Chapter 5

SIMULTANEOUS SPECTROPHOTOMETRIC
DETERMINATION OF SOME TRANSITION METAL IONS
INTRODUCTION:

The two important characteristic features of a spectrophotometric method are sensitivity and specificity or selectivity. Sensitive methods are now a common feature for every metal ion. However, there exists few specific methods. The degree of selectivity of a method is dependent on the complexity of the material or the sample being analysed.

In the world of ever increasing technological progress the modern analytical chemist is confronted with the analysis of materials that are highly complex in nature. In many cases the simultaneous presence of other elements is in fact a potential source of interference whenever quantitative determinations are carried out. In such cases the simultaneous determination of two or more metal ions enhances the selectivity of the method to a greater extent. Simultaneous spectrophotometric analysis eliminates wavelength independent matrix system effect as well as the analysis of two or more component systems. It also saves the sample, time and thereby enhances the speed of analysis.

From this point of view spectrophotometric methods for the simultaneous determination of metal ions is of immense value and is receiving the attention of many chemists in recent times. Hence the author has developed several spectrophotometric methods for the simultaneous determination of two metal ions when present in admixture with each other in several real samples. The results are presented in this chapter.

However, before this a brief account of the theoretical aspects of the simultaneous spectrophotometric procedures are presented here under.

Principles of multi-component spectrophotometric analysis:

If a system containing several absorbing components follows the principle of additivity of absorbances, i.e., the absorbance of the system is equal to the sum of the absorbances of all individual components at a particular wavelength, and if no mutual interaction takes place between them, then

$$A_j = \sum_{i=1}^{n} \epsilon_{ij} C_i A_j$$

Where $\epsilon$ is the molar absorptivity of the component $i$ at the wavelength $J$.

The additivity of absorbance is easily tested by calculation of absorbances for mixture of standard solutions containing known analyte concentrations. The determination of a mixture comprising of several absorbing components in solution is carried out by measuring the absorbance at selected wavelengths and solving a set of simultaneous equations of the type

$$A_j = \epsilon_1 C_1 + \epsilon_2 C_2 \ldots \epsilon_n C_n \lambda J$$
The accuracy and precision of the calculations is dependent on the number of components present and the selection of suitable wavelengths. At the wavelength selected, always one component absorbs strongly, whereas other components absorb less intensely.

\[ r_{ij} = r_1 > > r_2, r_3, r_4 \ldots \]

The molar absorptivities of all the components at the selected wavelengths are previously determined from solutions of the pure components under the same conditions as for the mixture.

The validity of Beer’s law for single components need not be maintained for their mixture. Even if the Beer’s law is not obeyed for one component of the mixture, the sum of the absorbances will still be correct for mixtures with a constant concentration of such a component.

The precision and accuracy of the calculated results depends on the number of unknown components in the mixture. The error of the resulting data considerably increases, if more than four absorbing species in unknown concentration are to be determined.

The set of equations containing \( i \) unknowns is solved by the familiar elimination procedures or with the aid of determinants but computer procedures are usually used if more than two absorbing components are to be determined.

**Two component analysis:**

For a system containing two absorbing species 1 and 2, the absorbance of the system at the selected wavelengths (\( \lambda_1 \) and \( \lambda_2 \)) i.e., \( A_1 \) and \( A_2 \) are given by the following equations.

\[
A_1 = r_1 r_{11} C_1 + r_2 r_{12} C_2 \quad \ldots \quad (1)
\]

\[
A_2 = r_1 r_{21} C_1 + r_2 r_{22} C_2 \quad \ldots \quad (2)
\]

Solving the equations (1) and (2) we get

\[
C_1 = \frac{A_1 r_{22} - A_2 r_{12}}{r_1 r_{22} - r_2 r_{12}} \quad \ldots \quad (3)
\]

\[
C_2 = \frac{A_1 r_{11} - A_2 r_{12}}{r_1 r_{11} - r_2 r_{12}} \quad \ldots \quad (4)
\]

Where \( C_1 \) and \( C_2 \) are the concentrations of analytes 1 and 2, \( r_1 r_{11}, r_2 r_{12} \) and \( r_1 r_{21}, r_2 r_{22} \) are the molar absorptivities of the components denoted by superscripts 1 and 2 at the wavelengths \( \lambda_1 \) and \( \lambda_2 \) respectively.
Section 1

SIMULTANEOUS SPECTROPHOTOMETRIC DETERMINATION OF TITANIUM(IV) AND
MOLYBDENUM (VI)

Titanium(IV) reacts with 2,4-DHBINH in the pH range 1.0 to 7.0 to form a red coloured water
insoluble complex, soluble in presence of a mixture of DMF and n-butanol. The colour intensity is
maximum in the pH range 1.2 to 3.5 (cf section 4.2). The complex shows two $\lambda_{\text{max}}$ one at 430 nm
and the other at 500 nm. Molybdenum(VI) reacts with 2,4-DHBINH giving an orange yellow
coloured complex in the pH range 1.0 to 4.0 and has maximum intensity in the pH range 1.0-3.0.
(cf section 4.3) with $\lambda_{\text{max}}$ at 445 nm.

It is thus clear that both Ti(IV) and Mo(VI) reacts with 2,4-DHBINH forming coloured complexes and have maximum intensities at pH 1.5. It is thus possible to determine Ti(IV) and
Mo(VI) simultaneously with 2,4-DHBINH. The following procedure is adopted for the purpose.

Absorption Spectra

The absorption spectra of the following solutions are recorded for selecting the
wavelengths for absorbance measurements.

Solution (a)

The absorption spectrum of 2,4-DHBINH is recorded by following the procedure in 3.a and
is presented in fig 5.1.1.c.

Solution (b)

The absorption spectrum of the Ti(IV)-2,4-DHBINH complex (2.5 X 10$^{-5}$ M) is recorded by
following the procedure in 3.b and presented in Fig.5.1.1.a.

Solution (c)

The absorption spectrum of the Mo(VI)-2,4-DHBINH complex is recorded by following the
procedure in 3.b. and presented in Fig. 5.1.1.b.

