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Extractive Spectrophotometric Method for Determination of Iron (II) using Ligand 2-(((Z)- 4-Hydroxy-3-Methoxy-5-((E)-Thiazol-5-Yldiazenyl) Benzylidene) Amino) Benzoic Acid As an Analytical Reagent

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Abstract: *Simple, economical, & feasible spectrophotometric method has been developed for the determination of Fe (II) by using schiff base ligand 2-(((Z)- 4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl) benzylidene) amino)benzoic acid (MThBABA). The schiff base has been synthesized conventionally as well as microwave method and was characterized by elemental analysis. MThBABA extracts Fe (II) quantitatively (99.65%) into n-butanol from an aqueous solution of pH range 5.7 – 6.9. An intense peak at 480 nm (λ max) was observed in the extract of n-butanol. Beer's law is obeyed over the concentration range 1 to 12 µg/ml for Fe (II). The molar absorptivity and Sandell's sensitivity for Fe - MThBABA system is 13449 L mole^{-1}cm^{-1} and 0.0705 µg cm^{-2} respectively. Job's Continuous Variation and Mole Ratio Method confirms that the extracted (Fe: MThBABA) complex has composition 1:1. The proposed method is rapid, sensitive, reproducible and accurate and it has been satisfactory applied for determination of iron in Ore and pharmaceutical samples.*

Keywords: Solvent Extraction, Extractive Spectrophotometry, Iron (II), Ores, pharmaceutical sample

I. INTRODUCTION

Iron is a transition element with atomic number 26. Iron belongs to group VIIIth and period 4th of the periodic table. Electronic configuration of iron is $1s^2 2s^2 2p^6 3s^2 3p^6 3d^6 4s^2$. Iron oxidizes readily in air and water to form Fe_2O_3 and is rarely found as a free element. It is necessary trace element, key component of haemoglobin found in red blood cells and carries oxygen to every cell in the body. Liver, bone marrow, spleen, and muscles stores extra iron. Iron is one of the most essential micronutrients for human beings however it is toxic when its concentration is > 0.3ppm in drinking water (U.S Public Health Service Drinking Water Standards). Lack of iron causes anemia in human being while excess of iron in the body causes Haemochromatosis (liver and kidney damage).According to the World Health Organization (WHO), iron deficiency is the number one nutritional disorder in the world. Hence, it is necessary to seek highly sensitive, accurate and selective analytical methods for quantitative determination of iron at trace levels.

Solvent extraction the highly efficient tool is used in separation technique. Due to its simplicity rapidity it is used in the separation of metal ions at trace level.[1,2] Solvent extraction coupled with spectrophotometrically plays a significant role in pharmaceutical science.[3] Schiff bases play crucial role as chelating agents in complexes of transition metal.[4] Various reagents [5-13] are available for the spectrophotometric determination of Iron (II).

The present communication describes the role of 2-(((Z)- 4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl) benzylidene) amino)benzoic acid (MThBABA) acts as a analytical reagent for the extractive spectrophotometric methods for the determination of iron.

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II. EXPERIMENTAL SECTION

ELICO - SL 159 spectrophotometer with optically matched quartz or glass cells of 1cm path length were used for absorbance measurement. An ELICO – LI 127 pH meter was employed for pH measurements.

2.1 General Procedure For Synthesis Of N - (O-Hydroxy Benzylidene) Pyridine -2-Amine (NOHBPA)

A. Conventional method of synthesis of ligand [2-((Z)-(4- hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl) benzenylidene) aminao) benzoic acid

The cold diazotized 2-Aminothiazol solution (0.005 moles) was poured into this ice cold vanillin (0.005 moles in 10% NaNO3) solution, very slowly with stirring. The red colour developed immediately, it was stirred till red crystals separates out. filtered it in a buchner. The product was washed with saturated solution of sodium chloride.

