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 (N₂O₂) 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, '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

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 anticancer6-8 antifungal9-10 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 heterogenous catalyst.12-17 Schiff base ligands are able to coordinate many different metals18-20 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 iron21-23 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-hydroxylaldehyde as chromogenic agent.

Materials and methods

All the reagents, O - phenylenediamine (Sigma Aldrich), 2 Hydroxybenzaldehyde (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’ Procedure24 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.25 5.41 g of (0.05 mole) of O - phenylenediamine was dissolved in dimethyl formamide (50 cm³) and 2 cm³ 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°C. The Schiff base was cooled, washed with ethanol, recrystallized and kept in a desiccator over fused CaCl₂. (91% yield, m.p. 112- 115°C) Figure 1.
Quantification and speciation of trace E amount of vanadium (iii) and vanadium (v) using 2-[(E)-2-[3-[(E)-(2-hydroxyphenyl)methyleneamino]phenyl]iminomethyl]phenol

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Figure 1 Synthesis of the Schiff base.

Synthesis of the metal complexes

The vanadium(III) and vanadium(V) complexes of 2-[(E)-2-[3-[(E)-(2-hydroxyphenyl)methyleneamino]phenyl]iminomethyl]phenol (HPMAPIP) were synthesized according to literature. In each case, a solution of the ligand 1.898 g (0.003 M) in ethanolic solution (25 cm³) was reacted with 0.500 g (0.00015 mole) solution of vanadium (III) and 0.350 g of vanadium (V) in ethanol (25 cm³). 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 CaCl₂.

Preparation of alloy steel sample solution

5 g of the alloy sample was dissolved in a mixture of 2 M HCl and 10 M HNO₃. The resulting solution was evaporated to a small volume. To this, 5 cm³ of 1:1 H₂O and H₂SO₄ mixture was added and evaporated to dryness. The residue was dissolved in 15 cm³ of distilled water and filtered through Whatman filter paper no. 40. The filtrate was collected in a 100 cm³ volumetric flask and made up to 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. 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 r² = 0.9733 indicating a good linear relationship between absorbance and concentration. 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.]
Quantification and speciation of trace amount of vanadium (III) and vanadium (V) using 2-[(E)-[3-[E)-(2-hydroxyphenyl)methyleneamino]phenyl]iminomethyl]phenol

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).

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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 S cm⁻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      | S     | 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⁻¹cm⁻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; IOSA(ppm) | 2.3         | 0.01         |
| Linear dynamic range (ppm) | 0.488 - 3.904 | 0.488 - 3.904 |

Sₐ = 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 cm⁻¹. This has been ascribed to the presence of molecular hydrogen bonding. The absence of absorption band in the 3509-3310 cm⁻¹ and 1740 – 1695 cm⁻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 3056 cm⁻¹, which shifted to higher intensity at 3061 cm⁻¹ in both V(III) and V(V) complexes. This is in conformity with the values given by literature. 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 attributable to ν(M-N).

The presence of bands at 547-544 cm⁻¹ was due to ν(M-O) in both complexes further supports coordination of the phenolic oxygen to the metal centre. In the IR 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.

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) | v(O-H) |
| 3056 (m) | 3061.13(w) | 3061.13(w) | v(C-H) of Aromatic |
| 2919 (w) | 2929.00(w) | 2929.04(w) | Aromatic overtones & combination bend |
| 1615.44(s) | 1596.15(s) | 1602.90(s) | v(C=N) |
| 1479.45(m) | 1452.45(m) | 1452.45(m) | v(C=C) |
| 1275.97(s) | 1312.60(s) | 1312.60(s) | v(C-O) |
| 1107.18(w) | 1144.79(m) | 1141.90(w) | v(C-H) out of plane |
| 901.75(m) | 980.84(m) | 982.76(m) | C-H Bending |
| 932.6(w) | 754.19(s) | 754.19(s) | v(C-H) out of plane |
| 640.39(w) | 635.57(w) | 621.10(w) | v(=C-H) deformation |
| 543.77(w) | 546.84(w) | 546.84(w) | M - O bending |
| 494.98(w) | 447.50(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/aplr.2021.10.00377
Table 5 ¹H NMR spectral Data of the HPMAPIP in CDCl₃ 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(%) |
|------------|---------------------|------------|-----------------------|
| Mg²⁺ Added as MgO | 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                    |
| Fe³⁺ added as FeCl₃ | 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                 |
| Cu²⁺ added as CuSO₄·SH₂O | 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                   |
| Ca²⁺ added as CaSO₄ | 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                  |

