CHAPTER 2

Solutions and Instruments
Section 2: Derivative spectrophotometry and its advantages

2.1. Introduction

Derivative spectrophotometry, which consists in the differentiation of a normal spectrum, offers a useful means for improving the resolution of mixtures, because it enhances the detectability of minor spectral features. Derivative spectrophotometry is an analytical technique of great utility for extracting both qualitative and quantitative information from spectra composed of unresolved bands by using the first or higher derivatives of absorbance with respect to wavelength. It tends to emphasize subtle spectral features by presenting them in a new and visually more accessible way, allowing the resolution of multi-component elements, and reducing the effect of spectral background interferences. This technique offers an alternative approach to the enhancement of sensitivity and specificity in mixture analysis. It consists of calculating and plotting one of the mathematical derivatives of a spectral curve. The derivative transformation does not increase the information content of a spectrum, but it permits discrimination against broad band interferences, arising from turbidity or non-specific matrix absorption. Various reviews are published dealing both with theoretical aspects [221-225] and practical [226-231] problems, since the principles of the derivative spectrophotometry are formulated.

The derivative spectra are always more complex than the zero-order spectrum. The first derivative is the rate of change of absorbance against wavelength. It starts and finished at zero, passes through zero at the same wavelength as $\lambda_{\text{max}}$ of the absorbance band with first a positive and then a negative band, with the maximum and minimum at the same wavelengths as the inflection points in the absorbance band. This bipolar function is characteristic of all the odd-order derivatives. The most characteristic feature of the second-order derivative is a negative band with the
minimum at the same wavelength as the maximum on the zero-order band. It also shows two additional positive satellite bands on either side of the main band. The fourth derivative shows a positive band. The presence of a strong negative or positive band, with the minimum or maximum at the same wavelength as \( \lambda_{\text{max}} \) of the absorbance band, is characteristic of the even-order derivatives. Note that the number of bands observed is equal to the derivative order plus one.

2.2. Theory

For spectrophotometry in the UV and visible regions, the differentiation of absorption spectra has many advantages. It is the key for the potential enhancement of resolution of overlapping bounds. It facilitates the detection of poorly resolved absorption peaks arising from admixtures (or) impurities in solution (or) for structural reasons and it enables the exact determination of \( \lambda_{\text{max}} \) of the particular analytical species and increases sensitivity of the spectrophotometric procedures. It is an excellent background elimination technique in addition to the aforesaid advantages. The resolution of signal to noise ratios improved [225, 232-239] by eliminating the influence of Rayleigh scattering constant, background absorbance, non-selective absorption of the matrix (or) accompanying components.

The enhancement of the resolution of overlapping spectral bonds is the main characteristic of derivative spectrophotometry. The amplitude ‘D’ of the \( n^{\text{th}} \) power of the inverse of the bandwidth \( W \), of the normal spectrum [240] for bands (Gaussian (or) Lorent Zian). So, the differentiations discriminate against broad bands emphasizing sharper features to an extent that increases parallel to derivative order.

\[
^n D \propto \frac{1}{W^n}
\]
Thus, for two bands (X and Y) of same intensity, but of different width, the derivative amplitude of the sharper band (X) is greater than that of the broader one (Y) by a factor that increases with increase in derivative order [241].

\[
\frac{n D(X)}{n D(Y)} \propto \left(\frac{W_Y}{W_X}\right)^n
\]

The use of derivative spectra can thus increases the detection sensitivity of minor spectral features.

