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Flow Injection Spectrophotometric Measurement of Ni (II) Nickel Using a Novel Reagent, MTMTCH, in an Analytical Sample

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P A P E R I N F O

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A B S T R A C T

This study describes flow-injection spectrophotometric methods for the determination of Ni (ll) Nickel in analytical sample by a new reagent MTDTCH.
An accurate and sensitive flow injection (FI) Spectrophotometric methods have been developed for the determination of ion Nickel (ll) in aqueous solution by preparing the new organic -Schiff base- reagent was prepared by reaction 3-methylthiophene-2-carbaldehyde with hydrazinecarbothiohydrazide. This reagent was worked as a ligand by reacting with the Nickel (II) ion to form a Nickel complex and determination trace amounts of it. The absorbance of the reaction was measured in the injection system which gave the wavelength of 357nm. With a limit of detection of 0.050 µg.mL^{-1} and a limit of quantitation of 0. 169 µg.mL^{-1} Relative standard deviation 0.495 and Recovery is 99.50, the metal concentration obeys Beer's law within the range0.5-9.mL^{-1} with a correlation coefficient value of 0.9997 . The complex composition was specific to the UV-visible spectra. the molar ratio of the metal to the reagent was (1:2).

1. INTRODUCTION

MTMTCH**(**N"-[(3-methylthiophen-2-yl) methyl-idene] thiocarbonohydrazide) is a Schiff bases are compounds containing an azomethine group, (-CH=N), can be formed by a condensation reaction of carbonyl compounds, (Aldehyde or ketone) with a primary amine in which the carbonyl group is replaced by a group (C = N-R)[1] ,[2] To measure Ni(ll) and other materials Such as: alloys [3], water [4] and soil [5] techniques have been devised [6]. They consist of the following: indirect flameless atomic absorption spectrophotometry [7]. Flow injection technique [8] Schiff's base creation, spectrophotometric [9]and colorimetric [10]In the field of pharmacological analysis, the flow injection technique has gained popularity [11]. Such methyldopa[11]. The present study describes new FIA-spectrophotometric methods [12]. To determination of Ni(ll) via reaction with (MTMTCH) forming yellow colored product that has absorption at λmax at 357 nm [2]. Definitions state that the flow-injection technique is a flow-through technology that applies particular thermodynamic conditions to the model region in a current stream. According to (Ruzicka and Hansen), the technique entails injecting a liquid into a suitable liquid stream that is continuously separated as the injected sample moves toward a detector that continuously records absorptivity or any other physical variable that results from the sample material passing through the transient flow cell. The injected sample forms the moving region of the model.

2. APPARATUS

Shimadzu 120 UV-VIS spectrophotometer equipped with a Cecil 10 uL flow cell was used. A Shimadzu 1650 PC UV-VIS double beam spectrophotometer was used for λmax determination**.** The carrier fluid was transported using a peristaltic pump (Gilsason minipuls (2)) fitted with flexible polyvinyl chloride tubes with an internal diameter of 0.8 mm.Using a tow-channel manifold (Figure 1), the FIA-spectrophotometer was used to determine Ni(ll). using FIA spectrophotometer. The Rheodyne -USA injection valve was utilized to administer standard solutions and samples at the proper injection volumes. The (MTMTCH) solution was transported via the Manifold's Channel A. While the alkaline oxalate solution stream was being transported via injection of ion Ni (II) via channel B, buffer solution (pH=6), which acted as an oxidizing agent**,** The reaction coil R.C as used to

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combine the product with a stream of buffer solution. The final product was mixed with the stream of alkaline oxalate at point (y), following the the mixing of coil RC.

