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DEVELOPMENT OF EXTRACTIVE SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF MANGANESE (II) WITH SCHIFF BASE 2-[(2-HYDROXYPHENYLIMINO) METHYL]-4-NITROPHENOL

Datta B. Mandhare and Vasant D. Barhate*

V.E. S. College of Art, Science and Commerce, Sindhi Society Chembur, Mumbai - 400071, India.

***Author for Correspondence: Vasant D. Barhate**
V.E. S. College of Art, Science and Commerce, Sindhi Society Chembur, Mumbai - 400071, India.

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ABSTRACT
A simple spectrophotometric method has been developed for the determination of Manganese (II) by using Schiff base 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP]. HPIMNP extracts Mn (II) quantitatively (99.78%) into n-Butyl alcohol from an aqueous solution of pH range 10.0-11.0. The n-Butyl alcohol extracts show maximum absorption at 485 nm (λ max). Beer's Law is obeyed over the Mn (II) concentration range of 0.1 to 8.0 µg/ ml. The Molar absorptivity and Sandell's sensitivity for Mn – HPIMNP system is 10998.08 L mol $^{-1}$ cm^{-1} and 0.005µg.cm^{-2} respectively. The composition of extracted species is found to be 1: 2 [Mn - HPIMNP] by Job's continuous variation and Mole - ratio method. Interference by various ions has been studied. The proposed method is rapid, sensitive, reproducible and accurate and it has been satisfactory applied for determination of Manganese in Ore and Alloy samples.

KEYWORDS: Solvent Extraction, Extractive Spectrophotometry, Manganese (II), Schiff base, 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP], Ores, Alloy sample.

INTRODUCTION
Various reagents[1] are available for the spectrophotometric determination of Manganese (II) of which Oximes, Schiff bases and it derivatives constitutes an important class.[2][3] Synthesis and Antimicrobial Activity of Schiff base 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP] has been reported.[4] However Analytical application of HPIMNP was not studied. In the present communication, we describe the extractive spectrophotometric determination of Mn (II) with 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP].

MATERIAL AND METHODS
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. The reagent HPIMNP was synthesized by condensation of 5-Nitro salicylaldehyde with 2 - amino phenol as per reported procedure.[4] The resulting product was recrystallized by using ethanol[5] and characterized by elemental and spectral analysis. Its 0.5% solution was prepared in dimethylformamide (DMF). A stock solution of Mn (II) was prepared by dissolving manganese sulfate in double distilled water containing dilute sulphuric acid and it was standardized by Potassium Periodate method.[6] Working solutions of Mn (II) were made by suitable dilution. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

Extraction and separation of Mn (II)
An aliquot of aqueous solution containing 500 µg of Mn (II) and 1ml of 1.0 % solution of HPIMNP prepared in DMF were mixed in 25 ml beaker. The pH of solution was adjusted to desired value with dilute solution of HCl/ NaOH, Keeping the total volume to 10 ml with distilled water and followed by digestion on boiling water bath for 5 minutes. After cooling the resulting solution was then transferred into 125 ml separatory funnel. The beaker was then washed with 5 ml portion of organic solvent twice and each washing was added to the solution in separatory funnel. The two phases were equilibrated for one minute and allowed to separate. After the separation of two phases, pH of the equilibrated aqueous phase was measured and Manganese content in each phase was determined by the Potassium Periodate method.[6] The extraction was carried out with different solvents to find out the best extracting solvent. On the basis of Manganese content in aqueous and organic phase, extraction coefficient and percent extraction was calculated.

Extractive Spectrophotometric Determination of Mn (II)

To an aliquot of aqueous solution containing 0.1-8.0 µg of Mn (II), 2 ml of Ammonium chloride & ammonia of Buffer solution pH 10.0 and 1ml of 0.5% solution of HPIMNP prepared in DMF were added. The volume of solution was made up to 10 ml with distilled water. The solution followed by digestion on boiling water bath for 5 minutes. After cooling the solution was equilibrated for half minute with 10 ml of n-Butyl alcohol and the phases were allowed to separate. The n-Butyl alcohol extract was collected in a 10 ml measuring flask and made up to mark with n-Butyl alcohol. The absorbance of n-Butyl alcohol extract was measured at 485 nm against a reagent blank prepared under identical conditions. The Mn (II) content of the sample solution was determined from 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.

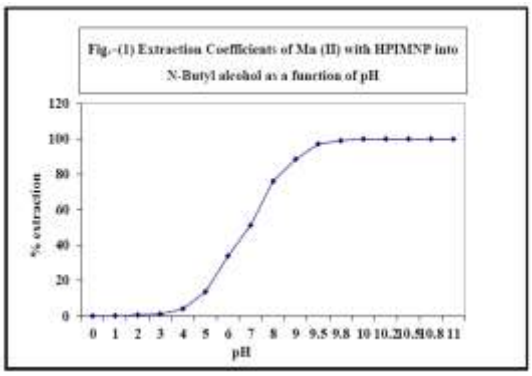
Determination of Manganese in Pyrolusite ore and Manganese steel sample

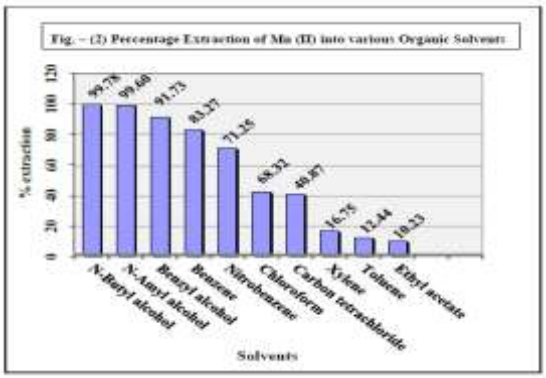
0.1 to 0.2 gm sample was dissolved in boiling with 10 ml aquaregia. The resulting solution was evaporated to dryness and the residue was then dissolved in 10 ml of 1 M HCl, filter if required and solution was diluted to 100 ml with doubly distilled water. To an aliquot of this solution (1 ml) was analyzed for Manganese by the by the procedure as described earlier.

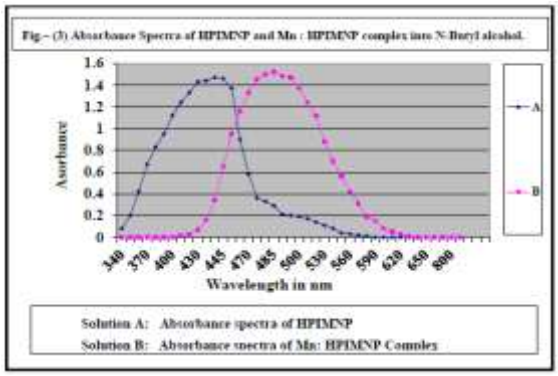
RESULTS AND DISCUSSION

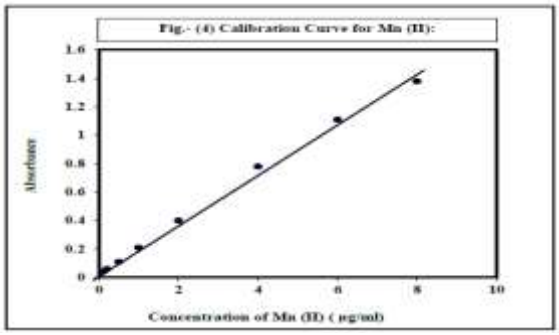
Manganese (II) could be extracted quantitatively (99.78%) by HPIMNP into n-Butyl alcohol from an aqueous solution of pH range 10.0 -11.0 (Fig. 1). Organic solvents used for extraction of Mn (II) can be arranged on the basis of their extraction coefficient values as N - Butyl alcohol > n-amyl alcohol > benzyl alcohol > benzene > nitro benzene > chloroform > carbon tetrachloride > xylene > toluene > ethyl acetate (Fig. 2). N-Butyl alcohol was found to be the best extracting solvent; hence, it was selected for extraction throughout the work.

The n-Butyl alcohol extract of Mn- HPIMNP complex showed an intense peak at 485 nm. The absorbance due to the reagent is negligible at this wavelength, so the absorption measurements were taken at this wavelength (Fig. 3). The result shows that the system confirmed to Beer's law at this wavelength over a Mn (II) concentration range 0.1 to 8.0 µg/ml (Fig- 4). The molar absorptivity and sandell's sensitivity of the extracted species on the basis of Mn (II) content were calculated to be 10998.08 L mol $^{-1}$ cm^{-1} and 0.005µg.cm^{-2} respectively. It was found that 1 ml of 0.5 % solution of HPIMNP prepared in DMF was sufficient to extract 80 µg of Mn (II). The color of the n-Butyl alcohol extract was found to be stable at least 48 hrs at room temperature.









