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Role of [2-((Z)-(4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl)benzenylidene)amino)phenol] (MThBAP) in Extractive Spectrophotometric Determination of Nickel (II)

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ABSTRACT : -

A rapid, sensitive, and straightforward spectrophotometric method using the MThBAP reagent has been developed to measure nickel at the trace level. Reagent was analysed using an elemental and spectral analytical technique. Ni (II) was quantitatively (99.64%) extracted from an aqueous solution with MThBAP and a pH range of 7.5 to 8.6 using the solvent n-amyl alcohol. The maximum peak of the N-amyl alcohol extract was seen at 450 and 650 nm. A concentration range of between 0.5 and 6 µg/ml for Ni (II) was declared. The Ni complex MThBAP's molar absorptivity and Sandell's sensitivity were calculated to be 118030.7 $Lmole^{-1}cm^{-1}$ and 0.0243 μgcm^{-2} , respectively. The Mole Ratio Method and Job's Continuous Variation (Ni: MThBAP) confirmed the complexity of the system. The suggested approach has been successfully applied for determination of Ni (II) in alloy and pharmaceutical samples.

KEYWORDS:- Solvent Extraction, Extractive Spectrophotometry, Nickel (II), Alloy sample, Pharmaceutical samples.

I. INTRODUCTION

Ni is a transition element and has an atomic number of 28. Nickel is more common in "Victuals" since Earthlings have generated more pollution. Human skin can be exposed to nickel through jewellery, coins, tobacco, and cleaning supplies. Although nickel can be removed from the body through urine, it is extremely poisonous and has been linked to cancer when consumed in large quantities. Solvent extraction is an important part of the separation process. Due to its ease of use and rapidity, it is used in the separation of metal ions at the trace level (Anovski T. et.al. 1972,Monior –Williams GW 1949). Pharmaceutical science relies heavily on solvent extraction in conjunction with a spectrophotometer(De AK et.al.1970). Schiff bases are crucial chelating agents in complexes of transition meta(Temel H et.al 2002). Spectrophotometry can be used to measure Nickel (II) using a number of reagents(Feig F 1949, Gili.P et.al 1997, Liu.J et.al 2006, Aliyu H.N et.al 2009, Makhijani RM et.al 2018,). In the present communication, we describe the extractive spectrophotometric determination of Ni(II) using

[2-((Z)-(4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl)benzenylidene)amino)phenol] (MThBAP)]

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II. EXPERIMENTAL SECTION

ELICO - SL 159 spectrophotometer with optically matched quartz or glass cells of 1cm path length were used for absorbance measurement. An ELICO – LI 127 pH meter was employed for pH measurements.

General procedure of synthesis:-

2-((Z)-(4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl)benzenylidene)amino) phenol] :-

2-Aminothiazol (0.005moles) solution was diazotized by adding sodium nitrite solution (0.005moles) in increments of 2 ml at a time, and maintaining a temperature below 5°C for 30 min.Then, stirring until red crystals separated, the cold diazonium salt (0.005moles) was gradually added to the vanilline solution (25cm^3 of 10% sodium hydroxide). It was suction-filtered in a Buchner funnel after an hour. A saturated solution of sodium chloride was used to clean the object.

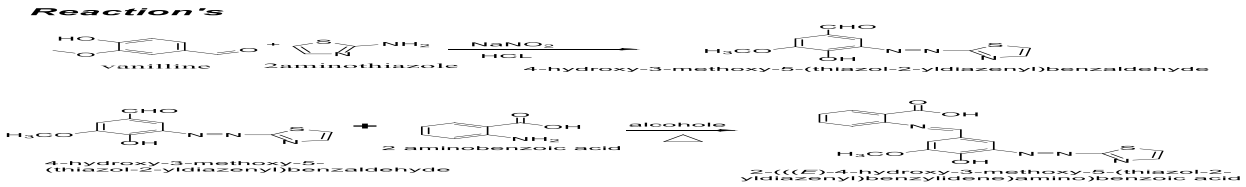

Figure.1:-synthesis of Azo Compound

Using 50 ml of ethyl alcohol as the solvent, 0.01 moles of the azo compound and 0.01 moles of 2-aminophenol are added to a flask with a circular bottom and a few shards of porcelain. It is recirculated for three hours while being connected to the water condenser. The combination is then put into a beaker and kept in the fridge for the remainder of the day. The final product underwent filtering and drying. Schiff's base compound 2-(((Z)-4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl)benzylidene)aminophenol (MThBAP) was created as brown crystals. The crystals have undergone regrowth..

