CHAPTER IV
CHAPTER - 4
SPECTROPHOTOMETRIC DETERMINATION OF IRON (III) USING 4-METHYL,-HYDROXY, 8-FORMYL COUMARIN.

Iron is one of the earliest known metal to mankind. Besides its industrial importance, it is now clearly established that element is essential in biological systems. Haemoglobin is an important oxygen carrier in virtebrata and is an iron complex. Iron is also an important constituent in the plant enzymes peroxidase, catalose, and cytochrome oxidase. Its presence is soils and plant tissue is well known.

Iron occurs in solution in oxidation states + 2 and + 3. Iron (iii) compounds are more stable than iron (ii). Several photometric methods' for the determination of iron employing numerous organic reagents are known. Since ferrous and ferric ions have chromophoric properties, many methods employed for the photometric determination of the metal ion involve the use of reagents without chromophoric group.

Block stated numerous methods have been employed (in attempts) to assess the availability status of iron in soils. However no one method has received wide usage of has been accepted as a standard. Much remains to be learned about the chemistry of iron in soils and iron nutrition of plants.
A systematic study of the reaction between iron (iii) and 4-methyl, 7-hydroxy, 8-formyl coumarin revealed that iron (iii) forms a complex with the reagent in solutions of acidic pH. The qualitative observation is studied systematically with a view to develop a method for the spectrophotometric determination of iron (III).

a) Absorption spectrum of 4-methyl-7-hydroxy 8-formyl Coumarin.

In a 10ml volumetric flask, 5ml of buffer solution (pH 4.0) and 1ml of 4-methyl-7-hydroxy, 8-formyl \(-2\) coumarin \((1 \times 10^{-2} \text{ M})\) solution and 2ml of dimethyl formamide (D.M.F.) are mixed and the contents of the flask are made up to the mark with distilled water. The absorbance of the resulting solution is measured in the wavelength region 350 to 450 nm against buffer solution as blank. The results are presented in Table 4.1.
### TABLE 4.1

**ABSORPTION SPECTRUM OF THE COUMARIN SOLUTION**

<table>
<thead>
<tr>
<th>Wavelength (nm)</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>350</td>
<td>0.878</td>
</tr>
<tr>
<td>360</td>
<td>0.603</td>
</tr>
<tr>
<td>370</td>
<td>0.200</td>
</tr>
<tr>
<td>380</td>
<td>0.097</td>
</tr>
<tr>
<td>390</td>
<td>0.030</td>
</tr>
<tr>
<td>400</td>
<td>0.020</td>
</tr>
<tr>
<td>410</td>
<td>0.009</td>
</tr>
<tr>
<td>420</td>
<td>0.008</td>
</tr>
<tr>
<td>430</td>
<td>0.006</td>
</tr>
<tr>
<td>440</td>
<td>0.006</td>
</tr>
</tbody>
</table>

(b) **Absorption spectrum of iron (III) - coumarin complex solution.**

1 ml of iron (III) (1 x 10^{-3} M) solution, 1 ml of 4-methyl, 7-hydroxy, 8-formyl coumarin (1 x 10^{-2} M) solution and 2 ml of dimethyl formamide (D.M.F.) are taken in a 10 ml volumetric flask containing 5 ml of buffer (pH 4.0) solution. The contents of the flask are made upto the mark with distilled water. The absorbance of the purple
coloured solution is measured against reagent blank in the wave length region 350 - 570 nm. The results are presented in table 4.2.

**TABLE 4.2**

Absorption spectrum of iron (III)-4-methyl-7-hydroxy-8-formyl coumarin complex solution.

[ Iron (III) ] = $1 \times 10^{-4}$ M

[ 4-methyl-7-hydroxy-8-formyl coumarin ] = $1 \times 10^{-3}$ M

Measured pH = 4.0

D.M.F. = 8% (by volume)