The first, second etc., derivative absorption spectrum of an analyte is defined as the first, second etc., derivative of the absorbance as a function of wavelength. The first (or) the second derivative is easily accessible instrumentally. On the basis of computer-generated functions, the higher derivatives are calculated. For the determination of the component more accurately in presence of matrix component, the first derivative spectrum is useful. For the multi component analysis (or) identification of derivatives with high spectral similarity as well, the first derivative spectrum is useful. Since bandwidth of a gaussian peak decreases to 51, 41 or 34% of the original bandwidth for the 2nd, 4th or 6th order derivatives respectively the resolution of overlapping bands is considerably improved. A greater analytical use is possessed by even order derivatives having a control peak of alternating sign narrower but coincident with the original peak. The odd functions [222,242] may have lesser analytical use. Thus, unwanted effects such as various scattering, instrumental effects, differences by replacing cells (or) base line shifts caused by continued background, can be eliminated by the first, second etc., order derivatives, which all allow a more accurate quantitative evaluation of data.
2.3. Quantitative evaluation of derivative plots

Various amplitudes of the derivative curves are proportional to the analyte concentration in a similar way as in the absorbance of the primary absorption peak, assuming that the Beer-Lambert’s law is obeyed for the zero order spectrums. The Beer’s law for first and second derivative methods can be written as follows respectively:

\[
\left( \frac{dA}{d\lambda} = -0.434 \frac{1}{l} \frac{dl}{d\lambda} = \frac{d\varepsilon}{d\lambda} Cl \right)
\]

\[
\frac{d^2A}{d\lambda^2} = 2.303C^2l^2 \left( \frac{d\varepsilon}{d\lambda} \right)^2 - 0.434 \frac{1}{l} \frac{dl}{d\lambda} = \frac{d^2\varepsilon}{d\lambda^2} Cl
\]

For quantitative measurement, the amplitude which is least affected by variation in the matrix and gives the best calibration statistics [196] is selected, assuming \(dl/d\lambda = 0\), (P) Peak-Peak, (t) peak-tangent or z-peak-base line methods which are shown in fig 1.2.1 are the procedures used for quantitative evaluation. In principle both the peak to valley amplitude, \(p_1, p_2\) and the base line to valley distance, \(z\) are proportional to the analyte concentration. For higher derivatives, the situation is more complicated. But the proportionality of the derived signal to the analyte concentration is often maintained [222, 243].

The derivative techniques have been used in pharmaceutical [244] environmental analysis [245] and in the finger print analysis of proteins [246], but only few data have been reported on the determination of metal ions.

In the present thesis, an attempt has been made by the author to develop new derivative spectrophotometric methods of the determination of Au(III), Fe(II), Ag(I), V(IV) by using HMBATSC. The results of the present investigations are reported in this thesis.
Fig. 1.2.1: Graphical measures for amplitudes in derivative spectrophotometry

(P) peak-peak;

(t) peak-tangent;

(z) peak-baseline method
Section 3: Importance of present investigations

Spectrophotometry is one of the most useful methods of quantitative analysis in various fields such as chemistry, physics, biochemistry, material and chemical engineering and clinical applications. Analytical chemistry seeks ever improved means of measuring the chemical composition of natural and artificial materials. The techniques of this science involve separation, identification and determination of the relative amounts of the components in simple or complex system(s). Analytical chemistry plays an important role in nearly all aspects of chemistry, like agriculture, chemical, environmental, forensic, manufacturing, metallurgical and pharmaceutical chemistry. Analytical chemists work to improve the reliability of existing techniques to meet the demands for better chemical measurements which arise constantly in our society.

An added impetus to the analytical chemist to discover simple, rapid and accurate methods was given by the fast growing progress in the area of analytical chemistry for the precise determination of metal ions at microgram level. The demand for newer methods of analysis is increasing in view of the problems constantly faced by the analytical chemists inspite of the availability of new methods and modern techniques for the determination of metal ions.

Atomic absorption, atomic fluorescence emission and X-ray fluorescence techniques were employed for the purpose in the past decade. The disadvantages with these techniques are that they are not within the reach of many laboratories. Also some of the interferences to which these methods are subject to are poorly understood and continue to cause problems. They are not amenable to easy operation besides the cost involved.
The advances made in the field of analytical chemistry have made possible the introduction of sophisticated methods for chemical analysis. Various chemical methods of analysis such as titrimetry, gravimetry, conductometry, potentiometry, turbidimetry, spectrophotometry, chromatography, polarography, neutron activation analysis, atomic absorption spectroscopy, ICP-atomic emission spectrometry etc., have played their respective roles in the determination of elements at trace levels. Spectrophotometric methods occupy special position due to their ease in application, simplicity and cost factor of the instrument, and at the same time high sensitivity and selectivity with proper manipulation of the experimental conditions. The colour reactions of metal ions with suitable analytical reagents form the basis of spectrophotometric determination.