From the spectra in Fig.5.1.1, it is clear that Ti(IV) has two $\lambda_{\text{max}}$ one at 430 nm and the other
at 500 nm. Mo(VI) shows the $\lambda_{\text{max}}$ at 445 nm. It is further noticed that Ti(IV) shows considerable
absorbance at and beyond 555 nm. Mo(VI) has maximum absorbance at 445 nm and negligible
or no absorbance at and beyond 555 nm. Ti(IV) on the other hand shows appreciable absorbance
at 445 nm or 560 nm. It is also noticed that the total absorbance at 445 nm or at 560 nm is equal
to the sum of the absorbance values due to individual Mo(VI) and Ti(IV) species at these
Fig. 5.1.1. Absorption spectra of
a. Ti(IV) - 2,4-DHBINH system Vs reagent blank
b. Mo(VI) - 2,4-DHBINH system Vs reagent blank
c. 2,4-DHBINH Vs buffer blank

\[ [\text{Ti(IV)}] = [\text{Mo(VI)}] = 2.5 \times 10^{-5} \text{ M} \]
\[ [\text{2,4-DHBINH}] = 7.5 \times 10^{-4} \text{ M}; \text{ pH} = 1.5 \]
wavelengths. This suggests that it is possible to determine Mo(VI) and Ti(IV) simultaneously using 2,4-DHBINH by measuring the absorbance at 445 nm and 560 nm.

Applicability of Beer's Law:

To establish whether Beer's law is applicable to the metal ions at 445 nm and 560 nm, the absorbance measurements with any metal ion are made at both these wavelengths (following the procedure in 3.h). The results are presented in Figs. 5.1.2 and 5.1.3.

From the linear plots it is observed that Beer's law is obeyed for Ti(IV) in the range 0.36 to 3.80 µg/ml both at 445 nm and 560 nm. Mo(VI) obeys Beer's law in the range 0.30 to 6.00 µg/ml at 445 nm. The molar absorptivities for Ti(IV) are 1.08 X 10^4 l mol⁻¹ cm⁻¹ and 0.72 X 10^4 l mol⁻¹ cm⁻¹ at 445 nm and 560 nm respectively. The molar absorptivities for Mo(VI) at 445 nm is 1.02 X 10^4 l mol⁻¹ cm⁻¹. It is also observed that the absorbance at 445 nm and 560 nm is equal to the sum of the absorbances due to the individual Ti(IV) and Mo(VI) species at these wavelengths. Hence simultaneous determination of Ti(IV) and Mo(VI) is carried out.

The absorbance of the solution mixture containing Ti(IV) and Mo(VI) complex species is recorded at 445 nm and 560 nm at pH 1.5. From the absorbance values at 560 nm (Where Ti(IV) alone absorbs) the amount of titanium is computed from the predetermined calibration plot (Fig.5.1.2). The amount of Mo(VI) is calculated from the absorbance values measured at 445 nm (A_445 nm after subtracting the absorbance due to Ti(IV) at this wavelength). The absorbance of Ti(IV) at 445 nm is 1.5 times to its absorbance at 560 nm. Therefore the amount of Ti(IV) is computed from the absorbance measured at 560 nm. The equation employed to calculate the amount of Mo(VI) at 445 nm is

\[
A_{445} - (A_{560} \times 1.5)
\]

\[
\text{Mo(VI)} = \frac{\text{Molar absorptivity of Mo(VI) at 445 nm}}{95.94 \times 10^3} \times \frac{A_{445}}{1.5}
\]

Where \(A_{445}\) and \(A_{560}\) are the absorbance values of the solution mixture at 445 nm and 560 nm respectively. \(\varepsilon_{445}\) is the molar absorptivity of Mo(VI) at 445 nm. 95.94 X 10^3 is the factor to convert the concentrations of Mo(VI) into amounts.

The simultaneous determination of Ti(IV) and Mo(VI) in synthetic mixtures is carried out by the following procedure.
Fig. 5.1.2. Absorbance Vs amount of Ti(IV)  
\[ [2,4\text{-DHBINH}] = 1.25 \times 10^{-3} \text{M}; \text{pH} = 1.5 \]

\[
A_{445} = 0.224 \, C + 0.0010 \\
A_{560} = 0.149 \, C + 0.0014
\]

Fig. 5.1.3. Absorbance Vs amount Mo(VI)  
\[ [2,4\text{-DHBINH}] = 1.25 \times 10^{-3} \text{M}; \text{pH} = 1.5 \]

\[
A_{445} = 0.102 \, C + 0.0033
\]
Procedure

In a 10ml volumetric flask containing 5 ml of buffer (pH1.5) solution, 1.25 ml of DMF, 1 ml of n-butanol, 1.25 ml of 2,4-DHBINH (1 X 10^{-2} M) solution and a known aliquots of the synthetic mixture of Ti(IV) and Mo(VI) are taken. The contents of the flask are made upto the mark with distilled water. The absorbance of the solution is measured at 445 nm and 560 nm against the reagent blank. The amount of Ti(IV) is computed from the absorbance measured at 560 nm. The amount of Mo(VI) is calculated from the equation 5.1.1. The results are presented in Table 5.1.1.

<table>
<thead>
<tr>
<th>Amount taken(μg/ml)</th>
<th>Amount found(μg/ml)</th>
<th>Relative Error(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti(IV)</td>
<td>Mo(VI)</td>
<td>Ti(IV)</td>
</tr>
<tr>
<td>0.479</td>
<td>0.959</td>
<td>0.472</td>
</tr>
<tr>
<td>0.479</td>
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<td>0.959</td>
<td>0.958</td>
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<td>1.420</td>
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</tr>
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<td>2.395</td>
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<td>2.379</td>
</tr>
<tr>
<td>2.876</td>
<td>0.959</td>
<td>2.660</td>
</tr>
</tbody>
</table>

Applications

The proposed method is applied to an alloy steel for the simultaneous determination of Ti(IV) and Mo(VI). The alloy steel is brought into solution by adopting the procedure in 3.r. The sample is analysed by the following procedure.

Procedure

A known aliquot of the sample solution is added to 5 ml of buffer solution (pH 1.5) in a 10 ml volumetric flask. 1.25 ml of DMF, 1 ml of n-butanol and 1 ml of 0.1 M ascorbic acid (to mask
Fe(II) and 1.25 ml of 2,4-DHBiNH (1 X 10^{-2} M) solution are added. The contents are made up to the mark with distilled water. The absorbance of the solution is measured at 445 nm and 560 nm against the reagent blank. The amount of Ti(IV) is computed from the absorbance measured at 560 nm. The amount of Mo(VI) is computed from equation 5.1.1. The results are presented in Table 5.1.2.