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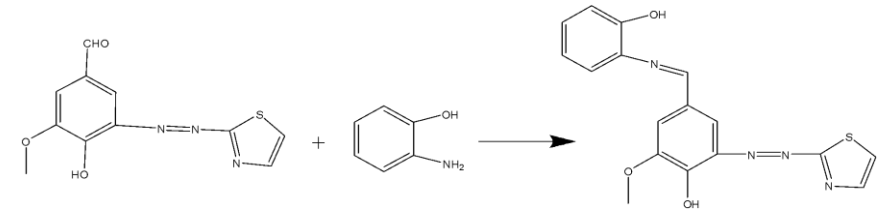

4-hydroxy-3-methoxy-5-thiazol-2-yldiazenyl benzaldehyde

2-aminophenol

[2-((Z)-(4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl)benzenylidene)amino)phenol]

Figure.2-Synthesis of Ligand MThBAP

Green synthesis of [2-((Z)-(4-hydroxy-3-methoxy-5-((E)-thiazol-5-yldiazenyl)benzenylidene)amino)phenol]

Microwave radiation was applied at 180 ^{0} C for 0.4 minutes to a beaker containing 0.005 moles of 2-aminophenol, 0.005 moles of azo compound, and a few drops of pure alcohol. In a short (2 min) reaction time, higher yields were attained. It is possible to create greenish-brown Schiffs base crystals. Elemental analysis & physical data are shown in table I (Vogel AI 1978)

Table-I The Analytical and Physical data of ligand

Compound (Colour)	Molecular Weight & melting point	Reaction period & %yield		% Elemental Analysis Found (Calculated)				
		Conventional methods	Micro synthesis	C	H	O	S	N
Ligand $C_{17}H_{14}N_4O_3S$ (greenish brown)	354.38 240^0C	4 hours 79%	0.4 Minutes 92%	57.00 (57.62)	3.90 (3.98)	13.50 (13.54)	9.08 (9.05)	15.78 (15.81)

Preparation of stock solution :

Nickel sulphate was dissolved in double-distilled water containing diluted sulphuric acid to create a stock solution of Ni (II), which was then standardized using the dimethylglyoxime technique.(AK.De et.al 1970) Ni (II) working solutions were created through the proper dilution.

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Extractive Spectrophotometric Determination of Ni (II)

2 mL of a buffer solution with a pH of 6.0, an aliquot of an aqueous solution containing 1-50 g of Ni (II), and 2 mL of a 1% solution of MThBAP prepared in DMF were also added. The volume of the solution was increased to 10 mL using distilled water. After the solution had been equilibrated for one minute with 10 mL of an organic solvent, the phases were then allowed to separate. In a 10 mL standard measuring flask, the organic solvent extract (n-amyl alcohol) was collected and, if necessary, brought up to the required concentration using n-amyl alcohol. The absorbance of an n-amyl alcohol extract was measured at 540 nm in comparison to a reagent blank produced under the identical conditions. Utilizing the measured absorbance, the amount of Ni (II) present in the sample solution from predetermined calibration curve

Determination of Nickel (II) in alloy sample- (Nickel-Aluminum based alloy):

In 10 ml of aqua regia, 0.1 to 0.2g of nickel sample were dissolved. When the solution was dry, the obtained residue was dissolved in 1N HCl. The filtrate after filtration was diluted up to 100 ml with distilled water. The solution's Ni(II) content was determined using a 1ml aliquot.(Amin AS 2012 , Basha S.K 2015)

III. RESULTS AND DISCUSSION:

Nickel (II) could be extracted quantitatively (99.64%) by MThBAP into n-amyl alcohol from an aqueous solution of pH range 7.5 -8.6

Based on the extraction coefficient values of the organic solvents used to extract Ni (II), they can be arranged as follows: n-amyl alcohol > n-Butyl alcohol > ethyl acetate > chloroform > carbon tetrachloride > chlorobenzene > bromobenzene > benzene > xylene

Since n-amyl alcohol is the best extracting solvent, it was employed throughout the entire project.

