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Quantification and speciation of trac. E amount of vanadium (iii) and vanadium (v) using 2-[(e)-[3-[(e)-(2 hydroxyphenyl) methyleneamino]phenyl] iminomethyl]phenol

Abstract

Some new transition metal complexes of Vanadium(III) and Vanadium(V) with tetradentate (N_2O_2) Schiff base – 2-[(*E*)-[3-[(*E*)-(2-hydroxyphenyl)methyleneamino]phenyl] iminomethyl]phenol (HPMAPIP) have been synthesized. The characterization of the ligand and the metal complexes were also reported. The bonding and the proposed structure of the newly synthesized complexes were also identified by elemental analysis, Infra red, UV – Vis, ^{1}H NMR, magnetic moment measurements and conductivity analysis. The Metal: Ligand Mole ratio indicated a 1:1 and their wavelength maximum were at 400 and 405 nm for Vanadium (III) and Vanadium(V) complexes respectively. From the calibration curve, Beer's law was valid for Vanadium (III) and Vanadium (V) between 0.488 – 3.904 ppm. The calibration and analytical sensitivity of Vanadium (III) complex are 0.074 and 0.32 while that of Vanadium (V) are 0.024 and 24 respectively. Optimum pH for the formation of the complex was determined to be 10 and 11 for Vanadium (III) and Vanadium(V) respectively. Very few elements were found to interfere with the method. The method was successfully applied in the determination of Vanadium in alloy steel.

Keywords: spectrophotometry, vanadium, alloy sample

Volume 10 Issue 4 - 2021

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Received: May 01, 2021 | **Published:** July 02, 2021

Introduction

The determination of vanadium has received extensive attention because of its increasing importance in biological and environmental studies. This metal is widely distributed in the earth's crust but in low abundance. Major sources for the emission of vanadium in the environment include combustion of fuel oils, dye, ceramics, ink, catalyst and steel manufacturing. Vanadium in trace amounts represents an essential element for normal cell growth, but it can be toxic when present in higher concentrations. It plays an important role in physiological systems including normalization of sugar levels and participation in various enzyme systems as an inhibitor and cofactor of the oxidation of amines.[1] Schiff base has been studied extensively because of their high – potential chemical permutation. Schiff base bases represent a class of important compounds in a medicinal and pharmaceutical field. They have biological activities such as antimicrobial,[2–5] anticancer[6–8] antifungal[9,10] and herbicidal activities.[11] More so, Schiff base offer more opportunities for inducing substrate chirality, tuning metals centred electronic factors, enhance solubilities and stability of either homogeneous or hetrogenous catalyst.[12–17] Schiff base ligands are able to coordinate many different metals[18–20] and stabilize their oxidation states. A good number of reviews have been made on the use of large number of chromogenic reagents for the spectrophotometric determination of iron and cobalt. Some of the recently proposed spectrophotometric methods for the determination of iron[21–24] are less sensitive and less selective. We are now proposing simple, sensitive and selective direct and derivative spectrophotometric methods for the determination of vanadium(III) and vanadium(V) in various complex materials using 2-[(*E*)-[3-[(*E*)-(2-Hydroxyphenyl)methyleneamino]phenyl]iminomethyl]phenol synthesized from the condensation reaction of 1,3-diaminobenzene and 2-hydroxyzaldehyde as chromogenic agent.

Materials and methods

All the reagents, *O* - phenylenediamine (Sigma Aldrich), 2 Hydroxylbenzylaldehyde (Sigma Aldrich), Concentrated Hydrochloric Acid (Sigma Aldrich), Ethanol (May and Baker), Ammonium trioxovanadate(V) (Sigma Aldrich), Vanadium (III) chloride (Sigma Aldrich) were of analytical grade and were used without further purification. Distilled water was used to prepare some solutions. Buffer solutions used covering the pH range 1-13 was prepared according to Clark and Lubbs' Procedure[25] All absorbance measurements and the spectra of Schiff base and the metal complexes solution were taken at 30°C with a double beam UV-VIS spectrophotometer (Shimadzu UV-1800). Measurements of pH were made using control digital pH meter equipped with a combination electrode.

Synthesis of the ligand

Synthesis of the Schiff base ligand was carried out according to reported method.[26] 5.41 g of (0.05 mole) of *O* - phenylenediamine was dissolved in dimethyl formamide (50 cm^3) and 2 cm^3 of 1.0 M NaOH was added and stirred. 10.44 g (0.1mole) of 2-hydroxy benzaldehyde was added to the resulting solution at room temperature. The mixture was refluxed for 2 hour at 60-65^{0}C. The Schiff base was cooled, washed with ethanol, recrystallized and kept in a desiccator over fused $CaCl_2$. (91% yield, m.p. 112- 115^{o}C) Figure 1.