**TABLE 5.1.2**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Amount taken(μg/ml)</th>
<th>Amount found(μg/ml)</th>
<th>Relative Error(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ti(IV)</td>
<td>Mo(VI)</td>
<td>Ti(IV)</td>
</tr>
<tr>
<td>BAS-387 alloy*</td>
<td>2.95</td>
<td>5.83</td>
<td>2.91</td>
</tr>
</tbody>
</table>

* : Average of six determinations.

a 41.9% Ni; 36% Fe; 12.46% Cr, 5.83% Mo; 2.95% Ti; 0.24% Al; 0.21% Co; 0.08% Mn; 0.032% Cu; 0.030% C;

**Conclusions :**

Titanium and molybdenum are industrially important metals and are found in association with each other. Hence their simultaneous spectrophotometric determination offers a more selective method for the analysis of steels and alloys. The present method is rapid, simple, and sensitive and does not involve extraction or separation for quantitative recovery of the metal ions.
Section 2:

SIMULTANEOUS DETERMINATION OF TITANIUM(IV) AND VANADIUM(V)

It is established in section 2 of chapter 4 that titanium(IV) reacts with 2,4-DHBINH forming red coloured complex with maximum colour intensity in the pH range 1.2 to 3.5 in presence of n-butanol and DMF mixture. Similarly in section 4 of Chapter 4, it is observed that vanadium(V) gives an orange yellow coloured complex with 2,4-DHBINH in the pH range 1.5 to 6.0. Further at pH 2.0 both Ti(IV) and V(V) complexes have maximum colour intensities. Therefore it is possible to determine Ti(IV) and V(V) simultaneously at pH 2.0. The following procedure is adopted for the purpose.

Absorption spectra

The absorption spectra of the following solutions are recorded to fix up the wavelengths for absorbance measurements.

Solution (a)

The absorption spectrum of solution containing Ti(IV)-2,4-DHBINH complex is recorded and presented in Fig.5.2.1a.

Solution (b)

The absorption spectrum of the solution containing V(V)-2,4-DHBINH complex is recorded by following the procedure in 3.b and the spectrum is presented in Fig. 5.2.1b.

Solution (c)

The absorption spectrum of the solution containing the reagent (2,4-DHBINH) is also recorded and presented in Fig.5.2.1c.

From the spectra in Fig.5.2.1, it is noticed that Ti(IV) shows maximum absorbance at 430 nm and 500 nm, while V(V) has maximum absorbance at 440 nm. At 500 nm Ti(IV) has more absorbance than V(V). Though V(V) has maximum absorbance at 440 nm than Ti(IV), the difference in the absorbance values between V(V) and Ti(IV) is higher at 450 nm than at 440 nm. Hence 450 nm and 500 nm are selected for carrying out simultaneous spectrophotometric determination of Ti(IV) and V(V).
Fig. 5.2.1. Absorption spectra of
a. Ti(IV) - 2,4-DHBINH system Vs reagent blank
b. V(V) - 2,4-DHBINH system Vs reagent blank
c. 2,4-DHBINH Vs buffer blank

[Ti(IV)] = [V(V)] = 2.5\times10^{-5}\text{M}
[2,4-DHBINH] = 7.5\times10^{-4}\text{M}; \text{pH} = 2.0
Applicability of Beer’s law

The absorbance measurements of the solutions containing varying amounts of Ti(IV) and V(V) are made at 450 nm and 500 nm by adopting the procedure in 3.h to establish the applicability of Beer’s law. The results are presented in Figs. 5.2.2 and 5.2.3.

The linear plots indicate that Ti(IV) obeys Beer’s law in the range 0.12 to 2.15 μg/ml, while V(V) obeys in the range 0.30 to 3.06 μg/ml at both 450 nm and 500 nm. The molar absorptivities for V(V) are $1.40 \times 10^4$ mol$^{-1}$ cm$^{-1}$ and $0.32 \times 10^4$ mol$^{-1}$ cm$^{-1}$ at 450 nm and 500 nm respectively, while the molar absorptivities for Ti(IV) are $1.34 \times 10^4$ mol$^{-1}$ cm$^{-1}$ and $1.07 \times 10^4$ mol$^{-1}$ cm$^{-1}$ at 500 nm and 450 nm respectively. It is also observed that the total absorbance at 450 nm and 500 nm is equal to the sum of the absorbance due to individual V(V) and Ti(IV) species at these wavelengths. Hence simultaneous determination of Ti(IV) and V(V) is attempted.

The absorbance of the solution containing the mixture of Ti(IV) and V(V) is measured at 450 nm and 500 nm at pH 2. From the absorbance data the amounts of Ti(IV) and V(V) are determined using the following simultaneous equations.

$$A_{450} = \varepsilon_{450} C_V + \varepsilon_{450}^{-1} C_{Ti} \tag{5.2.1}$$
$$A_{500} = \varepsilon_{500} C_V + \varepsilon_{500}^{-1} C_{Ti} \tag{5.2.2}$$

Converting the concentrations into amounts (μg/ml) and solving the equations 5.2.1 and 5.2.2, we get

$$Ti = \frac{A_{500} \varepsilon_{500} \varepsilon_{450} C_V - A_{450} \varepsilon_{450} \varepsilon_{500} C_{Ti}}{\varepsilon_{450} \varepsilon_{450}^{-1} - \varepsilon_{450} \varepsilon_{500}^{-1}} \times 47.90 \times 10^3 \tag{5.2.3}$$

$$V = \frac{A_{450} \varepsilon_{450} \varepsilon_{500} C_{Ti} - A_{500} \varepsilon_{500} \varepsilon_{450} C_V}{\varepsilon_{450} \varepsilon_{450}^{-1} - \varepsilon_{450} \varepsilon_{500}^{-1}} \times 50.94 \times 10^3 \tag{5.2.4}$$

Where $A_{450}$ and $A_{500}$ are the absorbance values of the solution mixture at 450 nm and 500 nm respectively. $\varepsilon$ values represent the molar absorptivities of Ti(IV) and V(V) complexes at the wavelengths specified. 50.94 and 47.90 are factors to convert the concentrations into amounts for V(V) and Ti(IV) respectively.
Fig. 5.2.2. Absorbance Vs amount of Ti(IV)
\[ [2,4-DHBINH] = 1.25 \times 10^{-3} \text{M}; \text{pH} = 2.0 \]

\[
\begin{align*}
A_{450} &= 0.224C + 0.0010 \\
A_{500} &= 0.292C + 0.0005
\end{align*}
\]

Fig. 5.2.3. Absorbance Vs amount of V(V)
\[ [2,4-DHBINH] = 1.25 \times 10^{-3} \text{M}; \text{pH} = 2.0 \]

\[
\begin{align*}
A_{450} &= 0.274C - 0.00004 \\
A_{500} &= 0.060C + 0.0003
\end{align*}
\]
The simultaneous determination of Ti(IV) and V(V) in synthetic mixtures is carried out by adopting the following procedure.