<table>
<thead>
<tr>
<th>WAVELENGTH (nm)</th>
<th>ABSORBANCE</th>
</tr>
</thead>
<tbody>
<tr>
<td>360</td>
<td>0.400</td>
</tr>
<tr>
<td>370</td>
<td>0.470</td>
</tr>
<tr>
<td>380</td>
<td>0.600</td>
</tr>
<tr>
<td>390</td>
<td>0.800</td>
</tr>
<tr>
<td>400</td>
<td>0.900</td>
</tr>
<tr>
<td>410</td>
<td>1.000</td>
</tr>
<tr>
<td>420</td>
<td>1.306</td>
</tr>
<tr>
<td>430</td>
<td>0.900</td>
</tr>
<tr>
<td>440</td>
<td>0.800</td>
</tr>
<tr>
<td>450</td>
<td>0.720</td>
</tr>
<tr>
<td>460</td>
<td>0.650</td>
</tr>
<tr>
<td>470</td>
<td>0.600</td>
</tr>
<tr>
<td>480</td>
<td>0.500</td>
</tr>
<tr>
<td>490</td>
<td>0.320</td>
</tr>
</tbody>
</table>
The absorbance values are plotted against wavelength and presented in fig. 4.1. The figure indicates that the purple coloured solution shows absorption maximum at 420 nm where the reagent has relatively low absorbance. Hence, all further studies are made at this wavelength using reagent blank.

C) Effect of pH :-

5ml of buffer solutions of different pH are taken in a series of 10 ml of volumetric flasks. To each flask, 1 ml of Iron (III) (1 x 10^-3 M) solution and 1 ml of 4-methyl-7-hydroxy, 8-formyl coumarin (1 x 10^-2 M) solution and 2ml of D.M.F. are added. The contents are diluted to the mark with distilled water and the absorbance is measured at 420 nm against reagent blank. The data is listed in Table 4.3.
TABLE 4.3

Effect of pH on the absorbance of Iron (III-coumarin complex solution.

-4
\[ \text{[Iron (III)]} = 1 \times 10^4 \text{ M} \]

complex or (4-Methyl-7-hydroxy-8-Formyl-coumarin)

-3
\[ = 1 \times 10^3 \text{ M} \]

D.M.F. = 8 % (by volume)

Wavelength = 420 nm.

<table>
<thead>
<tr>
<th>pH</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0</td>
<td>0.800</td>
</tr>
<tr>
<td>2.5</td>
<td>0.850</td>
</tr>
<tr>
<td>3.0</td>
<td>0.900</td>
</tr>
<tr>
<td>3.5</td>
<td>0.980</td>
</tr>
<tr>
<td>4.0</td>
<td>1.306</td>
</tr>
<tr>
<td>4.5</td>
<td>1.220</td>
</tr>
<tr>
<td>5.0</td>
<td>0.800</td>
</tr>
<tr>
<td>5.5</td>
<td>0.700</td>
</tr>
<tr>
<td>6.0</td>
<td>0.600</td>
</tr>
</tbody>
</table>

A plot is constructed (Fig. 4.2) between the absorbance and pH. It is evident from Fig. 4.2 that the reaction mixture is showing maximum and absorbance in the pH range 4.0 - 4.2. Hence, further studies are carried out in a buffer solution of pH 4.0.
Fig. 4.2. Effect of pH on the absorbance of the reaction mixture.
d) Effect of 4-methyl-7-hydroxy-8-formyl coumarin concentration:

The effect of 4-methyl, 7-hydroxy, 8-formyl coumarin concentration on the absorbance of the reaction mixture is studied by adopting the following procedure.

5 ml. buffer (pH 4.0) 2ml of D.M.F. solution, 1ml \(1 \times 10^{-3}\) Iron (III) solution are taken in each at a set of 10ml volumetric flasks. To these flasks, known aliquots of 4-Methyl-7-hydroxy-8-formyl coumarin solution added. The contents are made up to the mark with distilled water and the absorbance of the solution in each flask is measured at 420 nm. The corresponding reagent blank. The results are presented in Table 4.4.
TABLE 4.4

Effect of 4-methyl-7-hydroxy concentration on absorbance.

<table>
<thead>
<tr>
<th>[Iron (III)]</th>
<th>pH</th>
<th>D.M.F.</th>
<th>Wave length</th>
</tr>
</thead>
<tbody>
<tr>
<td>$1 \times 10^{-4}$</td>
<td>4.0</td>
<td>8% (by volume)</td>
<td>420 nm</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Iron (III):Coumarin</th>
<th>ABSORBANCE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 : 1</td>
<td>0.200</td>
</tr>
<tr>
<td>1 : 2</td>
<td>0.350</td>
</tr>
<tr>
<td>1 : 5</td>
<td>0.980</td>
</tr>
<tr>
<td>1 : 10</td>
<td>1.306</td>
</tr>
<tr>
<td>1 : 15</td>
<td>1.300</td>
</tr>
<tr>
<td>1 : 20</td>
<td>1.300</td>
</tr>
<tr>
<td>1 : 25</td>
<td>1.306</td>
</tr>
<tr>
<td>1 : 30</td>
<td>1.306</td>
</tr>
<tr>
<td>1 : 35</td>
<td>1.305</td>
</tr>
<tr>
<td>1 : 40</td>
<td>1.300</td>
</tr>
<tr>
<td>1 : 50</td>
<td>1.306</td>
</tr>
</tbody>
</table>