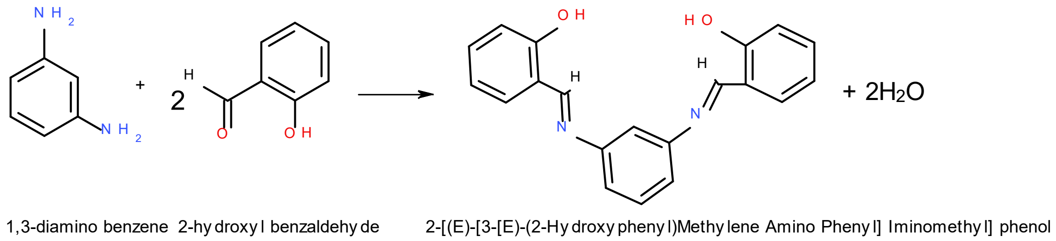

Figure 1 Synthesis of the Schiff base.

Synthesis of the metal complexes

The vanadium(III) and vanadium(V) complexes of 2-[(E)-[3-[(E)-(2-hydroxyphenyl)methyleneamino]phenyl]iminomethyl]phenol (HPMAPIP) were synthesized according to literature.[27] In each case, a solution of the ligand 1.898 g (0.003 M) in ethanolic solution (25 cm^3) was reacted with 0.500 g (0.00015 mole) solution of vanadium (III) and 0.350 g of vanadium (V) in ethanol (25 cm^3). The reaction mixture was refluxed for 2 hour at 60°-65°C. There was observable color change. The mixture was then cooled, the product formed collected, washed with absolute ethanol, recrystallised, dried and kept in desiccator over fused $CaCl_2$.

Preparation of alloy steel sample solution

5 g of the alloy sample was dissolved in a mixture of 2 M HCl and 10 M HNO_3. The resulting solution was evaporated to a small volume. To this, 5 cm^3 of 1 : 1 H_2O and H_2SO_4 mixture was added and evaporated to dryness. The residue was dissolved in 15 cm^3 of distilled water and filtered through Whatman filter paper no. 40. The filtrate was collected in a 100 cm^3 volumetric flask and made upto the mark with distilled water. The solution was further diluted as required.

Results and discussion

Vanadium(III) and vanadium(V) react with HPMAPIP forming Greenish- yellow and Orange- green complexes respectively. The colour of the complexes was stable. The texture of the complexes is powdery in nature and the percentage yield were higher and in tandem with literatures.

Direct method of determination of vanadium(III) and vanadium(V) complexes

The absorption spectrum of [V(III)- HPMAPIP] and [V(V)-HPMAPIP] shows maximum absorbance at 405 nm and 399 nm respectively. This is slightly higher from the value given in literature.[28] The composition of the complex [V(III): HPMAPIP] and [V(V): HPMAPIP] were determined as 1 : 1 by Slope ratio method. The optimization investigations indicate that the absorbance of the complexes were maximum and stable in pH range of 10 and 11 respectively Figure (2 & 3). Hence these pH's were chosen for further studies. The calibration curve of V(III) shows a good linear relationship. A straight line graph passing through the origin was obtained for the complex, indicating that spectrophotometric analysis of complex on donor- acceptor complex formation can be used for quantitative analysis of V(III) complex Figure 4. Beer's law was obeyed from 0.488ppm to 3.904ppm with r^2 =0.9733 indicating a good linear relationship between absorbance and concentration. The plot is shown in Figure 5. While the calibration curve of V(V) complex shows adherence to Beer's law over the concentration range of 0.488–3.904 ppm with r^2 =0.9801 indicating a good linear relationship between absorbance and concentration. The linearity of the plot suggests that it can obey Beer's law at concentrations beyond 3.904 ppm. The plot is shown in Figure 6. Variable amounts of V(III) and V(V) were treated with suitable amounts of reagent and buffer and the validity of Beer's law was tested by plotting the measured absorbance values of the prepared solutions against concentrations of V(III) and V(V). Other analytical results are presented in Table 1, 2 & 3.

