**INTERNATIONAL JOURNAL
OF
PHARMACEUTICAL SCIENCES
AND
RESEARCH**

A RAPID SPECTROPHOTOMETRIC DETERMINATION OF NI(II) IN MEDICINAL PLANT MATERIALS AND WATER SAMPLES USING N',N'''-((1E,1'E)-(PROPANE-1,3-DIYLBIS (SULFANEDIYL)) BIS(1-(4-BROMOPHENYL) ETHAN-2-YL-1-YLIDENE)) BIS(2-HYDROXY-BENZOHYDRAZIDE) AS A SELECTIVE AND SENSITIVE ANALYTICAL REAGENT

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ABSTRACT: N',N'''-((1E,1'E)-(propane-1,3-diylbis (sulfanediyl)) bis (1-(4-bromophenyl) ethan-2- yl-1-ylidene)) bis (2-hydroxybenzohydrazide) (BAPSSHZ) is found to be a selective and sensitive analytical reagent for the determination of trace levels of nickel (II) in environmental samples like medicinal plant materials and water samples (potable and polluted) by spectro-photometry. BAPSSHZ reacts with Ni (II) at pH 5.0 in aqueous DMF to form a yellow-colored (Ni-BAPSSHZ) complex that shows maximum absorbance (λ_{max}) at 343 nm. Immediate color reaction and the absorbance remain constant for about 48 h. The molar absorptivity coefficient and Sandell's sensitivity are found to be 7.21×10^4 L.mol^{-1} cm^{-1} and 0.064 µg cm^{-2} of Ni(II), respectively. The system obeys Beer`s law in the range of 2.37-23.71 µg mL^{-1} of Ni (II). The linear regression coefficient is 0.9991, and the standard deviation is 0.01086. A large number of cations, anions, and complexing agents do not interfere in this method. The developed method is applied for the determination of Ni(II) in medicinal plant materials and water samples (potable and polluted).

INTRODUCTION: Nickel is an essential trace metal in animals, microorganisms, and plants [1]. It is an important constituent in the enzyme urease which is used for the hydrolysis of urea in plant cells [2, 3]. Nickel has numerous industrial and commercial applications, and the development of industrialization leads to increased emission of pollutants into ecosystems [4, 5]. Generally, people may be exposed to nickel through the air, water, and food [6, 7]. Inhalation is an important route for nickel-induced toxicity and may cause toxic effects in the respiratory tract and immune system [8].

Hence, there is a great need to develop a simple, sensitive, selective, and cost-effective method for the determination of Ni(II) in environmental matrices.

In this point of view, several methods have been developed for the determination of Ni(II) at trace levels using various analytical techniques such as ICP-MS, ICP-OES, X-ray fluorescence, Spectro-photometry, Spectro fluorometry, AAS, and other techniques. Among these, the spectrophotometric methods are preferred as they are cheaper, suitable for automation, and have comparable sensitivity. The extensive literature survey reveals that a variety of hydrazone derivatives are used as spectro-photometric reagents for the determination of Ni (II) in various environmental matrices **Table 1** [9-21]. However, the reported spectrophotometric methods suffer from one or more disadvantages such as severe interferences, less sensitivity, and less selectivity, *etc.*

To the best of my knowledge, none has been reported on spectrophotometric determination of Ni (II) in medicinal plant materials and water samples using N', N'''-((1E, 1'E)-(propane-1, 3-diylbis (sulfanediyl)) bis(1- (4-bromophenyl) ethan-2-yl-1-ylidene)) bis (2-hydroxybenzohydrazide) as a sensitive analytical reagent.

Herein, we report a rapid spectrophotometric method for the determination of Ni(II) in medicinal plant materials and water samples by using N',N'''-((1E, 1'E)-(propane-1, 3-diylbis (sulfanediyl)) bis (1-(4-bromophenyl) ethan-2- yl-1-ylidene)) bis(2-hydroxybenzohydrazide) as a selective and sensitive ligand.