In a round bottom flask 0.005 moles of azo compound was taken with 0.005 moles of 2-aminobenzoic acid, 50ml of pure alcohol was added, with few porcelain pieces. It was attached to water condenser and refluxed for 3 hours. The mixture was poured into a beaker and left overnight in the fridge. Then mixture was filtered and dried. The product obtained was recrystallised as recommended by Vogel.[14] The product was greenish-brown crystals of schiff base, 2-(((Z)-4-hydroxy-3- methoxy-5-((E)-thiazol-5-yldiazenyl)benzylidene)amino) benzoic acid. The reaction is shown in Fig. 1. (Preparation of ligand)

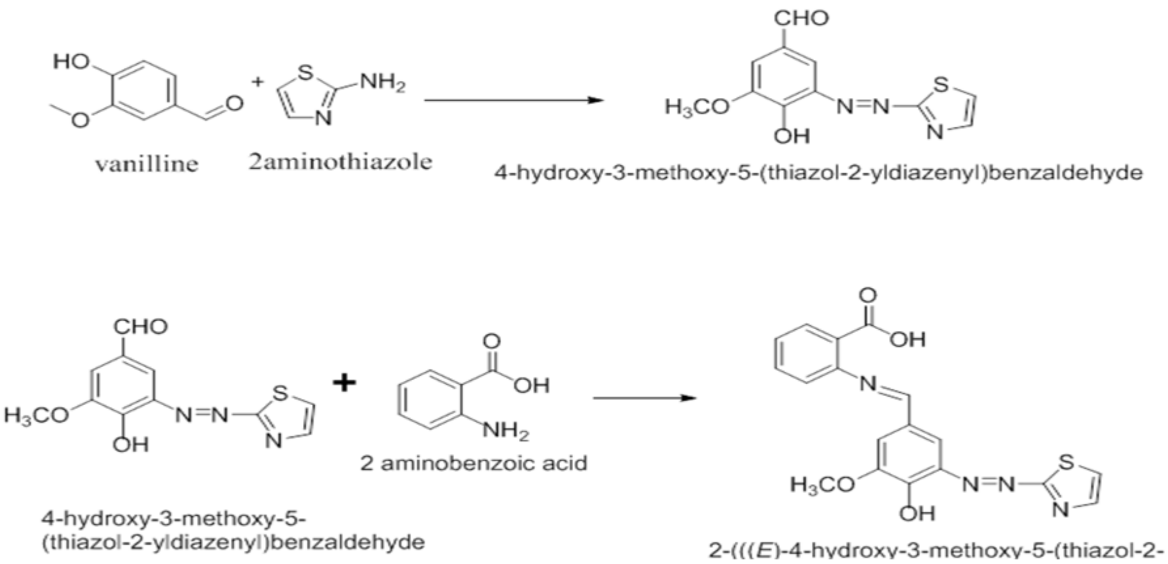

Figure 1: Synthesis of Ligand MThBABA

B. Green Method of Preparation of Schiff Base and Complex

In a beaker ingredients (0.005 moles of azo compound & 0.005 moles of 2-aminobenzoic) were mixed and few drops of pure alcohol was added. It was then irradiated in the microwave oven at 180^0 for 2 minutes. The reaction was completed in a short time (2 min) with higher yields.

Table 1: The Analytical and Physical data of ligand

Compounds (Colour)	Molecular Weight	Reaction period & %yield Conventional methods	Reaction period & %yield microwave methods	**Melting point**	**% Elemental Analysis Found (Calculated)**				
					C	**H**	**O**	**S**	**N**
Ligand (greenis brown)	382.39	3 -4 hours 70%	2-3Minutes 92%	**244^0C**	**56.53 (56.54)**	**3.70 (3.69)**	**16.78 (16.79)**	**8.40 (8.39)**	**14.67 (14.65)**

C. Preparation of Stock Solution

A stock solution of Fe (II) was prepared by dissolving accurately weighed ammonium ferrous sulphate in water containing sulphuric acid and it was standardized by gravimetrically.[15-16] Working solutions of Fe (II) were made by diluting the stock solution to an appropriate volume. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

D. Extractive Spectrophotometric Determination of Fe (Ii)

Aqueous solution containing 1 to 120 µg of Fe (II), 2ml of 5% hydroxylamine hydrochloride, 2ml of buffer solution (sodium acetate and acetic acid) of pH 6.0 and 2ml of 2% solution of MThBABA prepared in DMF were added. The volume of solution was made up to 10 ml with distilled water. The solution was then equilibrated for one minute with 10 ml of n butanol and the phases were allowed to separate. The n-butanol extract was collected in a 10 ml measuring flask and made up to mark with n butanol, if necessary. The absorbance of n butanol extract was measured at 480 nm against a reagent blank prepared under identical conditions. The measured absorbance was used to compute the amount of Fe (II) present in the sample solution from predetermined calibration curve. To study the effect of other ions, the respective foreign ions were added to aqueous phase before the extraction and adjustment of pH.