Spectrophotometric analysis of metal ions at microgram level involves synthesis of selective and sensitive reagents. Thiosemicarbazones constitute an important class of analytical reagents. The complexes formed by them usually are highly stable. Though a large number of spectrophotometric methods are available, these suffer either from the lack of specificity or sensitivity. Further, it is also difficult to develop a highly selective spectrophotometric procedure for a given metal ion making use of a given organic reagent in view of the matrix differences. The simultaneous presence of other elements is infact a potential cause of interference whenever quantitative determinations are carried out. Therefore, in order to achieve greater degree of selectivity, the emphasis is now being devoted to develop spectrophotometric procedures for the simultaneous determination of two metal ions when present in a mixture. The spectrophotometric procedures for the simultaneous determination of metal ions in a mixture are sporadically reported in literature.
An excellent background elimination technique which enables the exact determination of $\lambda_{\text{max}}$ of the particular analyte and facilitates the detection of poorly resolved absorption peaks and increases the sensitivity and the selectivity of the spectrophotometric procedures is the derivative spectrophotometry. Without any pre-separation or pre-concentration this technique further enables the simultaneous determination of trace elements of similar chemical properties present in mixtures. The sensitivity and selectivity of the method is enhanced by the use of micellar media and derivative spectrophotometry inspite of the convenient solution to a number of analytical procedures such as avoiding extraction procedures, resolution of multi component system and matrix background offered by the technique.

Thiosemicarbazones have recently attracted considerable attention due to their ability to form tridentate chelates with transition metal ions through either two nitrogen and sulfur atoms, N–N–S or oxygen, nitrogen and sulfur atoms, O–N–S. Considerable interest in thiosemicarbazones and their transition metal complexes has also grown in the areas of biology and chemistry due to biological activities such as antitumoral, fungicidal, bactericidal, antiviral and nonlinear optical properties. They have been used for metal analyses, for device applications related to telecommunications, optical computing, storage and information processing.

The versatile applications of metal complexes of thiosemicarbazones in various fields prompted us to synthesize the tridentate NNS-donor thiosemicarbazones and their metal complexes. As a part of our studies on transition metal complexes with these ligands, we undertook the current work with the following objectives.
**Section 4: Objectives of present investigations**

Thiosemicarbazones have emerged as an important class of sulfur ligands particularly for transition metal ions in the last two decades. The real impetus towards developing the coordination chemistry of these thiosemicarbazones has been provided by remarkable biological activities of these compounds which have since been shown to be related to their metal complexing ability. The recent literature shows that this general class of compounds exhibits a wide range of stereochemistries on complexation with metal ions.

Analytical methods play a vital role in checking the composition of the raw materials and finished products in controlling various processes in industry, in the metallurgical products, in identifying new sources for rare elements and in the analysis of environmental pollutants. The analytical chemistry of certain metal ions like gold, silver, iron and vanadium are important in chemical, biological, pharmaceutical, clinical and industrial process. The determination of trace amounts of iron and vanadium which are present as micronutrients in plants and biological samples is very important. The metals gold and silver are used as catalysts. The metal gold in recent years is in considerable demand as a coating on electric welding rods for stabilizing the arc.

The survey presented in section (1) of this chapter shows that thiosemicarbazones are widely used as analytical spectrophotometric reagents. A careful inspection of the observations reported in the literature reveals that the sensitivity of the spectrophotometric determination with aromatic thiosemicarbazones is generally enhanced when a phenolic group is present in the benzene ring, ortho to the chromophoric group. Further thiosemicarbazones derived form aldehydes are not fully exploited for the purpose and analytical potentialities of 2-hydroxy-3-methoxy...
benzaldehyde thiosemicarbazone (HMBATSC) have not been explored. In order to achieve greater selectivity, the need for procedures for the simultaneous determination of two metal ions in a mixture is also established in section (2).