The data in table 4.4 indicates that a 10-fold molar excess of reagent is essential for complete complexation. Excess of reagent does not produce any adverse effect.
e) **Effect of D.M.F. on colour reaction:**

The colour intensity of the reaction mixture between the metal ion and reagent increases in the presence of D.M.F. In absence of D.M.F. an purple coloured insoluble precipitate is formed to avoid precipitate.

f) **Effect of time on colour reaction:**

When iron (III), solution is mixed with 4-methyl-7-hydroxy, 8-formyl coumarin solution in the buffer medium of pH 4.0, and 2 ml. of D.M.F. maximum colour is developed within 5 minutes. The absorbance of the complex remains constant for over 24 hours.

g) **Order of addition of reactants:**

The order in which various reactant constituents (Iron (III), buffer, D.M.F., and coumarin are mixed has no effect on absorbance of reaction mixture.

g) **Applicability of Beer's Law:**

To examine the applicability of Beer's law for the present system, the following procedure is adopted.

Known volumes of standard solutions of iron (III) are taken in a set of ten 25ml volumetric flasks. 10ml of buffer solution (pH 4.0) 3ml of 4-methyl-7-hydroxy, 8-1 formyl coumarin 1 x 10^-1 M solution, 5 ml. of D.M.F. are added to each flask and the contents are finally made up to the mark with distilled water. The absorbance of these
solutions is measured at 420 nm. against the reagent blank prepared under identical conditions. The results are presented in Table 4.5.

**TABLE 4.5**

**BEER'S LAW**

\[
\text{[ 4-methyl-7-hydroxy 8-formyl coumarin ]} = 1.2 \times 10^{-2} \text{ M}
\]

Measured pH = 4.0

D.M.F. = 8 % by volume

Wavelength = 420 nm

<table>
<thead>
<tr>
<th>Amount of iron (III) (ppm)</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.560</td>
<td>0.130</td>
</tr>
<tr>
<td>1.120</td>
<td>0.260</td>
</tr>
<tr>
<td>1.680</td>
<td>0.380</td>
</tr>
<tr>
<td>2.24</td>
<td>0.520</td>
</tr>
<tr>
<td>2.80</td>
<td>0.650</td>
</tr>
<tr>
<td>3.36</td>
<td>0.780</td>
</tr>
<tr>
<td>3.92</td>
<td>0.910</td>
</tr>
<tr>
<td>4.48</td>
<td>1.050</td>
</tr>
<tr>
<td>5.04</td>
<td>1.170</td>
</tr>
<tr>
<td>5.60</td>
<td>1.300</td>
</tr>
<tr>
<td>6.16</td>
<td>1.430</td>
</tr>
<tr>
<td>6.72</td>
<td>1.560</td>
</tr>
<tr>
<td>7.28</td>
<td>1.600</td>
</tr>
</tbody>
</table>
A calibration plot is constructed between the absorbance and amount of iron (III). A linear plot as shown in fig. 4.3 is obtained indicating the applicability of the method. The graph suggests that Beer's law is obeyed in the range 0.50 - 8.00.

The relative standard deviation of the method for the determination of 10 measurements is obtained as 2.7 ppt.

The present method is having a molar absorptivity of $1.3 \times 10^{-1}$ liter mole$^{-1}$ cm$^{-1}$. The sandell's sensitivity in the determination of 2.00 ppm. of iron (III) is found to be 0.00443 ug/cm$^2$.

2) Tolerance limit of foreign ions:

In order to study the applicability of the present method for the determination of iron (III) in natural samples and alloys, the effect of various diverse ions on the determination of iron is studied by measuring the absorbance of the complex in presence of different amounts of foreign ions. The amount of the foreign ion which causes a change in the absorbance value by +/- 0.04 units is taken as the tolerance limit. The results obtained are presented in table 4.6.
Fig. 4.2. Absorbance Vs Amount of Iron (III) (ppm).

Fig. 4.3 Beer's law
TABLE 4.6

Tolerance limit of foreign ion amount of iron (III) = 2.0 ppm taken.

