5 ml
Polyurethane foam : 7 pieces
Shaking Time : 120 sec

C. EFFECT OF REAGENT SOLUTION :

Different amounts of the reagent solution were prepared for the investigation of the effect of the reagent concentration on the absorbance of vanadium (v) complex. It was prepared by the sample solution containing 60 μ g of vanadium (v). It was observed at pH 3.0 . Almost same absorbance was obtained when 1.5 to 6.0 ml of reagent solution were added therefore, 2.5 ml of reagent solution was considered the suitable quantity to be used for all absorbance measurements as shown in fig. 4 and table 2.

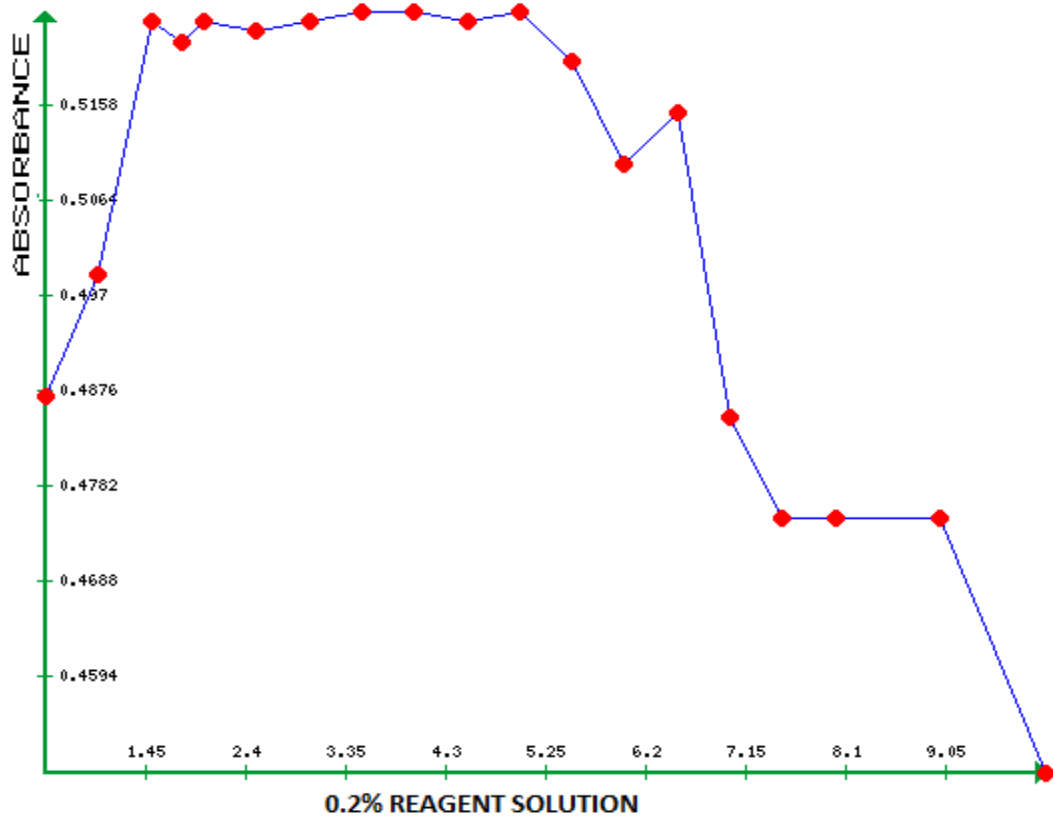


Figure. 4: Effect of Reagent

[VANADIUM (V) :60 µg, WAVELENGTH : 400 nm, pH : 3.0, POLYURETHANE FOAM : 7 PIECES, SHAKING TIME :120 sec]

TABLE (2) : Effect of Reagent Concentration

0.2% Reagent	Absorbance at 400 nm
0.5	0.487
1.0	0.499
1.5	0.524
1.8	0.522
2.0	0.524
2.5	0.523
3.0	0.524
3.5	0.525
4.0	0.525
4.5	0.524
5.0	0.525
5.5	0.520
6.0	0.510
6.5	0.515
7.0	0.485
7.5	0.475
8.0	0.475
9.0	0.475
10.0	0.450

Vanadium (V): 60µG
 pH : 3.0
 Polyurethane foam : 7 pieces
 Shaking Time : 120 sec

D. EFFECT OF ADSORBENT :

This effect was determined by adding different number of prepared polyurethane foam pieces in the vanadium (v) complex solution. The absorbance increased with the addition of 1-3 polyurethane pieces. The value of absorbance was found almost constant upto 9.0 therefore seven foam pieces were taken for all absorbance measurements(Fig. 5 and table 3.)

