

Spectrophotometric Determination of Nickel(II) Using 5-Bromo Salicylaldoxime as an Analytical Reagent

R.S. LOKHANDE* and LEENA M. KHADKE

*Department of Chemistry, University of Mumbai, Vidyana-gari,
Santacruz (East), Mumbai-400 098
E-mail: lokhanders@yahoo.com*

5-Bromo salicylaldoxime is proposed as a new sensitive, analytical reagent for the separation and extractive spectrophotometric determination of trace amounts of nickel. The reagent reacts with nickel(II) to produce greenish coloured complex. The complex can be extracted in ethyl acetate at pH 10.2 at 390 nm. Beer's law is obeyed over the range of 10–90 µg. The molar absorptivity and Sandell sensitivity are 0.428×10^3 L mol⁻¹ cm⁻¹ and 0.7×10^{-5} µg cm⁻², respectively. The stoichiometric ratio of the complex was found to be 1 : 2. The method has been successfully applied in the analysis of commercial and synthetic samples.

Key Words: 5-Bromo salicylaldoxime, Nickel(II), Spectrophotometric determination.

INTRODUCTION

Many organic reagents such as oximes^{1–5}, semicarbazones and thiosemicarbazones^{6–8} that have been used for the extractive spectrophotometric determination of transition metal ions.

The literature survey reveals that a wide variety of reagents have been employed for its spectrophotometric determination but most of them suffer from limitations such as heating for more time for colour development, critical pH, more time for equilibrium and the stability of the complex formed.

In the present investigation, 5-bromo salicylaldoxime (5-BSO) has been explored as a new reagent for the separation and spectrophotometric determination of nickel. 5-BSO reacts with nickel to give a greenish coloured complex which is extracted in ethyl acetate at 390 nm.

EXPERIMENTAL

The stock solution of nickel was prepared by dissolving weighed quantity of nickel sulphate with double distilled water containing dilute sulphuric acid and diluted to the desired volume with distilled water. A Shimadzu UV-Visible 2100 spectrophotometer with 1 cm quartz cell was used for all absorbance measurements. pH measurements were carried out by using buffer solution and an ELICO Li 120 model pH-meter was used.

The ligand 5-bromo salicylaldoxime (5-BSO) was synthesized as described in the literature⁹.

Extraction procedure

Extraction experiments were carried out by shaking the appropriate organic and

aqueous solution at an organic/aqueous phase ratio 1 : 1 for 10 min. The distribution study¹⁰ was carried out at 25°C in various organic solvents and it was observed that ethyl acetate gives maximum extraction of the Ni-BSO complex. The measured amount of Ni(II) was taken in a 50 mL beaker and to this 2 mL of 5-BSO was added and stirred for 2 min. The green coloured complex formed was extracted twice with 5 mL of ethyl acetate using a separating funnel. The complex was then transferred to a 10 mL volumetric flask. The combined extract was diluted to 10 mL (if required) and its absorbance was measured at 390 nm against the reagent blank solution prepared under the same conditions using the same quantity of the reagent.

RESULTS AND DISCUSSION

Effect of reagent concentration: Various volumes of 0.4% reagent solution were added to the sample solution containing 30 µg of nickel at the respective pH values. The absorbances remained constant when the volume of the reagent solution used was more than 0.8 mL (Table-1). Therefore, 0.8 mL of 0.4% reagent was chosen for the quantitative determination of nickel.

TABLE-1
EFFECT OF REAGENT CONCENTRATION ON THE
ABSORBANCE OF Ni(II) IN ETHYL ACETATE

Sr. No.	5-BSO (mL)	Absorbance
1	0.1	0.052
2	0.2	0.104
3	0.3	0.156
4	0.4	0.208
5	0.5	0.260
6	0.6	0.312
7	0.7	0.364
8	0.8	0.416
9	0.9	0.416
10	1.0	0.417
11	1.1	0.417
12	1.2	0.419

Total amount of nickel taken: 30 µg

Aqueous phase: 10.0 mL containing 1.0 mL of 0.4% 5-BSO in ethanol

Organic phase: 10.0 mL (2 × 5 mL) of ethyl acetate

Wavelength: 390 nm; pH: 10.2

Effect of pH on the extraction of Ni(II): The percentage extraction of Ni(II) with 5-BSO increased from 9.0 and maximum extraction was observed at pH 10.2 and further the percentage extraction of the complex decreases.

Absorption spectrum: The absorption spectrum of Ni(II): 5-BSO in ethyl acetate shows an intense absorption peak at 390 nm. Hence, the absorption measurements were taken at this wavelength using a reagent blank.

Nature of extracted species: The nature of extracted species was ascertained from the plot of $\log D$ vs. $\log R$. The slope of the graph is 2.0674 for 5-BSO. Again Job's continuous variation method, slope ratio method and mole ratio method show the formation of the complex having the composition Ni(II) : 5-BSO complex is 1 : 2.