TABLE 1: COMPARISON OF THE PRESENT METHOD WITH OTHER REPORTED SPECTROPHOTOMETRIC METHODS

| Name of the reagent	λ_{max} (nm)	pH	Beer's Law range (ppm)	Molar absorptivity (ε) (L mol^{-1}cm^{-1})	Ref.
Di-2-pyridyl methanone-1- pthalazinyl hydrazone	500	4.3-10.7	0.1-1.0	5.4×10^4	9
Anthrone phenyl hydrazone(APH)	426	10	-	2.0×10^4	10
3,5-Dimethoxy-4-hydroxy benzaldehyde isonicotinoyl hydrazone(DMHBIH)	386	9.0	0.234-2.94	1.22×10^4	11
3-methoxysalcilaldehyde-4-hydroxybenzoylhydrazone (MSHBH)	425	5.5	0.117-2.94	1.27×10^4	12
2-aminoacetophenone Isonicotinoylhydrazone	470	9.0	0.29-6.16	1.05×10^4	13
5-Bromo-2- hydroxyl -3-methoxy benzaldehyde-4-hydroxy benzoichydrazone	440	6.0	0.117 - 2.64	2.013×10^4	14
2,4-Dimethoxybenzaldehyde Isonicotinoyl hydrazone (DMBIH)	410	8.5-9.5	0.1467-1.760	5.92×10^4	15
Diacetylmonoxime isonicotinoyl hydrazone	431	8.5	0.23-1.88	1.75×10^4	16
Diacetylmonoxime benzoyl hydrazone	362	9.0	0.12-2.58	2.13×10^4	17
4-Hydroxy-3, 5-dimethoxy benzaldehyde-4-hydroxy benzoylhydrazone	450	9.0	0.117-0.528	3.2×10^4	18
4-Hydroxy 3,5 dimethoxy benzaldehyde 4-hydroxy benzoyl hydrazone	408	8.0-9.0	0.058-0.586	3.22×10^4	19
Thiazole-2-carbaldehyde 2-quinolyl hydrazone	522	8.7-9.5	-	7.17×10^4	20
(E)-N^1-(2-hydroxy-5-nitrobenzylidene) isonicotinoylhydrazone (HNBISNH)	480	4.0	0.81-19.7	5.1×10^4	21
N',N'''-((1E,1'E)-(propane-1, 3-diylbis (sulfanediyl))bis(1-(4-bromophenyl)ethan-2-yl-1-ylidene))bis(2-hydroxybenzohydrazide) (BAPSSHZ)	343	5.0	2.37-23.71	7.21×10^4	PM

MATERIALS AND METHODS: A Double beam UV-Visible spectrophotometer (Shimadzu model UV-2203) with a 1.0 cm quartz cell is used for absorbance studies, and a pH meter (Systronics model 3305) is used for the measurement of pH.

The melting point is determined and is uncorrected. For comparative analysis, atomic absorption spectrophotometer (Shimadzu model No: AA-6300) is used. 1H NMR spectrum of the ligand is recorded on Jeol 400 MHz NMR Spectrometer (JNM-400), and Mass spectrum of the ligand is recorded on Shimadzu-LCMS with ESI probe (LC-2010EV).

All chemicals and solvents are purchased from Sigma Aldrich, Acros Organics Ltd., and Avra Chemicals Ltd., and are used as received. Millipore double distilled water is used throughout the analysis.

Preparation of N', N'''-((1E,1'E)-(propane-1, 3-diylbis (sulfanediyl)) bis (1-(4- bromophenyl) ethan-2 -yl-1-ylidene)) bis (2-hydroxybenzo-hydrazide) (BAPSSHZ): 2, 2'-(propane-1, 3-diylbis (sulfanediyl)) bis(1-(4-bromophenyl) ethan-1-one) (I) (4.8 mmol) and salicylic acid hydrazide (II) (9.6 mmol) and 20 mL isopropanol are taken into a 100-mL two necked RB flask.