**Procedure**

A known aliquot of the synthetic mixture of Ti(IV) and V(V) is added to a 10 ml volumetric flask, containing 5 ml of buffer (pH 2.0) solution, 1.25 ml of DMF and 1.25 ml of 2,4-DHBINH (1 x 10^{-2} M) solution and 1 ml n-butanol is added. The contents are made upto the mark with distilled water. The absorbance is measured at 450 nm and 500 nm against the reagent blank. The amounts of Ti(IV) and V(V) are computed from the equations. 5.2.3. and 5.2.4 respectively. The results are presented in Table 5.2.1.

**TABLE 5.2.1.**

Simultaneous determination of Ti(IV) and V(V) in synthetic mixtures:

<table>
<thead>
<tr>
<th>Amount taken (μg/ml)</th>
<th>Amount found (μg/ml)</th>
<th>Relative Error(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti(IV)</td>
<td>V(V)</td>
<td>Ti(IV)</td>
</tr>
<tr>
<td>0.383</td>
<td>0.764</td>
<td>0.386</td>
</tr>
<tr>
<td>0.383</td>
<td>1.019</td>
<td>0.385</td>
</tr>
<tr>
<td>0.383</td>
<td>1.527</td>
<td>0.387</td>
</tr>
<tr>
<td>0.383</td>
<td>1.782</td>
<td>0.376</td>
</tr>
<tr>
<td>0.383</td>
<td>2.038</td>
<td>0.379</td>
</tr>
<tr>
<td>0.479</td>
<td>0.764</td>
<td>0.479</td>
</tr>
<tr>
<td>0.766</td>
<td>0.764</td>
<td>0.770</td>
</tr>
<tr>
<td>0.958</td>
<td>0.764</td>
<td>0.945</td>
</tr>
<tr>
<td>1.149</td>
<td>0.764</td>
<td>1.135</td>
</tr>
<tr>
<td>1.532</td>
<td>0.764</td>
<td>1.520</td>
</tr>
</tbody>
</table>

Conclusions:

Titanium and vanadium are industrially important metals and are found association with each other. The present method for the simultaneous spectrophotometric determination of Ti(IV) and V(V) is simple, and sensitive, and does not involve any extraction for quantitative determination of the metal ions.
Section 3

SIMULTANEOUS DETERMINATION OF IRON(III) AND VANADIUM(V)

It is established in chapter 4 section 5 that iron(III) reacts with 2,4-DHBINH to form an yellowish brown coloured complex in the pH range 1.5-7.0. The complex shows maximum absorbance in the pH range 2.5 to 5.5. Further in chapter 4.4 it is observed that vanadium forms an orange yellow coloured complex with 2,4- DHBINH in the pH range 1.5 to 6.0. The complex shows two isobestic points one in the pH range 1.5 to 2.5 and the other in the pH range 4.0 to 6.0. Therefore it is possible to carry out a simultaneous determination of Fe(III) and V(V) at pH 5.0.

Absorption spectra :

To fix up the wavelengths for absorbance measurements, the spectra of the following solutions are recorded.

The absorption spectra of the solutions containing Fe(III)-2,4- DHBINH complex and V(V)-2,4-DHBINH complex are recorded by following the procedure in 3.b and presented in Figs. 5.3.1a and 5.3.1b respectively. The absorption spectrum of 2,4-DHBINH is also recorded by adopting the procedure in 3.a and presented in Fig. 5.3.1c.

From the spectra in Fig. 5.3.1, it is observed that Fe(III) and V(V) show maximum absorbance at 390 nm and 410 nm respectively. The reagent shows appreciable absorbance at 390 nm. It is further noticed from Fig 5.3.1 that the difference in absorbance between Fe(III) and V(V) complexes is maximum at 420 nm and 460 nm, such that V(V) has more absorbance than Fe(III) at 420 nm and less absorbance at 460 nm. The total absorbance is equal to the sum of the absorbance values due to individual Fe(III) and V(V) species at these wavelengths. Hence 420 nm and 460 nm are selected for the purpose of carrying out the simultaneous determination of Fe(III) and V(V).

Applicability of Beer’s law :

To establish the applicability of Beer’s law, the absorbance measurements of the solutions containing varying amounts of Fe(III) and V(V) are made at 420 nm and 460 nm by adopting the procedure in 3.h. The results are presented in Figs. 5.3.2. and 5.3.3.

From the linear plots it is observed that Fe(III) obeys Beer’s law in the range 0.30 to 3.50 µg/ml while V(V) obeys in the range 0.50 to 2.55 µg/ml at both 420 nm and 460 nm. The molar absorptivities for Fe(III) are 0.97 X 10^4 l mol^-1 cm^-1 and 0.70 X 10^4 l mol^-1 cm^-1 at 420 nm and 460 nm respectively. The molar absorptivities for V(V) are 1.34 X 10^4 l mol^-1 cm^-1 and 0.20 X 10^4 l mol^-1 cm^-1 at 420 nm.
Fig. 5.3.1. Absorption spectra of

a. Fe(III) - 2,4-DHBINH system Vs reagent blank

b. V(V) - 2,4-DHBINH system Vs reagent blank

c. 2,4-DHBINH Vs buffer blank

$[\text{Fe(III)}] = [\text{V(V)}] = 2.5 \times 10^{-5} \text{ M}$

$[2,4\text{-DHBINH}] = 2.5 \times 10^{-4} \text{ M}; \text{ pH} = 5.0$
Fig. 5.3.2. Absorbance Vs amount of Fe(III)

\[ [2,4\text{-DHBINH}] = 3.00 \times 10^{-4} \text{M}; \text{pH} = 5.0 \]

Fig. 5.3.3. Absorbance Vs amount of V(V)

\[ [2,4\text{-DHBINH}] = 3.00 \times 10^{-4} \text{M}; \text{pH} = 5.0 \]
and 460 nm respectively. It is also noticed that the total absorbance at 420 nm and 460 nm is equal to the sum of the absorbances due to the individual Fe(III) and V(V) species at the wavelengths specified. Hence the possibility of simultaneous determination of Fe(III) and V(V) is explored.

