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DEVELOPMENT OF METHOD FOR EXTRACTIVE SPECTROPHOTOMETRIC DETERMINATION OF Ni (II) WITH 2-HYDROXY-1-NAPHTHALENE CARBOXALDEHYDE PHENYL HYDRAZONE AS AN ANALYTICAL REAGENT

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ABSTRACT

A spectrophotometric method has been developed for the determination of Ni (II) using 2-hydroxy-1-naphthalene carboxaldehyde phenyl hydrazone as an extractive reagent. The reagent forms a coloured complex, which has been quantitatively extracted into n-butanol at pH 8. The method obeys Beer's law over arrange from 1 to 12 ppm. The Molar absorptivity and Sandell's sensitivity were 1.736×10^4 L mol^{-1} cm^{-1} and 0.1613 µg cm^{-2}, respectively. The proposed method is very sensitive and selective. The method has been successfully applied to synthetic and commercial samples.

Key words: Nickel spectrophotometric determination, n-Butanol**,** 2-hydroxy-1-naphthalene carboxaldehyde phenyl hydrazone.

INTRODUCTION

Nickel is a naturally occurring element. Pure nickel is a hard, silvery-white metal used to make stainless steel and other metal alloys. Nickel can combine with other elements such as chlorine, sulfur, and oxygen to form nickel compounds. The massive forms of chromium, aluminum and titanium, nickel is a very reactive element, but is slow to react in air at normal temperatures and pressures. Due to its permanence in air and its inertness to oxidation, it is used in coins, for plating iron, brass, etc., for chemical apparatus, and in certain alloys, such as German silver. Ni. Many nickel compounds dissolve fairly easy in water and have a green color. Nickel compounds are used for nickel plating, to color ceramics, to make some batteries, and as substances known as catalysts that increase the rate of chemical reactions. A wide variety of reagent has been reported for the spectrophotometric determination of nickel. However, these methods suffer from limitations

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such as critical pH[1-3], requirement of masking agent[1] or other agents[4,5], requirement of heating[6], and interference from some ions[1,7].

EXPERIMENTAL

The reagent 2-hydroxy-1-naphthalene carboxaldehyde phenyl hydrazone was synthesized by the given procedure. The stock solution of Ni (II) was prepared by dissolving a weighed amount of sulphate in double distilled water containing dilute sulphuric acid, which was diluted to the desired volume with double distilled water and standardized by DMG method[8]. Absorbance and pH measurements were carried out on a Shimadzu UV-Visible 2100 spectrophotometer with 1 cm quartz cells and digital pH meter with combined glass electrode respectively.

Procedure for the extraction

1.0 mL of aqueous solution containing 0.1 mg of nickel metal and 1 mL of reagent were mixed in a 50 mL beaker. The pH of the solution adjusted to 8.0 with 0.2 M boric acid and potassium chloride, keeping the volume 10 mL. The solution was transferred to 100 mL separatory funnel. The beaker was washed twice with n-butanol and transferred to the same funnel. The two phases were shaken for two minutes and allowed to separate. The organic phase was collected in 10 mL measuring flask and made up to the mark with organic solvent if required. After separation of the two phases, the pH of the aqueous phase was measured and the Ni (II) in each phase was determined by DMG method.

RESULTS AND DISCUSSION

The reagent HNPH forms dark yellow coloured complex with Ni (II), which was extracted into organic phase. The extraction of Ni (II) forms an aqueous phase by HNPH in n-butanol is studied over a wide range of experimental condition. The results of various studies are discussed below.

Extraction as a function of pH

The extraction of cobalt with 2-hydroxy-1-naphthalene carboxaldehyde phenyl hydrazone has been studied over the pH range 1-10 and was observed that percentage extraction of Ni (II) is maximum at pH 8.

Absorption spectrum

The absorption spectrum of Ni (II): 2-hydroxy-1-naphthalene carboxaldehyde phenyl hydrazone in n-butanol shows the maximum absorption at 395 nm. The absorption due to

reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 395 nm.