<table>
<thead>
<tr>
<th>Foreign Ions</th>
<th>Tolerance limit (ppm)</th>
<th>Foreign Ion</th>
<th>Tolerance Ion (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EDTA</td>
<td>2000</td>
<td>Zn</td>
<td>800</td>
</tr>
<tr>
<td></td>
<td></td>
<td>V</td>
<td>600</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Pb</td>
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<tr>
<td></td>
<td></td>
<td>Mg</td>
<td>200</td>
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<td></td>
<td></td>
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<td>Pt</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Mn</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Co</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Cr</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ni</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Pd</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ru</td>
<td>10</td>
</tr>
<tr>
<td>Thiourea</td>
<td>80</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bromide</td>
<td>50</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Citrate</td>
<td>50</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Thiocyanide</td>
<td>50</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phosphate</td>
<td>50</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
It is observed that in the presence of 1000 ppm. of EDTA, the interference of Ni, Co, Mn, can be marked. Hence the present method determination of Iron (III) in presence of nickel, cobalt and manganese.

K) Composition and stability constant of the complex:

In the present study, the author observed that a single complex species is forming between iron (III)-4-methyl-7-hydroxy, 8-formyl coumarin in the concentration study. This is conformed by the Cooper's and Hooper's method based on the above observations, the composition of iron (III) and 4-methyl-7-hydroxy, 8-formyl coumarin complex is determined by using job's method, mole ratio method, and slope ratio method.

Job's continuous variation method:

Equimolar (3.0 x 10^{-4} M) solutions of iron (III) and 4-methyl-7-hydroxy-8-formyl coumarin are mixed in different volume proportions, keeping the total volume of the mixture constant (10 ml.) in series of 25ml volumetric flasks, 10ml of buffer pH (4.0) solutions, 2ml of D.M.F. are added to each flask and the resulting solutions are made up to the mark with distilled water. The absorbance of the solutions is measured at 420 nm. against the corresponding reagent blank. The data is tabulated in table 4.7.
A plot is drawn between absorbance and volume of iron (III) and presented in fig. 4.4. It is seen from the fig. 4.4 that the composition of the complex is 1:1 (iron (III) : 4-methyl-7-hydroxy-8-formyl coumarin).

**TABLE 4.7**

**Job’s continuous variation method:**

- $[\text{Iron (III)}] = 3.0 \times 10^{-4} \text{ M}$
- $[4$-methyl, 7-hydroxy, 8-formyl coumarin$] = 3.0 \times 10^{-4} \text{ M}$
- D.M.F. = 8% by volume
- Measured pH = 4.0
- Wave length = 420 nm.

<table>
<thead>
<tr>
<th>Iron (III) (ml)</th>
<th>Ml</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9</td>
<td>0.360</td>
</tr>
<tr>
<td>2</td>
<td>8</td>
<td>0.450</td>
</tr>
<tr>
<td>3</td>
<td>7</td>
<td>0.500</td>
</tr>
<tr>
<td>4</td>
<td>6</td>
<td>0.600</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
<td>0.600</td>
</tr>
<tr>
<td>6</td>
<td>4</td>
<td>0.580</td>
</tr>
<tr>
<td>7</td>
<td>3</td>
<td>0.550</td>
</tr>
<tr>
<td>8</td>
<td>2</td>
<td>0.450</td>
</tr>
<tr>
<td>9</td>
<td>1</td>
<td>0.360</td>
</tr>
</tbody>
</table>

**Molar ratio method:**

5ml of iron (III) solution ($1.5 \times 10^{-4} \text{ M}$), 10ml of buffer (pH 4.0) solution and 2ml of D.M.F. are taken in each
Fig. 4.4. Job's method

Mole fraction of metal ion

Absorbance

0.8 0.7 0.6 0.5 0.4
of different 25ml volumetric flasks known and different aliquots of 4-methyl-7-hydroxy, 8-formyl coumarin.

\[-4\]

\[1.5 \times 10^{-4}\] M solution are added to these flasks and the contents are made up to the mark to the distilled water. The absorbance of these solutions is measured at 420 nm against the corresponding reagent blank prepared under identical conditions. The results are presented in Table 4.8.

**TABLE 4.8**

MOLAR RATIO METHOD

D.M.F. = 8 % by volume

Wavelength = 420 nm.