The absorbance of the solution mixture containing Fe(III) and V(V) is measured at 420 nm and 460 nm at pH 5.0. From these absorbance values the amounts of Fe(III) and V(V) are calculated using the following simultaneous equations.

\[
\begin{align*}
A_{420} &= \varepsilon_{420}^V CV + \varepsilon_{420}^{Fe} C_{Fe} \\
A_{460} &= \varepsilon_{460}^V CV + \varepsilon_{460}^{Fe} C_{Fe}
\end{align*}
\] ... 5.3.1

Converting the concentrations into amounts (\(\mu g/ml\)) and solving the equations 5.3.1. and 5.3.2.

\[
\begin{align*}
Fe &= \frac{A_{460} \varepsilon_{420}^V - A_{420} \varepsilon_{460}^V}{\varepsilon_{420}^V \varepsilon_{460}^{Fe} + \varepsilon_{420}^{Fe} \varepsilon_{460}^V} \times 58.94 \times 10^3 \\
V &= \frac{A_{420} \varepsilon_{460}^V - A_{460} \varepsilon_{420}^V}{\varepsilon_{420}^V \varepsilon_{460}^{Fe} + \varepsilon_{420}^{Fe} \varepsilon_{460}^V} \times 50.94 \times 10^3
\end{align*}
\] ... 5.3.3

Where \(A_{420}\) and \(A_{460}\) are the absorbance values of the solution mixture at 420 nm and 460 nm respectively. \(\varepsilon\) values represent the molar absorptivities of Fe(III) and V(V) complexes at the wavelength specified. 58.94 and 50.94 are the factors to convert the concentrations into amounts for Fe(III) and V(V) respectively.

The simultaneous determination of Fe(III) and V(V) in synthetic mixtures is carried out by adopting the following procedure.

**Procedure**

A known aliquot of the synthetic mixture of Fe(III) and V(V) is added to 5 ml of buffer (pH 5.0) solution, 3 ml of DMF and 0.2 ml of 2,4-DHBINH (1 X 10^{-2} M) solution taken in a 10 ml volumetric flask. The contents of the flask are made up to the mark with distilled water. The absorbance is measured at 420 nm and 460 nm against the reagent blank.
The amounts of Fe(III) and V(V) are computed from equations 5.3.3. and 5.3.4. respectively. The results are presented in Table 5.3.1.

**TABLE 5.3.1.**

<table>
<thead>
<tr>
<th>Amount taken (µg/ml)</th>
<th>Amount found (µg/ml)</th>
<th>Relative Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe(III)</td>
<td>V(V)</td>
<td>Fe(III)</td>
</tr>
<tr>
<td>0.353</td>
<td>0.509</td>
<td>0.353</td>
</tr>
<tr>
<td>0.353</td>
<td>0.764</td>
<td>0.350</td>
</tr>
<tr>
<td>0.353</td>
<td>1.019</td>
<td>0.356</td>
</tr>
<tr>
<td>0.353</td>
<td>1.273</td>
<td>0.351</td>
</tr>
<tr>
<td>0.353</td>
<td>1.528</td>
<td>0.358</td>
</tr>
<tr>
<td>0.589</td>
<td>0.764</td>
<td>0.580</td>
</tr>
<tr>
<td>0.884</td>
<td>0.764</td>
<td>0.890</td>
</tr>
<tr>
<td>1.178</td>
<td>0.764</td>
<td>1.170</td>
</tr>
<tr>
<td>1.472</td>
<td>0.764</td>
<td>1.464</td>
</tr>
<tr>
<td>1.767</td>
<td>0.764</td>
<td>1.755</td>
</tr>
<tr>
<td>2.360</td>
<td>0.764</td>
<td>2.345</td>
</tr>
</tbody>
</table>

**Applications**

The proposed method is applied to the determination of Fe(III) and V(V) in ferro-vanadium steel sample. The steel sample is brought into solution by adopting the procedure in 3.r. The amounts of Fe(III) and V(V) present in the sample are determined by employing the procedure described below.

**Procedure**

A known aliquot of the steel sample solution is added to a 10 ml volumetric flask containing 5 ml buffer solution pH 5.0, 3 ml of DMF, 0.2 ml of 2,4-DHBINH (1 X 10^-2M). The contents are made up to the mark with distilled water and the absorbance of the solution is measured at 420 nm and 460 nm against the reagent blank. The amounts of Fe(III) and V(V) are computed from the equations 5.3.3. and 5.3.4. respectively. The results are presented in Table 5.3.2.
TABLE 5.3.2.

Analysis of alloy steel sample

<table>
<thead>
<tr>
<th>Sample</th>
<th>Amount taken (µg/ml)</th>
<th>Amount found* (µg/ml)</th>
<th>Relative Error(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe(III)</td>
<td>V(V)</td>
<td>Fe(III)</td>
</tr>
<tr>
<td>Ferro-vanadium*</td>
<td>42.22</td>
<td>50.16</td>
<td>41.28</td>
</tr>
</tbody>
</table>

* : Average of six determinations.

a : 0.053% Ni; 0.414% Al; 0.054 Cu; 42.22 Fe; 50.16% V.

Conclusions:

Iron and vanadium are important alloying metals and are found in association with each other in various alloys and steels. Hence their simultaneous spectrophotometric determination is important as it enhances the selectivity of the method. Kavilentis.E1. Proposed a spectrophotometric method for the simultaneous determination of Fe(III) and V(V) in water samples. The present method is simple and rapid and sensitive for the analysis of ferro-vanadium steel.
Section 4

SIMULTANEOUS SPECTROPHOTOMETRIC DETERMINATION OF COPPER(II) AND VANADIUM(V)

Copper(II) reacts with 2,4-DHBINH forming a greenish yellow coloured complex in the pH range 1.5 to 7.0. Similarly vanadium(V) gives an orange coloured complex in DMF medium in pH range 1.5 - 6.0.