E. Determination of Iron in Pyrolusite Ore

Sample weighing 0.1gm to 0.2 gm was dissolved in10 ml aquaregia. The resulting solution was evaporated to dryness and the residue was then dissolved in 10 ml of 1 M HCl. The final solution was diluted up to the mark in a 100 ml volumetric flask with doubly distilled water. Using an aliquot of this solution (1 ml) Iron was analyzed by the procedure as described earlier.

F. Determination of Iron in Pharmceutical Sample

Procedure for the Determination of Fe(II) in Pharmaceutical Sample 0.5-1.0 gm sample of pharmaceutical product was dissolved in boiling with 10 ml of aquaregia. The resulting solution was evaporated to dryness and the residue was dissolved in 10 ml of 6N HCl filter, if required. The resulting solution was diluted to 250 ml of distilled water. 1ml aliquot of this solution was analyzed for Fe (II) by the procedure as described earlier.

III. RESULTS AND DISCUSSION

Fe (II) could be extracted quantitatively (99.67%) by MThBABA into N-butanol from an aqueous solution of pH 5.7 to 6.9 in presence of 2ml of 5% hydroxylamine hydrochloride [Fig I]

Extraction of Fe (II) was done in organic solvents. The values of extraction coefficient were in the order n-butanol> n amyl alcohol > ethyl acetate > carbon tetra chloride > chloroform > chlorobenzene > bromobenzene > nitrobenxenxe >benzene [figure I1] Due to the highest extraction coefficient, n-butanol was the choice of solvent for the entire extraction process..

The n-btanol extract of Fe: MThBABA complex showed an intense peak at 480 nm [Fig - III]. The absorbance due to the reagent is negligible at this wavelength, (wavelength for reagent = 400) Hence the entire experiment was performed at wavelength 480 nm.

Calibration curve confirms that the system obeys Beer's law at this wavelength over a Fe (II) concentration range of 0.1 to 12.0 µg/ml [Fig -IV]. The molar absorptivity of the extracted complex on the basis of Fe (II) content was calculated to be 13449 L mol^{-1} cm^{-1}.

It was found that 2 ml of 2.0% DMF solution of MThBABA was sufficient to extract 120 µg of Fe (II). The colour of the n butanol extract was found to be stable at least 24 hrs. at room temperature.

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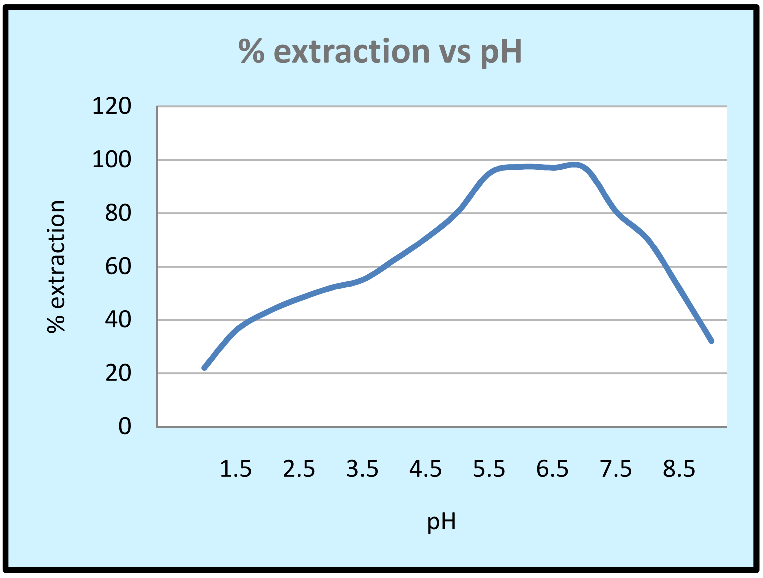