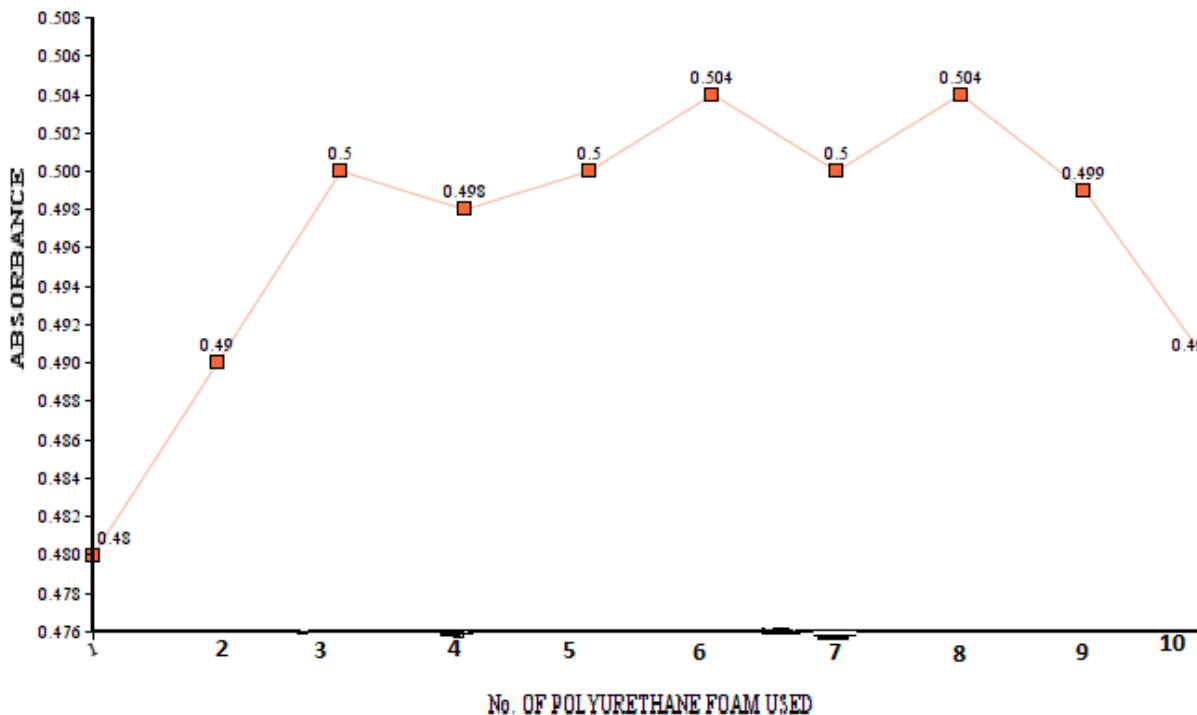


Figure. 5: Effect of Adsorbant

[VANADIUM (V) : 60 µg, WAVELENGTH : 400 nm, pH : 3.0, 0.2% REAGENT SOLUTION : 2.5 ml, SHAKING TIME : 120 sec]

TABLE (3) : Effect of Adsorbent

No. of Polyurethane foam Pieces used	Absorbance at 400 nm
1	0.480
2	0.490
3	0.500
4	0.498
5	0.500
6	0.504
7	0.500
8	0.504
9	0.499
10	0.490

Vanadium (V) : 60µG
 pH : 3.0
 0.2% Reagent : 2.5 ml
 Shaking Time : 120 sec

E. EFFECT OF SHAKING TIME :

Vanadium (v) solution containing 60 µg of vanadium on was allowed to satnd in contact with schiff base solution for two minutes. Then the complex was shaken from 5-300 seconds. Absorbance first increased, attained maximum and

became constant from 20 to 160 seconds. So, 120 second was chosen the shaking time for all absorbance measurements as in fig. 6 and table 4.

