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USE OF HYDRAZINE CARBOXAMIDE-2-[(2-HYDROXY-1-NAPHTHALENYL) METHYLENE] AS AN ANALYTICAL REAGENT FOR THE EXTRACTIVE SPECTRO-PHOTOMETRIC DETERMINATION OF NICKEL (II)

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ABSTRACT

The hydrazine carboxamide-2-[(2-hydroxy-1-naphthalenyl) methylene] is used as a reagent for extraction and spectrophotometric determination of Nickel (II). Nickel forms dark yellow colored complex, which can be quantitatively extracted in n-Butanol as solvent at pH 6.2. Beer's law is obeyed in the range 3-50 μg giving a linear and reproducible graph under optimum conditions. The λ_{max} is observed to be 395 nm. The complex obtained is studied by Job's continuos variation method, mole ratio method and log-log method. The effect of pH as function of extraction, effect of solvents, effects of salting out agents, effect of reagent concentrations and interference study of foreign ions has been also studied. The molar absorptivity is found to be 0.4785 x 10^5 L mol^{-1} cm^{-1} and Sandell sensitivity is observed to be 0.0585 $\mu g/cm^2$. The method developed is successfully applied to various commercial samples.

Key words: HCHNM, Nickel, Spectrophotometric determination.

INTRODUCTION

The significance of nickel as a transition metal lies in its wide spectrum of applications covering many frontier areas of study, particularly in industrial and consumer products. Even though nickel is not considered to be as toxic as most of the heavy metals, it is an equally harmful element. Hence, owing to the significance of nickel, it's determination from associated elements by extractive spectrophotometry has been of considerable importance.

A wide variety of reagent has been reported for the spectrophotometric determination

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of nickel. However, these methods suffer from limitations 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] etc. A method, far superior in sensitivity and selectivity to those reported in the literature, is developed for the extractive spectrophotometric determination of nickel with hydrazine carboxamide-2-[(2-hydroxy-1-naphthalenyl) methylene] (HCHNM). A close literature survey indicates that HCHNM has so far not been employed for either coordination or analytical studies. The proposed method is free from limitations.

EXPERIMENTAL

The absorption measurements were made on a Shimadzu UV-Visible 2100 Spectrophotometer with 1 cm quartz cells and standard buffer solutions and the digital pH meter Li-120 model of Elico Pvt. Ltd. was used for pH measurement study. The chemicals used were of analytical reagent grade. Stock solution of nickel was prepared by dissolving $NiSO_4.7H_2O$ in double distilled water and was standardized[8] by known method. The working solutions were prepared by appropriate dilution as required. The reagent was prepared as reported in the literature[8].

Procedure for 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 6.2 with 0.2 M potassium dihydrogen orthophosphate, 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 known method[8].

RESULTS AND DISCUSSION

The reagent forms dark yellow coloured complex with Ni (II), which was extracted in organic phase and the results obtained are as follows.

Extraction as a function of pH

The extraction of nickel with hydrazine carboxamide-2-[(2-hydroxy-1-naphthalenyl) methylene] has been studied over the pH range 1-10 and was observed that percentage extraction of Ni (II) is maximum at pH 6.2.

Absorption spectrum

The absorption spectrum of Ni (II): Hydrazine carboxamide-2-[(2-hydroxy-1-naphthalenyl) methylene] 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, ethyl methyl ketone, diethyl ether, toluene, n-butanol, carbon tetrachloride, MIBK, nitrobenzene, etc. The extraction of nickel (II) was quantitative with HCHNM 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 nitrate salts of alkali and alkaline metals did not show any effect over the absorbance value of Ni (II): Hydrazine carboxamide-2-[(2-hydroxy-1-naphthalenyl) methylene] complex extract.

Effect of reagent concentration

Various volumes of 0.1% reagent solution were added to the sample solution containing 100 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

The change in absorbance with variation in equilibrium time for extraction of Ni (II) shows that equilibrium time of 50 sec. are sufficient for quantitative extraction of nickel.

Stability of the complex with time

The study of stability of colour of the Ni (II): Hydrazine carboxamide-2-[(2-hydroxy-1-naphthalenyl) methylene] complex with respect to time shows that the absorbance due to extracted species is stable up to 50.0 hrs, 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 concentration of Ni (II) gives linear and reproducible graph in the concentration range 0.3 to 5 ppm of nickel indicating that the Beer's law is obeyed in this range. Fig. 1, The molar absorptivity and Sandell sensitivity were calculated to be is 0.4785 x 10^4 L $mol^{-1}cm^{-1}$ and 0.0585 μg cm^{-2}.

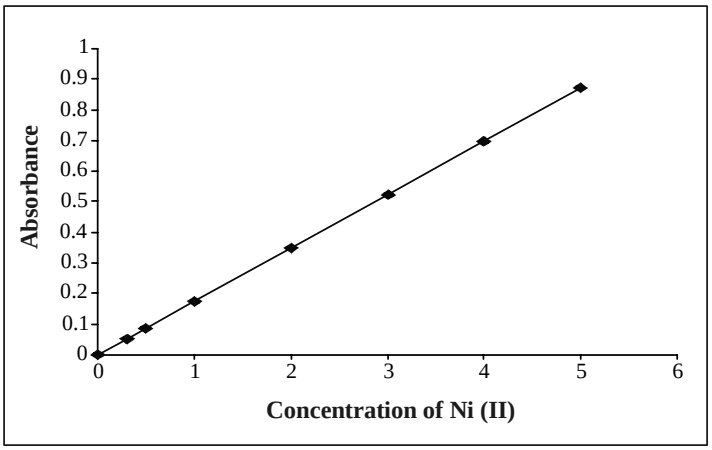

Fig. 1: Calibration plot of Ni (II) with HCHNM

Nature of extracted species

The composition of extracted species has been determined by Job's continuous variation method Fig. 2, slope ratio method Fig. 3 and mole ratio method. It shows that the composition of Ni (II): HCHNM complex is 1 : 2