Figure 2: % Extraction of Iron at Different pH

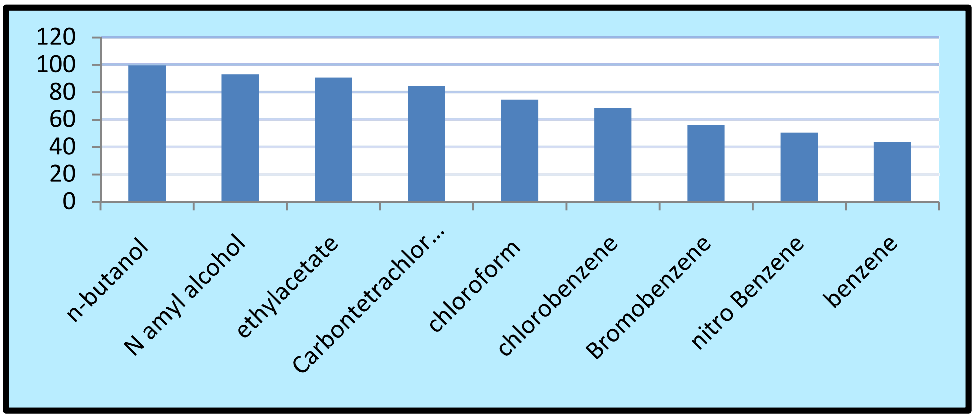

Figure 3: % Extraction of Iron into Various Solvents

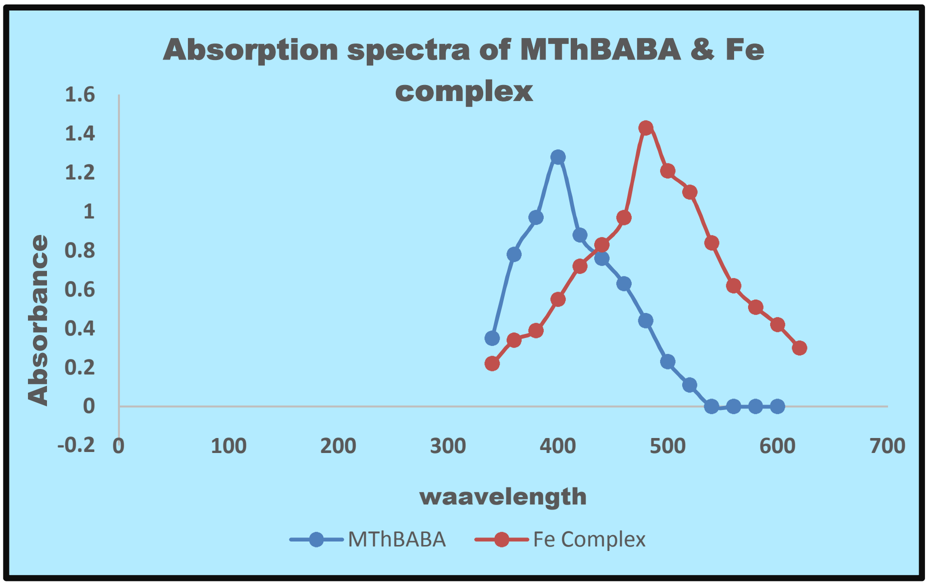

Figure 4: Absorption Spectra of MThBABA and Fe Complex

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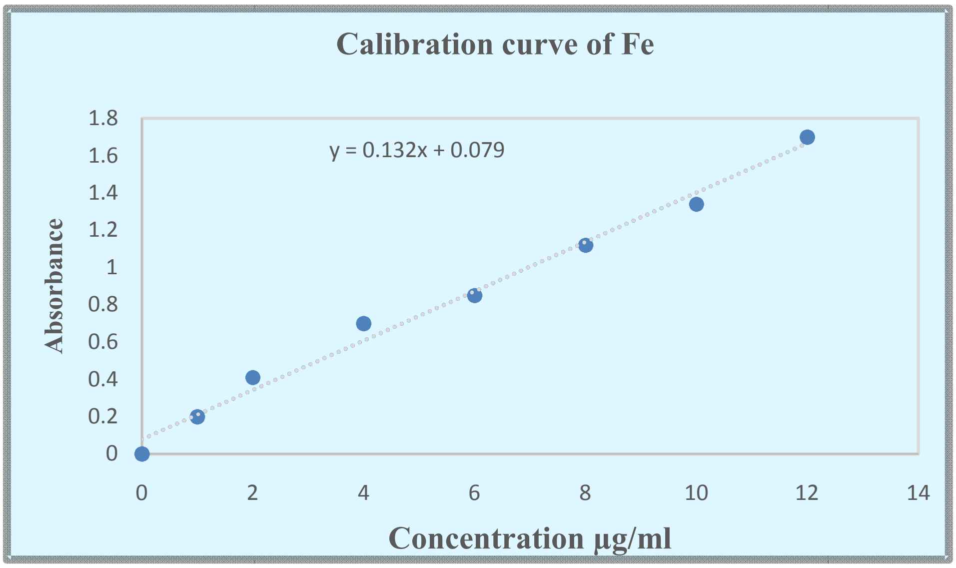

Figure 5: Calibration curve of Fe: MThBABA Complex

IV. EFFECT OF OTHER IONS

Fe (II) (100 µg) was determined in the presence of various ions. The following ions, did not interfere .

