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Analytical method development for extractive spectrophotometric determination of Ni(II) using 1,2-propanedione-1-phenyl-1-(2-hydroxy-5-bromo-benzilidineazine)-2-oxime [PDPHBBAO]

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ABSTRACT

1,2-propanedione-1-phenyl-1-(2-hydroxy-5-bromo-benzilidineazine)-2-oxime [PDPHBBAO] is proposed as a new sensitive and selective reagent for the spectrophotometric determination of trace amount of nickel. The optimum extraction conditions were evaluated by studying various parameters like pH, solvent, reagent concentration, equilibration time and stability of extracted complex. The reagent reacts with nickel to form a light yellow colored 1:1 chelate, in the pH range 9.5 to 10.5. The complex is extracted in chloroform. The absorption spectrum shows maxima at 435nm. Beer's law is obeyed in the concentration range 0.01-10.00 μg ml^{-1} nickel. The molar absorptivity and Sandell's Sensitivity of the extracted species are 5.3388X 10^3 Lit mol^{-1} cm^{-1} and 10.99 X 10^{-3} μg cm^{-2} respectively. The variation from mean at 95% confidence limit is 5.00 $\pm$ 0.12. The proposed method is highly sensitive, selective, simple, rapid, accurate, and has been satisfactorily applied for the determination of nickel in the synthetic mixtures, and real samples. © 2013 Trade Science Inc. - INDIA

KEYWORDS

PDPHBBAO;
Nickel;
Spectrophotomertic determination.

INTRODUCTION

Nickel has many industrial and Biological applications. It is used in many industrial and consumer products, including stainless steel, magnets, coinage and special alloys such as nickel brasses and bronzes, and alloys with copper, chromium, aluminium, lead, cobalt, silver, and gold. is also used for plating and as a green tint in glass.

Nickel plays numerous roles in biology. In fact urease (an enzyme which assists in the hydrolysis of urea) contains nickel. The NiFe-hydrogenases contain nickel in addition to iron-sulfur clusters. Such [NiFe]-hydrogenases characteristically oxidize H_2. A nickel-tetrapyrrole coenzyme F430 is present in the methyl coenzyme M reductase which powers methanogenic archaea. One of the carbon monoxide dehydrogenase enzymes consists of a Fe-Ni-S cluster. Other nickel-containing enzymes include a class of superoxide dismutase and a glyoxalase.

Literature survey reveal a number of reagents have been employed for the Spectrophotometric determination of Ni (II), but suffer from limitations such as critical pH[3-7], requirement of masking agent[3] or other agents[1,4], requirement of heating[2], interference from some ions[3,8], less sensitive[9]etc. In this paper a new method has been developed using 1,2-propanedione-1-phenyl-1-(2-hydroxy-5-bromo-

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benzilidineazine)-2-oxime [PDPHBBAO] for extraction and Spectrophotometric determination of nickel, which is simple, selective and sensitive.

EXPERIMENTAL

A Shimadzu UV visible 2401spectrophotometer with 1 cm quartz cell and a digital ELICO LI-120 pH meter were used for the absorbance and pH measurement respectively. The stock solution of nickel was prepared and standardized[10] and working solution of lower concentration was obtained by suitable dilution. The reagent, PDPHBBAO was synthesized by condensation of 1,2-Propanedione-1-phenyl-1-azine-2-oxime with 5-Bromosalicyldehyde in equimolar proportion and recrystalised from ethanol The reagent was confirmed by IR, NMR, Mass and elemental analysis. A 0.005% reagent solution of PDPHBBAO in ethanol was prepared and used for extraction studies.

RECOMMENDED PROCEDURE

The extraction experiments were performed by shaking the appropriate organic and aqueous solution at O/A phase ratio of 1. The reagent PDPHBBAO formed a light yellow coloured complex with nickel (II) which was transferred in a separating funnel. It was extracted into 10 cm^3 chloroform and then transferred to 10mL volumetric flask by passing through some amount of sodium sulphate in order to absorb trace amount of water. The amount of nickel present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 435 nm and that in the aqueous phase was determined by known method.

TABLE 1 : Results and discussion

Condition	Results
Absorption Maxima	435 nm
Solvent	Chloroform
pH range	9.5–10.5.
Equilibration time	1 min
Stability of Nickel-PDPHBBAO	5 h
Beer's range	0.1 to 10.0 μg ml^{-1}
Molar absorptivity	1.4297 X 10^3 Lit mol^{-1} cm^{-1}
Sandell's sensitivity	4.10 X 10^{-3} μg / cm^2
Mole Ratio of Ni : PDPHBBAO	1 : 1