The reaction mixture is stirred at reflux temperature for 3.0 h. The progress of the reaction is monitored by TLC. After completion of the reaction, the solvent is removed under reduced pressure. The formed product (III) is washed with a mixture of water and ethyl acetate, filtered, and dried. The obtained crude product is recrystallized from aq. ethanol and dried. The structure of the synthesized compound is confirmed by 1H NMR and Mass spectral data.
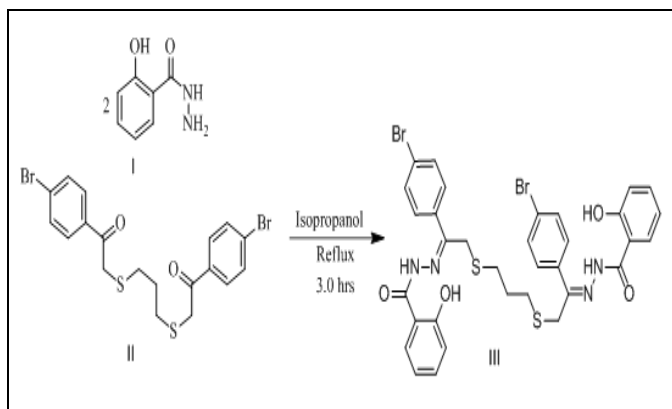
SCHEME 1: PREPARATION OF N', N'''-((1E, 1'E)-(PROPANE-1, 3-DIYLBIS (SULFANEDIYL)) BIS(1-(4-BROMOPHENYL) ETHAN-2-YL-1-YLIDENE)) BIS(2-HYDROXYBENZOHYDRAZIDE)

Physical and Spectral data of N', N'''-((1E, 1'E)-(propane-1, 3-diylbis (sulfanediyl)) bis (1-(4-bromophenyl) ethan-2-yl-1-ylidene)) bis(2-hydroxybenzohydrazide): Colour: yellow; yield: 90 %; mp: 250 °C; 1H NMR (400 MHz, DMSO-d6): δ 11.62 (s, 2H, arom-OH), 11.50 (s, 2H, NH), 8.10(d, 2H, J= 7.2 Hz, arom H), 7.90(d, 4H, J=8.0 Hz, arom H), 7.51 (d, 4H, J=8.0 Hz, arom H), 7.45 (t, 2H, J=7.2 Hz, arom H), 7.0 (d, 2H, J=7.6 Hz, arom H), 6.90 (t, 2H, J=7.6 Hz, arom H), 3.90 (s, 4H, 2 - N=C-CH$_2$-S), 2.60 (t, 4H, J=7.2 Hz, 2-S-CH$_2$-), 1.90 (quint, 2H, J = 6.8 Hz, -CH$_2$-CH$_2$-CH$_2$); MS (ESI): (M+H)+ 771.

Preparation of 0.01 M N', N'''-((1E, 1'E)-(propane-1, 3-diylbis (sulfanediyl)) bis (1-(4-bromophenyl) ethan-2-yl-1-ylidene)) bis (2-hydroxybenzohydrazide): 770 mg of reagent is dissolved in 100-mL standard flask with DMF and made up to the mark. Further required dilutions are prepared from this solution.

Preparation of Ni(II) Stock Solution, Tartrate Solution and Buffer Solutions: 0.01M Ni (II) solution, (0.1% w/v) tartrate solution, and pH 1.0-7.0 buffer solutions are prepared as per the standard procedure [22].

General Analytical Procedure for the Determination of Nickel (II): An aliquot (1.0 mL) of the solution containing a known amount of Nickel (II), 4.0 mL of ammonium acetate buffer solution (pH 5.0) and 1.0 mL of BAPSSHZ reagent of required concentration are mixed in a 10-mL volumetric flask and the resulting solution is diluted up to the mark with double distilled water.

The absorbance of this solution is measured at 343 nm, against ligand blank.

RESULTS AND DISCUSSION: The ligand reacts with nickel (II) forms the yellow-colored complex in aqueous DMF at pH 5.0. The Ni-BAPSSHZ complex shows maximum absorbance (λ_{max}) at 343 nm against the reagent blank. Hence, a detailed study has been undertaken for the determination of Ni (II) using BAPSHZ by spectro-photometric method.