<table>
<thead>
<tr>
<th>Volume of (ml)</th>
<th>4-methyl 7-hydroxy iron (III)</th>
<th>8-formyl coumarin</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5</td>
<td>5</td>
<td>0.150</td>
</tr>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td>0.200</td>
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<tr>
<td>3</td>
<td></td>
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<td>0.250</td>
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<td></td>
<td></td>
<td>0.300</td>
</tr>
<tr>
<td>5</td>
<td></td>
<td></td>
<td>0.350</td>
</tr>
<tr>
<td>6</td>
<td></td>
<td></td>
<td>0.3801</td>
</tr>
<tr>
<td>7</td>
<td></td>
<td></td>
<td>0.400</td>
</tr>
<tr>
<td>8</td>
<td></td>
<td></td>
<td>0.430</td>
</tr>
<tr>
<td>9</td>
<td></td>
<td></td>
<td>0.450</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td></td>
<td>0.470</td>
</tr>
</tbody>
</table>
Fig. 4.5 Molar ratio method

![Fig. 4.5. Molar Ratio Plot.](image-url)

Fig. 4.5. Molar Ratio Plot.
A graph is drawn between the absorbance and the volume of 4-methyl, 7-hydroxy, 8-formyl coumarin added and presented in fig.4.5.

It is seen from the curve (fig.4.5) that the inflection point occurs at a mole ratio of 1:1 (iron (III) : 4-methyl-7-hydroxy-8-formyl coumarin). Hence, the metal to reagent ratio in the complex is 1:1.

**Slope ratio method :**

To various aliquots of iron (III) (1 x 10^{-4} M) solution taken in a set of 25ml volumetric flask, 10ml of buffer (pH 4.0) solution, 2ml of D.M.F. and 5ml of 4-methyl, 7-hydroxy, 8-formyl coumarin (1 x 10^{-3} M) are added to each flask. To contents are made up to the mark with distilled water and the absorbance of the solution in each flask is measured at 420 nm against the reagent blank. The values are presented in table 4.9.
TABLE 4.9

([4-methyl 7-hydroxy
8-formyl Coumarin]) = 1 x 10^-3 M
[iron (III)] = 1 x 10^-4 M
Measured pH = 4.0
D.M.F. = 8 % by volume
Wavelength = 420 nm.

<table>
<thead>
<tr>
<th>Volume of (ml)</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>iron (III)</td>
<td>4-methyl 7-hydroxy 8-formyl coumarin</td>
</tr>
<tr>
<td>1</td>
<td>5</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
</tr>
<tr>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>6</td>
<td>5</td>
</tr>
<tr>
<td>7</td>
<td>5</td>
</tr>
<tr>
<td>8</td>
<td>5</td>
</tr>
<tr>
<td>9</td>
<td>5</td>
</tr>
</tbody>
</table>

A graph is drawn between absorbance and volume of iron (III) solution added. A linear plot is obtained as shown in fig. 4.6. The slope of the plot (m) is calculated.

In another set of 25ml volumetric flasks, 10ml of buffer solution (pH 4.0), 2ml of D.M.F., and 5ml of iron (III) (1 x 10^-4 M) solution are taken in each flask. Known aliquots of 4-
methyl, 7-hydroxy, 8-formyl coumarin (1 × 10⁻³ M) solution are added to these flasks and the contents are made up to the mark with distilled water. The absorbance of the solutions was measured against buffer blank. The experimental data is presented in Table 4.10.

TABLE 4.10

| [4-methyl, 7-hydroxy, 8-formyl Coumarin] | = 1 × 10⁻³ M |
| [iron (III)] | = 1 × 10⁻⁴ M |
| Measured pH | = 4.0 |
| D.M.F. | = 8 % by volume |
| Wavelength | = 420 nm |

<table>
<thead>
<tr>
<th>Volume of (ml)</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-methyl, 7-hydroxy, iron (III) 8-formyl coumarin</td>
<td>0.108</td>
</tr>
<tr>
<td>1 5</td>
<td>0.120</td>
</tr>
<tr>
<td>2 5</td>
<td>0.140</td>
</tr>
<tr>
<td>3 5</td>
<td>0.165</td>
</tr>
<tr>
<td>4 5</td>
<td>0.180</td>
</tr>
<tr>
<td>5 5</td>
<td>0.200</td>
</tr>
<tr>
<td>6 5</td>
<td>0.220</td>
</tr>
<tr>
<td>7 5</td>
<td>0.250</td>
</tr>
<tr>
<td>8 5</td>
<td>0.260</td>
</tr>
</tbody>
</table>

A plot of absorbance vs the volume of 4-methyl-7-hydroxy, 8-formyl coumarin is presented in fig. 4.7. The slope (n) of the straight line is calculated. From the
Fig. 4.6. Slope ratio method
ratio of the slopes \( m \) and \( n \) of the two plots, the composition of the complex is determined and found to be 1:1 (iron (III) : 4-methyl-7-hydrox-8-formyl coumarin).