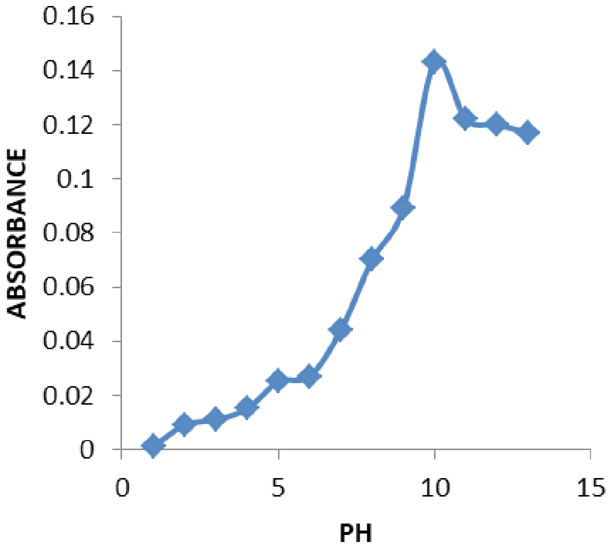

Figure 2 Shows the variation of absorbance with pH for the formation of the V(III) Complex.

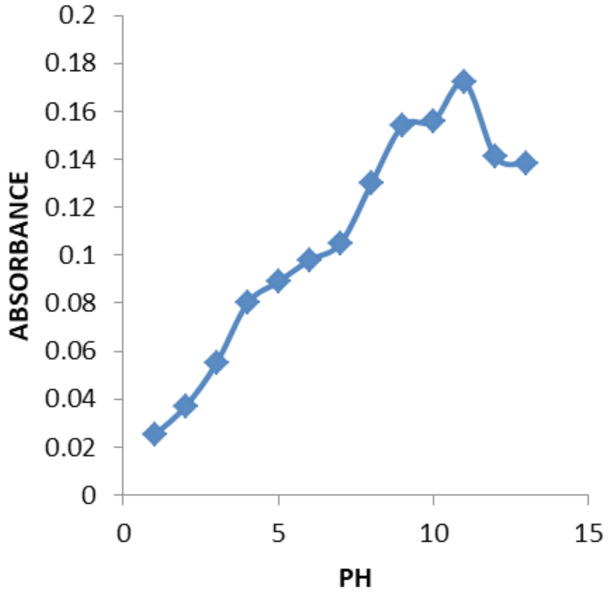

Figure 3 Shows the variation of absorbance with pH for the formation of the V(V) Complex.

Citation: Okenwa CJ, Ukoha PO, Chinyere EC, et al. Quantification and speciation of trac. E amount of vanadium (iii) and vanadium (v) using 2-[(e)-[3-[(e)-(2 hydroxyphenyl) methyleneamino]phenyl]iminomethyl]phenol. *J Anal Pharm Res.* 2021;10(4):136–145. DOI: 10.15406/japlr.2021.10.00377

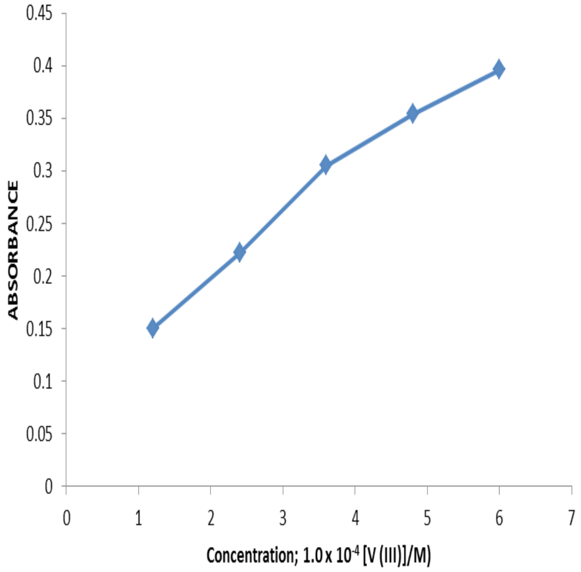

Figure 4 Effect of reagent concentration on the formation of vanadium(III) complex.

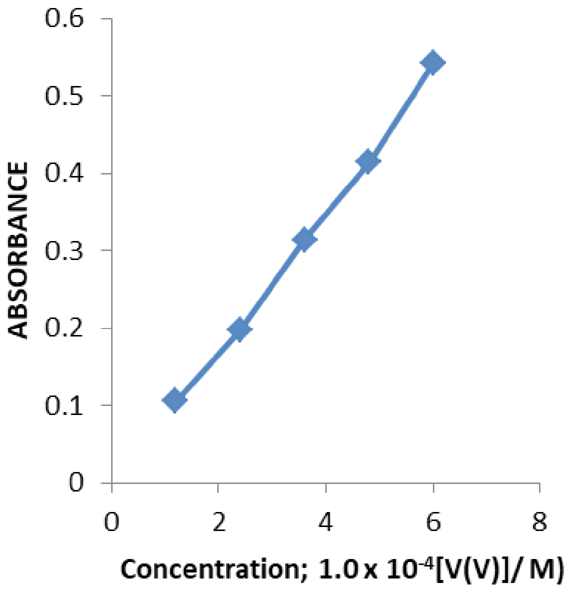

Figure 5 Effect of the reagent concentration on the formation of vanadium(V) Complex.

