— АНАЛИТИЧЕСКАЯ ХИМИЯ —

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Spectrophotometric determination of nickel (II) using 5-(2-bromo-5-methoxybenzylidene)-thiazolidine-2,4-dione

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Abstract. A fast and precise spectrophotometric approach was made to measure nickel (II) using the chromogenic compound 5-(2-bromo-5-methoxybenzylidene)-thiazolidine-2,4-dione (BMBT), which forms a yellow-brown complex when combined with other chemicals. It has been investigated whether bmbt can be used to measure Ni (II) through photometry. In the ph range of 7.1 to 8.9, the nickel (II) complex is roduced. A BMBT concentration of 510-4 mol/l is offered for the optimal complex formation and extraction. Water (as the base medium), methyl alcohol, ethyl alcohol, chloroform, hexane, dichloromethane, and acetonitrile were among the solvents investigated in the study. Methanol performed best among all, making it the most appropriate solvent for the experiment. At 482 nm, the complexation of nickel with bmbt reveals the strongest analytical signal. The bathochromic shift in this instance is 146 nm, which is the shift of the absorption peak to the long wavelength side. $1.75 \ 10^4$ is the molar absorption coefficient. Ni: BMBT = 1:2 is the component ratio in the complex. It is known that the complexing form of nickel, Ni²⁺, removes one hydrogen atom from a BMBT molecule. The complex's structure was investigated by empoying thermogravimetry and infrared spectroscopy. The research demonstrated that a number of metal ions, containing Fe(III), Cu(II), Mo(VI), Se(IV), Pt(II), Mn(II), Cd(II), Zn(II), Pb(II), and Pd(II), as well as form colored complexes when bmbt is used as a chromogenic reagent. The presence of masking agents or a change in the solution's ph considerably improves the accuracy of nickel measurement in the presence of these metal ions.at nickel concentrations of 0.3–13 g/ml, the fundamental rule of light absorption is observed. By utilizing masking agents, adjusting the medium's pH, or using extraction, the disruptive influence of irrelevant ions is removed. Methods for figuring out the amount of nickel in different things, like bottom deposits, wastewater, and steels, have been created based on the discoveries of a spectrophotometric analysis of the nickel (II) complex with BMBT.

Keywords: nickel (II), 5-(2-bromo-5-methoxybenzylidene)-thiazolidine-2,4-dione, spectrophotometry.

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Спектрофотометрическое определение никеля (II) с использованием 5-(2-бром-5-метоксибензилиден)-тиазолидин-2,4-диона

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Abstract. Разработан простой и селективный спектрофотометрический метод определения никеля (II) с использованием 5-(2-бром-5-метоксибензилиден)тиазолидин-2,4-диона (БМБТ) в качестве хромогенного реагента, образующего желто-коричневый комплекс. Изучена возможность использования БМБТ для фотометрического определения никеля (II). Комплекс никеля (II) образуется в интервале рн 7,1-8,9. Максимальный аналитический сигнал комплексообразования никеля с БМБТ наблюдается при 482 нм. Молярный коэффициент поглощения составляет 1,75·10⁴. Соотношение компонентов в комплексе Ni:БМБТ = 1:2. Соблюдение основного закона светопоглощения наблюдается при концентрации никеля 0,3–13 мкг/мл. Мешающее влияние посторонних ионов устранено с помощью маскирующих веществ, изменением pH среды или применением экстракции. По результатам спектрофотометрического исследования комплекса никеля (II) с БМБТ разработаны методы определения никеля в различных объектах.

Ключевые слова: спектрофотометрия, никель (II), 5-(2-бром-5-метоксибензилиден)-тиазолидин-2,4-дион

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Nickel involves in enzymatic activities and builds up in bird feathers and nickel makes it a necessary nutrient for both animals and humans. Nevertheless, having too much "nickel" in the body can trigger a number of medical problems, such as tachycardia, anemia, pulmonary and cerebral edema, and allergic reactions. Electroplating, nickel-cadmium batteries, ceramics, paint pigments, welding rods, ceramics, dental and surgical prostheses, magnetic tapes, computer components, and nickel catalysts are onlysome of the applications for "Ni".