Absorption Spectra of Ligand, BAPSSHZ and Ni (II)-BAPSSHZ Complex (MLC): The absorption spectrum of the BAPSSHZ is recorded against the solvent blank initially. The absorption spectrum of Ni (II)-BAPSSHZ complex is recorded against the reagent blank.

The absorption spectrum of both complex and reagent are shown in **Fig. 1**. From the absorption spectra, it is clear that the reagent shows maximum absorption at 315 nm whereas the complex shows maximum absorption at 343 nm. Therefore, all the spectral measurements are carried out at 343 nm.
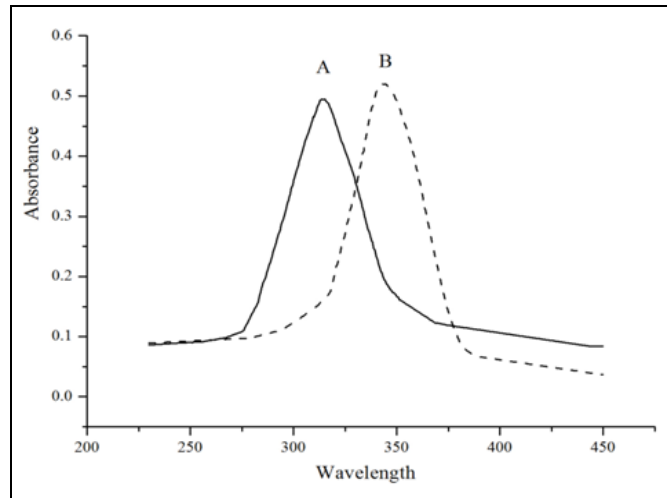
FIG. 1: (A) ABSORPTION SPECTRUM OF REAGENT (BAPSSHZ) *vs.* **SOLVENT (DMF) BLANK (B) ABSORPTION SPECTRUM OF NI(II)-BAPSSHZ COMPLEX (MLC)** *vs.* **REAGENT BLANK; NI(II) =1.0 ML OF 1.0 × 10^{-4} M, BAPSSHZ = 1.0 mL OF 1.0 × 10^{-4} M AND pH = 5.0**

Effect of pH: The absorbance of the M-L complex increases as the pH is increased from pH 1.0 to 5.0, and after that, it decreases **Fig. 2**. Therefore, all the experiments are carried out at pH 5.0 and considering it as the optimum pH.
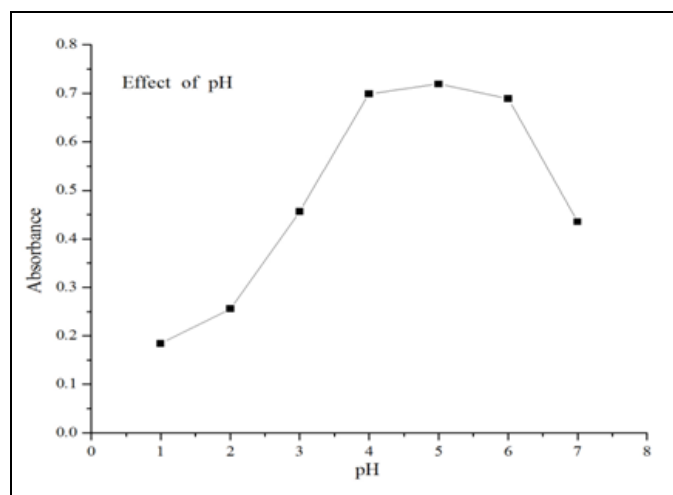
FIG. 2: EFFECT OF PH ON THE ABSORBANCE OF NI (II)-BAPSSHZ COMPLEX; NI (II) = 1.0 ML OF 1.0×10^{-4} M; BAPSSHZ =1.0 ML OF 1.0×10^{-4} M; Λ_{MAX} = 343 NM

Applicability of Beer's Law: Beer's law is obeyed in the range of 2.37-23.71 μg mL^{-1}, with a correlation coefficient of 0.9991, which indicates the linearity between the two variables **Fig. 3**.