In view of these facts the author has

(a) Undertaken the synthesis of 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone to study its potentiality as a new spectrophotometric reagent.

(b) Developed sensitive methods for gold (III), silver (I), iron (III) and vanadium (IV) and the developed methods are used for the determination of metal ions in various real samples of industrial importance.

(c) Developed new derivative spectrophotometric methods for the determination of above mentioned metal ions.

(d) Developed simultaneous derivative spectrophotometric methods for the determination of two metal ions in a mixture without the need for prior separation or masking.
References


Section 1: Synthesis and Characterization of 2-Hydroxy-3-Methoxy Benzaldehyde Thiosemicarbazone

(a) Preparation of 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone

The reagent 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone (HMBATSC) was prepared by the Sah and Daniels [1] procedure. 11.25 g of 2-hydroxy-3-methoxy benzaldehyde (I) and 4.55 g of thiosemicarbazide (II) are dissolved in sufficient volume of methanol and the mixture is refluxed for 60 minutes. The contents were allowed to cool and the product separated by filtration. A crude sample (yield 80%) was obtained (C_9H_11O_2NS_3). The resultant product was recrystallized twice from hot methanol. Pure light yellowish green crystals of 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone (III) (m.p. 220-222°C) were obtained.

The compound was characterized by IR and NMR spectral studies.

IR Spectral Studies [2,3]

The infrared (IR) spectrum of the compound is recorded in KBr pellet and presented in Fig. 2.1.1. The peaks observed at 3458 cm\(^{-1}\) and 3342 cm\(^{-1}\) may be due to symmetric and asymmetric –N-H stretching frequency of primary amino group. The peak noticed at 3164 cm\(^{-1}\) may be assigned to –OH stretching frequency of phenolic group due to intramolecular hydrogen bonding. The peak observed at 3028 cm\(^{-1}\) is assigned to Ar-H stretching frequency of aromatic proton. The peak observed at 1595
cm⁻¹ is assigned to –C=N stretching frequency of azomethine. The peaks observed in the range 1530-1361 cm⁻¹ are characteristic of aromatic ring stretching frequency. A strong peak observed at 1056 cm⁻¹ is assigned to C=S stretching frequency.

**¹H-NMR Spectral Studies [2,3]**

The ¹H-NMR spectrum of the compound in DMF (Dimethyl formamide) solvent is shown in Fig. 2.1.2. The peak observed at δ value 8.2 (1H) is characteristic of phenolic –OH group. The peak found at δ value 7.88 (3H) is due to aromatic protons. The peak observed at δ value 4.0 (3H) is due to methyl group attached to hetero atom (oxygen atom). The peak shows at δ value 6.8 (2H) is due to –NH₂ protons attached to thionyl group (C = S). The peak observed at δ value 9.0 is due to aldehydic proton. The peak at δ values 11.4 is due to –NH proton (Azomethine).

The IR and ¹H-NMR spectral studies indicate that the position of the spectral peaks clearly suggest the formation of the compound, 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone (HMBATSC) and its structure is shown below.
Fig. 2.1.1: I.R. Spectrum of 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone (HMBATSC)
Fig. 2.1.2: $^1$H-NMR Spectrum of 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone (HMBATSC)
Section 2: Preparation of the reagent solution and other solutions employed in the present investigations

All the chemicals used were of analytical grade unless otherwise specified.

a. Stock solution of HMBATSC (reagent)

HMBATSC solution \((1\times10^{-2} \text{ M})\) was prepared in dimethyl formamide (DMF). 0.225 g of the reagent (HMBATSC) was weighed and transferred into a 100 ml volumetric flask and made up to the mark with DMF. The stock solution was suitably diluted to get the required concentration wherever necessary. Fresh reagent solution was prepared every day before use.