The stability constant of iron (III) and 4-methyl-7-hydroxy-8-formyl coumarin complex is determined by using the following formula given by job.

\[
\beta = \frac{A}{A_P} = \frac{m}{m+n} \frac{m}{m+n-1} \frac{m}{n} \frac{1}{[(1-A/A)^m]} [C]
\]

Where

\( A \) = absorbance corresponding to the point of interaction of extrapolated lines.
\( A \) = Observed absorbance of concentration \( C \).
\( C \) = Concentration corresponding to the point of interaction.
\( \beta \) = Stability constant.

By substituting the values \( A, A \) and \( C \) in the above formula, the stability constants of the complex is calculated and found to be \( 5.3 \times 10^4 \).

**Application:**

The method is successfully applied for determination of iron (III) in alloys, steels and plant samples.
Analysis of Steels, alloys and plant samples:

By employing the present method iron present in cement sample is determined and the results are presented in the following paragraphs.

Preparation of the sample solution:

Required amount of cement is taken, dissolved in concentrated hydrochloric acid and digested for half an hour by heating on a hot plate. The solution is cooled and filtered and the filtrate is collected quantitatively in a 50ml standard flask and made up to the mark with distilled water.

Procedure:

Known aliquots of the sample solutions are taken in different 10ml standard flasks containing 5ml of buffer solution (pH 4.0), 2ml of D.M.F. and 0.5ml of the reagent (0.01 M). The contents are made up to the mark with distilled water. The absorbance of the solutions is measured at 420nm against reagent blank. The amount of iron present in the sample solutions is computed from the predetermined calibration plot drawn between the amount of iron v/s absorbence and the results are presented in Table 4.11.
### TABLE 4.11

Analysis of cement, steel, alloys and plant samples:

<table>
<thead>
<tr>
<th>Samples and (%) composition</th>
<th>Amount of iron (µg/ml)</th>
<th>Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Cement</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( Ca_{0} = 50.60, Si_{0} = 20.25 )</td>
<td>0.600</td>
<td>0.608</td>
</tr>
<tr>
<td>( Al_{0} = 5.10, Mg_{0} = 2.3 )</td>
<td>0.813</td>
<td>0.814</td>
</tr>
<tr>
<td>( Fe_{0} = 1.2, So_{0} = 1.2 )</td>
<td>0.927</td>
<td>0.927</td>
</tr>
<tr>
<td>( Na_{0} = 1.0, K_{0} = 1.0 )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( +* )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2. BAS = 179/2</td>
<td>0.525</td>
<td>0.525</td>
</tr>
<tr>
<td>( Fe = 1.02, Cu = 58.5 )</td>
<td>0.720</td>
<td>0.728</td>
</tr>
<tr>
<td>( Ni = 0.56, Mn = 0.86 )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( Sn = 0.7, Si = 0.044 )</td>
<td>0.800</td>
<td>0.800</td>
</tr>
<tr>
<td>( Pb = 0.35, Al = 2.22 )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zn = 35.8,</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3. Plant Sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(Guajava L)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( 0.780 )</td>
<td>0.770</td>
<td>-1.3</td>
</tr>
<tr>
<td>( 0.925 )</td>
<td>0.925</td>
<td>0.00</td>
</tr>
<tr>
<td>( 0.970 )</td>
<td>0.976</td>
<td>+0.6</td>
</tr>
</tbody>
</table>

* Average of six determinations

+ In presence of 1000 ppm of citrate
DISCUSSION:

From the analytical data obtained in the present method, it can be seen that the present method is sensitive, rapid, simple and selective. The purple colour formation between Iron (III) and 4-methyl, 7-hydroxy, 8-formyl coumarin is instantaneous and the colour is stable for more than 48 hours. The molar absorptivity of the method ($\varepsilon = 1.3 \times 10^{-1}$ lit. mole $^{-1}$ cm$^{-1}$), indicates that the method is more sensitive.

The method gives quite accurate results in the determination of Iron (III) in steels and plant samples.
REFERENCES