The molar absorptive and Sandell's sensitivity of the complex are found to be 7.21×10^4 L mol^{-1} cm^{-1} and 0.064 μg cm^{-2}, respectively.
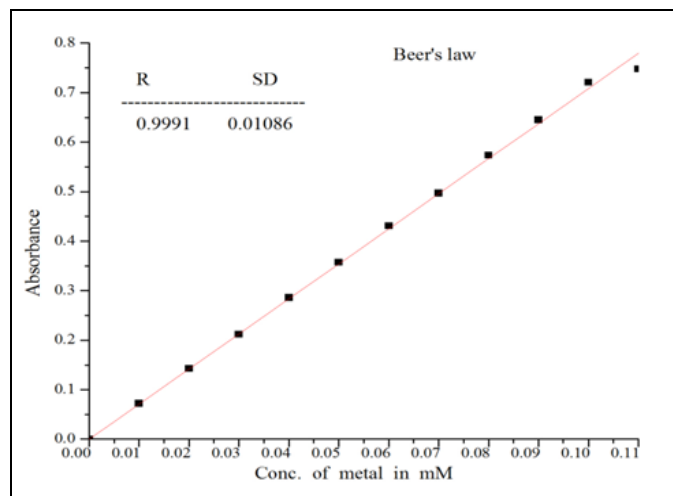

FIG. 3: APPLICABILITY OF BEER'S LAW: BAPSSHZ CONC. 1.0×10^{-4} M AND LIGAND CONC. 0.10×10^{-4} M TO 1.0×10^{-4} M; PH = 5.0 AND Λ_{MAX} = 343 NM

Composition of the Metal-ligand (M-L) Complex: The composition of the M-L complex has been determined by Job's method of continuous variation **Fig. 4** and mole ratio method **Fig. 5** & **6**.

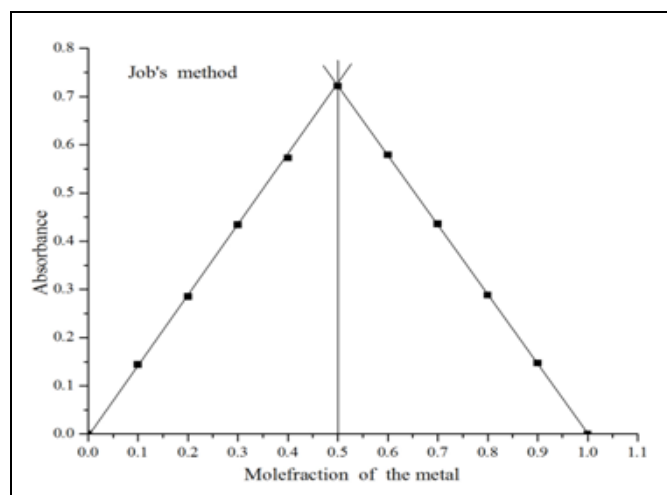Based on the above two methods, the composition of the Ni(II)-BAPSSHZ complex is confirmed as 1:1 ratio.

FIG. 4: JOBS METHOD OF CONTINUOUS VARIATIONS FOR STOICHIOMETRIC RATIO BETWEEN NI(II) AND BAPSSHZ (1.0×10^{-4} M CONCENTRATION EACH); PH = 5.0 AND Λ_{MAX} = 343 NM
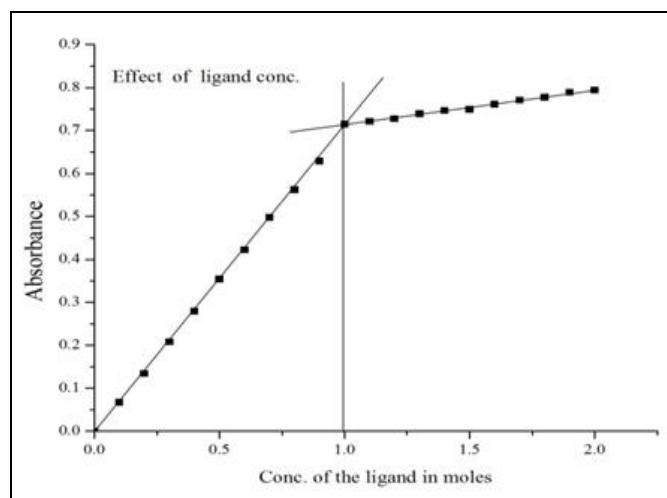