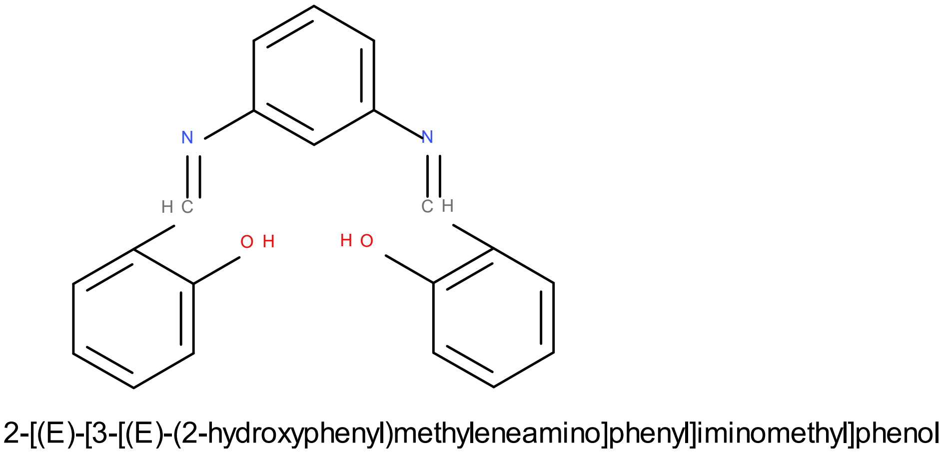

Figure 6 The proposed structure of the Ligand (HPMAPIP).

Citation: Okenwa CJ, Ukoha PO, Chinyere EC, et al. Quantification and speciation of trac. E amount of vanadium (iii) and vanadium (v) using 2-[(e)-[3-[(e)-(2 hydroxyphenyl) methyleneamino]phenyl]iminomethyl]phenol. *J Anal Pharm Res.* 2021;10(4):136‒145. DOI: 10.15406/japlr.2021.10.00377

Table 1 Physical Characteristic and Molar Conductivity Data of the Ligand and its metal Complex

Compound	Colour	Texture	Yield (gram)	% Yield	Composition Temperature (°C)	Molar Conductivity (μ Scm^{-1})	Remark
HPMAPIP	Orange	Crystalline	12.3	78	112-115	14.6	Non Electrolyte
V(III)- HPMAPIP	Greenish- yellow	Powdery	0.905	38	250-252	18.8	Non Electrolyte
V(V)- HPMAPIP	Orange- green	Powdery	0.833	37	240-242	47.8	Non Electrolyte
CuSO4.5H2O	-	-	-	-	-	570	Electrolyte

Table 2 Solubility of the Schiff Base and its Complexes in Various Solvents

Compound	Water	Methanol	Ethanol	Acetone	DMF
HPMAPIP	SS	S	S	SS	S
V(III)- HPMAPIP	S	S	S	SS	S
V(V)- HPMAPIP	S	S	S	SS	S

NS = Not Soluble, SS = Slightly Soluble, S = Soluble

Table 3 Analytical Characteristics of the metal complexes

Characteristics	V(III) complex	V(V) complex
λ (nm)	405	399
Molar absorptivity (Lmol^{-1}cm^{-1}	4955.69	3924.05
Calibration sensitivity; m (ppm)	0.074	0.024
Analytical sensitivity; m/SA	0.32	24
Limit of Detection; 3SA	0.69	0.003
Limit of Quantification; CQ; 10SA(ppm)	2.3	0.01
Linear dynamic range (ppm)	0.488 - 3.904	0.488 – 3.904

S_A = Standard Deviation of the blank: ±0.23 ±0.001

Optimization of conditions

Effect of pH on the absorbance of the complexes

The plot of absorbance against pH gave the highest absorbance of 0.214 for the formation of V(III) complex which was obtained at pH 10.0.

Effect of the concentration of the reagent on formation of vanadium(III) complex

The maximum absorption was obtained with 1.0 x 10^{-4} M

The highest absorbance obtained is 1.0×10^{-4}M for all the metal complexes.