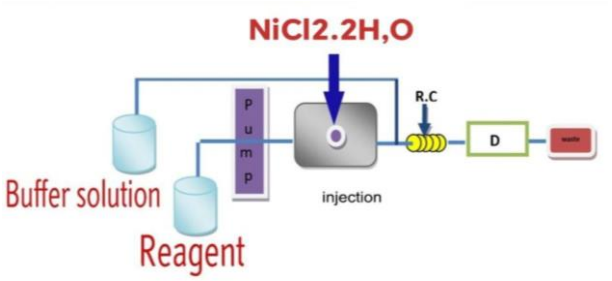


Figure 1. Manifold Used to Measure Ni (II) Using FIA Spectrophotometric Analysis

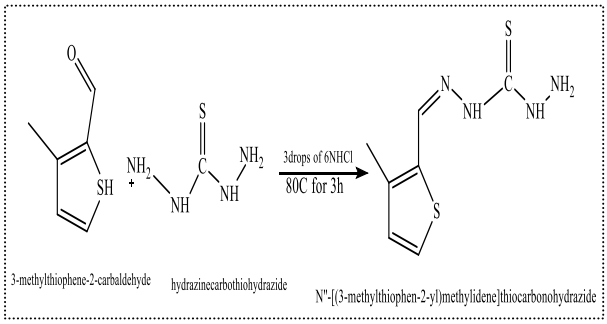
3. EXPERMINTAL

3.1 Materials

The B.D.H., Fluka, and Merck firms provided all of the high-purity solvents and chemical reagents utilized in this investigation; no further purification procedures were required.

3.1.1 Synthesis of the Ligand

Firstly, 1.06 g of thiocarbohydrazide (0.01mmoles) was dissolved in 58 mL of 95% EtOH and 20 mL of H2O. Next, 1.26g of 3-methylthiophene-2-carbaldehyde (0.01mmoles) was added, and last, three drops of 6N HCl were added. After three hours of stirring at 80 °C, the reaction mixture was allowed to crystallize at room temperature for twenty-four hours After filtering and a 1:1 EtOH/H2O wash, the solid was produced. and recrystallized from EtOH 95% to give N"-[(3-methylthiophen-2-yl)methylidene] thiocarbono-hydrazide in the form of a slightly ochre solid in 72.52% yield (0.7 1 g) [13] Scheme 1, and measure the degree of its fusion is.(233-235) °C, and Table 1 shows the chemical composition of ligand and some physical properties.



Scheme 1: Describes the steps for preparing the ligand (MTMTCH)

TABLE 1. The Physical Properties and Molecular Formulas of The Prepared Ligands

Color	Proportion of product	M.P (C^0)	M.W g / mol	M.F	Name
Yellow	72.52%	233-235	214.12	C_7S_2 N_4H_{10}	N"[(3methylthiophen2yl)methylidene] thiocarboenohydrazid

3.1.2 Preparation of Buffer Solutions and Ni (II) Nickel

The buffer solutions were prepared by dissolving 0.778 g of ammonium acetate at a concentration of 0.01M in 1000 mL of distilled water in a one-liter capacity tank.

This solution was used to create a number of solutions with a variety of acidic functions, ranging from (pH =3–10). by incorporating concentrated acetic acid or ammonia solution into the ammonium acetate that has been made[14]. and the Nickel (II) di Nickel compound (0.00134 g) was dissolved in distilled water to create the solution, and the volume was then increased to 100 mL using the same solvent. By gradually diluting this standard solution with distilled water, other standard solutions were created.

4. PRODUCT DESCRIPTION

UV-VIS was used to characterize the synthesized reagent and complex products that were recovered from the process, and FT-IR and $_1$HNMR spectra were obtained for the novel reagent.

5. METHODS

5.1 Procedure for the FIA Method

A 100.00µl sample is injected into a stream of 1×10^{-4} M MTMTCH reagent solution at a rate of 0.60 ml/min. The stream is allowed to combine with another stream of (pH=6) solution in a 50 cm reaction coil. Next, a valve was injected with salt ($NiCl_2.2H_2O$). Mixture is passed while sustaining the reaction, and absorbance is gauged at 375.nm [15].