- 10 mg of each of, Li (I), Be (II), Ba (II), Ca (II), Sr (II), Al (III), Ti (III), V (V), Mo (VI), U (VI)
- 0.1mg each of Ag(I) & Pt (IV) and 20 mg each of chloride, bromide, iodide, phosphates, tartrate, acetate, citrate and thiosulphate, thiocyanide, tri ethanol amine, ascorbic acid.

Interference by various ions was removed by using appropriate masking agent.

- 10 mg of Cu (II) was masked by I ml of 2M EDTA or Sodium dihydrogen phosphate
- 10 mg of Ni(II) was masked by I ml of 2M 5-sulphosalicylic acid
- 10 mg of Cr(III was masked by I ml 2 M Tri ethanol amine
- 10 mg of Co(II) was masked by I ml of 2M Ascorbic acid

IV. COMPOSITION OF THE EXTRACTED COMPLEX

The composition of the extracted complex was found to be 1:1 (Fe: MThBABA) by Job's continuous variation and Mole ratio methods (Fig- 6 & Fig- 7).

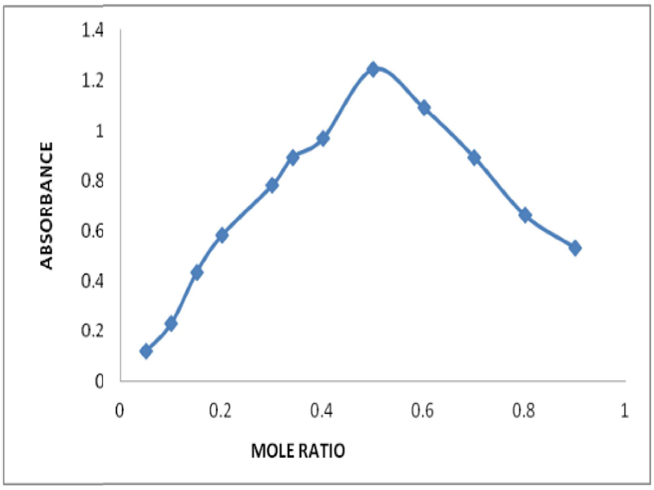

Figure 6: Composition of Complex by Job's Method

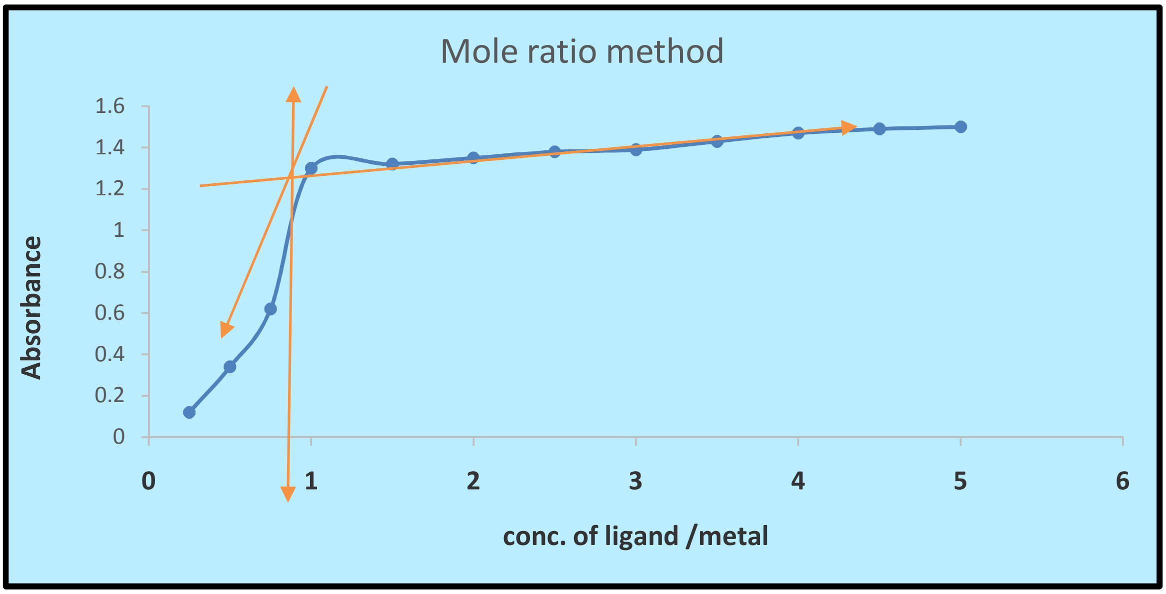

Figure 7: Composition of Complex by Mole ratio Method

V. ACCURACY, PRECISION, SENSITIVITY AND APPLICATIONS OF METHOD

In order to determine the accuracy and precision of the proposed method, the experiment was repeated ten times. 50 µg of Fe (II) in 10 cm^3 solutions was taken. The average of 10 determination of 50 µg of Fe (II) in 10 cm3 solutions was 49.85 µg, which is varied between 49.61 and 50.09 at 95% confidence limit Standard deviation and Sandell's sensitivity of the extracted species is found to be ±0.337 and 0.0176µgcm-2 respectively.

The proposed method has been applied for the determination of Fe (II) in pharmaceutical samples. The results of the analysis of the samples were comparable with those obtained by the standard method.[15] for Fe (II) (Table - II).

Table II: Determination of Iron in pharmaceutical sample and Pyrolusite ore

SAMPLES	Pyrolusite ore	FeriumXT
Present method (Based on the mean of three determinations)	5.93%	99.92mg
1:10 Phenonthroline method [16]	5.97%	99.95mg
Reported value (mg)		100mg

VI. CONCLUSION