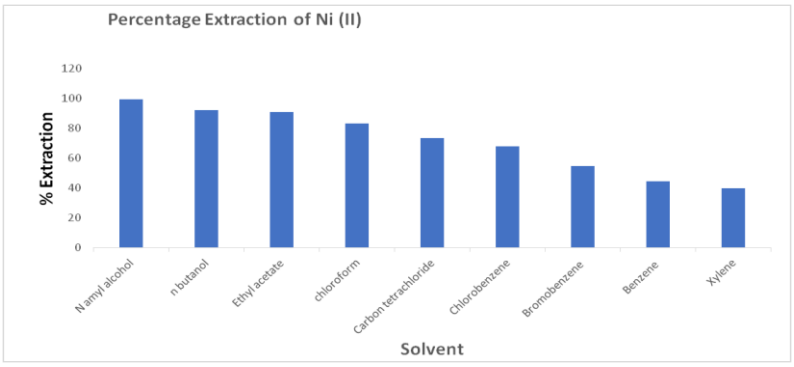

Figure 3:- Percentage extraction of Ni into various solvents

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(MThBAP) was successful in converting Ni(II) from an aqueous solution with a pH range of 7.5 to 8.6 into n-amyl alcohol.

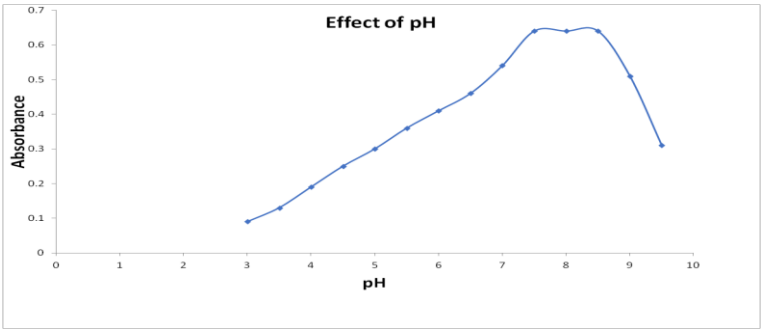

Figure 4:- Effect of pH

A calibration curve was established between absorbance reading and Ni complex concentration. Within the concentration range of 0.5 to 0.6 µg/ml, the calibration curve is linear.

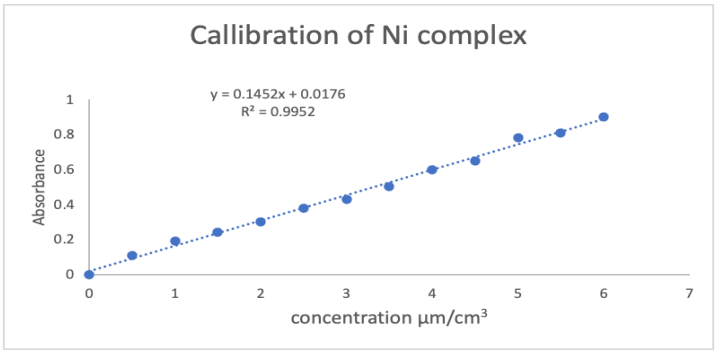

Figure 5 :- Calibration curve of Ni: MThBAP Complex

IV. EFFECT OF OTHER IONS

It was found that following ions did not interfere in the study of Ni(II)