Influence of diluents

The suitability of diluents was investigated using organic solvents such as chloroform, ethyl acetate, isoamyl alcohol, xylene, hexane, diethyl ether, toluene, n-butanol, carbon tetrachloride, MIBK, nitrobenzene, etc. The extraction of Ni (II) was quantitative with HNPH in n-butanol. Hence, n-butanol was used for further extraction studies as it gave better and quicker phase separation.

Effect of salting out agents

The presence of 0.1 M salts of various alkali and alkaline metals does not show any effect over the absorbance value of Ni (II) : 2-hydroxy-1-naphthalene carboxaldehyde phenyl hydrazone complex extract. Therefore, no salting out agent was required during the extraction.

Effect of reagent concentration

Various volumes of 0.1% reagent solution were added to the sample solution containing 80 μg of nickel at respective pH values. The absorbance remained nearly constant when the volume of the reagent solution used was more than 1 mL. Therefore, 1 mL of 0.1% reagent was chosen for the quantitative determination of the metal.

Effect of equilibration time and stability of the complex:

The study of change in absorbance with variation in equilibrium time for extraction of extraction of the complex into organic solvent shows that equilibration time of 1 min is sufficient for the quantitative extraction of Nickel. The study of stability of colour of the Ni (II): HNPH complex with respect to time shows that the absorbance due to extracted species is stable up to 40 hours, after which slight decrease in absorbance is observed. Throughout the experimental work, for practical convenience, the measurements have been carried out within one hour of extraction of nickel.

Calibration plot

A calibration plot of absorbance against varying nickel concentration and fixed HNPH concentration gives linear and reproducible graph in the concentration range 1 to 12 ppm of nickel (Fig. 1). This shows that the Beer's law is obeyed in this range. The molar absorptivity and Sandell's sensitivity were calculated to be is 1.736×10^4 L mol^{-1} cm^{-1} and 0.1613 μg cm^{-2}, respectively.

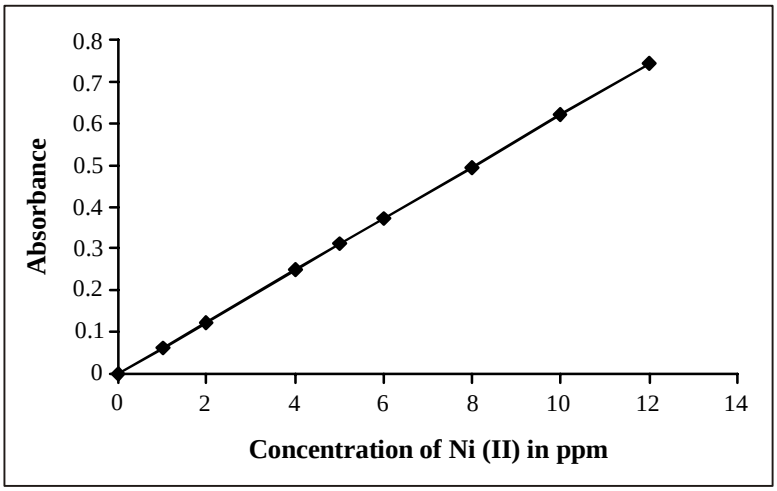

Fig. 1: Calibration plot of Ni (II): HNPH complex

Nature of extracted species

The composition of extracted species has been determined by Job's continuous variation method, Slope ratio method (Fig. 2) and Mole ratio method. It shows that the composition of Ni (II) : HNPH complex is 1 : 2.