FIG. 5: EFFECT OF LIGAND CONCENTRATION: BAPSSHZ =1.0 MOLE OF (0.1×10^{-4} M-2.0×10^{-4} M, NI(II)=1.0 MOLE OF (1.0×10^{-4} M), PH = 5.0 AND Λ_{MAX} = 343 NM
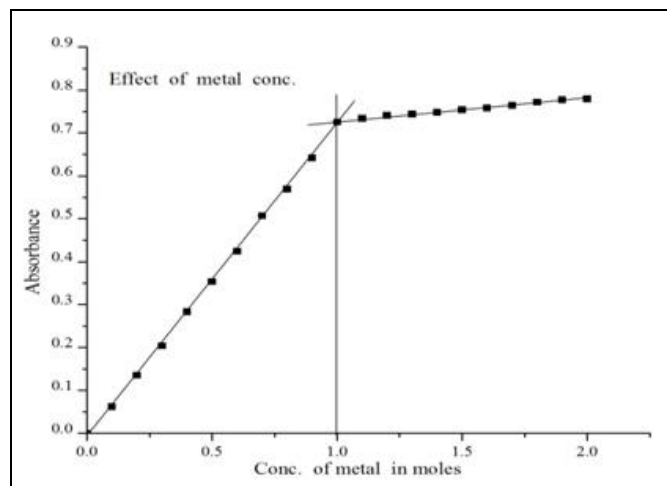

FIG. 6: EFFECT OF METAL CONCENTRATION: BAPSSHZ = 1.0MOLE OF (1.0×10^{-4} M), NI(II) = 1.0 MOLE OF (0.1×10^{-4} M-2.0×10^{-4} M), PH=5.0 AND Λ_{MAX}= 343 NM

Effect of Foreign Ions: The effect of diverse ions in the determination of Ni(II) has been studied by using 10 μg mL^{-1} of Ni(II) in solution, and various amounts of each diverse ion being discussed. The effect of foreign ions on complexation is studied by taking 1.0 mL of Ni(II) solution, 1.0 mL of required concentration of the foreign ion solution, 4.0 mL of ammonium acetate buffer (pH 5.0), and 1.0 mL of BAPSSHZ solution in a 10-mL standard flask. The total volume of the solution is brought to 10.0 mL with double distilled water. The experiment is repeated by changing the concentration of the diverse ions. The absorbance is measured at 343 nm. A change of ± 0.02 is taken as the tolerance limit for interference. The results indicated that Ca(II), Mg(II), Fe(II), and Mn(II) do not interfere even when present up to 5000 μg. Interference due to Al(III) and Cr(III) can be tolerated up to 4000 μg, whereas Mo(VI) and W(V) can be tolerated up to 2000 μg only. Determination of Ni (II) is not possible in the presence of Cu(II), Zn(II), Pb(II), Pd(II), and Cd(II) due to their severe interference, even when present in trace amount.

These cations are removed by using 2.0 mL of 0.5% thiosulphate solution as a masking agent.

Scope of the Method: The optimized method is successfully applied for the determination of nickel in medicinal plant materials and water samples either alone or in the presence of diverse ions.

Determination of Ni(II) in Medicinal Plant Materials: Medicinal plant materials are collected from various villages around Kadapa, A.P. India. The samples are cleaned and dried in the open air. The dried sample is pulverized in a mortar for the purpose of analysis to a convenient size. One gram of powdered plant materials is taken in a 100-mL beaker and digested with 10.0 mL of concentrated nitric acid and hydrochloric acid mixture. Further, added few drops of 1:2 HCl solutions and then filtered by using Whatman no. 42 filter paper. The filtrate is taken in a 10-mL standard flask, made up to the mark with double distilled water, and analyzed as per the general procedure. The obtained results are shown in **Table 2**.