Sewage which is containing nickel is toxic after adding the water and this fact defined the need of monitoring the quantity of nickel in samples of waste and natural water. Precise and fast measurement of nickel in natural and waste waters is made possible by spectrophotometric techniques and atomic absorption spectrometry by utilizing flame and graphite furnaces [2]. Yet, often, low analyte concentrations or matrix interferences prevent a direct determination from being used.

Various analytical techniques, such as flame atomic absorption spectrophotometry [3], graphite furnace atomic absorption spectrometry [4], electrothermal atomic absorption spectrometry [5], atomic fluorescence spectrometry [6], inductively coupled plasma-optical emission spectrometry [7], and spectrophotometry [8–12], have been utilized to define the concentration "nickel". Nonetheless, the procedures mentionedrequire high-purity solvents and some expensive equipments or ingredients for operation.

Due to their low cost of equipment, simpilicity, and ease of automation, spectrophotometry-based procedures are quite common. Apart from that, the in-tense absorption in the optical spectra, in the visible region, that is distinctive of the ligand itself is produced by nickel(II) chelates of various ligands, such as di-phenylcarbazone, 8-quinolinoldiphenylthiocarbazone, and its substituted equivalents [13–15].

Methods for determining the presence of nickel in distinct objects have been enhanced and it uses mixed - ligand complexes with oxythiophenols and dithiophenols in the presence of hydrophobic amines [16–19]. The use of 5-(2-bromo-5methoxybenzylidene)thiazolidine-2,4-dione (BMBT) for the photometric measurement of nickel(II) has been examined.

2. Experimental

2.1. Reagents and Solutions

dissolved We а precise quantity of (NH₄)₂Ni(SO₄)₂·6H₂O in water contained 2 ml of concentrated H₂SO₄, and we were able to make a standard solution of nickel(II) with a concentration of 1 mg/ml. After this, the solution was diluted and we continued until the time it had a final volume of 1 liter and the standard nickel (II) solution was diluted with redistilled deionized water right and after this process we use it to make working solutions. The ligand, BMBT, was yielded employing a certain method. Pure reagent was dissolved in 1 mol/l sodium hydroxide to produce a reagent solution containing a concentration of 0.002 mol/l and we used Britton-Robinson buffer in the pH range of 5-12 or 1 mol/l solutions of KOH and NaOH for obtaining the best pH conditions for experiments.

We used 1H NMR and IR spectra to confirm the structure of the ligand (BMBT) and the structure was verified. At 3364 cm⁻¹ (N-H), 2925 cm⁻¹ (Ar-H), 1674 cm⁻¹ (C=O), 1573 cm⁻¹ (C=N), 1449 cm⁻¹ (C=C), 1380 cm⁻¹ (C-N), and 733 cm⁻¹ (C-S-C), the IR spectrum indicated distinctive peaks at these locations. Peaks at 2.2 ppm (6H, s), 7.18 ppm (2H, s), 7.61 ppm (1H, s), 9.16 ppm (1H, s), and 12.45 ppm (1H, s) were observed in the 1H NMR spectrum in C_6D_6 solvent.

When we wanted to produce stock solutions, the essential metal salts were dissolved in the distilled water or diluted with the appropriate acids to a predetermined volume. We also produced 1% solutions of various ions by dissolving measured amounts of the appropriate salts in distilled water. Besides, we added acids to metal ion solutions to stop hydrolysis in some of the instances.