From the above experiments, it is found that Schiff base, [MThBABA] is a good sensitive reagent for development of rapid and sensitive extractive spectrophotometric method for the determination of Fe (II) and it has been competently applied for determination of Fe (II) in Pharmaetisical pharmaceutical samples sample & Pyrolusite ore.

VII. ACKNOWLEDGEMENT

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REFERENCES

[1]. Monior –Williams GW, Trace Elements in Food, Ess Ex Street W.C. London Chapman and Hall Ltd. 1949.

[2]. Anovski T, Mamedovic T, Rastvovac M, "Determination of certain elements in human serum albumin by neutron activation analysis", J Radioanal Chem., 1972; 12(1): 483-489.

[3]. De AK, Khopkar SM, Chalmers RA, Solvent extraction of Metals. Van Nostrad Reinhold Co. London: 1970.
[4]. Temel, H.; Hosgoren, H. New Cu(II), Mn(III), Ni(II) and Zn(II) complexes with chiral quadridentate Schiff base. Transition Metal Chemistry 2002, 27, 609-612
[5]. Singh R.B, Garg BS, and Singh RP, Talanta,(1979); 26(2): 525 - 526
[6]. Karthikeyan J, Naik P.P, Shetty AN, Environ Monit. Assess, (2011); 176(1 - 4): 419
[7]. Amer SM, El-Saharty YS, Metwally FH, Younes KM, Spectrophotometric study of Etodolac complexes with copper (II) and iron (II), J Agric Food Chem;(2005); 53(14):5492-5498
[8]. Lingayat SK, Development of New Spectrophotometric Method for the Determination of Some Metal Ions from Transition Metals. Ph. D. Thesis, University of Mumbai, (2006)
[9]. Bhaskare CK, Kawatkar SG, Extractive photometric simultaneous determination of Iron (II) and copper (II) with syn-phenyl-α-pyridyl ketoxime and analysis of ferrites, Talanta,(1975); 22(2): 189-193
[10]. Mahajan HA and Patil MR, Chim Anal, (1992); 37: 239
[11]. Lokhande RS, Makhijani Mamta and Barhate V D, Indian J. Chem, (2003); 15 (1): 535-537
[12]. Khambekar AM and Sawant AD, J. Indian Chem. Soc. (1997); 36A (5): 459 - 460
[13]. Nath R, Nantial SC and Singh H, Labdev, Part-A, (1974);12A (2): 53- 58
[14]. Vogel AI, Practical Organic chemistry, 5th Ed. Longman group limited London,(1989)
[15]. Vogel AI, Text Book of Quantitative Inorganic Analysis Longman Green and Co.Ltd. London;(1978)
[16]. Sandell EB, Colorimetric Determination of Traces of Metals 3rd Edn, Interscience, NY;(1965); 41