b. Gold(III) solution

A stock solution of 0.01 M gold(III) was prepared by dissolving precise amount of \(\text{HAuCl}_4\cdot3\text{H}_2\text{O}\) in 1 M Hydrochloric acid and standardized using standard procedure.

c. Iron (III) solution

The stock solution of Fe(III) was prepared by dissolving calculated amount of ferric alum, \(\text{NH}_4\ \text{Fe(SO}_4)_2\cdot12\ \text{H}_2\text{O}\) in distilled water containing small amount of concentrated \(\text{H}_2\text{SO}_4\). The solution was diluted to 100 ml with distilled water. A working standard solution was prepared by an appropriate dilution of the standard solution as required.

d. Silver (I) solution

A silver stock solution (0.001 M) was prepared by dissolving required amount of \(\text{AgNO}_3\) in double distilled water and diluting to the mark in a 100 ml volumetric flask. A suitable volume of this solution was diluted to obtain working solutions.
e. Vanadium (IV) solution

A stock solution of 0.01 M vanadium (IV) was prepared by dissolving required amount of VOSO$_4$·5H$_2$O in 100 ml standard flask with 0.01M Hydrochloric acid.

f. Preparation of other inorganic salt solutions

The inorganic salt solutions are prepared by dissolving the suitable salt in requisite quantity in doubly distilled water. A few drops of suitable acid are added to avoid hydrolysis before dilution wherever necessary. Solutions thus prepared are standardized if needed by standard procedures. The solvents employed are distilled and purified by standard procedures. The formulae, quality, molecular weight or specific gravity and the quantity of chemicals used are presented in the Table 2.2.1.