Beer's law and sensitivity: Calibration plot for nickel(II) was constructed under the optimum conditions. The graph obeys Beer's law in the range of 10–90 μg for nickel. The molar absorptivity and Sandell sensitivity were calculated to be $0.428 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$ and $0.7 \times 10^{-5} \mu\text{g cm}^{-2}$, respectively as shown in Fig. 1.

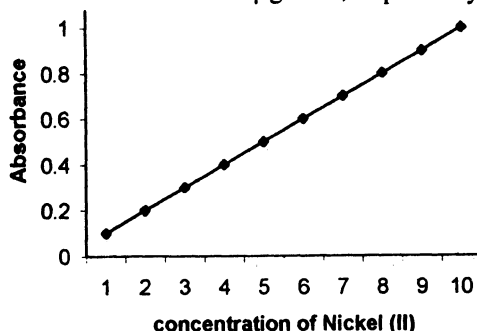


Fig. 1. Calibration Plot of Ni(II) with 5-BSO

Effect of solvent: Nickel(II) was extracted with 5-BSO in different solvents like ethyl acetate, ethyl methyl ketone, *n*-butanol, chloroform, diethyl ether, toluene, carbon tetrachloride (Table-2). The extraction was found to be quantitative in ethyl acetate which was used throughout the work.

Effect of equilibrium time: The study of changes in absorbance with variation in equilibrium time for the extraction of nickel shows that the equilibrium time of 1 min is sufficient for the quantitative extraction of nickel(II).

TABLE-2
EXTRACTION OF Ni(II) WITH 5-BROMO SALICYLALDOXIME INTO
VARIOUS SOLVENTS

Sr. No.	Solvent	% Extraction	Absorbance
1.	Chloroform	25.00	0.333
2.	Diethyl ether	0.000	0.000
3.	<i>n</i> -Butanol	85.00	5.666
4.	Toluene	0.000	0.000
5.	Carbon tetrachloride	0.000	0.000
6.	Ethyl methyl ketone	92.00	11.5
7.	Ethyl acetate	99.90	999
8.	Hexane	0.000	0.000
9.	Methylisobutylketone	95.00	19.0
10.	Nitrobenzene	0.000	0.000

Total amount of nickel taken : 30.0 μg

Aqueous phase : 10.0 mL containing 1.0 mL of 0.4% 5-BSO in ethanol

Wavelength : 390 nm; pH : 10.2

Applications

The proposed method has been successfully applied for the determination of nickel(II) from commercial samples and synthetic binary mixtures. The results are shown in Table-3.

TABLE-3
ESTIMATION OF Ni(II) WITH 5-BSO FROM VARIOUS SYNTHETIC AND COMMERCIAL SAMPLES

Sr. No.	Sample	Nickel found	
		DMG method	Present method
1.	Synthetic mixture		
	(a) Ni (50), Ag (50), Hg (50)	49.97 μg	49.98 μg
	(b) Ni (5), Zr (5)	4.99 μg	5.01 μg
2.	Monel nickel alloy 400 (Ita Lab, Mumbai)	18.45%	13.51%
3.	Steel alloy	3.04%	3.02%
4.	Brass alloy	0.54%	0.58%

*Average of three determinations.

Conclusion

The proposed method is more selective than the reported methods for the spectrophotometric determination of microgram amounts of nickel. The method is very precise, simpler and faster. The results show good agreement with the known standard method. The equilibrium time required is very little, *i.e.*, only 1 min.

REFERENCES

1. U. Muralikrishna and A. Sivaramakrishna, *Asian J. Chem.*, **13**, 289 (2001).
2. A.B. Bhatt and K.K. Desai, *J. Inst. Chemists (India)*, **61**, 5 (1989).
3. D. Vijaykumar, R.C. Hussain and N. Appalaraja, *J. Indian Chem. Soc.*, **67**, 786 (1980).
4. K.K. Desai and H.B. Naik, *Indian J. Chem.*, **25A**, 297 (1980).
5. B.D. Desai and K.K. Desai, *Asian J. Chem.*, **13**, 366 (2001).
6. M.V. Naik and N.V. Thakkar, *Indian J. Chem.*, **34A**, 410 (1995).
7. B.J. Desai and V.M. Shinde, *Microchim. Acta*, **10**, 93 (1993).
8. R.S. Lokhande, S.V. Poman and U.R. Kapadi, *Asian J. Chem.*, **13**, 1223 (2001).
9. S.K. Sindhvani, Y. Dutt and R.P. Singh, *Indian J. Chem.*, **12**, 110 (1973).
10. A.K. De, S.M. Khopkar and R.A. Chalmers, *Solvent Extraction of Metals*, Van Nostrand Reinhold Co., London (1970).

(Received: 5 October, 2004; Accepted: 15 June 2005)

AJC-4256