6. RESULTS AND DISCUSSION

6.1. Study of Ultra Violet – Visble Spectra

A spectroscopic of the Nickel complex was carried out in the 190-1100nm range and in the ultraviolet–visible portion of the spectrum. At a concentration of 1×10^{-5}M, the electronic absorption spectra of the novel reagent and complex were examined in an absolute ethanol solution. Within the reagent spectrum, the electronic spectrum of the reagent showed three bands at (λ=280) nm, (λ=354) nm and

(λ=213) nm. In complex spectrum, the Ni(II) complex, showed three absorption bands at (λ=375) nm, (λ=276) nm, (λ=290) nm, (λ=250) nm, as show in Figure 2, 3.

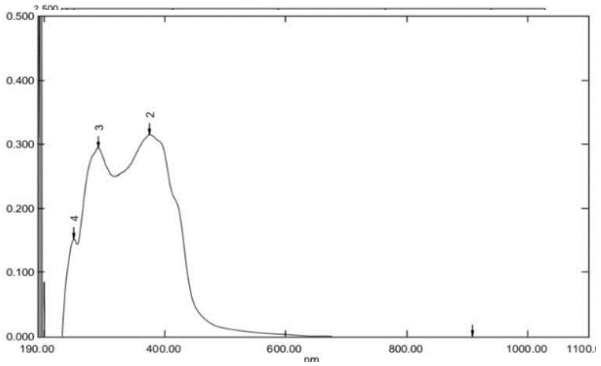


Figure 2. Absorption Spectrum of Ligand (MTMTCH)

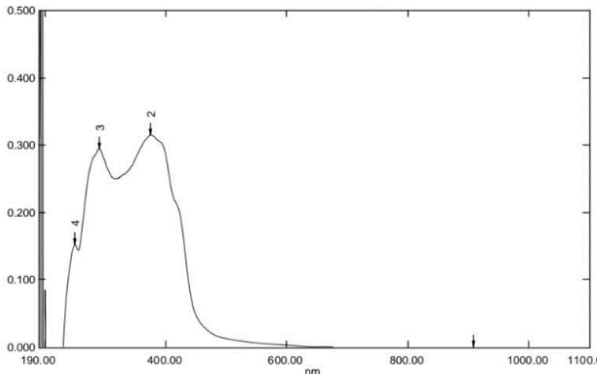


Figure 3. Absorption Spectrum of Nickel Complex (II) with Ligand MTMTCH

6.2. Study FT-IR Spectra for Reagent

The FT-IR spectrum of the prepared new reagent shows a peak belonging to (NH) at 3271 cm^{-1}, a peak at 1593 cm^{-1} belonging to (C=N), and the (C=C) peak at 1647 cm^{-1}. (NH_2)at 3151,and(C=s)1200-1100 cm^{-1}, a peak at700 cm^{-1} belonging to (C-S), as show in Figure 4.

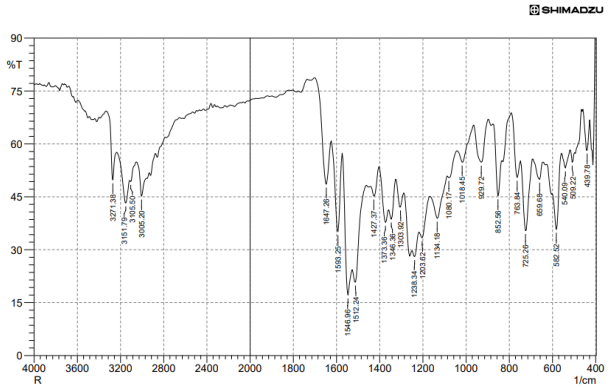


Figure 4. FT-IR Spectrum of Reagent

6.3. Proton NMR Spectrum for Reagent

From the signals appearing in the spectrum ^{1}H-NMR, the singlet signal at (δ=6.97 ppm,2H) refers to the proton of the NH_2 group, , the singlet signal at (δ=8.47 ppm,1H) refers to the proton of the (CH=N) group, the singlet signal at (δ=11.27 ppm,1H) refers to the proton of the NH group, , the signal at (2.32 ppm,3H) refers to the proton of the CH_3, the signal at 3.39 ppm indicates the presence of moisture H_2O and the signal at 2.5 ppm indicates the solvent DMSO, as show in Figure 5.