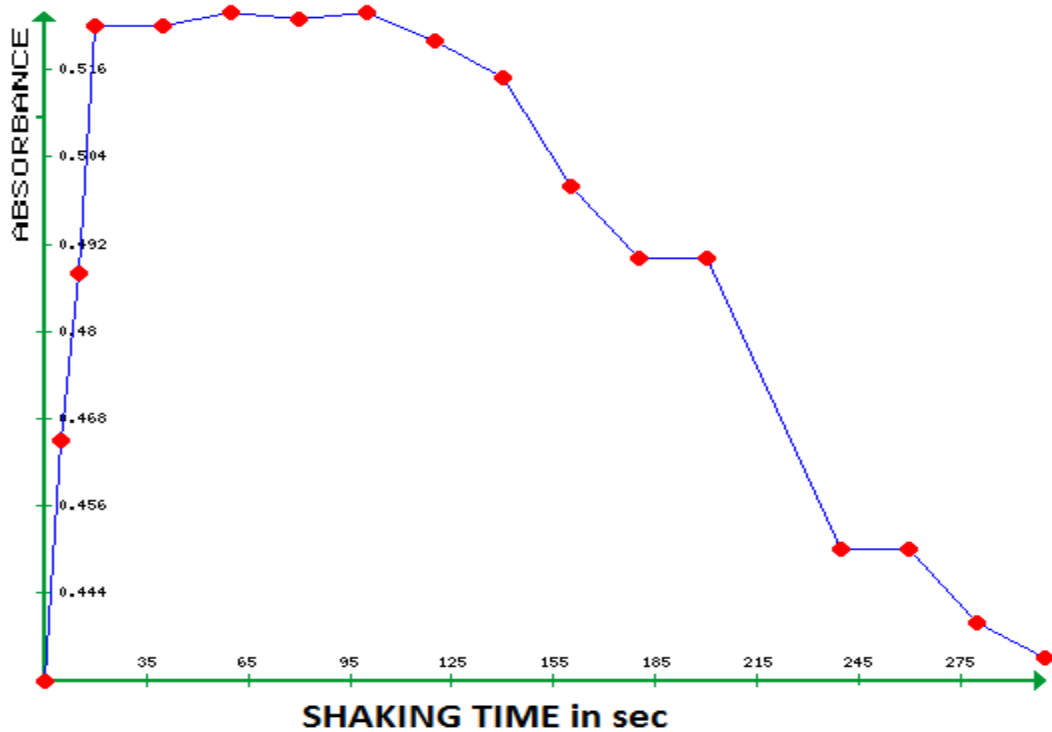


Figure. 6: Effect of Shaking Time

[VANADIUM (V) : 60 µg, WAVELENGTH : 400 nm, pH : 3.0, 0.2% REAGENT SOLUTION : 2.5 ml, POLYURETHANE FOAM : 7 PIECES]

TABLE (4) : Effect of Shaking Time

Shaking Time (sec.)	Absorbance at 400 nm
5	0.432
10	0.465
15	0.488
20	0.522
40	0.522
60	0.524
80	0.523
100	0.524
120	0.520
140	0.515
160	0.500
180	0.490
200	0.490
240	0.450
260	0.450
280	0.440
300	0.435

Vanadium (V) : 60µG
 pH : 3.0
 Polyurethane foam : 7 pieces
 0.2% Reagent : 2.5 ml

F. CALIBRATION CURVE FOR VANADIUM (V) :

Calibration curve was obtained by the recommended procedure under the optimum condition according to Fig. 7. Beer’s law was obeyed in the range 5-90 µg. The molar absorptivity was found to be $2.310 \times 10^4 \text{ Lmol}^{-1} \text{ cm}^{-1}$ at 400 nm and sensitivity being $1.58 \times 10^{-2} \text{ µg cm}^{-2}$ of vanadium (v) for the absorbance of 0.001.(Table 5)