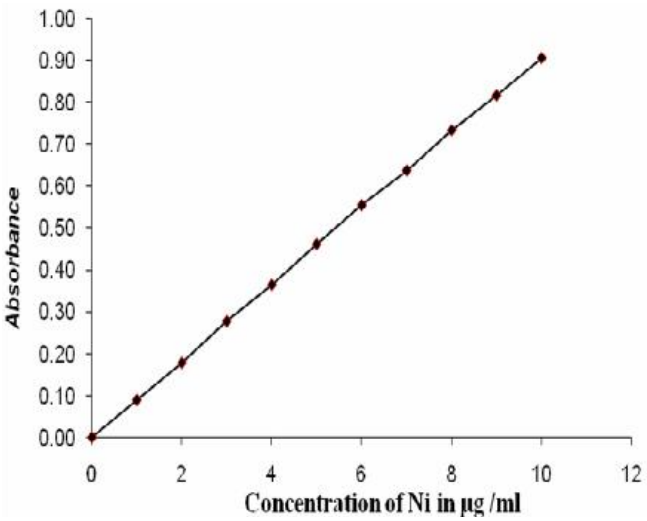

Figure 1 : Calibration Plot

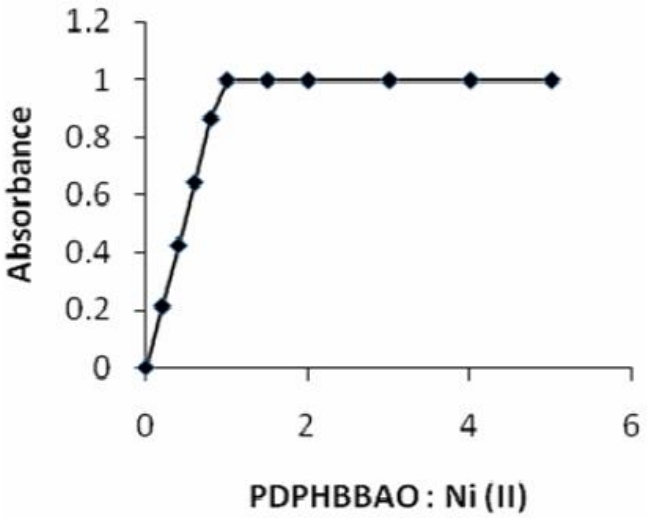

Figure 2 : Mole Ratio Plot

Effect of foreign ions

The tolerance limit of different metal ions was studied by carrying out determinations of 100 μg of Ni (II) in presence of large number of foreign ions. TABLE 1 shows the tolerance limit of foreign ion to cause an error of not more than ± 2% in recovery of the Ni (II). EDTA interfered strongly for the determination Ni (II). EDTA was masked with calcium chloride. Cu(II) was reduced to Cu(I) with sodium metabisulfite. Fe(II) and Fe(III) were masked with tartarate for the determination of Ni(II). Pd(II) was separated in the acidic range (0.1N H_2SO_4). Sulphate interfered seriously for Pb(II). Co(II)

TABLE 2 : Interference study

Sr. No.	Interfering ions	Tolerance Limit (mg)
1	Acetate, Oxalate, Citrate, SCN^-, $S_2O_7^{2-}$, NO_3^-, Urea, NO_2^2, $S_2O_8^{--}$, SO_4^{--}, SO_3^{--}, IO_3^-, BrO_3^-, ClO_3^-, Cl^-, Br^-, I^-, F^-.	20
2	Phosphate,	18
3	Thiourea	15
4	Zr(II), Ba(II), Mo(VI), Mg(II), Zn(II),	11
5	Ca(II), Al(III), Ce(IV), Mn(II)	8
6	Tl(I), Ag(I), Th(IV), Cr(VI), Li(I),	5
7	U(VI)	4
8	V(VI), Cd(II), Pb(II),	2
9	Rh(III), As(III)	1
10	Co^{+2},Cu^{+2}, Fe^{+2}, Fe^{+3}, Pd^{+2}, EDTA, Hg^{+2}	Interfere strongly

was separated sequentially for the determination of Ni(II).

Precision and accuracy

The precision and accuracy of the method were studied by analyzing a series of solutions containing known amounts of nickel (II). The average of 10 replicate analyses for nickel was 5.0 µg which varies between 5.0 µg which varies between 5.00 $\pm$ 0.12 at 95% confidence limit. The standard deviation was 0.16 and the variance was 0.03.

Application to the determination of nickel in synthetic mixtures

The present method was applied to the determination of nickel (10µg) in various synthetic mixtures and real samples. The results are shown in TABLE 3 & 4

TABLE 3 : Analysis of synthetic mixtures

Sr. No.	Synthetic mixture Composition[a]	Amount Found[b]
1	Ni (10)+Zn (10)	9.98
2	Ni (10) + Mo (10)	9.98
3	Ni (10) +Mg(10)	9.98
4	Ni (10) + Pb (10)	9.97
5	Ni (10) + U (10)	9.91

TABLE 4 : Analysis of real samples

Sr. No.	Sample	Amount of Ni (II)	
I	Nickel alloys	Certified Value	Found
1	Cupronickel	55.01%	54.995%
2	Steel	25.58%	25.545%
II	Vegetable Oil	0.0022%	0.0021%

[a] Values given are in microgram amount of the metals.[b] Average of five determination

CONCLUSION

The proposed method is more highly sensitive and selective than the reported methods for the extractive Spectrophotometric determination of microgram amounts of nickel. It offers advantages like reliability and reproducibility in addition to its simplicity, instant color development and suffers from less interference. It has been successfully applied to the determination of nickel at trace level in synthetic mixtures, vegetable oils and alloys.

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