10 mg each of Li(I), Ba(II), Hg(II), Sn(II), Sr(II), Zr(II), Ca(II), Zn(II), V(V), Mg(II), Cd(II) and 5 mg each of Th (IV), Ce (IV) and W (VI). 0.1mg each of Ru (III), Rh (III), Mo(IV) 20 mg each of sulphate, sulphide, nitrate, nitrite, chloride, bromide, iodide, fluoride, phosphate, citrate, triethanolamine, thiocyanate, acetate and 5-sulphosalicylic acid.Appropriate masking agent were used to remove the interference by the various ions shown in Table II

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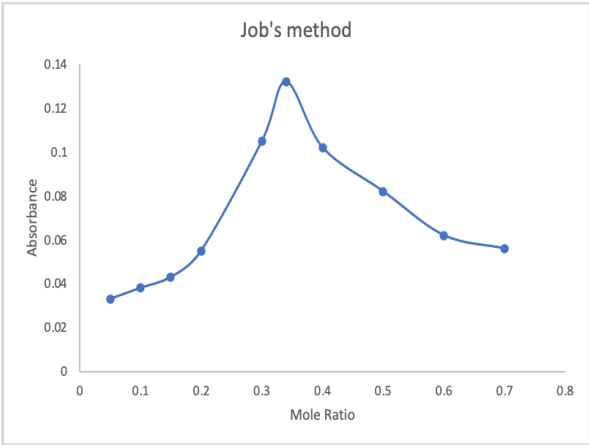
Table – II. Masking Agents

Sr No	Interfering ion	Amount added in mg	Masking agent added 1 ml of 0.5 M solution
1	Mn(II)	10	Potassium tartarrate
2	Ag(I) & Pd (II)	10	Potassium thiocyanante
3	Mn (II)	10	Thiocyanante
4	Fe(III) , Cr(III) & V(II)	10	Triethanol amine

V. COMPOSITION OF THE EXTRACTED COMPLEX

The composition of the extracted complex was found to be 1:2 (Ni: MThBAP) by Job's continuous variation and Mole ratio methods (Fig- 6 & Fig- 7).



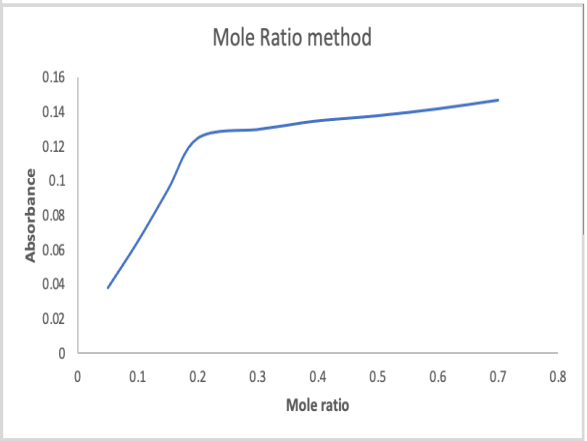


Figure 6. Composition of Complex by Job's Method Figure 7. Composition of Complex by Mole ratio Method

VI. Accuracy, Precision, Sensitivity and Applications of Method

Ten times of the experiment were conducted in order to assess the precision and accuracy of the suggested approach. 20 µg of Ni (II) in solutions of 10 cm3. The average of 10 measurements of 20 µg of Ni (II) in 10 cm3 solutions was 19.89 µg, which, at the 95% confidence level, ranged from 19.72 to 20.19 g. Calculated values for Sandell's sensitivity and standard deviation were 0.0243 μgcm^{-2} and ±0.368, respectively.

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Table – III. Determination of Nickel in Alloy sample.

Percentage of Ni (II)	Proposed method	Dimethyl glyoxime method
Alloy Sample (Nickel- Aluminium based alloy) BAS 20 (Nickel-1.90%)	1.89	0.90*
Alloy Sample (Nickel- Aluminium based alloy) BAS 85 (Nickel -0.92%)	1.92	0.94

VII. CONCLUSIONS

According to the results of the aforementioned investigations, Schiff base [MThBAP] is a good sensitive reagent for the development of an extraction spectrophotometric approach that is quick and sensitive for the determination of Ni (II), and it has been successfully used for the analysis of Ni (II) in alloy samples.

VIII. ACKNOWLEDGEMENT

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