Infrared spectral data of 2-[(E)-[3-[(E)-(2-hydroxyphenyl) methyleneamino]phenyl] iminomethyl]phenol (HPMAPIP) V(III) and V(V) metal complexes

One significant feature of the IR spectra of the ligand and its V(III) and V(V) complex is the appearance of broad band between 3400 and 3420.8 cm^{-1}. This has been ascribed to the presence of molecular hydrogen bonding. The absence of absorption band in the 3509-3310cm^{-1} and 1740 – 1695cm^{-1} region of the spectrum assignable to N-H vibration of the amines and C=O of the salicyaldehyde from the reactant respectively provide a strong evidence for the formation of ligand. The ligand showed vibration due to C-H of aromatic of medium intensity at 3056cm^{-1}, which shifted to higher intensity at 3061cm $^{-1}$ in both V(III) and V(V) complexes. This is in conformity with the values given by literature.[31] The C=N stretching vibration appeared at 1615 cm^{-1} in ligand and shifted to 1596 cm^{-1} in V(III) complex and 1602 cm^{-1} in V(V) complex. The decrease in frequency in both complexes suggests coordination of the azomethine nitrogen to the metal centres. This is also supported by the emergence of new bands at 495-448 cm^{-1} assignable to v(M-N).

The C-O stretching vibration was observed in the ligand at 1276-1107 cm^{-1} in both complexes, shifted to higher frequency which suggests that the phenolic oxygen was coordinated to the metal centres after deprotonation. This assertion agrees with what has been documented for other complexes in literature of diamino benzene complex,[29-31]

The presence of bands at 547-544 cm^{-1} was due to v(M-O) in both complexes further supports coordination of the phenolic oxygen to the metal centre. In the I.R spectra of the ligand and complexes, bands assignable to C-H aromatic stretching deformation, even ring breathing and out of plane vibration were observed to have weak, strong and moderate bands. The IR data of the ligand and complexes generally imply that the HPMAPIP is bidentate coordinating through N and O.

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Proton(^{1}H) NMR assignment for 2-[(E)-[3-[(E)-(2-hydroxyphenyl) methyleneamino] phenyl]iminomethyl]phenol (HPMAPIP)

The proton magnetic spectral of the ligand display a singlet peak at 8.85 ppm and integrated to two protons is the signal of hydroxyl protons attached to the aromatic ring. At 7.6 ppm, the singlet peak integrated for two protons is due to the azomethine protons Figure 7. The peak appearing at 7.4 ppm may be due to the non-equivalent aromatic proton. The aromatic proton is deshielded by the two azomethine nitrogens attached to the same ring. The doublet around 7.2 ppm and integrated for two protons is assigned to the symmetric phenyl proton. More so, the multiplet peaks around 6.9 ppm is integrated for eight protons. This arises from the symmetric aromatic protons of the phenyl group. It can be deduced from the proton magnetic resonance spectral that the ligand is symmetrical with some functional group N=C-H and Ar-OH appearing in both sides.Following the result of a 1:1 stoichiometry of the metal to ligand and its spectral data, it reveal that coordination of the metal to the ligand takes place through two azomethine N and two phenolato oxygen atoms in each of the metal complexes after deprotonation, giving a square planar geometry of the complexe in Figure 8.

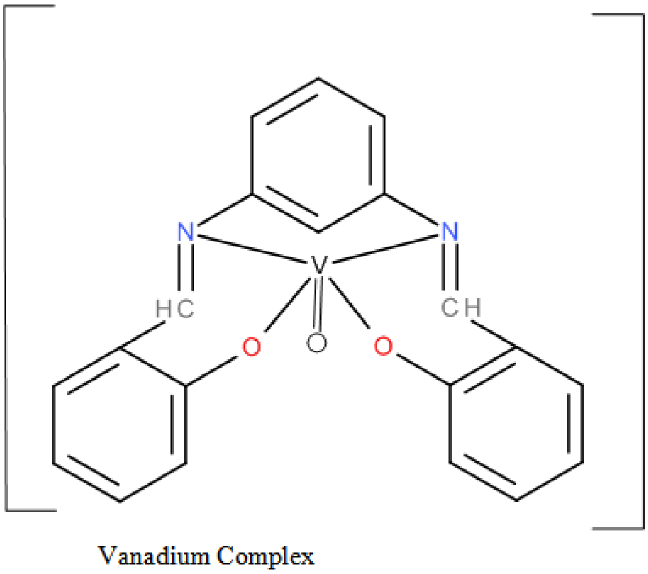

Figure 7 The proposed structure of the Metal Complexe.