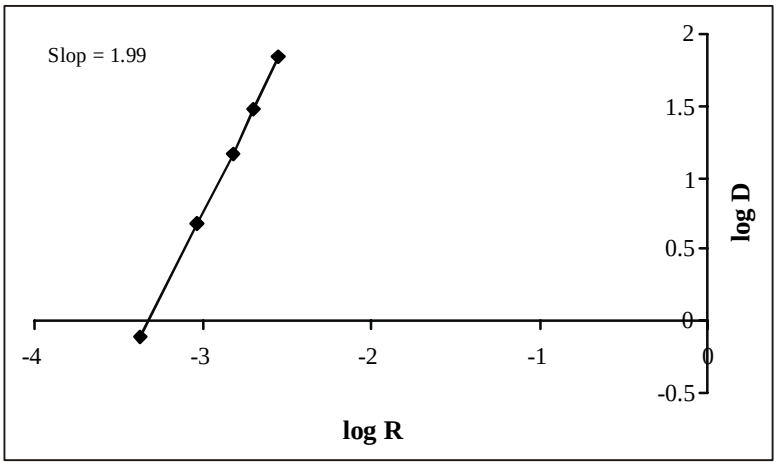

Fig. 2: Slope ratio method for Ni (II): HNPH complex

Effect of divalent ions and foreign ions

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 40 μg of nickel. The ions which show interference in

the spectrophotometric determination of nickel were overcome by using appropriate masking agents (Table 2).

Precision and accuracy

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing five solutions each containing 20 μg of nickel in the aqueous phase. The average of five determinations was 20.068 and variation from mean at 95% confidence limit was ± 0.228.

Applications

Various commercial samples and synthetic mixtures containing Ni (II) were prepared and analyzed according to the recommended procedure and the results were compared to those obtained by standard method. The proposed method facilitates separation of nickel (II) from synthetic mixtures. These metal ions do not extract and remain quantitatively in the aqueous phase under the optimum extraction conditions of nickel (II) with HNPH system facilitating separation of bivalent nickel quantitatively by the proposed method. The results found to be in good agreement with those obtained by the standard known method[10] (Table 1).

Table 1: Determination of Ni (II) using HNPH from different samples

S. No.	Sample	Amount of Ni (II)	
		Standard method	Present method
I	Nickel alloys		
1	Cupronickel	65.0%	64.9%
2	Steel	30.1%	29.9%
4	Nichrom	23.3%	23.2%
II	Vegetable oil	0.0016%	0.0015%
III	Synthetic mixture		
1	Ni (10) + Zn (10)	9.98 ppm	9.96 ppm
2	Ni (10) + Mo (10)	9.98 ppm	9.97 ppm
3	Ni (10) + Mg (10)	9.98 ppm	9.97ppm

Table 2: Effect of divalent ions and foreign ions

Ion	Tolerated ratio	Ion	Tolerated ratio
Cl^-	1 : 18	Zn^{2+}	1 : 10
Br^-	1 : 10	Ag^+	1 : 15
F^-	1 : 12	K^+	1 : 17
ClO^-_3	1 : 10	Mg^{2+}	1 : 16
BrO_3^-	1 : 15	Ca^{2+}	1 : 15
IO_3^-	1 : 15	Ba^{2+}	1 : 20
SO_3^{2-}	1 : 11	Bi^{2+}	1 : 20
SO_4^{2-}	1 : 19	V^{+5}	1 : 12
NO_2^-	1 : 08	Cr^{3+}	Masked
NO_3^-	1 : 10	Mn^{2+}	Masked
PO_4^{3-}	1 : 14	Ce^{4+}	Masked
$P_2O_7^{2-}$	1 : 20	CN^-	Masked
ClO_4^-	1 : 08	Tartarate	Masked

CONCLUSION

The results obtained show that HNPH in n-Butanol can be effectively used for quantitative extraction from aqueous media. The proposed method is found to be quantitative as compared to other standard method s. The equilibrium time required is very little, i.e. only 1 min and the complex is stable for 40 hrs. The results show good agreement with the standard method. The method is very fast, accurate and precise. The 2-Hydroxy-1-Naphthalene carboxaldehyde phenyl hydrazone was used for the first time for extraction of Ni (II) from various binary mixtures.

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