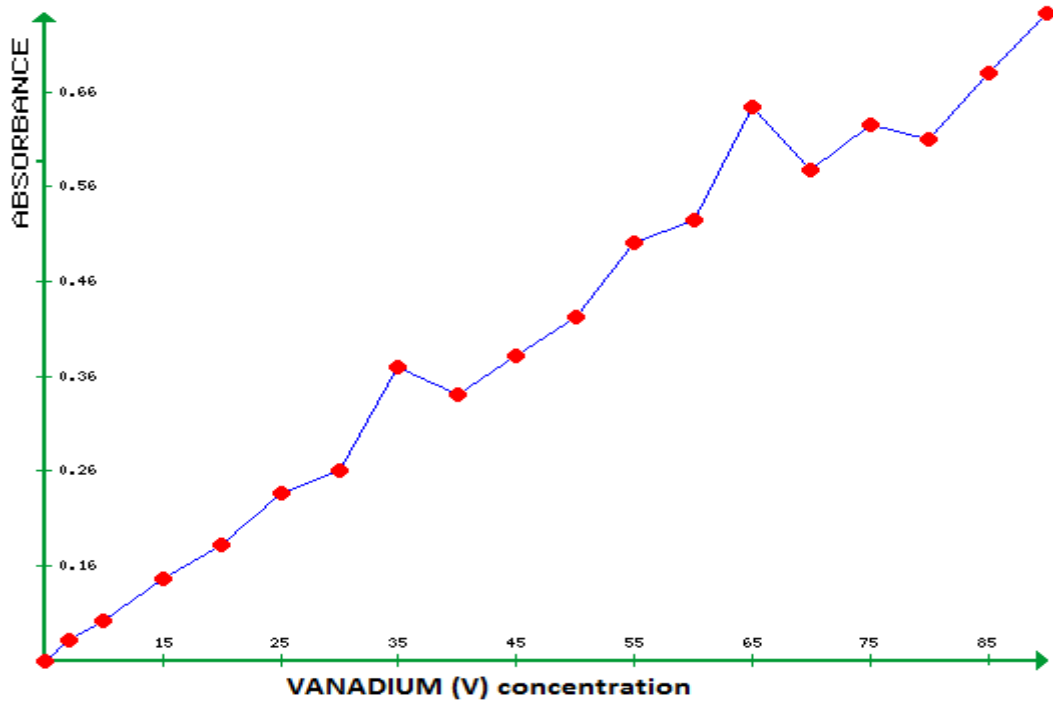


Figure. 7: Calibration Curve of Vanadium

[WAVELENGTH : 400 nm, pH : 3.0, 0.2% REAGENT SOLUTION : 2.5 ml, POLYURETHANE FOAM : 7 PIECES]

TABLE (5) : Calibration Data for Vanadium (V)

Vanadium (V) Concentration	Absorbance at 400 nm
5	0.060
7	0.082
10	0.101
15	0.146
20	0.182
25	0.236
30	0.261
35	0.369
40	0.341
45	0.382
50	0.423
55	0.501
60	0.524
65	0.644
70	0.577
75	0.625
80	0.610
85	0.680
90	0.742

pH : 3.0

0.2% Reagent : 2.5 ml

Polyurethane foam : 7 pieces

Shaking Time : 120 sec

G. EFFECT OF DIVERSE IONS :

Interference due to the presence of various amounts of alkali metal salts and metal ions on the absorbance was studied according to the table 6 and 7.

TABLE (6) : Effect of Diverse Alkali Metal Ion Salts

Alkali Metal Salts	Amount Added mg	Vanadium (V) Found μg
NaCl	50	60.8
	100	60.5
KCl	50	60.2
	100	60.1
KNO ₃	50	59.9
	200	59.2
NaNO ₃	50	60.8
	250	59.2
Na ₂ CO ₃	50	60.2
	100	60.3
K ₂ SO ₄	40	61.2
	150	61.3
Na ₂ HPO ₄	40	60.2
	200	59.8
Sodium Oxalate	40	60.1
	100	60.3

Vanadium (V) : 60 μg ;
 pH : 3.0;
 0.2% Reagent : 2.5 ml;
 Polyurethane foam : 7 pieces;
 Shaking Time : 120 sec.

TABLE (7) : Effect of Diverse Metal Ions

Diverse Metal Ions	Amount of Ion Added (mg)	Vanadium (V) Found μg
Co (II)	40	61.2
	120	62.3
Zn (II)	30	61.0
	80	60.9
Pd (II)	100	60.1
	150	61.6
Mn (II)	50	60.9
	150	59.8
Cu (II)	40	59.2
	200	58.6
Mg (II)	100	59.3
	250	60.1

Sn (II)	50	60.0
	100	59.9
Fe (III)	50	60.2
	100	60.0
Cr (III)	100	60.0
	200	59.9
Al (III)	40	59.8
	100	58.6
Bi (III)	30	59.2
	60	60.6

Vanadium (V) : 60 µg ;
pH : 3.0;
0.2% Reagent : 2.5 ml;
Polyurethane foam : 7 pieces;

V. PRECISION

Ten examples of the arrangement containing 60 µg of vanadium (v) were set up by weakening the stock arrangement and the complex shaped with the reagent was gotten. The absorbance was estimated at 400 nm. Mean absorbance of 0.524 with a standard deviation of 0.28% was acquired.

VI. CONCLUSION

In this examination, Schiff base ligand and its complex with vanadium(v) particle were researched. It is found from the above conversation that Schiff base got from pyridine-2-carboxaldehyde and 2- amino pyridine is a decent touchy reagent for the spectrophotometric assurance of vanadium(v) particle by the methods for different parameters like pH, reagent focus, adsorbent, shaking time, assorted particle expansion. It likewise can be acceptable applied for the assurance of vanadium(v) in pharmaceutical samples.

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