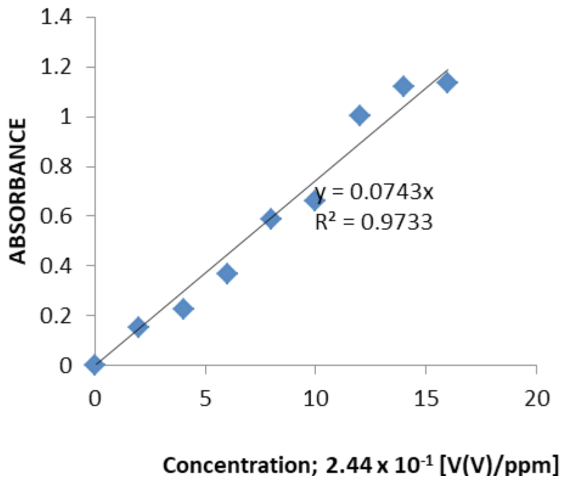

Figure 8 V(III) Complex.

Calibration Plots-Beer's law of V(III) and V(V) complexes

Effect of some interfering ions on the determination of V(III) and V(V) complexes

Under optimum conditions of the procedure, the selectivity of the proposed method was investigated by the determination of a fixed amount of V(III) and V(V) (1.2 ppm) in the presence of a series of cations that could as well form similar complex with HPMAPIP Table 4. These ions were chosen due to their occurrence in alloys in which vanadium III and V were also present. The absorbance was compared with the absorbance of the complex in the absence of foreign ion and percentage interference calculated. The limiting value of concentration of the foreign ion was taken as that which caused an error in the absorbance corresponding to twice the standard deviation of the absorbance of the V(III)- HPMAPIP or V(V)- HPMAPIP complexes Figure 9. The results are summarized in Table 5 and 6. In the determination of V(III)- HPMAPIP complex, the mean absorbance obtained for six determinations was 0.138±0.051. The tolerance limit was set as the amount of foreign ion causing an error of ±10.2 % in the absorbance of V(III)- HPMAPIP complex. The result of the interference study show that Mg(II), Fe(III), Cu(II), Ca(II) and Cr(III) interference seriously in the determination of V(III) complex and Ca(II) could be tolerable below 0.3 ppm. The interference of these ions were checked by masking and/or pre-extraction (where applicable) in the direct determination of V(III) in various alloys. KCN was used to enhance selectivity and for the pre-extraction of Fe(III) in steel.

Table 4 IR bands of ligand and its V(III) and V(V) complexes

Ligand	V(III) Complex	V(V) Complex	Band Assignment
3411 (w)	3400.0(m)	3420.87(m)	ν(O-H)
3056 (m)	3061.13(w)	3061.13(w)	ν(C-H) of Aromatic
2919 (w)	2929.00(w)	2929.04(w)	Aromatic overtones & combination bend
	2381.20(w)	2381.20(w)	
1615.44(s)	1596.15(s)	1602.90(s)	ν(C=N)
1479.45(m)	1452.45(m)	1452.45(m)	ν(C=C)
1275.97(s)	1312.60(s)	1312.60(s)	ν(C-O)
1107.18(w)	1144.79(m)	1141.90(w)	ν(C-O)
901.75(m)	980.84(m)	982.76(m)	ν(C-H) Out of plane C-H Bending
	932.6(w)		
754.19(s)	756.12(s)	754.19(s)	ν(C-H) out of plane
640.39(w)	635.57(w)	621.10(w)	ν(=C-H) deformation
	543.77(w)	546.84(m)	M – O bending
	494.98(w)	447.50(w)	M - N (ring breathing)

Legend: S=strong, m=medium, w= weak, br = broad

Citation: Okenwa CJ, Ukoha PO, Chinyere EC, et al. Quantification and speciation of trac. E amount of vanadium (iii) and vanadium (v) using 2-[(e)-[3-[(e)-(2 hydroxyphenyl) methyleneamino]phenyl]iminomethyl]phenol. *J Anal Pharm Res.* 2021;10(4):136–145. DOI: 10.15406/japlr.2021.10.00377

Table 5 ^{1}H NMR spectral Data of the HPMAPIP in $CDCl_3$ solvent

Signal ppm	Assignment
8.85 ppm (2H,s)	SymAr – OH hydroxyl protons
7.6 ppm (2H,s)	Sym, HC=N, azomethine protons
7.4 ppm (1H,d)	Ar-H, aromatic proton
7.2 ppm (2H,d)	Phenyl proton.
6.98 ppm (8H,m)	Sym.Ar-H aromatic protons of the phenyl.