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Quantification and speciation of trace amount of vanadium (III) and vanadium (V) using 2-[[e]-[3-[[e]-[2-(hydroxyphenyl)]methyleneamino]phenyl]iminomethyl]phenol

| Ions Added | Concentration (ppm) | Absorbance | Interference level(%) |
|------------|---------------------|------------|-----------------------|
| Cr<sup>3+</sup> added as Cr(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O | 0.6 | 0.141 | 2.2 |
| | 0.7 | 0.158 | 14.5 |
| | 1.0 | 0.166 | 20.3 |
| | 1.15 | 0.169 | 22.5 |
| | 1.5 | 0.194 | 40.6 |
| | 1.7 | 0.209 | 51.4 |
| | 2.0 | 0.220 | 59.4 |
| Al<sup>3+</sup> | 0.5 | 0.190 | 37.7 |
| | 0.8 | 0.170 | 23.2 |
| | 1.1 | 0.174 | 26.1 |
| | 1.38 | 0.160 | 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.

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

| Ions Added | Concentration (ppm) | Absorbance | Interference level (%) |
|------------|---------------------|------------|------------------------|
| Mg\(^{2+}\) Added as Mg\(_2\)O | 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\(_2\)O | 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\(_2\)O | 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(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, Patil and Dhuley, Nagabhushana et al., Wang et al. 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.

**References**

1. Pyrzyska Krystyna, Wierzbicki, Tomasz. Separation of Vanadium Species in Natural Waters. *Microchimica Acta*. 2004;147:59–64.
2. Karia FD, Parsania PH. Synthesis, biological and thermal properties of Schiff bases of bisphenol-C. *Asian Journal of Chemistry*. 1999;11:991–995.
3. More PG, Bhalvankar RB, Pattar SC. Schiff bases derived from substituted-2-aminothiazole and substituted salicylaldehyde and 2-hydroxy-1-naphthaldehyde exhibits antibacterial and antifungal activity. *Journal of Indian Chemical Society*. 2001;78:474–475.
4. El-Masyr AH, Fahmy HH, Abdelwahed SHA. Synthesis and antimicrobial activity of some new benzimidazole derivatives. *Molecules*. 2000;5:1429–1438.
5. Baseer MA, Jadhav VD, Phule RM, et al. Synthesis and antimicrobial activity of some new Schiff bases. *Asian Journal of Chemistry*. 2000;16:553–556.
6. Desai SB, Desai PB, Desai KR. Synthesis of Some Schiff bases, thiadiazolones, and azetidinones derived from 2,6-diamino benzol[1,2-d,4,5-d’] Bis thiazole and their anticancer activities. *Heterocyclic Communication*. 2001;7:83–90.
7. Hodnett EM, Dann WJ. Structure-antitumor activity correlation of some Schiff bases. *Journal of Medicinal Chemistry*. 1970;13:768–770.
8. Pathak P, Jolly VS, Sharma KP. Synthesis and biological activities of some new substituted aryl azo Schiff bases. *Asian Journal of Chemistry*. 2000;16:161–162.
9. Pandeya SN, Siriram D, Nath G, et al. Synthesis and antimicrobial activity of Schiff and Mannich bases of inatin and its derivatives with pyrimidine. *Il Farmaco*. 1999;54:624–628.
10. Singh WM, Dash BC. Synthesis of some new Schiff bases containing thiazole and oxazole nuclei and their fungicidal activity. *Pesticides*. 1988;22:33–37.
11. Samadihya S, Halve A. Synthetic utility of Schiff bases as potential herbicidal agents. *Asian Journal of Chemistry*. 2001;17:119–122.
12. Clercq BD, Verpoort F. Atom transfer radical polymerization of vinyl monomers mediated by Schiff base ruthenium-alkylidene catalysts and the adventitious effect of water in polymerizations with the analogous cationic complexes. *Macromolecules*. 2002;35:8943–8947.
13. Opstal T, Verpoort F. Ruthenium indenyldiene and vinylidene complexes bearing Schiff bases: potential catalysts in Enol –Ester Synthesis. *Synlett*. 2002;6:935–941.
14. Opstal T, Verpoort F. Synthesis of highly active Ruthenium indenyldiene complexes for atom transfer radical polymerization and ring opening metathesis polymerization. *Angew. Chemical*. 2003;42:2876–2879.
15. Pal SN, Pal SA. A Diruthenium(III) Complex possessing a diazine and two chloride bridges: synthesis, structure, and properties. *Inorganic Chemistry*. 2001;40:4807–4810.
16. Clercq BD, Verpoort F. Assessing the scope of the introduction of Schiff bases as co-ligands for monometallic and homo bimetallic ruthenium ring-opening metathesis polymerisation and ring-closing metathesis initiators. *Advanced Synthesis & Catalysis*. 2002;34:639–648.