Cu(II) and V(V) have $\lambda_{\text{max}}$ at 390 nm and 410 nm respectively in the pH range 4.0-6.0. This suggests that it is possible to determine Cu(II) and V(V) simultaneously at pH 5.0. The following procedure is followed for the purpose.

Absorption Spectra

The absorption spectra of the following solutions are recorded to fix up the wavelengths for absorbance measurements.

The absorption spectra of solutions containing Cu(II)-2,4-DHBINH complex and V(V)-2,4-DHBINH complex are recorded by following the procedure in 3.b and presented in Fig. 5.4.1a and 5.4.1b respectively. The absorption spectrum of 2,4-DHBINH is also recorded by following the procedure in 3.a and presented in Fig. 5.4.1c.

The spectra in Fig. 5.4.1, indicate that Cu(II) and V(V) show $\lambda_{\text{max}}$ at 390 nm and 410 nm respectively. However, Fig. 5.4.1 shows that the difference in absorbance between Cu(II) and V(V) complexes is maximum at both 400 nm and 430 nm, such that Cu(II) has more absorbance at 400 nm and less absorbance at 430 nm than V(V). The reagent shows negligible absorbance at both the wavelengths. Hence 400 nm and 430 nm are selected for the purpose of carrying out simultaneous determination of Cu(II) and V(V).

Applicability of Beer's law

The absorbance measurements of the solutions containing varying amounts of Cu(II) and V(V) are made at 400 nm and 430 nm by following the procedure in 3.h. The results are presented in Figs. 5.4.2 and 5.4.3.

From the linear plots it is observed that Cu(II) obeys Beer's law in the range 0.38 to 2.50 $\mu$g/ml, while V(V) obeys in the range 0.20 to 2.50 $\mu$g/ml at both 400nm and 430 nm. The molar absorptivities for Cu(II) are $1.93 \times 10^4$ l mol$^{-1}$ cm$^{-1}$ and $0.98 \times 10^4$ l mol$^{-1}$ cm$^{-1}$ at 400 nm and 430 nm respectively. The molar absorptivities for V(V) are $1.44 \times 10^4$ l mol$^{-1}$ cm$^{-1}$ and $0.98 \times 10^4$ l mol$^{-1}$ cm$^{-1}$ respectively. It is noticed that the total absorbance at 400 nm and 430 nm is equal to...
Fig. 5.4.1. Absorption spectra of

a. Cu(II) - 2,4-DHBINH system Vs reagent blank
b. V(V) - 2,4-DHBINH system Vs reagent blank
c. 2,4-DHBINH Vs' buffer blank

$[\text{Cu(II)}] = [\text{V(V)}] = 2.5 \times 10^{-5} \text{ M}$

$[2,4-\text{DHBINH}] = 2.5 \times 10^{-4} \text{ M}; \text{ pH} = 5.0$
Fig. 5.4.2. Absorbance Vs amount of Cu(II)

\[ [2,4-DHBINH] = 3.00 \times 10^{-4} \text{M}; \text{pH} = 5.0 \]

\[
\begin{align*}
A_{400} &= 0.302C + 0.0011 \\
A_{430} &= 0.079C + 0.0009
\end{align*}
\]

Fig. 5.4.3. Absorbance Vs amount of V(V)

\[ [2,4-DHBINH] = 3.00 \times 10^{-4} \text{M}; \text{pH} = 5.0 \]

\[
\begin{align*}
A_{400} &= 0.268C + 0.004 \\
A_{430} &= 0.186C + 0.004
\end{align*}
\]
the sum of the absorbance values due to individual Cu(II) and V(V) species at these wavelengths. Hence the possibility of the simultaneous determination of Cu(II) and V(V) is explored.

The absorbance of the solution mixture containing Cu(II) and V(V) is measured at 400 nm and 430 nm at pH 5.0. From these absorbance values the amounts of Cu(II) and V(V) are calculated using the following simultaneous equations.

\[ A_{400} = \varepsilon_{400}^{\text{Cu}} C_{\text{Cu}} + \varepsilon_{400}^{\text{V}} C_{\text{V}} \]  
\[ A_{430} = \varepsilon_{430}^{\text{Cu}} C_{\text{Cu}} + \varepsilon_{430}^{\text{V}} C_{\text{V}} \]

converting the concentrations into amounts (\(\mu g/ml\)) and solving the equations 5.4.1 and 5.4.2 we get

\[ Cu = \frac{A_{400} \varepsilon_{430}^{\text{V}} - A_{430} \varepsilon_{400}^{\text{V}}}{\varepsilon_{400}^{\text{Cu}} \varepsilon_{430}^{\text{V}} - \varepsilon_{400}^{\text{V}} \varepsilon_{430}^{\text{Cu}}} \times 63.54 \times 10^3 \]  
\[ V = \frac{A_{430} \varepsilon_{400}^{\text{Cu}} - A_{400} \varepsilon_{430}^{\text{Cu}}}{\varepsilon_{400}^{\text{Cu}} \varepsilon_{430}^{\text{V}} - \varepsilon_{400}^{\text{V}} \varepsilon_{430}^{\text{Cu}}} \times 50.94 \times 10^3 \]

Where \(A_{400}\) and \(A_{430}\) are absorbance values of solution mixture at 400 nm and 430 nm respectively. \(\varepsilon\) value represents the molar absorptivity of Cu(II) and V(V) complexes at the wavelengths specified. 63.54 and 50.94 are factors to convert the concentrations into amounts for Cu(II) and V(V) respectively.

The simultaneous determination of Cu(II) and V(V) in synthetic mixture is carried out by adopting the following procedure.