Legend: H= hydrogen; s= singlet; d=doublet; m= multiplet

Table 6 Effect of some Interfering Ions on V (III) Complex

Ions Added	Concentration (ppm)	Absorbance	Interference level (%)
Mg^{2+} Added as M_gO	0.5	0.184	33.3
	0.8	0.18	30.4
	1.1	0.207	50
	1.2	0.218	58
	1.5	0.244	77
	1.15	0.268	94.2
	2.2	0.215	55
Fe^{3+} added as $Fecl^3$	0.5	0.181	31.2
	0.8	0.223	61.5
	1	0.23	67
	1.2	0.266	93.1
	1.5	0.286	107.2
	1.9	0.165	19.7
	2.3	0.312	126.1
Cu^{2+} added as $CuSO_4.5H_2O$	0.4	0.261	89.1
	0.75	0.215	55.7
	0.9	0.293	112.3
	1	0.336	143.5
	1.32	0.33	139.1
	1.64	0.341	147.1
	1.93	0.363	163
Ca^{2+} added as $CaSO_4$	0.3	0.157	13.8
	0.5	0.163	18.1
	0.8	0.158	14.5
	1	0.159	15.2
	1.3	0.17	23.2
	1.5	0.164	18.8
	1.9	0.172	24.6

Table Continued...

Ions Added	**Concentration (ppm)**	**Absorbance**	**Interference level (%)**
Cr^{3+} added as $Cr(NO_3)3.9H_2O$	0.6	0.141	2.2
	0.7	0.158	14.5
	1	0.166	20.3
	1.15	0.169	22.5
	1.5	0.194	40.6
	1.7	0.209	51.4
	2	0.22	59.4
Al^{3+}	0.5	0.19	37.7
	0.8	0.17	23.2
	1.1	0.174	26.1
	1.38	0.16	15.9
	1.5	0.179	29.7
	1.8	0.174	26.1
	2.1	0.152	10.1

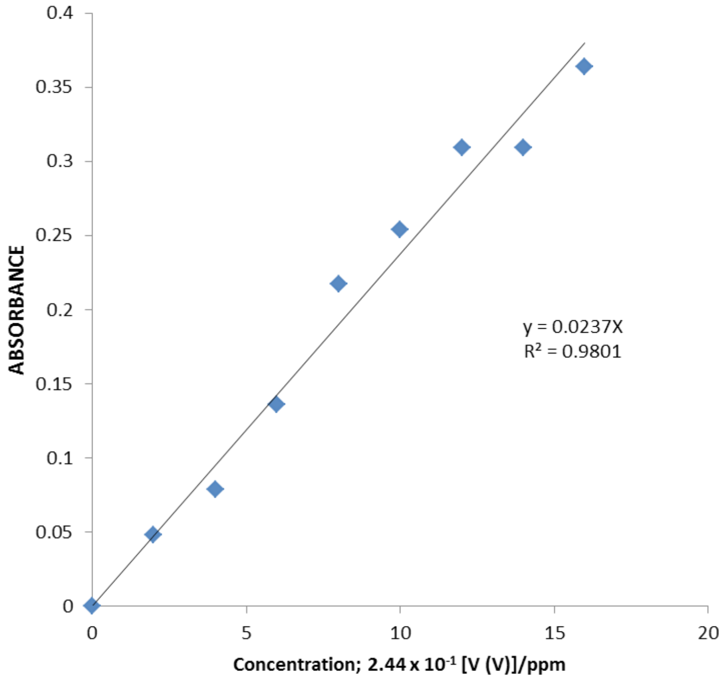

Figure 9 Calibration Curve of V(V) Complex.

V (V) complex

1.20 ppm V(V) ion was determined in the absence of foreign ions. The mean absorbance for four determinations was 0.167 ±0.074. The tolerance limit was taken as the concentration of foreign ion causing an interference of ± 14.8% in the V(V) complex. The interference levels in the determination of 1.20 ppm V(V) ion with various proportions of foreign ions are shown in Table 7 In the Table 7, the main interferences in the determination of V(V) ion are Fe(III), Cr(III), Ca(II), Al(III) and Cu(II) ions. Their interference levels were not within ±14.8%. The interference of Fe ion can be eliminated by masking with potassium cyanide.