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

17. Clercq BD, Lefebvre F, Verpoort F. Immobilization of multifunctional Schiff base containing ruthenium complexes on MCM-41. Applied catalysis. 2000;247:345–364.
18. Khalil SME. Dioxouranium(VI) Schiff base complexes as ligands towards Cu(II) and Ni(II) ions. Chemistry Papers. 2000;54:12–18.
19. Osman AH. Synthesis and Characterization of Cobalt (II) and Nickel(II) Complexes of Some Schiff Bases derived from 3-hydrazino-6-methyl[1,2,4] triazin-5(4H)one. Transition Metal Chemistry. 2006;31:35–41.
20. Sousa C, Freire C, De Castro B. Synthesis and Characterization of Benzo-15-Crown-5 Ethers with appended N2O Schiff bases. Molecules. 2003;8:894–900.
21. Pyrzyńska K, Wierzbicki T. Trends in speciation analysis of vanadium in Environment. Talanta. 2004;64.
22. Pyrzyńska K, Wierzbicki T, Chen. Spectrophotometric methods and vanadium analysis. 2004.
23. Soete D, Gijbels JH. Neutron Activation Analysis. Wiley Interscience. New York. 1972.
24. Kučera J. Methodological developments and applications of neutron activation analysis. Journal of Radioanalytical and Nuclear Chemistry. 2007;273(2):273–280.
25. Kolthoff IM, Sandell EB, Mecham EJ. Quantitative chemical analysis. Macmillan. 1969.
26. A Papaioannon, M Manos, S Karkabanas. Journal of Inorganic. Biochemistry. 2004;959–996.
27. Marina Cindri, Mirta Rub-ı, Gerald Giester. Oxovanadium(V) Complexes of Salicylaldehyde, Croatica Chimica Acta. 2007;583–590.
28. Petya Racheva, Kiril Blazhev Gavazov, Vanya Dimitrova Lekova. Journal of Iran Chemical Research. 2008;1:113–121.
29. Pyrzyńska K, Wierzbicki T, Chen. Spectrophotometric methods and vanadium analysis. 2004.
30. Soete D, Gijbels JH. Neutron Activation Analysis. Wiley Interscience. New York. 1972.
31. Raman N, Raja JJ, Joseph J, et al. Molecular designing, structural elucidation and comparison of the cleavage ability of oxovanadium(IV) Schiff base complexes. Journal of the Chilean Chemical Society. 2009;52:1099–1103.
32. Sadeek SA, Teleg SM, Refat MS, et al. Preparation, thermal and vibrational studies of UO(acac-o-phdn)(L)(L=H2O, py, DMF and Et3N). Journal of Coordination Chemistry. 2005;58:1077–1085.
33. Silverstein RM, Bassler GC, Morrill TC. Spectroscopic Identification of Organic Compounds, 5th Edition, Wiley: New York. 1991.
34. Turel I, Gruber K, Leban I, et al. Synthesis, crystal structure and characterization of three novel compounds of the quinolone family member (norfloxacin). Journal of Inorganic Biochemistry. 1996;61:197–212.
35. Turel I, Leban I, Klintschar G, et al. Synthesis, crystal structure, and characterization of two metal-quinolone compounds. Journal of Inorganic Biochemistry. 1997;66:77–82.
36. Turel I, Bukovec P, Quiros M. Crystal structure of ciprofloxacin hexahydrate and its characterization. International Journal of Pharmaceutics. 1997;152:59–65.