TABLE 2: DETERMINATION OF NI(II) IN MEDICINAL PLANT MATERIALS

S. no.	Scientific name	Conc. of Ni(II) by ICP-MS method (ppm)	Ref.	Conc. of Ni(II) by present method*(ppm)	SD	% of RSD
1	*Phyllanthus amarus*	13.48	23	13.18	0.21	1.59
2	*Tinosporia cardifolia*	8.13	24	8.01	0.08	1.05
3	*Eclipta alba*	11.00	24	10.74	0.18	1.69
4	Satavari root	12.27	25	12.11	0.11	0.93
5	Amla	8.65	25	8.35	0.21	2.50
6	Flax seed	9.01	26	8.65	0.25	2.88

*Average of five readings

Determination of Ni(II) in Water Samples: Different water samples are collected from in and around Kadapa, A.P. India. Each filtered water sample is evaporated nearly to dryness with a mixture of 5.0 mL of concentrated HCl and 10.0 mL of concentrated HNO$_3$ in a fume cupboard and then cooled to room temperature. The solution is cooled and neutralized with dilute NH4OH in the presence of 1.0-2.0 mL of 0.01% (w/v) tartarate solution. The resulting solution is filtered and quantitatively transferred into a 25-mL calibrated flask and made up to the mark with double distilled water and analyzed as per the general procedure [27]. The results obtained are shown in **Table 3**.

TABLE 3: DETERMINATION OF NI(II) IN WATER SAMPLES

S. no.	Sample	Ni(II) Spiked (ppm)	Conc. of Ni(II) by AAS method (ppm)	Conc. of Ni(II) by present method* (ppm)	SD	% of RSD
1	[1]Water sample-1	100	108.23	108.02	0.15	0.14
2	[1]Water sample-2	100	109.11	108.54	0.40	0.37
3	[1]Water sample-3	100	104.56	104.24	0.23	0.22
4	[1]Water sample-4	100	110.02	109.65	0.26	0.24
5	[1]Water sample-5	100	108.69	108.28	0.29	0.27
6	[2]Water sample-6	100	101.06	100.59	0.33	0.33
7	[2]Water sample-7	100	100.56	100.21	0.25	0.25
8	[2]Water sample-8	100	100.98	100.28	0.49	0.49
9	[2]Water sample-9	100	101.23	100.85	0.27	0.27
10	[2]Water sample-10	100	102.59	102.02	0.40	0.39

[1]Collected from industrial area, Kadapa.; [2]Collected from residential area, Kadapa. *Average of five readings

CONCLUSION: A rapid spectrophotometric method for the determination of Ni(II) in medicinal plant materials and water samples has been developed by using a fairly sensitive and selective BAPSSHZ as an analytical reagent. The proposed method offers advantages like good sensitivity, selectivity, reliability, reproducibility, less inter-ference, and immediate color development.

The developed method is found to be a quantitative comparison to other standard methods. The molar absorptivity of the complex 7.21×10^4 L mol^{-1} cm^{-1} reveals that the ligand is highly sensitive for Ni (II) when compared with other hydrazones. Hence, BAPSSHZ is an alternative ligand for the spectro-photometric determination of trace amount of Ni(II) in various environmental matrices.

CONFLICTS OF INTEREST: The authors declared that there are no conflicts of interest.

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How to cite this article:
Reddy NCG: A rapid spectrophotometric determination of Ni (II) in medicinal plant materials and water samples using N', N'''-((1E,1'E)-(propane-1,3-diylbis (sulfanediyl))bis(1-(4-bromophenyl) ethan-2-yl-1-ylidene))bis(2-hydroxy benzohydrazide) as a selective and sensitive analytical reagent. Int J Pharm Sci & Res 2021; 12(2): 838-44. doi: 10.13040/IJPSR.0975-8232.12(2).838-44.