### Table 2.2.1
Salt solutions used with qualitative and quantitative studies

<table>
<thead>
<tr>
<th>Ion</th>
<th>Formula</th>
<th>Quality</th>
<th>Sp. Gr./Mol. wt.</th>
<th>Weight/Volume</th>
<th>Molarity of the stock solution</th>
</tr>
</thead>
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<td>Na$^+$</td>
<td>NaNO$_3$</td>
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<td>0.8499</td>
<td>0.01</td>
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<tr>
<td>Ag$^+$</td>
<td>AgNO$_3$</td>
<td>AR BDH</td>
<td>169.87</td>
<td>0.1699</td>
<td>0.01</td>
</tr>
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<td>Mn$^{2+}$</td>
<td>MgSO$_4$.7H$_2$O</td>
<td>AR BDH</td>
<td>246.50</td>
<td>0.2465</td>
<td>0.01</td>
</tr>
<tr>
<td>Mn$^{2+}$</td>
<td>MnCl$_2$.4H$_2$O</td>
<td>Riedel</td>
<td>197.91</td>
<td>0.1979</td>
<td>0.01</td>
</tr>
<tr>
<td>Fe$^{2+}$</td>
<td>(NH$_4$)$_2$SO$_4$.FeSO$_4$.6H$_2$O</td>
<td>AR BDH</td>
<td>392.14</td>
<td>0.3921</td>
<td>0.01</td>
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<td>Co$^{2+}$</td>
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<td>AR BDH</td>
<td>291.04</td>
<td>0.2910</td>
<td>0.01</td>
</tr>
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<td>Ni$^{2+}$</td>
<td>NiSO$_4$.7H$_2$O</td>
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<td>280.80</td>
<td>0.2808</td>
<td>0.01</td>
</tr>
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<td>Cu$^{2+}$</td>
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<td>0.01</td>
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<td>Zn$^{2+}$</td>
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<td>0.2875</td>
<td>0.01</td>
</tr>
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<td>Pb$^{2+}$</td>
<td>Pb(NO$_3$)$_2$</td>
<td>AR BDH</td>
<td>331.20</td>
<td>0.3312</td>
<td>0.01</td>
</tr>
<tr>
<td>Al$^{3+}$</td>
<td>Al$_2$(SO$_4$)$_3$.3(NH$_4$)$_2$SO$_4$.24$H_2$O</td>
<td>AR BDH</td>
<td>453.33</td>
<td>0.4533</td>
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<td>Cr$^{3+}$</td>
<td>CrCl$_3$.3H$_2$O</td>
<td>E.Merck</td>
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<td>0.2664</td>
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</tr>
<tr>
<td>Fe$^{3+}$</td>
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<td>0.4822</td>
<td>0.01</td>
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<td>Ru$^{3+}$</td>
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<td>0.2615</td>
<td>0.01</td>
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<td>Ion</td>
<td>Formula</td>
<td>Quality</td>
<td>Sp. Gr./ Mol. wt.</td>
<td>Weight/ Volume</td>
<td>Molarity of the stock solution</td>
</tr>
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<td>---------</td>
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<td>Ti$^{4+}$</td>
<td>K$_2$TiO$_2$(C$_2$O$_4$)$_2$.2H$_2$O</td>
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<td>0.3542</td>
<td>0.01</td>
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<td>Na$_2$MoO$_4$.2H$_2$O</td>
<td>E.Merck</td>
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<td>0.2419</td>
<td>0.01</td>
</tr>
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<td>Ce$^{4+}$</td>
<td>(NH$_4$)$_2$[Ce(NO$_3$)$_6$]</td>
<td>AR BDH</td>
<td>548.23</td>
<td>0.5482</td>
<td>0.01</td>
</tr>
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<td>Th$^{4+}$</td>
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<td>AR BDH</td>
<td>588.05</td>
<td>0.5881</td>
<td>0.01</td>
</tr>
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<td>V$^{5+}$</td>
<td>NH$_4$VO$_3$</td>
<td>AR Riedel</td>
<td>116.98</td>
<td>0.1170</td>
<td>0.01</td>
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<td>Cr$^{6+}$</td>
<td>K$_2$CrO$_4$</td>
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<td>194.19</td>
<td>0.194</td>
<td>0.01</td>
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<td>Moly.Chem</td>
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<td>42.00</td>
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<td>KCl</td>
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<td>0.7465</td>
<td>0.1</td>
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<td>Br$^-$</td>
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<td>1.0291</td>
<td>0.1</td>
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<td>KI</td>
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<td>AR BDH</td>
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<td>1.0600</td>
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<td>AR BDH</td>
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<td>1.7425</td>
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<td>PO$_4^{3-}$</td>
<td>Na$_2$HPO$_4$.2H$_2$O</td>
<td>Renel, Hungary</td>
<td>177.99</td>
<td>1.7799</td>
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<td>Citrate</td>
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<td>2.4322</td>
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<td>Tartrate</td>
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<td>Oxalate</td>
<td>K$_2$C$_2$O$_4$.2H$_2$O</td>
<td>AR BDH</td>
<td>184.24</td>
<td>1.8424</td>
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<tr>
<td>EDTA</td>
<td>Na$_2$ClO$_4$.H$_2$O$_5$.2H$_2$O</td>
<td>AR BDH</td>
<td>372.24</td>
<td>3.7224</td>
<td>0.1</td>
</tr>
<tr>
<td>Ascorbic acid</td>
<td>C$_6$H$_8$O$_6$</td>
<td>GRSM</td>
<td>176.13</td>
<td>1.7613</td>
<td>0.1</td>
</tr>
<tr>
<td>DMF</td>
<td>HCON(CH$_3$)$_2$</td>
<td>ER E.Merck</td>
<td>73.09</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Acetic Acid</td>
<td>CH$_3$COOH</td>
<td>AR BDH</td>
<td>60.05</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>In$^{3+}$</td>
<td>In$_2$(SO$_4$)$_3$.H$_2$O</td>
<td>Himedia</td>
<td>517.8</td>
<td>0.5178</td>
<td>0.01</td>
</tr>
<tr>
<td>Pd$^{2+}$</td>
<td>PdCl$_2$</td>
<td>AR Loba</td>
<td>117.4</td>
<td>0.1774</td>
<td>0.01</td>
</tr>
</tbody>
</table>
g. Buffer solutions

Buffer solutions are prepared by adopting the standard procedures reported in the literature [4]. The solutions employed for the preparation are given below.