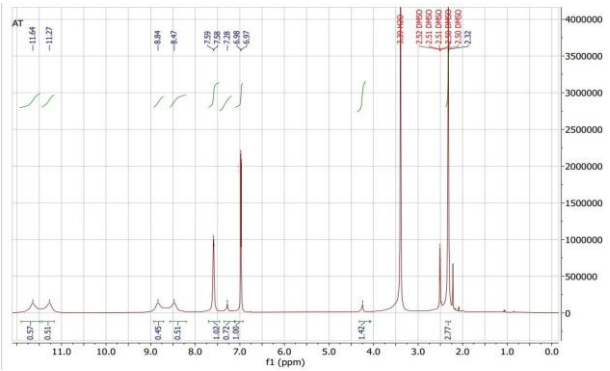


Figure 5. ^{1}H-NMR Spectrum for Reagent.

6.4. Study of the Variables of the Flow Injection System for the Determination of the Nickel (II) Ion.

The following system conditions have been investigated in relation to the effect of changing the reaction coil length range (25-125)cm: and the system conditions are: $NiCl_2.2H_2O$ concentration is 1×10^{-5} M and the concentration of MTMTCH reagent is 1×10^{-4}M, Figure 6 show the results obtained.. The regulator buffer solution is 6, the cell size is 10 µL loaded in the standard model loading link, the first and second carrier currents flow at 2.1 ml min-1, and the impact of changing total flow-rate within the range of (0.4-2.8) ml/min was investigated. concentration of $NiCl_2.2H2O$ (1×10^{-5}) M, while the reagent has a concentration of(1×10^{-4}) M. buffer solution has an (pH=6) and a cell size of 10 µL. model link, the maximum wavelength for Ni is 375 nm, and the flow velocities of the first and second load currents are 2.1 ml.min-1. Figure 7 shows the results obtained. A loop has been used in a pump with steel metal of various lengths and sizes (50 ml to 250 ml), as demonstrated by the results in Figure 8. The loop passes the current carrier detector and the buffer solution, which has a pH equivalent of (6) at a fixed concentration of $1 * 10^{-5}$, and absorbs readings for each length using a 10 ML spectrometer [16]

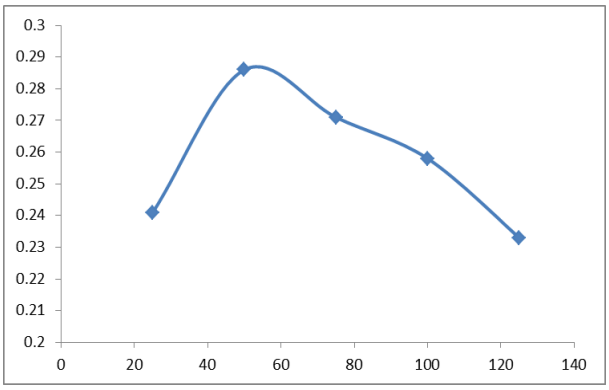


Figure 6. Effect of Reaction Coil Length.

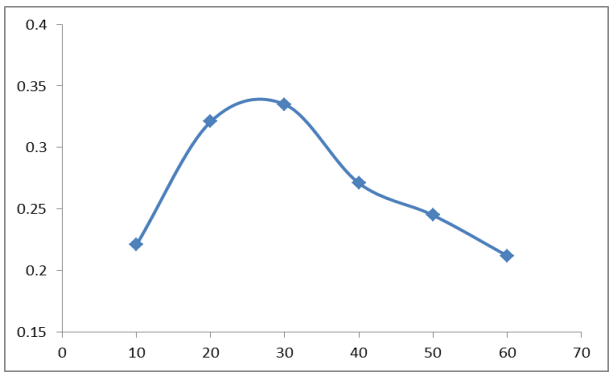


Figure 7. Effect of Total Flow-Rate.