Procedure

To a known aliquot of the synthetic mixture of Cu(II) and V(V) taken in a 10ml volumetric flask, 5ml of buffer (pH 5.0) solution, 3 ml of DMF and 0.2ml of 2,4-DHBINH (1 X 10^{-2} M) solution are added. The contents of the flask are made up to the mark with distilled water. The absorbance is measured at 400 nm and 430 nm against the reagent blank. The amount of Cu(II) and V(V) are computed from equations 5.4.3 and 5.4.4 respectively. The results are presented in Table 5.4.1.
### TABLE 5.4.1.  
Simultaneous determination of Cu(II) and V(V) in synthetic mixtures:

<table>
<thead>
<tr>
<th>Amount taken (µg/ml)</th>
<th>Amount found (µg/ml)</th>
<th>Relative Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu(II)</td>
<td>V(V)</td>
<td>Cu(II)</td>
</tr>
<tr>
<td>0.381</td>
<td>0.305</td>
<td>0.380</td>
</tr>
<tr>
<td>0.381</td>
<td>0.610</td>
<td>0.383</td>
</tr>
<tr>
<td>0.381</td>
<td>0.916</td>
<td>0.385</td>
</tr>
<tr>
<td>0.381</td>
<td>1.222</td>
<td>0.381</td>
</tr>
<tr>
<td>0.381</td>
<td>1.528</td>
<td>0.389</td>
</tr>
<tr>
<td>0.381</td>
<td>1.832</td>
<td>0.388</td>
</tr>
<tr>
<td>0.762</td>
<td>0.305</td>
<td>0.764</td>
</tr>
<tr>
<td>1.143</td>
<td>0.305</td>
<td>1.148</td>
</tr>
<tr>
<td>1.524</td>
<td>0.305</td>
<td>1.514</td>
</tr>
<tr>
<td>1.905</td>
<td>0.305</td>
<td>1.895</td>
</tr>
<tr>
<td>2.288</td>
<td>0.305</td>
<td>2.276</td>
</tr>
</tbody>
</table>

**Conclusions:**

Copper and vanadium are industrially important metals and are found in association with each other in alloys and steels. The present method is simple, direct and sensitive for the simultaneous determination of Cu(II) and V(V).
Section 5

SIMULTANEOUS SPECTROPHOTOMETRIC DETERMINATION OF IRON(III) AND COPPER(II)

Iron(III) reacts with 2,4-DHBINH giving an yellowish brown coloured complex in the pH range 2.5 to 5.5 with $\lambda_{\text{max}}$ at 390 nm (cf section 4.5). Copper(II) reacts with 2,4-DHBINH giving a greenish yellow coloured complex in the pH range 1.5 to 7.0 (cf section 4.7). Investigations are therefore carried out to explore the possibility of simultaneous determination of Fe(III) and Cu(II) at pH 5.0. The following procedure is adopted for the purpose.

Absorption spectra

To fix up the wavelengths for absorbance measurements, the spectra of the following solutions are recorded.

The absorption spectra of the solutions containing Fe(III)-2,4-DHBINH complex and Cu(II)-2,4-DHBINH complex are recorded by the following procedure in 3.b and presented in Fig.5.5.1a and 5.5.1b respectively. The absorption spectrum of 2,4-DHBINH solution is also recorded by adopting the procedure in 3.a and presented in Fig.5.5.1c.

From the spectra 5.5.1, it is noticed that both Fe(III) and Cu(II) systems show $\lambda_{\text{max}}$ at 390 nm. Though both Fe(III) and Cu(II) show $\lambda_{\text{max}}$ at 390 nm, at 400 nm Fe(III) has less absorbance and Cu(II) has more absorbance, while at 440 nm Cu(II) has less absorbance and Fe(III) has more absorbance. The difference in absorbance between Fe(III) and Cu(II) complexes is maximum at these wavelengths. Hence 400 nm and 440 nm are selected for the purpose of carrying out the simultaneous determination of Fe(III) and Cu(II).

Applicability of Beer's law

To establish whether Beer's law is applicable to the metal ions at 400 nm and 440 nm, the absorbance measurements with any metal ion are made at both these wavelengths by employing the procedure in 3.h. The results are presented Figs. 5.5.2 and 5.5.3.

From the linear plots it is observed that Fe(III) obeyed Beer's law in the range 0.24 to 2.40 $\mu$g/ml, while Cu(II) obeys in the range 0.40 to 3.17 $\mu$g/ml at both 400 nm and 440 nm. The molar absorptivities for Fe(III) are $1.68 \times 10^4$ I mol$^{-1}$ cm$^{-1}$ and $0.8 \times 10^4$ I mol$^{-1}$ cm$^{-1}$ at 400 nm and 440 nm respectively. The molar absorptivity for Cu(II) are $1.93 \times 10^4$ I mol$^{-1}$ cm$^{-1}$ and $0.26 \times 10^4$ I mol$^{-1}$ cm$^{-1}$ at 440 nm respectively. It is also noticed that the total absorbance at 400 nm and 440 nm is equal to the sum of the absorbance due to individual Fe(III) and Cu(II) species at these wavelengths. Hence the possibility of simultaneous determination of Fe(III) and Cu(II) is explored.
Fig. 5.5.1. Absorption spectra of
a. Fe(III) - 2,4-DHBINH system Vs reagent blank
b. Cu(II) - 2,4-DHBINH system Vs reagent blank
c. 2,4-DHBINH Vs buffer blank

\[
[\text{Fe(III)}] = [\text{Cu(II)}] = 2.5 \times 10^{-5} \text{ M} \\
[2,4-\text{DHBINH}] = 2.5 \times 10^{-4} \text{ M}; \text{ pH } = 5.0
\]
Fig. 5.5.2. Absorbance Vs amount of Fe(III)
$[2,4$-DHBINH$] = 3.00 \times 10^{-4}$ M; pH = 5.0

$A_{400} = 0.284C + 0.0008$
$A_{440} = 0.137C + 0.0001$

Fig. 5.5.3. Absorbance Vs amount of Cu(II)
$[2,4$-DHBINH$] = 3.00 \times 10^{-4}$ M; pH = 5.0

$A_{400} = 0.302C + 0.0011$
$A_{440} = 0.041C + 0.00001$
The absorbance of the solution mixture containing Fe(III) and Cu(II) are measured at 400 nm and 440 nm at pH 5.0. From these absorbance values the amounts of Fe(III) and Cu(II) are calculated using the following equations.