Table 7 Effect of some Interfering Ions on V(V) Complex

Ions Added	Concentration (ppm)	Absorbance	Interference level (%)
Mg^{2+} Added as M_gO	0.3	0.163	6
	0.5	0.184	10
	0.7	0.201	20
	0.96	0.196	17.4
	1.2	0.198	18.6
	1.6	0.186	11.4
	2	0.168	0.6
Fe^{3+} added as $Fecl_3$	0.5	0.233	39.5
	0.8	0.189	13.2
	1	0.213	27.5
	1.2	0.173	3.6
	1.5	0.204	22.2
	1.9	0.172	3
	2.3	0.176	5.4
Cu^{2+} added as $CuSO_4.5H_2O$	0.4	0.238	42.5
	0.75	0.207	24.1
	0.9	0.214	28.1
	1	0.23	38
	1.32	0.21	26
	1.64	0.177'	6.1
	1.93	0.153	8.4
Ca^{2+} added as $CaSO_4$	0.3	0.265	59.1
	0.5	0.247	48
	0.8	0.274	64
	1	0.33	98
	1.3	0.229	37.1
	1.5	0.25	50.1
	1.9	0.271	62.3
Cr^{3+} added as $Cr(NO_3)3.9H_2O$	0.6	0.119	19.2
	0.7	0.222	33
	1	0.21	26
	1.15	0.213	28
	1.5	0.237	42
	1.7	0.213	38
	2	0.221	32.3
Al^{3+}	0.5	0.205	23.1
	0.8	0.123	26.3
	1.1	0.166	0.6
	1.38	0.158	5.4
	1.5	0.146	12.6
	1.8	0.17	2
	2.1	0.177	6

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Application to the analysis of alloys

An aliquot of the solutions of various alloys already analyzed by flame AAS was taken and the content of V(III) and V(V) complexes and each case determined spectrophotometrically, under the prevailing conditions according to the general procedure. The concentration of the V(III) and V(V) was extrapolated from the calibration curve.

Table 8 and 9 summarizes the results obtained for the determination of V(III) and V(V) in alloys with HPMAPIP. The above results show that the direct determination of V(III) and V(V) in Steel gave a very good recovery at concentrations above 0.064 ppm. It appears that a prior separation of V(III) before determination in steel is necessary. The ability of the method to determine small amounts of V(III) and V(V) has been testified and tells more about its sensitivity advantage over the flame atomic spectrophotometric method (which of cause is not able to quantify the actual amount of various vanadium ions present in the samples. Therefore, no firm quantitative comparison of the selectivity of the present method is possible with AAS due to differences in the methods used for their assessment.

Table 8 Determination of V(III) in Steel with the reagent/UV-Vis spectrophotometry

Sample	Amount added (ppm)	Amount found (ppm)	Recovery (%)	RSD (%)
Steel I	0.068	0.064	94	± 0.20
Steel II	0.085	0.08	95.5	± 0.17
Steel III	0.102	0.097	96	± 0.40

Table 9 Determination of V(V) with the reagent/ UV- Vis spectrophotometry

Sample	Amount added (ppm)	Amount found (ppm)	Recovery (%)	RSD (%)
Steel I	0.068	0.061	90	± 1.74
Steel II	0.085	0.079	93	± 0.88
Steel III	0.102	0.097	95.5	± 1.53

Conclusion

The applicability of the proposed method was evaluated by its application to the direct determination of V(III) and (V) ion on steel alloy, previously analyzed by flame atomic absorption spectrophotometry. The results of determination of vanadium in steel with Flame AAS, determination of V(III) in Steel with the reagent/ UV-, Vis spectrophotometry determination of V(V) with the Reagent/ UV- Vis spectrophotometry respectively indicate that the proposed method proposed method of determination of vanadium is more sensitive than those reported by Malik and Rao[32], Patil and Dhuley[33] Nagabhushana et al.[34] Wang et al.[35] Zhang et al.[36] and can be used to determine vanadium metal ions in various alloys without extracting procedures. The proposed method has been prove to be promising in the determination of V(III) and V(V) ions and could be applied to several simple and complex sample. It is rapid, selective and sensitive. Above all most of the reported methods involve extraction into spurious organic solvents where as the present methods are simple, non extractive, and reasonably accurate.

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Citation: Okenwa CJ, Ukoha PO, Chinyere EC, et al. Quantification and speciation of trac. E amount of vanadium (iii) and vanadium (v) using 2-[(e)-[3-[(e)-(2 hydroxyphenyl) methyleneamino]phenyl]iminomethyl]phenol. *J Anal Pharm Res.* 2021;10(4):136–145. DOI: 10.15406/japlr.2021.10.00377

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