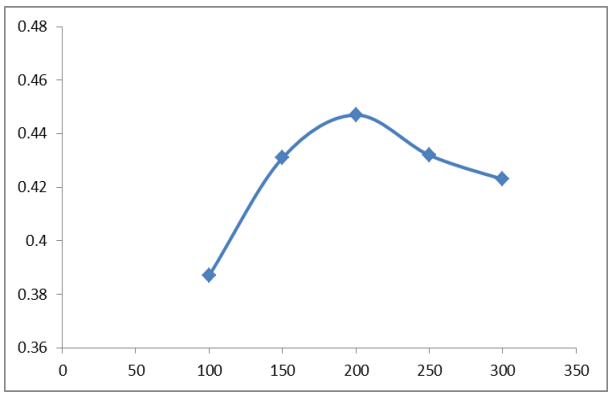


Figure 8. Effect of Loop Volume Injected.

By creating a calibration curve, the concentrations of the nickel complex that follow the Beer-Lambert law were found. Due to their departure from the Beer-Lambert law and the emergence of absorption peaks outside the measurement limits, many concentrations were eliminated. Consequently, (0.5–9) µg/mL are the concentrations that follow the Beer-Lambert law. Figure (9) shows the calibration curve for cobalt complex.

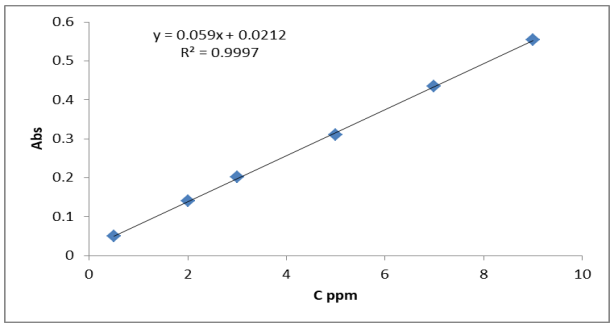


Figure 9. Calibration Curve for Ni (II) Complex

$$S.D = \frac{\sqrt{\sum(xi - x')^2}}{(N-1)}$$

$$R.S.D\% = \frac{(S.D)}{(x')} \times 100$$

$$L.O.Q = 10\ \frac{S.D}{Slope}$$

$$L.O.D = 3\ \frac{S.D}{Slop}$$

7. CONCLUSION

A spectrophotometer detector was employed in tandem with an accurate and sensitive F1A system during its design, construction, and operation. Flow-injection spectrophotometric methods were created for the determination of ion Ni(II) in a new simple reagent. This proposed approach can be carried out without the requirement for additional stages such as solvent extraction or pH control and other conditions.

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Arabic Abstract

توصف هذا الدراسة الطرق الطيفية للحقن الجرياني لتقدير النيكل الثنائي في عينة تحليلية بواسطة كاشف MTMTCH الجديد طريقة القياس الطيفية طريقة دقيقة وحساسة لحقن الجرياني التي تم تطويرها لتقدير النيكل ايون النيكل في محلول مائي من خلال تحضير كاشف جديد ناتج من تفاعل 3-ميثيلثوفين-2-كربلديهايد مع الهيدرازين كاربوتيو هيدرازيد. كان هذا الكاشف بمثابة ليكند من خلال تفاعله مع ايون النيكل لتكوين معقد النيكل وتحديد كميات قليلة منه. تم قياس امتصاص التفاعل في نظام الحقن الذي اعطى طول موجي ٤٠٠ نانومتر. مع حد كشف قدره ٠.٠٥٠ميكرو جرام مل-١ وحد كشف كمي ٠.٠١٦٩ميكروغرام.مل-١.ويخضع تركيز المعدن لقانون بير لامبرت ضمن النطاق ٠.٥-٩مل مع قيمة معامل ارتباط تبلغ. ٠.٩٩٩٧كان تركيب المعقد خاصا بالأطياف المرئية فوق البنفسجية. وكانت النسبة المولية المعدن للكاشف ١:٢).