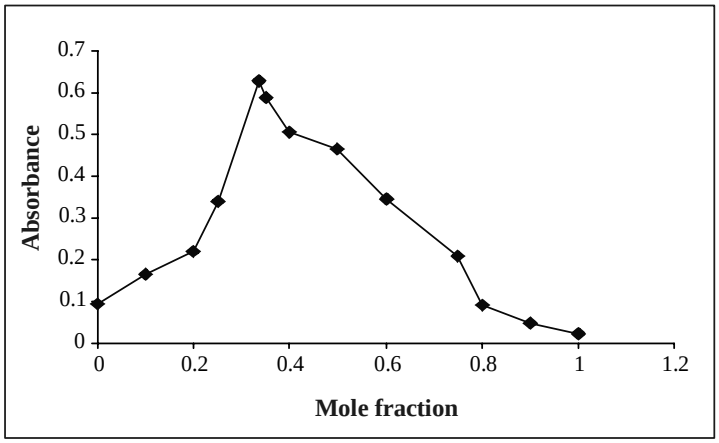

Fig. 2: Job's continuous variation method

Effect of divalent ions and foreign ions

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 30 µg of Nickel Table 1.

Table 1: Effect of the interference of some cations on absorbance of Ni (II): HCHNM complex in n-butanol

S. No.	Metal	Amount added in mg	Absorbance at 395 nm
1	--	--	0.522
2	Na (I)	11	0.522
3	K (I)	15	0.522
4	Ca (II)	7	0.522
5	Sr (II)	12	0.522
6	Mo (II)	6	0.522
7	Mn (II)	8	0.522
8	Mg (II)	13	0.522
9	V (VI)	6	0.522
10	Rh (IV)	16	0.522
11	Tl (I)	10	0.522
12	Th (II)	8	0.522
13	Li (I)	9	0.522
14	Be (II)	5	0.522
15	Zr (II)	8	0.522
16	Bi (III)	9	0.522
17	Al (III)	8	0.522
18	Hg (II)	9	0.522
19	Ce (III)	15 µg	0.522
20	Cd (II)	15 µg	0.522
21	Zn (II)	15 µg	0.522

Table 2: Determination of Ni (II) using HCHNM from different samples

S. No.	Sample	Amount of Ni (II)	
		Standard method	**Present method**
1.	**Vegetable oil**	0.0014%	0.0013%
2.	Alloys		
	1) cupro-nickel	65.0%	64.97%
	2) Steel	30.1%	30.07%
	3) Nickel cast iron	11.5%	11.48%
	4) Nichrome	23.3%	23.29%
3.	Synthetic mixtures		
	1) Ni (10) + Cu (10)	9.99 ppm	9.98 ppm
	2) Ni (10) + V (10)	9.98 ppm	9.97ppm
	3) Ni (10) + Zn (10)	10.0 ppm	9.99 ppm
Every result is an average of three independent determinations			

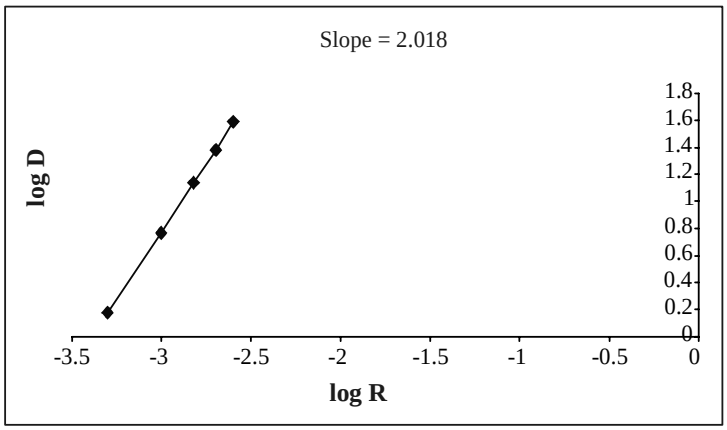

Fig. 3: Slope ratio method for Ni (II): HCHNM complex

Precision and accuracy

The precision and accuracy of the spectrophotometric method have been studied by analyzing five solutions each containing 40 µg of Nickel. Aliquot used is 40 µg/mL.

Standard deviation is 0.1480

Confidence limit at 99% is 39.98 ± 0.3046

Applications

The newly developed method has been successfully applied for the determination of Nickel from various alloys, ores and pharmaceutical samples. The results indicate that the developed method is compatible with the standard known method Table 2.

CONCLUSION

The results obtained show that hydrazine carboxamide-2-[(2-hydroxy-1-naphthalenyl) methylene] in n-butanol can be effectively used for quantitative extraction of Ni (II) from aqueous media. The proposed method is quick and requires less amount of organic solution. The equilibrium time required is very less and the complex is stable for 50 hours. The results show good agreement with the standard method. The method is very fast, accurate and precise.

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