\[
A_{400} = \varepsilon_{\text{Cu}400} C_{\text{Cu}} + \varepsilon_{\text{Fe}400} C_{\text{Fe}} \quad \ldots \text{5.5.1}
\]

\[
A_{440} = \varepsilon_{\text{Cu}440} C_{\text{Cu}} + \varepsilon_{\text{Fe}440} C_{\text{Fe}} \quad \ldots \text{5.5.2}
\]

Converting the concentrations into amounts of (µg/ml) and solving the equations 5.5.1 and 5.5.2, we get

\[
\frac{A_{440} \varepsilon_{\text{Cu}400} - A_{400} \varepsilon_{\text{Cu}440}}{\varepsilon_{\text{Cu}400} \varepsilon_{\text{Fe}440} - \varepsilon_{\text{Fe}400} \varepsilon_{\text{Cu}440}} \times \frac{58.94 \times 10^3}{\varepsilon_{\text{Fe}}400 \varepsilon_{\text{Fe}}440} = \text{Fe} \quad \ldots \text{5.5.3}
\]

\[
\frac{A_{400} \varepsilon_{\text{Fe}440} - A_{440} \varepsilon_{\text{Fe}400}}{\varepsilon_{\text{Cu}400} \varepsilon_{\text{Fe}440} - \varepsilon_{\text{Fe}400} \varepsilon_{\text{Cu}440}} \times \frac{63.54 \times 10^3}{\varepsilon_{\text{Fe}}400 \varepsilon_{\text{Cu}440}} = \text{Cu} \quad \ldots \text{5.5.4}
\]

Where \(A_{400}\) and \(A_{440}\) are absorbance values of solution mixture at 400 nm and 440 nm respectively. \(\varepsilon\) value represents the molar absorptivity of Fe(III) and Cu(II) complexes at the wavelengths specified. 58.94 and 63.54 are the factors to convert the concentrations into amounts for Fe(III) and Cu(II) respectively.

The simultaneous determination of Fe(III) and Cu(II) in synthetic mixtures is carried out by employing the following procedure.

**Procedure**

A known aliquot of synthetic mixture of Fe(III) and Cu(II) is added to 5 ml of buffer, (pH 5.0) solution, 3 ml of DMF and 0.2 ml of 2,4-DHBINH (1 X 10^{-2} M) taken in 10 ml volumetric flask. The contents of the flask are made up to the mark with distilled water. The absorbance is measured at 400 nm and 440 nm against the reagent blank. The amount of Fe(III) and Cu(II) are computed from equations 5.5.3 and 5.5.4 respectively. The results are presented in Table 5.5.1.
TABLE 5.5.1.

Simultaneous determination of Fe(III) and Cu(II) in synthetic mixtures:

<table>
<thead>
<tr>
<th>Amount taken(µg/ml)</th>
<th>Amount found(µg/ml)</th>
<th>Relative Error(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe(III)</td>
<td>Cu(II)</td>
</tr>
<tr>
<td>0.381</td>
<td>0.353</td>
<td>0.383</td>
</tr>
<tr>
<td>0.381</td>
<td>0.706</td>
<td>0.389</td>
</tr>
<tr>
<td>0.381</td>
<td>1.060</td>
<td>0.382</td>
</tr>
<tr>
<td>0.381</td>
<td>1.413</td>
<td>0.381</td>
</tr>
<tr>
<td>0.381</td>
<td>1.767</td>
<td>0.379</td>
</tr>
<tr>
<td>0.635</td>
<td>1.589</td>
<td>0.630</td>
</tr>
<tr>
<td>0.762</td>
<td>0.353</td>
<td>0.768</td>
</tr>
<tr>
<td>1.143</td>
<td>0.353</td>
<td>1.143</td>
</tr>
<tr>
<td>1.524</td>
<td>0.353</td>
<td>1.514</td>
</tr>
<tr>
<td>1.905</td>
<td>0.353</td>
<td>1.895</td>
</tr>
<tr>
<td>2.286</td>
<td>0.353</td>
<td>2.266</td>
</tr>
</tbody>
</table>

Applications

The proposed method is applied for the simultaneous determination of Fe(III) and Cu(II) in grape leaf sample. The sample is analysed by the following procedure.

Procedure

A known aliquot of the grape leaf sample is taken in a 10 ml volumetric flask containing 5 ml of buffer solution pH 5.0. 3 ml of DMF and 0.2 ml of 2,4-DHBINH (1 X 10^-2 M), are added. The contents are made upto the mark with distilled water. The absorbance is measured at 400 nm and 440 nm against the reagent blank. The amounts of Fe(III) and Cu(II) are computed from the equations 5.5.3 and 5.5.4, respectively. The results are presented in Table 5.5.2.

TABLE 5.5.2.

Analysis of grape leaf sample

<table>
<thead>
<tr>
<th>Sample</th>
<th>Amount taken(µg/ml)</th>
<th>Amount found(µg/ml)</th>
<th>Relative Error(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe(III)</td>
<td>Cu(II)</td>
<td>Fe(III)</td>
</tr>
<tr>
<td>Leaf sample of grape a</td>
<td>33.70</td>
<td>55.40</td>
<td>33.40</td>
</tr>
</tbody>
</table>

*: Average of six determinations.  a: ppm Zn 6.08; Cu 55.40; Fe 33.7; Mn 20.0; Ca 1100; Mg 110; P 300; K 1000.
Conclusions:

Iron and copper are found associated with each other in many industrially important materials and minerals. These are widely distributed in foods of plant and animal origin. These two metal are also distributed in blood and body tissues in human system. Hence a their simultaneous spectrophotometric determination is important as it enhances the selectivity of the method. Simultaneous spectrophotometric determination of Fe(III) and Cu(II) in chalco pyrate mono minerals is carried out with EDTA by Wang and Dimin². Wang et al.³ determined Fe(III) and Cu(II) simultaneously in brass samples. Chen et al.⁴ determined Cu(II) and Fe(III) in aluminium alloys by dual wavelength spectrophotometry. Deshmuk⁵ proposed a simultaneous spectrophotometric for the determination of Fe(III) and Cu(II). Simultaneous spectrophotometric determination of Fe(III) and Cu(II) is carried out by Uday Kumar⁶.

The present method for the simultaneous spectrophotometric determination of Fe(III) and Cu(II) is however simple, rapid and sensitive for analysis of biological samples and copper, iron ores.
REFERENCES: