ESTIMATION OF MANGANESE (II) FROM WATER, FOOD AND PHARMACEUTICAL SAMPLES USING 1-PHENYL-1-HYDRAZONYL-2-OXIMINO PROPANE –1, 2 –DIONE REAGENT

Tekale P P, S P Tekale and S K Lingayat
Department of Chemistry, G. N. Khalsa College, Matunga. Mumbai-400019
ptekale@rediffmail.com,
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ABSTRACT

Determination of Manganese (II) was carried out using 1-phenyl-1-hydrazonyl-2-oximino propane-1, 2-dione (HPHOPD) reagent by extractive spectrophotometric method. HPHOPD reagent formed a complex with Manganese (II). A systematic study for optimum extraction conditions has been carried out. Maximum extraction was obtained using chloroform as an organic phase at pH 7.8. Extracted species has absorption maxima at 430 nm and obeyed Beer's law over the range 1-100 ppm of Manganese. The molar absorptivity at this wave length is 0.89×10^3 liters $mole^{-1}$ cm^{-1}. The proposed method was selective for Manganese (II) and was successfully applied to determine its content in water, food, alcohol beverage and pharmaceutical samples.

Keywords: Manganese (II), extractive spectrophotometric, vegetable oils

INTRODUCTION

Minerals are important for biological systems. They are required for body structure, fluid balance, protein structures and to produce hormones. They act as co-factors, catalysts or inhibitors of all enzymes in the body (Groff 1995).

In the human body, manganese functions as an enzyme activator and as a component of metalloenzymes (Aschner 2000; Crowley 2000; Yoder 2000). Manganese deficiency is associated with nausea, vomiting, poor glucose tolerance (high blood sugar levels), skin rash, loss of hair color, excessive bone loss, low cholesterol levels, dizziness, hearing loss, and compromised function of the reproductive system etc. Severe manganese deficiency in infants can cause paralysis, convulsions, blindness, and deafness. Therefore, Manganese is essential for human body in trace level. Although daily intake of manganese is recommended for health but when the uptake is too high health problems will also occur. Over consumption of manganese is also associated with impotency. High doses of manganese may inhibit the absorption of iron, copper, and zinc. Manganese toxicity is most likely to occur in people with chronic liver disease, as the liver plays an important role in eliminating excess manganese from the body (Keen 2000). Therefore, Manganese is a trace element, which is not only necessary for humans to survive, but it is also toxic when too high concentrations are present in a human body.

Balanced food is a key source for manganese (Groff 1995). Water and food containing unsafe levels of amount of minerals **can pose substantial health risks to consumers** (McLaughlin 1995; Tekale 2010). **It** can enter food during harvesting, production, storage and cooking etc. Hence analysis of water, food even in alcoholic beverages is important (González 2005; Orriss 2000; Ibanez 2008) for chemical contaminates. Current work deals with analysis of water, food and pharmaceutical samples for determination of Manganese by extractive spectrophotometric method. Different parameters were varied to get optimum conditions, where maximum extraction of manganese is possible.

MATERIALS AND METHODS

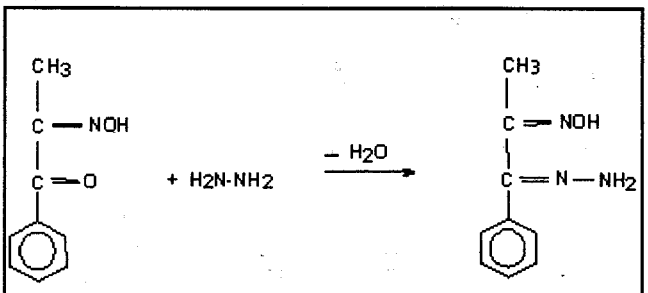
The Stock Solution of Mn (II) were prepared from manganese (II) sulphate purified monohydrate (loba Chemie). The stock solution was standardized volumetrically by EDTA (Vogel 1998).

A digital pH meter, (Elico Private Ltd, India) with a combined glass and calomel electrode (Toshniwal - Mollar, India) and UV 2100 spectrophotometer (Shimadzu) with glass cells of path length 1 cm was used.

Synthesis of 1-phenyl-1-hydrazonyl-2-oximino propane – 1,2 – dione (HPHOPD) reagent

The reagent HPHOPD was synthesized (Tekale 2010) by carrying out a reaction between *iso*-nitrosopropiophenone and 85 % hydrazine hydrate. The purity of the product was checked by melting point and GC-MS technique.

Chemical reaction:



iso-nitrosopropiophenone 1-phenyl-1-hydrazonyl-2-oximinopropane–1,2–dione

Extraction Procedure

An aqueous solution (10.0 cm^3) containing 0.1 mg Mn (II) metal and 0.05 M of 1-phenyl-1-hydrazonyl-2-oximino propane -1,2- dione reagent in n-butanol, after adjusting the pH = 7.8 was equilibrated with 10.0 cm^3 of chloroform for 1 min. After separation of the phases, the absorbance of the Mn (II): HPHOPD complex in organic phase was directly measured at 430 nm.

Preparation of Pharmaceutical samples for determination of Mn (II)

To a 40.0 –50.0 cm^3 Multivitamin Syrup or a 5 gm of tablet powder, 1.0 cm^3 of concentrated HCl : HNO_3 (1:1) was added and evaporated to dryness. It was treated with 5.0 cm^3 of 30 % H_2O_2 until solution became colorless. The colourless solution was then treated with dil. HCl and evaporated to dryness. The residue was dissolved in 10.0 cm^3 of distilled water and an aliquot of this was used for further analysis.

RESULTS AND DISCUSSION

a) Absorption spectrum

Mn: HPHOPD complex after extraction from aqueous phase into organic phase was scanned from 300 nm to 600 nm against reagent blank (Figure 1). Maximum Absorbance value was observed at 430 nm. Therefore, 430 nm was selected for the absorbance measurement for all experiments.

a) Systematic Study of extraction of Mn (II)

A systematic study of the extraction of Mn (II) was carried to obtain the conditions where maximum extraction was obtained. Different parameters were studied.

b) Effect of pH

The extraction of Mn (II) was carried out over the 1.0 - 10.0 pH range (Figure 2). It was observed that the extraction was increased upto 7.8 pH and decreased beyond it. Therefore, pH 7.8 was used throughout the exp

a) Effect of different solvents

Different organic solvents like chloroform, carbon tetrachloride, ethanol were used for the extraction of Mn (II) : HPHOPD complex. Maximum extraction and quick phase separation was obtained when chloroform was used as organic phase. Therefore, chloroform was used as organic phase for all experiments.

b) Effect of HPHOPD reagent concentration

The concentration of HPHOPD reagent was varied from 0.001 M to 0.05 M (Figure 3). It was observed that as the reagent concentration increased percent extraction increased. Maximum extraction was observed at 0.05 M concentration of HPHOPD reagent. Therefore, 0.05 M HPHOPD was used for extraction.

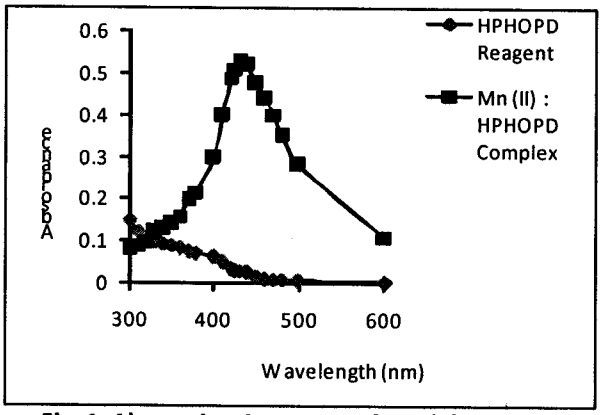


Fig. 1. Absorption Spectrum of Mn (II): HPHOPD Complex

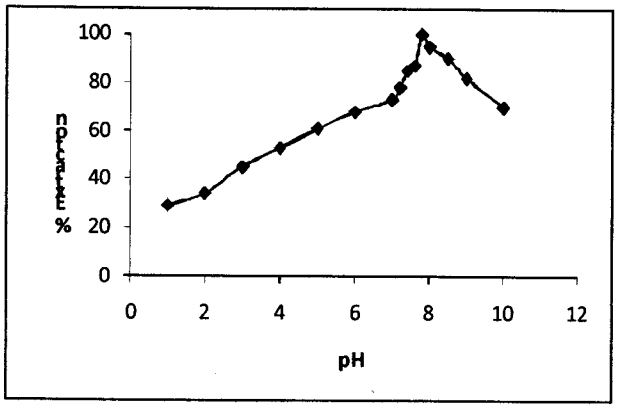


Fig. 2 Effect of pH on extraction

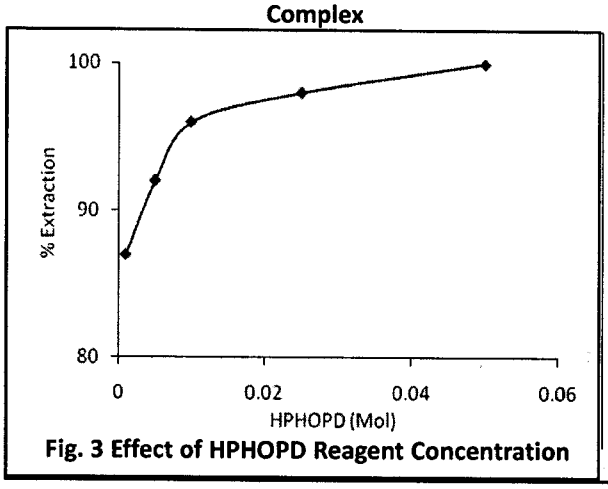


Fig. 3 Effect of HPHOPD Reagent Concentration

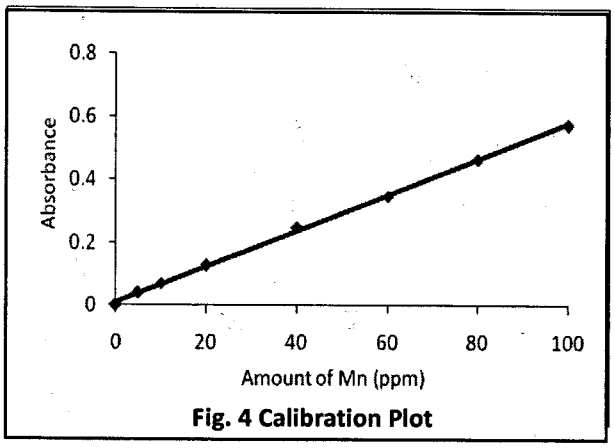


Fig. 4 Calibration Plot

a) Effect of equilibration time

Equilibration time for the extraction of Mn (II) : HPHOPD complex from aqueous phase to organic phase was varied from 0.5, 1 and 2 minutes. It was observed that 1.0 minute was sufficient equilibration time for maximum extraction. Therefore, 1 minute was used as equilibration time for all experiments.

b) Calibration plot

A calibration plot of absorbance against concentration of Mn (II) : HPHOPD complex gave a linear and reproducible graph in the concentration range of 1-100 ppm (Figure 4). The Beer's law is obeyed in this range. The molar absorptivity was 0.89 X 10^3 Lit $Mol^{-1}cm^{-1}$. **a) Effect of diverse ion concentration**

Extraction of Mn (II) was carried out in presence of different metals ions (Table 1). The tolerance limit was set to that amount of foreign ion causing ± 2% error in recovery of Mn (II). It was observed that metal ions like Ni^{+2}, Mg^{+2}, Ca^{+2}, Pb^{+2}, Zn^{+2}, Al^{+3} and Fe^{+3} showed high tolerance limit. Therefore the optimized parameters can use for maximum extraction of Mn (II) even in presence of diverse ions.

Quantitative determination of Mn (II) from Samples

Water Sample

Extraction of Mn (II) was carried out from water (Municipal tap) (Table 2). It was found that there was presence of Mn (II) in water.

Food Samples and Alcoholic beverage

Juices (from local Juice centre) like Orange Juice, Pineapple Juice, Strawberry Juice, Mix Fruit Juice and alcoholic Alcoholic beverage were analyzed for Manganese contents (Table 2). The percent extraction was in the 85 – 95 % range.

Pharmaceutical Samples

Extraction of Zinc was carried out from Multivitamin Syrup and tablet (Branded). The results are included in Table 3.

CONCLUSIONS

An extractive spectrophotometric technique is a separation method which allows the determination of a metal in organic phase without using stripping solvent. It is simple and economical method. Mn (II) formed a complex with HPHOPD which was extracted in chloroform at pH = 7.8 quantitatively. The method is simple, quick and reliable. The interference of diverse ions was studied and optimum conditions were developed for the determination of Mn in water, food and Pharmaceutical samples.

Table 1. Effect of diverse ion concentration Mn (II) concentration: 10 ppm

Ion	Tolerance limit (ppm)
Ni^{+2}	20.0
Mg^{+2}	25.0
Ca^{+2}	32.0
Pb^{+2}	35.0
Zn^{+2}	28.0
Al^{+3}	39.1
Fe^{+3}	38.2

Table 2. Determination of Mn (II) from Water, Food and Alcoholic beverage Samples

Sample	% Recovery of Mn (II)*
Tap Water	89.0
Orange Juice	75.0
Pineapple Juice	89.5
Mixed Fruit Juice	88.6
Strawberry Juice	92.3
Grape Juice	93.0
Alcoholic beverage	85.3

*The values of percent extraction of Mn are mean of three readings.

Table 3. Determination of Mn (II) from Pharmaceutical Samples

Sample	% Recovery of Zn (II)*
Multivitamin Syrup	97.9
Multivitamin Tablet Powder	98.5

*The values of percent extraction of Mn are mean of three readings.

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