<table>
<thead>
<tr>
<th>pH</th>
<th>Constituents</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5 – 3.0</td>
<td>1M sodium acetate + 1 M hydrochloric acid</td>
</tr>
<tr>
<td>3.2 – 6.0</td>
<td>2 M sodium acetate + 0.2 M acetic acid</td>
</tr>
<tr>
<td>7.0 – 8.0</td>
<td>1.0 M sodium acetate + 0.2 M acetic acid</td>
</tr>
<tr>
<td>8.0 – 12.0</td>
<td>2.0 M ammonia + 2.0 M ammonium chloride</td>
</tr>
</tbody>
</table>
Section 3: Instruments employed in the present investigations

a. UV visible recording spectrophotometer (UV-160 A)

Shimadzu Corporation Spectrophotometric Instrument Plant, Analytical Instruments Division, Kyote, Japan developed a versatile and indigenous microprocessor based UV visible recording spectrophotometer (UV 160 A).

Operational principle and constructional features

UV-160 A is a double-beam microprocessor based spectrophotometer designed for the quantitative analysis. Its main features are

1. Wavelength scanning system by CPU control without using sine bar to realize high speed wavelength scanning.
2. All in one type of spectrophotometer with CRT and printer incorporated.
3. Back up mode parameters are provided so as to enable single action operation.
4. Easy data processing, since the obtained spectrum is available by the conversation with CRO.

Specifications of UV 160 A spectrophotometer

<table>
<thead>
<tr>
<th>Specification</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measuring wavelength range</td>
<td>200 – 1100 nm</td>
</tr>
<tr>
<td>Spectral bandwidth</td>
<td>2 nm</td>
</tr>
<tr>
<td>Wavelength readability</td>
<td>0.1 nm increment</td>
</tr>
<tr>
<td>Wavelength scanning speed</td>
<td>monochromator setting speed is</td>
</tr>
<tr>
<td></td>
<td>nearly 3600 nm/min</td>
</tr>
<tr>
<td></td>
<td>fast – nearly 2400 nm/min</td>
</tr>
<tr>
<td></td>
<td>medium – nearly 1500 nm/min</td>
</tr>
<tr>
<td></td>
<td>slow – nearly 480 nm/min</td>
</tr>
<tr>
<td>Wavelength accuracy</td>
<td>± 0.5 nm with automatic wavelength correction</td>
</tr>
<tr>
<td>Light source switching</td>
<td>Automatic switching according to wavelength can be selected between 295 nm and 364 nm.</td>
</tr>
</tbody>
</table>
Photometric system : Double beam system.
Recording mode : Printout of measured data and calculated results.
Multi-component : Mixed samples up to eight components can be determined. Mixed samples can be used as standards. Standard sample data can be stored in the back up memory (up to 16 standards).
Light sources : 50 W long life halogen lamp (2000 hrs) and socket type deuterium lamp (500 hrs) with automatic control of maximum sensitivity.
Monochromator : Aberration corrected concave holographic grating with f = 4.2.
Detector : A matched pair of silicon photodiode.
Recorder : Computer controlled thermal graphic printer.
CRT dots : 9-inch with graphic function 240 x 320 dots.
Sample compartment : Inner size: 1100 mm wide; 230mm deep and 105 mm high, distance between sample and reference beam: 100 mm.
Power requirements : With line voltage selector for 100, 115, 220 and 240 V.
Weight : 42 Kgs.

b. ELICO digital pH meter

ELICO digital pH meter of model LI 613 (manufactured by M/s ELICO Private Limited, Hyderabad, India) is used for measuring the pH of buffer solutions. The instrument has a temperature compensate arrangement. The reproducibility of measurements is within ± 0.01 pH.
References