2.2. Instrumentation

Two spectrophotometers were used to measure the absorbance of the samples and these spectrophotometers are the SF-26 from the USSR and the KFK-2, also from the USSR, for the measurements, glass cells were used with optical path lengths of 10 mm or 5 mm. We used an I-120.2 potentiometer with a glass electrode to measure the pH of the aqueous solution. Thus a muffle furnace was used to dissolve the samples. In addition to this, 1H-NMR spectra were collected using a "Bruker" Fourier Transform apparatus which is running at 300,18 MHz in C6D6 solvent and 1H-NMR spectra were infrared (IR) spectra and were recorded using a German "Specord M 80" spectrophotometer.

2.3. General Procedure

2.3.1. General procedure for the determination of Ni(II)

A solution containing $5-100 \text{ mg of Ni}^{2+}$ was added to 2 mL of a 5-(2-bromo-5-methoxybenzylidene)-thiazolidine-2,4-dione reagent solution in a 10 mL volumetric flask. Methanol is used to change, the total volume to 10 mL. At 490 nm, the absorbance of the final mixture was measured with the reagent blank serving as a reference (path length: 0.5 cm). The absorbance readings were placed against various Ni²⁺ concentrations in the solutions to make calibration graphs.

2.3.2. Determination of nickel(II) in steel

Sulfuric acid (H_2SO_4) and a sample weighing 0.2 g were added to a 20 ml volume in a 1:1 ratio then this mixture was dissolved. To oxidize the solution were used after that a few drops of concentrated HNO₃ (nitric acid). Thus SO₃ (sulfur trioxide) was produced by evaporating the mixture twice. Then in 20 cc of heated 15% tartaric acid, the precipitate that acquired was dissolved. Water was added to make a volumetric flask's total volume 100 ml after cooling process and then the mixture was shaken. After this, the mixture was filtered. 5 ml of the filtered solution was moved to a separating funnel in order to verify nickel by employing the suggested methods.

2.3.3 Determination of Ni(II) in sewage water and bottom sediments

By evaporating 1 liter of waste water without boiling it a sample of wastewater was exposed to examination to produce a sloid residue. After being dissolved in 5 ml of pure nitric acid (HNO₃), this residue was then moved to a 50 ml flask. Ultimately, water was added to dilute the solution to the mark on the flask.

3. Results and Discussion

3.1. Selection of the reaction solvent

The stable complex that BMBT forms with Ni(II) in a distinct of aqueous and non-aqueous solvents has a reddish-yellow-brown color and both the BMBT reagent and the Ni-BMBT complex should be able to dissolve in the ideal solvent and enabling the measurement of discrete absorption spectra for the complexes without interference from the reagent. Water (as the base medium), hexane, methyl alcohol, chloroform, acetonitrile, ethyl alcohol and dichloromethane were among the solvents studied. Methanol performed best among all which made it the most suitable solvent for the experiment.

3.2.Effect of pH

The complex between Ni (II) and BMBT (an organic molecule) occurs in the pH range of 2.6 to 10, with the ideal pH for complex formation being between 7.1 and 8.9 (see Figure 1). The extraction of Ni(II) gradually declines with going down pH, primarily because of the drop in the concentration of the ionized form of BMBT, which is probably present in solution in an undissociated form. The complex is unable to form due to theNi ion's hydrolysis at a pH of 10.5. A BMBT concentration of $5 \cdot 10^{-4}$ mol/l is recommended for the optimal complex formation and extraction.



Fig.1. The dependence of the optical density of the complex on the pH of the aqueous phase. $C_{Ni(II)} = 3.44 \cdot 10^{-5} \text{ mol/l}; C_{BMBT} = 5.0 \cdot 10^{-4} \text{ mol/l};$ KFK-2, λ =490 nm l = 1 cm

Withour breaking down the Ni-BMBT complex may be kept unspoiled for two days and the Ni-BMBT complex is stable in both aqueous and organic solvents. Within five minutes of the compound forming, there is a maximum light absorption. Additionally, the compound keeps its stability at 80°C. 3.3.Absorption spectra of reagent solutions and metal complexes

Using a reference blank which included water, the reagent solution's absorbance was calculated. Basedon Figure 2, the greatest signal was observed during the complexation of nickel with BMBT turns out at a wavelength of 482 nm and from the Figure 2 the highest absorbance of BMBT is 336 nm. Bathochromic shift is the term for the shift in the absorption peak towards longer wavelengths. Thus in this instance, it equals to 146 nm. Besides, the substance's capacity to absorb light is found to be $1,75 \cdot 10^4$, as measured by the molar absorption coefficient.



Fig.2. Absorption spectra: $C_{Ni(II)}=3.44\cdot10^{-5} \text{ mol/l}; C_{BMBT} = 5.0\cdot10^{-4} \text{ mol/l};$ SF-26, l = 1 cm

3.4. Stoichiometry of Ni(II) – BMBT complexes

The molecular structure of the Ni(II) - BMBT complex was investigated by the researchers using a variety of techniques and these techniques involves the equilibrium shift, Starik Barbanel, and straight line approaches and based on all available methodologies, the complex's Ni:BMBT ratio is 1:2 (fig. 3). By using this information, they determined the complex's stability stable.

To manufacture and study the Ni(II) – BMBT complex in further detail, chemical analysis, IR

spectroscopy and the complex's IR spectra was compared with the reagent's. In the complex's IR spectra, prominent absorption bands were observed in the ranges of 1593–1448 cm⁻¹, which corresponds to the aromatic ring C = C, and 3050–3020 cm⁻¹, which is connected to v_{CH} in the aromatic core. Other absorption bands at particular wavenumbers were assigned to different molecular vibrations.



Fig. 3. Determination of the ratio of components by equilibrium shift method

According to a thermogravimetric investigation, the Ni-BMBT complex thermally decomposes in two steps. While one of the steps is dehydration and dehydration (90° to 115° C) is the first stage, which results in a loss of 4.93% of body weight, the elimination of BMBT causes the second stage, which takes place between 385 and 450 °C, to lose mass at the highest rate (41.78%). The end result of thermolysis is nickel (II) oxide.

It was found that one BMBT molecule displaces one hydrogen atom and that nickel complexes as Ni²⁺. The researchers suggested a potential structure for the Ni-BMBT complexes based on the ratio of components in the complexes, the quantity of protons being displaced, and the ionic form of nickel.



The research presentedthat utilizing the particular reagent, BMBT, allows the creation of colored complexes and ions of different metal elements, containing Fe(III), Cu(II), Mo(VI), Se(IV), Pt(II), Mn(II), Cd(II), Zn(II), Pb(II), and Pd(II). The accuracy of detecting these metal ions develops noticeably, if masking chemicals are present or the pH of the solution is altered.

Fluoride, chloride, bromide, oxalate, thiosulfate, sulfate, acetate, tartrate, and citrate ions do not prevent the process of determining the metal ions.

Nevertheless, even in modest concentrations, a number of particular ions, like thiourea phosphate, and thiocynate, might affect the result of the test. On top of that, due to the nickel(II)-EDTA complex's great stability Ni (II) can be completely hidden by EDTA.

3.5.Beer's law and sensitivity of Ni(II) – BMBT

To measure the "Ni" concentration, a calibration graph was used in an experiment and the ideal circumstances were used to manage the experiment. In the concentration range of 0.3–13 g/mL, the relationship between the concentration of nickel (Ni(II)) and the absorbance at 482 nm (A₄₈₂) followed Beer's law. It was discovered that A₄₈₂ = 0.045 + 0.071x, where 'x' stands for the concentration of nickel, is the calibration equation. The calibration curve was created by using data resulted utilizing the least squares method [26].

3.6.Analytical applications:

We determined the limits of photometric detection and quantitative nickel determination by employing calibration graphs. The primary spectroscopic features of our technique for Ni(II) detection using BMBT are shown in Table 1.

In Table 2, we contrasted our photometric nickel(II) approaches' analytical attributes with those of a few other methods that are already in use [3, 9, 18]. The discoveries presents the extreme selectivity, sensitivity, and impact of our technologies, which shows their capacity to reliably detect even minute levels of nickel ions. As our suggested strategy is straightforward, quick, sensitive, and selective, it is superior to existing approaches.