EFFECT OF OTHER IONS

Mn (II) (100 µg) was determined in the presence of various ions. The following ions in the amount indicated, did not interfere in the spectrophotometric determination of Mn (II)(60 µg) :10 mg each of, Li (I), Be (II), Ba (II), Ca (II), Sr (II), Al (III), Ti (III), V (V), Fe (II), Fe (III), Mo (VI), U (VI) And 0.1 mg each of Ru (III), Pt (IV) and Rh (III). And 20 mg each of chloride, bromide, iodide, fluoride, chlorate, bromate, iodate, sulphide,

phosphates, tartrate, acetate, citrate and thiosulphate, thiocyanide, triethanol amine, ascorbic acid.

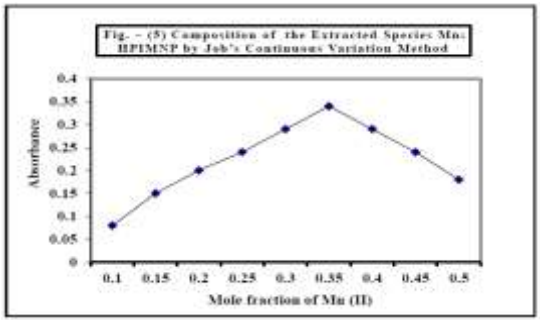
Interference by various ions was removed by using appropriate masking agent (Table 1).

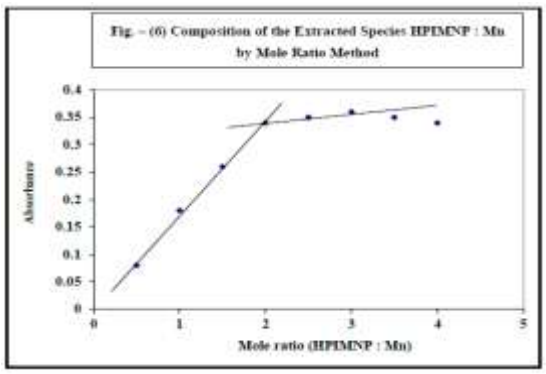
Table – 1 Masking agents required for suppressing the interference by other ions.

Interfering ion	Amount added in mg	Masking agent added 1ml of 2M solution
Cu(II)	10	Sodium Thiosulphate
Co (II)	10	Ascorbic acid
Ag (I) & Pb (II)	10	Potassium Bromide
Cr (III)	10	Tri ethanol Amine
Ni(II)	10	5-sulphosalicylic acid
Zn (II)	10	Sodium Fluoride

COMPOSITION OF THE EXTRACTED COMPLEX

The composition of the extracted complex was found to be 1:2 (Mn: HPIMNP) by Job's continuous variation and Mole ratio methods (Fig- 5 & Fig- 6).





PRECISION, ACCURACY, SENSITIVITY AND APPLICATION OF METHOD

The precision and accuracy of the method were tested by analyzing the solution containing a known amount of Mn (II) following the recommended procedure. The average of 10 determination of 5 µg of Mn (II) in 10 cm^3 solutions was 4.999 µg, which is varied between 4.995 and 5.003 at 95% confidence limit and standard deviation is ±0.006. The present method has been successfully applied to the determination of Mn (II) in Manganese steel samples and Synthetic Mixture. The Results of analysis of samples are comparable with standard method [6] and reported value (Table 2).

Table – 2 Determination of Manganese in Manganese steel sample and Synthetic Mixture.

Samples	Certified value of Mn	Present method	Known method
Pyrolusite ore	35.0 %	34.80%	35.00%
Mo-Mn steel	1.61%	1.60%	1.60%

Results are the average of three independent determinations.

CONCLUSIONS

From the above discussions, it is found that Schiff base, 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP] is a good sensitive reagent for development of rapid and sensitive extractive spectrophotometric method for the determination of Mn (II) and it has been satisfactory applied for determination of Mn (II) in Pyrolusite ore and Manganese steel sample.

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