Table 1.

Formation conditions and some chemical and analytical

properties of Ni(II) with BMBT

Parameter	Value	
Color	yellow brown	
pH _{Op}	7.1-8.9	
λ_{\max} (nm)	482	
Bathochromic shift	146	
Molar absorptivity (L· mol ⁻¹ cm ⁻¹)	$1.65 \cdot 10^4$	
Sandell's sensitivity $(ng \cdot cm^{-2})$	3.51	
The equation of calibration curves	0.045+0.071x	
Correlation coefficient	0.9954	
Stability constant (β)	10.61	
Beer's law range ($\mu g \cdot m l^{-1}$)	0.3-13	
Limit of detection (LOD): $ng \cdot mL^{-1}$	12	
Limit of quantification (LOQ):	39	
ng∙mL ⁻¹		

Table 2.

Reagent	рН	λ, nm	ε·10 ⁻⁴	Beer's law range
Dimethylglyoxime [27]	12	470		0.26-2.1
N-ethyl-3-carbazolecarboxaldehyde-3- thiosemicarbazone [28]	6.0	400	1.114	-
7-Methl-2-chloroquinoline-3-carbalde- hyde thiosemicarbazone [29]	6.0	410	1.67	-
Thiazole-2-carbaldehyde 2-quinolylhydrazone [30]	8.7–9.5	522	7.17	0-0.7
Pyridoxal-4-phenyl-3-thiosemicarbazone[31]	4–6	430	1.92	0.5-5
4-hydroksibenzaldehid-4-bromphenilhydrazin [32]	4.0	497	12.85	0.01-0.1
BMBT	7.1-8.9	482	1.75	0.3-13

Comparative characteristics of the procedures for determining nickel

With BMBT's spectroscopic research of the nickel(II) complex as a foundation, to determine nickel in a variety of samples, including bottom deposits, wastewater, and steels, we formed extrac-

tion spectrophotometric methods. Tables 3 and 4 describe how our techniques were successfully used to investigate real samples.

Table 3.

Reagent	Average result (%)	Probable relative error (ε)	Relative standard deviation (S _r)	Confidence interval (µ)
Dimethylglyoxime	0.255	0.0065	0.024	0.2550 ± 0.0065
Ni - BMBT	0.257	0.0114	0.042	0.2570±0.0114

Determination of nickel in steel $8X\Phi$ (C16⁶) (n=5, P=0.95)

4. Conclusions

The results describes that the newly created approach, which makes use of the reagent 5-(2-bromo-5-methoxybenzylidene)-thiazolidine-2,4-

dione (BMBT), is very effective for detecting Ni(II) in water-based solutions quantitatively. The interaction between nickel(II) and BMBT was investigated by using spectrophotometry.

Table 4.

Analysis object	Added, μg	Found (along with supplement), μg	Relative standard deviation (S _r)	Found in the sample, μg / kg			
Sewage water							
Sample 1	2.0	2.55	0.039	1.55 ± 0.06			
Sample 2	5.0	6.64	0.032	$1.64{\pm}0.12$			
Bottom sediments							
Sample 1	2.0	3.46	0.07	1.46 ± 0.07			
Sample 2	5.0	6.25	0.05	1.25±0.14			

Determination results of nickel (II) in the sewage water and bottom sediments (n = 6, P = 0.95)

The pH range of 7.1 to 8.9 is where the nickel(II) complex with BMBT forms with the greatest intensity. Besides, this process stands out for being quick and requiring less organic solvent and the component ratios in the complexes as well as the ideal complex formation conditions have been determined.It was possible to measure trace levels of nickel with accuracy in the concentration range of 0.3 to 13 g/ml because to the Beer's law. The suggested method is straightforward, quick, and extremely sensitive. Consequently, it may be used to identify nickel in both steel samples and bottom sediments.

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